In judging the suitability of alternate GC columns or the effects of altering chromatographic conditions, one can employ the area overlap as the resolution parameter with a specific maximum permissible value.

The use of Gaussian functions to describe chromatographic elution curves is widespread. However, some elution curves are highly asymmetric. In cases where the sample peak is followed by a contaminant that has a leading edge that rises sharply but the curve then falls off, it may be possible to define an effective width for t_r as "twice the distance from the leading edge to a perpendicular line through the maxim of the contaminant curve, measured along a perpendicular bisection of that line."

Procedure 2—Procedure for Field Auditing GC Analysis

Responsibilities of audit supervisor and analyst at the source sampling site include the following:

A. The audit supervisor verifies that audit cylinders are stored in a safe location both before and after the audit to prevent vandalism.

B. At the beginning and conclusion of the audit, the analyst records each cylinder number and pressure. An audit cylinder is never analyzed when the pressure drops below 200 psi.

C. During the audit, the analyst performs a minimum of two consecutive analyses of each audit cylinder gas. The audit must be conducted to coincide with the analysis of source test samples, normally immediately after GC calibration and prior to sample analyses.

D. At the end of audit analyses, the audit supervisor requests the calculated concentrations from the analyst and compares the results with the actual audit concentrations. If each measured concentration agrees with the respective actual concentration within ±10 percent, he directs the analyst to begin analyzing source samples. Audit supervisor judgment and/or supervisory policy determine action when agreement is not within ±10 percent. When a consistent bias in excess of 10 percent is found, it may be possible to proceed with the sample analysis, with a corrective factor to be applied to the results at a later time. However, every attempt should be made to locate the cause of the discrepancy, as it may be misleading. The audit supervisor records each cylinder number, cylinder pressure (at the end of the audit), and all calculated concentrations. The individual being audited must not under any circumstance be told actual audit concentrations until calculated concentrations have been submitted to the audit supervisor.

Field Audit Report

Part A.—To be filled out by organization supplying audit cylinders.

1. Organization supplying audit sample(s) and shipping address

_____

2. Audit supervisor, organization, and phone number

_____

3. Shipping instructions: Name, Address, Attention

_____

4. Guaranteed arrival date for cylinders_____

5. Planned shipping date for cylinders_____

6. Details on audit cylinders from last analysis

	Low conc.	High conc.
a. Date of last analysis		
b. Cylinder number		
c. Cylinder pressure, psi		
d. Audit gas(es)/balance gas		
e. Audit gas(es), ppm		
f. Cylinder construction		

Part B.—To be filled out by audit supervisor.

1. Process sampled_____

2. Audit location_____

3. Name of individual audit_____

4. Audit date_____

5. Audit results:

	Low conc. cylinder	High conc. cylinder
a. Cylinder number		
b. Cylinder pressure before audit, psi		
c. Cylinder pressure after audit, psi		
d. Measured concentration, ppm Injection #1* Injection #2* Average		
e. Actual audit concentration, ppm (Part A, 6e)		
f. Audit accuracy:[1] Low Conc. Cylinder		
High Conc. Cylinder		

$$\text{Percent accuracy} = \frac{\text{Measured Conc.} - \text{Actual Conc.}}{\text{Actual Conc.}} \times 100$$

g. Problems detected (if any)		

[1] Results of two consecutive injections that meet the sample analysis criteria of the test method.

[FR Doc. 82-24881 Filed 9-3-82; 8:45 am]

BILLING CODE 6560-50-M

FEDERAL EMERGENCY MANAGEMENT AGENCY

44 CFR Part 65

[Docket No. FEMA-6396]

List of Communities With Special Hazard Areas Under the National Flood Insurance Program

AGENCY: Federal Emergency Management Agency.

ACTION: Final rule.

SUMMARY: This rule identifies communities with areas of special flood, mudslide, or erosion hazards as authorized by the National Flood Insurance Program. The identification of such areas is to provide guidance to communities on the reduction of property losses by the adoption of appropriate flood plain management or other measures to minimize damage. It will enable communities to guide future construction, where practicable, away from locations which are threatened by flood or other hazards.

EFFECTIVE DATES: The effective date shown at the top right of the table or October 7, 1982, whichever is later.

FOR FURTHER INFORMATION CONTACT: Mr. Richard E. Sanderson, Chief, Natural Hazards Division, (200) 287-0270, 500 C Street Southwest, Donohoe Building, Room 505, Washington, DC 20472.

SUPPLEMENTARY INFORMATION: The Flood Disaster Protection Act of 1973 (Pub. L. 93-234) requires the purchase of flood insurance on and after March 2, 1974, as a condition of receiving any form of Federal or federally related financial assistance for acquisition or construction purposes in an identified flood plain area having special flood hazards that is located within any community participating in the National Flood Insurance Program.

One year after the identification of the community as flood prone, the requirement applies to all identified special flood hazard areas within the United States, so that, after the date, no such financial assistance can legally be provided for acquisition and construction in these areas unless the community has entered the program. The prohibition, however, does not apply in respect to conventional mortgage loans by federally regulated, insured, supervised, or approved lending institutions.

This 30 day period does not supersede the statutory requirement that a community, whether or not participating in the program, be given the opportunity for a period of six months to establish that it is not seriously flood prone or that such flood hazards as may have existed have been corrected by floodworks or other flood control methods. The six months period shall be considered to begin 30 days after the date of publication in the Federal Register or the effective date of the Flood Hazard Boundary Map, whichever is later. Similarly, the one year period a community has to enter the program under section 201(d) of the Flood Disaster Protection Act of 1973 shall be considered to begin 30 days after publication in the Federal Register or the effective date of the Flood Hazard Boundary Map, whichever is later.

This identification is made in accordance with Part 64 of Title 44 of the Code of Federal Regulations as

ENVIRONMENTAL PROTECTION AGENCY REGULATIONS ON NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS

(40 CFR 61; 38 FR 8820, April 6, 1973; Amended as shown in Code of Federal Regulations, Volume 40, Revised as of July 1, 1978; 43 FR 47692, October 16, 1978; 44 FR 7714, February 7, 1979; 44 FR 55174, September 25, 1979; 44 FR 65399, November 13, 1979; 45 FR 13074, February 28, 1980; 46 FR 27342, May 19, 1981; 46 FR 29262, June 1, 1981; 46 FR 39422, July 31, 1981; 46 FR 49853, October 8, 1981)

PART 61—NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS

Subpart A—General Provisions

Subpart B—National Emission Standard for Asbestos

Subpart C—National Emission Standard for Beryllium

Subpart D—National Emission Standard for Beryllium Rocket Motor Firing

Subpart E—National Emission Standard for Mercury

Subpart F—National Emission Standard for Vinyl Chloride

AUTHORITY: Sec. 112, 301(a), Clean Air Act as amended (42 U.S.C. 7412, 7601(a)), unless otherwise noted.

SOURCE: 38 FR 8826, Apr. 6, 1973, unless otherwise noted.

Subpart A—General Provisions

§ 61.01 Applicability.

The provisions of this part apply to the owner or operator of any stationary source for which a standard is prescribed under this part.

§ 61.02 Definitions.

[61.02 amended by 44 FR 55174, September 25, 1979]

The terms used in this part are defined in the Act or in this section as follows:

"Act" means the Clean Air Act (42 U.S.C. 1857 et seq.).

"Administrator" means the Administrator of the Environmental Protection Agency or his authorized representative.

"Alternative method" means any method of sampling and analyzing for an air pollutant which is not a reference or equivalent method but which has been demonstrated to the Administrator's satisfaction to, in specific cases, produce results adequate for his determination of compliance.

"Commenced" means, with respect to the definition of "new source" in section 111(a)(2) of the Act, that an owner or operator has undertaken a continuous program of construction or modification or that an owner or operator has entered into a contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or modification.

"Compliance schedule" means the date or dates by which a source or category of sources is required to comply with the standards of this part and with any steps toward such compliance which are set forth in a waiver of compliance under §61.11.

"Construction" means fabrication, erection, or installation of an affected facility.

"Effective date" is the date of promulgation in the Federal Register of an applicable standard or other regulation under this part.

"Equivalent method" means any method of sampling and analyzing for an air pollutant which has been demonstrated to the Administrator's satisfaction to have a consistent

and quantitatively known relationship to the reference method, under specified conditions.

"Existing source" means any stationary source which is not a new source.

"Modification" means any physical change in, or change in the method of operation of, a stationary source which increases the amount of any hazardous air pollutant emitted by such source, or which results in the emission of any hazardous air pollutant not previously emitted, except that:

(a) Routine maintenance, repair, and replacement shall not be considered physical changes, and

(b) The following shall not be considered a change in the method of operation:

(1) An increase in the production rate, if such increase does not exceed the operating design capacity of the stationary source;

(2) An increase in hours of operation.

"New source" means any stationary source, the construction or modification of which is commenced after the publication in the Federal Register of proposed national emission standards for hazardous air pollutants which will be applicable to such source.

"Owner or operator" means any person who owns, leases, operates, controls, or supervises a stationary source.

"Reference method" means any method of sampling and analyzing for an air pollutant, as described in Appendix B to this part.

"Standard" means a national emission standard for a hazardous air pollutant proposed or promulgated under this part.

"Startup" means the setting in operation of a stationary source for any purpose.

"Stationary source" means any building, structure, facility or installation which emits or may emit any air pollutant which has been designated as hazardous by the Administrator.

§61.03 Units and abbreviations.

Used in this part are abbreviations and symbols of units of measure. These are defined as follows:

(a) System International (SI) units of measure:

A = ampere
g = gram
Hz = hertz
J = joule
K = degree Kelvin
kg = kilogram
m = meter
m³ = cubic meter
mg = milligram = 10^{-3} gram
mm = millimeter = 10^{-3} meter
Mg = megagram = 10^{6} gram
mol = mole
N = newton
ng = nanogram = 10^{-9} gram
nm = nanometer = 10^{-9} meter
Pa = pascal
s = second
V = volt
W = watt
Ω = ohm
μg = microgram = 10^{-6} gram

(b) Other units of measure:

°C = degree Celsius (centigrade)
cfm = cubic feet per minute
cc = cubic centimeter
d = day
°F = degree Fahrenheit
ft² = square feet
ft³ = cubic feet
gal = gallon
in = inch
in Hg = inches of mercury
in H_2O = inches of water
l = liter
lb = pound
lpm = liter per minute
min = minute
ml = milliliter = 10^{-3} liter
oz = ounces
psig = pounds per square inch gage
°R = degree Rankine
μl = microliter = 10^{-6} liter
v/v = volume per volume
yd² = square yards
yr = year

(c) Chemical nomenclature:

Be = beryllium
Hg = mercury
H_2O = water

(d) Miscellaneous:

act = actual
avg = average
I.D. = inside diameter
M = molar
N = normal
O.D. = outside diameter
% = percent
std = standard

[42 FR 51574, September 29, 1977]

§61.04 Address.

[61.04(a) amended by 46 FR 39422, July 31, 1981]

(a) All requests, reports, applications, submittals, and other communications to the Administrator pursuant to this part shall be submitted in duplicate and addressed to the appropriate Regional Office of the Environmental Protection Agency, to the attention of the Director, Enforcement Division. The regional offices are as follows:

Region I (Connecticut, Maine, New Hampshire, Massachusetts, Rhode Island, Vermont), John F. Kennedy Federal Building, Boston, Massachusetts 02203.

Region II (New York, New Jersey, Puerto Rico, Virgin Islands), Federal Office Building, 26 Federal Plaza (Foley Square), New York, N.Y. 10007.

Region III (Delaware, District of Columbia, Pennsylvania, Maryland, Virginia, West Virginia), Curtis Building, Sixth and Walnut Streets, Philadelphia, Pennsylvania 19106.

Region IV (Alabama, Florida, Georgia, Mississippi, Kentucky, North Carolina, South Carolina, Tennessee), Suite 300, 1421 Peachtree Street, Atlanta, Georgia 30309.

Region V (Illinois, Indiana, Minnesota, Michigan, Ohio, Wisconsin), 230 South Dearborn Street, Chicago, Illinois 60604.

Region VI (Arkansas, Louisiana, New Mexico, Oklahoma, Texas), 1600 Patterson Street, Dallas, Texas 75201.

Region VII (Iowa, Kansas, Missouri, Nebraska), 324 East 11th Street, Kansas City, Missouri 64106.

Region VIII (Colorado, Montana, North Dakota, South Dakota, Utah, Wyoming), 196 Lincoln Towers, 1860 Lincoln Street, Denver, Colorado 80203.

Region IX (Arizona, California, Hawaii, Nevada, Guam, American Samoa), 100 California Street, San Francisco, California 94111.

Region X (Washington, Oregon, Idaho, Alaska), 1200 Sixth Avenue, Seattle, Washington 98101.

(b) Section 112(d) directs the Administrator to delegate to each State, when appropriate, the authority to implement and enforce the national emission standards for hazardous air pollutants for stationary sources located in such State. All information required to be submitted to EPA under paragraph (a) of this section, must also be submitted to the appropriate State Agency of any State to which this authority has been delegated (provided, that each specific delegation may exempt sources from a certain federal or State reporting requirement). The appropriate mailing address for those States whose delegation request has been approved is as follows:

(A) [reserved]

(B) State of Alabama, Air Pollution Control Division, Air Pollution Control Commission, 645 S. McDonough Street, Montgomery, Alabama 36104.

(C) [reserved]

(D) Arizona:

Maricopa County Department of Health Services, Bureau of Air Pollution Control, 1825 East Roosevelt Street, Phoenix, AZ 85006.

Pima County Health Department, Air Quality Control District, 151 West Congress, Tucson, AZ 85701.

(E) [reserved]

F—California:

(F) California:

[61.04(b)(F) amended by 46 FR 49853, October 8, 1981]

Amador County Air Pollution Control District, P.O. Box 430, 810 Court Street, Jackson, CA 95642

Bay Area Air Pollution Control District, 939 Ellis Street, San Francisco, CA 94109.

Butte County Air Pollution Control District, P.O. Box 1229, 316 Nelson Avenue, Oroville, CA 95965

Calaveras County Air Pollution Control District, Government Center, El Dorado Road, San Andreas, CA 95249

Colusa County Air Pollution Control District, 751 Fremont Street, Colusa, CA 95932

Del Norte County Air Pollution Control District, 909 Highway 101 North, Crescent City, CA 95531

El Dorado Air Pollution Control District, 360 Fair Lane, Placerville, CA 95667

Fresno County Air Pollution Control District, P.O. Box 11867, 1246 L Street, Fresno, CA 93721

Glenn County Air Pollution Control District, P.O. Box 351, 720 North Colusa Street, Willows, CA 95988

Great Basin Unified Air Pollution Control District, 860 North Main Street, Suite 213, Bishop, CA 93514

Humboldt County Air Pollution Control District, 5600 S. Broadway, Eureka, CA 95501.

Imperial County Air Pollution Control District, County Services Building, 939 West Main Street, El Centro, CA 92243

Kern County Air Pollution Control District, 1700 Flower Street (P.O. Box 997), Bakersfield, CA 93302.

Kings County Air Pollution Control District, 330 Campus Drive, Hanford, CA 93230

[See. 61.04(b)]

Lake County Air Pollution Control District, 255 North Forbes Street, Lakeport, CA 95453

Lassen County Air Pollution Control District, 175 Russell Avenue, Susanville, CA 96130

Madera County Air Pollution Control District, 135 W. Yosemite Avenue, Madera, CA 93637.

Mariposa County Air Pollution Control District, Box 5, Mariposa, CA 95338

Mendocino County Air Pollution Control District, County Courthouse, Ukiah, CA 94582.

Merced County Air Pollution Control District, P.O. Box 471, 240 East 15th Street, Merced, CA 95340

Modoc County Air Pollution Control District, 202 West 4th Street, Alturas, CA 96101

Monterey Bay Unified Air Pollution Control District, 1270 Natividad Road, Room 108, Salinas, CA 93906

Nevada County Air Pollution Control District, H.E.W. Complex, Nevada City, CA 95959

Northern Sonoma County Air Pollution Control District, 134 "A" Avenue, Auburn, CA 95448

Placer County Air Pollution Control District, 11491 "B" Avenue, Auburn, CA 95603

Plumas County Air Pollution Control District, P.O. Box 480, Quincy, CA 95971

Sacramento County Air Pollution Control District, 3701 Branch Center Road, Sacramento, CA 95827.

San Bernardino County Air Pollution Control District, 15579–8th, Victorville, CA 92392

San Diego County Air Pollution Control District, 9150 Chesapeake Drive, San Diego, CA 92123.

San Joaquin County Air Pollution Control District, 1601 E. Hazelton Street (P.O. Box 2009), Stockton, CA 95201.

San Luis Obispo County Air Pollution Control District, P.O. Box 637, San Luis Obispo, CA 93406

Santa Barbara County Air Pollution Control District, 300 North San Antonio Road, Santa Barbara, CA 93110

Shasta County Air Pollution Control District, 2650 Hospital Lane, Redding, CA 96001

Sierra County Air Pollution Control District, P.O. Box 286, Downieville, CA 95936

Siskiyou County Air Pollution Control District, 525 South Foothill Drive, Yreka, CA 96097

South Coast Air Quality Management District, 9150 Flair Drive, El Monte, CA 91731

Stanislaus County Air Pollution Control District, 1030 Scenic Drive, Modesto, CA 95350

Sutter County Air Pollution Control District, Sutter County Office Building, 142 Garden Highway, Yuba City, CA 95991

Tehama County Air Pollution Control District, P.O. Box 38, 1760 Walnut Street, Red Bluff, CA 96080

Trinity County Air Pollution Control District, P.O. Box AK, Weaverville, CA 96093

Tulare County Air Pollution Control District, County Civic Center, Visalia, CA 93277

Tuolumne County Air Pollution Control District, 9 North Washington Street, Sonora, CA 95370

Ventura County Air Pollution Control District, 800 South Victoria Avenue, Ventura, CA 93009

Yolo-Solano Air Pollution Control District, P.O. Box 1006, 323 First Street, #5, Woodland, CA 95695

[43 FR 20988, May 16, 1978]

(G)—State of Colorado, Colorado Air Pollution Control Division, 4210 East 11th Avenue, Denver, Colorado 80220.

