

DEPARTMENT OF ENVIRONMENT, GREAT LAKES, AND
ENERGY ENVIRONMENTAL QUALITY

AIR QUALITY DIVISION

AIR POLLUTION CONTROL

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(By authority conferred on the director of the department of **environment, Great Lakes, and energy environmental quality** by sections 5503 and 5512 of **the natural resource and environmental protection act**, 1994 PA 451, MCL 324.5503 and 324.5512, and Executive Reorganization Order Nos. 1995-168, **2009-31, 2011-1, 2019-1**, MCL 324.99903, **324.99919, 324.99921, and 324.99923**)

R 336.2001, R 336.2003, R 336.2004, R 336.2011, R 336.2012, R 336.2014, R 336.2033, R 336.2040, and R 336.2041 of the Michigan Administrative Code are amended, as follows:

PART 10. INTERMITTENT TESTING AND SAMPLING

R 336.2001 Performance tests by owner.

Rule 1001. (1) The department may require the owner or operator of ~~any~~ a source of air contaminant to conduct acceptable performance tests, at the owner's or operator's expense, in accordance with R 336.2003 under any ~~1~~ of the following conditions:

- (a) ~~Prior to~~ **Before** issuance of a permit to operate.
- (b) The source is determined to be in violation of R 336.1301 and the potential emissions exceed 100 tons per year.
- (c) The owner or operator of the source has not submitted an acceptable performance test, in accordance with R 336.2003, that demonstrates ~~that the source is in compliance~~ **complies with either** the department's rules ~~and/or with~~ the conditions specified in the permit to install, **or both**.
- (d) The source of air contaminant is located in an area designated as nonattainment for 1 or more air pollutants, and more than 12 months have expired since the date of the last performance test for such designated nonattainment pollutants.
- (e) The source of air contaminant has potential emissions in excess of 100 tons per year, is located in an area designated as attainment for 1 or more air pollutants, and more than 36 months have expired since the date of the last performance test for ~~such~~ **the** designated attainment pollutants.
- (f) After completion of a compliance program.

(2) Performance tests required by subrule (1) of this rule ~~shall~~ **must** be conducted within 60 days following receipt of written notification from the department, unless otherwise authorized by the department.

(3) **Not less than 30 days before** ~~For~~ a performance test, **as** required by subrule (1) of this rule, the owner or operator, **or an authorized agent**, shall **do both of the following**:

(a) ~~s~~Submit a site-specific test plan ~~not less than 30 days before a performance test~~ for approval ~~of~~ **by** the department. The plan ~~will~~ **must** include a test program summary, test schedule, and the quality assurance measures to be applied.

~~(4)(b) Not less than 7 days before performance tests are conducted, the owner of a source of air contaminant, or his or her authorized agent, shall n~~Notify the department, in writing, of the time and place of the performance tests and who shall conduct them **as provided in the site-specific test plan required under subdivision (a) of this subrule**. A representative of the department shall have the opportunity to witness these tests.

~~(5)~~ Results of performance tests ~~shall~~ **must** be submitted to the department in the format prescribed by the applicable reference test method within 60 days after the last date of the test.

R 336.2003 Performance test criteria.

Rule 1003. (1) Performance tests ~~shall~~ **must** be conducted and data reduced according to the reference test methods listed in R 336.2004, unless the department does any of the following:

(a) Specifies or approves, in specific cases, the use of a reference test method with minor changes in procedures or equipment.

(b) Approves the use of an equivalent method.

(c) Specifies or approves the use of an alternative method if an applicable reference test method does not exist for a specific air contaminant or source of air contaminant.

(2) **Unless otherwise approved by the department, a** performance test ~~shall~~ **must** consist of a minimum of 3 separate samples of a specific air contaminant conducted within a 36-hour period **that starts once the probe enters the stack. Any data measured within the 36-hour period must be recorded and provided to the department.** ~~unless otherwise authorized by the department.~~ Each of the 3 separate samples ~~shall~~ **must** be obtained while the source is operating at a similar production level, **as described under subrule (3) of this rule**. For the purpose of determining compliance with an applicable emission limit, rule, or permit condition, the arithmetic mean of results of the 3 samples ~~shall~~ **must** apply. If a sample is accidentally lost or conditions occur in which 1 of the 3 samples must be discontinued because of forced shutdown, failure of an irreplaceable portion of the sampling train, extreme meteorological conditions, or other circumstances beyond the owner's or operator's control, then compliance may, upon the approval of the department, be determined using the arithmetic mean of the results of 2 samples.

(3) All performance tests ~~shall~~ **must** be conducted while the source of air contaminant is operating at maximum routine operating conditions, or under ~~such~~ other conditions, within the capacity of the equipment, as may be requested by the department. Other conditions may include source operating periods of startup, shutdown, or ~~such~~ other operations, excluding malfunction, specific to certain sources. Routine operating conditions ~~shall~~ **must** also include those specified within a permit to install or a permit to

operate. The owner or operator shall make available to the department ~~such~~ **the** records ~~as that~~ may be necessary to determine the conditions of source operation that occurred during the period of time of the performance test.

(4) For ~~any~~ sources that ~~is~~ **are** subject to an emission limitation calculated to 50% excess air, the multipoint, integrated sampling procedure of method 3 ~~shall~~ **must** be used for gas analysis. For all other sources that require a determination of the molecular weight of the exhaust, ~~any~~ **an** optional sampling procedure of method 3 may be used. Alternatives or modifications to procedures are subject to the approval of the department.

(5) For reference test methods 5B, **which is described in R 336.2011**, ~~and reference test method 5C, which is described in R 336.2012, and reference test method 5E, which is described in R 336.1014~~, the minimum volume per sample ~~shall~~ **must** be 30 cubic feet of dry gas corrected to standard conditions, (70 degrees Fahrenheit, 29.92 in-ches Hgmercury). Minimum sample time ~~shall~~ **must** be 60 minutes, which may be continuous or a combination of shorter sampling periods for sources that operate in a cyclic manner. Smaller sampling times or sample volumes, when necessitated by process variables or other factors, may be approved by the department.

R 336.2004 ~~Appendix A; r~~ **Reference test methods; adoption of federal reference test methods.**

Rule 1004. (1) The ~~following~~ federal ~~reference~~ test methods; described in the provisions of 40 ~~C.F.R.~~ **CFR** part 60, appendix A ~~(2007)~~, **adopted by reference in R 336.1902**, are ~~the reference~~ test methods for performance tests required pursuant to the provisions of this part **and include, but are not limited to, the following:**

- (a) Method 1 - Sample and velocity traverse for stationary sources.
- (b) Method 1A - Sample and velocity traverses for stationary sources with small stacks or ducts.
- (c) Method 2 - Determination of stack gas velocity and volumetric flow rate (type-S pitot tube).
- (d) Method 2A - Direct measurement of gas volume through pipes and small ducts.
- (e) Method 2C - Determination of stack gas velocity and volumetric flow rate in small stacks and ducts (standard pitot tube).
- (f) Method 2D - Measurement of gas volumetric flow rates in small pipes and ducts.
- (g) Method 3 - Gas analysis for the determination of dry molecular weight.
- (h) Method 4 - Determination of moisture content in stack gases.
- (i) Method 5 - Determination of particulate matter emissions from stationary sources.
- (j) Method 6 - Determination of sulfur dioxide emissions from stationary sources.
- (k) Method 7 - Determination of nitrogen oxide emissions from stationary sources.
- (l) Method 8 - Determination of sulfuric acid mist and sulfur dioxide emissions from stationary sources.
- (m) Method 9 - Visual determination of the opacity of emissions from stationary sources.
- (n) Method 10 - Determination of carbon monoxide emissions from stationary sources.
- (o) Method 10B - Determination of carbon monoxide emissions from stationary sources.
- (p) Method 18 - Measurement of gaseous organic compound emissions by gas chromatography.

- (q) Method 21 - Determination of volatile organic compound leaks.
- (r) Method 24 - Determination of volatile matter content, water content, density, volume solids, and weight solids of surface coatings.
- (s) Method 24A - Determination of volatile matter content and density of printing inks and related coatings.
- (t) Method 25 - Determination of total gaseous nonmethane organic emissions as carbon.
- (u) Method 25A - Determination of total gaseous organic concentration using a flame ionization analyzer.
- (v) Method 27 - Determination of vapor tightness of gasoline delivery tank using pressure-vacuum test.
- (w) Method 29 - Determination of metals emissions from stationary sources.
- (x) Method 30A - Determination of total vapor phase mercury emissions from stationary sources (instrumental analyzer procedure).
- (y) Method 30B - Determination of total vapor phase mercury emissions from coal-fired combustion sources using carbon sorbent traps.

(2) **The federal test methods in the following provisions, adopted by reference in R 336.1902, are test methods for performance tests required pursuant to the provisions of this part:**

- (a) **40 CFR part 51, appendix M.**
- (b) **40 CFR part 61, appendix B.**
- (c) **40 CFR part 63, appendix A.**

~~The reference test methods listed in subrule (1) of this rule are adopted by reference in this rule. Copies of the test methods may be inspected at the Lansing office of the air quality division of the department of environmental quality. A copy of title 40 of the Code of Federal Regulations, part 60, appendix A, may be obtained from the Department of Environmental Quality, Air Quality Division, P.O. Box 30260, Lansing, Michigan 48909-7760, at a cost at the time of adoption of these rules of \$67.00; from the Superintendent of Documents, United States Government Printing Office, P.O. Box 979050, St. Louis, Missouri 63197-9000, at a cost at the time of adoption of these rules of \$57.00; or on the United States government printing office internet web site at <http://www.gpoaccess.gov>.~~

(3) All alternatives that are subject to the approval of the administrator in the adopted federal reference methods are subject to the approval of the department.

(4) Determinations of compliance with visible emission standards for stationary sources ~~shall~~ **must** be conducted as specified in **40 CFR part 60, appendix A**, ~~reference~~ test method 9 or ~~other~~ **another** alternative method approved by the department, with the following exceptions:

(a) Visible emissions from a scarfing operation at a steel manufacturing facility ~~shall~~ **must** be determined as specified in reference test method 9A, which is described in R 336.2030.

(b) Visible emissions from a coke oven pushing operation and fugitive coke oven visible emissions ~~shall~~ **must** be determined as specified in reference test method 9B, which is described in R 336.2031.

(c) Visible emissions, fugitive and nonfugitive, from basic oxygen furnace operations, hot metal transfer operations, and hot metal desulfurization operations ~~shall~~ **must** be determined as specified in reference method 9C, which is described in R 336.2032.

(5) Determinations of particulate emission rates for stationary sources ~~shall~~ **must** be conducted as specified in 1 or more of the following reference test methods:

(a) Reference test method 5B, which is described in R 336.2011.

(b) Reference test method 5C, which is described in R 336.2012.

(c) Reference test method 5D, which is described in R 336.2013.

(d) Reference test method 5E, which is described in R 336.2014.

(e) "Standard Methods for the Examination of Water and Wastewater," (14th ~~23rd~~ edition), ~~section 208C~~, as described and modified in R 336.2033.

(6) Determinations of total gaseous nonmethane organic emissions as carbon, using the alternate version of federal ~~reference~~ test method 25 **under 40 CFR part 60, appendix A**, incorporating the Byron analysis, ~~shall~~ **must** be conducted as specified in R 336.2006.

R 336.2011 Reference test method 5B.

Rule 1011. Reference test method 5B, in-stack filtration method, reads as follows:

(a) The principle, applicability, and performance test criteria are as follows:

(i) Principle. Particulate matter is withdrawn isokinetically from the source and collected on solid filtering media maintained at stack temperature. The particulate matter mass is determined gravimetrically after removal of uncombined water.

(ii) Applicability. This method is applicable for the determination of particulate emissions from stationary sources as identified in table 31 of R 336.1331. The method is also applicable when specifically provided for in the department's rules, orders, a permit to install, or a permit to operate.

(iii) Performance test criteria **as follows**:

(A) A performance test ~~must~~ ~~shall~~ ~~consist~~ **meet the requirements under R 336.2003(2)**. ~~of a minimum of 3 separate samples of a specific air contaminant conducted within a 36-hour period, unless otherwise authorized by the department. Each of the 3 separate samples shall be obtained while the source is operating at a similar production level. For the purpose of determining compliance with an applicable emission limit, rule, or permit condition, the arithmetic mean of results of the 3 samples shall apply. If a sample is accidentally lost or conditions occur in which 1 of the 3 samples must be discontinued because of forced shutdown, failure of an irreplaceable portion of the sampling train, extreme meteorological conditions, or other circumstances beyond the owner's or operator's control, compliance may, upon the approval of the department, be determined using the arithmetic mean of the results of 2 samples.~~

(B) For ~~any~~ sources that ~~are~~ is subject to an emission limitation calculated to 50% excess air, the multipoint, integrated sampling procedure of R 336.2004(1)(c) ~~shall~~ **must** be used for gas analysis. For all other sources that require a determination of the molecular weight of the exhaust, ~~any~~ **an** optional sampling procedure of R 336.2004(1)(c) may be used. Alternatives or modifications to procedures are subject to the approval of the department.

(C) The minimum volume per sample ~~shall~~ **must** be 30 cubic feet of dry gas corrected to standard conditions, (70 degrees Fahrenheit, **and** 29.92 inches mercury). Minimum sample time ~~shall~~ **must** be 60 minutes, which may be continuous or a combination of

shorter sampling periods for sources that operate in a cyclic manner. Smaller sampling times or sample volumes, if necessitated by process variables or other factors, may be approved by the department.

(D) For ~~any~~ a source whose emission control device alters the moisture content of the exhaust gas, a moisture determination ~~shall~~ **must** be performed in a location upstream from the emission control device and in accordance with R 336.2004(1)(d) or an alternative method approved by the department.

(b) The following provisions apply to apparatus:

(i) Sampling train. A schematic of the sampling train used in this method is shown in figure 102 **under R 336.2021**. Construction details for many, but not all, of the train components are given in APTD-0581, **adopted by reference in R 336.1902**. (See subdivision (g)(ii) of this rule.) For changes from the APTD-0581 document and for allowable modifications to figure 102, the user shall consult with the department. The operating and maintenance procedures for many, but not all, of the sampling train are described in APTD-0576, **adopted by reference in R 336.1902**. (See subdivision (g)(iii) of this rule.) Since correct usage is important in obtaining valid results, all users shall read APTD-0576 and adopt the applicable operating and maintenance procedures outlined in it, unless otherwise specified ~~herein~~. The sampling train ~~shall~~ **must** consist of the following components:

(A) Probe nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper ~~shall~~ **must** be less than 30 degrees and the taper ~~shall~~ **must** be on the outside to preserve a constant internal diameter. The probe nozzle ~~shall~~ **must** be of the button-hook design, unless otherwise specified by the department. If made of stainless steel, the nozzle ~~shall~~ **must** be constructed from seamless tubing. Other materials of construction may be used, subject to the approval of the department. A range of nozzle sizes suitable for isokinetic sampling ~~shall~~ **must** be available, for example, 0.32 to 1.27 centimeters, (1/8 to 1/2 inch), or larger if higher volume sampling trains are used inside diameter (~~ID~~) nozzles in increments of 0.16 centimeters, (1/16 inches). Each nozzle ~~shall~~ **must** be calibrated according to the procedures outlined in subdivision (e) of this rule.

(B) Probe liner. Interior surface may be constructed of stainless steel, (no specific grade), glass, ~~teflon~~ **Teflon**, or ~~such~~ other material that maintains proper flow at the stack conditions experienced.

(C) Pitot tube. Type S, as described in ~~section 2.1 of method 2~~, or other device approved by the department. The pitot tube ~~shall~~ **must** be attached to the probe, as shown in figure 102 **under R 336.2021**, to allow constant monitoring of the stack gas velocity. The impact, (high pressure,) opening plane of the pitot tube ~~shall~~ **must** be even with or above the nozzle entry plane, (see method 2, figure 2-6b,) during sampling. The type S pitot tube assembly ~~shall~~ **must** have a known coefficient, determined as outlined in ~~section 4 of method 2~~.

(D) Differential pressure gauge. **Two incline** ~~Incline~~ manometer or equivalent devices (~~2~~) as described in ~~section 2.2 of method 2~~. One manometer ~~shall~~ **must** be used for velocity head (p) readings and the other ~~shall~~ **must** be used for orifice differential pressure readings.

(E) Filter holders. Two separate filter holders in series or 1 filter holder with separate filter supports and seals for 2 filters. One filter holder with 2 filters held in contact with

each other is not acceptable. Materials of construction may be stainless steel (316), glass, ~~teflon~~ Teflon, or other material approved by the department.

(F) Filter heating system. Auxiliary heating of the filter media is not acceptable. For saturated stack gases, the operator may opt to use filters that do not blind when wet and that do not require heating, (see subdivision (c)(i)(A) of this rule).

(G) Condenser. The following system ~~shall~~ **must** be used to determine the stack gas moisture content: Three impingers connected in series with leak-free ground glass fittings or ~~any~~-similar leak-free noncontaminating fittings. All impingers ~~shall~~ **must** be of the Greenburg-Smith design and ~~shall~~ **must** be modified by replacing the tip with a 1.3 centimeters, (1/2 inch,) ~~to~~ **inside diameter** glass tube extending to about 1.3 centimeters, (1/2 inch,) from the bottom of the flask. Modifications, such as using flexible connections between the impingers or using materials other than glass, are ~~permitted~~ **allowed**, subject to the approval of the department. The first impinger ~~shall~~ **must** contain a known quantity of water, (~~as described in~~ subdivision (d)(i)(C) of this rule.); ~~the~~ **The second impinger shall must** be empty; and the third ~~shall~~ **must** contain a known weight of silica gel or equivalent desiccant. Alternatively, ~~any~~-a system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 milliliter or 1 gram, may be used, subject to the approval of the department. In any case, the means for measuring the moisture leaving the condenser ~~shall~~ **must** be by passing the sample gas stream through a tared silica gel, or equivalent desiccant, trap with exit gases kept below 20 degrees Centigrade, (68 degrees Fahrenheit,) and determining the weight gain. If a determination of the particulate matter collected in the impingers is required by the department's rules, a permit to install, or a permit to operate, then the impinger system described above ~~shall~~ **must** be used without modification. Contact the department as to the sample recovery and analysis of the impinger contents.

(H) Metering system. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3 degrees Centigrade, (5.4 degrees Fahrenheit), dry-gas meter capable of measuring volume to within 2%, and related equipment as shown in figure 102 **under R 336.2021**. Other metering systems capable of maintaining sampling rates within 10% of isokinetic and capable of determining sample volumes to within 2% may be used, subject to the approval of the department. When the metering system is used in conjunction with a pitot tube, the system ~~shall~~ **must** enable checks of isokinetic rates. Sampling trains utilizing metering systems designed for higher flow rates than those described in APTD-0581 or APTD-0576, **both adopted by reference in R 336.1902**, may be used if the specifications of this method are met.

(I) Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 millimeters ~~Hg~~mercury, (0.1 inch ~~Hg~~mercury). In many cases, the barometric reading may be obtained from a nearby national weather service station. In this case, the station value, which is the absolute barometric pressure, ~~shall~~ **must** be requested and an adjustment for elevation differences between the weather station and sampling point ~~shall~~ **must** be applied at a rate of minus 2.5 millimeters ~~Hg~~mercury, (0.1 inch ~~Hg~~mercury,) per 30 ~~M~~meters, (100 feet,) elevation increase or vice versa for elevation decrease.

(J) Gas density determination equipment. Temperature sensor and pressure gauge, as described in sections 2.3 and 2.4 of method 2, and gas analyzer, if necessary, as described

in method 3. The temperature sensor ~~shall~~ **must**, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration ~~such so~~ that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just before use in the field. If the temperature sensor is attached in the field, then the sensor ~~shall~~ **must** be placed in an interference-free arrangement with respect to the type S pitot tube openings, (see method 2, figure 2-76 **Velocity Traverse Data**). As a second alternative, if a difference of not more than 1% in the average velocity measurement is to be introduced, then the temperature gauge need not be attached to the probe or pitot tube. This alternative is subject to the approval of the department. "Construction Details of Isokinetic Source Sampling Equipment," APTD-0581, April 1971, (PB203-060-LL), and "Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment," APTD-0576, March 1972, (PB209-022-LL), are adopted by reference in **R 336.1902**. ~~this rule. Copies of these documents may be inspected at the Lansing office of the air quality division of the department of environmental quality. Copies of APTD-0581 and APTD-0576 may be obtained from the Department of Environmental Quality, Air Quality Division, P.O. Box 30260, Lansing, Michigan 48909-7760, or from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161, at a cost at the time of adoption of these rules of \$28.50 each.~~

(ii) Sample recovery. The following items are required:

(A) Probe-liner and probe-nozzle brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush ~~shall~~ **must** have extensions, at least as long as the probe, made of stainless steel, nylon, ~~teflon~~ **Teflon**, or similarly inert material. The brushes ~~shall~~ **must** be properly sized and shaped to brush out the probe liner and nozzle.

(B) Wash bottles. ~~2. Glass~~ **Two glass** wash bottles are recommended; ~~the~~. **The** tester may use polyethylene wash bottles, but the acetone should not be stored in polyethylene bottles for longer than 1 month.

(C) Glass sample storage containers. Chemically resistant, borosilicate glass bottles for acetone washes, 500 **milliliters** or 1000 **milliliters**. Screw cap liners ~~shall~~ **must** either be rubber-backed ~~teflon~~ **Teflon** or ~~shall~~ **must** be constructed ~~so as~~ to be leak-free and resistant to chemical attack by acetone. Narrow-mouth glass bottles are less prone to leakage. Alternatively, polyethylene bottles may be used.

(D) Filter containers. Glass, polyethylene, or aluminum tube containers, unless otherwise specified by the department.

(E) Graduated cylinder or balance. To measure condensed water to within 1 **milliliter** or 1 **gram**, graduated cylinders ~~shall~~ **must** have subdivisions of not more than 2 **milliliters**. Most laboratory balances are capable of weighing to the nearest 0.5 **gram** or less. Any of these balances may be used here and in paragraph (iii)(D) of this subdivision.

(F) Plastic storage containers. Airtight containers to store silica gel.

(G) Funnel and rubber policeman. To aid in the transfer of silica gel to container; **but** not necessary if silica gel is weighed in the field.

(H) Funnel. Glass or polyethylene, to aid in sample recovery.

(iii) Analysis. The following equipment is required for analysis:

(A) Glass weighing dishes.

(B) Desiccator.

- (C) Analytical balance. To measure to within 0.1 milligrams.
- (D) Balance. To measure to within 0.5 milligrams.
- (E) Beakers. 250 milliliters.
- (F) Hygrometer. To measure the relative humidity of the laboratory environment.
- (G) Temperature gauge. To measure the temperature of the laboratory environment.
- (c) The following provisions ~~shall~~ **must** apply to reagents:
 - (i) Sampling. The reagents used in sampling are as follows:
 - (A) Filters. Two in-stack filters may be any combination of alundum ceramic thimble filters, type RA-98, or glass fiber filters, type A without organic binder. The size of ~~such~~ **the** filters ~~shall~~ **must** allow proper sampling rates to maintain iso-kinetics using the nozzle sizes specified in subdivision (b)(i)(A) of this rule. Alternatively, other types of filters may be used, subject to the approval of the department.
 - (B) Silica gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175 degrees Centigrade, (350 degrees Fahrenheit,) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants that are equivalent or better may be used, subject to the approval of the department.
 - (C) Water. When analysis of the material caught in the impingers is required, distilled water ~~shall~~ **must** be used. Run blanks before field use to eliminate a high blank of test samples.
 - (D) Crushed ice.
 - (E) Stopcock grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with ~~teflon~~ **Teflon** sleeves, or equivalent, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the department.
 - (ii) Sample recovery. Washing solvent. Either acetone or distilled water may be used for sample recovery. If acetone is used for washing solvent, then reagent grade, less than 0.001% residue, in glass bottles is required. Acetone from metal containers generally has a high residue blank and ~~shall~~ **must** not be used. If suppliers transfer acetone to glass bottles from metal containers, then acetone blanks ~~shall~~ **must** be run before field use and only acetone with low blank values, (less than 0.001%,) ~~shall~~ **must** be used. In no case ~~shall~~ **must** a blank value of more than 0.001% of the weight of acetone used be subtracted from the sample weight. If distilled water is used for washing solvent, use distilled water with less than 0.001% residue. Run blanks before field use to eliminate a high blank on test samples.
 - (iii) Analysis. Two reagents are required for the analysis:
 - (A) Solvent. Same as paragraph (ii) of this subdivision for quantitative transfer.
 - (B) Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the department.
- (d) The following provisions ~~shall~~ **must** apply to procedure:
 - (i) Sampling. The complexity of this method is such that, in order to obtain reliable results, testers shall be trained and experienced with the test procedures. Sampling ~~shall~~ **must** comply with the following provisions:
 - (A) Pretest preparation- **provisions are as follows:**
 - (I). All the components ~~shall~~ **must** be maintained and calibrated according to the applicable procedures described in APTD-0576, **adopted by reference in R 336.1902**, unless otherwise specified in this rule.

