

4.3 Cross-contamination may occur whenever high-level and low-level samples are analyzed sequentially. The order of sample analyses specified in Section 11.7 is designed to minimize this problem.

4.4 Cross-contamination may also occur if the devices used to transfer coating during the sample preparation process or for injecting the sample into the GC are not adequately cleaned between uses. All such devices should be cleaned with acetone or other suitable solvent and checked for plugs or cracks before and after each use.

## 5. Safety

5.1 Many solvents used in coatings are hazardous. Precautions should be taken to avoid unnecessary inhalation and skin or eye contact. This method may involve hazardous materials, operations, and equipment. This test method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations in regards to the performance of this test method.

5.2 Dimethylformamide is harmful if inhaled or absorbed through the skin. The user should obtain relevant health and safety information from the manufacturer. Dimethylformamide should be used only with adequate ventilation. Avoid contact with skin, eyes, and clothing. In case of contact, immediately flush skin or eyes with plenty of water for at least 15 minutes. If eyes are affected, consult a physician. Remove and wash contaminated clothing before reuse.

5.3 User's manuals for the gas chromatograph and other related equipment should be consulted for specific precautions to be taken related to their use.

## 6. Equipment and Supplies

Note: Certified product data sheets (CPDS) may also include information relevant to the analysis of the coating sample including, but not limited to, separation column, oven temperature, carrier gas, injection port temperature, extraction solvent, and internal standard.

### 6.1 Sample Collection.

6.1.1 Sampling Containers. Dual-seal sampling containers, four to eight fluid ounce capacity, should be used to collect the samples. Glass sample bottles or plastic containers with volatile organic compound (VOC) impermeable walls must be used for corrosive substances (e.g., etch primers and certain coating catalysts such as methyl ethyl ketone (MEK) peroxide). Sample containers, caps, and inner seal liners must be inert to the compounds in the sample and must be selected on a case-by-case basis.

6.1.1.1 Other routine sampling supplies needed include waterproof marking pens, tubing, scrapers/spatulas, clean rags, paper towels, cooler/ice, long handle tongs, and mixing/stirring paddles.

6.1.2 Personal safety equipment needed includes eye protection, respiratory protection, a hard hat, gloves, steel toe shoes, etc.

6.1.3 Shipping supplies needed include shipping boxes, packing material, shipping labels, strapping tape, etc.

6.1.4 Data recording forms and labels needed include coating data sheets and sample can labels.

Note: The actual requirements will depend upon the conditions existing at the source sampled.

### 6.2 Laboratory Equipment and Supplies.

6.2.1 Gas Chromatograph (GC). Any instrument equipped with a flame ionization detector and capable of being temperature programmed may be used. Optionally, other types of detectors (e.g., a mass spectrometer), and any necessary interfaces, may be used provided that the detector system yields an appropriate and reproducible response to the analytes in the injected sample. Autosampler injection may be used, if available.

6.2.2 Recorder. If available, an electronic data station or integrator may be used to record the gas chromatogram and associated data. If a strip chart recorder is used, it must meet the following criteria: A 1 to 10 millivolt (mV) linear response with a full scale response time of 2 seconds or less and a maximum noise level of  $\pm 0.03$  percent of full scale. Other types of recorders may be used as appropriate to the specific detector installed provided that the recorder has a full scale response time of 2 seconds or less and a maximum noise level of  $\pm 0.03$  percent of full scale.

6.2.3 Column. The column must be constructed of materials that do not react with components of the sample (e.g., fused silica, stainless steel, glass). The column should be of appropriate physical dimensions (e.g., length, internal diameter) and contain sufficient suitable stationary phase to allow separation of the analytes. DB-5, DB-Wax, and FFAP columns are commonly used for paint analysis; however, it is the responsibility of each analyst to select appropriate columns and stationary phases.

6.2.4 Tube and Tube Fittings. Supplies to connect the GC and gas cylinders.

6.2.5 Pressure Regulators. Devices used to regulate the pressure between gas cylinders and the GC.

6.2.6 Flow Meter. A device used to determine the carrier gas flow rate through the GC. Either a digital flow meter or a soap film bubble meter may be used to measure gas flow rates.

6.2.7 Septa. Seals on the GC injection port through which liquid or gas samples can be injected using a syringe.

6.2.8 Liquid Charging Devices. Devices used to inject samples into the GC such as clean and graduated 1, 5, and 10 microliter ( $\mu$ l) capacity syringes.

6.2.9 Vials. Containers that can be sealed with a septum in which samples may be prepared or stored. The recommended size is 25 ml capacity. Mininert® valves have been found satisfactory and are available from Pierce Chemical Company, Rockford, Illinois.

6.2.10 Balance. Device used to determine the weights of standards and samples. An analytical balance capable of accurately weighing to 0.0001 g is required.

## 7. Reagents and Standards

7.1 Purity of Reagents. Reagent grade chemicals shall be used in all tests. Unless otherwise specified, all reagents shall

conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of determination.

7.2 Carrier Gas. Helium carrier gas shall have a purity of 99.995 percent or higher. High purity nitrogen may also be used. Other carrier gases that are appropriate for the column system and analyte may also be used. Ultra-high purity grade hydrogen gas and zero-grade air shall be used for the flame ionization detector.

7.3 Dimethylformamide (DMF). Solvent for all standards and samples. Some other suitable solvent may be used if DMF is not compatible with the sample or coelutes with a target analyte.

Note: DMF may coelute with ethylbenzene or p-xylene under the conditions described in the note under Section 6.2.3.

7.4 Internal Standard Materials. The internal standard material is used in the quantitation of the analytes for this method. It shall be gas chromatography spectrophotometric quality or, if this grade is not available, the highest quality available. Obtain the assay for the internal standard material and maintain at that purity during use. The recommended internal standard material is 1-propanol; however, selection of an appropriate internal standard material for the particular coating and GC conditions used is the responsibility of each analyst.

7.5 Reference Standard Materials. The reference standard materials are the chemicals cited in Section 1.1 which are of known identity and purity and which are used to assist in the identification and quantification of the analytes of this method. They shall be the highest quality available. Obtain the assays for the reference standard materials and maintain at those purities during use.

7.6 Stock Reference Standards. Stock reference standards are dilutions of the reference standard materials that may be used on a daily basis to prepare calibration standards, calibration check standards, and quality control check standards. Stock reference standards may be prepared from the reference standard materials or purchased as certified solutions.

7.6.1 Stock reference standards should be prepared in dimethylformamide for each analyte expected in the coating samples to be analyzed. The concentrations of analytes in the stock reference standards are not specified but must be adequate to prepare the calibration standards required in the method. A stock reference standard may contain more than one analyte provided all analytes are chemically compatible and no analytes coelute. The actual concentrations prepared must be known to within 0.1 percent (e.g.,  $0.1000 \pm 0.0001$  g/g solution). The following procedure is suggested. Place about 35 ml of dimethylformamide into a tared ground-glass stoppered 50 ml volumetric flask. Weigh the flask to the nearest 0.1 mg. Add 12.5 g of the reference standard material and reweigh the flask. Dilute to volume with dimethylformamide and reweigh. Stopper the