



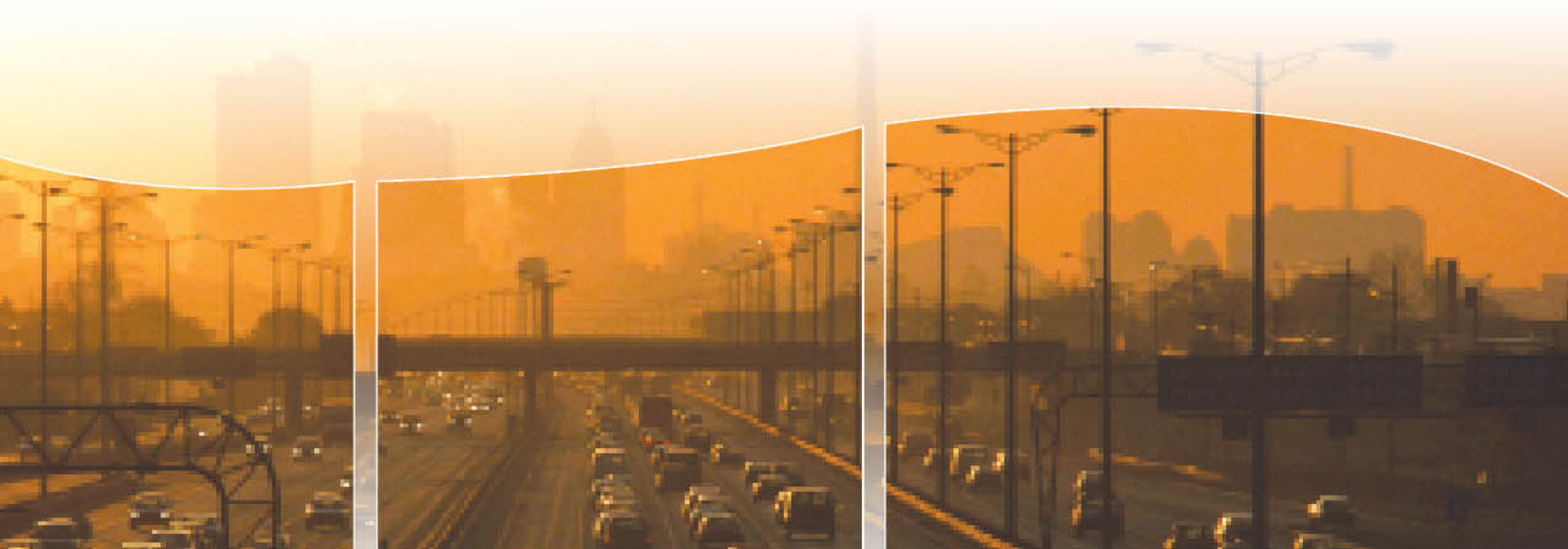
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## Guidance Document

# *Analytical Methods for Determining Volatile Organic Compound Concentration and Other Parameters for the VOC Regulations*



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**Guidance Document**  
***Analytical Methods for Determining Volatile Organic Compound Concentration and  
Other Parameters for the VOC Regulations***

Following stakeholder consultations regarding volatile organic compound (VOC) regulations, this guidance document was developed to inform the regulated community of the analytical methods that will be used by Environment Canada to verify regulatory compliance.

Specifically, this guidance document comments on the analytical methods for determination of VOC concentration and other parameters for the following VOC regulations:

- A. *Volatile Organic Compound (VOC) Concentration Limits for Automotive Refinishing Products Regulations*,<sup>1</sup> S.O.R./2009-197.
- B. *Volatile Organic Compound (VOC) Concentration Limits for Architectural Coatings Regulations*,<sup>2</sup> S.O.R./2009-264.

Mention of trade names or commercial products in this document does not constitute endorsement by Environment Canada. Although care has been taken to ensure that this document reflects the requirements of the *Canadian Environmental Protection Act, 1999* (CEPA 1999), the *Volatile Organic Compound (VOC) Concentration Limits for Automotive Refinishing Products Regulations* and the *Volatile Organic Compound (VOC) Concentration Limits for Architectural Coatings Regulations*, the Act and these regulations prevail over the text of this document in case of any discrepancies or inconsistencies. This document does not supersede or modify the Act or these regulations. It is ultimately the responsibility of regulatees to be familiar with the full text and application of these regulations.

