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TYPICAL EXAMPLE OF ANALYSES ACTUALLY CARRIED OUT BY CUSTOMS

(Item III.2 on Agenda)

Reference documents :

41.678 (SSC/13)

1. Following the publication of Doc. 41.678, the Secretariat received information on typical examples of analyses from the Indian Customs Laboratory.

2. The above information has been arranged under the same titles as those in Doc. 41.678 and set out in the Annex to this document.

3. The Sub-Committee is invited to take the above information into account while examining this Agenda Item.

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File No. 2134

IV. TYPICAL EXAMPLE OF ANALYSES (contd.)

No.	Description of the product	HS Code(*)
47	Calcium Carbonate	2836.50
48	Titanium Dioxide	28.23
49	Stainless Steel	72.18
50	Ferro Silicon (Ferro Alloy)	7202.21
51	Low density polyethylene	3901.10
52	Polypropylene Wide Spec (granules)	3902.10
53	Lignarome - 4 Hydroxy - 5 Methyl 1,3 Mthoxy Benzaldehyde - Aromatic Chemical	2912.41
54	Woven fabric of synthetic staple fibres containing 85% or more by weight of synthetic staple fibres.	55.12
55	Woolen Yarn containing 85% or more by weight of wool	5107.10
56	White circular tablets scored on one side. Each tablet is purported to contain Sulfadoxine USP 500 mg, Pyrimethamine USP 25 mg	3004.20
57	Pale yellow liquid with characteristic odour, declared as Peppermint oil	3301.24

(*) The HS Code of the products are merely suggested classification, not authorised by the WCO

Example No. 47

1. Description of the product :
   Calcium Carbonate

2. Purpose of analyses :

   In order to confirm -
   (I) Whether the sample is Calcium Carbonate
   (ii) Purity of Sample.

3. Analytical methods and procedures :
   (1) Physical appearance
   (2) thermal decomposition
   (3) Solubility
   (4) Moisture Content
   (5) Test for Calcium
   (6) Test for carbonate
   (7) Loss on ignition
   (8) Purity determination (as per ANALAR Standards for Laboratory chemicals - 6th Ed., ANALAR STANDARD LTD., London P. 132).

4. Results of analyses :
   (1) White, fine crystalline powder
   (2) Non-combustible
   (3) Insoluble in water, soluble in HCl
   (4) Moisture Content at (100±2 °C) = 0.21 % W/W
   (5) Test for Calcium (+ve)
   (6) Test for Carbonate (+ve)
   (7) Loss on ignition - 43.7 % (W/W)
   (8) Purity = 98.6 % W/W.

5. Discussion and conclusion :
   The sample is calcium carbonate, Purity - 98.6 % W/W.

6. Suggested HS Classification :
   Subheading 2836.50

1. Description of the product :
   Titanium Dioxide

2. Purpose of analyses :

   To confirm -
   (I) Whether the sample is TiO_2
   (ii) Purity of Sample.

3. Analytical methods and procedures :
   (1) Physical appearance
   (2) Heating in a dry test tube
   (3) Solubility
   (4) Moisture Content
   (5) Test for Titanium
   (6) Relative Density determination
   (7) Purity (determined by Aluminium reduction method : A solution of dry sample is made with conc. H_2SO_4 and Aluminium Sulphate. Titanium is reduced to trivalent state by Aluminium & titrated against Ferric Ammonium Sulphate using Ammonium Thiocyanate as indicator).

4. Results of analyses :
   (1) White, amorphous fine powder
   (2) Turns yellow when heated in a dry test tube and white on cooling
   (3) Insoluble in water, soluble in conc. H_2SO_4
   (4) Moisture Content at (100±2 °C) = 0.2 % W/W
   (5) Test for titanium (+ve)
   (6) Relative density = 4.1
   (8) Purity = 97.6 % W/W.

5. Discussion and conclusion :
   The sample is Titanium Dioxide, Purity - 97.6 % W/W.

6. Suggested HS Classification :
   Heading 28.23

1. Description of the product :
   Stainless Steel

2. Purpose of analyses :

   To confirm whether stainless steel as per the requirements of Note 1 (c) for Chapter 72.