(II) Weigh several 200 to 300 **gram** portions of silica gel in airtight containers to the nearest 0.5 **gram**. Record the total weight of the silica gel plus container on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just before train assembly.

(III) Check filters visually against light for irregularities, flaws, pinhole leaks, or cracks. Label filters of the proper size on the back side using numbering machine ink. As an alternative, label the shipping containers, **as described under** (subdivision (b)(ii)(D) of this rule), and keep the filters in these containers at all times, except during sampling and weighing.

(IV) Dry the filters in an oven at 105 degrees Centigrade, (220 degrees Fahrenheit,) for a minimum of 2 hours, cool for at least 1 hour in a desiccator containing anhydrous calcium sulfate, and individually weigh and record ~~their weights~~ **each weight** to the nearest 0.1 **milligram**. During the weighing, the filter ~~shall~~ **must** not be exposed to the laboratory atmosphere for a period of more than 2 minutes and a relative humidity above 50%.

(V) Procedures, other than those specified, that account for relative humidity effects may be used, subject to the approval of the department.

(B) Preliminary determinations- **provisions are as follows:**

(I) Select the sampling site and the minimum number by the department.

(II) Determine the stack pressure, temperature, and the range of velocity heads using method 2; ~~It~~ **It** is recommended that a leak check of the pitot lines, (see method 2, section 3.1A) be performed.

(III) Determine the moisture content using approximation method 4, or its alternatives, for the purpose of making isokinetic sampling rate settings.

(IV) Determine the stack gas dry molecular weight, as described in method 2, ~~section 3.6;~~ if integrated method 3 sampling is used for molecular weight determination, the integrated bag sample ~~shall~~ **must** be taken simultaneously with, and for the same total length of time as, the particulate sample run.

(V) Select a nozzle size based on the range of velocity heads so that it is not necessary to change the nozzle size to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered, (see ~~section 2.2 of method 2~~).

(VI) Select a suitable probe liner and probe length so that all traverse points may be sampled. For large stacks, sampling from opposite sides of the stack may reduce the length of probes.

(VII) Select a total sampling time greater than or equal to the minimum total sampling time specified in the department's rules so that the sampling time per point is not less than 5 minutes, unless approved by the department, or some greater time interval as specified by the department, and so that the sample volume taken, corrected to standard conditions, exceeds the required minimum total gas sample volume. The latter is based on an approximate average sampling rate. The number of minutes sampled at each point may be an integer or an integer plus 1/2 minute to avoid timekeeping errors. In some circumstances, such as in batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the department's approval ~~shall~~ **must** first be obtained.

(C) Preparation of collection train- **provisions are as follows:**

(I) During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just before assembly or until sampling is about to begin.

(II) Place 100 **milliliters** of water in the first impinger, leave the second impinger empty, and transfer approximately 200 to 300 **grams** of preweighed silica gel from its container to the third impinger. More silica gel may be used, but care ~~shall~~ **must** be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 **gram** and recorded.

(III) Using tweezers or clean disposable surgical gloves, place a labeled, (identified,) and weighed filter in each filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter.

(IV) Install the selected nozzle using a Viton A 0-ring when stack temperatures are less than 260 degrees Centigrade, (500 degrees Fahrenheit,) and an asbestos string gasket when temperatures are higher. See APTD-0576, **adopted by reference in R 336.1902**, for requirements. Other connecting systems using either 310 stainless steel or ~~teflon~~ **Teflon** ferrules may be used to form a leak-free direct mechanical connection.

(V) Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

(VI) Set up the train as in figure 102 **under R 336.2021**.

(VII) If necessary, use a very light coat of silicone grease on all ground glass joints. Grease only the outer portion, (see APTD-0576,) to avoid the possibility of contamination by the silicone grease.

(VIII)- Place crushed ice around the impingers.

(D) Leak check procedures:

(I+) Pretest leak check. A pretest leak check is strongly recommended, but not required, to prevent invalid sampling and wasted time. If the tester opts to conduct the pretest leak check, the following procedure ~~shall~~ **must** be used: Perform the leak check on the entire system, including filter housings and nozzle, by plugging the nozzle and pulling a 380 **millimeter Hgmercury**, (15 **inch Hgmercury**,) vacuum. Alternatively, a lower vacuum may be used if it is not exceeded during the test. Leakage rates in excess of 4% of the average sampling rate or 0.00057 m^3/min **cubic meters per minute**, (0.02 ~~cfm~~ **cubic feet per minute**), whichever is less, are unacceptable. The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581, **adopted by reference in R 336.1902**, may be helpful. Start the pump with the bypass valve fully open and the coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve, **as** this will cause water to back up into the flexible sample tube and the probe. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check and start over. When the leak check is completed, first slowly remove the plug from the inlet to the nozzle and immediately turn off the vacuum pump. This prevents the water in the first impinger from being forced backward into the sample tube and prevents silica gel from being entrained backward into the second impinger.

(II2) Leak checks during sample run. If, during the sampling run, a component, (such as a filter assembly or impinger,) change becomes necessary, a leak check ~~shall~~ **must** be conducted immediately before the change is made. The leak check ~~shall~~ **must** be done according to the procedure outlined in paragraph (i)(D)(4I) of this subdivision, except that it ~~shall~~ **must** be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is not more than 0.00057 ~~m³/min~~ **cubic meters per minute**, (0.02 ~~cfm~~ **cubic feet per minute**,) or 4% of the average sampling rate, whichever is less, then the results are acceptable and no correction need be applied to the total volume of dry gas metered. If a higher leakage rate is obtained, then the tester shall either record the leakage rate and plan to correct the sample volume, as shown in subdivision (f)(iii) of this rule, or ~~shall~~ void the sampling run. Immediately after component changes, leak checks may be performed. If leak checks are done, then the procedure outlined in paragraph (i)(D)(4I) of this subdivision ~~shall~~ **must** be used.

(III3) Post-test leak check. A leak check is required at the conclusion of each sampling run. The leak check ~~shall~~ **must** be performed in accordance with the procedures in paragraph (i)(D)(4I) of this subdivision, except that it ~~shall~~ **must** be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is not more than 0.00057 ~~m³/min~~ **cubic meters per minute**, (0.02 ~~cfm~~ **cubic feet per minute**,) or 4% of the average sampling rate, whichever is less, then the results are acceptable and no correction need be applied to the total volume of dry gas metered. If a higher leakage rate is obtained, then the tester shall either record the leakage rate and correct the sample volume, as shown in subdivision (f)(iii) of this rule, or ~~shall~~ void the sampling run.

(E) Particulate train operation. During the sampling run, maintain an isokinetic sampling rate that is within 10% of true isokinetic, unless otherwise specified by the department. For each run, record the data required on a data sheet such as the data sheet in figure 104 **under R 336.2021**. Record the initial dry-gas meter reading. Record the dry-gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by figure 104 **under R 336.2021** at least once at each sample point during each time increment, and take additional readings when significant changes, (20% variation in velocity head readings,) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse. Clean the portholes before the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap and verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs that aid in the rapid adjustment of the isokinetic sampling rate without excessive computations are available. These nomographs are designed for use when the type S pitot tube coefficient is 0.85 ± 0.02 and the stack gas equivalent density, (dry molecular weight,) is equal to 29 ± 4 . APTD-0576, **adopted by reference in R 336.1902**, details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps, (see subdivision (g)(iv) of this rule,) are taken to compensate for the deviations. When the stack is under significant negative pressure, (height of impinger

stem), take care to pull low-flow when inserting the probe into the stack to prevent water from backing into the sample tubing and to avoid pulsation through the filter and possible loss of materials. When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream. Traverse the stack cross section, as required by method 1 or as specified by the department, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes; this minimizes the chance of extracting deposited material. During the test run, add more ice and, if necessary, salt to maintain a temperature of less than 20 degrees Centigrade, (68 degrees Fahrenheit,) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer. If the pressure drop across the filter becomes too high and makes isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak check, ~~(as described under~~ paragraph (i)(D)(~~2II~~) of this subdivision). The total particulate weight ~~shall~~ **must** include the summation of all filter assembly catches. A single train ~~shall~~ **must** be used for the entire sample run, except in cases where simultaneous sampling is required in 2 or more separate ducts, at 2 or more different locations within the same duct, or where equipment failure necessitates a change of trains. In all other situations, the use of 2 or more trains ~~shall~~ **must** be subject to the approval of the department. When 2 or more trains are used, separate analyses of the front-half and, if applicable, impinger catches from each train ~~shall~~ **must** be performed, unless identical nozzle sizes were used on all trains. If identical nozzle sizes were used, the front-half catches from the individual trains may be combined, as may the impinger catches, and 1 analysis of front-half catch and 1 analysis of impinger catch may be performed. Consult with the department for details concerning the calculation of results when 2 or more trains are used. At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry-gas meter reading, and conduct a post-test leak check, as outlined in paragraph (i)(D)(~~3III~~) of this subdivision. Leak-check the pitot lines as described in method 2, ~~section 3.1;~~ ~~the~~ **The** lines ~~shall~~ **must** pass this leak check to validate the velocity head data.

(F) Calculation of percent isokinetic. Calculate percent isokinetic, (see subdivision (f) of this rule,) to determine if the run was valid or if another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the department for possible variance on the isokinetic rates.

(ii) Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this creates a vacuum in the filter holder and draws water from the impingers into the sample tube. Before moving the sampling train to the cleanup site, make sure all condensed water in the probe and flexible sample lines are drained into the first impinger. Disconnect all sample lines and remove the nozzle-filter set assembly from the probe. Cap all openings to prevent contamination or accidental loss of sample. Remove all excess particulate from the exterior of the nozzle-filter assembly to prevent contamination during disassembly.

Transfer the nozzle-filter set assembly and impinger set to the cleanup area. The cleanup area ~~shall~~ **must** be clean and protected from the wind so that the chances of contaminating or losing the sample are minimized. Save a portion of the solvent used for cleanup as a blank. Take 200 **milliliters** of this solvent directly from the wash bottle being used and place it in a glass sample container labeled "solvent blank". Inspect the train before and during disassembly and note any abnormal conditions. Treat the samples in the following manner: Container ~~numbers~~ **Nos. 1, 1A**. Carefully remove the filters from the filter holders and place **each filter in its** ~~in their~~ identified containers. Use a pair of tweezers or clean disposable surgical gloves, or both, to handle the filters. Carefully transfer to the container any particulate matter or filter fibers, or both, that adhere to the filter holder gasket by using a dry nylon bristle brush or sharp-edged blade, or both. Seal the containers. Container ~~number~~ **No. 2**. Taking care to see that particulate on the outside of the nozzle and filter holders does not get into the sample, the test~~er~~ shall carefully remove the nozzle and clean the inside surface by rinsing with solvent from a wash bottle and brushing with a nylon bristle brush. Brush until the solvent rinse shows no visible particles and then make a final rinse of the inside surface with solvent. After ensuring that all joints have been cleaned of all extraneous material, the test~~er~~ shall quantitatively remove particulate from the filter holders by rubbing the surfaces with a nylon bristle brush and rinsing with solvent. Rinse each surface 3 times, or more if needed, to remove visible particulate. Make a final rinse of the brush and filter holder set. After all solvent washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that solvent will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine if leakage occurred during transport. Label the container to clearly identify its contents. Container ~~number~~ **No. 3**. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the third impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling it. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that adhere to the impinger wall and are difficult to remove. Since the gain in weight will be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for container ~~number~~ **No. 3** in paragraph (iii) of this subdivision. Impinger water. Treat the impingers in the following manner: Make a notation of any color or film in the liquid catch. Measure the liquid that is in the first 2 impingers to within ± 1 **milliliter** by using a graduated cylinder or by weighing it to within ± 1.0 **g gram** by using a balance if one is available. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required, (~~see subdivision (b)(i)(G) of this rule~~). If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically. If possible, containers ~~shall~~ **must** be shipped in a manner that keeps them upright at all times.

(iii) Analysis. Record the data required on a sheet such as the sheet in figure 106 **under R 336.2021**. Handle each sample container in the following manner: Container ~~numbers~~ **Nos. 1, 1A**. Analyze and report each filter separately. Transfer the filter and any loose particulate from the sample container to a tared-glass weighing dish. Dry the filter

in an oven at 105 degrees Centigrade, (220 degrees Fahrenheit,) for a minimum of 2 hours, cool for at least 1 hour in a desiccator containing anhydrous calcium sulfate, and weigh and record its weight to the nearest 0.1 **milligram**. During the weighing the filter ~~shall~~ **must** not be exposed to the laboratory atmosphere for a period greater than 2 minutes or a relative humidity above 50%. Procedures, other than those specified, that account for relative humidity effects may be used, subject to the approval of the department. The method used for drying and weighing of filters ~~shall~~ **must** be consistent before and after the test. Container ~~No. number~~ 2. Note the level of liquid in the container and confirm on the analysis sheet if leakage occurred during transport. If a noticeable amount of leakage has occurred, then either void the sample or use methods, subject to the approval of the department, to correct the final results. Measure the liquid in this container either volumetrically to ± 1 **millileter** or gravimetrically to ± 1.0 **gram**. Transfer the contents to a tared 250-**millileter** beaker and evaporate to dryness either at ambient temperature and pressure for acetone or at 95 degrees Centigrade, (203 degrees Fahrenheit,) in an oven for distilled water. Then subject the sample to 250 degrees Centigrade, (482 degrees Fahrenheit,) in an oven for 2 to 3 hours. Desiccate 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 **milligram**. Container ~~No. number~~ 3. Weigh the spent silica gel, or silica gel plus impinger, to the nearest 0.5 **gram** using a balance. This step may be conducted in the field. "Solvent blank" container. Measure solvent in this container either volumetrically or gravimetrically. Transfer the contents to a tared 250-**milliliters** beaker and evaporate to dryness either at ambient temperature and pressure for acetone or at 95 degrees Centigrade, (203 degrees Fahrenheit,) in an oven for distilled water. Then subject the sample to 250 degrees Centigrade, (482 degrees Fahrenheit,) in an oven for 2 to 3 hours. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 ~~mg~~ **milligram**. If acetone is used, the contents of Container ~~No. number~~ 2, as well as the acetone blank container, may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, then the temperature ~~shall~~ **must** be closely supervised, and the contents of the beaker ~~shall~~ **must** be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

(e) Calibration. Maintain a laboratory log of all calibrations. Calibrations ~~shall~~ **must** comply with the following provisions:

(i) Probe nozzle. ~~A probe nozzle~~ ~~Probe nozzles~~ ~~shall~~ **must** be calibrated before its initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 **millimeter**, (0.001 ~~in.-ch~~). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers ~~shall~~ **must** not exceed 0.1 **millimeter**, (0.004 ~~in.-ch~~). When nozzles become nicked, dented, or corroded, ~~they shall~~ **the nozzles must** be reshaped, sharpened, and recalibrated before use. Each nozzle ~~shall~~ **must** be permanently and uniquely identified.

(ii) Pitot tube. The type S pitot tube assembly ~~shall~~ **must** be calibrated according to the procedures in ~~section 4 of~~ method 2.

(iii) Metering system. Before its initial use in the field, the metering system ~~shall~~ **must** be calibrated according to the procedure in APTD-0576, **adopted by reference in R 336.1902**. Instead of physically adjusting the dry-gas meter dial readings to correspond to the wet-test meter readings, calibration factors may be used to mathematically correct the

gas meter dial readings to the proper values. Before calibrating the metering system, a leak check may be conducted. For metering systems having diaphragm or rotary pumps, the normal leak check procedure will not detect leakages within the pump. For these cases, the following leak check procedure may be used: Make a 10-minute calibration run at $0.00057 \text{ m}^3/\text{min}$ **cubic meters per minute**, (~~0.02 cfm cubic feet per minute.~~); ~~at~~ **At** the end of the run, take the difference of the measured wet-test meter and dry-gas meter volumes; **and** divide the difference by 10 to get the leak rate. The leak rate ~~shall~~ **must** not exceed $0.00057 \text{ m}^3/\text{min}$ **cubic meters per minute** (~~0.02 cfm cubic feet per minute~~). After each field use, the calibration of the metering system ~~shall~~ **must** be checked by performing 3 calibration runs at a single, intermediate orifice setting, based on the previous field test, with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet-test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5%, then recalibrate the meter over the full range of orifice settings, as outlined in APTD²-0576. Alternatively, a spirometer may be substituted for a wet-test meter in the above calibration procedures. Alternative procedures, such as using the orifice meter coefficients, may be used, subject to the approval of the department. If the dry-gas meter coefficient values obtained before and after a test series differ by more than 5%, then the test series ~~shall~~ **must** be performed using whichever meter coefficient value, (~~before or after,~~) gives the lower value of total sample volume.

(iv) Temperature gauges. Use the procedure in ~~section 4.3~~ of method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as those used for the dry-gas meter and condenser outlet, ~~shall~~ **must** be calibrated against mercury-in-glass thermometers.

(v) Leak check of metering system shown in figure 102 **under R 336.2021**. That portion of the sampling train from the pump to the orifice meter ~~shall~~ **must** be leak-checked before initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested, **also** (~~see figure 107 under R 336.2021~~): Close the main valve on the meter box. Insert a 1-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 **centimeters**, (~~5 to 7 in-~~**ches**), water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 minute. A loss of pressure on the manometer indicates a leak in the meter box; ~~Leaks~~ **Leaks**, if present, ~~shall~~ **must** be corrected.

(vi) Barometer. Calibrate against a mercury barometer.

(f) Calculations. When carrying out calculations, retain at least 1 extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used if ~~they~~ **the other forms of the equations** give equivalent results. The following provisions apply to calculations:

(i) -Nomenclature:

A_n = Cross-sectional area of nozzle, **meters²** (~~or the equivalent feet²~~).

A = Cross-sectional area of stack or flue at the point of sampling, **feet²**.

B_{ws} = Water vapor in the gas stream, proportion by volume, expressed as a fraction.

B_{wi} = Percent water vapor in gas entering source particulate control device determined by method 4.

- B_{wo} = Percent water vapor in gas exiting source particulate control device.
- C_a = Wash blank residue concentration, **milligrams per gram**.
- C_s = Concentration of particulate matter in stack gas, pounds per 1,000 pounds of actual stack gas.
- C_{sD} = Concentration of particulate matter in stack gas, moisture excluded, pounds per 1000 pounds of dry stack gas.
- C_{s50} = Concentration of particulate matter corrected to 50% excess air, pounds per 1000 pounds of stack gas.
- C_{s50D} = Concentration of particulate matter corrected to 50% excess air, excluding any water addition from a collector, pounds per 1000 pounds of stack gas.
- E = Mass emission rate of particulate, **lbpounds/hour**.
- F_{50} = Concentration conversion factor to 50% excess air with no moisture alterations in exhaust.
- F_{50D} = Concentration conversion factor to 50% excess air, excluding any moisture added to exhaust gas by pollution collection system.
- F_D = Concentration conversion factor to dry basis, excluding any water in the stack gas.
- I = Percent of isokinetic sampling.
- L_a = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 **meters³/minute (0.02 cubic feet per minute)** or 4% of the average sampling rate, whichever is less.
- L_i = Individual leakage rate observed during the leak check conducted before the "ith" component change ($i = 1, 2, 3 \dots n$), **meters³/minute (cubic feet per minute)**.
- L_p = Leakage rate observed during the post-test leak check, **meters³/minute (cubic feet per minute)**.
- M_d = Molecular weight of dry stack gas, **gram/gram mole (lbpound/lbpound-mole)**, calculated by method 3, equation 3-12, using data from integrated method 3.
- m_n = Total amount of particulate matter collected, **milligram**.
- M_w = Molecular weight of water, 18.0 **gram/gram-mole (18.0 lbpound/lbpound-mole)**.
- m_a = Mass of residue of solvent after evaporation, **milligram**.
- m_g = Total weight of gas samples through nozzle, **lbpound**.
- P_{bar} = Barometric pressure at the sampling site, **millimeter mercury Hg (inches Hgmercury)**.
- P_s = Absolute stack gas pressure.
- P_{std} = Standard absolute pressure, 760 **millimeters Hgmercury (29.92 inches Hgmercury)**.
- R = Ideal gas constant, 0.06236 ~~mm /°K-g-mole~~ **millimeters of mercury-cubic meters per kelvin-gram-mole, (21.85 in.Hg-ft.³/R²lb-mole inches of mercury-cubic feet per Rankine-pound-mole)**.
- T_m = Absolute average dry-gas meter temperature, see figure 104 **under R 336.2021, °Kelvin, (°Rankine)**.
- T_s = Absolute average stack gas temperature, see figure 104 **under R 336.2021, °Kelvin, (°Rankine)**.

- T_{std} = Standard absolute temperature, **294.1°Kelvin, (530°Rankine)**.
 V_a = Volume of solvent blank, **millileters**.
 V_{aw} = Volume of solvent used in wash, **millileters**.
 V_{lc} = Total volume of liquid collected in impingers and silica gel (see figure 106 **under R 336.2021**), **millileters**.
 V_m = Volume of gas sample as measured by the dry-gas meter, **deci-centimeter, (deci-cubic-foot)**.
 $V_{m(std)}$ = Volume of gas sample measured by the dry-gas meter, corrected to standard conditions, **deci-standard cubic meter, (deci-standard cubic foot)**.
 $V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, **standard cubic meter, (standard cubic foot)**.
 V_s = Stack gas velocity, calculated by method 2, ~~equation 2-9~~, using data obtained from method 5, **meters/second (feet/second)**.
 W_a = Weight of residue in solvent wash, **milligram**.
 Y = Dry-gas meter calibration factor.
 ΔH = Average pressure differential across the orifice meter (see figure 104 **under R 336.2021**), **millimeter water H₂O (inches waterH₂O)**.
 $\%O_2$ = Percent oxygen in stack gas by volume (dry basis).
 $\%N_2$ = Percent nitrogen in stack gas by volume (dry basis).
 p_a = Density of solvent, **milligrams/millileter**.
 $p_{s(std)}$ = Density of all sampled gas at standard conditions, **lbpounds/feet.³**
 p_w = Density of water, **0.9982 grams/millileter (0.002201 lbpounds/millileter)**.
 θ = Total sample time, **minute**.
 θ_1 = Sample time, interval, from the beginning of a run until -the -first component change, **minute**.
 θ_i = Sampling time interval, between 2 successive component changes, beginning with the interval between the first and second changes, **minute**.
 θ_p = Sampling time interval, from the final (nth) component change until the end of the sampling run, **minute**.
13.6 = Specific gravity of mercury.
60 = **Seconds/minute**.
100 = Conversion to percent.
386.9 = Cubic feet per **lbpound**-mole of ideal gas at standard conditions.
453.6 = Conversion of pounds to grams.
3600 = Conversion of hours to **seconds**.
1000 = Conversion of 1000 **lbpound** units to **lbpound** units.

(ii) Average the dry-gas meter temperature and average the orifice pressure drop. See data sheet, (figure 5-2 **104 under R 336.2021**).

(iii) Dry gas volume. Correct the sample volume measured by the dry-gas meter to standard conditions, (21.11 degrees Centigrade, 760 **millimeters Hgmercury** or 70 degrees Fahrenheit, 29.92 **inches Hgmercury**), by using equation 5-1.

