

CHAAP Data Verification

Laboratory and SDG#: TADenver 280-130115

AECOM Chemist: Jared DeSadier

Date Verified: 11/21/2019

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
SP-S2	7/31/2019	8/1/2019	Water	VOCs (8260B), Explosives (8330A), TSS (SM2540D), pH (9040C)
SP-E1	7/31/2019	8/1/2019	Water	VOCs (8260B), Explosives (8330A), Metals (6020A), pH (9040C)
SP-E11	7/31/2019	8/1/2019	Water	Metals (6020A)
SP-S22	7/31/2019	8/1/2019	Water	VOCs (8260B), Explosives (8330A), TSS (SM2540D), pH (9040C)
SP-S6	7/31/2019	8/1/2019	Water	Explosives (8330A)
SP-S8	7/31/2019	8/1/2019	Water	Explosives (8330A)
TB-1074	7/31/2019	8/1/2019	Water	VOCs (8260B)

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 case narrative indicated that some ICV and CCV %Ds were outside of evaluation criteria. These issues are discussed further in Sections 5.0 and 6.0. Sample SP-S2 was analyzed with headspace greater than 6 mm in some sample VOAs. The RPD between the primary and confirmation column for some explosives samples was above evaluation criteria. These issues are discussed further in Section 13.0.

Some LCS and surrogate recoveries and hold times were outside evaluation criteria. These issues are discussed further in the ADR report.

The cooler receipt form indicated a discrepancy between the type of some sample containers received by lab and the COC. Sufficient volume was received in the containers and samples were properly preserved; no qualification of data was required.

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

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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	

3.0 Instrument Performance Check (Tuning)

Method 8260B Instrument Tuning Criteria (Filename)	MS1_52396.D		
Instrument:	VMS_MS1		
Date of Tuning:	10/28/2019		
	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)	MS1_52410.D		
Instrument:	VMS_MS1		
Date of Tuning:	10/29/2019		
	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)	MS1_52720.D		
Instrument:	VMS_MS1		
Date of Tuning:	11/4/2019		
	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		

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Method 6020A Instrument Tuning Criteria (Date)		LI110819.b		
Instrument:		MT_077		
Date of Tuning:		11/8/2019		
		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 $\leq 5\%$ for at least four replicate analytes?		X		

4.0 Initial Calibration

Method 8260B Initial Calibration Criteria				
Instrument:		VMS_MS1		
Date of Calibration:		10/28/2019		
		Yes	No	N/A
Option 1: RSD for each analyte $\leq 15\%$?		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 8260B Initial Calibration Criteria				
Instrument:		VMS_MS1		
Date of Calibration:		10/29/2019		
		Yes	No	N/A
Option 1: RSD for each analyte $\leq 15\%$?		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:		11/8/2019		
		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		

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Method 8330A Initial Calibration Criteria			
Instrument:	CHHPLC_X3		
Date of Calibration:	7/1/2019		
	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 $\leq 20\%$?	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:	CHHPLC_X3		
Date of Calibration:	7/1/2019		
	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 $\leq 20\%$?	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:	CHHPLC_G2_LUNA		
Date of Calibration:	10/25/2019		
	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 $\leq 20\%$?	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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Applicable Analytical Methods: 8260B, 8330A, 6020A, SM2540D, 9040C

Method 8330A Initial Calibration Criteria			
Instrument:	CHHPLC_G2_LUNA		
Date of Calibration:	11/6/2019		
	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 $\leq 20\%$?	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:	CHHPLC_G2_LUNA		
Date of Calibration:	11/15/2019		
	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 $\leq 20\%$?	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:	CHHPLC_G2_LUNA		
Date of Calibration:	11/18/2019		
	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 $\leq 20\%$?	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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5.0 Initial Calibration Verification [(ICV) Second Source]

Method 8260B ICV Criteria (Filename)	MS1_52407.D		
Instrument:	VMS MS1		
Date of Initial Calibration Verification:	10/28/2019		
	Yes	No	N/A
Was the ICV analyzed after each calibration?	X		
Were all reported analytes within $\pm 20\%$ of true value?		X	

The %D for dichlorodifluoromethane (20.7%) was outside of evaluation criteria with a high bias. All associated results were nondetect and no qualification of data was required.

Method 8260B ICV Criteria (Filename)	MS1_52421.D		
Instrument:	VMS MS1		
Date of Initial Calibration Verification:	10/29/2019		
	Yes	No	N/A
Was the ICV analyzed after each calibration?	X		
Were all reported analytes within $\pm 20\%$ of true value?	X		

Method 6020A ICV Criteria (Date)	11/8/2019 16:21		
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 $\pm 10\%$ of true value?	X		

Method 8330A ICV Criteria (Filename)	07010015.D		
Instrument:	CHHPLC X3		
Date of Initial Calibration Verification:	7/1/2019		
	Yes	No	N/A
Was the ICV analyzed after each calibration?	X		
Was the ICV for all analytes within $\pm 15\%$ of the true value?	X		

Method 8330A ICV Criteria (Filename)	07010033.D		
Instrument:	CHHPLC X3		
Date of Initial Calibration Verification:	7/2/2019		
	Yes	No	N/A
Was the ICV analyzed after each calibration?	X		
Was the ICV for all analytes within $\pm 15\%$ of the true value?	X		

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Method 8330A ICV Criteria (Filename)	10250015.D		
Instrument:	CHHPLC_G2_ LUNA		
Date of Initial Calibration Verification:	10/26/2019		
	Yes	No	N/A
Was the ICV analyzed after each calibration?	X		
Was the ICV for all analytes within $\pm 15\%$ of the true value?		X	

The %D for 3-nitrotoluene (33.4%) was outside of evaluation criteria with a high bias. Associated results were nondetect and no qualification of data was required.

