

**NARRATIVE REPORT**

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**Job Code:** 209003

**Report Date:** May 6, 2004

**Client:** Samuel J. Goldberg, American Formulation & Manufacturing

**Product I.D.:** 50606-SR60 Safecoat Polyureseal BP Coating

**Manufacturer's I.D.:** DA94

**Production Date:** April 19, 2004

**Product Received:** April 22, 2004

**Report Prepared By:** R.S. Tannous, Laboratory Director

**Report Approved By:** 

Alfred T. Hodgson, Research Director

**Date:** 5/7/04

**OBJECTIVE**

The objective of this test was to measure the emissions of formaldehyde and total volatile organic compounds (TVOC) from a clear coating sample at 24 hours. The test conformed to the guidance of the Japanese Industrial Standard, JIS A 1901 (2003), "Determination of the emission of volatile organic compounds and aldehydes for building products-Small chamber method."

**SUMMARY**

The coating was tested for emissions of formaldehyde and TVOC. The test specimen was prepared by applying 1.73 grams of coating to a 178-mm x 178-mm (0.0316 m<sup>2</sup>) stainless steel plate with a roller. The coating was allowed to cure for one hour before transferring to the test chamber. The environmental parameters for the test were 28±1°C temperature, 50±5% relative humidity and 1.0±0.05 air change rate. Measurements of chamber VOC concentrations were made at 24 hours after initiating the test. Area-specific emission rates (EF<sub>a</sub>) were calculated by mass balance. The coating did not emit formaldehyde above the lower limit of quantitation of <2.1 µg m<sup>-2</sup> h<sup>-1</sup>. The TVOC area-specific emission rate was 6,270 µg m<sup>-2</sup> h<sup>-1</sup>.

**METHODS**

***Description of Product Specimen***

On April 22, 2004, the laboratory received a product sample consisting of a container of a clear coating. The sample was identified as: manufacture's ID # DA94; product name Safecoat Polyureseal BP-SR60; sample ID 50606-SR60; manufacturing date 4/19/04. The sample was handled in accordance with BAA-SOP-2000.02, "Selection, Collection and Handling of Material Specimens for Testing to Determine the Emissions of Volatile Organic Compounds." On April 26, 2004, the container was opened. The coating was first thoroughly mixed in its container by stirring. Approximately 100 ml volume was transferred to an aluminum tray. A paint roller with a 10-cm cover was saturated with coating by running the roller back and forth in the tray. The coating was applied to one side of a 178-mm x 178-mm (0.0316 m<sup>2</sup>) stainless steel plate using four strokes, two in the vertical direction and two in the horizontal direction, so that the entire area

was uniformly covered. 1.73 Grams of coating were applied in a single film. The coated surface area was 0.0316 m<sup>2</sup>. Thus, the product loading was 55 g m<sup>-2</sup>. The test specimen was allowed to dry for one hour before placing it on an open wire shelf in the test chamber.

### **GC/MS Analysis of TVOC**

The methods used for the sampling and analysis of VOCs are based on U.S. EPA Methods TO17, "Determination of Volatile Organic Compounds in Ambient Air Using Active Sampling Onto Sorbent Tubes" and TO1, "Determination of Volatile Organic Compounds in Ambient Air using Tenax Adsorption and Gas Chromatography/Mass Spectrometry (GC/MS)."

The multisorbent samplers (Model No. ST-032, Envirochem, Inc.) used for the collection of VOC samples contain Tenax-TA, Amborsorb XE-340 and activated charcoal, in series. With these samplers, C<sub>4</sub> to C<sub>16</sub> nonpolar and moderately polar volatile organic compounds (VOCs), and many C<sub>1</sub> to C<sub>3</sub> compounds, depending upon functional group, are quantitatively collected. Highly reactive and very volatile compounds, such as formaldehyde, cannot be analyzed using these samplers.

The samplers are thermally desorbed, and the samples are introduced into a Hewlett-Packard 5971A GC/MS system using a UNACON 810 concentrating system (Envirochem, Inc.). Prior to analysis, an internal standard (ISTD) is added to each sampler. The ISTD is 121 ng of bromofluorobenzene (BFB). The ISTD is used to check on the operation of the system, to provide a retention-time marker, and for quantitative analysis. The GC/MS is operated in the SCAN mode over a mass range of *m/z* 33-300.

For the quantitative analysis of total VOCs (TVOC) in a sample, the GC/MS total-ion-current (TIC) chromatogram is integrated over a retention-time (RT) interval of 15 - 50 minutes using parameters that capture almost all of the TIC area in a sample. The integrated areas less the area of the ISTD are summed. The mass of the compounds represented by the sum is calculated relative to the known amount of the ISTD that is added to the sampler. The calculation uses the response factor for toluene relative to BFB, the ISTD. This relative response factor (RRF) is 1.85. Because there can be substantial variation in the TIC response of different classes of compounds, the measurement of TVOC is less accurate than the calibrated measurement of individual VOCs.

### **Formaldehyde Analysis**

The methods used for the sampling and analysis of formaldehyde and other carbonyl compounds are based on ASTM Method D-5197, "Standard Test Method for Determination of Formaldehyde and Other Carbonyl Compounds in Air (Active Sampler Methodology)."