[40 FR 50719, October 31, 1975]

(H) State of Connecticut, Department of Environmental Protection, State Office Building, Hartford, Connecticut 06115

[41 FR 11820, March 22, 1976]

(I) State of Delaware (for asbestos, beryllium and mercury only): Delaware Department of Natural Resources and Environmental Control, Edward Tatnall Building, Dover, Delaware 19901.

[43 FR 6771, February 16, 1978]

(J)-(K) [reserved]

(L) State of Georgia, Environmental Protection Division, Department of Natural Resources, 270 Washington Street, S.W., Atlanta, Georgia 30334.

[41 FR 24885, June 21, 1976]

(M)-(O) [reserved]

(P) State of Indiana, Indiana Air Pollution Control Board, 1330 West Michigan Street, Indianapolis, Indiana 46206.

[41 FR 43148, September 30, 1976]

(S) Division of Air Pollution Control, Department for Natural Resources and Environmental Protection, U.S. 127, Frankfort, Ky. 40601.

[43 FR 3361, January 25, 1978]

(U) State of Maine, Department of Environmental Protection, State House, Augusta, Maine 04330.

[40 FR 59729, December 30, 1975]

(V) State of Maryland, Bureau of Air Quality and Noise Control, Maryland State Department of Health and Mental Hygiene, 201 West Preston Street, Baltimore, Maryland 21201.

[51.04(b)(V) added by 45 FR 13074, February 28, 1980]

(W) Massachusetts Department of Environmental Quality Engineering, Division of Air Quality Control, 600 Washington Street, Boston, Massachusetts 02111.

(X) State of Michigan, Air Pollution Control Division, Michigan Department of Natural Resources, Stevens T. Mason Building, 8th Floor, Lansing, Michigan 48926.

(Y) Minnesota Pollution Control Agency, Division of Air Quality, 1935 West County Road B-2, Roseville, Minn. 55113.

[43 FR 10, January 3, 1978]

(Z) [reserved]

(AA) Missouri Department of Natural Resources, Post Office Box 1368, Jefferson City, Missouri 65101.

[51.04(b)(AA) added by 46 FR 27342, May 19, 1981]

(BB) State of Montana, Department of Health and Environmental Sciences, Cogswell Building, Helena, Mont. 59601.

[42 FR 44545, September 6, 1977]

(CC) State of Nebraska, Nebraska Department of Environmental Control, P.O. Box 94877, State House Station, Lincoln, Nebraska 68509.

[51.04(b)(CC) added by 46 FR 39422, July 31, 1981]

(DD) Nevada:

Nevada Department of Conservation and Natural Resources, Division of Environmental Protection, 201 South Fall Street, Carson City, NV 89710.

Clark County County District Health Department, Air Pollution Control Division, 625 Shadow Lane, Las Vegas, NV 89106.

Washoe County District Health Department, Division of Environmental Protection, 10 Kirman Avenue, Reno, NV 89502.

[43 FR 20988, May 16, 1978]

(EE) New Hampshire Air Pollution Control Agency, Department of Health and Welfare, State Laboratory Building, Hazen Drive, Concord, New Hampshire 03301.

[41 FR 19633, May 13, 1976]

(FF)—State of New Jersey: New Jersey Department of Environmental Protection, John Fitch Plaza, P.O. Box 2807, Trenton, New Jersey 08625.

[42 FR 37387, July 21, 1977]

(GG) [reserved]

(HH)—New York: New York State Department of Environmental Conservation, 50 Wolf Road, Albany, New York 12233, attention: Division of Air Resources.

[40 FR 48347, October 15, 1975]

(II) North Carolina Environmental Management Commission, Department of Natural and Economic Resources, Division of Environmental Management, P.O. Box 27687, Raleigh, North Carolina 27611. Attention: Air Quality Section.

[41 FR 56805, December 30, 1976]

(JJ)—State of North Dakota, State Department of Health, State Capitol, Bismarck, North Dakota 58501.

[41 FR 44859, October 13, 1976]

(KK) Ohio

Montgomery County: Regional Air Pollution Control Agency, Montgomery County Combined General Health District, 451 West Third Street, Dayton, Ohio 45402.

Clarke, Darke, Greene, Miami and Preble Counties (except for all information required under §51.22(d) and (e)): Montgomery County Combined General Health District, 451 West Third Street, Dayton, Ohio 45402.

[60.04(b)(KK) added by 44 FR 65400, November 13, 1979]

(LL) [Reserved]

(MM)—State of Oregon, Department of Environmental Quality, 1234 SW Morrison Street, Portland, Oregon 97205.

[41 FR 7750, February 20, 1976]

(NN)(a) Commonwealth of Pennsylvania (except for City of Philadelphia and Allegheny County)

Pennsylvania Department of Environmental Resources,
Bureau of Air Quality and Noise Control,
Post Office Box 2063,
Harrisburg, Pennsylvania 17120.

(NN)(b) City of Philadelphia,
Philadelphia Department of Public Health,
Air Management Services,
501 Arch Street,
Philadelphia, Pennsylvania 19107.

[42 FR 6812, February 4, 1977]

(OO) State of Rhode Island, Department of Environmental Management, 83 Park Street, Providence, R.I. 02903

[43 FR 47692, October 16, 1978]

(PP) State of South Carolina, Office of Environmental Quality Control, Department of Health and Environmental Control, 2600 Bull Street, Columbia, South Carolina 29201.

[42 FR 4124, January 24, 1977]

[Sec. 51.04(b)]

(QQ) [reserved]
(RR) Division of Air Pollution Control, Tennessee Department of Public Health, 256 Capitol Hill Building, Nashville, Tennessee 37219.
[61.04(b)(RR) added by 46 FR 29262, June 1, 1981]

(SS) State of Texas, Texas Air Control Board, 8520 Shoal Creek Boulevard, Austin, Texas 78758.

[44 FR 7714, February 7, 1979]

(TT) [reserved]

(UU)—State of Vermont, Agency of Environmental Protection, Box 489, Montpelier, Vermont 05602.
[42 FR 1215, January 6, 1977]

(VV) Commonwealth of Virginia, Virginia State Air Pollution Control Board, Room 1106, Ninth Street Office Building, Richmond, Virginia 23219.

[41 FR 8346, February 26, 1976]

(WW) (i) Washington: State of Washington, Department of Ecology, Olympia, Washington 98504.

(ii) Northwest Air Pollution Authority, 207 Pioneer Building, Second and Pine Streets, Mount Vernon, Washington 98273.

(iii) Puget Sound Air Pollution Control Agency, 410 West Harrison Street, Seattle, Washington 98119.

(iv) Spokane County Air Pollution Control Authority, North 811 Jefferson, Spokane, Washington 99201.

(v) Yakima County Clean Air Authority, County Courthouse, Yakima, Washington 98901.
[40 FR 58640, December 18, 1975]

(vi) Olympic Air Pollution Control Authority, 120 East State Avenue, Olympia, Washington 98501.

(vii) Southwest Air Pollution Control Authority, Suite 7801 H, NE Hazel Dell Avenue, Vancouver, Washington 98665.
[41 FR 4264, January 29, 1976]

(XX) [reserved].

(YY) Wisconsin—
Wisconsin Department of Natural Resources, P.O. Box 7921, Madison, Wisconsin 53707.
[42 FR 16778, March 30, 1977]

(ZZ) [reserved].

(AAA) [reserved]

(BBB) — Commonwealth of Puerto Rico: Commonwealth of Puerto Rico Environmental Quality Board, P.O. Box 11785, Santurce, P.R. 00910.

[42 FR 62137, December 9, 1977]

(CCC)—U.S. Virgin Islands: U.S. Virgin Islands Department of Conservation and Cultural Affairs, P.O. Box 578, Charlotte Amalie, St. Thomas, U.S. Virgin Islands 00801.

[41 FR 34629, August 16, 1976]

(DDD) [reserved].

[39 FR 37987, October 25, 1974; 40 FR 18169, April 25, 1975; 40 FR 42195, September 11, 1975]

§ 61.05 Prohibited activities.

(a) After the effective date of any standard prescribed under this part, no owner or operator shall construct or modify any stationary source subject to such standard without first obtaining written approval of the Administrator in accordance with this subpart, except under an exemption granted by the President under section 112(e)(2) of the act. Sources, the construction or modification of which commenced after the publication date of the standards proposed to be applicable to such source, are subject to this prohibition.

(b) After the effective date of any standard prescribed under this part, no owner or operator shall operate any new source in violation of such standard except under an exemption granted by the President under section 112(c)(2) of the act.

(c) Ninety days after the effective date of any standard prescribed under this part, no owner or operator shall operate any existing stationary source in violation of such standard, except under a waiver granted by the Administrator in accordance with this subpart or under an exemption granted by the President under section 112(c)(2) of the act.

(d) No owner or operator subject to the provisions of this part shall fail to report, revise reports, or report source test results as required under this part.

§ 61.06 Determination of construction or modification.

Upon written application by an owner or operator, the Administrator will make a determination of whether actions taken or intended to be taken by such owner or operator constitute construction or modification or the commencement thereof within the meaning of this part. The Administrator will within 30 days of receipt of sufficient information to evaluate an application, notify the owner or operator of his determination.

§ 61.07 Application for approval of construction or modification.

(a) The owner or operator of any new source to which a standard prescribed under this part is applicable shall, prior to the date on which construction or modification is planned to commence, or within 30 days after the effective date in the case of a new source that already has commenced construction or modification and has not begun operation, submit to the Administrator an application for approval of such construction or modification. A separate application shall be submitted for each stationary source.

(b) Each application shall include:

(1) The name and address of the applicant.

(2) The location or proposed location of the source.

(3) Technical information describing the proposed nature, size, design, operating design capacity, and method of operation of the source, including a description of any equipment to be used for control of emissions. Such technical information shall include calculations of emission estimates in sufficient detail to permit assessment of the validity of such calculations.

§ 61.08 Approval by Administrator.

(a) The Administrator will, within 60 days of receipt of sufficient information to evaluate an application under § 61.07, notify the owner or operator of approval or intention to deny approval of construction or modification.

(b) If the Administrator determines that a stationary source for which an application pursuant to § 61.07 was submitted will, if properly operated, not cause emissions in violation of a standard, he will approve the construction or modification of such source.

(c) Prior to denying any application for approval of construction or modification pursuant to this section, the Administrator will notify the owner or operator making such application of the Administrator's intention to issue such denial, together with:

(1) Notice of the information and findings on which such intended denial is based, and

(2) Notice of opportunity for such owner or operator to present, within such time limit as the Administrator shall specify, additional information or arguments to the Administrator prior to final action on such application.

(d) A final determination to deny any application for approval will be in writing and will set forth the specific grounds on which such denial is based. Such final determination will be made within 60 days of presentation of additional information or arguments, or 60 days after the final date specified for presentation, if no presentation is made.

(e) Neither the submission of an application for approval nor the Administrator's granting of approval to construct or modify shall:

(1) Relieve an owner or operator of legal responsibility for compliance with any applicable provision of this part or of any other applicable Federal, State, or local requirement, or

(2) Prevent the Administrator from implementing or enforcing this part or taking any other action under the act.

§ 61.09 Notification of startup.

(a) Any owner or operator of a source which has an initial startup after the effective date of a standard prescribed under this part shall furnish the Administrator written notification as follows:

(1) A notification of the anticipated date of initial startup of the source not more than 60 days nor less than 30 days prior to such date.

[Sec. 61.09(a)(1)]

(2) A notification of the actual date of initial startup of the source within 15 days after such date.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

§ 61.10 Source reporting and waiver request.

(a) The owner or operator of any existing source, or any new source to which a standard prescribed under this part is applicable which had an initial startup which preceded the effective date of a standard prescribed under this part shall, within 90 days after the effective date, provide the following information in writing to the Administrator:

(1) Name and address of the owner or operator.

(2) The location of the source.

(3) The type of hazardous pollutants emitted by the stationary source.

(4) A brief description of the nature, size, design, and method of operation of the stationary source including the operating design capacity of such source. Identify each point of emission for each hazardous pollutant.

(5) The average weight per month of the hazardous materials being processed by the source, over the last 12 months preceding the date of the report.

(6) A description of the existing control equipment for each emission point.

(i) Primary control device(s) for each hazardous pollutant.

(ii) Secondary control device(s) for each hazardous pollutant.

(iii) Estimated control efficiency (percent) for each control device.

(7) A statement by the owner or operator of the source as to whether he can comply with the standards prescribed in this part within 90 days of the effective date.

(b) The owner or operator of an existing source unable to operate in compliance with any standard prescribed under this part may request a waiver of compliance with such standard for a period not exceeding 2 years from the effective date. Any request shall be in writing and shall include the following information:

(1) A description of the controls to be installed to comply with the standard.

(2) A compliance schedule, including the date each step toward compliance will be reached. Such list shall include as a minimum the following dates:

(i) Date by which contracts for emission control systems or process modifications will be awarded, or date by which orders will be issued for the purchase of component parts to accomplish emission control or process modification;

(ii) Date of initiation of onsite construction or installation of emission control equipment or process change;

(iii) Date by which onsite construction or installation of emission control equipment or process modification is to be completed; and

(iv) Date by which final compliance is to be achieved.

(3) A description of interim emission control steps which will be taken during the waiver period.

(c) Changes in the information provided under paragraph (a) of this section shall be provided to the Administrator within 30 days after such change, except that if changes will result from modification of the source, as defined in § 61.02(j), the provisions of § 61.07 and § 61.08 are applicable.

(d) The format for reporting under this section is included as appendix A of this part. Advice on reporting the status of compliance may be obtained from the Administrator.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 61.11 Waiver of compliance.

(a) Based on the information provided in any request under § 61.10, or other information, the Administrator may grant a waiver of compliance with a standard for a period not exceeding 2 years from the effective date of such standard.

(b) Such waiver will be in writing and will:

(1) Identify the stationary source covered.

(2) Specify the termination date of the waiver. The waiver may be terminated at an earlier date if the conditions specified under paragraph (b) (3) of this section are not met.

(3) Specify dates by which steps toward compliance are to be taken; and impose such additional conditions as the Administrator determines to be necessary to assure installation of the necessary controls within the waiver period, and to assure protection of the health of persons during the waiver period.

(c) Prior to denying any request for a waiver pursuant to this section, the Administrator will notify the owner or operator making such request of the Administrator's intention to issue such denial, together with:

(1) Notice of the information and findings on which such intended denial is based, and

(2) Notice of opportunity for such owner or operator to present, within such time limit as the Administrator specifies, additional information or arguments to the Administrator prior to final action on such request.

(d) A final determination to deny any request for a waiver will be in writing and will set forth the specific grounds on which such denial is based. Such final determination will be made within 60 days after presentation of additional information or arguments, or 60 days after the final date specified for such presentation, if no presentation is made.

(e) The granting of a waiver under this section shall not abrogate the Administrator's authority under section 114 of the act.

§ 61.12 Emission tests and monitoring.

(a) Emission tests and monitoring shall be conducted and reported as set forth in this part and appendix B to this part.

(b) The owner or operator of a new source subject to this part, and at the request of the Administrator, the owner or operator of an existing source subject to this part, shall provide or cause to be provided, emission testing facilities as follows:

(1) Sampling ports adequate for test methods applicable to such source.

(2) Safe sampling platform(s).

(3) Safe access to sampling platform(s).

(4) Utilities for sampling and testing equipment.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 61.13 Waiver of emission tests.

(a) Emission tests may be waived upon written application to the Administrator if, in his judgment, the source is meeting the standard, or if the source is operating under a waiver of compliance or has requested a waiver of compliance.

(b) If application for waiver of the emission test is made, such application shall accompany the information required by § 61.10. The appropriate form is contained in appendix A to this part.

(c) Approval of any waiver granted pursuant to this section shall not abrogate the Administrator's authority under the act or in any way prohibit the Administrator from later canceling such waiver. Such cancellation will be made only after notice is given to the owner or operator of the source.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 61.14 Source test and analytical methods.

(a) Methods 101, 102, and 104 in appendix B to this part shall be used for all source tests required under this part, unless an equivalent method or an alternative method has been approved by the Administrator.

(b) Method 103 in appendix B to this part is hereby approved by the Administrator as an alternative method for sources subject to § 61.32(a) and § 61.42 (b).

(c) The Administrator may, after notice to the owner or operator, withdraw approval of an alternative method granted under paragraphs (a), (b) or (d) of this section. Where the test results using an alternative method do not adequately indicate whether a source is in compliance with a standard, the Administrator may require the use of the reference method or its equivalent.

(d) Method 105 in Appendix B to this part is hereby approved by the Administrator as an alternative method for sources subject to § 61.52(b).

[40 FR 48292, October 14, 1975]

§ 61.15 Availability of information.

The availability to the public of information provided to, or otherwise obtained by, the Administrator under this part shall be governed by Part 2 of this chapter.

[41 FR 36918, September 1, 1976]

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 61.16 State authority.

(a) The provisions of this part shall not be construed in any manner to preclude any State or political subdivision thereof from:

[Sec. 61.16(a)]

(1) Adopting and enforcing any emission limiting regulation applicable to a stationary source, provided that such emission limiting regulation is not less stringent than the standards prescribed under this part.

(2) Requiring the owner or operator of a stationary source, other than a stationary source owned or operated by the United States, to obtain permits, licenses, or approvals prior to initiating construction, modification, or operation of such source.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414.)

§ 61.17 Circumvention.

No owner or operator subject to the provisions of this part shall build, erect, install, or use any article, machine, equipment, process, or method, the use of which conceals an emission which would otherwise constitute a violation of an applicable standard. Such concealment includes, but is not limited to, the use of gaseous diluents to achieve compliance with a visible emissions standard, and the piecemeal carrying out of an operation to avoid coverage by a standard that applies only to operations larger than a specified size.

