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VIII. APPENDIX I
METHOD FOR SAMPLING NITRIC ACID

Introduction

The recommended sampling method employs a 25-ml midjet impinger filled with distilled water sorbent. Concentrations of airborne nitric acid are determined by measuring the ultraviolet absorption at 220 nm by means of an ultraviolet spectrophotometer. The National Institute for Occupational Safety and Health (NIOSH) is currently evaluating the collection efficiency and recovery of a number of filters including a Gelman Type A (glass-fiber filter) or equivalent for use in sampling airborne nitric acid. It was believed that the filter collection device would be superior to the impinger because the former would have greater durability in the field and lower interference from ambient oxides of nitrogen. However, preliminary findings indicate that 4 filters tested thus far have poorer collection efficiency and reliability than the impinger method. However, results are incomplete at this time. A final report will be published in a future edition of the NIOSH Manual of Analytical Methods.

General Requirements

In order to evaluate compliance with the standard, half-hour breathing zone samples, representative of the individual worker's exposure, shall be collected. The sample data sheet shall include:

- (a) Date and time of sample.
- (b) Sample duration.
- (c) Volumetric flow rate of sampling.
- (d) Description of sampling location.
- (e) Other pertinent information.

Air Sampling Requirements

(a) Half-hour breathing zone samples representative of the worker's exposure shall be collected to characterize the exposure from each job or specific operation in each production area.

(b) Samples shall be collected using personal sampling pumps and a 25-ml midjet impinger containing 20 ml of doubly distilled water sorbent. A teflon baffle should be installed on the impinger stem at the 25-ml level.

(c) The sample shall be taken at a flow rate of 2.8 liters/minute (lpm). A sample size of 200 liters is required.

(d) A sufficient number of samples shall be taken to allow determination of compliance or noncompliance with the recommended limit at the 95% confidence level. Determination of compliance or noncompliance given the sampling accuracy, analytic accuracy, and type of sampling (eg, full-period consecutive sampling, partial-period consecutive sampling), has been described by NIOSH. [64]

(e) Blank impingers, filled from the same distilled water source as the used bubblers, shall be carried to the sampling site to provide a background correction which must be applied to the analytical results. The number of blank impingers required shall be as follows: 3 for 20 or fewer

samples, and 1 for each additional 10 samples.

Sample Procedure

(a) Connect the impinger to the pump with a piece of flexible tubing. If personal sampling is being done, the midget impinger is placed in a midget-impinger holder and fastened to the worker in his breathing zone.

(b) Turn on the pump and set the flow rate to 2.8 liters/minute according to the manufacturer's directions. Periodically check the flow rate and readjust as needed. Terminate sampling if unable to readjust. In addition, check the level of solution in the impinger. If less than 5 ml of solution remains in the impinger, fill the impinger back up to the 20-ml mark.

(c) After sampling, quantitatively transfer the impinger solution to the glass vial. Use 1 or 2 ml of solution to wash the impinger. Transfer the wash to a glass vial. Tightly screw on the cap and seal with vinyl tape.

(d) Record ambient temperature and pressure. If ambient pressure is unavailable, record the elevation.

(e) Use one impinger as a blank. Handle it the same way as any other impinger but do not draw air through it. Label this solution as a blank.

Interferences

The following substances will interfere with NO₃ analysis: nitrite, dissolved organic matter, hexavalent chromium, surfactants, suspended

particles, hydroxide, and carbonate.

Shipping

Ship in a container designed to prevent damage in transit.

Calibration of Sampling Trains

Accurate calibration of sampling pumps is essential for correct interpretation of the measured concentration. Sampling pumps should be initially calibrated or recalibrated if subjected to misuse, repair, or frequent hard usage.

Ordinarily, pumps should be calibrated in the laboratory both before and after they are used to collect large numbers of field samples. The accuracy of the calibration is dependent on the quality of the standard reference instrument. For laboratory calibration, a 1-liter buret is recommended. Other standard calibrating instruments can be used for field calibration.

XI. APPENDIX II

ANALYTICAL METHOD FOR NITRIC ACID

The recommended analytical method is based on an ultraviolet spectrophotometric procedure described by the American Public Health Association, the American Water Works Association, and the Water Pollution Control Federation. [50]

Principle of the Method

Measurement of the ultraviolet absorption at 220 nm enables a rapid means of determining nitrate. The nitrate calibration curve follows Beer's law up to 15.5 $\mu\text{g NO}_3/\text{ml}$ nitrogen. Because dissolved organic matter may also absorb at 220 nm and nitrate does not absorb at 275 nm, a second measurement is made at 275 nm to correct the nitrate value. The extent of this empirical correction is related to the nature and concentration of the organic matter.

