ORGANICS

Arizona Department of Health Services
Mary Cornell

Method Updates

- ADHS will no longer license method 8141A. Method 8141B will be offered instead. 14
- Both 8260B and 8260C will be offered as well as both 8270C and 8270D.
- The version of method 8000 you will reference will depend on which determinative method you will be using. 15

Method 8000	Determinative Methods
8000C	8141B 8260B 8270C
8000D	8260C 16 8270D

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I4	May director approve 8141A if permit requires it...check with Steve. labuser, 9/2/2016
I5	May not pick and choose requirements/allowances from 8000C and 8000D. Must follow one or the other. labuser, 9/2/2016
I6	8000D not explicitly assigned to 8141B in the draft rules. This was our mistake. Too late to correct. We will be addressing this. labuser, 9/2/2016

General SW-846 Method Updates

- Improved overall method formatting for consistency with SW-846 methods style guidance.
- Formatting updated to Microsoft Word.docx
- Many minor editorial and technical revisions made throughout to improve method clarity. l2

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I2 This presentation will not cover these minor revisions in detail. Only major revisions will be covered.
labuser, 9/9/2016

Method 8000D

Determinative Chromatographic Separations

Revision 4, July 2014

8000D Updates

Carrier Gas – Optional use of hydrogen gas for GC to address helium shortages (Section 1.3.1)

Initial Demonstration of Proficiency (IDP)

- Replaces “Initial Demonstration of Capability (IDC)” in 8000C
- Section 9.3.3 – Additional text to indicate that samples used for IDP analyses should be from the same source as the calibration standard.
  - Now consistent with the language in methods 8260 and 8270 (Appendix A)
- Separate language for preparation/extraction chemists and instrument chemists.
  - This allows the chemist to only perform an IDP for the procedure that they actually do. (Appendix A)

8000D (cont.)

- **MQL vs. LLOQ -** Method 8000D will now use the terminology Lower Limit of Quanitation (LLOQ) in place of Method Quantitation Limit (MQL).

- **Relative Standard Error (RSE) -** Included in Section 11.5.4.2 as an alternative for the determination of calibration acceptability.
  - RSE acceptance criteria is the same as the RSD limit for RF in the determinative method.
  - If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for good performing compounds and ≤30% for poor performing compounds.

8000D Calibration Verification

- LCS may serve as the CCV (Section 9.4.1)

- For multi-analyte methods, you will not have to reanalyze if ≤10% of compounds fall outside of the acceptance criteria. (Section 11.7.1)
  - Any detected analytes exceeding the limit **must be reported as estimated**.
  - If the analyte recovers below the acceptance criteria and the analyte is non-detect in the sample, a sensitivity verification standard ≤ LLOQ should be analyzed. The analyte should be detected in the LLOQ standard and meet qualitative identification criteria (e.g. RT, qualifier ions, signal-to-noise, etc.).

Method 8260C

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry

Revision 3, August 2006

8260 ICAL

8260B	8260C
Calibration Check Compounds (CCC)	No CCCs
System Performance Check Compounds (SPCC)	No SPCCs
%RSD ≤15 for all target compounds; %RSD for CCCs ≤30 (Section 7.3.6.3)	%RSD criteria for all targets ≤20 (Section 11.3.4.1)
No minimum RF requirement for target analytes	Recommended minimum RF values specified in Table 4 (Section 11.3.4.1)
No re-fitting of calibration standards required	If linear calibration used must verify the RL by re-calculating concentrations in the lowest calibration standard using calibration curve. Acceptance criteria is 70-130% Rec. (Section 11.4.5.6)
Must recalibrate if 1 or more CCCs out or >20% of targets exceed 30% RSD (Section 7.3.6.4)	Must recalibrate if >10% of targets exceed the %RSD or regression criteria (Section 11.3.4.2)

8260C ICAL – Additional Requirements

I8

- May use a single surrogate concentration in ICAL if autosampler is used (Section 11.3.3 Note)
- SIM - must monitor a minimum of 2 ions per analyte, primary and confirmation (Section 11.5.12)

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I8 Most labs already complying with the following. These are just specifically addressed now.
labuser, 9/2/2016

8260 Calibration Verification

8260B	8260C
No ICV requirement	Must be a second source standard. Suggested acceptance criteria 70-130% (Section 11.4.2)
No RF requirement for CCVs	Evaluate minimum RFs specified in Table 4 of 8260C (Section 11.4.5.1)
No IS requirement for CCVs	The IS area counts must be -50-200% of area counts in the associated mid-level ICAL std. (Section 11.4.7)
For a CCV, CCC %Drift criteria ≤20 (Section 7.4.5.2)	%D ≤20 for all targets. Recalibrate if >20% targets exceed the %D criteria. (Section 11.4.5.4) • Failed compounds may be reported as non-detects if there is adequate sensitivity to detect the compound at the quantitation limit. • Failed compounds that are present in the sample must be reported as estimated values.

