U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT COMMAND
CHEMICAL BIOLOGICAL CENTER

ABERDEEN PROVING GROUND, MD 21010-5424

CCDC CBC-TR-1639

Test Service Agreement 1911T Report – CAM2 Technologies, LLC

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March 2020

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REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. REPORT DATE *(DD-MM-YYYY)*	**2. REPORT TYPE**	**3. DATES COVERED** *(From - To)*
XX-03-2020	Final	Jan 2020–Jan 2020

4. TITLE AND SUBTITLE
Test Service Agreement 1911T Report–CAM2 Technologies, LLC

5a. CONTRACT NUMBER
TSA 1911T

5b. GRANT NUMBER

5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)
Curtiss, Justin; Pardoe, Ian; and Emge, Darren

5d. PROJECT NUMBER
TSA 1911T

5e. TASK NUMBER

5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
Director, CCDC CBC, ATTN: FCDD-CBR-CP, APG, MD 21010-5424

8. PERFORMING ORGANIZATION REPORT NUMBER
CCDC CBC-TR-1639

9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES)
CAM2 Technologies; 6 Finance Drive, Danbury, CT 06810-4132

10. SPONSOR/MONITOR'S ACRONYM(S)

11. SPONSOR/MONITOR'S REPORT NUMBER(S)

12. DISTRIBUTION / AVAILABILITY STATEMENT
Approved for public release: distribution unlimited.

13. SUPPLEMENTARY NOTES
The U.S. Army Combat Capabilities Development Command Chemical Biological Center (CCDC CBC) was previously known as U.S. Army Edgewood Chemical Biological Center (ECBC).

14. ABSTRACT: (Limit 200 words)
Documented in this report are the tasks executed by members of the U.S. Army Combat Capabilities Development Command Chemical Biological Center for CAM2 Technologies (Danbury, CT) under contract and project numbers Test Service Agreement (TSA) 1911T. The objectives of the TSA were to collect and document infrared spectra for the purposes of algorithm development of a selection of chemical materials for CAM2 Technologies using their RedWave ThreatID infrared spectrometer.

15. SUBJECT TERMS
Infrared Test Service Agreement (TSA)

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
a. REPORT	**b. ABSTRACT**	**c. THIS PAGE**			Renu B. Rastogi
U	U	U	UU	18	**19b. TELEPHONE NUMBER** *(include area code)* (410) 436-7545

Standard Form 298 (Rev. 8-98)
Prescribed by ANSI Std. Z39.18

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PREFACE

The work described in this report was authorized under contract and project numbers TSA 1911T. The work was started in January 2020 and completed in January 2020.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

The text of this report is published as received and was not edited by the Technical Releases Office, U.S. Combat Capabilities Development Command Chemical Biological Center.

This report has been approved for public release.

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Test Service Agreement 1911T Report – CAM2 Technologies, LLC

1. PURPOSE

The report herein documents the work performed by U.S. ARMY Combat Capabilities Development Command Chemical Biological Center (CCDC CBC) Chemical Analysis and Physical Properties (CAPP) Branch of the Chemical Sciences Division under Test Service Agreement (TSA) 1911T for CAM2 Technologies, LLC.

2. SCOPE

CAPP personnel executed the statement of work (SOW) detailed in TSA 1911T by providing personnel, facilities, materials, supplies, and equipment to conduct data collection requested by CAM2. The goal of the TSA was to collect infrared spectra of chemical materials with the CAM2 RedWave ThreatID infrared spectrometer. This effort was conducted in Building E3400 at Aberdeen Proving Ground, Edgewood Area, MD on January 6, 2020. During the data collection period, CAPP personnel prepared samples in accordance with requested sample conditions specified in the matrix, recorded laboratory data, operated the system, and communicated results to the vendor in real-time.

