# CHAAP Data Verification

**Laboratory and SDG#: TADenver 280-119704** **AECOM Chemist: Jared DeSadier**
**Date Verified: 2/25/2019** **AECOM ITR: Jeff Aust**
**Guidance: DoD QSM Version 5.1 (January 2017)**
**Applicable QAPP: Cornhusker Army Ammunition Plant QAPP (Brice and AECOM, October 2018)**
**Applicable Analytical Methods: 8260B, 8330A, 6020A, SM2540D, 9040C**

| Sample Identification # | Date Collected | Date Received | Matrix | Analysis |
|---|---|---|---|---|
| TB-1036 | 1/30/2019 | 1/31/2019 | Water | VOCs (8260B) |
| SP-S2 | 1/30/2019 | 1/31/2019 | Water | VOCs (8260B), Explosives (8330A), TSS (SM2540D), pH (9040C) |
| SP-E1 | 1/30/2019 | 1/31/2019 | Water | VOCs (8260B), Explosives (8330A), Metals (6020A), pH (9040C) |
| SP-E11 | 1/30/2019 | 1/31/2019 | Water | Metals (6020A) |
| SP-S22 | 1/30/2019 | 1/31/2019 | Water | VOCs (8260B), Explosives (8330A), TSS (SM2540D), pH (9040C) |
| SP-S6 | 1/30/2019 | 1/31/2019 | Water | Explosives (8330A) |
| SP-S8 | 1/30/2019 | 1/31/2019 | Water | Explosives (8330A) |

## 1.0 Laboratory Case Narrative \ Cooler Receipt Form

| Verification Criteria | Yes | No | N/A |
|---|---|---|---|
| Were any DoD QSM deviations noted in the laboratory case narrative? | X | | |
| Were DoD QSM corrective actions followed if deviations were noted? | X | | |
| Were any issues noted in the cooler receipt form? | | X | |

The laboratory case narrative indicated LCS RPDs were above evaluation criteria for some VOCs. Results were not qualified based on RPD alone.

The case narrative also indicated samples SP-E1 and SP-S22 had a pH above 2 for VOCs and that the RPD between the primary and confirmation columns for some explosives results were above evaluation criteria. These issues are discussed further in Section 15.0.

The cooler receipt form indicated explosives analysis was subcontracted to TestAmerica Sacramento.

No other issues were noted in the case narrative or cooler receipt form.

## 2.0 Sample Documentation

| Verification Criteria | Yes | No |
|---|---|---|
| Were all samples documented correctly on the chain-of-custody (COC) and samples labels? | X | |
| Were all sample identifications (IDs) documented correctly on sample labels? | X | |
| Did samples listed on COCs match the sample labels? | X | |
| Were samples relinquished properly on the COC? | X | |

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## 3.0 Instrument Performance Check (Tuning)

| Method 8260B Instrument Tuning Criteria (Filename) | R0482.D | | |
|---|---|---|---|
| Instrument: | VMS_R1 | | |
| Date of Tuning: | 2/6/2019 | | |
| | Yes | No | N/A |
| Was instrument tuning completed prior to calibration? | X | | |
| Were all samples analyzed under an acceptable 12 hour clock tune? | X | | |
| Were ion relative abundances for each target mass within the required intensity limits listed in Table 4 of SW-846 Method 8260B? | X | | |

| Method 8260B Instrument Tuning Criteria (Filename) | R0551.D | | |
|---|---|---|---|
| Instrument: | VMS_R1 | | |
| Date of Tuning: | 2/7/2019 | | |
| | Yes | No | N/A |
| Was instrument tuning completed prior to calibration? | X | | |
| Were all samples analyzed under an acceptable 12 hour clock tune? | X | | |
| Were ion relative abundances for each target mass within the required intensity limits listed in Table 4 of SW-846 Method 8260B? | X | | |

| Method 6020A Instrument Tuning Criteria (Date) | li020519.b | | |
|---|---|---|---|
| Instrument: | MT_077 | | |
| Date of Tuning: | 2/5/2019 | | |
| | Yes | No | N/A |
| Was instrument tuning completed prior to calibration? | X | | |
| Was mass calibration ≤ 0.1 amu from true value? | X | | |
| Was resolution < 0.9 amu full width at 10% peak height? | X | | |
| For stability, RSD was ≤ 5% for at least four replicate analytes? | X | | |