[40 FR 48292, October 14, 1975]

Subpart B—National Emission Standard for Asbestos

§ 61.20 Applicability.

The provisions of this subpart are applicable to those sources specified in § 61.22.

§ 61.21 Definitions.

Terms used in this subpart are defined in the act, in subpart A of this part, or in this section as follows:

(a) "Asbestos" means actinolite, amosite, anthophyllite, chrysotile, crocidolite, tremolite.

(b) "Asbestos material" means asbestos or any material containing asbestos.

(c) "Particulate asbestos material" means finely divided particles of asbestos material.

(d) "Asbestos tailings" means any solid waste product of asbestos mining or milling operations which contains asbestos.

(e) "Outside air" means the air outside buildings and structures.

(f) "Visible emissions" means any emissions which are visually detectable without the aid of instruments and which contain particulate asbestos material.

(g) "Asbestos mill" means any facility engaged in the conversion or any intermediate step in the conversion of asbestos ore into commercial asbestos. Outside storage of asbestos materials is not considered a part of such facility.

(h) "Commercial asbestos" means any variety of asbestos which is produced by extracting asbestos from asbestos ore.

(i) "Manufacturing" means the combining of commercial asbestos, or in the case of woven friction products the combining of textiles containing commercial asbestos, with any other material(s), including commercial asbestos, and the processing of this combination into a product as specified in § 61.22(c).

(j) "Demolition" means the wrecking or taking out of any load-supporting structural member and any related removing or stripping of friable asbestos materials.

(k) "Friable asbestos material" means any material that contains more than 1 percent asbestos by weight and that can be crumbled, pulverized, or reduced to powder, when dry, by hand pressure.

(l) "Control device asbestos waste" means any asbestos-containing waste material that is collected in a pollution control device.

(m) "Renovation" means the removing or stripping of friable asbestos materials used on any pipe, duct, boiler, tank, reactor, turbine, furnace, or structural member. Operations in which load-supporting structural members are wrecked or taken out are excluded.

[Paragraph (m) revised by 43 FR 26373, June 19, 1978]

(n) "Planned renovation" means a renovation operation, or a number of such operations, in which the amount of friable asbestos material that will be removed or stripped within a given period of time can be predicted. Operations that are individually non-scheduled are included, provided a number of such operations can be predicted to occur during a given period of time based on operating experience.

(o) "Emergency renovation" means a renovation operation that results from a sudden, unexpected event, and is not a planned renovation. Operations necessitated by non-routine failures of equipment are included.

(p) "Adequately wetted" means sufficiently mixed or coated with water or an aqueous solution to prevent dust emissions.

(q) "Removing" means taking out friable asbestos materials used on any pipe, duct, boiler, tank, reactor, turbine, furnace, or structural member from any building, structure, facility, or installation.

(r) "Stripping" means taking off friable asbestos materials from any pipe, duct, boiler, tank, reactor, turbine, furnace, or structural member.

[Paragraph (q) and (r) revised by 43 FR 26373, June 19, 1978]

(s) "Fabricating" means any processing of a manufactured product containing commercial asbestos, with the exception of processing at temporary sites for the construction or restoration of buildings, structures, facilities or installations.

(t) "Inactive waste disposal site" means any disposal site or portion thereof where additional asbestos-containing waste material will not be deposited and where the surface is not disturbed by vehicular traffic.

(u) "Active waste disposal site" means any disposal site other than an inactive site.

(v) "Roadways" means surfaces on which motor vehicles travel including, but not limited to, highways, roads, streets, parking areas, and driveways.

(w) "Asbestos-containing waste material" means any waste which contains commercial asbestos and is generated by a source subject to the provisions of this subpart, including asbestos mill tailings, control device asbestos waste, friable asbestos waste material, and bags or containers that previously contained commercial asbestos.

[40 FR 48292, October 14, 1975]

(x) "Structural member" means any load-supporting member, such as beams and load-supporting walls; or any non-load-supporting member, such as ceilings and non-load-supporting walls.

[42 FR 12127, March 2, 1977]

§ 61.22 Emission standard.

(a) Asbestos mills: There shall be no visible emissions to the outside air from any asbestos mill except as provided in paragraph (f) of this section.

[39 FR 15936, May 3, 1974]

(b) Roadways: The surfacing of roadways with asbestos tailings or with asbestos-containing waste that is generated by any source subject to paragraphs (c), (d), (e) or (h), of this section is prohibited, except for temporary roadways on an area of asbestos ore deposits. The deposition of asbestos tailings or asbestos-containing waste on roadways covered with snow or ice is considered "surfacing."

(c) Manufacturing: There shall be no visible emissions to the outside air, except as provided in paragraph (f) of this section, from any of the following operations if they use commercial asbestos or from any building or structure in which such operations are conducted.

[40 FR 48292, October 14, 1975]

(1) The manufacture of cloth, cord, wicks, tubing, tape, twine, rope, thread, yarn, roving, lap, or other textile materials.

(2) The manufacture of cement products.

(3) The manufacture of fireproofing and insulating materials.

(4) The manufacture of friction products.

(5) The manufacture of paper, millboard, and felt.

(6) The manufacture of floor tile.

(7) The manufacture of paints, coatings, caulks, adhesives, sealants.

(8) The manufacture of plastics and rubber materials.

(9) The manufacture of chlorine.

(10) The manufacture of shotgun shells.

(11) The manufacture of asphalt concrete.

(d) *Demolition and renovation.* The requirements of this paragraph shall apply to any owner or operator of a demolition or renovation operation who intends to demolish any institutional, commercial, or industrial building (including apartment buildings having more than four dwelling units),

operator of a source subject to the standard in § 61.52(b) shall test emissions from that source. Such tests shall be conducted in accordance with the procedures set forth either in paragraph (d) of this section or in § 61.54.

(2) Method 101 in Appendix B to this part shall be used to test emissions as follows:

(i) The test shall be performed within 90 days of the effective date of these regulations in the case of an existing source or a new source which has an initial startup date preceding the effective date.

(ii) The test shall be performed within 90 days of startup in the case of a new source which did not have an initial startup date preceding the effective date.

(3) The Administrator shall be notified at least 30 days prior to an emission test, so that he may at his option observe the test.

(4) Samples shall be taken over such a period or periods as are necessary to determine accurately the maximum emissions which will occur in a 24-hour period. No changes shall be made in the operation which would potentially increase emissions above the level determined by the most recent stack test, until the new emission level has been estimated by calculation and the results reported to the Administrator.

(5) All samples shall be analyzed, and mercury emissions shall be determined within 30 days after the stack test. Each determination shall be reported to the Administrator by a registered letter dispatched before the close of the next business day following such determination.

(6) Records of emission test results and other data needed to determine total emissions shall be retained at the source and shall be made available, for inspection by the Administrator, for a minimum of 2 years.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 61.54 Sludge sampling.

(a) As an alternative means for demonstrating compliance with § 61.52 (b), an owner or operator may use Method 105 of Appendix B and the procedures specified in this section.

(1) A sludge test shall be conducted within 90 days of the effective date of these regulations in the case of an existing source or a new source which has an initial startup date preceding the effective date; or

(2) A sludge test shall be conducted within 90 days of startup in the case of a new source which did not have an initial startup date preceding the effective date.

(b) The Administrator shall be notified at least 30 days prior to a sludge sampling test, so that he may at his option observe the test.

(c) Sludge shall be sampled according to paragraph (c)(1) of this section. sludge charging rate for the plant shall be determined according to paragraph (c)(2) of this section, and the sludge analysis shall be performed according to paragraph (c)(3) of this section.

(1) The sludge shall be sampled after dewatering and before incineration or drying, at a location that provides a representative sample of the sludge that is charged to the incinerator or dryer. Eight consecutive grab samples shall be obtained at intervals of between 45 and 60 minutes and thoroughly mixed into one sample. Each of the eight grab samples shall have a volume of at least 200 ml but not more than 400 ml. A total of three composite samples shall be obtained within an operating period of 24 hours. When the 24-hour operating period is not continuous, the total sampling period shall not exceed 72 hours after the first grab sample is obtained. Samples shall not be exposed to any condition that may result in mercury contamination or loss.

(2) The maximum 24-hour period sludge incineration or drying rate shall be determined by use of a flow rate measurement device that can measure the mass rate of sludge charged to the incinerator or dryer with an accuracy of ±5 percent over its operating range. Other methods of measuring sludge mass charging rates may be used if they have received prior approval by the Administrator.

(3) The handling, preparation, and analysis of sludge samples shall be accomplished according to Method 105 in Appendix B of this part.

(d) The mercury emissions shall be determined by use of the following equation:

$$E_{Hg} = 1 \times 10^{-3}\, cQ$$

where

E_{Hg} = Mercury emissions, g/day.

c = Mercury concentration of sludge on a dry solids basis, $\mu g/g$ (ppm).

Q = Sludge charging rate, kg/day.

(e) No changes in the operation of a plant shall be made after a sludge test has been conducted which would potentially increase emissions above the level determined by the most recent sludge test, until the new emission level has been estimated by calculation and the results reported to the Administrator.

(f) All sludge samples shall be analyzed for mercury content within 30 days after the sludge sample is collected. Each determination shall be reported to the Administrator by a registered letter dispatched before the close of the next business day following such determination.

(g) Records of sludge sampling, charging rate determination and other data needed to determine mercury content of wastewater treatment plant sludges shall be retained at the source and made available, for inspection by the Administrator, for a minimum of 2 years.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 61.55 Emission monitoring.

(a) Wastewater treatment plant sludge incineration and drying plants. All such sources for which mercury emissions exceed 1600 g/day, demonstrated either by stack sampling according to § 61.53 or sludge sampling according to § 61.54, shall monitor mercury emissions at intervals of at least once per year by use of Method 105 of Appendix B, or the procedures specified in § 61.54(c) and (d). The results of monitoring shall be reported and retained according to § 61.53(d) (5) and (6), or § 61.54(f) and (g).

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

Subpart F—National Emission Standard for Vinyl Chloride

[41 FR 46559, October 21, 1976]

§ 61.60 Applicability.

(a) This subpart applies to plants which produce:

(1) Ethylene dichloride by reaction of oxygen and hydrogen chloride with ethylene,

(2) Vinyl chloride by any process, and/or

(3) One or more polymers containing any fraction of polymerized vinyl chloride.

(b) This subpart does not apply to equipment used in research and development if the reactor used to polymerize the vinyl chloride processed in the equipment has a capacity of no more than 0.18 m³ (50 gal).

(c) Sections of this subpart other than §§ 61.61; 61.64 (a)(1), (b), (c), and (d); 61.67; 61.68; 61.69; 61.70; and 61.71 do not apply to equipment used in research and development if the reactor used to polymerize the vinyl chloride processed in the equipment has a capacity of greater than 0.18 m³ (50 gal) and no more than 1.00 m³ (265 gal).

[42 FR 29006, June 7, 1977]

§ 61.61 Definitions.

Terms used in this subpart are defined in the Act, in subpart A of this part, or in this section as follows:

(a) "Ethylene dichloride plant" includes any plant which produces ethylene dichloride by reaction of oxygen and hydrogen chloride with ethylene.

(b) "Vinyl chloride plant" includes any plant which produces vinyl chloride by any process.

(c) "Polyvinyl chloride plant" includes any plant where vinyl chloride alone or

in combination with other materials is polymerized.

(d) "Slip gauge" means a gauge which has a probe that moves through the gas/liquid interface in a storage or transfer vessel and indicates the level of vinyl chloride in the vessel by the physical state of the material the gauge discharges.

(e) "Type of resin" means the broad classification of resin referring to the basic manufacturing process for producing that resin, including, but not limited to, the suspension, dispersion, latex, bulk, and solution processes.

(f) "Grade of resin" means the subdivision of resin classification which describes it as a unique resin, i.e., the most exact description of a resin with no further subdivision.

(g) "Dispersion resin" means a resin manufactured in such away as to form fluid dispersions when dispersed in a plasticizer or plasticizer/diluent mixtures.

(h) "Latex resin" means a resin which is produced by a polymerization process which initiates from free radical catalyst sites and is sold undried.

(i) "Bulk resin" means a resin which is produced by a polymerization process in which no water is used.

(j) "Inprocess wastewater" means any water which, during manufacturing or processing, comes into direct contact with vinyl chloride or polyvinyl chloride or results from the production or use of any raw material, intermediate product, finished product, by-product, or waste product containing vinyl chloride or polyvinyl chloride but which has not been discharged to a wastewater treatment process or discharged untreated as wastewater.

(k) "Wastewater treatment process" includes any process which modifies characteristics such as BOD, COD, TSS, and pH, usually for the purpose of meeting effluent guidelines and standards; it does not include any process the purpose of which is to remove vinyl chloride from water to meet requirements of this subpart.

(l) "In vinyl chloride service" means that a piece of equipment contains or contacts either a liquid that is at least 10 percent by weight vinyl chloride or a gas that is at least 10 percent by volume vinyl chloride.

(m) "Standard operating procedure" means a formal written procedure officially adopted by the plant owner or operator and available on a routine basis to those persons responsible for carrying out the procedure.

(n) "Run" means the net period of time during which an emission sample is collected.

(o) "Ethylene dichloride purification" includes any part of the process of ethylene dichloride production which follows ethylene dichloride formation and in which finished ethylene dichloride is produced.

(p) "Vinyl chloride purification" includes any part of the process of vinyl chloride production which follows vinyl chloride formation and in which finished vinyl chloride is produced.

(q) "Reactor" includes any vessel in which vinyl chloride is partially or totally polymerized into polyvinyl chloride.

(r) "Reactor opening loss" means the emissions of vinyl chloride occurring when a reactor is vented to the atmosphere for any purpose other than an emergency relief discharge as defined in § 61.65(a).

(s) "Stripper" includes any vessel in which residual vinyl chloride is removed from polyvinyl chloride resin, except bulk resin, in the slurry form by the use of heat and/or vacuum. In the case of bulk resin, stripper includes any vessel which is used to remove residual vinyl chloride from polyvinyl chloride resin immediately following the polymerization step in the plant process flow.

(t) "Standard temperature" means a temperature of 20° C (68° F).

(u) "Standard pressure" means a pressure of 760 mm of Hg (29.92 in. of Hg).

[42 FR 29905, June 7, 1977]

§ 61.62 Emission standard for ethylene dichloride plants.

[42 FR 29905, June 7, 1977]

(a) Ethylene dichloride purification: The concentration of vinyl chloride in all exhaust gases discharged to the atmosphere from any equipment used in ethylene dichloride purification is not to exceed 10 ppm, except as provided in § 61.65(a). This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement in § 61.65(b)(6)(i) before being opened.

(b) Oxychlorination reactor: Except as provided in § 61.65(a), emissions of vinyl chloride to the atmosphere from each oxychlorination reactor are not to exceed 0.2 g/kg (0.0002 lb/lb) of the 100 percent ethylene dichloride product from the oxychlorination process.

§ 61.63 Emission standard for vinyl chloride plants.

An owner or operator of a vinyl chloride plant shall comply with the requirements of this section and § 61.65.

(a) Vinyl chloride formation and purification: The concentration of vinyl chloride in all exhaust gases discharged to the atmosphere from any equipment used in vinyl chloride formation and/or purification is not to exceed 10 ppm, except as provided in § 61.65(a). This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement in § 61.65(b)(6)(i) before being opened.

§ 61.64 Emission standard for polyvinyl chloride plants.

An owner or operator of a polyvinyl chloride plant shall comply with the requirements of this section and § 61.65.

(a) Reactor: The following requirements apply to reactors:

(1) The concentration of vinyl chloride in all exhaust gases discharged to the atmosphere from each reactor is not to exceed 10 ppm, except as provided in paragraph (a)(2) of this section and § 61.65(a).

(2) The reactor opening loss from each reactor is not to exceed 0.02 g vinyl chloride/Kg (0.00002 lb vinyl chloride/lb of polyvinyl chloride product, with the product determined on a dry solids basis. This requirement applies to any vessel which is used as a reactor or as both a reactor and a stripper. In the bulk process, the product means the gross product of prepolymerization and postpolymerization.

(3) Manual vent valve discharge: Except for an emergency manual vent valve discharge, there is to be no discharge to the atmosphere from any manual vent valve on a polyvinyl chloride reactor in vinyl chloride service. An emergency manual vent valve discharge means a discharge to the atmosphere which could not have been avoided by taking measures to prevent the discharge. Within 10 days of any discharge to the atmosphere from any manual vent valve, the owner or operator of the source from which the discharge occurs shall submit to the Administrator a report in writing containing information on the source, nature and cause of the discharge, the date and time of the discharge, the approximate total vinyl chloride loss during the discharge, the method used for determining the vinyl chloride loss, the action that was taken to prevent the discharge, and measures adopted to prevent future discharges.

(b) Stripper: The concentration of vinyl chloride in all exhaust gases discharged to the atmosphere from each stripper is not to exceed 10 ppm, except as provided in § 61.65(a). This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement in § 61.65(b)(6)(i) before being opened.

(c) Mixing, weighing, and holding containers: The concentration of vinyl chloride in all exhaust gases discharged to the atmosphere from each mixing, weighing, or holding container in vinyl chloride service which precedes the stripper (or the reactor if the plant has no stripper) in the plant process flow is not to exceed 10 ppm, except as provided in § 61.65(a). This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement in § 61.65(b)(6)(i) before being opened.

(d) Monomer recovery system. The concentration of vinyl chloride in all exhaust gases discharged to the atmosphere from each monomer recovery system is not to exceed 10 ppm, except as provided in § 61.65(a). This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement in § 61.65(b)(6)(i) before being opened.

