In the Matter of

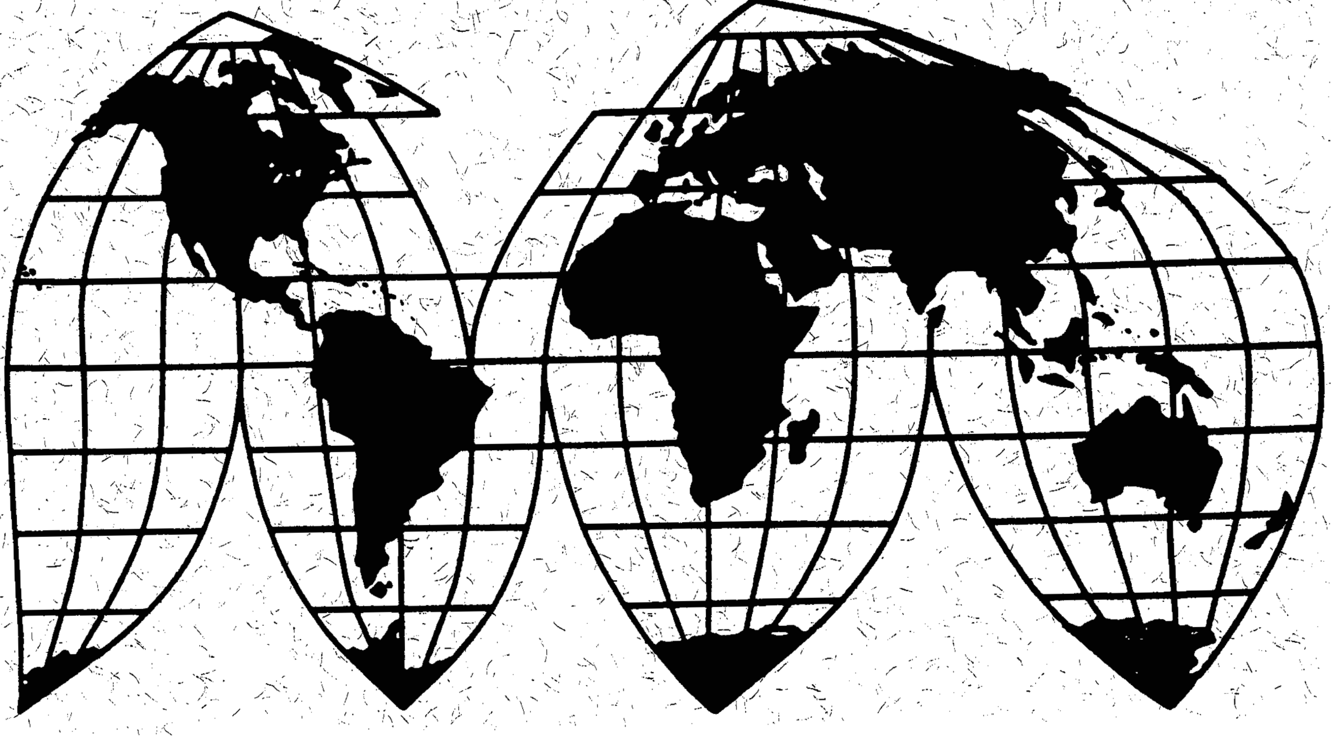
Certain Curable Fluoroelastomer Compositions and Precursors Thereof

Investigation No. 337-TA-364

Publication 2890

May 1995

U.S. International Trade Commission



Washington, DC 20436

U.S. International Trade Commission

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UNITED STATES INTERNATIONAL TRADE COMMISSION
Washington, DC 20436

In the Matter of

CERTAIN CURABLE
FLUOROELASTOMER
COMPOSITIONS AND
PRECURSORS THEREOF

Investigation No. 337-TA-364

95 FEB -1 P4:59

NOTICE OF DECISION NOT TO REVIEW INITIAL DETERMINATION FINDING A VIOLATION OF SECTION 337 AND SCHEDULE FOR THE FILING OF WRITTEN SUBMISSIONS ON REMEDY, THE PUBLIC INTEREST, AND BONDINGS

AGENCY: U.S. International Trade Commission.

ACTION: Notice.

SUMMARY: Notice is hereby given that the U.S. International Trade Commission has determined not to review the initial determination (ID) issued on December 15, 1994, by the presiding administrative law judge (ALJ) in the above-captioned investigation finding a violation of section 337 in the importation into the United States and the sale within the United States after importation of certain curable fluoroelastomer compositions and precursors thereof.

FOR FURTHER INFORMATION CONTACT: Mark D. Kelly, Esq., Office of the General Counsel, U.S. International Trade Commission, telephone 202-205-3106. Copies of the nonconfidential version of the ID and all other nonconfidential documents filed in connection with this investigation are or will be available for inspection during official business hours (8:45 a.m. to 5:15 p.m.) in the Office of the Secretary, U.S. International Trade Commission, 500 E Street S.W., Washington, D.C. 20436, telephone 202-205-2000. Hearing-impaired persons are advised that information on the matter can be obtained by contacting the Commission's TDD terminal on 202-205-1810.

SUPPLEMENTARY INFORMATION: On March 16, 1994, the Commission instituted an investigation of a complaint filed by Minnesota Mining and Manufacturing Company ("3M") under section 337 of the Tariff Act of 1930. The complaint alleged that Ausimont, S.p.A., of Milan, Italy, and Ausimont U.S.A., Inc., of Morristown, NJ, imported, sold for importation, or sold in the United States after importation certain curable fluoroelastomer compositions and precursors thereof that infringed certain claims of U.S. Letters Patent 4,287,320 ("the '320 patent"). The Commission's notice of investigation named as respondents Ausimont Italy and Ausimont U.S.A., each of which was alleged to have committed one or more unfair acts in the importation or sale of curable fluoroelastomer compositions and precursors thereof that infringe claims of the asserted patent.

The ALJ conducted an evidentiary hearing commencing on September 23, 1994, and issued his final ID on December 15, 1994. He found that: (1) the '320 patent is not invalid; (2) respondents' imported products infringe the claims in issue of the '320 patent; and (3) complainant 3M satisfied the economic requirements for existence of a domestic industry. Based upon his findings of validity, infringement, and domestic industry, the ALJ concluded that there was a violation of section 337.

Respondents filed a petition for review of the ALJ's findings on the questions of validity of the '320 patent and infringement. Complainant and the Commission investigative attorneys filed responses to the petition for review. No other petitions for review of the ID or government comments were received by the Commission.

In connection with final disposition of this investigation, the Commission may issue (1) an order that could result in the exclusion of the subject articles from entry into the United States, and/or (2) cease and desist orders that could result in respondents being required to cease and desist from engaging in unfair acts in the importation and sale of such articles. Accordingly, the Commission is interested in receiving written submissions that address the form of remedy, if any, that should be ordered.

If the Commission contemplates some form of remedy, it must consider the effects of that remedy upon the public interest. The factors the Commission will consider include the effect that an exclusion order and/or cease and desist orders would have on (1) the public health and welfare, (2) competitive conditions in the U.S. economy, (3) U.S. production of articles that are like or directly competitive with those that are subject to investigation, and (4) U.S. consumers. The Commission is therefore interested in receiving written submissions that address the aforementioned public interest factors in the context of this investigation.

If the Commission orders some form of remedy, the President has 60 days to approve or disapprove the Commission's action. During this period, the subject articles would be entitled to enter the United States under a bond, in an amount determined by the Commission and prescribed by the Secretary of the Treasury. The Commission is therefore interested in receiving submissions concerning the amount of the bond that should be imposed, if remedial orders are issued.

WRITTEN SUBMISSIONS: The parties to the investigation, interested government agencies, and any other interested persons are encouraged to file written submissions on the issues of remedy, the public interest, and bonding. Complainant and the Commission investigative attorneys are also requested to submit proposed remedial orders for the Commission's consideration. The written submissions and proposed remedial orders must be filed no later than the close of business on February 13, 1995. Reply submissions must be filed no later than the close of business on February 21, 1995. No further submissions will be permitted unless otherwise ordered by the Commission.

Persons filing written submissions must file the original document and 14 true copies thereof with the Office of the Secretary on or before the deadlines stated above. Any person desiring to submit a document (or portion thereof) to the Commission in confidence must request confidential treatment unless the information has already been granted such treatment during the proceedings. All such requests should be directed to the Secretary of the Commission and must include a full statement of the reasons why the Commission should grant such treatment. See 19 C.F.R. § 201.6. Documents for which confidential treatment is granted by the Commission will be treated accordingly. All nonconfidential written submissions will be available for public inspection at the Office of the Secretary.

This action is taken under the authority of section 337 of the Tariff Act of 1930, as amended (19 U.S.C. § 1337), and sections 210.53 and 210.58 of the Commission's Interim Rules of Practice and Procedure (19 C.F.R. §§ 210.53 and 210.58).

By order of the Commission.

Donna R. Koehnke

Donna R. Koehnke
Secretary

Issued: February 2, 1995

UNITED STATES INTERNATIONAL TRADE COMMISSION
Washington, DC 20436

In the Matter of

CERTAIN CURABLE
FLUOROELASTOMER
COMPOSITIONS AND
PRECURSORS THEREOF

Investigation No. 337-TA-364

'95 MAR 16 A8:56
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NOTICE OF ISSUANCE OF LIMITED EXCLUSION ORDER AND CEASE AND DESIST ORDER

AGENCY: U.S. International Trade Commission.

ACTION: Notice.

SUMMARY: Notice is hereby given that the U.S. International Trade Commission has issued a limited exclusion order and a cease and desist order in the above-captioned investigation.

FOR FURTHER INFORMATION CONTACT: Mark D. Kelly, Esq., Office of the General Counsel, U.S. International Trade Commission, 500 E Street, S.W., Washington, D.C. 20436, telephone 202-205-3106.

SUPPLEMENTARY INFORMATION: The authority for the Commission's determinations is contained in section 337 of the Tariff Act of 1930, as amended (19 U.S.C. § 1337), and in section 210.58 of the Commission's Interim Rules of Practice and Procedure (19 C.F.R. § 210.58).

The Commission instituted this investigation on March 16, 1994, based upon a complaint filed by Minnesota Mining and Manufacturing Company ("3M") alleging that Ausimont, S.p.A., of Milan, Italy, and Ausimont U.S.A., Inc., of Morristown, NJ (collectively referred to as "respondents" or "Ausimont") had violated section 337 in the sale for importation, the importation, and the sale within the United States after importation of certain curable fluoroelastomer compositions and precursors thereof, by reason of infringement of one or more claims of U.S. Letters Patent 4,287,320 ("the '320 patent") assigned to 3M. 59 Fed. Reg. 12344 (March 16, 1994).

On December 15, 1994, the presiding administrative law judge (ALJ) issued his final initial determination (ID) finding that respondents had violated section 337, based on his findings that (1) the claims in issue of the '320 patent are not invalid; (2) the accused products imported by respondents infringe the claims in issue of the '320 patent under the doctrine of equivalents; and (3) a domestic industry exists. On February 2, 1995, the Commission determined not to review the ALJ's final ID and requested written submissions on the issues of remedy, the public interest, and bonding. 60 Fed. Reg. 7581 (February 8, 1995).

Submissions on remedy, the public interest, and bonding were received from complainant 3M, respondents, and the Commission investigative attorney (IA). Complainant, respondents, and the IA also filed reply submissions on these issues.

Having reviewed the record in this investigation, including the written submissions of the parties, the Commission made its determinations on the issues of remedy, the public interest, and bonding. The Commission determined that the appropriate form of relief is a limited exclusion order prohibiting the unlicensed importation of infringing fluoroelastomer compositions or precursors thereof manufactured and/or imported by or on behalf of Ausimont, S.p.A. of Milan, Italy or Ausimont U.S.A, Inc., of Morristown, New Jersey. In addition, the Commission issued a cease and desist order directed to the domestic respondent, Ausimont U.S.A, ordering it to cease and desist from the following activities in the United States: importing, selling, marketing, distributing, offering for sale, or otherwise transferring (except for exportation) in the United States infringing imported curable fluoroelastomer compositions or precursors thereof. The orders apply to any of the affiliated companies, parents, subsidiaries, licensees, contractors, or other related business entities, or their successors or assigns, of the above-named companies.

The Commission also determined that the public interest factors enumerated in 19 U.S.C. § 1337(d) and (f) do not preclude the issuance of the limited exclusion and cease and desist orders, and that the bond during the Presidential review period shall be in the amount of 48 percent of the entered value of the articles in question.

Copies of the Commission orders, the Commission opinion in support thereof, and all other nonconfidential documents filed in connection with this investigation are or will be available for inspection during official business hours (8:45 a.m. to 5:15 p.m.) in the Office of the Secretary, U.S. International Trade Commission, 500 E Street, S.W., Washington, D.C. 20436, telephone 202-205-2000. Hearing-impaired persons are advised that information on this matter can be obtained by contacting the Commission's TDD terminal on 202-205-1810.

By order of the Commission.

Donna R. Koehnke

Donna R. Koehnke
Secretary

Issued: **March 16, 1995**

UNITED STATES INTERNATIONAL TRADE COMMISSION
Washington, DC 20436

In the Matter of CERTAIN CURABLE FLUOROELASTOMER COMPOSITIONS AND PRECURSORS THEREOF	Investigation No. 337-TA-364

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ORDER

The Commission instituted this investigation on March 16, 1994, based upon a complaint filed by Minnesota Mining and Manufacturing Company ("3M") alleging that Ausimont, S.p.A., of Milan, Italy, and Ausimont U.S.A., Inc., of Morristown, NJ (collectively referred to as "respondents" or "Ausimont") had violated section 337 in the sale for importation, the importation, and the sale after importation of certain curable fluoroelastomer compositions and precursors thereof, by reason of infringement of one or more claims of U.S. Letters Patent 4,287,320 ("the '320 patent") assigned to 3M. 59 Fed. Reg. 12344 (March 16, 1994).

On December 15, 1994, the presiding administrative law judge (ALJ) issued his final initial determination (ID) finding that respondents had violated section 337, based on his findings that (1) the claims in issue of the '320 patent are not invalid; (2) the accused products imported by respondents infringe the claims in issue of the '320 patent under the doctrine of equivalents; and (3) a domestic industry exists. On February 2, 1995, the Commission determined not to review the ALJ's final ID and requested written submissions on the issues of remedy, the public interest, and bonding. 60 Fed. Reg. 7581 (February 8, 1995).

Submissions on remedy, the public interest, and bonding were received from complainant 3M, respondents, and the Commission investigative attorney (IA). Complainant, respondents, and the IA also filed reply submissions on these issues.

Having reviewed the record in this investigation, including the written submissions of the parties, the Commission has made its determinations on the issues of remedy, the public interest, and bonding. The Commission has determined that the appropriate form of relief is a limited exclusion order prohibiting the unlicensed importation of infringing fluoroelastomer compositions or precursors thereof manufactured and/or imported by or on behalf of Ausimont, S.p.A. of Milan, Italy or Ausimont U.S.A, Inc. of Morristown, New Jersey. In addition, the Commission has issued a cease and desist order directed to the domestic respondent, Ausimont U.S.A, ordering it to cease and desist from the following activities in the United States: importing, selling, marketing, distributing, offering for sale, or otherwise transferring (except for exportation) in the United States infringing imported curable fluoroelastomer compositions or precursors thereof. The orders apply to any of the affiliated companies, parents, subsidiaries, licensees, contractors, or other related business entities, or their successors or assigns, of the above-named companies.

The Commission has also determined that the public interest factors enumerated in 19 U.S.C. § 1337(d) and (f) do not preclude the issuance of the limited exclusion order and the cease and desist order, and that the bond during the Presidential review period shall be in the amount of 48 percent of the entered value of the articles in question.

Accordingly, the Commission hereby **ORDERS THAT** --

1. Curable fluoroelastomer compositions or precursors thereof, covered by claims 1, 2, 4, 5, 6, 11, 12, 14, or 15 of U.S. Letters Patent 4,287,320, and manufactured and/or imported by or on behalf of Ausimont, S.p.A. of Milan, Italy, or Ausimont U.S.A., Inc. of Morristown, New Jersey, or any of their affiliated companies, parents, subsidiaries, licensees, contractors, or other related entities, or their successors or assigns, are excluded from entry for consumption into the United States for the remaining term of the patent, i.e., until September 1, 1998, except under license of the patent owner or as provided by law.

2. Curable fluoroelastomer compositions or precursors thereof, manufactured and/or imported by or on behalf of Ausimont, S.p.A. or Ausimont U.S.A., Inc., identified in paragraph 1 above, are entitled to entry into the United States under bond in the amount of forty-eight (48) percent of the entered value of such items pursuant to subsection (j) of section 337 of the Tariff Act of 1930, as amended (19 U.S.C. 1337(j)), from the day after this Order is received by the President, until such time as the President notifies the Commission that he approves or disapproves this action, but no later than 60 days after the date of receipt of this Order by the President.

3. Pursuant to procedures to be specified by the U.S. Customs Service, as the Customs Service deems necessary, persons seeking to import curable fluoroelastomer compositions or precursors thereof, manufactured and/or imported by or on behalf of Ausimont S.p.A. or Ausimont U.S.A., Inc., shall certify that they are familiar with the terms of this Order, that they have made appropriate inquiry, and thereupon state that, to the best of their knowledge and belief, the curable fluoroelastomer compositions or precursors thereof being imported are not excluded from entry under paragraph 1 of this Order.

4. In accordance with 19 U.S.C. § 1337(l), the provisions of this Order shall not apply to curable fluoroelastomer compositions or precursors thereof imported by and for the use of the United States, or imported for, and to be used for, the United States with the authorization or consent of the Government.

5. The Commission may modify this Order in accordance with the procedure described in section 211.57 of the Commission's Interim Rules of Practice and Procedure (19 C.F.R. § 211.57).

6. The Secretary shall serve copies of this Order upon each party of record in this investigation and upon the Department of Health and Human Services, the Department of Justice, the Federal Trade Commission, and the U.S. Customs Service.

7. Notice of this Order shall be published in the Federal Register.

By order of the Commission.

Donna R. Koehnke

Donna R. Koehnke
Secretary

Issued: **March 16, 1995**

UNITED STATES INTERNATIONAL TRADE COMMISSION
Washington, DC 20436

In the Matter of

CERTAIN CURABLE
FLUOROELASTOMER
COMPOSITIONS AND
PRECURSORS THEREOF

Investigation No. 337-TA-364

ORDER TO CEASE AND DESIST

IT IS HEREBY ORDERED THAT Ausimont U.S.A., Inc., 44 Whippany Road, Morristown, New Jersey 07962, cease and desist from importing, selling, marketing, distributing, offering for sale, or otherwise transferring (except for exportation) in the United States certain curable fluoroelastomer compositions or precursors thereof, covered by claims 1, 2, 4, 5, 6, 11, 12, 14, or 15 of U.S. Letters Patent 4,287,320, in violation of section 337 of the Tariff Act of 1930, as amended, 19 U.S.C. § 1337.

I.

(Definitions)

As used in this Order:

(A) "Commission" shall mean the United States International Trade Commission.

(B) "Complainant" shall mean Minnesota Mining and Manufacturing Company, 3M Center, St. Paul, Minnesota 55133.

(C) "Respondent" shall mean Ausimont U.S.A., Inc., 44 Whippany Road, Morristown, New Jersey 07962.

(D) "Person" shall mean an individual, or non-governmental partnership, firm, association, corporation, or other legal or business entity other than the above Respondent or its majority owned and/or controlled subsidiaries, their successors, or assigns.

(E) "United States" shall mean the fifty States, the District of Columbia, and Puerto Rico.

(F) "Covered product" shall mean curable fluoroelastomer compositions or precursors thereof covered by claims 1, 2, 4, 5, 6, 11, 12, 14, or 15 of U.S. Letters Patent 4,287,320.

(G) The terms "import" and "importation" refer to importation for entry for consumption under the Customs laws of the United States.

II.

(Applicability)

The provisions of this Cease and Desist Order shall apply to Respondent and to its principals, stockholders, officers, directors, employees, agents, licensees, distributors, controlled (whether by stock ownership or otherwise) and/or majority owned business entities, successors, and assigns, and to each of them, in accordance with Section VII hereof.

III.

(Conduct Prohibited)

The following conduct of Respondent in the United States is prohibited by this Order. Respondent shall not:

(A) import into the United States curable fluoroelastomer compositions or precursors thereof covered by claims 1, 2, 4, 5, 6, 11, 12, 14, or 15 of U.S. Letters Patent 4,287,320 for the remaining term of the patent, or

(B) sell, market, distribute, offer for sale, or otherwise transfer (except for exportation) in the United States imported curable fluoroelastomer compositions or precursors thereof covered by claims 1, 2, 4, 5, 6, 11, 12, 14, or 15 of U.S. Letters Patent 4,287,320 for the remaining term of the patent.

IV.

(Conduct Permitted)

Notwithstanding any other provision of this Order, specific conduct otherwise prohibited by the terms of this Order shall be permitted if, in a written instrument, the owner of the U.S. Letters Patent 4,287,320 licenses or authorizes such specific conduct, or such specific conduct is related to the importation or sale of covered products by or for the United States.

V.

(Reporting)

For purposes of this reporting requirement, quarterly reporting periods shall commence on March 1, June 1, September 1, and December 1 of each year, and shall end on the subsequent May 31, August 31, November 30, and the last day of February, respectively. However, the first report required under this section shall cover the period March 17, 1995, through May 31, 1995. The reporting requirement shall continue in force until the expiration of U.S. Letters Patent 4,287,320, on September 1, 1998, unless, pursuant to subsection (j)(3) of section 337 of the Tariff Act of 1930, the President notifies the Commission within 60 days after the date he receives this Order, that he disapproves this Order; provided, however, that Respondent's reporting requirement hereunder shall cease if, in a timely filed report, Respondent shall report no sales of imported covered product during two successive quarterly reporting periods and no remaining inventory of imported covered product.

Within thirty (30) days of the last day of each reporting period, Respondent shall report to the Commission the quantity in pounds and the value in dollars of foreign-made covered product that Respondent has imported or sold in the United States during the reporting period and/or that remains in inventory at the end of the reporting period.

Any failure to report shall constitute a violation of this Order.

VI.

(Recordkeeping and Inspection)

(A) For the purpose of securing compliance with this Order, Respondent shall retain any and all records relating to the importation, sale, marketing, distribution, offering for sale, or otherwise transferring in the United States of imported covered products, made and received in the usual and ordinary course of business, whether in detail or in summary form, for a period of two (2) years from the close of the fiscal year to which they pertain.

(B) For the purposes of determining or securing compliance with this Order and for no other purpose, and subject to any privilege recognized by the Federal Courts of the United States, duly authorized representatives of the Commission, upon reasonable written notice by the Commission or its staff, shall be permitted access and the right to inspect and copy in the principal offices of Respondent during office hours, and in the presence of counsel or other representatives if Respondent so chooses, all books, ledgers, accounts, correspondence, memoranda, financial reports, and other records and documents, both in detail and in summary form, for the purpose of verifying any matter or statement contained in the reports required to be retained under subparagraph VI(A) of this Order.

VII.

(Service of Cease and Desist Order)

Respondent is ordered and directed to:

(A) Serve, within fifteen (15) days after the effective date of this Order, a copy of this Order upon each of its officers, directors, managing agents, agents, and employees who have any responsibility for the importation, sale, marketing, or distribution of imported covered products in the United States;

(B) Serve, within fifteen (15) days after the succession of any persons referred to in subparagraph VII(A) of this Order, a copy of the Order upon each successor; and

(C) Maintain such records as will show the name, title, and address of each person upon whom the Order has been served, as described in subparagraphs VII(A) and VII(B) of this Order, together with the date on which service was made.

The obligations set forth in subparagraphs VII(B) and VII(C) of this Order shall remain in effect until the date of expiration of U.S. Letters Patent 4,287,320.

VIII.

(Confidentiality)

Information obtained by means provided for in Sections V and VI of this Order will be made available only to the Commission and its authorized representatives, will be entitled to confidential treatment, and will not be divulged by any authorized representative of the Commission to any person other than duly authorized representatives of the Commission, except as may be required in the course of securing compliance with this Order, or as otherwise required by law. Disclosure hereunder will not be made by the Commission without ten (10) days prior notice in writing to Respondent.

IX.

(Enforcement)

Violation of this Order may result in any of the actions specified in section 211.56 of the Commission's Interim Rules of Practice and Procedure, 19 C.F.R. § 211.56 (1994), including an action for civil penalties in accordance with section 337(f) of the Tariff Act of 1930, 19 U.S.C. § 1337(f), and any other action as the Commission may deem appropriate. In determining whether Respondent is in violation of this Order, the Commission may infer facts adverse to Respondent if Respondent fails to provide adequate or timely information.

X.

(Modification)

The Commission may amend this Order on its own motion or in accordance with the procedure described in section 211.57 of the Commission's Interim Rules of Practice and Procedure, 19 C.F.R. § 211.57 (1994).

XI.

(Bonding)

The conduct prohibited by Section III of this Order may be continued during the period which this Order is under review by the President pursuant to section 337(j) of the Tariff Act of 1930 (19 U.S.C. § 1337(j)), subject to Respondent posting of bond in the amount of forty-eight (48) percent of the entered value of the imported covered product. This bond provision does not apply to conduct that is otherwise permitted by Section IV of this Order. Covered products imported on or after March 16, 1995, are subject to the entry bond as set forth in the limited exclusion order issued by the Commission on March 16, 1995, and are not subject to this bond provision.

This bond is to be posted in accordance with the procedures established by the Commission for the posting of bonds by complainants in connection with the issuance of temporary exclusion orders. See Commission Interim Rule 210.58, 19 C.F.R. § 210.58. The bond and any accompanying documentation is to be provided to and approved by the Commission prior to the commencement of conduct which is otherwise prohibited by Section III of this Order.

The bond is to be forfeited in the event that the President approves, or does not disapprove within the Presidential review period, the Commission's Orders of March 16, 1995, or any subsequent final order issued after the completion of Investigation No. 337-TA-364, unless the U.S. Court of Appeals for the Federal Circuit, in a final judgment, reverses any Commission final determination and order as to Respondent on appeal, or unless the products subject to this bond are exported or destroyed by Respondent, and Respondent provides certification to that effect satisfactory to the Commission.

The bond is to be released in the event the President disapproves this Order and no subsequent order is issued by the Commission and approved, or not disapproved, by the President, upon service on Respondent of an Order issued by the Commission based upon application therefor made to the Commission.

By order of the Commission.

Donna R. Koehnke

Donna R. Koehnke
Secretary

Issued: **March 16, 1995**

UNITED STATES INTERNATIONAL TRADE COMMISSION
Washington, DC 20436

In the Matter of

CERTAIN CURABLE
FLUOROELASTOMER
COMPOSITIONS AND
PRECURSORS THEREOF

Investigation No. 337-TA-364

COMMISSION OPINION

I. BACKGROUND

The Commission instituted this investigation on March 16, 1994, based upon a complaint filed by Minnesota Mining and Manufacturing Company ("3M") alleging that Ausimont, S.p.A., of Milan, Italy, and Ausimont U.S.A., Inc., of Morristown, NJ (collectively referred to as "respondents" or "Ausimont") had violated section 337 of the Tariff Act of 1930 (19 U.S.C. § 1337) in the sale for importation, the importation, and the sale after importation of certain curable fluoroelastomer compositions and precursors thereof, by reason of infringement of one or more claims of U.S. Letters Patent 4,287,320 ("the '320 patent") assigned to 3M.[1]

Fluoroelastomer compositions are rubber-like polymers that have wide application in the automotive and aerospace industries as a material from which component parts such as O-rings, gaskets, seals, hoses, and the like are made. Complainant 3M's '320 patent claims a mixture of four ingredients in specific proportions -- a, b, c, and d -- in specified amounts (essentially a recipe), which may be processed into a cured fluoroelastomer product.[2]

The products covered by the '320 patent, as well as the infringing imported products, are "cure incorporated" precursors of curable fluoroelastomer compositions, i.e., a proportional mixture of all of the ingredients required for curing (except an acid acceptor and an optional base).[3] A rubber processor is then able to produce finished goods from the mix using any one of a variety of rubber processing methods, such as injection molding or transfer molding, which involve the final step of curing the product by the addition of an acid acceptor and an optional base (which corresponds to ingredient c of the claims in issue of the '320 patent).[4]

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[1] 59 Fed. Reg. 12344 (March 16, 1994).

[2] The crosslinking or curing (i.e., hardening) of an elastomer such as a fluorinated polymer is commonly referred to as "vulcanization."

[3] Individual ingredients a, b, c, and d are not covered by the patent.

[4] Although there are at least 15 hypothetical "precursors" of curable fluoroelastomer compositions, consisting of the set of the four ingredients and all possible combinations thereof, and arguably each ingredient and combination thereof could be regarded as a "precursor," there is no basis in the record of this investigation to conclude that precursors other than the "cure incorporated" mixtures of ingredients a, b, and d of the claims in issue (leaving out the acid acceptor and optional base to be added by the customer in a final step) have been manufactured and sold by either complainant or respondents.

In his final initial determination ("ID"), the presiding administrative law judge ("ALJ") concluded that respondents had violated section 337, based on his findings that (1) the claims in issue of the '320 patent are not invalid; (2) the accused products imported by respondents infringe the claims in issue of the '320 patent under the doctrine of equivalents; and (3) a domestic industry exists.

On February 2, 1995, the Commission determined not to review the ALJ's final ID, thereby finding a violation of section 337 to exist. The Commission also requested written submissions on the issues of remedy, the public interest, and bonding. 60 Fed. Reg. 7581 (February 8, 1995). Complainant 3M, respondents, and the Commission investigative attorney (IA) each filed submissions and reply submissions on these three issues.

On March 16, 1995, the Commission made its determinations on the issues of remedy, the public interest, and bonding. The Commission determined that the appropriate form of relief is a limited exclusion order prohibiting the entry for consumption of the infringing curable fluoroelastomer compositions and precursors thereof. In addition, the Commission issued an order directed against respondent Ausimont U.S.A. to cease and desist from the following activities: importing, selling, marketing, distributing, offering for sale, or otherwise transferring (except for exportation) in the United States curable fluoroelastomer compositions or precursors thereof, determined to be infringing the claims in issue of the '320 patent, in violation of section 337 of the Tariff Act of 1930. The orders apply to respondents and to their principals, stockholders, officers, directors, employees, agents, licensees, distributors, controlled (whether by stock ownership or otherwise) and/or majority owned business entities, successors, and assigns. The Commission also determined that the statutory public interest factors enumerated in sections 337(d) and 337(f) of the Tariff Act of 1930 do not preclude issuance of such orders and that the bond during the 60-day Presidential review period should be in the amount of 48 percent of the entered value of the infringing compositions.

II. DISCUSSION

Where a violation of section 337 has been found, the Commission must consider the issues of remedy, the public interest, and bonding. Under subsections 337(d) and (f), the Commission may issue an exclusion order, a cease and desist order, or both, depending on the circumstances. The Commission has broad discretion in selecting the form, scope, and extent of the remedy in a section 337 proceeding.[5] The Commission may make factual determinations in the remedy phase of a section 337 investigation, to the extent necessary, in order to reach its determination, which may be based on the evidence of record, or on the basis of submissions of the parties on remedy, the public interest, and bonding.[6]

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[5] Viscofan, S.A. v. United States International Trade Commission, 787 F.2d 544, 548 (Fed. Cir. 1986) (affirming Commission remedy determination in Certain Processes for the Manufacture of Skinless Sausage Casings and Resulting Products, Inv. Nos. 337-TA-148/169, USITC Pub. 1624 (December 1984)); Hyundai Electronics Industries Col, Ltd. v. U.S. International Trade Commission, 899 F.2d 1204 (Fed. Cir. 1990) (affirming Commission remedy issued in Certain Erasable Programmable Read-Only Memories, Components Thereof, Products Containing Such Memories, and Processes for Making Such Memories, Inv. No. 337-TA-276, USITC Pub. 2196 (May 1989)).

[6] Sealed Air Corporation v. U.S. Int'l Trade Comm'n, 645 F.2d 976 (C.C.P.A. 1981).

A. REMEDY

1. The Exclusion Order.

There are two types of exclusion orders: a general exclusion order and a limited exclusion order. A general exclusion order instructs the U.S. Customs Service to exclude from entry all articles which infringe the involved patent, without regard to source. Thus, a general exclusion order applies to persons who were not parties to the Commission's investigation and, indeed, to persons who could not have been parties, such as persons who decide to import after the Commission's investigation is concluded. See, Certain Airless Paint Spray Pumps, Inv. No. 337-TA-90, 216 U.S.P.Q. 465 (ITC 1981).

A limited exclusion order instructs the Customs Service to exclude from entry all articles which infringe the involved patent claims and that originate from a firm that was a party to the Commission investigation.

A cease and desist order is an order to a person who was a party to the Commission investigation to cease its unfair acts. Unlike an exclusion order, it is enforced by the Commission, through the courts, not by the Customs Service.

No evidence has been presented in this investigation which would provide a basis for issuance of a general exclusion order and none of the parties has requested issuance of a general exclusion order. We have therefore determined to issue a limited exclusion order prohibiting from entry into the United States certain fluoroelastomer compositions and precursors thereof manufactured abroad by Ausimont S.p.A. and covered by claims 1, 2, 4, 5, 6, 11, 12, 14 or 15 of the '320 patent. We expect that the U.S. Customs Service will exclude respondents' precursor curable fluoroelastomer compositions containing ingredients a, b, and d as specified in the claims in issue of the '320 patent, as well as respondents' curable fluoroelastomer compositions containing ingredients a, b, c, and d. We do not expect that Customs will exclude any other possible "precursors" of curable fluoroelastomer compositions since there is no evidence in the record of this investigation to indicate that precursors, other than those missing ingredient c, have been imported and sold by respondents. We have determined not to include a provision expressly excluding from the coverage of the exclusion order any cured fluoroelastomer products (such as O-rings) since such products are not covered by the claims in issue of the '320 patent.[7]

2. Type of Entry.

Although respondents admitted in their initial submission on remedy, the public interest, and bonding that they have engaged in transshipments of infringing product across the United States to a customer in Canada,[8] neither complainant nor the IA has argued in favor of expanding the scope of the exclusion order to cover entries other than "entry for consumption." As the Commission stated in Certain Devices for Connecting Computers Via Telephone Lines, Inv. No. 337-TA-360, Commission Opinion, December 12, 1994, although the Commission's remedial authority is quite broad, it has applied this authority "in measured fashion and has issued only such relief as is adequate to redress the harm caused by the prohibited imports." Id. at page 9. Here, complainant has not alleged that it has been adversely affected by such transshipments or is likely to be so affected. Id. at page 10.

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[7] Indeed, "cured" or "vulcanized" fluoroelastomer products differ chemically from the curable fluoroelastomer mix of the claims in issue of the '320 patent.

[8] Respondents' submission at pages 3-4.

3. Certification and the Scope of the Limited Exclusion Order.

The parties are in agreement that the limited exclusion order contain a certification provision whereby an importer seeking to import goods manufactured by Ausimont, S.p.A. may do so by providing a written certification to Customs that the goods are not covered by the exclusion order. Such certification provisions are intended to facilitate Customs' administration of the order by eliminating the need to test every shipment of goods sought to be imported. In Hyundai Electronics Industries Co., Ltd. v U.S. Int'l Trade Comm'n, 899 F.2d 1204 (Fed. Cir. 1990), the Federal Circuit concluded that the inclusion of a certification requirement was "both reasonable and well within [the Commission's] authority." Id., 899 F.2d at 1210. In this case, certification appears to be a practical means for identifying product that may be imported since it is not possible to determine readily whether a curable fluoroelastomer product is covered by the claims in issue of the '320 patent.[9]

Complainant, however, seeks to expand the certification beyond merely identifying goods covered by the exclusion order. It seeks to require prospective importers to certify that they have not and will not recommend to users of the fluoroelastomer compositions to be imported that such users add or mix components into the imported fluoroelastomer compositions so as to result in a composition covered by the claims in issue of the '320 patent. Complainant also seeks to require prospective importers to certify that they do not know of any U.S. persons or companies that will add or mix components into the imported fluoroelastomer compositions where said mixing will result in a composition covered by the claims in issue of the '320 patent. As a basis for the inclusion of such an expanded certification, complainant argues that such a provision must be included to prevent induced or contributory infringement of the '320 patent.[10]

While complainant correctly notes that the ALJ made findings on induced infringement and contributory infringement (which the Commission has adopted), the factual basis for those findings was that the products covered by the '320 patent, as well as the allegedly infringing imported products, are properly proportioned mixtures incorporating all of the ingredients required for an infringing product, except an acid acceptor and an optional base (which correspond to the ingredient c called for by claim 1 of the '320 patent).[11] Since it has been established that this final ingredient is always added by the customer,[12] we agree with complainant that the limited exclusion order should cover curable fluoroelastomer compositions and precursors consisting essentially of ingredients a, b,

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[9] However, we note that Customs has testing laboratories available and is capable of determining by chemical analysis whether a given shipment of imported compositions is covered by the claims in issue of the '320 patent.

[10] 35 U.S.C. § 271(b) and (c) provide the standards for contributory and induced infringement of U.S. patents. The standards in relevant part are as follows:

> (b) Whoever actively induces infringement of a patent shall be liable as an infringer.
> (c) Whoever sells a component of a patented ... composition ... constituting a material part of the invention, knowing the same to be especially made or especially adapted for use in an infringement of such patent, and not a staple article or commodity of commerce suitable for substantial noninfringing use, shall be liable as a contributory infringer.

See also Certain Headboxes, Inv. No. 337-TA-82 (1981).

[11] Final Initial Determination, filed December 15, 1994, pages 188-192.

[12] The record demonstrates that the infringing compositions are useful only when the acid acceptor and optional base are added. Final Initial Determination, Findings of Fact Nos. 343-345.

and d of claim 1 of the '320 patent.[13] Complainant's expanded certification, however, goes beyond what is necessary to prevent respondents from importing cure incorporated product. Complainant apparently seeks to cover the hypothetical situation where some other precursor of a curable fluoroelastomer composition is sought to be imported, or where some or all of the ingredients are imported separately for mixture in the United States into curable fluoroelastomer compositions. There is no evidence in the record that such situations have occurred in the past, nor has complainant provided evidence that such situations are likely to occur in the future. We therefore decline to extend the certification to cover such a hypothetical situation and note that such a certification would not aid Customs in identifying covered products and would potentially bar entry of non-infringing ingredients.

The four individual chemical ingredients (a, b, c, and d) listed in claim 1 of the '320 patent may well have non-infringing applications. We see no basis for requiring prospective importers to make a certification concerning products which would not fall within the scope of an exclusion order. We therefore determine that prospective importers shall be permitted to import goods upon certifying that the goods are not covered by the exclusion order. However, if complainant learns that respondents have engaged, or are about to engage, in acts which would be a circumvention of the Commission's limited exclusion order, e.g., suggesting to customers that they mix an ingredient or ingredients into compositions imported by respondents which would result in a fluoroelastomer composition covered by the claims in issue of the '320 patent, then complainant may file a petition for modification of the exclusion order pursuant to 19 C.F.R. § 211.57.

4. The Cease and Desist Order.

The Commission normally issues cease and desist orders when the circumstances indicate that the respondents have in U.S. inventory a "commercially significant" amount of infringing imported product that they can sell, thus undercutting the effect of any exclusion order. See, e.g., Certain Crystalline Cefadroxil Monohydrate, Inv. No. 337-TA-293, USITC Pub. 2391 (March 15, 1990). Unlike an exclusion order which is enforced by the U.S. Customs Service, a cease and desist order is typically an in personam order directed to a party in the United States and enforced by the Commission. Thus, unless a party in the United States can be compelled to do some act or to refrain from doing some act, a cease and desist order is inappropriate since the jurisdiction of the Commission (and United States courts) does not extend abroad.

Respondents argue that their U.S. inventory should not be deemed to include product that ****. We have therefore determined to include such goods within the term "inventory" for purposes of determining whether to issue a cease and desist order.

We next address the question of whether respondents' inventory is sufficient to warrant the issuance of a cease and desist order. Although respondents argue that they have not engaged in "stockpiling" of inventory, that is not the issue. The test for determining whether to issue a cease and desist order affecting inventory is whether the domestic respondent maintains a "commercially significant" level of inventories in the United States.[14] ****

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[13] Complainant's proposed exclusion order specifically lists only the ingredients in clauses a, b, and d of claim 1 of the '320 patent and does not include the acid acceptor or optional base called for in clause c.

[14] Certain Crystalline Cefadroxil Monohydrate, Inv. No. 337-TA-293, Commission Opinion on Remedy, the Public Interest and Bonding at page 40, USITC Publication 2391, (June 1991).

On balance, we have determined to issue a cease and desist order. Although there may be circumstances warranting modification of the cease and desist order, ****, we believe that the burden should be on respondents to come forward with evidence showing why a modification would be appropriate.

The cease and desist order we have issued prohibits respondent Ausimont U.S.A. from importing, selling, marketing, distributing, offering for sale, or otherwise transferring (except for exportation) in the United States curable fluoroelastomer compositions or precursors thereof covered by the claims in issue of the '320 patent. The order also requires respondent Ausimont U.S.A. to file quarterly reports with the Commission on the quantity in pounds and the value in dollars of foreign-made covered product imported by respondent or sold in the United States and/or that remains in inventory during the reporting period.

B. THE PUBLIC INTEREST

Prior to issuing relief, the Commission is required to consider the effect of such relief on the public health and welfare, competitive conditions in the U.S. economy, the production of like or directly competitive articles in the United States, and U.S. consumers. 19 U.S.C. 1337(d). Complainant and the IA argue that the issuance of relief in this case would have no adverse impact on the public interest in this case. We agree that the statutory public interest factors do not preclude issuance of relief in this investigation.

Respondents have urged the Commission to postpone the operation of the limited exclusion order for a period of six months because **** As the Commission stated in Microwave Filters, the public policy of enforcing valid U.S. intellectual property rights overrides a request for an exception to an exclusion order based on hardship.

C. BONDING

Section 337(j)(3) provides for the entry of infringing articles upon the payment of a bond during the 60-day Presidential review period.[15] The bond is to be set at a level sufficient to "offset any competitive advantage resulting from the unfair method of competition or unfair act enjoyed by persons benefitting from the importation."[16] The bond should not be set so high as to effectively prevent importation during the Presidential review period. However, the period of Presidential review is relatively short, and the consequences of any bond are therefore likely to be short-lived.

Complainant urges that the bond during the 60-day Presidential review period should be set at 100 percent of the entered value of the imported curable fluoroelastomers, arguing that there is insufficient price information to make a price comparison between the imported goods and those of the domestic industry. The IA, however, utilized information of record which permits comparison of respondents' prices to those of complainant with respect to three of respondents' infringing imports and complainant's competing products. The IA calculates that a bond in the amount of **** percent of the entered value of the goods is appropriate to offset the price advantage enjoyed by the infringing imports. Respondents note, however, that the IA's pricing information ****. After taking into consideration ****, respondents arrive at a bond in the amount of 47.9 percent of the entered value of the goods.[17] We believe that a bond in the amount of 48 percent, in line with the ****

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[15] 19 U.S.C. § 1337(j)(3).

[16] S. Rep. No. 1298, 93rd Cong., 2d Sess. 198 (1974).

[17] Respondents' Reply at page 10. ****

information provided by respondents and the unweighted average method proposed by the IA, reasonably approximates the competitive advantage enjoyed by respondents. Such a method is preferable to the arbitrary figure of 100 percent recommended by complainant.

Customs has, in the past, expressed a strong preference for imposing a single bond against all infringing imported products during the 60-day Presidential review period. We have therefore determined that the bond be set in the amount of 48 percent of the entered value of the imported goods covered by the claims in issue of the '320 patent.

PUBLIC VERSION

UNITED STATES INTERNATIONAL TRADE COMMISSION
Washington, D.C.

In the Matter of

CERTAIN CURABLE FLUOROELASTOMER COMPOSITIONS AND PRECURSORS THEREOF

Investigation No. 337-TA-364

Initial Determination

Paul J. Luckern, Administrative Law Judge

Pursuant to the Notice of Investigation (59 Fed. Reg. 12,344 (March 16, 1994)), this is the administrative law judge's initial determination, under Commission interim rule 210.53 (19 C.F.R. § 210.53). The administrative law judge hereby determines, after a review of the record developed, that there is a violation of subsection (a)(1)(B)(i) of the Tariff Act of 1930, as amended (19 U.S.C. § 1337), in the importation into the United States, the sale for importation, or the sale within the United States after importation, of certain curable fluoroelastomer compositions and percussors thereof.

