## 9. Quality Control

9.1 Laboratories using this method should operate a formal quality control program. The minimum requirements of the program should consist of an initial demonstration of laboratory capability and an ongoing analysis of blanks and quality control samples to evaluate and document quality data. The laboratory must maintain records to document the quality of the data generated. When results indicate atypical method performance, a quality control check standard (see Section 9.4) must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

9.2 Before processing any samples, the analyst must demonstrate, through analysis of a reagent blank, that there are no interferences from the analytical system, glassware, and reagents that would bias the sample analysis results. Each time a set of analytical samples is processed or there is a change in reagents, a reagent blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement steps.

9.3 Required instrument quality control parameters are found in the following sections:

9.3.1 Baseline stability must be demonstrated to be  $\leq$ 5 percent of full scale using the procedures given in Section 10.1.

9.3.2 The GC calibration is not valid unless the retention time (RT) for each analyte at each concentration is within  $\pm 0.05$ min of the retention time measured for that analyte in the stock standard.

9.3.3 The retention time (RT) of any sample analyte must be within  $\pm 0.05$  min of the average RT of the analyte in the calibration standards for the analyte to be considered tentatively identified.

9.3.4 The GC system must be calibrated as specified in Section 10.2.

9.3.5 A one-point daily calibration check must be performed as specified in Section 10.3.

9.4 To establish the ability to generate results having acceptable accuracy and precision, the analyst must perform the following operations.

9.4.1 Prepare a quality control check standard (QCCS) containing each analyte expected in the coating samples at a concentration expected to result in a response between 25 percent and 75 percent of the limits of the calibration curve when the sample is prepared as described in Section 11.5. The QCCS may be prepared from reference standard materials or purchased as certified solutions. If prepared in the laboratory, the QCCS must be prepared independently from the calibration standards.

9.4.2 Analyze three aliquots of the QCCS according to the method beginning in Section 11.5.3 and calculate the weight percent of each analyte using Equation 1, Section 12.

9.4.3 Calculate the mean weight percent  $(\tilde{X})$  for each analyte from the three results obtained in Section 9.4.2.

9.4.4 Calculate the percent accuracy for each analyte using the known concentrations (Ti) in the QCCS using Equation 3, Section 12.

9.4.5 Calculate the percent relative standard deviation (percent RSD) for each analyte using Equation 7, Section 12, substituting the appropriate values for the relative response factors (RRF's) in said equation.

9.4.6 If the percent accuracy (Section 9.4.4) for all analytes is within the range 90 percent to 110 percent and the percent RSD (Section 9.4.5) for all analytes is  $\leq$ 20 percent, system performance is acceptable and sample analysis may begin. If these criteria are not met for any analyte, then system performance is not acceptable for that analyte and the test must be repeated for those analytes only. Repeated failures indicate a general problem with the measurement system that must be located and corrected. In this case, the entire test, beginning at Section 9.4.1, must be repeated after the problem is corrected.

9.5 Great care must be exercised to maintain the integrity of all standards. It is recommended that all standards be stored at -10 °C to 0 °C in screw-cap amber glass bottles with Teflon liners.

9.6 Unless otherwise specified, all weights are to be recorded within 0.1 mg.

## 10. Calibration and Standardization.

10.1 Column Baseline Drift. Before each calibration and series of determinations and before the daily calibration check, condition the column using procedures developed by the laboratory or as specified by the column supplier. Operate the GC at initial (i.e., before sample injection) conditions on the lowest attenuation to be used during sample analysis. Adjust the recorder pen to zero on the chart and obtain a baseline for at least one minute. Initiate the GC operating cycle that would be used for sample analysis. On the recorder chart, mark the pen position at the end of the simulated sample analysis cycle. Baseline drift is defined as the absolute difference in the pen positions at the beginning and end of the cycle in the direction perpendicular to the chart movement. Calculate the percent baseline drift by dividing the baseline drift by the chart width representing full-scale deflection and multiply the result by 100.

10.2 Calibration of GC. Bring all stock standards and calibration standards to room temperature while establishing the GC at the determined operating conditions.

10.2.1 Retention Times (RT's) for Individual Compounds.

Note: The procedures of this subsection are required only for the initial calibration. However, it is good laboratory practice to follow these procedures for some or all analytes before each calibration. The procedures were written for chromatograms output to a strip chart recorder. More modern instruments (e.g., integrators and electronic data stations) determine and print out or display retention times automatically.

The RT for each analyte should be determined before calibration. This provides a positive identification for each peak observed from the calibration standards. Inject an appropriate volume (see Note in Section 11.5.2) of one of the stock reference standards into the gas chromatograph and record on the chart the pen position at the time of the injection (see Section 7.6.1). Dilute an aliquot of the stock reference standard as required in dimethylformamide to achieve a concentration that will result in an on-scale response. Operate the gas chromatograph according to the determined procedures. Select the peak(s) that correspond to the analyte(s) [and internal standard, if used] and measure the retention time(s). If a chart recorder is used, measure the distance(s) on the chart from the injection point to the peak maxima. These distances, divided by the chart speed, are defined as the RT's of the analytes in question. Repeat this process for each of the stock reference standard solutions.

Note: If gas chromatography with mass spectrometer detection (GC–MS) is used, a stock reference standard may contain a group of analytes, provided all analytes are adequately separated during the analysis. Mass spectral library matching can be used to identify the analyte associated with each peak in the gas chromatogram. The retention time for the analyte then becomes the retention time of its peak in the chromatogram.

10.2.2 Calibration. The GC must be calibrated using a minimum of three concentration levels of each potential analyte. (See Section 7.7 for instructions on preparation of the calibration standards.) Beginning with the lowest concentration level calibration standard, carry out the analysis procedure as described beginning in Section 11.7. Repeat the procedure for each progressively higher concentration level until all calibration standards have been analyzed.

10.2.2.1 Calculate the RT's for the internal standard and for each analyte in the calibration standards at each concentration level as described in Section 10.2.1. The RT's for the internal standard must not vary by more than 0.10 minutes. Identify each analyte by comparison of the RT's for peak maxima to the RT's determined in Section 10.2.1.

10.2.2.2 Compare the retention times (RT's) for each potential analyte in the calibration standards for each concentration level to the retention times determined in Section 10.2.1. The calibration is not valid unless all RT's for all analytes meet the criteria given in Section 9.3.2.

10.2.2.3 Tabulate the area responses and the concentrations for the internal standard and each analyte in the calibration standards. Calculate the response factor for the internal standard (RF<sub>is</sub>) and the response factor for each compound relative to the internal standard (RRF) for each concentration level using Equations 5 and 6, Section 12.

10.2.2.4 Using the RRF's from the calibration, calculate the percent relative standard deviation (percent RSD) for each analyte in the calibration standard using Equation 7, Section 12. The percent RSD for each individual calibration analyte must be *less* than 15 percent. This criterion must be met in order for the calibration to be valid. If the criterion is met, the mean RRF's determined above are to be used until the next calibration.

10.3 Daily Calibration Checks. The calibration curve (Section 10.2.2) must be checked and verified at least once each day that samples are analyzed. This is