(e) Sources following the stripper(s): The following requirements apply to emissions of vinyl chloride to the atmosphere from the combination of all sources following the stripper(s) (or the reactor(s) if the plant has no stripper(s)) in the plant process flow including but not limited to, centrifuges, concentrators, blend tanks, filters, dryers, conveyor air discharges, baggers, storage containers, and inprocess wastewater:

(1) In polyvinyl chloride plants using stripping technology to control vinyl chloride emissions, the weighted average residual vinyl chloride concentration in all grades of polyvinyl chloride resin

processed through the stripping equipment, or each subsequent day, measured immediately after the stripping operation is completed, shall not exceed:

(i) 2000 ppm for polyvinyl chloride dispersion resins, including latex resins, averaged separately for each type of resin; or

(ii) 400 ppm for all other polyvinyl chloride resins, including latex resins, averaged separately for each type of resin; or

(2) In polyvinyl chloride plants controlling vinyl chloride emissions with technology other than stripping or in addition to stripping, emissions of vinyl chloride to the atmosphere may not exceed:

(i) 2 g/kg (0.002 lb/lb) product from the stripper(s) [or reactor(s) if the plant has no stripper(s)] for dispersion polyvinyl chloride resins, excluding latex resins, with the product determined on a dry solids basis;

(ii) 0.4 g/kg (0.0004 lb/lb) product from the strippers [or reactor(s) if the plant has no stripper(s)] for all other polyvinyl chloride resins, including latex resins, with the product determined on a dry solids basis.

§ 61.63: Emission standard for ethylene dichloride, vinyl chloride, and polyvinyl chloride plants.

An owner or operator of an ethylene dichloride, vinyl chloride, and/or polyvinyl chloride plant shall comply with the requirements of this section.

(a) **Relief valve discharge:** Except for an emergency relief discharge, there is to be no discharge to the atmosphere from any relief valve on any equipment in vinyl chloride service. An emergency relief discharge means a discharge which could not have been avoided by taking measures to prevent the discharge. Within 10 days of any relief valve discharge, the owner or operator of the source from which the relief valve discharge occurred shall submit to the Administrator a report for writing containing information on the source, nature and cause of the discharge, the date and time of the discharge, the approximate total vinyl chloride loss during the discharge, the method used for determining the vinyl chloride loss, the action that was taken to prevent the discharge, and measures adopted to prevent future discharges.

(b) **Fugitive emission sources:**

(1) **Loading and unloading lines:** Vinyl chloride emissions from loading and unloading lines in vinyl chloride service which are opened to the atmosphere after each loading or unloading operation are to be minimized as follows:

[42 FR 29065, June 7, 1977]

(i) After each loading or unloading operation and before opening a loading or unloading line to the atmosphere, the quantity of vinyl chloride in all parts of each loading or unloading line that are to be opened to the atmosphere is to be reduced so that the parts combined contain no greater than 0.0050 m³ (176 ml) of vinyl chloride, at standard temperature and pressure; and

(ii) Any vinyl chloride removed from a loading or unloading line in accordance with paragraph (b)(1)(i) of this section is to be ducted through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm or equivalent as provided in § 61.66.

(2) **Slip gauges:** During loading or unloading operations, the vinyl chloride emissions from each slip gauge in vinyl chloride service are to be minimized by ducting any vinyl chloride discharged from the slip gauge through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm or equivalent as provided in § 61.66.

(3) **Leakage from pumps, compressors, and agitator seals:**

(i) **Rotating pumps:** Vinyl chloride emissions from seals on all rotating pumps in vinyl chloride service are to be minimized by installing sealless pumps, pumps with double mechanical seals, or equivalent as provided in § 61.66. If double mechanical seals are used, vinyl chloride emission from the seals are to be minimized by maintaining the pressure between the two seals so that any leak that occurs is into the pump; by ducting any vinyl chloride between the two seals through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm or equivalent as provided in § 61.66.

(ii) **Reciprocating pumps:** Vinyl chloride emissions from seals on all reciprocating pumps in vinyl chloride service are to be minimized by installing double outboard seals, or equivalent as provided in § 61.66. If double outboard seals are used, vinyl chloride emissions from the seals are to be minimized by maintaining the pressure between the two seals so that any leak that occurs is into the pump; by ducting any vinyl chloride between the two seals through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm; or equivalent as provided in § 61.66.

(iii) **Rotating compressors:** Vinyl chloride emissions from seals on all rotating compressors in vinyl chloride service are to be minimized by installing compressors with double mechanical seals, or equivalent as provided in § 61.66. If double mechanical seals are used, vinyl chloride emissions from the seals are to be minimized by maintaining the pressure between the two seals so that any leak that occurs is into the compressor; by ducting any vinyl chloride between the two seals through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm or equivalent as provided in § 61.66.

(iv) **Reciprocating compressors:** Vinyl chloride emissions from seals on all reciprocating compressors in vinyl chloride service are to be minimized by installing double outboard seals, or equivalent as provided in § 61.66. If double outboard seals are used, vinyl chloride emissions from the seals are to be minimized by maintaining the pressure between the two seals so that any leak that occurs is into the compressor; by ducting any vinyl chloride between the two seals through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm; or equivalent as provided in § 61.66.

(v) **Agitator seals:** Vinyl chloride emissions from seals on all agitators in vinyl chloride service are to be minimized by installing agitators with double mechanical seals, or equivalent as provided in § 61.66. If double mechanical seals are used, vinyl chloride emissions from the seals are to be minimized by maintaining the pressure between the two seals so that any leak that occurs is into the agitated vessel; by ducting any vinyl chloride between the two seals through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm or equivalent as provided in § 61.66.

(4) **Leakage from relief valves:** Vinyl chloride emissions due to leakage from each relief valve on equipment in vinyl chloride service are to be minimized by installing a rupture disk between the equipment and the relief valve, by connecting the relief valve discharge to a process line or recovery system, or equivalent as provided in § 61.66.

(5) **Manual venting of gases:** Except as provided in § 61.64(a)(3), all gases which are manually vented from equipment in vinyl chloride service are to be ducted through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm or equivalent as provided in § 61.66.

(6) **Opening of equipment:** Vinyl chloride emissions from opening of equipment (including loading or unloading lines that are not opened to the atmosphere after each loading or unloading operation) are to be minimized as follows:

(i) **Before opening any equipment for any reason,** the quantity of vinyl chloride is to be reduced so that the equipment contains no more than 2.0 percent by volume vinyl chloride or 0.0050 m³ (320 gal) of vinyl chloride, whichever is larger, at standard temperature and pressure; and

(ii) Any vinyl chloride removed from the equipment in accordance with paragraph (b)(6)(i) of this section is to be ducted through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm, or equivalent as provided in § 61.66.

(7) **Sampling:** Unused portions of samples containing at least 10 percent by weight vinyl chloride are to be returned to the process, and sampling techniques are to be such that sample containers in vinyl chloride service are purged into a closed process system.

(8) **Leak detection and elimination:** Vinyl chloride emissions due to leaks from equipment in vinyl chloride service are to be minimized by instituting and implementing a formal leak detection and elimination program. The owner or operator shall submit a description of this program to the Administrator for approval. This program is to be submitted, within 45 days of the effective date of these regulations, unless a waiver of compliance is granted under § 61.11. If a waiver of compliance is granted, the program is to be submitted on a date

scheduled by the Administrator. Approval of a program will be granted by the Administrator provided he finds:

(i) It includes a reliable and accurate vinyl chloride monitoring system for detection of major leaks and identification of the general area of the plant where a leak is located. A vinyl chloride monitoring system means a device which obtains air samples from one or more points on a continuous sequential basis and analyzes the samples with gas chromatography or, if the owner or operator assumes that all hydrocarbons measured are vinyl chloride, with infrared spectrophotometry flame ion detection, or an equivalent or alternative method.

(ii) It includes a reliable and accurate portable hydrocarbon detector to be used routinely to find small leaks and to pinpoint the major leaks indicated by the vinyl chloride monitoring system. A portable hydrocarbon detector means a device which measures hydrocarbons with a sensitivity of at least 10 ppm and is of such design and size that it can be used to measure emissions from localized points.

(iii) It provides for an acceptable calibration and maintenance schedule for the vinyl chloride monitoring system and portable hydrocarbon detector. For the vinyl chloride monitoring system, a daily span check is to be conducted with a concentration of vinyl chloride equal to the concentration defined as a leak according to paragraph (b)(8)(vi) of this section. The calibration is to be done with either:

(A) A calibration gas mixture prepared from the gases specified in sections 5.2.1 and 5.2.2 of Test Method 106 and in accordance with section 7.1 of Test Method 106, or

(B) A calibration gas cylinder standard containing the appropriate concentration of vinyl chloride. The gas composition of the calibration gas cylinder standard is to have been certified by the manufacturer. The manufacturer must have recommended a maximum shelf life for each cylinder so that the concentration does not change greater than ±5 percent from the certified value. The date of gas cylinder preparation, certified vinyl chloride concentration and recommended maximum shelf life must have been affixed to the cylinder before shipment from the manufacturer to the buyer. If a gas chromatograph is used as the vinyl chloride monitoring system, these gas mixtures may be directly used to prepare a chromatograph calibration curve as described in section 7.3 of Test Method 106. The requirements in section 5.2.3.1 and 5.2.3.2 of Test Method 106 for certification of cylinder standards and for establishment and verification of calibration standards are to be followed.

[42 FR 29005, June 7, 1977]

(iv) The location and number of points to be monitored and the frequency of monitoring provided for in the program are acceptable when they are compared with the number of pieces of equipment in vinyl chloride service and the size and physical layout of the plant.

(v) It contains an acceptable plan of action to be taken when a leak is detected.

(vi) It contains a definition of leak which is acceptable when compared with the background concentrations of vinyl chloride in the areas of the plant to be monitored by the vinyl chloride monitoring system. Measurements of background concentrations of vinyl chloride in the areas of the plant to be monitored by the vinyl chloride monitoring system are to be included with the description of the program. The definition of leak for a given plant may vary among the different areas within the plant and is also to change over time as background concentrations in the plant are reduced.

(9) *Inprocess wastewater*: Vinyl chloride emissions to the atmosphere from inprocess wastewater are to be reduced as follows:

(i) The concentration of vinyl chloride in each inprocess wastewater stream containing greater than 10 ppm vinyl chloride measured immediately as it leaves a piece of equipment and before being mixed with any other inprocess wastewater stream is to be reduced to no more than 10 ppm by weight before being mixed with any other inprocess wastewater stream which contains less than 10 ppm vinyl chloride; before being exposed to the atmosphere, before being discharged to a wastewater treatment process; or before being discharged untreated as a wastewater. This paragraph does apply to water which is used to displace vinyl chloride from equipment before it is opened to the atmosphere in accordance with § 61.64(a)(2) or paragraph (b)(6) of this section, but does not apply to water which is used to wash out equipment after the equipment has already been opened to the atmosphere in accordance with § 61.64(a)(2) or paragraph (b)(6) of this section.

(ii) Any vinyl chloride removed from the inprocess wastewater in accordance with paragraph (b)(9)(i) of this section is to be ducted through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm, or equivalent as provided in § 61.66.

(c) The requirements in paragraphs (b)(1), (b)(2), (b)(5), (b)(6), (b)(7) and (b)(8) of this section are to be incorporated into a standard operating procedure, and made available upon request for inspection by the Administrator. The standard operating procedure is to include provisions for measuring the vinyl chloride in equipment 4.75 m³ (1,250 gal) in volume for which an emission limit is prescribed in § 61.65(b)(6)(i) prior to opening the equipment and using Test Method 106, a portable hydrocarbon detector, or an equivalent or alternative method. The method of measurement is to meet the requirements in § 61.67(g)(5)(i)(A) or (g)(5)(i)(B).

[41 FR 53017, December 3, 1976]

§ 61.66 Equivalent equipment and procedures.

Upon written application from an owner or operator, the Administrator may approve use of equipment or procedures which have been demonstrated to his satisfaction to be equivalent in terms of reducing vinyl chloride emissions to the atmosphere to those prescribed for compliance with a specific paragraph of this subpart. For an existing source, any request for using an equivalent method as the initial measure of control is to be submitted to the Administrator within 30 days of the effective date. For a new source, any request for using an equivalent method is to be submitted to the Administrator with the application for approval of construction or modification required by § 61.07.

§ 61.67 Emission tests.

(a) Unless a waiver of emission testing is obtained under § 61.13, the owner or operator of a source to which this subpart applies shall test emissions from the source,

(1) Within 90 days of the effective date in the case of an existing source or a new source which has an initial startup date preceding the effective date, or

(2) Within 90 days of startup in the case of a new source, initial startup of which occurs after the effective date.

(b) The owner or operator shall provide the Administrator at least 30 days prior notice of an emission test to afford the Administrator the opportunity to have an observer present during the test.

(c) Any emission test is to be conducted while the equipment being tested is operating at the maximum production rate at which the equipment will be operated and under other relevant conditions as may be specified by the Administrator based on representative performance of the source.

(d) [Reserved]

(e) When at all possible, each sample is to be analyzed within 24 hours, but in no case in excess of 72 hours of sample collection. Vinyl chloride emissions are to be determined within 30 days after the emission test. The owner or operator shall report the determinations to the Administrator by a registered letter dispatched before the close of the next business day following the determination.

[42 FR 29005, June 7, 1977]

(f) The owner or operator shall retain at the plant and make available, upon request, for inspection by the Administrator, for a minimum of 2 years records of emission test results and other data needed to determine emissions.

(g) Unless otherwise specified, the owner or operator shall use test Test Methods in Appendix B to this part for each test as required by paragraphs (g)(1), (g)(2), (g)(3), (g)(4), and (g)(5) of this section, unless an equivalent method or an alternative method has been approved by the Administrator. If the Administrator finds reasonable grounds to dispute the results obtained by an equivalent or alternative method, he may require the use of a reference method. If the results of the reference and equivalent or alternative methods do not agree, the results obtained by the reference method prevail, and the Administrator may notify the owner or

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operator that approval of the method previously considered to be equivalent or alternative is withdrawn.

(f) Test Method 106 is to be used to determine the vinyl chloride emissions from any source for which an emission limit is prescribed in §§ 61.62(a) or (b) § 61.63(a), or §§ 61.64(a)(1), (b), (c), or (d), or from any control system to which reactor emissions are required to be ducted in § 61.64(a)(2) or to which fugitive emissions are required to be ducted in §§ 61.65(b)(1)(ii), (b)(2), (b)(5), (b)(6)(ii), or (b)(9)(ii).

(i) For each run, one sample is to be collected. The sampling site is to be at least two stack or duct diameters downstream and one half diameter upstream from any flow disturbance such as a bend, expansion, contraction, or visible flame. For a rectangular cross section an equivalent diameter is to be determined from the following equation:

$$\text{equivalent diameter} = 2\,\frac{(\text{length})\,(\text{width})}{\text{length}+\text{width}}$$

The sampling point in the duct is to be at the centroid of the cross section. The sample is to be extracted at a rate proportional to the gas velocity at the sampling point. The sample is to be taken over a minimum of one hour, and is to contain a minimum volume of 50 liters corrected to standard conditions.

(ii) Each emission test is to consist of three runs. For the purpose of determining emissions, the average of results of all runs is to apply. The average is to be computed on a time weighted basis.

(iii) For gas streams containing more than 10 percent oxygen the concentration of vinyl chloride as determined by Test Method 106 is to be corrected to 10 percent oxygen (dry basis) for determination of emissions by using the following equation:

$$C_{b\,(\text{corrected})} = C_b\,\frac{10.9}{20.9 - \text{percent } O_2}$$

where:

$C_{b\,(\text{corrected})}$ = The concentration of vinyl chloride in the exhaust gases, corrected to 10-percent oxygen.

C_b = The concentration of vinyl chloride as measured by Test Method 106.

20.9 = Percent oxygen in the ambient air at standard conditions.

10.9 = Percent oxygen in the ambient air at standard conditions, minus the 10.0-percent oxygen to which the correction is being made.

Percent O_2 = Percent oxygen in the exhaust gas as measured by Reference Method 3 in Appendix A of Part 60 of this chapter.

(iv) For those emission sources where the emission limit is prescribed in terms of mass rather than concentration, mass emissions in kg/100 kg product are to be determined by using the following equation:

$$C_{RX} = \frac{[C_b\,(2.60)\,Q\,10^{-6}]\,[100]}{Z}$$

where:

C_{RX} = kg vinyl chloride/100 kg product.

C_b = The concentration of vinyl chloride as measured by Test Method 106.

2.60 = Density of vinyl chloride at one atmosphere and 20° C in kg/m³.

Q = Volumetric flow rate in m³/hr as determined by Reference Method 2 of Appendix A to Part 60 of this chapter.

10^{-6} = Conversion factor for ppm.

Z = Production rate (kg/hr).

[42 FR 29006, June 7, 1977]

(2) Test Method 107 is to be used to determine the concentration of vinyl chloride in each inprocess wastewater stream for which an emission limit is prescribed in § 61.65(b)(9)(i).

(3) Where a stripping operation is used to attain the emission limit in § 61.64(e), emissions are to be determined using Test Method 107 as follows:

(i) The number of strippers and samples and the types and grades of resin to be sampled are to be determined by the Administrator for each individual plant at the time of the test based on the plant's operation.

(ii) Each sample is to be taken immediately following the stripping operation.

(iii) The corresponding quantity of material processed by each stripper is to be determined on a dry solids basis and by a method submitted to and approved by the Administrator.

(iv) At the prior request of the Administrator, the owner or operator shall provide duplicates of the samples required in paragraph (g)(3)(i) of this section.

(4) Where control technology other than or in addition to a stripping operation is used to attain the emission limit in § 61.64(e), emissions are to be determined as follows:

(i) Test Method 108 is to be used to determine atmospheric emissions from all of the process equipment simultaneously. The requirements of paragraph (g)(1) of this section are to be met.

(ii) Test Method 108 is to be used to determine the concentration of vinyl chloride in each inprocess wastewater stream subject to the emission limit prescribed in § 61.64(e). The mass of vinyl chloride in kg/100 kg product in each in process wastewater stream is to be determined by using the following equation:

$$C_{RX} = \frac{[C_c\,R\,10^{-6}]\,[100]}{Z}$$

where:

C_{RX} = kg vinyl chloride/100 kg product.

