# 6020 QUALITY ASSURANCE/QUALITY CONTROL FOR ORGANIC COMPOUNDS

## 6020 A. Introduction

Quality assurance (QA) and quality control (QC) for organic compound analysis include the operating principles stated in Sections 1020 Quality Assurance, 1030 Data Quality, and 1040 Method Development and Evaluation. This section consolidates the additional requirements common to the methods in Part 6000. The requirements are recommended minimum QA/QC activities; they should be followed unless the individual method gives different, but comparable, specifications. Some methods may have additional QA/QC requirements. Others may have broader acceptance criteria because of the unique difficulties associated with the determination of a constituent, e.g., the extraction efficiency for phenols.

## 6020 B. Quality Control Practices

This section describes the elements of a quality control program deemed necessary to maintain proper control for organic analyses. The related data quality objectives (DQOs), i.e., the rationale for sampling and analyses, should define which elements are necessary for each individual analytical design.

### 1. Calibration

*a. Initial calibration:* Perform initial calibration with a minimum of five concentrations of analytical standards for the analyte(s) of interest (for example: 1, 5, 10, 20, 40). The lowest concentration should be at the minimum reporting level; for example, if the minimum reporting level is 1, the lowest concentration should be 1. The highest concentration should be near the upper end of the calibration range; for example, if the upper end of the calibration range is 40, the high standard should be 40. Concentration ranges should reflect concentrations in actual samples. Choose calibration concentrations with no more than one order of magnitude between concentrations; for example, for a calibration range of 1 to 1000, choose concentrations of 1, 10, 100, and 1000.

Use any of the following calibration functions, as appropriate: response factor for internal standard calibration, calibration factor for external standard calibration, or calibration curve. Calibration curves may be linear or quadratic, and may or may not pass through the origin. Use the following recommended acceptance criteria for the various calibration functions.

If response factors or calibration factors are used, relative standard deviation (RSD) for each analyte should be less than 20%. If RSD is not less than 20% for any analyte, then identify and correct source of lack of linearity before sample quantitation. When using response factors (i.e., for GC/MS analysis), check performance or sensitivity of instrument for the analyte against minimum acceptance values for response factors. Refer to specific analytical method for the acceptance criteria for response factors for each analyte.

If a linear regression is used, the correlation coefficient should be >0.995. If a calibration curve has been constructed, recalculate each calibration point compared to curve. Values should be within ±20%. If any of the recalculated values are not within ±20%, identify and correct source of outlier(s) before sample quantitation.

Use initial calibration with any of the above functions (response factor, calibration factor, or calibration curve) for quantitation of analytes in samples. Use continuing calibration, ¶ *b* below, only for checks on initial calibration and not for sample quantitation. Perform initial calibration when instrument is set up and whenever continuing calibration criteria are not met.

*b. Continuing calibration:* Continuing calibration is the periodic verification, by analysis of a calibration standard, that instrument performance has not changed significantly from initial calibration. Perform continuing calibration every 10 samples for GC analysis, every 20 samples for GC/MS analysis, or every 12 h, whichever is more frequent. Perform continuing calibration with one or more of the concentrations of analytical standards in the initial calibration, varying actual concentration of continuing calibration standard over the calibration range. The acceptance criteria for continuing calibration should be ±20% (80 to 120% recovery) compared to the known or expected value of the calibration standard. If the acceptance criteria are not met, reanalyze continuing calibration standard or repeat initial calibration. When using response factors (i.e., GC/MS analysis), check performance or sensitivity of instrument for analytes against minimum acceptance values for the response factors. Refer to the specific analytical method for acceptance criteria for response factors for each analyte.

*c. Closing standard:* Finish all runs with a laboratory-fortified blank (LFB) for VOC analyses or closing standard (for methods with procedural standards) to demonstrate that performance was still acceptable for last sample analyzed. A LFB is a reagent blank to which a known concentration of analytes has been added. See ¶ 3*b* below. All samples must be bracketed by acceptable continuing calibrations.

### 2. Initial Quality Control

*a. Initial demonstration of capability:* Before analysis of any sample, require each analyst to demonstrate proficiency with the method of choice. Include at least the analysis of a laboratory reagent blank (LRB) and four laboratory-fortified blanks (LFBs) that have added concentrations between 5 times the minimum

reporting level and the midpoint of the calibration curve. The blank should not contain any analyte at a concentration greater than one-fourth the minimum quantitation level. The precision and percent recovery calculated from the four LFBs should be at least as good as the values listed in the method of choice.