All the methods described herein were developed, validated and approved by recognized organizations such as the American Society for Testing and Materials (ASTM), the United States Environmental Protection Agency (U.S. EPA), the California Air Resource Board (CARB), the Bay Area Air Quality Management District (BAAQMD), the South Coast Air Quality Management District (SCAQMD), the National Institute for Occupational Safety and Health (NIOSH) and others. All of these methods are recognized by Environment Canada as reference methods; however, some of these methods unavoidably refer to techniques and instrumentation that with time have been replaced with modern equivalents. With this in mind, any reference to a specific methodology described in this text should be seen as guidance only. Industry and testing laboratories may choose to use other functionally equivalent methods that have been properly tested and validated and that produce equivalent or more accurate results. However, any laboratory that performs an analysis for the purposes of these regulations

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<sup>1</sup> Available at: [www.ec.gc.ca/lcpe-cepa/eng/regulations/detailReg.cfm?intReg=118](http://www.ec.gc.ca/lcpe-cepa/eng/regulations/detailReg.cfm?intReg=118)

<sup>2</sup> Available at: [www.ec.gc.ca/lcpe-cepa/eng/regulations/detailReg.cfm?intReg=117](http://www.ec.gc.ca/lcpe-cepa/eng/regulations/detailReg.cfm?intReg=117)

shall be accredited under the International Organization for Standardization standard ISO/IEC 17025: 2005, and its accreditation shall include the analysis of the applicable parameter within its scope of testing.

The *Volatile Organic Compound (VOC) Concentration Limits for Architectural Coatings Regulations* refers to test methods pertaining to drying times and surface chalkiness. These methods will be used to determine compliance and are not discussed further within this guidance document.

## **ANALYTICAL METHODS**

The analytical method commonly referred to in the United States for the determination of VOCs in consumer and commercial products is CARB Method 310 [1]. The method applies to the determination of the percent by weight of

- VOCs in a wide range of consumer products, including antiperspirant and deodorant products as well as different aerosol products
- VOCs in products with low vapour pressure (LVP-VOCs)

Components of a product that do not meet the definition of a VOC or are exempt from the regulations are subtracted from the total volatile material to determine the final VOC content of a product.

CARB Method 310 incorporates approximately 22 independent analytical reference methods developed by the ASTM, the NIOSH and the U.S. EPA. Some of these methods, while still technically part of CARB Method 310, are no longer practical and need to be either replaced by more modern, validated standards, or brought in line with modern technology by changes in instrumentation. As long as these methods follow the original ASTM protocol, they may be recognized and accepted as “ASTM-based” methods.

### **A. AUTOMOTIVE REFINISHING PRODUCTS**

The *Volatile Organic Compound (VOC) Concentration Limits for Automotive Refinishing Products Regulations* establish national VOC concentration limits for 14 categories of automotive refinishing products. These regulations prohibit the manufacture, import, sale and offer for sale in Canada of products containing VOCs in concentrations above the prescribed limit. Automotive refinishing products are applied to motor vehicles and mobile equipment to service, maintain, repair, restore or modify their surface. These include pre-treatment wash primers, primers, sealers, colour and clear coatings, truck bed liner coatings, and surface cleaners. The manufacture and import prohibitions of products that exceed the VOC concentration limits came into force on June 19, 2010, while the sale and offer for sale prohibitions came into force on December 19, 2010.

VOC exclusions:

Any compound excluded from the VOC definition in Schedule 1 of CEPA 1999 [4] is exempted for the purpose of determining the VOC concentration of the product. Tertiary-

butyl acetate (TBAC) or acetic acid, 1,1-dimethylethyl, was also listed as an “excluded compound” for the purpose of the Regulations. However, it should be noted that TBAC is on Schedule 1 of CEPA 1999.

### **Determination of VOC concentration**

In accordance with the Regulations, the VOC concentration in a coating and a surface cleaner are determined by the following equations:

– VOC concentration in an automotive refinishing coating:

$$\text{VOC concentration (g/L)} = (\text{Ws} - \text{Ww} - \text{Wec}) / (\text{Vm} - \text{Vw} - \text{Vec})$$

– VOC concentration in an automotive refinishing surface cleaner:

$$\text{VOC concentration (g/L)} = (\text{Ws} - \text{Ww} - \text{Wec}) / \text{Vm}$$

Where:

VOC concentration = concentration of VOCs, in grams per litre of coating or surface cleaner