3. OBJECTIVES OF TSA

Specific tasks to be completed were outlined in Section 3.0 of TSA 1911T and are as follows:

1. CCDC CBC will set up ThreatID according to CAM2 Technology specifications and perform all necessary calibrations as required
2. CCDC CBC will provide testing, facilities, personnel, chemicals of interest and laboratory materials necessary to execute the SOW
3. CCDC CBC will collect infrared spectra of each chemical, preparing three replicate samples for each, using the ThreatID instrument. The sample will be placed in the instruments' sample holder and a spectrum will be collected and saved. Once complete, the sample will be removed and the sample holder will be cleaned using the appropriate solvent. Collection procedures will be conducted per training/operation materials and instructions of CAM2 Technologies personnel.
4. CCDC CBC will purchase all analytes required for Sample Quality Analyses and provide all instrumentation for the execution of same
5. CCDC CBC will perform Quality Analysis for all procured chemicals and associated materials, to include the concentration/purity of the chemicals (by IMS, FT-IR, IR, NMR, GC/MS, or GC/FPD) and the identity of the chemicals' sources, if allowed. CCDC CBC will provide details of the Quality Assurance standards and system used in the laboratory. As a minimum, the laboratory will comply with the Quality Management System described by ISO 9001:2015.
6. CCDC CBC will perform necessary decontamination, cleanup, and waste disposal

7. CCDC CBC will perform risk assessment as required CAM2 Technologies representative(s) desiring to observe or participate in activities at CCDC CBC associated with the performance of this TSA shall make prior arrangements with the CCDC CBC POC listed in SOW 8.1 to address safety and security requirements. CAM2 TECHNOLOGIES representative(s) agree to comply with all pertinent safety precautions in accordance with applicable regulations, procedures, and policies. The CCDC CBC POC will provide applicable installation/facility security policies and procedures and assist with a visit request. The CCDC CBC POC requires a minimum 48 hour notice for coordination with CCDC CBC Security Office. CAM2 TECHNOLOGIES will provide the necessary information to gain access prior to the visit. A CCDC CBC Visitor's guide containing installation access information can be found at: http://www.ecbc.army.mil/about/mapdir.html. CAM2 Technologies should be aware of gate access and documentation requirements and comply with all personal identity verification requirements. Should the Force Protection Condition at any individual facility or Aberdeen Proving Ground change, CAM2 Technologies will adhere to changes in security matters or processes, as applicable. Only U.S. citizens are permitted area access without Foreign Disclosure approval. Dual citizens are considered foreign nationals.
8. CAM2 Technologies shall provide any Purchaser items necessary and any required support equipment 10 days prior to provision of technology support under this TSA.

4. SYSTEM DESCRIPTION

A portable infrared spectrometer, RedWave ThreatID, was provided by CAM2 to CAPP personnel for collection of the infrared spectra. The ThreatID consisted of a single pelican case, measuring 16.4 L, weighing 6.4 kgs., with a power supply cord; the instrument had an internal battery that was not used. Control of the instrument was performed through an integrated touch screen, operating on an Android based application. Wireless viewing of the instrument screen was performed with the Teamviewer program through a wireless access point (MiFi) provided by CAPP.

5. TEST EQUIPMENT

Described in the following sections are the infrastructure and equipment used by CAPP personnel to conduct necessary data collection operations. The usage and justification of the equipment is detailed as well as any calibration or verification techniques.

5.1 Laboratory Infrastructure

The data collection was conducted in building E3400 of the Edgewood Area of Aberdeen Proving Ground, room 310, Chemical, Biological, Radiological, Nuclear, and Explosive (CBRNE) Assessment, Science, and Technology Laboratory Edgewood (CASTLE).

A chemical fume hood was used for engineering controls to contain and exhaust any vapor from the chemicals being used. The vendor personnel observed the instrument operation from a room adjacent to room 310 and was in communication with lab personnel via encrypted, wireless headsets.

5.2 Pipettes

All liquid sample depositions were performed with calibrated and verified volumetric pipettes. A Gilson M10 and M100 were used for sample depositions with volumes deposited of 5 µL and 20 – 100 µL, respectively. For each sample deposition a new pipette tip was used and disposed of after each trial.

5.3 Chemicals

CAM2 requested the chemicals listed in Table 1 for infrared spectral collection using the ThreatID.

