

CHAAP Data Verification

Laboratory and SDG#: Eurofins 280-162386

AECOM Chemist: D. Casagrande

Date Verified: 7/20/2022

AECOM ITR: S. Louie

Guidance: DoD QSM Version 5.1 (January 2017)

Applicable QAPP: Cornhusker Army Ammunition Plant QAPP (Brice and AECOM, October 2019)

Applicable Analytical Methods: 8330A, 353.2, 350.1, 351.2, RSK-175, 9060A, 2320B, 9056A, 9034

Sample Identification #	Date Collected	Date Received	Matrix	Analysis
PZ007-22A	5/13/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)
G0087-22A	5/13/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)
G0117-22A	5/13/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)
G0022-22A	5/13/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)
G0088-22A	5/13/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)
G0116-22A	5/13/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)
PZ014-22A	5/13/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)
G0092-22A	5/12/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)
PZ013-22A	5/13/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)
G0081-22A	5/13/2022	5/14/2022	Water	Explosives (8330A), Nitrate, Nitrite (353.2), Ammonia (350.1), TKN (351.2), Methane (RSK-175), DOC (9060A), Sulfate (9056A), Alkalinity (2320B), Sulfide (9034)

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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		

Validator comments in italics.

The container label for the following samples did not match the information listed on the Chain-of-Custody (COC): PZ007-22A (280-162386-1), PZ007-22A (280-162386-1[MS]), PZ007-22A (280-162386-1[MSD]), G0117-22A (280-162386-3), G0116-22A (280-162386-6), PZ014-22A (280-162386-7) and PZ013-22A (280-162386-9). The container labels lists the sample collection date as 06/13/2022, while the COC lists 05/13/2022. Logged per the COC. *This issue is discussed further in Section 2.0.*

One out of three VOA vials received does not have the sample collection date or time recorded on the client label. Logged per the COC. PZ007-22A (280-162386-1[MS]). The container label for the following sample did not match the information listed on the Chain-of-Custody (COC): G0092-22A (280-162386-8). The container labels lists the sample collection date as 6/12/22, while the COC lists 05/12/2022. Logged per the COC. *This issue is discussed further in Section 2.0.*

Method RSK-175:

The following sample(s) was collected in a properly preserved vial; however, the pH was 7 and outside the required criteria when verified by the laboratory. The sample was analyzed outside the 7-day holding time specified for unpreserved samples but within the 14-day holding time specified for preserved samples: G0116-22A (280-162386-6). *This issue is discussed further in Section 7.0.*

Method 8330A:

Surrogate recovery for the following sample in preparation batch 280-575516 and analytical batch 280-575712 for method 8330 was outside the lower control limits: G0116-22A (280-162386-6). Evidence of matrix interference is present; therefore, re-extraction and/or re-analysis was not performed. Surrogate recovered within control limit in the primary instrument. *No analytes detected in sample G0116-22A, therefore results qualified (UJ). This issue is further discussed in the ADR report.*

Method 2320B:

Total Alkalinity as CaCO₃ was detected in method blank MB 280-576106/32 at a level that was above one half the LOQ but below the LOQ. Associated sample results are greater than 10x the method blank concentration. *No data are considered affected or qualified.*

Method 351.2:

Nitrogen, Total Kjeldahl failed the recovery criteria high for the MS of sample PZ007-22AMS (280-162386-1) in batch 280-576217. Nitrogen, Total Kjeldahl failed the recovery criteria high for the MSD of sample PZ007-22AMSD (280-162386-1) in batch 280-576217. *Since the MS/MSD was recovered above the control limit and TKN was not detected in sample G0098-22A, no data are affected or qualified. This issue is further discussed in the ADR report.*

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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	

The container label for the following samples did not match the information listed on the Chain-of-Custody (COC): PZ007-22A (280-162386-1), PZ007-22A (280-162386-1[MS]), PZ007-22A (280-162386-1[MSD]), G0117-22A (280-162386-3), G0116-22A (280-162386-6), PZ014-22A (280-162386-7) and PZ013-22A (280-162386-9). The container labels lists the sample collection date as 06/13/2022, while the COC lists 05/13/2022. Per the AECOM chemist, the sample was logged via the time on the COC and no qualification of data was required.

