



Paint Material Odor Assessment - Olfactometry

1 Introduction

Note: Nothing in this standard supercedes applicable laws and regulations.

Note: In the event of conflict between the English and domestic language, the English language shall take precedence.

1.1 Purpose. This standard covers the requirements for assessing the relative odor potential of automotive paint materials. It includes liquid paint materials that are applied by spraying, electrodeposition primers and solvents. It also includes three methods for sample collection and the appropriate use of these methods.

1.2 Applicability. Paint Materials including liquid paint materials that are applied by spraying, electrodeposition primers and solvents.

1.3 Remarks. None.

2 References

Note: Only the latest approved standards are applicable unless otherwise specified.

2.1 External Standards/Specifications.

ASTM 679

ASTM E544

2.2 GM Standards/Specifications.

None

2.3 Additional References.

Air and Waste Management Association (AWMA) E6 Odor Committee Standard

Harper's Scale of Odor-Quality Descriptors as a Guide

3 Resources

3.1 Facilities. Environment suitable for test.

3.1.1 Calibration. The test facilities and equipment shall be in good working order and shall have a valid calibration label.

3.1.2 Alternatives. Alternative test facilities and equipment may also be used. However, all measuring variables as specified in this standard shall be determined correctly with respect to their physical definition.

3.2 Equipment. Equipment requirements are separated into four categories: General Equipment, Method A Equipment, Method B Equipment and Method C Equipment.

3.2.1 General Equipment.

- Triangular dynamic dilution olfactometer– capable of supplying regulated concentrations of sample to the test port at calibrated flow rates.
- Butanol sample bottles from 15 ppm to 2000 ppm with a step factor of two (2) for each intensity (eight (8) point scale).
- Tedlar sampling bags with a 10 L to 40 L volume capacity or as determined by olfactometer setup.
- Volumetric flow rate measurement equipment capable of measuring to ± 0.01 L/minute.
- Teflon Tubing. 3.2 mm ($\frac{1}{8}$ in) and 6.4 mm ($\frac{1}{4}$ in) Outer Diameter (OD).
- Thermometer (for ambient temperature).
- Barometer.

- h. Stop Watch.
- i. Ultrasonic Cleaner.
- j. Pure Nitrogen Cylinder.
- k. Matheson Dual Stage Regulator with 0 psig to 200 psig delivery pressure range.

3.2.2 Method A Equipment.

- a. Dry filter spray booth.
- b. Cup guns or pressure pots (0.8 L to 2 L).
- c. Rigid container odor sampling lung and evacuation pump.
- d. Sampling probe with minimum 6.4 mm (¼ in) Inner Diameter (ID).
- e. 100 mm × 300 mm phosphate plain steel panels.

Note: Pretreatment as defined by responsible engineer.

3.2.3 Method B Equipment.

- a. Force-dry, gas-fired oven capable of 200 °C (400 °F).
- b. 100 mm × 300 mm phosphate plain steel panels.

Note: Use pretreatment as defined by responsible engineer.

3.2.4 Method C Equipment.

- a. Oven for liquid evaporation. Varian Aerograph Series 1200 Chromatograph or equivalent.
- b. Digital flow meter capable of measuring 1 L/minute to 2 L/minute with a resolution of 0.001 L/minute (Gilian Primary Standard Airflow Calibrator (Giliblator) or equivalent).
- c. Teflon tubing with internal diameter of 3.2 mm (⅛ in).
- d. Analytical balance capable of weighing 20 g within 0.0001 g.
- e. Syringes; 10 µL to 250 µL with spare needles. (Hamilton Model 1801RN and Model 1825RN Gastight Syringes or equivalent).
- f. Ultrasonic Cleaner.
- g. Septa for Gas Chromatograph (GC) injection port.

3.3 Test Vehicle/Test Piece. Required amount of automotive paint materials for adequate assessment.

3.4 Test Time. Necessary time to complete assessment.

3.5 Test Required Information. Required assessments.

3.6 Personnel/Skills. Test technicians to test to requirements.

4 Procedure

4.1 Preparation. See 4.3.1.

4.2 Conditions.

4.2.1 Environmental Conditions. Not applicable.

4.2.2 Test Conditions. Deviations from the requirements of this standard shall have been agreed upon. Such requirements shall be specified on component drawings, test certificates, reports, etc.

4.3 Instructions.

4.3.1 Test Procedure. Basic methodology for sample collection and odor measurement protocol is per guidelines set forth in the Air and Waste Management Association (AWMA) E6 Odor Committee Standard (for Method A). Exceptions and clarifications for GM testing and data reporting are contained in this procedure.