## 4.0 Initial Calibration

| Method 8260B Initial Calibration Criteria | | | |
|---|---|---|---|
| Instrument: | VMS_R1 | | |
| Date of Calibration: | 2/6/2019 | | |
| | Yes | No | N/A |
| Option 1: RSD for each analyte ≤ 15%? | X | | |
| Option 2: If linear least squares regression was used was the $r^2 \geq 0.99$? | X | | |
| Option 3: If non-linear regression was used was the coefficient of determination $r^2 \geq 0.99$? | | | X |
| If non-linear regression was used were 6 points used for second order and 7 points for third order? | | | X |

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| **Method 8260B Initial Calibration Criteria** | | | |
|---|---|---|---|
| **Instrument:** | **VMS_R1** | | |
| **Date of Calibration:** | **2/7/2019** | | |
| | **Yes** | **No** | **N/A** |
| Option 1: RSD for each analyte ≤ 15%? | X | | |
| Option 2: If linear least squares regression was used was the $r^2 \geq 0.99$? | X | | |
| Option 3: If non-linear regression was used was the coefficient of determination $r^2 \geq 0.99$? | | | X |
| If non-linear regression was used were 6 points used for second order and 7 points for third order? | | | X |

| **Method 6020A Initial Calibration Criteria** | | | |
|---|---|---|---|
| **Instrument:** | **MT_077** | | |
| **Date of Calibration:** | **2/5/2019** | | |
| | **Yes** | **No** | **N/A** |
| Was a minimum of two standards and a calibration blank used for ICAL? | X | | |
| Was $r^2 \geq 0.99$ for all target metals? | X | | |

| **Method 8330A Initial Calibration Criteria** | | | |
|---|---|---|---|
| **Instrument:** | **LC9** | | |
| **Date of Calibration:** | **1/30/2019** | | |
| | **Yes** | **No** | **N/A** |
| Was at least a five point calibration completed for all analytes prior to sample analysis and one option below? | X | | |
| Option 1: RSD for each analyte ≤ 15%? | X | | |
| Option 2: If linear least squares regression was used was the $r^2 \geq 0.99$? | X | | |
| Option 3: If non-linear regression was used was the coefficient of determination $r^2 \geq 0.99$? | | | X |
| If non-linear regression was used were 6 points used for second order and 7 points for third order? | | | X |

| **Method 8330A Initial Calibration Criteria** | | | |
|---|---|---|---|
| **Instrument:** | **LC12** | | |
| **Date of Calibration:** | **12/6/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was at least a five point calibration completed for all analytes prior to sample analysis and one option below? | X | | |
| Option 1: RSD for each analyte ≤ 15%? | X | | |
| Option 2: If linear least squares regression was used was the $r^2 \geq 0.99$? | | | X |
| Option 3: If non-linear regression was used was the coefficient of determination $r^2 \geq 0.99$? | | | X |
| If non-linear regression was used were 6 points used for second order and 7 points for third order? | | | X |

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| **Method 8330A Initial Calibration Criteria** | | | |
|---|---|---|---|
| **Instrument:** | **LC12** | | |
| **Date of Calibration:** | **12/7/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was at least a five point calibration completed for all analytes prior to sample analysis and one option below? | X | | |
| Option 1: RSD for each analyte ≤ 15%? | X | | |
| Option 2: If linear least squares regression was used was the $r^2 \geq 0.99$? | | | X |
| Option 3: If non-linear regression was used was the coefficient of determination $r^2 \geq 0.99$? | | | X |
| If non-linear regression was used were 6 points used for second order and 7 points for third order? | | | X |

| **Method 8330A Initial Calibration Criteria** | | | |
|---|---|---|---|
| **Instrument:** | **LC11** | | |
| **Date of Calibration:** | **1/22/2019** | | |
| | **Yes** | **No** | **N/A** |
| Was at least a five point calibration completed for all analytes prior to sample analysis and one option below? | X | | |
| Option 1: RSD for each analyte ≤ 15%? | X | | |
| Option 2: If linear least squares regression was used was the $r^2 \geq 0.99$? | | | X |
| Option 3: If non-linear regression was used was the coefficient of determination $r^2 \geq 0.99$? | | | X |
| If non-linear regression was used were 6 points used for second order and 7 points for third order? | | | X |

| **Method 8330A Initial Calibration Criteria** | | | |
|---|---|---|---|
| **Instrument:** | **LC11** | | |
| **Date of Calibration:** | **1/22/2019** | | |
| | **Yes** | **No** | **N/A** |
| Was at least a five point calibration completed for all analytes prior to sample analysis and one option below? | X | | |
| Option 1: RSD for each analyte ≤ 15%? | X | | |
| Option 2: If linear least squares regression was used was the $r^2 \geq 0.99$? | X | | |
| Option 3: If non-linear regression was used was the coefficient of determination $r^2 \geq 0.99$? | | | X |
| If non-linear regression was used were 6 points used for second order and 7 points for third order? | | | X |