C_c = the concentration of vinyl chloride as measured by Test Method 107.

R = waste flow rate in l/hr, determined in accordance with a method which has been submitted to and approved by the Administrator.

10^{-6} = Conversion factor for ppm.

Z = Production rate (kg/hr), determined in accordance with a method which has been submitted and approved by the Administrator.

(5) The reactor opening loss for which an emission limit is prescribed in § 61.64(a)(3) is to be determined. The number of reactors for which the determination is to be made is to be specified by the Administrator for each individual plant at the time of the determination based on the plant's operation. For a reactor that is also used as a stripper, the determination may be made immediately following the stripping operation.

(i) Except as provided in paragraph (g)(5)(ii) of this section, the reactor opening loss is to be determined using the following equation:

$$C_b = \frac{W\,(2.60)\,(10^{-6})\,(Cb)}{YZ}$$

where:

C_b = kg vinyl chloride emissions/kg product.

W = capacity of the reactor in m³.

2.60 = Density of vinyl chloride at one atmosphere and 20° C in kg/m³.

10^{-6} = Conversion factor for ppm.

Cb = ppm by volume vinyl chloride as determined by Test Method 108 or a portable hydrocarbon detector which measures hydrocarbons with a sensitivity of at least 10 ppm.

Y = Number of batches since the reactor was last opened to the atmosphere.

Z = Average kg of polyvinyl chloride produced per batch in the number of batches since the reactor was last opened to the atmosphere.

(A) If Method 108 is used to determine the concentration of vinyl chloride (Cb), the sample is to be withdrawn at a constant rate with a probe of sufficient length to reach the vessel bottom from the manhole. Samples are to be taken for 5 minutes within 6 inches of the vessel bottom, 5 minutes near the vessel center, and 5 minutes near the vessel top.

(B) If a portable hydrocarbon detector is used to determine the concentration of vinyl chloride (Cb), a probe of sufficient length to reach the vessel bottom from the manhole is to be used to make the measurements. One measurement will be made within 6 inches of the vessel bottom, one near the vessel center and one near the vessel top. Measurements are to be made at each location until the reading is stabilized. All hydrocarbons measured are to be assumed to be vinyl chloride.

(C) The production rate of polyvinyl chloride (Z) is to be determined by a method submitted to and approved by the Administrator.

(ii) A calculation based on the number of evacuations, the vacuum involved, and the volume of gas in the reactor is hereby approved by the Administrator as an alternative method for determining reactor opening loss for postpolymerization reactors in the manufacture of bulk resins.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

§ 61.65 Emissions monitoring.

(a) A vinyl chloride monitoring system is to be used to monitor on a continuous basis, the emissions from the sources for which emission limits are prescribed in § 61.62(a) and (b), § 61.63(a), and § 61.64(a)(1), (b), (c), and (d), and for any control system to which reactor emissions are required to be ducted in §61.64(a)(2) or to which fugitive emissions are required to be ducted in §61.65 (b)(1)(ii), and (b)(2), (b)(5), (b)(6)(ii), and (b)(9)(ii).

[41 FR 53017, December 3, 1976]

(b) The vinyl chloride monitoring system is to be used to meet the requirement in paragraph (a) of this section is to be a device which obtains air samples from one or more points on a continuous sequential basis and analyzes the samples with gas chromatography or, if the owner or operator submits that all hydrocarbons measured are vinyl chloride, with a

[See. 61.62(b)]

infrared spectrophotometry, flame ion detection, or an equivalent or alternative method. The vinyl chloride monitoring system used to meet the requirements in § 61.65(b)(6)(i) may be used to meet the requirements of this section.

(c) A daily span check is to be conducted for each vinyl chloride monitoring system used. For all of the emission sources listed in paragraph (a) of this section, except the one for which an emission limit is prescribed in § 61.62(b), the daily span check is to be conducted with a concentration of vinyl chloride equal to 10 ppm. For the emission source for which an emission limit is prescribed in § 61.62(b), the daily span check is to be conducted with a concentration of vinyl chloride which is determined to be equivalent to the emission limit for that source based on the emission test required by § 61.67. The calibration is to be done with either:

[41 FR 53017, December 3, 1976]

(1) A calibration gas mixture prepared from the gases specified in sections 5.2.1 and 5.2.2 of Test Method 106 and in accordance with section 7.1 of Test Method 106, or

[42 FR 29006, June 7, 1977]

(2) A calibration gas cylinder standard containing the appropriate concentration of vinyl chloride. The gas composition of the calibration gas cylinder standard is to have been certified by the manufacturer. The manufacturer must have recommended a maximum shelf life for each cylinder so that the concentration does not change greater than ±5 percent from the certified value. The date of gas cylinder preparation, certified vinyl chloride concentration and recommended maximum shelf life must have been affixed to the cylinder before shipment from the manufacturer to the buyer. If a gas chromatograph is used as the vinyl chloride monitoring system, these gas mixtures may be directly used to prepare a chromatograph calibration curve as described in section 7.2 of Test Method 106. The requirements in sections 5.2.3.1 and 5.2.3.2 of Test Method 106 for certification of cylinder standards and for establishment and verification of calibration standards are to be followed.

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414))

[42 FR 29006, June 7, 1977]

§ 61.69 Initial report.

(a) An owner or operator of any source to which this subpart applies shall submit a statement in writing notifying the Administrator that the equipment and procedural specifications in §§ 61.65 (b)(1), (b)(2), (b)(3), (b)(4), (b)(5), (b)(6), (b)(7), and (b)(8) are being implemented.

(b)(1) In the case of an existing source or a new source which has an initial startup date preceding the effective date, the statement is to be submitted within 90 days of the effective date, unless a waiver of compliance is granted under § 61.11, along with the information required under § 61.10. If a waiver of compliance is granted, the statement is to be submitted on a date scheduled by the Administrator.

(2) In the case of a new source which did not have an initial startup date preceding the effective date, the statement is to be submitted within 90 days of the initial startup date.

(c) The statement is to contain the following information:

(1) A list of the equipment installed for compliance.

(2) A description of the physical and functional characteristics of each piece of equipment.

(3) A description of the methods which have been incorporated into the standard operating procedures for measuring or calculating the emissions for which emission limits are prescribed in §§ 61.65 (b)(1)(i) and (b)(6)(i).

(4) A statement that each piece of equipment is installed and that each piece of equipment and each procedure is being used.

§ 61.70 Emission monitoring.

(a) The owner or operator of any source to which this subpart applies shall submit to the Administrator on [illegible] and March 15 of each year a report in writing containing the information required by this section. The first semi-annual report is to be submitted following the first full 6 month reporting period after the initial report is submitted.

[41 FR 53017, December 3, 1976]

(b)(1) In the case of an existing source or a new source which has an initial startup date preceding the effective date, the first report is to be submitted within 180 days of the effective date, unless a waiver of compliance is granted under § 61.11. If a waiver of compliance is granted, the first report is to be submitted on a date scheduled by the Administrator.

(2) In the case of a new source which did not have an initial startup date preceding the effective date, the first report is to be submitted within 180 days of the initial startup date.

(c) Unless otherwise specified, the owner or operator shall use the Test Methods in Appendix B to this part to conduct emission tests as required by paragraphs (c)(2) and (c)(3) of this section, unless an equivalent or an alternative method has been approved by the Administrator. If the Administrator finds reasonable grounds to dispute the results obtained by an equivalent or alternative method, he may require the use of a reference method. If the results of the reference and equivalent or alternative methods do not agree, the results obtained by the reference method prevail, and the Administrator may notify the owner or operator that approval of the method previously considered to be equivalent or alternative is withdrawn.

(1) The owner or operator shall include in the report a record of any emissions which averaged over any 3 hour period (commencing on the hour) are [illegible] the emission limits prescribed in §§ 61.62(a) or (b), § 61.63(a), or §§ 61.64(a)(1), (b), (c), or (d), or for any control system to which reactor emissions are required to be ducted in § 61.64(a)(2) or to which fugitive emissions are required to be ducted in § 61.65 (b)(1)(ii), (b)(2), (b)(5), (b)(6)(ii), or (b)(9)(ii). The emissions are to be measured in accordance with § 61.68.

(2) In polyvinyl chloride plants for which a stripping operation is used to attain the emission level prescribed in § 61.64(c), [illegible] shall include in the report a record of the vinyl chloride content in the polyvinyl chloride resin. Test Method 107 is to be used to determine vinyl chloride content as follows:

(i) At each stripping [illegible] one representative sample of polyvinyl chloride resin is to be taken from each batch or each grade of resin immediately following the completion of the stripping operation, and identified by resin type and grade and the date and time the batch is completed. The corresponding quantity of material processed in each stripper batch is to be recorded and identified by resin type and grade and the date and time the batch is completed.

[42 FR 29006, June 7, 1977]

(ii) If continuous stripping is used, one representative sample of polyvinyl chloride resin is to be taken for each grade of resin processed or at intervals of 8 hours for each grade of resin which is being processed, whichever is more frequent. The sample is to be taken as the resin flows out of the stripper and identified by resin type and grade and the date and time the sample was taken. The corresponding quantity of material processed by each stripper over the time period representing by the sample during the eight hour period, is to be recorded and identified by resin type and grade and the date and time it represents.

(iii) The quantity of material processed by the stripper is to be determined on a dry solids basis and by a method submitted to and approved by the Administrator.

(iv) At the prior request of the Administrator, the owner or operator shall provide duplicates of the samples required in paragraphs (c)(2)(i) and (c)(2)(ii) of this section.

(v) The report to the Administrator by the owner or operator shall include the vinyl chloride content found in each sample required by paragraphs (c)(2)(i) and (c)(2)(ii) of this section, averaged separately for each type of resin, over each calendar day and weighted according to the quantity of each grade of resin processed by the stripper(s) that calendar day, according to the following equation:

$$Av_t = \frac{\sum_{i=1}^{n} Pv_i Mv_i}{Qr_t} = \frac{Pv_1 Mv_1 + Pv_2 Mv_2 + \cdots + Pv_n Mv_n}{Qr_t}$$

where:

A = 24-hour average concentration of type T_i resin in ppm (dry weight basis).

Q = Total production of type T_i resin over the 24-hour period, in kg.

T_i = Type of resin; $i = 1, 2 \ldots m$ where m is total number of resin types produced during the 24-hour period.

Mf = Concentration of vinyl chloride in one sample of grade G_i resin, in ppm.

P = Production of grade G_i resin represented by the sample, in kg.

G_i = Grade of resin; e.g., G_1, G_2 and G_3.

n = Total number of grades of resin produced during the 24-hour period.

[42 FR 29006, June 7, 1977]

(vi) The owner or operator shall retain at the source and make available for inspection by the Administrator for a minimum of 2 years records of all data needed to furnish the information required by paragraph (c)(2)(v) of this section: The records are to contain the following information:

(A) The vinyl chloride content found in all the samples required in paragraphs (c)(2)(i) and (c)(2)(ii) of this section, identified by the resin type and grade and the time and date of the sample, and

(B) The corresponding quantity of polyvinyl chloride resin processed by the stripper(s), identified by the resin type and grade and the time and date it represents.

(3) The owner or operator shall include in the report a record of the emissions from each reactor opening for which an emission limit is prescribed in § 61.64(a)(2). Emissions are to be determined in accordance with § 61.67(g)(5), except that emissions for each reactor are to be determined. For a reactor that is also used as a stripper, the determination may be made immediately following the stripping operation.

(Sec 114 of the Clean Air Act as amended (42 U.S.C. 7414)

§ 61.72 [illegible]

(a) The owner or operator of any source to which this subpart applies shall retain the following information at the source and make it available for inspection by the Administrator for a minimum of two years:

(1) A record of the leaks detected by the vinyl chloride monitoring system as required by § 61.65(b)(5), including the concentrations of vinyl chloride as measured, analyzed, and recorded by the vinyl chloride detector, the location of each measurement and the date and approximate time of each measurement.

(2) A record of the leaks detected during routine monitoring with the portable hydrocarbon detector and the actions taken to repair the leaks, as required by § 61.65(b)(5), including a brief statement explaining the location and cause of each leak detected with the portable hydrocarbon detector, the date and time of the leak, and any action taken to eliminate that leak.

[42 FR 29006, June 7, 1977]

(3) A record of emissions measured in accordance with § 61.68

[42 FR 29006, June 7, 1977]

(4) A daily operating record for each polyvinyl chloride reactor, including pressures and temperatures.

[42 FR 29006, June 7, 1977]

(Sec. 114 of the Clean Air Act as amended (42 U.S.C. 7414)

APPENDIX A

National Emission Standards for Hazardous Air Pollutants

Compliance Status Information

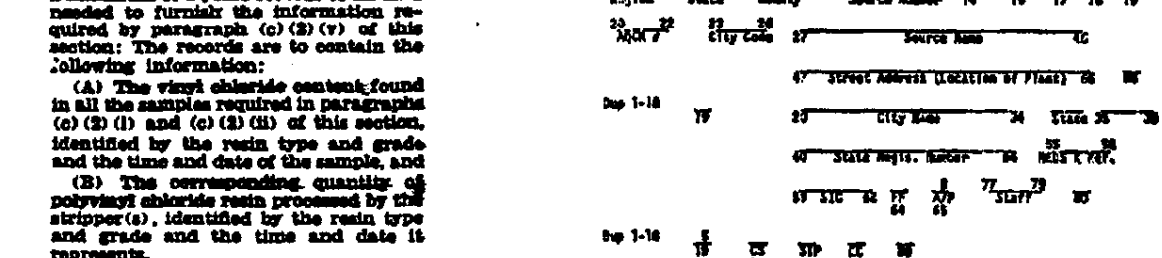
I. SOURCE REPORT

INSTRUCTIONS: Owners or operators of sources of hazardous pollutants subject to the National Emission Standards for Hazardous Air Pollutants are required to submit the information contained in Section I to the appropriate U.S. Environmental Protection Agency Regional Office prior to 90 days after the effective date of any standards or amendments which require the submission of such information.

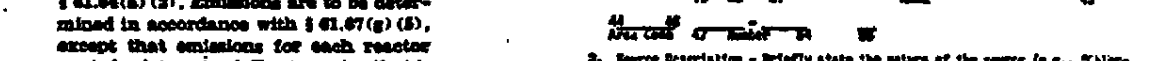
A list of regional offices is provided in §61.04.

A. SOURCE INFORMATION

1. Identification/Location - Indicate the name and address of each source.

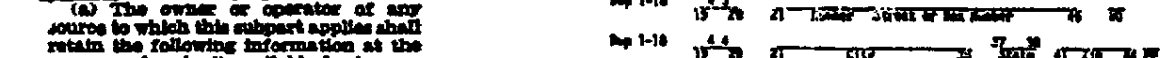


2. Contact - Indicate the name and telephone number of the owner or operator or other responsible official whom EPA may contact concerning this report.



3. Source Description - Briefly state the nature of the source (e.g., "Chlor-alkali Plant" or "Plastics Shop").

4. Alternative Mailing Address - Indicate an alternative mailing address if correspondence is to be directed to a location different than that specified above.

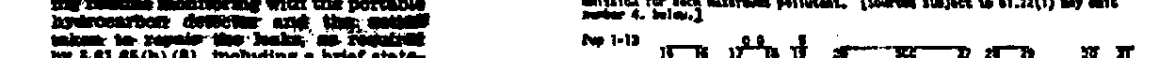


5. Compliance Status - The emissions from this source ___ can ___ cannot meet the emission limitations contained in the National Emission Standards on or prior to 90 days after the effective date of any standards or amendments which require the submission of such information.

Signature of Owner, Operator or Other Responsible Official

NOTE: If the emissions from the source will exceed State limits set by the National Emission Standards for Hazardous Air Pollutants, the source will be in violation and subject to Federal enforcement action unless granted a waiver of compliance by the Administrator of the U.S. Environmental Protection Agency. The information needed for such waivers is listed in Section II of this form.

II. PROCESS INFORMATION. Part B should be completed separately for each point of emission for each hazardous pollutant. [Sources subject to 61.12(1) may omit number 4. below.]



[Appendix A]

1. Pollutant Emitted - Indicate the type of hazardous pollutant emitted by the process. Indicate "AS" for asbestos, "BE" for beryllium, or "HG" for mercury.

32 33 Pollutant | 34 Regulation 48 | 49 EC

2. Process Description - Provide a brief description of each process (e.g., "hydrogen end box" in a mercury chlor-alkali plant, "grinding machine" in a beryllium machine shop). Use additional sheets if necessary.

50 Process Description 74 | 80

Dup 1-18 | 6 1 (19 20) | 21 50

51 79 | 80

Dup 1-18 | 6 2 (19 20) | 21 50

51 79 | 80

3. Amount of Pollutant - Indicate the average weight of the hazardous material named in Item 1 which enters the process in pounds per month (based on the previous twelve months of operation).

Dup 1-18 | 6 3 (19 20) | 21 27 | lbs./mo. 29 36 | 80

4. Control Devices

a. Indicate the type of pollution control devices, if any, used to reduce the emissions from the process (e.g., venturi scrubber, baghouse, wet cyclone) and the estimated percent of the pollutant which the device removes from the process gas stream.

Dup 1-18 | 6 4 (19 20) | PRIMARY CONTROL DEVICE: 21 43

45 Primary Device Name 64 | 66 70 Percent Removal Efficiency | 72 78

80

Dup 1-18 | 6 5 (19 20) | SECONDARY CONTROL DEVICES: 21 45

47 Secondary Device Name 64 | 66 70 Percent Removal Efficiency | % EFFIC. 72 78 | 80

b. Asbestos Emission Control Devices Only

i. If a baghouse is specified in Item 4a, give the following information:

- The air flow permeability in cubic feet per minute per square foot of fabric area.

  Air flow permeability = _____ cfm/ft^2

- The pressure drop in inches water gauge across the filter at which the baghouse is operated.

  Operating pressure drop = _____ inches w.g.