Eequation 5-1:

$$V_{m(std)} = \frac{V_m Y T_{std} (P_{bar} + \Delta H / 13.6)}{T_m P_{std}} = K_1 V_m Y \frac{(P_{bar} + \Delta H / 13.6)}{T_m}$$

Where:

$$K_1 = 0.3869 \text{ }^\circ\text{K/mm Hg for metric units.}$$

$$= 17.71 \text{ }^\circ\text{R/in. Hg for English units.}$$

Equation 5-1 may be used as written. However, if the leakage rate observed during any of the mandatory leak checks, (for example, the post-test leak check or leak checks conducted before component changes,) exceeds L_a , equation 5-1 **must shall** be modified **as follows: in the following manner:**

(A) Case I. No component changes made during sampling run. In this case, replace V_m in equation 5-1 with the following expression:

$$V_m - (L_p - L_a) \theta$$

(B) Case II. One or more component changes made during the sampling run. In this case, replace V_m in equation 5-1 by the following expression:

$$V_m - (L_i - L_a) \theta_1 - \sum_{i=2}^n (L_i - L_a) \theta_i - (L_p - L_a) \theta_p$$

and substitute only for those leakage rates (L_i or L_p) that exceed L_a .

(iv) Volume of water vapor.

Equation 5-2

$$V_{w(std)} = V_{lc} (p_w / M_w) (R T_{std} / P_{std}) = K_2 V_{lc}$$

Where:

$$K_2 = 0.001338 \text{ m}^3/\text{ml for metric units.}$$

$$= \mathbf{0.04733 \text{ ft.}^3/\text{ml for English units.}$$

(v) Moisture content.

Equation 5-3

$$B_{ws} = V_{w(std)} / (V_{m(std)} + v_{w(std)})$$

In saturated or water droplet-laden gas streams, 2 calculations of the moisture content of the stack gas **shall must** be made: 1 from the impinger analysis, (equation 5-3), and a second from the assumption of saturated conditions. The lower of the 2 values of B_{ws} **shall-must** be considered correct. The procedure for determining the moisture content based ~~upon on the~~ assumption of saturated conditions **as described in-is given in the**

~~note of section 1.2 of 40 CFR part 60 appendix A~~ method 4. For the purpose of this method, the average stack gas temperature from figure 104 **under R 336.2021** may be used to make the determination, if the accuracy of the in-stack temperature sensor is ± 1 degree Centigrade, (± 2 degrees Fahrenheit).

(vi) Solvent blank concentration.

Eequation 5-4

$$C_a = m_a / (V_a P_a)$$

(vii) Solvent wash blank.

Eequation 5-5

$$W_a = C_a V_{aw} P_a$$

(viii) Total particulate weight. Determine the total particulate catch from the sum of the weights obtained from containers 1, 1A, and 2 less the wash solvent blank, (~~see figure 106 under R 336.2021~~). Refer to subdivision (d)(i)(E) of this rule to assist in the calculation of results involving 2 or more pairs of filters or 2 or more sampling trains.

(ix) Sampled gas density. Determine the density of the gas sampled from the stack, at standard conditions **in pounds per cubic foot**, (lb/ft^3).

Eequation 5-6

$$P_{s(std)} = (M_d (1 - B_{ws}) + M_w B_{ws}) / 386.9$$

(x) Total weight of gas sampled, (lbs).

Eequation 5-7

$$m_g = (V_{m(std)} + V_{w(std)}) P_{s(std)}$$

(xi) Particulate concentration, ($\text{lbs}/1000 \text{ lbs}$).

Eequation 5-8

$$C_s = m_n / (453.6 m_g)$$

(xii) Excess air and moisture correction factors:

(A) Correction factor to 50% excess air for those sources with or without ~~any~~ a particulate collector where no increase in moisture content of the exhaust gas occurs after the process and before the point of sampling.

Equation 5-9

$$F_{50} = \frac{M_d + 18 B_{wo} / (100 - B_{wo})}{0.1826 \% N_2 - 2.0592 \% O_2 + M_d + 18 B_{wo} / (100 - B_{wo})}$$

(B) Correction factor to 50% excess air for those sources with a wet collection device, (scrubber,) that increases the moisture content of the exhaust gas after the process and before the point of sampling.

Equation 5-10

$$F_{50D} = \frac{M_d + 18 B_{wo} / (100 - B_{wo})}{0.1826 \% N_2 - 2.0592 \% O_2 + M_d + 18 B_{wi} / (100 - B_{wi})}$$

(C) Correction factor to convert the actual concentration, C_s , to dry conditions.

Equation 5-11

$$F_D = \frac{M_d + 18 B_{wo} / (100 - B_{wo})}{M_d}$$

(xiii) Converted particulate concentrations, where applicable under the department's rules or permit.

Equation 5-12

$$C_{s50} = C_s F_{50}$$

Equation 5-13

$$C_{s50D} = C_s F_{50D}$$

Equation 5-14

$$C_{sD} = C_s F_D$$

(xiv) Mass emission rate **in pounds per hour**, (lb/hr).

Equation 5-15

$$E = \frac{3600 A V_s C_s P_s T_{std} P_{s(std)}}{1000 T_s P_{std}} = K_3 A V_s C_s P_s P_{s(std)} / T_s$$

Where:

$K_3 = 63.77$ for English units.

(xv) Isokinetic variation **using 1 of the following methods:**

(A) Calculation from raw data.

Equation 5-16

$$I = \frac{100 T_s (K_4 V_{lc} + (V_m / T_m)(P_{bar} + \Delta H / 13.6))}{60 \theta V_s P_s A_n}$$

Where:

$K_4 = 0.003458$ mm Hg - m³ml - °K for metric units.

$= 0.002672$ in. Hg - ft.³/ml - °R for English units.

(B) Calculation from intermediate values.

Equation 5-17

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} V_s \theta A_n P_s 60(1 - B_{ws})} = K_5 \frac{T_s V_{m(std)}}{P_s V_s A_n \theta (1 - B_{ws})}$$

Where:

$K_5 = 4.307$ for metric units.

$= 0.09409$ for English units.

(xvi) Acceptable results. If 90%≤I≤110%, then the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or if I is less than 90%, then the department may opt to accept the results. Otherwise, reject the results and repeat the test.

(g) Bibliography:

(i) Federal Register, Volume 42, No. 160, Part 160, Chapter 1, Title 40, Appendix A, Method 5, August 18, 1977.

(ii) Martin, Robert M. Construction Details of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0581. April, 1971.

(iii) Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0576. March, 1972.

(iv) Shigehara, R. T. "Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights." Stack Sampling News, 2:4 - 11. October, 1974.

(v) Guidelines for Source Testing of Particulate. Michigan Department of Natural Resources, Air Quality Division. June 1, 1977.

R 336.2012 Reference test method 5C.

Rule 1012. Reference test method 5C, out-stack filtration method, reads as follows:

(a) The principle, applicability, and performance test criteria are as follows:

(i) Principle. Particulate matter is withdrawn iso-kinetically from the source and collected on solid filtering media maintained at a temperature in the range of 120 ± 14 degrees Centigrade, (248 ± 25 degrees Fahrenheit,) or ~~such other~~ **another** temperature as specified by the department's rules or a permit condition, or as approved by the department for a particular application. The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after removal of uncombined water.

(ii) Applicability. This method is applicable for the determination of particulate emissions from stationary sources as identified in table 31 of R 336.1331. The method is also applicable when specifically provided for in the department's rules, orders, a permit to install, or a permit to operate.

(iii) Performance test criteria **as follows**:

(A) A performance test ~~must~~**shall** ~~consist~~ **meet the requirements under R 336.2003(2)**. ~~Of a minimum of 3 separate samples of a specific air contaminant conducted within a 36-hour period, unless otherwise authorized by the department. Each of the 3 separate samples shall be obtained while the source is operating at a similar production level. For the purpose of determining compliance with an applicable emission limit, rule, or permit condition, the arithmetic mean of results of the 3 samples shall apply. If a sample is accidentally lost or conditions occur in which 1 of the 3 samples must be discontinued because of forced shutdown, failure of an irreplaceable portion of the sampling train, extreme meteorological conditions, or other circumstances beyond the owner's or operator's control, compliance may, upon the approval of the department, be determined using the arithmetic mean of the results of 2 samples.~~

(B) For ~~any~~ **any** sources that ~~is~~ **are** subject to an emission limitation calculated to 50% excess air, the multipoint, integrated sampling procedure of R 336.2004(1)(c) ~~shall~~ **must** be used for gas analysis. For all other sources that require a determination of the molecular weight of the exhaust, ~~any~~ **an** optional sampling procedure of R 336.2004(1)(c) may be used. Alternatives or modifications to procedures are subject to the approval of the department.

(C) The minimum volume per sample ~~shall~~ **must** be 30 cubic feet of dry gas corrected to standard conditions, (70 degrees Fahrenheit, 29.92 inches mercury). Minimum sample time ~~shall~~ **must** be 60 minutes, which may be continuous or a combination of shorter sampling periods for sources that operate in a cyclic manner. Smaller sampling times or sample volumes, when necessitated by process variables or other factors, may be approved by the department.

(D) For ~~any~~ **a** source whose emission control device alters the moisture content of the exhaust gas, a moisture determination ~~shall~~ **must** be performed in a location upstream from the emission control device and in accordance with R 336.2004(1)(d) or an alternative method approved by the department.

(b) The following provisions apply to apparatus:

(i) Sampling train. A schematic of the sampling train used in this method is shown in figure 103 **under R 336.2021**. Construction details for many, but not all, of the train components are given in APTD-0581, (subdivision (g)(ii) of this rule). For changes from

the APTD-0581 document and for allowable modifications to figure 103 **under R 336.2021**, consult with the department. The operating and maintenance procedures for many, but not all, of the sampling train are described in APTD-0576, **adopted by reference in R 336.1902, and referenced under** (subdivision (g)(iii) of this rule). Since correct usage is important in obtaining valid results, all users shall read APTD-0576 and adopt the applicable operating and maintenance procedures outlined in it, unless otherwise specified ~~herein~~. The sampling train consists of the following components:

(A) Probe nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper ~~shall~~ **must** be less than 30 degrees and the taper ~~shall~~ **must** be on the outside to preserve a constant internal diameter. The probe nozzle ~~shall~~ **must** be of the buttonhook design, unless otherwise specified by the department. If made of stainless steel, the nozzle ~~shall~~ **must** be constructed from seamless tubing. Other materials of construction may be used, subject to the approval of the department. A range of nozzle sizes suitable for isokinetic sampling ~~shall~~ **must** be available, for example, 0.32 to 1.27 centimeters, (1/8 to 1/2 in-~~ch~~,) or larger if higher volume sampling trains are used inside diameter (ID) nozzles in increments of 0.16 centimeters, (1/16 in-~~ches~~). Each nozzle ~~shall~~ **must** be calibrated according to the procedures outlined in subdivision (e) of this rule.

(B) Probe liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of 120 ± 14 degrees Centigrade, (248 ± 25 degrees Fahrenheit), another temperature as specified by the department's rules, or a temperature approved by the department for a particular application. The tester may opt to operate the equipment at a temperature lower than that specified. Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581, **adopted by reference in R 336.1902**, which utilize the calibration curves of APTD-0576, or calibrated according to the procedure outlined in APTD-0576, **adopted by reference in R 336.1902**, are acceptable. Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480 degrees Centigrade, (900 degrees Fahrenheit); quartz liners ~~shall~~ **must** be used for temperatures between 480 and 900 degrees Centigrade, (900 and 1,650 degrees Fahrenheit). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the department. The softening temperature for borosilicate is 820 degrees Centigrade, (1,508 degrees Fahrenheit,) and for quartz it is 1,500 degrees Centigrade, (2,732 degrees Fahrenheit). When practical, every effort ~~shall~~ **must** be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners, such as 316 stainless steel, Incoloy 825, or other corrosion resistant materials made of seamless tubing, may be used, subject to the approval of the department.

(C) Pitot tube. Type S, as described in ~~section 2.1 of method 2~~, or ~~other~~ **another** device approved by the department. The pitot tube ~~shall~~ **must** be attached to the probe, as shown in figure 103 **under R 336.2021**, to allow constant monitoring of the stack gas velocity. The impact, (high pressure,) opening plane of the pitot tube ~~shall~~ **must** be even with or above the nozzle entry plane, (see method 2, figure 2-6b **Velocity Traverse Data**,) during sampling. The type S pitot tube assembly ~~shall~~ **must** have a known coefficient, determined as outlined in ~~section 4 of method 2~~.

(D) Differential pressure gauge. Incline manometer or equivalent devices (2), as described in ~~section 2.2 of~~ method 2. One manometer ~~shall~~ **must** be used for velocity head (p) readings, and the other ~~shall~~ **must** be used for orifice differential pressure readings.

(E) Filter holders. Two separate filter holders in series or 1 filter holder with separate filter supports and seals for 2 filters. One filter holder with 2 filters held in contact with each other is not acceptable. Materials of construction may be stainless steel (316), glass, ~~teflon~~ **Teflon**, or ~~such other~~ **another** material approved by the department.

(F) Filter heating system. Any heating system capable of maintaining a temperature around the filter holder during sampling of 120 ± 14 degrees Centigrade, (248 ± 25 degrees Fahrenheit), another temperature as specified by the department's rules or a permit condition, or a temperature approved by the department for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3 degrees Centigrade, (5.4 degrees Fahrenheit,) ~~shall~~ **must** be installed so that the temperature around the filter holders can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

(G) Condenser. The following system ~~shall~~ **must** be used to determine the stack gas moisture content: Three impingers connected in series with leak-free ground glass fittings or any similar leak-free non-contaminating fittings. All impingers ~~shall~~ **must** be of the Greenburg-Smith design and ~~shall~~ **must** be modified by replacing the tip with a 1.3 centimeters, ($1/2$ in.-ch.) ~~ID~~ **inside diameter** glass tube extending to about 1.3 centimeters, ($1/2$ in.-ch.) from the bottom of the flask. Modifications, such as using flexible connections between the impingers or using materials other than glass, are ~~permitted~~ **allowed** subject to the approval of the department's staff. The first impinger ~~shall~~ **must** contain a known quantity of water-, (~~as described in~~ subdivision (d)(i)(C) of this rule), the second ~~shall~~ **must** be empty, and the third ~~shall~~ **must** contain a known weight of silica gel or equivalent desiccant. Alternatively, ~~any~~ a system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, ~~each~~ to within 1 milliliter or 1 gram, may be used subject to the approval of the department. In any case, the means for measuring the moisture leaving the condenser ~~shall~~ **must** be by passing the sample gas stream through a tared silica gel, or equivalent desiccant, trap with exit gases kept below 20 degrees Centigrade, (68 degrees Fahrenheit,) and determining the weight gain. If a determination of the particulate matter collected in the impingers is required by the department's rules, a permit to install, or a permit to operate, the impinger system described in this subparagraph ~~shall~~ **must** be used, without modification. Contact the department as to the sample recovery and analysis of the impinger contents.

(H) Metering system. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3 degrees Centigrade, (5.4 degrees Fahrenheit), drygas meter capable of measuring volume to within 2%, and related equipment as shown in figure 103 **under R 336.2021**. Other metering systems capable of maintaining sampling rates within 10% of isokinetic and capable of determining sample volumes to within 2% may be used, subject to the approval of the department. When the metering system is used in conjunction with a pitot tube, the system ~~shall~~ **must** enable checks of isokinetic rates. Sampling trains utilizing metering systems designed for higher flow rates than

those described in APTD-0581 or APTD-0576, **both adopted by reference in R 336.1902**, may be used if the specifications of this method are met.

(I) Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within **2.5 millimeters Hgmercury, (0.1 in.-ch Hgmercury)**. In many cases, the barometric reading may be obtained from a nearby national weather service station. When obtained from this source, the station value, which is the absolute barometric pressure, ~~shall~~ **must** be requested and an adjustment for elevation differences between the weather station and sampling point ~~shall~~ **must** be applied at a rate of minus **2.5 millimeters Hgmercury per 30 meters, (0.1 in.-ch Hgmercury,) per 30 m (100 foot-)**, elevation increase or vice versa for elevation decrease.

(J) Gas density determination equipment. Temperature sensor and pressure gauge, as described in ~~sections 2.3 and 2.4 of method 2~~, and gas analyzer, if necessary, as described in method 3. The temperature sensor ~~shall~~ **must**, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration ~~such so~~ that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just ~~prior to~~ **before** use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor ~~shall~~ **must** be placed in an interference-free arrangement with respect to the type S pitot tube openings, (see method 2, figure 2.7). As a second alternative, if a difference of not more than 1% in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube. This alternative is subject to the approval of the department.

(ii) Sample recovery. The following items ~~are needed~~:

(A) Probe-liner and probe-nozzle brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush ~~shall~~ **must** have extensions, at least as long as the probe, made of stainless steel, nylon, ~~teflon~~ **Teflon**, or similarly inert material. The brushes ~~shall~~ **must** be properly sized and shaped to brush out the probe liner and nozzle.

(B) Wash ~~bottles - 2~~ **bottles - 2**. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

(C) Glass sample storage containers. Chemically resistant, borosilicate glass bottles, for acetone washes, ~~500 ml~~ or **1000 milliliters**. Screw cap liners ~~shall~~ **must** either be rubber-backed ~~teflon~~ **Teflon** or ~~shall~~ **must** be constructed so as to be leak-free and resistant to chemical attack by acetone. Narrow-mouth glass bottles have been found to be less prone to leakage. Alternatively, polyethylene bottles may be used.

(D) Filter containers. Glass, polyethylene, or aluminum tube containers, unless otherwise specified by the department.

(E) Graduated cylinder or balance. To measure condensed water to within **1 milliliter** or **1 gram**. Graduated cylinders ~~shall~~ **must** have subdivisions of not more than **2 milliliters**. Most laboratory balances are capable of weighing to the nearest **0.5 gram** or less. Any of these balances are suitable for use here and in paragraph (iii)(D) of this subdivision.

(F) Plastic storage containers. Airtight containers to store silica gel.

(G) Funnel and rubber policeman-~~F~~, to aid in the transfer of silica gel to container; not necessary if silica gel is weighed in the field.

(H) Funnel: ~~made from G~~ **made from G** glass or polyethylene, to aid in sample recovery.

(iii) Analysis: ~~must include~~ ~~the following equipment is needed for analysis:~~

- (A) Glass weighing dishes.
- (B) Desiccator.
- (C) Analytical balance, ~~to~~ to measure to within 0.1 milligrams.
- (D) Balance, ~~to~~ to measure to within 0.5 milligrams.
- (E) Beakers, 250 milliliters.
- (F) Hygrometer, ~~to~~ to measure the relative humidity of the laboratory environment.
- (G) Temperature gauge, ~~to~~ to measure the temperature of the laboratory environment.

(c) The following provisions apply to reagents:

(i) ~~Sampling~~. The reagents used in sampling are as follows:

(A) Filters. Two outstack filters may be any combination of alundum ceramic thimble filters, type RA-98 or glass fiber filters, type A without organic binder. The size of ~~such~~ ~~the~~ filters ~~shall~~ **must** allow proper sampling rates to maintain isokinetics using the nozzle sizes specified in subdivision (b)(i)(A) of this rule. Alternatively, other types of filters may be used, subject to the approval of the department.

(B) Silica gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175 degrees Centigrade, (350 degrees Fahrenheit), for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants, (equivalent or better,) may be used, subject to the approval of the department.

(C) Water. When analysis of the material caught in the impingers is required, distilled water ~~shall~~ **must** be used. Run blanks prior to field use to eliminate a high blank on test samples.

(D) Crushed ice.

(E) Stopcock grease. Acetone-insoluble, heatstable silicone grease. This is not necessary if screw on connectors with ~~teflon~~ **Teflon** sleeves, or equivalent, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the department.

(ii) Sample recovery, ~~W~~ washing solvent. Either acetone or distilled water may be used for sample recovery. If acetone is used for washing solvent, then reagent grade, less than 0.001% residue, in glass bottles is required. Acetone from metal containers generally has a high residue blank and ~~shall~~ **must** not be used. Suppliers sometimes transfer acetone to glass bottles from metal containers; ~~thus,~~ **so** acetone blanks ~~shall~~ **must** be run ~~prior to~~ **before** field use and only acetone with low blank values, (less than 0.001%,) ~~shall~~ **must** be used. A blank value of more than 0.001% of the weight of acetone used ~~shall~~ **must** not be subtracted from the sample weight. If distilled water is used for washing solvent, use distilled water with less than 0.001% residue. Run blanks before field use to eliminate a high blank on test samples.

(iii) ~~Analysis~~. Two reagents are required for the analysis:

(A) Solvent. Same as paragraph (ii) of this subdivision for quantitative transfer.

(B) Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the department.

(d) The following provisions apply to procedure:

(i) Sampling. The complexity of this method is such that, in order to obtain reliable results, testers shall be trained and experienced with the test procedures. Sampling ~~shall~~ **must** comply with the following provisions:

(A) Pretest preparation. All the components ~~shall~~ **must** be maintained and calibrated according to the applicable procedures described in APTD-0576, **adopted by reference in R 336.1902**, unless otherwise specified in this rule. Weigh several 200 to 300 **gram** portions of silica gel in airtight containers to the nearest 0.5 **gram**. Record the total weight of the silica gel plus container on each container. As an alternative, the silica gel need not be pre-weighed, but may be weighed directly in its impinger or sampling holder just ~~prior to~~ **before** train assembly. Check filters visually against light for irregularities, flaws, pinhole leaks, or cracks. Label filters of the proper size on the back side using numbering machine ink. As an alternative, label the shipping containers, **described in** (subdivision (b)(ii)(D) of this rule,) and keep the filters in these containers at all times, except during sampling and weighing. Dry the filters in an oven at 105 degrees Centigrade, (220 degrees Fahrenheit), for a minimum of 2 hours, cool for at least 1 hour in a desiccator containing anhydrous calcium sulfate, and individually weigh and record **each weight** ~~their weights~~ to the nearest 0.1 **milligram**. During the weighing, the filters ~~shall~~ **must** not be exposed to the laboratory atmosphere for a period of more than 2 minutes and a relative humidity above 50%. Procedures, other than those specified, that account for relative humidity effects may be used, subject to the approval of the department.

(B) Preliminary determinations. Select the sampling site and the minimum number of sampling points according to method 1 or as specified by the department. Determine the stack pressure, temperature, and the range of velocity heads using method 2. ~~It is~~ recommended that a leak check of the pitot lines, (see method 2, ~~section 3.1~~), be performed. Determine the moisture content using approximation method 4, or its alternatives, for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in method 2, ~~section 3.6~~; ~~if~~ **If** integrated method 3 sampling is used for molecular weight determination, the integrated bag sample ~~shall~~ **must** be taken simultaneously with, and for the same total length of time as, the particulate sample run. Select a nozzle size based on the range of velocity heads so that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered, (see ~~section 2.2 of method 2~~). Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes. Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry so that the sampling time per point is not less than 5 minutes, unless approved by the department, or some greater time interval as specified by the department, and so that the sample volume taken, corrected to standard conditions, exceeds the required minimum total gas sample volume. The latter is based on an approximate average sampling rate. It is recommended that the number of minutes sampled at each point be an integer or an integer plus 1/2 minute to avoid timekeeping errors. In some circumstances, such as in batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the department's approval ~~shall~~ **must** first be obtained.

(C) Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just before

assembly or until sampling is about to begin. Place 100 **milliliters** of water in the first impinger, leave the second impinger empty, and transfer approximately 200 to 300 **grams** of pre-weighed silica gel from its container to the third impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 **gram** and recorded. Using tweezers or clean disposable surgical gloves, place a labeled, (~~identified~~), and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed. When glass liners are used, install the selected nozzle using a Viton A **O**-**O**-ring when stack temperatures are less than 260 degrees Centigrade, (~~500 degrees Fahrenheit~~), and an asbestos string gasket when temperatures are higher. See APTD-0576, **adopted by reference in R 336.1902**, for details. Other connecting systems using either 310 stainless steel or ~~teflon~~ **Teflon** ferrules may be used. When metal liners are used, install the nozzle in the same manner as for glass liners or by a leak-free direct mechanical connection. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Set up the train as in figure 103 **under R 336.2021**. If necessary, use a very light coat of silicone grease on all ground glass joints. Grease only the outer portion, (~~see APTD-0576, adopted by reference in R 336.1902,~~) to avoid the possibility of contamination by the silicone grease. Place crushed ice around the impingers.