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ABBREVIATIONS

CB	Complainant's Initial Posthearing Brief
CBR	Complainant's Reply Posthearing Brief
CX	Complainant's Documentary Exhibit
CPX	Complainant's Physical Exhibit
FF	Findings of Fact
RB	Ausimont's Initial Posthearing Brief
RBR	Ausimont's Reply Posthearing Brief
RPX	Ausimont's Physical Exhibit
RRS	Ausimont's Proposed Rebuttal To Staff's Finding
RRX	Ausimont's Rebuttal Exhibit
RX	Ausimont's Documentary Exhibit
SB	Staff's Initial Posthearing Brief
SSF	Staff's Proposed Finding
SX	Staff's Documentary Exhibit
Tr.	Transcript

I. PROCEDURAL HISTORY

By notice dated March 8, 1994, the Commission instituted an investigation pursuant to subsection (b) of section 337 of the Tariff Act of 1930, as amended, to determine whether there is a violation of section 337(a)(1)(B)(i) in the importation into the United States, the sale for importation, or the sale within the United States after importation of certain curable fluoroelastomer compositions and precursors thereof, by reason of alleged infringement of claims 1-2, 4-6, 11-12, and 14-15 of U.S. Letters Patent No. 4,287,320 (the '320 patent) and whether there exists an industry in the United States as required by subsection (a)(2) of section 337.

During the evidentiary hearing respondents moved to strike "massive portions" of the witness statement and testimony of Robert Engel, who was called by complainant as an expert witness. Also complainant opposed a proposed expert qualification of Ausimont's Tommasi. Those items are treated in Order No. 15, filed herewith.

The matter is now ready for a final initial determination by the administrative law judge.

The initial determination is based on the entire record complied at the hearing and the exhibits admitted into evidence. The administrative law judge has also taken into account his observation of the witnesses who appeared before him during the hearing. Proposed findings submitted by the parties participating in the hearing not herein adopted, in the form submitted or in substance, are rejected either as not supported by the evidence or as involving immaterial matters. The findings of fact of this initial determination include references to supporting evidentiary items in the record. Such references are intended to serve as guides to the testimony and exhibits supporting the findings of fact of the administrative law judge, and they do not necessarily represent complete summaries of the evidence supporting said findings.

II. PARTIES

The parties, complainant Minnesota Mining and Manufacturing Company (3M) and respondents Ausimont S.p.A. and Ausimont U.S.A., Inc. (Ausimont), are identified in the findings (FF 1 to 3).

III. IMPORTATION

Section 337(a)(1)(B) prohibits "[t]he importation into the United States, sale for importation, or the sale within the United States after importation" of articles which infringe a valid and enforceable United States patent. 19 U.S.C. § 1337(a)(1)(B)(i). Ausimont has stipulated in this investigation that it has imported into the United States and is currently selling the following accused products: FOR 421, FOR 423, FOR 5351, FOR 65BI/R and FOR 800HE (FF 8); that it has imported and sold FOR 420 in the United States, although it has not done so since 1991 (FF 9); and that it has substantial sales of the accused compositions, excluding FOR 9550, in the United States (FF 30). With respect to FOR 9550, Ausimont stipulated that it has imported FOR 9550 into, although it has not sold FOR 9550 within, the United States (FF 12, 24). Thus it is found that complainant has met its burden of showing that Ausimont has imported each of the accused products into the United States within the meaning of section 337(a)(1)(B).

IV. JURISDICTION

The Commission has in rem and subject matter jurisdiction in this investigation under section 337 of the Tariff Act of 1930, as amended, 19 U.S.C. § 1337, since the alleged unfair methods of competition and unfair acts involve the importation into the United States of certain fluoroelastomer compositions and precursors thereof alleged to infringe certain claims of the '320 patent. The Commission has in personam jurisdiction based on the appearance of counsel for all the parties.

V. THE PRODUCTS AT ISSUE

The products involved in this investigation are fluoroelastomer compositions. A fluoroelastomer is a rubber-like polymer in which some of the hydrogen atoms of the polymer have been substituted with fluorine (FF 4). The products covered by the '320 patent and the accused products are "incorporated cure" products,[1] meaning that chemicals required to vulcanize the fluoroelastomer polymer are incorporated with a mix containing the polymer by the compounder or molder and then placed in a mold, cured and sold as a product (FF 4). Fluoroelastomers are used in automotive, aerospace, pollution control, chemical processing and food processing applications (FF 4). The specific components of the accused compositions are set forth in the findings (FF 4, 14, 15, 18 to 23, 25, 26, 29, 33, 36, 37).

Complainant sells fluoroelastomers products in the United States which are covered under the '320 patent (FF 39). Neither Ausimont nor the staff has disputed FF 39 which corresponds to complainant's proposed finding 259.

VI. OPINION

Complainant alleges that Ausimont S.p.A. (Ausimont Italy) has contributorily infringed and induced infringement of claims 1, 2, 4, 5, 6, 11, 12, 14 and 15 of the '320 patent and that Ausimont U.S.A. has contributorily infringed, induced infringement and directly infringed said claims literally and, if not literally, under the doctrine of equivalents. The staff argued

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[1] Curing a product refers to vulcanizing or cross-linking the product (FF 190).

that there is infringement under the doctrine of equivalents.

The parties have stipulated that all proofs relating to the infringement issue in this investigation may be limited to claim 1 of the '320 patent. (CX-495, Stipulation No. 16). Thus if claim 1 is infringed, the remaining claims 2, 4, 5, 11, 12, 14 and 15 in issue are also infringed.

Complainant has the burden of proving infringement by a preponderance of the evidence. Smithkline Diagnostics, Inc. v. Helena Lab. Corp., 859 F.2d 878, 889, 8 USPQ2d 1468, 1477 (Fed. Cir. 1988) (Smithkline). If Ausimont's accused fluoroelastomer compositions fall clearly within the language of claim 1 of the '320 patent, then literal infringement is established. Envirotech Corp. v. Al George, Inc., 730 F.2d 753, 759, 221 USPQ 473,477 (Fed. Cir. 1984). The claims define the metes and bounds of an invention and the protection afforded to a patentee. Raytheon Co. v Roper Corp., 724 F.2d 951, 957, 220 USPQ 592, 597 (Fed. Cir. 1983).

A. Claim Construction

A threshold question in determining the issue of infringement is to ascertain the scope of the claims. Minnesota Mining and Manufacturing Co. v. Johnson & Johnson, 976 F.2d 1559, 1576, 24 USPQ2d 1321, 1335 (Fed. Cir. 1992). Claims are construed in the same manner when determining both validity and infringement. W.L. Gore & Associates, Inc. v. Garlock, Inc., 842 F.2d 1275, 1279, 6 USPQ2d 1277, 1280 (Fed. Cir. 1988).

In dispute on the infringement issue is the meaning of the term "quaternary phosphonium . . . compound" in clause (b) of independent claims 1, 12, 14 and 15 (FF 53, 54). Complainant contends that a "quaternary phosphonium compound" is a phosphorus containing compound in which the phosphorus atom is attached to four substituents, which can be radicals except hydrogen or an isotope of hydrogen, and where the phosphorus bears a positive charge and is ionically associated with a negatively charged atom (CB at 27-28). Complainant's counsel at closing argument argued that there is nothing in the specification of the '320 patent that restricts the interpretation of the language "quaternary phosphonium compound" to a compound containing phosphorus only linked to carbon, i.e., a compound having four phosphorus carbon (P-C) covalent bonds as contended by Ausimont and the staff; and that while in the prior art patents as of the October 16, 1973, filing date of the '320 patent, when a quaternary phosphonium compound was recited in a claim the quaternary phosphonium compound was limited to a compound having four P-C bonds, the independent claims of the '320 patent have no such limitation (Tr. at 2717, 2718); and that it is "pretty well established" in patent law that patent claims can be drawn broader than the specific disclosure and it is not uncommon for patent claims to be interpreted to cover a later discovered compound (Tr. at 2717, 2718, 2832, 2833).

The '320 patent issued on September 1, 1981, to Robert E. Kolb on an initial application filed on October 16, 1973, and is assigned on its face to complainant (FF 50). The parties are in agreement (Tr. at 2863 to 2869) that for any interpretation of the words of a claim in issue the administrative law judge must look to how one of ordinary skill in the revelant art, at the time of the initial filing of the '320 patent, viz. October 16, 1973 (FF 50), would interpret any words in dispute. The meaning that an inventor gives to a word in a claim of an application as filed cannot be changed to conform to subsequent events. See Intellicall, Inc. v. Phonometrics, Inc., 852 F.2d 1384, 21 USPQ2d 1383 (Fed. Cir. 1985).

The principal field in which the Kolb patent would be practiced is the compounding and curing of fluoroelastomers (FF 173, 174). In 1973 a person involved in compounding or formulating fluoroelastomer compositions would typically have been a person with experience in rubber compounding and in the use of equipment to process rubbers (FF 173). He or she would be what is referred to in the industry as "rubber compounders" (FF 179). Moreover, in 1973 formulation and compounding work in fluoroelastomers was generally conducted by persons whose training and experience was developed "at the bench" or "on the job." Such a person would not necessarily have had a college degree but typically would have had at least a high school degree, and many would have taken college courses. While the experience level for a man skilled in the art would vary, it would typically be about three to five years of practice, assuming that the person's work was not exclusively formulation but also involved routine compounding and testing. However, depending on how smart the person was, the person could have had only from six months to two years experience at the work bench. With respect to practicing in the involved art, a person skilled in the art would have to know where bisphenol-type compounds could be obtained and what fluoroelastomer and quaternary phosphonium compounds were available in 1973 so he could use them (FF 172 to 175). Accordingly, the administrative law judge finds that a person of ordinary skill in the relevant art, viz. the field of compounding fluoroelastomers and curing them, would have had a high school education, perhaps have taken some college chemistry courses, and would have had some on the bench training, which training would vary from two years to up to some five years, depending on how smart the person was.

It is hornbook patent law that words of a claim are given their ordinary and accustomed meaning unless it appears from the specification and prosecution history that the inventor intended a different meaning. Smithkline, 859 F.2d at 878, 8 USPQ2d at 1471-72. The claims in issue merely recite for component b "quaternary phosphonium or ammonium compound," and hence those claims give no indication of the meaning of that phrase (FF 53, 54). Claim 13, which is not in issue does, however, recite triphenyl benzyl phosphonium chloride for the quaternary phosphonium compound (FF 55), which all parties agree is a compound having four phosphorus carbon covalent bonds. Under the doctrine of claim differentiation, the presence of an express limitation in one claim of a patent negates an intent to limit similarly by implication a claim in which the limitation is not expressed. Kalman v. Kimberly-Clark Corp., 713 F.2d 760, 770, 218 USPQ 781, 788 (Fed. Cir. 1983), cert. denied, 465 U.S. 1026 (1984). It is improper to read into an independent claim a limitation that another dependent claim sets forth explicitly. Whittaker Corp. v. UNR Industries, Inc., 911 F.2d 709, 712, 15 USPQ2d 1742, 1744 (Fed. Cir. 1990).

The prosecution history of the '320 patent provides no assistance in defining the scope of the term "quaternary phosphonium compound" (FF 89). The '320 specification does disclose, however, that the quaternary phosphonium compounds useful in preparing curable fluoroelastomer compositions are compounds which contain at least one phosphorus atom covalently bonded through carbon-phosphorus single bonds to four organic radicals, and that such materials are described, for example, in "Organo Phosphorus Compounds," G. M. Kosolapoff (John Wiley and Sons, New York, 1959), particularly chapter five (FF 111). No other portion of the 1950 Kosolapoff book is mentioned in the '320 patent (FF 114). Chapter five of the 1950 Kosolapoff is entitled "Quaternary Phosphonium Compounds." Kosolapoff describes the contents of chapter five as phosphorus containing chemical compounds wherein the compounds are limited to four carbon-phosphorus bonds (FF 113). Kosolapoff identifies over two hundred phosphorus compounds in chapter five, each of which involves a phosphorus atom bonded to four carbon atoms (FF 118).

All of the working examples in the '320 patent that use a phosphonium compound use a compound in which the phosphorus atom is bonded directly to four carbon atoms (FF 127). The de Brunner U.S. Patent No. 3,752,787 and Patel et al. U. S. Patent No. 3,712,877, cited in the '320 patent teaches that ammonium compounds are compounds having four carbon-nitrogen bonds (FF 108, 136). Patents cited by the Examiner in the prosecution of the '320 patent, such as Patel et al. U.S. 3,655,727 and Schmiegel U.S. Patent No. 3,933,732 (FF 57, 78) disclose that the term "quaternary" as it relates to quaternary ammonium and/or phosphonium compound concerns compounds having four car-nitrogen or four carbon-phosphorus bonds (FF 58, 97, 135, 136). Inventor Kolb, when asked whether the only description he gave in the '320 patent for quaternary phosphonium compounds is the description wherein the compound contains four phosphorus-carbon bonds, answered in the negative and for support referred only to claim 1 of the '320 patent (FF 158, 159). However, claim 1 does not tell a person of ordinary skill in the art in 1973 what a quaternary phosphonium compound is. Inventor Kolb also agreed that the reason he did not refer in the '320 patent to a phosphonium compound containing a nitrogen-phosphorus bond was because he did not contemplate such compounds (FF 159).

Complainant's expert Harwood (FF 45) testified that the term "quaternary" is used in the same manner when relating to either a phosphonium or ammonium compound (FF 137) and that it is appropriate to look at the nomenclature of quaternary ammonium compounds for guidance as to the appropriate nomenclature for quaternary phosphonium compounds (FF 138). Chemical dictionaries and chemistry textbooks published before the initial October 16, 1973 filing date of the '320 patent define "quaternary ammonium base" and/or "quaternary ammonium" (the component b of claim 1 in issue includes as an alternative compound a "quaternary ammonium compound" (FF 53)) as having nitrogen covalently bonded to four carbon atoms (FF 141, 143, 144 to 147, 148 to 150, 164 to 168).

Complainant's outside expert witnesses Harwood (FF 45) and Engel (FF 48) could not identify any chemical or technical dictionary, or even any literature reference, as of the initial October 16, 1973 filing date of the '320 patent that contained a definition of quaternary ammonium or phosphonium compound which supported complainant's assertion that the four substituents could be anything but hydrogen or one of its isotopes (FF 145, 146). Complainant's expert Engel did not develop complainant's definition of quaternary phosphonium compound until after he obtained his Ph.D and began working in the organophosphorus field (FF 156). Ausimont's expert Schlosser (FF 47) testified that quaternary phosphonium compounds have four phosphorus-carbon bonds (FF 169). Under the nomenclature rules of Chemical Abstracts and the International Union of Pure and Applied Chemistry, a compound having a phosphorus-nitrogen bond could not be called a quaternary phosphonium compound (FF 170, 171).

Complainant has referenced certain scientific articles (CX-51 to CX-99 to support its claim interpretation. However, as complainant's expert Harwood agreed, a person who is not delving into research but who wants to determine the meaning of chemical terms would likely rely on dictionary definitions and would remember no more than what the person learned in any undergraduate chemistry courses which the person may have taken (FF 182). When Harwood was asked if a person of ordinary skill in the art in 1973 wanting to determine what a quaternary phosphonium compound is in the fluoroelastomer art would refer to scientific research articles, such as complainant's exhibits CX-51 through CX-99, Harwood was of the opinion that in order to do so the person would have to be "pretty good" in the use of a library or have a librarian help and, if the person really wanted to know about quaternary phosphonium compounds, he or she "probably could find these articles, [although] [t]hey probably would be a little advanced for many of them, to be honest" (FF 182). Harwood further testified that a person with only a few years of college education in 1973 would not have consulted scientific articles CX-51 through CX-99 (FF 176); that people not active in the organophosphorus or nitrogen chemistry fields would be likely to rely on chemical dictionaries to determine the definitions of the chemical term "quaternary phosphonium or ammonium" (FF 177); and that it was the people "who do <u>research</u> in this area [who] would be interested in materials that had subconstituents [sic] other than carbon" (FF 182). Inventor Kolb admitted that prior to the filing of the '320 patent application he had not reviewed any of the scientific articles set forth in CX-51 to CX-99 (FF 181).

Complainant's counsel at closing argument argued that it is "pretty well established" in patent law that patent claims can be drawn broader than the specific disclosure and that it is not uncommon for patent claims to be interpreted to cover something that is later discovered (Tr. at 2832, 2833). Complainant appears to be confusing claim construction with the doctrine of equivalents. Under the doctrine of equivalents, a patent obtained on a modification does not necessarily preclude the modification from being considered an equivalent of an invention previously patented. Thus in Atlas Powder Co. v. E. I. du Pont de Nemours & Co., 750 F.2d 1569, 1581, 224 USPQ 409, 417 (Fed. Cir. 1984) (Atlas Powder), the patent claimed a water resistant blasting agent. One claimed element was a certain "water-in-oil" emulsion. The accused infringer produced a blasting agent that, unlike the patentee's agent, involved adding the emulsion in situ. The accused infringer used a certain "oil-in-water" emulsion that turned into a water-in-oil type when applied in situ. The Court upheld a finding of equivalence even though the accused infringer had obtained a patent on its improved blasting agent. In issue in this section of the initial determination is not the doctrine of equivalence but rather claim construction.

Based on the '320 patent specification, the prior art and the testimony of witnesses, the administrative law judge finds that a person of ordinary skill in the art, as of the initial October 16, 1973 filing date of the '320 patent, would not have referred to scientific research articles to determine the meaning of the term "quaternary phosphonium ... compound" recited in the claims of the '320 patent but rather, based on the '320 patent specification and standard chemical references, would have interpreted that compound as a phosphorus containing compound wherein the phosphorus atom is attached to four carbon atoms.

B. Literal Infringement

Independent claim 1 requires the presence of a "quaternary phosphonium . . . compound" (component b of claim 1). The administrative law judge has construed the claimed phrase "quaternary phosphonium . . . compound" as limited to a phosphorus containing compound wherein the phosphorus atom is attached to four carbon atoms.

The accused compositions do not contain a phosphorus containing compound wherein the phosphorus atom is attached to four carbon atoms. Rather the accused compositions have either or XA-51 is an adduct from a compound or GM 102E which is a compound, wherein the phosphorus atom is attached to only three carbon atoms and one nitrogen atom. Since the accused compositions do not have a compound containing four phosphorus-carbon bonds literally required by claim 1 of the '320 patent, the administrative law judge finds that Ausimont's accused compositions do not literally infringe said claim 1.

C. Doctrine of Equivalents

Even if an accused composition does not literally infringe a patent, it can infringe under the doctrine of equivalents if it performs substantially the same function in substantially the same way to obtain substantially the same result as the patented invention. Graver Tank & Mfg. Co. v. Linde Air Products Co., 339 U.S. 605, 608, 85 USPQ 328, 330 (1950); London v. Carson Pirie Scott & Co., 946 F.2d 1534, 1538, 20 USPQ2d 1456, 1458 (Fed. Cir. 1991); Pennwalt Corp. v. Durand-Wayland, Inc., 833 F.2d 931, 934, 4 USPQ2d 1737, 1739 (Fed. Cir. 1087) (en banc), cert. denied, 485 U.S. 961 (1988); Spectra Corp. v. Lutz, 839 F.2d 1579, 1582, 5 USPQ2d 1867, 1869 (Fed. Cir. 1988) (Spectra). Equivalence is determined by comparing the claimed matter as a whole and the accused composition. Hughes Aircraft Co. v. United States, 717 F.2d 1351, 1364, 219 USPQ 473, 480-81 (Fed. Cir. 1985); 4 Chisum, Patents 18.04 [1] at 18-90 to 18-93 (1994). However, where the accused product avoids literal infringement by changing one ingredient of a claimed composition, it is appropriate to consider in assessing equivalence whether the changed ingredient has the same purpose, quality and function as the claimed ingredient. Atlas Powder, 750 F.2d at 1579-80, 224 USPQ at 416.[2] The doctrine of equivalents can be used to extend patent protection to products that would have been unknown to those skilled in the art at the time of filing of the application that resulted in the patent in issue, since the question of equivalence is determined at the time infringement takes place.[3] Atlas Powder, 750 F.2d at 1569, 1581, 224 USPQ at 417.

Each of complainant and the staff has argued that the equivalence of GM 102E or XA 51 in the accused compositions (FF 14, 15, 18 to 23, 25, 26, 29, 33, 36, 37) to quaternary phosphonium compounds (component b of claim 1), with respect to the claimed curable fluoroelastomer composition (FF 53) and in terms of their function, way, and result, is supported by ample evidence in the record, including published literature, the testimony of witnesses (including Ausimont's own researchers'), Ausimont's own internal research reports, and experimental work performed by 3M's and Ausimont's experts. Ausimont's counsel at closing arguments (Tr. at 2848 to 2861) agreed that if the accused compositions infringe the '320 patent under the doctrine of equivalents, then Ausimont U.S.A. directly infringes the '320 patent. Ausimont's counsel argued, however, that complainant failed to establish even

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[2] Ausimont's accused curable fluoroelastomer compositions are to a fluoroelastomer composition as recited in claim 1 with the exception that component (b) of the claimed imposition, viz. "quaternary phosphonium or ammonium compound" is changed to GM 102E or XA-51. See infra.

[3] Ausimont developed its P-N based curing system and filed for patent protection in 1977 (FF 200). However, Ausimont's U.S. sales of sulfone-containing accused compositions did not commence until 1986 (FF 38). Ausimont imported the accused TECNOFLON FOR-420 into the United States at least until 1991, and its accused TECNOFLON FOR-9550 was imported into the United States in 1990 and 1992. In addition, the following specific Ausimont articles have been imported into the United States and are presently being sold: the accused FOR-423, FOR-5351, FOR-65BI/R and FOR 800 HE (FF 38). Hence, the administrative law judge reject. Ausimont's contention that the approximate time for assessing equivalency is 1977 (RBR at 24).

a prima facie case of infringement under the doctrine of equivalents.

1. The Claimed Composition And The Accused Compositions

The claimed invention in independent claim 1 recites a "curable fluoroelastomer composition" (FF 53). Like the claimed invention, the accused compositions (FF 14, 15, 18 to 23, 25, 26, 29, 33, 36, 37) are curable compositions which are cured, i.e. vulcanized (crosslinked) to make elastomeric molded parts (FF 4). Complainant's and Ausimont's molded parts are in competition with each other (FF 42). Independent claim 1, as well as all of the claims in issue, however, does not limit or even specify that the claimed "curable composition" has any particular property, for example an increase in cure rate (FF 53, 54). In addition the claimed curable fluoroelastomer composition is not limited to, and does not even specify, any utility for the cured product obtained from the claimed "curable composition" (FF 53, 54). To the contrary, complainant's agent in an amendment filed on June 23, 1977 in Ser. No. 493,537 in the prosecution of the '320 patent, argued that the term "a curable fluoroelastomer composition" in the claims makes it clear that it is not the final cured material which is claimed but the composition which is subsequently cured and that inventor Kolb recognizes similarity in properties of his composition after curing with other good cured fluoroelastomers (FF 79). There is also not even a disclosure in the '320 patent of a specific utility of the cured product obtained from the claimed curable composition.[4]

Independent claim 1 of the '320 patent is also a combination claim to a

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[4] The utility of cured fluoroelastomer compositions is well known in the art. For example, the Bowman '143 patent cited by the Examiner in the prosecution of the '320 patent (FF 57) teaches that cured highly fluorinated elastomers have found application in areas such as high temperature resistant gaskets, seals, diaphragms and tubing (FF 61).

curable fluoroelastomer consisting essentially of specific amounts of an elastomeric co-polymer of vinylidene fluoride and terminally ethylenically unsaturated fluorinated comonomer <u>and</u> of (a) a particular diorganosulfur oxide,[5] (b) a quaternary phosphonium or ammonium compound, (c) acid acceptor and/or base acceptor, and (d) an aromatic hydroxy or amino compound (FF 53). The '320 specification teaches that the use of the particular diorganosulfur oxide in the multicomponent claimed composition "frequently" allows reduction in the amount of calcium hydroxide with both improved rate of cure and retention of desirable physical characteristics as compared to the "presently" known formulations (FF 77). However, the '320 specification also discloses that the use of the particular diorganosulfur oxide may provide self-lubricating characteristics leading to relatively low pressure extrusions with excellent surface finish and provide moldings which are readily removed from the molds and that mold release agents may not be required the claimed composition (FF 77).

The '320 specification in Tables 3 and 4 further provides a comparison of the claimed compositions, which included the particular diorganosulfur

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[5] The Patent Office Board of Appeals, in reversing the Examiner's rejection of the claimed subject matter over a Schmiegel patent corresponding to U.S. Pat. No. 3,933,732 (the '732 patent), did find that "all aspects" of the claimed curable composition are in the prior art except for the dioragnosulfur oxide which is recited in the claimed subject matter and which is limited to a diorganosulfur oxide "which contains two like or unlike aliphatic, cycloaliphatic or aromatic groups, which are unsubstituted or substituted only with halogen" (FF 85). Inventor Kolb and complainant's expert Worm testified that Kolb's '320 patent is not based on making a different cure system but instead is to an improvement of the existing bisphenol-phosphonium type cure systems (FF 51. <u>See also</u> FF 311, 312). However, the administrative law judge finds persuasive expert Worm's testimony that one of the significant aspects of the process of using the diorgano sulfur oxide, as developed by Kolb in his '320 patent, is that it is an additive which does not significantly affect the fluoroelastomer system in typical use (FF 104).

oxide recited in independent claim 1 with an identical composition without any diorganosulfur oxide (i.e. run 9 which was a control run). It was concluded in the '320 patent specification that control run 9 required three (3) minutes press cure in commercial equipment, whereas runs 10 through 14 (runs according to the claimed curable composition) require one (1) minute (FF 65). Runs 10, 11, 12, 13 and 14 have, respectively, a 5.0, 4.1, 4.5, 4.5 and 4.9 time to 50 lb. rise (min.) while the control run 9 has a 7.4 time to 50 lb. rise (min.) (FF 64). Run 15, which was also according to the claimed curable invention, but not referenced by inventor Kolb, and uses bis (p-chlorophenyl) sulfone (FF 64) which Ausimont uses in its accused compositions (FF 14, 15, 18 to 23, 25, 26, 29, 33, 36, 37,) had as much as a 6.8 time to 50 lb. rise (min.). The '320 specification teaches also that run 16 in Tables 3 and 4, which is according to the claimed composition and thus contains all the components of the claimed composition, including the particular diorganosulfur oxide within the amounts claimed, "requires 3 minutes [in press cure as did control run 9 with no diorganosulfur oxide] but the reduction in amount of phosphonium chloride result in improved properties" (Emphasis added) (FF 65). Thus the administrative law judge finds that the '320 specification teaches to one skilled in the art that the claimed composition, depending on the specific amounts of the particular components within the range specified in the claimed composition, may or may not result in a reduction of press cure time in commercial equipment, although improved properties do result.

While Kolb's agent argued, during the prosecution of the '320 patent, that the novelty in the claimed invention arises from the use of neutral diorgano sulfur oxides which results in a more rapid cure, and relied on Table 4 of the '320 patent (FF 63), Table 4 also has run 16, which is to a claimed curable composition containing tetramethylene sulfone and which showed no increase in press cure in commercial equipment in comparison with control run 9, which was to a curable composition containing no diorganosulfur oxide (FF 65). In addition, Kolb's agent in the prosecution of the '320 patent argued that the diorganosulfur oxides employed by Kolb in the claimed curable composition are "cure accelerators or processing aids"[6] (FF 77), and that a Schmiegel patent, cited by protester DuPont, does not suggest that Kolb's diorganosulfur oxides are processing aids which improve mold release and surface finish (FF 79). Moreover, Kolb's agent in prosecution of the '320 patent relied on page 22, line 22-28 of Kolb's patent application which, inter alia, showed that Run 16 pursuant to the claimed invention required the same amount of time for press cure as control run 9 which related to a composition containing no diorganosulfur oxide (FF 79, 80).

Based on the foregoing, the administrative law judge finds that while the particular diorganosulfur oxide in the claimed curable composition of a combination of components in issue frequently allows for an improved rate of cure, an improved rate of cure is not a necessary limitation inherent in the claimed curable composition.

2. The Components Of The Claimed Composition and The Accused Compositions

With respect to the components of the accused compositions, those components are set forth in the findings (FF 14, 15, 18 to 23, 25, 26, 29, 33, 36, 37). Referring to the introductory portion of independent claim 1 (FF 53)

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[6] Ausimont argued that the "accused compositions ... contain small amounts of dichlorodiphenyl sulfone as a processing aid" (RB at 24). There is expert testimony that before the Kolb '320 patent most process aids affected properties, as compression set, of the final cured product while the dioganosulfur oxide of the Kolb patent does not (FF 308). A process aid is helpful in the compounding of fluoroelastomers (FF 303 to 307)..

the findings show that the each of the accused compositions is a fluoroelastomer composition consisting essentially of an elastomeric copolymer of vinylidene fluoride and terminally ethylenically unsaturated fluorinated comonomer, viz. hexafluoropropene. As to components a, c and d of claim 1, (FF 52) the findings show that within the ranges specified in independent claim 1 each of the accused compositions contain (a) dichlorodiphenyl sulfone which is a diorganosulfur oxide that contains two aromatic groups which are substituted only with a halogen (component a of claim 1) and which is specifically claimed in dependent claim 11 of the '320 patent and (d) Bisphenol AF (component d of claim 1). Also Ausimont recommends that their customers add calcium hydroxide and magnesium oxide (component c of claim 1) to the commercially available product within the amounts specified in independent claim 1 (FF 32, 332 to 345) and Ausimont U.S.A. has supplied accused compositions containing calcium hydroxide and magnesium oxide to customers (FF 321 to 331). Remaining for consideration, under the doctrine of equivalents, is component b of independent claim 1, viz. the quaternary phosphonium compound.

a. The Functions Of The Quaternary Phosphonium Compound In Claim 1 And Of GM 102 E And XA-51 In The Accused Compositions

The administrative law judge finds that the record supports complainant's argument that in all of the accused compositions with the exception of one, viz, FOR-423, Ausimont uses benzyl diethylamino diphenyl phosphonium chloride, viz. GM 102E, which material differs from triphenyl benzyl phosphonium chloride which is employed as the quaternary phosphonium compound in examples of the '320 patent in that GM 102E has

Substituted for the GM 102E in the accused FOR 423

is

Acelerator technology for curable fluoroelastomers[7] was developed at least in the 1960's and early 1970's (FF 107). For example accelerators for the vulcanization of saturated, fluorinated polymers were referred to as enabling a more rapid cure with conventional vulcanization systems in the U.S. Patent No. 3,502,628 (the '628 patent) which issued on March 24, 1970 and has an initial filing date of August 17, 1967 (FF 110). <u>See</u> also the '143 patent which is based on an initial application filed on April 11, 1969 and states that catalysts which accelerate vulcanization or the curing of elastomers are generally referred to as "vulcanization accelerators" (FF 61).

The '320 patent does not specifically disclose the function of the quaternary phosphonium compound. However, it does disclose that a curing system based on quaternary ammonium derivatives has been developed in U.S. Patent No. 3,655,727 (the '727 patent). The '320 patent discloses that although the curing system in the '727 patent allows safe milling at 90°-120°C and molding temperatures in the range of 160°-170°C with good flow and a short cure cycle, cure is so rapid at the curing temperature that the stock tends to be "scorchy" requiring careful handling; and that to a considerable extent those difficulties are overcome by use of the quaternary phosphonium curing system based on the presence of a compound in which the phosphorus atom

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[7] It was known at least by November 30, 1961 that a fluoroelastomer copolymer of vinylidene fluoride with other fluorinated monomers as hexafluoropropene (perfluoropropene), which is specifically recited in dependent claim 2 in issue of the '320 patent (FF 54), was curable (FF 109).

is covalently bonded to an anion as described in U.S. Pat. No. 3,712,877 (the '877 patent) (FF 96). It also disclosed that the quaternary ammonium compounds useful in preparing curable fluoroelastomer compositions are described not only in the '727 patent but also in U.S. Patent No. 3,752,787 (FF 97).[8]

The '727 patent to Patel and Maier which was based on an initial application filed on April 19, 1968, and issued on April 11, 1972, discloses a fluorinated elastomeric copolymer composition curable in reactive association with an inorganic acid acceptor capable of generating water upon reacting with hydrogen fluoride to produce a cured elastomer comprising (a) the fluorinated elastomer copolymer, (b) at least one quaternary ammonium or quaternary phosphonium compound where the phosphonium compound has a formula showing a cation and an anion and wherein the phosphorus atom in the cation is covalently bound to four specified alkyl radicals, and (c) at least one aromatic hydroxy or amino compound which has a certain oxidation potential. The '727 patent teaches that the quaternary phosphonium compound may be premixed with a suitable amine and when used in amounts as small as 0.05 pphr., produce a measurable <u>acceleratory</u> effect on cure rate at a given amine concentration. The claims of the '727 patent are limited to a curable composition containing (a) an elastomeric vinylidene fluoride copolymer, (b) at least one quaternary ammonium compound of a particular formula and (c) at least one particular aromatic hydroxy or amino compound (FF 57 to 60). Claim 1 of U.S. Patent No. 3,752,787 (the '787 patent), which is based on an initial

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[8] Independent claim 1 in issue discloses the alternative use of a quaternary phosphonium compound and a quaternary ammonium compound. Thus component b of claim 1 reads "0.1 to 5 parts quaternary phosphonium or ammonium compound" (FF 53).

application filed on June 9, 1970, and which issued on August 14, 1973 has a claim merely directed to a fluoroelastomer composition comprising (a) an elastomeric copolymer of vinylidene fluoride and at least one other fluorinated monomer, and (b) as a vulcanization accelerator, a triarylphosphorane compound. However, it teaches that cross-linking of highly fluorinated polymers with aromatic polyhydroxylic compounds must be carried out in the presence of a catalyst which catalysts accelerate vulcanization or curing of elastomers and are generally referred to as "vulcanization accelerators" (FF 130).

U.S. Patent No. 3,876,654 (the '654 patent), which is based on an initial application filed June 9, 1970, and which issued on April 8, 1975 discloses that for many applications of highly fluorinated elastomers the resultant fluoroelastomer articles must be resilient and have low compression set which is accomplished by vulcanizing or cross-linking the elastomer and that cross linking of highly fluorinated polymers with aromatic polyhroxylic compounds must be carried out in the presence of a catalyst, i.e. a vulcanization accelerator viz. a quaternary phosphonium compound which accelerates vulcanization or curing of the elastomer, are directed to a curable fluoroelastomer composition comprising the fluoroelastomer, the quaternary phosphonium compound "as a vulcanization accelerator," a divalent metal oxide and a polyhydroxylic-aromatic compound cross-linking agent for the fluoroelastomer (FF 190 to 194).

The Schmiegel '732 patent, which was cited by protester DuPont in the prosecution of the '320 patent (FF 74), and has an initial filing date of December 27, 1971 discloses as a suitable accelerator for curing fluoroelastomer composition a quaternary phosphonium compound wherein four carbon containing radicals are covalently bonded with a preferred compound such as benzyl triphenyl phosphonium chloride (FF 75, 76), which compound is specifically recited in claim 13 of the '320 patent (FF 55). The same Schmiegel, who obtained the '732 patent, presented a paper in Germany in 1978, which paper was later published in 1979 (FF 189, 195 to 198). The paper has been extensively cited in scientific articles by complainant and Ausimont (FF 219, 225, 227, 232, 237, 239, 240, 254). The Schmiegel paper, titled "Crosslinking of Elastomeric Vinylidene Fluoride Copolymers with Nucelophiles," presented data involving a curing system for vinylidene fluoride hexafluoropropylene copolymers consisting of vinylidene fluoride copolymer, carbon black, calcium hydroxide magnesium oxide, benzyltriphenyl phosphonium chloride (FF 189, 195 to 198) which Schmiegel characterized as an accelerator in his '732 patent, and bisphenol AF (FF 75, 76). Schmiegel, in his paper referring to the '654 patent, disclosed that the vinylidene fluoride based fluoroelastomers are generally vulcanized by basic curatives (FF 189 to 198). Schmiegel's data in his paper showed that while the bisphenol AF in the curing procedure became attached to the polymer chain the phosphonium compound accelerates the curing although it does not become a part of the polymer as does the bisphenol AF. Based on the foregoing patents and Schmiegel article, the administrative law judge finds that the evidence supports a finding that quaternary phosphonium compound in the claimed curable composition in issue serves as an accelerator when used with a bisphenol in the vulcanization of a fluoroelastomer (FF 189 to 198).

Ausimont, in challenging the staff's proposed finding that the quaternary phosphonium compound (component b of claim 1) functions as an accelerator or catalyst for curing fluoroelastomers (SFF 107) argued that the

Patel and Maier '877 patent suggest that in the claim in issue, the quaternary phosphonium compound is the cross-linker and the biphenyl acts as an accelerator (RRS 107(a)).

The '877 patent cited by Ausimont in RRS107(b) which also issued to Patel and Maier and which is based on the same initial application filed on August 19, 1968 as the '727 patent, has claims only drawn to a curable vinylidene fluoride composition comprising a particular fluorinated elastomeric copolymer of vinylidene fluoride and at least one quaternary phosphonium compound having at least one phosphorus atom covalently bonded through carbon-phosphorus single bonds to certain organic radicals. The '877 patent discloses that the quaternary phosphonium compounds are useful in preparing curable fluoroelastomer compositions and, although useful vulcanizates can be obtained using the quaternary phosphonium compounds alone "as curative," it is frequently desirable to use in addition an accelerator, i.e. a material which significantly increases the rate of cure under curing conditions without unduly accelerating the rate of cross-linking during mixing and milling (FF 108). The '877 patent excludes quaternary ammonium compounds and its claims merely recite the fluorinated elastomeric copolymer and the quaternary phosphonium compound. The claims of the '877 patent do not recite any aromatic hydroxy or amino compound as did the claims of the '727 patent. In issue in claim 1 of the '320 patent is a curable composition that containing a combination of compounds one of which must be an aromatic hydroxy or amino compound (FF 53).

With respect to the function of GM 102E and XA-51, Ausimont developed its P-N based curing system and filed for patent protection in 1977 (RBR at 24, FF 200). It was successful in obtaining Moggi U.S. Patent No. 4,259,463 (the '463 patent) which issued on March 31, 1981 (FF 200 to 215). The Moggi patent disclosed that the vulcanized elastomers based on vinylidene fluoride copolymer are well known to the prior art and that according to the most advanced prior art for the vulcanization of the elastomeric copolymers of vinylidene fluoride, polynucleophilic compounds, and in particular polyhydroxylic aromatic compounds (which bisphenol AF is) are used as vulcanizing agents, but that while such products lead to absolutely satisfying physical-mechanical characteristics and altogether satisfy thermal resistance, they have the disadvantage of requiring long vulcanizing times, wherefore "they are used in combination with substances having an accelerating action" (FF 200 to 215). The '463 patent discloses that the substances that develop an accelerating action according to the most advanced prior art include those described derivatives of tertiaryphosphines containing four covalent phosphorus-carbon linkages, citing French Patent No. 2,096,115 (FF 202) which is based on a U.S. priority application which led to the issuance of the Pattison '654 patent (FF 200 to 215). Moggi discloses that he has found that some compounds containing one or more simple phosphorus-nitrogen linkages can be used as "vulcanization accelerating agents for elastomers," and discloses preferred classes of products to be used "according to the invention as accelerators," and Tables 1, 4 and 5 refer to the use of accelerators according to the Moggi invention (FF 200 to 215). Independent claim 1 of the '463 patent is to a vulcanizable composition consisting of a plurality of components, one of which is a vulcanization accelerator, <u>viz</u>. an aminophosphinic derivative of a formula which

Ausimont's Tommasi has referred to a "new class of catalysts (accelerators) for the bisphenol AF curing system [for fluoroelastomers]" illustrated by GM

102E (FF 199).

Ausimont's U.S. Patent No. 4,544,708 (the '708 patent) discloses, as a vulcanization <u>accelerator</u> for curing fluoroelastomers, a cation of phosphonium or of amino-phosphonium which formula thereof is "selected amongst those already known in the art and broadly described for example in .... [the '654 and '463 patents]" (FF 216, 217).

Ausimont's U.S. Patent No. 4,612,351 (the '351 patent) discloses vulcanizable fluoroelastomer compositions providing vulcanizates having high adhesion to metals wherein the vulcanization <u>accelerator</u> is a salt composed of a cation of phosphonium as described in the Pattison '654 patent or the cation of an amino-phosphonium as described in the Moggi '463 patent with a particular counteranion (FF 218). Hence the '351 patent not only recognized that the salt of a phosphonium cation, as disclosed in the '654 patent, which specifies quaternary phosphonium compounds utilized in the '320 patent, is an accelerator in the vulcanization of fluoroelastomer but equated, as accelerators, the phosphonium cation as disclosed in the '320 patent with the phosphonium cation as disclosed in the Moggi '463 patent.

An Ausimont's 1984 presentation, a February 1987 publication, and a 1986 Ausimont research report disclose a phosphonium compound containing a phosphorus-nitrogen bond as an accelerator in the vulcanization of fluoroelastomers (FF 219 to 224). An Ausimont 1985 research report equates the quaternary phosphonium compound of the '320 patent with GM 102 E "as an accelerant" in the vulcanization of fluoroelastomers (FF 225). Another Ausimont 1985 report refers to "Accelerant system GM 102E" in the vulcanization of fluoroelastomers (FF 226). An Ausimont 1988 report discloses that the MF accelerator (GM 102E) is more efficient than a quaternary phosphonium compound of the '320 patent (FF 237). Moreover, Ausimont's Tommasi testified that Ausimont's internal research reports stated that the "P-N" compounds can be used as accelerator agents in the crosslinking reaction in fluoroelastomers (FF 245). In addition, an Ausimont 1989 report equates

U.S. Patent No. 4,912,171, which issued on March 27, 1990, equates amino phosphonium compounds, as described in the Moggi et al. '463 patent, with quaternary phosphonium compounds as described on the Patel et al. '727 patent, as capable of functioning as a vulcanization accelerator (FF 160, 161).

In view of the foregoing, the administrative law judge finds that the evidence supports a finding that GM 102E

in the accused compositions containing fluoroelastomers function as accelerators, as does the quaternary phosphonium compound of the '320 patent.

b. The Way The Quaternary Phosphonium Accelerator And The Way The Ausimont Accelerators Function

Ausimont argued that the accused compositions function in a different way. At the outset the quaternary phosphonium compound included in claim 1 of the '320 patent and GM 102E are similar with respect to structure (FF 181 to 187). Moreover, several U.S. patents relating to curable fluoroelastomer compositions group quaternary phosphonium compounds and compounds illustrated by GM 102E together as accelerators for curing fluoroelastomer compounds containing a bisphenol (FF 188).

The '727 patent which is based on an initial application filed on August 19, 1968, disclosed at that time that the reaction mechanism for curing vinylidene fluoride elastomers, which involved the use of a quaternary phosphonium compound as an accelerator, was not fully understood; that existing evidence suggests that the initial press cure involved a base-catalyzed release of hydrogen fluoride to generate double bonds in the polymer, which double bonds then provide a limited number of crosslinks between the polymer chains that serve to stabilize the shape and form of the polymer while a subsequent cure step results in the formation of further ethylenically unsaturate structures which combine to form benzenoid crosslinks, acting to aid in the release of hydrogen fluoride (FF 59, 60).

The '654 patent which is based on an initial application filed on June 9, 1970 involved curing a fluoroelastomer in a solid phase state polymerization[9] using as a vulcanization accelerator a quaternary phosphonium compound and in the presence of a dwalent metal oxide and a polydroxylic aromatic compound cross-linking agent for the fluoroelastomers (FF 190, 192 to 194). It disclosed, however, that the exact nature "is not yet known of the chemical reaction involving the accelerator during curing" (FF 194). Likewise, a '787 patent also based on an initial application filed on June 9, 1970 (FF 136) and involving a reaction, as in the '654 patent, indicated that the exact nature of the chemical reaction involving the accelerator during curing is not known (FF 136).

In 1978 Schmiegel, who had earlier obtained the '732 patent presented a paper in Germany, which was later published in 1979, which involved the crosslinking of elastomeric vinylidene fluoride copolymers with nucleophiles[10]

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[9] Solid phase means that all of the ingredients of a reaction mixture are in a solid phase.

[10] Nucleophiles are oxidizable aromatic hydroxy or amino compounds (FF 108). A bisphenol is an aromatic hydroxy compound (FF 283).