Table 1: Chemicals for Infrared Spectra Collection

Compound Name	CAS No.
Acetone	67-64-1
GA (Tabun)	77-81-6
GB (Sarin)	107-44-8
GD (Soman)	96-64-0
GF (cyclo-Sarin)	329-99-7
Half Mustard (2-chloroethyl ethyl sulfide)	693-07-2
HD (Distilled Sulfur Mustard)	505-60-2
HN-1 (Nitrogen Mustard)	538-07-8
HN-3 (Nitrogen Mustard)	555-77-1
L (Lewisite)	541-25-3
VX	50782-69-9
A-230	2387496-12-8
A-232	2387496-04-8
A-234	2387496-06-0

6. TEST PROCEDURES

Described in the following sections are CAM2 conducted training, data collection matrix, instrument operation, data transfer and data export.

6.1 Training

Upon arrival, after setting up the instrument outside of the engineering controls, Dave Schiering from CAM2 conducted a training session for CAPP personnel on operating the ThreatID, transferring data and exporting data to a USB drive. Once training was completed to

satisfaction of CAM2, the instrument was moved into the chemical fume hood for data collection by CAPP personnel.

6.2 Data Collection Matrix

A macro-enabled Excel spreadsheet was used to record and document parameters during data collection. Included in the matrix were details of the sample, start and stop times of the data collection by the instrument and comments by the data recorder. Detailed in Table 2 are details for the information contained in the matrix.

Table 2: Matrix Information

Tab Label	Column Label	Definition
Matrix	Trial Number	Number denoting a new sample was been prepared, unique for each sample preparation
	Test Condition	Number that defines the parameters of the sample and allows for a reference of multiple sample preparations, each number is unique set of conditions
	Chemical	Chemical material deposited on the crystal surface for analysis
	Sample Volume	Volume of sample deposited on the crystal, measure in microliters (µL)
	Sample Start	Time at which the instrument started the collecting the spectra
	Sample Stop	Time at which the instrument stopped collecting the spectra
	Sample ID	File name generated by instrument for each collected spectra

6.3 System Operation

The ThreatID was operated using the integrated touchscreen, cycling through a series of steps as per the training. After the instrument verified that the sample space was clean, a 60 second reference spectrum was collected by pressing the “Next” button on the touchscreen. The user was then prompted that the reference was complete and to proceed to the next step, collecting the sample spectrum. A sample of the chemical, according to the matrix, was deposited on to the sample space using the appropriate pipette and new pipette tip. A 60 second sample spectrum was collected after which the spectrum was displayed. If the spectrum collected was the first of the three replicates, it was sent via email to the vendor to confirm data quality; subsequent spectra were collected without emailing for quality confirmation. The sample space was cleaned twice with a Kim-wipe wetted with methanol, then dried with an additional Kim-wipe. The user then navigated to the home screen and repeated the above steps to complete the data collection matrix.

6.4 Data Transfer

To ensure data quality, the first spectra collected in the series of three replicates of each chemical was emailed to the vendor, prior to collecting the two additional spectra. The emailing operation was performed from within the instrument control program and sent directly to the vendor using the wireless connection provided by the MiFi access point.

6.5 Data Export

Once the data collection matrix had been completed all spectra were transferred to a USB thumb drive and given to the vendor, who verified all data was present and met the requirements stated in the TSA.

6.6 Quality Assurance and Quality Control

Prior to conducting data collection of the infrared spectra the purity of each chemical was measured. Purity was taken to be the peak area of the Gas Chromatography (GC) or Liquid Chromatography (LC) curve associated with the chemical as a fraction of the total area under the GC or LC curve. The purity for each chemical are listed in Appendix 8.2. Samples were prepared by placing 1 uL of chemical into 1 mL of methylene chloride. Samples analyzed on 1/6/2020 under the following conditions:

- Oven Profile: 45C (3 min) 15C/min 280C (0 min)
- Inlet: Splitless; On @ 0.1 min at a flowrate of 100mL/min
- Inlet Temp: 250C
- Column: DB-5MS; 30m X 0.25mm ID; 0.25um film thickness
- Column Flow: 1.2 mL/min
- Injection volume: 0.2 uL
- Transfer Line: 280C
- EM Voltage: 1718
- Scan Range: 40 – 400 amu
- MS Source Temp: 230C
- MS Quad Temp: 150C
- Solvent Delay: 2.75 minutes

