accomplished by analyzing a calibration standard that is at a concentration near the midpoint of the working range and performing the checks in Sections 10.3.1, 10.3.2, and 10.3.3.

10.3.1 For each analyte in the calibration standard, calculate the percent difference in the RRF from the last calibration using Equation 8, Section 12. If the percent difference for each calibration analyte is less than 10 percent, the last calibration curve is assumed to be valid. If the percent difference for any analyte is greater than 5 percent, the analyst should consider this a warning limit. If the percent difference for any one calibration analyte exceeds 10 percent, corrective action must be taken. If no source of the problem can be determined after corrective action has been taken, a new three-point (minimum) calibration must be generated. This criterion must be met before quantitative analysis begins.

10.3.2 If the RF_{is} for the internal standard changes by more than ±20 percent from the last daily calibration check, the system must be inspected for malfunctions and corrections made as appropriate.

10.3.3 The retention times for the internal standard and all calibration check analytes must be evaluated. If the retention time for the internal standard or for any calibration check analyte changes by more than 0.10 min from the last calibration, the system must be inspected for malfunctions and corrections made as required.

11. Procedure

11.1 All samples and standards must be allowed to warm to room temperature before analysis. Observe the given order of ingredient addition to minimize loss of volatiles.

11.2 Bring the GC system to the determined operating conditions and condition the column as described in Section 10.1.

Note: The temperature of the injection port may be an especially critical parameter. Information about the proper temperature may be found on the CPDS.

11.3 Perform the daily calibration checks as described in Section 10.3. Samples are not to be analyzed until the criteria in Section 10.3 are met.

11.4 Place the as-received coating sample on a paint shaker, or similar device, and shake the sample for a minimum of 5 minutes to achieve homogenization.

11.5 Note: The steps in this section must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory hood free from solvent vapors. All weights must be recorded to the nearest 0.1 mg.

11.5.1 Add 16 g of dimethylformamide to each of two tared vials (A and B) capable of being septum sealed.

11.5.2 To each vial add a weight of coating that will result in the response for the major constituent being in the upper half of the linear range of the calibration curve.

Note: The magnitude of the response obviously depends on the amount of sample injected into the GC as specified in Section 11.8. This volume must be the same as used for preparation of the calibration curve, otherwise shifts in compound retention times may occur. If a sample is prepared that results in a response outside the limits of the calibration curve, new samples must be prepared; changing the volume injected to bring the response within the calibration curve limits is not permitted.

11.5.3 Add a weight of internal standard to each vial (A and B) that will result in the response for the internal standard being between 25 percent and 75 percent of the linear range of the calibration curve.

11.5.4 Seal the vials with crimp-on or Mininert® septum seals.

11.6 Shake the vials containing the prepared coating samples for 60 seconds. Allow the vials to stand undisturbed for ten minutes. If solids have not settled out on the bottom after 10 minutes, then centrifuge at 1,000 rpm for 5 minutes. The analyst also has the option of injecting the sample without allowing the solids to settle.

11.7 Analyses should be conducted in the following order: daily calibration check sample, method blank, up to 10 injections from sample vials (*i.e.*, one injection each from up to five pairs of vials, which corresponds to analysis of 5 coating samples).

11.8 Inject the prescribed volume of supernatant from the calibration check sample, the method blank, and the sample vials onto the chromatographic column and record the chromatograms while operating the system under the specified operating conditions.

Note: The analyst has the option of injecting the unseparated sample.

12. Data Analysis and Calculations

12.1 Qualitative Analysis. An analyte (e.g., those cited in Section 1.1) is considered tentatively identified if two criteria are satisfied: (1) elution of the sample analyte within ±0.05 min of the average GC retention time of the same analyte in the calibration standard; and (2) either (a) confirmation of the identity of the compound by spectral matching on a gas chromatograph equipped with a mass selective detector or (b) elution of the sample analyte within ±0.05 min of the average GC retention time of the same analyte in the calibration standard analyzed on a dissimilar GC column.

12.1.1 The RT of the sample analyte must meet the criteria specified in Section 9.3.3.

12.1.2 When doubt exists as to the identification of a peak or the resolution of two or more components possibly comprising one peak, additional confirmatory techniques (listed in Section 12.1) must be used.

12.2 Quantitative Analysis. When an analyte has been identified, the quantification of that compound will be based on the internal standard technique.

12.2.1 A single analysis consists of one injection from each of two sample vials (A and B) prepared using the same coating. Calculate the concentration of each identified analyte in the sample as follows:

$$HAP_{wt\%} = 100 \times \frac{(A_x)(W_{is})}{(A_{is})(\overline{RRF_x})(W_x)} \qquad \text{Eq. (1)}$$

where:

$HAP_{wt\%}$ = weight percent of the analyte in coating.
A_x = Area response of the analyte in the sample.
W_{is} = Weight of internal standard added to sample, g.
A_{is} = Area response of the internal standard in the sample.
$\overline{RRF_x}$ = Mean relative response factor for the analyte in the calibration standards.
W_x = Weight of coating added to the sample solution, g.

12.2.2 Report results for duplicate analysis (sample vials A and B) without correction.

12.3 Precision Data. Calculate the percent difference between the measured concentrations of each analyte in vials A and B as follows.

12.3.1 Calculate the weight percent of the analyte in each of the two sample vials as described in Section 12.2.1.

12.3.2 Calculate the percent difference for each analyte as:

$$\%Dif_i = 100 \times \frac{|A_i - B_i|}{\frac{(A_i + B_i)}{2}} \qquad \text{Eq. (2)}$$

where A_i and B_i are the measured concentrations of the analyte in vials A and B.