- If the baghouse material contains synthetic fill yarn, check whether this material is / / spun / / or not spun.

- If the baghouse utilizes a felted fabric, give the minimum thickness in inches and the density in ounces per square yard.

  Thickness = _____ inches Density = _____ oz/yd^2

ii. If a wet collection device is specified in Item 4a, give the designed unit contacting energy in inches water gauge.

- Unit contacting energy = _____ inches w.g.

C. DISPOSAL OF ASBESTOS-CONTAINING WASTES. Part C should be completed separately for each asbestos-containing waste generation operation arising from sources subject to §61.22(a), (c), (e), and (h).

Dup 1-13 | 14 15 | 0 0 (17 18) | 5 (19) | 20 SCC 27 | 28 29 NEDS X Ref | 30 CS | 31 SIP

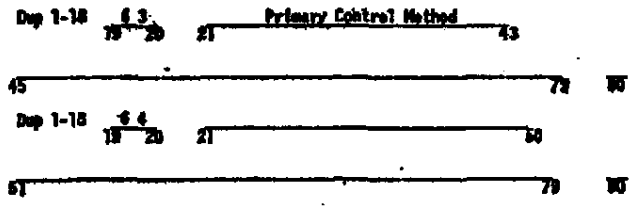
A S 32 33 Pollutant | 34 Regulation 48 | 49 EC

1. Waste Generation - Provide a brief description of each process that generates asbestos-containing waste (e.g. disposal of control device wastes).

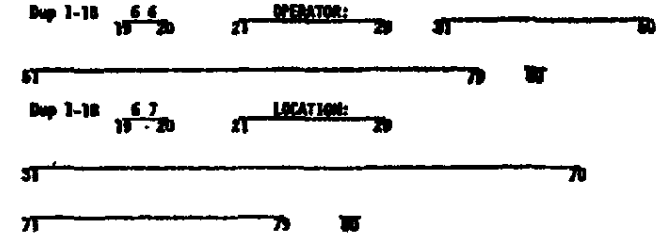
2. Asbestos Concentration - Indicate the average percentage asbestos content of these materials.

3. Amount of Wastes - Indicate the average weight of asbestos-containing wastes disposed of, measured in kg/day.

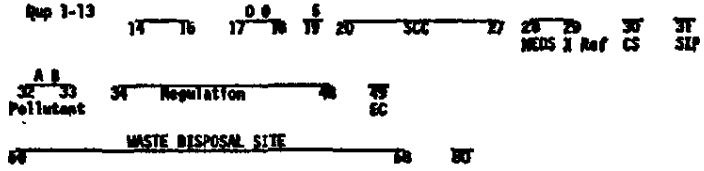
4. Control Methods - Indicate the emission control methods used in all stages of waste disposal, from collection, processing, and packaging to transporting and deposition.



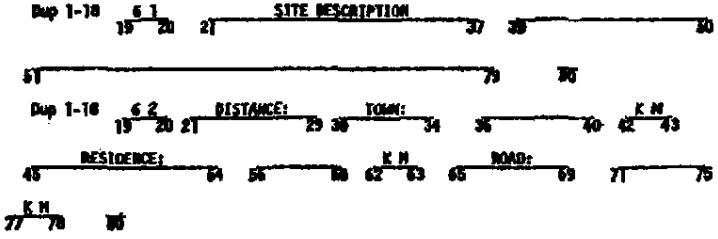
5. Waste Disposal - Indicate the type of disposal site (sanitary landfill, open, covered) or incineration site (municipal, private) where the waste is disposed of and who operates the site (company, private, municipal). State the name and location of the site (closest city or town, county, state).



B. WASTE DISPOSAL SITES. Part B should be completed separately for each asbestos waste disposal site subject to section 61.22(1).



1. Description - Provide a brief description of the site, including its size and configuration, and the distance to the closest city or town, closest residence, and closest primary road.



2. Inactivation - After the site is inactivated, indicate the method or methods used to comply with the standard and send a list of the actions that will be undertaken to maintain the inactivated site.

Dup 1-16 6 8 COMPLIANCE METHOD/INACTIVE SITE:
19 20 21 52

54 79 80

II. WAIVER REQUESTS

A. WAIVER OF COMPLIANCE. Owners or operators of sources unable to operate in compliance with the National Emission Standards for Hazardous Air Pollutants prior to 90 days after the effective date of any standards or amendments which require the submission of such information may request a waiver of compliance from the Administrator of the U.S. Environmental Protection Agency for the time period necessary to install appropriate control devices or make modifications to achieve compliance. The Administrator may grant a waiver of compliance with the standard for a period not exceeding two years from the effective date of the hazardous pollutant standards, if he finds that such period is necessary for the installation of controls and that steps will be taken during the period of the waiver to assure that the health of persons will be protected from imminent endangerment.

The report information provided in Section I must accompany this application. Applications should be sent to the appropriate EPA regional office.

1. Processes Involved - Indicate the process or processes emitting hazardous pollutants to which emission controls are to be applied.

2. Controls

a. Describe the proposed type of control device to be added or modification to be made to the process to reduce the emissions of hazardous pollutants to an acceptable level. (Use additional sheets if necessary.)

b. Describe the measures that will be taken during the waiver period to assure that the health of persons will be protected from imminent endangerment. (Use additional sheets if necessary.)

3. Increments of Progress - Specify the dates by which the following increments of progress will be met.

Date by which contracts for emission control systems or process modifications will be awarded; or date by which orders will be issued for the purchase of the component parts to accomplish emission control or process modification.

Dup 1-16 0 1 7
17 19 53 54 55 60 61 MO/DY/YR 66 80

Date of initiation of on-site construction or installation of emission control equipment or process change.

Dup 1-16 0 2 7
17 19 53 54 55 60 61 MO/DY/YR 66 80

- Date by which on-site construction or installation of emission control equipment or process modification is to be completed.

Dup 1-16 0 3 7
17 19 53 54 55 60 61 MO/DY/YR 66 80

- Date by which final compliance is to be achieved.

Dup 1-16 0 4 7
17 19 53 54 55 60 61 MO/DY/YR 66 80

B. WAIVER OF EMISSION TESTS. A waiver of emission testing may be granted to owners or operators of sources of beryllium or mercury pollutants if, in the judgment of the Administrator of the Environmental Protection Agency the emissions from the source comply with the appropriate standard or if the owners or operators of the source have requested a waiver of compliance or have been granted a waiver of compliance.

This application should accompany the report information provided in Section I.

1. Reason - State the reasons for requesting a waiver of emission testing. If the reason stated is that the emissions from the source are within the prescribed limits, documentation of this condition must be attached.

Date

Signature of the owner or operator

[40 FR 48292, October 14, 1975]

METHOD 105. METHOD FOR DETERMINATION OF MERCURY IN WASTEWATER TREATMENT PLANT SEWAGE SLUDGES

1. *Principle and applicability.* 1.1 Principle—A weighed portion of the sewage sludge sample is digested in aqua regia for 2 minutes at 95°C, followed by oxidation with potassium permanganate. Mercury in the digested sample is then measured by the conventional spectrophotometer cold vapor technique. An alternative digestion involving the use of an autoclave is described in paragraph 4.5.2 of this method.

1.2 Applicability—This method is applicable for the determination of total organic and inorganic mercury content in sewage sludges, soils, sediments, and bottom-type materials. The normal range of this method is 0.2 to 5 μg/g. The range may be extended above or below the normal range by increasing or decreasing sample size and through instrument and recorder control.

2. *Apparatus.* 2.1 Analysis—The conventional cold vapor technique(5) is used to analyse the sample.

2.1.1 Atomic Absorption Spectrophotometer[1]—Any atomic absorption unit having an open sample presentation area in which to mount the absorption cell is suitable. Instrument settings recommended by the particular manufacturer should be followed.

2.1.2 Mercury Hollow Cathode Lamp—Westinghouse WL-22847, argon filled, or equivalent.

2.1.3 Recorder—Any multirange, variable-speed recorder that is compatible with the UV detection system is suitable.

2.1.4 Absorption Cell—Standard spectrophotometer cells 10 cm long, having quartz end windows may be used. Suitable cells may be constructed from plexiglass tubing, 2.5 cm O.D. x 11.4 cm (ca. 1" O.D. x 4½"). The ends are ground perpendicular to the longitudinal axis, and quartz windows [2.5 cm diameter x 0.16 cm thickness (ca. 1" diameter x 1/16" thickness)] are cemented in place. Gas inlet and outlet ports [also of plexiglass but 0.6 cm O.D. (ca. ¼" O.D.)] are attached approximately 1.3 cm (½") from each end. The cell is strapped to a burner for support and aligned in the light beam to give the maximum transmittance. NOTE: Two 5.1 cm x 5.1 cm (ca. 2" x 2") cards with 2.5 cm (ca. 1") diameter holes may be placed over each end of the cell to assist in positioning the cell for maximum transmittance.

2.1.5 Air Pump—Any peristaltic pump capable of delivering 1 liter of air per minute may be used. A Masterflex pump with electronic speed control has been found to be satisfactory. (Regulated compressed air can be used in an open one-pass system.)

2.1.6 Flowmeter—Capable of measuring an air flow of 1 liter per minute.

2.1.7 Aeration Tubing—Tygon tubing is used for passage of the mercury vapor from the sample bottle to the absorption cell and return. Straight glass tubing terminating in a coarse porous frit is used for sparging air into the sample.

2.1.8 Drying Tube—15 cm long x 1.9 cm diameter (ca. 6" long x ¾" diameter) tube containing 20 grams of the desiccant magnesium perchlorate. The apparatus is assembled as shown in Figure 105-1. In place of the magnesium perchlorate drying tube, a small reading lamp with 60W bulb may be used to prevent condensation of moisture inside the cell. The lamp is positioned so as not to interfere with the measurement and to shine on the absorption cell maintaining the air temperature about 5°C above ambient.

3. *Reagents.* 3.1 Analysis.

3.1.1 Aqua Regia—Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

3.1.2 Sulfuric Acid, 0.5N—Dilute 14.0 ml of concentrated sulfuric acid to 1.0 liter.

3.1.3 Stannous Sulfate—Add 25 g stannous sulfate to 250 ml of 0.5N sulfuric acid. This mixture is a suspension and should be stirred continuously during use. Stannous chloride may be used in place of the stannous sulfate.

3.1.4 Sodium Chloride—Hydroxylamine Sulfate Solution—Dissolve 12 grams of sodium chloride and 12 grams of hydroxylamine sulfate in distilled water and dilute to 100 ml. Hydroxylamine hydrochloride may be used in place of the hydroxylamine sulfate.

3.1.5 Potassium Permanganate—5% solution, w/v. Dissolve 5 grams of potassium permanganate in 100 ml of distilled water.

3.1.6 Stock Mercury Solution—Dissolve 0.1354 grams of reagent grade mercuric chloride (Assay >95%) in 75 ml. of distilled water. Add 10 ml of concentrated nitric acid and adjust the volume to 100.0 ml. 1 ml = 1 mg Hg.

3.1.7 Working Mercury Solution—Make successive dilutions of the stock mercury solution to obtain a working standard containing 0.1 μg per ml. This working standard and the dilutions of the stock mercury solution should be prepared fresh daily. Acidity of the working standard should be maintained at 0.15% nitric acid. This acid should be added to the flask as needed before the addition of the aliquot. Mercuric solutions should not be prepared in plastic containers.

4. *Procedures.* Samples for mercury analysis are subject to contamination from a variety of sources. Extreme care must be taken to prevent contamination. Certain interferences may occur during the analysis procedures. Extreme caution must be taken to avoid inhalation of mercury.

4.1 Sample Handling and Preservation.

4.1.1 Because of the extreme sensitivity of the analytical procedure and the omnipresence of mercury, care must be taken to avoid extraneous contamination. Sampling devices, sample containers, and reagents should be ascertained to be free of significant amounts of mercury; the sample should not be exposed to any condition in the laboratory that may result in contact or airborne mercury contamination. Sample containers to be used for collection and shipment of mercury samples should be properly cleaned before use. These should be rinsed with at least 20% v/v HNO_3, followed by distilled water.

4.1.2 While the sample may be analyzed without drying, it has been found to be more convenient to analyse a dry sample. Moisture may be driven off in a drying oven at a temperature of 60°C. No significant mercury losses have been observed by using this drying step. The dry sample should be pulverized and thoroughly mixed before the aliquot is weighed.

4.2 Interferences.

4.2.1 Interferences that may occur in sludge samples are sulfides, high copper, high chlorides, etc. A discussion of possible interferences and suggested preventative measures to be taken is given in Reference (6) (7).

4.2.2 Volatile materials which absorb at the 253.7 nm will cause a positive interference. In order to remove any interfering volatile materials, the dead air space in the BOD bottle should be purged with nitrogen before the addition of stannous sulfate.

4.3 Handling Sample Mercury Vapors After Analysis.

4.3.1 Because of the toxic nature of mercury vapor, precaution must be taken to avoid its inhalation. Therefore, a bypass should be included in the analysis system to either vent the mercury vapor into an exhaust hood or pass the vapor through some absorbing media, such as:

(a) equal volumes of 0.1N $KMnO_4$ and 10% H_2SO_4;

(b) 0.25% iodine in a 3% KI solution.

A specially treated charcoal that will absorb mercury vapor is also available from Barnebey and Cheney, E. 6th Ave. and North Cassidy St., Columbus, Ohio 43219, Catalog No. 580-12 or No. 580-22.[2]

4.4 Calibration.

4.4.1 Transfer 0, 0.5, 1.0, 2.0, 5.0 and 10 ml aliquots of the working mercury solution containing 0 to 1.0 μg of mercury to a series of 300-ml BOD bottles. Add enough distilled water to each bottle to make a total volume of 10 ml. Add 5 ml of aqua regia and heat 2 minutes in a water bath at 95°C. Allow the sample to cool and add 50 ml distilled water and 15 ml of $KMnO_4$ solution to each bottle and return to the water bath for 30 minutes. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Add 50 ml of distilled water. Treating each bottle individually, add 5 ml of stannous sulfate solution and immediately attach the bottle to the aeration apparatus. At this point, the sample is allowed to stand quitely without manual agitation. The circulating pump, which has previously been adjusted to a rate of 1 liter per minute, is allowed to run continuously. The absorbance, as exhibited either on the spectrophotometer or the recorder, will increase and reach maximum within 30 seconds. As soon as the recorder pen levels off, approximately 1 minute, open the bypass valve and continue the aeration until the absorbance returns to its minimum value. Close the bypass valve, remove the fritted tubing from the BOD bottle and continue the aeration. Proceed with the standards and construct a standard curve by plotting peak height versus micrograms of mercury.

4.5 Analysis.

4.5.1 Weigh triplicate 0.2g±0.001 g portions of dry sample and place in bottom of a BOD bottle. Add 5 ml of distilled water and 5 ml of aqua regia. Heat 2 minutes in a water bath at 95°C. Cool and add 50 ml distilled water and 15 ml potassium permanganate solution to each sample bottle. Mix thoroughly and place in the water bath for 30 minutes at 95°C. Cool and add 6 ml of sodium chloride-hydroxylamine sulfate to reduce the excess permanganate. Add 55 ml of distilled water. Treating each bottle individually, add 5 ml of stannous sulfate and immediately attach the bottle to the aeration apparatus. With each sample, continue as described in paragraph 4.4.1 of this method.

4.5.2 An alternative digestion procedure using an autoclave may also be used. In this method 5 ml of concentrated H_2SO_4 and 2 ml of concentrated HNO_3 are added to the 0.2 grams of sample. 5 ml of saturated $KMnO_4$ solution are added and the bottle is covered with a piece of aluminum foil. The samples are autoclaved at 121°C and 2.1 kg/cm² (ca. 15 psig) for 15 minutes. Cool, make up to a volume of 100 ml with distilled water, and add 6 ml of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate. Purge the dead air space and continue as described in paragraph 4.4.1 of this method.

[1] Instruments designed specifically for the measurement of mercury using the cold vapor technique are commercially available and may be substituted for the atomic absorption spectrophotometer.

[2] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

[Appendix B]

5. *Calculation.* 5.1 Measure the peak height of the unknown from the chart and read the mercury value from the standard curve.

5.2 Calculate the mercury concentration in the sample by the formula:

$$\mu g\ Hg/gm = \frac{\mu g\ Hg \text{ in the aliquot}}{\text{wt. of the aliquot in g}}$$

5.3 Report mercury concentrations as follows: Below 0.1 μg/g; between 0.1 and 1 μg/g. to the nearest 0.01 μg/g; between 1 and 10 μg/g. to nearest 0.1 μg; above 10 μg/g. to nearest μg.

6. *Precision and accuracy.* 6.1 According to the provisional method in reference number 5, the following standard deviations on replicate sediment samples have been recorded at the indicated levels: 0.29 μg/g ± 0.08 and 0.85 μg/g ± 0.33. Recovery of mercury at these levels, added as methyl mercuric chloride, was 97 and 94%, respectively.

7. *References.*

1. Bishop; J. N. "Mercury in Sediments," Ontario Water Resources Comm., Toronto, Ontario, Canada, 1971.

2. Saima, M. Private communication, EPA Cal/Nev Basin Office, Alameda, California.

3. Hatch, W. R., and Ott, W. L. "Determination of Sub-Microgram Quantities of Mercury by Atomic Absorption Spectrophotometry," Ana. Chem. 40, 2085 (1968).

4. Bradenberger, H. and Bader, H. "The Determination of Nanogram Levels of Mercury in Solution by a Flameless Atomic Absorption Technique," Atomic Absorption Newsletter 6, 101 (1967).

5. Analytical Quality Control Laboratory (AQCL), Environmental Protection Agency, Cincinnati, Ohio, "Mercury in Sediment (Cold Vapor Technique)," Provisional Method, April 1973.

6. Kopp, J. F., Longbottom, M. C. and Lobring, L. B. "Cold Vapor Method for Determining Mercury," Journal AWWA, 64, 1 (1972), pp. 20–25.