One out of three VOA vials received does not have the sample collection date or time recorded on the client label. Logged per the COC. PZ007-22A (280-162386-1[MS]) The container label for the following sample did not match the information listed on the Chain-of-Custody (COC): G0092-22A (280-162386-8). The container labels lists the sample collection date as 6/12/22, while the COC lists 05/12/2022. Per the AECOM chemist, the sample was logged via the date and time on the COC and no qualification of data was required.

3.0 Initial Calibration

Method 8330A Initial Calibration Criteria			
Instrument:	CHHPLC X3		
Date of Calibration:	1/4/2022		
	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

%RSD was met for all target analytes.

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Method 8330A Initial Calibration Criteria			
Instrument:	CHHPLC_X3		
Date of Calibration:	1/5/2022		
	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_X5		
Date of Calibration:	3/2/2022		
	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_X5		
Date of Calibration:	3/3/2022		
	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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Method RSK-175 Initial Calibration Criteria			
Instrument:	VGC J		
Date of Calibration:	9/24/2021		
	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 $\leq 25\%$?			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

No %RSD was provided for methane; Methane met the r2 criteria.

Method 9056A Initial Calibration Criteria			
Instrument:	WC IonChrom11		
Date of Calibration:	5/26/2022		
	Yes	No	N/A
Was a minimum of three standards and a calibration blank used for ICAL?	X		
Was $r^2 \geq 0.99$?	X		

Method 9056A Initial Calibration Criteria			
Instrument:	WC IonChrom13		
Date of Calibration:	5/16/2022		
	Yes	No	N/A
Was a minimum of three standards and a calibration blank used for ICAL?	X		
Was $r^2 \geq 0.99$?	X		

Method 350.1 Initial Calibration Criteria			
Instrument:	WC_SKALAR_01		
Date of Calibration:	6/1/2022		
	Yes	No	N/A
Was a minimum of three standards and a calibration blank used for ICAL?	X		
Was $r^2 \geq 0.99$?	X		

Method 353.2 Initial Calibration Criteria			
Instrument:	WC Alp 2		
Date of Calibration:	6/2/2022		
	Yes	No	N/A
Was a minimum of three standards and a calibration blank used for ICAL?	X		
Was $r^2 \geq 0.99$?	X		

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Method 351.2 Initial Calibration Criteria			
Instrument:	WC_GAL1		
Date of Calibration:	5/25/2022		
	Yes	No	N/A
Was a minimum of three standards and a calibration blank used for ICAL?	X		
Was $r^2 \geq 0.99$?	X		

Method 9060A Initial Calibration Criteria			
Instrument:	WC_SHI5		
Date of Calibration:	12/10/2021		
	Yes	No	N/A
Was a minimum of three standards and a calibration blank used for ICAL?	X		
Was $r^2 \geq 0.99$?	X		

4.0 Initial Calibration Verification [(ICV) Second Source]

Method 8330A ICV Criteria (Filename)	280-562503/20		
Instrument:	CHHPLC_X3		
Date of Initial Calibration Verification:	1/4/2022		
	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)	280-562503/38		
Instrument:	CHHPLC_X3		
Date of Initial Calibration Verification:	1/5/2022		
	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)	280-567560/19		
Instrument:	CHHPLC_X5		
Date of Initial Calibration Verification:	3/3/2022		
	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)	280-567560/28		
Instrument:	CHHPLC_X5		
Date of Initial Calibration Verification:	3/3/2022		
	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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Applicable QAPP: Cornhusker Army Ammunition Plant QAPP (Brice and AECOM, October 2019)

Applicable Analytical Methods: 8330A, 353.2, 350.1, 351.2, RSK-175, 9060A, 2320B, 9056A, 9034

