IV. ENVIRONMENTAL DATA

Environmental Concentrations

Data on occupational environmental concentrations of sulfuric acid are very meager, possibly because the corrosive action of the acid on the skin and eyes is so commonly recognized from splash and spray that environmental levels have been overlooked. The few data that are available have generally been collected only on a single day; therefore, it is extremely difficult to relate the reported environmental information to actual conditions. At best, only rough estimates of environmental concentrations, especially ranges, can be made. Occupational environmental levels reported by Malcolm and Paul, [20] El-Sadik et al, [31] and Anfield and Warner [29] are as meaningful as could be obtained.

Malcolm and Paul's study [20] concerned dental erosion in workers. Reported acid mist concentrations in forming process areas to a mixture of dilute sulfuric acid (specific gravity 1.020 to 1.100) varied from 3.0 to 16.6 mg/cu m of air. Measurements were made on a dry day with low relative humidity. Forming tanks contained a foaming agent on the top of the acid which coalesced acid-containing gas bubbles, thus reducing acid mist escaping into the air. In addition, impervious sheets were used to cover the tanks which condensed the spray and permitted it to run back into the tanks. It was stated that the amount of acid present in the air on a cold humid day often exceeded 16 mg/cu m. In the charging process, the acid specific gravities were mostly about 1.265, higher than those in the forming process, and airborne acid levels varied from less than 0.8 to 2.5 mg/cu m. The method of analysis was not given; however, the standard error of the method was reported to be ±25%. These same environmental data were the only ones referred to in the epidemiologic study on dental erosion reported by ten Bruggen Cate [19] in 1968.

El-Sadik et al [31] reported environmental sulfuric acid concentrations, again in the manufacture of storage batteries. Concentrations ranged from 26.12 to 35.02 mg/cu m of air in 1 plant and 12.55 to 13.51 mg/cu m in another. No information was given as to what processes or what locations were involved, nor was temperature, humidity, particle size, etc. mentioned. Air samples were collected at 2 liters/minute in a bubbler containing sodium hydroxide absorbing solution and methyl red indicator. Twelve samples were collected daily at various times (unspecified) and analyzed for excess sodium hydroxide by standard acid titration. The method employed was a common acid-base titration and was not specific for sulfuric acid; however, acid exposures in battery manufacturing processes are almost exclusively due to sulfuric acid mist.

Anfield and Warner, [29] in response to the limited information available for sulfuric acid mist concentrations in industrial atmospheres, reported on environmental monitoring of sulfuric acid, sulfur dioxide, and ferrous sulfate in 5 industrial operations. The departments reported were Department A, a continuous sheet strip acid cleaning (pickling) operation of a large integrated steel plant; Department B, an acid recovery plant at the same steel facility; Department C, an acid cleaning (pickling) operation which treated small steel components for the automobile industry; and Departments D and E, lead-acid battery plate forming operations at 2 separate plants. A sampling train consisting of a filter head with filter, an impinger containing hydrogen peroxide absorbing solution, a gas meter, and a vacuum pump was used for sample collection. The air was sampled approximately 5 feet above floor level for periods varying from 1/2 hour up to several hours at flow rates up to 20 liters/minute. Because the sulfuric acid and particulate sulfate collected on the filter could not be determined separately, 2 samples were taken so that each substance could be analyzed separately. The sulfur dioxide passed through the filter and was absorbed in the hydrogen peroxide solution. The results of the sulfuric acid determinations are listed in Table X-4. Considerable ranges in acid levels occurred, reportedly due to different processes in various parts of the departments, changes in rates of production, and the effects of natural and forced ventilation systems. The importance of enclosure, ventilation, or a combination of these practices was emphasized. Department C, from a series of 85 samples around an open, unventilated tank, showed average sulfuric acid concentrations of approximately 3 mg/cu m. The 6 highest samples averaged more than 14 mg/cu m, and 49 samples exceeded 1 mg/cu m. The 15 lowest samples averaged 0.36 mg/cu m. Department A, also employing a pickling process, but utilizing enclosure and exhaust ventilation, showed only 2 of 48 samples which exceeded 1 mg/cu m, and the overall average concentration was 0.33 mg/cu m. The plate-forming operations at the 2 battery manufacturing plants, Departments D and E, showed average concentrations of 1.38 and 0.97 mg/cu m, respectively. In both installations, the operations were partly enclosed or a detergent was used to provide a frothing seal.

