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TITLE/TITRE:

Accident Investigation involving a UXO in Rivers MB

PROJECT NUMBER
NUMÉRO DU PROJET: C007307

DATE: May 2008

PROJECT MANAGER
ADMINISTRATEUR DU PROJET:

Mr. KENT BRADY
Lead Chemist, Metals and Inorganic Chemicals
QETE 3-2

APPROVED BY
APPROUVÉ PAR:

Dr. H. Fanous
Group Leader, General Chemistry and
Environmental Issues
for SUPERINTENDENT

NATIONAL DEFENCE
DÉFENSE NATIONALE CANADA

ABSTRACT

An accident involving a 16-year-old agricultural worker occurred in River, MB on 29 April 2007. The Ammo Tech from ASU Shilo recovered remnants of an AN-M23 White Phosphorus (WP) igniter with an AN-M173 initiator and the coveralls worn by the employee. Significant quantities of Tetryl were found on both the remnants of the UXO and the coveralls that link the two items to the accident. Additionally, the phosphorus levels around the burns on the coveralls indicate the burns are likely caused by phosphorus.

KEYWORDS

UXO, Rivers Manitoba, Accident, White Phosphorus, Tetryl, AN-M173, AN-M23, igniter, Evidence, Chain of Custody, GC-MS, SEM-EDX, ED-XRF

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF GRAPHS

LIST OF ANNEXES

REFERENCES

A. Tasking e-mail from Major Mathew Braid, P.Eng Project Director – DND UXO and Legacy Sites Program ADM(IE) DRFMS, dated 19 June 2007.

B. ASTM D 4840 Standard Guide for Sampling Chain-Of-Custody Procedures.

C. Robert J. Fellows Scott D. Harvey Dominic A. Cataldo, Environmental Fate and Behavior of Tetryl (U.S. Army Medical Research and Development Command under a Related Services Agreement with the U.S. Department of Energy, March 1992).

D. TOXICOLOGICAL PROFILE FOR TETRYL (2,4,6-Trinitrophenyl-N-methylnitramine), Public Health Service, Agency for Toxic Substances and Disease Registry, June 1995.

E. TOXICOLOGICAL PROFILE FOR WHITE PHOSPHORUS, Public Health Service, Agency for Toxic Substances and Disease Registry, September 1997.

DEFINITIONS

1. Acronyms

ED-XRF – Energy Dispersive X-Ray Florescence
SEM – Scanning Electron Microscope
EDX – Energy Dispersive X-Ray
WDX – Wavelength Dispersive X-Ray
GC-MS – Gas Chromatography Mass Spectrometry
ASU – Area Support Unit
WP – White Phosphorus
MB – Manitoba
UXO – Unexploded Explosive Ordinance
QETE – Quality Engineering Test Establishment
DND – Department of National Defense
RCMP – Royal Canadian Mounted Police

TERMINOLOGY

2. AN-M23 White Phosphorus (WP) igniter - An initiator used to ignite smoke bombs and the napalm fillers for firebombs. It consists of a steel body and 0.567 kg of white phosphorus.

3. AN-M173 initiator - This fuse is used with firebomb igniters. It is vane armed and impact fired. It consists of a primer (M26), Detonator (M31A1) and a booster containing 14 grams of Tetryl

4. Tetryl - Tetryl is an odorless, synthetic, yellow crystal-like solid that is not found naturally in the environment. The chemical name for tetryl is 2,4,6-trinitrophenyl-n-methylnitramine. As of 1979, Tetryl is no longer manufactured or used in the United States, but can still be found in legacy munitions.

5. White Phosphorus - White phosphorus is a waxy solid that reacts rapidly with oxygen, easily catching fire. It is used in chemical manufacturing and certain types of munitions. Exposure to white phosphorus may cause burns and irritation, liver, kidney, heart, lung, or bone damage, and death.

BACKGROUND

6. At Rivers MB an incident involving a UXO occurred on land that was once owned by the DND. On the 29 April 2007, the incident involving a 16-year-old employee of Redfern Farm Services occurred while the individual was mowing corn stalks. The mower ran over a pyrotechnic item that caught on fire. While trying to extinguish the fire, the individual saw a softball size item, which subsequently detonated in his face. On 30 April 2007, ASU Shilo was apprised of the incident and on 1 May 2007 they visited the site where the incident occurred as well as Rivers Fire hall. They also visited the Rivers Fire hall were the Ammo Tech from ASU Shilo retrieved the remnants of the alleged UXO. The item was later identified as a M23 WP igniter. On 2 May 2007, DND's UXO and Legacy Sites Program were tasked with investigating the incident. In June 2007 Major Matt Braid, program director of the DND UXO and Legacy Site Program contacted QETE. QETE was tasked to investigate a linkage between the UXO and the injury to the individual (Ref. A).

EVIDENCE

7. When the samples were retrieved from the UXO offices in Ottawa a chain of custody form was setup in accordance with ASTM D 4840 – 88 "Standard Practice for Sampling Chain of Custody Procedures"(ref. B). A Copy of the chain of custody form for the coveralls and the UXO can be found in ANNEX A.

8. The coveralls were retrieved from the Ottawa offices of the DND UXO and Legacy Sites Program at 1100 hrs on the 3 July, 2007. The coveralls were packaged in plastic bags and sealed with tape. The coveralls were removed from the packaging and photographed by the photo unit at QETE. The photos can be found in ANNEX B. Anchor Textiles manufactured the coveralls. The material consisted of 65% polyester and 35% cotton. There were extensive burns on the front of the coveralls with the majority being in

the upper left thigh and groin area. There were almost no burns on the back of the coveralls with the exception of one small (~2-3 cm) burn in the left rear pocket.

9. Samples were taken from eight different locations on the coveralls. At each location one to four samples were taken. Figure 1 shows the location on the coveralls were the samples were removed.

Figure 1. Coveralls showing the sampling points.

10. The following table summarizes all of the samples that were taken from the coveralls. Note that not all of the samples were used for testing. Five samples (1 sample from locations 4, 5, 6, 7 and 8) were placed in vials and are stored in a secure location. Additionally, some samples were used for method development.

Sample Location	Sample ID	Sample Description
1	C007307-COV-1A, B, C & D	Four samples were taken from the front collar. The collar had burns that appeared free from soil/dirt.
2	C007307-COV-2A	One sample was taken from the lower front right leg. The section had small burns and was heavily soiled.
3	C007307-COV-3A	One sample was taken from the front left thigh and was heavily damaged.
4	C007307-COV-4A, B, C, & D	Four samples were taken from the upper left back shoulder. This area appeared to be the cleanest section on the coveralls.
5	C007307-COV-5A, B, C & D	Four samples were taken from the groin area. The groin area appeared to be the most damaged section on the coveralls.
6	C007307-COV-6A, & B	Two samples were taken from the lower front right leg area.
7	C007307-COV-7A & B	Two samples were taken from the left front thigh area. The left thigh area was heavily damaged.
8	C007307-COV-8A & B	Two samples were taken from the upper left front chest area. The upper chest area was lightly damaged.

Table 1: Coverall Sample Description

11. The remnants of the UXO were also retrieved from the Ottawa offices of the DND UXO and Legacy Sites Program at 1100 hrs on the 3 July 2007. The remnants, which were packaged in a plastic RCMP evidence bag, were removed from the packaging and photographed by the photo unit at QETE. The photos can be found in ANNEX C. The device was identified as being an AN-M23 WP igniter with an AN-M173 ALL-WAYS fuse. The remnants of the UXO consisted of the main body that contained the fuse and several smaller fragments, which were covered with dirt and appeared to have an orange and black coating on them.

12. The UXO remnants were sampled in four places by swiping small areas on the surface using an acetonitrile soaked swap. The following table describes where the swabs were taken.

Sample Location	Sample ID	Sample Description
1	C007307-UXO-1	One swab of the exterior of the outer casing.
2	C007307-UXO-2	One swab of the interior of the outer casing.
3	C007307-UXO-3	One swab of the interior of the initiator.
4	C007307-UXO-4	One swab of the exterior tip of the initiator.

Table 2: UXO Remnants Sample Description

TEST PROCEDURES

SEM

13. The SEM analysis on the coverall samples was performed with both WDX detection and EDX detection. The purpose of the analysis was to determine the phosphorus levels around the burnt areas of the fabric and compare the results to a clean undamaged section. Further details can be found in the two reports included in ANNEX D and ANNEX E.

14. C007307-COV-4A (Rear Left Shoulder): The clean, undamaged coverall sample removed from the rear left shoulder (labeled C007307-COV-4A) was analyzed for phosphorus and silicon using the WDX analyser.

15. C007307-COV-3A (Left Thigh): A section of the coveralls was removed from the left thigh area (labeled C007307-COV-3A) and provided for WDX analysis. The sample consisted of three zones (a burnt zone, an undamaged zone, and a transition zone) and various areas across the three zones (labeled k, j, 1, 2, 3, 4, and 5) which were all analyzed for phosphorus and silicon. The analysis started at the burnt edge (labeled ‘k’) and moved towards the undamaged edge (labeled ‘5’).

16. C007307-COV-1A (Left Collar Piece A): A section of the coveralls was removed from the left collar area (labeled C007307-COV-1A) and provided for WDX analysis. This sample consisted of three zones (a burnt zone, an undamaged zone, and a transition zone) and various areas across the three zones (labelled b, c, d, e, f, and h) were also analyzed for phosphorus and silicon. The analysis started at the burnt edge (labeled ‘b’) and moved towards the undamaged edge (labelled ‘h’).