Ws = weight of volatiles, in grams

Ww = weight of water, in grams

Wec = weight of excluded compounds, in grams

Vm = volume of coating or surface cleaner, in litres

Vw = volume of water, in litres

Vec = volume of excluded compounds, in litres

## **ANALYTICAL METHODS OVERVIEW**

### *Determination of the concentration of VOCs in automotive refinishing coatings*

CARB Method 310 refers to EPA Method 24 [5] for the procedure to determine the concentration of VOCs in automotive refinishing coatings. The latter will be used to verify compliance with the VOC concentration limits for automotive refinishing coatings. EPA Method 24 describes procedures for sample preparation and treatment divided between several steps, while referring to different ASTM methods in due process. The method gives best results when used to determine the total volatile portion of traditional solvent-borne coatings, especially when combined with direct analysis of the exempt solvents. Some exempt solvents are not addressed by Method 24, but appropriate ASTM testing methods exist and can be used; for example, acetone can be analyzed using ASTM methods D6133-02 or D6438-05.

The main steps to follow in order to determine the concentration of VOCs in automotive refinishing coatings are:

### **Step 1 – Determination of volatile content by weight difference**

EPA Method 24 describes the procedure for determining the volatile content of solvent-borne and water-borne coatings by weight difference while referring to ASTM D2369-07 for air drying paints and to ASTM D5403-93 (2007) for radiation curable coatings. The designated quantity of coating (typically 0.3 to 1.0 g) is dispensed with a syringe into a preconditioned aluminum foil (Al-) dish. Preconditioning is achieved by heating the Al-dishes for 30 min at  $110 \pm 5^\circ\text{C}$ , after which they are transferred and stored in a desiccator prior to use (see ASTM D2369-07, section 5.2 for details). The sample is dispersed and heated at  $110 \pm 5^\circ\text{C}$  for 60 min. If the sample in the dish does not form a thin film, a small volume of an appropriate solvent, typically water, acetone or methanol, is used to facilitate its dispersion before heating. If the sample appears to be reacting with the aluminum dish, a Teflon dish should be used instead. The percent of volatile compounds is calculated from the weight loss displayed. A desiccator is used for cooling samples to ambient temperature before weighing and for vial conditioning to avoid absorption of water vapour from air.

ASTM D5403-93 (2007) is the standard test method for determining volatile content of coatings, inks and adhesives designed to be cured by exposure to ultraviolet light or to an accelerated electron beam. The amount of volatiles in the cured coating is established by weight difference. Except for the use of special equipment required to cure the paint, the procedures prescribed in this method are identical to those described in Method 24.

### **Step 2 – Determination of water content**

For the determination of the water content in water-borne coatings, EPA Method 24 calls for using either ASTM D3792-05 or ASTM D4017-02 (2008) method. In practice both methods are used independently and their results are later averaged.

ASTM D3792-05 is the standard test method for the determination of water content by direct injection gas chromatography. A small amount (0.6 g) of water-borne paint is spiked with the same amount of an internal standard (either anhydrous 2-propanol or anhydrous methanol) and diluted with 7 g of anhydrous dimethylformamide (DMF). After shaking for 15 minutes, a small aliquot (1-2  $\mu\text{L}$ ) of the mixture is injected into a gas chromatograph equipped with a thermal conductivity detector (TCD) and a 4 to 6 ft long column, 1/8" outside diameter (O.D.) stainless steel, lined with TFE-fluorocarbon coating and packed with 60–80 mesh (180 to 250  $\mu\text{m}$ ) porous polymer packing material (HayeSep R, C or similar). Such columns are available as a special order from several suppliers.

The ASTM D3792-05 method gives the best results for products with water content between 15–75%. For samples with either a very low or a very high water concentration, the Karl Fisher (KF) method is typically more accurate. The ASTM D3792-05 method is often applied with some modifications to solvent type and/or column packing material. Such modifications are acceptable, assuming that the modified method was properly validated before use. Certain components of aerosol propellants are known to interfere with the analysis, and if such interference occurs KF titration would yield lower water concentration than that obtained from method D3792. Screening of the composition of

the propellant by gas chromatography – mass spectrometry method prior to the analysis is recommended, as knowledge of the sample composition would help in determining the method best suited for a given product matrix.