3. Analytical methods and procedures :
   (1) Appearance - Shining white metallic piece
   (2) Test for Iron
   (3) Test for Carbon
   (4) Test for Chromium
   (5) Estimation of Carbon :
   Volumetric method by Strohlein apparatus - The sample is burnt in a current of pure oxygen in presence of a suitable flux. After removal of sulphurous gases by suitable absorbents, the carbon dioxide gas is collected in a specially jackated burette along with excess of oxygen. The carbon dioxide is then absorbed in alkali. On passing the excess oxygen back to the burette, the contraction in volume is read against a scale calibrated directly to the percentage of carbon.
   (6) Estimation of Chromium : Chromium by the Peroxydisulphate oxidation - Titration Method. Ref.-ASTM 1990 - E 353-89, Vol. 03.05 Page 515.

4. Results of analyses :
   (1) Test for Iron - Positive
   (2) Test for Carbon - Positive
   (3) Test for Chromium - Positive
   (4) Carbon Content = 0.06 % (W/W)
   (5) Chromium Content = 18.6 % (W/W).

5. Discussion and conclusion :
   Product conforms the composition of stainless steel.

6. Suggested HS Classification :
   Heading 72.18

1. Description of the product :
   Ferro Silicon (Ferro Alloy)

2. Purpose of analyses :

   To confirm whether Ferro Silicon - a ferro alloy as per the requirements of Note 1 (c) for Chapter 72.

3. Analytical methods and procedures :
   (1) Appearance - Metallic Lump
   (2) Test for Silicon
   (3) Test for Iron
   (4) Estimation of Silicon - Silicon by the Sodium Peroxide Fusion - Perchloric Acid dehydration Method. Ref.-ASTM 1990, E 360-85, Vol. 03.05 Page 553
   (5) Estimation of Iron - By Stannous Chloride reduction - Potassium dichromate volumetric titration method.

4. Results of analyses :
   (1) Test for Silicon (Positive)
   (2) Test for Iron (Positive)
   (3) Silicon Content = 65.4 % (W/W)
   (4) Iron Content = 34.1 % (W/W).

5. Discussion and conclusion :
   The product conforms the composition of Ferro Silicon (Ferro Alloy). Silicon = 65.4 % (W/W).

6. Suggested HS Classification :
   Subheading 7202.21

1. Description of the product :
   Low density polyethylene.

2. Purpose of analyses :

   To confirm whether the product is low density polyethylene polymer or not.

3. Analytical methods and procedures :
   (1) Appearance
   (2) Solubility test
   (3) Test for polyethylene
   (4) Determination of softening point (Ref. - Simple method for identification of plastics by Dietrich Brown, Hanser Publication, Page 50 - 1982)
   (5) Determination of specific gravity (Ref. - I.S. 2530 - 1991)
   (6) I.R. Spectrophotometry.

4. Results of analyses :
   (1) White translucent granules.
   (2) Soluble in hot xylene
   (3) Test for polyethylene (+ve)
   (4) Softening point, 108 °C
   (5) Specific Gravity, 0.925
   (6) I.R. Spectral pattern of the sample indicates that it is low density polyethylene polymer.

5. Discussion and conclusion :
   The sample is in the form of white translucent granules. Product is polyethylene polymer having specific gravity less than 0.94.

6. Suggested HS Classification :
   Subheading 3901.10

1. Description of the product :
   Polypropylene Wide Spec(granules).

2. Purpose of analyses :

   To confirm whether the product is polypropylene polymer or not.

3. Analytical methods and procedures :
   (1) Appearance
   (2) Solubility test
   (3) Test for polypropylene (Ref. - Simple Method for Identification of Plastic by Dietrich Brown, Hanser Publisher, Page 67 - 1982)
   (4) Determination of Softening point (Ref. - Simple method for identification of plastics by Dietrich Brown, Hanser Publication, Page 50 - 1982)
   (5) I.R. Spectrophotometry.