*b. Method detection level:* Determine method detection level (MDL) before any samples are analyzed, using procedure described in Section 1030C or other specified method[1] required for the type of samples the laboratory is intending to analyze. As a starting point for determining concentration to use in performing the laboratory's MDL calculation, try about five times the estimated instrument detection level or refer to the selected method. Determine MDL as an iterative process. Repeat determinations if calculated MDL is not within a factor of 10 of the fortified value. Determine MDL at least annually. Analyze samples for MDL determination over a 3- to 5-d period to generate a more realistic value. Include all applicable sample preparatory techniques in MDL determinations.

*c. Minimum quantitation level (MQL):* The MQL is the lowest level that can be quantitated accurately. MQL is defined as four times the MDL. Report samples containing compounds of interest at a level less than the MQL as <MQL. Report samples containing compounds of interest at a level less than the MDL as ND (not detected).

*e. Sample (batch) set:* A sample or batch set is defined as those samples extracted in a single day, not to exceed 20 samples.

*f. Analytical day:* An analytical day is defined as a 12-h analytical period.

### 3. Batch Quality Control

*a. Reagent blank:* A reagent blank consists of all reagents and preservatives that normally contact a sample when it is carried through the entire analytical procedure. Use a reagent blank to determine contribution of reagents and preparative analytical steps to error in the observed value. No analyte of interest should be present in a reagent blank at a level greater than one fourth the MQL. Include a minimum of one reagent blank with each sample set or batch.

*b. Laboratory-fortified blank (LFB):* An LFB, also known as spiked blank, is a reagent blank containing all the same reagents and preservatives as samples and to which a known concentration of analytes has been added. Use LFB to evaluate laboratory performance and analyte recovery in a blank matrix. Make addition concentration at least 5 times the MQL or the midpoint of the calibration curve, and use to calculate recovery limits and to plot control charts as in Section 1020B. Prepare known-addition solution for blanks and samples from a different primary mix than that used to develop working standard mix. Include a minimum of one LFB with each sample set or batch. Ensure that LFB meets performance criteria in the method of choice.

*c. Internal standard (IS):* An internal standard is a compound of known concentration added to each standard and sample extract just before sample analysis. This compound should have chromatographic characteristics similar to those of the analytes of interest. Use IS to monitor retention time, relative response, and quantity of analytes in each extract. When quantifying by the internal standard method, measure all analyte responses relative to this standard. Internal standard response should be in the range of $\pm 30\%$ compared to calibration curve response. The retention time of this compound should separate from all analytes of interest and elute in a representative area of the chromatogram. If a single compound cannot be found to meet these criteria, use additional compounds.

*d. Surrogate standard:* A surrogate standard is a compound of a known concentration added to each environmental and blank sample before extraction. Use compound(s) that have characteristics similar to those of the analytes of interest and that are unlikely to be found in environmental samples. Carry surrogate standard through entire sample extraction and analytical process to monitor extraction efficiency of the method for each sample. Refer to method of choice for specific surrogates and acceptance criteria.

*e. Quality control sample:* Analyze an externally generated quality control sample of known quantity as a laboratory-fortified blank at least quarterly or whenever new stock solutions are prepared. This sample is used to validate the laboratory's standards both qualitatively and quantitatively.

*f. Laboratory-fortified sample (LFS):* A laboratory-fortified sample, also known as laboratory-fortified matrix or matrix spike, is another portion of a sample fortified with the analytes of interest at a concentration at least 5 times the MQL or around the midpoint of the calibration range. Include a minimum of one LFS with each sample set (batch). Make LFSs of sufficient concentrations that sample background levels do not adversely affect the recovery calculations. (Adjust addition concentrations if this is a known sample to be about five times background level.) Base sample batch acceptance on results of LFBs rather than on LFSs, because the sample matrix may interfere with method performance. Prepare addition solution for blanks and samples from a different primary mix than that used to develop working standard mix.

*g. Laboratory-fortified sample duplicates (LFSDs):* A laboratory-fortified sample duplicate, also known as laboratory-fortified matrix duplicate, spiked sample duplicate, or matrix spike duplicate, is a second portion of the sample to which a known amount of analyte is added. If sufficient sample volume is collected, add to a second portion of fortified sample and compare to first. If sufficient sample volume is not collected, use a second sample to obtain results on two separate LFSs rather than LFSDs. Include a minimum of one LFSD with each sample set (batch). Compare precision and bias to those listed in the method. Base sample batch acceptance on results of reagent blank additions rather than laboratory-fortified sample duplicates.

### 4. Reference

1. U.S. Environmental Protection Agency. 1995. Definition and procedure for the determination of the method detection limit, revision 1.11. 40 CFR Part 136, Appendix B. *Federal Register* 51:23703.