ASTM D4017-02 is the standard test method for the determination of water in paints by KF titration. The method requires the use of an automated KF titrator, KF reagents and a KF sample oven, all available from reputable suppliers. The method is applicable over a wide range of water content and is preferred for analyzing samples with very low water concentrations. Typically a small sample of product is added to a few mL of anhydrous methanol and mixed in the KF cell. The sample is then titrated with the appropriate reagent, and the titration end point is established from a drop in the electrical current between the poles of the platinum electrode. However, if the products are insoluble in methanol, they have a tendency to form balls, which contaminate the electrode, thus interfering with the analysis. Latex polymers in modern coatings are prone to cause this effect. In such situations, the use of a KF drying oven in connection with the KF titrator is necessary. Using this procedure, a sample sealed in a preparation vial is heated in the oven before moisture is removed from the vial headspace with a stream of dry inert gas. After passing through the vial, the gas is directed to the KF titrator where water vapour is retained and its amount is calculated. This technique avoids the interference between the sample matrix, KF reagent and electrode, and is preferable over the direct injection of the sample to the KF titrator.

### **Step 3 – Determination of excluded compounds**

For determining the weight fraction of compounds specifically excluded by CEPA 1999 or the Regulations, the EPA Method 24 refers to the ASTM D4457-02 (2008) method.

ASTM D4457 is the standard method for the determination of dichloromethane (DCM) and 1,1,1-trichloroethane (TCE) in paints and coatings by direct injection in a gas chromatograph (GC). The method was last reviewed and reapproved for use in 2008. The method has been evaluated for cellulose nitrate, alkyd, vinyl and styrene-butadiene systems. It has not yet been evaluated for other formulations, but it is believed to be applicable. The established working range of this test method is 32–78% for DCM and 31–65% for TCE; however, there is no reason to believe that the method will not work above and below these ranges. 1-propanol is used in the method as an internal standard; if the paint contains this compound, then a different alcohol should be used. Sample preparation is simple with minimal steps: 5 g of the paint together with 16 g of dimethylformamide are added to a vial and spiked with 2 g of internal standard. The vial is then sealed with a crimp-on or septum seal, shaken and centrifuged for 5 minutes at 1000 rpm to facilitate settling. A 1 µL specimen of the supernatant from the solution is then injected in the GC equipped with two packed columns and a TCD. The first column is a 40" × 1/8" (100 cm × 3.2 mm) O.D. stainless steel column packed with glass wool to trap any non-volatile material and minimize sludge build-up in the second column. The second column is a 4 ft × 1/8" (122 cm × 3.2 mm) O.D. stainless steel column packed with 80–100 mesh (150–180 µm) porous polymer packing material. The amount of analytes in the coating is calculated from the detector response compared to standards.



A more recent method relies on gas chromatography with either flame ionization (FID) or mass selective detection (MSD). The suite of volatile exempt solvents including DCM and TCE are separated on a standard DB-624, 30 m × 0.32 mm internal diameter (I.D.) capillary column (1.8 µm film thickness). The product samples are prepared as 1:10 weight/volume dilutions in an organic solvent, most commonly 1-methoxy-2-propanol (MPA). After mixing, the solution is filtered if necessary to remove any insoluble material, and the diluted samples are then analyzed by GC-FID or GC-MSD. The data are reported as weight fraction of analyte in the product. This method can analyze relatively large suites of VOCs including ethanol, acetone and volatile methyl siloxanes (VMS) in addition to DCM and TCE, and TBAC.

#### *Determination of the concentration of VOCs in surface cleaners*

The concentration of VOCs in surface cleaners is determined by EPA Method 24 as described above.

#### *Determination of the acid concentration and the weight of solids of a pre-treatment wash primer*

In the schedule of the Regulations, a pre-treatment wash primer is defined as a coating that contains a minimum of 0.5% acid by weight and not more than 16% solids by weight that is formulated to be applied directly to a bare metal surface to provide corrosion resistance and to facilitate adhesion of subsequent coatings. The determination of the acidity content and the solid by weight are required to evaluate whether the coating falls in this category.

Determination of the acid content in a pre-treatment wash primer may be based on the ASTM D1613-06 technique. This test method has been developed for samples with an acid content below 0.05%, and a slight modification to the procedure may be necessary to accommodate higher acid concentrations in these products. Reducing the pre-treatment wash primer sample volume and increasing the titrant concentration (sodium hydroxide, NaOH) allow to increase the acidity limit of the method. The mass of the acid neutralized in the sample is calculated from the acid molecular weight and stoichiometry of its reaction with NaOH.

The determination of the weight of solids present in the sample follows the guideline set by EPA Method 24.