7. "Manual of Methods for Chemical Analysis of Water and Wastes," Environmental Protection Agency, EPA–625/3–74–003, pp. 118–138.

[40 FR 48292, October 14, 1975]

Method 106—Determination of Vinyl Chloride From Stationary Sources

[41 FR 46559, October 21, 1976]

Introduction

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph, nor by those who are unfamiliar with source sampling, as there are many details that are beyond the scope of this presentation. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, a carcinogen.

1. Principle and Applicability.

1.1 An integrated bag sample of stack gas containing vinyl chloride (chloroethene) is subjected to chromatographic analysis, using a flame ionization detector.

[42 FR 29005, June 7, 1977]

1.2 The method is applicable to the measurement of vinyl chloride in stack gases from ethylene dichloride, vinyl chloride and polyvinyl chloride manufacturing processes, except where the vinyl chloride is contained in particulate matter.

2. Range and Sensitivity.

The lower limit of detection will vary according to the chromatograph used. Values reported include 1×10^{-9} mg and 4×10^{-9} mg.

[1] Mention of trade names on specific products does not constitute endorsement by the Environmental Protection Agency.

3. *Interferences.* Acetaldehyde, which can occur in some vinyl chloride sources, will interfere with the vinyl chloride peak from the Chromasorb 102[1] column. See sections 4.2.2 and 5.4. If resolution of the vinyl chloride peak is still not satisfactory for a particular sample, then chromatograph parameters can be further altered with prior approval of the Administrator. If alteration of the chromatograph parameters fails to resolve the vinyl chloride peak, then supplemental confirmation of the vinyl chloride peak through an absolute analytical techniqus, such as mass spectroscopy, must be performed.

[42 FR 29005, June 7, 1977]

4. Apparatus.

4.1 Sampling (Figure 106–1).

4.1.1 Probe—Stainless steel, Pyrex glass, or Teflon tubing according to stack temperature, each equipped with a glass wool plug to remove particulate matter.

4.1.2 Sample line—Teflon, 6.4 mm outside diameter, of sufficient length to connect probe to bag. A new unused piece is employed for each series of bag samples that constitutes an emission test.

4.1.3 Male (2) and female (2) stainless steel quick-connects, with ball checks (one pair without) located as shown in Figure 106–1.

[42 FR 29005, June 7, 1977]

4.1.4 Tedlar bags; 100 liter capacity—To contain sample. Tyflon bags are not acceptable. Aluminized Mylar bags may be used, provided that the samples are analyzed within 24 hours of collection.

4.1.5 Rigid leakproof containers for 4.1.4, with covering to protect contents from sunlight.

4.1.6 Needle valve—To adjust sample flow rate.

4.1.7 Pump—Leak-free. Minimum capacity 2 liters per minute.

4.1.8 Charcoal tube—To prevent admission of vinyl chloride to atmosphere in vicinity of samplers

4.1.9 Flow meter—For observing sample flow rate; capable of measuring a flow range from 0.10 to 1.00 liter per minute.

4.1.10 *Connecting tubing.* Teflon, 6.4 mm outside diameter, to assemble sample train (Figure 106–1).

[42 FR 29005, June 7, 1977]

4.1.11 Pitot tube—Type S (or equivalent), attached to the probe so that the sampling flow rate can be regulated proportional to the stack gas velocity.

4.2 Sample recovery.

4.2.1 Tubing—Teflon, 6.4 mm outside diameter, to connect bag to gas chromatograph sample loop. A new unused piece is employed for each series of bag samples that constitutes an emission test, and is to be discarded upon conclusion of analysis of those bags.

4.3 Analysis.

4.3.1 Gas chromatograph—With flame ionization detector, potentiometric strip chart recorder and 1.0 to 5.0 ml heated sampling loop in automatic sample valve.

4.3.2 *Chromatographic column.* Stainless steel, 2 m × 2.2 mm, containing 80/100 mesh Chromasorb 102. A secondary column of OS SP–96, 20 percent on 80/86 mesh AW Chromasorb P, stainless steel, 2 m × 2.2 mm or Porapak T, 80/100 mesh, stainless steel, 1 m × 2.2 mm is required if acetaldehyde is present. If used, a secondary column is placed after the Chromasorb 102 column. The combined columns should then be operated at 120° C.

[42 FR 29005, June 7, 1977]

4.3.3 Flow meters (2)—Rotameter type, 0 to 100 ml/min capacity, with flow control valves.

4.3.4 Gas regulators—For required gas cylinders.

4.3.5 Thermometer—Accurate to one degree centigrade, to measure temperature of heated sample loop at time of sample injection.

4.3.6 Barometer—Accurate to 5 mm Hg, to measure atmospheric pressure around gas chromatograph during sample analysis.

4.3.7 Pump—Leak-free. Minimum capacity 100 ml/min.

4.4 Calibration.

4.4.1 Tubing—Teflon, 6.4 mm outside diameter, separate pieces marked for each calibration concentration.

4.4.2 Tedlar bags—Sixteen-inch square size, separate bag marked for each calibration concentration.

4.4.3 Syringe—0.5 ml, gas tight.

4.4.4 Syringe—50 ml, gas tight.

4.4.5 Flow meter—Rotameter type, 0 to 1000 ml/min range accurate to ±1%, to meter nitrogen in preparation of standard gas mixtures.

4.4.6 Stop watch—Of known accuracy, to time gas flow in preparation of standard gas mixtures.

5. Reagents. It is necessary that all reagents be of chromatographic grade.

5.1 Analysis.

5.1.1 Helium gas or nitrogen gas.—Zero grade, for chromatographic carrier gas.

5.1.2 Hydrogen gas—Zero grade.

5.1.3 Oxygen gas, or Air, as required by the detector—Zero grade.

5.2 *Calibration.* Use one of the following options: either 5.2.1 and 5.2.2, or 5.2.3.

5.2.1 *Vinyl chloride, 99.9+ percent.* Pure vinyl chloride gas certified by the manufacturer to contain a minimum of 99.9 percent vinyl chloride for use in the preparation of standard gas mixtures in Section 7.1. If the gas manufacturer maintains a bulk cylinder supply of 99.9+ percent vinyl chloride, the certification analysis may have been performed on this supply rather than on each gas cylinder prepared from this bulk supply. The date of gas cylinder preparation and the certified analysis must have been affixed to the cylinder before shipment from the gas manufacturer to the buyer.

5.2.2 *Nitrogen gas.* Zero grade, for preparation of standard gas mixtures.

5.2.3 *Cylinder standards* (3). Gas mixture standards (50, 10, and 5 ppm vinyl chloride in nitrogen cylinders) for which the gas composition has been certified by the manufacturer. The manufacturer must have recommended a maximum shelf life for each cylinder so that the concentration does not change greater than ±5 percent from the certified value. The date of gas cylinder preparation, certified vinyl chloride concentration and recommended maximum shelf life must have been affixed to the cylinder before shipment from the gas manufacturer to the buyer. These gas mixture standards may be directly used to prepare a chromatograph calibration curve as described in section 7.3.

5.2.3.1 *Cylinder standards certification.* The concentration of vinyl chloride in nitrogen in each cylinder must have been certified by the manufacturer by a direct analysis of each cylinder using an analytical procedure that the manufacturer had calibrated on the day of cylinder analysis. The calibration of the analytical procedure shall, as a minimum, have utilized a three-point calibration curve. It is recommended that the manufacturer maintain two calibration standards and use these standards in the following way: (1) a high concentration standard (between 50 and 100 ppm) for preparation of a calibration curve by an appropriate dilution technique; (2) a low concentration standard (between 5 and 10 ppm) for verification of the dilution technique used.

5.2.3.2 *Establishment and verification of calibration standards.* The concentration of each calibration standard must have been established by the manufacturer using

[Appendix B]

reliable procedures. Additionally, each calibration standard must have been verified by the manufacturer by one of the following procedures, and the agreement between the initially determined concentration value and the verification concentration value must be within ± 5 percent: (1) verification value determined by comparison with a calibrated vinyl chloride permeation tube, (2) verification value determined by comparison with a gas mixture prepared in accordance with the procedure described in section 7.1 and using 99.9+ percent vinyle chloride, or (3) verification value obtained by having the calibration standard analyzed by the National Bureau of Standards. All calibration standards must be renewed on a time interval consistent with the shelf life of the cylinder standards sold.

[42 FR 29005, June 7, 1977]

6. Procedure.

6.1 Sampling. Assemble the sample train as in Figure 106–1. Perform a bag leak check according to Section 7.4. Observe that all connections between the bag and the probe are tight. Place the end of the probe at the centroid of the stack and start the pump with the needle valve adjusted to yield a flow of 0.5 lpm. After a period of time sufficient to purge the line several times has elapsed, connect the vacuum line to the bag and evacuate the bag until the rotameter indicates no flow. Then reposition the sample and vacuum lines and begin the actual sampling, keeping the rate proportional to the stack velocity. Direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Protect the bag container from sunlight.

6.2 *Sample storage.* Sample bags must be kept out of direct sunlight. When at all possible analysis is to be performed within 24 hours, but in no case in excess of 72 hours of sample collection.

[42 FR 29005, June 7, 1977]

6.3 Sample recovery. With a piece of Teflon tubing identified for that bag, connect a bag inlet valve to the gas chromatograph sample valve. Switch the valve to withdraw gas from the bag through the sample loop. Plumb the equipment so the sample gas passes from the sample valve to the leak-free pump, and then to a charcoal tube, followed by a 0–100 ml/min rotameter with flow control valve.

6.4 Analysis. Set the column temperature to 100° C the detector temperature to 150° C, and the sample loop temperature to 70° C. When optimum hydrogen and oxygen flow rates have been determined verify and maintain these flow rates during all chromatograph operations. Using zero helium or nitrogen as the carrier gas, establish a flow rate in the range consistent with the manufacturer's requirements for satisfactory detector operation. A flow rate of approximately 40 ml/min should produce adequate separations. Observe the base line periodically and determine that the noise level has stabilized and that base line drift has ceased. Purge the sample loop for thirty seconds at the rate of 100 ml/min, then activate the sample valve. Record the injection time (the position of the pen on the chart at the time of sample injection), the sample number, the sample loop temperature, the column temperature, carrier gas flow rate, chart speed and the attenuator setting. Record the laboratory pressure. From the chart, select the peak having the retention time corresponding to vinyl chloride, as determined in Section 7.2. Measure the peak area, A_m, by use of a disc integrator or a planimeter. Measure the peak height, Hm. Record Am, Hm and the retention time. Repeat the injection at least two times or until two consecutive vinyl chloride peaks do not vary in area more than 5%. The average value for these two areas will be used to compute the bag concentration.

Compare the ratio of H_m to A_m for the vinyl chloride sample with the same ratio for the standard peak which is closest in height. As a guideline, if these ratios differ by more than 10%, the vinyl chloride peak may not be pure (possibly acetaldehyde is present) and the secondary column should be employed (see Section 4.3.3).

[41 FR 53017, December 3, 1976]

6.5 Measure the ambient temperature and barometric pressure near the bag. (Assume the relative humidity to be 100 percent.) From a water saturation vapor pressure table, determine the record and water vapor content of the bag.

7. Calibration and Standards.

7.1 *Preparation of vinyl chloride standard gas mixtures.* Evacuate a sixteen-inch square Tedlar bag that has passed a leak check (described in Section 7.4) and meter in 5 liters of nitrogen. While the bag is filling, use the 0.5 ml syringe to inject 250ul of 99.9+ percent vinyl chloride through the wall of the bag. Upon withdrawing the syringe needle, immediately cover the resulting hole with a piece of adhesive tape. The bag now contains a vinyl chloride concentration of 50 ppm. In a like manner use the other syringe to prepare gas mixtures having 10 and 5 ppm vinyl chloride concentrations. Place each bag on a smooth surface and alternately depress opposite sides of the bag 50 times to further mix the gases. These gas mixture standards may be used for 10 days from the date of preparation, after which time preparation of new gas mixtures is required. (CAUTION.—Contamination may be a problem when a bag is reused if the new gas mixture standard contains a lower concentration than the previous gas mixture standard did.)

[42 FR 29005, June 7, 1977]

7.2 Determination of vinyl chloride retention time. This section can be performed simultaneously with Section 7.3. Establish chromatograph conditions identical with those in Section 6.3, above. Set attenuator to X 1 position. Flush the sampling loop with zero helium or nitrogen and activate the sample valve. Record the injection time, the sample loop temperature, the column temperature, the carrier gas flow rate, the chart speed and the attenuator setting. Record peaks and detector responses that occur in the absence of vinyl chloride. Maintain conditions. With the equipment plumbing arranged identically to Section 6.3, flush the sample loop for 30 seconds at the rate of 100 ml/min with one of the vinyl chloride calibration mixtures and activate the sample valve. Record the injection time. Select the peak that corresponds to vinyl chloride. Measure the distance on the chart from the injection time to the time at which the peak maximum occurs. This quantity, divided by the chart speed, is defined as the retention time. Record.

7.3 *Preparation of chromatograph calibration curve.* Make a gas chromatographic measurement of each gas mixture standard (described in section 5.3.2 or 7.1) using conditions identical with those listed in sections 6.3 and 6.4. Flush the sampling loop for 30 seconds at the rate of 100 ml/min with each standard gas mixture and activate the sample valve. Record C_c, the concentration of vinyl chloride injected, the attenuator setting, chart speed, peak area, sample loop temperature, column temperature, carrier gas flow rate, and retention time. Record the laboratory pressure. Calculate A_c, the peak area multiplied by the attenuator setting. Repeat until two injection areas are within 5 percent, then plot these points v. C_c. When the other concentrations have been plotted, draw a smooth curve through the points. Perform calibration daily, or before and after each set of bag samples, whichever is more frequent.

[42 FR 29005, June 7, 1977]

7.4 Bag leak checks. While performance of this section is required subsequent to bag use, it is also advised that it be performed prior to bag use. After each use, make sure a bag did not develop leaks as follows. To leak check, connect a water manometer and pressurize the bag to 5–10 cm H_2O (2–4 in H_2O). Allow to stand for 10 minutes. Any displacement in the water manometer indicates a leak. Also check the rigid container for leaks in this manner.

(NOTE: An alternative leak check method is to pressurize the bag to 5–10 cm H_2O or 2–4 in. H_2O and allow to stand overnight. A deflated bag indicates a leak.) For each sample bag in its rigid container, place a rotameter in-line between the bag and the pump inlet. Evacuate the bag. Failure of the rotameter to register zero flow when the bag appears to be empty indicates a leak.

8. Calculations.

8.1 Determine the sample peak area as follows:

$$A_c = A_m A_f$$

Equation 106–1

where:

A_c = The sample peak area.
A_m = The measured peak area.
A_f = The attenuation factor.

8.2 Vinyl chloride concentrations. From the calibration curve described in Section 7.3, above, select the value of C_c that corresponds to A_c, the sample peak area. Calculate C_b as follows:

$$C_b = \frac{C_c P_r T_f}{P_f T_r (1 - B_{ws})}$$

Equation 106–2

Where:

B_{ws} = The water vapor content of the bag sample, as analyzed.
C_b = The concentration of vinyl chloride in the bag sample in ppm.
C_c = The concentration of vinyl chloride indicated by the gas chromatograph, in ppm.
P_r = The reference pressure, the laboratory pressure recorded during calibration, mm Hg.
T_f = The sample loop temperature, on the absolute scale at the time of analysis, °K.
P_f = The laboratory pressure at time of analysis, mm Hg.
T_r = The reference temperature, the sample loop temperature recorded during calibration, °K.

[Appendix B]

9. References.
1. Brown, D. W., Loy, E. W. and Stephenson, M. E. "Vinyl Chloride Monitoring Near the B. F. Goodrich Chemical Company in Louisville, Kentucky." Region IV, U.S. Environmental Protection Agency, Surveillance and Analysis Division, Athens, Georgia, June 24, 1974.

2. "Evaluation of A Collection and Analytical Procedure for Vinyl Chloride in Air," by G. D. Clayton and Associates, December 13, 1974. EPA Contract No. 68-02-1408, Task Order No. 2, EPA Report #N. TS-VCL-1.

3. "Standardization of Stationary Source Emission Method for Vinyl Chloride," by Midwest Research Institute, 1976. EPA Contract No. 68-02-1098, Task Order No. 7.

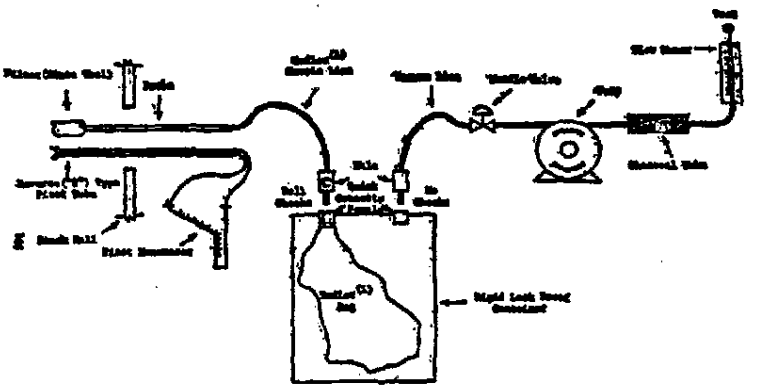


Figure 204-1. Integrated bag sampling train.

[1] Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

Appendix 107—Determination of Vinyl Chloride Content of Inprocess Wastewater Samples, and Vinyl Chloride Content of Polyvinyl Chloride Resin, Slurry, Wet Cake, and Latex Samples

[41 FR 46559, October 20, 1976]

Introduction

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph, nor by those who are unfamiliar with sampling, as there are many details that are beyond the scope of this presentation. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, a carcinogen.

1. Principle and Applicability.

1.1 The basis for this method relates to the vapor equilibrium which is established between RVCM, PVC, resin, water, and air in a closed system. It has been demonstrated that the RVCM in a PVC resin will equilibrate in a closed vessel quite rapidly, provided that the temperature of the PVC resin is maintained above the glass transition temperature of that specific resin.