4. Results of analyses :
   (1) White translucent granules.
   (2) Soluble in hot xylene
   (3) Test for polypropylene (+ve)
   (4) Softening point 155 °C
   (5) I.R. Spectral pattern of the sample indicates that it is polypropylene polymer.

5. Discussion and conclusion :
   The sample is a white translucent granules. The product is polypropylene polymer.

6. Suggested HS Classification :
   Subheading 3902.10

1. Description of the product :
   Lignarome - 4 Hydroxy - 5 Methyl 1, 3 Methoxy Benzaldehyde - Aromatic Chemical

2. Purpose of analyses :

   To ascertain nature and composition.

3. Analytical methods and procedures :
   (1) Appearance
   (2) Burning Characteristics
   (3) Solubility
   (4) Test for elements (Sodium fusion test)
   (5) Melting Point
   (6) Identification by colour test - Aqueous solution of the sample + Ferric Chloride solution - A blue colour appears which turns brown on heating, white ppt. is produced on cooling.
   (7) UV-VIS Spectrum of the sample in 0.1 N NaOH
   (8) FTIR spectrum of the sample in Kbr.
   (9) Estimation of Carbon, Hydrogen and Oxygen by Elemental Analyser.

4. Results of analyses :
   (1) White crystalline powder.
   (2) Burning characteristics - organic, aromatic
   (3) Insoluble in water. Soluble in Ether, Chloroform, Methanol, Ethanol.
   (4) Found negative for Nitrogen, Sulphur, Halogens and Phosphorous
   (5) Melting Point 81.5 °C to 82.5 °C (Reported Melting Point of Vanillin 81 °C to 83 °C)
   (6) Identification by colour test - positive for Vanillin
   (7) UV-VIS Spectrum of the sample compares well to that of Standard Vanillin solution (in 0.1 N NaOH) - (ANNEX I)
   (8) FTIR Spectrum of the sample is superimposable to FTIR spectrum of Standard Vanillin - (ANNEX II)
   (9) Percentage of C, H and O in sample is in agreement with theoretical values for C, H and O in Vanillin

5. Discussion and conclusion :
   Sample is an aromatic organic compound, identified as Vanillin (4 Hydroxy - 3 - Methoxy Benzaldehyde).

6. Suggested HS Classification :
   Subheading 2912.41

1. Description of the product :
   Woven fabric of synthetic staple fibres containing 85 % or more by weight of synthetic staple fibres.

2. Purpose of analyses :

   To confirm whether the woven fabric falls under sub-heading 55.12 or 55.13.

3. Analytical methods and procedures :
   (1) Physical Form : The sample is a cut piece of woven fabric.
   (2) Microscopic Examination : Fibres of yarns, shows tubular as well as convolution structures.
   (3) Burning Test : The sample burn with cellulosic odour and forms bead.
   (4) Solubility : Partly soluble in H_2SO_4 (75 % W/W) and partly in methyl salicylate
   (5) Quantitative Analysis : Followed analytical procedure for binary mixtures of polyester fibres and cellulosic fibres (Ref. - ISO-1833 - 1977).

4. Results of analyses :
   (1) Physical Form : Woven fabric.
   (2) Microscopic Examination : Observed
   (3) Burning Test : Confirms the presence of cotton and synthetic fibres.
   (4) Solubility : Confirms the presence of polyester and cotton fibres.
   (5) Quantitative Analysis : Polyester fibres = 86.2 % (W/W) - Cotton fibres = balance (W/W)

5. Discussion and conclusion :
   The sample is woven fabric, composed of blended yarns of 86.2 % of synthetic staple fibres of polyester and rest being cotton.

6. Suggested HS Classification :
   Heading 55.12

1. Description of the product :
   Woolen Yarn containing 85 % or more by weight of wool.

2. Purpose of analyses :

   To confirm whether the yarn falls under sub-heading 5107.10 or 5107.20.