## **B. ARCHITECTURAL COATINGS**

*The Volatile Organic Compound (VOC) Concentration Limits for Architectural Coatings Regulations* establish national VOC concentration limits for 53 categories of architectural coatings. These regulations prohibit the manufacture, import, sale and offer for sale in Canada of products containing VOCs in concentrations above the prescribed limit. The Regulations apply with some exceptions to general architectural, high-performance

industrial maintenance and traffic marking coatings. These include paints, stains, lacquers and other coatings.

Architectural coatings include paints, stains, varnishes, primers, sealing products, etc., which are intended for field applications in residential, commercial, institutional and industrial settings. The sector is subdivided in three broad sub-sectors: general architectural coatings (consumer trade), traffic marking coatings and industrial maintenance coatings. General architectural coatings include those applied to interior and exterior surfaces of buildings, furniture, fences, concrete, asphalt and other surfaces by either consumers or professionals. Traffic marking coatings are used on roads, normally by institutional and private applicators. Industrial maintenance coatings are used to protect surfaces subject to harsh conditions not found in residential settings; they are typically not available to consumers and are most often applied for maintenance operations.

VOC emissions from architectural coatings result from the use of solvent-based or water-based coatings. Due to the field application of these coatings, it is not feasible to control (capture) VOC emission at the point of use. To protect the environment and the health of Canadians from the effects of air pollution, the Regulations set the VOC concentration limit for 53 categories of architectural coatings [6]. The expectation is that industry will reduce VOC emissions from architectural coatings by reformulating their products to contain lower levels of VOCs.

VOC exclusions:

Any compound excluded from the VOC definition in Schedule 1 of CEPA 1999 [4] is exempted for the purpose of determining the VOC concentration of the product. TBAC was also listed as an “excluded compound” within the Regulations. However, it should be noted that TBAC is still on Schedule 1 of CEPA 1999.

## DETERMINATION OF VOC CONCENTRATION

In accordance with the Regulations, the VOC concentration in an architectural coating and a low solids coating are determined by the following equations:

– VOC concentration in an architectural coating:

$$\text{VOC concentration (g/L)} = (W_s - W_w - W_{ec}) / (V_m - V_w - V_{ec})$$

– VOC concentration in a low solids coating:

$$\text{VOC concentration (g/L)} = (W_s - W_w - W_{ec}) / V_m$$

Where:

VOC concentration = concentration of VOCs, in grams per litre of architectural coating or low solids coating

Ws = weight of volatiles, in grams  
Ww = weight of water, in grams  
Wec = weight of excluded compounds, in grams  
Vm = volume of architectural coating or low solids coating, in litres  
Vw = volume of water, in litres  
Vec = volume of excluded compounds, in litres

## ANALYTICAL METHODS OVERVIEW

### *Determination of the concentration of VOCs in architectural coatings*

EPA Method 24 [5] is often used for the determination of the volatile matter and water contents in architectural coatings. The method describes the procedure for determining the volatile content of solvent-borne and water-borne coatings by weight difference while referring to ASTM D2369-07 for air drying paints and to ASTM D5403-93 (2007) for radiation curable coatings. The designated quantity of coating (typically 0.3 to 1.0 g) is dispensed with a syringe into an Al-dish. Preconditioning is achieved by heating the Al-dishes for 30 min at  $110 \pm 5^\circ\text{C}$ , after which they are transferred and stored in a desiccator prior to use (see ASTM D2369-07, section 5.2 for details). The sample is dispersed and heated at  $110 \pm 5^\circ\text{C}$  for 60 min. If the sample in the dish does not form a thin film, a small volume of an appropriate solvent, typically water, acetone or methanol, is used to facilitate its dispersion before heating. If the sample appears to be reacting with the aluminum dish, a Teflon dish should be used instead. The percent of volatile compounds is calculated from the weight loss displayed. A desiccator is used for cooling samples to ambient temperature before weighing and for vial conditioning to avoid absorption of water vapour from air.

It was found that Method 24 is not as precise when applied to low-VOC water-borne architectural coatings as it is for solvent-borne coatings [7]. For these coatings ASTM D6886-03 is better suited. ASTM D6886-03 has been specifically developed for coatings having less than 5% VOC by weight, and this method is recommended whenever Method 24 produces inconsistent or otherwise doubtful results. Contrary to Method 24, which relies on bulk measurements of VOC content by weight loss, method D6886 analyzes each VOC separately and then adds them together to obtain the total value. According to the D6886 procedure, a paint sample is diluted with tetrahydrofuran (THF) prior to injection in a GC. Several modifications to this methodology exist [8], and water was reported as a good alternative solvent to THF. The use of the static headspace sampling technique was tested as a possible replacement for direct sample injection, with promising results [8].

