

SCIENCE APPLICATIONS INTERNATIONAL CORPORATION
Organic Data Review Checklist - Standard Validation

Project: Harley-Davidson

Page 1 of 11

SDG No: 180-40481-1

Analysis: VOC

Method: 8260 LL

Laboratory: TestAmerica Pittsburgh

Matrix: Water

The above data package has been reviewed and the analytical quality control/quality assurance performance data have been summarized. The general criteria used to assess the analytical integrity of the data were based on an examination of the following:

- | | |
|---------------------------|------------------------------------|
| Case Narrative | Analytical Surrogate Recoveries |
| Analytical Holding Times | Internal Standard Performance |
| Sample Preservation | MS/MSD Recoveries and Differences |
| Method Calibration | LCS Recoveries |
| Method and Project Blanks | Re-analysis and Secondary Dilution |

Project Specific QA/QC or contract requirements may take priority over validation criteria in this procedure.

Overall Remarks: Initial and Continuity Calibration and
LCS ISSUES.

Fixed limits used for LCS recovery criteria
instead of Lab limits (Compound specific) which
result in potentially unnecessary J-flags. - OAR

Definition of Qualifiers:

- "U", not detected at the associated level
- "UJ", not detected and associated value estimated
- "J", associated value estimated
- "R", associated value unusable or analyte identity unfounded
- "=", compound properly identified and value positive

Reviewed by: Alex G. Miller Jr. [Signature]

Date: 2/26/15

QA Reviewed by: [Signature]

Date: 6-23-15

3/3/15
AGM

I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks: No issues

II. Re-analysis and Secondary Dilutions

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

Remarks: No issues

III. Holding Times

VOC - Waters - unpreserved: aromatic within 7 days, non-aromatic within 14 days of sample collection
 VOC - Waters - preserved: aromatic and non-aromatic within 14 days of sample collection
 VOC - Soils - preserve or analyze within 48 hours of sample collection; analyze within 14 days of preservation

SVOC, Pest., PCB - Waters - extract within 7 days of sample collection, analyze within 40 days of extraction
 SVOC, Pest., PCB - Soils - extract within 14 days of sample collection, analyze within 40 days of extraction

Deviations:

| Sample # | VOC | | SVOC | | | Pest/PCB | | |
|----------|----------------|---------------|----------------|----------------|---------------|----------------|----------------|---------------|
| | Date Collected | Date Analyzed | Date Collected | Date Extracted | Date Analyzed | Date Collected | Date Extracted | Date Analyzed |
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Actions:

- 1. If holding times are exceeded, all results are qualified as estimated (J/UJ)
- 2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)

Remarks:

_____ No issues see attached

IV. System Monitoring Compounds (SMC) Recoveries (VOC, SVOC, Pesticides, PCBs)

List SMC compounds with unacceptable recoveries:

Deviations:

| Sample # | VOC | | | SVOC B/N Compounds | | | SVOC Acid Compounds | | | Pest | PCB |
|--------------|-----|-----|-----|-----------------------|-----|-----|------------------------|-----|-----|------|-----|
| | TOL | BFB | DCE | NBZ | FBP | TPH | PHL | 2FP | TBP | TCX | DCB |
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| QC Limits | | | | | | | | | | | |

- Actions:**
1. If any SMC recovery is <10%, qualify all positive results in associated fractions as estimated (J)
 2. If any SMC recovery is <10%, qualify all nondetects in associated fractions as unusable (R)
 3. If SMC recoveries fall between 10% and the lower recovery limit, qualify results as estimated (J/UJ)
 4. If SMC recoveries fall above the upper recovery limit, qualify positive results as estimated (J)
 5. Use professional judgement to qualify Pest/PCB results when SMC recoveries are >10%
 6. Use professional judgement to qualify results when SMC recoveries have been diluted out of spec.
 7. For SVOC, qualification of the data is required only when 2 or more SMC per fraction are not within control limits
 8. Note: SMC formerly known as surrogates.

Remarks: No issues

V. Internal Standards Performance (VOC, SVOC)

VOC internal standard area counts within -50% to +100% of standard (Y/N)
VOC internal standard retention times within \pm 30 seconds of standard (Y/N)

SVOC internal standard area counts within -50% to +100% of standard (Y/N)
SVOC internal standard retention times within + 30 seconds of standard (Y/N)

Deviations:

| Sample # | IS Affected | Area Counts | Acceptable Range | RT | Std. RT Value |
|----------|-------------|-------------|------------------|----|---------------|
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Actions:

- 1. If area counts are outside limits, qualify positive results associated with that IS as estimated (J)
- 2. Non-detected compounds quantitated using an IS area count >100% should not be qualified
- 3. Non-detected compounds quantitated using an IS area count <50%, qualify as estimated (UJ)
- 4. If extremely low area counts are reported (<50% of the lower limit), qualify non-detects as unusable (R)
- 5. If an IS retention time varies more than 30 seconds, review the chromatographic profile for shifts and irregularities. Use professional judgement to qualify the data estimated (J/UJ) or unusable (R)

Remarks: No issues

VI. Blanks

All blanks were reported per matrix per concentration level for each 12 hour period on each GC/ MS system used to analyze VOCs and SVOCs Yes No

Review associated laboratory and project blank samples. List documented contamination below:

Laboratory Method Blanks:

| Date: | Lab ID # | Fraction | Compound | Conc. (ppb) |
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Associated Project Blanks (e.g., equipment rinsates, trip blanks, etc.)

| Date | Lab ID # | Fraction | Compound | Conc. (ppb) |
|------|----------|----------|----------|-------------|
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Remarks: no detections

VI. Blanks (continued)

Calculate action levels based on 10X the highest blank concentration of "common laboratory solvents", VOCs (methylene chloride, acetone, toluene, 2-butanone, cyclohexane) or SVOCs (phthalates), and 5X the highest blank concentration for all other VOC, SVOC, Pesticides, and PCB compounds. Sample weights, volumes, and dilution factors must be taken into account when applying the 5X and 10X criteria. This allows the total amount of contaminant present to be considered.

Deviations:

| Compound | Maximum Conc. Detected, (ppb) | Action Level (ppb) | Samples Affected |
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Actions:

- 1. If compound results exceed the action levels, the data are not qualified
- 2. If compound results are below the required reporting level, report results as non-detect (U) at the reporting level
- 3. If the compound is detected above the reporting level, but below the action level, qualify as not-detected (U)
- 4. If gross contamination exists in blanks (i.e., saturated peaks by GC/ MS), all affected compounds in the associated samples should be qualified as unusable (R) due to interference.
- 5. If blanks were not analyzed per matrix per concentration level for each 12 hour period on each GC/MS system used to analyze VOCs and SVOCs use professional judgement to qualify data. Data may be rejected (R).

Remarks: No deviations

VII. Initial & Continuing Calibration (VOC, SVOC)

GC/MS instrument performance checks (BFB / DFTPP) Acceptable Y or N
 All compounds must have and RRF > 0.01, %RSD < 30, and %D < 25

VOC - Date of initial calibration: 12/15/14
 VOC - Date(s) of continuing calibration: 1/16/15, 1/19/15
 Was the 12 hour criteria met? Y or N

SVOC- Date of initial calibration: _____
 SVOC - Date(s) of continuing calibration: _____
 Was the 12 hour criteria met? Y or N

NA

Deviations:

| Compound | Date | RRF | %RSD | %D | Samples Affected |
|------------------------|----------|-------|------|------|-------------------------------|
| 1,4-Dioxane | 12/15/14 | 0.003 | | | 1, 2, 3, 4, 5, 6, 7, 8, 9 - R |
| Trichlorofluoromethane | 1/19/15 | | | 35.5 | None |
| 2,2-Dichloropropane | 1/19/15 | | | 45.7 | None |
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* % Difference = ((RF_{CCV} - RF_{ICAL AVG})/RF_{ICAL AVG}) x 100. In instances where the bias of the CCV impacts validation qualifiers, review the RF values or amount reported to confirm that the % Difference or % Drift are reported with the correct negative or positive value.

Actions:

1. If any compound has an initial or continuing RRF of < 0.01, qualify positive results as estimated (J)
2. If any compound has an initial or continuing RRF of < 0.01, qualify non-detects as unusable (R)
3. If any compound has a %RSD >30 or a %D >25, qualify positive results as estimated (J)
4. If any compound has a %RSD >40 or a %D >40, qualify non-detects as estimated (UJ)
5. If BFB or DFTPP mass assignment / ION abundance criteria are all associated data as unusable (R).
6. If samples were analyzed outside the 12 hour BFB or DFTPP performance check time period, qualify the affected sample data as estimated (J/UJ).
7. If separate calibration for water and soil were not performed, use professional judgement to evaluate the data. Data may be rejected (R).
8. If calibrations were not completed within the 12 hour criterion, qualify all associated data as estimated (J/UJ). If the 12 hour criterion was grossly exceeded, reject all associated data (R).

Remarks: see above.

VIII. Initial & Continuing Calibration (Pesticides, PCBs)

Linearity evaluation, are %RSD <20? (Y/N) _____

Is the RPD between calibration factors ≤25? (Y/N) _____

Are multicomponent calibration data provided for each analysis date? (Y/N) _____

Is the difference between columns check ≤ 25%D? (Y/N) _____

Are 4, 4'- DDT and endrin breakdown (PEM) ≤ 20% and combined breakdown ≤ 30% (Y/N)

Deviations:

| Compound | %RSD | RPD | Samples Affected |
|----------|------|-----|------------------|
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* % Difference = ((RF_{CCV} - RF_{ICAL AVG})/RF_{ICAL AVG}) x 100. In instances where the bias of the CCV impacts validation qualifiers, review the RF values or amount reported to confirm that the % Difference or % Drift are reported with the correct negative or positive value.

