VIII. APPENDIX I

METHOD FOR SAMPLING AMMONIA IN AIR

General Requirements

Ammonia concentrations shall be determined within the worker's breathing zone and shall meet the following criteria in order to evaluate conformance with the recommended standard:

(a) Samples collected shall be representative of the individual worker's exposure.

(b) Sampling data sheets shall include a log of

(1) The date and time of sample collection

(2) Sampling duration

(3) Volumetric flowrate of sampling

(4) A description of the sampling location

(5) Other pertinent information

Equipment for Air Sampling

(a) Stopwatch

(b) Vacuum pumps with calibrated rotameter

(c) Midget impingers

(d) Solid Standard Taper 24/40 stoppers

(e) Polyvinyl tubing

(f) Dispensing glassware and supporting equipment as may be deemed necessary. Glassware should be borosilicate quality, washed thoroughly with detergent, and followed by rinses with tap water and distilled water.

Reagents

Cautiously add 56 ml of concentrated reagent grade sulfuric acid to 500 ml of distilled water; dilute to 1 liter to obtain 2 N sulfuric acid. Cool to room temperature and dilute 50 ml of the 2 N sulfuric acid to 1 liter with distilled water to obtain 0.1 N sulfuric acid absorbing solution.

Breathing Zone Sampling

Breathing zone samples shall be collected as near as practicable to the worker's face without interfering with his freedom of movement and shall characterize the exposure from each job or specific operation in each production area.

(a) Sampling Equipment

A calibrated personal sampling pump with flowmeter (range up to 2 liters a minute), a midget impinger containing 10 ml of 0.1 N sulfuric acid absorbing solution.

(b) Sampling Procedure

The impinger outlet is connected to the personal sampling pump inlet by a piece of tubing of convenient length, but not in excess of 3 feet. The impinger assembly is attached to the worker's clothing in order to sample from the worker's breathing zone. The sample is collected at a rate of 1 liter a minute for 5 minutes.

A minimum of 3 samples shall be taken for each operation (more samples if the concentrations are close to the recommended standard). At least one blank impinger shall be provided containing sulfuric acid

absorbing solution through which no air has been sampled. One additional blank impinger shall be supplied with every 10 samples obtained.

Shipping

After sampling, remove the glass stopper and impinger stem from the impinger bottle. Tap the stem gently against the inside wall of the impinger bottle to recover as much of the sampling solution as possible. Wash the stem with a small amount of unused absorbing solution from a wash bottle, adding the wash to the impinger to bring to a final volume of 20 ml. Stopper the impinger tightly with plastic caps (do not seal with rubber), place in an upright position, and ship the impinger samples to the analytical laboratory in a suitable container to prevent damage in transit. Special impinger shipping containers designed by NIOSH are available. Be certain that the impinger bottles are sealed very tightly to prevent leakage and loss of samples.

Calibration of Sampling Trains

Since the accuracy of an analysis can be no greater than the accuracy of the volume of air which is measured, the accurate calibration of a sampling pump is essential to the correct interpretation of the volume indicated. The frequency of calibration is dependent on the use, care, and handling to which the pump is subjected. In addition, pumps should be recalibrated if they have been misused or if they have just been repaired or received from a manufacturer. If the pump receives hard usage, more frequent calibration may be necessary.

Ordinarily, pumps should be calibrated in the laboratory both before they are used in the field and after they have been used to collect a large number of field samples. The accuracy of calibration is dependent on the type of instrument used as a reference. The choice of calibration instrument will depend largely upon where the calibration is to be performed. For laboratory testing, primary standards such as a spirometer or soapbubble meter are recommended, although other standard calibrating instruments such as a wet test meter or dry gas meter can be used. The actual setups will be similar for all instruments.

Instructions for calibration with the soapbubble meter follow. If another calibration device is selected, equivalent procedures should be used. The calibration setup for personal sampling pumps with a midget impinger is shown in Figure XI-1.

(a) Check the voltage of the pump battery with a voltmeter to assure adequate voltage for calibration. Charge the battery if necessary.

(b) Fill the impinger with 10 ml of the absorbing solution.

(c) Assemble the sampling train as shown in Figure XI-1.

(d) Turn the pump on and moisten the inside of the soapbubble meter by immersing the buret in the soap solution and draw bubbles up the inside until they are able to travel the entire buret length without bursting.

(e) Adjust the pump rotameter to provide a flowrate of 1 liter a minute.

(f) Check the water manometer to insure that the pressure drop across the sampling train does not exceed 13 inches of water (approximately 1 inch of mercury).

