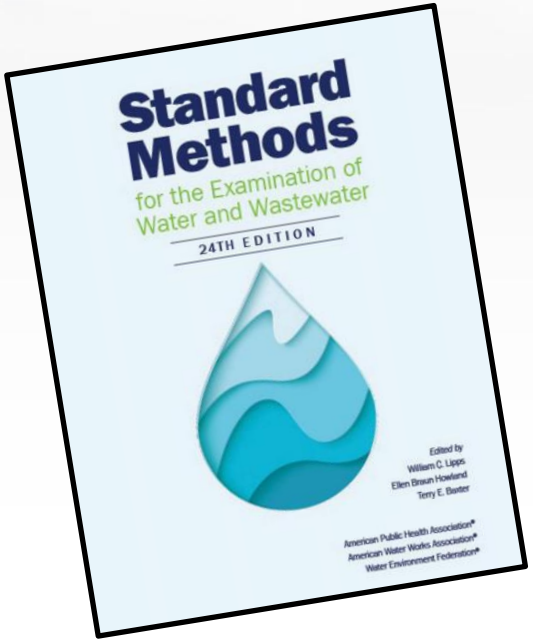
Updating the Quality Control Requirements for Organic Methods in Standard Methods



Jerry Parr
Catalyst Information Resources

Background

- 6020 provides the Quality Assurance (QA) and Quality Control (QC) practices for organic methods.
- 6020 was last revised in 2011 and does not address newer techniques and practices such as GC/MS/MS, LC/MS/MS, or isotope dilution.
- The discussion on instrument calibration and surrogates was also outdated.

Charge

- Revise Section 6020 to make it more consistent with 3020, 4020, and 5020.
- Ensure that terms and definitions do not conflict with Part 1000.
- Terms and definitions may be added.
- (Provide consistency to the TNI Standard where possible.)
- (Emphasize the use of data qualification.)

Specific Tasks

- Make SM 6020 a resource for laboratories regarding discussion on organic analysis including extractions, quality control procedures including those largely unique to organic methods, calibration models and when to use them, and instrument techniques.
- Review Table 6020:I for accuracy and include any methods that are not listed in the Table.

Process

- Create a Joint Task Group to develop the language
- JTG must have a balance of interest of Producer, User, and General Interest.
- Draft standard then balloted by full Standards Method Committee.
- Any negative votes must be resolved by revising the standard or ruling the vote non-persuasive.

Joint Task Group

• Jerry Parr, Catalyst (Chair)	General Interest
• Hunter Adams, Wichita Falls	User
• Eric Davis, Clinisys	Producer
• Mike Delaney, Retired	General Interest
• Matt Graves, ERA	Producer
• Judy Morgan, Pace	User
• Heather Lord, ESI	General Interest
• Marlene Moore, ASI	General Interest
• Paul Junio, Pace	User

William Lipps (Shimadzu) participated as a non-voting member

Terminology

Standard Methods

- LFB
- MRL
- LFM

TNI

- LCS
- LOQ
- MS

6020 Organization

- 6020A – Introduction
- 6020B – QC Practices
  - 15 subsections
- 6020C – Additional QC
  - 4 subsections

6020A

- Added emphasis on Quality Management System (QMS).
- Recommends written Quality Manual or SOPs.
- Recommended QC practices are the minimum and can be superseded by methods or regulations.
- References include TNI accreditation standard and ISO/IEC 17025.

^020C – Overview (1)

- Initial analyst and method performance
  1. Initial Demonstration of Capability (IDC)
  2. Ongoing Demonstration of Capability (ODC)
  3. Method Detection Limit (MDL)
  4. Minimum Reporting Limit (MRL)
  5. Instrument calibration (ICAL, ICV, and CCV)

IDC

- Applies to every analyst.
- 2 options
  - Blank plus 4 replicates spiked between 2 * MRL and mid-point
  - Analyze a PT sample
- Repeat test for any analyte with unacceptable performance.
- Use 70-130% as a default.

ODC

- Routinely meet the QC requirements in the method.
- Includes the 5 options in the 2016 TNI Standard.