Actions:

1. If %RSD criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
2. If RPD criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
3. If %D criteria is not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
4. If breakdown criteria are not met, positive 4, 4'-DDT and endrin should be qualified as estimated (J).
And non-detects should be rejected (R).

Remarks:

IX. Matrix Spike/Matrix Spike Duplicate Information

General MS/MSD Criteria:
 percent recovery (%R)
 relative percent difference (RPD)

| | | | |
|--------|--------|--------|--------|
| VOC | SVOC | Pest | PCB |
| 70-130 | 45-135 | 40-140 | 40-140 |
| <30 | <50 | <50 | <50 |

Project Sample(s) Spiked: _____

Deviations:

| Compound | %R | %R Limits | RPD | RPD Limits | Samples Affected |
|----------|----|-----------|-----|------------|------------------|
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Actions:

1. If the spike recovery is above the upper control limit (UCL), qualify all positive values in the unspiked sample as estimated (J) and non-detects as estimated (UJ).
2. If the spike recovery is below the lower control limit (LCL), qualify positive values as estimated (J).
And non-detects as estimated (UJ).
3. If the spike recovery is <10%, qualify non-detect values as unusable (R)
4. If the RPD does not meet criteria, qualify positive values in the unspiked sample as estimated (J)
5. Use professional judgement to qualify additional samples in the analytical group based on MS/MSD results
6. Use professional judgement for qualification of data for unspiked compounds

Remarks: None Collected,

X. Laboratory Control Sample Information

General LCS Criteria:
percent recovery (%R)

| | | | |
|--------|--------|--------|--------|
| VOC | SVOC | Pest | PCB |
| 80-120 | 60-120 | 50-130 | 50-130 |

Laboratory LCS Identifications: LCS 180-130947/9, LCS 180-131060/8, LCS 180-130947/10

Deviations:

| Compound | Date | %R | Samples Affected/Qualifiers Applied |
|----------------------|---------|----|-------------------------------------|
| 2 - Hexanone | 1/19/13 | 78 | 6, 7, 8 - J or UJ |
| 1,1 - Dichloroethane | ↓ | 75 | ↓ |
| Chloroethane | ↓ | 75 | ↓ |
| Vinyl Chloride | ↓ | 74 | ↓ |
| Chloroethane | ↓ | 73 | ↓ |
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these are all within the lab's QC limits

Actions:

- Action should be based on both the number of compounds outside the criterion and the magnitude of the exceedance.
1. If the LCS recovery is below limits but > one-half the lower limit, qualify valves as estimated (J/UJ).
 2. If the LCS recovery is < one-half the lower limit, qualify all data for that analyte as unusable (R).
 3. If the LCS recovery is greater than the upper limit, qualify positive valves for that analyte as estimated (J).
 4. If more than half the compounds in this LCS are not within recovery criteria, then qualify associated detected compounds as estimated (J).
 5. Use professional judgement for qualification of data for compounds with no LCS information

Remarks: See above.