(FF 189 to 198). What Schmiegel did was to investigate the reactions of basic nucleophiles with vinylidene fluoride copolymers in solution and therefrom determine some reactivity principles which could be used to interpret the vulcanization behavior of related fluoroelastomers <u>such as those disclosed in the '654 patent</u> (FF 189 to 198). Thus it was the purpose of Schmiegel's presentation to consider the reactions of normal vinylidene copolymers toward basic nucleophiles in solution and "to develope some structure-reactivity generalizations which can be compared to experience with <u>practical</u> vulcanization" (FF 196, 189 to 195, 197, 198) (emphasis added).[11]

In his study Schmiegel employed a curing system consisting of a vinylidene fluoride copolymer, carbon black, calcium hydroxide magnesium oxide, a quaternary phosphonium compound and bisphenol AF (FF 189 to 198). In a section of the study titled "Behavior of Polymer Solutions Toward Basic Bis-Nucleophiles" he demonstrated through solution polymerization studies in a system containing a fluoroelastomer copolymer, a bisphenol and a phosphonium cation, <u>viz</u>. positively charged "R_4P"[12] which was originally present as a phosphonium chloride and which Schmiegel in his '732 patent disclosed was an accelerator in the vulcanization of fluoroelastomer (FF 189 to 198), that phenols actually become attached to the polymer chains. Schmiegel disclosed that because of that attachment of the mono-hydroxy analog of bisphenol-AF to the vinylidene copolymer, and in view of the nucleophilic attack of hydroxide

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[11] Schmiegel does disclose that it appears that a substantial different and inferior kind of crosslinked network is obtained in the curing of fluoroelastomer copolymers when a quaternary phosphonium compound is used in the absence of a bisphenol (FF 189 to 198). However, each of the claimed composition and the accused compositions contain a phosphonium compound <u>and</u> a bisphenol (FF 14, 15, 18 to 23, 25, 26, 29, 33, 36, 37, 53).

[12] It is common practice that "R" is used for a carbon bonded moiety (FF 116, 117).

ion on the unsaturated polymer, one can conclude that in "practical vulcanization" a bisphenol-derived phenolate probably also attacks the intermediate diene and leads to dienic phenyl ether crosslinks; that attack on the diene by phenoxide, unlike attack by hydroxide, is not expected to proceed beyond vinylic nucleophilic substitution and that therefore the expected product is the phenylether that corresponds to the dienol proposed as an intermediate in attack by the hydroxide; that the phosphonium ion of the quaternary phosphonium compound, originally present as a chloride, is believed to undergo several cycles of conversion from fluoride or bifluoride to intermediate hydroxide to phenoxide to fluoride before exhaustion of the bisphenol; and that ultimately the phosphonium ion of the quaternary phonium compound is converted to triphenylphosphine oxide (FF 189 to 198). Schmiegel proposed in his paper that the bisphenol/phosphonium complex was responsible for the initial dehydrofluorination of the fluoroelastomer, which forms double bonds in the fluoroelastomer. He further proposed that due to the reactivity of the phenoxide complex, the dehydrofluorination and eventually the crosslinking, only occurred at selective sites on the polymer backbone, specifically sites where a vinylidene fluoride moiety was surrounded by two hexafluororopropene moities (FF 189 to 198).

Schmiegel's study presented in Germany in 1978 has been accepted by other scientists in the fluoroelastomeric field. Dr. Pothapragada Venkateswarlu (FF 253) in a study reported in 1989 and with coauthors R. E. Kolb, R. A. Guenthner and T. A. Kestner did refer to a 1982 publication of S. Smith which was said to characterize the scheme of crosslinking reactions of Schmiegel as based on an analysis of early stages of the vulcanization and to conclude that the later stages of the crosslinking reactions, when some crosslinking has rendered the polymer insoluble, are much more difficult to study, with further work needed to uncover details of the reactions which occur. It was precisely toward those goals that the efforts in the Venkateswarlu et al. study was addressed. Venkateswarlu et al. reported that the method developed in their study has been used to uncover and elucidate certain chemical events occurring during the curing of a fluorocarbon elastomer with bisphenol AF and using a quaternary phosphonium compound "as the phase transfer catalyst" as well as calcium hydroxide and magnesium oxide as acid receptors and cure activators. Significantly, Venkateswarlu et al. reported that their work "complements the earlier pioneering work done by Schmiegel" (FF 254 to 265). Thus the Venkateswarlu et al. paper indicated that in the initial dehydrofluorination in the curing of a fluoroelastomer using a bisphenol crosslinking agent and a phosphonium ion with a base the initial dehydrofluorination occurs by way of the phosphonium/bisphenol complex; and that the bisphenol is then attached to polymer chains at double bond sites created by the dehydorfluorination (FF 254 to 265).

In addition to the Schmiegel and Venkateswarlu studies, complainant's expert Worm gave testimony, which the administrative law judge finds persuasive. Thus he testified, consistent to what is shown in the Schmiegel 1978 paper, that fluoroelastomers are cured through a vulcanization process involving the generation of cross-links through a curative; that in the products in issue sold by complainant and Ausimont, the crosslinking agent utilized to form the bridge is Bisphenol AF; that in the presence of a phosphonium compound and a base, Bisphenol AF is involved in a dehydrofluorination of the fluoroelastomer, i.e. elimination of hydrogen fluoride from the fluoroelastomer; that the Bishpenol AF then adds to the reaction site where the hydrogen fluoride was eliminated to become attached to the fluoroelastomer chain with the Bisphenol AF reacting at each of its ends to form a bridge (cross-link) between two chains of fluoroelastomer (FF 99); that Bisphenol AF however does not react very rapidly with a fluoroelastomer, unless an accelerator is present because Bisphenol AF is not very soluble in the fluoroelastomer and cannot migrate freely to the potential reactive sites of the fluoroelastomer (FF 99); that when the phosphonium compound of the '320 patent is provided in the fluoroelastomer polymer mixture, in the presence of bisphenol AF and base, an association between the phosphonium cation of the phosphonium compound and the bisphenol AF anion takes place; that the phosphonium cation is mobile in the polymer phase, and as it moves through the polymer phase it can also transport the associated bisphenol AF anion through the polymer moving through the polymer phase and into locations where reaction is probable and crosslinking can occur; that the phosphonium cation operates as a catalyst, so it can recycle repeatedly and move a number of bisphenol AF anions to the polymer phase for reaction; and that while the mechanistic details of the vulcanization reaction, at a molecular level, are not fully understood, such a characterization is a widely accepted model for picturing the operation of the phosphonium compound of the '320 patent (FF 99, 101). As complainant's expert Grootaert testified, phase transfer catalysis is the transfer of one component from one phase into a second phase and only involves bringing reactants together for a later reaction, <u>i.e.</u> only involves a migration (FF 292, 293).

In addition, complainant's expert Harwood gave testimony, which the administrative law judge also finds persuasive, that a fluoroelastomer is a rubber, which is a very, very viscous liquid, and that its molecules can thus move; that when one wants to carry out a reaction between two phases, and one component of the reaction is soluble in one of the phases, which is inorganic, and the other component is soluble only in the remaining organic phase, it helps to promote the reaction between the two phases if the component soluble in the inorganic phase can be brought into the organic phase, which is the role, as phase transfer catalyst, of the phosphonium compound of the '320 patent; that phase transfer catalysts are really nothing more than solubilizing materials for a medium one wants to work in; that a phase transfer catalyst takes ions from an interface of one phase and brings the ions into another phase so reaction takes place, and this is what happens when a fluoroelastomer is cured using a bisphenol; that initially the bisphenol reacts at the surface of a base, such as calcium hydroxide, and is neutralized to become the bisphenol anion; that if there was nothing around to take the bisphenol anion into the fluoroelastomer copolymer phase there would be no reaction of the bisphenol anion with the fluoroelastomer; that the phase transfer catalyst brings the bisphenol anion into the viscous liquid fluoroelastomer and reaction occurs between the bisphenol anion first through dehydrofluoronation of the polymer and then to establish a bisphenol crosslink structure; that the phase transfer catalyst also brings the resulting fluoride ion, from the dehydrofluorination, to the inorganic phase; and that phase transfer in organic chemistry facilitates a reaction that occurs across phases and is basically predictable technology (FF 100, 102).

Harwood, in persuasive testimony, testified that the phosphonium salt is not connected to the fluoroelastomer to form crosslinks when Bisphenol AF is present; that the phosphonium compound is required for crosslinking because it is a phase transfer catalyst; that the type of curing for example that occurs in the second curve in Figure 2 of Schmiegel's 1978 presentation with the triangles shows that the phosphonium salt brings the hydroxide up to the fluoroelastomer backbone to cause dehydrofluorination which then allows cross-linking of the fluoroelastomer to occur even <u>in the absence of Bisphenol AF</u> to form an inferior kind of crosslinked network but that with Bisphenol AF present, the phosphonium salt is only a carrier catalyst for causing dehydrofluorination and unsaturation which are necessary before any crosslinking takes place through the Bisphenol AF to form the crosslinks (FF 281, 282).

In connection with the curing of a fluoroelastomer composition, Ausimont's scientists in 1984 reported that the vinylidene fluoride copolymers seems to be crosslinked by a three step process; that in the first step hydrogen fluoride is eliminated from the polymers upon treatment with bases; that the second step involved addition of the curing agent to the site of unsaturation; and the final step in the cure involved the thermal formation of additional unsaturations and subsequent crosslinking (FF 219 to 224). Those three steps are as Schmiegel disclosed in his 1978 paper, <u>viz</u>. initial dehydrofluorination, attachment of the bisphenol curing agent to the site of unsaturation and the thermal formation of additional unsaturations and subsequent crosslinking (FF 189 to 198).[13] Ausimont scientists in 1984 acknowledged that the cure chemistry and mechanism was investigated by Schmiegel (FF 189 to 198, 219 to 224), although they suggested that cross-

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[13] Ausimont's Tommasi agreed that Ausimont's scientists in 1984 confirmed that in the curing of a fluoroelastomer the bisphenol links that polymer chains together, stating that "[y]ou have to crosslink bisphenol" and further agreed that the Ausimont scientists in 1984 did not say that the phosphonium cation they reported doesn't work the same way as Schmiegel in his 1978 presentation although he testified that the unsaturation sites on the fluoroelastomer are different than what is disclosed in Schmiegel (FF 224).

linking occurred at random vinylidene fluoride moieties rather than only at those sites surrounded by hexafluoropropene moieties (FF 219 to 224). In a March 20, 1985 report Ausimont's Arcella relied on the Schmiegel mechanism to explain vulcanization chemistry (FF 225). It is indicated that the crosslinking can be explained "in the presence of a phosphonium salt (C 20 [which is a DuPont product containing triphenyl benzyl phosphonium chloride and which is recited in claim 13 of the '320 patent], GM 102 E, etc.) and similar molecules." Arcella further acknowledged that Schmiegel carried out curing tests with accelerators <u>alone</u> and from the compression set of the vulcanizate concluded that the accelerator generated a "poor" network and therefore it is necessary to use it in amounts barely sufficient to promote crosslinking.[14] (FF 225).

Ausimont researchers in another report in April 1985 described a mechanism for the curing of a fluoroelastomer composition as essentially what Schmiegel had proposed in 1978 (FF 226). Ausimont's scientists in the introduction of a further report dated May 15, 1986 (FF 227, 228) stated that amino-phosphonium or phosphoranamine derivatives are characterized by having the structure of "onium" salts and by the presence of one or more phosphorus-nitrogen bonds that the industrial importance of those class of compounds resides in the fact that they act as accelerator agents <u>in the crosslinking reaction that results in the formation of cross bonds between the fluoroelastomer chains</u>, relying on the Schmiegel 1978 reaction mechanism; that the analogy noted between active catalysts in <u>phase transfer catalysis</u> and accelerator compounds from "this bisnucleophile

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[14] Neither the accused compositions nor the claimed composition in issue caries out the curing with only accelerators (FF 14, 15, 18 to 23, 25, 26, 29, 33, 36, 37, 53).

reaction led, on the one hand, to close examination in the system in question of the relations between the structure and catalytic activity of the aminophosphonium compounds and, on the other, to evaluation of their activity in certain typical reactions carried out in phase transfer catalysis;" and that the accelerant action which indeed showed a strict parallel with phase transfer catalysis seemed to occur in both passages of the crosslinking reaction, which can be diagramed as a dehydrofluorination and an attack by a bisnucleophile agent with two dehydrofluorinated polymer chains. The scientists stated that a modified and simplified version of the pattern of reactions is found in the 1978 Schmiegel presentation. Ausimont's Tommasi at the hearing testified that the May 15, 1986, report talks about the importance of compounds with phosphorus nitrogen bonds because they can act as accelerator agents in the crosslinking reaction in fluoroelastomers; that the report suggests that the accelerator and the crosslinking reaction shows a parallel with phase transfer catalyst; and that Table 1 of the report is a simplified version of the Schmeigel proposed mechanism for the curing of fluoroelastomers (FF 228).

Ausimont scientists, in still another paper published in February 1987, stated that the most common vulcanization system for fluoroelastomers is based on formulations consisting of (a) inorganic bases, (b) bisphenol AF, (c) a "vulcanizing accelerator," generally a quaternary salt of ammonium or phosphonium and (d) filler; that according to the currently accepted mechanism the cross linking reaction consists of two steps: (1) polymer dehydrofluorination by the base to give double bonds in the backbone chain and (2) nucleophilic addition of bisphenol AF to the double bonds yielding crosslinks; and that the accelerator which has the structure typical of phase transfer catalysts is thought to act as the cation of the base and/or bisphenate making them able to diffuse through the rubber although it concluded that the reaction pattern in the vulcanization of fluoroelastomers "seems to be more complex than that suggested by Schmiegel" (FF 231 to 236). However, the reference to polymer dehydrofluorination and nucleophile addition is consistent with the mechanism postulated by Schmiegel in his 1978 paper (FF 189 to 198).

A subsequent May 5, 1988 Ausimont report involving the vulcanization mechanism for Ausimont's TECNOFLON and the influence of the recipe on vulcanization and properties concluded that the data of the report is in good agreement with those obtained by Schmiegel in his 1978 presentation, although it represented that GM 102E is more efficient than the quaternary compound benzyltriphenylphosphonium chloride; and that the best network stability is obtained by a vulcanization recipe very similar to the standard (2 phr BISAF and 0.4 phr GM 102E) (FF 237). An Ausimont 1988 publication, consistent with the presentation by Schmiegel in 1978, stated that the cross-linking reaction of vinylidene fluoride-hexafluoropropene copolymers can be summarized in a three-step process: (1) base-induced dehydrofluorination producing polymer chain unsaturation, (2) primary network formation by reaction of unsaturated chains with a bisnucleophilic agent; and (3) ultimate network formation after heating (FF 239). The work was premised on homogeneous phase dehyrofluorination involving polymerization in solution, phase transfer catalysis dehydrofluorination also involving polymerization in solution and bulk dehydrofluorination (FF 239). It was pointed out that while the Schmiegel presentation found that either of two monomer sequences was the only selective base-sensitive site leading to the diene group in the vulcanization, two kinds of unsaturation were detected by the Ausimont publication. A 1991 paper of Ausimont scientists studied the cross-linking chemistry of vinylidene fluoride fluorocarbon elastomers by bis-nucleophiles and concluded that while the chemical reaction mechanism through which cross-linking develops in "practical vulcanization" is not completely understood since some conflicting conclusions have been drawn, the proposed chemical mechanism for cross-linking "agrees with most of the published data." Also it was concluded that formation of a particular double bond by elimination of tertiary fluorine agrees with the early findings of Schmiegel, who described the formation of the initial double bond region selectivity from base sensitive site (FF 240 to 244). In addition, the paper noted that investigations by Ausimont scientists confirmed the high selective dehydrofluorination of vinylidenefluoride units isolated between two hexafluoropropylene units "as previously reported by Schmiegel," which results "have been confirmed independently by Venkateswarlu" (FF 244). Ausimont's Tommasi testified that the emphasis of the Arcella paper was the particular location of the cure sites on the polymer backbone (FF 241). The report following solid state studies stated that results therefrom are in a very good agreement with the previously reported findings obtained by samples in solution and represent a good support to the proposed vulcanization mechanism and that the experimental conditions tested are very close to that of common practical vulcanization (FF 240 to 244).

An Ausimont 1989 report disclosed that

can be used in the curing of fluoroelastomers. It also confirmed that the results observed with adducts are consistent with what has been found many times in the case of fluoroelastomers using the "traditional system of separate accelerant and bisphenol" (FF 27, 28).

Ausimont's '463 patent, in commenting on the reaction mechanism of the disclosed accelerators containing the phosphorus-nitrogen linkage, disclosed that there forms in the vulcanization of the fluoroelastomer a compound whose action mechanism is still unknown but which presumably behaves as a ionic couple on the interface between an organic phase represented by the fluoroelastomer and an inorganic phase represented by charges of oxides and alkalinepearthy hydrates present in the vulcanization formula (FF 200 to 213).

Ausimont's scientist Chiodini (FF 229) sketched the scheme of the crosslinking of fluoroelastomers involving the use of GM 102E and bisphenol AF (FF 230), which scheme is similar to the reaction mechanism proposed in the Schmiegel 1978 paper (FF 230). She testified that GM 102E and the triphenyl benzyl phosphonium chloride (recited as the phosphonium compound in claim 13 of the '320 patent), would act "in the same way" as far as concerns the scheme that she sketched (FF 247 to 249). Moreover, she testified that the reaction mechanism is the same for all of Ausimont's accused compositions (FF 247 to 249). Chiodini has a doctorate degree in chemistry from the University of Milan and at Ausimont has been very much involved with GM 102E and the curing of fluoroelastomers (FF 229, 250).

In addition to the testimony of experts and scientists in the fluoroelastomer art, publications and Ausimont's own documents which support the finding that Ausimont's accelerators, like the quaternary phosphonium compound in the '320 patent, operate as phase transfer catalysts in the vulcanization of fluoroelastomer, complainant performed a series of experiments which further support this conclusion.

As a first part of complainant's experimental program the general characteristics of benzyl triphenyl phosphonium chloride, a quaternary phosphonium compound specifically recited in claim 13 of the '320 patent and thus specific to the generic term "quaternary phosphonium compound" recited in claim 1, was investigated by complainant's expert Engel. He reported that spectral properties indicative of a positive charge on the phosphorus atom was found. Moreover, comparing the phosphorus-NMR and proton NMR spectra on GM 102E to the analogous spectra of benzyl triphenyl phosphonium chloride, he found very little difference with respect to the positive charge on the phosphorus anion (FF 296).

To investigate whether there would be any difference between the two phosphonium cations of interest (i.e., benzyl triphenyl phosphonium cation and benzyl diethylamino diphenyl phosphonium cation), complainant conducted experiments to see if there was any difference in the two cations with respect to their respective abilities to form adducts or ion association with bisphenol AF anion. Complainant's investigations found, through synthesis of the compounds, that both materials readily form an adduct with the bisphenol AF anion (FF 284, 285, 292, 294).

As indicated, supra, in order for the fluoroelastomer vulcanization to take place, testimony of witnesses, published articles and research reports show that it is necessary that the bisphenol AF anion be transported from the inorganic phase through the organic material represented by the fluoroelastomer to the reaction sites on the fluoroelastomer which is the role of the phosphonium cation and which transport can be termed "phase transfer catalysis" because the material is moved between two phases i.e., the inorganic phase of the base and the organic phase of the fluoroelastomer. Thus complainant conducted several experiments comparing the operations of the benzyl triphenyl phosphonium cation (a cation of the accelerator of the '320 patent) and the benzyl diethylamino diphenyl phosphonium cation (the cation in the accused compositions) under phase transfer circumstances and found that in no instance was there any difference observed between the two compared phosphonium cations in their operation as phase transfer catalysts. The systems evaluated included an aqueous phase/organic phase transfer of a colored ion conducted by complainant's expert Harwood, an aqueous phase/organic phase transfer of bromide ion conducted by complainant's Grootaert and certain vulcanization experiments, involving a fluoroelastomer copolymer of vinylidene fluoride and hexafluoroprepene (FF 273) which is specific to claim 1 of the '320 patent (FF 53),[15] and which included the presence and absence of dichorodiphenylsulfone used in the accused compositions conducted under the direction of Harwood (FF 280).

Based on the foregoing, the administrative law judge finds that testimony of scientists in the relevant field, published material, internal Ausimont reports and experimental evidence support a finding that the phosphonium compounds of the claimed composition and of the accused compositions operate as phase transfer catalysts, and hence perform in substantially the same way when used with a bisphenol and base in the vulcanization of a fluoroelastomer.

c. The Results Obtained Using The Quaternary Phosphonium Accelerator And The Ausimont Accelerators

Ausimont argued that the accused compositions obtain a different result. The testimony on record, the published literature, Ausimont's internal research reports, and experimental evidence, supra, indicate that the

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[15] The accused compositions have copolymers of vinylidene fluoride and hexafluoropropene (FF 25, 274).

accelerators set forth in the '320 patent, as well as those used in the accused compositions, produce a fluorinated polymer that is crosslinked with bisphenol. Moreover, both the claimed composition in issue and the accused curable compositions produce a product that permits a manufactures to produce molded parts with good physical properties (FF 4). In fact, both curable compositions of complainant which are covered by independent claim 1 in issue are in competition with Ausimont's accused compositions (FF 42). Accordingly, the administrative law judge finds that the evidence supports a finding that the results, when using the quaternary phosphonium component accelerator and the Ausimont accelerators in the vulcanization of a fluoroelastomer in the presence of a bisphenol and a base are substantially the same.

d. Ausimont's Arguments

Ausimont has argued that the U.S. Patent Office in granting Ausimont's '463 patent to Moggi (FF 200 to 214) has recognized that Ausimont's amino-phosphonium type curing systems is patentably distinct from the "state-of-the-art" curing systems that Kolb had in mind when he filed the '320 patent and that and hence a finding of equivalency can not be justified (RB at 44,45). The Moggi '463 patent found that certain compounds containing one or more simple phosphorus-nitrogen linkages may be substituted for the quaternary phosphonium compound of claim 1 of the '320 patent and used as vulcanization accelerating agents for fluoroelastomers (FF 200 to 214). Under the doctrine of equivalents a patent obtained on a modification of a prior patent however does not necessarily preclude the modification from being considered an equivalent of the invention previously patented. <u>Atlas Powder</u>, 750 F.2d at 1581, 224 USPQ at 417.

Ausimont argued that its expert witness, Jerry Leyden, concluded that

Ausimont's rebuttal tests established that Ausimont's dichlorodiphenyl sulfone, which is specifically recited in claim 11 of the '320 patent as the dioragnosulfur oxide, had no effect on the cure rate of Ausimont's compositions while the diorganosulfur oxide in the claimed compositions somehow facilitates the curing action of quaternary phosphonium compounds with the aromatic hydroxy compound and the copolymer in the claimed compositions. Hence Ausimont relying on Spectra, 839 F.2d at 1579, 5 USPQ2d at 1867, argued that any equivalency cannot be justified. (RB at 45-46).[16]

In Spectra the claim in issue recited a developer consisting of particles of magnetic material in a mixture of at least one polymer with a wax and at least one sublimable or vaporizable dyestuff which release the vapors of said dyestuff if heated at the sublimation or vaporization temperature of said dyestuff. The parties in Spectra agreed that in order for complainant Spectra to prove literal infringement Spectra would have to show that the accused toners contained four components: magnetic particles, sublimation dye, wax and polymer. The Federal Circuit, in referring to Spectra's argument that there was a genuine issue of material fact as to infringement under the doctrine of equivalence and that the binding function of the polymer in the patent in issue in Spectra is performed by the wax in the accused toner, asserted that such fact, if true, is immaterial because, as admitted in Spectra's brief, "What distinguishes the toners claimed . . . is not polymer,

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[16] Complainant argued that Ausimont's alleged defense involving Ausimont's "rebuttal tests" referred to by Leyden was first raised during complainant's deposition of Pizzi in Italy on August 26, 1994 and that Ausimont should not be permitted to rely on those tests since Ausimont has "deliberately withheld production of these documents during discovery" (CB at 52). Complainant however filed no motions for additional discovery, for extension of the discovery date and/or for sanctions. Hence complainant's argument that Ausimont should not be permitted to rely on what complainant has characterized as "rebuttal tests" is rejected.

but rather <u>wax</u>" (Emphasis in original). The Federal Circuit also stated that Spectra's brief further says "The patent in suit teaches the use of wax in the coating to overcome [the polymer's] affinity . . . " and thus to avoid the "danger that the polymer would have too strong an affinity for the dyestuff, interfering with the release of the dyestuff"; that when the wax is removed the wax can no longer perform the function assigned it in the patent, namely the amelioration of the polymer's affinity for the dyestuff; and that if, as Spectra says, the wax in the accused toner performs the binding function of the polymer in the claimed invention, there remains nothing in the accused toner to perform the function of the wax in the claimed invention. Hence the Federal Circuit concluded that the clear and uncontroverted statements made in the specification of the patent in issue in <u>Spectra</u> coupled with Spectra's admission of the function of the wax in the claimed invention, precluded Spectra from successfully arguing that toners <u>without</u> polymer, and without a wax-reduced affinity between polymer and dyestuff, are an equivalent of the invention claimed in the patent in issue. <u>Spectra</u> 839 F.2d at 1581-82, 5 USPQ2d at 1868-69. In contrast to <u>Spectra</u>, independent claim 1 in issue does not limit, or even specify that the claimed composition has an effect on cure rate. In addition, the administrative law judge has found that while the particular diorganosulfur oxide in the claimed curable composition frequently allows for an improved rate of cure, an improved rate of cure is not a necessary limitation inherent in the claimed curable composition.
Moreover, Ausimont in the accused compositions does not omit an ingredient but rather substitutes an ingredient of the claimed composition with another ingredient. Hence he finds that <u>Spectra</u> is inapposite.

Ausimont argued that none of complainant's witnesses pointed to any evidence as to what occurs during vulcanization of a composition containing a fluoroelastomer, a quaternary phosphonium, bisphenol AF and a sulfone to support infringement (RRS 106(a)) and that there are no published literature studies or any study addressing how a quaternary phosphonium behaves or how a P-N compound behaves in a solid phase vulcanization in the presence of a sulfone (RRS 111(b)). It is argued that proof of infringement requires testing of the accused composition, citing In re Certain Molded Golf Balls, Inv. No. 337-TA-35 (USITC Publication 897, July 1978) (Molded Golf Balls); Genentech Inc. v. The Wellcome Foundation Ltd., 29 F.3d 1555, 1556, 31 USPQ2d 1161, 1167 (Fed. Cir. 1994) (Genentech); and Perkin Elmer Corp. v. Westinghouse, Elec. Corp., 822 F.2d 1528, 3 USPQ2d 1321 (Fed. Cir. 1987) (Perkin-Elmer) (RB at 31, 32).

The administrative law judge finds Molded Golf Balls, Genentech and Perkin-Elmer not controlling. Referring to the underlying facts in the three cases, in issue in Molded Golf Balls was the importation of certain molded golf balls and whether those golf balls infringed U.S. Patent No. 3,313,545 (the '545 patent). The Commission agreed with the administrative law judge's finding that seven respondents violated section 337 by infringement of claim 1 of the '545 patent because samples of their imported golf balls were tested by an independent testing laboratory and were found to infringe "directly and literally" claim 1 of the '545 patent.[17] While the administrative law judge

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[17] The administrative law judge in his recommended determination, in connection with the accused samples obtained, found that visual inspection in cross section of a sampling of ten imported golf balls showed nearly identical appearances and textures to the domestic product with the exception of some variations in pigmentation, and that chemical tests performed at an independent testing lab showed an infringing formulation of the constituent ingredients; that the patented golf balls contained an elastomer, to provide the resiliency, a filler to impart the requisite density and a monomer that is polymerized to form flexible cross links that cross-link the elastomer into a

had found that eighteen respondents from whom samples of molded golf balls were not obtained and tested should be included with those seven respondents who were in violation of section 337, the Commission dismissed the eighteen respondents from whom samples were not obtained and tested because complainant had failed to sustain its burden of coming forward with evidence to prove that the imported articles from those eighteen respondents infringe the '545 patent, stating that it believed that complainant could have obtained samples of those eighteen respondents' accused balls and that "[o]nly testing actual samples prove infringement." However the Commission concluded that the exclusion order "which we determined is the appropriate remedy in this case, is a remedy that will prevent any person, including the respondents who were dismissed from the investigation, from importing molded golf balls which infringe this patent." Molded Golf Balls, Commission determination at 7-10.

In issue in Genentech, was whether the accused tissue plasminogen activator, FE1X infringes one or more patents under the doctrine of equivalents. The Federal Circuit concluded that the jury's implied conclusion that the specific limitation appearing in the certain claims in issue either literally or equivalently was not supported by substantial evidence. The Court found that while certain measurements were made "using the chromogenic

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three dimensional network; that all of the spectra from spectrographic tests obtained from pyrolyzed samples taken by an independent lab from three of the domestic products and the ten accused golf balls showed peaking characteristics of both a methacrylate monomer and a cis-1-4 polybutadiene elastomer; that those lab tests demonstrated graphically that claim 1 of the '545 patent read on the composition of the samples of certain respondents but that the test results are conclusive only as to the presence of a methacrylate monomer as taught in (claim 1 and not the specific substance suggested elsewhere in the '545 patent. Molded Golf Balls, recommended determination at 4 (Feb. 10, 1978). Finding 24 of the recommended determination found that the failure by the respondents to reveal the formulations used by them in their manufacture of the balls had precluded any direct evidence and had necessitated reliance on secondary evidence to show infringement.

substrate assay," there was no evidence that such measurements made using this assay were "comparable to those made using the bovine fibrin plate assay" and in fact "all the evidence of record addressing the subject indicates that the two methods [of measurement] are not comparable." Genentech, 29 F.2d at 1560-61, 1565-66, 31 USPQ2d at 1164, 1169.

In issue in Perkin-Elmer was a resonator coupler for coupling a source of r-f electrical power into an electrodeless discharge lamp for starting and operating the lamp. The district court found that the claimed tap-coupling enabled the claimed invention to implement a specific manner or way of impedance matching a frequency tuning, which manner of operation was based on a positioning of the tap point location along the length of the helical cord which was "one of the 'cardinal structural-functional-operational interrelationships' of the claimed invention." The Federal Circuit (Chief Judge Markey and Judge Archer with Judge Newman dissenting), in affirming the district court's finding that the accused devices and the claimed invention do not operate in substantially the same way, found no such tap point in the accused devices for impedance matching, for frequency tuning, or for any purposes; that on the contrary, as the district court found, there is a frequency mismatch in the accused devices which was remedied by varying the length of an external cable (not an element in the claimed invention) connecting the EDLs to their r-f power source and by the positioning of an "iris capacitor"; that the accused devices are not tuned by anything relating to the internal r-f power coupling means and are tuned in a substantially different way from that required by the claim in issue; and that the patentee did not challenge the district court's findings of those structural and functional differences. The Federal Circuit made reference to the district court's finding that to compensate for an impedance mismatch, the external cable connecting the accused EDLs to their r-f power source must be of a specific length and is an "integral element used for impedance matching" and that while the patentee's response to that finding was to argue that the invention and the accused devices all match impedance, that argument missed the mark for it disregarded the way impedance is matched in its claimed invention and the different way impedance is matched in the accused devices. The Federal Circuit concluded that while the patentee argued that an impedance match could be achieved in the accused devices by varying the number of turns in the primary coil of the transformer circuit, assuming arguendo that that were true, the district court failed to detect the relevance of a modified version of the accused devices, even if the patentee had pointed, as it did not, to evidence that the modified version would operate substantially the same as the claimed invention. Perkin-Elmer, 822 F.2d at 1534-35, 3 USPQ2d at 1326.

The administrative law judge finds that neither Molded Golf Balls, Genentech nor Perkin-Elmer requires that there must be testing of the accused composition in order to establish infringement under the doctrine of equivalents and that any other evidence relied on to establish infringement under the doctrine of equivalents must, in effect, be disregarded. Thus in Molded Golf Balls in issue was whether certain golf balls were found to infringe "directly and literally" claim 1 of the patent in issue, and whether the failure of respondents to reveal the formulation used by them in their manufacture of the golf balls had precluded any direct evidence. The record in Molded Golf Balls had no evidence, other than the spectra from spectrographic tests performed by an independent lab, to determine whether there was a direct or literal infringement of the claimed subject matter. In Genentech "all the evidence of record" indicated that two methods for obtaining certain measurements to demonstrate infringement were not "comparable." In Perkin Elmer the district court failed to detect the relevance of a modified version of the accused devices, even if the patentee had pointed, as it did not, to evidence that the modified version would operate substantially the same as the claimed invention.

In contrast to the underlying facts in Molded Golf Balls, Genentech, and Perkin-Elmer, in issue in this investigation is whether under the doctrine of equivalents the claimed composition which contains a plurality of ingredients is infringed by the accused compositions which are within the literal language of independent claim 1, with the exception that component b of claim 1 is substituted by another substance. In support of complainant's allegation of infringement under the doctrine of equivalents, there is evidence, not only from complainant but also evidence involving Ausimont, supra, that the substituted substances in the accused compositions and component b of the claimed composition are accelerating agents in the presence of bisphenol AF and base for the curing of fluoroelastomers. There is also evidence not only from complainant but also evidence involving Ausimont, Schmiegel and Venkateswarlu, supra, that the curing of a fluoroelastomer composition involves an initial dehydrofluorination, attachment of the bisphenol curing agent and the thermal formation of additional unsaturations and subsequent crosslinking of the fluoroelastomer and that the component b of the claimed composition and the substituted substances in the accused compositions perform as a phase transfer catalyst in the curing. Ausimont's Chiodini testified that GM 102E and triphenyl benzyl phosphonium chloride, which is specifically recited in claim 13 of the '320 patent (FF 55), "act in the same way" in the vulcanization of fluoroelastomers using a bisphenol (FF 248). Moreover, Ausimont's Tommasi has testified that the diorganosulfur oxide used in the compositions claimed in the '320 patent is not a cross-linker[18] and it does not form a salt with bisphenol AF but rather has a solvating effect in the curing of fluoroelastomers (FF 246). Complainant's expert Harwood further testified that the dioganosulfur oxide recited in independent claim 1 in issue does not react with the fluoroelastomer to crosslink it and also does not work like the phosphonium cation as a phase transfer catalyst to form a salt with the Bisphenol AF to improve transport (FF 106). Hence, the administrative law judge concludes that the evidence shows that the diorganosulfur oxide of independent claim 1 and of the accused compositions are distinct from the role of the phosphonium accelerators in claim 1 and in the accused compositions.

Ausimont has criticized certain tests of complainant, some of which did involve a diorganosulfur oxide, on the ground that they did not involve solid-solid phase polymerization studies. However, as demonstrated by Schmeigel, Venkateswarlu and even Ausimont's own scientists, it is common place for an understanding of what is happening in a solid/solid phase vulcanization of fluoroelastomers to refer to work using other systems. Thus the Schmiegel 1978 presentation, which referenced the '654 patent involving solid/solid phase polymerization and which 1978 presentation employed solution polymerization experiments (FF 189 to 198), and the Venkateswarlu 1989 presentation which was not limited to solid/solid phase polymerization studies

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[18] Tommasi's testimony is consistent with the argument of Kolb's agent in the prosecution of the '320 patent that the diorganosulfur oxide recited in claim 1 in issue is not a vulcanizing agent (FF 84).

(FF 254 to 265), have been relied upon to explain reactions involving solid/solid phase polymerization. Moroever, Ausimont's scientists have accepted the Schmiegel and/or Venkateswarlu presentations (FF 219 to 223, 225 to 227, 231 to 237, 239, 240, 243, 244). Also, Ausimont's Arcella in 1991 reported, following solid state studies under approximate vulcanization conditions, that:

> These results are in a very good agreement with the previously reported findings obtained by FT-IR analysis performed on samples <u>in solution</u> and represent a good support to the proposed vulcanization mechanism, since the experimental conditions tested are very close to that of <u>common practical vulcanization</u>. (FF 243) (Emphasis added)

In addition, there is testimony from expert witnesses in the curing of fluoroelastomer compositions that if a compound acts as a phase transfer catalyst in a liquid system, there is no reason to expect that it would act differently in a solid system and that there is no reason to believe that phosphonium compounds acting as phase transfer catalysts in the curing of fluoroelastomers would behave any differently in a solid/solid phase as compared to a liquid/liquid phase (FF 298 to 300).

At closing argument, Ausimont made reference to the "chain transfer technology" involved in making the "current" co-polymers in the accused compositions and has argued that the accused compositions contain a "more advanced and far superior fluorinated copolymer" (RB at 49). The claimed invention however is not directed to a more advanced and far superior fluorinated copolymer but rather to as combination of components. In addition Ausimont has not denied that any possible "more and far superior fluorinated copolymer" is included in the literal language for the fluoroelastomer recited in claim 1 in issue. Moreover, assuming <u>arguendo</u> the accused compositions had such a copolymer complainant has demonstrated, in certain of its tests, that complainant used a copolymer of vinylidene fluoride-hexafluoropropene copolymer, as are the copolymers of the accused compositions. Also complainant showed that the mole ratio of the copolymer used in complainant's tests approximates the mole ratio of the copolymer in the accused compositions (FF 274). Moreover, even if the accused compositions contained a "more advanced and far superior fluorinated copolymer" there is nothing in the record to support a finding that GM 102 and XA 51 would not function as an accelerator and act as a phase transfer catalyst in the presence of a bisphenol and base in the vulcanization of a fluoroelastomer composition to result in a crosslinked fluoroelastomer, consistent with the teachings of the literature, Ausimont's own reports, the testimony of scientists in the relevant field and the tests performed by complainant.

Ausimont argued that the reverse doctrine of equivalents precludes a finding of infringement and that the accused compositions are so far changed in principle from the patented compositions that they function in a substantially different way; that the accused compositions contain a "more advanced and far superior fluorinated copolymer" as compared with the fluorinated copolymers available at the time of Kolb's invention; that by using non-ionic chain transferrers, Ausimont was able to make new copolymers, which gave Ausimont's compositions faster cure rates in comparison with compositions containing old copolymers made from ionic chain transferrers; that the dichlorodiphenyl sulfone in Ausimont's compositions does not affect an improved cure rate (i.e., with Ausimont's "advanced polymers" in combination with Ausimont's P-N compounds); and that clearly the way in which Ausimont achieves an improved cure rate was not contemplated by inventor Kolb, and is so far changed in principle from Kolb's use of diorgano sulfoxide or sulfones that it cannot be regarded to infringe the claimed invention, citing SRI International v. Matsushita Electric Corp. of America, 775 F.2d 1107, 1122-23, 227 USPQ 577, 587 (Fed. Cir. 1985) (SRI International).[19] (RB at 49 to 52).

When a patentee establishes literal infringement, the accused infringer may undertake the burden of going forward to establish the fact of non-infringement under the reverse doctrine of equivalents. SRI International 775 F.2d at 122-24, 227 USPQ at 587. Judge Newman in her order in Texas Instruments, Inc. v. U.S.I.T.C., 846 F.2d 1369, 1371, 6 USPQ2d 1886, 1889 (Fed. Cir. 1988), stated that the invocation of the doctrine of the reverse doctrine of equivalents requires both that (1) there must be apparent literal infringement of the words of the claims; and (2) the accused device must be sufficiently different from that which is patented that despite the apparent literal infringement, the claims are interpreted to negate infringement. In this initial determination the administrative law judge has found there is no literal infringement. Hence, Ausimont's reliance on the reverse doctrine of equivalents is rejected.

3. Conclusion

Based on the foregoing, the administrative law judge finds that complainant has established that each of component b of the claimed composition in issue, and XA 51 and GM 102E in the accused compositions, function as accelerators in the curing of fluoroelastomers in the presence of a bisphenol; that the quaternary phosphonium compound of the claimed compositions, and the XA-51 and GE 102E, perform in substantially the same way

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[19] Ausimont has not denied that the "advanced polymers" in the accused compositions literally read on the definition of an "elastomer copolymer" in independent claim 1 of the '320 patent.

when used with a bisphenol and a base in the vulcanization of a fluoroelastomer; and that the results when using the quaternary phosphonium compound and the Ausimont accelerators are substantially the same in the curing of a fluoroelastomer in the presence of a bisphenol. Accordingly, the administrative law judge finds that complainant has established by a preponderance of the evidence that Ausimont infringes the claims in issue under the doctrine of equivalents.

D. Validity Of The '320 Patent

Ausimont asserts that the '320 patent is invalid for obviousness under 35 U.S.C. § 102 and invalid under 35 U.S.C. § 112 for indefiniteness, best mode and nonenablement. The '320 patent is presumed to be valid under 35 U.S.C. § 282. Accordingly, complainant can rely upon the presumption of validity, which Ausimont must overcome by clear and convincing evidence. Uniroyal, Inc. v. Rudkin-Wiley Corp., 837 F.2d 1044, 1059, 5 USPQ2d 1434, 1438 (Fed. Cir.), cert denied, 488 U.S. 825 (1988) (Uniroyal). The presumption of validity is applicable to any bases for challenging a patent's validity. Panduit Corp. v. Dennison Mfg., 810 F.2d 1561, 1570, 1 USPQ2d 1593, 1599 (Fed. Cir.), cert. denied, 481 U.S. 1052 (1987). The circumstances surrounding the prosecution of a patent can make the patent challenger's burden of proving invalidity a heavier one. Custom Accessories Inc. v. Jeffrey-Allan Indus. Inc., 807 F.2d 995, 961, 1 USPQ2d 1196, 1200 (Fed. Cir. 1986) (reexamination and reissue of a patent makes burden of showing invalidity heavier). In the prosecution of the '320 patent, the patent issued over a protest from duPont, a major competitor in the fluoroelastomer market (FF 74, 88).

1. Alleged Invalidity Under 35 U.S.C. § 103

The determination of obviousness under 35 U.S.C. 103 is a determination of law based on the underlying factual inquiries required by Graham v. John Deere Co., 383 U.S. 1, 17, 148 USPQ 459,467 (1966), viz. (1) the scope and content of the prior art; (2) the differences between the prior art and the claims at issue; (3) the level of ordinary skill in the art; and (4) any objective evidence of secondary considerations. Thus the obviousness under section 103 is determined by whether the claimed invention would have been obvious to a person of ordinary skill in the appropriate art at the time the invention was made. Uniroyal, 837 F.2d at 1051, 5 USPQ2d at 1438. In determining whether the differences between the claims at issue and the prior art are sufficiently minor to render the claimed invention obvious, it must be established whether one skilled in the art would have been motivated to select and combine features from each cited reference in order to make the claimed invention at the time it was made. In re Fritch, 972 F.2d 1260, 1266, 23 USPQ2d 1780, 1783 (Fed. Cir. 1992). Thus there must be something in the prior art suggesting the desirability of combining the references. In re Beattie, 974 F.2d 1309, 1312, 24 USPQ2d 1040, 1042 (Fed. Cir. 1992).

The two principle prior art references relied upon by Ausimont are de Brunner U.S. Patent No. 3,752,787 (the '787 patent) (RX 9) and Pattison U.S. Patent No. 3,876,654 (the '654 patent) (RX 10) (RB at 65). Ausimont relies on de Brunner or Pattison taken in combination with any one of the following further references, none of which, it is argued, were considered by the PTO during prosecution of the '320 patent: Morrison & Boyd, "Organic Chemistry", Third Edition, 1973 (RX 13), Carpenter et al. U.S. Patent No. 2,964,503 (RX 8), and Busse U.S. Patent No. 2,522,776 (RX 7) (RB at 66, 67). Ausimont argued that all of the components set forth in the '320 patent claims in issue, apart from the sulfur oxide (sulfone) additive, were disclosed in the literature and used in the fluoroelastomer field before the claimed invention in issue; and that the deficiency of the sulfone is remedied by the express disclosures of the use of sulfones to accelerate the reaction rates of a wide variety of chemical reactions by solvating anions (Morrison & Boyd and Carpenter et al.), or to plasticize and/or tackify curable synthetic rubbers to improve their processability without impairing their properties after curing (Carpenter et al. and Busse) (RB at 65, 67, 68).

Complainant argued that there is absolutely no testimony whatsoever which indicates that the differences between the '320 claimed invention and the prior art would have been obvious to one of ordinary skill in the art (CB at 8,9). The staff submitted that the Morrison & Boyd, Carpenter et al. or Busse references do not relate to fluoroelastomers and that none suggest combination with fluoroelastomer systems such as those described in the prior references (SB at 35).

a. The Scope And Content Of The Prior Art And The Differences Between The Prior Art And The Claims In Issue

At the outset, none of the references asserted by Ausimont as rendering the '320 patent obvious discloses the use of any type of a diorganosulfur oxide with a fluoroelastomer (FF 314). Moreover, the deBrunner '787 patent was before the Patent Office during the prosecution of the '320 patent (FF 136). In addition, the prosecution of the '320 patent demonstrates the criticality of the limitation placed on the claimed diorganosulfur oxide, viz. a specific class of diorganosulfur oxides, consisting of a diorganosulfur oxide containing two like or unlike aliphatic, cycloaliphatic or aromatic groups which are unsubstituted or substituted with halogens (FF 85).