VOC determination in multi-component traffic marking coatings [9] is described in Appendix A, subpart D, 40 CFR 59. The method is applicable specifically to multi-component methacrylate paints used for traffic marking. The paint is cured as described in EPA Method 24 and cooled in a desiccator to ambient temperature. The film formed at the surface of the paint is then broken and the paint is cured in the oven for a second time.

The sum of the weight loss during both curing steps is used to calculate the volatile content of the paint.

In addition to method ASTM D4457 referenced in Method 24, the determination of excluded compounds in architectural coatings can be achieved using the BAAQMD and/or the SCAQMD reference methods:

- BAAQMD Method 41 for the determination of parachlorobenzotrifluoride [10]
- BAAQMD Method 43 for the determination of methylated siloxanes [11]
- SCAQMD Method 303-91 for the determination of excluded compounds [12]

BAAQMD Method 41 [10] applies to the determination of VOCs in solvent-based coatings and related materials containing parachlorobenzotrifluoride. The method describes the procedures for the determination of the volatile content of coatings, which are essentially the same as described in EPA Method 24. In addition, the method describes the determination of parachlorobenzotrifluoride (PCBTF) in these coatings by GC-FID, using ethanol as an internal standard. Chromatographic separation is achieved using a 12 feet  $\times$  1/8" O.D. stainless steel column packed with one of these specified adsorbents (SP 2100, Carbowax 1500 or Supelcoport), or alternatively the DB-WAX capillary column, 60 m  $\times$  0.32 mm I.D., 0.5  $\mu$ m film thickness. The use of the DB-WAX capillary column is recommended over a packed column for GC separation.

BAAQMD Method 43 [11] applies to the determination of VMS in solvent-based coatings. The method describes the determination of five VMS excluded from the VOC definition in the U.S. and Canada: hexamethyldisiloxane, octamethyltrisiloxane, decamethyltetrasiloxane, octamethylcyclotetrasiloxane and decamethylcyclopentasiloxane. The concentration of these compounds is determined by GC-FID using *n*-octane as an internal standard. Separation is achieved on a 60 m  $\times$  0.32 mm I.D., 1  $\mu$ m film thickness DB-1 capillary column. The sample is dissolved in carbon disulfide (CS<sub>2</sub>) and spiked with the internal standard prior to injection in the GC. An alternative GC-FID method that uses the DB-624 column (30 m  $\times$  0.32 mm I.D.  $\times$  1.8  $\mu$ m film thickness) and MPA as a solvent is also applicable. The latter method is preferable whenever toxicity and flammability of the solvent are of concern.

SCAQMD Method 303-91 [12] applies to the determination of two excluded compounds: DCM and TCE within the volatile portion of coatings. Coatings are first distilled by following SCAQMD Method 302-91 (Distillation of solvents from paints, coatings and inks). The distillate is then diluted with a mixture of perchloroethylene and isooctane. The presence of DCM and/or TCE is first screened by GC-MS and, if confirmed, the quantification is done using GC-TCD. The chromatographic separation is achieved using a 20 ft  $\times$  1/8" O.D. stainless steel column packed with 10% SP1000 on 80–100 mesh Supelcoport.

SCAQMD Method 302-91 [13] describes the vacuum distillation of solvents from paints, coatings and inks in order to collect the volatile fraction for further chemical analysis. A sample is heated in a silicone oil bath under vacuum (2 Torr) until the bath temperature

reaches 200°C or the sample solidifies. A cold liquid nitrogen trap is used for collecting the distillate.

*Determination of the acid concentration of a pre-treatment wash primer*

Acid concentration in a pre-treatment wash primer can be analyzed based on the ASTM Method D1613-06. This test method has been developed for samples with an acid content below 0.05%, and a slight modification to the procedure may be necessary to accommodate higher acid concentrations in some products. Reducing the sample volume and increasing the titrant concentration (sodium hydroxide, NaOH) allow to increase the acidity limit of the method. The mass of the acid neutralized in the sample is calculated from the acid molecular weight and stoichiometry of its reaction with NaOH.

