

SMOKE CAUSING EMISSIONS FROM PAINTS AND SEALERS

GM9828P

1 SCOPE. This procedure is established to standardize the analysis and reporting of the mass of the emissions from paint and sealer materials during bake in an oven as a function of temperature and number of bakes. This procedure specifically addresses the smoke-causing part of the oven emissions, which includes solids and semi-volatile constituents. Emissions of volatile compounds are not intended to be completely quantified by this procedure.

2 REFERENCED STANDARDS.

None

3 TEST EQUIPMENT.

3.1 BAKE OVEN SET-UP. The schematic of the set-up for baking and collecting the emissions from a given sample material is shown in Figure 1. A Thermolyne 21135 oven or equivalent (electrical) is used to bake the sample material. The oven is a cylindrical unit equipped with temperature control in the range 100 to 300°C. A custom Pyrex tube liner (2" OD, 1.5" ID, 22" length, 50/50 ground glass joint on one end, 12/5 ball joint on the other end) is placed snugly into the oven. A 50/50 to 24/50 reducer is used to connect a 24/40 adapter with inlet hose connection to the liner. Bottled air (hydrocarbon free) is connected to a mass flow controller that meters the air flow to the adapter. A glass wool plug is placed at the adapter-liner junction to smooth out the air flow. To eliminate heat loss and condensation, insulation or heating tape is used to cover the exposed glass in the adapter-liner assembly.

3.1.1 The sample is placed in the middle of the oven liner on top of a metal grid (1.25 x 6"). The metal grid rests on the inner wall of the oven liner (See Figure 2). A thermocouple wire (K-type, exposed junction, fiberglass insulated) is passed through the adapter and is placed in contact with the under side of the metal grid or sample panel/container. A pair of magnets is used to hold the thermocouple in place. The thermocouple is connected to a temperature recorder (See Figure 1).

3.1.2 The exhaust end of the oven liner is connected through a 12/5 ball connection to an L-shaped adapter (end tube). The adapter has a 12/5 socket on one end and a 12/5 ball on the other end. The adapter is then connected to a 24/40 25-mL midjet impinger with a 24/40 bubbler having 12/5 socket and ball joints. The oven end assembly up to the impinger is fully insulated (heating tape can be used). The oven exhaust bubbles through the impinger, which is placed in an ice-water bath. The air from the impinger passes through a short tygon tube into a filter holder assembly. The filter holder houses a 47 mm filter (Pallflex, type T60A20). The air flows out of the filter into an exhaust hood.

NOTE: All joints, reducers, adapters, connections and sockets are standard taper.

3.2 MISCELLANEOUS. Additional equipment and laboratory items required for testing include small pieces of stiff wire

(or paper clips), 50 x 9 mm sterilized disposable petri dishes, 20 mL screw thread glass vials with open top cap and Teflon/silica septa, film thickness gauge (CMI CGX-A2 or equivalent), four place balance (precision: ± 0.1 mg), desiccator, 50 mL aluminum dishes, 47 mm filters (Pallflex, type T60A20), and analytical grade methanol.

4 SAMPLE PREPARATION.

4.1 ELECTROCOAT AND LIQUID COATINGS. For testing of electrocoat material (ELPO), arrangements have to be made with the supplier to coat preweighed cold roll steel panels (uncoated panel weight = W_i) of size 1.5 x 4" with the ELPO material (the bath conditions and the film thickness should match the actual production conditions). The coated panels are then used for emissions testing. Note that the testing of the precoated panels should be made within 0 to 48 h from the time the panels are coated. Furthermore, the precoated panels should be received in an air-tight container (minimizing exposure to air and loss of volatiles) and kept in the container, preferably refrigerated, while waiting to be tested.

4.1.1 Preweigh the metal grid and 2 small pieces of stiff wire or paper clips (risers) (W_2). Place the risers on the grid. With as little touching as possible, place the coated panel over the risers on the support plate (note that the ELPO-panels are coated both sides). Weigh the assembly (W_f). The initial sample weight (W_{si}) is then:

$$W_{si} = W_f - W_i - W_2 \quad (1)$$

4.1.2 For testing of liquid coatings (primer, basecoat, or clearcoat), arrangements have to be made with the supplier to coat panels (1.5 x 4"). They can also be prepared in the laboratory by placing the coating material on the panel and smoothing it out to the desired thickness using the simple panel-slide test unit. The coated area should be at least 1" wide by 3" long at a thickness comparable to the prebaked production settings. The panel has to be weighed before (W_i) and after coating (W_f). The initial sample weight is then:

$$W_{si} = W_f - W_i \quad (2)$$

4.1.3 If the initial weight of the cold roll steel panel is not available, then the weight of the cured sample (W_{sf}) can be estimated using measured film thickness, coated surface area, and estimated specific gravity of the baked coating on the panel. Initial sample weight can then be estimated by adding the measured weight loss of the sample during bake (ΔW_s) to W_{sf} , i.e.:

$$W_{si} = W_{sf} + \Delta W_s \quad (3)$$

4.2 POWDER PAINT AND SEALERS. For powder paint, the sample is tested using an aluminum dish. Trim off the sides of an aluminum weighing dish so that it will fit inside the oven liner. Weigh the dish (W_i). The powder sample is then sprinkled in the dish and smoothed out to cover at least a (1.5 x 2") area at a thickness comparable to the prebake production settings (the amount of powder needed can be estimated from the

SMOKE CAUSING EMISSIONS FROM PAINTS AND SEALERS

GM9828P

coverage area, desired thickness, and specific gravity of the cured powder). The dish is then weighed again (Wf).

4.2.1 For a sealer that comes as paste, place about 1.0 g of the sample in a preweighed aluminum dish (Wi). The sample is then smoothed out to cover at least (1.5 x 2") area. The dish is then weighed again (Wf). The initial weight of the sample is then given by Eq. 2.

4.2.2 For a sealer that comes in a solid form, cut a (1.5 x 2") piece and place it in an aluminum dish. Weigh the dish before (Wi) and after (Wf). The initial weight of the sample is then given by Eq. 2.

5 TEST PROCEDURE.

5.1 INITIAL PREPARATIONS.

- Make sure all the components of the system are clean.
- Turn the oven on and set to the desired temperature. Allow 1 h for temperature stabilization.
- Put an aluminum dish in the desiccator for 1 h and then weigh (Ai).
- Preweigh the bottom half of a petri dish, then set the Pallflex filter in the dish and place uncovered in the desiccator for 1 h. The filter is then weighed with the petri dish (Fi). Place the filter in the filter holder. Care must be taken to ensure that both parts of the filter holder are lined up correctly before tightening or leaks will occur, nullifying the filter data.
- Fill the impinger with 20 mL methanol and place it in the ice bath. Clamp the impinger inlet to the exhaust end of the oven liner and the impinger outlet to the filter holder.
- Turn on the purified air and set the flow rate at 1 L/minute. Verify that there are no leaks throughout the system. (one way to check this is by measuring the air flow out of the filter).

5.2 EMISSIONS TEST. Open the adapter assembly. With as little touching as possible, place the metal grid supporting the coated panel or the aluminum dish in the oven. With a pair of magnets, attach the thermocouple to the sample container or the coated panel underside near its middle; for ELPO panels, place the thermocouple in contact with the grid. The magnets should not touch the thermocouple junction. Using tongs, place the metal grid in the center in the oven. Push the metal grid into the oven using a glass rod or similar tool until the sample is centered in the oven. Close the adapter assembly and connect the thermocouple to the recorder.

5.2.1 Set the timer for 25 minutes and begin recording the temperature of the sample. After 25 minutes, open the adapter assembly and remove the metal grid with the sample. Close the adapter assembly. Set the temperature of the oven to 240°C and flush the system with air for 40 minutes. Leave the sample to cool to room temperature.

5.2.2 After the 40-minute flush run, turn the oven and air off. Remove the filter and place it in the petri dish that was used in the initial weighing of the filter; leave the dish in the desiccator to cool for 60 minutes. Also, remove the impinger and the end tube. Leave the end for 15 minutes to cool. Pour the impinger solution into a 20-mL vial. Rinse the end tube twice, each time with 2 mL fresh methanol, and pour the rinse into the impinger. Then pour this rinse solution (4 mL) into the 20-mL vial. Use 2 mL fresh methanol to clean rinse the end tube into the impinger and add the rinse to the 20-mL vial. Use additional methanol to rinse the impinger to bring the total solution volume in the vial to 20 mL. Keep 5 mL of the vial solution for analytical speciations (not part of this standardized test), and retain the remaining 15 mL for emissions analysis.

6 ANALYSIS.

6.1 TOTAL WEIGHT LOSS.

Weigh the baked sample/panel (Wff). The weight of the sample after bake is then:

$$W_{sf} = W_{ff} - W_i \quad (4)$$

The total weight loss of the sample during the bake (ΔW_s) is:

$$\Delta W_s = W_{sf} - W_{si} \quad (5)$$

The total weight loss of the sample in wt% is given by:

$$w_i = \Delta W_s / W_{si} * 100 \quad (6)$$

$$w_f = \Delta W_s / W_{sf} * 100 \quad (7)$$

where w_i and w_f are based on the initial sample weight (wet) and the final weight (baked), respectively.