Hold Time Summary

| Sample Number | Method | Date Collected | Analysis Date | Date Extracted | Days to Analysis |
|---------------|-------------|----------------|---------------|----------------|------------------|
| 180-40481-1 | MCAWW 300.0 | 1/14/2015 | 1/15/2015 | | 1 |
| 180-40481-2 | MCAWW 300.0 | 1/14/2015 | 1/15/2015 | | 1 |
| 180-40481-3 | MCAWW 300.0 | 1/14/2015 | 1/15/2015 | | 1 |
| 180-40481-4 | MCAWW 300.0 | 1/14/2015 | 1/15/2015 | | 1 |
| 180-40481-5 | MCAWW 300.0 | 1/14/2015 | 1/15/2015 | | 1 |
| 180-40481-6 | MCAWW 300.0 | 1/14/2015 | 1/15/2015 | | 1 |
| 180-40481-7 | MCAWW 300.0 | 1/14/2015 | 1/15/2015 | | 1 |
| 180-40481-8 | MCAWW 300.0 | 1/14/2015 | 1/15/2015 | | 1 |
| 180-40481-1 | SM SM 2320B | 1/14/2015 | 1/23/2015 | | 9 |
| 180-40481-2 | SM SM 2320B | 1/14/2015 | 1/23/2015 | | 9 |
| 180-40481-3 | SM SM 2320B | 1/14/2015 | 1/23/2015 | | 9 |
| 180-40481-4 | SM SM 2320B | 1/14/2015 | 1/23/2015 | | 9 |
| 180-40481-5 | SM SM 2320B | 1/14/2015 | 1/23/2015 | | 9 |
| 180-40481-6 | SM SM 2320B | 1/14/2015 | 1/23/2015 | | 9 |
| 180-40481-7 | SM SM 2320B | 1/14/2015 | 1/23/2015 | | 9 |
| 180-40481-8 | SM SM 2320B | 1/14/2015 | 1/23/2015 | | 9 |
| 180-40481-1 | SW846 6020A | 1/14/2015 | 1/21/2015 | 1/16/2015 | 7 |
| 180-40481-2 | SW846 6020A | 1/14/2015 | 1/21/2015 | 1/16/2015 | 7 |
| 180-40481-3 | SW846 6020A | 1/14/2015 | 1/21/2015 | 1/16/2015 | 7 |
| 180-40481-4 | SW846 6020A | 1/14/2015 | 1/21/2015 | 1/16/2015 | 7 |
| 180-40481-5 | SW846 6020A | 1/14/2015 | 1/21/2015 | 1/16/2015 | 7 |
| 180-40481-6 | SW846 6020A | 1/14/2015 | 1/21/2015 | 1/16/2015 | 7 |
| 180-40481-7 | SW846 6020A | 1/14/2015 | 1/21/2015 | 1/16/2015 | 7 |
| 180-40481-8 | SW846 6020A | 1/14/2015 | 1/21/2015 | 1/16/2015 | 7 |
| 180-40481-1 | SW846 8260C | 1/14/2015 | 1/16/2015 | | 2 |
| 180-40481-2 | SW846 8260C | 1/14/2015 | 1/16/2015 | | 2 |
| 180-40481-3 | SW846 8260C | 1/14/2015 | 1/16/2015 | | 2 |
| 180-40481-4 | SW846 8260C | 1/14/2015 | 1/16/2015 | | 2 |
| 180-40481-5 | SW846 8260C | 1/14/2015 | 1/16/2015 | | 2 |
| 180-40481-6 | SW846 8260C | 1/14/2015 | 1/16/2015 | | 2 |
| 180-40481-6 | SW846 8260C | 1/14/2015 | 1/19/2015 | | 5 |
| 180-40481-7 | SW846 8260C | 1/14/2015 | 1/19/2015 | | 5 |
| 180-40481-8 | SW846 8260C | 1/14/2015 | 1/19/2015 | | 5 |
| 180-40481-9 | SW846 8260C | 1/14/2015 | 1/16/2015 | | 2 |

Trip Blank Detections

| Sample ID | Sample | Analyte | Result | Method | Units | Qual |
|-----------|--------|---------|--------|--------|-------|------|
|-----------|--------|---------|--------|--------|-------|------|

SCIENCE APPLICATIONS INTERNATIONAL CORPORATION
Inorganic Data Review Checklist - Standard Validation
 (Chloride, Fluoride, Nitrate/Nitrite, Sulfate, Sulfide, Phosphate, etc.)

Project: Harley - Davidson

Page 1 of 8

SDG No: 180-40481-1

Analysis: Chloride, Nitrate, Sulfate, Alkalinity

Laboratory: TestAmerica Pittsburgh

Method: 9320B, 300.0

Matrix: Water

The above data package has been reviewed and the analytical quality control/quality assurance performance data have been summarized. The general criteria used to assess the analytical integrity of the data were based on an examination of the following:

- | | |
|--------------------------|------------------------------------|
| Case Narrative | Method and Project Blanks |
| Analytical Holding Times | Matrix Spike Recoveries |
| Sample Preservation | Duplicate Differences |
| Method Calibration | LCS Recoveries |
| | Re-analysis and Secondary Dilution |

Overall Remarks: No major issues

Definition of Qualifiers:

- "U", not detected at the associated level
- "UJ", not detected and associated value estimated
- "J", associated value estimated
- "R", associated value unusable or analyte identity unfounded
- "=", compound properly identified and value positive

Reviewed by: Alan G. Miller II

Date: 3/2/15

QA Reviewed by: CR

Date: 6-23-15

3/2/15
AGM

I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks: No major issues

II. Re-analysis and Secondary Dilutions

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

Remarks: No issues

III. Holding Times

Sample should be preserved and analyzed according to the appropriate analytical method
In general the following preservations and holding times for waters can be applied:

- Sulfate, 4 degrees C, 28 days
- Sulfide, 4 degrees C, pH ≥ 9 with zinc acetate/sodium hydroxide, 7 days
- Bromide/Chloride/Fluoride, no preservative required, 28 days
- Nitrate/Nitrite or Ammonia, 4 degrees C, pH ≤ 2 with sulfuric acid, 28 days
- Nitrate or Nitrite, 4 degrees C, 48 hours
- Alkalinity, 4 degrees C, 14 days
- TDS/TSS, 4degrees C, 7 days
- Phosphate (total), 4 degrees C, pH < 2 with sulfuric acid, 28 days
- Hexavalent Chromium, Cool 4 degrees C, water- 24 hours, soil - 30 days