17. C007307-COV-1B (Left Collar Piece B): A second section of the coveralls was removed from the left collar area (labeled C007307-COV-1B) and provided for WDX analysis. It consisted of three zones (a burnt zone, an undamaged zone, and a transition

zone). Two sets of analyses were carried out for phosphorus and silicon across the three zones. Analysis 1 analyzed areas z, y, x, w, v, u, and t (with z closest to the burnt edge and t furthest from the burnt edge) while analysis 2 analyzed areas r, q, p, o, n, m, and l (with r closest to the burnt edge and l furthest from the burnt edge). Figure 2 shows the points on the fabric that were analyzed by the SEM.

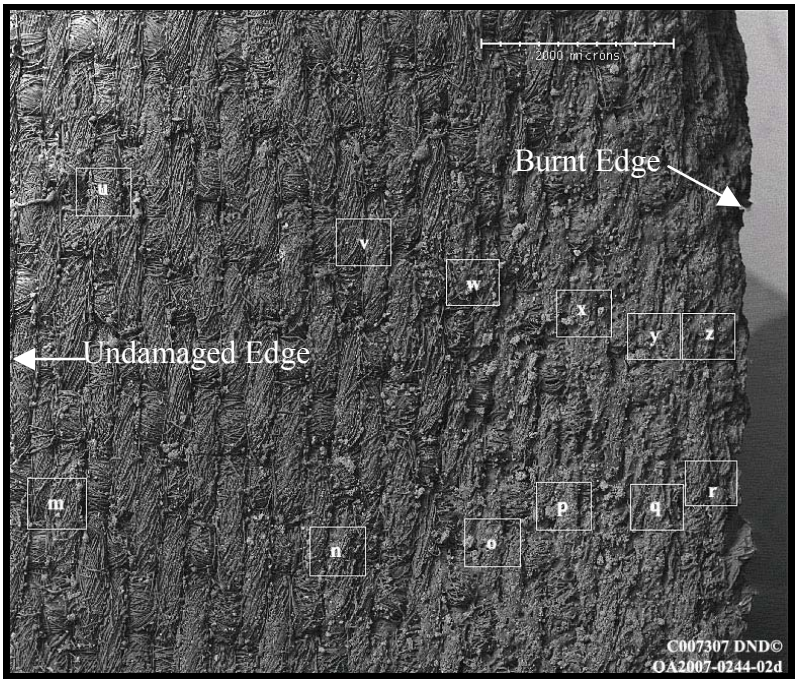


Figure 2: SEM Sample Locations

18. C007307-COV-4A: Energy dispersive X-ray (EDX) analysis was carried out on an undamaged coverall section removed from the rear left shoulder (labeled C007307-COV-4A).

19. C007307-COV-1A: EDX analysis was carried out on a burnt section of the coveralls removed from the left collar (labeled C007307-COV-1A).

20. C007307-COV-1B: EDX analysis was performed out on a burnt coverall sample removed from the left collar piece B (labeled C007307-COV-1B).

21. C007307-COV-2A: EDX analysis was carried out on a burnt coverall section removed from the right leg (labeled C007307-COV-2A).

22. Phosphorus Profile for C007307-COV-2A: Using sample C007307-COV-2A, a phosphorus line profile was acquired (using the EDX analyzer) from the burn hole to an area away from the burn. The analysis was carried out across three consecutive areas. The same equipment parameters (i.e. acquisition time, voltage, current, magnification, etc) were used for each analysis.

ED-XRF

23. In order to analyze the phosphorus concentrations at various distances from the burnt edge of fabric, a thin 2 mm wide strip was cut from sample C007307-1C. This sample was burnt at one end and undamaged at the other end. The strip was then cut into five sections 2-3 mm in length. The five sections were mounted onto sample holders using double-sided tape. The sections were analyzed using an Oxford Instrument ED2000 ED-XRF.

GC-MS

24. Coverall samples were prepared by placing 10 to 40 mg of sample into separate clean extraction vials and adding 500 µL of acetone using a glass syringe. The samples were mixed on a shaker then allowed to settle for an hour then mixed again. Immediately after the second shaking a portion of the acetone from each sample was transferred to an auto-sampler insert vial (~200 µL capacity) using a glass pipette. The vials were heated gently to less than 60°C and evaporated under a gentle stream of nitrogen. When the majority acetone was evaporated the remaining acetone from the extraction vials was transferred into the insert vials where they were evaporated to a final volume of 20±5 µL.

25. The sample extracts were analyzed with an Agilent 6890 GC-MS. The instrument parameters for the analysis of the coverall extracts are included in ANNEX F.

26. The tetryl concentrations in the coverall samples were determined by comparing the response from the samples to a calibration curve. Standard concentrations for the calibration curve were prepared using AccuStandard Tetryl (Lot# B5080227-1A).

27. Samples from the remnants of the UXO were prepared by swiping the area with cotton swabs soaked in acetonitrile. The swabs were then placed in vials and 1.0 mL of acetonitrile was added to each vial. The vials were mixed on a shaker for ~30 seconds. 0.5 ml of the acetonitrile was transferred from the extraction vials to GC vials and evaporated under a gentle stream of nitrogen to a final volume of ~20 µL.

28. The sample extracts were analyzed with an Agilent 6890 GC-MS. The instrument parameters for the analysis of the UXO extracts are included in ANNEX G.

29. The tetryl concentrations on the samples from the UXO were determined by comparing the instrument response from the samples to the instrument response from a single standard of known concentration. The standard of known concentration was prepared using Restek 8095 Calibration Mix A (Lot# A034040) and Restek 8095 Calibration Mix B (Lot# A033026).

TEST RESULTS

SEM

30. WDX analysis of the burnt coverall samples showed higher phosphorus levels at the burnt edges with a gradual decrease in phosphorus as the analysis moved towards the cleaner, undamaged areas. No phosphorus was detected in the clean and undamaged coverall section (C007307-COV-4A).

31. WDX analysis determined that the silicon levels for three of the analyses followed a similar trend to the phosphorus and that the fourth analysis showed an increase in silicon near the undamaged edge. Small levels of silicon were detected in the clean and undamaged coverall sample.

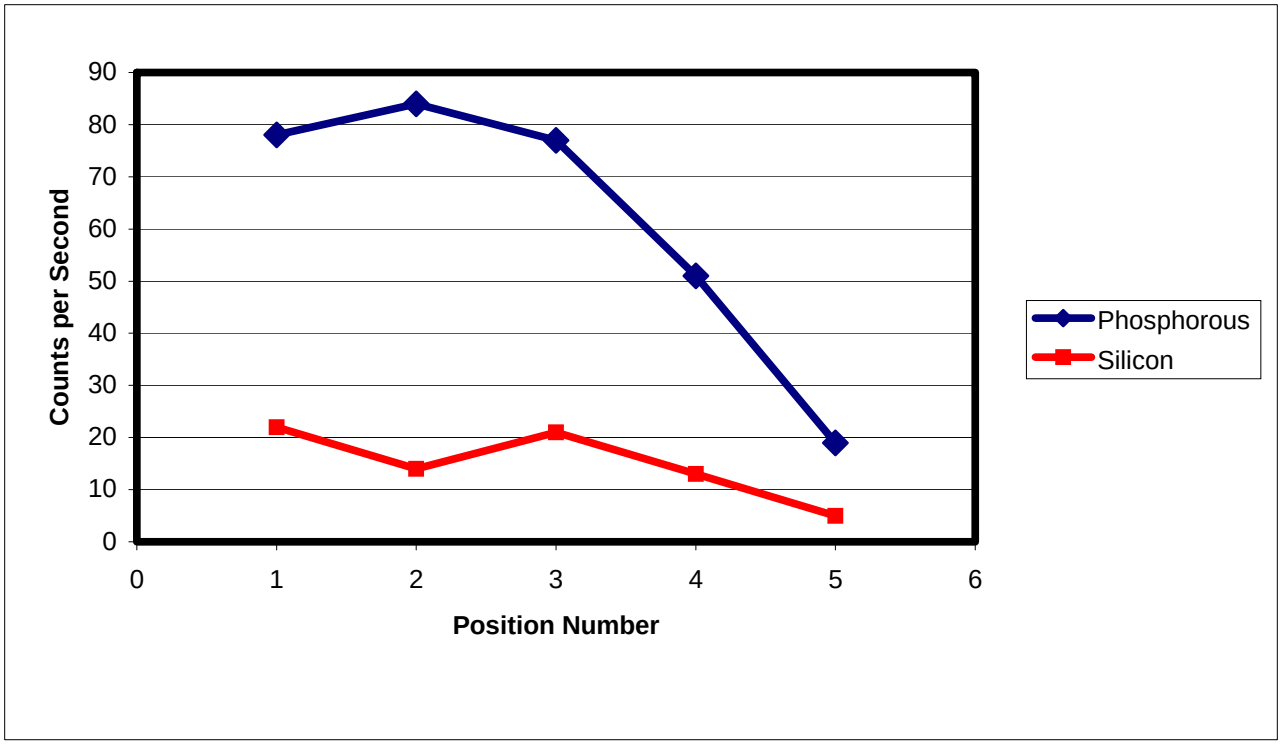
32. It must be stated that WDX analysis is a very localized analysis and that the detected levels of phosphorus and silicon may not exactly represent their overall distribution in the samples. Further analysis was carried out using EDX analysis (see ANNEX E).

33. EDX analysis was carried out on the burnt section of samples C007307-COV-1A, C007307-COV-1B, and C007307-COV-2A they were similar to the parent material (spectrum “c007307_cov_1a_left rear shoulder”) except that the former three spectra had a major amount of phosphorus, no chlorine, and a slightly higher aluminum, magnesium, sodium, and silicon content.

34. The line profile analysis determined that the phosphorus levels decreased as the analysis moved away from the burn mark on sample C007307-COV-2A. This was consistent with the WDX analysis that was carried out on sections C007307-COV-1A, C007307-COV-1B, and C007307-COV-3A.