(g) Start a soapbubble up the buret and, with a stopwatch, measure the time it takes for the bubble to move from one calibration mark to another. For a 1,000-ml buret, a convenient calibration volume is 500 ml.

(h) Repeat the procedure in (g) above at least twice, average the results, and calculate the flowrate by dividing the volume between the preselected marks by the time required for the soapbubble to traverse the distance.

(i) Data for the calibration include the volume measured, elapsed time, pressure drop, air temperature, atmospheric pressure, serial number of the pump, date, and name of the person performing the calibration.

IX. APPENDIX II

METHOD FOR ANALYSIS OF AIR SAMPLES

Equipment

(a) Spectrophotometer

(b) 50-ml beakers

(c) 1 each 1-, 2-, 5-, 10-, 20-ml pipets

(d) 1-cm cuvettes

(e) 1-liter volumetric flasks

Reagents

(a) Nessler Reagent

Dissolve 100 g of mercury (II) iodide and 70 g of potassium iodide in a small quantity of water, and add this mixture slowly, with stirring, to a cool solution (15-20 C) of 160 g sodium hydroxide in 500 ml water. Dilute to 1 liter. Store in rubber-stoppered borosilicate glassware and out of sunlight to maintain reagent stability for periods up to a year under normal laboratory conditions.

(b) Ammonium chloride, stock solution (1.0 ml = 1.0 mg ammonia)

Dissolve 3.141 g of ammonium chloride in water and dilute to 1 liter with distilled, ammonia-free water.

(c) Ammonium chloride, standard solution (1.0 ml = 10.0 μg ammonia)

Dilute 10.0 ml of 1.0 mg ammonia/ml stock solution to 1000 ml with distilled, ammonia-free water.

Preparation of Standard Curve

To a series of 50-ml beakers add 25.0, 23.0, 20.0, 17.0, and 13.0 ml of absorbing solution (Appendix I). To these beakers in the same order add 0.0, 2.0, 5.0, 8.0, and 12.0 ml of ammonium chloride standard solution, so that the final volume is 25 ml. Add 2.0 ml of Nessler reagent, mix well, and cover beakers. After 20 minutes the color intensity is measured spectrophotometrically in a 1-cm cell at 425 nm against the reagent blank.

Analysis of Samples

Samples should be analyzed within a week of collection. Transfer an aliquot of the blank and sample solutions to 50-ml beakers. Add absorbing solution to adjust the final volume to 25 ml in each beaker. Add 2.0 ml of Nessler reagent, mix well, and cover beakers. After 20 minutes the color intensity is determined by the same procedure as the standards.

Calculations

From the standard curve determine the micrograms of ammonia in the sample solution aliquot. Calculate the concentration of ammonia in the air as ppm:

$$\text{ppm ammonia} = \frac{1.44\ \mu l\ NH_3}{\mu g\ NH_3} \times \frac{AB}{CDE}$$

Where

$$\frac{1.44\ \mu l\ NH_3}{\mu g\ NH_3} = \frac{\mu mole}{17\ \mu g\ NH_3} \times \frac{22.4\ \mu l}{\mu mole} \times \frac{298\ K}{273\ K}$$

A = Sample solution volume in milliliters = 20 ml

(10 ml scrubber solution plus 10 ml rinse)

B = μg of ammonia in aliquot analyzed

C = Volume of aliquot in milliliters

D = Time of sampling period in minutes

E = Sampling impinger flow rate in liters/min.

X. APPENDIX III

MATERIAL SAFETY DATA SHEET

The following items of information which are applicable to a specific product or material containing ammonia shall be provided in the appropriate section of the Material Safety Data Sheet or other approved form. If a specific item of information is inapplicable (eg, flash point), the initials "na" (not applicable) should be inserted.

(a) Section I. Source and Nomenclature.

(1) The name, address, and telephone number of the manufacturer or supplier of the product.

(2) The trade name and synonyms for a mixture of chemicals, a basic structural material, or for a process material; and the trade name and synonyms, chemical name and synonyms, chemical family, and formula for a single chemical.

(b) Section II. Hazardous Ingredients.

(1) Chemical or widely recognized common name of all hazardous ingredients.

(2) 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, eg, 10-20% V; 10% max. W.

(3) Basis for toxicity for each hazardous material such as an established standard, in appropriate units.

(c) Section III. Physical Data.

Physical properties of the total product including boiling point and melting point in degrees Fahrenheit; vapor pressure, in millimeters of

mercury, vapor density of gas or vapor (air = 1), solubility in water in parts per hundred parts of water by weight; specific gravity (water = 1); percent volatile, indicate if by weight or volume, at 70 Fahrenheit; evaporation rate for liquids (indicate whether butyl acetate or ether = 1); and appearance and odor.