The deBrunner '787 patent and the Pattison '654 patent contain no better disclosure than the Schmiegel '732 patent which was the subject of the protest by duPont (FF 74). In fact, the '732 patent, appears to be closer to the Kolb claimed invention than the '787 and '654 patent because the '732 patent contains examples of bisphenol-type crosslinking compounds with sulfur oxide substituents (FF 74, 75).

The Morrison and Boyd reference discloses the development and widespread use of <u>aprotic</u> solvents, as for example "dimethyl sulfoxide, N,N-dimethylformamide and sulfolane" for dissolving both organic and inorganic regents and in dissolving ionic compounds with solvating cations and that reports of dramatic solvent effects on a wide variety of reactions, since about 1958, have appeared about dimethylforamide and about dimethyl sulfoxide (FF 315, 316). The Carpenter reference incorporates into a curing composition composed essentially of a base mixture of polyalkylene polysulfide polymers and a soluble curing agent adapted to liberate anions of chromium when in solution, a modifying and solubilizing agent from the group of "amides, sulfoxides, sulfones, sulfonamides, phosphoramides, esters of phosphoric acid, esters of boric acid and esters of monobasic and polybasic organic acids" (FF 317, 318). Busse found that the processing of synthetic rubbers, exemplified by polymerized diolifins, such as the copolymers of butadiene with styrene and butadiene, can be improved by incorporating in the rubber an aryl sulfone or a polysulfone with the sulfones operating to tackify <u>and</u> plasticize the synthetic rubber (FF 319, 320). However, as the Patent Office Board of Appeals found (FF 85), only by reference to Kolb's own disclosure would the class of diorganosulfur oxides recited in Kolb's claims have been obvious,[20]

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[20] <u>See</u> FF 72, 79 and 82 which shows that Kolb's agent in the prosecution of the '320 patent argued the criticality of the class of diorganosulfur oxides claimed in the '320 patent.

even assuming, arguendo, the Morrison and Boyd, Busse and Carpenter references disclosed a fluoroelastomer, which they do not. Moreover, Busse's sulfone tackifies the synthetic rubber which Busse teaches renders the surface of a substance to which the tackifier is added sticky and tacky. In addition Morrison and Boyd shows the equivalence of N,N-dimethylformamide (not even a diorganosulfoxide) and dimethylsulfoxide and Carpenter teaches the equivalence of a wide variety of components, e.g. amides and esters of boric acid far removed from the class of diorganosulfur oxide recited in claim 1 in issue. In addition there is no indication in Carpenter that the indispensable soluble curing agent adapted to liberate anions of chornium when in solution and the polyalkylene polysulfide polymers (FF 317, 318) can be dispensed with. Also, there is expert testimony that the diorganosulfide oxide of claim 1 in issue when added to fluoroelastomers is not simply a plasticizer as Ausimont contends Busse shows.

Based on the foregoing, the administrative law judge finds that Ausimont's combinations of prior art do not suggest the claimed invention.

b. Objective Evidence Of Nonobviousness

The evidence shows that Kolb began his work which resulted in the '320 patent in direct response to a customer complaint about curable fluoroelastomer compositions (FF 91). Furthermore, the products based on the patented improvement have been commercially successful (FF 42). In some instances rubber processors switched to the patented products specifically because their previous fluoroelastomers did not incorporate a sulfone (FF 39).

c. Conclusion

Based on the scope and content of the prior art relied upon, the differences between the prior art and the claims in issue, and certain objective evidence of secondary considerations, the administrative law judge finds that Ausimont has not sustained its burden of establishing that the claims in issue of the '320 patent are not valid under 35 U.S.C. § 103.

2. Best Mode Defense

Ausimont argued that the "best mode" requirement of 35 U.S.C. §112, first paragraph, provides that a patent specification must "set forth the best mode contemplated by the inventor of carrying out his invention." It is argued that inventor Kolb developed the sulfone additives of his invention working as a technical service representative of complainant with one if its customers which was testing an "incorporated cure product" of complainant's FC 2170, for the manufacture of O-rings, and that Kolb proposed the addition of the sulfones to improve the curing characteristics of the curable fluoroelastomer composition; that prior to Kolb's October 16, 1973, filing date, his work on the sulfone-containing curable fluoroelastomer compositions was directed only to this O-ring application; that all of the experiments carried out by or on behalf of Kolb utilized curable compositions incorporating a "raw gum" prepared from a particular copolymer of vinylidene fluoride and perfluoropropene known at 3M by the internal code designation JC 3163; that Kolb preferred JC 3163 in the curable compositions intended for the manufacture of O-rings because of that copolymer's desirable Mooney viscosity and other properties meeting existing specifications for O-ring manufacture; that although the '320 specification discloses that any copolymer within a broad class of materials may be utilized as the elastomer component of the curable composition, only a single specific copolymer was used in each of the compositions cured in the comparative runs described in Tables 1-4; that in reality each of the examples (runs) of the '320 patent was carried out with a curable composition in which the copolymer was JC 3163, but yet the specification of the '320 patent does not identify JC 3163 or the commercial incorporated cure product (FC 2170) which contained that copolymer, nor does the '320 specification describe any properties of the copolymer; and that JC 3163 was not sold to customers as a raw gum, but was only sold as a component of the FC 2170 incorporated cure product (RB 52 to 59).

Complainant argued that while inventor Kolb, in performing the examples set forth in Tables 1 to 4 of the '320 patent, used a raw gum having an internal designation JC 3163, which was the gum used in complainant's commercial incorporated cure fluoroelastomer composition FC-2170, the claimed invention in issue is not a new or improved "copolymer" or "fluoroelastomer gum"; that as Kolb has indicated, he was not knowledgeable about the specifics of the complainant's JC 3163 "raw gum" at the time he filed the patent application that resulted in the '320 patent; and that the '320 patent is not limited to any specific copolymer, but rather the invention is a combination of old fluoroelastomers with the addition of a diorganosulfur oxide (CB at 11-13).

The staff argued that a person of ordinary skill in the art would have known which specific polymer to use in "Kolb's preferred embodiment"; that given the few commercially available polymers, details concerning the copolymer would have been known in the art; and that the particular characteristics of a desired polymer vary widely depending on the final product being manufactured (SB at 39).

The best mode requirement is subjective: did the inventor disclose what he believed to be the best way to practice his or her invention. <u>Chemcast Corp. v. Arco Indus. Corp.</u>, 913 F.2d 923, 927-28, 16 USPQ2d 1033, 1036 (Fed.

Cir. 1990); In re Sherwood, 613 F.2d 809, 204 USPQ 537 (CCPA 1980), cert. denied, 450 U.S. 944 (1981). As Kolb's agent argued before the Patent Office (FF 79), the claimed phrase "[a] curable fluoroelastomer composition" makes it clear that it is not the final cured material, for example the O-ring application referred to by Ausimont, but the "curable" composition which is subsequently cured which is being claimed, and that inventor Kolb recognized similarity in properties of his composition after curing with other good cured fluoroelastomers (FF 79). The '320 patent contains a lengthy disclosure of the saturated polymers which may be cross-linked in accordance with the Kolb invention and makes specific reference to the particularly preferred fluorinated elastomers produced by copolymerizing perfluoropropene and vinylidene fluoride as described in U.S. Patent Nos. 3,051,677 and 3,318,854, and those terpolymers produced by copolymerizing perfluoropropene, vinylidene fluoride and tetrafluoroethylene as described in U.S. Patent No. 2,968,649 (FF 129 to 134).

Based on the foregoing, the administrative law judge finds that Ausimont has not established that the '320 patent is not valid under the best mode requirement of 35 U.S.C. § 112.

3. Undue Breadth and Indefiniteness Defenses

Ausimont argued that given the unpredictability regarding which P-N compounds would work and Kolb's failure to identify any P-N compounds in his specification which would work, the claims are invalid under 35 U.S.C § 112, second paragraph, because the specification "utterly fails to describe the invention in 'such full, clear, concise and exact terms' as to enable one skilled in the art to practice it without undue experimentation" (RB at 83).

Ausimont also argued that complainant's own evidence shows that neither the inventor nor complainant can "make up their minds" on the meaning of the term "quaternary phosphonium compound" in the claims in issue, and that said inconsistent positions compel the conclusion that the term is hopelessly indefinite. Hence it is argued that all of the claims of the '320 patent are invalid for failing to particularly point out and distinctly claim the invention under 35 U.S.C. § 112, second paragraph (RB at 86).

In view of the finding of the administrative law judge, <u>supra</u>, that the claims in issue do not literally encompass amino-phosphonium compounds, <u>i.e.</u>, compounds wherein the phosphorus atom is bonded to a nitrogen, the administrative law judge finds that the undue breath and indefinite defenses have been mooted.

E. Direct Infringement

Complainant and the staff argue that Ausimont U.S.A. and its customers directly infringe the asserted claims of the '320 patent when mixing an acid acceptor and an optional base with the accused products (CB at 39-40; SB at 80-82). Ausimont maintains that even when the acid acceptor is mixed with the accused compositions there is still no direct infringement because the compositions do not infringe literally or under the doctrine of equivalents the asserted claims of the '320 patent (<u>see</u> RRS 167-172).

It is not contested that an acid acceptor and an optional base must be added to the accused compositions in order to crosslink the polymer (FF 323). Ausimont Italy manufactures and exports to the United States the accused compositions (FF 2), and Ausimont U.S.A. imports said compositions and sells them to its customers (FF 3). The record demonstrates that Ausimont U.S.A. personnel recommend to customers recipes for fluoroelastomers which include amounts of acid acceptors and optional bases within the range of the '320 patent, and that Donald J. Myer, Ausimont U.S.A.'s technical manager for TECNOFLON, testified that although Ausimont U.S.A.'s customers may not tell it what recipe they use, some of its customers follow the recommendations of Ausimont U.S.A. (FF 322, 324, 333). The record also demonstrates that the accused compositions are useful only when the acid acceptor and optional base are added (FF 343 to 345).

With respect to direct infringement by Ausimont U.S.A., the record establishes that Ausimont U.S.A. has supplied to accused compositions with amounts of magnesium oxide and calcium hydroxide within the ranges of the '320 patent already mixed in. In addition, the record shows that Ausimont U.S.A. has prepared slabs containing accused compositions mixed with amounts of magnesium oxide and calcium hydroxide within the range of the '320 patent for purposes of testing the compositions (FF 327).

With respect to FOR 9550, Ausimont's counsel argued at oral argument that Ausimont has not sold any FOR 9550 but maintains "a small research quantity" of FOR 9550 at Ausimont U.S.A., and that since said FOR 9550 has not been sold to or used by any customer, there has been no infringing use thereof (Tr. at 2849-52). Assuming, arguendo, that Ausimont U.S.A. has maintained "a small research quantity" of FOR 9550 in the United States and such use is not an infringement, the record also shows that Ausimont U.S.A. shipped a compound 5029 A to a company called which compound contained a mixture of FOR 9550 with calcium hydroxide and magnesium oxide within the levels of the '320 patent (FF 328, 329). In addition, Myer testified that tests were performed at Ausimont U.S.A. on slabs of fluoroelastomer containing FOR 9550 that were prepared using a recipe specifying 3 and 6 parts of magnesium oxide and calcium hydroxide as required by the '320 patent (FF 330, 331).

Accordingly, in view of the foregoing, the administrative law judge finds that complainant has established that Ausimont U.S.A. and its customers have directly infringed the asserted claims of the '320 patent in mixing the accused compositions, including FOR 9550, with amounts of acid acceptor and an optional base within the levels of the '320 patent.

F. Induced Infringement

Complainant and the staff argued that Ausimont Italy and Ausimont U.S.A. induce their customers to infringe the '320 patent. At oral argument on November 2, counsel for Ausimont conceded that if there is direct infringement of the '320 patent, then both Ausimont Italy and Ausimont U.S.A. induce infringement (Tr. at 2847-48).

35 U.S.C. § 271(b) provides that "[w]hoever actively induces infringement of a patent shall be liable as an infringer." The Federal Circuit has held that a "person <u>induces</u> infringement under § 271(b) by actively and knowingly aiding and abetting another's direct infringement," <u>C.R. Bard, Inc. v. Advanced Cardiovascular Sys., Inc.</u>, 911 F.2d 670, 675, 15 USPQ2d 1540, 1544 (Fed. Cir. 1990) (emphasis in original), and that in order to prevail, the complainant must show that Ausimont's "actions induced infringing acts <u>and</u> that [Ausimont] knew or should have known that [its] actions would induce actual infringements." <u>Manville Sales Corp. v. Paramount Systems, Inc.</u>, 917 F.2d 544, 553, 16 USPQ2d 1587, 1594 (Fed. Cir. 1990) (emphasis in original).

In addition to the admission of Ausimont's counsel at oral argument, <u>supra</u>, the record demonstrates that Ausimont Italy manufactures and exports to the United States the accused compositions (FF 2), and that Ausimont U.S.A. imports said compositions and sells them to its customers (FF 3); that numerous product brochures and technical data sheets from Ausimont U.S.A. provide test recipes in which an acid acceptor and an optional base are mixed with the commercial product in amounts within the ranges provided in the claims of the '320 patent (FF 332); that Ausimont U.S.A. personnel recommend to customers recipes with respect to fluoroelastomers which include amounts of acid acceptors and optional bases within the patent ranges (FF 333); and that, while customers do not always report to Ausimont U.S.A. the recipe they actually use, Ausimont U.S.A.'s Myer (FF 322), admitted that some customers of Ausimont U.S.A. do follow its recommendations (FF 333). Moreover, Myer further testified that he has never recommended using levels of acid acceptor and optional base outside the limits contained in the '320 patent (FF 334).

With respect to FOR 9550, there is substantial evidence in the record that Ausimont U.S.A. has provided samples of FOR 9550 to customers and/or potential customers (FF 335-342). Thus, the record demonstrates that as of May 1988, Ausimont U.S.A. had shipped 2.5 kg of FOR 9550 to (FF 336), and that on approximately October 18, 1988, Ausimont U.S.A. shipped 5 lbs. of FOR 9550 to In addition, Myer testified that FOR 9550 was sent to other customers, although he could specifically remember how many (FF 338), and that Ausimont is still attempting to sell FOR 9550 (FF 335).

Accordingly, for the foregoing reasons and in view of the statement of Ausimont's counsel at oral argument, the administrative law judge finds that complainant has established that both Ausimont Italy and Ausimont U.S.A. induce infringement of the '320 patent.

G. Contributory Infringement

Complainant and the staff argue that Ausimont Italy and Ausimont contribute to infringement of the asserted claims of the '320 patent by their customers. As with induced infringement, counsel for Ausimont stated at oral argument on November 2 that if there is found to be direct infringement of the '320 patent, then both Ausimont S.p.A. and Ausimont U.S.A. contributorily infringe the asserted claims of the '320 patent (Tr. at 2847-48).

35 U.S.C. § 271(c), concerning contributory infringement, provides in relevant part as follows:

> (c) Whoever sells a component of a patented ... composition, ... constituting a material part of the invention, knowing the same to be especially made or especially adapted for use in an infringement of such patent, and not a staple article or commodity of commerce suitable for substantial non-infringing use, shall be liable as a contributory infringer.

In addition to the admission of counsel for Ausimont, <u>supra</u>, the record demonstrates that Ausimont Italy manufactures and exports to the United States the accused compositions (FF 2), and that Ausimont U.S.A. imports said compositions and sells them to its customers (FF 3); and that said accused compositions are intended to be cured by addition of an acid acceptor and/or an optional base in a manner within the asserted claims of the '320 patent (FF 343). Indeed, Ausimont's Myer testified that he <u>never</u> recommended to customers to use levels of acid acceptor and optional base <u>outside</u> the limits contained in the '320 patent (FF 334). In addition, complainant's Brullo testified that he knew of no applications for the accused compositions that do not involve curing with a base (FF 345). Although Ausimont's Tommasi testified that he was not sure "if there are some minor applications that the customers might have invented or designed or decided or agreed upon with our research assistant," he testified further he knew of no such applications (FF

344). Moreover, assuming if there are such applications, mere theoretical or occasional aberrant uses would not qualify the accused compositions as staple articles. See Fromberg, Inc. v. Thornhill, 315 F.2d 407, 415, 137 USPQ 84, 90 (5th Cir. 1963); Dennison Mfg. Co. v. Ben Clements and Sons, Inc., 467 F.Supp. 391, 428, 203 USPQ 895, 925 (S.D.N.Y. 1979). Since Ausimont U.S.A. has made no sales in the United States of FOR 9550 (FF 24), there is no contributory infringement by Ausimont Italy or Ausimont U.S.A. with respect to that composition.

Accordingly, for the foregoing reasons, and in view of the statement of Ausimont's counsel during oral argument, the administrative law judge finds that complainant has established that Ausimont Italy and Ausimont U.S.A. contributorily infringe the asserted claims of the '320 patent.

H. Domestic Industry

Pursuant to section 337(a)(3), a complainant may prove the existence of a domestic industry by showing that

> . . . there is in the United States, with respect to the articles protected by the patent . . . concerned --
>
> (A) significant investment in plant and equipment;
>
> (B) significant employment of labor or capital; or
>
> (C) substantial investment in its exploitation, including engineering, research and development, or licensing.

19 U.S.C. § 1337(a)(3). The burden of proving the existence of a domestic industry is on the complainant. Certain Methods of Making Carbonated Candy Products, Inv. No. 337-TA-292, Commission Opinion at 34-35 (March 8, 1990); Certain Concealed Cabinet Hinges and Mounting Plates, Inv. No. 337-TA-289, Commission Opinion at 22 (Dec. 28, 1989).

Neither Ausimont nor the staff contests that there exists a domestic industry in complainant's activities relating to the '320 patent, indeed the parties have stipulated to certain facts which establish the existence of a domestic industry under section 337(a)(3) (FF 351 to 355). Robert A. Brullo, who is the manager of complainant's Specialty Fluoropolymers Department, testified that all of 3M's domestically sold FLUOREL and FLUOREL II fluoroelastomers with incorporated cure compounds, specifically the ones which include a sulfone, are produced at 3M's plant (FF 346, 357). As demonstrated in the record and the testimony of Brullo, complainant has made significant investment in plant and equipment (FF 353, 361, 362) and significant employment of labor and capital in the manufacture of products covered by the '320 patent (FF 354, 358, 360, 363), as well as substantial investment in engineering and research and development concerning compositions produced under the '320 patent (FF 349, 355, 359). Moreover, as stipulated to by Ausimont and the staff (FF 356), it is found that complainant practices the '320 patent in the production of certain of its FLUOREL and FLUOREL II products. Accordingly, the administrative law judge finds that complainant has established there exists a domestic industry with respect to the fluoroelastomer compositions protected by the '320 patent pursuant to section 337(a)(3)(A) through (C).

VII. FINDINGS OF FACT

A. Parties

1. Complainant is Minnesota Mining and Manufacturing Company (3M), a Delaware corporation having its principal place of business at 3M Center, St. Paul, Minnesota. 3M manufactures, among other things, curable fluoroelastomer compositions and precursors thereof useful in the manufacture of molded products. (SX-30 at 2-4; RX-1 at 1-2).

2. Respondent Ausimont S.p.A. (Ausimont Italy) is an Italian corporation having its principal place of business at Viale Lombardia 20, 20021 Bollate (MI), Italy. Ausimont S.p.A. manufactures, among other things, curable fluoroelastomer compositions and precursors thereof useful in the manufacture of molded products and exports fluroelastomers compositions into the United States. (CX-479 at 4-6; CX-262 ¶ 10). Ausimont Italy is manufacturing TECNOFLON FOR-421, FOR-423, TECNOFLON FOR-65 BI/R, TECNOFLON FOR-800 HE and TECNOFLON FOR-5351 in Italy. (CX-262 at ¶ 11; CX-495). Ausimont Italy has manufactured TECNOFLON FOR-420 and TECNOFLON FOR-9550. (CX-495, Stipulations 14 and 15).

3. Respondent Ausimont U.S.A., Inc. (Ausimont U.S.A.) is a Delaware corporation having its principal place of business at 44 Whippany Road, Morristown, New Jersey. Ausimont U.S.A. tests, sells, and markets curable fluoroelastomer compositions and precursors thereof in the United States. Ausimont U.S.A. is a wholly-owned subsidiary of Ausimont S.p.A. (CX-479 at 4-6). Ausimont U.S.A. is importing TECNOFLON FOR-421, FOR-423, TECNOFLON FOR-65 BI/R, TECNOFLON FOR-800 HE and TECNOFLON FOR-5351 into the United States and selling the products to customers within the United States. (CX-262, at ¶ 11; CX-495, Stipulation No. 13). Ausimont U.S.A. has imported TECNOFLON FOR-

420 and TECNOFLON FOR-9550 into the United States. (CX-495, Stipulations 14 and 15).

B. The Products At Issue

4. The products at issue are fluoroelastomer compositions. A fluoroelastomer is a rubber-like polymer where some of the hydrogen atoms of the polymer have been substituted with fluorine. Complaiant's products which it contends are covered by the '320 patent and the accused products are "incorporated cure" products, meaning that many of the chemicals required to vulcanize the polymers into final products are incorporated into the commercial products. Fluoroelastomers are used in automotive, aerospace, pollution control, chemical processing, and food processing applications. (Brullo, CX-100 at 4-6 and 10).

5. Fluoroelastomer polymers are used for a variety products, such as o-rings, gaskets, and fuel connectors. (Brullo, CX-100 at 9).

6. Complainant and Ausimont produce fluoroelastomer compositions. The fluoroelastomer compositions must be compounded and molded to produce products. (Brullo, CX-100 at 5-6).

7. An incorporated cure fluoroelastomer compound is sold with all components necessary to achieve a cured fluoroelastomer, with the exception of an acid acceptor or base. The compounder or molder may add the acid acceptor or base and then mix the fluoroelastomer compound, with the mixed fluoroelastomer compound placed in a mold, cured, and sold as a product. (Brullo, CX-100 at 16-18).

8. Ausimont Italy is manufacturing TECNOFLON FOR-421, FOR-423, TECNOFLON FOR-65 BI/R, TECNOFLON FOR-800 HE and TECNOFLON FOR-5351 in Italy. (CX-262 at ¶ 11; CX-495, Stipulation 13).

9. Ausimont U.S.A. is importing TECNOFLON® FOR-421, FOR-423, TECNOFLON® FOR-65 BI/R, TECNOFLON® FOR-800 HE and TECNOFLON FOR-5351 into the United States and selling the products to customers within the United States. (CX-262 at ¶ 11; CX-495, Stipulation 13).

10. Ausimont Italy has manufactured TECNOFLON® FOR-420 and TECNOFLON® FOR-9550. (CX-495, Stipulations 14 and 15).

11. Ausimont TECNOFLON FOR-420 was imported into the United States from 1986 to 1991. (CX-495, Stipulation 14).

12. Ausimont TECNOFLON FOR-9550 was imported into the United States in 1990 and 1992. (CX-495, Stipulation 15).

13. The following commercial designations of Ausimont products are accused of infringing the '320 patent,

FOR 420;
FOR 421;
FOR 423;
FOR 5351;
FOR 65BI/R;
FOR 800 HE; and
FOR 9550.

(CX-495, p. 4, ¶ 13, 14, 15.)

14. FOR 420 has the following composition,

(CX-495, Exh. 3; Tommasi Tr. at 984, 985; Ferro CPX-5, Vol I. at 43-44).

15. The name of GM102E under the International Union of Pure and Applied Chemistry (INPAC) rules is

16. FOR 420 has not been imported into or sold in the United States since 1991. (CX-495, p. 4, ¶ 14.)

17. Ausimont has no current plans to import FOR 420 into the United States. (CX-496; Alisio CPX at 57).

18. FOR 421 has the following composition,

(CX-495, Exh. 3; Tommasi Tr. at 984, 985; Ferro CPX-5, Vol 1 at 43-44).

19. FOR 423 has the following composition,

(CX-495, Exh. 3).

20. FOR 65BI/R has the following composition,

(CX-495, Exh. 3; Tommasi, Tr. at 984, 985; Ferro CPX-5, Vol. 1 at 43-44).

21. FOR 800HE has the following composition,

(CX-495, Exh. 3; Tommasi, Tr. at 984, 985; Ferro CPX-5, Vol. 1 at 43-44).

22. FOR 5351 has the following composition,

(CX-495, Exh. 3; Tommasi, Tr. at 984, 985; Ferro CPX-5, Vol. 1 at 43-44).

23. FOR 9550 has the following composition,

(CX-495, Exh. 3; Tommasi, Tr. at 984, 985; Ferro CPX-5, Vol. 1 at 43-44).

24. FOR 9550 has not been sold in the United States. (CX-495, p. 4, ¶ 15.)

25. The abbreviations in the above formulations have the following meanings:

NMA }
NM/R }
NML/R } All are copolymers of vinylidene fluoride and hexafluoropropene
NML }
NMLA }
NH }
N535 }
NX215 }

Bisphenol AF 4,4'-[2,2,2-trifluoro-1-(trifluoromethyl)ethylidene] bisphenol

GM102E - diphenylbenzyldiethylaminophosphonium chloride; phosphoraneamine; also known as diphenyl benzyl diethyl aminophosphonium chloride

XA-51 -

DCDPS - Dichlorodiphenyl sulfone

YO6 - Fomblin Y commercial fraction (an oil used in vacuum pumps, added to fluoroelastomer compositions as plasticizer).

(CX-495 Exh. 3: Tommasi, Tr. at 985-985; Ferro CPX-5, Vol. I at 43-44; CX-476 at 27-31 (Ausimont's answer to 3M's Interrogatory No. 217), Harwood CX-231 at 16-20; Tommasi CPX-12, Vol. I at 51-52).

26. XA-51 is

27. In an Ausimont report of Chiodini and Minutillo (CX-294) titled "Synthesis and Characterization of New Ionic Type Curing Systems For Fluoroelastomers" dated November 30, 1989,

were prepared. The conclusion of the report states (AI00 9533 and AI009534):

28. With respect to characterization of blends using a readily available fluoroelastomer as the polymer matrix the final analysis in CX-244 as to the rheometric properties was (A100 9548):

> In the final analysis, the results observed are consistent with what has been found many times in the case of copolymers using the traditional system of separate accelerant and bisphenol. The most significant improvement in terms of rheumatic properties lies essentially in the higher crosslinking speed for the same scorch times; this obviously means shorter molding times with substantial economic advantages. Moreover, of considerable importance for the improvement of processability, especially in injection molding, is the reduction in the viscosity of the blend which is 3-4 points less on the average than blends with the same concentration in mols of bisphenol. The decrease in viscosity was always rather limited and seems to vary in terms of the initial viscosity of the polymer: it is greater in the case of polymers with a higher viscosity ... and if an adduct with a higher bisphenol ratio is used.

29.

30. Ausimont has substantial sales of the accused compositions, excluding FOR 9550, in the United States. (CX-495, Stipulation 15 at 4; SX-17; SX-18; SX-19; SX-20; SX-21; SX-22; SX-23).

31. The ingredients of complainant's compositions covered by the '320 patent are set forth at CX-495, Exh. 1 and 2.

32. Complainant and Ausimont recommend that their customers add calcuim hydroxide and magnesium oxide (the acid acceptor and optional base of the patented formulation) to the commercially available product. (Tommasi, Tr. at 986-988; Harwood CX-231 at 7-8; Brullo CX-100 at 6 and 16; *see generally* CX-121 (FLUOREL technical information sheets) and CX-315, CX-316, CX-317, CX-318, and CX-339 (Ausimont technical information and specification sheets)).

33.

34. GM 102E <u>may</u> be represented by certain resonance structures (Schlosser, Tr. at 2036-39; RPX-9; Schlosser RX-145, at 3).

35. While the parties have stipulated that the Ausimont TECNOFLON products listed on confidential Exhibit 3 of CX-495 have been imported into the United States and have the formulation shown on said Exhibit 3, (CX-495 at 4 §12), it is a fact that certain chemicals listed in Exhibit 3 are sometimes added by the compounder or molder or even Ausimont U.S.A. in the United States. <u>See</u> (FF 321 to 345).

36. Ausimont's Giulio Tommasi agreed that each of FOR 420, FOR 421 FOR FOR 5351.65 B1/R, FOR 800HE, and FOR 70BI is a curable fluoroelastomer consisting essentially of an elastomeric copolymer of vinylidene fluoride and

terminally ethylenically unsaturated fluorinated comonomer; that in FOR 420 for every hundred parts of the copolymer there would be between 0.2 to five parts of a diorganosulfur oxide which contains two like or unlike aliphatic, cycloaliphatic or aromatic groups which are unsubstituted or substituted only with halogen; that FOR 420 would have 0.2 to five parts of an aromatic hydroxy or amino compound (CPX-12 at 36); and that to test the FOR 420 for properties after curing the suggested composition to add is six percent calcium hydroxide and three percent magnesium oxide. (CPX-12 at 38, 44, 45, 50, 65, 70, 71).

37. Ausimont's Tommasi testified that FOR 423 contains the fluoroelastomer, a dioganosulfur oxide and XA 51. (CPX-12 at 51, 52).

38. Ausimont's U.S. sales of sulfone containing accused compositions commenced at least as early as 1986 (RX-4, Exh. I). However, Ausimont's TECHNOFLON FOR-420 has been imported into the United States at least until 1991. Also its TECHNOFLON FOR-9550 was imported into the United States in 1990 and 1992. Moreover, the following specific Ausimont articles have been imported into the United States and are presently being sold: FOR-421, FOR-423, FOR-5351, FOR-65 BI/R and FOR-800 HE. (CX-495, Stipulations 13, 14, 15).

39. Complainant sells fluoroelastomer products in the United States which are covered under the '320 patent. Those products includes FLUOREL (registered trademark) FC-2121, FC-2123, FX-2124Q, FC-2144, FC-2174, FC-2173, FC-2176, FC-2180, FC-2181, FC-2181 PS, FC-2182, FT-2350, FT-9038, FX-9143. FE-5620Q, FE-5260-23, FE-5640Q, FE-5620-40, FE-5660Q, FE-5840Q, FC-2182P, FLS-2330, FX-11700 and FX-11900. (CX-102 and CX-103). Also in some instances, rubber processors switched to the patented products specifically because their previous fluoroelastomers did not incorporate a sulfone. (Brullo, CX-100 at

22).

40. Complainant, with respect to some of its fluoroelastomer compositions sold under the trade name FLUOREL has included not only dichlorodiphenyl sulfone but also tetramethylene sulfone. (CX-102).

41. Complainant does not recommend to its customers the use of dichloro diphenyl sulfone for an improvement in cure rate. In its "Technical Fundamentals Manual FLUOREL Fluoroelastomers" the dichlorodiphenyl sulfone advantages in fluoroelastomers is described as follows:

> DCIDPS In Fluoroelastomers
>
> What it does do:
>
> - Provides mold release without mold buildup.
> - Provides faster extrusion rates.
> - Improved mold flow for transfer or injection applications
> - Increase mold shrinkage rate.
> - Causes "Bloom" on non-post cured parts.
>
> What it does not do:
>
> - Improve surface appearance on extrusions.
> - Alter original physical properties, compression set resistance or heat or heat aging results when used within recommended limits.

Nowhere is there any allegations about the effect of DCIDPS on "cure rate." (CX-120 at 47).

42. The accused compositions are sold in competition with complainant's patented compositions to the same customers for the same applications (CX-440). For example, the accused FOR 65/BI/R was developed to compete with

complainant's FC 2174 (Ferro, CPX-5, Vol. 1 at 22; CX-425).

C. Experts

43. Alan T. Worm, based on his vitae (CX-133), was qualified as an expert on fluoroelastomer compositions. (Tr. at 674, CX-133). He started work with complainant after he graduated from Purdue in 1969 with a Ph.D in Organic Chemistry. When Worm first began work with complainant, his research was with fluorochemical compounds. After 1975 Worm started to do basic research in the area of elastomer cure chemistry. Throughout the years to the present time, he has had various roles with respect to fluoroelastomer products. (CX-132 at 1, 2).

44. Werner Grootaert, based on his vitae (CX-494), was qualified as an expert in fluoroelastomer and fluoroelastomer compositions and analysis. (Tr. at 929).

45. Professor James Harwood on the basis of his curriculum vitae (CX-232) and voir dire (Tr. at 1077 to 1109) was qualified as an expert in polymer chemistry, organophosphorus chemistry, analyses on fluoroelastomers systems and the curing of the fluoroelastomers. (Tr. at 1070, 1086, 1100, 1109).

46. Jerry Leyden, in view of his viate (RX-65) was qualified as an expert in the testing and interpretation of rheology data respecting curable fluoroelastomer compositions. (Tr. at 1651, 1652).

47. Professor Manfred Schlosser, based on his vitae (RX-105), was qualified as an expert in the fields of fluorine chemistry, organophosphorus chemistry and analytical techniques. (Tr. at 1985).

48. Professor Robert Engel was offered as an expert by complainant. He was the subject of an oral motion at the evidentiary hearing with respect to his expertise. Engel is qualified as an expert with respect to the effect of

other components on reactions involving organophosphorus compounds, catalyes involving organophosphorus compounds and phase transfers containing organophosphorus compounds and including fluoroelastomers. However, <u>see</u> Order No. 15, which issued herewith.

49. Giulio Tommasi was offered as an expert by Ausimont. His expertise was challenged at the evidentiary hearing. Tommasi is presently director of the R & D Center of Ausimont which was formed out of Montedison and he had been with Montedison since April 15, 1968. He received a "doctor degree" from the Polytechnic Institute of Milan. He then was an assistant at the University of Milan, Polytechnic Institute of Milan for almost one year. Thereafter, he spent one and a half years at Syracuse University in assistant teaching and as a research assistant in chemical engineering. He also received a degree from Syracuse. After Syracuse University he was hired by Montedison as a process design engineer (CPX-12 of July 25, 1994 at 6 to 8). Tommasi is qualified as an expert in the field of curable fluoroelastomers and compositions including same. However, <u>see</u> Order No. 15, which issued herewith.

D. The '320 Patent And Claims At Issue

50. The patent in issue is U.S. Letters Patent No. 4,287,320, entitled "Composition of Fluoroelastomer and Diorganosulfuroxide," which issued on September 1, 1981, to inventor Robert E. Kolb and is assigned on its face to complainant. The '320 patent is based on Application Serial No. 122,513 filed February 19, 1980, which was a continuation of abandoned Ser. No. 493,537 filed August 1, 1974 which was a continuation of abandoned Ser. No. 406,832 filed October 16, 1973. (CX-1).

51. Kolb's '320 patent is not based on making a different cure system,

but instead is based on an improvement of the existing bisphenol-phosphonium type cure system. (Kolb, RX-121 Dep. Tr. at 111; Worm, Tr. at 666, 677).

52. The claims at issue are claims 1, 2, 4, 5, 6, 11, 12, 14, and 15. Claims 1, 12, 14 and 15 are independent claims. The remaining asserted claims are dependent on Claim 1. (CX-495, Stipulation No. 16).

53. Claim 1 of the '320 patent reads:

> 1. A curable fluoroelastomer composition consisting essentially of elastomeric co-polymer of vinylidene fluoride and terminally ethylenically unsaturated fluorinated comonomer, and for each hundred parts of said co-polymer:
>
> a. 0.2 to 5 parts diorganosulfur oxide, which contains two like or unlike aliphatic, cycloaliphatic or aromatic groups which are unsubstituted or substituted only with halogen;
>
> b. 0.1 to 5 parts quaternary phosphonium or ammonium compound;
>
> c. a total of 3 to 40 parts consisting of 0 to 25 parts acid acceptor, and 0 to 25 parts optional base; and
>
> d. 0.2 to 5 parts aromatic hydroxy or amino compound.

(CX-1, column 12, line 64 to column 13, line 11).

54. Claims 2, 4, 5, 6, 11, 12, 14 and 15 in issue read:

> 2. A curable fluoroelastomer composition according to claim 1 wherein the elastomeric copolymer comprises vinylidene fluoride and perfluoropropene.
>
> 4. A curable fluoroelastomer composition according to claim 1 wherein the diorganosulfur oxide is diorganosulfone.
>
> 5. A curable fluoroelastomer composition according to claim 1 wherein the acid acceptor is magnesium oxide and the optional base is calcium hydroxide.
>
> 6. A curable fluoroelastomer composition

according to claim 1 wherein the aromatic hydroxy compound is 2,2-bis-(p-hydroxyphenyl) hexafluoropropane.

11. A curable fluoroelastomer composition according to claim 1 wherein the diorganosulfur oxide is bis(p-chlorophenyl) sulfone.

12. A curable fluoroelastomer composition consisting essentially of elastomeric co-polymer of vinylidene fluoride and terminally ethylenically unsaturated fluorinated commonomer, and for each hundred parts of said copolymer:

a. 0.2 to 5 parts diorganosulfur oxide, which contains two like or unlike aliphatic, cycloaliphatic or aromatic groups which are unsubstituted or can be substituted only with halogen, aliphatic hydroxyl, nitro, and cyano substituents;

b. 0.1 to 5 parts quaternary phosphonium or ammonium compound;

c. a total of 3 to 40 parts consisting of 0 to 25 parts acid acceptor, and 0 to 25 parts optional base; and

d. 0.2 to 5 parts aromatic hydroxy or amino compound.

14. A curable fluoroelastomer composition comprising an elastomeric copolymer of vinylidene fluoride and at least one terminally unsaturated fluoromonolefin containing at least one fluorine atom substituent on each double-bonded carbon atom, each carbon atom of said fluoromonoolefin being substituted only with fluorine, chlorine, hydrogen or a lower fluoroalkyl or fluoroalkoxy radical, at least 10% of the chain carbon atoms of said copolymer being --CH_2-- groups, and for each 100 parts of said copolymer:

a. 0.2 to 5 parts diogranosulfur oxide, which contains two like or unlike aliphatic, cycloaliphatic or aromatic groups which are substituted or substituted only with halogen;

b. 0.1 to 5 parts quaternary phosphonium or ammonium compound;

c. a total of 3 to 40 parts consisting of 0 to 25 parts acid acceptor, and 0 to 25 parts optional base; and

d. 0.2 to 5 parts aromatic hydroxy or amino compound.

15. A curable fluoroelastomer composition comprising an elastomeric copolymer or vinylidene fluoride and at least one terminally unsaturated fluoromonoolefin containing at least one fluorine atom substitutent on each double-bonded carbon atom, each carbon atom of said fluromonolefin being substituted only with fluorine, chlorine, hydrogen or a lower fluoroalkyl or fluoroalkoxy radical, at least 10% of the chain carbon atoms of said copolymer being $-CH_2-$ groups, and for each 100 parts of said copolymer:

a. 0.2 to 5 parts diogranosulfur oxide, which contains two like or unlike aliphatic, cycloaliphatic or aromatic groups which are unsubstituted or can be substituted only with halogen, aliphatic hydroxyl, nitro, and cyano substituents;

b. 0.1. to 5 parts quaternary phosphonium or ammonium compound;

c. a total of 3 to 40 parts consisting of 0 to 25 parts acid acceptor, and 0 to 25 parts optional base; and

d. 0.2. to 5 parts aromatic hydroxy or amino compound.

55. The claims of the '320 patent in issue do not define the term "quaternary phosphonium," although independent claim 13, not in issue, recites as said compound, "triphenyl benzyl phosphonium chloride." (RX-5, col. 13, 14).

E. Prosecution Of The '320 Patent

56. Original independent claim 1 of Kolb's Serial No. 406,832 filed October 16, 1973 read:

1. A fluoroelastomer composition comprising:

(A) an elastomeric copolymer of vinylidene fluoride and at least one other terminally ethylenically unsaturated fluorinated comonomer,

(B) at least one quaternary phosphonium or ammonium compound and

(C) at least one diorganosulfur oxide.

(CX-4 at 26).

57. The Examiner in an office action on Ser. No. 406,832 in June 1974, _inter alia_, rejected independent original claim 1 on Patel and Maier U. S. 3,655,727 (the '727 patent) taken with Bowman U. S. 3,686,143 ('the '143 patent) on the ground that the '727 patent at col. 4, lines 43 to 64 teaches the use of polyols in general with "onium" compounds and that it is obvious to use sulfur oxides since Bowman equates non sulfur oxide to sulfur oxide containing polyols. The '727 patent (CX-11) at col. 4, lines 30 to 70 teaches that a critical ingredient in the final curable composition is for example bisphenol A and that the composition includes at least one quaternary ammonium or quaternary phosphonium compound (the "onium" compound). (CX-4)

58. The '727 patent, which is referred to also in the '320 patent in describing a curing system based on quaternary ammonium derivatives (col. 2, line 59, col. 6, line 56), and in referring to well-known co-curatives for curing fluoroelastomers with a particularly preferred class comprising aromatic hydroxy or amino compounds (col. 7, lines 55-57) issued to complainant's Patel and Maier on April 11, 1972. It is based on an initial application filed August 19, 1968 and discloses quaternary compounds, including phosphonium and ammonium, and their use in fluoroelastomer curing systems. It is titled "Curing Systems For Vinylidene Fluoride Elastomers". The patent limits the constituents on the quaternary phosphonium compound to compounds having a phosphorus atom covalently bonded to four carbon atoms, _viz_. alkyl, aralkyl, or alkylene radicals with a preferred number of carbons in the chain. (CX-11, column 2, lines 52-69).

59. The '727 patent has composition claims which recite an admixture

comprising (a) elastomeric copolymer of vinylidine fluoride (b) a quaternary ammonium or phosphonium compound and (c) at least one aromatic hydroxy or amino compound. The '727 patent states (CX-11, col. 9, lines 58):

> Quaternary ammonium and phosphonium compounds, when used in amounts as small as 0.05 pphr., produce a measurable acceleratory effect on cure rate at a given amine concentration. An excess of the quaternary compound, over 0.4 pphr., particularly when more than about 0.5 pphr., tends to effect an overcured vulcanizate, although in general the higher molecular weight quaternary compound can be used in greater amounts than those of lower molecular weights. Generally, the lower the amount of amine employed, the greater the optimum amount of the quaternary compound....

60. The '727 patent, on the mechanism of curing vinylidine fluoride elastomers, states (RX-24 col. 5, lines 74, 75, col. 6, lines 16):

> The mechanism of curing saturated copolymers of vinylidene fluoride with, for example, perfluoropropene, is not fully understood. Existing evidence suggests that the initial press cure involves a base-catalyzed release of hydrogen fluoride to generate double bonds in the polymer, these double bonds then providing a limited number of crosslinks between the polymer chains which serve to stabilize the shape and form of the polymer, while the subsequent post cure step results in the formation of further ethylenically unsaturated structures which combine to form benzenoid crosslinks of high themostability. This is consistent with our findings that a variety of compositions function as curing or crosslinking agents, acting to aid in the release of hydrogen fluoride. Most free primary or secondary aliphatic amines (insofar as they are not in themselves amine generators), free hydrazine or free ammonia are not suitable as curing agents, and their use as such results in either too rapid a cure rate or no cure at al.

The claims of the '727 patent are limited to a curable composition containing (a) an elastomeric vinyledene fluoride copolymer, (b) at least one quaternary ammonium compound of a particular formula and (c) at least one particular aromatic hydorxy or amino compound (RX-24).

61. The '143 patent issued on August 22, 1972 to Bowman on an initial application filed on April 11, 1969. It is assigned on its face to DuPont (CX-12). The patent is titled "Guanidine and Amidine Accelerators For

Vulcanization of Fluoroelastomers." The '143 patent teaches that elastomeric copolymers of two or more fluorinated monomers can be cross-linked with certain aromatic polyhydroxylic compounds such as bisphenols in the presence of a certain metal compound; that crosslinking of highly fluorinated polymers with aromatic polyhydroxylic compounds must be carried out in the presence of a catalyst; and that catalysts which accelerate vulcanization or the curing of elastomers are generally referred to as "vulcanization accelerators." The '143 patent discloses that highly fluorinated elastomers have found increasing application in specialized areas, such as high temperature resistent gaskets, seals, diaphragms, and tubing; that it is necessary for many applications that the fluoroelastomer article be resilient and have low compression set which is accomplished by curing the article which comprises vulcanization or cross-linking of the elastomer. Table VI of Bowan shows time for 5.5 lb. rise as 15 minutes and for 53.3 lb. rise as 30 minutes under an ODR test. (col. 1, lines 42-61, col. 3, lines 52 to 60, col. 9, Table VI).

62. On August 1, 1974 Kolb's agent prosecuting Serial No. 406,832 filed an express abandonment in view of the filing of a continuation application Ser. No. 493,537 on August 1, 1974. (CX-4, CX-5).