ED-XRF

35. The results of the analysis by ED-XRF were in agreement with the results from the analysis by SEM. The following graph shows the level of phosphorus (in counts per second) found in each of the 5 sections. Section 1 was at the burnt edge and section 5 was at the clean edge.



Graph 1. Phosphorus Levels vs. Position from Burnt Edge

GC-MS

36. The GC-MS analysis determined that there were significant levels of tetryl on certain areas both the coveralls and the UXO. Figure 3 shows a printout with comments of the results from a coverall analysis.

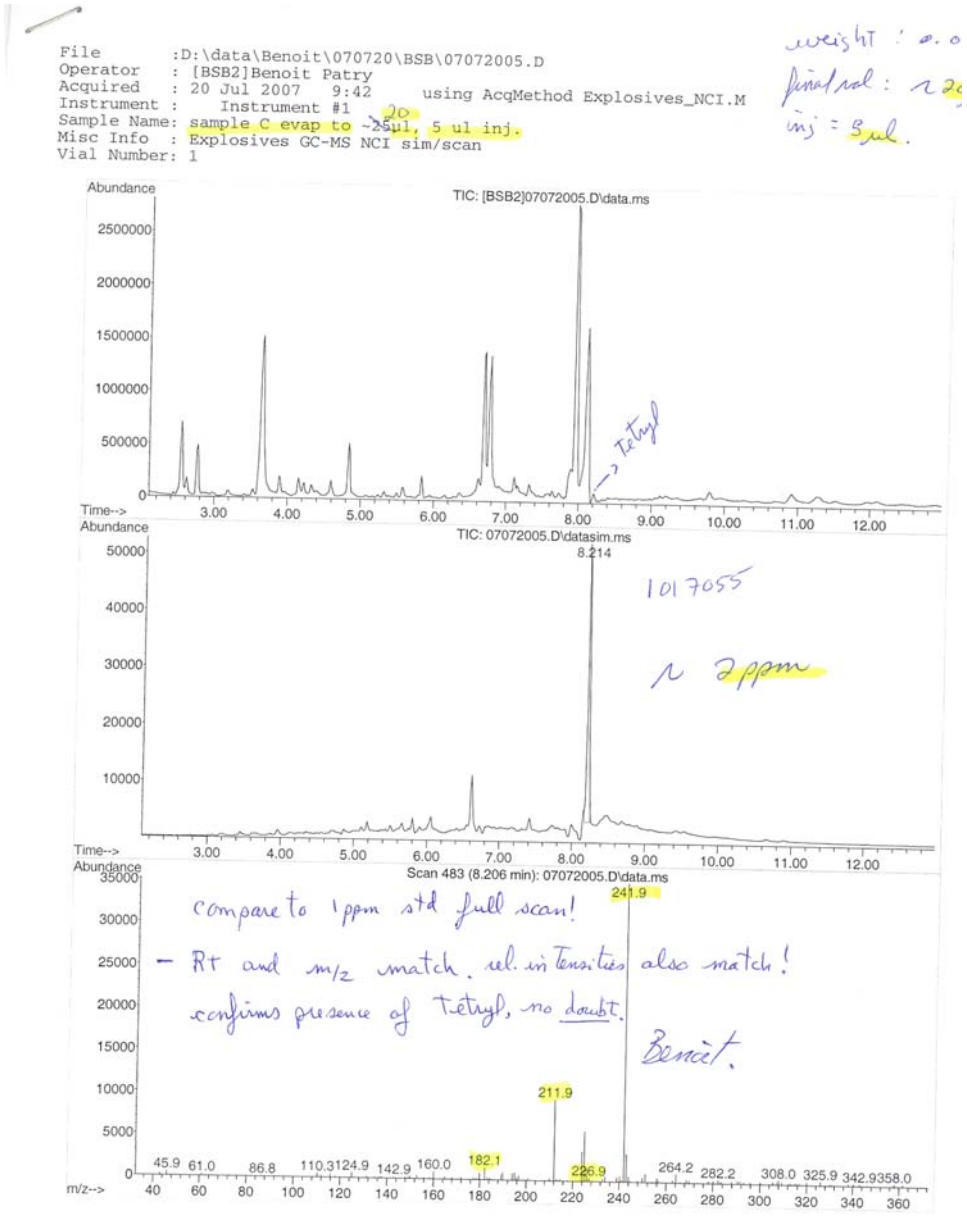


Figure 3. GC-MS Results

37. Tetryl concentrations (ng per µL) in the extracts from the coveralls were multiplied by the final volume of the extracts (20 µL) yielding the total mass of tetryl in the extract. The mass of tetryl in the extract was divided by the mass of the coverall sample (in milligrams) giving a tetryl concentration from the sample in parts per million (ppm). The following table summarizes the results of the coverall analysis.

Sample ID	Sample Mass milligrams	Extract Concentration ng/µL	Tetryl Concentration on Fabric ppm
C007307-COV-4C	21.7	0.7	0.6
C007307-COV-5C	16.6	272	327
C007307-COV-6B	13.5	2.6	4.0
C007307-COV-7B	41.0	54	26
C007307-COV-8B	30.7	1.5	1.0

Table 3: Tetryl Concentration on Coveralls

38. Tetryl concentrations (ng per µL) in the extracts on the UXO remnants were multiplied by the final volume of the extracts (20 µL) yielding the mass of tetryl in the portions of the extracts that were analyzed. The mass of tetryl in the portion of the extract represents half of the total extract therefore the mass of tetryl was multiplied by two. The total mass of tetryl in the extract was divided by surface area of the UXO that was swabbed (in mm^2) giving a tetryl concentration from the sample in ng per mm^2. The following table summarizes the results of the UXO analysis.

Sample ID	Area Swabbed mm^2	Extract Concentration ng/µL	Tetryl Concentration on Fragment ng/mm^2
C007307-UXO-1	100	Not Detected	N/A
C007307-UXO-2	100	Not Detected	N/A
C007307-UXO-3	100	Not Detected	N/A
C007307-UXO-4	378	37.8	4

Table 4: Tetryl Concentrations on UXO Remnants

OBSERVATIONS

39. Burn patterns on the coveralls were concentrated on the front of the coveralls around the waist and upper thigh. The burns on the coveralls ranged from large damaged sections to small circular burns.

40. When the coveralls were removed from the plastic bag there was no odour of gasoline or diesel detected.

41. The interior of the UXO appeared to be covered with a black and orange coating. Figure 3 shows the condition of the UXO.

Figure 4: Interior of UXO Remnant

42. The section that contained the fuse appeared to be intact with the exception of the booster portion, of which there was no trace. The fuse was dismantled and the photos are shown below.

Figure 5: UXO with the Fuse Casing Removed

Figure 6: Fuse Casing

Figure 7: Object Removed from Fuse Casing

DISCUSION

43. The primary use of tetryl was from 1916 to 1979 as a component of military explosives (Ref. D). The U.S. stopped using tetryl in 1979 (Ref. D). It is therefore not a common substance. From the shape and size of the UXO recovered it was determined that the device was an AN-M23 WP igniter with an AN-M173 ALL-WAYS fuse. The fuse in this device contains 14 grams of tetryl. Tetryl degrades rapidly in soil (Ref. C). Therefore it is reasonable to assume that the integrity of the UXO has only recently been breached.

44. One theory as to how the accident occurred was that the case of the UXO was cracked by the plow igniting the WP. The heat generated by the WP then set off the initiator. I found no evidence to contradict this hypothesis.

45. White phosphorus oxidizes violently in the presents of oxygen (air). Phosphorus oxide reacts with water (moisture) to form various phosphorus containing acids. Phosphoric acid will etch metal surfaces making them more susceptible to oxidation (rusting). The rust on the remnants of the UXO was superficially and could have occurred in a short period of time.

CONCLUSIONS

46. Significant quantities of Tetryl were found on both the remnants of the UXO and the coveralls that link the two items to the accident. Additionally, the phosphorus levels around the burns on the coveralls indicate that the burns were likely caused by phosphorus.

ACKNOWLEDGEMENTS

47. I would like to thank Mr. Benoit Patry for his work on the GC-MS and Ms. Christina Butler-Jones for her work on the SEM-EDX.

48. In addition I would like to thank Mr. Dan Normandin for his UXO advice and comments.

LABORATORY CHAIN OF CUSTODY FORM

QETE 3-2
General Chemistry and Environmental Issues
Ottawa ON, K1A 0K2

Phone: (819) 994-1675
Fax: (819) 997-4096
Web: http://www.forces.gc.ca

Project No.:	Project Name:	Date:	Sampling Site:	Sample Date and Time:
C007307	Rivers Manitoba	3 July 07	Rivers Manitoba	29 May 2007

Samplers Name:	Sample Description:
Matt Braid	Initiator
Organization: ADM(IE)	**Phone Number:** 613-995-2227
Address: 141 Laurier Ave W. Suite 901 Ottawa Ontario	**Comments:**

Attention: All persons **relinquishing and receiving** the sample must provide their signature, organization and time/date.

	Relinquished by:	Organization	Date/Time	Received by:	Organization	Date/Time
1.	M.P. Braid, Maj	ADM(IE) DREM	3 July 2007 1100 hrs	Kent Brady	QETE-32	July 3, 2007 11:00
2.	Kent Brady	QETE-3-2	July 24, 2007 09:35	Steve Barrett	QETE 3-2	July 24 2007 09:33
3.	Steve Barrett	QETE 3-2	Aug 02/07 1610	H. Fawcus	QETE 3-2	07/08/2 1640
4.	HANK FAWCUS	QETE 3-2	07/08/7	K. Braid	QETE 3-2	Aug 7, 2007 13:10
5.						
6.						