(D) Leak check procedures **as follows**:

(I) Pretest leak check. A pretest leak check is strongly recommended, but not required, to prevent invalid sampling and wasted time. If the tester opts to conduct the pretest leak check, the following procedure ~~shall~~ **must** be used: After the sampling train has been assembled, turn it on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A **O****O**-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak check the train at the sampling site by plugging the nozzle and pulling a 380 **millimeter mercury, Hg** (~~15 inch- Hgmercury,~~) vacuum. A lower vacuum may be used, if it is not exceeded during the test. If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak check the train by first plugging the inlet to the filter holder, (~~and cyclone, if applicable,~~) and pulling a 380 **millimeter Hgmercury, (15 inch- Hgmercury),** vacuum. A lower vacuum may be used if it is not exceeded during the test. Then connect the probe to the train and leak check at about a 25 **millimeter Hgmercury, (1 inch- Hgmercury),** vacuum. ~~Alternatively~~ **alternatively**, the probe may be leak checked with the rest of the sampling train, in 1 step, at a 380 **millimeter Hgmercury, (15 inch- Hgmercury),** vacuum. Leakage rates in excess of 4% of the average sampling rate or 0.00057 **cubic meters per minute** ~~m³/min~~ (0.02 **cubic feet per minute** ~~cfm~~), whichever is less, are unacceptable. The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with the bypass valve fully open and the coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve, ~~;~~ **as** this will cause water to back up into the filter holder. If the desired vacuum is

exceeded, either leak check at this higher vacuum or end the leak check and start over. When the leak check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone, (if applicable), and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and prevents silica gel from being entrained backward into the third impinger.

(II2) Leak checks during sample run. If, during the sampling run, a component, (such as a filter assembly or impinger), change becomes necessary, a leak check ~~shall~~ **must** be conducted immediately before the change is made. The leak check ~~shall~~ **must** be done according to the procedure outlined in ~~paragraph (i)(D)(II) of this subdivision~~ **subparagraph (D) (I) of this paragraph**, except that it ~~shall~~ **must** be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be not more than 0.00057 **cubic meters per minute, m^3/min** (0.02 **cubic feet per minute, cfm**) or 4% of the average sampling rate, whichever is less, the results are acceptable and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume, as shown in subdivision (f)(iii) of **R 336.2011** ~~this rule~~, or shall void the sampling run. Immediately after component changes, leak checks are optional. ~~If the if such~~ **if such** leak checks are done, the procedure outlined in paragraph (i)(D)(II) of this subdivision ~~shall~~ **must** be used.

(III3) Post-test leak check. A leak check is mandatory at the conclusion of each sampling run. The leak check ~~shall~~ **must** be done in accordance with the procedures outlined in paragraph (i)(D)(II) of this subdivision, except that it ~~shall~~ **must** be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be not more than 0.00057 **m^3/min cubic meters per minute, (0.02 cfm cubic feet per minute,)** or 4% of the average sampling rate, whichever is less, the results are acceptable and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume, as shown in subdivision (f)(iii) of **R 336.2011** ~~this rule~~, or shall void the sampling run.

(E) Particulate train operation. During the sampling run, maintain an isokinetic sampling rate that is within 10% of true isokinetic, unless otherwise specified by the department. For each run, record the data required on a data sheet such as the one shown in figure 104 **under R 336.2021**. Be sure to record the initial dry-gas meter reading. Record the dry-gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by figure 104 **under R 336.2021** at least once at each sample point during each time increment, and take additional readings when significant changes, (20% variation in velocity head readings), necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse. Clean the portholes ~~prior to~~ **before** the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap and verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs that aid in the rapid adjustment of the isokinetic sampling rate without excessive

computations are available. These nomographs are designed for use when the type S pitot tube coefficient is 0.85 ± 0.02 and the stack gas equivalent density, (dry molecular weight,) is equal to 29 ± 4 . APTD-0576, **adopted by reference in R 336.1902**, details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps, (see subdivision (g)(iv) of this rule,) are taken to compensate for the deviations. When the stack is under significant negative pressure, (height of impinger stem,) take care to pull low flow when inserting the probe into the stack to prevent water from backing into the sample tubing and to avoid pulsation through the filter and possible loss of materials. When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream. Traverse the stack cross section, as required by method 1 or as specified by the department, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes.; ~~this~~ **This** minimizes the chance of extracting deposited material. During the test run, add more ice and, if necessary, salt to maintain a temperature of less than 20 degrees Centigrade, (68 degrees Fahrenheit,) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer. If the pressure drop across the filter becomes too high and makes isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak check, (see ~~subparagraph paragraph (i)(D)(2II)~~ **paragraph subdivision**). The total particulate weight ~~shall~~ **must** include the summation of all filter assembly catches. A single train ~~shall~~ **must** be used for the entire sample run, except in cases where simultaneous sampling is required in 2 or more separate ducts, at 2 or more different locations within the same duct, or where equipment failure necessitates a change of trains. In all other situations, the use of 2 or more trains ~~shall~~ **must** be subject to the approval of the department. Note that when 2 or more trains are used, separate analyses of the front half catches from the individual trains may be combined, as may the impinger catches, and 1 analysis of the front half catch and 1 analysis of impinger catch may be performed. Consult with the department for details concerning the calculation of results when 2 or more trains are used. At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak check, as outlined in ~~paragraph~~ **subparagraph (i)(D)(3III) of this paragraph**. Also, leak check the pitot lines as described in method 2, ~~section 3.1; the~~. **The** lines ~~shall~~ **must** pass this leak check to validate the velocity head data.

(F) Calculation of percent isokinetic. Calculate percent isokinetic, (see subdivision (f) of this rule,) to determine whether the run was valid or whether another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the department for possible variance on the isokinetic rates.

(ii) Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this creates a vacuum in the filter holder and draws water from the impingers into the filter holder.

Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Ground-glass stoppers, plastic caps, or serum caps may be used to close these openings. Transfer the probe and filter-impinger assembly to the cleanup area. This area ~~shall~~ **must** be clean and protected from the wind so that the chances of contaminating or losing the sample are minimized. Save a portion of the solvent used for cleanup as a blank. Take 200 milliliters of this solvent directly from the wash bottle being used and place it in a glass sample container labeled "solvent blank." Inspect the train prior to and during disassembly and note ~~any~~ abnormal conditions. Treat the samples as follows:

(A) Container ~~Nos. numbers~~ 1, 1A. Carefully remove the filters from the filter holders and place **each filter in its** ~~in their~~ identified containers. Use a pair of tweezers or clean disposable surgical gloves, or both, to handle the filters. Carefully transfer to the container any particulate matter or filter fibers, or both, that adhere to the filter holder gasket by using a dry nylon bristle brush or sharp-edged blade, or both. Seal the container.

(B) Container ~~No. number~~ 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, the ~~tester~~ shall quantitatively recover from particulate matter or any condensate from the nozzle, probe fitting, probe liner, and from both filter holders by washing these components with solvent and placing the wash in a glass container. Perform the solvent rinses as follows: Carefully remove the probe nozzle and clean the inside surface by rinsing with solvent from a wash bottle and brushing with a nylon bristle brush. Brush until the solvent rinse shows no visible particles and then make a final rinse of the inside surface with solvent. Brush and rinse the inside parts of the Swagelok fitting with solvent in a similar way until no visible particles remain. Rinse the probe liner with solvent by tilting and rotating the probe while squirting solvent into its upper end so that all inside surfaces are wetted with acetone. Let the solvent drain from the lower end into the sample container. A glass or polyethylene funnel may be used to aid in transferring liquid washes to the container. Follow the solvent rinse with a probe brush. Hold the probe in an inclined position and squirt solvent into the upper end as the probe brush is being pushed with a twisting action through the probe. ~~;~~ ~~hold~~ **Hold** a sample container underneath the lower end of the probe and catch any solvent and particulate matter that is brushed from the probe. Run the brush through the probe 3 or more times until no visible particulate matter is carried out with the solvent or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through, in the above prescribed manner, not less than 6 times, since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with solvent and quantitatively collect these washings in the sample container. After the brushing, make a final solvent rinse of the probe as described above. It is recommended that 2 people ~~be used to~~ clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints have been wiped clean of silicone grease, clean the inside of both filter holders by rubbing the surfaces with a nylon bristle brush and rinsing with solvent. Rinse each surface 3 times, or more if needed, to remove visible particulate. Make a final rinse of the brush and filter holder. After all solvent washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that solvent will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

(C) Container No-number 3. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the third impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling it. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for container ~~No-number~~ 3 in paragraph (iii)(C) of this subdivision. Impinger water. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid that is in the first 2 impingers to within ± 1 milliliter by using a graduated cylinder or by weighing it to within ± 1.0 g **gram** by using a balance if none is available. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required, (see subdivision (b)(i)(G) of this rule). If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically. Whenever possible, containers ~~shall~~ **must** be shipped in a manner that keeps them upright at all times.

(iii) Analysis. Record the data required on a sheet such as the one shown in figure 106 **under R 336.2021**. Handle each sample container as follows:

(A) Container Nos-numbers 1, 1A. Analyze and report each filter separately. Transfer the filter and any-loose particulate from the sample container to a tared-glass weighing dish. Dry the filter in an oven at 105 degrees Centigrade, (220 degrees Fahrenheit,) for a minimum of 2 hours, cool for at least 1 hour in a desiccator containing anhydrous calcium sulfate, and weigh and record its weight to the nearest 0.1 milligram. During the weighing, the filter ~~shall~~ **must** not be exposed to the laboratory atmosphere for a period of more than 2 minutes or a relative humidity above 50%. Procedures, other than those specified, that account for relative humidity effects may be used, subject to the approval of the department. The method used for the drying and weighing of filters ~~shall~~ **must** be consistent before and after the test.

(B) Container No-number 2. Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the department, to correct the final results. Measure the liquid in this container either volumetrically to ± 1 milliliter or gravimetrically to ± 1.0 gram. Transfer the contents to a tared 250- milliliter beaker and evaporate to dryness either at ambient temperature and pressure for acetone or at 95 degrees Centigrade, (203 degrees

Fahrenheit,) in an oven for distilled water. Then subject the sample to 250 degrees Centigrade, (482 degrees Fahrenheit,) in an oven for 2 to 3 hours. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 milligram.

(C) Container ~~No-number~~ 3. Weigh the spent silica gel, or silica gel plus impinger, to the nearest 0.5 gram using a balance. This step may be conducted in the field. "Solvent blank" container. Measure solvent in this container either volumetrically or gravimetrically. Transfer the contents to a tared 250- milliliter beaker and evaporate to dryness either at ambient temperature and pressure for acetone or at 95 degrees Centigrade, (203 degrees Fahrenheit), in an oven for distilled water. Then subject the sample to 250 degrees Centigrade, (482 degrees Fahrenheit), in an oven for 2 to 3 hours. Desiccate for 24-hours and weigh to a constant weight. Report the results to the nearest 0.1 milligram. If acetone is used, the contents of container ~~No-number~~ 2, as well as the acetone blank container, may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature ~~shall~~ **must** be closely supervised, and the contents of the beaker ~~shall~~ **must** be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

(e) Calibration. Maintain a laboratory log of all calibrations. Calibrations ~~shall~~ **must** comply with all of the following provisions:

(i) Probe nozzle. ~~A probe nozzle Probe nozzles shall~~ **must** be calibrated before ~~their~~ its initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 millimeter, (0.001 in-~~ch~~). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers ~~shall~~ **must** not exceed 0.1 millimeter, (0.004 in-~~ch~~). When nozzles become nicked, dented, or corroded, ~~they shall~~ **the nozzles must** be reshaped, sharpened, and recalibrated before use. Each nozzle ~~shall~~ **must** be permanently and uniquely identified.

(ii) Pitot tube. The type S pitot tube assembly ~~shall~~ **must** be calibrated according to the procedure outlined in ~~section 4 of~~ method 2.

(iii) Metering system. Before its initial use in the field, the metering system ~~shall~~ **must** be calibrated according to the procedure outlined in APTD -0576, **adopted by reference in R 336.1902**. Instead of physically adjusting the dry gas meter dial readings to correspond to the wet test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values. Before calibrating the metering system, it is suggested that a leak check be conducted. For metering systems having diaphragm or rotary pumps, the normal leak check procedure will not detect leakages within the pump. For these cases, the following leak check procedure is suggested: Make a 10-minute calibration run at 0.00057 **cubic meters per minute, m^3/min (0.02 cubic feet per minute. ~~cfm~~)**; ~~at~~ **At** the end of the run, take the difference of the measured wet test meter and dry gas meter volumes, **and**; divide the difference by 10 to get the leak rate. The leak rate ~~shall~~ **must** not exceed 0.00057 **cubic meters per minute, m^3/min (0.02 cubic feet per minute ~~cfm~~)**. After each field use, the calibration of the metering system ~~shall~~ **must** be checked by performing 3 calibration runs at a single, intermediate orifice setting, based on the previous field test, with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate

the average value of the calibration factor. If the calibration has changed by more than 5%, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576. Alternatively, a spirometer may be substituted for a wettest meter in the above mentioned calibration procedures. Alternative procedures, such as using the orifice meter coefficients, may be used, subject to the approval of the department. If the dry gas meter coefficient values obtained before and after a test series differ by more than 5%, the test series ~~shall~~ **must** be performed using whichever meter coefficient value, (before or after,) gives the lower value of total sample volume.

(iv) Probe heater calibration. The probe heating system ~~shall~~ **must** be calibrated before its initial use in the field according to the procedures outlined in APTD-0576, **adopted by reference in R 336.1902**. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

(v) Temperature gauges. Use the procedure in ~~section 4.3 of~~ method 2 to calibrate in stack temperature gauges. Dial thermometers, such as those used for the dry gas meter and condenser outlet, ~~shall~~ **must** be calibrated against mercury in glass thermometers.

(vi) Leak check of metering system shown in figure 103 **under R 336.2021**. That portion of the sampling train from the pump to the orifice meter ~~shall~~ **must** be leak checked before initial use and after each shipment. Leakage after the pump ~~will result~~ **results** in less volume being recorded than is actually sampled. The following procedure is suggested, **also** (see figure 107 **under R 336.2021**): Close the main valve on the meter box. Insert a 1-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 centimeters, (5 to 7 in-ches,) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 minute. A loss of pressure on the manometer indicates a leak in the meter box. ~~Leak~~ **leaks**, if present, ~~shall~~ **must** be corrected.

(vii) Barometer. Calibrate against a mercury barometer.

(f) Calculations. When carrying out calculations, retain at least 1 extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used if ~~they~~ **the other forms of the equations** give equivalent results. All of the following provisions **under R 336.2011 (f)** apply to calculations: ~~for this rule.~~

—(i) Nomenclature:

~~A_n = Cross-sectional area of nozzle, $m^2(ft.^2)$.~~

~~A = Cross-sectional area of stack or flue at the point of sampling, ft^2 .~~

~~B_{ws} = Water vapor in the gas stream, proportion by volume, expressed as a fraction.~~

~~B_{wi} = Percent water vapor in gas entering source particulate control device determined by method 4.~~

~~B_{wo} = Percent water vapor in gas exiting source particulate control device.~~

~~C_a = Wash blank residue concentration, mg/g .~~

~~C_s = Concentration of particulate matter in stack gas, pounds per 1,000 pounds of actual stack gas.~~

~~C_{sD} = Concentration of particulate matter in stack gas, moisture excluded, pounds per 1000 pounds of dry stack gas.~~

~~C_{s50} = Concentration of particulate matter corrected to 50% excess air, pounds per 1000 pounds of stack gas.~~

C_{s50D} — Concentration of particulate matter corrected to 50% excess air, excluding any water addition from a collector, pounds per 1000 pounds of stack gas.

E — Mass emission rate of particulate, lb/hr.

F_{50} — Concentration conversion factor to 50% excess air with no moisture alterations in exhaust.

F_{50D} — Concentration conversion factor to 50% excess air, excluding any moisture added to exhaust gas by pollution collection system.

F_D — Concentration conversion factor to dry basis, excluding any water in the stack gas.

I — Percent of isokinetic sampling.

L_a — Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m³/min (0.02 cfm) or 4% of the average sampling rate, whichever is less.

L_i — Individual leakage rate observed during the leak check conducted before the "ith" component change ($i = 1, 2, 3 \dots n$), m³/min (cfm).

L_p — Leakage rate observed during the post test leak check, m³/min (cfm).

M_d — Molecular weight of dry stack gas, g/g mole (lb/lb-mole), calculated by method 3, equation 3-21, using data from integrated method 3.

m_h — Total amount of particulate matter collected, mg.

M_w — Molecular weight of water, 18.0 g/g mole (18.0 lb/lb-mole).

m_a — Mass of residue of solvent after evaporation, mg.

m_g — Total weight of gas samples through nozzle, lb.

P_{bar} — Barometric pressure at the sampling site, mm Hg (in. Hg).

P_s — Absolute stack gas pressure.

P_{std} — Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

R — Ideal gas constant, 0.06236 mm Hg m³/°K g mole (21.85 in.Hg-ft.³/°R²lb-mole).

T_m — Absolute average dry gas meter temperature (see figure 104), °K (°R).

T_s — Absolute average stack gas temperature (see figure 104), °K (°R).

T_{std} — Standard absolute temperature, 294.1°K (530°R).

V_a — Volume of solvent blank, ml.

V_{aw} — Volume of solvent used in wash, ml.

V_{le} — Total volume of liquid collected in impingers and silica gel (see figure 106), ml.

V_m — Volume of gas sample as measured by the dry gas meter, dem (dcf).

$V_{m(std)}$ — Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dsem (dsef).

$V_{w(std)}$ — Volume of water vapor in the gas sample, corrected to standard conditions, sem (sef).

V_s — Stack gas velocity, calculated by method 2, equation 2-9, using data obtained from method 5, m/sec (ft./sec).

W_a — Weight of residue in solvent wash, mg.

Y — Dry gas meter calibration factor.

ΔH — Average pressure differential across the orifice meter (see figure 104), mm H₂O (in. H₂O).

$\%O_2$ — Percent oxygen in stack gas by volume (dry basis).

- %N₂ = Percent nitrogen in stack gas by volume (dry basis).
- p_a = Density of solvent, mg/ml.
- p_{s(std)} = Density of all sampled gas at standard conditions, lb/ft.³
- p_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).
- θ = Total sample time, min.
- θ₁ = Sample time, interval, from the beginning of a run until the first component change, min.
- θ_i = Sampling time interval, between 2 successive component changes, beginning with the interval between the first and second changes, min.
- θ_p = Sampling time interval, from the final (nth) component change until the end of the sampling run, min.
- 13.6 = Specific gravity of mercury.
- 60 = Sec/min.
- 100 = Conversion to percent.
- 386.9 = Cubic feet per lb-mole of ideal gas at standard conditions.
- 453.6 = Conversion of pounds to grams.
- 3600 = Conversion of hours to sec.
- 1000 = Conversion of 1000 lb units to lb units.

—(ii) Average the dry gas meter temperature and average the orifice pressure drop. See data sheet (figure 104).

—(iii) Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (21.1 degrees Centigrade, 760 mm Hg or 70 degrees Fahrenheit, 29.92 in. Hg) by using equation 5-1.

equation 5-1

$$V_{m(std)} = \frac{V_m Y T_{std} (P_{bar} + \Delta H/13.6)}{T_m P_{std}} = K_1 \frac{V_m Y (P_{bar} + \Delta H/13.6)}{T_m}$$

Where:

—K₁ = 0.3869 °K/mm Hg for metric units.

— = 17.71 °R/in. Hg for English units.

Equation 5-1 can be used as written. However, if the leakage rate observed during any of the mandatory leak checks (for example, the post-test leak check or leak checks conducted prior to component changes) exceeds L_a, equation 5-1 shall be modified as follows:

—(A) Case I. No component changes made during sampling run. In this case, replace V_m in equation 5-1 with the expression:

$$V_m - (L_p - L_a) \theta$$

—(B) Case II. One or more component changes made during the sampling run. In this case, replace V_m in equation 5-1 by the expression:

$$V_m - (L_i - L_a)\theta_i - \sum_{i=2}^n (L_i - L_a)\theta_i - (L_p - L_a)\theta_p$$

and substitute only for those leakage rates (L_i or L_p) that exceed L_a .
 —(iv) Volume of water vapor.

equation 5-2

$$V_{w(std)} = V_{Tc} (p_w / M_w) (R T_{std} / P_{std}) = K_2 V_{Tc}$$

Where:

$K_2 = 0.001338 \text{ m}^3/\text{ml}$ for metric units.
 $= 0.04733 \text{ ft.}^3/\text{ml}$ for English units.

—(v) Moisture content.

equation 5-3

$$B_{ws} = V_{w(std)} / (V_{m(std)} + v_{w(std)})$$

In saturated or water droplet laden gas streams, 2 calculations of the moisture content of the stack gas shall be made: 1 from the impinger analysis (equation 5-3), and a second from the assumption of saturated conditions. The lower of the 2 values of B_{ws} shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the note of section 1.2 of method 4. For the purpose of this method, the average stack gas temperature from figure 104 **under R 336.2021** may be used to make the determination, if the accuracy of the in-stack temperature sensor is ± 1 degree Centigrade (2 degrees Fahrenheit).

—(vi) Solvent blank concentration.

equation 5-4

$$C_a = m_a / (V_a P_a)$$

—(vii) Solvent wash blank.

equation 5-5

$$W_a = C_a V_{aw} P_a$$

—(viii) Total particulate weight. Determine the total particulate catch from the sum of the weights obtained from containers 1, 1A, and 2 less the wash solvent blank (see figure 106).

Refer to subdivision (d)(i)(E) of this rule to assist in the calculation of results involving 2 or more pairs of filters or 2 or more sampling trains.

—(ix) Sampled gas density. Determine the density of the gas sampled from the stack, at standard conditions ($\text{lb}/\text{ft.}^3$).

equation 5-6

$$P_{s(std)} = (M_d(1 - B_{ws}) + M_w B_{ws}) / 386.9$$

—(x) Total weight of gas sampled (lbs).

equation 5-7

$$m_g = (V_{m(std)} + V_{w(std)}) P_{s(std)}$$

—(xi) Particulate concentration (lbs/1000 lbs).

equation 5-8

$$C_s = m_p / (453.6 m_g)$$

—(xii) Excess air and moisture correction factors:

—(A) Correction factor to 50% excess air for those sources with or without any particulate collector where no increase in moisture content of the exhaust gas occurs after the process and before the point of sampling.

equation 5-9

~~$$F_{50} = \frac{M_d + 18 B_{wo} / (100 - B_{wo})}{0.1826 \% N_2 - 2.0592 \% O_2 + M_d + 18 B_{wo} / (100 - B_{wo})}$$~~

—(B) Correction factor to 50% excess air for those sources with a wet collection device (scrubber) that increases the moisture content of the exhaust gas after the process and before the point of sampling.

equation 5-10

~~$$F_{50D} = \frac{M_d + 18 B_{wo} / (100 - B_{wo})}{0.1826 \% N_2 - 2.0592 \% O_2 + M_d + 18 B_{wi} / (100 - B_{wi})}$$~~

—(C) Correction factor to convert the actual concentration, C_s , to dry conditions.

equation 5-11

~~$$F_D = \frac{M_d + 18 B_{wo} / (100 - B_{wo})}{M_d}$$~~

—(xiii) Converted particulate concentrations, where applicable under the department's rules or permit.

equation 5-12

$$C_{s50} = C_s F_{50}$$

equation 5-13

$$C_{s50D} = C_s F_{50D}$$

equation 5-14

$$C_{sD} = C_s F_D$$

—(xiv) Mass emission rate (lb/hr).

equation 5-15

$$E = \frac{3600 A V_s C_s P_s T_{std} P_{s(std)}}{1000 T_s P_{std}} = K_3 A V_s C_s P_s P_{s(std)} / T_s$$

Where:

K₃=63.77 for English units.