In a simple laboratory experiment conducted in an exhaust hood with a beaker of 15% w/v sulfuric acid solution and a filter sampler located "a few inches" above the beaker, Anfield and Warner [29] demonstrated that although heating and agitation of fluid with an air bubbler would increase airborne sulfuric acid concentrations, processes involving the evolution of hydrogen produced enormously high acid levels by comparison. Heating alone produced concentrations of 0.045 mg/cu m at 90 C, 4.1 mg/cu m with agitation at the same temperature, but with hydrogen bubble evolution, concentrations of 3.2 mg/cu m were noted at 20 C, 278.9 mg/cu m at 60 C, and complete collapse of the filter resulted at 90 C. The additional use of floating plastic balls to blanket the reaction in the 60 C range reduced sulfuric acid mist emanation by 50% (from 278.9 to 136.0 mg/cu m).

If exhaust ventilation is necessary for control of sulfuric acid mist, design principles which give useful guidelines are published for general industrial ventilation practices, [40] open-surface tanks, [41] and design and operation of local exhaust systems. [42]

Environmental Sampling and Analytical Method

Early collection methods for sulfuric acid in air involved the use of water or alkaline solutions in a scrubber or impinger followed by some form of acid-based titration. [1,43] Such methods also absorbed sulfate salts and possibly acid gases. Sulfate measurements using filtration or impaction techniques have been used in air pollution studies by titration for total acidity [13,44] or by a related procedure of sulfate analysis through the use of a barium sulfate turbidimetric determination. [45] The use of selective filters to separate sulfuric acid (with sulfates) from sulfur dioxide has also been reported. [46,47]

In 1969, Scaringelli and Rehme [47] reported a method for measuring sulfuric acid aerosol in microgram quantities which had application for community air measurements. The method successfully separated sulfuric acid from sulfur dioxide and other sulfates by filter collection with controlled temperature (400 C) in a nitrogen atmosphere, followed by conversion to sulfur dioxide with hot copper which could then by determined by spectrophotometric, coulometric, or flame photometric technics. The method, although satisfactory for sulfuric acid isolation, required controlled heat, a rather special setup of apparatus, and a zirconium oxide combustion tube, thus entailing a rather complicated preparation procedure prior to the analytical determination. Dubois et al [48] devised a microseparation of sulfuric acid from other airborne sulfates by the microdiffusion of sulfuric acid at 200 C into sodium hydroxide absorbing solution using glass petri dishes. Subsequently, following the preliminary separation, the isolated sulfuric acid was then measured by a method specific for sulfate. [49]

Because gravimetric determinations with sulfate were both time-consuming and tedious, and turbidimetric procedures were often unreliable and difficult to reproduce, direct titration methods for sulfate were devised which were rapid, accurate, and widely applicable. [49,50] Fritz and Freeland [50] in 1954 described the direct titration of sulfate in an alcoholic solution with barium chloride or barium perchlorate using Alizarin Red S or Thorin [o-(2-hydroxy-3,6-disulfo-1-naphthylazo)-benzenearsonic acid] as the indicator. A sharp, vivid color change from yellow to pink was described as marking the endpoint with results being as precise as gravimetric procedures and considerably faster. Later Fritz and Yamamura [49] in 1955 improved the method so as to be capable of determining very low concentrations of sulfate, as low as 10 ppm in water samples. The barium perchlorate titration method has been capable of measuring sulfuric acid concentrations at 0.1 mg/cu m (see Appendix I) and results from a micromethod have been reported in the range of 1 microgram/cu m of air. [48]

The filtration method accompanied by direct titration with barium perchlorate using Thorin as the indicator is the recommended compliance method as outlined in Appendix I. Metal ion interferences are eliminated by use of an ion exchange column and phosphate can be removed by precipitation with magnesium carbonate. If circumstances are such that airborne sulfate occurs, it can be separated [48] from sulfuric acid prior to the titration with barium perchlorate.