All testing is to be done using a side-by-side relative comparison approach. For example, new materials should be compared to the existing materials they intend to replace.

4.3.1.1 Sample Collection. All odor samples must be collected using odor-free, chemically inert, nonreactive, low permeability, smooth materials. Line lengths from sampling probe to the sampling bag must be kept to a minimum.

4.3.1.1.1 Sample Bags. Bags must be Tedlar with a 10 L to 40 L capacity unless otherwise agreed upon prior to start of test.

4.3.1.1.2 Conditioning. Preconditioning must be done with both inert gas and sample gas. Use an agreed upon method.

4.3.1.1.3 Replication. Three (3) runs per sample must be done for each collection method.

4.3.1.1.4 Documentation. Information requirements are shown in Appendix A, Table A3 and Table A4.

4.3.1.2 Collection Methods. There are three (3) methods for collecting odor samples based on the type of material being evaluated.

4.3.1.2.1 Method A; Paint Spray Application. This method is used for simulation of relative odor potential of liquid paints that are spray-applied. Sampling for this method is done in the exhaust stack from the booth per Appendix A, Figure A1. Typical materials evaluated are liquid basecoats, clearcoats or primers.

4.3.1.2.1.1 Sample Preparation. Reduce paint as required to simulate production or use a production sample at spray viscosity. Determine initial paint properties Volatile Organic Compounds (VOC), weight solids, etc., and the amount of paint sprayed by both weight and volume.

4.3.1.2.1.2 Replication. Spray three (3) 100 mm × 300 mm panels per run. There should be three (3) runs per sample unless otherwise agreed upon.

4.3.1.2.1.3 Sampling Time is One (1) Minute. Initiate sampling simultaneously with the initiation of spraying. Sample the exhaust stack continuously during spraying. Specify draw rate in Liters per minute.

4.3.1.2.1.4 Paint Colors. Odor analysis for a new topcoat technology must be done with a minimum palette of White, Medium Red Mica and Light Metallic plus clearcoat. Odor analysis for a new primer-surfacer technology must be done on a minimum of Gray and Red. Specific color codes (i.e., WAXXXX or L), will be agreed upon at test initiation. Testing of additional colors may be required prior to plant implementation, depending on color-to-color solvent variation and plant location.

4.3.1.2.2 Method B; Oven Bake. This method is typically used for electrodeposition primer, powder paint, liquid paints that may give off odorous by-products, sealers and adhesives. Materials are baked at upper bake limits and samples are collected from the oven exhaust air per Appendix A, Figure A2.

4.3.1.2.2.1 Sample Preparation. Apply target films of electrodeposition primer or powder. A minimum of six (6) panels per run must be used.

4.3.1.2.2.2 Sampling Time. Heat oven to upper limit of bake schedule and insert test specimens. Bake for 30 minutes and sample continuously at constant flow rate to the bag.

4.3.1.2.3 Method C; Head Space. This method should be used for determining odor threshold of solvents and for screen testing liquid paints for impact of formulation changes. See Appendix A, Figure A3 for equipment layout.

4.3.1.2.3.1 Sample Preparation. Paint samples must be at spray viscosity and thoroughly mixed.

4.3.1.2.3.2 Test Conditions. For paint samples, the following settings of the gas chromatograph should be used:

- Program Mode: ISO (International Organization for Standardization (ISO))
- Injector Temperature: 45 °C (113 °F)
- Column Oven: Oven and Fan
- Oven Temperature: 45 °C (113 °F)
- Other Settings: Not Used

4.3.1.2.3.3 Sample Size. Use 50 µL for paint unless otherwise agreed upon. Quantity of sample should be such that the resulting odor Detection Threshold (T_d) (Dilution/Threshold (D/T)) is between 50 OU and 500 OU; (Odor Units (OU)). Select a clean syringe in the proper size (250 µL for 50 µL to 500 µL liquid volume, 10 µL for 10 µL liquid volume) and flush the syringe with the liquid sample at least three (3) times.

4.3.1.2.3.4 General Procedure. Connect a clean, dry Teflon tube to the gas chromatograph (approximately 0.3 cm OD by 100 cm long). With power on, turn nitrogen cylinder on and adjust the regulator for delivery pressure of about 100 psig. Extract the exact appropriate volume of liquid. Weigh the filled syringe and record the weight as the initial weight.