—(xv) Isokinetic variation:

—(A) Calculation from raw data.

equation 5-16

$$I = \frac{100 T_s (K_4 V_{ic} + (V_m / T_m)(P_{bar} + \Delta H / 13.6))}{60 \theta V_s P_s A_n}$$

Where:

K₄=0.003458 mm Hg m³ ml⁻¹ °K for metric units.=0.002672 in. Hg ft.³/ml⁻¹ °R for English units.

—(B) Calculation from intermediate values.

equation 5-17

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} V_s \theta A_n P_s 60(I - B_{WC})} = K_5 \frac{T_s V_{m(std)}}{P_s V_s A_n \theta (I - B_{WC})}$$

Where:

K₅ = 4.307 for metric units.

= 0.09409 for English units.

—(xvi) Acceptable results. If 90% ≤ I ≤ 110%, the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or if I is less than

90%, the department may opt to accept the results. Otherwise, reject the results and repeat the test.

(g) Bibliography:

(i) Federal Register, Volume 42, No. 160, Part 60, Chapter 1, Title 40, Appendix A, Method 5. August 18, 1977.

(ii) Martin, Robert M. Construction Details of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C.APTD-0581. April, 1971.

(iii) Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0576. March, 1972.

(iv) Shigehara, R. T. "Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights." Stack Sampling News 2:4-11. October, 1974.

(v) Guidelines for Source Testing of Particulate. Michigan Department of Natural Resources, Air Quality Division. June 1, 1977.

R 336.2014 Reference test method 5E.

Rule 1014. Reference method 5E, determination of particulate matter emissions from positive pressure fabric filters, reads as follows:

(a) The principle, applicability, and performance test criteria are as follows:

(i) Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature at or above the exhaust gas temperature up to a nominal 248 ± 25 degrees Fahrenheit. The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after the removal of uncombined water.

(ii) Applicability. This method is applicable for the determination of particulate emissions from the stationary sources as identified in table 31 of R 336.1331. The method is also applicable when specifically provided for in the department's rules, orders, a permit to install, or a permit to operate.

(iii) Performance test criteria **as follows**:

(A) A performance test ~~must~~**shall consist meet the requirements under R 336.2003(2)** of a minimum of 3 separate samples of a specific air contaminant conducted within a 36-hour period, unless otherwise authorized by the department. Each of the 3 separate samples shall be obtained while the source is operating at a similar production level. For the purpose of determining compliance with an applicable emission limit, rule, or permit condition, the arithmetic mean of results of the 3 samples shall apply. If a sample is accidentally lost or conditions occur in which 1 of the 3 samples must be discontinued because of forced shutdown, failure of an irreplaceable portion of the sampling train, extreme meteorological conditions, or other circumstances beyond the owner's or operator's control, compliance may, upon the approval of the department, be determined using the arithmetic mean of the results of 2 samples.

(B) For ~~any~~ sources that ~~are~~ is subject to an emission limitation calculated to 50% excess air, the multipoint, integrated sampling procedure of R 336.2004(1)(c) ~~shall~~ **must** be used for gas analysis. For all other sources that require a determination of the molecular weight of the exhaust, ~~any~~ **an** optional sampling procedure of R

336.2004(1)(c) may be used. Alternatives or modifications to procedures are subject to the approval of the department.

(C) The minimum volume per sample ~~shall~~ **must** be 30 cubic feet actual gas. Minimum sample time ~~shall~~ **must** be 60 minutes, which may be continuous or a combination of shorter sampling periods for sources that operate in a cyclic manner. Smaller sampling times or sample volumes, when necessitated by process variables or other factors, may be approved by the department.

(D) For ~~any~~ a source whose emission control device alters the moisture content of the exhaust gas, a moisture determination ~~shall~~ **must** be performed in a location upstream from the emission control device and in accordance with R 336.2004(1)(d) or an alternative method approved by the department.

(b) The following provisions apply to apparatus:

(i) Sampling train. A schematic of the sampling train used in this method is shown in figure 103 **under R 336.2021**. Construction details for many, but not all, of the train components are given in APTD-0581, (subdivision (g)(ii) of this rule). For changes from the APTD-0581 document and for allowable modifications to figure 103 **under R 336.2021**, consult with the department. The operating and maintenance procedures for many, but not all, of the sampling train are described in APTD-0576, **adopted by reference in R 336.1902 and as referenced under** (subdivision (g)(iii) of this rule). Since correct usage is important in obtaining valid results, all users shall read APTD-0576 and adopt the applicable operating and maintenance procedures outlined in it, unless otherwise specified in these rules. The sampling train consists of the following components:

(A) Probe nozzle. Stainless steel (316) or glass with sharp, tapered leading edge. The angle of taper ~~shall~~ **must** be less than 30 degrees and the taper ~~shall~~ **must** be on the outside to preserve a constant internal diameter. The probe nozzle ~~shall~~ **must** be of the button-hook design, unless otherwise specified by the department. If made of stainless steel, the nozzle ~~shall~~ **must** be constructed from seamless tubing. Other materials of construction may be used, subject to the approval of the department. A range of nozzle sizes suitable for isokinetic sampling ~~shall~~ **must** be available, for example, 0.32 to 1.27 centimeters, (1/8 to 1/2 in.-~~ch.~~) or larger if higher volume sampling trains are used inside diameter (~~HD~~) nozzles in increments of 0.16 centimeter, (1/16 in.-~~ch.~~). Each nozzle ~~shall~~ **must** be calibrated according to the procedures outlined in subdivision (e) of this rule.

(B) Probe liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a gas temperature at the exit end during sampling of 120 ± 14 degrees Centigrade, (248 ± 25 degrees Fahrenheit), another temperature as specified by the department's rules, or a temperature approved by the department for a particular application. The tester may opt to operate the equipment at a temperature lower than that specified. Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 ~~which~~ **that** utilize the calibration curves of APTD-0576, or calibrated according to the procedure outlined in APTD-0576, are acceptable. Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480 degrees Centigrade, (900 degrees Fahrenheit); ~~quartz~~ **Quartz** liners ~~shall~~ **must** be used for temperatures between 480 and 900 degrees Centigrade, (900 and 1,650 degrees Fahrenheit). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the

department. The softening temperature for borosilicate is 820 degrees Centigrade, (1,508 degrees Fahrenheit,) and for quartz it is 1,500 degrees Centigrade, (2,732 degrees Fahrenheit). When practical, every effort ~~shall~~ **must** be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners, such as 316 stainless steel, Incoloy 825, or other corrosion-resistant materials made of seamless tubing, may be used, subject to the approval of the department.

(C) Pitot tube. Type S, as described in ~~section 2.1 of method 2,~~ or other device approved by the department. The pitot tube ~~shall~~ **must** be attached to the probe, as shown in figure 103 **under R 336.2021**, to allow constant monitoring of the stack gas velocity. The impact, (high pressure,) opening plane of the pitot tube ~~shall~~ **must** be even with or above the nozzle entry plane, (see method 2, figure 2-6b **Velocity Traverse Data**) during sampling. The type S pitot tube assembly ~~shall~~ **must** have a known coefficient, determined as outlined in ~~section 4 of method 2.~~

(D) Differential pressure gauge. Incline manometer or equivalent devices, (**quantity** 2), as described in ~~section 2.2 of method 2.~~ One manometer ~~shall~~ **must** be used for velocity head (p) readings, and the other ~~shall~~ **must** be used for orifice differential pressure readings.

(E) Filter holders. Two separate filter holders in series or 1 filter holder with separate filter supports and seals for 2 filters. One filter holder with 2 filters held in contact with each other is not acceptable. Materials of construction may be stainless steel (316), glass, ~~teflon~~ **Teflon**, or ~~such~~ other material approved by the department.

(F) Filter heating system. Any heating system capable of maintaining a temperature around the filter holder during sampling of 120 ± 14 degrees Centigrade, (248 ± 25 degrees Fahrenheit), another temperature as specified by the department's rules or a permit condition, or a temperature approved by the department for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3 degrees Centigrade, (5.4 degrees Fahrenheit), ~~shall~~ **must** be installed so that the temperature around the filter holders can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

(G) Condenser. The following system ~~shall~~ **must** be used to determine the stack gas moisture content: Three impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. All impingers ~~shall~~ **must** be of the Greenburg-Smith design and ~~shall~~ **must** be modified by replacing the tip with a 1.3 centimeters, (1/2 in.-ch,) ~~ID~~ **inside diameter** glass tube extending to about 1.3 centimeters, (1/2 in.-ch,) from the bottom of the flask. Modifications, such as using flexible connections between the impingers or using materials other than glass, are ~~permitted~~ **allowed** subject to the approval of the department. The first impinger ~~shall~~ **must** contain a known quantity of water, (**as described in** subdivision (d)(i)(C) of this rule), the second ~~shall~~ **must** be empty, and the third ~~shall~~ **must** contain a known weight of silica gel or equivalent desiccant. Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 milliliter or 1 gram, may be used subject to the approval of the department. In any case, the means for measuring the moisture leaving the condenser ~~shall~~ **must** be by passing the sample gas stream through a tared silica gel, or equivalent desiccant, trap with exit gases kept below 20 degrees Centigrade, (68 degrees

Fahrenheit,) and determining the weight gain. If a determination of the particulate matter collected in the impingers is required by the department's rules, a permit to install, or a permit to operate, the impinger system described in this subdivision ~~shall~~ **must** be used, without modification. Contact the department as to the sample recovery and analysis of the impinger contents.

(H) Metering system. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3 degrees Centigrade, (~~5.4 degrees Fahrenheit~~), dry-gas meter capable of measuring volume to within 2%, and related equipment as shown in figure 103 **under R 336.2021**. Other metering systems capable of maintaining sampling rates within 10% of isokinetic and capable of determining sample volumes to within 2% may be used, subject to the approval of the department. When the metering system is used in conjunction with a pitot tube, the system ~~shall~~ **must** enable checks of isokinetic rates. Sampling trains utilizing metering systems designed for higher flow rates than those described in APTD-0581 or APTD-0576, **adopted by reference in R 336.1902**, may be used if the specifications of this rule are met.

(I) Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 millimeters Hgmercury, (~~0.1-inch- Hgmercury~~). In many cases, the barometric reading may be obtained from a nearby national weather service station. When obtained from this source, the station value, which is the absolute barometric pressure, ~~shall~~ **must** be requested and an adjustment for elevation differences between the weather station and sampling point ~~shall~~ **must** be applied at a rate of minus 2.5 millimeters Hgmercury, (~~0.1-in.-ch Hgmercury,~~) per 30 meters, (~~100 foot,~~) elevation increase or vice versa for elevation decrease.

(J) Gas density determination equipment. Temperature sensor and pressure gauge, as described in ~~sections 2.3 and 2.4 of~~ method 2, and gas analyzer, if necessary, as described in method 3. The temperature sensor ~~shall~~ **must**, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration ~~such so~~ that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch ~~any~~-metal. Alternatively, the sensor may be attached just before use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor ~~shall~~ **must** be placed in an interference-free arrangement with respect to the type S pitot tube openings, (~~see method 2, figure 2.76 Velocity Traverse Data~~). As a second alternative, if a difference of not more than 1% in the average velocity measurement is to be introduced, the temperature gauge need not be attached to the probe or pitot tube. This alternative is subject to the approval of the department.

(ii) Sample recovery. The following items are needed:

(A) Probe-liner and probe-nozzle brushes. Nylon bristle brushes with stainless steel wire handles. The probe brush ~~shall~~ **must** have extensions, at least as long as the probe, made of stainless steel, nylon, ~~teflon~~ **Teflon**, or similarly inert material. The brushes ~~shall~~ **must** be properly sized and shaped to brush out the probe liner and nozzle.

(B) Wash ~~bottles—2bottles - 2~~. Glass wash bottles are recommended.; ~~polyethylene~~ **Polyethylene** wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

(C) Glass sample storage containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 milliliters or 1000 milliliters. Screw cap liners ~~shall~~ **must** either be rubber-backed ~~teflon~~ **Teflon** or ~~shall~~ **must** be constructed so as to be leak-free and

resistant to chemical attack by acetone. Narrow-mouth glass bottles have been found to be less prone to leakage. Alternatively, polyethylene bottles may be used.

(D) Filter containers. Glass, polyethylene, or aluminum tube containers, unless otherwise specified by the department.

(E) Graduated cylinder or balance. To measure condensed water to within 1 milliliter or 1 gram. Graduated cylinders ~~shall~~ **must** have subdivisions of not more than 2 milliliters. Most laboratory balances are capable of weighing to the nearest 0.5 gram or less. Any of these balances are suitable for use here and in paragraph (iii)(D) of this subdivision.

(F) Plastic storage containers. Airtight containers to store silica gel.

(G) Funnel and rubber policeman, ~~To~~ to aid in the transfer of silica gel to container, **but** not necessary if silica gel is weighed in the field.

(H) Funnel. Glass or polyethylene, to aid in sample recovery.

(iii) Analysis. The following equipment is needed for analysis:

(A) Glass weighing dishes.

(B) Desiccator.

(C) Analytical balance. To measure to within 0.1 milligrams.

(D) Balance. To measure to within 0.5 milligrams.

(E) Beakers. 250 milliliters.

(F) Hygrometer, ~~To~~ to measure the relative humidity of the laboratory environment.

(G) Temperature gauge, ~~To~~ to measure the temperature of the laboratory environment.

(c) The following provisions apply to reagents:

(i) Sampling. The reagents used in sampling are as follows:

(A) Filters. Two outstack filters may be any combination of alundum ceramic thimble filters, type RA-98 or glass fiber filters, type A without organic binder. The size of such filters ~~shall~~ **must** allow proper sampling rates to maintain isokinetics using the nozzle sizes specified in subdivision (b)(i)(A) of this rule. Alternatively, other types of filters may be used, subject to the approval of the department.

(B) Silica gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175 degrees Centigrade, (350 degrees Fahrenheit,) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants, the equivalent or better of silica gel, may be used, subject to the approval of the department.

(C) Water. When analysis of the material caught in the impingers is required, distilled water ~~shall~~ **must** be used. Run blanks before field use to eliminate a high blank on test samples.

(D) Crushed ice.

(E) Stopcock grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with ~~teflon~~ **Teflon** sleeves, or equivalent, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the department.

(ii) Sample recovery. Washing solvent. Either acetone or distilled water may be used for sample recovery. If acetone is used for washing solvent, then reagent grade, less than 0.001% residue, in glass bottles is required. Acetone from metal containers generally has a high residue blank and ~~shall~~ **must** not be used. Suppliers sometimes transfer acetone to glass bottles from metal containers, ~~thus~~ **Thus**, acetone blanks ~~shall~~ **must** be run before field use, and only acetone with low blank values, less than 0.001%, ~~shall~~ **must** be used.

In no case ~~shall~~ **must** a blank value of more than 0.001% of the weight of acetone used be subtracted from the sample weight. If distilled water is used for washing solvent, use distilled water with less than 0.001% residue. Run blanks before field use to eliminate a high blank on test samples.

(iii) Analysis. Two reagents are required for the analysis:

(A) Solvent. Same as paragraph (ii) of this subdivision for quantitative transfer.

(B) Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the department.

(d) The following provisions apply to procedures:

(i) Determination of single measurement sites. The measurement site for a positive pressure fabric filter with an exhaust stack meeting method 1 criteria ~~shall~~ **must** be in accordance with ~~section 2.1 of~~ method 1. The measurement site for positive pressure fabric filters with short stacks or physical configuration not amenable to the requirements of method 1 ~~shall~~ **must** be determined from the following alternatives, or as approved by the department:

(A) Short stacks not meeting method 1 criteria: Short stacks may be extended in accordance with the procedures set forth in method 1 or by the use of flow straightening vanes. The flow straightening vanes ~~shall~~ **must** be of the egg crate design, (see figure 109 **under R 336.2021**). The measurement site, when using straightening vanes, ~~shall~~ **must** be at a distance not less than 2 times the average equivalent diameter of the vane opening and not less than ~~1/2~~ **half** of the overall stack diameter upstream of the stack outlet.

(B) Roof monitor or monovent exhaust outlets: For positive pressure fabric filters equipped with peaked roof monitors, ridge vents, or other types of monovalents, use a measurement site at the base of the monovent. Examples of ~~such~~ **the** locations are shown in figure 108 **under R 336.2021**. The measurement site ~~shall~~ **must** be upstream of any exhaust point.

(C) Measurement site in fabric filter compartment housing. Sample immediately downstream of the filter bags directly as shown in the examples in figure 108 **under R 336.2021**. Depending on the housing design, use sampling ports in the housing walls or locate the sampling equipment within the compartment housing.

(ii) Determination of number and location of traverse points. The number and location of traverse points for single exhaust stacks on positive pressure fabric filters meeting method 1 criteria ~~shall~~ **must** be in accordance with ~~section 2.3 of~~ method 1. The number of traverse points for other single measurement sites not meeting method 1 criteria ~~shall~~ **must** not be less than 24. For example, a rectangular measurement site, such as a monovent, would require the use of a balanced 5-by-5 traverse point matrix. All traverse points ~~shall~~ **must** be sampled for each test run.

(iii) Multiple measurement sites. Sampling from 2 or more stacks or measurement sites may be combined for a test run, if all of the following requirements are met:

(A) All measurement sites up to 12 ~~shall~~ **must** be sampled. For more than 12 measurement sites, conduct sampling on not less than 12 sites or 50% of the sites, whichever is greater. The measurement sites sampled ~~shall~~ **must** be evenly, or nearly evenly, distributed among the available sites, if not all of the sites are to be sampled.

(B) The same number of measurement sites ~~shall~~ **must** be sampled for each test run.

(C) The minimum number of traverse points per test run is 24. An exception to the 24-point minimum would be a test combining the sampling from 2 stacks meeting method 1 criteria for acceptable stack length, and method 1 specifies fewer than 12 points per site.

(D) As long as the 24 traverse points per test run criterion is met, the number of traverse points per measurement site may be reduced to 8. Alternatively, conduct a test run for each measurement site individually using the criteria in this paragraph and paragraph (ii) of this subdivision for **the** number of traverse points. Each test ~~shall~~ **must** count toward the total of 3 required for a performance test. If more than 3 measurement sites are sampled, the number of traverse points per measurement site may be reduced to 8 if not less than 72 traverse points are sampled for all 3 tests.

(iv) Sampling. The complexity of this method is such that, in order to obtain reliable results, testers ~~shall~~ **must** be trained and experienced with the test procedures. Sampling ~~shall~~ **must** comply with the following provisions:

(A) Pretest preparation. All the components ~~shall~~ **must** be maintained and calibrated according to the applicable procedures described in APTD-0576, **adopted by reference in R 336.1902**, unless otherwise specified in this rule. Weigh several 200 to 300 **gram** portions of silica gel in airtight containers to the nearest 0.5 **gram**. Record the total weight of the silica gel plus container on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just before train assembly. Check filters visually against light for irregularities, flaws, pinhole leaks, or cracks. Label filters of the proper size on the back side using numbering machine ink. As an alternative, label the shipping containers, (subdivision (b)(ii)(D) of this rule,) and keep the filters in these containers at all times, except during sampling and weighing. Dry the filters in an oven at 105 degrees Centigrade, (220 degrees Fahrenheit,) for a minimum of 2 hours, cool for at least 1 hour in a desiccator containing anhydrous calciumsulfate, and individually weigh and record ~~their weights~~ **each weight** to the nearest 0.1 **milligram**. During the weighing, the filters ~~shall~~ **must** not be exposed to the laboratory atmosphere for a period of more than 2 minutes and a relative humidity above 50%. Procedures, other than those specified, that account for relative humidity effects may be used, subject to the approval of the department.

(B) Preliminary determinations. Select the sampling site and the minimum number of sampling points according to method 1 or as specified by the department. Determine the stack pressure, temperature, and the range of velocity heads using method 2; ~~it~~ **It** is recommended that a leak check of the pitot lines, (see method 2, ~~section 3.1~~), be performed. Determine the moisture content using approximation method 4, or its alternatives, for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in method 2, ~~section 3.6~~; ~~if~~ **If** integrated method 3 sampling is used for molecular weight determination, the integrated bag sample ~~shall~~ **must** be taken simultaneously with, and for the same total length of time as, the particulate sample run. Select a nozzle size based on the range of velocity heads so that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered, (see ~~section 2.2 of method 2~~). Select a suitable probe liner and probe length so that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes. Select a total sampling time greater than or equal to the minimum

total sampling time specified in the test procedures for the specific industry so that the sampling time per point is not less than 5 minutes, unless approved by the department, or some greater time interval as specified by the department, and so that the sample volume taken, corrected to standard conditions, exceeds the required minimum total gas sample volume. The latter is based on an approximate average sampling rate. It is recommended that the number of minutes sampled at each point be an integer or an integer plus 1/2 minute to avoid timekeeping errors. In some circumstances, such as in batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the department's approval ~~shall~~ **must** first be obtained.

(C) Preparation of collection train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just before assembly or until sampling is about to begin. Place 100 **milliliters** of water in the first impinger, leave the second impinger empty, and transfer ~~approximately~~ 200 to 300 **grams** of preweighed silica gel from its container to the third impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 **gram** and recorded. Using tweezers or clean disposable surgical gloves, place a labeled, (~~identified,~~) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed. When glass liners are used, install the selected nozzle using a Viton A **O**-ring when stack temperatures are less than 260 degrees Centigrade, (~~500 degrees Fahrenheit,~~) and an asbestos string gasket when temperatures are higher. See APTD-0576, **adopted by reference in R 336.1902**, for details. Other connecting systems using either 310 stainless steel or ~~teflon~~ **Teflon** ferrules may be used. When metal liners are used, install the nozzle in the same manner as for glass liners or by a leak-free direct mechanical connection. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Set up the train as in figure 103 **under R 336.2021**. If necessary, use a very light coat of silicone grease on all ground glass joints. Grease only the outer portion, (~~see APTD-0576,~~) to avoid the possibility of contamination by the silicone grease. Place crushed ice around the impingers.

(D) Leak check procedures:

(I+) Pretest leak check. A pretest leak check is strongly recommended, but not required, to prevent invalid sampling and wasted time. If the tester opts to conduct the pretest leak check, the following procedure ~~shall~~ **must** be used: After the sampling train has been assembled, turn it on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A **O**-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak check the train at the sampling site by plugging the nozzle and pulling a 380 **millimeter Hgmercury**, (~~15 in.-ch Hgmercury,~~) vacuum. A lower vacuum may be used if it is not exceeded during the test. If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak check the train by first plugging the inlet to the filter holder, (~~cyclone, if applicable,~~) and pulling a 380 **millimeter Hgmercury**, (~~15~~

in-ch Hgmercury,) vacuum. A lower vacuum may be used if it is not exceeded during the test. Then connect the probe to the train and leak check at about a 25 millimeter Hgmercury, (1 in-ch Hgmercury,) vacuum.; alternatively Alternatively, the probe may be leak checked with the rest of the sampling train, in 1 step, at a 380 millimeter Hgmercury, (15 in-ch Hgmercury,) vacuum. Leakage rates in excess of 4% of the average sampling rate or 0.00057 cubic meters per minute, m^3/min (0.02 cubic feet per minute, cfm), whichever is less, are unacceptable. The following leak check instructions for the sampling train described in APTD-0576 and APTD-058 may be helpful. Start the pump with the bypass valve fully open and the coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve, as; this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak check at this higher vacuum or end the leak check and start over. When the leak check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone, if applicable, and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and prevents silica gel from being entrained backward into the third impinger.

(II2) Leak checks during sample run. If, during the sampling run, a component, such as a filter assembly or impinger, change becomes necessary, a leak check shall must be conducted immediately before the change is made. The leak check shall must be done according to the procedure outlined in paragraph (iv)(D)(I4) of this subdivision, except that it shall must be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be not more than 0.00057 cubic meters per minute, m^3/min (0.02 cubic feet per minute, cfm) or 4% of the average sampling rate, whichever is less, the results are acceptable and no correction need be applied to the total volume of dry gas metered.; if-If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and plan to correct the sample volume, as shown in subdivision (f)(iii) of R 336.2011this rule, or shall void the sampling run. Immediately after component changes, leak checks are optional.; if such If the leak checks are done, the procedure outlined in paragraph (iv)(D)(I4) of this subdivision shall must be used.