Deviations:

| Sample # | Analyte | Date Collected | Date Extracted | Date Analyzed | Notes: |
|----------|---------|----------------|----------------|---------------|--------|
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Actions:

1. If holding times are exceeded, all results are qualified as estimated (J/UJ)
2. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)
3. If samples were not properly preserved, use professional judgement to qualify the data

Remarks:

No issues.

IV. Initial & Continuing Calibration

A blank and at least three standards should be analyzed, with one of the standards being within 2X the MDL
Correlation coefficients must be ≥ 0.995
Initial calibration check recoveries must be within 90-110%
Continuing calibration check recoveries must be within 85-115%

Deviations:

| Compound | Correlation Coefficient | ICV/CCV | %R | Samples Affected |
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Actions:

- 1. If any compounds initial calibration linearity is < 0.995 , qualify the data as estimated (J/UJ)
- 2. If any compounds initial calibration linearity is < 0.95 , qualify the data as unusable (R)
- 3. If ICV or CCV criteria are not met, qualify positive results as estimated (J) and non-detects as estimated (UJ)
- 4. If ICV or CCV recoveries fall below 50%, qualify results as unusable (R)

Remarks:

V. Blanks (Method Blanks and Project Blanks)

An analytical method blank must be analyzed with each batch of samples

Calculate action levels based on 5X the highest blank concentration of any given analyte

Sample weights, volumes, and dilution factors must be taken into account when applying the 5X criteria

Deviations:

| Analyte | Maximum Conc. Detected, (ppb) | Action Level (ppb) | Samples Affected |
|------------------------|-------------------------------|--------------------|------------------|
| Total Alkalinity | 3.96 | 19.8 | None |
| Bicarbonate Alkalinity | 3.96 | 19.8 | ↓ |
| Nitrate as N | 0.00912 | 0.00456 | ↓ |
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Actions:

- 1. If analyte results exceed the action levels, the data are not qualified
- 2. If analyte results are below the required reporting level, report results as non-detect (U) at the reporting level
- 3. If the analyte is detected above the reporting level, but below the action level, qualify as not-detected (U)

Remarks: No samples impacted

VI. Laboratory Control Sample Information

Each analyte's LCS % recovery must be within the control limits established by the laboratory
In general LCS % recoveries should all be within 85-115%

Deviations:

| Analyte | Date | %R | Samples Affected/Qualifiers Applied |
|---------|------|----|-------------------------------------|
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Actions:

- 1. If the LCS recovery is outside limits but >10%, qualify all positive values as esimated (J)
- 2. If the LCS recovery is outside limits but >10%, qualify non-detect values as estimated (UJ)
- 3. If the LCS recovery is <10%, qualify all data for that analyte as unusable (R)
- 4. Use professional judgement for qualification of data for compounds with no LCS information

Remarks: No issues

VII. Matrix Spike Information

Each analyte's Matrix Spike % recovery should be within the laboratory established control limits
In general matrix spike % recoveries should all be within 75-125%

Deviations:

| Analyte | %R | %R Limits | Samples Affected |
|---------|----|--------------|------------------|
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Actions:

- 1. If the spike recovery is outside limits, qualify all values in the unspiked sample as estimated (J/UJ)
- 2. If the spike recovery is <10%, qualify non-detect values as unusable (R)
- 3. Use professional judgement to qualify additional samples in the analytical group based on MS results
- 4. Use professional judgement for qualification of data for unspiked analytes

Remarks: No Issues

SCIENCE APPLICATIONS INTERNATIONAL CORPORATION
Metals Data Review Checklist - Standard Validation

Project: Harley-Davidson

Page 1 of 13

SDG No: 180-40481-1

Analysis: Na,Ca,Mg

Method: 6020A

Laboratory: TestAmerica Pittsburgh

Matrix: Water

The above data package has been reviewed and the analytical quality control/quality assurance performance data have been summarized. The general criteria used to assess the analytical integrity of the data were based on an examination of the following:

- | | |
|---------------------------|---|
| Case Narrative | MS/MSD Recoveries and Differences |
| Analytical Holding Times | Duplicate Relative Percent Differences |
| Sample Preservation | ICP Serial Dilution |
| Method Calibration | Furnace Atomic Absorption QC |
| Method and Project Blanks | Re-analysis and Secondary Dilution |
| LCS Recoveries | Internal Standard Performance (if applicable) |

Project specific QA/QC or contract requirements may take priority over validation criteria in this procedure.