63. In a preliminary amendment dated July 29, 1974 and referring to the '727 and '143 patents applied in the parent Serial No. 406,832, it was argued:

> ... The novelty in the present case arises from the use of neutral diorgano sulfur oxides. This results in a more rapid cure than is possible in their absence....Neither reference suggests the use of <u>neutral</u> diorgano sulfur compounds as adjuvants, hence the combination is in nowise [sic] anticipatory. Furthermore, applicant attains significantly more rapid cures. The figures which point to this particularly are the "times to 50.1b rise" in Table 4, page 25 [col. 12 of the '320 patent], which are in the approximate range of 4 to 7.5 minutes. By contrast note in Bowman, column 9, Table VI, [CX-12] under the ODR test (the same as applicant's rheometer test which in Example 7 is at 177°C, as used in applicant's test) that the time for 50 lb rise would be

> between 15 and 30 minutes, probably close to 25 minutes. Thus applicant's procedure gives about four times as rapid cure. This is clearly not to be expressed from the references.

(CX-3, RX-6, p. 3 of Amendment dated July 29, 1974).

64. Table 4 of the '320 patent (CX-1, col. 12) shows that the time to 50 lb. rise in minutes for the control (run 9) is 7.4, while for run 10 (tetramethylene sulfoxide) is 5, for run 11 (tetramethylene sulfoxide) is 4.1, for run 12 (dimethyl sulfone) is 4.5, for runs 13 and 14 (dimethylsulfoxide) are 4.5 and 4.9, and for run 15 bis(p-chlorophenyl) sulfone is 6.8.

65. In discussing improvements in cure times by the use of diorgano sulfur oxides, the Kolb '320 patent states at column 10, line 68 - column 11, line 5:

> As one point of comparison, material of run 9 (control) require three minutes press cure in commercial equipment whereas runs 10 through 14 (runs according to the invention) require 1 minute. Run 16 also requires 3 minutes but the reduction in amount of phosphonium chloride [from 0.6 to 0.4] results in improved properties.

The '320 patent makes no reference to any improvement in the cure with respect to run 15, namely, the run where the diorganosulfur oxide is dichloro diphenyl sulfone (which Ausimont employs in its accused compositions). Runs 15 and 16 are within the scope of independent claim 1.

66. In the prosecution of Serial No. 493,537 original independent claim 11 was amended to read:

> A curable fluoroelastomer composition consisting essentially of elastomeric copolymer of vinylidene fluoride and terminally ethylenically unsaturated fluorinated comonomer and for each hundred parts thereof:
>
> (a) 0.2 to 5 parts unsubstituted or neutrally substituted diorganosulfur oxide,
>
> (b) 0.1 to 5 parts quaternary phosphonium or ammonium compound,

(c) a total of 3 to 40 parts consisting of

i. 0 to 25 parts acid acceptor
ii. 0 to 25 parts optional base

(d) 0.2 to 5 parts aromatic hydroxy or amino compound.

(CX-3, RX-6).

67. The Examiner, in an office action dated November 6, 1974, <u>inter alia</u>, rejected independent claim 11 of Ser. No. 493,537 on the Patel et. al. '727 patent and the Bowman '143 patent on the ground that Patel "teaches the use of phosphonium cpds. in VF polymers" and it is obvious to use "(a) [diorganosulfur oxide] and (d) [aromatic hydroxy or amino compound], since the total amt. of (a) and (d) embraces the range of cpd. in C [Bowman] (col. 2, lines 58; col. 5, line 6). The neutral grp. of the 'neutrally substituted diorganosulfur oxide' may be hydroxy, inter alia, and thus (a) and (b) [In an amendment filed April 28, 1975 it was stated that the "concensus was that (b) ... should have been (d)"] may be identical with respect to the scope of the term 'organo' and the total parts at lower limits." (CX-3, RX-6).

68. The Examiner in his office action dated November 6, 1974 also rejected independent claim 11 of Ser. No. 493,537 on the Patel and Maier '727 patent and Bowan '143 patent taken with Barth U.S. Pat. No. 3,400,065 (the '065 patent) on the ground that the '065 patent further "renders the combination of organosulfuroxides with VF [vinylende fluoride] polymers unobvious [sic]. This combination improves the property of VF polymers." (CX-3, RX-6).

69. U.S. Patent No. 3,400,065 (the '065 patent) issued September 3, 1968 to Barth on an application filed August 23, 1965 and is assigned on its face to Union Carbide. The patent relates to polymeric mixtures of improved

strength and resistance to thermal stress embrittlement comprising from about 0.1 to 30 parts of a vinylidene fluoride-hexafluoropropene copolymer and a particular linear thermoplastic polyarylene polyether. (CX-9).

70. Barth teaches that his invention can be used in molding powder formulations either alone or mixed with various fillers to make molded parts such as gear, ratchets, bearings, cams, impact parts, gaskets, valve seats, bottles, and containers and can be applied to a broad spectrum of uses in form of sheets, rods, tapes and the like and are useful in electrical applications. (CX-9, col. 6, lines 37-50).

71. On April 28, 1975 there was filed in the prosecution of Ser. No. 493,537 an affidavit under Patent Office Rule 132 by John E. Maier, a coinventor on the '727 patent, wherein he stated that "[a]lthough dihydroxydiphenyl sulfone is useful as a curing agent and cross-linking agent, I do not consider dihydroxydiphenyl sulfone to be a neutrally substituted diorganosulfur oxide which could increase the rate of cure as it is known to be acidic" and that certain data (which Maier presented in the affidavit) showed that the "addition of dihydroxydiphenyl sulfone results in an undesirable retardation of curing whereas the data in application, Serial No, 493,537 show that desirable improvements in rate of cure are obtained when unsubstituted or neutrally substituted diorganosulfur oxides are included in compositions of this sort." (CX-3, RX-6).

72. In an amendment filed April 28, 1975 in the prosecution of Serial No. 493,537 it was argued that Bowman requires the presence of a vulcanization accelerator which may be a guanidine, a salt thereof or an amidine; and that none of those are included by Kolb and there is no suggestion in the cited references that the indispensable part of Bowman can be dispensed with or

substituted by other accelerators. It was also argued:

> In Applicant's Preliminary Amendment, he pointed out that acidic hydroxy aromatic sulfones cannot take the place of his neutral diorganosulfur compounds. Applicant further pointed to the figures in the examples which demonstrate the superiority of curing by the present process.

In addition it was argued that dihydroxydiphenyl sulfone is excluded as a diorganosulfuroxide because it is not neutrally substituted, as called for in the claim, but is actually excessively acidic as compared to the standard for neutrally substituted compounds set forth in the specification, page 11, lines 6-16 which states that the chain in the diorgano sulfur oxides may be substituted or unsubstituted, but the substituents, if any, should preferably be neutral, <u>i.e.</u> neither strongly acidic nor basic radicals; and that the affidavit of Maier recites that the first hydrogen ion from dihydroxydiphenyl sulfone has a KA of about the same as benzoic acid and hence is about six times the maximum acceptable value and thus that this sulfone does not improve and actually harms the curing properties of a vinylidene fluoride elastomer.

73. The Patent Office in a communication mailed on June 23, 1975 in Ser. No. 493,537 stated that all of the claims were allowable in view of the response filed on April 28, 1975. A notice of allowance was mailed on October 23, 1975. (CX-3, RX-6).

74. DuPont in a paper titled "Protest Under Trial Voluntary Protest Program" dated June 8, 1976 in the prosecution of Serial No. 493,537 argued that compositions within the scope of claim 1 of the Kolb application are disclosed by Schmiegel in U. S. Patent 3,933,732 (the '732 patent) and in particular that the Schmiegel Example 1 composition contains 2.67 parts of 4,4'-sulfonyldiphenol diacetate, which is a diorgano sulfur oxide (sulfone) containing two neutrally substituted (ester substituted) aromatic groups.

Thereafter the Patent Office in an action mailed September 21, 1976 rejected the claimed subject matter on Schmiegel. (CX-3, RX-6).

75. The Schmiegel '732 patent issued on January 20, 1976 to Walter Werner Schmiegel on an initial application filed on December 27, 1971 and is assigned in its face to duPont (CX-15). The '732 patent discloses that a fluoroelastomer composition which has beneficial utility in the rapid manufacture of cured fluoroelastomer articles having good strength and resistance to compression set can be prepared by blending (a) an elastomer copolymer of vinylidene fluoride and another fluorine-containing monomer and (B) as a vulcanization agent a carboxylic acid ester of 4,4'-sulfonyldiphenol; and when making the cured articles it is also preferred to add (C) a suitable metal compound and (D) a suitable accelerator, for example a certain type of quaternary phosphonium compound (CX-15). The '732 patent also discloses that one very useful type of component D accelerator is a compound wherein four carbon containing radicals are covalently bonded to the phosphorus, with a preferred compound of this type being benzyl triphenyl phosphonium bromide or chloride. The '732 patent teaches that this type of accelerator is described in greater detail in Pattison's Can. Pat. No. 945,300 granted April 9, 1974 (col. 5, lines 3-30); and that other useful accelerators include "methyltrioctylammonium chloride and other quaternary ammonium compounds" as those described in the Patel et al. '727 patent and by Pattison in a French Pat. No. "71-18215" (col. 5, lines 38-42). The '732 patent also discloses that by using the composition of the invention one can overcome the prior art problem regarding the slow cure rates obtained when using 4,4'-sulfonyldiphenol as the vulcanization agent for fluoroelastomers and one can still obtain cured products having excellent physical properties with the

faster cure rates permitting the use of shorter and more economical press-curing operations. (col. 6, lines 42-48).

76. Pattison's Can. Pat. No. 945,300 with a U. S. priority dates of June 9, 1970 (44,883) and Dec. 23, 1970 (101,170) (RX-26) discloses that cross-linking of highly fluorinated polymers with aromatic polyhydroxylic compounds must be carried out in the presence of a catalyst; that catalysts which accelerate vulcanization or curing of elastomers are generally referred to as "vulcanization accelerators;" that according to the invention in the Canadian patent, it is an important feature of the process to employ a quaternary compound of phosphorus, arsenic or antimony, each of which has four carbon radicals covalently bonded to the phosphorus, arsenic or antimony as the vulcanization accelerator; and that it is quite unexpected that this compound functions so effectively as a vulcanization accelerator for a fluoroelastomer in the presence of a metal compound and a polyhydroxylic aromatic compound cross-linking agent for the copolymer. (at pages 3, 9).

77. Kolb's agent in a response filed on December 17, 1976, in Ser. No. 493,577 argued that it is clear that the substituted compounds, <u>i.e.</u> the claimed diorgansulfur oxide in Ser. No. 493,577, do not include the bis(acetoxyphenyl)sulfone disclosed by Schmiegel as curing agents or the "putative meta-hydroxy aryl-sulfinyl compound postulated by the Examiner" and that:

> [i]t should be noted that the bis(acetoxyphenyl)sulfone was employed by Schmiegel in place of all or part of a bis(hydroxphenyl)sulfone which is used as a curing agent. There is a totally different purpose from that of the diorganosulfur oxides employed by Applicant as cure accelerators or processing aids (page 7, lines 17-26) in presently claimed compositions. The diorganosulfur oxides do not act as curing agents.

Page 7, lines 17 to 26 of Ser. No. 493,537 which corresponds to col. 3, lines

47 to 57 of the '320 patent (CX-1) discloses:

> It is found that the use of diorgano sulfoxides and/or sulfones frequently allows reduction in amount of $Ca(OH)_2$ with both improved rate of cure and retention of desirable physical characteristics, including improved resistance to compression set, as compared to the presently known formulations. They may provide self-lubricating characteristics leading to relatively low pressure extrusions with excellent surface finish and provide moldings which are readily removed from the molds. Mold release agents may not be required with the instant compositions.

(CX-3, RX-6).

78. The Patent Examiner, in a final rejection in Ser. No. 493,577 responding to the response filed December 17, 1976, again rejected the claimed subject matter over the Schmiegel '732 patent. It was stated that Schmiegel discloses that diorganosulfones may be employed in the preparation of fluoroelastomer compositions and there is an improvement in the curing properties; that the cured sample show improvement in the characteristic such as tensile strength, compression set etc.; that the results obtained in the cured samples of the Schmiegel patent and those of Kolb's specification are substantially the same; that the diorganosulfones of the Kolb application as well as the Schmiegel patent may be classified as organo esters of diorganosulfones, i.e. that the substituent on the para-position of the aromatic nucleus is a residue of an acid which is the anion portion of the acid; and that both substituents on the aryl sulfone nucleus would be expected to produce a vulcanizate of a fluoroelastomer of the scope covered by Kolb's claims. (CX-3, RX-6).

79. Kolb's agent, responding to the final rejection, in an amendment filed on June 23, 1977, in Ser. No. 493,537 argued:

> The amendment makes the claims consistent in calling in each case for "A curable fluoroelastomer composition." This may make it

> clearer to the Examiner that it is not the final cured material which is claimed but the composition which is subsequently cured. Applicant recognizes similarity in properties of his composition <u>after curing</u> with other good cured fluoroelastomers. It is the faster rate of curing as described page 22 lines 22-28 [col. 10, line 68 - col. 11, line 7] which will be recognized by those of skill in the art as an invention. For this reason the claims are directed to "curable fluoroelastomer compositions."
>
> * * *
>
> ...In his explanation of the rejection [over Schmiegel] the Examiner has quite evidently overlooked the important difference in rate of curing obtained by Applicant and has merely compared properties of cured compositions. Accordingly, it is submitted he has failed to act on the merits of the case <u>which is not directed to cured products</u>, and the final rejection is therefore submitted to be premature.
>
> Applicant [Kolb] has quite clearly pointed to his invention by stating (page 7, first paragraph):
>
>> 'It is an object and aim of this invention to provide curable fluoroelastomer compositions having <u>shortened time for press curing without adversely affecting the physical properties of the cured product</u>.' (Emphasis added [in original])
>
> The more rapid cure is of significance in processing in that time between successive moldings can be reduced and overhead cots thereby reduced.

It was also argued that the Examiner was in error in ascribing to Schmiegel a broad disclosure as to diorgano sulfones; that Schmiegel did not use the term, which is believed first used by Kolb; that Schmiegel discloses only carboxylic esters of 4,4'-disulfonyl diphenol while sulfones containing those groups are not included in compositions claimed by Kolb; and that Schmiegel does not suggest that applicant's unsubstituted or halogen-substituted aliphatic, cycloaliphatic or aromatic sulfones or sulfoxides, "which are processing aids which decrease process time and improve mold release and surface finish, can be used or are equivalent in any way to the esters he uses as 'a vulcanization agent', <u>i.e.</u> which participate in the actual curing." (CX-3, RX-6).

80. The page 22, lines 22-28 (col. 10, line 68, col. 11, line 7 of the '320 patent) referred to by Kolb's agent in the amendment filed June 23, 1977, supra, reads:

> As one point of comparison, material of run 9 (control run) require 3 minutes press cure in commercial equipment whereas runs 10 through 14 (runs according to the invention) require 1 minute. Run 16 also requires 3 minutes but the reduction in amount of phosphonium chloride results in improved properties. Compounding is performed in a roll mill and curing is effected as described above.

Run 9 (control run) contains no diogranosulfur oxide.

81. The Examiner, in an advisory action, responding to the response filed June 23, 1977 in Ser. No. 493,537 stated:

> The rejection is maintained as set forth in Paper No. 17 dated January 10, 1977. Applicant's [Kolb's] arguments have been considered but does not overcome the rejection. Applicant's statement that the composition enable one to prepare fluoro elastomer compositions using sulfones or sulfoxides in amount that the press curing time would be short. However in examination of the Schmiegel patent (col. 8, Table I) states that the curing time could be shorten in the same matter set forth by applicant's in their tables 3 and 4 in the instant specification. Since the times for the curing of the polymers are substantially the same applicant's argument directed to the embodiment as being an unexpected and/or unobvious result is without merit.
>
> Additionally the instant specification fails to present any comparative data with the compounds set forth in the Schmiegel, therefore it is not well taken that applicant's alleged unexpected and/or unobvious results and/or improvements in the curing as related to pressing time.

Thereafter Kolb's agent filed a notice of appeal to the Patent Office Board of Appeals. (CX-3, RX-6).

82. Kolb's agent in his brief before the Patent Office Board of Appeals in Ser. No. 493,537 filed on November 16, 1977 argued that the Examiner appears to have lost sight of the fact that Kolb has amended his claims so that the disulfur oxide, e.g. diaryl sulfone or sulfoxide, which is employed

in addition to an aromatic hydroxy or amino compound may only bear halogen substituents or be unsubstituted, which halogen substituents were argued to be patentably distinct from all other neutral substituents. It was also argued:

> Applicant [Kolb] has pointed out (page 22, lines 22-27 [col. 10, line 68 - col. 11, line 7]) how his compositions cure as compared to prior art materials. They cure in [sic] one-third the time at the same catalyst level. The times are considerably less than the pressures (sic, press cures) used by Schmiegel (col. 7, lines 15-45) of 15-20 minutes at 177°C.

(RX-6, CX-3).

83. In the prosecution of Serial No. 493,537 protester DuPont in a letter dated December 13, 1977 to the Commissioner of Patents and Trademarks concerning foreign patents and/or literature which has been published relating to Schmiegel U. S. Patent 3,933,732 referred, inter alia to British Patent 1,413,837 (3/10/76). (RX-6, CX-3).

84. Kolb's agent in a reply brief before the Patent Office Board of Appeals filed October 4, 1978, in Ser. No. 493,537 argued that although it may not have been sufficiently emphasized, the carboxylic acid esters used by Schmiegel are vulcanizing agents per se and the only vulcanizing agents present in his formulations; that they either include large amounts of the bisphenol, which could act as a vulcanizing agent, or are themselves functioning as vulcanizing agents in all the examples in which they are introduced as 100 percent esters; that it is well known that the vulcanization reaction cannot proceed in the absence of a vulcanizing agent, e.g., an hydroxy or amino compound; that a material such as Kolb's halogen substituted components is not itself a vulcanizing agent and cannot give a phenol under curing conditions to act as a vulcanizing agent and cannot be the equivalent of Schmiegel's curative; that Kolb requires addition of a vulcanizing agent, e.g., (d) of the claim, i.e. an aromatic hydroxy or amino compound; that

Kolb's halogen substituted diorgano sulfur oxides are therefore in no way analogous to Schmiegel's carboxylic esters and could not substitute for the latter to give any basis for affidavit evidence; that the curing times given by Kolb are at least partially supported by the affidavit of John E. Maier which resulted in allowance before the opposition procedure; and that Schmiegel shows no working example in which both bisphenol and carboxylic ester are used together so that he made no disclosure suggesting that there might be advantages to such a combination. It is argued:

> The Examiner continues not to face the fact that cure time is a significant feature in the real world of commerce. A decrease in cure time results in greater through-put in equipment and hence an increase in productivity. It is submitted that the record shows adequately that Appellant's invention possesses this unexpected advantage and that the comparison of data of Appellant's specification and the reference is sufficient to overcome assertions of obviousness made by the Examiner without any additional affidavit evidence.

(CX-3, RX-6)

85. The Patent Office Board of Appeals in a decision mailed on December 11, 1979 in Ser. No. 493,537 asserted that it is uncontroverted that Schmiegel (British) 1,413,837 (the British counterpart to the Schmiegel '732 patent) discloses "all aspects of appellant's claimed composition except for appellant's component 'a,' [diorganosulfur oxide] which the examiner contends would have been rendered obvious by Schmiegel's component 'B';" that the Examiner has stated that, with regard to this component, Schmiegel discloses "diorganosulfones in general;" that the relevant Schmiegel disclosure, however, is limited to a "a carboxylic acid ester of 4,4'-sulfonyldiphenol;" that the Board is constrained to agree with Kolb that only by reference to Kolb's own disclosure would the class of diorganosulfur oxides recited in Kolb's claims have been obvious from the quoted reference disclosure; and thus

no prima facie case of obviousness has been made out on the record before the Board. Hence the decision of the Examiner was reversed. The Board also noted that "claim 16 appears to be identical in scope to claim 15 and is thus redundant." Thereafter a notice of allowance was mailed on August 7, 1980. (RX-6, CX-3).

86. Kolb's agent on February 19, 1980 filed continuation application Ser. No. 122,513, which resulted in the issuance of the '320 patent, to cure a "redundancy" and to seek the added protection provided by claims reciting "said five species." Kolb's agent cancelled the claims of the predecessor application and added claims 11 to 23. Added claim 23 (now claim 13 of the '320 patent) specified the diorganosulfur oxide as bis(p-chlorophenyl) sulfone. (Amendment filed Feb. 19, 1980 CX-2).

87. The Patent Office, in an action mailed on July 31, 1980, allowed certain claims of Ser. No. 122,513 but rejected other claims on French patent 2,096,115 or British patent 1,356,344 stating that the references suggest blends of compounds containing sulfoxide and/or sulfone compounds may be used to prepare curable compositions. (CX-2).

88. Kolb's agent, in an amendment filed October 27, 1980 in Ser. No. 122,513, argued that the Examiner's rejection under 35 USC § 103 in view of the French and British patents is traversed since the basis for the Examiner's reliance on those foreign patents is identical to that found in the patents overcome in the parent application Ser. No. 493,537 in which a notice of allowance was issued following reversal by the Board of Appeals of the Examiner's final rejection therein. It was argued that to the extent that Kolb's diorganosulfur oxide component "a" is unsubstituted, Kolb's claims patentably distinguish over the hydroxy ring-substituted compounds of the

French and British patents although to the extent that Kolb's recited diorganosulfur oxide component is "halogen-substituted" or "substituted with halogen", the Examiner at an interview contended that those claims would not be patentably distinct in that respect over the certain portions of the French and British patents unless it was made clear that when Kolb's diorganosulfur oxide compounds were substituted, the substituents were recited as the only substituents; and that although Kolb believes that this is clearly implied by the original language (e.g., "unsubstituted or halogen-substituted"), Kolb, in order to advance prosecution, has amended certain claims to make it clearer that when Kolb's diorganosulfur oxide compounds are substituted, they are substituted only with the recited substituents. Thereafter on March 5, 1981 the Patent Office mailed a notice of allowance. (CX-2).

89. The prosecution history of the '320 patent provides no assistance in defining the scope of the term "quaternary phosphonium" as used in the claims of the patent. (RX-6).

F. Inventer Kolb

90. Inventor Kolb went to Iowa State University for three years prior to coming to 3M as a technician in 1960. There he took math, zoology and chemistry courses. He started at complainant in the analytical lab and about six years later transferred to the central research pilot plant where he worked about six years. In the fall of 1972 Kolb became a technical service representative for elastomers, and in that function Kolb made customer calls. Kolb is presently a Division Scientist with complainant. It is a level "T6" on a total technical scale of "T7" which is the top. In about 1984 Kolb won the Corporate Technical Excellence Award because of Kolb's work in fluoroelastomers. (CX-128 at 1 to 6).

91. Kolb was on a customer call in early 1973. At that time, the customer was evaluating a 3M fluoroelastomer product designated FC 2170. In discussing this product with the customer, the customer mentioned that he would be interested in seeing an improved FC 2170-type material which would have the faster cure rate as well as improved processability. The improved processability means that it would have better process properties such as extrusion, smoothness, and better mold release characteristics while retaining equivalent or better physical properties. In general, it would just have improved properties over the existing FC 2170-type material. Kolb had done some work using phosphate esters as additives to 2170-type materials. The curative package in the 2170 fluoroelastomer is formulated with the bisphenol cross-linking agent and a phosphonium cure activator. Kolb found that the addition of phosphate esters accelerated the cure. After researching the problem for some time, Kolb believed that the cure was accelerated as a result of a solvation effect created by the phosphate esters in the fluoroelastomer FC 2170 product. The result seemed to be an increased cure rate in the fluoroelastomer, probably resulting from the faster delivery of the Bisphenol AF to the fluoropolymer backbone. Kolb began to look at other compounds and chemicals that he thought would have this solvation effect. Kolb's concerns were to find a compound which would help accelerate the cure while providing better processing without adversely affecting physical properties. The compound Kolb sought had to be effective at low concentrations in the fluoroelastomer system. Further, the compound would have to be compatible and effective in providing the advantages Kolb sought given the ionic nature of the cure package and the physical properties of the base fluoroelastomer gum. Kolb screened any number of compounds including tetrahydrofuran, furfuryl

alcohol, pyridine, piperidine, phosphate esters, picoline, dimethylacetimide, nitrobenzene, nitromethane, and various sulfones and sulfoxides. All of those compounds were reviewed to determine whether they had some type of solvation effect in the composition under investigation. As part of Kolb's review of compounds, he realized that various sulfoxides and sulfones had this effect. Of those compounds, Kolb generally reviewed dimethyl sulfoxide, tetra methylene sulfone, tetra methylene sulfoxide, various sulfonamides and various aryl or aromatic sulfoxides and sulfones. In reviewing the sulphur compounds, Kolb found that those compounds provided an unexpected and surprising balance of cure activation, processing improvements, and maintained the physical properties of the compositions. (Kolb, CX-128 at 7 to 8).

G. The '320 Patent Specification And The Term "Quaternary"

92. The '320 patent states as to how to obtain a cured product from a fluoroelastomer:

> In order to provide a molded, cured product from a fluoroelastomer ... a complex procedure must be followed, each step of which influences both the properties and the cost of the final product. Initially, a uniform mixture of the desired formulation of gum stock and filler, curatives, processing aids, etc. must be prepared. ... At the molding temperature initial curing takes place and must continue to a sufficient stage of cure so that the molded part will maintain its integrity without distortion or tearing when it is released and removed from the molds. The higher the mold temperature, the more rapidly such a "press cured" state can be attained. However, sufficient time must be allowed for the mold to be filled uniformly, and hence there is a practical maximum temperature. Ideally, the viscosity should not increase significantly during the initial flow which fills the mold but should then increase very rapidly to attain a sufficiently stable state for removal.
>
> After removal from the mold, the part is customarily post cured or oven cured. If the cure has not advanced sufficiently during the molding step, the

> gases released during final cure may result in bubbling, blistering and distortion.

(CX-1, col. 1, lines 45-69, col. 2, lines 1-28)

93. The '320 patent discloses as to the utility of cured fluoroelastomers:

> The utility of cured fluoroelastomers depends upon solvent resistance, good compression set characteristics, and resistance to degradation of properties at elevated temperatures. These properties, combined with the generally good elastomeric characteristics of fluoroelastomers, have resulted in the commercial use of a relatively narrow range of compositions in which the major molar component, commonly vinylidene fluoride, is combined with lesser molar amounts of one or more of perfluoropropylene, tetrafluoroethylene, trifluorochloroethylene or monohydroperfluoropropylene. ...

(CX-1, col. 2, lines 28 to 39)

94. The '320 patent discloses that fillers are often added to the polymers to improve the molding characteristics and other properties and that "[p]lasticizers, softeners and processing aids, preferably esters or ketones, may also be added if desired". (CX-1, col. 10, lines 10 to 20).

95. Reference is made in the '320 patent for determining compression set of the cured compositions "in accordance with "ASTM D 395-61B using 25 mm. diameter 0-rings, 3,4 mm. thick, compressed to 27 mm, under the indicated conditions" (CX-1, col. 12, lines 5 to 8).

96. On the use of curing systems, the '320 patent discloses:

> Because the characteristics of the cured product are determined largely by the particular curing system used, most of the improvements in the characteristics of the final product have resulted from improved curing systems. The curing system dictates the conditions of mixing, forming and curing, which have a major influence on the cost of the final product as well as exerting its influence on the physical and chemical properties.

> The polyamine curing systems that first enabled the achievement of satisfactory commercial products continue to be of importance. Long experience with polyamine systems has allowed safe handling and reliable processing, with the major difficulty being the tendency toward permanent deformation after extended use, that is, "compression set".
>
> A curing system based on quaternary ammonium derivatives has been developed (U.S. Pat. No. 3,655,727) which has many of the desirable handling characteristics of the polyamine system, but produces a final product with much higher resistance to compression set. This curing system allows safe milling at 90°-120° C. (200°-250°L.F.) and molding temperatures in the range of 160°-170°C. (320°-335°F). with good flow and a short cure cycle. Cure is so rapid, in fact, at these temperatures that the stock tends to be "scorchy" and careful handling is required to avoid partial curing during the milling operation.
>
> To a considerably *[sic]* extent these difficulties [with quaternary ammonium based curing systems] are overcome by use of quaternary phosphonium curing system based on the presence of a compound in which the phosphorus atom is covalently bonded to 4 carbon atoms and ionically bonded to an anion, as described in U.S. Pat. No. 3,712,877.

(CX-1, column 2, lines 43-68, col. 3, lines 1-7).

97. The '320 patent, on quaternary ammonium compounds, discloses:

> Quaternary ammonium compounds useful in preparing curable fluoroelastomer compositions are compounds which contain at least one nitrogen atom covalently bonded through carbon-nitrogen single bonds to four organic radicals and additionally through an ionic bond to an anion. Such materials are described, for example, in U.S. Pat. No. 3,655,727, column 4, line 65 to column 5, line 2, and U.S. Pat. No. 3,752,787, at column 6, lines 1 through 22. The four organic radicals bonded to each nitrogen atom may be the same or different, and each radical may contain from one to twenty or more carbon atoms, although 2 to about 8 carbon atoms are preferred, with a total of not more than about 30.

(CX-1, col. 6, lines 50 to 62).

98. The Bisphenol AF is the actual cross-linking agent in the claimed

composition in issue. If one takes the base gum with the phosphonium and the Bisphenol AF in it and heats it there will be no cure activation. An inorganic base such as calcium hydroxide or magnesium oxide must be added to activate the cure system. (Kolb, CX-128 at 12).

99. In general, fluoroelastomers are cured through a vulcanization process involving the generation of cross-links through a curative. In typical systems such as the products sold by complainant and Ausimont, the crosslinking agent utilized to form the bridge is Bisphenol AF. In the presence of an organo onium and a base, Bisphenol AF is involved in a dehydrofluorination of the polymer chain, i.e. the elimination of hydrogen fluoride (HF) from the polymer. The Bisphenol AF then adds to the reaction site where the HF was eliminated, to become attached to the polymer chain. Since a material such as Bisphenol AF has two hydroxy groups that can react with polymer chains, the Bisphenol AF can react at each of its ends to become attached to a polymer chain. Thus, the Bispheol AF can form a bridge between two chains, i.e. a cross-link. In general, Bisphenol AF does not react very rapidly with fluoroelastomer polymer, unless an accelerator is provided. The reason is that Bisphenol AF is not very soluble in the polymer, and cannot migrate freely through the polymer to potential reactive sites. In order for the reaction to take place, generally an accelerator is provided. (Worm CX-132 at 3).

100. Phase transfer in organic chemistry facilitates a reaction that occurs across phases. It is basically predictable technology. (Harwood, Tr. at 1237).

101. When the phosphonium compound of the '320 patent is provided in the fluoroelastomer polymer mixture, in the presence of Bisphenol AF and base,

an association between the phosphonium cation and the Bisphenol AF anion takes place. The phosphonium cation is mobile in the polymer phase, and as it moves through the polymer phase it can also transport the associated bisphenol AF through the polymer. This is a method of moving the Bisphenol AF anion into locations where reaction is probable and crosslinking can occur. The phosphonium cation operates as a catalyst, so it can recycle repeatedly and move a number of Bisphenol AF anions into reaction. While the mechanistic details of the vulcanization reaction, at a molecular level, are not fully understood, this characterization is a widely accepted model for picturing the operation of phosphonium compounds of the '320 patent as catalysts in the vulcanization of fluoroelastomers with dihydroxy or diamino compounds. The work of Schmiegal (CX-134) generally summarizes this. Some of the studies which have been done in order to investigate the phenomena include the report of Dr. Pothapragada Venkateswarlu (CX-223). (Worm, CX-132 at 4, 5).

102. There is testimony from Harwood that a fluoroelastomer is really a very, very viscous liquid and its molecules can move. When one wants to carry out a reaction between two liquids (phases) and one component of the reaction is soluble in one of the liquids (for example water) and the other component is soluble only in the remaining organic liquid, in order to have catalysis to promote that reaction, i.e. to have catalysis of it, it helps if the water soluble component can be brought into the organic liquid phase which is the role of the phosphonium compound of the '320 patent, i.e. there is a catalysis to promote the reaction. Phase transfer catalysts are really nothing more than solubilizing materials for a medium which one wants to work in. A phase transfer catalyst takes ions from an interface of the phase and brings the ions into another phase so reaction can take place. In a solid-solid phase

this is what happens when a fluoroelastomer is cured using a bisphenol. Thus initially the bisphenol reacts at the surface of a base such as calcuim hydroxide and is neutralized to become the bisphenol anion. If there is not something around to take the bisphenol anion into the fluoroelastomer copolymer phase thre would be no reaction of the bisphenol anion with the copolymer. The phase transfer catalyst brings the bisphenol anion into the viscous liquid fluoroelastomer and reaction occurs between the bisphenol anion and the polymer backbone first through dehydrofluorination of the polymer and then to establish a bisphenol crosslink sructure. Moreover the bisphenol phase transfer catalyst can bring the fluoride out into the inorganic phase. The phosphonium compound solubilizes the bisphenol A in the organic phase such that it can react with the polymer backbone in the organic phase. Hence in the curing of fluoroelastomers, the bisphenol goes to the polymer backbone and the fluoride comes out and this movement is catalyzed or promoted by the phase transfer catalyst. (Harwood, 1345 to 1351).

103. The invention of the Kolb '320 patent concerns providing for advantageous cure in the presence of certain diorganosulfur oxides. Those materials can be used to accelerate cure (although the process and precise manner in which it operates to accelerate cure is not understood) or as process aids, or both. For example, when added to fluoroelastomers as a process aid, the diorganosulfur oxides modify the polymer flow characteristics in either the extruder or the mold. Also, the surface appearance of the resulting compound is generally smoother in the presence of said oxides than in their absence. Mold release is also generally improved by the diorganosulfur oxide. With respect to the operation of the dioganosulfur oxide as a process aid, it is not clear exactly how the diorganosulfur oxide

works. However, it is not simply a plasticizer, which would be a material used merely to modify viscosity. Rather it modifies the surface of the fluoroelastomer compound during processing. (Worm, CX-132 at 5).

104. One of the significant aspects of the process of using the diorganosulfur oxide, as developed by Kolb, is that it is an additive which does not significantly adversely affect the fluoroelastomer system in typical use, i.e. the properties of the resulting product are generally not undersirably affected by the presence of the diorganosulfur oxide and there is generally no effect which would be greater than is tolerable in typical applications. In other words the material can generally be tolerated within the system, according to the needed process parameters and product parameters. (Worm, CX-132 at 6).

105. The Kolb '320 patent concerns fluoroelastomer compositions which are cured or vulcanized by cross-linking reactions involving either quaternary phosphonium compounds or quaternary ammonium compounds, as phase transfer catalysts. These compounds operate to facilitate reactions of the polymer with certain aromatic hydroxy or amino compounds for cure. (Harwood, CX-231, Tr. at 13).

106. The Kolb '320 patent provides an improvement involving the use of certain diogranosulfur oxides in curing fluoroelastomers. The diorgano sulfur oxide does not react with the polymer to cross-link and also does not work like the phosphonium cation as a phase transfer catalyst to form a salt with the bisphenol AF, to improve transport. (Harwood, CX-231 at 13, 14).

107. Accelerator technology was developed in the 1960's and early 1970's at complainant. In general, this technology involves the provision of a phase transfer catalyst, in order to assist movement of the Bisphenol AF

molecule through the polymer. The particular phase transfer catalysts, developed at by complainant, were quaternary phosphonium and ammonium compounds. (Worm, CX-132 at 3, 4).

108. The '877 patent (CX-13, RX-251), which issued to Patel and Maier and is assigned on its face to complainant is titled "Curable Vinylidene Fluoride Elastomers Containing Phosphonium Curing Agents". It is listed under the "References Cited" in the '320 patent (CX-1). The '877 patent discloses (col. 4, lines 17 to 30, lines 64 to 75, col. 5, lines 1-12, 25-27, 52-65):

> The quaternary phosphonium compounds useful in preparing curable fluoroelastomer compositions are compounds which contain at least one phosphorus atom covalently bonded through carbon-phosphorus single bonds to four organic radicals and, additionally, through an ionic bond to an anion. Such materials, their characteristics and several methods of preparation are described, for example, in "Organo Phosphorus Compounds," G.M. Kosolapoff (John Wiley and Sons, New York, 1950), particularly chapter five. The four organic radicals bonded to each phosphorus atom may be the same or different, and each radical may contain from one to twenty or more carbon atoms, although two to about eight carbon atoms are preferred....
>
> Although useful vulcanizates can be obtained using the quaternary phosphonium compounds alone as curatives, it is frequently desirable to use in addition an accelerator, i.e. a material which significantly increases the rate of cure under curing conditions without unduly accelerating the rate of cross-linking during mixing and milling. The resulting combination generally has a more desirable balance of scorch and cure rate than compositions containing only the quaternary phosphonium compound. Such accelerators are well known and are described in the literature, for example, in U.S. Pats. Nos. 3,243,411 [CX-7] and 3,502,628 [CX-10]. Heretofore, these accelerators have been used with curing systems based on curatives such as amines rather than the quaternary phosphonium compounds of this invention. A particularly preferred class of accelerators is the oxidizable aromatic hydroxy or amino compounds, that is nucleophilic compounds in which one or more hydroxyl, primary amino, or secondary amino radical is

> bonded through the oxygen or nitrogen atom of the radical to an aromatic nucleus, such as phenyl, naphthyl, and the like.
>
> A further component which desirably is contained in the final curing recipe is an acid acceptor, preferably an inorganic acid acceptor ...
>
> Although not necessary, the composition may also contain as cocuratives at least one aromatic amine (primary, secondary or tertiary, aliphatic tertiary amine, or a compound which is stable in the absence of water at temperatures below about 75°C, and which at temperatures above about 125°C, in the presence of water generates a basic nitrogen atom-containing compound.
>
> The mechanism of curing saturated copolymers of vinylidene fluoride with, for example, perfluoropropene, is not fully understood. Existing evidence suggests that the initial press cure involves a base-catalyzed release of hydrogen fluoride to generate double bonds in the polymer, these double bonds then providing a limited number of crosslinks between the polymer chains which serve to stabilize the shape and form of the polymer, while the subsequent postcure step results in the formation of further ethylenically unsaturated structures which combine to form benzenoid crosslinks of high thermostability. This is consistent with our findings that a variety of compositions function as curing or crosslinking agents, acting to aid in the release of hydrogen fluoride... [Emphasis added]

The two claims of the '877 patent are directed to a curable vinylidene fluoride copolymer composition comprising a fluorinated elastomeric copolymer and at least one quaternary phosphonium compound having phosphous-carbon single bonds (col. 11, 12).

109. U. S. Patent No. 3,243,411 (the '411 patent) referred to in the '877 patent, supra, issued March 29, 1966 to Tawney and Conger on an application filed November 30, 1961 and is assigned on its face to United States Rubber Company (CX-7). It is titled "Cross-linking Fluorocarbon Elastomers" and listed in the '320 patent under the heading "References

Cited." The '411 patent discloses that it has been proposed to cure the fluorocarbon elastomers, such as the elastomeric copolymers of vinylidene fluoride with other fluorinated monomers such as hexafluoropropene, with diamines or with monoamines but the vulcanizates so obtained frequently give off unduly large quantities of gas upon heating and also the vulcanizates seem to continue to cure during high temperature ageing with the result that the properties change markedly; that it has been proposed to cure those elastomers with inorganic hydroxides as well as by modifying the elastomer with a secondary amine containing other functional groups and then subsequently cross-linking through those other functional groups; that curing with organic peroxides, using metal oxides as accelerators and polyisocyanantes, polyamines and isocyanate-amine combinations has been described in the literature; and that in general such conventional cures of the fluorocarbon elastomers suffer from various disadvantagers, such as inconvenience or inefficiany, or undesirable side effects or deficiencies in the properties or behavior of the products. The invention in the '411 patent is based on the discovery that fluorocarbon elastomers can be vulcanized in the presence of novel vulcanizing agents which are chemicals that ionize in water to yield an entity which has two or more negative charges with a basic strength greater than that of the acetoxy ion. To cure the fluorocarbon elastomer one or more of the described curing agents are admixed with the elastomer. Typically the curing chemical is prepared beforehand but it is also possible, in many cases, to prepare the vulcanizing agent in situ within the elastomer, particularly if the elastomer is being cured in solution. Among the commercially available fluorocarbon elastomers useful in the invention are Viton A, Viton A-HV, Viton B, Kel-F 2140 and Fuorel which are copolymers of vinylidene fluoride and perfluoropropene and Kel-F 3700 and Kel-F 5500 which are copolymers of vinylidene fluoride and chlorotrifluoroethylene. (CX-7, col. 1, 5, 6).

110. U. S. Patent 3,502,628 (the '628 patent) referred to in the '877 patent, <u>supra</u> and also in the '320 patent (for a preferred class of co-curatives (col. 7, line 55)) issued on March 24, 1970 on an initial application filed on August 17, 1967. It is assigned on its face to duPont. It discloses the acceleration of the vulcanization of saturated, fluorinated polymers by the use of tropolone and 1,10-phenanthroline; and that those accelerators enable a more rapid cure with conventional amine-based vulcanization systems and also enable a satisfactory rate and state of cure with weakly basic bis-nucleophiles otherwise incapable of adequately curing fluorinated polymers. It is disclosed that the invention is particularly useful because it offers routes to faster vulcanization with a lower amount of polyamine compound, thus reducing the cost and reducing the amounts of excess polyamines which are believed to be deleterious for best heat resistance, and because the accelerator compounds do not by themselves affect curing even when as much as 5 parts are used. (CX-10)

111. The '320 patent, referring to quaternary phosphonium compounds, states:

> The quaternary phosphonium compounds useful in preparing curable fluoroelastomer compositions are compounds which contain at least one phosphorus atom covalently bonded through carbon-phosphorus single bonds to four organic radicals and, additionally, through an ionic bond to an anion. Such materials, their characteristics and several methods of preparation are described, for example in "Organophosphorus Compounds", G. M. Kosolapoff, (John Wiley and Sons, New York 1950), particularly chapter five. [CX-21, RX-33, RX-103)

(CX-1, column 5, lines 66-68 and column 6, lines 1-7).

112. The language cited in the foregoing finding is identical to portions of the specification of Patel U.S. Patent 3,712,877, supra. (CX-1, CX-13).

113. Chapter 5 of the 1950 Kosolapoff text is entitled "Quaternary Phosphonium Compounds", and describes only compounds in which a phosphorus atom bears a positive charge and is covalently bonded to four carbon atoms. (Kolb, Tr. at 469; RX-103, 84-95).

114. No other portion of the 1950 Kosolapoff book is mentioned in the '320 patent (CX-1) Chapter five of the 1950 Kosolapoff book is entitled "Quaternary Phosphonium Compounds." Kosolapoff describes the contents of chapter five as follows:

> The substances discussed in this chapter are represented by the general formula R_4PX, where X may be a halogen or a hydroxyl (free or in the form of a salt with an acid).

(CX-21 at 78).

115. Chapter 11 of the 1950 Kosolapoff text, which is not cited in the Kolb '320 patent, is entitled "Quasi-Phosphonium Compounds," and describes compounds containing a phosphorus atom bonded through covalent bonds to both carbon and nitrogen atoms. (RX-103 at 327; Kolb, Tr. at 469-470; RX-5).

116. Complainant's Harwood agreed that at least in the 1950 Kosolapoff book, referred to in the '320 patent, it would appear that the term "R" would be limited to organic radicals which are attached by a carbon phosphorus bond (Harwood, Tr. at 1280, 1281).

117. With respect to the use of "R", Ausimont's expert Schlosser testified:

> A. It is both common practice and recommendation that you should use R for organic moiety, a carbon bonded moiety and X for an electronegative substituent amino

> group, hydroxy and so on.
>
> If you use simply R without any specific mention, it would be misleading to come later and say, well, I meant nitrogen, I meant oxygen, I meant thallium.
>
> If you use R and you specify as I have done R equals, then there is no harm even if somebody may come and say you should not have used R but another letter but it is unequivocal.
>
> Say there is no specification given with a general form of R, people can rely R means something organic and X means something electronegative. [Schlosser Tr. at 2122-2123]

118. Kosolapoff identifies over 200 phosphonium compounds in chapter five, all of which involve a phosphorus atom bound to four carbon atoms. (CX-21 at 86-97; Harwood, Tr. 1279).