Additional Information:
(1) Munition to be cut into pieces to ship to RCMP.

LABORATORY CHAIN OF CUSTODY FORM

QETE 3-2
General Chemistry and Environmental Issues
Ottawa ON, K1A 0K2

Phone: (819) 994-1675
Fax: (819) 997-4096
Web: http://www.forces.gc.ca

Project No.:	Project Name:	Date:	Sampling Site:	Sample Date and Time:
C007307	Rivers Manitoba	3 July 07	Rivers Manitoba	29 May 2007.

Samplers Name:	Sample Description:
Matt Braid.	1) Coveralls
Organization: ADM(IE)	**Phone Number:** 613-995-2227
Address: 141 Laurier Ave W. Suite 901 Ottawa Ontario	**Comments:**

Attention: All persons relinquishing and receiving the sample must provide their signature, organization and time/date.

	Relinquished by:	Organization	Date/Time	Received by:	Organization	Date/Time
1.	M.P. Braid (1) Maj.	ADM(IE) DREM	3 July 2007 1100 hrs	Kent Brady	QETE 3-2	July 3, 2007 11:00
2.	Kent Brady	QETE-3-2.	July 24, 2007 10:23	MacBride F.G. MacBride	DREM Maj 3	1025 hrs 24 July 07
3.	Relinquished by:	Organization	Date/Time	Received by:	Organization	Date/Time
4.	Relinquished by:	Organization	Date/Time	Received by:	Organization	Date/Time
5.	Relinquished by:	Organization	Date/Time	Received by:	Organization	Date/Time
6.	Relinquished by:	Organization	Date/Time	Received by:	Organization	Date/Time

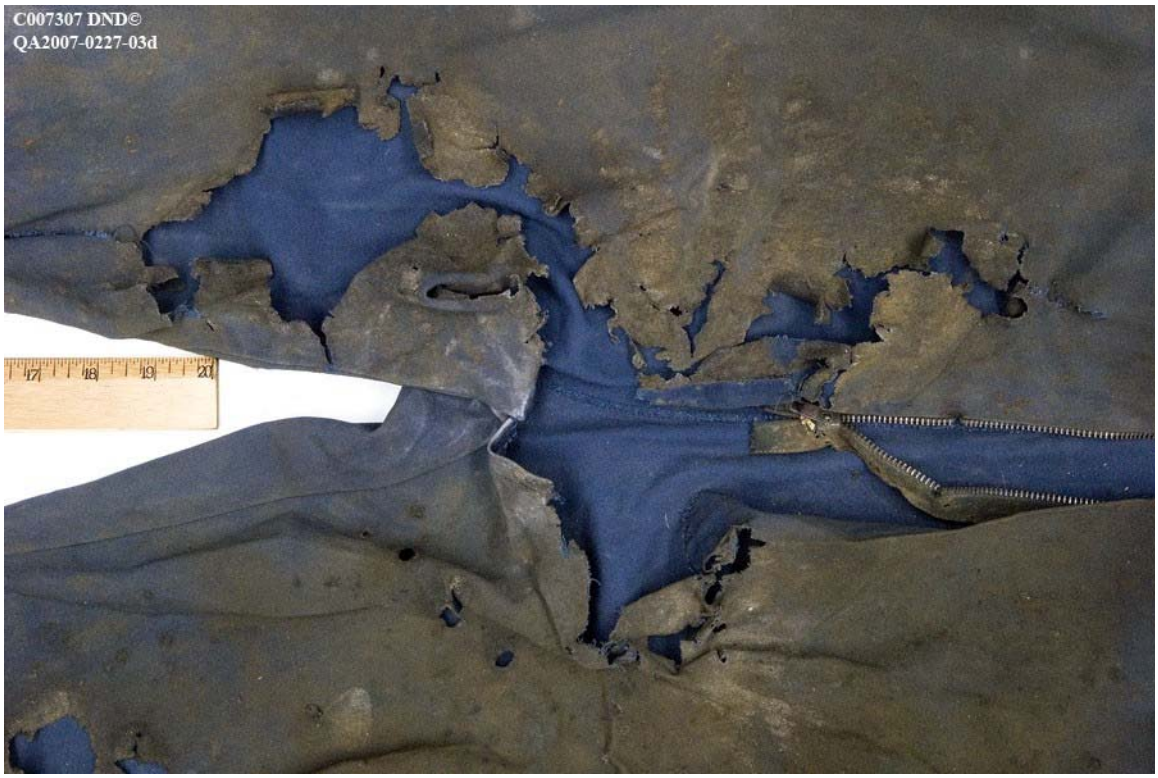
Additional Information:
Samples were removed for Rivers, Manitoba

C007307 DND©
QA2007-0227-01d

A007307 DND©
QA2007-0227-05d

C007307 DND©
QA2007-0227-02d

A007307 DND©
QA2007-0227-04d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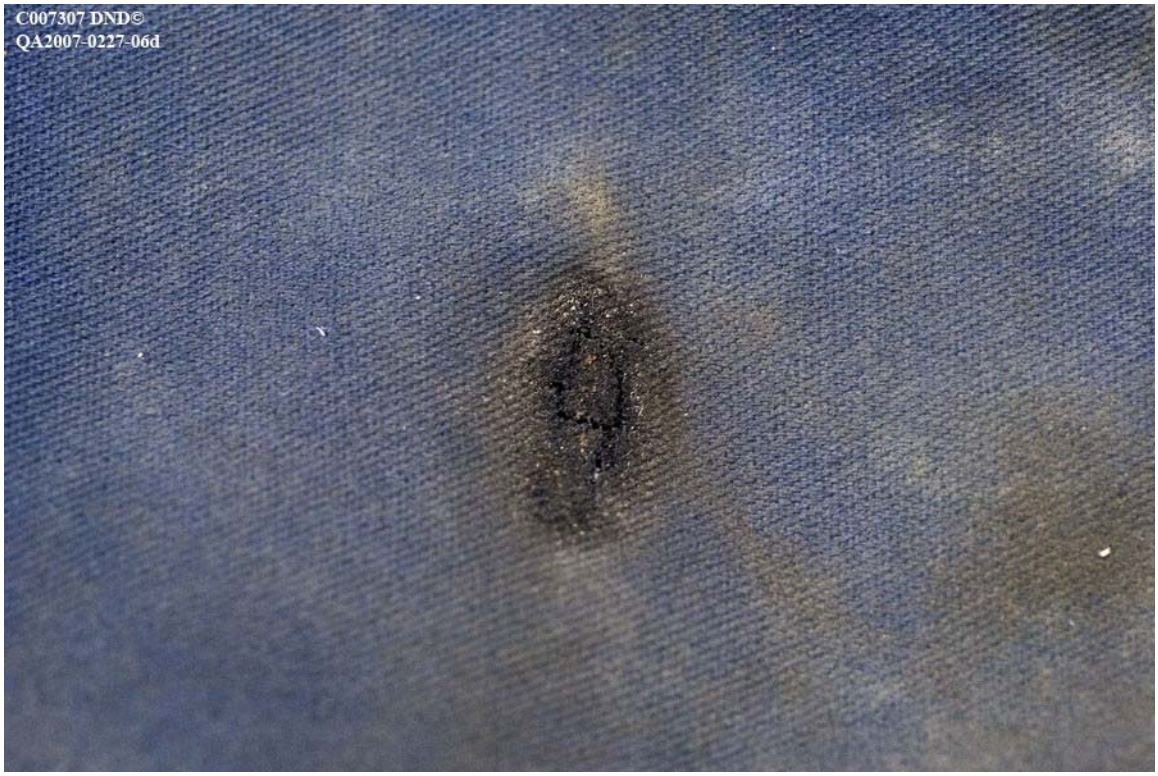
C007307 DND©
QA2007-0227-06d


PERTH'S SERVICES
4/07
341 46760 #25
U/4/07/40--REDFERNS, RIVERS
MATTHEW DICKINSON
C007307 DND©
QA2007-0227-10d


MADE IN CANADA / FABRIQUÉ AU CANADA
UNION MADE
FABRICATION SYNDICALE
40
SY/PAR
anchor textiles
CR 07688 65% POLYESTER
2165
C007307 DND©
QA2007-0227-11d


C007307 DND©
QA2007-0227-07d


C007307 DND©
QA2007-0227-09d


C007307 DND©
QA2007-0227-08d

OBJECTIVE:

(A) Determine the silicon and phosphorus content on the clean, undamaged coverall piece (labelled C007307-COV-4A).
(B) Determine the silicon and phosphorus profiles (starting at a burnt edge and moving away from it) on the following coverall pieces: (1) C007307-COV-3A, (2) C007307-COV-1A, and (3) C007307-COV-1B.

EXAMINATION:

Calibration: A gallium phosphide (GaP) sample on a multi-element standard[1] was examined in the scanning electron microscope (SEM) at a working distance (WD) of 15mm and analysed using the wavelength dispersive X-ray (WDX) Microspec system. The phosphorus $K\alpha_1$ wavelength peak was located at 6.1521 Å (slightly offset from 6.1570 Å - the expected wavelength value) at which point approximately 550 counts per second were detected. The data is stored on the WDX Microspec system under the filename "c007307a".

(A) C007307-COV-4A (Rear Left Shoulder): The clean, undamaged coverall piece removed from the rear left shoulder (labelled C007307-COV-4A) was analysed (at a WD of 15mm and a magnification of 200X) for phosphorus and silicon using the WDX analyser. Only small amounts of silicon were detected (see Figure 1 below and file "c007307l" on the WDX Microspec system).

