flask and mix the contents by inverting the flask several times. Calculate the concentration in grams per gram of solution from the net gain in weights, correcting for the assayed purity of the reference standard material.

Note: Although a glass-stoppered volumetric flask is convenient, any suitable glass container may be used because stock reference standards are prepared by weight.

7.6.2 Transfer the stock reference standard solution into one or more Teflonsealed screw-cap bottles. Store, with minimal headspace, at -10°C to 0°C and protect from light.

7.6.3 Prepare fresh stock reference standards every six months, or sooner if analysis results from daily calibration check standards indicate a problem. Fresh stock reference standards for very volatile HAP's may have to be prepared more frequently.

7.7 Calibration Standards. Calibration standards are used to determine the response of the detector to known amounts of reference material. Calibration standards must be prepared at a minimum of three concentration levels from the stock reference standards (see Section 7.6). Prepare the calibration standards in dimethylformamide (see Section 7.3). The lowest concentration standard should contain a concentration of analyte equivalent either to a concentration of no more than 0.01% of the analyte in a coating or to a concentration that is lower than the actual concentration of the analyte in the coating, whichever concentration is higher. The highest concentration standard should contain a concentration of analyte equivalent to slightly more than the highest concentration expected for the analyte in a coating. The remaining calibration standard should contain a concentration of analyte roughly at the midpoint of the range defined by the lowest and highest concentration calibration standards. The concentration range of the standards should thus correspond to the expected range of analyte concentrations in the prepared coating samples (see Section 11.5). Each calibration standard should contain each analyte for detection by this method expected in the actual coating samples (e.g., some or all of the compounds listed in Section 1.1 may be included). Each calibration standard should also contain an appropriate amount of internal standard material (response for the internal standard material is within 25 to 75 percent of full scale on the attenuation setting for the particular reference standard concentration level). Calibration Standards should be stored for 1 week only in sealed vials with minimal headspace. If the stock reference standards were prepared as specified in Section 7.6, the calibration standards may be prepared by either weighing each addition of the stock reference standard or by adding known volumes of the stock reference standard and calculating the mass of the standard reference material added. Alternative 1 (Section 7.7.1) specifies the procedure to be followed when the stock reference standard is added by volume. Alternative 2 (Section 7.7.2) specifies the procedure to be followed when the stock reference standard is added by weight.

Note: To assist with determining the appropriate amount of internal standard to add, as required here and in other sections of this method, the analyst may find it advantageous to prepare a curve showing the area response versus the amount of internal standard injected into the GC.

7.7.1 Preparation Alternative 1. Determine the amount of each stock reference standard and dimethylformamide solvent needed to prepare approximately 25 ml of the specific calibration concentration level desired. To a tared 25 ml vial that can be sealed with a crimp-on or Mininert® valve, add the total amount of dimethylformamide calculated to be needed. As quickly as practical, add the calculated amount of each stock reference standard using new pipets (or pipet tips) for each stock reference standard. Reweigh the vial and seal it. Using the known weights of the standard reference materials per ml in the stock reference standards, the volumes added, and the total weight of all reagents added to the vial, calculate the weight percent of each standard reference material in the calibration standard prepared. Repeat this process for each calibration standard to be prepared.

7.7.2 Preparation Alternative 2. Determine the amount of each stock reference standard and dimethylformamide solvent needed to prepare approximately 25 ml of the specific calibration concentration level desired. To a tared 25 ml vial that can be sealed with a crimp-on or Mininert® valve, add the total amount of dimethylformamide calculated to be needed. As quickly as practical, add the calculated amount of a stock reference standard using a new pipet (or pipet tip) and reweigh the vial. Repeat this process for each stock reference standard to be added. Seal the vial after obtaining the final weight. Using the known weight percents of the standard reference materials in the stock reference standards, the weights of the stock reference standards added, and the total weight of all reagents added to the vial, calculate the weight percent of each standard reference material in the calibration standard prepared. Repeat this process for each calibration standard to be prepared.

8. Sample Collection, Preservation, Transport, and Storage

8.1 Copies of material safety data sheets (MSDS's) for each sample should be obtained prior to sampling. The MSDS's contain information on the ingredients, and physical and chemical properties data. The MSDS's also contain recommendations for proper handling or required safety precautions. 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.

8.2 A copy of the blender's worksheet can be requested to obtain data on the exact coating being sampled. A blank coating data sheet form (see Section 18) may also be used. The manufacturer's formulation information from the product data sheet should also be obtained.

8.3 Prior to sample collection, thoroughly mix the coating to ensure that a

representative, homogeneous sample is obtained. It is preferred that this be accomplished using a coating can shaker or similar device; however, when necessary, this may be accomplished using mechanical agitation or circulation systems.

8.3.1 Water-thinned coatings tend to incorporate or entrain air bubbles if stirred too vigorously; mix these types of coatings slowly and only as long as necessary to homogenize.

8.3.2 Each component of multicomponent coatings that harden when mixed must be sampled separately. The component mix ratios must be obtained at the facility at the time of sampling and submitted to the analytical laboratory.

8.4 Sample Collection. Samples must be collected in a manner that prevents or minimizes loss of volatile components and that does not contaminate the coating reservoir. A suggested procedure is as follows. Select a sample collection container which has a capacity at least 25 percent greater than the container in which the sample is to be transported. Make sure both sample containers are clean and dry. Using clean, long-handled tongs, turn the sample collection container upside down and lower it into the coating reservoir. The mouth of the sample collection container should be at approximately the midpoint of the reservoir (do not take the sample from the top surface). Turn the sample collection container over and slowly bring it to the top of the coating reservoir. Rapidly pour the collected coating into the sample container, filling it completely. It is important to fill the sample container completely to avoid any loss of volatiles due to volatilization into the headspace. Return any unused coating to the reservoir or dispose as appropriate.

Note: If a company requests a set of samples for its own analysis, a separate set of samples, using new sample containers, should be taken at the same time.

- 8.5 Once the sample is collected, place the sample container on a firm surface and insert the inner seal in the container by placing the seal inside the rim of the container, inverting a screw cap, and pressing down on the screw cap which will evenly force the inner seal into the container for a tight fit. Using clean towels or rags, remove all residual coating material from the outside of the sample container after inserting the inner seal. Screw the cap onto the container.
- 8.5.1 Affix a sample label (see Section 18) clearly identifying the sample, date collected, and person collecting the sample.
- 8.5.2 Prepare the sample for transportation to the laboratory. The sample should be maintained at the coating's recommended storage temperature specified on the Material Safety Data Sheet, or, if no temperature is specified, the sample should be maintained within the range of 5°C to 38°C.
- 8.9 The shipping container should adhere to U.S. Department of Transportation specification DOT 12–B. Coating samples are considered hazardous materials; appropriate shipping procedures should be followed.