119. Chapter 10 of the 1950 Kosolapoff book (RX-33) is entitled "Compounds with Phosphorus to Nitrogen Bonds," and chapter 11 (RX-33 and RX-103) is entitled "Quasi-Phosphonium Compounds." Neither chapter 10 nor chapter 11 is referred to in the '320 patent.

120. Quasi-phosphonium compounds are described by Kosolapoff in the book cited in the '320 patent as follows:

> The substances that are the subject of this chapter may be regarded as variants of the usual phosphonium compounds, R_4PX, in which one or more radicals R are replaced by ester groups, OR, or amino groups, RNH or R_2N, or as variants of the polyhalides, RPX_4, R_2PX_3, and R_3PX_2, in which radicals R are similarly substituted.

(RX-103 at 324).

121. The Introduction of the 1950 Kosolapoff book, cited in the '320 patent, contains the following definitions:

> **R_4PX.** Quaternary phosphonium compounds. Et_4PCl - tetraethylphosphonium chloride; $MeEt_3POH$ - methyltriethylphosphonium hydroxide. * * *

> **Quasi-phosphonium compounds.** Substances analogous to the true phosphonium compounds and to the phosphorus halides, but containing ester or amide groups. $PhOPCl_4$ -phenoxyphosphorus tetrachloride; $(MeO)_2Et_2PI$ -dimethoxydiethylphosphorus iodide.

(RX-103 at 4-5).

122. Kolb, the inventor of the '320 patent and one of ordinary skill in the art at the time, did not consult a 1972 Kosolapoff and Maier monograph prior to filing the application leading to the '320 patent. (Kolb, Tr. 614-15).

123. Kolb has <u>never</u> consulted <u>any</u> of references relied upon by complainant's experts to support their asserted definitions of quaternary phosphonium compound. (Kolb, Tr. at 617-621).

124. The '320 patent does not refer to the 1972 Kosolapoff and Maier monograph.

125. The list of compounds following the text of Chapter 4 of the 1972 Kosolapoff and Maier monograph is titled "Quaternary Phosphonium Compounds" and has "A", "B", "C", "D", "E", "F" and "G" subheadings. The "G" with the subheading "List Of Compounds" is divided into "(A) Quaternary phosphonium salts; (B) Di-quaternary salts: (C) Betaines; and (D) Salts with less than four carbon atoms attached to phosphorus (quasi-phosphonium salts)." (CX-86 at 189, 190, 215).

126. The authors of the 1972 Kosolapoff and Maier monograph state before the subheading "A":

> The substances discussed in this chapter are represented by the general formula R_4PX, where R may be a radical linked to phosphorus by carbon or another atom; X may be any compound, simple or complex, inorganic or organic, serving as an anion. Also included are the substances which bear the negative charge in one of the ligands. They are referred to as betaines.
>
> All the substances which appear in the literature under the

> heading "phosphonium" have been taken into the list of compounds. No critical selection has been done, whether they belong to the tetra- or pentacovalent state of phosphorus. For the compounds with less than four carbon bonds to phosphorus it can often be assumed that there is an equilibrium between both states. Certain quaternary phosphonium salts are soluble in ethers. This may indicate that a pentacovalent state is valid even here.

(CX-86 at 190).

127. All of the working examples in the '320 and '877 patents that use a phosphonium compound, as distinguished from an ammonium compound, use a compound in which the phosphorus atom is bound directly to four carbon atoms. (CX-1, column 12, Tables 1 and 3).

128. The ten "representative quaternary phosphonium compounds" disclosed in the '320 patent all contain a phosphorus atom bound to four carbon atoms. (CX-1, column 6, lines 31-48).

129. The '320 patent states that particularly preferred polymers which may be crosslinked in accordance with the invention are the fluorinated elastomers produced by copolymerizing perfluoropropene and vinylidene fluoride, as described in U. S. Pat. Nos. 3,051,677 (the '677 patent) which issued to Rexford and 3,318,854 (the '854 patent) which issued to Honn and those terpolymers produced by copolymerizing perfluoropropene, vinylidene fluoride and tetrafluoroethylene as described in U. S. Pat. No. 2,968,649 (the '649 patent) which issued to Pailthrop. (CX-1, col. 4, lines 58 to 64).

130. The '649 patent <u>supra</u> issued on Jan. 17, 1961 on an initial application filed on Dec. 4, 1958 and is assigned on its face to du Pont (CX-5). The '677 patent issued on August 28, 1962 on an initial application filed on April 27, 1955 and is also assigned on its face to duPont (CX-6). The '854 patent issued on May 9, 1967 on an initial application filed Feb. 5, 1960 and is assigned on its face to complainant. (CX-8).

131. The '649 patent is directed to terpolymers formed by copolymerizing vinylidene fluoride, hexafluopropene, and tetrafluoroethylene in certain ratios to give novel elastomers with improved physical and chemical properties. The patent discloses that the terpolymers may be cured with the same curing agents by the same procedures as for curing the vinylidene fluoride and hexafluoropropene copolymer. Suitable curing agents are hexamethylenediamine carbamate, benzoyl peroxide, high energy radiation, N,N'-aryldienediamines, aliphatic and cycloaliphatic diamines. An acid acceptor such as magnesium oxide, zinc oxide and disodium phosphate is used in combination with the curing agents. It is also taught that the terpolymer may be compounded with reinforcing agents such as carbon black and silica and that pigments may be incorporated for color effects. (CX-5, col. 1, lines 12-15, col. 2, lines 30 to 41).

132. The '677 patent discloses that the claimed elastomeric copolymers can be further treated to produce elastomers of exceptionally good physical and chemical properties; that this subsequent treatment is a curing process which probably causes cross-links to be established throughout the copolymer; that curing and compounding of polymers is well known in the art and can conventionally be carried out by the usual methods for this type of polymer; that particularly useful in curing the copolymers of the invention are the peroxides, specifically benzoyl peroxide, or polyamines; that also useful is a cure obtained with ionizing irradiation; and that when using peroxides improved results are obtained by adding free radical acceptors. (CX-6, col. 4, lines 20-40).

133. The '854 patent relates to the crosslinking of fluorinated linear, saturated polymers and to the vulcanization of fluorinated, linear,

saturated elastomers. The patent teaches that while linear or chain polymers are thermoplastic in nature, vulcanized or cross-linked elastomers may have equal, greater, or lesser extensibility than the linear elastomers, depending on the number and nature of the crosslinkages and that in any case they have lessened plasticity and solubility and increased toughness and heat resistance. (CX-8, col. 1, lines 12-16, lines 25-30).

134. The '854 patent discloses that the copolymerization may be carried out in either a water suspension type system or in a mass polymerization system; and that in the latter system organic peroxide promoters are used. It is taught that in general the crosslinking agents react to remove a hydrogen or halogen atom from a carbon atom on the polymer chain and thereby produce a free radical spot on the chain which is capable of linking to a similar free radical spot on another chain, either directly or indirectly; that among the crosslinking agents which may be used are the peroxy-type compounds, basic metal oxides and inorganic polysulfides; and that while it is not desired to be bound by any particular theory of operation, it is believed that the peroxy-type compounds remove a hydrogen atom from a carbon atom on the linear chain and thereby produce an activated free radical spot on the chain which spot links directly to a similar free radical spot on another chain and thus produces a cross-linked polymer. (CX-8, col. 2, lines 68-71, column 3, lines 15 to 65).

135. The '320 patent discloses that quaternary ammonium compounds useful in preparing curable fluoroelastomer compositions are compounds which contain at least one nitrogen atom covalently bonded through carbon-nitrogen single bonds to four organic radicals and additionally through an ionic bond to an anion, and that such materials are described for example in U.S. Pat.

No. 3,655,727, col. 4, line 65 to col. 5, line 2 and U.S. Pat. No. 3,752,787 at col. 6, lines 1 through 22. (CX-1, col. 6, lines 50 to 58).

136. U. S. Patent No. 3,752,787 (the '787 patent) to de Brunner and assigned on its face to duPont issued on August 14, 1973 on an initial application Ser. No. 44,884 filed on June 9, 1970 and application Ser. No. 128,184 filed March 25, 1971 (CX-14, RX-5). It is cited under "References Cited" in the '320 patent (CX-1). The '787 patent is titled "Fluoroelastomer Composition Containing A Triarylphosphorane Vulcanization Accelerator." The '787 patent discloses that a fluoroelastomer composition which has beneficial utility in the manufacture of cured fluoroelastomer articles having good resistance to compression set can be prepared by blending the following components: (A) an elastomeric copolymer of vinylidene fluoride and another fluorinated monomer; (B) a vulcanization accelerator comprised of a triarylphosphorane compound such as (carbethoxyethylidene)-triphenylphosphorane which is used either alone or in combination with a certain quaternary ammonium compound or a certain guanidine or amidine compound; and that when making the cured articles it is also preferred to add (C) a suitable metal compound such as divalent metal oxide or hydroxide and optionally a metal salt of a weak acid; and (D) a cross-linking agent composed of a suitable polyhydroxylic-aromatic compound (Col. 1, lines 18 to 32) which radicals are alkyl, aralkyl, fluoroalkyl, polyoxyalkylene on polyoxyfluoroalkylene. The quaternary ammonium compound has a formula wherein four carbon containing radicals are covalently bonded to the nitrogen which radicals are alkyl, aralkyl, fluoroalkyl, polyoxyalkylene or polyoxyfluoroalkylene (col. 6, lines 1 to 20). The '787 patent discloses that cross-linking of highly fluorinated polymers with aromatic polyhydroxylic

compounds must be carried out in the presence of a catalyst and that catalysts which accelerate vulcanization or curing of elastomers are generally referred to as "vulcanization accelerators" (col. 4, lines 44-54). The '787 patent also discloses that one skilled in the art would realize that the best curing time and temperature for a particular application will depend on such factors as the nature and proportion of ingredients and the properties needed in the final product; and that the exact nature is not yet known of the chemical reaction involving the accelerator during cure of the composition and the accelerator reaction products present in the cured material. (col. 6, lines 74-75, col. 7, lines 1 to 7).

137. The term "quaternary" is used in the same manner when relating to either a phosphonium or ammonium compound. (Harwood, Tr. 1266-67).

138. It is appropriate to look to the nomenclature of quaternary ammonium compounds for guidance as to the appropriate nomenclature for quaternary phosphonium compounds. (Harwood, Tr. at 1117-18, 1126).

139. French patent 2,096,115, issued to Pattison of duPont on January 17, 1972, and describes an accelerator having a phosphorus, arsenic, or antimony atom connected to four groups, which groups can be substituted or unsubstituted aryl, aralkyl or alkylene groups. (CX-16). French patent 2,091,806, which also issued to Pattison, gives similar limitations for quaternary ammonium compounds. (RX-11).

140. The terms alkyl, aryl, alkylene, fluoroalkyl, and the like, all refer to organic radicals attached to the structure in question by a carbon bond. (Harwood, Tr. 1267-69).

141. The *Standard Chemical and Technical Dictionary*, published in 1939, defines an "ammonium base, quaternary" as a "[c]ompound that may be

regarded as derived from ammonium hydroxide by replacing the four hydrogen atoms by hydrocarbon radicals, e.g. R_4NOH." (SX-8, at 27).

142. The term "hydrocarbon" means a compound having only carbon and hydrogen atoms, and the hydrocarbon radicals attached to the ammonium are attached through carbon atoms. (Harwood, Tr. 1269-71).

143. *Chambers's Technical Dictionary*, published in 1961, defines a "quaternary ammonium base" as a compound derived from ammonium hydroxide "in which the four hydrogen atoms attached to the nitrogen are replace by alkyl radicals. . . ." (SX-9 at 693).

144. The *Dictionary of Rubber Technology*, published in 1969, defines "quaternary ammonium compounds" as "[o]rganic compounds having four alkyl groups attached to nitrogen." (SX-10 at 125).

145. Complainant's outside expert witnesses could not identify any chemical or technical dictionary that contained a definition of quaternary ammonium or phosphonium compound which supported complainant's asserted definition that the four substituents could be anything but hydrogen or one of its isotopes. (Engel, Tr. at 1404-05; Harwood, Tr. at 1163-64). Complainnant's outside expert witnesses could not identify <u>any</u> literature references which gave such a definition for quaternary phosphonium or ammonium compounds. (Harwood, Tr. at 1170-72; Engel, Tr. at 1405, 1853).

146. Complainant's Harwood and Engel could point to no literature reference, as of October 16, 1973, that defines a quaternary phosphonium compound as a phosphorus compound bearing a positive charge on the phosphorus atom and being covalently bonded to four constituents none of which is hydrogen. (Harwood, Tr. at 1170 to 1172, 1405, 1853).

147. *Organic Nomenclature: A Programmed Introduction*, published in

1966, states that "[a]mmonium compounds in which four alkyl groups are bound to nitrogen are called quaternary ammonium compounds." (SX-13 at 72) (underlining in original).

148. *Organic Chemistry*, by Morrison and Boyd, published in 1973, is a textbook for college students. (Harwood, Tr. at 1173).

149. The Morrison and Boyd textbook defines a primary carbon atom as a carbon attached "to only one other carbon atom." (SX-15 at 85). Similarly, cations are deemed primary, secondary, or tertiary by "the number of alkyl groups attached to the electron-deficient carbon." (SX-15 at 164). Similar terminology is used for alcohols (SX-15 at 166) and amines (SX-15 at 731). Quaternary ammonium compounds are described as having the formula $R_4N^+X^-$, where the nitrogen is covalently bonded to "[f]our organic groups." All of the examples of quaternary ammonium compounds in the text with full structural formulae, as opposed to the abbreviated R for radical, involve nitrogen atoms bound to 4 carbon atoms. (SX-15 at 452-53).

150. Linstromberg's "Organic Chemistry", published in 1970, indicates that a quaternary ammonium salt is obtained by progressive alkylation of ammonia. (RRX-171 at 351).

151. Baker & Engel, *Organic Chemistry*, published in 1992, indicates that "[c]arbon atoms are classified [as primary, secondary, tertiary, and quaternary] on the basis of the number of other carbon atoms bound to them." (SX-14 at 57; Engel, Tr. 1948). "Quaternary ammonium ions" are described as "ions in which four alkyl (or aryl) groups are attached to a positively charged nitrogen, ..." (SX-14 at 735; Engel, Tr. 1570-74 and 1897-1903).

152. Technical chemistry references ranked in complexity from least sophisticated to the most sophisticated are (1) chemical or technical

dictionaries, (2) undergraduate textbooks, (3) treatises or compendia, (4) reviews and monographs, and (5) original research literature. (Engel, Tr. 1949-51).

153. The book *Phosphorus and its Compounds*, by Van Wazer of Monsanto, published in 1958, addresses quaternary phosphonium compounds and quasiphosphonium compounds separately. (SX-5 at 211-215). Van Wazer describes a compound where a phosphorus is attached to one carbon (a phenyl group) and three nitrogens (NHC_6H_5 groups) as a "[m]ixed phosphonium-quasiphosphonium compound." (SX-5 at 215).

154. Complainant's Harwood and Engel are unfamiliar with the fluoroelastomer art in 1973. (Harwood, Tr. at 1053-54; Engel, Tr. at 1904-09).

155. The definitions of quaternary phosphonium compound offered by Harwood and Engel are limited to chemists. (Harwood, CX-231 at 39-41; Engel CX-242 at 17-26). Neither Harwood nor Egnel testified that their definitions were used by persons of ordinary skill in the fluoroelastomer compounding art in 1973.

156. Engel did not develop his own definition for quaternary phosphonium compound until after he obtained his Ph.D. and began working in the organophosphorus field. (Engel, Tr. at 1955).

157. Neither the rules of the "International Union Of Pure and Applied Chemistry" (IUPAC) (RX 36) nor of Chemical Abstracts call a compound having a phosphorus- nitrogen bond in it a quaternary phosphonium salt. (Schlosser, Tr. at 2014).

158. Inventor Kolb testified as to the definition of quaternary phosphonium compound:

Q	Isn't correct -- let me start again. The only description you give in your patent for quaternary phosphonium compounds are useful in preparing a fluoroelastomers is the description starting at column 5, line 66; isn't that right?

A	No, it's not right.

Q	Where else do you describe quaternary phosphonium compound in your patent?

A	Claim 1.

Q	Okay. In claim 1, element B you simply recite quaternary phosphonium compound; isn't that correct?

A	Yes.

Q	So your quaternary phosphonium compound is quaternary phosphonium compound in claim 1; isn't that correct?

A	Yes.

Q	You consider that a description of quaternary ammonium compound?

A	I consider that a description, yes, for quaternary phosphonium?

Q	That is like saying a bear is a bear, or red is red, or green is green. Those are descriptions.

A	The difference between the claim 1 in the line 66, is line 66 calls for --

JUDGE LUCKERN: Claim 66 are you talking about, line 66 at column 5?

THE WITNESS: Yes. Starting there. There it calls out for carbon radical to the phosphorus and in claim 1 it did not necessarily state that.

MR. STIEFEL: So, now, let's go back to my original question. The only description where you describe what you mean by quaternary phosphonium compound, other than saying, as you do in claim one, quaternary phosphonium compound, appears at column 5, starting at line 66; isn't that correct?

A	Column line -- that description describes a quaternary phosphonium, yes.

Q	Is there any other description in your patent of quaternary phosphonium compound apart from what appears starting at column 5, line 66?

* * *

THE WITNESS: I thought I answered that. In the column in the body of this patent, we describe a quaternary phosphonium compound and, in that particular instance, it has four substituents that are carbon, that are bonded jointly to the phosphorus.

That is what is stipulated in the body of the text. Now, in claim 1 we also describe a quaternary phosphonium but in this case it does not stipulate that there be four carbon radicals attached to the phosphorus. That is my answer.

(Kolb, Tr. at 462 to 465).

159. Inventor Kolb testified as to what is included in his '320 patent:

Q	The reason you didn't refer to what is sometimes referred to as -- as amino phosphonium compound is that, at the time you filed your patent application on October 16th, 1973, you didn't contemplate amino phosphonium compounds; isn't that right?
A	Yes.

* * *

Q	What does the patent specifically say way it describes quaternary phosphonium compound?
A	The patent simply states an example of a quaternary phosphonium compound --
Q	What is that example that the patent specifically states?
A	Well, the patent specifically states that there would be four organic radicals connected to the phosphorus by definition of a quaternary phosphonium compound.
Q	And it further indicates that those organic radicals are bonded to the phosphorus each through a carbon atom; isn't that correct?
A	In Claim 1, the answer would be no. In the body of the text, the answer would be yes.

Q Claim 1 defines quaternary phosphonium compound by the same term, quaternary phosphonium compound?

A Yes, it does.

Q Is that your idea of a definition?

A That can be, yes.

(Kolb, Tr. at 470, 479, 480).

160. Robert Kolb and Werner Grootaert were the named co-inventors of U.S. Patent No. 4,912,171 (the '171 patent) based on an application filed April 1, 1988 and which issued on March 27, 1990. The patent is assigned on its face to 3M. (RX-16 at 1).

161. The '171 patent is directed to curable fluoroelastomer compositions incorporating organo-onium compounds such as quaternary phosphonium compounds and also amino phosphonium compounds which are capable of functioning as vulcanization accelerators in the vulcanization of fluoroelastomers. (RX-16, col. 4, lines 51-54).

162. Quaternary organo-onium compounds disclosed in the '171 patent are said to be described in the Patel et al. '727 patent. (RX-16, col. 4, lines 23-45).

163. The '171 patent discloses that the aminophosphonium compounds are described in Ausimont's Moggi et al. '463 patent. (RX-16, col. 4, lines 23-45).

164. The 1939 Standard Chemical and Technical Dictionary defines a quaternary ammonium base as a compound that may be regarded as derived from ammonium hydroxide by replacing the four hydrogen atoms by hydrocarbon radicals. (SX-8).

165. The 1961 Chambers Technical Dictionary defines a quaternary ammonium base as one in which the four hydrogen atoms attached to the nitrogen

of ammonium hydroxide are replaced by alkyl radicals. (SX-9).

166. The 1969 Dictionary of Rubber Technology defines quaternary ammonium compounds as organic compounds having four alkyl groups attached to nitrogen. (SX-10).

167. The Morrison and Boyd 1973 college text, Organic Chemistry, describes quaternary ammonium compounds as those having the formula $R_4N^+X^-$, wherein the nitrogen is covalently bonded to four organic groups. The 1966 edition of the same book gives the same definition. (RX-13, at 752-753; RRX-169).

168. Pertinent literature, consisting of (1) patents such as the de Brunner U.S. 3,752,787 at col. 6, lines 1-14 (CX-14), Pattison U.S. 3,876,654 at col. 1, lines 50-56 (RX-10), Patel et. al. U.S. 3,712,877 at col 4, lines 17-63 (RX-25), Patel U.S. 3,655,727, col. 2, lines 52-69 (CX-11), and (2) technological dictionaries, and basic textbooks such as Bennett, Standard Chemical and Technical Dictionary, The Chemical Publishing Co., Inc. (1939), pp. xxi, 27-28, 439, 483 (SX-8) Chambers, Technical Dictionary, 3d ed., MacMillan Company (1961), pp. 636 and 693 (SX-9), Craig, Dictionary of Rubber Technology, Philosophical Library Inc. (1969), pg. 125 (SX-10), Morrison and Boyd, "Organic Chemistry", 3rd Ed. 1973, pp. 31-32, 752-753 (RX-13) Kosolapoff, G.M., Organophosphorus Compounds, (John Wiley & Sons 1950), pp. 1-9, 78-97, 324-355 (RX-103), Morrison and Boyd, "Organic Chemistry," Second Ed., pp. 733, 748 and 749 (1966) (RRX-169), and Nomenclature of Organic Chemistry, I.U.P.A.C., Organic Chemistry Division, Commission on Nomenclature of Organic Chemistry, Section D5, Prepared by J. Rigaudy and S.P. Klesney, Pergamon Press: Oxford, pp. 323, 371, 393-393 (1979) (CRX-36) shows that quaternary ammonium compounds are compounds having four carbon-nitrogen bonds.

(SX 8-10; RX-103 at 78; RX-13 at 752-753; RRX-169).

169. Quaternary phosphonium compounds have four carbon-phosphorus bonds. The phosphorus carries partial or total positive charge. (Schlosser, Tr. at 1993; Schlosser RX-145 at 1; RX-36).

170. Under the nomenclature rules of the International Union of Pure and Applied Chemistry, a compound having a phosphorus nitrogen bond could not be called a quaternary phosphonium compound. (Schlosser, Tr. at 2014; RX-36).

171. Under the nomenclature rules of Chemical Abstracts a compound having a phosphorus-nitrogen bond could not be called a quaternary phosphonium compound. (Schlosser, Tr. at 2014).

H. Person Of Ordinary Skill In The Art

172. The Kolb '320 invention would be developed or practiced by persons involved in rubber compounding, and typically by people who were using fluoroelastomer materials or who were working for suppliers of such materials. In about the 1973 time frame, formulation and compounding work in fluoroelastomers was generally conducted by persons whose training and experience was developed "at the bench" or "on the job", i.e. much of their knowledge resulted from actual practice and practical experience, rather than from a theoretical classroom approach. They would not necessarily have had a bachelor's degree, but typically they would have at least graduated from high school. Many would have had some chemical or science education in college or technical school, but not necessarily a degree. While the experience level on the job, before they were involved in formulating on their own, would vary, it would typically be about three to five years of practice, assuming that their work was not exclusively formulation but also involved in routine compounding

and testing. If the job required them to be constantly involved in formulation, with relatively few other duties or expectations, they might have developed sufficient skill to be involved in their own formulation work with as little as about one to two years of practice. (Harwood, CX-231 at 52, 53).

173. The principal field in which the Kolb patent would be practiced is in the field of compounding fluoroelastomers and curing them. In general, the particular application would be practiced by those who are interested in modifying fluoroelastomer compounds for processing, to develop improved processing characteristics. In about 1973, a person involved in compounding or formulating fluoroelastomer compositions would typically have been a person having experience in rubber compounding and in the use of equipment to process rubbers. If that was their only focus of work, in about 1 to 2 years they would have had sufficient experience to be involved on their own in formulating compositions such as those described in the Kolb '320 patent. With respect to their educational level, they would typically have at least a high school education. Many of the people would have several years of college, but not necessarily a college degree. (Worm, CX-132 at 6, 7).

174. In about October of 1973, the field to which the art of the Kolb '320 patent pertained would have been the rubber compounding field. Thus, users of the kind of technology of concern in the Kolb '320 patent would have either been in companies or industries which use fluoroelastomers as raw materials to generate products such as gaskets or seals, or they would be involved with companies such as complainant, duPont or Ausimont who manufacture and supply the raw materials. In about the 1973 time frame, formulation and compounding work in fluoroelastomers and other polymers was generally conducted by persons whose training and expertise was developed on

the job. That is, they would become experienced and knowledgeable about fluoroelastomer compounding as a result of field or industrial exposure and activities. They would not necessarily have a Bachelor's degree, but would typically, at least, have graduated from high school and often would have had some chemical or science background through post-high school education, for example, technical school education or a few years of college. Their experience level on the job would vary, but typically they would have had at least about three to five years experience in formulating compounding and curing systems. If their work was particularly intensive, and they were associated with a department which comprised others who were well experienced, they may have developed their skills to the level of ordinary skill in less time. (Engel, CX-242 at 41, 42).

175. A person skilled in the art to practice the '320 patent probably would have to have at least a high school education and would have to have some experience with processing equipment that is used in the rubber industry, so he could tell the difference between whether the process was good or bad, and he would have to have some compounding experience. Thus he would have to have experience either in determining where he could obtain certain chemicals or additives to a composition by looking at data that is supplied by suppliers. He would have to be able to understand, for example, what carbon blacks he needed to use. The same thing would be involved if he is practicing in the art. Thus he would have to know where he could obtain the bisphenol-type compounds and which ones are commercially available. The same thing with the phosphonium component, <u>i.e.</u> he would have to know where he could go to obtain the phosphonium component or who to ask as to which would be the best or what phosphoniums components are available in 1973, so he could use them.

The same thing with the fluoroelastomers, i.e. he would have to go and request information on the fluoroelastomers and make a choice on which one he would want to use. Depending upon how smart he is, he would have anywhere from six months to two years at the work bench. (Worm, Tr. at 700 to 702).

176. The scientific journal articles admitted as exhibits CX-51 through CX-99 would not have been consulted by a person with only a few years of college education. (Harwood, Tr. at 1335-36).

177. People not active in the organophosphorus or nitrogen chemistry fields would be likely to rely on chemical dictionaries to determine the definitions of the chemical terms quaternary phosphonium or ammonium. (Harwood, Tr. at 1277-78 and 1332).

178. Persons below the graduate level of chemistry education would retain the definitions of chemical terms learned during undergraduate courses although such persons might not even retain that much information. (Harwood, Tr. at 1277-78).

179. People of ordinary skill in the art in the field in issue would be "what we call rubber compounders" (Brullo, Tr. at 448).

180. Prior to the filing of the '320 patent application, inventor Kolb did not review any of the scientific journal articles submitted by 3M that were published before 1973, specifically CX-51, CX-52, CX-53, CX-56, CX-57, CX-58, CX-59, CX-63, CX-67, CX-68, CX-70, CX-73, CX-78, CX-83, CX-84, CX-88, CX-93, CX-94, CX-95, CX-96, CX-97, and CX-99. (Kolb, Tr. at 616-21). Kolb has never reviewed most of this literature. (Kolb, Tr. at 617-621).

181. Kolb, prior to his testimony at the hearing, had never read or seen any of the references relied upon by complainant's experts, in forming their opinion as to the meaning of "quaternary phosphonium compound," either

before or after October 16, 1973. (Kolb, Tr. at 617-621).

182. Harwood testified as to the knowledge of one skilled in the art (Tr. at 1335, 1336):

> JUDGE LUCKERN: Let me ask you this: Would a person as you have described this person on page 52 in your witness statement in 1973, if that person wanted to know what a -- what quaternary phosphonium compound embraces, would he go to something like this CX-86, each of these things you testified to on 40 and 41?
>
> * * *
>
> JUDGE LUCKERN: In your opinion would such a person, if they wanted to determine what a quaternary phosphonium compound is in the fluoroelastomer art, would they know enough to go and go into these articles in order to come to some meaning as to what this term includes, embraces, consists of?
>
> THE WITNESS: I think they would have to be pretty good, you know, in the use of a library or have a librarian help them. If they really wanted to know about quaternary phosphonium compounds, he would -- he probably could find these articles. They probably would be a little advanced for many of them, to be honest.

Harwood also testified (Tr. at 1112):

> I believe that the general impression of people really working in this area [on October 16, 1993] would have a broad definition of it. Most of the quaternary phosphonium salts that were, let's say, available around at the time had four carbon-phosphorus bonds, but there was no limitation to that side. I've never seen in any article or anything like that a limitation that you must have four carbon phosphorus bonds. I mean, the people who do research in this area would be interested in materials that had subconstituents other than carbon.

Harwood further testified (Tr. at 1277, 1278):

> Q But for someone who is not delving into research in those particular areas they might be more likely to actually rely on the type of dictionary definitions that we've just reviewed?
>
> A I'm afraid that probably would be true, yes.
>
> Q And someone who is not a graduate student in chemistry would be likely to remember no more than what they learned in their undergraduate chemistry courses?
>
> A I think so.

Q Certainly that's true for me.

A Most people even try to forget that.

I. Phosphonium Compounds

183. Phosphonium compounds are generally members of a class of cations, often referred to as "oniums". These are cations in which the positive charge is associated with an element of group 5A or 6A of the periodic table, most typically nitrogen, phosphorus, oxygen or sulfur, <u>i.e.</u>, phosphonium, ammonium, oxonium and sulfonium compounds. Onium compounds are salts of those various cations, with an associated anion which has a negative charge. (Engel, CX-242 at 16, 17).

184. The phosphorus and the nitrogen atoms at least to some extent, the lone pair of electrons which is ordinarily associated with the nitrogen atom. As a result, the phosphorus-nitrogen bond takes on a partial double bond characteristic, and the positive charge is delocalized between the phosphorus and nitrogen atoms. (Schlosser, Tr. at 20377-39; RPX-9).

185. The cation of GM 102 E has a single positive charge. (Engel, CX-242 at 23).

186. Quaternary phosphonium cations have a single positive charge. (Engel, CX-242 at 17).

187. GM 102E and quaternary phosphonium compounds have four organic substituents which shield the positive charge from polar solvents and permit the molecule to move between polar and non-polar phases, thereby acting as phase-transfer catalysts. (Engel, Tr. at 1504-10). This was observed by the phase transfer experiments carried out by Harwood in solution. (Harwood, CX-231 at 45 to 47).

188. Several U.S. patents relating to curable fluoroelastomer compositions group several types of "onium" accelerators together, including ammonium, phosphonium, and amino-phosphonium accelerators. See, e.g., SX-1, U. S. Pat. No. 4,882,390 col. 4, lines 33-44 which issued Nov. 21, 1989 on an application initially filed on April 1, 1988 and is assigned on its face to complainant; SX-3, U. S. Pat. No. 5,086,123 column 4, line 63 to column 5, line 18 which issued Feb. 4, 1992 on an application initially filed on Feb. 27, 1984 and is assigned on its face to complainant; SX-4, U. S. Pat. No. 5,216,085 column 5, lines 3-31 which issued in June 1993 on an initial application filed on Feb. 27, 1984 and is assigned on its face to complainant; and RX-16 U.S. Pat. No. 4,912,171 column 4, lines 37-49 which issued on March 27, 1990 on an initial application filed on April 1, 1988 and is assigned on its face to complainant.

J. Schmiegel Article

189. Walter Werner Schmiegel in *Die Angerwandte Makromolekulare Chemie* 76/77, Nr. 1122 at 39-65 (1979) (RX-97), which article was received on April 14, 1978 and was presented in Germany on April 17, 1978 and which article was titled "Crosslinking of Elastomeric Vinylidene Fluoride Copolymers with Nucleophiles" under the heading "Summary" states:

> The monomer sequence $CF^2CF(CF^3)CH^2CF^2CF^2CF(CF^3)$ has been identified ... as the selectively base-sensitive site of poly(vinylidene fluoride/hexafluoropropylene). Similar studies of the other vinylidene fluoride (VF^2) copolymers with either tetrafluoroethylene (TFE) or perfluoro(methyl vinyl ether) (PMVE) show that the generalized base-sensitive of VF^2 copolymers which contain one or more perfluorinated monomers can be formulated as....
>
> Depending on the nature of A, treatment with bases can lead to elimination of HF or the elements of CF^3OH ...

* * *

> The relative reactivity of HFP-VF^2-HFP and TFE-VF^2TFE sites toward basic nucleophiles was determined in solution at room temperature. Whereas the two sites undergo HF elimination with similar ease, subsequent nucleophilic attack on the formed unsaturation by unreacted base or another nucleophile clearly differentiates these two sites....

(RX-97 at 39-40) (emphasis added). Under the heading "Introduction", Schmiegel states:

> We have investigated the reactions of basic nucleophiles with vinylidene fluoride (VF^2) copolymers in solution and have determined some reactivity principles which can be used to interpret the vulcanization behavior of related fluoroelastomers.
>
> Copolymers of VF_2 and perfluorinated monomers such as hexafluoropropylene (HFP), tetrafluoroethlene (TFE) and perfluoro(methylvinylether)(PMVE) are prepared by emulsion polymerization using peroxide initiators ... With the exception of VF_2/TFE, choice of composition allows the preparation of all such dipolymers and terpolymers at sufficiently low levels of crystallinity and low glass transition temperatures to afford elastomers, as shown in Tab. 1. [Table 1 refers to the following commercial vinylidene fluoride copolymers base-curable: Viton A, 3M's Fluorel, Montecatini-Edison's Tecnoflon, Daiel and Viton B]. These fluoroelastomers, or more properly hydrofluoroelastomers in order to distinguish them from the ultra-high performance perfluoroelastomer poly(TFE/PMM VE/cure site monomer) ... would be expected to, and indeed do, possess outstanding resistance toward fluids and a wide variety of aggressive chemicals, as well as exceptional thermo-oxidative stability. The VF^2-based fluoroelastomers are generally vulcanized by basic curatives such as diamines and metal oxides and hydroxides.... [referring to U.S. Pat. No. 3,876,654 [RX-10] It is the purpose of this presentation, however, to consider only the reactions of normal VF_2 copolymers toward basic nucleophiles in solution and to develope some structure-reactivity generalizations which can be compared to experience with practical vulcanization.

(RX-97 at 40-42)

190. U.S. Patent No. 3,876,654 (RX-10) (the '654 patent), which issued on April 8, 1975 to D.B. Pattison on an initial application filed on June 9, 1970 (Ser. No. 44,883), on its face is assigned to Du Pont and is titled "Fluoroelastomer Composition." The '654 patent discloses, under the heading "Background Of the Invention," that it is necessary for many applications of highly fluorinated elastomers that the resultant fluoroelastomer article be

resilient and have low compression set which is accomplished by curing the article, <u>i.e.</u> by vulcanizing or cross-linking the elastomer. It is taught that prior art processes for vulcanization of fluoroelastomers use mainly aliphatic diamines as cross-linking agents, or "they carry out free radical initiated vulcanization in the presence of organic peroxides or high energy radiation". The '654 patent then disclosed a need for a fluoroelastomer vulcanization process which combines satisfactory cure rates with good processing safety and good storage stability of unvulcanized formulations and which yields vulcanized polymers of good stress/strain properties and low compression set. (RX-10, col. 1, lines 10 to 42).

191. The '654 patent under the heading "Summary of the Invention" discloses that expressed broadly, the "present invention" provides a fluoroelastomer composition comprising (A) an elastomeric copolymer of vinylidene fluoride and at least one other fluorinated monomer, and (B) as a "vulcanization accelerator" a quaternary phosphonium compound which is an alkyl- or aralkyl-triarylphosphonium compound. (RX-10, col. 1, lines 45-67, col. 2, lines 1 to 6).

192. The '654 patent, under the heading "Description of Preferred Embodiments" discloses that a useful approach is for the supplier of the fluoroelastomer composition to add one or both of a component (C) and component (D) before the composition is delivered to the person who makes the cured articles. Component C is a metal compound such as a divalent metal oxide and component D is a polyhydroxylic-aromatic compound cross-linking agent for the copolymer (col. 2, lines 28 to 38). All of the claims of the '654 patent are directed to a fluoroelastomer composition comprising components (A), (B) i.e. "as a vulcanization accelerator, a quaternary

phosphonium compound which is an alkyl-or aralkyl-triarylphosphnium compound," (C) and (D) (col. 15, 16).

193. The '654 patent describes quaternary phosphonium compounds as those compounds having four carbon groups bonded to a phosphorus atom. (RX-10, col. 1, lines 50-66).

194. The '654 patent discloses (RX-10, col. 5, lines 25 to 40, Col. 6, lines 55-68, col. 7, lines 1-4)):

> Cross-linking of highly fluorinated polymers with aromatic polyhydroxylic compounds must be carried out in the presence of a catalyst. Catalysts which accelerate vulcanization or curing of elastomers are generally referred to as "vulcanization accelerators." According to this invention, it is an important feature of the process to employ a quaternary compound of P, As of Sb as described above as the vulcanization accelerator. It is quite unexpected that the compound functions so effectively as a vulcanization accelerator for a fluoroelastomer in the presence of components C and D, with some of the best results of the invention being obtained with a component B content of only about 0.2-0.8 part per 100 parts of the elastomer.
>
> * * *
>
> The initial curing of the curable composition is preferably carried out by heating the composition for about 3-30 minutes at about 149°-204°C; conventional rubber-curing presses, molds, extrudes, and the like provided with suitable heating and curing means can be used. Also, if one wants a product having maximum heat resistance and dimensional stability, it is preferred to carry out a post-curing operation wherein the article is heated in an oven or the like for an additional period of about 1-48 hours at about 204°-260°C. One skilled in the art will realize that the best curing time and temperature for a particular application will depend on such factors as the nature and proportion of ingredients and the properties needed in the final product. The exact nature is not yet known of the chemical reaction involving the accelerator during curing of the present composition, and the accelerator reaction products present in the cured material.

195. Schmiegel in his article (RX-97 at 42, 43) under the subheading "1. Curing Behavior" refers to a curing system for vinylidene fluoride hexafluoropropylene copolymers and optimized for maximum resistance to high temperature compression set as consisting of a vinylidene fluoride copolymer

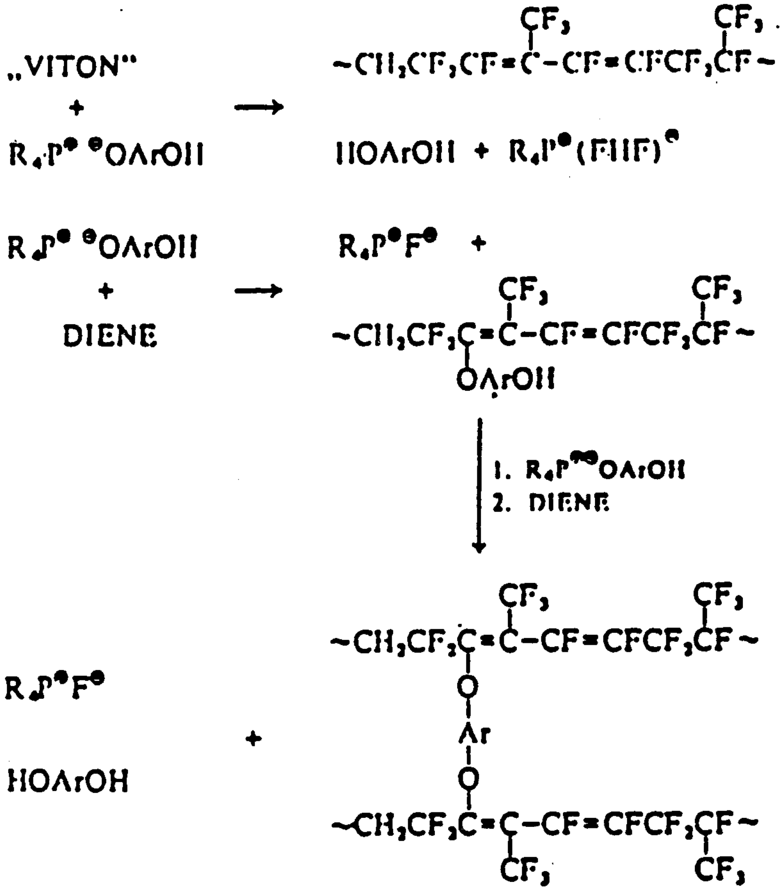
("VITON" E-60 - (3.5 vinylidene fluoride/hexafluoropropylene)), carbon black, calcium hydroxide magnesium oxide, benzyltriphenylphosphonium chloride (BTPPC) and bisphenol AF chloride. It is stated that all of the "curatives" are solids and only the bisphenol has a melting point below the cure temperature; that the ODR response is characterized by a distinct induction period whose length can be regulated easily by manipulation of the levels of inorganic bases or benzyltriphenylphosphonium chloride if a fairly constant cure state is desired, or by the level of the bisphenol, if sufficient freedom exists with respect to cure state; that high Bisphenol-AF levels markedly increase the length of the induction period and lead to high cure states; that the formation of crosslinks, once the process begins, is extremely rapid; that in the compound shown, over seventy five percent of the crosslinks are formed within one minute; that maximum cure rate is reached rapidly and without reversion; that only a two percent increase in cure state occurs between 13 and 60 minutes; and that after the press cure, to obtain optimum resistance to high-temperature compression set, the vulcanizate is subjected to an air oven cure between 230 and 260 degrees centigrade for 16 to 24 hours. It is stated that when BTPPC is omitted from the standard recipe, no cure occurs within one hour at 177 degrees and that if Bisphenol-AF is omitted, about seven percent of the cure state of the full compound is attained. Schemiegel concludes at 44:

> ... The highest BTPPC concentration examined leads to a cure state which is only 43% of that of the standard compound and which is characterized by very poor high temperature compression set resistance. The bisphenol-free compounds are also extremely scorch sensitive at 121°C. Thus, it appears that a substantial different and inferior kind of network can be formed in the absence of bisphenol and that, at the modest accelerator levels required for practical Bp-AF cures, the formation of the inferior network is greatly suppressed.

196. The Schmiegel paper has a section "3. Behavior of Polymer Solutions Toward Basic Bis-Nucleophiles" where solutions of poly(vinylidene fluoride and hexafluoropropylene) in the dipolar, aprotic solvent DMAC (dimethylacetamide) were treated with a cyclic amidine base and Bisphenol AF at room temperature. (RX-97 at 48). In that section he compared the results of "dehyrofluorination and gelation experiments run in solution with the results of vulcanization," and demonstrated that phenols actually became attached to the polymer chain and do not merely promote another process which forms crosslinks. Later he stated (RX-97 at 50, 51):

> To demonstrate that phenols actually become attached to the polymer chain and do not merely promote another process which forms crosslinks when a VF_2HFP polymer is treated with a bisphenol in the presence of base, the monofunctional analog of Bp-AF and [a cyclic amidine] DBU were used to treat the polymer (Fig. 8). After twice precipitating the polymer with an excellent solvent for free phenol or any unreacted phenolate, the washed polymer clearly showed the presence of the geminal trifluoromethyl groups. Under the conditions shown in Fig. 8, about 40% of the phenolate is incorporated based on the internal standard p-fluoroanisole.
>
> Because of the attachment of the mono-hydroxy analog of bisphenol-AF to the VF_2HFP polymer and in view of the nucleophilic attack of hydroxide ion on the unsaturated polymer, one concludes that in <u>practical vulcanization</u> a bisphenol-derived phenolate probably also attacks the intermediate diene and leads to the

dienic phenyl ether crosslinks shown in the following scheme [wherein positively charged R_4P is a phosphonium ion]:



Attack on the diene by phenoxide, unlike attack by hydroxide, is not expected to proceed beyond vinylic nucleophilic substitution and, therefore, the expected product is the phenyl ether that corresponds to the dienol proposed as an intermediate in attack by hydroxide.

The phosphonium ion, originally present as a chloride, is believed to undergo several cycles of conversion from fluoride or bifluoride to intermediate hydroxide to phenoxide to fluoride before exhaustion of the bisphenol. Ultimately the phosphonium ion is converted to triphenylphosphine oxide. [Emphasis added].

197. Schmiegel's paper proposed that the bisphenol/phosphonium complex was responsible for the initial dehydrofluorination, which forms the double bonds in the polymer. Due to the reactivity of the phenoxide complex, as

opposed to a hydroxide, Schmiegel also proposed that the dehydrofluorination, and eventually the crosslinking, only occurred at selective sites on the polymer backbone, specifically sites where a vinylidene fluoride moiety was surrounded by two hexafluoropropene moieties. (RX-97 at 47-51).