4.3.1.2.3.4.1 Measure the flow rate at the sample line outlet using the Gilibrator. Adjust the cylinder regulator to give a steady flow rate of 0.8 L/minute to 1.2 L/minute. Record this as the initial flow rate.

4.3.1.2.3.4.2 Connect a cleaned and labeled 30 L Tedlar bag to the outlet of the sample line. Collect some nitrogen and flush the bag. Reconnect the bag to the sample line outlet and start timing immediately. Collect the nitrogen for approximately two (2) minutes and inject the liquid sample into the injection port. Allow the needle to reside in the injector port for at least one (1) minute to evaporate the droplet at the tip. Re-weigh the syringe and record as the final weight.

4.3.1.2.3.4.3 Continue running for 30 minutes with the nitrogen on. Remove the Tedlar bag at the end of the sampling period and record the sampling time.

4.3.1.2.3.4.4 Measure the flow rate immediately and record this as the final flow rate. If the difference between initial and final rate is greater than 10%, the sample must be discarded and the cause of variance must be ascertained.

4.3.1.2.3.4.5 Remove the sample line and flush it with acetone and then distilled water. Then clean it in an ultrasonic cleaner for at least 15 minutes. Re-clean with distilled water and dry with clean, dry air.

4.3.1.2.3.5 Calculations. The volume of nitrogen used to fill the sample bag is calculated using the average of the initial and final flow rates multiplied by the total sampling time, correcting for temperature and pressure. The concentration of liquid in the sample bag is calculated by dividing the weight of the liquid injected in milligrams by the volume of nitrogen in cubic meters. This concentration, divided by the threshold odor value gives the threshold concentration in milligram per cubic meter (mg/m^3).

4.3.1.3 Odor Test Protocol. The time interval between sample collection and odor panel evaluation must be no more than 24 h. As a general practice, analysis should be conducted with 8 h of sample collection.

4.3.1.3.1 Panel Selection. Panelists must be screened for sensitivity to n-Butanol, which is the reference standard, and trained in the use of olfactometer equipment. Report an average sensitivity for each group.

4.3.1.3.2 Olfactometer. A dynamic olfactometer should be used in accordance with ASTM 679. Any deviation must be discussed with the appropriate engineer for approval prior to testing.

4.3.1.3.3 Flow Rate. The sample gas flow rate must be between 5 L/minute and 10 L/minute per sniff port.

4.3.1.3.4 Sample Dilution. The maximum step factor between subsequent concentrations is two (2). The same dilution factor must be used for each replicate. Report any variance in step factor from concentration to concentration.

4.3.1.3.5 Odor Measurements. The following odor parameters will be determined by measurement or calculation and reported.

4.3.1.3.5.1 Dilution to Detection Threshold (D/T_d). Where the Detection Threshold (T_d) is the lowest concentration of an odorant at which an odorized sample can be distinguished from non-odorous air by 50% of the group of observers.

Note: This may also be referred to as an Effective Dose at 50% of the population (ED_{50}).

4.3.1.3.5.2 Dilution to Recognition Threshold (D/T_r). Where the Recognition Threshold (T_r) is the lowest concentration of an odorant at which the odor character can be described/characterized by 50% of the group of observers.

4.3.1.3.5.3 Odor Unit (OU). An odor unit is the amount of odor produced by a given Dilution to Detection Threshold (D/T_d) by the volume or weight of paint sprayed. Report OU_v , and OU_w when using collection Method A (see 3.2.2). Report by volume when using collection Method B (see 3.2.3) and Method C (see 3.2.4).

Note: OU_v = Odor units based on volume of paint sprayed in milliliters for odor sample collection in the stack.

Note: OU_w = Odor units based on weight in gram (g) of paint sprayed for odor sample collection in the stack.

4.3.1.3.5.4 Intensity (I). Intensity is the comparison of the odorant strength, T_r , of n-Butanol in water. Butanol samples are prepared per ASTM E544, Method B on an eight (8) point scale which starts at a concentration of 15 ppm and goes to 2000 ppm. See Appendix A, Table A1 for the relationship between the rating scale and the concentration level of n-Butanol in water.

4.3.1.3.5.5 Odor Character Description. This is a general description of odor using Harper's Scale of Odor-Quality Descriptors as a Guide. See Appendix A, Table A2.

4.3.1.4 Other Definitions and Calculations.

4.3.1.4.1 Dilution Air. The odor-free air used to dilute the odor sample and serve as the blanks against which the samples are judged. The quality of the dilution air must be judged to be odor-free by all panel members and the panel moderator. A typical procedure for obtaining odor-free air is to filter it through a charcoal filter.