Interferences

Dissolved organic matter, suspended particles, hydroxide, carbonate, nitrite, chromium(VI), and surfactants interfere. The latter three substances may be compensated for by the preparation of individual correction curves.

All glassware must be thoroughly cleaned and rinsed in order to reduce the errors which might result from streaks or particles on the outside of the cuvettes, as well as traces of surfactants or dichromate cleaning solution which might adhere on the interior glass surfaces.

The ultraviolet method yields best results in the presence of very low color interference and appreciable nitrate content. Colored samples should accordingly be treated with aluminum hydroxide, or diluted to minimize color interference.

Apparatus

(a) Spectrophotometer: A spectrophotometer for use at 220 nm and 275 nm with matched silica cells having 1-cm or longer light path. A Beckman Model DU spectrophotometer with a photomultiplier attachment and hydrogen lamp source, or equivalent.

(b) Impinger: A midget impinger, 25-ml size, containing 20 ml of redistilled water.

Reagents

(a) Redistilled water: Use redistilled water for the preparation of all solutions and dilutions.

(b) Standard nitrate solution: Dissolve 850.0 mg of anhydrous sodium nitrate and dilute to 1000 ml; 1.00 ml = 620 μ g NO₃.

(c) Aluminum hydroxide suspension: Dissolve 125 g of aluminum potassium sulfate or ammonium sulfate redistilled water. Warm to 60C and add 55 ml concentrated NH₄OH slowly, with stirring. After 1 hour, transfer to a large bottle and wash the precipitate by successive additions (with thorough mixing) and decantations of distilled water until free from ammonia, chloride, nitrate, and nitrite. Finally, after settling, decant off as much clear liquid as possible, leaving only the concentrated suspension.

Analysis Procedure

(a) Midget impinger: Absorbance can be measured directly from the solution. However, if the solution has a high color or contains organic substance, decant the solution into a 15 ml graduated centrifuge tube and add 0.5 ml aluminum hydroxide suspension into the sample. Cap the centrifuge tube with a parafilm strip and mix thoroughly. Centrifuge at 2,000 rpm for 5 minutes.

(b) Preparation of standard curve: Prepare nitrate calibration standards in the range 0-15.5 $\mu\text{g NO}_3/\text{ml}$ by diluting to 50 ml the following volumes of the standard nitrate solution: 0, 0.05, 0.1, 0.2....1.25 ml. Treat the nitrate standards in the same manner as the samples.

(c) Spectrophotometric measurement: Read the absorbance or transmittance against redistilled water set at zero absorbance or 100% transmittance. Use a wavelength of 220 nm to obtain the nitrate reading and a wavelength of 275 nm to obtain the interference due to dissolved organic matter.

(d) Preparation of correction curves for nitrite, chromium(VI) and surfactants: When nitrite, chromium(VI) and anionic surfactants are known to be present in the sample, prepare correction curves for each of these substances at 2 mg/liter intervals up to 10 mg/liter. Use potassium nitrite, potassium dichromate, and linear alkylate sulfonate, with redistilled water for this purpose. Measure the absorbances given by each substance at a wavelength of 220 nm against redistilled water and plot a separate curve for each of these interfering materials.

Calculations

(a) Correction for dissolved organic matter: Subtract 2 times the reading at 275 nm from the reading at 220 nm to obtain the absorbance due to nitrate. Convert this absorbance value into equivalent nitrate by reading the nitrate value from a standard calibration curve obtained at 220 nm.

(b) Correction for nitrite, hexavalent chromium, or surfactants: Deduct the equivalent nitrate values for each of these interfering substances from the gross nitrate result.

(c) Calculations: Plot absorbance against concentration, and obtain the concentration from the standard curve.

$$\mu\text{g HNO}_3 = (\mu\text{g/ml from the curve}) \times (\text{sample vol}) \times (1.016)$$

Convert the volume of air sampled to standard conditions of 25(C) and 760 mmHg.

$$V_s = V \times \frac{P}{760} \times \frac{298}{T+273}$$

V_s = volume of air in liters at 25 C and 760 mmHg.

V = volume of air in liters as measured.

P = barometric pressure in mmHg.