*Determination of the flame spread index of a fire retardant coating*

ASTM E84-07 method can be used for determination of the flame spread index of a fire retardant coating. This is a historical standard that was recently replaced by a newer E84-10b version. The standard is used to measure the response of tested materials to heat and flame under controlled test conditions. The standardized specimen sample is heated in a combustion chamber with a flame from a gas torch. Flame spread and temperature in a chamber are measured over the specified distance and time intervals. A photometer employed at the end of the combustion chamber is used to measure change in attenuation of incident light due to a passing smoke, particulate and other effluent present in exhaust air. The flame spread distance, temperature of the chamber and changes in photoelectric cell readings are used to calculate flame spread and smoke-developed indexes.

*Determination of the fire resistance of a fire resistant coating*

ASTM E119-07a method can be used for determination of the fire resistant rating of a fire resistive coating. Paragraphs 45 through 49 of this method refer to performance of protecting membranes in wall, partition and roof assemblies. This standard has been currently replaced by its new version with designation E119-10b. Several laboratories are accredited to do the testing according to E119 method and can be contacted to obtain more specific information.

*Determination of the gloss of a calcimine recoater, a quick-dry enamel, any flat coating other than one set out in items 1 to 50, any non-flat coating other than one set out in items 1 to 50 and any high-gloss coating other than one set out in items 1 to 50*

The gloss of certain architectural coatings set out in the Regulations can be determined in accordance with ASTM D523-89 (Reapproved 1999) method. This standard has been currently superseded by D523-08. Measured gloss ratings by these test methods are obtained by comparing the specular (mirror) reflectance from the specimen to that from a black glass standard.

#### *Determination of the metallic concentration of a metallic pigmented coating*

SCAQMD Method 318-95 can be used for metallic concentration of metallic pigment coating [14]. As crystalline particles exhibit unique X-ray diffraction patterns characteristic of their crystalline structure, a dried sample of specimen is ground into a homogeneous powder and analyzed by X-ray diffraction. The concentration of elemental metal particles is determined by comparing the integrated intensities of the sample with those of known standard. The method has been validated for the determination of percent weight of elemental aluminum in coatings, but may also be applicable for the determination of other elemental metals or crystalline materials for which appropriate standards are available. The addition of silicon as an internal standard is used to normalize the measured intensity of the analyte and to minimize effects of packing, and partially compensate for density variations.

#### *Determination of the radiation resistance of a nuclear coating*

The radiation resistance of a nuclear coating should be determined in accordance with ASTM D4082-02 method. This standard has been currently replaced by ASTM with its newer version D4082-10. The method covers a standard procedure for evaluating the lifetime radiation tolerance of coatings to be used in nuclear power plants. It is designed to provide a uniform test to assess the suitability of coatings, used in nuclear power facilities, under radiation exposure for the life of these facilities. The gamma energy field at the position of the test specimen of  $1 \times 10^6$  rads/h is recommended unless specified otherwise by the licence. Total irradiation accumulated dose of  $1 \times 10^9$  rads is recommended for most of the tests.

#### *Determination of the chemical resistance of a nuclear coating*

The chemical resistance of a nuclear coating should be determined in accordance with ASTM D3912-95 (R2001) method. This standard has been currently replaced by ASTM with its newer version D3912-10. The method is intended to be used as a screening test to evaluate coatings on steel and concrete substrates. It addresses coatings intended to be exposed to short-term/occasional chemical exposure such as chemical splash or spill and coatings intended for near-continuous chemical immersion. Tests are performed at room temperature and evaluate changes in physical properties such as delamination, blistering, cracking, softening or discoloration. The tests are qualitative in nature; often ratings such as “slight, moderate or extreme” are used to report the severity of the effects for which no ASTM reference exists.