(B1) C007307-COV-3A (Left Thigh): A piece of the coveralls was removed from the left thigh area (labelled C007307-COV-3A) and provided for WDX analysis (see Photo 1). The piece consisted of three zones (a burnt zone, an undamaged zone, and a transition zone) and various areas across the three zones (labelled k, j, 1, 2, 3, 4, and 5) were analysed for phosphorus and silicon (at a WD of 15mm, a magnification of 200X, and a scan speed of 5). The analysis started at the burnt edge (labelled 'k') and moved towards the undamaged edge (labelled '5'). The results (shown in Figure 1) show that higher phosphorus and silicon levels were detected near the burnt edge and that the levels decreased as the analysis moved towards the undamaged edge. Note that the analysed areas were spaced unevenly apart. The data is stored on the WDX Microspec system under the filenames "c007307k", "c007307j", "c0073071", etc.

[1] *Multi-Element Standard*, Charles M. Taylor Co. Stanford California, 25 July 1978

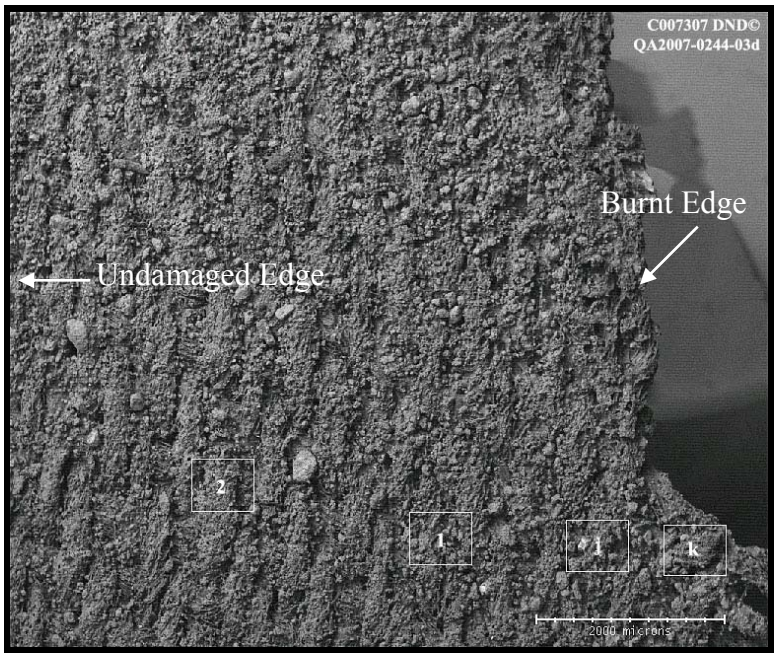

Photo 1: A micrograph of the left thigh piece (C007307-COV-3A) showing some of the areas that were analysed.

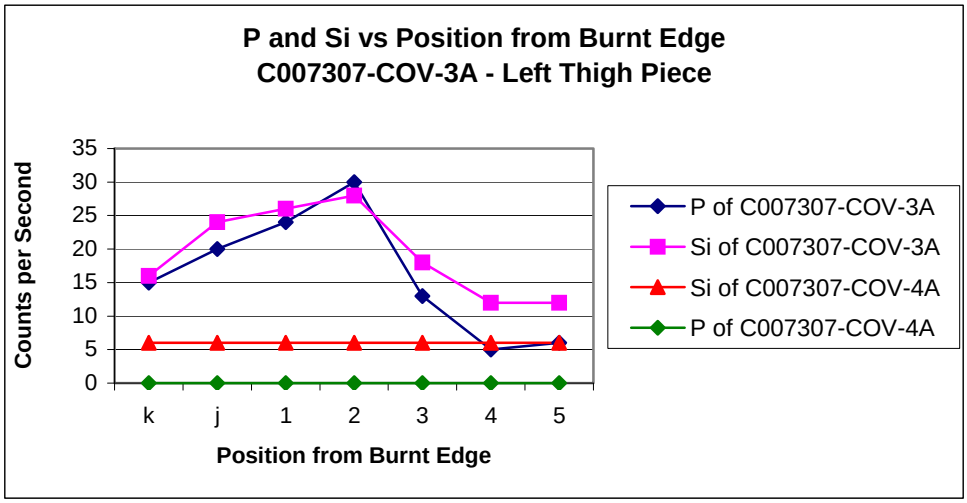

Figure 1: Phosphorus and silicon content on the left thigh piece (C007307-COV-3A). Position 'k' is closest to the burnt edge and '5' is furthest from the burnt edge. The results for the rear left shoulder (C007307-COV-4A) are also shown for comparative purposes.

(B2) C007307-COV-1A (Left Collar Piece A): A piece of the coveralls was removed from the left collar area (labelled C007307-COV-1A) and provided for WDX analysis (see Photo 2). The piece consisted of three zones (a burnt zone, an undamaged zone, and a transition zone) and various areas across the three zones (labelled b, c, d, e, f, and h) were analysed for phosphorus and silicon (at a WD of 15mm, a magnification of 200X, and a scan speed of 5). The analysis started at the burnt edge (labelled 'b') and moved towards the undamaged edge (labelled 'h'). The results (shown in Figure 2) show that higher phosphorus and silicon levels were detected near the burnt edge and that the levels decreased as the analysis moved towards the undamaged

edge. Note that the analysed areas were spaced unevenly apart. The data is stored on the WDX Microspec system under the filenames "c007307b", "c007307c", etc.

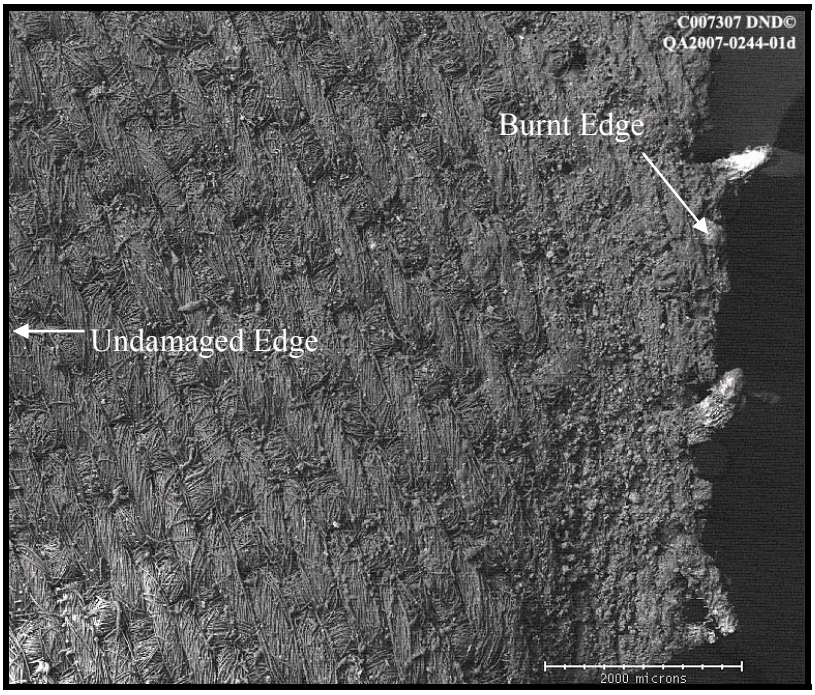

Photo 2: A micrograph of the left collar piece A (C007307-COV-1A).

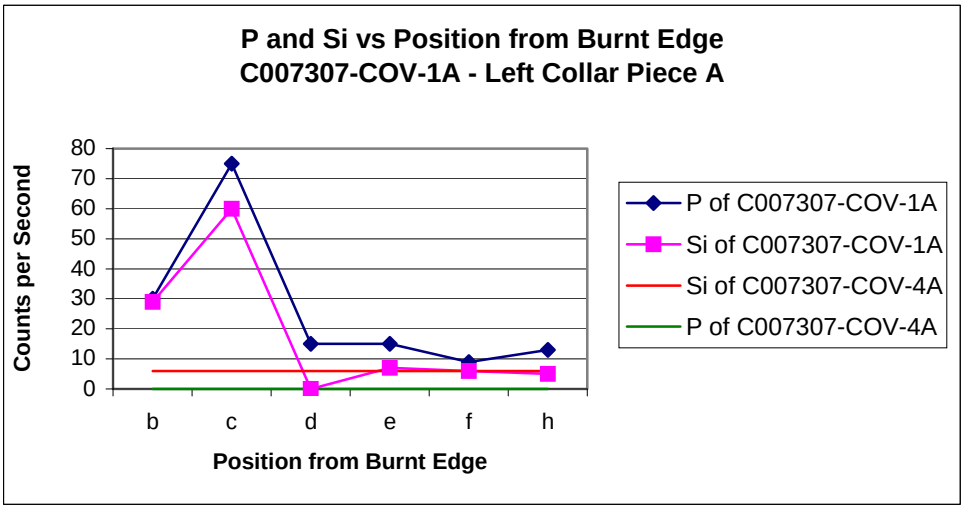

Figure 2: Phosphorus and silicon content on the left collar piece A (labelled C007307-COV-1A). Position 'b' is closest to the burnt edge and 'h' is furthest from the burnt edge. The Si and P content of the rear left shoulder (C007307-COV-4A) are also shown for comparative purposes.

(B3) C007307-COV-1B (Left Collar Piece B): A second piece of the coveralls was removed from the left collar area (labelled C007307-COV-1B) and provided for WDX analysis (see Photo 3). The piece consisted of three zones (a burnt zone, an undamaged zone, and a transition zone) and two sets of analyses were carried out for phosphorus and silicon across the three zones (at a WD of 15mm, a magnification of 200X, and a scan speed of 5). Analysis 1 analysed areas z, y, x, w, v, u, and t (with z closest to the burnt edge and t furthest from the burnt edge) and analysis 2 analysed areas r, q, p, o, n, m, and l (with r closest to the burnt edge and l furthest from the burnt edge). The results (shown in Figures 3 and 4) show that higher phosphorus levels were detected near the burnt edge and that the levels decreased as the analysis moved towards the undamaged edge. The silicon levels for the two analyses followed a similar trend to the phosphorus however the second analysis showed an increase in silicon near the undamaged edge. Note that the analysed areas were spaced unevenly apart. The data is stored on the WDX Microspec system under the filenames "c007307z", "c007307y", etc.