8260C Additional Updates

- **Section 7.9** – Dibromofluoromethane no longer listed as a recommended surrogate, but may be used depending upon the analysis requirements.

- **Section 7.13** - LCS and MS should be prepared from the same source as the calibration standards.I1

- **Section 7.14** - Standards may be stored <6°C (previously I9 < -10°C) unless otherwise specified by the manufacturer.

- **Section 11.6.1.4** - Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <50% (previously <25%) of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

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I1 Section 7.13 already allowed by Director Approved Method Modification #20.
labuser, 9/2/2016

I9 Recommend to continue storing volatile standards <-10 degrees C.
labuser, 9/2/2016

Method 8270D

Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry

Revision 5, July 2014

8270 DFTPP Tune Criteria

	8270C	8270D
51	30-60% of mass 198	**10-80%** of mass 198
68	< 2% of mass 69	< 2% of mass 69
70	< 2% of mass 69	< 2% of mass 69
127	40-60% of mass 198	**10-80%** of mass 198
197	< 1% of mass 198	**< 2%** of mass 198
198	Base peak, 100% relative abundance	Base peak, **or > 50% of mass 442**
199	5-9% of mass 198	5-9% of mass 198
275	10-30% of mass 198	**10-60%** of mass 198
365	> 1% of mass 198	> 1% of mass 198
441	Present but less than mass 443	Present but **< 24% mass 442**
442	> 40% of mass 198	**Base peak, or > 50% of** mass 198
443	17-23% of mass 442	**15-24%** of mass 442

8270 ICAL

8270C	8270D
Calibration Check Compounds (CCC)	No CCCs
System Performance Check Compounds (SPCC)	No SPCCs
%RSD ≤15 for all target compounds; %RSD for CCCs ≤30 (Section 7.3.5.2)	%RSD criteria for all targets ≤20 (Section 11.3.4.1)
No minimum RF for target compounds	Recommended minimum RF values specified in Table 4 (Section 11.3.4.1)
No re-fitting of calibration standards required	If linear calibration used must verify the RL by re-calculating concentrations in the lowest calibration standard using calibration curve. Acceptance criteria is 70-130% Rec. (Section 11.4.5.6)
Must recalibrate if 1 or more CCCs out or >20% of targets exceed 30% RSD (Section 7.3.5.3)	Must recalibrate if >10% of targets exceed the %RSD or regression criteria (Section 11.3.4.2)

Identical to 8260C ICAL updates

8270 Calibration Verification

8270C	8270D
No ICV requirement	Must be a second source standard. Suggested acceptance criteria 70-130% (Section 11.4.2)
No RF requirement for CCVs	Evaluate minimum RFs specified in Table 4 of 8260C (Section 11.4.5.1)
No IS requirement for CCVs	The IS area counts must be 50-200% of area counts in the associated mid-level ICAL std. (Section 11.4.7)
For a CCV, CCC %Drift criteria ≤20 (Section 7.4.5.2) Also identical to 8260C	%D ≤20 for all targets. Recalibrate if >20% targets exceed the %D criteria. (Section 11.4.5.4) • Failed compounds may be reported as non-detects if there is adequate sensitivity to detect the compound at the quantitation limit. • Failed compounds that are present in the sample must be reported as estimated values.

8270D Additional Updates

- **Section 8.2** - Standards may be stored <6°C (previously < -10°C) unless otherwise specified by the manufacturer.

- **Section 11.6.1.4** - Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is <50% (previously <25%) of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

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I10 Section 7.13 already allowed by Director Approved Method Modification #20.
labuser, 9/2/2016

I11 Recommend to continue storing volatile standards <-10 degrees C.
labuser, 9/2/2016

Method 8141B

Organophosphorus Compounds by Gas Chromatography

Revision 2, February 2007

8141B Updates

- Solid samples may now be extracted by EPA Method 3550 - Ultrasonic Extraction (Section 2.1.2)
- When an internal standard calibration is used, the IS should be no more than 50% different from the average area of the calibration standards (Section 11.4.2)
- Calibration verification recommended every 10 samples, but only required every 20 samples. Method blanks and control standards are counted in the total. (Section 11.4.1)

Questions?

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