## REFERENCES:

- [1] CARB Method 310 – Determination of Volatile Organic Compounds (VOC) in Consumer Products and Reactive Organic Compounds in Aerosol Coating Products, California Environmental Protection Agency, Air Resources Board, adopted September 25, 1997, amended May 5, 2005.
- [2] *Regulation for Reducing Ozone from Aerosol Coating Product Emissions*, Title 17, CCR, sections 94520-94528.
- [3] *VOC Concentration Limits for Automotive Refinishing Products Regulations*, Regulatory Impact Analysis Statement, *Canada Gazette* Part II, Vol. 143, No. 14, p. 1348-1399.
- [4] *Canadian Environmental Protection Act, 1999*, Amendment to Schedule 1, SOR/DORS/2003-229, *Canada Gazette* Part II, Vol. 142, No. 14, p. 1864-1865.
- [5] EPA Method 24 – Determination of Volatile Matter Content, Water Content, Density, Volume Solids and Weight Solids of Surface Coatings, Appendix A-7, Part 60, Chapter 1 of Title 40 of the Code of Federal Regulations of the United States.
- [6] *VOC Concentration Limits for Architectural Coatings Regulations*, *Canada Gazette* Part II, Vol. 143, No. 20, p. 1855-1915.
- [7] Brickweg *et al.*, Comprehensive VOC analysis method for architectural coatings, JCT Coatings Tech, *Technology Today*, Jan. 2007.
- [8] Dane R. Jones and Max T. Wills, Development of an improved VOC analysis method for architectural coating, Quarterly Progress Report for California Air Resources Board and California Environmental Protection Agency, February 28, 2006.
- [9] 40 CFR 59, subpart D, appendix A, Determination of Volatile Matter Content of Methacrylate Multicomponent Coatings Used as Traffic Marking Coatings, 64 Fed. Reg. 35002 (June 30, 1999).
- [10] Bay Area Air Quality Management District (BAAQMD) Method 41, Determination of Volatile Organic Compounds in Solvent-Based Coatings and Related Materials Containing Parachlorobenzotrifluoride, proposed amendment February 4, 2004.
- [11] Bay Area Air Quality Management District (BAAQMD) Method 43, Determination of Volatile Methylsiloxanes in Solvent-Based Coatings, Inks, and Related Materials, proposed amendment February 4, 2004.
- [12] South Coast Air Quality Management District (SCAQMD) Method 303-91, Determination of Exempt Compounds, revised February 1993.
- [13] South Coast Air Quality Management District (SCAQMD) Method 302-91, Distillation of Solvents from Paints, Coatings and Inks, approved June 1, 1991, revised February 1993.
- [14] South Coast Air Quality Management District (SCAQMD) Method 318-95, Determination of Weight Percent Elemental Metal in Coatings by X-Ray Diffraction, approved July 1996.

## ASTM methods\*

D523-89	(current standard D523-08) Standard Test Method for Specular Gloss
D1613-06	Standard Test Method for Acidity in Volatile Solvents and Chemical Intermediates Used in Paint, Varnish, Lacquer, and Related Products
D2369-07	Standard Test Method for Volatile Content of Coatings
D3792-05	Standard Test Method for Water Content of Coatings by Direct Injection into a Gas Chromatograph
D3912-95	Standard Test Method for Chemical Resistance of Coatings Used in Light-Water Nuclear Power Plants
D3912-10	Standard Test Method for Chemical Resistance of Coatings and Linings for Use in Nuclear Power Plants
D4017-02	(2008) Standard Test Method for Water in Paints and Paint Materials by Karl Fischer Method
D4082-02	Standard Test Method for Effects of Gamma Radiation on Coatings for Use in Light-Water Nuclear Power Plants
D4082-10	Standard Test for Effects of Gamma Radiation on Coatings for Use in Nuclear Power Plants
D4359-90	(2006) Standard Test Method for Determining Whether a Material Is a Liquid or a Solid
D4457-02	(2008) Standard Test method for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph
D5403-93	(2007) Standard Test Method for Volatile Content of Radiation Curable Materials
D6133-02	(2008) Standard Test Method for Acetone, <i>p</i> -Chlorobenzotrifluoride, Methyl Acetate or <i>t</i> -Butyl Acetate Content of Solventborne and Waterborne Paints, Coatings, Resins, and Raw Material by Direct Injection into a Gas Chromatograph
D6438-05	Standard Test Method for Acetone, Methyl Acetate, and Parachlorobenzotrifluoride Content of Paints, and Coatings by Solid Phase Microextraction-Gas Chromatography
D6886-03	Standard Test Method for Speciation of Volatile Organic Compounds in Low VOC Content Waterborne Air-Dry Coatings by Gas Chromatography
E84-07	(current standard E84-10b) Standard Test Method for Surface Burning Characteristics of Building Material
E119-07	(current standard E119-10b) Standard Test Methods for Fire Tests of Building Construction and Materials

\* The methods are issued under the fixed designation with a capital letter followed by a number. The second number immediately following the designation indicates the year of original adoption or, in the case of a revision, the year of last revision. A number in parentheses indicates the year of last reapproval. As a rule the most current method revision should be used unless specified otherwise.



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