(d) Section IV. Fire and Explosion Hazard Data.

Fire and explosion hazard data about a single chemical or a mixture of chemicals, including flash point, in degrees Fahrenheit; flammable limits, in percent by volume in air; suitable extinguishing media or agents; special fire fighting procedures; and unusual fire and explosion hazard information.

(e) Section V. Health Hazard Data.

Toxic level for total compound or mixture, effects of exposure, and emergency and first aid procedures.

(f) Section VI. Reactivity Data.

Chemical stability, incompatibility, hazardous decomposition products, and hazardous polymerization.

(g) Section VII. Spill or Leak Procedures.

Detailed procedures to be followed with emphasis on precautions to be taken in cleaning up and safe disposal of materials leaked or spilled. This includes proper labeling and disposal of containers containing residues, contaminated absorbants, etc.

(h) Section VIII. Special Protection Information.

Requirements for personal protective equipment, such as respirators, eye protection and protective clothing, and ventilation such as local

exhaust (at site of product use or application), general, or other special types.

(i) Section IX. Special Precautions.

Any other general precautionary information.

U.S. DEPARTMENT OF LABOR
Occupational Safety and Health Administration

Form Approved
OMB No. 44-R1387

MATERIAL SAFETY DATA SHEET

Required under USDL Safety and Health Regulations for Ship Repairing, Shipbuilding, and Shipbreaking (29 CFR 1915, 1916, 1917)

SECTION I

MANUFACTURER'S NAME	EMERGENCY TELEPHONE NO.
ADDRESS *(Number, Street, City, State, and ZIP Code)*	
CHEMICAL NAME AND SYNONYMS	TRADE NAME AND SYNONYMS
CHEMICAL FAMILY	FORMULA

SECTION II - HAZARDOUS INGREDIENTS

PAINTS, PRESERVATIVES, & SOLVENTS	%	TLV (Units)	ALLOYS AND METALLIC COATINGS	%	TLV (Units)
PIGMENTS			BASE METAL		
CATALYST			ALLOYS		
VEHICLE			METALLIC COATINGS		
SOLVENTS			FILLER METAL PLUS COATING OR CORE FLUX		
ADDITIVES			OTHERS		
OTHERS					

HAZARDOUS MIXTURES OF OTHER LIQUIDS, SOLIDS, OR GASES	%	TLV (Units)

SECTION III - PHYSICAL DATA

BOILING POINT (°F.)		SPECIFIC GRAVITY (H_2O=1)	
VAPOR PRESSURE (mm Hg.)		PERCENT, VOLATILE BY VOLUME (%)	
VAPOR DENSITY (AIR=1)		EVAPORATION RATE (_____ =1)	
SOLUBILITY IN WATER			
APPEARANCE AND ODOR			

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT (Method used)	FLAMMABLE LIMITS	Lel	Uel

EXTINGUISHING MEDIA
SPECIAL FIRE FIGHTING PROCEDURES
UNUSUAL FIRE AND EXPLOSION HAZARDS

SECTION V - HEALTH HAZARD DATA

THRESHOLD LIMIT VALUE

EFFECTS OF OVEREXPOSURE

EMERGENCY AND FIRST AID PROCEDURES

SECTION VI - REACTIVITY DATA

STABILITY	UNSTABLE		CONDITIONS TO AVOID
	STABLE		

INCOMPATABILITY *(Materials to avoid)*

HAZARDOUS DECOMPOSITION PRODUCTS

HAZARDOUS POLYMERIZATION	MAY OCCUR		CONDITIONS TO AVOID
	WILL NOT OCCUR		

SECTION VII - SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED

WASTE DISPOSAL METHOD

SECTION VIII - SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION *(Specify type)*

VENTILATION	LOCAL EXHAUST	SPECIAL
	MECHANICAL *(General)*	OTHER

PROTECTIVE GLOVES	EYE PROTECTION

OTHER PROTECTIVE EQUIPMENT

SECTION IX - SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING

OTHER PRECAUTIONS

TABLE XI-1

PROPERTIES OF AMMONIA

Property	Anhydrous	Aqua Ammonia (ammonia hydroxide)
Chemical formula	NH_3	NH_4OH
Formula weight	17.03	35.05
Boiling point	-33.35 C	varies with concentration
Melting point	-77.7 C	----
Autoignition temperature	651 C	----
Flammable limits (by volume in air)	16-25%	----
Solubility		
Cold water (0 C)	89.9 g/100 cc	
Hot water (100 C)	7.4 g/100 cc	
Color	Colorless	Colorless