For the cold roll steel and wet coatings, measure the cured film thickness using a calibrated film thickness gauge. Report the results in mils.

6.2 TOTAL SMOKE CAUSING EMISSIONS.

Place 15 mL of the impinger solution that was collected in the 20-mL vial in the desiccated aluminum dish (initial weight= A_i). The sample is then allowed to sit in a hood @ 20°C for 24 h. The sample is then desiccated for about 1 h and weighed (A_f). The increase in the weight of the dish represents the mass of solids collected in the vial. This number multiplied by 4/3 gives the solids collected in the impinger and the end tube (E_1), i.e.

$$E_1 = 4/3 (A_f - A_i) \quad (8)$$

Take the petri dish which contains the filter out of the desiccator and weigh it (F_f). The weight increase over its initial weight (F_i) gives the mass of solids collected on the filter (E_2), i.e.

$$E_2 = F_f - F_i \quad (9)$$

The smoke-causing emissions from the sample (E) is the sum of the solids collected in the impinger as well as on the filter:

$$E = E_1 + E_2 \quad (10)$$

The emissions as wt% of the samples are:

$$E_i = E / W_{si} * 100 \quad (11)$$

$$E_f = E / W_{sf} * 100 \quad (12)$$

where e_i and e_f are based on the initial sample weight (wet) and the final weight (baked), respectively.

SMOKE CAUSING EMISSIONS FROM PAINTS AND SEALERS

GM9828P

7 TEST MATRIX. A given sample material must be tested for emissions a number of times to determine the effects of bake temperature and number of bakes. The following lists the required test matrix for ELPO, powder / liquid primer, and sealers. For any test, the oven temperature should be controlled within $\pm 3^{\circ}\text{C}$ of the specified temperature for the test. The oven temperature is calculated to be the average temperature recorded by the thermocouple during the test over the 6 to 25 minute period.

7.1 ELPO. The test matrix for ELPO panels is given in Table 1. ELPO panels should be run at 3 different temperatures

based on the bake window specified for the material: the lowest temperature (Emin), maximum temperature (Emax), and target temperature (Etar). Four (4) ELPO panels are run at each temperature. After taking all necessary measurements, these panels then go through a second bake for 25 minutes. The temperature for the second bake is defined by the operating window for powder or liquid primer that is going to be used with the ELPO. The rebake is done at the primer oven target (Ptar) and maximum (Pmax) temperatures. The 12 ELPO panels, which were baked at Emin, Etar, and Emax in the first bake, are rebaked at Ptar and Pmax with 2 runs per temperature.

TABLE 1 - TEST MATRIX FOR ELPO PANELS

	First Bake at ELPO Oven Temperatures	Second Bake at Primer Oven Temperatures
ELPO	Emin (4)* +	Ptar (2), Pmax (2)
	Etar (4)	Ptar (2), Pmax (2)
	Emax (4)*	Ptar (2), Pmax (2)

* Number in brackets denotes the number of runs at the temperature.

+ Prepare 6 panels instead of 4 if also running the test matrix for primer (Table 2).

7.2 PRIMER. The test matrix for powder/liquid primer is given in Table 2. The primer is tested at three temperatures based on the operating window for the primer: minimum

(Pmin), target (Ptar), and maximum (Pmax). The primer is also prepared over ELPO panels that have been baked at Emin and Emax. These panels are then baked for 25 minutes at Pmax.

TABLE 2 - TEST MATRIX FOR POWDER/LIQUID PRIMER

Bake by itself	Over ELPO baked at Emin	Over ELPO baked at Emax
Pmin (2)		
Ptar (2)		
Pmax (2)	Pmax (2)	Pmax (2)

7.3 SEALERS. The test matrix for sealers is given in Table 3. The sealers that are applied in the body shop (before paint) are tested at Emin, Etar, and Emax. The samples that are baked at Emin and Emax are then rebaked at Pmax for 25 minutes.

ELPO) are baked at Pmin, Ptar, and Pmax.

7.3.2 The sealers that are applied after the primer oven are baked at three temperatures based on the operating window for the sealers: minimum (Smin), target (Star), and maximum (Smax).

7.3.1 The sealers that are applied in the paint shop (after

TABLE 3 - TEST MATRIX FOR SEALERS

Body Shop Sealers		Sealers after ELPO	Sealers after Primer
First Bake at ELPO Oven Temp	Second Bake at Primer Oven Temp	Bake at Primer Oven Temp	Bake at Sealers Oven Temp
Emin (2)	Pmax (2)	Pmin (2)	Smin (2)
Etar (2)		Ptar (2)	Star (2)
Emax (2)	Pmax (2)	Pmax (2)	Smax (2)