198. The Schmiegel paper appears in the record as a number of exhibits. RX-97 and RX-152 are English versions of the paper. CX-134 is a German version with a certified English translation. RRX-186 and RRX-187 are German versions with the same English translation although the translation is not certified.

K. Ausimont's Work

199. On Ausimont's work Tommasi testified:

> Ausimont started its activity on fluoroelastomers in the late 60's.... The monomer hexafluoropropene (HFP) was available because Ausimont was using it in its new proprietary process for production of perfluoropolyethers (FOMBLIN fluids). Tetrafluorethylene (TFE) was being used for PTFE [polytetrafluoroethylene] production (ALGOFLON) and also for different grades of FOMBLIN. So a pilot plant for production of vinylidenefluoride (VDF) and one for production of elastomeric copolymers (TECNOFLON) were built at the Research Center of Linate, near Milano.

In 1977 Tommasi found large pilot plants at Ausimont that were being used more for sale of polymer than for experimental purposes and for development of suitable industrial plants. He realized that the whole production process had to be revolutionized in all its stages: polymerization, coagulation, washing, drying.

200. On March 31, 1981 Ausimont's U.S. Patent No. 4,259,463 (the '463 patent) issued to Giovanni Moggi. It is based on an application filed on Dec. 8, 1978 which has foreign Italian priority dates of Dec. 14, 1977 and Nov. 20, 1978 and is titled "Vulcanizable Compositions Based On Copolymers Of Vinylidene Fluoride And Containing Vulcanization Accelerators Which Are Aminophosphinic Compounds." It is assigned on its face to Montedison S.p.A., Milan (CX-31). It is Ausimont's first patent application on its proprietary aminophosphonium- containing fluoroelastomer curing system (RX-22, CX-31; RRS 109(a)).

201. The '463 patent, in discussing the prior art, states (CX-31, col. 1, lines 10 to 50):

> The vulcanized elastomers based on vinylidene fluoride copolymer are well known to the Prior Art and are widely used in numerous applicative fields, where exceptional resistance to chemicals and solvents, lubricants, fuels, acids and similar products is required, even at very high temperatures.
>
> The vulcanized articles obtained from such elastomeric copolymers find their most suited application as sealing gaskets or packings in general, both in static as well as dynamic

> conditions, in the motor-engineering, aeronautical, missilistic, naval, mechanical, chemical field, in protective impermeabilizations of various supports such as for instance: protective garments for contact with aggressive chemical agents, sheathes for electrical cables exposed to high thermal radiation and lastly as protective coatings of industrial containers.
>
> According to the most advanced Prior Art for the vulcanization of the elastomeric copolymers of vinylidene fluoride, as vulcanizing agents there are used polynucleophilic compounds and in particular polyhydroxylic aromatic compounds (or analogous thioderivatives), either as such or in a salified form.
>
> These products leads, in fact, to vulcanized products of absolutely satisfying physical-mechanical characteristics and of an altogether satisfying thermal resistance.
>
> Such products have, however, the disadvantage of requiring extremely long vulcanizing times, <u>wherefore they are used in combination with substances having an accelerating action</u>.
>
> With the known vulcanizing systems, however, it is not possible to obtain a satisfactory adhesion to the metals of the vulcanized compositions, nor is it possible to carry out the injection molding of articles with short runs and in the absence of 'scorching" phenomena.
>
> Amongst the substances that develop an <u>accelerating action</u> according to the most advanced Prior Art, there are described derivatives of tertiary amines containing 4 (four) covalent nitrogen-carbon linkages and derivatives of tertiaryphosphines containing 4 (four) covalent phosphorous-carbon linkages (French Pat. Nos. 2,091,806 and 2,096,115). [Emphasis added]

202. French Patent No. 2,096,115 also referenced in FF 139 is based on U.S. priority application dated June 9, 1970 (Ser. No. 44, 883) and December 23, 1970 (Ser. No. 101,770) which issued to Dexter Brayton Pattison (CX-16). Serial No. 44,883 was the initial application that led to issuance of U.S. Patent No. 3,876,654 (RX-10) referred to in the Schmiegel article (RX-97). (<u>See</u> FF 190).

203. The '463 patent under the subheading "The Present Invention" states (CX-31, col. 1 lines 52 to 68, col. 2, lines 1 to 65):

> We have surprisingly found that some compounds containing 1 or more simple phosphorous-nitrogen linkages may be conveniently

used as vulcanization accelerating agents for fluoroelastomers.

Not all the compounds containing the P-N linkage do exert an accelerating action in the vulcanization of fluoroelastomers; for instance, non active are compounds such as phosphin-imine $(C^6H^5)^3P=N--C^6H^5$, while the corresponding isosteric phosphorane (see Brit. Pat. No. 1,413,857) $(C^6H^5)^3P=N--CH--R$ is described as an accelerating agent.

On the contrary, according to this invention there may be conveniently used the compounds obtained from the reaction of some tri-coordinated aminophosphinic derivatives such as for instance the reaction product between tri-coordinated aminophosphinic derivatives such as for instance the reaction product between tris-diethylamine of phosphorous acid $[(CH^3)^2N]^3P$ and alkyl halides.

There thus forms a compound whose action mechanism is still unknown but which presumably behaves as a ionic couple on the interface between an organic phase represented by the elastomer and an inorganic phase represented by charges of oxides and alkaline-earthy hydrates present in the vulcanization formula.

Thus, object of this invention is that of providing vulcanizable compositions based on elastomeric copolymers of vinylidene fluoride, containing as vulcanizing agents polynucleophilic compounds that be free of the above mentioned drawbacks.

Still another object of this invention is that of providing a vulcanizing process for compositions based on elastomeric copolymers of vinylidene fluoride free of the above mentioned drawbacks and that shall yields vulcanized products having a high degree of adhesion to metal substrate.

A third object of this invention is that of providing vulcanized compositions based on elastomeric compositions of vinylidene fluoride, and that be free of the above mentioned drawbacks and that shall yields vulcanized products having a high degree of adhesion to metal substrate.

These and still other objects may be attained with vulcanizable compositions consisting of:

(I) 100 parts by weight of an elastomer copolymer of vinylidene fluoride, with one or more fluorinated or chloro-fluorinated monomers, ethylenially unsaturated, such as for instance: 1-hydropenta-fluoropropene, 2-hydropenta-fluoropropene, 1,1-dihdrotetrafluoropropene, hexafluoropropene, tetre-fluoroethylene, trifluorochloroethylene, alkyl- and arylvinyl ethers, partially or totally fluorinated, and the like.

> (II) 1-40 parts by weight of an acceptor of inorganic acids ...
>
> (III) 0.5-10 parts by weight of one or more basic compounds ...
>
> (IV) 0.5-15 parts by weight, but preferably 1-6 parts by weight of a vulcanizing agent based on one or more polyhdroxyl and/or polythiol compounds ...
>
> (V) 0.05-5 parts by weight of a vulcanization accelerator based on aminophosphinic derivatives ... [Emphasis added]

204. The '463 patent on commenting on the anion of the Aminophosphinic compounds state (CX-31, col. 3, lines 50-53):

> Anion Y of valency m may be either organic or inorganic, as halide, perchlorate, nitrate, tetrafluoroborate, hexafluophosphate, oxalate, acetate, stearate, haloacetate, p.toluensulphonate, or as OH.

205. The '463 patent on commenting on the claimed vulcanizable compositions state (CX-31, col. 3, lines 63 to 69, col. 4 lines 1 to 50):

> The vulcanizable compositions of the above described type, are vulcanized by means of a process, likewise object of this invention, which consists in first heating said compositions under pressure and at temperatures comprised between 130° C, and 230° C, but preferably between 160° and 200° C, for a period of from 0.5 to 60 minutes, but preferably comprised between 1 and 20 minutes; by then post-vulcanizing the manufactured articles thus obtained in an oven or furnace, at atmospheric pressure, at temperatures comprised between 130° and 315° C, but preferably between 200°C, and 275°C, for a period of between 5 and 48 hours, but preferably between 10 and 24 hours.
>
> It has now surprisingly been found that the compositions vulcanizable according to this invention, may be transformed into manufactured articles of any shape and dimension, by extrusion forming and successive vulcanization, also using highly automatized injection molding techniques. In fact, at the usual plasticizing temperatures for injection molding no drawbacks are experienced because of scorchings or tearings under heat.
>
> Said manufactured articles display an excellent resistance to permanent set and to compression, a minimum tendency to scorching in relationship to time and storing temperature or to the temperature of particular processing techniques, such as for instance extrusion, and they also show a high resistance to

> thermal ageing. Moreover, they may be coupled to metal supports or supports of alloys thereof, towards which they show an exceptional adhesion even at high temperature, for instance at 250° C. and more.
>
> It was also noticed that the vulcanizable compositions, comprising the additives from (1) to (V) as indicated above, do not cause any phenomena of stickiness and soilability of the molds, wherefore production wastes are practically eliminated thereby allowing high production standards and extremely regular processing cycles.
>
> The process according to this invention process particularly indicated in the case of copolymers containing from 30 to 70 mols% of vinylidene fluoride and from 70 to 30 mols % of 1-hydropentafluoropropene and/or hexafluoropropane or of vinylidene fluoride/tetrafluoroethylene/hexafluoropropene (or 1 hydorpentafluoropene) terpolymers, in which the percentual quantities of the three monomers are comprised respectively between 40 and 80, 30 and 10 and 30 and 10 mols percent.
>
> More generally, the process according to this invention may be conveniently applied to all fluorinated polymeric materials of the elastomeric type, possibly containing substituents different from fluorine and chlorine, and also to mixtures of two or more fluorinated elastomers.

206. The '463 patent teaches that that polyhydroxyl and or polythiol compounds used in the claimed compositions "as vulcanizing agents" are those well known in the prior art (CX-31, col. 4, lines 51 to 54).

207. The '463 patent discloses preferred classes of products to be used "according to the invention as accelerators" (CX-31, col. 5, lines 1 to 3).

208. The '463 patent discloses that the quantity of "accelerating agent" to be used in the process according to the invention can vary (CX-31, col. 7 lines 37 to 45).

209. The '463 patent discloses as to another advantage of the claimed invention (CX-31, col. 8, lines 4 to 28):

> Lastly, another advantage, according to this invention, consists in the complete elimination of the undesired "flash shrinkage" in the closed pressurized vulcanizing mold on the fluorinated elastomer products, in particular in the case of O-ring gaskets.

> In a preferential form of embodiment of the process object of this invention, the mixture of the amino-phosphoric of this invention, the mixture of the amino-phosphoric compound or its metal complex (0.1-1 parts by weight), and the vulcanizing agent (1-6 parts by weight) is additioned in the fluorinated elastomer (100 parts by weight), before the addition of the acid acceptor (2-10 parts by weight), with the basic compounds (1-7 parts by weight), with the reinforcing and inert fillers, lubricants, plasticizers and with other possible addivites.
>
> Operating in this way, one achieves a fast, controlled and uniform vulcanization, without the danger of the appearance of undesired phenomena such as, for instance, scorchings during the various processing stages of the mix or during it storage. Similarly, any danger of losses due to volatilization during preparation and preservation of the vulcanizable compositions, is avoided, while, moreover, no special precautions are required on the part of the personnel in charge of the processing operations.

210. Table 1 of the '463 patent refers to the use of "Accelerator" with regard to the characteristics of the vulcanized articles. (CX-31, col. 10).

211. Example 2 of the '463 patent makes reference to different vulcanization mixes containing the polyhydroxyl "vulcanizing agents" and certain "accelerators" referenced in Table 2. (CX-31, col. 11, 12).

212. Example 4 of the '463 patent, with reference to Table 4, refers to the use of "accelerators" (CX-31, col. 17).

213. The '463 patent discloses (CX-31, col. 18, lines 10 to 67, col. 18, lines 1-12):

> It is necessary that there be a perfect balance between the vulcanization speed of the mix and the action of the attack agent.
>
> In the case of too high a vulcanization speed, the elastomeric part reticulates within a very short time, before the adhesive exerts its action. In such a case there occurs the complete coming off the elastomeric part from the metal insert. It is therefore absolutely necessary that the action of the <u>accelerator</u> develops in perfect concomitance with that of the adhesive. In other words, the vulcanization curve should show a not completely vertically upwards rising graph, so that from the minimum value to the maximum value there shall lapse a certain stretch of time (3-9 minutes).

> This requirement is completely satisfied by the <u>accelerators</u> of this invention.
>
> On Table 5, there have been recorded the values of the adhesive force, values that have been determined by means of an AMSLER dynamometer, for formulations of Tecnoflon containing <u>accelerators</u> A^1 and A^4 according to this invention (formulations 37 and 38), already indicated in example no. 1, and for comparative purposes, those obtained by using an <u>accelerator</u> of the Prior Art, of the type of quaternary ammonium salt (formulation 39) which exerts a <u>pronounced accelerating action</u>. [Emphasis added]

214. Independent claim 1 of the '463 patent claim vulcanizable compositions consisting of (I) 100 parts of weight of an elastomeric copolymer of vinylidene fluoride, with at least one fluorinated or chlorofluorinated, ethylenically unsaturated monomer, (II) 1-40 parts by weight of an acceptor, (III) 0.5-10 parts by weight of at least one basic compound, (IV) 0.5-15 parts weight of a vulcanizing agent based on at least one polyhydroxyl and/or polythiol compound and (V) 0.05-5 parts by weight of a vulcanization <u>accelerator</u> which is an aminophosphinic derivative that has a formula generic to GM 104E. (CX-31, col. 19 lines 40 to 69).

215. Ausimont's Moggi has referred to GM102E as a new accelerator (Moggi CPX-7 at 36). It was developed to replace a quaternary phosphonium accelerator which Ausimont was using in the 1970's. (Tommasi RX-162 at 15, 16).

216. Ausimont's U.S. Patent No. 4,544,708, (the '708 patent) issued to Moggi and Cirillo on October 1, 1985 which is titled "Vulcanizable Fluoroelastomer Compositions Providing Vulcanizates Having High Adhesion To Metals" and assigned to Montedison with a foreign Italian priority date of Feb. 24, 1983 claims a fluoroelastomer composition which includes metal complex anions bound to the traditional vulcanization accelerators. The specification indicates that the composition comprises, among other things:

> (D) as a vulcanization accelerator, a salt composed by a cation of phosphonium or of amino-phosphonium or of ammonium, the formula thereof is selected from amongst those already known in the art and broadly described for example in U.S. Pat. Nos. 3,876,654 [RX-10 to Pattison]; 4,259,463 [CX-31 to Moggi] and in GB Pat. No. 1,356,344 respectively. . .

Examples 34-45 use the accelerator where the cation is "benzyltriethylammonium" whereas examples 46-51 use the accelerator where the cation is "diethylamino-diphenyl-benzyl-phosphonium". (CX-266, column 2, lines 13-18).

217. The '708 patent stated that according to the disclosed invention it has been found that it is possible to obtain high rubber/metal adhesion values when there is used for example as a vulcanization accelerator a salt composed of a cation of phosphonuim as decribed in the Pattison '654 patent or of an amino-phophominium as described in the Moggi '463 patent with a particular counteranion (CX-266, col. 2, lines 12-25).

218. Ausimont's U.S. Patent No. 4,612,351, which issued on September 16, 1986, to Caporiccio, Monza, Bonardelli, Moggi and Cirillo, and assigned to Montedison with Italian priority dates of June 30, 1983 and May 23, 1984 claims a fluoroelastomer composition where the polymer contains a small amount of chlorotrifluoroethylene which promotes adhesion to metal. The specification notes that fluoroelastomers can be cured in compositions which include, among other things:

> (5) a vulcanization accelerator having the structure of a quaternary compound of nitrogen, of phosphorus, of arsenic and of antimony in an amount ranging from 0.2 to 3 parts, as described in the following patents:
> GB No. 1,354,344 (Du Pont), ammonium salts
> U.S. Pat. No. 3,876,654 (Du Pont) [RX-10], phosphonium salts
> U.S. Pat. No. 3,655,727 (EM) *[sic 3M]*, ammonium salts
> U.S. Pat. No. 3,752,787 (Du Pont), phosphoranes
> GB No. 2,010,285 (ME), amino-phosphonium salts.

(CX-488, column 3, lines 54-62).

219. Ausimont's Moggi, Bonardelli and Cirillo in a paper titled "Fluoroelastomers, Dependence Of Crosslinking Behaviour On Vinylidene Fluoride Content" (SX-16) at the International Rubber Conference in Moscow (1984) stated that the vinylidene fluoride copolymers seems to be crosslinked by a three step process; that in the first step hydrogen fluoride is eliminated from the polymers upon treatment with bases. The second step involves addition of the curing agent to the site of unsaturation. The final step in the cure involves the thermal formation of additional unsaturations and subsequent crosslinking. The paper further addressed Schmiegel's selective curing site proposal as reported in Kautsch, Gummi Kunstat., 31, 137 (1978) and Angew, Makromol. Chemie 76/77, 39 (1979).

220. Moggi et al. in SX-16 performed experiments using solid samples which approximated the receipe used during vulcanization, although the level of ingredients was chosen to enhance the experimental results, not to result in an end product with optimal physical properties. (SX-16 at 4).

221. The "accelerator" used in the experiments in SX-16

222.

223. Moggi et al.'s paper (SX-16) suggested that cross-linking occurred at random vinylidene fluoride moieties rather than only at those surrounded by hexafluoropropene moieties. (SX-16 at 4-8).

224. Ausimont's Tommasi testified, as to SX-16, (Tr. at 2520-21):

Q. He is suggesting that Schmiegel's theory on exactly which site the crosslinking occurs may be incorrect?

A. Yes, this is what he does.

Q. He doesn't say the accelerator doesn't work the same way, does he?

A. No, he certainly does not. Actually, he says what (sic, that) the results are different.

Q. He says the sites --

A. The sites are different, the sites of attack are different.

Q. But he also confirms that essentially by the substance of his paper, he confirms that the bisphenol links the polymer chains together?

A. Of course. This is the object. You have to crosslink bisphenol. This as the (sic, This is -- the) mechanism in his opinion is completely different, because it is not the Schmiegel sites that are attacked but other sites.

225. In a report titled "Chain Endings In the Curing Of Technoflon And Compression Set" dated March 20, 1985, Ausimont's Arcella relies on the Schmiegel mechanism to explain the vulcanization chemistry. (CX-272 at Bates No. AI007755-57). Arcella under the heading "Summary And Conclusions indicates that the crosslinking can be explained in the presence of a phosphonium salt (C 20 which is a DuPont product containing a Du Pont fluoroelastomer and triphenyl benzyl phosphonium chloride (Aloisio CPX-4 at 115-16), GM 102E, etc.) and similar molecules (CX-272 at 1 (Bates No. AI007748)). Thus under said heading Arcella states:

> A crosslinking mechanism for Tecnoflon is proposed using chain endings of ionic nature. The crosslinking explained in the presence of a phosphonium salt (C 20, GM 102 E, etc.) and similar molecules.
>
> It is obvious how in ionic curing via bisphenol, in which the phosphonium salt is necessarily present as an accelerant, both types of crosslinking coexist in competition with each other.

> In this case, the conditions are such that the final crosslinking is, for the most part, generated by phenol and only minimally by the endings.
>
> This minimum part has however a substantial impact on the compression set of the vulcanizate. In fact, the crosslink formed with the endings has poor compression set characteristics which have repercussions on the final characteristics.
>
> Reducing the endings of an ionic nature causes a reduction in the fraction of the crosslink with poor properties and clear improvements in the compression set are obtained.
>
> For the purpose of obtaining later verification of the mechanism proposed, this work will continue with experimentation using model molecules in solution and curing tests with bi-functional fluoridated molecules $-CH_2OH$ and $-COOH_4$ (for example, Fomblin oils) which, in accordance with the mechanism, should be capable of crosslinking in the presence of a phosphonium salt.
>
> Also to be investigated are the characteristics of the secondary network and its mechanism of degradation through the study of the vulcanizates with accelerant alone.

Under the heading "Introduction" it is stated:

> The mechanism of curing the elastomers using bisphenol was studied by Schmiegel ... In his study, he notes that both bisphenol and the ionic accelerant are necessary for curing. However, while bisphenol alone is not capable of generating a crosslink, the accelerant is.
>
> Schmiegel therefore carried out curing tests with accelerants alone and noticed that the compression set of the vulcanizate was poor. From this, he concludes that the accelerant generates a "poor network" and therefore is it necessary to use it in amounts barely sufficient to promote crosslinking.
>
> Schmiegel does not supply indications on the type of crosslink generated by the accelerant or on the reaction mechanism involved.

226. In a report presented on or about April 5, 1985, Ausimont researchers Arcella and Brioni studied the the discontinuous washing of TECNOFLON (the commercial name for Ausimont's fluoroelastomers) through the introduction of aluminum complexing agents (CX-450). In the course of the report, the authors described "the mechanism of reaction in the normal case of

the absence of OH ions from NaOH." (CX-450 at 12, Bates Nos. AI007709). The mechanism described is essentially the Schmiegel proposal including the requirement of the diene curing site. The report at A1007709 refers to "Accelerant system GM102E" and "Curing system BISPHENOL AF". It also refers to the "interaction of the accelerant" with the base calcium hydroxide.

227. Ausimont's research report dated 5/15/86 (CX-421) and with Moggi in charge of the research and the report written by Moggi, Grazielle Chiodini and Gionna Cirillo and titled "Preparation and characterization of phosphonium compounds containing phosphorus-nitrogen bonds. Kinetic study of their catalytic activity in nucleophilic substitution reactions, particularly in the crosslinking of fluoroelastomers" stated under the heading "Summary":

> The research was conducted mainly in accordance with the following guidelines:
>
> - Synthesis and characterization of new amino-phosphonium or phosphoranamine compounds
> - Study of their activity in certain reactions conducted in phase transfer catalysis (PTC)
> - Evaluation of catalytic activity in the reaction on 1,1,difluororethylene-based fluoroelastomers, leading to the formation of cross bonds through by attack of a bisnucleophile agent.

Under the heading "Introduction" it stated:

> Amino-phosphonium or phosphoranamine derivatives are characterized by having the structure of "onium" salts and by the presence of one or more phosphorus-nitrogen bonds. The industrial importance of this class of compounds resides in the fact that they act <u>as accelerator agents</u> in the crosslinking reaction that results in the formation of cross bonds between the 1,1,difluoroethylene-based copolymer elastomer chains. ... [the '463 patent] The analogy noted between active catalysts in PTC [phase transfer catalysis] and <u>accelerator compounds</u> from this bisnucleophile reaction led, on the one hand, to close examination in the system in question of the relations between structure and catalytic activity of the amino-phosphonium compounds and, on the other, to evaluation of their activity in certain typical reactions carried out in PTC. <u>The accelerant action, which indeed shows a strict</u>

> parallel with phase transfer catalysis, seems to occur in both passages of the crosslinking reaction which can be diagrammed as a dehydrofluorination and an attack by a bisnucleophile agent, for example hydroquinone, with two dehydrofluorinated polymer chains. A diagram of these reactions that take place in the presence of calcium hydroxide and magnesium hydroxide at temperatures ranging between 160 and 190°C is given in Table 1. It is a modified and simplified version of a pattern described in the literature .... [W.W. Schmiegel, Angew, Makron, Chem., 1979, 76.]

228. According to Ausimont's Tommasi the title for CX-421 suggests that said phosphonium compounds are presumably "going to be used as accelerating agents in the crosslinking reaction". Tommasi testified that the report talks about the importance of compounds with phosphorus nitrogen bonds because they can act as accelerator agents in the crosslinking reaction in fluoroelastomers; that the report also suggests that the accelerator and the crosslinking reaction shows a parallel with phase transfer catalyst or catalysis; that the report deals with the preparation of GM102E; and that Table 1 referred to in the "Introduction" of the report is a simplified version of the Schmiegel proposed mechanism of crosslinking (Bates No. A1007808) (Tommasi, Tr. at 2524 to 2527).

229. Ausimont's Granzille Chiodini received an organic chemistry degree from the University of Milan. She has synthesized

Chiodini has a doctorate degree in chemistry from the University of Milan received in 1982. Chiodini began employment at Ausimont in 1983. After starting at Ausimont, she soon worked with phosphorus compounds, and then in particular worked with the synthesis of GM102E (Chiodini, CPX-15 at 6 to 8). Chiodini's next assignment for Ausimont was the purification of Bisphenol AF. Her current position at Ausimont is a researcher. After Chiodini finished work on purification of Bisphenol AF, she worked on the reaction on fluorinated polymers dissolved in solvent. She also worked on the vulcanization with peroxide of fluoroelastomers and with the synthesis of the accelerating system called XA. Chiodini has more than five patents. (Chiodini, CPX-15 at 9 to 12, 15).

230. The chemistry or scheme of the crosslinking reaction for GM102E and bisphenol AF is sketched out by Chiodini on CX-411. The "I" on page 3 of CX-411 refers to the formula I on page 2 of CX-411. The various steps on CX-411 can occur at the same time. (Chiodini, CPX-16 at 129, 130, 131). On comparison with the reaction mechanism proposed by Schmiegel in his 1978 paper the two are similar. Chiodini also testified that

231. Ausimont's Pianca, Bonardelli, Tato, Cirillo and Moggi in a paper titled "Composition and sequence distribution of vinylidene fluoride copolymer and terpolymer fluoroelastomers. Determination by 19F nuclear magentic resonance spectroscopy and correlation with some properties" Vol. 28 Polymers 224 to 230 (Feb. 1987) under the subheading "vulcanization" at 223 stated that the most common vulcanization system for fluoroelastomers is based on formulations consisting of (a) inorganic bases (b) Bisphenol AF, (c) a "vulcanizing accelerator, generally a quaternary salt of 'onium (ammonium, phosphonium, etc.)" and (d) fillers, such as carbon black. The authors stated that according to the currently accepted mechanism, the crosslinking reaction consists of two steps: (1) polymer dehydrofluorination by the base, to give double bonds in the backbone chain and (2) nucleophilic addition of bisphenol

AF to the double bonds, yielding crosslinks; and that it is worth reminding "ourselves" that carbon-carbon double bonds in fluorinated compounds are electrophilic. They also stated that the "accelerator which has the structure typical of phase transfer catalysts, is thought to act as the cation of the base and/or bisphenate, making them able to diffuse through the rubber." (SX-34).

232. In the Feb. 1987 publication (SX-34) at 229-230 referring to the Schmiegel 1978 presentation, the authors state that Schmiegel has observed that solution reaction with bases of vinylidene fluoride-hexafluoropropene copolymers results in the decrease of an integrated intensity of signals in a certain spectrum and that after the basic treatment Schemiegel also observed the appearance of two low-field resonances and hence suggested that dehydrofluorination takes place selectively to give conjugated dienes which are the site of vulcanization. It was concluded that as a whole the experimental results of ODR measurements do not agree with Schmiegel's mechanism and suggested that the vulcanization behaviour depends more likely on the overall content of vinylidene fluoride, the moiety that can be dehydrofluorinated, than on the presence of hexafluoropropene-vinylidene fluoride - hexafluoropropene sequences; and that though Schmiegel's observations make it difficult to think of a fully random dehydrofluorination, at least in solution, the reaction pattern "seems to be more complex than that suggested by Schemiegel."

233. The substance and conclusions reached in Moggi et al. 1984 paper (SX-16) were essentially repeated in more statistical detail in SX-34.

234. The Pianca et al. paper (SX-34) did not involve any new experimental work; it relied on the previous experimental work presented in

the 1984 Moggi paper. (Tommasi, Tr. at 2531-32).

235. The accelerator used in the Pianca et al. paper was benzyl trisdimethylamino phosphonium tetrafluoroborate. (SX-34 at 225).

236. The Pianca et al. paper repeats the conclusion of the 1984 Moggi et al. paper (SX-16) that crosslinking occurs at more sites than merely those suggested by Schmiegel. (SX-34 at 229-30).

237. Ausimont's research report "The Vulcanization Mechanism for Tecnoflon-The Influence of the Recipe on Vulcanization and Properties for the Copolymer NM/R," May 5, 1988 (AI008988-A00908) (SX-24) which included Cirillo as an author. NM/R is a medium viscosity copolymer TECNOFLON (A1008996). Under the heading "AIM OF THE REPORT", it was stated that the Schmiegel 1978 presentation dealt with the influence of vulcanization receipe on the crosslinking reaction and end use properties of vulcanizates in a very comprehensive way, but "both the raw rubber and the accelerator were different from those produced by Montefluos;" and that it seemed necessary to do a similar work on the Montefluous rubber and receipe. The report contained the following conclusions, inter alia (A1008993):

> a. The real crosslinking agent is BISAF [Bisphenol AF] as shown by several different techniques (ODR, DSC, solvent swelling, mechanical properties) and the accelerator plays only a minor role at a very low BISAF concentration.
>
> BISAF reacts in a practically quantitative way and the degree of crosslinking increased with its concentration.
>
> b. The MF accelerator (GM102E) is more efficient than GM200 (Benzyl-triphenylphosphonium Chloride) in that a lower concentration is required to achieve a given vulcanization rate; however the concentration dependence of the relevant vulcanization parameters... was found to be practically the same for the two accelerators.

> c. Our data are in good agreement with those obtained by Schmiegel on Viton so that any influence of the raw rubber on the vulcanization behaviour can be excluded.
>
> d. The crosslinking reaction rate is decreased by BISAF and increased up by accelerator concentration. [Emphasis supplied]

The conclusions ended with the statement that "[v]ery surprisingly it was found that the best network stability is obtained by a vulcanization receipe very similar to the standard (2 phr BISAF, 0.4 phr GM102E).

238. VITON is a DuPont fluoroelastomer product. (Brullo, Tr. at 446).

239. Ausimont's Cirillo, Chiodini, Del Fanti and Moggi in a paper titled "Fluoroelastomers Reaction Products in Early Stages of Network Formation" from Biological and Synthetic Polymer Network, edited by Kramer at 255 to 265 (Elsevier Applied Science Publisher 1988) (RX-96) investigated reactions occurring in the first steps of crosslinking of vinylidene fluoride (VDF)-hexafluoropropene (HFP) elastomer copolymers (motor ratio 4/1) operating in homogeneous solution, phase transfer catalysis conditions and bulk. Specifically, the paper analyzed Schmiegel's proposed limitation that the polymers cure only at diene groups (RX-96 at 256). Under the heading "Introduction" the authors state:

> The crosslinking reaction of vinylidene fluoride (VDF)-hexafluorpropene (HFP) copolymers can be summarized in a three-step process:
>
> - base-induced dehydrofluorination producing polymer chain unsaturation;
> - primary network formation by reaction of unsaturated chains with a bisnucleophilic agent;
> - ultimate network formation after heating.

Under the heading "Experimental" the authors stated:

> Homogeneous Phase Dehydrofluorination
> Dehydrofluorination was investigation by dissolving the copolymer

> in suitable solvents, such as acetone. THF, DMAc, and DMSO in the presence of alcoholic KOH. The reaction was monitored by analyzing the ...[fluoride] ions in the solution. It proceeded to completion within a few minutes and, at the same base concentration, its rate was found to depend on the nature of the solvent Homogeneous phase dehydrofluorination has been studied in more detail using THF as a solvent.
>
> Phase Transfer Catalysis (PTC) Dehydrofluorination
> The reaction was carried out by dissolving the copolymer in a water insoluble solvent such as methyl-t-butyl ether, and treating the stirred solution in the presence of a PTC agent, usually an onium salt such as ... [a phosphorus containing compound wherein the phosphorus is covalently bonded to four carbon atoms and which contains a bromide ion]
>
> Bulk Dehydorfluorination
> Copolymers containing 6% ... [calcium hydroxide] and 0-5% onium salt were mixed on a cold lab mill and then heated to 150°C for 30 min. Only the MEK-soluble fraction has been used for the analysis.

Under the heading "Conclusions" the authors stated:

> Alkaline treatment of VDH/HFP copolymers (molar ration 4/1) in homogeneous, PTC and bulk systems induces in all cases at least two kinds of unsaturation. FT-1R spectra show absorptions (proportional to the added base) at:
>
> > (1) 1720cm-$_1$ due to-CF=CH-groups. This absorption is also the only one detected after alkaline treatment of chlortrifluoro-ethylene-vinylidene fluoride and tetrafluorethylene-vinylidene fluoride copolymers.
> >
> > (2) 1680 cm-$_1$ related to -(CF^3)C=CH-. Its relative intensity increases with the HFP content in the copolymer.
>
> Both these kinds of unsaturation are detected ...
>
> * * *
>
> A signal at 1640cm$_{-1}$ is present only in the copolymers treated in alkaline homogeneous solution, which is probably related to conjugated double bonds. This band seems to be peculiar to this treatment, as it does not appear in the copolymers treated in the heterogeneous systems.
>
> Polymer chain scission is also observed; this has to be related to the HFP units. No degradation is in fact observed; this has to be related to the HFP units. No degradation is in fact observed in similar experimental conditions with

> poly(vinylidene fluoride) and tetrafluoroethylene-vinylidene fluoride copolymers. FT-1R spectra of the copolymer treated with sodium p-chlorophenate suggest that -CF=CH- should be the curing site in the bisnucleophile crosslinks of the fluoroelastomers.

The authors (at 256) stated that the dehydrofluorination reactions have been investigated in great deal by Schmiegel _Kautsch Gummi Kunststst_. 31, 137 (1978) _Angew Makromol Chem_., 76/77, 39 (1979) in which it was found that either of two certain monomer sequence was the _only_ selective base-sensitive site leading to the diene group.

240. In a paper presented by V. Arcella, G. Chrodini, N. Del Fanti and M. Pianca at a meeting of the Rubber Division of the American Chemical Society, dated October 1991 titled "Cross-Linking Chemistry of Vinylidene Fluoride Fluorocarbon Elastomers By Bis-Nucleophiles" (Bates Nos. A031035-A031060)(RX-151; CX-297) there was the following "abstract".

> The chemical events occurring during the cross-linking of vinylidene fluoride/hexafluoropropylene (VF2/HFP) fluorocarbon elastomers by bis-nucleophiles have been studied in the past both in solution and solid state vulcanization. Model compounds have been used to support mechanistic assumptions and elucidate the chemistry involved. It appears from these works that the chemical reaction mechanism through which cross-linking develops in practical vulcanization is not completely understood since some conflicting conclusions have been drawn. In the present paper the cross-linking chemistry is reviewed and a chemical mechanism is proposed which agrees with most of the published data. Furthermore, an original experimental approach is presented, which was devised to follow structure evolution during solid state cross-linking in conditions very close to practical vulcanization. It is shown that the proposed mechanism appears to be consistent with this new investigation. [Emphasis added]

Under the heading "Proposed Cross-linking Mechanism" (A031044) it was disclosed:

> On the basis of the solution investigation using NMR and FT-IR techniques coupled with solid state vulcanization experiments (ODR tests and F^- evolution) a modified reaction mechanism can be proposed:
>
> a) Formation of $-C(CF_3)=CH-$ double bond by elimination of

> "tertiary fluorine". This agrees with the early findings of Schmiegel who described the formation of the initial double bond regioselectively from base sensitive sites. This double bonded structure is supported by ... investigations.
>
> b) Double bond shift catalyzed by fluoride ion and formation of -CH=CF double bond in a similar way of that described by Schmiegel, again supported by FT-IR and 1H-NMR investigation. Notably, the data do not distinguish between -CH = CF- formation directly from elimination or indirectly from -C(CF_3)=CH-. Experimentation now in progress hopefully will clarify the kinetic/thermodynamic preferences of the system.
>
> c) Nucleophilic addition to the -CH = CF-double bond (intensity decrease of the 1720 cm^{-1} band) with:
>
> c') allylic displacement of fluoride affording the new -C(CF3)=CH-double bond ... and/or
>
> c") additional fluoride elimination from the same double bond yielding another double bond ...

241. The emphasis of the Arcella paper (CX-297) was the particular location of the cure sites on the polymer backbone (Tommasi, Tr. at 2523).

242. The first section of the Arcella paper reviews the previously published works on Schmiegel, Moggi, et al. and Pothapragada Venkateswarlu et al. (Dr. V.). (CX-297 at 2-7). The Arcella paper notes that Dr. V. et al.'s studies utilized triphenyl benzyl phosphonium chloride, and Moggi's research used a "quaternary 'onium' salt." (CX-297 at 6).

243. Following solid state studies under approximate vulcanization conditions, Arcella reported additional results, and stated that:

> These results are in a very good agreement with the previously reported findings obtained by FT-IR analysis performed on samples in solution and represent a good support to the proposed vulcanization mechanism, since the experimental conditions tested are very close to that of common practical vulcanization.

(CX-297 at 13).

244. The paper (RX 151, CX-297) referred to the "fundamental study" of Schmiegel on the cross-linking reaction of vinylidene fluoride/hexafluoropropylene copolymers by bis-nucleophiles, particularly with bisphenols in the presence of a quaternary phosphonium salt and an acid acceptor and his proposed mechanism. It noted that further investigations by Cirillo, Chiodine, Moggi and Serverini in <u>Biol. Synth. Polym. Networks</u> (1988) confirmed the high selective dehydrofluorination of vinylidene fluoride units isolated between two hexafluoropropylene units "as previously reported by Schmiegel" and that these "results have been confirmed independently by Venkateswarlu" in (CX-223).

245.

246. The diorganosulfur oxide used in the compositions claimed in the '320 patent is not a cross-linker and it does not form a salt with Bisphenol AF. Also it is well know from general chemistry textbooks that diorgano sulfur dioxides have a solvating effect on ions. This effect may increase reaction rates even by orders of magnitude. (Tommasi, RPX-193 at 3).

247. The chemistry would not be different as to what takes place during each of the crosslinking of the accused Ausimont compositions. (Chiodini, CPX-16, Vol. II at 124, 125).

248. Chiodini testified that "the three accelerators," <u>viz</u>. GM102E,

would "act in the same way as far as concerns the scheme [CX 411] of the reaction "(CX-297). The three "accelerators" do have different activity (Chiodini, CPX-16, Vol. II at 125 to 129).

249. Chiodini testified that the reaction mechanism is the same for all of Ausimont's compositions. (CPX-16, Vol. II at 125).

250. Chiodini was responsible for developing a feasible method to synthesize GM102E production. (Chiodini, CPX-15, Vol. 1 at 7 to 9).

251. The term TECNOFLON is a commercial designation for the accused compositions (Tommasi, RX-162 at 15, 16).

252. Chiodini testified that the reaction mechanism is essentially the same when (CPX-17, Vol. III at 153-54).

L. Complainant's Publications And Tests

253. Pothapragada Venkateswarlu (Dr. V.) is employed at complainant and his present position is Senior Research Specialist. He has a Doctorate of Science in Biochemistry and a Ph.D. in Physiological Chemistry. The degree in Physiological Chemistry was obtained from the University of Minnesota in 1962. The degree in Biochemistry, was obtained from Andhra University, Waltair, India, in 1955. A list of his publications is at CX-459. He has been employed full time by 3M since about 1978. His positions, duties and responsibilities have generally included: premanufacture notification coordinating, especially of toxicological and environmental studies concerning fluorochemicals; development of trace fluorine analytical methods for body fluids and tissues; development of trace fluorine analytical methods for preparing FDA submissions; and, study of cure mechanisms of fluoroelastomers. He has conducted studies concerning chemical actions occurring in the solid phase during the curing of fluoroelastomers with bisphenol AF. One of his

studies was reported at a meeting of the Rubber Division, American Chemical Society, Detroit, Michigan, in 1989. (CX-223). CX-223 is authored by Venkateswarlu, R.E. Kolb, R.A. Guenthner and T.A. Kestner. In that study "we did both solid phase and liquid phase work." (CX-222 at 1, 2).

254. Dr. V. in his report (CX-223) at 1 states that cure chemistry pertaining to the crosslinking of the vinylidene fluoride/hexafluoropropylene copolymer with bisphenol AF has not been extensively reported in open literature, except for the "early work of Schmiegel" in <u>Angew, Macromolek, Chem</u>, 76/77 at 39 to 65 (1979). The proposed scheme of crosslinking reactions of Schmiegel is referred to. Dr. V's report then states:

> ... However, according to Smith... [a 1982 publication of S. Smith], "this is based on analysis of early stages. The later stages of the reaction - when some crosslinking has rendered the polymer insoluble - are much more difficult to study, and further work will be needed to uncover details of the reactions which occur". It is precisely toward these goals that the efforts in the present studies are addressed. New methodologies have now been developed to overcome the difficulties of investigating reactions in insoluble solid matrices, the problem alluded to by Smith...
>
> The method so developed have now been used to uncover and elucidate certain chemical events occurring during the curing of a fluorocarbon elastomer (VF_2/HFP copolymer) with BPAF, using benzyltriphenylphosphonium chloride (PCI) as the phase transfer catalyst, and calcium hydroxide and magnesium oxide as acid receptors and cure activators.
>
> In addition, this report includes studies on release of fluoride ion from VF_2HFP copolymer in solutions by inorganic bases and also the release of fluoride ion from VF_2HFP copolymer in the solid state by each of the individual components of the cure system (calcium hydroxide, magnesium oxide, BPAF and PC1), as well as by certain combinations thereof.
>
> The work reported in this paper complements the earlier pioneering work done by Schmiegel... [RX-97], and provides a new dimension to the overall picture of cure mechanisms of fluorocarbon elastomers with BPAF.

> The new approaches described in this paper should stimulate additional fresh investigations of reaction mechanisms pertaining to curing of fluorocarbon elastomers and other polymers, when the material to be investigated is rendered progressively insoluble. [Footnote omitted]

255. Dr. V.'s report (CX-223) confirmed the work of Schmiegel which dealt with liquid phase studies. (Tr. at 859).

256. Fig. 8 of Dr. V's CX-223 is a suggested mechanism of what is going on in the cure of fluoroelastomers (a vinylidene fluoride hexapropylene copolymer of 78:22 mole percent) and is based on a solid phase (Worm, Tr. at 792, 793, 812). The figure shows removal of hydrogen fluoxide (HF) from the backbone next to the tertiary fluorine on the backbone to get a double bond in the backbone. There is formed the hydrogenated form of Bisphenl AF and the fluoride salt of the phosphonium. Then salt is reformed of the phosphonium and the Bisphenol AF. In the next step the phosphonium takes the Bisphenol AF and attaches it where the double bond was to form another different double bond. All of the Bisphenol AF is attached on one side until it's used up (Worm, Tr. at 760, 825).

257. Dr. V. did the actual steps in investigation 2 (B. Dehydrofluorination of Vinylidene fluoride hexafluoropropylene (VF_2/HFP) copolymer in the solid state) of his study (CX-223) and he also developed its protocol (Tr. at 878, 879). Investigation 2 was preliminary (Tr. at 883). Investigation 3 of CX-223 is titled "C, VF_2/HFP Copolymer Cure Mechanisms" and here Dr. V. was varying the amounts of Disphenol AF and tribenzylphenylphosphonium chloride to get an understanding of the kinetics of the reaction (Tr. at 896).

258. Investigation 1 of CX-223 concerned solution phase study (Tr. at 898). The solid phase experiments carried out in connection with CX-223 were

quite tedious in comparison with the solution phase experiments employed by Schmiegel (Tr. at 903). Dr. V. in CX-223 repeated Schmiegel's solution phase experiments and confirmed them. (Tr. at 903). Dr. V. felt it was necessary to carry out the solid phase curing studies of CX-223 to gain a better understanding of the chemical actions occurring in the solid phase (Tr. at 905).

259. One of the studies in CX-223 involved actual vulcanization recipes using triphenyl benzyl phosphonium chloride as an accelerator. The vulcanization reaction was periodically stopped and measurements were taken for amounts of fluoride ion released, free Bisphenol AF, and phosphonium ion. (CX-223 at 5).

260. The paper CX-223 makes additional revisions to the Schmiegel mechanism, but generally confirms the substance of Schmiegel's original theory. (Dr. V., Tr. at 858, 911-21).

261. The paper CX-223 indicates that the initial dehydrofluorination in the presence of a base occurs by way of the phosphonium/bisphenol complex. The bisphenol is then all attached to polymer chains at double bond sites created by the dehydrofluorination. Once there is no more free bisphenol, the phosphonium forms a complex with the unattached end of the linked bisphenol. The phosphonium end of the linked bisphenol then defluorinates a site on another polymer chain, forming the cross-linked polymer and releasing phosphonium fluoride. (CX-223 at 15-16 and Fig. 8; Worm, Tr. at 794-802).

262. The paper CX-223 suggests that a diene is not necessary for crosslinking. (CX-223 at 16).

263. During the vulcanization experiments reported in the paper CX-223 the bisphenol concentration gradually decreases to zero, and once it reaches

low concentrations, the ODR curve begins to increase, indicating that the cross-linking is beginning to occur. From that it can be concluded that the bulk of the bisphenol is attached to the polymer chains before the chains are connected to each other. (CX-223, Figure 5; Worm, Tr. 803-04).