Method RSK-175 ICV Criteria (Filename)	280-550959/13		
Instrument:	VGC J		
Date of Initial Calibration Verification:	9/24/2021		
	Yes	No	N/A
Was the ICV analyzed after each calibration?	X		
Was the ICV for all analytes within $\pm 25\%$ of the true value?	X		

Method 9056A ICV	WC IonChrom10		
Date of Initial Calibration Verification:	5/26/2022		
	Yes	No	N/A
Was the ICV analyzed after each ICAL, prior to the beginning of a sample analysis?	X		
Was the ICV for all analytes within $\pm 10\%$ of the true value?	X		

Method 9056A ICV	WC IonChrom13		
Date of Initial Calibration Verification:	5/16/2022		
	Yes	No	N/A
Was the ICV analyzed after each ICAL, prior to the beginning of a sample analysis?	X		
Was the ICV for all analytes within $\pm 10\%$ of the true value?	X		

Method 350.1 ICV Criteria	WC SKALAR 01		
Date of Initial Calibration Verification:	6/1/2022		
	Yes	No	N/A
Was the ICV analyzed after each ICAL, prior to the beginning of a sample analysis?	X		
Was the ICV for all analytes within $\pm 10\%$ of the true value?	X		

Method 353.2 ICV Criteria	WC Alp 2		
Date of Initial Calibration Verification:	6/2/2022		
	Yes	No	N/A
Was the ICV analyzed after each ICAL, prior to the beginning of a sample analysis?	X		
Was the ICV for all analytes within $\pm 10\%$ of the true value?	X		

Method 351.2 ICV Criteria	WC GAL1		
Date of Initial Calibration Verification:	5/25/2022		
	Yes	No	N/A
Was the ICV analyzed after each ICAL, prior to the beginning of a sample analysis?	X		
Was the ICV for all analytes within $\pm 10\%$ of the true value?	X		

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Applicable QAPP: Cornhusker Army Ammunition Plant QAPP (Brice and AECOM, October 2019)

Applicable Analytical Methods: 8330A, 353.2, 350.1, 351.2, RSK-175, 9060A, 2320B, 9056A, 9034

Method 9060A ICV Criteria	WC_SHI5		
Date of Initial Calibration Verification:	5/26/2022		
	Yes	No	N/A
Was the ICV analyzed after each ICAL, prior to the beginning of a sample analysis?	X		
Was the ICV for all analytes within $\pm 10\%$ of the true value?	X		

5.0 Continuing Calibration Verification (CCV)

Method 8330A CCV Criteria (Filename)	280-575712/7-8		
Instrument:	CHHPLC_X5		
Date of Calibration Verification:	5/20/2022		
	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)	280-575712/20-21		
Instrument:	CHHPLC_X5		
Date of Calibration Verification:	5/20-21/2022		
	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 CCV criteria were met for all target analytes.

Method 8330A CCV Criteria (Filename)	280-575600/7,9		
Instrument:	CHHPLC_X3		
Date of Calibration Verification:	5/19/2022		
	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 CCV criteria were met for all target analytes.

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Method 8330A CCV Criteria (Filename)	280-575600/21,23		
Instrument:	CHHPLC_X3		
Date of Calibration Verification:	5/20/2022		
	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 CCV criteria were met for all target analytes.

Method 8330A CCV Criteria (Filename)	280-575600/33,35		
Instrument:	CHHPLC_X3		
Date of Calibration Verification:	5/20/2022		
	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 CCV criteria were met for all target analytes.

