

NARRATIVE REPORT

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Job Code: 209005

Report Date: November 30, 2004
Client: Samuel J. Goldberg, American Formulation & Manufacturing
Product I.D.: 5601Q-K814 Safecoat Water Based Resin Wax
Manufacturer's I.D.: 5601Q
Production Date: November 10, 2004
Product Received: November 15, 2004
Report Prepared By: R.S. Tannous, Laboratory Director

Report Approved By: 

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Date: Nov. 30, 2004

OBJECTIVE

The objective of this test was to measure the emissions of a target list of volatile organic compounds (VOCs) and total volatile organic compounds (TVOC) from a clear coating sample at seven days. 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 clear coating was tested for emissions of TVOC and the individual target compounds, toluene, ethylbenzene, m-/p-xylene isomers, o-xylene, styrene, p-dichlorobenzene and n-tetradecane. The test specimen was prepared by applying 1.43 grams of coating to a 178-mm x 178-mm (0.0316 m²) 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 7 days. Area-specific emission rates (EF_a) were calculated by mass balance. The TVOC EF at 7 days was below the lower limit of quantitation. None of the target VOCs was detected.

METHODS

Description of Product Specimen

On November 15, 2004, the laboratory received a product sample consisting of a container of a clear coating. The sample was identified as: manufacture's ID # 5601Q; product name Safecoat Water Based Resin Wax; sample ID 5601Q-K814; manufacturing date 11/10/04. 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 November 17, 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 m²) 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. 1.43 Grams of coating were applied in a single film. The coated surface area was 0.0316 m². Thus, the product loading was 45 g m⁻². 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 VOCs

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, C₄ to C₁₇ nonpolar and moderately polar volatile organic compounds (VOCs), and many C₁ to C₃ 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 qualitative analysis, the spectra of peaks from the total-ion-current chromatograms are first compared to spectra contained in a database of commonly occurring VOCs. This database was created by BAA from analyses of pure compounds. If a spectrum of a compound matches a spectrum of a compound in the database and if the compounds' retention times also match, then the identification is considered to be "confirmed." If no match is obtained, then the spectrum is compared to spectra contained in the NIST database of approximately 129,000 spectra. Such identifications are considered to be "tentative."

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. This relative response factor (RRF) is 2.03. 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.

For the quantitative analysis of individual VOCs, multi-point internal-standard calibrations are prepared using pure compounds. Standards of the more volatile compounds are prepared by injecting microliter quantities of mixtures of the compounds into static dilution bottles. Gas volumes are withdrawn from the static dilution bottles and transferred to conditioned samplers using gas-tight syringes. Standards of the less volatile compounds are prepared by making serial dilutions in an appropriate low-boiling solvent and injecting microliter quantities of the dilute standards directly onto samplers containing Tenax. Samplers containing standards are analyzed in the same manner as field samples.

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 ± 0.06 standard L min^{-1} is regulated with electronic mass-flow controllers (calibrated at 25°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°C and 1 atm. pressure). Samples for VOCs are collected on multisorbent samplers at a flow rate of 100 standard $\text{cm}^3 \text{min}^{-1}$. For this test, 1.5-L gas samples for the analysis of TVOC and individual target VOCs were collected 7 days (168 hours) 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, m^3	0.067
Coating substrate	Stainless steel
Substrate dimensions, mm	178 x 178
Coated surface area, m^2	0.0316
Loading ratio, $\text{m}^2 \text{m}^{-3}$	0.472
Weight of coating applied, g	1.43
Inlet flow rate, $\text{m}^3 \text{h}^{-1}$	0.067 ± 0.003
Average temp, $^\circ \text{C}$	28 ± 1
Atmosphere	Humidified Air
Average humidity, %RH	50 ± 5
Test duration, days	7

Data Analysis and Reporting for Emission Tests

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

$$ER = Q (C - C_o) \quad (1)$$

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 (m^2).

$$EF_a = ER / A \quad (2)$$

RESULTS**Emissions of TVOC and Individual Target VOCs**

The 7-day chamber sample was quantitatively analyzed for toluene, ethylbenzene, combined m-/p-xylene isomers, o-xylene, styrene, p-dichlorobenzene and n-tetradecane. The chamber concentration and area-specific emission rate results are presented Table 2. None of these target VOCs was detected in the sample. The lower limit of detection for each of these compounds is approximately 1 ng or $1.0 \mu\text{g m}^{-3}$ for a one-liter air sample. The TVOC chamber blank concentration of $7.6 \mu\text{g m}^{-3}$ was subtracted from the TVOC chamber concentration of the sample. The TVOC lower limit of quantitation is approximately $10 \mu\text{g m}^{-3}$.

Table 2. Chamber concentrations and emission factors of TVOC and individual target VOCs measured at 7 days for a test of 5601Q-K814 Safecoat Water Based Resin Wax.

Compound	Chamber Conc. ($\mu\text{g m}^{-3}$)	Emission Factor ($\mu\text{g m}^{-2} \text{h}^{-1}$)
TVOC	<10	<21
Toluene	ND*	ND
Ethylbenzene	ND	ND
m-/p-Xylene	ND	ND
o-Xylene	ND	ND
Styrene	ND	ND
p-Dichlorobenzene	ND	ND
n-Tetradecane	ND	ND

*ND = Indicates value is below detection. The ND for the individual compounds tested is $1.0 \mu\text{g m}^{-3}$, based on a 1 ng limit for a 1-liter sample. This is equivalent to a $2.1 \mu\text{g m}^{-2} \text{h}^{-1}$ emission factor limit of detection.