264. The vulcanization experiments reported in paper CX-223 indicate that the phosphonium concentration remains steady until cross-linking is well underway. After crosslinking begins and the bisphenol concentration has dropped to almost zero, the phosphonium begins to degrade. (CX-223, at 17 and Fig. 5).

265. The experiments reported in the paper CX-223 indicate that several equivalents of fluoride are released per equivalent of phosphonium. (CX-223 at 8).

266. In connection with this investigation, Dr. V. was asked to develop a study, in the liquid phase, similar to the one reported in his report (CX-223) in order to examine and compare the operation of the bisphenol AF salt of benzyl triphenyl phosphonium cation and the bisphenol AF salt of benzyl triphenyl phosphonium cation and the bisphenol AF salt of benzyl diethylamino diphenyl phosphonium cation, in the cure of fluoroelastomers. The protocol developed for conducting the experiment is in CX-227 and was based upon the published work of Dr. V. (CX-223). The experiment was designed by Dr. V. in consultation with Engel. The techniques Dr. V. used for conduct of the experiments were the same as those used in the report (CX-223), except that the fluoride ion released was determined with a fluoride ion electrode, instead of by a spectraphotometic method. The experiments were done in two groups. In the first group, experiments were conducted by Dr. V. in the absence of any diorgano sulfur oxide component. In a second group of

experiments, bis(p-chlorophenyl) sulfone was included in the reaction mixtures. (Engel, CX-242, addendum at 1). In order to run the experiments, Dr. V. needed samples of the two phosphonium compounds. They were provided to him by Dr. Werner Grootaert, who had synthesized them. The documentation generated during the conduct of the experiments is contained in CX-243, 147-163, 224, 206, 207, 208, 209, 210, 168, 169, 170, 171, 172. (Dr. V. CX-222 at 1 to 3).

267. In CX-243, Dr. V. created all pages, as records of his first experiments with the benzyl diethylamino diphenyl phosphonium, and the benzyl triphenyl phosphonium, with the bisphenol AF anion. In CX-147-163, are contained the analytical results he obtained from Tom Kestner, to create CX-243. All pages in CX-224 were created by Dr. V. as a record of data from certain of his studies. The pages in CX-207, 208, 209 and 210 were given to him by Kestner and Schultz and are pages of data collected by them, and used by Dr. V. in generating CX-206. The pages of CX-168 were created by Dr. V. to record the data collected in one of his experiments. The pages of CX-169, 170, 171, and 172, were given to Dr. V. by Kestner, and were used by Dr. V. in generating CX-168. For some experimentation, Dr. V. was asked to conduct the same experiments as in CX-243, but in the presence of a sulfone. Documentation created by Dr. V. relating to this experiment is included in CX-206 and CX-168. (Dr. V. CX-222 at 3).

268. In conducting the set of experiments of CX-168, Dr. V. noticed that the second set of experiments differed from the first, in that after he had mixed the samples for the first experiment, he had stored them in the refrigerator at 4°C, after they were put in the bath for reaction, to quench the reaction. For the second set of experiments, CX-168, however, Dr. V.

inadvertently allowed the samples to remain at room temperature, after the reaction in the bath, for an extended period of time, before they were analyzed. Therefore, Dr. V. decided to run a third set of experiments, CX-206, analogous to the second set of experiments, except wherein the samples were quenched and stored in the refrigerator after reaction. Also, Dr. V. believed there may have been some problem with the pipette used in the second set of experiments. He adjusted for this in conducting the third set of experiments, CX-206. In order to conduct the third set of experiments, Dr. V. needed to have further samples of the two phosphonium materials. He asked Dr. Grootaert for the materials and the synthesis is provided in CX-164 to 167. Dr. V. has discussed how the experiments were conducted, and the data obtained with Engel. Dr. V. personally gave study 1, CX-243 and 147-163, to Engel. Studies 3 and 2, CX-206-210 and 168-172, respectively Dr. V. requested others to give to him. (CX-222 at 3, 4).

269. Engel and Dr. V. collaborated on experiments in which a fluoroelastomer was dissolved in solvent along with differing concentrations of a complex of an accelerator and bisphenol. Experiments were run with two different accelerators: GM 102E and triphenyl benzyl phosphonium chloride. The level of dehydrofluorination achieved by the complex increased with the increase in concentration of accelerator. The increase measured was similar with the two different complexes. (Engel, CX-242, addendum; CX-243).

270. The documentation of CX-243, 147, 1553, 154-158, and 161-163 reflect the procedure conducted and results of the first group of experiments involving Dr. V and Engel. The formulations used in the first group of experiments are provided in CX-243. Each reaction mixture was heated and stirred in a water bath at 70°C for two hours, after which it was quenched by

cooling to 4°C. Then, fluorine-NMR spectra were run. Fluorine-nMR spectra were then run. The spectra provides a manner in which the changes in the fluoroelastomer system, under the curing the conditions, can be evaluated. The changes for the compositions are shown in the fluorine-NMR spectra printed on two pages of CX-243. On one of the pages, the results of the compositions in which benzyl triphenyl phosphonium bisphenol AF complex was used, are shown. On the other, the results using benzyl diethylamino diphenyl phosphonium bisphenol AF salt are shown. The fundamental experiment was to follow what happens to fluorine nuclei in the system, as more and more phosphonium/bisphenol AF salt was provided, over the same reaction time and more and more dehydrofluorination occurs. Dehydrofluorination is the first step in the vulcanization reaction of fluoroelastomers. The difference between the two F-NMR summary pages of CX-243 is that in the experiments on one page, the phosphonium cation is benzyl triphenyl phosphonium cation; and, in the experiments on the other page, the phosphonium cation is

The measured spectra show there was virtually no difference in the two sets of reactions. Both proceeded in the same manner, to the same extent. (Engel, CX-242 addendum at 2 at 5).

271. In CX-147-152, 154-58, 161-163, specific documentation used to generate the summary documents of CX-243 involving Dr. V. and Engel were provided. CX-161 provides the fluorine-NMR peak assignments, for the various fluorine atoms in the system. Pages 2 and 3 of CX-162, 163 are the full size plots which were shown in reduced form in CX-243. The remaining 12 pages of CX-147-152, 154-158, 161 include the individual NMR spectra for each of the samples. The designations in the upper right hand corner of each spectra identify uniquely that spectrum. The same indications, for cross-referencing

purposes, are identified in CX-243, for each corresponding spectra. The dehydrofluorination occurs very slowly, and essentially not at all, at room temperature. Thus, it was expected that no significant reaction would have occurred before the samples were immersed in the 70°C bath or after removal and before the fluorine-NMR were taken. To ensure this, after reaction the samples were stored in the refrigerator, at 4°C, before they were analyzed. (Engel, CX-242, addendum at 5, 6).

272. In a follow-up group of experiments involving Dr. V. and Engel, in the experiments in the previous finding, (bis(p-chlorphenyl) sulfone) was added to the reaction mixture. Two sets of experiments of this type were performed, one in which the samples were refrigerated at 4°C immediately after their 2 hour heating period at 70°C, (set sulfone-added A), <u>viz</u>. CX-206 and 207-210 and one in which the samples were inadvertently allowed to stay for two days at room temperature prior to measurement of their F-19 NMR spectra (and fluoride ion concentration) (set sulfone-added B), <u>viz</u>. CX-168-172. For set sulfone-added A, the results for the triphenyl benzyl phosphonium and the are, within experimental error, identical to each other, and differ only slightly from those runs noted earlier in the absence of sulfone, in the rate of fluoride ion generation. The two materials behave essentially identically under the same reaction conditions. For set sulfone-added B, the results for the triphenyl benzyl phosphonium and the are, within experimental error, identical to each other with the exception of those at the highest concentration where a deviation in the amount of fluoride ion present is noted. With the exception of the highest concentration system, the runs also differ only slightly from those runs noted earlier in the absence of

sulfone, in the rate of fluoride ion generation. The two materials do the same thing in the same manner. (Engel, CX-242, addendum at 6-7).

273. The function and manner of operation of a phosphonium compound is described, for example, in the separate publications of Schmiegel & Pothapragada et al. and is a generally accepted model for the operation of the phosphonium compounds as cure accelerators in fluoroelastomer cure systems. (CX-223; RX-186; Worm CX-132, at 4-5). The operation of

and benzyl triphenyl phosphonium chloride have been directly compared to one another, as accelerators in the solid phase cure of fluoroelastomer compositions. This study was undertaken by persons at complainant, under the direction of Harwood. The studies were conducted with a fluoroelastomer copolymer of vinylidene fluoride and hexafluoropropene of complainant. The operation of the two phosphonium chlorides was compared, in the solid phase vulcanization of otherwise identical fluoroelastomer compositions. A series of comparisons were made, involving variations in order components or in the presence of various relative amounts of other components. The studies show that the Ausimont phosphonium compound,

has essentially the same influence on cure performance as does benzyl triphenyl phosphonium chloride. There is no difference observed in the way the two phosphonium chlorides were operating to accelerate the cure. The result in each instance was a cured or vulcanized fluoroelastomer exhibiting similar physical characteristics such as hardness, elongation, tensile, and modulus. (CX-231, Harwood at 47-50, addendum, at 23-33; CX-214, CX-225).

274. For complainant's experiments a series of fluoroelastomer compositions were prepared using as the fluoroelastomer co-polymer a copolymer

of vinylidene fluoride and herafluoropropene, viz. FC 2230 which contained 60 wt. percent vinylidene fluoride and 40 wt. percent hexafluoropropene which is the same as a 78: 22 mole ratio. (CX-231 at 47). Comparison of the infrared, H-NMR and F-NMR spectra of the accused FOR 421, FOR 800 HE, FOR 5351, FOR 423 and FOR 65 B1/R with corresponding spectra VITON A and with corresponding spectra published for vinylidene fluoride-hexafluoropropene copolymmer showed that the Ausimont materials are clearly copolymers of vinylidene fluoride and hexafluoropropene and contain approximately 20 mole percent hexafluoropene units. (Harwood, Tr. at 1202; CX-231, addendum at 20, 21).

275. Harwood conducted analytical studies to characterize GM102E. He received two samples of GM 102E for analysis, and the first he understood to have been a sample of GM 102E that was obtained from an Ausimont source in the United States. The second GM 102E sample he received was given to him on about September 3, 1994. He understood that sample to be a sample of GM 102E that was obtained from Ausimont in Italy and this was a certified authentic sample. The conclusion from his studies on these samples is that both GM 102E samples are The characterizations which resulted in these conclusions include proton-NMR analysis; and, a chloride ion analysis conducted with an ion analyzer. (Harwood, CX-231, addendum at 2).

276. Harwood's work confirmed that the composition of XA51 samples was as expected, The analysis shows that XA51 was made from a mixture containing

277. Harwood observed that both

and benzyl tripehnyl phosphonium chloride show the characteristic behavior of phosphonium compounds in a simple phase transfer system. A similar observation was made in a system tested by Dr. Grootaert. In each instance the phosphonium cation acted as a phosphonium phase transfer catalyst (CX-231 at 45 to 47).

278. Harwood studied the operation of:

benzyl triphenyl phosphonium chloride; and, the salt of bisphenol AF anion and benzyl triphenyl phosphonium cation, as accelerators in the cure of fluoroelastomer compositions. The experiments show that all four compounds function as accelerators in the same manner to give the same cured result. (CX-215, CX-241 and CX-247).

279. Harwood's experiments were conducted using a

In those compositions in which one of the two phosphonium chlorides was used as phosphonium accelerator, bisphenol AF was provided in an amount of 1.29 moles per 100 grams, co-polymer. For each phosphonium chloride, several different compositions differing in the amount of phosphonium chloride present, per 100 grams co-polymer were prepared. Formulations were prepared both with and without the sulfone additive, of the Kolb '320 patent. When a sulfone additive was used, the selected sulfone was bis(p-chlorophenyl)sulfone. Harwood's study showed that the Ausimont phosphonium compound,

had essentially the same influence

on cure performance as did benzyl triphenyl phosphonium chloride. On this basis Harwood concluded that both are clearly phosphonium compounds which function in the same manner. A similar conclusion was reached in regard to the nearly identical behavior of the

and the benzyl triphenyl phosphonium ion. That is, both of those phosphorus compounds were clearly operating as quaternary phosphonium compounds, performing the same function in the same manner, to give the same cure. (Harwood, CX-231 at 45 to 49).

280. No difference in the cure (comparing the operation of the two phosphoniums) was obtained, either in the presence or the absence of the sulfone, i.e., while the presence of the sulfone had a detectable effect on the cure rate, the effect was essentially the same for both of the phosphonium compounds. When a "poison" in the form of nitrobenzoic acid was added, both systems failed to cure. (Harwood, CX-231 at 47-50, addendem at 23-33).

281. The phosphonium salt is not connected to the polymer to form crosslinks when Bisphenol AF is present. The salt is required for crosslinking because it is a phase transfer catalyst and the type of curing that occurs in the second curve in Figure 2 of Schmiegel with the triangles shows that the phosphonium salt does bring hydroxide up to cause dehydrofluorination which then allows cross-polymer linking to occur in the absence of bisphenol AF. Schmiegel (RRX-187, CX-134 at 44) concludes that "it appears that a substantially different and inferior kind of network can be formed in the absence of the bisphenol AF and that, at the modest accelerator levels required for practical ... [bisphenol AF] cures, the formation of the inferior network is greatly suppressed." Harwood interprets that statement of Schmiegel as the quaternary phosphonium compound in the absence of bisphenol

as being "responsible for the formation of the crosslinks ... [which is] different than form crosslinks." (Harwood, Tr. at 1222).

282. The inferior kind of network formed with an increased concentration of the phosphonium salt and with no Bisphenol AF is the type of cure that occurred in the "very early days," and with the Bisphenol AF present the inferior network is suppressed. (Harwood, Tr. at 1216 to 1219). With bisphenol AF present, the phosphonium compound is only a carrier catalyst for the reaction. Both processes (with or without bisphenol AF) require dehydrofluorination for the polymer before the crosslinking can take place and when bisphenol AF is present the crosslinks are present because the bisphenol AF adds to the unsaturation (Harwood, Tr. at 1222).

283. Solid phase means that all the ingredients of a mixture are in a solid phase. The date plotted in Figure 5 of CX-223 was based upon a solid phase study. (Worm, Tr. at 811). Schmiegel's work was done in liquid phase. (Worm, Tr. at 822). The temperature affects the rate of reactions (Worm, Tr. at 825).

284. Grootaert synthesized

and the bisphenol AF salt of benzyl triphenyl phosphonium cation. (CX-225 at 1).

285. Harwood tested Ausimont's organophosphorus material to see whether it can operate as a phase transfer agent. Grootaert tested it as a phase transfer catalyst. If it can operate as a phase transfer agent, it can operate as a phase transfer catalyst. Harwood's test demonstrated the ability of the transfer agent to transfer a chloride anion into a chloride phase. Grootaert's experiments had two components in it. It had the phase transfer

capability which brought a bromide ion into the organic phase and since that occurred, then catalysis occurred and the reaction occurred. Grootaret's test involved a reaction by bromide ion which is water soluble with an organic compound that is soluble in the organic phase. Grootaert demonstrated the transfer of the bromide ion from the aqueous phase into the organic phase so the reaction could take place. Hence there was the transfer of the bromide into the aqueous phase and the reaction of the bromide with the chloride to form octyl bromide. Grootaret went to benzylchloride in his second experiment because it is a much more reactive chloride than octyl chloride. It is incorrect to characterize Grootaert's first experiment as a failure. It was just something that needed improved design. If one wanted to wait long enough the reaction with octyl chloride would proceed. (Harwood, Tr. at 1231-36).

286. CX-227 is the protocol which was developed to compare the performance of two types of phosphonium compounds in this investigation. (Harwood, Tr. at 1303, 1304).

287. The experiments that are discussed at (b) (page 4 of CX-227) are reported in the table of CX-492 which is related to the experiments in CX-492 which have tables "Effect Of Tetramentylene Sulfone" and "Effect of p-chlorophenyl sulfone." In said section (b) of CX-227, there is an identification of the ODR instrument and the protocol that was used in operating it. (Harwood, Tr. at 1307, 1308).

288. The use of carbon black in fluoroelastomer compositions is to make the stock a little less expensive and as a reinforcing agent to make stronger material. A sulfone does not do the same thing as the carbon black. While both are adjuvants, the sulfone improves the processing of the material. (Harwood, Tr. at 1304, 1310).

289. The presence of phosphonium compound after the cure has taken place can promote additional curing reaction and this causes poor compression set characteristics. (Harwood, Tr. at 1338).

290. GM102E and Kolb's quaternary phosphonium compound are accelerators and hence speed up any crosslinking reaction. "[T]hey behave in exactly the same way." (Harwood, Tr. at 1355).

291. Harwood's studies show that the Ausimont phosphonium compound, has essentially the same influence on cure performance as does benzyl triphenyl phosphonium chloride. There was no difference observed in the way the two phosphonium chlorides were operating, to accelerate the cure. The result in each instance (wherein no poison was added to prevent the operation of the catalyst,) was a cure or vulcanized fluoroelastomer exhibiting similar physical characteristics such as hardness, elongation at break and modulus. (Harwood, CX-231, at 47-50, addendum at 23-33).

292. Phase transfer catalysis is the transfer of one component from one phase into a second phase. Phase transfer catalysis only involves bringing the reactants together for a later reaction, <u>i.e.</u> only involves a migration. Grootaert's tests were conducted in a two phase solution system at room temperature. (Grootaert, Tr. at 966, 974).

293. If one attempts to perform a chemical reaction between two chemicals which are not in the same phase, one would not be able to have reaction unless one brings the reactants together. (Grootaert, Tr. at 944, 945).

294. Grootaert compared GM102E and a quaternary phosphonium salt named in the '320 patent (Tr. at 945). He showed that there was a reaction between

the bromide ion of sodium bromide in water and benzylchloride to form benzyl bromide by adding GM102E or said quaternary phosphonium salt which compounds were able to transfer the bromide to the organic phase for subsequent reaction. (Grootaert, Tr. at 944 to 965).

295. The (Engel, Tr. at 1506 to 1508). In a fluoroelastomer cure in nonvalent system in a mold it would be expected that they would both act in the same way, <u>i.e.</u> in a fundamentally similar manner based on an extrapolation of the solution system, the phase transfer studies of Grootaert, phase transfer experiment of Harwood and published reports. (Harwood, CX-231 at 47-50, addendum at 22-33; Engel, Tr. at 1555 to 1557).

296. Engel in his experiments with the quaternary phosphonium compound benzyl triphenyl phosphonium chloride and GM 102E reported spectral properties indicative of a positive charge on the phosphorus atom. Also, comparting the phosphorus-NMR and protein NMR spectra on GM 102E to the analogous spectra of benzyl triphenyl phosphonium chloride, Engel found little difference with respect to the positive charge on the phosphorus. (Engel, CX-242 at 29 to 31; Tr. at 1437 to 1458).

297. Dr. V. cannot recall any published works or presentations that dispute Schmiegel's proposed mechanism in RX-152. (Tr. at 91).

298. If a compound acts as a phase transfer catalyst in a liquid system, there is no reason to expect that it would act differently in a solid system, <u>i.e</u>. if there is no difference between the operation of two materials as phase transfer catalysts in liquid systems, there is no reason to believe that the two materials would operate differently as phase transfer catalysts under vulcanization conditions. (Grootaert, Tr. at 976 to 980).

299. Grootaert knows of no reason to believe that phosphonium compounds acting as phase transfer catalysts in the curing of fluoroelastomer would behave any differently in a solid/solid phase as compared to a liquid/liquid phase. (Tr. at 979, 980).

300. Harwood knows of no reason to believe that phosphonium compounds acting as phase transfer catalysts in the curing of fluoroelastomers would behave any differently in a solid/solid phase as compared to a liquid/liquid phase. (Tr. at 1345 to 1351).

301. None of the products identified in CX-102 and CX-103 (Tables showing 3M Fluorel products under the '320 patent), and CX-121 (3M Technical information sheets) were commercially available in 1973. (Worm, Tr. at 686, 687). A copolymer of vinylidene fluoride and perfluoropene in molar proportions of 78:22 (CX-1, col. 10, line 65) was commercially available as of October 13, 1993. (Worm, Tr. at 687). At that time 3M was selling three raw gums, viz. high viscosity gum (KF2140), medium viscosity gum and low viscosity gum (2147). The medium viscosity gum would correspond the closest to JC-3163. (Worm, Tr. at 687).

302. JC-3163 gum was not sold prior to 1973, other than as part of an incorporated cure product. In 1975 FC 2175 was introduced and it continued to be sold. FC 2175 was prepared from JC-3163 but is no longer prepared from JC-3163. (Worm, Tr. at 689, 690).

303. In rubber compounding, a process aid gives one improved flow in filling a mold or in extruding or in improved mixing of compound ingredients (less time in the mixer). (Worm, Tr. at 705).

304. The diorgansulfur oxide serves as a process aid in extrusion. Thus it comes to the surface of the compound in extrusion and acts as a

lubricant along the metal wall of the extruder dye. This effect will vary depending on the temperatures and what the temperature is. (Worm, Tr. at 710, 711).

305. The diorganosulfur oxide of the Kolb '320 patent also serves as a process aid in compression molding. Thus it will help with the flow of the gum in the cavity so that it will flow evenly before it starts curing. (Worm, Tr. at 711).

306. With compression set, the diorganosulfur oxide serves as a process aid in giving the release from the mold. (Worm, Tr. at 711, 712).

307. In injection molding the diorgansulfur oxide helps the flow of the gum into the cavity aid after the part is cured it helps to release it. (Worm, Tr. at 712).

308. Most process aids before the Kolb '320 patent affected things like compression set. The dioganosulfur oxide of the Kolb patent does not. (Worm, Tr. at 722).

309. If one was making O rings with the claimed '320 compositions, he would take the mixture out of the press as soon as the maximum torque was reached (starts flattening out) and then do post curing. (Worm. Tr. at 807, 808).

M. Validity

310. At the time inventor Kolb ran the experiments that is the basis for his '320 patent the quaternary phosphonium compound used were the oniums of the state-of-the art that were cheap to make and low cost and would be the phosphonium compound of choice. Kolb did not go out and try to evaluate every quaternary phosphonium salt because there is a "list from forever and that I did not do. This was not part of the invention. The invention was based on

trying to improve existing cure system." The existing cure system Kolb's invention was trying to improve was the one using a quaternary phosphonium compound. (Kolb, Dep. Tr. at RX-121 at 111).

311. Worm's understanding as to the difference between the subject matter claimed in the Kolb '320 patent and that which is disclosed or claimed in the Patel '877 patent is that the '877 patent only claims the polymer and the quaternary phosphonium compound. (Worm, Tr. at 66).

312. Worm agreed that it is correct that with the exception of the diorganosulfur oxide compound, all of the other ingredients recited in the claims of the '320 patent were known in the art prior to the date of filing of the Kolb '320 patent. (Worm, Tr. at 666).

313. Referring to claim 1 of the '320 patent, the deBrunner '787 patent (RX-9) shows the elastomeric copolymer (col. 2, links 8-9 and col. 2, line 58 to col. 3, line 3), the quaternary phosphonuim or ammonium compound (col. 5, lines 57-61 and col. 6, lines 1-22) the acid acceptor and/or base (col. 2, lines 32-35 and col. 3, lines 4-32) and the aromatic hydroxy or amino compound. The Pattison '654 patent (RX-10) shows the same components (col. 1, lines 47-48 and col. 2, line 50 - col. 3, line 11; col. 1, line 50 - col. 2, line 6, col. 5, line 24 - col. 6, line 10, and col. 6, lines 42-46; col. 2, lines 32-35 and col. 3, lines 12-41; and col. 2, lines 36-37 and col. 3, line 42 - col. 4, line 26).

314. None of the references asserted by Ausimont as rendering the '320 patent obvious discloses the use of a diorgano sulfur oxide with a fluoroelastomer. (Engel, CX-242 at 45-46).

315. Morrison and Boyd which is a textbook titled "Organic Chemistry" (Third Edition) (1973). (CX-27, RX-13) discloses at 31, 32 that "[r]ecent

years have seen the development and widespread use of aprotic solvents: polar solvents of moderately high dielectric constants, which do not contain acidic hydrogen"; that for example dimethyl sulfoxide, N,N-dimethylformamide and sulfolane dissolve both organic and inorganic reagents but, in dissolving ionic compounds, solvate cations most strongly, and leave anions relatively unencumbered and highly reactive; and that anions are more basic and more nucleophilic; that since about 1958 reports of dramatic solvent effects on a wide variety of reactions have appeared, first about dimethylformamide and more recently about dimethyl sulfoxide; that reactions that, in most solvents, proceed slowly at high temperature to give low yields may be found, in an parotic solvent, to proceed rapidly - often at room temperature - to give high yields; and that the "solvent is intimately involved in any reaction that takes place in it, and we are just beginning to find out how much it is involved, and in what way."

316. The description Morrison and Boyd reference (RX-13, CX-27) is not related to addition of small amounts of diorgano sulfur oxide to cure systems, or information from which one could predict the effects of such additions. It has no relevance to the fluoroelastomer cures in the Kolb '320 patent and is directed to a clearly different kind of use than the Kolb '320 patent is directed to. (Engel, CX-242 at 49-50).

317. The Carpenter reference, U. S. Patent No. 2,964,503 issued on December 13, 1960 on an application filed March 29, 1957. (CX-25). The disclosed invention relates to polysulfide synthetic rubber and polysulfide polymers and is based on the concept that control over the rate of cure and a modification of the characteristics of the cured composition may be achieved by incorporating into a curing composition composed essentially of a base

mixture of polyalkylene polysulfide polymers and a soluble curing agent adapted to liberate anions of chromium when in solution, between about 2 and 50 parts of a modifying and solubilizing agent from the group of amides, sulfoxides, sulfones, sulfonamides, phosphoramides, esters of phosphoric acid, esters of boric acid, and esters of monobasic and polybasic organic acids per each 100 parts of polyalkylene polysulfide polymer. (col. 3, lines 4-14). It is disclosed that the presence, with a curing component, comprising a soluble salt of chromic acid, of said solubilizing agent not only modifies the rate of cure of polyaklylene polysulfide polymers but also uniquely modifies the properties of the cured composition, i.e. enhancing resistance to swelling and to loss of physical properties in the presence of water and at elevated temperatures, and correspondingly greater resistance to thermal extrusion and interfacial sponging. (col. 2, lines 25 to 43). It is also disclosed that the novel method and desirable results of the invention are preferably obtained by use of curing components conforming to the many necessary requirements set forth in Patent No. 2,787,608 which curing components, comprising broadly the soluble salts of chromic acid which upon solution liberate anions containing chromium, not only effect a cure at normal atmospheric temperatures but also produce in the cured elastomers enhanced properties of resistance to high temperature, compression set and to the solvent action of hydrocarbons. (col. 4, lines 6 to 17).

318. There is no recitation in the Carpenter patent (CX-25) to the use of any of the Carpenter materials to effect cures which are catalyzed by quaternary phosphonium or quaternary ammonium compounds The Carpenter patent does not disclose whether using a sulfone or sulfoxide in a fluoroelastomer would result in a faster cure, whether scorch would be less of a problem, and

whether mold release would be made easier. (Engel, CX-242 at 51-52).

319. The Busse U.S. Patent No. 2,522,776 issued on September 19, 1950 on an application filed on October 11, 1946 and relates to the "tackification" and plasticization of synthetic rubbers "of all kinds," particularly in order to improve the processing of the synthetic rubber and to improve the properties of the cured products obtained therefrom. It is disclosed that cured synthetic rubber products were found to have a number of troublesome objections, the most important of which are their tendency to generate a large amount of heat on flexing, their low elongation at high temperatures, their tendency to crack rapidly after cracking is initiated and their tendency to become hard and brittle at low temperature. (col. 2, lines 1 to 9). Busse found that some of those manifold objections to the processing of synthetic rubber and to the properties of the cured products may be overcome by incorporating in the synthetic rubber an aryl sulfone or a polysulfone. Examples of polysulfones are polyvinyl phenyl sulfone and polypropylene sulfone. (col. 3, lines 1 to 35). The inventor discloses that the invention is applicable to any of the known synthetic rubbers such as polymerized diolefines. The improvement is most marked in the case of for example the copolymers of butadiene with styrene and butadiene. (col. 3, lines 55-70). The sulfones in the Busse invention operate both to tackify and plasticize the synthetic rubber. Plasticization involves fundamentally a softening action whereas tackification involves the rendering of the surface of a substance to which the tackifier is added sticky and tacky. (col. 3, lines 7-75, col. 4, lines 1-5).

320. The rubbers in the Busse patent (CX-24) are not fluoroelastomers and have very different cure characteristics and involve different reaction

systems as compared to the fluoroelastomers. Reactions in the Busse patent do not involve catalysis through the use of a quaternary phosphonium or quaternary ammonium compound. The Busse patent does not have any relationship to the '320 patent and even in combination with the other references relied upon by Ausimont does not predict the invention of the '320 patent. (Engel, CX-242 at 52-53).

N. Direct Infringement

321. Salvatore Aloisio is the business manager for TECHNOFLON fluoroelastomers for respondent Ausimont U.S.A. (Aloisio, CPX-4 at 7).

322. Donald J. Myer is the technical service manager for TECHNOFLON for respondent Ausimont U.S.A. (Myer, CPX-9 at 5).

323. An acid acceptor and an optional base must be added to the accused compositions in order to crosslink the polymer. (Tommasi, Tr. at 986-89; Aloisio, CPX-4 at 53-54).

324. Some Ausimont customers follow the recommended recipe for vulcanization of the accused compositions, which includes addition of an acid receptor and optional base within the limits of the '320 patent. (Myer, CPX-9 (Vol I) at 19-30; Myer, CPX-10 (Vol 11) at 104-05).

325. Ausimont's Myer testified that CX-358 is a packing slip indicating a shipment of 10 lbs. of a compound

which compound had the following composition:

(CX-358). Myer Testified that the ingredients "were all mixed together" in the product that was sent to (Myer, CPX-10 at

101-02).

326. CX-359 is a packing slip indicating the shipment of 5.5 lbs of a compound which compound had the following formulation:

(CX-359). Myer testified that what was sent to

327. CX-353 is a report by Myer of mixing tests done at Ausimont U.S.A. in the United States on Ausimont's compositions 800 HE and 65BI/R, the purpose of which tests was to determine the effect of various mixing procedures on the finished product. (Myer, CPX-9 at 72; CX-353). Several batches were produced as shown by the following formulations of CX-353:

(CX-353 at A012958). After mixing, CX-353 indicates that "slabs were cured" and "Mooneys and ODR's were run on each compound." (CX-353 at A012957) Myer testified that no parts were molded from the compounds. (Myer, CPX-9 at 73). CX-353 also reflects Myer's observations concerning the cure times of the various compounds. (CX-353 at A012957).

328. CX-370 is an Ausimont packing slip, dated May 3, 1990, indicating the shipment of two compounds,

Under a section headed "Comments," CX-370 identifies the formulation of lot A as follows:

(CX-370).

329. Regarding CX-170, Ausimont's Myer testified at his deposition that the above formulation for lot A was the formulation of compound

(Myer, CPX-10 at 141).

330. CX-380 is a document headed "TECHNOFLON Tech. Ser. Project List 3/12/92," which was developed by Ausimont U.S.A. to show the laboratory projects in progress at Ausimont U.S.A. as of March 12, 1992. (CX-380; Myer, CPX-10 at 160).

331. Regarding CX-380, Ausimont's Myer testified as follows:

> Q. Was 9550 supplied to
>
> A. I'm not sure.
>
> Q. Now from this document, would it indicate that perhaps TH 340 and 9550 were the two compounds that were to be supplied to
>
> A. Two compounds were supplied according to this document, and it would be reasonable to expect that one of the compounds contained TH 340 and the other one contained 9550.
>
> Q. Okay. Now over the column that says "TESTING," you'll notice that with respect to Santech, first it says "Physicals" and then it says "Rheology." Okay? Now does that mean, in accordance with how you understand these documents were kept, does that mean that both physicals and rheology were to be conducted on each of the TH 340 and the 9550, or does it mean that physicals were to be

done on the TH 340 and rheology was to be done on the 9550?

A. Both were to be done on both compounds.

Q. Both were to be done on both compounds, okay.

To do the physical you would have to produce test slabs with these compounds; is that correct?

A. That's correct.

Q. Do you know the formulations or the recipes that were used with the 9550 to make the test slabs?

A. I don't remember what they were.

Q. Would it be what you keep referring to a the standard test recipe?

A. It could be.

Q. Let me ask you the question because maybe it will clear some of these things up: When you're running these tests like this, does everybody formulate to the same test recipe so that theoretically the results would be repeatable over and over regardless of who ran the tests?

A. "Everybody" as --

Q. Like in the industry. If one of your customers, assuming they have the test equipment, or 3M or Dupont or whoever, was going to be running -- you know, making test slabs, would they all use the same recipe to make the slabs?

A. 3M and Dupont especially will use the same basic test recipe.

Q. The same recipe that Ausimont would use?

A. Yes.

Q. Does the test recipe for these type of fluoroelastomers specify three and six parts of the mag oxide and the calcium hydroxide?

A. Yes.

(Myer, CPX-10 at 161-63).

O. Induced Infringement

332. Ausimont's product brochures and technical data sheets provide

test recipes in which an acid acceptor and an optional base are included with the commercial product in amounts within the ranges provided in the claims of the '320 patent. Along with the test recipe, Ausimont provides ODR data and physical characteristic measurements for products vulcanized according to the test recipe. (<u>See</u> <u>e.g.</u>, CX-239; CX-245; CX-260; CX-261; CX-315; CX-316; CX-317; CX-318; CX-339; Brullo, CX-100 at 18).

333. Ausimont U.S.A. personnel recommend to customers recipes for fluoroelastomers which include amounts of acid acceptors and optional bases within the patent ranges. (Aloisio, CPX-4 at 50; Myer, CPX 9 at 21-22). Although customers may not tell Ausimont what recipe they use, some Ausimont customers follow the Ausimont recommendations. (Myer, CPX-9 (Vol. I) at 19-30; Myer, CPX 10 (Vol. II) at 104-05).

334. Myer has never recommended using levels of acid acceptor and optional base outside the limits contained in the '320 patent. (Myer, CPX-9 (Vol. I) at 22).

335. Myer testified that he has recommended the use of FOR 9550 to two or three customers, and that he has sent samples of FOR 9550 to customers and that Ausimont U.S.A. is still attempting to sell FOR 9550. (Myer, CPX-9 at 23).

336. CX-362 is a sample sheet which indicates that by May 1988 Ausimont had shipped (CX-362 at A001220). With respect to such sample shipments, Ausimont's Myer testified during his deposition as follows:

> Q. When you sent out samples to customers to try or evaluate, was it customary to send along a product data sheet with it?
>
> A. On occasion.
>
> Q. And did those product data sheets include a suggested

recipe?

A. Most properties are developed on a standard test recipe.

Q. So would the product data sheet have that recipe?

A. Of course.

Q. And that test recipe would list an acid acceptor and base somewhere in the range between three to 40 parts per hundred?

A. Yes.

(Myer, CX-10 at 111-12).

337. CX-364 indicates that Ausimont had 114 lbs. of FOR-9550 in inventory as of November 30, 1992. (CX-364 at A010831; Myer, CPX-10 at 118-19).

338. With respect to the 114 lbs. of FOR-9550 in Ausimont's inventory in November 1992, Myer testified as follows:

Q. Has any of the FOR 9550 been sold, do you know?

A. Not that I know of.

Q. It has been sent to customers though; is that correct?

A. Could you clarify the question a little bit?

Q. Well, at some time or other --

Well we know FOR, for example, 9550 was sent to a company called

A.

Q.

A. Yes.

Q. Was it sent to other customers as well, do you know?

A. 9550? Yes.

Q. Do you know how many?

A. Not off the top of my head, no.

(Myer, CPX-10 at 119).

339. CX-373 is a "Call Report," dated July 3, 1990, concerning a sales call by Ausimont sales representative Ron Westfall and Sal Aloisio to which states, in part, as follows:

> 70 and 90 duro 65BIR and 9550 compounds worked well in plant trial. There were no differences seen when compared to 3M FLUOREL.
>
> * * *
>
> NOTE: Do we have FOR 9550 in USA stock?

There is also a handwritten notation on CX-373, which notation is dated July 5, 1990, and which states as follows:

> Ron -- We have 63 lbs!! Count on a lead time of 6-8 wks. from receipt of order. Regards -- Angela

(CX-373).

340. Regarding CX-373, Myer testified in his deposition that "plant trial" refers to a plant trial done at and further that he does not know what recipe or formulation was used by (Myer, CPX-10 at 145).

341. CX-375 is a letter to a from Ausimont's Myer, dated October 18, 1988, concerning "samples" of TECHNOFLON fluoroelastomers sent to by Ausimont. The samples sent are described therein as follows:

Grade	Quantity	Type	Cure Containing	% Fluorine (typical)
NH	5 lbs.	Copolymer	No	65
FOR-9550	5 lbs.	Copolymer	Yes	65
FOR-LHF	2 lbs.	Copolymer	Yes	65

CX-375 further provides, in part, as follows:

> We have also included FOR-9550 which is an o-ring type grade containing a Bisphenol cure system. FOR-9550 is based on a high level of NH and also contains a process aid like FOR-65BIR. You may wish to blend 65BIR and 9550 or blend 65BIR with NH adding M1 and M2 to get the necessary state of cure.

(CX-375).

342. Ausimont's Myer testified in his deposition that he does not know whether ever used the FOR-9550 to mold any products or make any test samples, and that he does not recall whether he sent a recipe to be used with the FOR-9550. (Myer, CPX-10 at 149).

P. Contributory Infringement

343. The accused compositions are intended to be cured by addition of an acid acceptor and/or an optional base. (Tommasi, Tr. 987-89, 993-94).

344. As to applications for the accused formulations which do not involve curing with a base, Ausimont's Tommasi testified as follows:

> Q ... Without an acid acceptor or base, the formulations aren't useful for anything, are they?
>
> A I said I don't know for sure if 100 percent of the applications are as cured items or if there are some minor applications that the customers might have invented or designed or decided or agreed upon with our research assistant.
>
> Q You know of any such applications?
>
> A No, I don't.
>
> Q Then based upon what you do know --
>
> A Excuse me, no, I don't, from these formulations.
>
> Q For the accused formulations?
>
> A I told you I know of applications for other kinds of formulations of rubber which is not being cured.
>
> Q I'm referring only to the accused formulations, Mr. Tommasi. You know of no other application for those formulations except as cured?
>
> A Yes, you're right.

(Tommasi, Tr. at 993-94).

345. Complainant's Brullo testified that there are no known

applications for the accused formulations which do not involve curing them with a base. (Brullo, CX-100 at 16-17).

Q. Domestic Industry

346. Robert A. Brullo is the manager of complainant's Specialty Fluoropolymers Department, which is part of the Industrial and Consumer business sector of complainant. (Brullo, CX-100 at 1).

347. Complainant has three business sectors: Life Sciences; Information, Imaging and Electronics; and Industrial and Consumer. (Brullo, CX-100 at 4).

348. The fluoroelastomer compositions that are the subject of this investigation fall under the responsibility of the Specialty Fluoropolymers Department of the Industrial and Consumer business sector. (Brullo, CX-100 at 4).

349. Complainant has been researching fluoroelastomers since 1953 and has been selling fluoroelastomers since 1959. (Brullo, CX-100 at 5).

350. Complainant sells fluoroelastomer products in the United States of which are covered under the '320 patent. These products include FLUOREL FC-2121, FC-2123, FX-2124Q, FC-2144, FC-2174, FC-2173, FC-2176, FC-2180, FC-2181, FC-2181PS, FC-2182, FT-2350, FT-9038, FX-9143, FE-5620Q, FE-5620-23, FE-5640Q, FE-5620-40, FE-5660Q, FE-5840Q, FC-2182P, FLS-2330 (a/k/a FC2330), FX-11700 and FX-11900. (CX-102; CX-103).

351. Complainant's FLUOREL products listed in the preceding finding are sold by 3M in the United States and have the formulations shown on CX-102 and CX-103. (CX-495, Stipulation No. 8).

352. About of complainant's FLUOREL and FLUOREL II sales are accounted for by FLUOREL products covered by the '320 patent. (Brullo, Tr. at

434).

353. For the products listed in CX-102 and CX-103, 3M has made significant investment in plant and equipment. (CX-495, Stipulation No. 9).

354. Complainant has, in the products listed in confidential exhibits CX-102 and CX-103, made significant employment of labor and capital. (CX-495, Stipulation No. 10).

355. Complainant has, in the products listed in CX-102 and CX-103, made substantial investments in the exploitation, including engineering research and development. (CX-495, Stipulation No. 11).

356. Ausimont agrees that complainant's fluoroelastomer products utilize the technology of the Kolb '320 patent. (Tr. at 386).

357. Brullo testified that all of complainant's domestically sold FLUOREL and FLUOREL II fluoroelastomers with incorporated cure compounds, specifically the ones which include a sulfone, are produced at

(Brullo, CX-100 at 23).

358. CX-122 reflects the amount of money spent by complainant on sales and marketing costs for the patented '320 compositions from 1987 through August 1994. (Brullo, CX-100 at 21; CX-122).

359. CX-123 reflects the approximate spending by complainant on its laboratory and engineering for the period 1987 through August 1994 for the patented '320 compositions. (Brullo, CX-100 at 21; CX-123).

360. CX-124 reflects complainant's sales of products that are the subject of this investigation and all FLUOREL products. (Brullo, CX-100 at 22; CX-124).

361. CX-125 reflects the square footage of the

the manufacture of FLUOREL products during the period 1987 through 1993.

(Brullo, CX-100 at 25; CX-125).

362. CX-126 reflects the capital expenditures made by complainant relating to the manufacture of FLUOREL products between 1977 and 1994. (Brullo, CX-100 at 25; CX-126).

363. CX-127 reflects the complainant's production history of the number of labor hours spent on the production of its FLUOREL products from 1987 through 1993. (Brullo, CX-100 at 25; CX-127).

VIII. CONCLUSIONS OF LAW

1. The Commission has _in rem_ jurisdiction and subject matter jurisdiction.

2. The Commission has _in personam_ jurisdiction over the respondents.

3. The '320 patent is not invalid.

4. Complainant has sustained its burden in establishing that respondents infringe the claims in issue under the doctrine of equivalents.

5. Complainant has not sustained its burden in establishing that respondents literally infringe the claims in issue.

6. There is a domestic industry with respect to the asserted claims of the '320 patent.

7. There is a violation of section 337.

IX. INITIAL DETERMINATION AND ORDER

Based on the foregoing findings of fact, conclusions of law, the opinion, and the record as a whole, and having considered all of the pleadings and arguments presented orally and in briefs, as well as certain proposed findings of fact, it is the administrative law judge's determination that there is a violation of section 337 in the importation into the United States and sale for importation, or the sale within the United States after importation of certain curable fluoroelastomer compositions and precursors thereof.

The administrative law judge hereby CERTIFIES to the Commission this initial determination, together with the record consisting of the following:

1. The transcript of the hearing; and
2. The exhibits admitted into evidence.[21]

The pleadings of the parties filed with the Secretary are not certified, since they are already in the Commission's possession in accordance with Commission's interim rules.

Further it is ORDERED that:

1. In accordance with Commission interim rule 210.44(b), all material heretofore marked <u>in camera</u> because of business, financial, and marketing data found by the administrative law judge to be cognizable as confidential business information under Commission interim rule 210.6(a) is to be given <u>in camera</u> treatment continuing after the date this investigation is terminated.

2. Counsel for the parties shall have in the hands of the administrative law judge those portions of the initial determination which

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[21] At the prehearing conference all exhibits were admitted without objection. However, <u>see</u> Order No. 15.

contain bracketed confidential business information to be deleted from the public version of the initial determination, and all attachments thereto, no later than Friday, December 30, 1994. Any such bracketed version shall not be served by telecopy on the administrative law judge. If no version is received from a party it will mean that the party has no objection to removing the confidential status, in its entirety, from this initial determination.

3. This initial determination shall become the determination of the Commission forty-five (45) days after the service thereof, unless the Commission, within forty-five (45) days after the date of filing of the initial determination shall have ordered review of the initial determination or certain issues therein pursuant to Commission interim rules 210.54(b) or 210.55 (19 C.F.R. § 210.54(b) or § 210.55) or by order shall have changed the effective date of the initial determination.

Paul J Luckern

Paul J. Luckern
Administrative Law Judge

Issued: December 15, 1994