1.2 This procedure is suitable for determining the vinyl chloride monomer (VCM) content of inprocess wastewater samples, and the residual vinyl chloride monomer (RVCM) content of polyvinyl chloride (PVC) resins, wet cake, slurry, and latex samples. It cannot be used for polymer in fused forms, such as sheet or cubes. If a resolution of the vinyl chloride peak is not satisfactory for a particular sample, then chromatograph parameters may be altered provided that the precision and reproducibility of the analysis of vinyl chloride cylinder standards are not impaired. If there is reason to believe that some other hydrocarbon with an identical retention time is present in the sample, then supplemental confirmation of the vinyl chloride peak through an absolute analytical technique, such as mass spectroscopy, should be performed.

[42 FR 29006, June 7, 1977]

2. Range and Sensitivity.

The lower limit of detection of vinyl chloride will vary according to the chromatograph used. Values reported include 1×10^{-6} mg and 4×10^{-7} mg. With proper calibration, the upper limit may be extended as needed.

3. Precision and Reproducibility.

An interlaboratory comparison between seven laboratories of three resin samples, each split into three parts, yielded a standard deviation of 2.63% for a sample with a mean of 2.09 ppm, 4.16% for a sample with a mean of 1.66 ppm, and 3.30% for a sample with a mean of 62.66 ppm.

4. Safety.

Do not release vinyl chloride to the laboratory atmosphere during preparation of standards. Venting or purging with VCM/air mixtures must be held to a minimum. When they are required, the vapor must be routed to outside air. Vinyl chloride, even at low ppm levels, must never be vented inside the laboratory. After vials have been analyzed, the pressure within the vial must be vented prior to removal from the instrument turntable. Vials must be vented into an activated charcoal tube using a hypodermic needle to prevent release of vinyl chloride into the laboratory atmosphere. The charcoal must be replaced prior to vinyl chloride breakthrough.

5. Apparatus.

5.1 Sampling.

5.1.1 Bottles—60 ml (2 oz). with waxed lined screw on tops, for PVC samples.

5.1.2 Vials—50 ml Hypo-vials[1] sealed with Teflon faced Tuf-Bond discs for water samples.

5.1.3 Electrical tape—or equivalent, to prevent loosening of bottle tops.

5.2 Sample recovery.

5.2.1 Vials—With seals and caps, Perkin-Elmer Corporation No. 105-0116, or equivalent.

5.2.2 Analytical balance—Capable of weighing to ±0.001 gram.

5.2.3. Syringe, 100 µl—Precision Series "A" No. 010035, or equivalent.

5.2.4 Vial Sealer, Perkin-Elmer No. 105-0106 or equivalent.

5.3 Analysis.

5.3.1 Gas chromatograph—Perkin-Elmer Corporation Model F-40, head-space analyzer, No. 104-0001, or equivalent.

5.3.2 *Chromatographic column.* Stainless steel, 6 m × 3.2 mm, containing 0.4 percent Carbowax 1500 on Carbopak A, Perkin-Elmer Corporation No. 105-0138, or equivalent. Carbopak C can be used in place of Carbopak A. If methanol and/or acetaldehyde is present in the sample, a pair of Peropak Q columns in series (1 m × 3.2 mm followed by 3 m × 3.2 mm) with provision for backflush of the first column has been shown to provide adequate separation of vinyl chloride.

[42 FR 29006, June 7, 1977]

5.3.3 Thermometer—0 to 100° C. accurate to ±0.1° C. Perkin-Elmer No. 105-0109 or equivalent.

5.3.4. Sample tray thermostat system—Perkin-Elmer No. 105-0108, or equivalent.

5.3.5 Septa—Sandwich type, for automatic dosing, 13 mm, Perkin-Elmer No. 108-1006, or equivalent.

5.3.6 Integrator - recorder — Hewlett - Packard Model 3380A, or equivalent.

5.3.7 Filter drier assembly (3)—Perkin-Elmer No. 2230117, or equivalent.

5.3.8 Soap film flowmeter—Hewlett Packard No. 0101-0112, or equivalent.

5.4 Calibration.

5.4.1 Regulators—for required gas cylinders.

6. Reagents.

6.1 Analysis.

6.1.1 Hydrogen gas—zero grade.

6.1.2 Nitrogen gas—zero grade.

6.1.3 Air—zero grade.

6.2 *Calibration.*

[41 FR 29005, June 7, 1977]

6.2.1 *Cylinder standards* (4). Gas mixture standards (50, 500, 2,000, and 4,000 ppm vinyl chloride in nitrogen cylinders) for which the gas composition has been certified by the manufacturer. Lower concentration standards should be obtained if lower concentrations of vinyl chloride samples are expected, as the intent is to bracket the sample concentrations with standards. The manufacturer must have recommended a maximum shelf life for each cylinder so that the concentration does not change greater than ±5 percent from the certified value. The date of gas cylinder preparation, certified vinyl chloride concentration and recommended maximum shelf life must have been affixed to the cylinder before shipment from the manufacturer to the buyer.

6.2.1.1 *Cylinder standards certification.* The concentration of vinyl chloride in nitrogen in each cylinder must have been certified by the manufacturer by a direct analysis of each cylinder using an analytical procedure that the manufacturer had calibrated on the day of cylinder analysis. The calibration of the analytical procedure shall, as a minimum, have utilized a three-point calibration curve. It is recommended that the manufacturer maintain two calibration standards and use these standards in the following way: (1) a high concentration standard (between 4,000 and 5,000 ppm) for preparation of a calibration curve by an appropriate dilution technique; (2) a low concentration standard (between 50 and 500 ppm) for verification of the dilution technique used.

6.2.1.2 *Establishment and verification of calibration standards.* The concentration of each calibration standard must have been established by the manufacturer using reliable procedures. Additionally, each calibration standard must have been verified by the manufacturer by one of the following procedures, and the agreement between the initially determined concentration value and the verification concentration value must be within ±5 percent: (1) verification value determined by comparison with a gas mixture standard generated in a similar manner to the procedure described in section 7.1 of Method 106 for preparing gas mixture standards using 99.9+ percent vinyl chloride, or (2) verification value obtained by having the calibration standard analyzed by the National Bureau of Standards. All calibration standards must be renewed on a time interval consistent with the shelf life of the cylinder standards sold.

7. Procedure.

7.1 Sampling.

7.1.1 PVC sampling—Allow the resin or slurry to flow from a tap on the tank or silo until the tap line has been well purged. Extend a 60 ml sample bottle under the tap, fill, and immediately tightly cap the bottle. Wrap electrical tape around the cap and bottle to prevent the top from loosening. Place an identifying label on each bottle, and record the date, time, and sample location both on the bottles and in a log book.

7.1.2 Water sampling—Prior to use, the 60 ml vials (without the discs) must be capped with aluminum foil and muffled at 400°C for at least one hour to destroy or remove any organic matter that could interfere with analysis. At the sampling location fill the vials bubble-free, to overflowing so that a convex meniscus forms at the top. The excess water is displaced as the sealing disc is carefully placed, Teflon side down, on the opening of the vial. Place the aluminum seal over the disc and the neck of the vial and crimp into place. Affix an identifying label on the bottle, and record the date, time, and sample location both on the vials and in a log book. All samples must be kept refrigerated until analyzed.

7.2 Sample recovery. Samples must be run within 96 hours.

7.2.1 Resin samples—The weight of the resin used must be between 0.1 and 4.5 grams. An exact weight must be obtained (±0.001 gram) for each sample. In the case of suspension resins a volumetric cup can be prepared which will hold the required amount of sample. The sample bottle is opened, and the cup volume of resin is added to the tared sample vial (including septum and aluminum cap). The vial is immediately sealed and the exact sample weight is then obtained. Report this value on the data sheet as it is required for calculation of RVCM. In the case of relatively dry resin samples (water content <0.2 weight %), 100 µl of distilled water must be injected into the vial, after sealing and weighing, using a 100 µl syringe. In the case of dispersion resins, the cup cannot be used. The sample is instead weighed approximately in an aluminum dish, transferred to the tared vial and weighed accurately in the vial. The sample is then placed in the Perkin-Elmer head space analyser (or equivalent) and conditioned for one hour at 90°C.

NOTE: Some aluminum vial caps have a center section which must be removed prior to placing into sample tray. If not removed, serious damage to the injection needle will occur.

7.2.2 Suspension resin slurry and wet cake samples—Slurry must be filtered using a small Buchner funnel with vacuum to yield wet cake. The filtering process must be continued only as long as a steady stream of water is exiting from the funnel. Excessive filtration time could result in some loss of VCM. The wet cake sample (0.10 to 4.5 grams) is added to a tared vial (including septum and aluminum cap) and immediately sealed. Sample weight is then determined to 3 decimal places. The sample is then placed in the Perkin-Elmer head space analyser (or equivalent) and conditioned for one hour at 90°C. A sample of wet cake is used to determine TS (total solids). This is required for calculating the RVCM.

7.2.3 Dispersion resin slurry samples.—This material should not be filtered. Sample must be thoroughly mixed. Using a tared vial (including septum and aluminum cap) add approximately 8 drops (0.25 to 0.35 grams) of slurry or latex using a medicine dropper. This should be done immediately after mixing. Seal the vial as soon as possible. Determine sample weight accurate to 0.001 grams. Total sample weight must not exceed 0.50 grams. Condition the vial for one hour at 90°C in the analyzer. Determine the TS on the slurry sample (Section 7.3.5).

7.2.4 Aqueous latex/water samples—Using a tared vial (including septum and aluminum cap) quickly add approximately 1 cc of water using a medicine dropper. Seal the vial as soon as possible. Determine sample weight accurate to 0.001 gram. Condition the vial for two hours at 90°C in the analyzer.

7.3 Analysis.

7.3.1 Preparation of gas chromatograph—Install the chromatographic column and condition overnight at 150°C. Do not connect the exit end of the column to the detector while conditioning.

7.3.1.1 Flow rate adjustments—Adjust flow rates as follows:

a. Nitrogen carrier gas—Set regulator on cylinder to read 50 psig. Set regulator on chromatograph to 1.5 kg/cm². Normal flows at this pressure should be 25 to 40 cc/minute. Check with bubble flow meter.

b. Burner air supply—Set regulator on cylinder to read 50 psig. Set regulator on chromatograph to supply air to burner at a rate between 250 and 500 cc/minute. Check with bubble flowmeter.

c. Hydrogen supply—Set regulator on cylinder to read 30 psig. Set regulator on chromatograph to supply approximately 25±5 cc/minute. Optimize hydrogen flow to yield the most sensitive detector response without extinguishing the flame. Check flow with bubble meter and record this flow

7.3.1.2 Temperature adjustments—Set temperatures as follows:

a. Oven (chromatographic column), 80° C.

b. Dosing line, 140° C.

c. Injection block, 140° C.

d. Sample chamber, water temperature, 90° C±1.0° C.

7.3.1.3 Ignition of flame ionization detector—Ignite the detector according to the manufacturer's instructions.

7.3.1.4 Amplifier balance—Balance the amplifier according to the manufacturer's instructions.

7.3.2 Programming the chromatograph—Program the chromatograph as follows:

a. I—Dosing time—The normal setting is 2 seconds.

b. A—Analysis time—The normal setting is 8 minutes. Certain types of samples contain high boiling materials which can cause interference with the vinyl chloride peak on subsequent analyses. In these cases the analysis time must be adjusted to eliminate the interference. An automated backflush system can also be used to solve this problem.

c. B—Flushing—The normal setting is 0.2 minutes.

d. W—Stabilization time—The nomal setting is 0.2 minutes.

e. N—Number of analyses per sample—The normal setting is 1.

7.3.3 Preparation of sample turntable—Before placing any sample into turntable, be certain that the center section of the aluminum cap has been removed. The numbered sample bottles should be placed in the corresponding numbered positions in the turntable. Insert samples in the following order:

Positions 1 & 2—Old 2000 ppm standards for conditioning. These are necessary only after the analyzer has not been used for 24 hours or longer.

Position 3—50 ppm standard, freshly prepared.

Position 4—500 ppm standard, freshly prepared.

Position 5—2000 ppm standard, freshly prepared.

Position 6—4000 ppm standard, freshly prepared.

[Appendix B]

Position 7—Sample No. 7 (This is the first sample of the day, but is given as 7 to be consistent with the turntable and the integrator printout.)

After all samples have been positioned, insert the second set of 50, 500, 2000, and 4000 ppm standards. Samples, containing standards must be conditioned in the bath of 90° C for 1 hour (not to exceed 8 hours).

7.2.4 Start chromatograph program—When all samples, including standards, have been conditioned at 90° C for 1 hour, start the analysis program according to the manufacturers' instructions. These instructions must be carefully followed when starting and stopping program to prevent damage to the dosing assembly.

7.2.5 Determination of total solids (TS). For wet cake, slurry, resin solution, and PVC latex samples, determine TS for each sample by accurately weighing approximately 2 to 4 grams of sample in an aluminum pan before and after placing in a draft oven (105 to 110° C). Samples must be dried to constant weight. After first weighing return the pan to the oven for a short period of time and then reweigh to verify complete dryness. TS is then calculated as the final sample weight divided by initial sample weight.

8. Calibration.

Calibration is to be performed each eight-hour period when the instrument is used. Each day, prior to running samples, the column should be conditioned by running two of the previous days 2000 ppm standards.

8.1 Preparation of Standards.

Calibration standards are prepared by filling the vials with the vinyl chloride/nitrogen standards, rapidly seating the septum and sealing with the aluminum cap. Use a stainless steel line from the cylinder to the vial. Do not use rubber or tygon tubing. The sample line from the cylinder must be purged (into hood) for several minutes prior to filling vials. After purging, reduce the flow rate to approximately 500–1000 cc/min. Place end of tubing into vial (near bottom) and after one minute slowly remove tubing. Place septum in vial as soon as possible to minimize mixing air with sample. After the standard vials are sealed, inject 100 μl of distilled water.

8.2 Preparation of chromatograph calibration curve.

Prepare two 50 ppm, two 500 ppm, two 2000 ppm, and two 4000 ppm standard samples. Run the calibration samples in exactly the same manner as regular samples. Plot A_s, the integrator area counts for each standard sample vs C_s, the concentration of vinyl chloride in each standard sample. Draw a line of best fit through the points.

9. Calculations.

9.1 Response factor.

From the calibration curve described in Section 8.2, above, select the value of C_s that corresponds to A_s for each sample. Compute the response factor, R_f, for each sample, as follows:

$$R_f = \frac{A_s}{C_s} \quad \text{Equation 107–1}$$

9.2 Residual vinyl chloride monomer concentration, or vinyl chloride monomer concentration.

Calculate C_{vm} as follows:

$$C_{vm} = \frac{A_s P_a}{R_f T_1}\left(\frac{M_v V_v}{m_s R} + K T_2\right) \quad \text{Equation 107–2}$$

where:

C_{vm} = Concentration of vinyl chloride in the sample, in ppm.

P_a = Laboratory atmosphere pressure, mm Hg.

T_1 = Room temperature, °K.

M_v = Molecular weight of VCM (62.5).

V_v = Volume of vapor phase (vial volume less sample volume).

m_s = Weight of sample, grams.

R = Gas constant [62,360 (cc-mm-mole-degrees Kelvin)]

K = Henry's Law constant. For VCM in PVC at 90° C, $K = 6.52 \times 10^{-4} = K_p$. For VCM in 1 cc (approximate) wastewater sample at 90° C, $K = 5.0 \times 10^{-4} = K_w$.

T_2 = Equilibration temperature, °K.

If the following conditions are met, Equation 107–2 can be simplified as follows:

1. $T_1 = 22°$ C (295° K)
2. $T_2 = 90°$ C (363° K)
3. $P_a = 750$ mm Hg.
4. $V_v = V_v - \frac{m_s}{1.4} = 23.5 - \frac{m_s}{1.4}$

where

V_v = Vial volume, cc (23.5).

5. Sample contains less than 0.5 percent water.

$$C_{vm} = \frac{A_s}{R_f}\left(4.197 \times 10^{-3} + \frac{5.988 \times 10^{-3}}{m_s}\right) \quad \text{Equation 107–3}$$

The following general equation can be used for any sample which contains VCM, PVC and water.

$$C_{vm} = \frac{A_s P_a}{R_f T_1} \times \left[\frac{M_v V_v}{R m_s} + K_p (TS) T_2 + K_w (1 - TS) T_2\right]$$

Equation 107–4

where:

TS = Total solids.

NOTE: K_w must be determined for samples with a vapor volume to liquid volume ratio other than 22.5 to 1. This ratio can be obtained by adjusting the sample weight through giving consideration to the total solids and density of the PVC.

Results calculated using Equation 107–4 represent concentration based on the total sample. To obtain results based on dry PVC content, divide by TS.

For a 1-cc wastewater sample (that is, 22.5 to 1 vapor volume to liquid volume ratio), K_w is 5.0×10^{-4}. Thus, Equation 107–4 can be simplified to the following:

$$C_{vm} = \frac{A_s}{R_f}\left[\frac{5.988 \times 10^{-3}}{m_s} + (2.066 \times 10^{-4})\right] \quad \text{Equation 107–5}$$

[42 FR 29006, June 7, 1977]

10. References.

1. Residual Vinyl Chloride Monomer Content of Polyvinyl Chloride Resins and Wet Cake Samples. B. F. Goodrich Chemical Co. Standard Test Procedure No. 1905–T. B. F. Goodrich Technical Center, Avon Lake, Ohio. January 30, 1975.

2. Berens, A. R., "The Solubility of Vinyl Chloride in Polyvinyl Chloride," ACS-Division of Polymer Chemistry, Polymer Preprints *15* (2): 197, 1974.

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4. Berens, A. R., L. B. Crider, C. J. Tounsley and J. M. Whitney, Analysis for Vinyl Chloride in PVC Powders by Head-Space Gas Chromatography," to be published.

[Appendix B]