(III3) Post-test leak check. A leak check is mandatory at the conclusion of each sampling run. The leak check shall must be done in accordance with the procedures outlined in paragraph (iv)(D)(I4) of this subdivision, except that it shall must be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be not more than 0.00057 cubic meters per minute, m^3/min (0.02 cubic feet per minute, cfm) or 4% of the average sampling rate, whichever is less, the results are acceptable and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume, as shown in subdivision (f)(iii) of R 336.2011this rule, or shall void the sampling run.

(E) Particulate train operation. During the sampling run, maintain an isokinetic sampling rate that is within 10% of true isokinetic, unless otherwise specified by the department. For each run, record the data required on a data sheet such as the one shown in figure 104 under R 336.2021. Be sure to record the initial dry-gas meter reading. Record the dry-gas meter readings at the beginning and end of each sampling time

increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by figure 104 **under R 336.2021** at least once at each sample point during each time increment, and take additional readings when significant changes, 20% variation in velocity head readings, necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse. Clean the portholes before the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap and verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs that aid in the rapid adjustment of the isokinetic sampling rate without excessive computations are available. These nomographs are designed for use when the type S pitot tube coefficient is 0.85 ± 0.02 and the stack gas equivalent density, (dry molecular weight,) is equal to 29 ± 4 . APTD-0576, **adopted by reference in R 336.1902**, details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps, (see subdivision (g)(iv) of this rule,) are taken to compensate for the deviations. When the stack is under significant negative pressure, (height of impinger stem), take care to pull low flow when inserting the probe into the stack to prevent water from backing into the sample tubing and to avoid pulsation through the filter and possible loss of materials. When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream. Traverse the stack cross section, as required by method 1 or as specified by the department, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes.; ~~this~~ **This** minimizes the chance of extracting deposited material. During the test run, add more ice and, if necessary, salt to maintain a temperature of less than 20 degrees Centigrade, (68 degrees Fahrenheit,) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer. If the pressure drop across the filter becomes too high and makes isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak check, (see paragraph (iv)(D)(~~2II~~) of this subdivision). The total particulate weight ~~shall~~ **must** include the summation of all filter assembly catches. A single train ~~shall~~ **must** be used for the entire sample run, except in cases where simultaneous sampling is required in 2 or more separate ducts, at 2 or more different locations within the same duct, or where equipment failure necessitates a change of trains. In all other situations, the use of 2 or more trains ~~shall~~ **must** be subject to the approval of the department. Note that when 2 or more trains are used, separate analyses of the front-half catches from the individual trains may be combined, as may the impinger catches, and 1 analysis of the front-half catch and 1 analysis of impinger catch may be performed. Consult with the department for details concerning the calculation of results when 2 or more trains are used. At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry-gas meter reading, and conduct a post-test leak check, as outlined in paragraph (iv)(D)(~~III3~~) of this subdivision. Also, leak-check the pitot lines as described in method

2, section 3.1; ~~the~~ **The** lines ~~shall~~ **must** pass this leak check to validate the velocity head data.

(F) Calculation of percent isokinetic. Calculate percent isokinetic, (see subdivision (f) of this rule,) to determine whether the run was valid or whether another test run should be made. If there was difficulty in maintaining isokinetic rates due to source conditions, consult with the department for possible variance on the isokinetic rates.

(v) Sample recovery. Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this creates a vacuum in the filter holder and draws water from the impingers into the filter holder. Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Ground-glass stoppers, plastic caps, or serum caps may be used to close these openings. Transfer the probe and filter-impinger assembly to the cleanup area. This area ~~shall~~ **must** be clean and protected from the wind so that the chances of contaminating or losing the sample are minimized. Save a portion of the solvent used for cleanup as a blank. Take 200 **milliliters** of this solvent directly from the wash bottle being used and place it in a glass sample container labeled "solvent blank." Inspect the train before and during disassembly and note any abnormal conditions. Treat the samples as follows: Container ~~No. numbers~~ **1, 1A**. Carefully remove the filters from the filter holders and ~~place in their~~ **each filter in its** identified containers. Use a pair of tweezers or clean disposable surgical gloves, or both, to handle the filters. Carefully transfer to the container any particulate matter or filter fibers, or both, that adhere to the filter holder gasket by using a dry nylon bristle brush or sharp-edged blade, or both. Seal the container. Container ~~No. number~~ **2**. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, the tester shall quantitatively recover from particulate matter or ~~any~~ condensate from the nozzle, probe fitting, probe liner, and from both filter holders by washing these components with solvent and placing the wash in a glass container. Perform the solvent rinses as follows: Carefully remove the probe nozzle and clean the inside surface by rinsing with solvent from a wash bottle and brushing with a nylon bristle brush. Brush until the solvent rinse shows no visible particles and then make a final rinse of the inside surface with solvent. Brush and rinse the inside parts of the Swagelok fitting with solvent in a similar way until no visible particles remain. Rinse the probe liner with solvent by tilting and rotating the probe while squirting solvent into its upper end so that all inside surfaces are wetted with acetone. Let the solvent drain from the lower end into the sample container. A glass or polyethylene funnel may be used to aid in transferring liquid washes to the container. Follow the solvent rinse with a probe brush. Hold the probe in an inclined position and squirt solvent into the upper end as the probe brush is being pushed

with a twisting action through the probe. ~~hold~~ **Hold** a sample container underneath the lower end of the probe and catch any solvent and particulate matter that is brushed from the probe. Run the brush through the probe 3 or more times until no visible particulate matter is carried out with the solvent or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through, in the manner set forth in this paragraph, not less than 6 times, since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with solvent and quantitatively collect these washings in the sample container. After the brushing, make a final solvent rinse of the probe as described above. It is recommended that 2 people be used to clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination. After ensuring that all joints have been wiped clean of silicone grease, clean the inside of both filter holders by rubbing the surfaces with a nylon bristle brush and rinsing with solvent. Rinse each surface 3 times, or more if needed, to remove visible particulate. Make a final rinse of the brush and filter holder. After all solvent washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that solvent will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents. Container ~~No. number~~ **3**. Note the color of the indicating silica gel to determine if it has been completely spent and make a notation of its condition. Transfer the silica gel from the third impinger to its original container and seal. A funnel may make it easier to pour the silica gel without spilling it. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for container ~~No. number~~ **3** in paragraph (vi) of this subdivision. Impinger water. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid that is in the first 2 impingers to within ± 1 milliliter by using a graduated cylinder or by weighing it to within ± 1.0 gram by using a balance if none is available. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required, (see subdivision (b)(i)(G) of this rule). If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically. When possible, containers ~~shall~~ **must** be shipped in a manner that keeps them upright at all times.

(vi) Analysis. Record the data required on a sheet such as the one shown in figure 106 ~~under R 336.2021~~. Handle each sample container as follows: Container ~~Nos. numbers~~ **1, 1A**. Analyze and report each filter separately. Transfer the filter and any loose particulate from the sample container to a tared-glass weighing dish. Dry the filter in an oven at 105 degrees Centigrade, (220 degrees Fahrenheit,) for a minimum of 2 hours, cool for at least 1 hour in a desiccator containing anhydrous calcium sulfate, and weigh and record its weight to the nearest 0.1 milligram. During the weighing, the filter ~~shall~~ **must** not be exposed to the laboratory atmosphere for a period of more than 2 minutes or a relative humidity above 50%. Procedures, other than those specified, that account for relative humidity effects may be used, subject to the approval of the department. The method

used for the drying and weighing of filters ~~shall~~ **must** be consistent before and after the test. Container ~~No. number~~ 2. Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the department, to correct the final results. Measure the liquid in this container either volumetrically to ± 1 milliliters or gravimetrically to ± 1.0 grams. Transfer the contents to a tared 250- milliliter beaker and evaporate to dryness either at ambient temperature and pressure for acetone or at 95 degrees Centigrade, (203 degrees Fahrenheit,) in an oven for distilled water. Then subject the sample to 250 degrees Centigrade, (482 degrees Fahrenheit,) in an oven for 2 to 3 hours. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 milligram. Container ~~No. number~~ 3. Weigh the spent silica gel, or silica gel plus impinger, to the nearest 0.5 gram using a balance. This step may be conducted in the field. "Solvent blank" container. Measure solvent in this container either volumetrically or gravimetrically. Transfer the contents to a tared 250- milliliter beaker and evaporate to dryness either at ambient temperature and pressure for acetone or at 95 degrees Centigrade, (203 degrees Fahrenheit,) in an oven for distilled water. Then subject the sample to 250 degrees Centigrade, (482 degrees Fahrenheit,) in an oven for 2 to 3 hours. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 milligram. If acetone is used, the contents of container ~~No. number~~ 2, as well as the acetone blank container, may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature ~~shall~~ **must** be closely supervised, and the contents of the beaker ~~shall~~ **must** be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

(e) Calibration. Maintain a laboratory log of all calibrations. The following provisions apply to calibrations:

(i) Probe nozzle. ~~Probe nozzles~~ **A probe nozzle shall must** be calibrated before ~~their~~ its initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 millimeter, (0.001 in.-ch). Make 3 separate measurements using different diameters each time and obtain the average of the measurements. The difference between the high and low numbers ~~shall~~ **must** not exceed 0.1 millimeter, (0.004 in.-ch). When nozzles become nicked, dented, or corroded, ~~they shall~~ **the nozzles must** be reshaped, sharpened, and recalibrated before use. Each nozzle ~~shall~~ **must** be permanently and uniquely identified.

(ii) Pitot tube. The type S pitot tube assembly ~~shall~~ **must** be calibrated according to the procedure outlined in ~~section 4 of~~ method 2.

(iii) Metering system. Before its initial use in the field, the metering system ~~shall~~ **must** be calibrated according to the procedure outlined in APTD-0576, **adopted by reference in R 336.1902**. Instead of physically adjusting the dry-gas meter dial readings to correspond to the wet-test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values. Before calibrating the metering system, it is suggested that a leak check be conducted. For metering systems having diaphragm or rotary pumps, the normal leak check procedure will not detect leakages within the pump. For these cases, the following leak check procedure is suggested: Make a 10-minute calibration run at 0.00057 **cubic meters per minute**, m^3/min (0.02 **cubic feet per minute**, cfm); at the end of the run, take the

difference of the measured wet-test meter and dry-gas meter volumes, **and**; divide the difference by 10 to get the leak rate. The leak rate **shall must** not exceed 0.00057 **cubic meters per minute, m³/min** (0.02 **cubic feet per minute, cfm**). After each field use, the calibration of the metering system **shall must** be checked by performing 3 calibration runs at a single, intermediate orifice setting, based on the previous field test, with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet-test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5%, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0576. Alternatively, a spirometer may be substituted for a wet-test meter in the above mentioned calibration procedures. Alternative procedures, such as using the orifice meter coefficients, may be used, subject to the approval of the department. If the dry-gas meter coefficient values obtained before and after a test series differ by more than 5%, the test series **shall must** be performed using whichever meter coefficient value, before or after, gives the lower value of total sample volume.

(iv) Probe heater calibration. The probe heating system **shall must** be calibrated before its initial use in the field according to the procedures outlined in APTD-0576, **adopted by reference in R 336.1902**. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used.

(v) Temperature gauges. Use the procedure in ~~section 4.3 of method 2~~ to calibrate instack temperature gauges. Dial thermometers, such as those used for the dry-gas meter and condenser outlet, **shall must** be calibrated against mercury-in-glass thermometers.

(vi) Leak check of metering system shown in figure 103 **under R 336.2021**. That portion of the sampling train from the pump to the orifice meter **shall must** be leak checked before ~~to~~ initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested, **also** (see figure 107 **under R 336.2021**): Close the main valve on the meter box. Insert a 1-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 **centimeters, (5 to 7 in-ches,)** water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for 1 minute. A loss of pressure on the manometer indicates a leak in the meter box.; **Leaks**, if present, **shall must** be corrected.

(vii) Barometer. Calibrate against a mercury barometer.

(f) Calculations. When carrying out calculations, retain at least 1 extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used if ~~they~~ **the other forms of the equations** give equivalent results. **All of the** ~~The following~~ provisions **under R 336.2011(f)** apply to calculations: **for this rule.**

—(i) Nomenclature:

— A_n = ~~Cross-sectional area of nozzle, m²(ft.²).~~

— A = ~~Cross-sectional area of stack or flue at the point of sampling, ft².~~

— B_{ws} = ~~Water vapor in the gas stream, proportion by volume, expressed as a fraction.~~

~~B_{wi} = Percent water vapor in gas entering source particulate control device determined by method 4.~~

~~B_{wo} = Percent water vapor in gas exiting source particulate control device.~~

~~C_a = Wash blank residue concentration, mg/g.~~

~~C_s = Concentration of particulate matter in stack gas, pounds per 1,000 pounds of actual stack gas.~~

~~C_{sD} = Concentration of particulate matter in stack gas, moisture excluded, pounds per 1000 pounds of dry stack gas.~~

~~C_{s50} = Concentration of particulate matter corrected to 50% excess air, pounds per 1000 pounds of stack gas.~~

~~C_{s50D} = Concentration of particulate matter corrected to 50% excess air, excluding any water addition from a collector, pounds per 1000 pounds of stack gas.~~

~~E = Mass emission rate of particulate, lb/hr.~~

~~F_{50} = Concentration conversion factor to 50% excess air with no moisture alterations in exhaust.~~

~~F_{50D} = Concentration conversion factor to 50% excess air, excluding any moisture added to exhaust gas by pollution collection system.~~

~~F_D = Concentration conversion factor to dry basis, excluding any water in the stack gas.~~

~~I = Percent of isokinetic sampling.~~

~~L_a = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m³/min (0.02 cfm) or 4% of the average sampling rate, whichever is less.~~

~~L_i = Individual leakage rate observed during the leak check conducted before the "ith" component change ($i = 1, 2, 3 \dots n$), m³/min (cfm).~~

~~L_p = Leakage rate observed during the post-test leak check, m³/min (cfm).~~

~~M_d = Molecular weight of dry stack gas, g/g mole (lb/lb mole), calculated by method 3, equation 3-21, using data from integrated method 3.~~

~~m_h = Total amount of particulate matter collected, mg.~~

- ~~M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).~~
- ~~m_a = Mass of residue of solvent after evaporation, mg.~~
- ~~m_g = Total weight of gas samples through nozzle, lb.~~
- ~~P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).~~
- ~~P_s = Absolute stack gas pressure.~~
- ~~P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).~~
- ~~R = Ideal gas constant, 0.06236 mm Hg $m^3/^\circ K$ g-mole (21.85 in.Hg ft.³/ $^\circ R$?lb-mole).~~
- ~~T_m = Absolute average dry gas meter temperature (see figure 104), $^\circ K$ ($^\circ R$).~~
- ~~T_s = Absolute average stack gas temperature (see figure 104), $^\circ K$ ($^\circ R$).~~
- ~~T_{std} = Standard absolute temperature, 294.1 $^\circ K$ (530 $^\circ R$).~~
- ~~V_a = Volume of solvent blank, ml.~~
- ~~V_{aw} = Volume of solvent used in wash, ml.~~
- ~~V_{le} = Total volume of liquid collected in impingers and silica gel (see figure 106), ml.~~
- ~~V_m = Volume of gas sample as measured by the dry gas meter, dem (dcf).~~
- ~~$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dsem (dscf).~~
- ~~$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, sem (scf).~~
- ~~V_s = Stack gas velocity, calculated by method 2, equation 2-9, using data obtained from method 5, m/sec (ft./sec).~~
- ~~W_a = Weight of residue in solvent wash, mg.~~
- ~~Y = Dry gas meter calibration factor.~~
- ~~ΔH = Average pressure differential across the orifice meter (see figure 104), mm H₂O (in. H₂O).~~

~~-%O₂ = Percent oxygen in stack gas by volume (dry basis).~~

~~-%N₂ = Percent nitrogen in stack gas by volume (dry basis).~~

~~-p_a = Density of solvent, mg/ml.~~

~~-p_{s(std)} = Density of all sampled gas at standard conditions, lb/ft.³~~

~~-p_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).~~

~~-θ = Total sample time, min.~~

~~-θ₁ = Sample time, interval, from the beginning of a run until the first component change, min.~~

~~-θ_i = Sampling time interval, between 2 successive component changes, beginning with the interval between the first and second changes, min.~~

~~-θ_p = Sampling time interval, from the final (nth) component change until the end of the sampling run, min.~~

~~-13.6 = Specific gravity of mercury.~~

~~-60 = Sec/min.~~

~~-100 = Conversion to percent.~~

~~-386.9 = Cubic feet per lb mole of ideal gas at standard conditions.~~

~~-453.6 = Conversion of pounds to grams.~~

~~-3600 = Conversion of hours to sec.~~

~~-1000 = Conversion of 1000 lb units to lb units.~~

~~-(ii) Average the dry gas meter temperature and average the orifice pressure drop. See data sheet (figure 104).~~

~~-(iii) Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (21.1 degrees Centigrade, 760 mm Hg or 70 degrees Fahrenheit, 29.92 in. Hg) by using equation 5-1.~~

equation 5-1

$$\cancel{V_{m(std)} = \frac{V_m Y T_{std} (P_{bar} + \Delta H/13.6)}{T_m P_{std}}} = \cancel{K_1 V_m Y \frac{(P_{bar} + \Delta H/13.6)}{T_m}}$$

—Where:

$$\cancel{K_1} = 0.3869 \text{ }^\circ\text{K/mm Hg for metric units.}$$

$$= 17.71 \text{ }^\circ\text{R/in. Hg for English units.}$$

—Equation 5-1 can be used as written. However, if the leakage rate observed during any of the mandatory leak checks (for example, the post test leak check or leak checks conducted prior to component changes) exceeds L_a , equation 5-1 shall be modified as follows:

—(A) Case I. No component changes made during sampling run. In this case, replace V_m in equation 5-1 with the expression:

$$\cancel{V_m - (L_p - L_a) \theta}$$

—(B) Case II. One or more component changes made during the sampling run. In this case, replace V_m in equation 5-1 by the expression:

$$\cancel{V_m - (L_1 - L_a) \theta_1 - \sum_{i=2}^n (L_i - L_a) \theta_i - (L_p - L_a) \theta_p}$$

and substitute only for those leakage rates (L_i or L_p) that exceed L_a .

—(iv) Volume of water vapor.

equation 5-2

$$\cancel{V_{w(std)} = V_{lc} (p_w / M_w) (R T_{std} / P_{std}) = K_2 V_{lc}}$$

—Where:

$$\cancel{K_2} = 0.001338 \text{ m}^3/\text{ml for metric units.}$$

$$= 0.04733 \text{ ft.}^3/\text{ml for English units.}$$

—(v) Moisture content.

equation 5-3

$$\cancel{B_{ws} = V_{w(std)} / (V_{m(std)} + V_{w(std)})}$$

—In saturated or water droplet laden gas streams, 2 calculations of the moisture content of the stack gas shall be made: 1 from the impinger analysis (equation 5-3), and a second from the assumption of saturated conditions. The lower of the 2 values of B_{ws} shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in the note of section 1.2 of method 4. For the purpose of this method, the average stack gas

temperature from figure 104 may be used to make the determination, if the accuracy of the in-stack temperature sensor is ± 1 degree Centigrade (2 degrees Fahrenheit).

—(vi) Solvent blank concentration.

equation 5-4

$$C_a = m_a / (V_a P_a)$$

—(vii) Solvent wash blank.

equation 5-5

$$W_a = C_a V_{aw} P_a$$

—(viii) Total particulate weight. Determine the total particulate catch from the sum of the weights obtained from containers 1, 1A, and 2 less the wash solvent blank (see figure 106).

—Refer to subdivision (d)(i)(E) of this rule to assist in the calculation of results involving 2 or more pairs of filters or 2 or more sampling trains.

—(ix) Sampled gas density. Determine the density of the gas sampled from the stack, at standard conditions (lb./ft.³).

equation 5-6

$$P_{s(std)} = (M_d(1 - B_{ws}) + M_w B_{ws}) / 386.9$$

—(x) Total weight of gas sampled (lbs).

equation 5-7

$$m_g = (V_{m(std)} + V_{w(std)}) P_{s(std)}$$

—(xi) Particulate concentration (lbs/1000 lbs).

equation 5-8

$$C_s = m_n / (453.6 m_g)$$

—(xii) Excess air and moisture correction factors:

—(A) Correction factor to 50% excess air for those sources with or without any particulate collector where no increase in moisture content of the exhaust gas occurs after the process and before the point of sampling.

equation 5-9

$$F_{50} = \frac{M_d + 18 B_{w0} / (100 - B_{w0})}{0.1826 \% N_2 - 2.0592 \% O_2 + M_d + 18 B_{w0} / (100 - B_{w0})}$$

~~—(B) Correction factor to 50% excess air for those sources with a wet collection device (scrubber) that increases the moisture content of the exhaust gas after the process and before the point of sampling.~~

~~equation 5-10~~

~~$$F_{50D} = \frac{M_d + 18 B_{w0} / (100 - B_{w0})}{0.1826 \% N_2 + 2.0592 \% O_2 + M_d + 18 B_{w1} / (100 - B_{w1})}$$~~

~~—(C) Correction factor to convert the actual concentration, C_s , to dry conditions.~~

~~equation 5-11~~

~~$$F_D = \frac{M_d + 18 B_{w0} / (100 - B_{w0})}{M_d}$$~~

~~—(xiii) Converted particulate concentrations, where applicable under the department's rules or permit.~~

~~equation 5-12~~

~~$$C_{s50} = C_s F_{50}$$~~

~~equation 5-13~~

~~$$C_{s50D} = C_s F_{50D}$$~~

~~equation 5-14~~

~~$$C_{sD} = C_s F_D$$~~

~~—(xiv) Mass emission rate (lb/hr).~~

~~equation 5-15~~

~~$$E = \frac{3600 A V_s C_s P_s T_{std} P_{s(std)}}{1000 T_s P_{std}} = K_3 A V_s C_s P_s P_{s(std)} / T_s$$~~

~~—Where:~~

~~$$K_3 = 63.77 \text{ for English units.}$$~~

~~—(xv) Isokinetic variation:~~

~~—(A) Calculation from raw data.~~

~~equation 5-16~~

~~$$I = \frac{100 T_s (K_4 V_{is} + (V_m / T_m)(P_{bar} + \Delta H / 13.6))}{60 \theta V_s P_s A_n}$$~~

Where:

~~$K_4 = 0.003458 \text{ mm Hg} \cdot \text{m}^3 \cdot \text{ml}^{-1} \cdot \text{K}$ for metric units.~~

~~$= 0.002672 \text{ in. Hg} \cdot \text{ft.}^3 / \text{ml}^{-1} \cdot \text{R}$ for English units.~~

~~(B) Calculation from intermediate values.~~

equation 5-17

~~$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} V_s \theta A_n P_s 60(1 - B_{vs})} K_5 \frac{T_s V_{m(std)}}{P_s V_s A_n \theta(1 - B_{vs})}$$~~

Where:

~~$K_5 = 4.307$ for metric units.~~

~~$= 0.09409$ for English units.~~

~~(xvii) Acceptable results. If $90\% \leq I \leq 110\%$, the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or if I is less than 90% , the department may opt to accept the results. Otherwise, reject the results and repeat the test.~~

(g) Bibliography:

(i) Federal Register, Volume 42, No. 160, Part 60, Chapter 1, Title 40, Appendix A Method 5, August 18, 1977.

(ii) Martin, Robert M. Construction Details of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0581. April, 1971.

(iii) Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, N.C. APTD-0576. March, 1972.

(iv) Shigehara, R. T. "Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights." Stack Sampling News 2:4-11. October, 1974.

(v) Guidelines for Source Testing of Particulate. Michigan Department of Natural Resources, Air Quality Division. June 1, 1977.

R336.2033 Test methods for coke oven quench towers.

Rule 1033. (1) Test methods as applicable to coke oven quench towers. The publication entitled "Standard Methods for the Examination of Water and Wastewater," (4th 23rd edition), section 208C, shall **must** apply to the measurement of total dissolved solids in coke oven quench tower water.