V. DEVELOPMENT OF STANDARD

Basis for Previous Standards

Cook, [51] in his comprehensive 1945 list of maximum allowable concentrations (MAC) of industrial atmospheric pollutants, cited 2 different values for sulfuric acid mist, 5 mg/cu m recommended by the Industrial Hygiene Division of the New York State Department of Labor and 2 mg/cu m recommended by the Industrial Hygiene Division of the Utah Department of Health. The documentation cited by Cook included references from Flury and Zernik's "Schadliche Gase,"[52] and one from Sterner [53] endorsing the 5 mg/cu m MAC. Cook also observed that individual human susceptibility differed widely, with the development of tolerance in workers habitually exposed.

In 1952, the American Conference of Governmental Industrial Hygienists (ACGIH) adopted 1.0 mg/cu m as their recommended Threshold Limit Value (TLV) for sulfuric acid mist. [54] This decision was based upon human experimental work reported by Amdur et al [16] in which it was found that concentrations below 1 mg/cu m could not be detected by odor, taste, or irritation by unacclimated persons. The threshold for odor and irritation was 1 mg/cu m in 2 persons and 3 mg/cu m in all subjects.

The ACGIH TLV has remained unchanged at 1.0 mg/cu m. In the latest documentation of TLV's, [55] the ACGIH Committee reviewed 8 published reports [16,20,21,23,33,34,35,37] from which the TLV of 1.0 mg/cu m was recommended to prevent irritation of respiratory passages and injury to the teeth.

In a report presented by the Czechoslovak Committee of MAC, [56] the majority of members of the Committee agreed on a MAC of 1 mg/cu m of air as a mean concentration and a peak MAC of 2 mg/cu m although it was recognized that slight irritation, though not necessarily unpleasant, might be experienced at 1 mg/cu m. The report of Amdur et al [16] was not sufficiently convincing to influence the opinion of the Committee at that time (1969). It was also commented that a great effort was necessary under the conditions of sulfuric acid production to adhere to their recommendations of 1 mg/cu m. The same standard exists in Soviet Russia, Hungary, and Poland. In the Federal Republic of Germany and the German Democratic Republic, the standard was listed as 13 mg/cu m and 10 mg/cu m, respectively.

The present federal standard for sulfuric acid is an 8-hour time-weighted average of 1 mg/cu m (29 CFR Part 1910.93 published in the Federal Register, volume 37, page 22139, dated October 18, 1972).

Basis for Recommended Environmental Standard

Although subjective responses such as throat tickling and scratching have been reported at sulfuric acid concentrations of less than 1 mg/cu m, [12,18] other investigators have reported no subjective responses until a level of 1 mg/cu m was reached. [16] Concentrations of about 5 mg/cu m may be very objectionable, usually causing cough, with marked alterations in respiration. [16] Overexposure to sulfuric acid by splash or spray has resulted in pulmonary edema and chronic pulmonary fibrosis, residual bronchiectasis, and pulmonary emphysema. [14]

Very few reports are available of occupational sulfuric acid concentrations especially reports from which exposure-effect relationships may be obtained. Environmental concentrations in the lead-acid battery industry have been reported ranging from 3 to 16.6 mg/cu m of air [20] in which relatively dilute sulfuric acid is used in forming processes. In addition, charging processes in the same industry have produced measured airborne sulfuric acid levels at about 0.8 to 2.5 mg/cu m. [20] Other studies have reported mean concentrations of 1.4 mg/cu m (range, 0.2 to 5.6 mg/cu m) [29] and findings ranging from 12.5 to as high as 35 mg/cu m. [31] These figures serve more to illustrate general circumstances for which many unknown variables exist rather than quantitative levels in attempts to derive exposure-effect relationships.