Adapted from references 1-3

TABLE XI-2
OCCUPATIONS WITH POTENTIAL EXPOSURE TO AMMONIA

Acetylene workers
Aluminum workers
Amine workers
Ammonia workers
Ammonium salt makers
Aniline makers
Annealers
Boneblack makers
Braziers
Bronzers
Calcium carbide makers
Case hardeners
Chemical laboratory workers
Chemical manufacturers
Coal tar workers
Coke makers
Color makers
Compressed gas workers
Corn growers
Cyanide makers
Decorators
Diazo reproducing machine operators
Drug makers
Dye intermediate makers
Dye makers
Electroplaters
Electrotypers
Explosive makers
Farmers
Fertilizer workers
Galvanizers
Gas purifiers
Gas workers, illuminating
Glass cleaners
Glue makers
Ice cream makers
Ice makers
Ink makers
Lacquer makers
Latex workers
Maintenance workers (janitors)
Manure handlers
Metal extractors
Metal powder processors
Mirror silverers
Nitric acid makers
Organic chemical synthesizers
Paper makers
Perfume makers
Pesticide makers
Petroleum refinery workers
Photoengravers
Photographic film makers
Plastic cement mixers
Pulp makers
Rayon makers
Refrigeration workers
Resin makers
Rocket fuel makers
Rubber cement mixers
Rubber workers
Salt extractors, coke oven byproducts
Sewer workers
Shellac makers
Shoe finishers
Soda ash makers
Solvay process workers
Stablemen
Steel makers
Sugar refiners
Sulfuric acid workers
Synthetic fiber makers
Tanners
Tannery workers
Textile (cotton) finishers
Transportation workers
Urea makers
Varnish makers
Vulcanizers
Water base paint workers
Water treaters
Wool scourers

Adapted from references 7-9

TABLE XI-3
SUBJECTIVE EVALUATIONS OF IRRITATION AND ODOR

	IRRITATION		ODOR	
SUBJECT	30 ppm	50 ppm	30 ppm	50 ppm
1	1	2	3	4
2	0	2	4	4
3	0	0	4	4
4	0	1	4	4
5	-	2	-	4
6	1	2	3	4

IRRITANT SCALE (NOSE AND EYE)

DEGREE	INTENSITY	DESCRIPTION
0	No irritation	Not detectable
1	Faint	Just perceptible, not painful
2	Moderate	Moderate irritation
3	Strong	Discomforting, painful, but may be endured
4	Intolerable	Exceedingly painful, cannot be endured

ODOR SCALE

DEGREE	INTENSITY	DESCRIPTION
0	No odor	No detectable odor
1	Very faint	Minimum, but positively perceptible odor
2	Faint	Weak odor, readily perceptible
3	Easily noticeable	Moderate intensity
4	Strong	Highly penetrating
5	Very strong	Intense effect

MacEwen et al [37]

TABLE XI-4
AMMONIA LEVELS FOUND FOR VARIOUS INDUSTRIAL PROCESSES

OPERATION	LEVEL	CONTROLS	REMARKS	REFERENCE
Machinery manufacturing (cleaning)	15	Not stated		Rispoli [86]
Diazo reproducing machine	8	Air conditioned room		"
Mildew-proofing	125	Not stated		Elkins [42]
Electroplating	55	"		"
Galvanizing, ammonium chloride flux	10-88	Natural ventilation, monitor roof	Ammonia formed from decomposition of flux	Pate [87]
Blueprint machine	30-35	Not stated		Pagnotto [written communication 1973]
Printing machine	1-45	"		"
Etching	36	"		"
Blueprint machine	10, 20	"		
Refrigeration equipment	9-37	"	Ice cream plant odor noticeable	"
Printing machine	3-29	"	Marked odor, not disagreeable	"
Printing machine	2-45	"		"

TABLE XI-4 (CONTINUED)
AMMONIA LEVELS FOUND FOR VARIOUS INDUSTRIAL PROCESSES

OPERATION	LEVEL	CONTROLS	REMARKS	REFERENCE
Blueprint machine	45	Not stated	Strong odor of ammonia, some eye irritation	Pagnotto [written communication 1973]
Cementing insoles	8-28	"	Latex cement, odor often distinct slight eye irritation	
Chemical mixing	60-440	"	35 gallons of 35% ammonia poured into open trough, intense exposure	"
Fabric impregnating	Not detectable	"		"

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FIGURE XI-1

CALIBRATION SETUP FOR PERSONAL SAMPLING PUMP WITH MIDGET IMPINGER