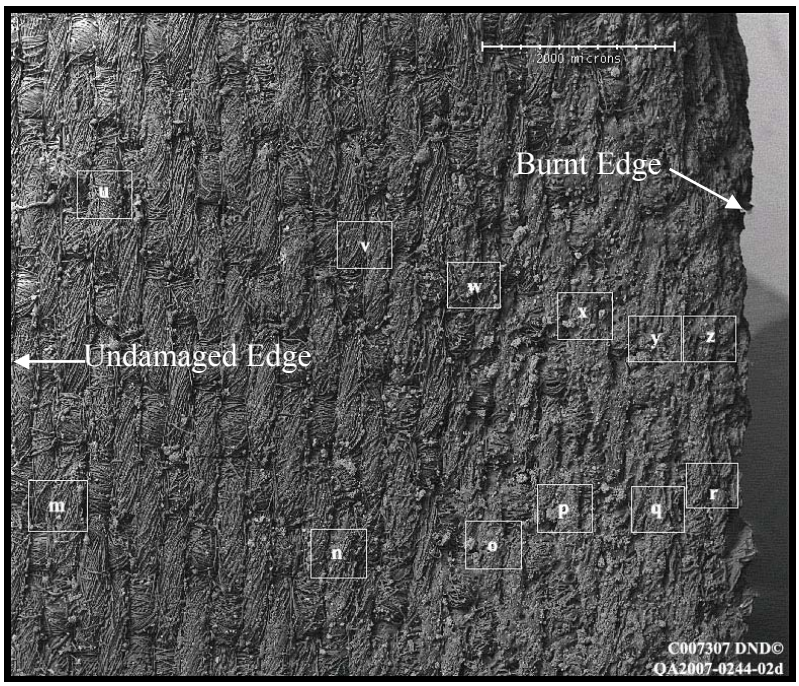

Photo 3: A micrograph of the left collar piece B (C007307-COV-1B) showing some of the areas that were analysed.

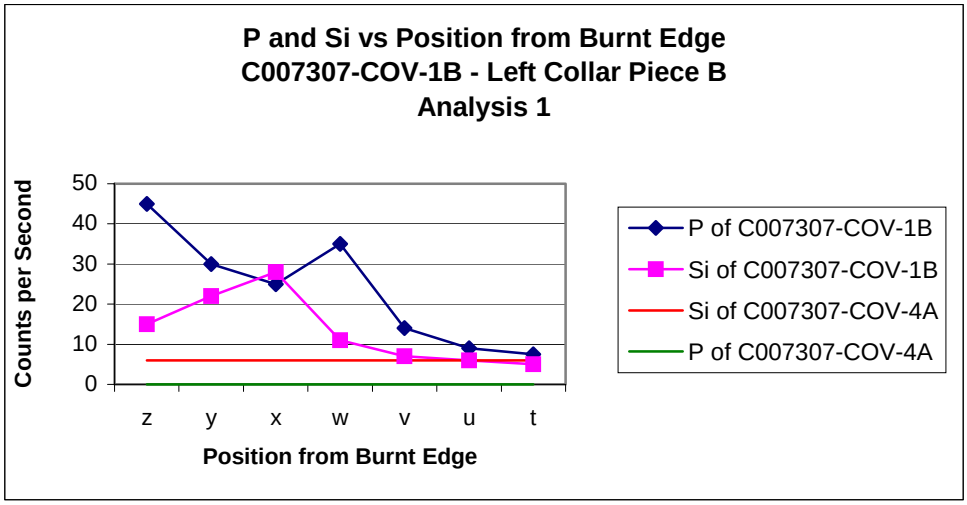

Figure 3: Phosphorus and silicon content for the first analysis of the left collar piece B (labelled C007307-COV-1B). Position 'z' is closest to the burnt edge and 't' is furthest from the burnt edge. The Si and P content of the rear left shoulder (C007307-COV-4A) are also shown for comparative purposes.

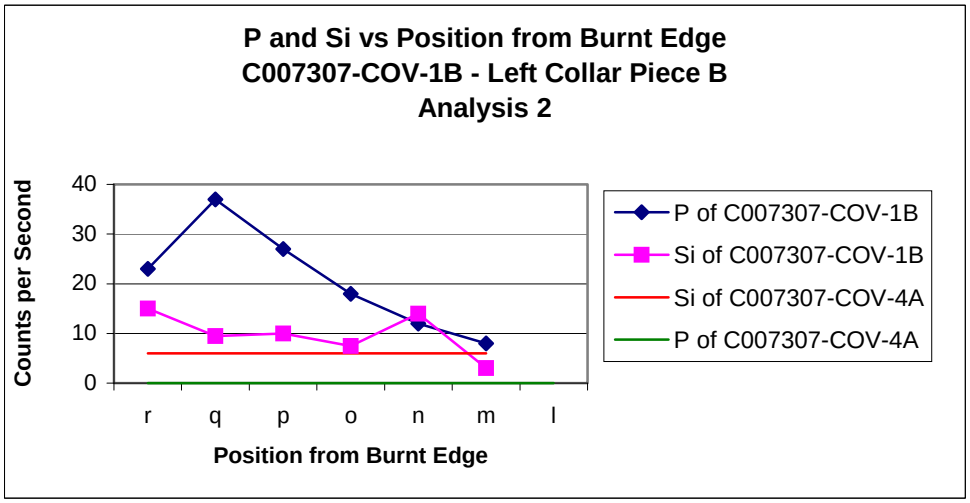

Figure 4: Phosphorus and silicon content for the second analysis of the left collar piece B (C007307-COV-1B). Position 'r' is closest to the burnt edge and 'm' is furthest from the burnt edge. The Si and P content of the rear left shoulder (C007307-COV-4A) are also shown for comparative purposes.

CONCLUSIONS:

WDX analysis of the different burnt coverall samples showed higher phosphorus levels at the burnt edges with a gradual decrease in phosphorus as the analysis moved towards the cleaner, undamaged areas. No phosphorus was detected in the clean and undamaged coverall piece (C007307-COV-4A).

WDX analysis found that the silicon levels for the three of the analyses followed a similar trend to the phosphorus and that the fourth analysis showed an increase in silicon near the undamaged edge. Small levels of silicon were detected in the clean and undamaged coverall piece.

In conclusion, it must be stated that WDX analysis is a very localized analysis and that the detected levels of phosphorus and silicon may not exactly represent their overall distribution in the samples. Further analysis was carried out using energy dispersive X-ray (EDX) analysis (see QETE 702 C007307 - SEM 026a 07).

OBJECTIVE:

(A) Determine if any of the following elements: chlorine, potassium, strontium, barium, aluminum, magnesium, and copper, are detected at the burnt area on coverall pieces C007307-COV-1A, C007307-COV-1B, and C007307-COV-2A. Compare the results with an unburnt coverall piece (labelled as C007307-COV-4A).
(B) Determine and plot the phosphorus line profile from the burnt hole edge of piece C007307-COV-2A to an unburnt area.

EXAMINATION:

(A1) C007307-COV-4A: Energy dispersive X-ray (EDX) analysis was carried out on an undamaged coverall piece removed from the rear left shoulder (labelled C007307-COV-4A). The spectrum (labelled "c007307_cov_4a_left rear shoulder" in Figure 1) indicated that the major[1] elements were carbon and oxygen with minor[2] amounts of iron, aluminum, silicon, potassium, chlorine, and calcium. Sodium and magnesium may also have been present.

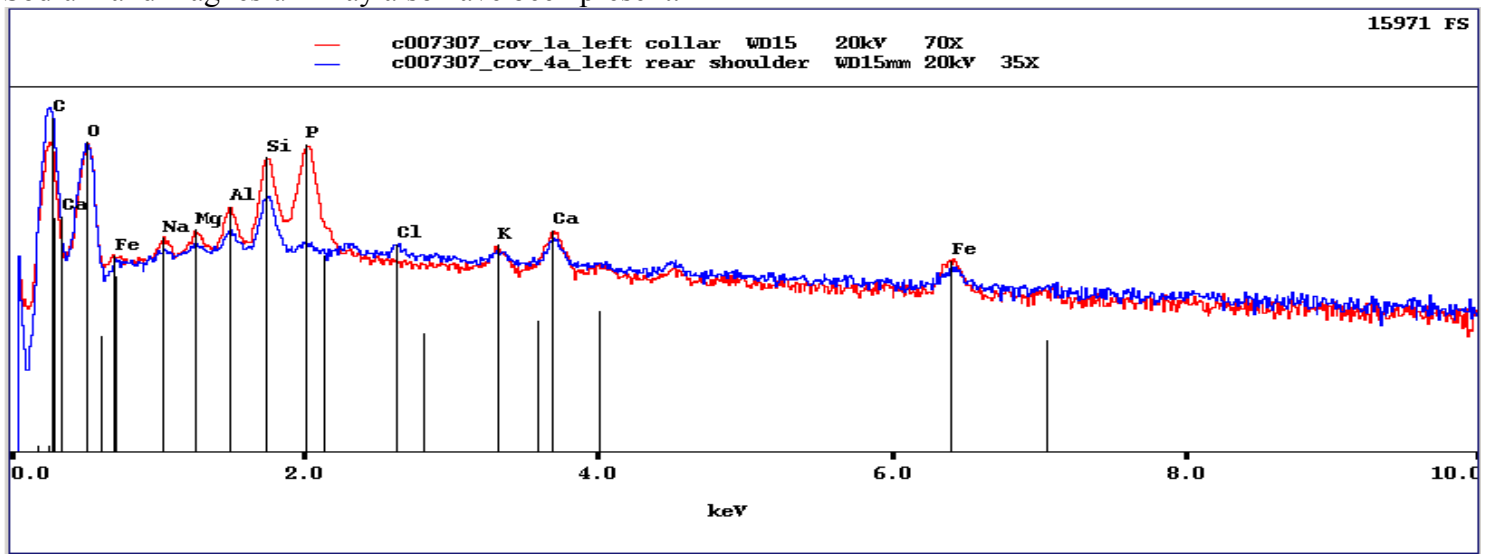

Figure 1: EDX spectra for pieces C007307-COV-1A and C007307-COV-4A.