Method RSK-175 CCVRT Criteria (Filename)	280-576169/2		
Instrument:	VGC J		
Date of Calibration Verification:	5/25/2022		
	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 $\pm 25\%$ of the true value?	X		

Method RSK-175 CCV Criteria (Filename)	280-576169/75		
Instrument:	VGC J		
Date of Calibration Verification:	5/25/2022		
	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 $\pm 25\%$ of the true value?	X		

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Method RSK-175 CCV Criteria (Filename)	280-576170/2		
Instrument:	VGC J		
Date of Calibration Verification:	5/25/2022		
	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 $\pm 25\%$ of the true value?	X		

Method RSK-175 CCV Criteria (Filename)	280-576170/89		
Instrument:	VGC J		
Date of Calibration Verification:	5/25/2022		
	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 $\pm 25\%$ of the true value?	X		

Method RSK-175 CCV Criteria (Filename)	280-576170/106		
Instrument:	VGC J		
Date of Calibration Verification:	5/25/2022		
	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 $\pm 25\%$ of the true value?	X		

Method RSK-175 CCV Criteria (Filename)	280-576170/120		
Instrument:	VGC J		
Date of Calibration Verification:	5/26/2022		
	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 $\pm 25\%$ of the true value?	X		

Method 9056A, Instrument: WC IonChrom11, All CCVs on 6/1/2022	Yes	No
Was a CCV analyzed after every 10 field samples and at the end of the analysis sequence?	X	
Were the CCVs for all analytes within $\pm 10\%$ of the true value?	X	

Method 9056A, Instrument: WC IonChrom13, All CCVs on 5/31/2022-6/1/2022	Yes	No
Was a CCV analyzed after every 10 field samples and at the end of the analysis sequence?	X	
Were the CCVs for all analytes within $\pm 10\%$ of the true value?	X	

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Method 350.1, Instrument: WC_SKALAR_01, All CCVs on 6/1/2022	Yes	No
Was a CCV analyzed after every 10 field samples and at the end of the analysis sequence?	X	
Were the CCVs for all analytes within $\pm 10\%$ of the true value?	X	

Method 353.2, Instrument: WC_Alp_2, All CCVs on 6/2/2022	Yes	No
Was a CCV analyzed after every 10 field samples and at the end of the analysis sequence?	X	
Were the CCVs for all analytes within $\pm 10\%$ of the true value?	X	

Method 351.2, Instrument: WC_GAL1, All CCVs on 5/25/2022	Yes	No
Was a CCV analyzed after every 10 field samples and at the end of the analysis sequence?	X	
Were the CCVs for all analytes within $\pm 10\%$ of the true value?	X	

Method 9060A, Instrument: WC_SHI5, All CCVs on 5/26/2022- 5/27/2022	Yes	No
Was a CCV analyzed after every 10 field samples and at the end of the analysis sequence?	X	
Were the CCVs for all analytes within $\pm 10\%$ of the true value?	X	

Method SM2320B, Instrument: WC_AT3, All CCVs on 5/24/2022	Yes	No
Was a CCV analyzed after every 10 field samples and at the end of the analysis sequence?	X	
Were the CCVs for all analytes within $\pm 10\%$ of the true value?	X	

6.0 Sensitivity

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

7.0 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		

The following sample(s) was collected in a properly preserved vial; however, the pH was 7 and outside the required criteria when verified by the laboratory. The sample was analyzed outside the 7-day holding time specified for unpreserved samples but within the 14-day holding time specified for preserved samples: G0116-22A (280-162386-6). The detected Methane result is qualified as an estimated concentration (J).

Sample ID	Analysis	Analyte	Qualification
G0116-22A	RSK-175	Methane	J

CHAAP Data Verification

Laboratory and SDG#: Eurofins 280-162386

AECOM Chemist: D. Casagrande

Date Verified: 7/20/2022

AECOM ITR: S. Louie

Guidance: DoD QSM Version 5.1 (January 2017)

Applicable QAPP: Cornhusker Army Ammunition Plant QAPP (Brice and AECOM, October 2019)

Applicable Analytical Methods: 8330A, 353.2, 350.1, 351.2, RSK-175, 9060A, 2320B, 9056A, 9034

8.0 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 requested sample analyses performed, the correct analyte lists used, and correct sample preparation and analyses methods and units utilized?	X		