Sep-Pak XPoSure Aldehyde Samplers (Part number WAT047205, Waters, Corp.) are used to collect air samples for formaldehyde and other low-molecular weight carbonyl compounds. Air is pulled through a sampler, and the acidified 2,4-dinitrophenylhydrazine (DNPH) reagent in the sampler reacts with carbonyl compounds to form the stable hydrazone derivatives that are retained by the sampler. The hydrazone derivatives are eluted from a sampler with acetonitrile. An aliquot of the sample is analyzed for the hydrazone derivatives of formaldehyde and acetaldehyde using reverse-phase high-performance liquid chromatography (HPLC) with UV detection. The absorbance of the derivatives is measured at 360 nm. The mass responses of the resulting peaks are determined using multi-point calibration curves prepared from standard solutions of the hydrazone derivatives.

### **Testing for Emissions of VOCs from Products Using Small-Volume Chambers**

The methods used for the measurement of emissions of VOCs from this product specimen are based on the Japanese Industrial Standard, JIS A 1901 (2003), "Determination of

the emission of volatile organic compounds and aldehydes for building products-Small chamber method," English edition.

The chamber consists of a 67-L, stainless steel cylindrical vessel with a stainless-steel lid equipped with three fittings. The chamber is held in an incubator that is maintained at  $28 \pm 1^\circ \text{C}$ . Purified air from a clean air generator is introduced into the chamber through one fitting with a stainless-steel tubing extension. The inlet flow rate of  $1.12 \pm 0.06$  standard  $\text{L min}^{-1}$  is regulated with electronic mass-flow controllers (calibrated at  $25^\circ \text{C}$  and 1 atm. pressure). The gas stream is split into two streams. One of these is passed through a bubbler containing distilled water. This saturated gas stream is mixed with the dry gas stream to produce a humidified gas stream with a relative humidity (RH) of  $50 \pm 5\%$  that is then introduced into the chamber. A humidity probe (Model HMD 30YB, Vaisala) is inserted into the chamber through a second fitting on the lid. Chamber temperature and humidity are measured and recorded throughout the test. Gas exits and is sampled for the analytes of interest at the other fitting on the lid. Atmospheric pressure at the laboratory is near 1 atm. Prior to use, the chamber and fittings are cleaned by washing them with hot water and a detergent.

A clean, empty chamber is operated at the same testing conditions for at least three hours prior to a test. Chamber background concentrations are measured. Then, the chamber is opened and the material specimen is positioned on a wire rack approximately near the center of the chamber. To initiate a test, the chamber is sealed and ventilated. At specified times, gas samples are collected at the chamber exhaust. The sample flow rates are regulated with electronic mass-flow controllers (calibrated at  $25^\circ \text{C}$  and 1 atm. pressure). Samples for VOCs are collected on multisorbent samplers at a flow rate of 100 standard  $\text{cm}^3 \text{min}^{-1}$ . Aldehyde samples are collected on XPoSure Aldehyde Samplers at a flow rate of 0.5 standard  $\text{L min}^{-1}$ . For this test, a 1-L gas sample for the analysis of TVOC and a 30-L sample for the analysis of formaldehyde were collected 24-h after initiating the test period. The parameters for the emissions test are summarized in Table 1.

**Table 1.** Parameters for a VOC emission test conducted in a small-scale environmental chamber.

Parameter	Value
Chamber volume, $\text{m}^3$	0.067
Coating substrate	Stainless steel
Substrate dimensions, mm	178 x 178
Coated surface area, $\text{m}^2$	0.0316
Loading ratio, $\text{m}^2 \text{m}^{-3}$	0.472
Weight of coating applied, g	1.73
Inlet flow rate, $\text{m}^3 \text{h}^{-1}$	$0.067 \pm 0.003$
Average temp, $^\circ \text{C}$	$28 \pm 1$
Atmosphere	Humidified Air
Average humidity, %RH	$50 \pm 5$
Test duration, h	24

**Data Analysis and Reporting for Emissions Tests**

Steady-state emission rates ( $\mu\text{g h}^{-1}$ ) are calculated for the quantified compounds using the following equation:

$$ER = Q (C - C_o) \tag{1}$$

where Q is the volumetric flow rate ( $\text{m}^3 \text{h}^{-1}$ ) through the chamber; C is the average chamber concentration for the sampling interval ( $\mu\text{g m}^{-3}$ ); and  $C_o$  is the chamber blank or inlet gas concentration ( $\mu\text{g m}^{-3}$ ). An area-specific emission rate or emission factor,  $EF_a$  ( $\mu\text{g m}^{-2} \text{h}^{-1}$ ) is calculated by dividing the emission rate by A, the exposed planar surface area of the product ( $\text{m}^2$ ).

$$EF_a = ER / A \tag{2}$$

**RESULTS**

**Emissions of TVOC and formaldehyde**

The 24-h chamber sample was quantitatively analyzed for TVOC and formaldehyde. The chamber concentration and area-specific emission rate results are presented Table 2. The TVOC chamber blank concentration of  $14.0 \mu\text{g m}^{-3}$  was subtracted from the TVOC chamber concentration of the sample. The blank subtracted chamber TVOC concentration was  $2,960 \mu\text{g m}^{-3}$ . The chamber blank formaldehyde concentration was below the quantitation limit of  $1 \mu\text{g m}^{-3}$ . The coating sample chamber concentration was also below the lower limit of quantitation of  $1 \mu\text{g m}^{-3}$ .

**Table 2.** Chamber concentrations and emission factors of TVOC and formaldehyde measured at 24 hours for a test of 50606-SR60 Safecoat Polyureseal BP Coating.

<b>Compound</b>	<b>Chamber Conc. (<math>\mu\text{g m}^{-3}</math>)</b>	<b>Emission Factor (<math>\mu\text{g m}^{-2} \text{h}^{-1}</math>)</b>
TVOC	2,960	6,270
Formaldehyde	ND	ND