(A2) C007307-COV-1A: EDX analysis was carried out on a burnt coverall piece removed from the left collar (labelled C007307-COV-1A). The spectrum (labeled "c007307_cov_1a_left collar" in Figure 1) was similar to the parent material (spectrum "c007307_cov_4a_left rear shoulder") except that the former spectrum had a major[1] amount of phosphorus, no chlorine, and a slightly higher aluminum, magnesium, sodium, and silicon content.

(A3) C007307-COV-1B: EDX analysis was carried out on a burnt coverall piece removed from the left collar piece B (labelled C007307-COV-1B). The spectrum (labelled "c007307_cov_1b_left rear shoulder" in Figure 2) was similar to the "c007307_cov_1a_left collar" spectrum.

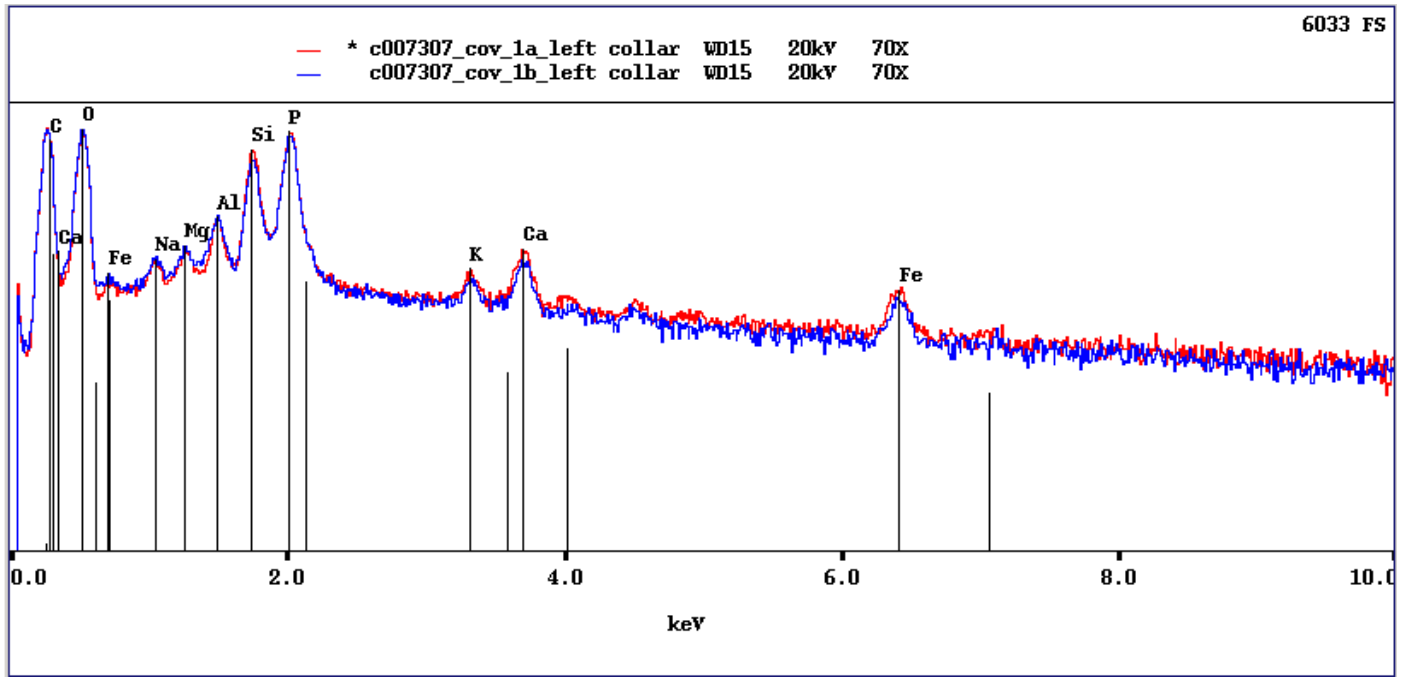

Figure 2: EDX spectra for pieces C007307-COV-1A and C007307-COV-1B.

(A4) C007307-COV-2A: EDX analysis was carried out on a burnt coverall piece removed from the right leg (labelled C007307-COV-2A). The spectrum (labelled "c007307_cov_2a_right_leg_hole" in Figure 3) was similar to the "c007307_cov_1a_left collar" spectrum.

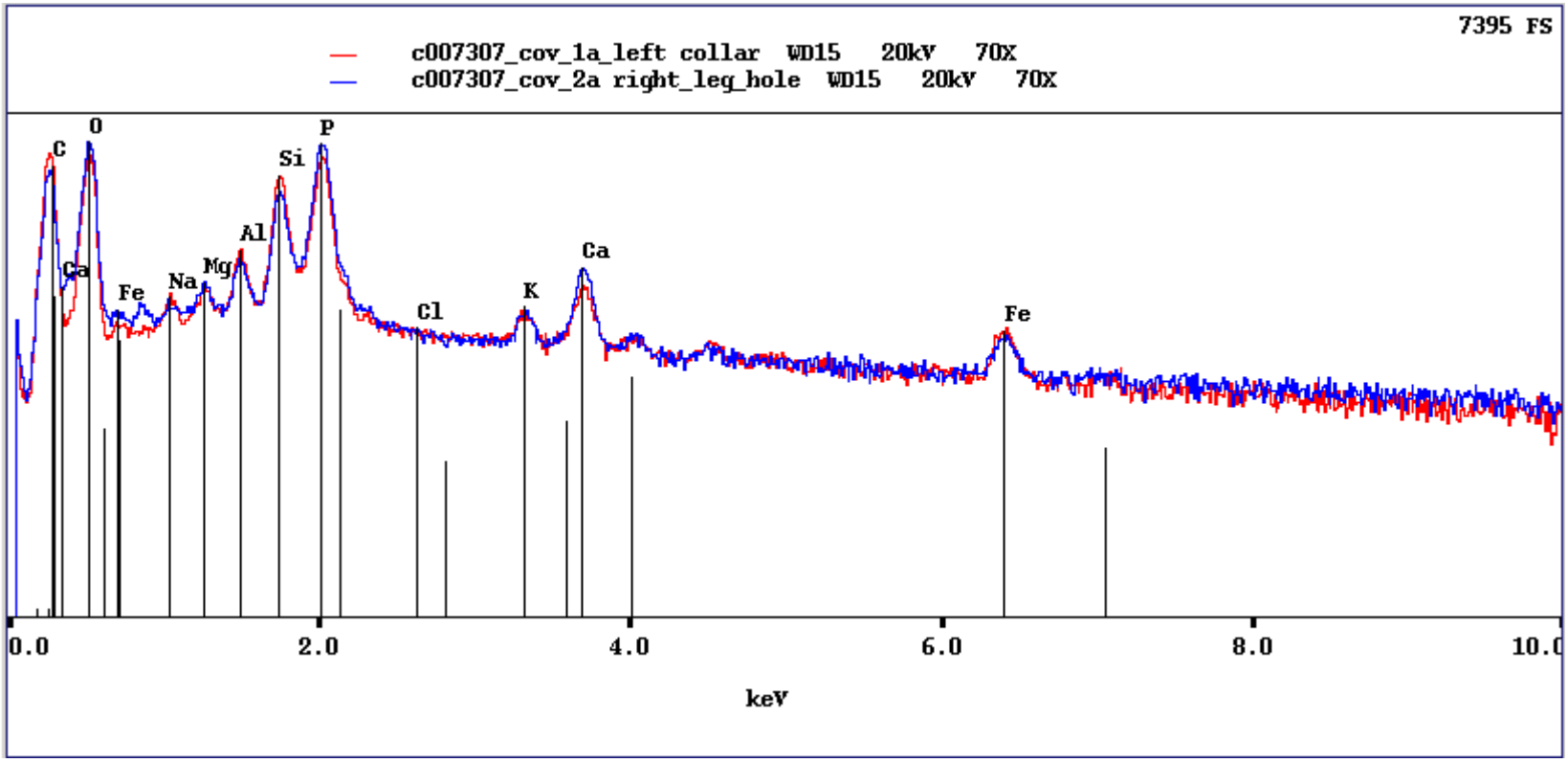

Figure 3: EDX spectra for pieces C007307-COV-1A and C007307-COV-2A.

(B) Phosphorus Profile for C007307-COV-2A: Using piece C007307-COV-2A, a phosphorus line profile was acquired (using the EDX analyzer) from the burn hole to an area away from the burn. The analysis was carried out across three consecutive areas

(as shown in Photo 1). The same equipment parameters (i.e. acquisition time, voltage, current, magnification, etc) were used for each analysis and each analysis was carried out along the bottom line of the red box (as shown in Photo 1). The profile showed that the overall phosphorus content decreased as the analysis moved away from the burnt area.