## 5.0 Initial Calibration Verification [(ICV) Second Source]

| **Method 8260B ICV Criteria (Filename)** | **R0492.D** | | |
|---|---|---|---|
| **Instrument:** | **VMS_R1** | | |
| **Date of Initial Calibration Verification:** | **2/6/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was the ICV analyzed after each calibration? | X | | |
| Were all reported analytes within ± 20% of true value? | X | | |

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| **Method 8260B ICV Criteria (Filename)** | **R0563.D** | | |
|---|---|---|---|
| **Instrument:** | **VMS_R1** | | |
| **Date of Initial Calibration Verification:** | **2/7/2019** | | |
| | **Yes** | **No** | **N/A** |
| Was the ICV analyzed after each calibration? | X | | |
| Were all reported analytes within ± 20% of true value? | X | | |

| **Method 6020A ICV Criteria (Date)** | **2/5/2019 12:21** | | |
|---|---|---|---|
| **Instrument:** | **MT_077** | | |
| | **Yes** | **No** | **N/A** |
| Was the ICV analyzed after each ICAL, prior to the beginning of a sample analysis? | X | | |
| Were all reported analytes within ± 10% of true value? | X | | |

| **Method 8330A ICV Criteria (Filename)** | **ZDA000010.D** | | |
|---|---|---|---|
| **Instrument:** | **LC9** | | |
| **Date of Initial Calibration Verification:** | **1/31/2019** | | |
| | **Yes** | **No** | **N/A** |
| Was the ICV analyzed after each calibration? | X | | |
| Was the ICV for all analytes within ± 15% of the true value? | X | | |

| **Method 8330A ICV Criteria (Filename)** | **F1000022.D** | | |
|---|---|---|---|
| **Instrument:** | **LC12** | | |
| **Date of Initial Calibration Verification:** | **12/7/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was the ICV analyzed after each calibration? | X | | |
| Was the ICV for all analytes within ± 15% of the true value? | X | | |

| **Method 8330A ICV Criteria (Filename)** | **F1000024.D** | | |
|---|---|---|---|
| **Instrument:** | **LC12** | | |
| **Date of Initial Calibration Verification:** | **12/7/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was the ICV analyzed after each calibration? | X | | |
| Was the ICV for all analytes within ± 15% of the true value? | X | | |

| **Method 8330A ICV Criteria (Filename)** | **V0000021.D** | | |
|---|---|---|---|
| **Instrument:** | **LC11** | | |
| **Date of Initial Calibration Verification:** | **1/23/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was the ICV analyzed after each calibration? | X | | |
| Was the ICV for all analytes within ± 15% of the true value? | X | | |

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| Method 8330A ICV Criteria (Filename) | V0000023.D | | |
|---|---|---|---|
| Instrument: | LC11 | | |
| Date of Initial Calibration Verification: | 1/23/2018 | | |
| | Yes | No | N/A |
| Was the ICV analyzed after each calibration? | X | | |
| Was the ICV for all analytes within ± 15% of the true value? | X | | |

## 6.0 Continuing Calibration Verification (CCV)

| Method 8260B Beginning CCV Criteria (Filename) | R0563.D | | |
|---|---|---|---|
| Method 8260B Ending CCV Criteria (Filename) | R0582.D | | |
| Instrument: | VMS_R1 | | |
| Date of Calibration Verification: | 2/7/2019 | | |
| | Yes | No | N/A |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 12 hours of analysis time? | X | | |
| Were all reported analytes and surrogates within ± 20% of true value? | X | | |
| Were all reported analytes and surrogates within ± 50% of true value for the end of analytical batch CCV? | X | | |

| Method 6020A CCV Criteria (Date) | All CCVs on 2/5/2019 | | |
|---|---|---|---|
| Instrument: | MT_077 | | |
| | Yes | No | N/A |
| Were the CCVs analyzed after every 10 samples and at the end of the analysis sequence? | X | | |
| Were all reported analytes within ± 10% of true value? | X | | |

| Method 8330A CCV Criteria (Filename) | H000003.D | | |
|---|---|---|---|
| Instrument: | LC9 | | |
| Date of Calibration Verification: | 2/8/2019 | | |
| | Yes | No | N/A |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | X | | |