7. CONCLUSIONS AND OBSERVATIONS

Data collection as described in the SOW was completed on January 6, 2020, fulfilling the objectives of the TSA, completing the data collection matrix. The instrument as operated per the training had no faults or issues during the data collection period. As the instruments' library was not populated with the chemicals listed in Table 1, the reported results were not recorded, however the instrument did generate a detection results for every sample. Overall the instrument was easy to use and no additional instructions were needed by the vendor during the data collection period, completing the TSA objectives in a single day.

ACRONYMS AND ABBREVIATIONS

CAPP	Chemical Analysis Physical Properties
CASTLE	CBRNE Assessment, Science, and Technology Laboratory Edgewood
CBRNE	Chemical, Biological, Radiological, Nuclear, and Explosive
GC	Gas Chromatography
GC-MS	Gas Chromatography Mass Spectrometry
QA/QC	Quality Assurance and Quality Control
SOW	Statement of Work
TSA	Test Service Agreement
U.S. Army Combat Capabilities Development Command Chemical Biological Center	CCDC CBC

APPENDIX A: SUPPLEMENTAL INFORMATION

A.1 Data collection Matrix

Displayed below in Table 3 is the data collection matrix as described in Section 6.2.

Table 3: Data Collection Matrix

Trial #	Test Condition	Chemical	Sample Start	Sample Stop	Date	Sample ID	Sample Volume (µL)	Comments
1	1	GA	1:02:04 PM	1:03:17 PM	1/6/2020	2020-01-06T12:58:05	5	
2	1	GA	1:06:04 PM	1:07:15 PM	1/6/2020	2020-01-06T13:04:17	5	
3	1	GA	1:10:08 PM	1:11:32 PM	1/6/2020	2020-01-06T13:08:25	5	
4	2	GB	11:18:59 AM	11:20:12 AM	1/6/2020	2020-01-06T11:16:21	5	
5	2	GB	11:23:11 AM	11:24:22 AM	1/6/2020	2020-01-06T11:21:26	5	
6	2	GB	11:26:54 AM	11:28:08 AM	1/6/2020	2020-01-06T11:25:01	5	
7	3	GD	1:25:52 PM	1:27:04 PM	1/6/2020	2020-01-06T13:24:15	5	
8	3	GD	1:29:44 PM	1:30:58 PM	1/6/2020	2020-01-06T13:28:07	5	
9	3	GD	1:33:08 PM	1:34:46 PM	1/6/2020	2020-01-06T13:31:27	5	
10	4	GF	11:05:14 AM	11:06:27 AM	1/6/2020	2020-01-06T11:03:36	5	
11	4	GF	11:09:49 AM	11:11:01 AM	1/6/2020	2020-01-06T11:07:57	5	