The published epidemiologic studies provide valuable information on the signs and symptoms resulting from occupational exposure to sulfuric acid. In studying sickness absence and ventilatory capacity in 461 lead-acid battery workers, Williams [28] concluded that an excess of spells of respiratory disease, especially bronchitis, occurred in forming process workers. Variation was noted in the number of spells of sickness absence which was contributed by different individuals; therefore, no tests of statistical significance were made. It was suggested that the increased number of spells of respiratory disease was due to an increased incidence of spells in attacked men rather than by an increased proportion of men attacked. It was also believed likely that one or more factors might have been present in the forming operation which was specifically associated with bronchitis and other respiratory disease. Interestingly, the same forming operation used for this study [28] was also the source of considerable dental erosion reported by Malcolm and Paul [20] in 1961. It was suggested by Williams [28] that the absence of lower respiratory tract disease observed in his study, where marked dental erosion had been earlier reported, might have been due to the large size of sulfuric acid mist particles or droplets, thus preventing their reaching the deep lung. Williams [28] stated that other operations similar to the one being reported showed median particle sizes of 14 m with only 4% of the particles being less than 4 μm in diameter. El-Sadik et al [31] found no significant difference in the prevalence of chronic bronchitis and/or chronic asthmatic bronchitis between 33 exposed workers and 20 controls; however, there was a greater group mean decrease in pulmonary function (FEV 1) in the exposed group than in the controls which the authors stated might be due to the inhalation of sulfuric acid mist. The consistent findings of dental erosion among sulfuric acid workers reported in 2 separate studies [19,20] indicates the problem to be one of definite health impairment. The consistent relationship that was observed [19] between the onset and advance of dental erosion and the length of employment emphasized this problem to be one of importance in the evaluation of sulfuric acid exposure. Progressive erosion among battery formation process workers was noted based on cases actually observed to advance under relatively modern (1964) industrial environmental conditions. Anfield and Warner [29] compared their environmental findings (see Table X-4) with those of ten Bruggen Cate [19] on tooth erosion incidence and stated that their 1.4 mg/cu m finding likely underestimated the dental risk to which workers had been exposed during their earlier years. A more realistic value of environmental acid concentration was suggested as being somewhere between 1.4 mg/cu m and that reported by Malcolm and Paul, 3 to 16.6 mg/cu m. [20] Even though dental erosion produced functional disability and disfigurement, [19] workers were able to compensate by using the canine teeth in place of the anterior teeth which could not be brought together. Because of the many uncertainties present in the reported environmental sulfuric acid levels, it is not possible to estimate an exposure level to sulfuric acid mist which would eliminate the occurence of dental etching and erosion.

Experimental studies have shown that sulfuric acid produces mucous membrane irritation and reflex bronchoconstriction with increased airway resistance. Most animal experimental work has been conducted in guinea pigs, considered to be the most sensitive of the standard laboratory animals to the respiratory effects of sulfuric acid. [33] At concentrations considered important for the evaluation of environmental standards, exposure of guinea pigs to 2 mg/cu m sulfuric acid for 1 hour produced increases in pulmonary airway resistance from reflex bronchoconstriction. [37] Bushtueva [36] found edema and thickening of the alveolar walls of guinea pigs also exposed to 2 mg/cu m, but for 5 days continuous exposure. Thomas et al [35] reported minor unspecified pathological changes for continuous exposure periods greater than 3 months. Similarly, continuous exposure of dogs to 0.9 mg/cu m sulfuric acid for 225 days produced decreased lung function as measured by diffusion capacity and changes in lung parenchyma. [39] However, effects produced by continuous exposure are difficult to evaluate in terms of intermittent exposures which are more representative of the occupational experience. In humans, Bushtueva [18] noted respiratory changes in amplitude and rhythm at sulfuric acid exposures of 1.8 to 2 mg/cu m. Slight changes at 1.0 to 1.1 mg/cu m, and no alterations in respiratory patterns, were noted at concentrations less than 1 mg/cu m. In another study, [24] measurements of sensory and central nervous responses from light sensitivity in the dark adapted eye or from reflex optical stimulation produced effects at 0.7 mg/cu m sulfuric acid. Both of these studies [18,24] are regarded as screening observations because results were observed in only 2 subjects. Furthermore, whether such optical changes, or for that matter, minimal respiratory changes represent undesirable effects is debatable. The increased respiratory effects reported by Amdur [16] to occur in subjects exposed to sulfuric acid concentrations as low as 0.35 mg/cu m are again uncertain as to their meaning. The findings were reported many years ago and have not been recently confirmed at the levels reported. In humans, a strong cortical influence exists to regulate respiration, and mechanical procedures, especially the use of a face mask, could influence the results markedly. In summary, the minimal changes reported in respiratory rate and on optical response to sulfuric acid concentrations below about 2 mg/cu m, remain unconvincing and unconfirmed.