Overall Remarks: No major issues

Definition of Qualifiers:

- "U", not detected at the associated level
- "UJ", not detected and associated value estimated
- "J", associated value estimated
- "R", associated value unusable or analyte identity unfounded
- "=", compound properly identified and value positive

Reviewed by: Alan G. Miller Jr

Date: 3/2/15

QA Reviewed by: APR

Date: 6-23-15

3/3/15
AGM

I. Case Narrative

Verify direct statements made within the Laboratory Case Narrative (note discrepancies).

Remarks: No major issues

II. Re-analysis and Secondary Dilutions

Verify that re-analysis and secondary dilutions were performed and reported as necessary. Determine appropriate results to report.

Remarks: _____

III. Holding Times

Metals - Waters - preserved to pH<2, 180 days from sample collection
 Metals - Soils - 180 days from sample collection
 Mercury - Waters - preserved to pH<2, 28 days from sample collection
 Mercury - Soils - 28 days from sample collection

Deviations:

| Sample # | Metals | | | | Mercury | | | |
|----------|----------------|---------------|----------|----------|----------------|---------------|----------|----------|
| | Date Collected | Date Analyzed | Days >HT | pH Check | Date Collected | Date Analyzed | Days >HT | pH Check |
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Actions:

- 1. If preserved samples exceed holding time, qualify all associated results as estimated (J/UJ).
- 2. If unpreserved samples exceed holding time, qualify all associated results as unusable (R).
- 3. If holding times are exceeded by more than 2X, reviewer may qualify non-detected results as unusable (R)
- 4. If water samples are not acidified, use professional judgement. Minimally, qualify data as estimated (J) and non-detects unusable (R).
- 5. If soil samples exceed holding time, use professional judgement to qualify data.

Remarks:

_____ *No issues* _____

IV. Initial & Continuing Calibration (ICP, GFAA, CVAA, etc.)

Initial calibration linearity criteria is $r \geq 0.995$
 ICV and CCV criteria are $\pm 10\%$ recovery, low level check standard allowed $\pm 30\%$
 ICP inter-element check standard criteria $\pm 20\%$

Deviations:

| Element | Date | Initial Calib. | ICV/CCV | %R | Samples Affected |
|---------|------|----------------|---------|----|------------------|
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Actions:

- 1. If any elements initial calibration linearity is < 0.995 , qualify the data as estimated (J/UJ)
- 2. If any elements initial calibration linearity is < 0.95 , qualify the data as unusable (R)
- 3a. If any elements ICV or CCV recovery is $< 90\%$, qualify the data as estimated (J/UJ)
- 3b. If any elements ICV or CCV recovery is $> 110\%$, qualify results \geq MDL as estimated (J), do not qualify non-detects
- 4a. If any elements ICV or CCV recovery is $< 75\%$, qualify the data as unusable (R)
- 4b. If any elements ICV or CCV recovery is $> 125\%$, qualify positive results as estimated (J) or non-detects unusable (R)
- 4c. If any elements ICV or CCV recovery is $> 160\%$, qualify positive results \geq MDL as unusable (R). Do not qualify non-detects.
- 5a. If any elements CRI recovery is 50-69% (30-49% for Sb, Pb, Ti), qualify results \geq MDL (but $< 2 \times$ CRQL) as estimated (J/UJ) and results $> 2 \times$ CRQL are not qualified.
- 5b. If any elements CRI recovery is $< 50\%$ ($< 30\%$ for Sb, Pb, Ti), qualify results \geq MDL (but $< 2 \times$ CRQL) as unusable (R) and results $> 2 \times$ CRQL as estimate (J).
- 5c. If any elements CRI recovery is $> 130\%$ but $< 180\%$ ($> 150\%$ but $\leq 200\%$ for Sb, Pb, Ti) quality results \geq MDL (but $< 2 \times$ CRQL) as estimated (J) and non-detects and results $> 2 \times$ CRQL are not qualified.
- 5d. If CRI or (R) $> 180\%$ ($> 200\%$ for Sb, Pb, Ti), qualify results that are \geq MDL as unusable (R).

Remarks:

_____ *No Issues* _____

IV. Initial & Conting Calibration (ICP, GFAA, CVAA, etc.) (continued)

Analytical Sequence and MS Tune

(Y/N)

- 1. Were the appropriate number of ICP standards used?
- 2. Were the appropriate number of AA standards used?
- 3. Was calibration performed and documented at the beginning of each run?
- 4. Were calibration check standards run at 10% frequency or every two hours?
- 5. Were low level standard checks analyzed at approximately 2X the PQL?
- 6. Was ICP-MS mass calibration within 0.1 AMU?
- 7. Was ICP-MS % RSD of the absolute signals for all analytes < 5%?