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| **Method 8330A CCV Criteria (Filename)** | **H000010.D** | | |
|---|---|---|---|
| **Instrument:** | **LC9** | | |
| **Date of Calibration Verification:** | **2/9/2019** | | |
| | **Yes** | **No** | **N/A** |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | X | | |

| **Method 8330A CCVRT Criteria (Filename)** | **E000003_4.D** | | |
|---|---|---|---|
| **Instrument:** | **LC12** | | |
| **Date of Calibration Verification:** | **2/7/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | | X | |

The %Ds for 3-nitrotoluene (15.2%) and tetryl (16.6%) were outside of evaluation criteria. The CCV RFs were greater than the mean RFs, indicating potentially high biases. The compounds 3-nitrotoluene and tetryl were not detected in any associated samples and no qualification of data was required.

| **Method 8330A CCV Criteria (Filename)** | **E000016_17.D** | | |
|---|---|---|---|
| **Instrument:** | **LC12** | | |
| **Date of Calibration Verification:** | **2/8/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | | X | |

The %D for tetryl (22.7%) was outside of evaluation criteria. The CCV RF was greater than the mean RF, indicating a potentially high bias. Tetryl was not detected in any associated samples and no qualification of data was required.

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| **Method 8330A CCV Criteria (Filename)** | **E000025.D** | | |
|---|---|---|---|
| **Instrument:** | **LC12** | | |
| **Date of Calibration Verification:** | **2/8/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | X | | |

| **Method 8330A CCV Criteria (Filename)** | **I000001.D** | | |
|---|---|---|---|
| **Instrument:** | **LC12** | | |
| **Date of Calibration Verification:** | **2/9/2018** | | |
| | **Yes** | **No** | **N/A** |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | | X | |

The %Ds for 2,6-dinitrotoluene (-15.8%) and tetryl (23.9%) were outside of evaluation criteria. The CCV RFs were greater than the mean RFs, indicating potential high biases. The compounds 2,6-nitrotoluene and tetryl were not detected in any associated samples and no qualification of data was required.

| **Method 8330A CCV Criteria (Filename)** | **F0000029_30.D** | | |
|---|---|---|---|
| **Instrument:** | **LC11** | | |
| **Date of Calibration Verification:** | **2/7/2019** | | |
| | **Yes** | **No** | **N/A** |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | X | | |

| **Method 8330A CCV Criteria (Filename)** | **F0000044_45.D** | | |
|---|---|---|---|
| **Instrument:** | **LC11** | | |
| **Date of Calibration Verification:** | **2/8/2019** | | |
| | **Yes** | **No** | **N/A** |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | X | | |

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| Method 8330A CCV Criteria (Filename) | F0000049_50.D | | |
|---|---|---|---|
| Instrument: | LC11 | | |
| Date of Calibration Verification: | 2/8/2019 | | |
| | Yes | No | N/A |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | X | | |

| Method 8330A CCVRT Criteria (Filename) | F0000003_4.D | | |
|---|---|---|---|
| Instrument: | LC11 | | |
| Date of Calibration Verification: | 2/9/2019 | | |
| | Yes | No | N/A |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | X | | |

| Method 8330A CCV Criteria (Filename) | F0000008_9.D | | |
|---|---|---|---|
| Instrument: | LC11 | | |
| Date of Calibration Verification: | 2/9/2019, 2/10/2019 | | |
| | Yes | No | N/A |
| Was the CCV analyzed daily before sample analysis? | X | | |
| Was the CCV analyzed every 10 field samples and at the end of the analysis sequence? | X | | |
| Was the CCV for all analytes within ± 15% of the true value? | X | | |

## 7.0 Blank Samples

| Blank Criteria | Yes | No | N/A |
|---|---|---|---|
| Were method blanks analyzed with every preparatory batch? | X | | |
| Were target analytes detected > ½ the LOQ and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater)? | | X | |
| Were target analytes detected in method, trip or calibration blanks? | | X | |

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## 8.0 Internal Standard (IS) Recoveries

| Methods 8260B Criteria | Yes | No | N/A |
|---|---|---|---|
| Were internal standards spiked for all samples and standards? | X | | |
| Were internal standard areas within -50% to + 100% of the ICAL midpoint standard area? | X | | |
| Were retention time ± 30 seconds from the retention time of the midpoint standard of the ICAL? | X | | |

| Method 6020A IS Criteria | Yes | No | N/A |
|---|---|---|---|
| Were internal standard intensities within 30-120% of intensity of the IS in the ICAL? | X | | |