(2) In addition to the provisions of in "Standard Methods for the Examination of Water and Wastewater" under subrule (1) section 208C, all of the following provisions

~~shall~~ **must** apply to the measurement of total dissolved solids in coke oven quench tower water:

- (a) The quench tower makeup water ~~shall~~ **must** be sampled at locations downstream of any makeup water additions.
- (b) The quench tower water ~~shall~~ **must** be sampled between the quench tower sump and the quench tower spray nozzles.
- (c) One sample of quench tower water for all operating quench towers ~~shall~~ **must** be collected once per day, 5 days per week.
- (d) Compliance with the applicable quench tower limits ~~shall~~ **must** be determined on a weekly basis.
- (e) For purposes of determining compliance, either individual analysis of the collected samples may be averaged or a weekly composite analysis may be performed.

R 336.2040 Method for determination of volatile organic compound emissions from coating lines and graphic arts lines.

Rule 1040. (1) The methods described in this rule ~~shall~~ **must** be used for the determination of volatile organic compound emissions from coating lines and graphic arts lines for the purpose of determining compliance, during the specified averaging period, with an emission limit. **For emission limits expressed as pounds of volatile organic compounds per gallon of coating, minus water, as applied. Concentrations of volatile organic compounds in coatings and inks must be determined by excluding water and compounds that are used as organic solvents and are excluded from the definition of volatile organic compound from both the volume of volatiles in the coatings and inks and the volume of the coatings and inks as applied. The emission limits can be contained in any of the following:**

- (a) These rules.
- (b) A permit to install.
- (c) A permit to operate.
- (d) A voluntary agreement.
- (e) A performance contract.
- (f) A stipulation.
- (g) An order of the department.

~~For emission limits expressed as pounds of volatile organic compounds per gallon of coating, minus water, as applied, the phrase "minus water" shall also include compounds which are used as organic solvents and which are excluded from the definition of volatile organic compound. Concentrations of volatile organic compounds in coatings and inks shall be determined by excluding water and compounds which are used as organic solvents and which are excluded from the definition of volatile organic compound from both the volume of volatiles in the coatings and inks and the volume of the coatings and inks as applied.~~

(2) Unless otherwise specified in these rules or in a legally enforceable permit, order, or contract as described in subrule (1) of this rule, for a particular coating line or graphic arts line, the applicable method for the determination of volatile organic emissions from coating lines and graphic arts lines is based ~~upon~~ **on** the form of the specified emission limit as follows:

(a) For coating lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating, minus water, as applied, use the method described in subrule (12)(a) of this rule.

(b) For coating lines that have 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating, minus water, as applied, use the method described in subrule (12)(b) of this rule.

(c) For coating lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating solids, as applied, use the method described in subrule (12)(c) of this rule.

(d) For coating lines with 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating solids, as applied, use the method described in subrule (12)(d) of this rule.

(e) For coating lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of applied coating solids, use the method described in subrule (12)(e) of this rule.

(f) For coating lines that have 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per gallon of applied coating solids, use the method described in subrule (12)(f) of this rule.

(g) For graphic arts lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per pound of solids, as applied, use the method described in subrule (12)(g) of this rule.

(h) For graphic arts lines that have 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per pound of solids, as applied, use the method described in subrule (12)(h) of this rule.

(i) For flatwood paneling coating lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per 1,000 square feet of coated finished product, use the method described in subrule (12)(i) of this rule.

(j) For flatwood paneling coating lines that have 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per 1,000 square feet of coated finished product, use the method described in subrule (12)(j) of this rule.

(3) The following nomenclature applies to the equations described in this rule:

(a) a = An individual duct through which gases containing volatile organic compounds are ducted to an add-on emissions control device.

(b) B = Modified emission limit, converted from pounds of volatile organic compounds per gallon of coating, minus water, as applied, to pounds of volatile organic compounds per gallon of coating solids as applied.

(c) C_{im} = Concentration of volatile organic compounds, as measured by the applicable method, in the effluent gas flowing through stack "m" leaving the add-on emissions control device, (parts per million by volume).

(d) C_{za} = Concentration of volatile organic compounds, as measured by the applicable method, in the influent gas flowing through duct "a" entering the add-on emissions control device, (parts per million by volume).

(e) D_{ci} = Density of each ink or coating "i" as received from the ink or coating supplier, (pounds per gallon of ink or coating).

(f) D_{sj} = Density of each volatile organic compound dilution solvent "j" added to the coating, (pounds per gallon of solvent, minus water).

(g) DE = Volatile organic compound destruction efficiency of the add-on emissions control device.

(h) E = Emission limit expressed in pounds of volatile organic compounds per gallon of coating, minus water, as applied.

(i) G_T = Total volume of all coatings "z" used during the averaging period, (gallons of coating, minus water, as applied).

(j) i = An individual coating used during the averaging period that belongs to the coating category for which a compliance determination is being made pursuant to the provisions of this rule. For a graphic arts line, "i" is an individual ink or coating used during the averaging period.

(k) j = An individual dilution solvent used during the averaging period that is used in conjunction with a coating "i".

(l) k = An individual method of application of coating solids.

(m) L_{ci} = Volume of each coating "i" used during the averaging period, (gallons of coating, minus water, as applied).

(n) L_{di} = Volume of each ink or coating "i" as received from the ink or coating supplier and used during the averaging period, (gallons of ink or coating).

(o) L_{ik} = Volume of each coating "i" used by each application method "k" during the averaging period, (gallons of coating, minus water, as applied).

(p) L_{sj} = Volume of each volatile organic compound dilution solvent "j" added to the coating during the averaging period, (gallons of solvent, minus water).

(q) M = Total weight of volatile organic compounds in all coatings "z" used during the averaging period for a coating line or a graphic arts line, (pounds).

(r) m = An individual stack through which gases containing volatile organic compounds are ducted to the ambient air from an add-on emissions control device.

(s) M_r = Total weight of volatile organic compounds recovered from a recovery-type add-on emissions control device during the averaging period, (pounds).

(t) N = Fraction, by weight, of the total volatile organic compounds emitted from an operation which is captured and enters the add-on emissions control device, (pound per pound).

(u) NT_r = Fraction, by weight, of the total volatile organic compounds in all coatings "z" used during the averaging period for a coating line or a graphic arts line which is controlled by an add-on emissions control device "r," (pound per pound).

(v) P = For an individual coating "i," pounds of volatile organic compounds per gallon of coating, minus water, as applied.

(w) P_s = For an individual coating "i," pounds of volatile organic compounds, minus water, as received from the ink or coating supplier.

(x) P_a = As used in subrule (12)(a) of this rule for coating lines that do not have an add-on emissions control device, volume-weighted average pounds of volatile organic compounds per gallon of coating, minus water, as applied, for a single coating category during the averaging period.

(y) P_b = As used in subrule (12)(b) of this rule for coating lines that have 1 or more add-on emissions control devices, volume-weighted average pounds of volatile organic

compounds per gallon of coating, minus water, as applied, for a single coating category during the averaging period.

(z) P_c = As used in subrule (12)(c) of this rule for coating lines that do not have an add-on emissions control device, volume-weighted average pounds of volatile organic compounds per gallon of coating solids, as applied, for a single coating category during the averaging period.

(aa) P_d = As used in subrule (12)(d) of this rule for coating lines that have 1 or more add-on emissions control devices, volume-weighted average pounds of volatile organic compounds per gallon of coating solids, as applied, for a single coating category during the averaging period.

(bb) P_e = As used in subrule (12)(e) of this rule for coating lines that do not have an add-on emissions control device, volume-weighted average pounds of volatile organic compounds per gallon of applied coating solids for a single coating category during the averaging period.

(cc) P_f = As used in subrule (12)(f) of this rule for coating lines that have 1 or more add-on emissions control devices, volume-weighted average pounds of volatile organic compounds per gallon of applied coating solids for a single coating category during the averaging period.

(dd) P_g = As used in subrule (12)(g) of this rule for graphic arts lines that do not have an add-on emissions control device, average pounds of volatile organic compounds per pound of solids, as applied, for all inks and coatings used during the averaging period.

(ee) P_h = As used in subrule (12)(h) of this rule for graphic arts lines that have 1 or more add-on emissions control devices, average pounds of volatile organic compounds per pound of solids, as applied, for all inks and coatings used during the averaging period.

(ff) P_i = As used in subrule (12)(i) of this rule for flatwood paneling coating lines that do not have an add-on emissions control devices, volume-weighted average pounds of volatile organic compounds per 1,000 square feet of coated finished product for a single-coating category during the averaging period.

(gg) P_j = As used in subrule (12)(j) of this rule for flatwood paneling coating lines that have 1 or more add-on emissions control devices, volume-weighted average pounds of volatile organic compounds per 1,000 square feet of coated finished product for a single-coating category during the averaging period.

(hh) Q_{im} = Volumetric flow rate of the effluent gas flowing through stack "m" leaving the add-on emissions control device, (dry standard cubic feet per hour).

(ii) Q_{za} = Volumetric flow rate of the influent gas flowing through duct "a" entering the add-on emissions control device, (dry standard cubic feet per hour).

(jj) Q_{3n} = Volumetric flow rate of the effluent gas leaving an uncontrolled stack "n," (dry standard cubic feet per hour).

(kk) r = An individual add-on emissions control device.

(ll) R_r = Reduction efficiency of a single add-on emissions control device.

(mm) R_t = Overall reduction efficiency of all add-on emissions control devices used for a coating line or a graphic arts line.

(nn) s = The total number of different add-on control devices "r" on a coating line or graphic arts line.

(oo) S_{sq} = The total surface area of coated finished product for a single-coating category for a flatwood paneling coating line during the averaging period, (square feet).

(pp) T = Overall transfer efficiency for all coatings "i" for a single-coating category on a coating line for the averaging period.

(qq) t = The total number of stacks "m" leaving an add-on emissions control device "r".

(rr) T_i = Transfer efficiency for application of coating "i," (%).

(ss) u = Total number of ducts "a" entering an add-on emissions control device "r".

(tt) U_{ci} = For representative colors and parts that are tested for transfer efficiency, " U_{ci} " is the volume of each representative color of coating that is applied to each representative part on a coating line during the averaging period.

(uu) V = For a coating line, the volume of solids in all coatings used "zc" during the averaging period, (gallons).

(vv) V_{ci} = Proportion of solids by volume in each coating "i," (gallon of solids per gallon of coating, minus water, as applied).

(ww) W = For a graphic arts line, the weight of solids in all inks and coatings used "zg" during the averaging period, (pounds).

(xx) W_{ci} = Proportion of volatiles, (volatile organic compounds, water, and exempt compounds), by weight in each ink or coating "i" as received from the ink or coating supplier, (pound of volatiles per pound of coating).

(yy) x = The total number of different application methods "k."

(zz) y = The total number of different dilution solvents "j."

(aaa) z = The total number of different coatings "i" used on a coating line or different number of inks and coatings "i" used on a graphic arts line during the averaging period. "z" is used generically in the equations specified in this rule for "zc" when the calculation is made for a coating line and for "zg" when the calculation is made for a graphic arts line.

(bbb) zc = The total number of different coatings "i" in the same coating category used during the averaging period.

(ccc) zg = The total number of different inks and coatings "i" used during the averaging period.

(4) The following provisions apply to the calculations for a coating line or graphic arts line made pursuant to the methods described in this rule:

(a) When carrying out calculations, carry not less than 5 significant digits in intermediate calculations. Round off figures after the final calculation, rounding off calculated emission numbers to not less than 2, but not more than 3, significant figures.

(b) The calculations for a coating line ~~shall~~ **must** include all of the coatings which are in the same coating category and which are used during the averaging period as specified in the applicable emission limit.

(c) Except as specified in R 336.1624(5)(d), the calculations for a graphic arts line ~~shall~~ **must** include all of the inks and coatings that are used during the averaging period as specified in the applicable emission limit.

(5) The volatile organic compound content of an ink or coating, minus water, as applied, "P," ~~shall~~ **must** be determined using any of the following methods:

(a) The volatile organic compound content of an ink or coating, minus water, as applied, "P," ~~shall~~ **must** be determined according to all of the following provisions:

(i) The volatile organic compound content, minus water, as applied, ~~shall~~ **must** be determined as follows:

(A) For a coating used on a coating line or a coating used on a graphic arts line, the volatile organic compound content, minus water, as applied, ~~shall~~ **must** be determined using federal reference method 24 or federal reference method 24Aa, as applicable to the coating, as described in R 336.2004, or an alternate method approved by the department.

(B) For an ink that is used on a graphic arts line, the volatile organic compound content, minus water, as applied, ~~shall~~ **must** be determined using **federal reference method 24 or federal reference method 24Aa, as applicable**, as described in R 336.2004.

(ii) The ink or coating sample ~~shall~~ **must** be taken at a point where the sample will be representative of the ink or coating material as applied.

(iii) The sample ~~shall~~ **must** be stored in an enclosed container that is not less than 1 pint in volume.

(iv) By using a procedure that is acceptable to the department, the amount of any compound in the sample that is excluded from the definition of volatile organic compound may be quantified and subtracted from the total amount of volatiles in the sample as determined by federal reference method 24, federal reference method 24Aa, or an alternate method that is approved by the department. In this case, the volume of any excluded compound in the sample ~~shall~~ **must** also be subtracted from the volume of the ink or coating sample.

(b) Upon written approval by the department, the volatile organic compound content of an ink or coating may be determined from formulation data, which includes batch composition information from the ink or coating manufacturer and the amount of volatile organic compound dilution solvent added to the ink or coating before application. In this case, "P" ~~shall~~ **must** be calculated using the following equation:

$$P = \frac{L_{di} P_s + L_{sj} D_{sj}}{L_{di} + L_{sj}}$$

(c) If a coating or ink is tested by a federal reference method 24 or 24Aa analysis or by an alternate method approved by the department and the results are different than calculated through formulation data review, then the test method results ~~shall~~ **must** be used for determining compliance with the emission limit.

(6) The weight of volatile organic compounds that are used during the averaging period ~~shall~~ **must** be calculated using the following equation, where "z" is the total number of coatings used:

$$M = \sum_{i=1}^z L_{ci} P$$

(7) The total volume of coating solids that are used during the averaging period ~~shall~~ **must** be calculated using the following equation, where "z" is the total number of coatings used:

$$V = \sum_{i=1}^z L_{ci} V_{ci}$$

(8) The total weight of ink and coating solids that are used during the averaging period ~~shall~~ **must** be calculated using the following equation, where "zg" is the total number of inks and coatings used during the averaging period:

$$W = \sum_{i=1}^{zg} L_{di} D_{ci} (1 - W_{ci})$$

(9) The transfer efficiency ~~shall~~ **must** be determined by the following method, if approved by the department, or by an alternate method approved by the department:

(a) A person ~~who~~ **that** is responsible for the transfer efficiency test shall identify all of the following in a coating operation transfer efficiency test proposal and ~~shall~~ submit the proposal to the department for approval not less than 30 days before the transfer efficiency test:

(i) All processing sequences. A processing sequence is the combination and order of paint booths, flash-off areas, ovens, and application equipment necessary to apply a coating.

(ii) The coating categories used on each processing sequence.

(iii) The representative coating color in each coating category. A representative color ~~shall~~ **must** be determined based ~~upon~~ **on** the volume of coating used in relation to the total volume of coating category coatings used and any other parameters acceptable to the department. If it is believed by the department that the transfer efficiency of the various coatings within the same coating category would be different as a result of different coating technologies, such as for metallic topcoat coatings and nonmetallic topcoat coatings, 2 or more representative coating colors may be required by the department to be tested.

(iv) At a minimum, 2 different representative parts coated in each processing sequence. A representative part ~~shall~~ **must** be determined based on the numbers of the part coated in relation to the total number of parts coated, the configuration of the part, and any other parameters acceptable to the department.

(b) For the initial transfer efficiency tests, a person shall test, at a minimum, the representative color or colors in each coating category used on each representative part in each processing sequence. To more closely represent actual process conditions, coatings applied wet-on-wet, such as basecoat and clearcoat, may be tested together. Also, identical colors or clear coats on identical parts in identical processing sequences need not be tested.

(c) A person ~~who~~ **that** is responsible for the transfer efficiency test shall review the operating conditions annually ~~thereafter~~ **after the initial transfer efficiency tests** and demonstrate, to the satisfaction of the department, that significant changes have not occurred in coating technology, the parts coated, or the processing sequence. The most recent test results ~~shall~~ remain valid for 5 years if the person demonstrates that significant change has not occurred. Significant product, processing, material, or application equipment changes ~~shall~~ necessitate retesting of the transfer efficiency of the operations that have been modified. The retesting ~~shall~~ **must** be done as soon as practicable, but not more than 180 days after the start-up and stabilization of the new product, process, material, or application equipment. New transfer efficiency values determined by the

retest ~~shall~~ **must** be used retroactively to the start-up of the new product, process, material, or application equipment.

(d) Retests on a coating line may be limited to a representative coating on not less than 2 representative parts in a representative processing sequence, as approved by the department. A representative processing sequence ~~shall~~ **must** be determined based ~~upon~~ **on** coating usage, application equipment, and any other parameters acceptable to the department.

(e) The area in the facility to be used for part weighing ~~shall~~ **must** be selected so as to provide for an area that has relatively constant temperature and minimal air movement.

(f) Except as allowed pursuant to the provisions of subdivision (b) of this subrule, the coating being tested ~~shall~~ **must** be the only coating applied during the transfer efficiency test. If the part is weighed, weight loss from all other materials ~~shall~~ **must** be accounted for in the initial test weight.

(g) A minimum of 10 parts per transfer efficiency test ~~shall~~ **must** be weighed to determine the weight of the solids applied. The average weight gain for the 10 parts tested ~~shall~~ **must** be used as the part weight gain for that transfer efficiency test. All transfer efficiency tests for a processing sequence ~~shall~~ **must** be completed within a 36-hour period.

(h) Each part to be painted ~~shall~~ **must** be identified and preweighed to the nearest 0.05 pound.

(i) The selected paint material at each paint system or paint pot dedicated for the transfer efficiency test ~~shall~~ **must** be isolated.

(j) The amount of material used during the transfer efficiency test ~~shall~~ **must** be determined by either of the following measurement procedures:

(i) The weight measurement procedure as follows:

(A) Weigh the tank of reduced paint, to the nearest 0.01 pound, after all supply and return lines have been filled.

(B) Connect the paint tank to the system and paint the test parts.

(C) Reweigh the tank to the nearest 0.01 pound.

(D) Determine the weight of paint material used by subtracting the final weight of the tank from the initial weight of the tank.

(E) Obtain paint samples for weight solids determination.

(F) Do both the initial and final weighings of the paint tank with the tank pressurized or with the tank not pressurized.

(ii) A volume measurement procedure that is acceptable to the department.

(k) Each painted test part ~~shall~~ **must** be reweighed to the nearest 0.01 pound after paint has cured and cooled.

(l) The weight of the solids in the paint samples ~~shall~~ **must** be determined using ASTM-D2369, **adopted by reference in R 336.1902.** ~~ASTM-D2369 is adopted in these rules by reference. A copy of this document may be inspected at the Lansing office of the air quality division of the department of environmental quality. A copy of this document may be obtained from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428, or from the Department of Environmental Quality, Air Quality Division, P.O. Box 30260, Lansing, Michigan 48909-7760, at a cost as of the time of adoption of these rules of \$25.00.~~

(m) The coating density in pounds per gallon ~~shall~~ **must** be determined using ASTM-D1475, **adopted by reference in R 336.1902.** ~~ASTM-D1475~~ is adopted in these rules by reference. A copy of this document may be inspected at the Lansing office of the air quality division of the department of environmental quality. A copy of this document may be obtained from the [American Society for Testing and Materials](#), 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428, or from the Department of Environmental Quality, Air Quality Division, P.O. Box 30260, Lansing, Michigan 48909-7760, at a cost as of the time of adoption of these rules of \$25.00.

(n) The following equation ~~shall~~ **must** be used to calculate the transfer efficiency for the application of coating "i":

$$T_i = \frac{\text{Total weight gain of parts tested}}{(\text{total weight of coating used per test}) \times (\text{weight fraction solids})}$$

$$T_i = \frac{\text{Total weight gain of parts tested}}{(\text{total weight of coating used per test}) \times (\text{weight fraction solids})}$$

(o) Where more than 1 part type or coating are tested on a coating line for a single coating category, the overall transfer efficiency "T" for the coating category ~~shall~~ **must** be determined by averaging the individual transfer efficiency values based ~~upon~~ **on** a volume-weighted average of coatings applied during the averaging period for each different color and part type tested. This overall transfer efficiency ~~shall~~ **must** be calculated by using the following equation, where "aa" is the number of coatings tested and "bb" is the number of part types tested:

$$T = \frac{\sum_{i=1}^{aa} \sum_{c=1}^{bb} T_{ci} U_{ci}}{\sum_{i=1}^{aa} \sum_{c=1}^{bb} U_{ci}}$$

(p) Baseline operating parameters of the paint application equipment and the paint booths ~~shall~~ **must** be established for each transfer efficiency test and ~~shall~~ **must** serve as a basis for determining compliance. These parameters ~~shall~~ **must** be included in a transfer efficiency test report and ~~shall~~ **must** include all of the following information:

- (i) Type of spray equipment.
- (ii) Electrostatic voltage.
- (iii) Size and geometry of the part coated.
- (iv) Gun-to-target distance, (nonmanual).
- (v) Number of parts per conveyor hook.
- (vi) Air velocity in spray booth, (linear feet per minute).
- (vii) Fluid flow settings, (by color).
- (viii) Bell revolutions per minute for minibells.
- (ix) Atomizing air pressure.

(10) The capture efficiency "N" ~~shall~~ **must** be determined as follows:

(a) The capture efficiency "N" ~~shall~~ **must** be determined by using the methods specified in 40 C.F.R.CFR §52.741(a)(4)(iii) entitled "Capture ~~system~~ **e**fficiency ~~t~~est

pprotocols" and in appendix B entitled "~~Volatile Organic Material~~ Measurement Techniques for Capture Efficiency," with the following modifications:

(i) The general modifications are as follows:

(A) Replace the requirements under 40 CFR 52.741(a)(4)(iii)(A)(2) with the following requirements: If a source owner or operator uses a control device designed to collect and recover ~~volatile organic compounds~~, for example, carbon adsorber, an explicit measurement of capture efficiency is not necessary if the conditions described in 40 ~~C.F.R.~~CFR §52.741(a)(4)(iii) are met. The overall emission reduction efficiency of the control system ~~shall~~ **must** be determined each day by directly comparing the input liquid ~~volatile organic compounds~~ to the recovered liquid ~~volatile organic compounds~~. The procedure for use in this situation is given in 40 ~~C.F.R.~~CFR §60.433, with the following modifications to 40 CFR 52.741(a)(4)(iii)(A)(2)(i) under paragraph **(B) of this rule.:**

(B) Replace the requirements under 40 CFR 52.741(a)(4)(iii)(A)(2)(i) with the following requirements: The source owner or operator shall obtain data each day for the solvent usage and solvent recovery and determine the solvent recovery efficiency of the system each day using a 7-day rolling period. The recovery efficiency for each day is computed as the ratio of the total recovered solvent for that day and the prior 6 consecutive operating days to the total solvent usage for the same 7-day period weighted average as given in 40 ~~C.F.R.~~CFR §60.433. This ratio ~~shall~~ **must** be expressed as a percentage. The ratio ~~shall~~ **must** be computed within 72 hours after each 24-hour period. With the approval of the administrator, a source that believes that the 7-day rolling period is not appropriate may use an alternative multi-day rolling period of not more than 30 days.

(C) Requirements in 40 CFR 52.741 Aappendix B**F**for procedures G.1, G.2, F.2, F.1, and L, ~~the following modifications:~~**sections titled "1.4 Sampling requirements" must be replaced with the following:** A capture efficiency test ~~shall~~ **must** consist of not less than 3 sampling runs. Each run ~~shall~~ **must** cover at least 1 complete production cycle, but ~~shall~~ **must** be not less than 3 hours long. The sampling time for each run need not be more than 8 hours, even if the production cycle has not been completed. Alternative sampling times may be used if approved by the administrator.