Method 8330A ICV Criteria (Filename)	11060015.D		
Instrument:	CHHPLC_G2_ LUNA		
Date of Initial Calibration Verification:	11/6/2019		
	Yes	No	N/A
Was the ICV analyzed after each calibration?	X		
Was the ICV for all analytes within $\pm 15\%$ of the true value?	X		

Method 8330A ICV Criteria (Filename)	11180015.D		
Instrument:	CHHPLC_G2_ LUNA		
Date of Initial Calibration Verification:	11/18/2019		
	Yes	No	N/A
Was the ICV analyzed after each calibration?	X		
Was the ICV for all analytes within $\pm 15\%$ of the true value?	X		

6.0 Continuing Calibration Verification (CCV)

Method 8260B Beginning CCV Criteria (Filename)	MS1_52722.D		
Method 8260B Ending CCV Criteria (Filename)	MS1_52750.D		
Instrument:	VMS MS1		
Date of Calibration Verification:	11/4/2019		
	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 $\pm 20\%$ of true value?	X		
Were all reported analytes and surrogates within $\pm 50\%$ of true value for the end of analytical batch CCV?	X		

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Method 6020A CCV Criteria (Date)	All CCVs on 11/8/2019		
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 $\pm 10\%$ of true value?	X		

Method 8330A CCV Criteria (Filename)	10310040 2.D		
Instrument:	CHHPLC X3		
Date of Calibration Verification:	11/1/2019		
	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 $\pm 15\%$ of the true value?	X		

Method 8330A CCV Criteria (Filename)	10310053 5.D		
Instrument:	CHHPLC X3		
Date of Calibration Verification:	11/1/2019		
	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 $\pm 15\%$ of the true value?	X		

Method 8330A CCV Criteria (Filename)	11040007 9.D		
Instrument:	CHHPLC X3		
Date of Calibration Verification:	11/4/2019		
	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 $\pm 15\%$ of the true value?	X		

Method 8330A CCV Criteria (Filename)	11040020 2.D		
Instrument:	CHHPLC X3		
Date of Calibration Verification:	11/4/2019		
	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 $\pm 15\%$ of the true value?	X		

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Method 8330A CCV Criteria (Filename)	11040026.D		
Instrument:	CHHPLC_G2_ LUNA		
Date of Calibration Verification:	11/5/2019		
	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 $\pm 15\%$ of the true value?		X	

The %D for 3-nitrotoluene (63.9%) was outside of evaluation criteria with a high bias and all associated results were nondetect. No qualification of data was required.

Method 8330A CCV Criteria (Filename)	11040036.D		
Instrument:	CHHPLC_G2_ LUNA		
Date of Calibration Verification:	11/5/2019		
	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 $\pm 15\%$ of the true value?	X		

Method 8330A CCV Criteria (Filename)	11080025_6.D		
Instrument:	CHHPLC_G2_ LUNA		
Date of Calibration Verification:	11/9/2019		
	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 $\pm 15\%$ of the true value?	X		

Method 8330A CCV Criteria (Filename)	11080037_8.D		
Instrument:	CHHPLC_G2_ LUNA		
Date of Calibration Verification:	11/9/2019		
	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 $\pm 15\%$ of the true value?	X		

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Method 8330A CCV Criteria (Filename)	11180016.D		
Instrument:	CHHPLC_G2_ LUNA		
Date of Calibration Verification:	11/18/2019		
	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 $\pm 15\%$ of the true value?	X		

Method 8330A CCV Criteria (Filename)	11180029_30.D		
Instrument:	CHHPLC_G2_ LUNA		
Date of Calibration Verification:	11/18/2019		
	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 $\pm 15\%$ 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 $> \frac{1}{2}$ the LOQ and $> \frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater)?		X	
Were target analytes detected in method, trip or calibration blanks?		X	

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		

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9.0 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 $\pm 10\%$ of the original measurement?			X
If the five-fold dilution did not agree within $\pm 10\%$ of the original measurement, was a post digestion spike sample analyzed?			X

10.0 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

11.0 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 $\pm 20\%$?	X		

12.0 Sensitivity

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

13.0 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		

There was headspace larger than 6mm in some sample VOAs. Qualification of data is provided in the table below.

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Field ID	Parameter	Analyte	Qualification
SP-S2	VOCs	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	Qual
SP-S6	Explosives	2-amino-dinitrotoluene	86.0	UJ
SP-S22	Explosives	RDX	42.8	UJ

14.0 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		