## 9.0 Matrix Duplicate

| Matrix Duplicate (MD) Criteria | Yes | No | N/A |
|---|---|---|---|
| Were MD samples analyzed with every preparatory batch? | X | | |
| Were MD samples collected for this SDG? | | X | |
| Were MD RPDs within acceptance criteria listed in the UFP-QAPP? | | | X |

## 10.0 Dilution Test

| Method 6020A Dilution Test Criteria | Yes | No | N/A |
|---|---|---|---|
| Was a dilution test sample analyzed with every preparatory batch? | X | | |
| Was a dilution test sample analyzed from this SDG? | X | | |
| Were metals concentrations > 50x the LOQ? | X | | |
| Did the five-fold dilution agree within ± 10% of the original measurement? | X | | |
| If the five-fold dilution did not agree within ± 10% of the original measurement, was a post digestion spike sample analyzed? | | | X |

Sample SP-E1 was diluted and analyzed for selenium.

## 11.0 Post Digestion Spike (PDS) Recoveries

| Method 6020A PDS Criteria | Yes | No | N/A |
|---|---|---|---|
| Was a PDS sample analyzed from this SDG? | X | | |
| Was a PDS sample analyzed if the dilution test failed or metals concentrations were > 50 x the LOD? | X | | |
| Were the PDS recoveries within 80-120%? | X | | |

Sample SP-E1 was spiked and analyzed for selenium.

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## 12.0 Interference Check Solutions (ICS)

| Method 6020A ICS Criteria | Yes | No | N/A |
|---|---|---|---|
| Were ICS-A and ICSAB samples analyzed at the beginning of the analytical run and every 12 hours? | X | | |
| Was the ICS-A absolute value concentration for all non-spiked metals < LOD (unless they are a verified trace impurity form one of the spiked metals) | X | | |
| Were the ICS-AB recoveries within ± 20%? | X | | |

## 13.0 Field Duplicate Samples

| Field Duplicate Criteria | Yes | No | N/A |
|---|---|---|---|
| Were field duplicate samples collected for this SDG? (if yes, list below) | X | | |
| Were parent sample / field duplicate RPDs ≤ 30% for water samples and ≤ 50% for soils for analytes that had concentrations > 5x the LOQ? | X | | |
| Were the differences between the parent sample / field duplicate < 2x the LOQ for analytes that had concentrations < 5x the LOQ? | X | | |

| Parent Sample ID | Field Duplicate Sample ID |
|---|---|
| SP-S2 | SP-E22 |
| SP-E1 | SP-E11 |

## 14.0 Sensitivity

| Sensitivity Criteria | Yes | No | N/A |
|---|---|---|---|
| Was the laboratory sensitivity consistent with project (QAPP) requirements? | X | | |
| Did all analytes meet sensitivity requirements? | X | | |

## 15.0 Additional Qualifications

| Additional Qualification Criteria | Yes | No | N/A |
|---|---|---|---|
| Were common laboratory contaminants detected? | | X | |
| Was professional judgment used to qualify data (if yes, list below) | X | | |

Samples SP-E1 and SP-S22 had pH levels above 2 for VOC analysis. Samples were analyzed outside the holding time for unpreserved samples (7 days) but within holding time for preserved samples (14 days). Qualification of data is shown in the table below.

| Sample ID | Analysis | Analyte | Qualifications |
|---|---|---|---|
| SP-E1 | VOC | All VOCs | **J/UJ** |
| SP-S22 | VOC | All VOCs | **J/UJ** |

The RPD between the primary and confirmation column for some explosives samples was above evaluation criteria. Qualification of data is shown in the table below.

| Sample ID | Analysis | Analyte | RPD | Qualifications |
|---|---|---|---|---|
| SP-S2 | Explosives | RDX | **181.2** | **J** |
| SP-S22 | Explosives | 2,4-dinitrotoluene | **49.7** | **J** |
| SP-S8 | Explosives | RDX | **40.2** | **J** |

## 16.0 Completeness

| Completeness Criteria | Yes | No | N/A |
|---|---|---|---|
| Were any data rejected during the verification process? | | X | |
| Were any samples lost, broken, or in any other manner in not verified? | | X | |
| Were samples analyses requested performed, the correct analyte lists used and correct sample preparation and analyses methods and units utilized? | X | | |