~~For procedure L, the following addition: 5.4 Audit procedure. Concurrently, analyze the audit sample and a set of compliance samples in the same manner to evaluate the technique of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for compliance samples and the EPA audit sample. If this condition is met, the auditing of subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set shall not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both enforcement agencies.~~

~~For procedures G.1, G.2, F.2, F.1, and L, the following additions:~~

~~5.5 (5.6 for procedure G.2) Audit samples. Audit sample availability. Audit samples will be supplied only to enforcement agencies for compliance tests. The availability of audit samples may be obtained by writing to the following address:~~

- ~~-Source Test Audit Coordinator (MD-77B)~~
- ~~-Quality Assurance Division~~
- ~~-Atmospheric Research and Exposure Assessment Laboratory~~
- ~~-U.S. Environmental Protection Agency~~
- ~~-Research Triangle Park, NC 27711~~

~~The availability of audit samples may also be obtained by calling the source test audit coordinator (STAC) at (919) 541-7834. The request for the audit sample shall be made not less than 30 days before the scheduled compliance sample analysis.~~

~~5.6 (5.7 for procedure G.2) Audit results. Calculate the audit sample concentration according to the calculation procedure described in the audit instructions included with the audit sample. Fill in the audit sample concentration and the analyst's name on the audit response form included with the audit instructions. Send 1 copy to the EPA regional office or the appropriate enforcement agency and a second copy to the STAC. The EPA regional office or the appropriate enforcement agency will report the results of the audit to the laboratory being audited. Include this response with the results of the compliance samples in relevant reports to the EPA regional office or the appropriate enforcement agency.~~

(ii) Owners or operators of coating lines that have multiple stacks may choose to apply **1 of the 4 protocols under 40 CFR 52.741(a)(4)(iii)(B) or the protocol described under subparagraph (A) of this paragraph**, following ~~modifications~~ in addition to the modifications listed in paragraph (i) of this subdivision **except for and to replace** the modification to 1.4 listed in paragraph (i)(C)(1) of this subdivision, **which is replaced with the following language as described under subparagraph (B) of this paragraph:**

~~(A)(a)(4)(iii)(B) The capture efficiency of a coating line shall be measured using 1 of the 5 protocols given below. Any error margin associated with a test protocol shall not be incorporated into the results of a capture efficiency test. If these techniques are not suitable for a particular process, then the source shall present an alternative capture efficiency protocol and obtain approval for it by the administrator as a sip or fip revision.~~

~~(5) Liquid/gas method measuring the captured emission, G_w , and liquid input, L . This procedure may only be used when the capture efficiency for a coating line is expected to be less than 50%. The capture efficiency equation to be used for this protocol is:~~

$$CE = G_w/L$$

Where:

CE = Coating line capture efficiency, decimal fraction.

G_w = Mass of vom captured and delivered to a control device.

L = Mass of liquid vom input to coating line.

Procedure G.1 contained in appendix B of this section is used to obtain G_w . Procedure L contained in appendix B or the alternate method in R 336.2007 ~~shall~~ **must** be used to determine L.

(B) Requirements in 40 CFR 52.741 ~~Appendix B~~ ~~For procedures G.1, G.2, F.2, F.1, and L, under the procedure section titled~~ the following modification: “1.4 Sampling requirements” **must be replaced with the following requirements**:- A capture efficiency test ~~shall~~ **must** consist of not less than 3 sampling runs. Each run ~~shall~~ **must** cover at least 1 complete production or processing cycle or ~~shall~~ **must** be at least 1 hour in duration. For automotive surface coating operations, the sampling time per test ~~shall~~ **must** be based on coating a minimum of 3 representative vehicles.

(b) The test protocols and **40 CFR 52.741(a)(4)(iii)** ~~appendix B of 40 C.F.R. §52.741(a)(4)(iii)~~ are adopted **by reference under R 336.1902**. ~~in these rules by reference. A copy of these regulations may be inspected at the Lansing office of the air quality division of the department of environmental quality. A copy of these regulations may be obtained from the Department of Environmental Quality, Air Quality Division, P.O. Box 30260, Lansing, Michigan 48909-7760, at no charge. A copy of 40 C.F.R. part 52 may be obtained from the Superintendent of Documents, Government Printing Office, P.O. Box 371954, Pittsburgh, Pennsylvania 15250-7954, at a cost as of the time of adoption of these rules of \$36.00, or on the United States government printing office internet web site at <http://www.access.gpo.gov>.~~

(c) Where multiple capture systems are used on a coating line or a graphic arts line, the appropriate capture efficiency for each application method ~~shall~~ **must** be determined and the overall capture efficiency for the coating line ~~shall~~ **must** be based ~~upon~~ **on** a mass-weighted average of all volatile organic compounds used on the coating line or the graphic arts line during the averaging period.

(11) The overall reduction efficiency of add-on emissions control devices ~~shall~~ **must** be determined using 1 or more of the following methods, as applicable:

(a) When a destructive-type add-on emissions control device is used, the reduction efficiency for the add-on emissions control device ~~shall~~ **must** be determined by using the following method:

(i) The destruction efficiency of the add-on emissions control device is calculated by using the following equation, where "u" is the total number of ducts entering the control device and "t" is the total number of stacks leaving the control device:

$$DE = \frac{\sum_{a=1}^u Q_{za} C_{za} - \sum_{m=1}^t Q_{im} C_{im}}{\sum_{a=1}^u Q_{za} C_{za}}$$

(ii) Using the destruction efficiency as determined in paragraph (i) of this subdivision, the reduction efficiency of the add-on emissions control device is calculated by using the following equation:

$$R_r = (DE) N$$

(iii) If there is only 1 add-on emissions control device used on a coating line or graphic arts line, use the value calculated for "R_r" pursuant to paragraph (ii) of this subdivision as the value for R_T in subsequent calculations.

(iv) The concentration of volatile organic compound emissions entering and exiting the add-on emissions control device ~~shall~~ **must** be determined by using federal reference methods 25 or 25aA, federal reference method 18 if approved by the department, the alternate version of federal reference method 25 incorporating the Byron analysis, **as described in R 336.2004**, or an alternate method that is acceptable to the department. ~~Federal reference methods 1, 2, 3, and 4, as described in R 336.2004, shall~~ **must** be used **as applicable** for the determination of the volumetric flow rate in the effluent gas. ~~Alternate federal reference method 1A, 2A, 2C, or 2D shall be used where appropriate.~~

(b) When an add-on emissions control device is used that recovers volatile organic compounds, the reduction efficiency of the device ~~shall~~ **must** be determined by using 1 of the following methods:

(i) A mass balance of the products used and the products recovered, using the following equation:

$$R_r = \frac{M_r}{M}$$

(ii) An alternate method that is acceptable to the department.

(iii) If there is only 1 add-on emissions control device used on a coating line or a graphic arts line, use the value calculated for "R_r" pursuant to paragraph (i) or (ii) of this subdivision as the value for R_T in subsequent calculations.

(c) If there is more than 1 add-on emissions control device used on a coating line or a graphic arts line, calculate the overall reduction efficiency by using the following equation:

$$R_T = \sum_{r=1}^s R_r (NT_r)$$

(12) Compliance with the specified emission limit ~~shall~~ **must** be determined using 1 of the following methods, as applicable, based ~~upon~~ **on** the form of the emission limit:

(a) For coating lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating, minus water, as applied, use either of the following methods:

(i) If only 1 coating is used on the coating line during the averaging time, use the following method:

(A) Determine the volatile organic compound content of the coating, minus water, as applied, "P," by using the method described in subrule (5) of this rule.

(B) If "P" is less than or equal to the specified emission limit, the coating line meets the emission limit.

(ii) If more than 1 coating of the same coating category is used on the coating line during the averaging period, use the following method:

(A) Determine the volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period by using the method described in subrule (5) of this rule.

(B) Determine the weight of volatile organic compounds used during the averaging period "M" by using the method described in subrule (6) of this rule.

(C) Determine the total volume of coatings used on the coating line during the averaging period "G_T" using the following equation:

$$G_T = \sum_{i=1}^z L_{ci}$$

(D) Determine the volume-weighted average weight of volatile organic compounds per gallon, minus water, as applied, by using the following equation:

$$P_a = \frac{M}{G_T}$$

(E) If "P_a" is less than or equal to the specified emission limit, the coating line meets the emission limit.

(b) For coating lines that have 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating, minus water, as applied, use the following method:

(i) Convert the specified emission limit to a modified emission limit "B" expressed in pounds of volatile organic compounds per gallon of coating solids, as applied, by using the following equation:

$$B = \frac{E}{1 - \frac{E}{7.36}}$$

(ii) Determine the volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period by using the method described in subrule (5) of this rule.

(iii) Determine the weight of volatile organic compounds used during the averaging period "M" by using the method described in subrule (6) of this rule.

(iv) Determine the total volume of coating solids used during the averaging period "V" by using the method described in subrule (7) of this rule.

(v) Determine the overall capture efficiency "N" by using the method described in subrule (10) of this rule.

(vi) Determine the overall reduction efficiency "R_T" by using the method described in subrule (11) of this rule.

(vii) Determine the volume-weighted average weight of volatile organic compounds per gallon of coating solids, as applied, "P_b," by using the following equation:

$$P_b = \frac{M}{V} (1 - R_T)$$

(viii) If " P_b " is less than or equal to the modified limit " B ," the coating line meets the emission limit.

(c) For coating lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating solids, as applied, use the following method:

(i) Determine the volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category " P " used during the averaging period by using the method described in subrule (5) of this rule.

(ii) Determine the weight of volatile organic compounds used during the averaging period " M " by using the method described in subrule (6) of this rule.

(iii) Determine the total volume of coating solids used during the averaging period " V " by using the method described in subrule (7) of this rule.

(iv) Determine the volume-weighted average weight of volatile organic compounds per gallon of coating solids, as applied, " P_c ," by using the following equation:

$$P_c = \frac{M}{V}$$

(v) If " P_c " is less than or equal to the specified limit, the coating line meets the emission limit.

(d) For coating lines that have 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating solids, as applied, use the following method:

(i) Determine the volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category " P " used during the averaging period by using the method described in subrule (5) of this rule.

(ii) Determine the weight of volatile organic compounds used during the averaging period " M " by using the method described in subrule (6) of this rule.

(iii) Determine the total volume of coating solids used during the averaging period " V " by using the method described in subrule (7) of this rule.

(iv) Determine the overall capture efficiency " N " by using the method described in subrule (10) of this rule.

(v) Determine the overall reduction efficiency " R_T " by using the method described in subrule (11) of this rule.

(vi) Determine the volume-weighted average weight of volatile organic compounds per gallon of coating solids, as applied, " P_d ," by using the following equation:

$$P_d = \frac{M}{V} (1 - R_T)$$

(vii) If " P_d " is less than or equal to the specified limit, the coating line meets the emission limit.

(e) For coating lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of applied coating solids, use the following method:

(i) Determine the volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period by using the method described in subrule (5) of this rule.

(ii) Determine the weight of volatile organic compounds used during the averaging period "M" by using the method described in subrule (6) of this rule.

(iii) Determine the total volume of coating solids used during the averaging period "V" by using the method described in subrule (7) of this rule.

(iv) Determine the overall transfer efficiency "T" by using the method described in subrule (9) of this rule.

(v) Determine the volume-weighted average weight of volatile organic compounds per gallon of applied coating solids "P_e" by using the following equation:

$$P_e = \frac{M}{V \frac{(T)}{100}}$$

(vi) If "P_e" is less than or equal to the specified limit, the coating line meets the emission limit.

(f) For coating lines that have 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per gallon of applied coating solids, use the following method:

(i) Determine the volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period by using the method described in subrule (5) of this rule.

(ii) Determine the weight of volatile organic compounds used during the averaging period "M" by using the method described in subrule (6) of this rule.

(iii) Determine the total volume of coating solids used during the averaging period "V" by using the method described in subrule (7) of this rule.

(iv) Determine the overall transfer efficiency "T" by using the method described in subrule (9) of this rule.

(v) Determine the overall capture efficiency "N" by using the method described in subrule (10) of this rule.

(vi) Determine the overall reduction efficiency "R_T" by using the method described in subrule (11) of this rule.

(vii) Determine the volume-weighted average weight of volatile organic compounds per gallon of applied coating solids "P_f" by using the following equation:

$$P_f = \frac{M}{V \frac{(T)}{100}} (1 - R_T)$$

(viii) If "P_f" is less than or equal to the specified limit, the coating line meets the emission limit.

(g) For graphic arts lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per pound of solids, as applied, use the following method:

(i) Determine the volatile organic compound content of each ink and coating, minus water, as applied, "P," used during the averaging period by using the method described in subrule (5) of this rule.

(ii) Determine the weight of volatile organic compounds used during the averaging period "M" by using the method described in subrule (6) of this rule.

(iii) Determine the weight of ink and coating solids used during the averaging period "W" by using the method described in subrule (8) of this rule.

(iv) Determine the average pounds of volatile organic compound per pound of solids, as applied, "P_g," by using the following equation:

$$P_g = \frac{M}{W}$$

(v) If "P_g" is less than or equal to the specified limit, the graphic arts line meets the emission limit.

(h) For graphic arts lines that have 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per pound of solids, as applied, use the following method:

(i) Determine the volatile organic compound content of each ink and coating, minus water, as applied, "P," used during the averaging period by using the method described in subrule (5) of this rule.

(ii) Determine the weight of volatile organic compounds used during the averaging period "M" by using the method described in subrule (6) of this rule.

(iii) Determine the weight of ink and coating solids used during the averaging period "W" by using the method described in subrule (8) of this rule.

(iv) Determine the overall capture efficiency "N" by using the method described in subrule (10) of this rule.

(v) Determine the overall reduction efficiency "R_t" by using the method described in subrule (11) of this rule.

(vi) Determine the average pounds of volatile organic compound per pound of solids, as applied, "P_h," by using the following equation:

$$P_h = \frac{M}{W} (1 - R_T)$$

(vii) If "P_h" is less than or equal to the specified limit, the graphic arts line meets the emission limit.

(i) For flatwood paneling coating lines that do not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compound per 1,000 square feet of coated finished product, use the following method:

(i) Determine the volatile organic compound content of each coating, minus water, as applied that belongs to the same coating category "P" used during the averaging period by using the method described in subrule (5) of this rule.

(ii) Determine the weight of volatile organic compounds used during the averaging period "M" by using the method described in subrule (6) of this rule.

(iii) Determine the total surface area of coated finished product for the coating category during the averaging period "sq".

(iv) Determine the volume-weighted average pounds of volatile organic compounds per 1,000 square feet of coated finished product "P_i" by using the following equation:

$$P_i = \frac{\frac{M}{sq}}{1,000}$$

Where the units for the term 1,000 are square feet.

(v) If "P_i" is less than or equal to the specified limit, the coating line meets the emission limit.

(j) For flatwood paneling coating lines that have 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per 1,000 square feet of coated finished product, use the following method:

(i) Determine the volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period by using the method described in subrule (5) of this rule.

(ii) Determine the weight of volatile organic compounds used during the specified averaging period "M" by using the method described in subrule (6) of this rule.

(iii) Determine the total surface area of coated finished product for the coating category during the averaging period "sq".

(iv) Determine the overall capture efficiency "N" by using the method described in subrule (10) of this rule.

(v) Determine the overall reduction efficiency "R_T" by using the method described in subrule (11) of this rule.

(vi) Determine the volume-weighted average pounds of volatile organic compounds per 1,000 square feet of coated finished product "P_j" by using the following equation:

$$P_j = \frac{\frac{M}{sq}}{1,000} (1 - R_T)$$

Where the units for the term 1,000 are square feet.

(vii) If "P_j" is less than or equal to the specified limit, the coating line meets the emission limit.

R 336.2041 Recordkeeping requirements for coating lines and graphic arts lines.

Rule 1041. (1) Unless otherwise specified in any of the following, the recordkeeping requirements specified in this rule ~~shall~~ **must** apply to coating lines and graphic arts lines subject to emission limits contained in any of the following:

- (a) These rules.
- (b) A permit to install.
- (c) A permit to operate.
- (d) A voluntary agreement.
- (e) A performance contract.
- (f) A stipulation.
- (g) An order of the department.

(h) A renewable operating permit.

(2) If a coating line does not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating, minus water, as applied, and if only 1 coating is used on the coating line during the averaging time, then a person shall keep records of the name, identification number, volume "Lci," and volatile organic compound content of the coating, minus water, as applied, "P," used during the averaging period, as described in R 336.204-0(5). If this single coating used during an averaging period is in compliance with all of the emission limits specified in subrule (1) of this rule, then the volume "Lci" for the averaging period may be calculated, based upon coating usage records during a time period of not more than 1 month, with the coating usage prorated to the specified averaging period using a method approved by the department for that coating line.

(3) If a coating line does not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating, minus water, as applied, and if more than 1 coating of the same coating category is used on the coating line during the averaging period, then a person shall keep all of the following records:

(a) The name, identification number, volume "Lci," and volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period, as described in R 336.2040(5). If all coatings used on the coating line during an averaging period are in compliance with all of the emission limits specified in subrule (1) of this rule, then the volume "Lci" for the averaging period may be calculated, based upon coating usage records during a time period of not more than 1 month, with the coating usage prorated to the specified averaging period using a method approved by the department for that coating line.

(b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).

(c) The total volume of coatings used on the coating line during the averaging period "GT," as described in R 336.2040(12).

(d) The volume-weighted average weight of volatile organic compounds per gallon, minus water, as applied, "Pa," as described in R 336.2040(12).

(4) If a coating line has 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating, minus water, as applied, then a person shall keep all of the following records:

(a) The name, identification number, volume "Lci," and volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period, as described in R 336.2040(5).

(b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).

(c) The total volume of coating solids and volume of ink or coating "Ldi" used during the averaging period "V," as described in R 336.2040(7).

(d) The overall capture efficiency "N," as described in R 336.2040(10).

(e) The overall reduction efficiency "Rt," as described in R 336.2040(11), including the parameters "Qza," "Cza," "Qim," "Cim," and "Mr."

(f) The volume-weighted average weight of volatile organic compounds per gallon of coating solids, as applied, "Pb," as described in R 336.2040(12).

(5) If a coating line does not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating solids, as applied, then a person shall keep all of the following records:

(a) The name, identification number, volume "Lci," and volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period, as described in R 336.2040(5).

(b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).

(c) The total volume of coating solids and volume of ink or coating "Ldi" used during the averaging period "V," as described in R 336.2040(7).

(d) The volume-weighted average weight of volatile organic compounds per gallon of coating solids, as applied, "Pc," as described in R 336.2040(12).

(6) If a coating line has 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per gallon of coating solids, as applied, then a person shall keep all of the following records:

(a) The name, identification number, volume "Lci," and volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period, as described in R 336.2040(5).

(b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).

(c) The total volume of coating solids and volume of ink or coating "Ldi" used during the averaging period "V," as described in R 336.2040(7).

(d) The overall capture efficiency "N," as described in R 336.2040(10).

(e) The overall reduction efficiency "Rt," as described in R 336.2040(11), including the parameters "Qza," "Cza," "Qim," "Cim," and "Mr."

(f) The volume-weighted average weight of volatile organic compounds per gallon of coating solids, as applied, "Pd," as described in R 336.2040(12).

(7) If a coating line does not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per gallon of applied coating solids, then a person shall keep all of the following records:

(a) The name, identification number, volume "Lci," and volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "Pp" used during the averaging period, as described in R 336.2040(5).

(b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).

(c) The total volume of coating solids and volume of ink or coating "Ldi" used during the averaging period "V," as described in R 336.2040(7).

(d) The overall transfer efficiency "T," as described in R 336.2040(9), including "Ti" and "Uci".

(e) The volume-weighted average weight of volatile organic compounds per gallon of applied coating solids "Pe," as described in R 336.2040(12).

(8) If a coating line has 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per gallon of applied coating solids, then a person shall keep all of the following records:

- (a) The name, identification number, volume "Lci," and volatile organic compound content of each coating, minus water, as applied, that belongs to the same coating category "P" used during the averaging period, as described in R 336.2040(5).
- (b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).
- (c) The total volume of coating solids and volume of ink or coating "Ldi" used during the averaging period "V," as described in R 336.2040(7).
- (d) The overall transfer efficiency "T," as described in R 336.2040(9), including "Ti" and "Uci".
- (e) The overall capture efficiency "N," as described in R 336.2040(10).
- (f) The overall reduction efficiency "Rt," as described in R 336.2040(11), including the parameters "Qza," "Cza," "Qim," "Vim," and "Mr."
- (g) The volume-weighted average weight of volatile organic compounds per gallon of applied coating solids "Pf," as described in R 336.2040(12).
- (9) If a graphic arts line does not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compounds per pound of solids, then a person shall keep all of the following records:
- (a) The name, identification number, volume "Lci," and volatile organic compound content of each ink and coating, minus water, as applied, "P," used during the averaging period, as described in R 336.2040(5).
- (b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).
- (c) The weight of ink and coating solids used during the averaging period "W," as described in R 336.2040(8), including "Wci" and "Ldi."
- (d) The average pounds of volatile organic compound per pound of solids, as applied, "Pg," as described in R 336.2040(12).
- (10) If a graphic arts line has 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per pound of solids, as applied, then a person shall keep all of the following records:
- (a) The name, identification number, volume "Lci," and volatile organic compound content of each ink and coating, minus water, as applied, "P," used during the averaging period, as described in R 336.2040(5).
- (b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).
- (c) The weight of ink and coating solids used during the averaging period "W," as described in R 336.2040(8), including "Wci" and "Ldi."
- (d) The overall capture efficiency "N," as described in R 336.2040(10).
- (e) The overall reduction efficiency "Rt," as described in R 336.2040(11), including the parameters "Qza," "Cza," "Qim," "Cim," and "Mr."
- (f) The average pounds of volatile organic compound per pound of solids, as applied, "Ph," as described in R 336.2040(12).
- (11) If a flatwood paneling coating line does not have an add-on emissions control device for which emission limits are expressed in pounds of volatile organic compound per 1,000 square feet of coated finished product, then a person shall keep all of the following records:

(a) The name, identification number, volume "Lci," and volatile organic compound content of each coating, minus water, as applied, "P," used during the averaging period, as described in R 336.2040(5).

(b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).

(c) The total surface area of coated finished product for the coating category during the averaging period "sq," as described in R 336.2040(3).

(d) The volume-weighted average pounds of volatile organic compounds per 1,000 square feet of coated finished product "Pi," as described in R 336.2040(12).

(12) If a flatwood paneling coating line has 1 or more add-on emissions control devices for which emission limits are expressed in pounds of volatile organic compounds per 1,000 square feet of coated finished product, then a person shall keep all of the following records:

(a) The name, identification number, volume "Lci," and volatile organic compound content of each coating, minus water, as applied, "P," used during the averaging period, as described in R 336.2040(5).

(b) The weight of volatile organic compounds used during the averaging period "M," as described in R 336.2040(6).

(c) The total surface area of coated finished product for the coating category during the averaging period "sq," as described in R 336.2040(3).

(d) The overall capture efficiency "N," as described in R 336.2040(10).

(e) The overall reduction efficiency "Rt," as described in R 336.2040(11), including the parameters "Qza," "Cza," "Qim," "Cim," and "Mr."

(f) The volume-weighted average pounds of volatile organic compounds per 1,000 square feet of coated finished product "Pj," as described in R 336.20-40(12).

(13) An owner or operator of primer surfacer or topcoat operations subject to emission limits in R 336.1610(11), table 62, **or R 336.1610a(4), table 64-a**, shall keep records as required in the publication entitled "Protocol for Determining the Daily Volatile Organic Compound Emission Rate of Automobile and Light-duty Truck Topcoat Operations," EPA-453/R-08/0020/3-88-018, December, 1988, which is referenced in R 336.1610(6)(b) **adopted by reference in R 336.1902.**

(14) The records that are required in this rule ~~shall~~ **must** be retained for a period of not less than 2 complete years ~~from~~ **after** the date of collection and, ~~upon~~ **on** request by the department, ~~shall~~ **must** be submitted to the department in an acceptable format.