Y

Y

Deviations:

| Element | Deviation | Samples Affected |
|---------|-----------|------------------|
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Actions:

- 1. If instrument calibration is questionable, use professional judgement, qualify the data as estimated (J/UJ)
- 2. If instrument calibration documentation can not be obtained or is inadequate, qualify the data as unusable (R)
- 3. If mass calibration for ICP-MS was not within 0.1 AMU, qualify analyte results as estimated (J/UJ).
- 4. If % RSD for ICP-MS was > 5% for any analyte in the tuning solution, qualify associated results as estimated (J/UJ).

Remarks:

V. Blanks (ICB, CCB, Method Blank, Equipment Rinsate Blank)**A. Blank Results**

If the blank level is > CRQL for any analyte check that the analyte's concentration in a sample is > 10 x the blank value. The highest blank concentration of observed elements is the action level.

Sample weights, volumes, and dilution factors must be taken into account when applying the action level.

Blank results given in ug/L must be converted to mg/kg to compare them with soil sample results.

use the following equation:

$$\text{ug/L} \times \text{V/W} \times 1\text{L}/1000\text{mL} \times 1000\text{g}/1\text{kg} \times 1\text{mg}/1000\text{ug} = \text{mg/kg}$$

where: V = volume of samples digest solution (usually 200 mL)

W = weight of sample digested (usually 1 g)

Deviations:

| Blank ID | Element | Max. Conc. Detected | Action Level | Samples Affected |
|-----------------|-----------|---------------------|--------------|------------------|
| MB180-13092/1-A | Calcium | 10.1 | 101 | None |
| ↓ | Magnesium | 2.86 | 28.6 | ↓ |
| ↓ | Potassium | 13.6 | 136 | |
| ↓ | Sodium | 5.65 | 56.5 | |
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If additional space is required, use next page

Actions:

1. For blank results \geq MDL but \leq CRQL, qualify sample results \geq MDL but < CRQL as CRQL U. Use professional judgement to qualify sample results exceeding the CRQL.
- 2a. If blank results are > CRQL: for sample values \geq MDL but \leq CRQL, qualify results as CRQL U; for sample values > CRQL but < 10 x the blank, qualify results as unusable (R) or estimated (J). No action is taken for sample results \geq 10 x the blank values.
- 2b. If ICB/ CCB results are > CRQL: for sample values \geq MDL but \leq CRQL, qualify results as CRQL U; for sample values > CRQL but < blank results, qualify results as not detected (U) at the level of the blank or unusable (R). Use professional judgement for sample results > blank results.

Remarks:

No samples impacted

V. Blanks (continued)

The highest blank concentration of observed elements is the action level.

Sample weights, volumes, and dilution factors must be taken into account.

Blank results given in ug/L must be converted to mg/kg to compare them with soil sample results.

use the following equation:

$$\text{ug/L} \times \text{V/W} \times 1\text{L}/1000\text{mL} \times 1000\text{g}/1\text{kg} \times 1\text{mg}/1000\text{ug} = \text{mg/kg}$$

where: V = volume of samples digest solution (usually 200 mL)

W = weight of sample digested (usually 1 g)

Deviations:

| Blank ID | Element | Max. Conc. Detected | Action Level | Samples Affected |
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V. Blanks (continued)

B. Frequency Requirements

(Y/N)

1. Was a method (preparation) blank analyzed for each matrix?
2. Was a method blank processed for every analytical batch (20 samples)?
3. Was a calibration blank analyzed at 10% frequency or every two hours?

Y
~~NA~~
~~NA~~

Deviations:

| Element | Deviation | Samples Affected |
|---------|-----------|------------------|
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Remarks: _____

C. Baseline Shift Evaluation

List the highest negative blank concentration for each analyte observed in laboratory or project blanks.

Deviations:

| Blank ID | Element | Max. Neg. Conc. | Action Level | Samples Affected |
|----------|---------|-----------------|--------------|------------------|
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Actions:

1. If the absolute value of the maximum negative blank result is > the CRQL, qualify positive results as estimated (J) and non-detects as estimated (UJ).

Remarks: _____

VI. Laboratory Control Sample Evaluation

All LCS recovery criteria are set at 80-120%

An LCS must be analyzed for each matrix and for each digestion batch or set of twenty samples

Deviations:

| Element | Date | %R | Matrix | Samples Affected |
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Actions:

1. If any element's LCS recovery is >120%, qualify positive results as (J) .
2. If any element's LCS recovery is 50-79%, qualify positive results as (J) and non-detect results as (UJ).
- 3a. If any element's LCS recovery is <50%, qualify positive results as (J) and non-detect results as (R).
- 3b. If the LCS recovery is > 150%, qualify all results as unusable (R).
4. For soil LCS recovery > upper limit, qualify sample results \geq MDL as estimated (J).
5. For soil LCS recovery < lower limit, qualify results \geq MDL as estimated (J) and non-detects estimated (UJ).
6. Use professional judgement to qualify data if the LCS frequency criteria are not met.

