

SCIENCE APPLICATIONS INTERNATIONAL CORPORATION
Organic Data Review Checklist - Standard Validation

Project: Harley-Davidson

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SDG No: 180-42445-2 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: Calibration 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: [Signature] Alan G. Miller Jr.

Date: 4/20/15

QA Reviewed by: [Signature]

Date: 5-14-15

Alan
5/27/15



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 _____

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

List SMC compounds with unacceptable recoveries:

Deviations:

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

| <u>Date:</u> | <u>Lab ID #</u> | <u>Fraction</u> | <u>Compound</u> | <u>Conc. (ppb)</u> |
|--------------|-----------------|-----------------|-----------------|--------------------|
| _____ | _____ | _____ | _____ | _____ |
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| _____ | _____ | _____ | _____ | _____ |

Associated Project Blanks (e.g., equipment rinsates, trip blanks, etc.)

| <u>Date</u> | <u>Lab ID #</u> | <u>Fraction</u> | <u>Compound</u> | <u>Conc. (ppb)</u> |
|-------------|-----------------|-----------------|-----------------|--------------------|
| _____ | _____ | _____ | _____ | _____ |
| _____ | _____ | _____ | _____ | _____ |
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Remarks: None

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 samles 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: None

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: 1/28/15
 VOC - Date(s) of continuing calibration: _____
 Was the 12 hour criteria met? Y or N

SVOC- Date of initial calibration: 4/1/15
 SVOC - Date(s) of continuing calibration: _____
 Was the 12 hour criteria met? Y or N

Deviations:

| Compound | Date | RRF | %RSD | %D | Samples Affected |
|------------------|---------|--------|------|------|------------------|
| 1,4 - Dioxane | 1/28/15 | 0.0026 | | | 13 = R |
| Isobutyl Alcohol | 4/1/15 | 0.0084 | | | None |
| 1,4 - Dioxane | 4/1/15 | 0.0030 | | 43.8 | 13 = R |
| 9 - Hexanone | 4/1/15 | | | 50.2 | 13 = J |
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* % Difference = $((RF_{CCV} - RF_{ICAL\ AVG}) / RF_{ICAL\ AVG}) \times 100$. 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 $\leq 25\%D$? (Y/N) _____

Are 4, 4'- DDT and endrin breakdown (PEM) $\leq 20\%$ and combined breakdown $\leq 30\%$ (Y/N) _____

Deviations:

| Compound | %RSD | RPD | Samples Affected |
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* % Difference = $((RF_{CCV} - RF_{ICAL AVG})/RF_{ICAL AVG}) \times 100$. 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: Project sample not spiked.

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:

180-137993/8

Deviations:

| Compound | Date | %R | Samples Affected/Qualifiers Applied |
|-----------------------------|--------|-----|-------------------------------------|
| 1,4 - Dioxane | 4/1/15 | 147 | None |
| 1,1,2,2 - Tetrachloroethane | | 123 | ↓ |
| trans-1,2 - Dichloroethane | | 78 | |
| Methylene Chloride | | 77 | |
| 1,1,1 - Trichloroethane | | 75 | |
| Carbon Tetrachloride | | 75 | |
| Carbon Dioxide | | 67 | |
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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 values 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 values 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

180-42445-9

| Sample Number | Method | Date Collected | Analysis Date | Date Extracted | Days to Analysis |
|---------------|-------------|----------------|---------------|----------------|------------------|
| 180-42445-13 | SW846 8260C | 3/26/2015 | 4/1/2015 | | 6 |
| 180-42445-14 | SW846 8260C | 3/26/2015 | 4/1/2015 | | 6 |

Trip Blank Detections

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