T = temperature of air in centigrade.

$$\frac{\text{mg HNO}_3}{\text{cu m air}} = \frac{\mu\text{g HNO}_3}{V_s}$$

X. APPENDIX III - MATERIAL SAFETY DATA SHEET

The following items of information which are applicable to a specific product or material shall be provided in the appropriate block of the Material Safety Data Sheet (MSDS).

The product designation is inserted in the block in the upper left corner of the first page to facilitate filing and retrieval. Print in upper case letters as large as possible. It should be printed to read upright with the sheet turned sideways. The product designation is that name or code designation which appears on the label, or by which the product is sold or known by employees. The relative numerical hazard ratings and key statements are those determined by the procedures described in Chapter V, Part B, of the NIOSH publication, An Identification System for Occupationally Hazardous Materials. [52] The company identification may be printed in the upper right corner if desired.

(a) Section I. Product Identification

The manufacturer's name, address, and regular and emergency telephone numbers (including area code) are inserted in the appropriate blocks of Section I. The company listed should be a source of detailed backup information on the hazards of the material(s) covered by the MSDS. The listing of suppliers or wholesale distributors is discouraged. The trade name should be the product designation or common name associated with the material. The synonyms are those commonly used for the product, especially formal chemical nomenclature. Every known chemical designation or competitor's trade name need not be listed.

(b) Section II. Hazardous Ingredients

The "materials" listed in Section II shall be those substances which are part of the hazardous product covered by the MSDS and individually meet any of the criteria defining a hazardous material. Thus, one component of a multicomponent product might be listed because of its toxicity, another component because of its flammability, while a third component could be included both for its toxicity and its reactivity. Note that a MSDS for a single component product must have the name of the material repeated in this section to avoid giving the impression that there are no hazardous ingredients.

Chemical substances should be listed according to their complete name derived from a recognized system of nomenclature. Where possible, avoid using common names and general class names such as "aromatic amine," "safety solvent," or "aliphatic hydrocarbon" when the specific name is known.

The "%" may be the approximate percentage by weight or volume (indicate basis) which each hazardous ingredient of the mixture bears to the whole mixture. This may be indicated as a range or maximum amount, ie, "10-40% vol" or "10% max wt" to avoid disclosure of trade secrets.

Toxic hazard data shall be stated in terms of concentration, mode of exposure or test, and animal used, ie, "100 mg/kg LD50-oral-rat," "25 mg/kilo LD50-skin-rabbit," "75 ppm LC man," or "permissible exposure from 29 CFR 1910.1000," or if not available, from other sources of publications such as the American Conference of Governmental Industrial Hygienists or the American National Standards Institute Inc. Flammability or reactivity data could be flash point, shock sensitivity, or other brief data

indicating nature of the hazard.

(c) Section III. Physical Data

The data in Section III should be for the total mixture and should include the boiling point and melting point in degrees Fahrenheit (Celsius in parentheses); vapor pressure, in conventional millimeters of mercury (mmHg); vapor density of gas or vapor (air = 1); solubility in water, in parts/hundred parts of water by weight; specific gravity (water = 1); percent volatiles (indicate if by weight or volume) at 70 F (21.1 C); evaporation rate for liquids or sublimable solids, relative to butyl acetate; and appearance and odor. These data are useful for the control of toxic substances. Boiling point, vapor density, percent volatiles, vapor pressure, and evaporation data are useful for designing proper ventilation equipment. This information is also useful for design and deployment of adequate fire and spill containment equipment. The appearance and odor may facilitate identification of substances stored in improperly marked containers, or when spilled.

(d) Section IV. Fire and Explosion Data

Section IV should contain complete fire and explosion data for the product, including flash point and autoignition temperature in degrees Fahrenheit (Centigrade in parentheses); flammable limits, in percent by volume in air; suitable extinguishing media or materials; special firefighting procedures; and unusual fire and explosion hazard information. If the product presents no fire hazard, insert "NO FIRE HAZARD" on the line labeled "Extinguishing Media."

(e) Section V. Health Hazard Data

The "Health Hazard Data" should be a combined estimate of the hazard of the total product. This can be expressed as a TWA concentration, as a

permissible exposure, or by some other indication of an acceptable standard. Other data are acceptable, such as lowest LD50, if multiple components are involved.

Under "Routes of Exposure," comments in each category should reflect the potential hazard from absorption by the route in question. Comments should indicate the severity of the effect and the basis for the statement if possible. The basis might be animal studies, analogy with similar products, or human experiences. Comments such as "yes" or "possible" are not helpful. Typical comments might be:

Skin Contact--single short contact with concentrated acid is painful and burns the skin.