3. Analytical methods and procedures :
   (1) Physical Form : The sample is in the form of yarn.
   (2) Microscopic Examination : The fibres of yarn shows fairly cylindrical cuticle of overlapping scales and some fibres shows tubular structure.
   (3) Burning Test : The sample burns with proteinaceous odour and forms brittle bead.
   (4) Solubility : The sample is partly soluble in Dimethyl formamide and partly in 2 % KOH solution.
   (5) Quantitative Analysis : As per procedure given in ISO-1833 - 1977.

4. Results of analyses :
   (1) Physical Form : The sample is in the form of yarn.
   (2) Microscopic Examination : Characteristic structures of wool and synthetic fibres.
   (3) Burning Test : Confirms presence of wool and synthetic fibres.
   (4) Solubility : Confirms presence of wool and acrylic fibres.
   (5) Quantitative Analysis : Obtained wool Content : 85.8 % (W/W), balance synthetic staple fibres of acrylic.

5. Discussion and conclusion :
   The sample is in the form of blended spun yarn composed of wool = 85.8 % (W/W) and balance being synthetic acrylic fibres.

6. Suggested HS Classification :
   Subheading 5107.10.

1. Description of the product :
White circular tablets scored on one side. Each tablet is purported to contain Sulfadoxine USP 500 mg, Pyrimethamine USP 25 mg.

2. Purpose of analyses :

To confirm the composition of the product.

3. Analytical methods and procedures :
(1) Qualitative chemical tests*
Tests for Sulfadoxine
(i) Thoroughly mixed powdered tablets equivalent to 50 mg. of sulfadoxine with 10 ml of hydrochloric acid diluted to 10 percent filtered, to the 2 ml of filtrate add 0.5 ml of sodium nitrite solution and dil. hydrochloric acid, after 3 minutes add 0.2 ml of alpha naphthol solution; an orange red colour produced.
(ii) Thoroughly mixed powdered tablets equivalent to 50 mg of sulfadoxine with 3 ml of 0.1N sodium hydroxide solution, filtered, heat filtrate. On a water bath for 5 minutes, cool and add 1 ml of copper sulphate solution; a green yellow precipitate formed, turns blue.
Tests for Pyrimethamine
(i) Mixed powdered tablets equivalent to 100 mg of pyrimethamine with 30 ml of ammonia diluted to 10 percent, filtered, precipitated pyrimethamine washed with water and dissolved in 50 ml of acetone, evaporate to dryness to carry out following tests :
(a) Dissolve 50 mg of residue in 10 ml of dilute sulphuric acid by heating to 60 °C. Add 0.2 ml of potassium tetraiodomercurate solution; a creamy white precipitate forms.
(b) Incinerate remainder residue with 0.25 gm of anhydrous sodium carbonate, dissolve the incinerated residue in 10 ml of water neutrialise with dilute nitric acid, filter, filtrate answers positive test for chloride.
* (Ref. - Syndicate National de L'Industrie Pharmaceutique, France, Jan., 1990).
(2) Thin Layer Chromatography (ANNEX III).

4. Results of analyses :
(1) Qualitative Chemical Tests confirmed the presence of sulfadoxine and pyrimethamine in the product.
(2) Thin Layer Chromatography also confirmed the presence of Sulfadoxine and Pyrimethamine in the product.

5. Discussion and conclusion :
The product contains Sulfadoxine and Pyrimethamine.

6. Suggested HS Classification :
Subheading 3004.20.

1. Description of the product :
   Pale yellow liquid with characteristic odour, declared as Peppermint oil.

2. Purpose of analyses :

   To ascertain whether product is peppermint oil.

3. Analytical methods and procedures :
   (1) Determination of weight per ml.
   (2) Determination of refractive index.
   (3) Determination of optical rotation.
   (4) Thin layer chromatography of the product. (ANNEX IV).

4. Results of analyses :
   (1) Wt. per ml obtained, 0.904 g (reported between 0.900 and 0.916 g)
   (2) Refracted index obtained, 1.462 (reported between 1.460 and 1.467).
   (3) Optical rotation obtained, -18 (reported between -10 and -30).
   (4) Thin Layer Chromatography of product is similar to that of authentic peppermint oil.

5. Discussion and conclusion :
   The product is peppermint oil.

6. Suggested HS Classification :
   Subheading 3301.24.