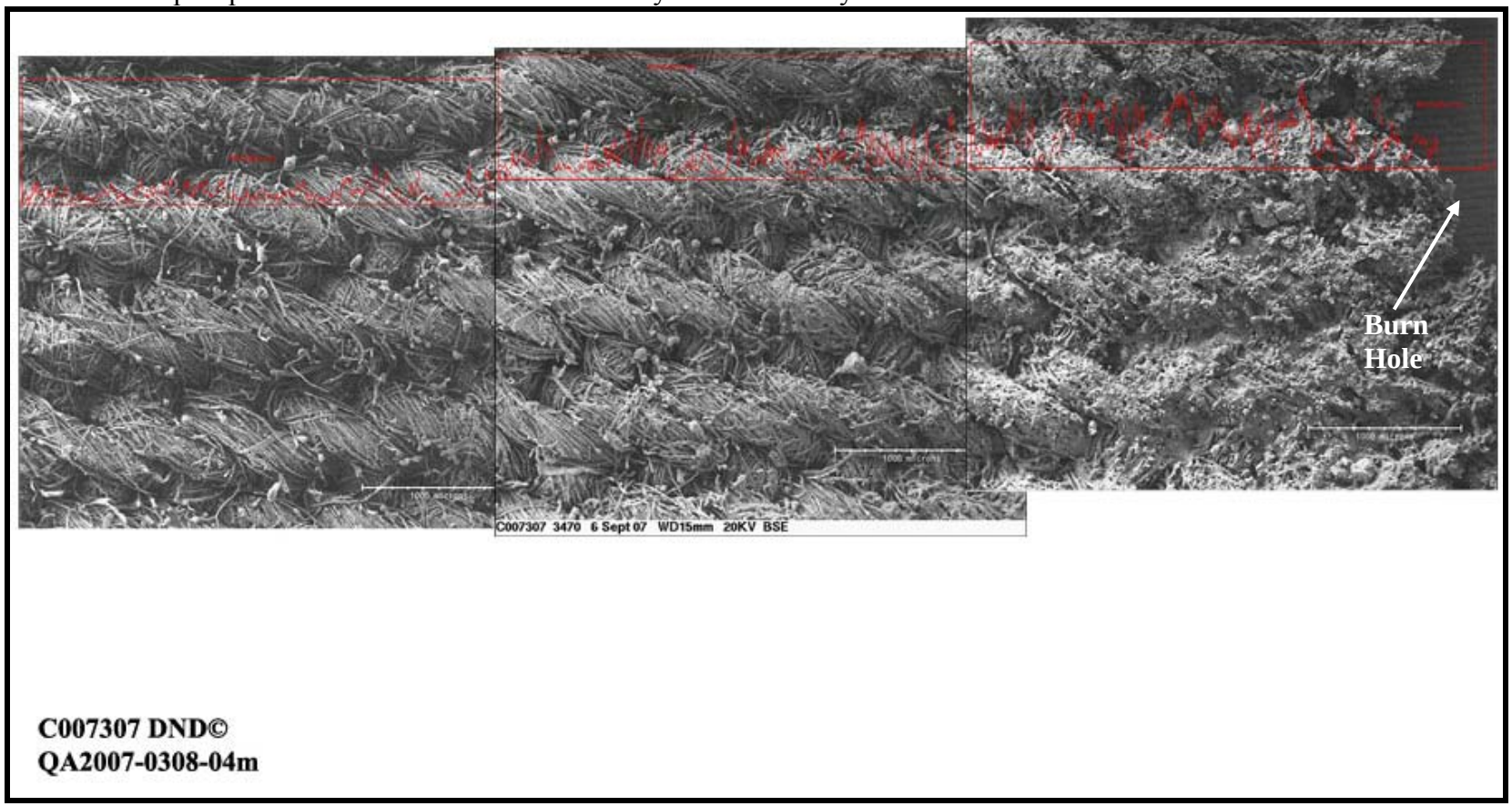

Photo 1: A phosphorus line profile on the C007307-COV-3A sample starting at the burn hole on the right.

CONCLUSIONS:

(A) EDX analysis was carried out on the burnt section of pieces C007307-COV-1A, C007307-COV-1B, and C007307-COV-2A they were similar to the parent material (spectrum "c007307_cov_1a_left rear shoulder") except that the former three spectra had a major amount of phosphorus, no chlorine, and a slightly higher aluminum, magnesium, sodium, and silicon content.

(B) The line profile analysis found that the phosphorus levels decreased away from the burn mark on the C007307-COV-2A piece. This was consistent with the wavelength dispersive X-ray (WDX) analysis that was carried out on pieces C007307-COV-1A, C007307-COV-1B, and C007307-COV-3A (see QETE 702 C007307 - SEM 026 07).

6890 GC METHOD for Coverall Analysis

OVEN

Initial temp: 50 °C (On)
Maximum temp: 320 °C
Initial time: 1.00 min
Equilibration time: 0.50 min
Ramps:

#	Rate (°C/min)	Final temp (°C)	Final time (min)
1	25.00	250	4.00
2	0.0(Off)		

Post temp: 0 °C
Post time: 0.00 min
Run time: 13.00 min

INLET (SPLIT/SPLITLESS)

Mode: Splitless
Initial temp: 220 °C (On)
Pressure: 2.20 psi (On)
Purge flow: 50.0 mL/min
Purge time: 1.00 min
Total flow: 64.0 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 2.00 min
Gas type: Helium

COLUMN

Capillary Column
Model Number: J&W DB-5
Max temperature: 320 °C
Nominal length: 7.5 m
Nominal diameter: 530.00 µm
Nominal film thickness: 1.50 µm
Mode: constant flow
Initial flow: 11.1 mL/min
Nominal init. pressure: 2.20 psi
Average velocity: 85 cm/sec
Outlet: MSD
Outlet pressure: ambient

THERMAL AUX 2

Use: MSD Transfer Line Heater
Initial temp: 210 °C (On)

GC Injector

Injection Volume 1.00 microliters
Syringe Size 10.0 microliters

MS ACQUISITION PARAMETERS

Acquisition Mode : Scan/SIM

Solvent Delay: 2.00 min

EM Absolute: False
EM Offset: 0
Resulting EM Voltage: 1200.0

Raw Scan Parameters

Low Mass: 40.0
High Mass: 400.0
Threshold: 150
Sample #: 2 A/D Samples 4

Sim Parameters

Group ID: 1
Resolution: Low
Plot 1 Ion: 242.00
Ions/Dwell In Group

(Mass, Dwell)	(Mass, Dwell)	(Mass, Dwell)
(123.00, 100)	(137.00, 100)	(168.00, 100)
(213.00, 100)	(242.00, 100)	

MS Zones

MS Quad : 150 C maximum 200 °C
MS Source : 150 C maximum 300 °C

6890 GC METHOD for UXO Analysis

OVEN

Initial temp: 90 °C (On)
Maximum temp: 325 °C
Initial time: 0.00 min
Equilibration time: 0.50 min
Ramps:

#	Rate (°C/min)	Final temp (°C)	Final time (min)
1	20.00	230	0.00
2	7.0	240	1.00
3	50.00	280	3.00
4	0.0(off)		

Post temp: 0 °C
Post time: 0.00 min
Run time: 13.23 min

INLET (SPLIT/SPLITLESS)

Mode: Splitless
Initial temp: 50 °C (off)
Pressure: 13.79 psi (On)
Purge flow: 66.4 mL/min
Purge time: 0.00 min
Total flow: 70.2 mL/min
Gas saver: On
Saver flow: 20.0 mL/min
Saver time: 2.00 min
Gas type: Helium

COLUMN

Capillary Column
Model Number: J&W DB-5
Max temperature: 320 °C
Nominal length: 7.5 m
Nominal diameter: 530.00 µm
Nominal film thickness: 1.50 µm
Mode: constant flow
Initial flow: 11.1 mL/min
Nominal init pressure: 2.20 psi
Average velocity: 85 cm/sec
Outlet: MSD
Outlet pressure: ambient

THERMAL AUX 2

Use: MSD Transfer Line Heater
Initial temp: 210 °C (On)

GC Injector

Injection Volume 1.00 microliters
Syringe Size 10.0 microliters

MS ACQUISITION PARAMETERS

Acquisition Mode: Scan/SIM

Solvent Delay: 1.50 min

EM Absolute: False
EM Offset: 0
Resulting EM Voltage: 1200.0

Raw Scan Parameters

Low Mass: 40.0
High Mass: 400.0
Threshold: 150
Sample #: 2 A/D Samples 4

Sim Parameters

Group ID: 1
Resolution: Low
Plot 1 Ion: 123.00
Ions/Dwell In Group

(Mass, Dwell)	(Mass, Dwell)	(Mass, Dwell)
(62.00, 50)	(123.00, 50)	(168.00, 50)

Group ID: 2
Resolution: Low
Group Start Time: 3.75
Plot 1 Ion: 168.00
Ions/Dwell In Group

(Mass, Dwell)	(Mass, Dwell)	(Mass, Dwell)
(62.00, 50)	(168.00, 50)	(182.00, 50)
(213.00, 50)	(227.00, 50)	

Group ID: 3
Resolution: Low
Group Start Time: 5.80
Plot 1 Ion: 102.00
Ions/Dwell In Group

(Mass, Dwell)	(Mass, Dwell)	(Mass, Dwell)
(62.00, 50)	(102.00, 50)	(183.00, 50)
(197.00, 50)	(242.00, 50)	

Group ID: 4
Resolution: Low
Group Start Time: 8.00
Plot 1 Ion: 102.00
Ions/Dwell In Group

(Mass, Dwell)	(Mass, Dwell)
(102.00, 100)	(176.00, 100)

MS Zones

MS Quad : 150 C maximum 200 °C
MS Source : 150 C maximum 300 °C