Eye Contact--painful, opacification of cornea, may result in blindness.

"Emergency and First Aid Procedures" should be written in lay language and should primarily represent first aid treatment that could be provided by paramedical personnel or individuals trained in first aid.

Information in the "Notes to Physician" section should include any special medical information which would be of assistance to an attending physician including required or recommended replacement and periodic medical examinations, diagnostic procedures, and medical management of overexposed workers.

(f) Section VI. Reactivity Data

The comments in Section VI relate to safe storage and handling of hazardous, unstable substances. It is particularly important to highlight instability or incompatibility with common substances or circumstances such as water, direct sunlight, steel or copper piping, acids, alkalies, etc.

"Hazardous Decomposition Products" shall include those products released as a result of fire. It must also include dangerous products produced by aging, such as peroxides in the case of some ethers. Where applicable, shelf life should also be indicated.

(g) Section VII. Spill or Leak Procedures

Detailed procedures for cleanup and disposal should be listed with emphasis on precautions to be taken to protect workers assigned to cleanup detail. Specific neutralizing chemicals or procedures should be described in detail. Disposal methods should be explicit including proper labeling of containers holding residues and ultimate disposal methods such as "sanitary landfill," or "incineration." Warnings such as "comply with local, state, and federal antipollution ordinances" are proper but not sufficient. Specific procedures shall be identified.

(h) Section VIII. Special Protection Information

Section VIII requires specific information. Statements such as "Yes," "No," or "If necessary" are not informative. Ventilation requirements should be specific as to type and preferred methods. Respirators shall be specified as to type and NIOSH or US Bureau of Mines approval class, ie, "Supplied air," "Organic vapor canister," "Suitable for dusts not more toxic than lead," etc. Protective equipment must be specified as to type and materials of construction.

(i) Section IX. Special Precautions

"Precautionary Statements" shall consist of the label statements selected for use on the container or placard. Additional information on any aspect of safety or health not covered in other sections should be inserted in Section IX. The lower block can contain references to published guides or in-house procedures for handling and storage.

Department of Transportation markings and classifications and other freight, handling, or storage requirements and environmental controls can be noted.

(j) Signature and Filing

Finally, the name and address of the responsible person who completed the MSDS and the date of completion are entered. This will facilitate correction of errors and identify a source of additional information.

The MSDS shall be filed in a location readily accessible to workers potentially exposed to the hazardous material. The MSDS can be used as a training aid and basis for discussion during safety meetings and training of new employees. It should assist management by directing attention to the need for specific control engineering, work practices, and protective measures to ensure safe handling and use of the material. It will aid the safety and health staff in planning a safe and healthful work environment and suggesting appropriate emergency procedures and sources of help in the event of harmful exposure of employees.

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MATERIAL SAFETY DATA SHEET

I PRODUCT IDENTIFICATION		
MANUFACTURER'S NAME	REGULAR TELEPHONE NO. EMERGENCY TELEPHONE NO.	
ADDRESS		
TRADE NAME		
SYNONYMS		
II HAZARDOUS INGREDIENTS		
MATERIAL OR COMPONENT	%	HAZARD DATA
III PHYSICAL DATA		
BOILING POINT, 760 MM HG		MELTING POINT
SPECIFIC GRAVITY (H ₂ O=1)		VAPOR PRESSURE
VAPOR DENSITY (AIR=1)		SOLUBILITY IN H ₂ O, % BY WT
% VOLATILES BY VOL.		EVAPORATION RATE (BUTYL ACETATE=1)
APPEARANCE AND ODOR		

IV FIRE AND EXPLOSION DATA				
FLASH POINT (TEST METHOD)		AUTOIGNITION TEMPERATURE		
FLAMMABLE LIMITS IN AIR, % BY VOL.	LOWER		UPPER	
EXTINGUISHING MEDIA				
SPECIAL FIRE FIGHTING PROCEDURES				
UNUSUAL FIRE AND EXPLOSION HAZARD				
V HEALTH HAZARD INFORMATION				
HEALTH HAZARD DATA				
ROUTES OF EXPOSURE				
INHALATION				
SKIN CONTACT				
SKIN ABSORPTION				
EYE CONTACT				
INGESTION				
EFFECTS OF OVEREXPOSURE				
ACUTE OVEREXPOSURE				
CHRONIC OVEREXPOSURE				
EMERGENCY AND FIRST AID PROCEDURES				
EYES				
SKIN				
INHALATION				
INGESTION				
NOTES TO PHYSICIAN				