The interaction of sulfuric acid with other gases and aerosols has been reported to produce effects on growth, lung changes, and respiration which were more marked than would have been predicted from either agent alone. [25] Bushtueva [24] found the effects of sulfuric acid combined with sulfur dioxide exposure to be merely additive as measured by reflex optical responses. In dogs, statistically significant reductions in mean diffusion capacity were also found between combinations of sulfuric acid and sulfur dioxide. [39] Although Toyama and Nakamura [27] reported increases in mean pulmonary airway resistance in humans to very low concentrations of sulfuric acid (0.01 to 0.1 mg/cu m), their method of producing sulfuric acid from the reaction between hydrogen peroxide and sulfur dioxide indicated a combination effect between the 2 substances rather than to sulfuric acid alone. Although sulfuric acid was identified, no data were given concerning the amount of unreacted sulfur dioxide or hydrogen peroxide which was present with the sulfuric acid. Other investigations of particle (droplet) size, [33,34,35,37] as well as temperature effects [29] and humidity, [17] emphasize a strong interplay between these factors, thus making interpretations of exposure-effect extremely difficult. The problems encountered in interpreting results from a combination of only 2 substances emphasizes the difficulty encountered when considering particle size, temperature, humidity, and multiple substance interrelationships.

It is concluded that the existing federal standard of 1 ppm TWA should be retained. It is believed that adherence to the present environmental federal standard in conjunction with a strong program of work practices to prevent skin and eye contact from sulfuric acid will prevent the irritant effects of sulfuric acid in workers.

VI. WORK PRACTICES

The corrosive, oxidizing, and sulfonating properties of sulfuric acid are such as to require that it be handled at all times with proper care. The work practices specified in the recommended standard are primarily for the purpose of preventing or minimizing sulfuric acid contact with the respiratory tract, skin, or eyes. These practices incorporate basic principles described in standard guides such as the Chemical Safety Data Sheet SD-20, [3] and the Chemical Hazards Bulletin on Sulfuric Acid. [8]

Sulfuric acid itself is not flammable; however, it can cause ignition when in contact with other combustible materials. It reacts with some metals to release hydrogen gas, which is potentially explosive. As the gas is released from the liquid surface, it may also entrain acid droplets which may be inhaled or burn the skin. Released hydrogen will also react with arsenic, selenium, or cyanides which may be present as impurities either in the acid or in metals, producing highly toxic arsine, hydrogen selenide, or hydrogen cyanide.

Sulfuric acid generates heat when mixed with water. Adding water to the acid can be extremely dangerous; therefore, when mixing is necessary, the acid should be added to the water; in special cases when water must be added to acid, suitable precautions should be taken.

Workers should not expose themselves unnecessarily to sulfuric acid mist or fumes. Because of its irritant properties, an individual is usually conscious of the presence of acid mist in the environment, especially when the acid occurs at high concentrations. Should a situation arise where it is essential to remain in an environment where high airborne acid levels exist, such as for repairs or in an emergency, the individual should be especially aware of the need for proper protective equipment. This should include impervious clothing, gloves, rubber shoes, goggles, face shields, and respiratory devices as appropriate to prevent acid contact with the skin, eyes, or respiratory tract.

Should an emergency make it necessary to enter a tank or closed space, reliance should never be placed on a canister-type gas mask. Only self-contained breathing apparatus in pressure-demand mode or a combination supplied air respirator, pressure-demand type, with auxiliary self-contained air supply should be used in such situations.

Employees should be trained at appropriate regular intervals in the proper techniques for handling, moving, and emptying carboys, drums, tank trucks, railroad cars, and barges of sulfuric acid. They should also be trained in the emergency procedures to be followed in case of accidents involving sulfuric acid.

All of the prescribed practices apply to oleum, which is a more hazardous form of sulfuric acid.

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