12	4	GF	11:13:44 AM	11:14:56 AM	1/6/2020	2020-01-06T11:11:48	5	
13	5	CEES	12:47:26 PM	12:48:38 PM	1/6/2020	missed	5	
14	5	CEES	12:51:55 PM	12:53:07 PM	1/6/2020	2020-01-06T12:49:38	5	
15	5	CEES	12:55:52 PM	12:57:08 PM	1/6/2020	2020-01-06T12:53:59	5	
16	6	HD	10:40:07 AM	10:41:20 AM	1/6/2020	2020-01-06T10:36:49	5	
17	6	HD	10:44:27 AM	10:45:36 AM	1/6/2020	2020-01-06T10:42:34	5	
18	6	HD	10:48:15 AM	10:49:27 AM	1/6/2020	2020-01-06T10:46:20	5	
19	7	HN-1	1:42:16 PM	1:43:27 PM	1/6/2020	2020-01-06T13:38:01	5	
20	7	HN-1	1:46:12 PM	1:47:23 PM	1/6/2020	2020-01-06T13:44:27	5	
21	7	HN-1	1:49:47 PM	1:51:00 PM	1/6/2020	2020-01-06T13:48:04	5	
22	8	HN-3	10:24:26 AM	10:25:38 AM	1/6/2020	2020-01-06T10:20:59	5	
23	8	HN-3	10:30:03 AM	10:31:15 AM	1/6/2020	2020-01-06T10:28:22	5	
24	8	HN-3	10:34:54 AM	10:36:05 AM	1/6/2020	2020-01-06T10:32:49	5	
43	8	HN-3	2:05:09 PM	2:06:19 PM	1/6/2020	2020-01-06T14:02:56	5	
44	8	HN-3	2:09:52 PM	2:11:06 PM	1/6/2020	2020-01-06T14:07:28	5	
45	8	HN-3	2:14:15 PM	2:15:28 PM	1/6/2020	2020-01-06T14:12:20	5	
25	9	L	1:14:40 PM	1:16:07 PM	1/6/2020	2020-01-06T13:13:09	5	
26	9	L	1:18:47 PM	1:20:02 PM	1/6/2020	2020-01-06T13:17:05	5	
27	9	L	1:22:30 PM	1:24:09 PM	1/6/2020	2020-01-06T13:20:58	5	
28	10	VX	1:53:15 PM	1:54:25 PM	1/6/2020	2020-01-06T13:51:42	5	
29	10	VX	1:57:08 PM	1:58:20 PM	1/6/2020	2020-01-06T13:55:27	5	
30	10	VX	2:00:43 PM	2:01:54 PM	1/6/2020	2020-01-06T13:59:10	5	
31	11	A-230	11:43:06 AM	11:44:18 AM	1/6/2020	2020-01-06T11:41:02	5	
32	11	A-230	11:47:28 AM	11:48:53 AM	1/6/2020	2020-01-06T11:45:47	5	
33	11	A-230	11:51:50 AM	11:53:07 AM	1/6/2020	2020-01-06T11:50:08	5	
34	12	A-232	11:30:52 AM	11:32:13 AM	1/6/2020	2020-01-06T11:28:46	5	
35	12	A-232	11:35:14 AM	11:36:25 AM	1/6/2020	2020-01-06T11:33:22	5	
36	12	A-232	11:38:52 AM	11:40:05 AM	1/6/2020	2020-01-06T11:37:12	5	
37	13	A-234	10:52:40 AM	10:53:52 AM	1/6/2020	2020-01-06T10:50:16	5	
38	13	A-234	10:56:58 AM	10:58:10 AM	1/6/2020	2020-01-06T10:54:51	5	

39	13	A-234	11:00:53 AM	11:02:07 AM	1/6/2020	2020-01-06T10:59:05	5	
40	14	Acetone	2:18:04 PM	2:19:23 PM	1/6/2020	2020-01-06T14:16:12	5uL	5uL evaporated quickly even with cup and lens, volume was changed to 100 µL
41	14	Acetone	2:21:20 PM	2:22:30 PM	1/6/2020	2020-01-06T14:19:37	100uL	
42	14	Acetone	2:24:58 PM	2:26:10 PM	1/6/2020	2020-01-06T14:23:03	100uL	
46	14	Acetone	2:29:25 PM	2:30:39 PM	1/6/2020	2020-01-06T14:26:37	100uL	

A.2 QA/QC Results

Displayed below in Table 4 are the QA/QC results for the chemicals used during data collection.

Table 4: Chemicals for Infrared Spectra Collection

Compound Name	Purity (%)
Acetone	99.9 (Sigma Aldrich)
GA (Tabun)	92.1
GB (Sarin)	98.5
GD (Soman)	99.5
GF (cyclo-Sarin)	93.7
Half Mustard (2-chloroethyl ethyl sulfide)	Not Performed
HD (Distilled Sulfur Mustard)	98.3
HN-1 (Nitrogen Mustard)	99.3
HN-3 (Nitrogen Mustard)	99.8
HN-3 (Nitrogen Mustard), second sample	98.6
L (Lewisite)	82.7
VX	92.1
A-230	98.6
A-232	98.7
A-234	99.1

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