VI REACTIVITY DATA	
CONDITIONS CONTRIBUTING TO INSTABILITY	
INCOMPATIBILITY	
HAZARDOUS DECOMPOSITION PRODUCTS	
CONDITIONS CONTRIBUTING TO HAZARDOUS POLYMERIZATION	
VII SPILL OR LEAK PROCEDURES	
STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED	
NEUTRALIZING CHEMICALS	
WASTE DISPOSAL METHOD	
VIII SPECIAL PROTECTION INFORMATION	
VENTILATION REQUIREMENTS	
SPECIFIC PERSONAL PROTECTIVE EQUIPMENT	
RESPIRATORY (SPECIFY IN DETAIL)	
EYE	
GLOVES	
OTHER CLOTHING AND EQUIPMENT	

IX SPECIAL PRECAUTIONS

PRECAUTIONARY
STATEMENTS

OTHER HANDLING AND
STORAGE REQUIREMENTS

PREPARED BY _____

ADDRESS _____

DATE _____

XI. TABLES

TABLE XI-1

PHYSICAL DATA ON CHEMICALLY PURE NITRIC ACID

%HNO ₃	Sp.Gr.at 15.5 C(60 F)	Melting Points	
		C	F
52.30	1.3303	-19.5	-3
56.52	1.3551	-20.0	-4
61.38	1.3810	-24.5	-12
67.18	1.4078	-33.0	-27
70.33	1.4216	-42.5	-45
80.04	1.4610	-39.0	-38
85.70*	1.4796	-47.0	-53
95.11	1.5026	-52.0	-62

* Considered "fuming acid" above this strength. "Red fuming acid" is high strength nitric acid containing varying percentages of oxides of nitrogen in solution at normal temperatures. At elevated temperatures all nitric acid will give off gaseous oxides of nitrogen (commonly called "nitrous fumes").

From reference 7

TABLE XI-2
 PROPERTIES AND CHARACTERISTICS OF NITRIC ACID

	100%	Aqueous Solution
Physical state	Liquid	Liquid
Explosive limits	Nonflammable	Nonflammable
Boiling point	86 C (186.9 F)	Constant boiling mixture 68 to 68.5% HNO ₃ 121.6 C (251 F)
Color	Colorless	Colorless to light brown
Corrosivity	Will vigorously attack most metals	
Hygroscopicity	Yes	Yes
Light sensitivity	Water white acid becomes amber to brown depending upon strength of acid and exposure	
Odor	HNO ₃ vapors acrid--Oxides of nitrogen sweet to acrid	
Reactivity	Will react readily with most chemicals. Strong nitric in contact with wood and some other organic materials may cause fire	

From reference 7

TABLE XI-3
VAPOR PRESSURES OF HNO3 AND NO2

HNO3		NO2	
Temp, C	Vp, mm Hg	Temp, C	Vp, mm Hg
20	42	21.3	760
30	77	30	1125
40	133	40	1733
50	215	50	2599
60	230	60	3823

From Gray et al 8

TABLE XI-4

POTENTIAL OCCUPATIONAL EXPOSURES TO NITRIC ACID

Aircraft workers	Lithographers
Ammonium nitrate makers	Mirror makers
Bleachers	Nitration workers
Brass cleaners	Nitric acid workers
Bright-dip workers	Nitrobenzene makers
Cellulose nitrate makers	Nitro-compound workers
Drugmakers	Ore flotation workers
Dyemakers	Organic chemical synthesizers
Electroplaters	Photoengravers
Etchers	Rocket fuel handlers
Jewelers	Steel etchers
Laboratory workers, chemical	Sulfuric acid makers

From reference 12

TABLE XI-5

EXPOSURE OF MALE RATS TO NITRIC ACID FUMES AND NITROGEN DIOXIDE

Chemical Agent	No. of Runs	LC50 in ppm NO2 (Total Concentration)
Red Fuming Nitric Acid	9	138 (310)*
Nitrogen Dioxide	10	174 (174)
White Fuming Nitric Acid (WFNA)	16	244 (334)*

*Total concentration computed by NIOSH. Based upon molecular weights and the percentage of nitrogen dioxide in white and red fuming nitric acid.

Adapted from Gray et al [17]

DEPARTMENT OF
HEALTH, EDUCATION, AND WELFARE
PUBLIC HEALTH SERVICE
CENTER FOR DISEASE CONTROL
NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH
ROBERT A. TAFT LABORATORIES
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