MDL

- Follow 40 CFR 136 Appendix B.
- Reporting rules
  - Report results below the MDL as “not detected” (ND) or <MDL (where MDL is replaced by the numerical value of the MDL).
  - Report results between the MDL and MRL with qualification that the value is below the MRL for the quantified value given.
  - Report results above the MRL with a value.

MRL and Range

- MRL established by
  - The concentration of the lowest calibration standard.
  - 3 times the MDL.
  - 3.18 times the MDL rounded to the nearest 1, 2, or 10.
- Quarterly verification required by analyzing a LFB spiked at or below the MRL
- Establish the upper limit.

ICAL

- 4 points for average RF, 5 for linear, 6 for non-linear, excluding isotope dilution.
- Various options allowed including weighting.
- Single point allowed for isotope dilution but not recommended.
- Evaluate RF using RSD; other curves use RE or RSE.
- Selective removal of calibration points is prohibited.

ICV

- Second source standard at mid-point (with options).
- Acceptance criteria of +/- 10%.

CCV

- Can be based on time or number of samples.
- Examples provided (12 hours or 20 samples) but not mandated.
- Corrective action required for a failed CCV.
- Ending CCV required for external standard methods.

On-Going QC

1. Method blank (MB)
2. Laboratory-Fortified Blank (LFB)
3. Laboratory-Fortified Matrix (LFM)
4. Laboratory-Fortified Matrix Duplicate (LFMD) or duplicate (DS)
5. Internal Standard (IS)
6. Surrogate Spike (SS)
7. Extracted Internal Standard (EIS), for isotope dilution methods
8. Mass Calibration (MS)
9. Proficiency Testing (PT)
10. Method Specific QC Practices

Blanks

- Method blank required per batch.
- Acceptance criteria are:
  - < MDL and results > MRL, then no qualification.
  - > MDL but < MRL and results > MRL, qualify the results.
  - Blank and sample > MDL and < MRL, reanalyze the samples or qualify.
  - > MRL, further corrective action and/or qualification is required.
- Other blanks (e.g., field, trip) discussed but not required.

LFB

- 1 per batch or 5 %.
- Used to evaluate batch performance.
- Marginal exceedances allowed per TNI.
- Low-level may be used limits are 50-150%.

LFM

- 1 per batch or 5%.
- Used to evaluate analyte recovery in a sample matrix.
- Spike at 5 * MRL.
- Take corrective action to rectify the matrix effect, use another method, or qualify the data if reported.
- Base sample batch acceptance on results of LFB rather than LFM, because the LFM may interfere with method performance.

Duplicates

- Optional: LFMD or DS required 1 per batch or 5%.
- Evaluate results for precision.
- If results are out of control, take corrective action to rectify the matrix effect, use another method, use the method of standard addition, or qualify the data.

IS

- Not required, but good practice.
- Limits such as 50-200% may be used.
- If limits exceeded, use an alternate technique (e.g., using a different internal standard or performing an external standard calibration).

SS

- Used to monitor extraction recovery.
- If results are out of control, then take corrective action, including re-preparation and reanalysis if possible, or reporting the data with qualifiers.

EIS

- Applies to isotope dilution methods.
- Recovery should be 70-130%.

Mass Calibration

- Follow manufacturer’s recommendation.

PT (QCS)

- Single blind (QCS) or double blind (PT) allowed.
- Recommended frequency is semi-annually.
- Investigate circumstances fully to find the cause for failed results.

Method QC

- Follow instructions in the method such as:
  - Second column confirmation
  - DDT/Endrin breakdown
  - Peak tailing

Table 6020:1

Section	LFM	LFMD or DS	IS	SS	EIS	MS	PT	Notes
6040 B	•	(•)	•	•			(•)	1
6040 C	•	(•)	•	•		•	(•)	
6040 D	•	(•)	•	•		•	(•)	
6040 E	•	(•)	•	•			(•)	
6200 B	•	(•)	•	•		•	(•)	

1. ICAL, ICV, CCV, IDC, ODC, MB, MDL, MRL, and LFB required for all methods.

• indicates a test is mandatory and (•) indicates it is optional.

Additional QC

1. Corrective action and root cause analysis
2. Frequency of QC activities and definitions of a batch
3. QC acceptance criteria, including control charts as applicable
4. Data qualifiers and case narratives

Thank You!

- Jerry Parr
  - jparr@catalystinforesources.com