Remarks:

No issues

VII. Matrix Spike Evaluation

All MS recovery criteria are set at 75-125%

An MS must be analyzed for each matrix and for each digestion batch or set of twenty samples

Verify that a field blank or PE sample was not used for spiked sample analysis.

Verify that a post-digestion spike was analyzed for those analytes where the pre-digestion spike recovery is outside control limits and the sample result is $< 4 \times$ the spike added.

Project Sample(s) Spiked: None

Deviations:

| Element | Spiked Sample Result | Sample Result | Spike Amount | %R | Samples Affected |
|---------|----------------------|---------------|--------------|----|------------------|
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Actions:

1. If the sample concentration exceeds the spiking level by a factor of 4X or more, do not qualify the data
2. If the spike recovery is $>125\%$, qualify all positive values as (J).
3. If the spike recovery is between 30-74%, qualify positive values as (J) and non-detect values as estimated (UJ)
4. If the spike recovery is $<30\%$, qualify positive values as (J) and non-detects are qualified unusable (R) if the post-digestion spike recovery is $< 75\%$ (or none was performed); non-detects are qualified as estimated (UJ) if the post-digestion spike recovery is $\geq 75\%$. There is no post-digestion spike performed for mercury.
5. Qualify all samples of similar matrix to the spiked sample in the same manner
6. Use professional judgement to qualify data if the MS frequency criteria are not met.
7. Use professional judgement for qualification of data for unspiked elements

Remarks: None Collected

VIII. Laboratory Duplicate Evaluation

Duplicate relative percent difference (RPD) for water is 20% (both results > 5 times CRDL) or < CRDL difference (if either result is < 5 times CRDL) and RPD for soil is 35% (if both results are > 5 times CRDL or < 2 times CRDL if either result is < 5 times CRDL).

When duplicate sample values are both less than the reporting level they are considered acceptable

When duplicate sample values are within 5X the reporting level they are acceptable if their absolute difference is within 3X the reporting level

Verify that a field blank or PE samples was not used for duplicate analysis.

Deviations:

| Element | Sample # | Duplicate # | RPD | Samples Affected |
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Actions:

1. If an element's RPD is >20% (water) / 35% (soil), qualify positive results as (J) and non-detect results as (UJ)
2. For low concentrations, if an element's duplicate absolute difference is > 3X the reporting level, qualify positive results as (J) and non-detect results as (UJ)
3. Use professional judgement to qualify data if the duplicate frequency criteria are not met.

Remarks:

None collected / analyzed

IX. Inductively Coupled Plasma (ICP) Serial Dilution Analysis

Verify that a field blank or PE sample was not used for serial dilution.
 Serial dilution of positive results are performed when values exceed 50X the IDL
 Results from serial dilutions should agree within 10% of the original undiluted analysis

Deviations:

| Element | Sample # | Sample Result | Serial Dilution | %D | Action |
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Actions:

1. If the serial dilution %D is >10 and the analyte results are >50X the IDL, qualify all positive results as estimated (J) and non-detects as estimated (UJ).

Remarks: None were run on samples

X. Furnace Atomic Absorption QC

A. Duplicate Precision

(Y/N)

- 1. Were duplicate injections performed for all samples?
- 2. Were one point analytical spikes performed for all samples?
- 3. Did duplicate injections agree within $\pm 20\%$?

Deviations:

| Element | Deviation | Sample Affected |
|---------|-----------|-----------------|
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Actions:

- 1. If duplicate injection results are outside $\pm 20\%$, qualify positive results as (J) and non-detect results as (UJ)

Remarks:

B. Post Digestion Spike Recoveries

(Y/N)

- 1. Did post digestion spike recoveries meet an 85-115% recovery criteria?
- 2. If spike recoveries did not meet recovery criteria were samples analyzed by MSA?
- 3. If MSA was used to analyze samples, was its' correlation coefficient ≥ 0.995 ?

Deviations:

| Element | Deviation | Sample Affected |
|---------|-----------|-----------------|
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Actions:

- 1. If post digestion spike recoveries are $>115\%$, qualify positive results as (J) and non-detect results as (U)
- 2. If post digestion spike recoveries are 11-84%, qualify positive results as (J) and non-detect results as (UJ)
- 3. If post digestion spike recoveries are $<10\%$, qualify positive results as (R) and non-detect results as (R)
- 4. If MSA was used to quantitate values and the correlation coefficient was <0.995 , qualify data as (J or UJ)
- 5. If MSA was used to quantitate values and the correlation coefficient was <0.95 , qualify data as (R)

Remarks:
