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Analysis of Aqueous Field Samples for Mustard Degradation Products: A Joint CA/US TTCP E-AG-42 Analytical Exercise

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January 1999

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National Defence Défense nationale

Canada

Suffield Memorandum No. 1520

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ABSTRACT i

CA and US participated in a joint TTCP E-AG-42 analytical exercise involving the analysis of aqueous field samples for the presence of mustard degradation products. The laboratories reported consistent results that clearly indicated the prior presence of mustard in a destruction site more than twenty years after the fact. Both laboratories used mass spectrometry for confirmation of the hydrolysis product of mustard, thiodiglycol. The US confirmed thiodiglycol as its bis(TMS) derivative during GC-MS and CA as the intact compound during GC-MS and LC-ESI-MS analysis. In addition both laboratories reported the presence of 1,4-thioxane and 1,4-dithiane, two compounds commonly associated with the degradation of mustard. CA also detected 6-oxa-3,9-dithia-1,11-undecanediol, the hydrolysis product of the longer chain sulfur vesicant, bis[(2-chloroethylthio)ethyl] ether (T) during LC-ESI-MS analysis.

Executive Summary ii

Title: P.A. D'Agostino, J.R. Hancock, L.R. Provost and K. Taylor, "Analysis of Aqueous Field Samples for Mustard Degradation Products: A Joint CA/US TTCP-E-AG-42 Analytical Exercise", Suffield Memorandum No. 1520, 1999, UNCLASSIFIED.

Introduction: During World War II over 700 tons of the chemical warfare agent mustard were shipped to Defence Research Establishment Suffield (DRES) and stored in lead-lined concrete vaults. In the early 1970's it was decided that this stockpile of mustard would be destroyed by hydrolysis. Batch hydrolysis using 1000 gallons of mustard, 5000 pounds of lime ($Ca(OH)_2$) and 2500 gallons of water was carried out according to a method developed at DRES. Although the majority of the mustard hydrolysate was removed from the site, a residual amount remained. The residual hydrolysate was contained and encased with the remains of the vaults after mustard destruction. Monitoring wells were established near the destruction site to enable future sampling. Sampling of the wells was performed in 1997 when TTCP E-AG-42 (Chemical Weapons Convention Analytical Technologies), as part of its mandate, analysed aqueous samples from three of the sampling wells. This report summarizes the results of the analyses performed on the aqueous samples distributed to the US (IIT Research Institute) and CA (DRES) laboratories for joint study.

Results: Both laboratories used mass spectrometry for the confirmation of thiodiglycol, the US as its bis(TMS) derivative during GC-MS and CA as the intact compound during GC-MS and LC-ESI-MS analysis. In addition both laboratories reported the presence of 1,4-thioxane and 1,4-dithiane, two compounds commonly associated with the degradation of mustard. CA also detected 6-oxa-3,9-dithia-1,11-undecanediol, the hydrolysis product of the longer chain sulfur vesicant, bis[(2-chloroethylthio)ethyl] ether (T) during LC-ESI-MS analysis.

Significance of Results: CA and US participated in a joint TTCP E-AG-42 analytical exercise involving the analysis of aqueous field samples for the presence of mustard degradation products. The participants reported consistent results that indicated the prior presence of mustard in a destruction site more than twenty years after the fact. The reported methods could be used for the identification of thiodiglycol and other sulfur vesicant degradation products in samples collected during military operations or in support of United Nations Chemical Weapons Convention inspections.

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INTRODUCTION

During World War II over 700 tons of the chemical warfare agent mustard were shipped to Defence Research Establishment Suffield (DRES) and stored in lead-lined concrete vaults (1). In the early 1970's it was decided that this stockpile of mustard would be destroyed by hydrolysis. Batch hydrolysis using 1000 gallons of mustard, 5000 pounds of lime ($Ca(OH)_2$) and 2500 gallons of water was carried out according to a method developed at DRES (2,3).

The principal reactions involved in the hydrolysis of mustard are shown in the equations below (4). Conversion of mustard, through hemisulfur mustard to thiodiglycol, was essentially complete provided the ratio of water to mustard was large, the temperature was elevated to 100°C and the pH was maintained above 7 (2).

$$Cl\text{-}CH_2CH_2\text{-}S\text{-}CH_2CH_2\text{-}Cl + H_2O \rightarrow Cl\text{-}CH_2CH_2\text{-}S\text{-}CH_2CH_2\text{-}OH + HCl$$

$$Cl\text{-}CH_2CH_2\text{-}S\text{-}CH_2CH_2\text{-}OH + H_2O \rightarrow HO\text{-}CH_2CH_2\text{-}S\text{-}CH_2CH_2\text{-}OH + HCl$$

$$Ca(OH)_2 + 2\,HCl \rightarrow Ca(Cl)_2 + 2\,H_2O$$

Following batch hydrolysis the mustard hydrolysate was transferred from the reaction vessel into one of five empty storage vaults. After a cooling and settling period the hydrolysate separated into two layers. The upper or liquid layer was very fluid and ranged in colour from clear to a pale yellow in colour. The lower or sludge layer was paste-like and yellow-brown in colour. Samples of the liquid and sludge layers from the vaults containing the mustard hydrolysate were analysed for thiodiglycol, mustard and other organic content. Thiodiglycol was found in the 2 to 10 mg/mL range in the liquid hydrolysate and 6 to 14 mg/g range in the sludge hydrolysate (5). Mustard was found at trace levels in two sludge samples (5) and a number of other sulfur containing compounds were identified in extracts of the sludge and liquid samples (6).

Although the majority of the mustard hydrolysate was removed from the vaults, a residual amount remained. The residual hydrolysate was contained and encased with the remains of the vault after mustard destruction. Monitoring wells were established near this site to enable future sampling. Sampling was performed in 1984, 1986 and 1987 and thiodiglycol was found at site #5 at 3.9 mg/mL, 0.6 mg/mL and 2.2 mg/mL, respectively (7). Hexane extracts of these aqueous samples were analysed by capillary column gas chromatography-mass spectrometry (GC-MS) under electron impact (EI) and chemical ionization (CI) conditions and found to contain a number of sulfur containing compounds including 1,4-thioxane and 1,4-dithiane (7).

Sampling of the wells was not performed again until 1997 when TTCP E-AG-42 (Chemical Weapons Convention Analytical Technologies), as part of its mandate, analysed aqueous from three of the sampling wells, including site #5. This report summarizes the results of the analyses performed on the aqueous samples distributed to the US (IIT Research Institute) and CA (DRES) laboratories for joint study under TTCP E-AG-42.

EXPERIMENTAL

a) Samples

Aqueous samples for the TTCP E-AG-42 exercise were taken from three sampling wells located at the mustard destruction site. Samples #1, #2 and #3 were collected from sites # 3, #5 and #6, respectively. The exact locations of the sampling wells have been reported internally.

b) CA analyses

GC-FID and GC-MS Analysis of Hexane Extracts

25 mL of each aqueous sample (#1, #2 and #3) were extracted in a 40 mL separatory funnel with 3x5 mL of hexane and the hexane removed and concentrated under a gentle stream of nitrogen to 5 mL. The hexane extracts were analysed by GC-FID and GC-MS (VG Trio-1 under EI conditions: 70 eV, 0.15 mA, 200°C) using a 15m x 0.32mm ID J+W DB-1701 capillary column and the following temperature program: 40°C (2 min) 10°C/min 280°C (2 min). Hewlett-Packard 5890 gas chromatographs were used for GC separations and injections were cool on-column at 40°C. The VG Trio-1 mass spectrometer was scanned from 40 to 340 u at 1 sec/scan.

GC-FID and GC-MS Analysis of Aqueous Samples

A Hewlett-Packard 5890 gas chromatograph was used for GC separations and injections were on-column at 110°C. The aqueous extracts were filtered through cotton gauze and analysed directly by GC-FID and GC-MS (EI) using a 15m x 0.32mm ID J+W DBWAX capillary column and the following temperature program: 110°C (2 min) 10°C/min 225°C (2 min).

LC-ESI-MS Analysis of Aqueous Samples

Sample #2 was filtered and analysed by packed capillary LC-MS using electrospray ionization. LC separations were performed with a Zorbax 150mm x 0.32mm i.d. C_{18} SB (5 μm) packed fused-silica capillary column and a Rheodyne 8125 injector equipped with a 5 μL sample loop. The following solvent compositions were prepared for sample introduction: Solvent A (0.1% trifluoroacetic acid (TFA) in water) and Solvent B (0.1% TFA in acetonitrile/water, 95:5). Chromatographic separations were performed with an Applied Biosystems model 140B dual syringe pump using a 1% to 75%B gradient over 30 minutes. In order to minimize dead volume effects and ensure reproducible mixing, the mobile phase was delivered at 200 μL/min and split prior to the injector such that the flow through the column was 5 μL/min.

All electrospray mass spectra were acquired using a Micromass Autospec-Q tandem mass spectrometer equipped with the Mark II electrospray interface. The electrospray needle was operated at 7.6 kV and ions were accelerated into the mass spectrometer at 4 kV. A sampling cone voltage of 40 V was utilized. Nitrogen (Very Dry) bath gas was introduced into the interface (80 °C) at a flow rate of 400 L/hr. Nitrogen nebulizer gas was introduced at a flow rate of 14 L/hr. The electrospray interface was pumped with both a rotary and a turbomolecular pump, which enabled maintenance of a $4x10^{-4}$ and $7x10^{-6}$ Pa within the source and analyzer regions of the instrument, respectively. LC-ESI-MS data were acquired in the continuum mode by scanning the magnetic sector from 340 to 50 u (7 sec/decade) or 600 to 100 u (7 sec/decade) with a resolution of 1000 (10% valley definition). Three to five scans were typically averaged to enhance the signal-to-noise ratio.

c) US Analyses

Liquid/Liquid Extraction

A 10 mL aliquot of the water sample was tested for pH using pH paper. All samples were between 6 and 8, so no pH adjustment was made. The sample was placed in a 60 mL separatory funnel and 1 mL of methylene chloride was added. The separatory funnel was capped and shaken for 30 seconds. The pressure was vented and the layers allowed to separate. The lower methylene chloride layer was withdrawn and placed in a 1 dram sample vial for GC-MS analysis. Sample #2 required filtration due to excessive particulate. This sample was filtered through a 0.45μm Teflon syringe filter into a clean sample vial. For quantitation 10 mL of sample #2 was extracted with 2 mL of methylene chloride and 80 μL of a 1.00 mg/mL hexachlorobenzene (HCB) internal standard was added to the sample extract.

Silylanation Procedure

A 5 mL aliquot of the water sample was placed in an 8 mL sample vial and the vial placed in a vial heater at 60°C, under a steady stream of nitrogen (about 200 mL/min). The sample was evaporated to dryness using this procedure. The dried residue was then mixed with 0.5 mL of tetrahydrofuran, capped and shaken and then 0.5 mL of bis(trimethylsilyl)trifluoroacetamide (BSTFA) was added. The sample was capped, shaken and heated to 60°C for 30 minutes. The sample was allowed to cool to room temperature and transferred to a 1 dram sample vial for analysis. For quantitation 40 μL of the 1.00 mg/mL hexachlorobenzene/pyrene internal standard was added to the sample #2.

Instrumental

All samples were analysed using a Hewlett-Packard Model 5890 Series II GC equipped with

electronic pressure control and coupled to a Hewlett-Packard Model 5972 mass selective detector (MSD). A Restek RTx-5 column; 30 m x 0.25 mm ID, 0.25 μm phase was used for component separation. Conditions were as follows:

GC carrier gas:	He at 35 cm/sec
GC injection:	1 μL splitless at 250°C
GC temperature program:	40°C (1 min) 10°C/min 280°C (10 min)
MS conditions:	EI at 70 eV
MS scan:	41 to 400 u at 2 sec/scan
MS solvent delay:	3.90 min for CH_2Cl_2 extract, 7.0 min for BSTFA/THF sample

Quantitation

Standards of 1,4 dithiane and thiodiglycol were prepared for quantitation. A stock 1,4 dithiane standard was prepared by weighing out 0.1179g of dithiane (Aldrich Lot # 09218CF, 97%) and diluting to 25 mL volume with methylene chloride. From the stock, two dilute solution standards were prepared, at 56.6 and 4.72 μg/mL dithiane each containing 40.0 μg/mL hexachlorobenzene as the internal standard.

A stock thiodiglycol standard was prepared by weighing out 0.0983g of thiodiglycol (Aldrich Lot # 16717JN, 99 + %) and diluting to 25 mL with tetrahydrofuran. One mL, of the stock was combined with 1 mL of BSTFA and heated to 60°C for 30 minutes to derivatize. The sample was cooled and diluted to a final volume of 25 mL with methylene chloride. This derivatized standard was then combined with the hexachlorobenzene (HCB) internal standard and diluted to volume with methylene chloride to prepare thiodiglycol at 50.3 and 4.72 μg/mL each containing 40.0 μg/mL hexachlorobenzene as the internal standard. No correction was made for the conversion of thiodiglycol to the bis(TMS) derivative. Therefore, all quantitation for thiodiglycol is based on the original mass of thiodiglycol and assumes 100% conversion to the bis(TMS) derivative.

RESULTS AND DISCUSSION

a) CA Results (Performed in September 1997)

Mustard was not detected in any of the three aqueous samples analysed by GC-FID and GC-MS (EI) analysis. If present mustard would be below the GC-FID detection limit of 0.0002 mg/mL (S/N = 3:1).

Only one sample, sample #2, contained organic content in the hexane extract. The other two samples, samples #1 and #3, were free of organic content and exhibited chromatograms consistent with the solvent blank. The following compounds were identified in the hexane extract of sample #2 based comparison of acquired MS data with library data: 1,4-thioxane, 1,4-dithiane, 1-oxa-4,5-dithiapane, 4-methylphenol (or another isomer) indole, 4-methyl indole (or another isomer). Quantitation was not performed on these compounds.

Thiodiglycol was confirmed only in sample #2 during direct aqueous analysis using a DBWAX column during GC-FID and GC-MS (EI) analysis. Thiodiglycol was detected at 0.2 mg/mL based on external calibration. A similar level was estimated during LC-ESI-MS analysis. No derivatization procedures were employed for thiodiglycol analysis.

The hydrolysis products of sulfur vesicants would generally be associated with aqueous samples. Development of capillary electrophoresis (CE) or liquid chromatography-mass spectrometry (LC-MS) for their detection and identification could reduce sample handling and derivatization requirements. Direct aqueous MS analysis methods for mustard hydrolysis products have made use of either loop injection MS or LC-MS. A notable exception involved the use of micellar electrokinetic chromatography with UV detection for the detection of thiodiglycol, 1,4-dithiane, 1,4-thioxane and 2,2'-sulfinyldiethanol (8). Thermospray (9), atmospheric pressure chemical ionization (APCI) (10) and electrospray (11) mass spectrometric interfaces have all been

used to facilitate the introduction and ionization of mustard hydrolysis products. In these cases, the investigations focussed on the analysis of thiodiglycol, half-mustard, thiodiglycol sulfone and thiodiglycol sulfoxide, compounds commonly associated with the degradation of mustard. More recently a packed capillary LC-ESI-MS method was developed at DRES to detect and identify thiodiglycol and the hydrolysis products of the longer chain sulfur vesicants contained in munitions grade mustard samples (12). ESI-MS provided ample molecular ion information and structurally important product ion information were generated by promoting collisionally activated dissociation (CAD) in the ESI interface. The method was applied to the analysis of aqueous sample #2.

Figure 1 illustrates the total-ion-current chromatogram obtained for sample #2 during LC-ESI-MS analysis. Thiodiglycol was detected along with 6-oxa-3,9-dithia-1,11-undecanediol, the hydrolysis product of the longer chain sulfur vesicant, bis[(2-chloroethylthio)ethyl] ether (T). Molecular masses were confirmed by the presence of $(M+H)^+$, $(M+NH_4)^+$ and $(M+Na)^+$ ions and both spectra contained product ions observed during analysis of these compounds in HT and HQ hydrolysis samples (12).

b) US Results (Performed in January 1998)

The water samples were prepared for analysis by two methods, liquid/liquid extraction with methylene chloride and evaporation of water followed by silylanation with bis-(trimethylsilyl)trifluoroacetamide (BSTFA) in tetrahydrofuran (THF). All sample preparations were analysed using GC-MS. Only sample #2 showed any organic content using the liquid/liquid extraction procedure. Compounds identified in sample #2 were: 1,4-thioxane, 1,4-dithiane, phenol, a methylphenol, 1,4-thioxane-4,4-dioxide, indole, a methylindole, nonadecene and hexadecanioc acid. 1,4-Dithiane and 1,4-thioxane were quantitated using the internal standard method with a standard containing 1,4-dithiane and hexachlorobenzene as the internal standard. The analysed concentrations were 2.7 and 10. 1 μg/mL, respectively.

The sample evaporation/silylanation procedure was used to confirm the presence of thiodiglycol as its bis(TMS) derivative in sample #2. Thiodiglycol was not detected in either samples #1 or #3. Quantitation of thiodiglycol was performed using the internal standard method with a standard containing thiodiglycol and hexachlorobenzene. The thiodiglycol concentration was calculated to be 0.0566 mg/mL, based on the TMS reaction going to completion. This assumption, the fact that the standard was about ten times lower in concentration than the sample or the fact that the analyses were completed over a fairly lengthy timeframe (about five months) may have contributed to the lower concentration estimate by the US. Figure 2a illustrates the total-ion-chromatogram for sample #2 following GC-MS analysis. Figure 2b illustrates the mass spectrum obtained for the bis(TMS) derivative of thiodiglycol obtained for this sample.

CONCLUSIONS

CA and US, as part of a TTCP E-AG-42 analytical exercise, analysed three aqueous sample for the present of mustard degradation products. Samples #1 and #3 did not contain thiodiglycol or other significant organic content. Sample #2 contained compounds indicative of the prior presence of mustard. Both laboratories reported the presence of thiodiglycol, the principal mustard hydrolysis product. Thiodiglycol was quantitated directly by CA and as the bis(TMS) derivative by the US. The thiodiglycol concentration was calculated to be 0.0566 mg/mL by the US, based on the TMS reaction going to completion. This assumption, the fact that the standard was about ten times lower in concentration than the sample or the fact that the analyses were completed over a fairly lengthy timeframe (about five months) may have contributed to the lower concentration estimate by the US. CA concluded the analyses prior to the US and recorded a higher thiodiglycol concentration, 0.2 mg/mL.

1,4-thioxane and 1,4-dithiane, two compounds commonly associated with the degradation of mustard were also detected in the organic extracts of sample #2 by both laboratories. Both laboratories also reported the presence of phenols and indoles, compounds that may be associated with decaying material at the site. The US used a more polar extraction solvent, methylene chloride, which may explain the presence of several additional extract components.

The TTCP E-AG-42 joint exercise yielded consistent results that clearly indicated the prior presence of mustard in a destruction site more than twenty years after the fact. Both laboratories used mass spectrometry to confirm thiodiglycol, the US as its bis(TMS) derivative during GC-MS and CA as the intact compound during GC-MS and LC-ESI-MS analysis. In addition CA detected 6-oxa-3,9-dithia-1,11-undecanediol, the hydrolysis product of the longer chain sulfur vesicant, bis[(2-chloroethylthio)ethyl] ether (T) during LC-ESI-MS analysis.

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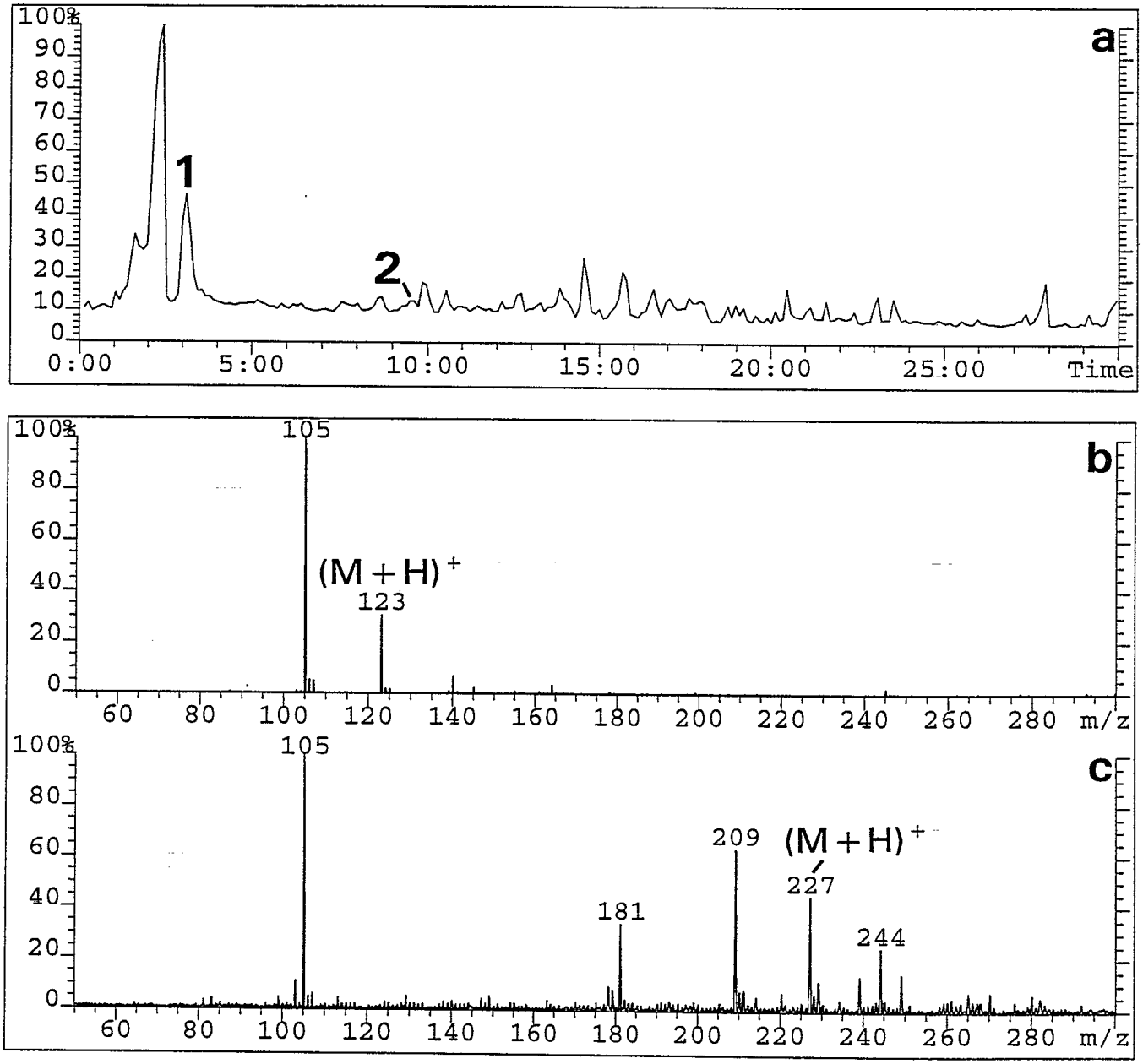

Figure 1: a) LC-ESI-MS total-ion-current (340 to 50 u) chromatogram obtained for aqueous sample #2 from a former mustard destruction site. b) ESI-MS data obtained for thiodiglycol (1) and c) 6-oxa-3,9-dithia-1,11-undecanediol (2).

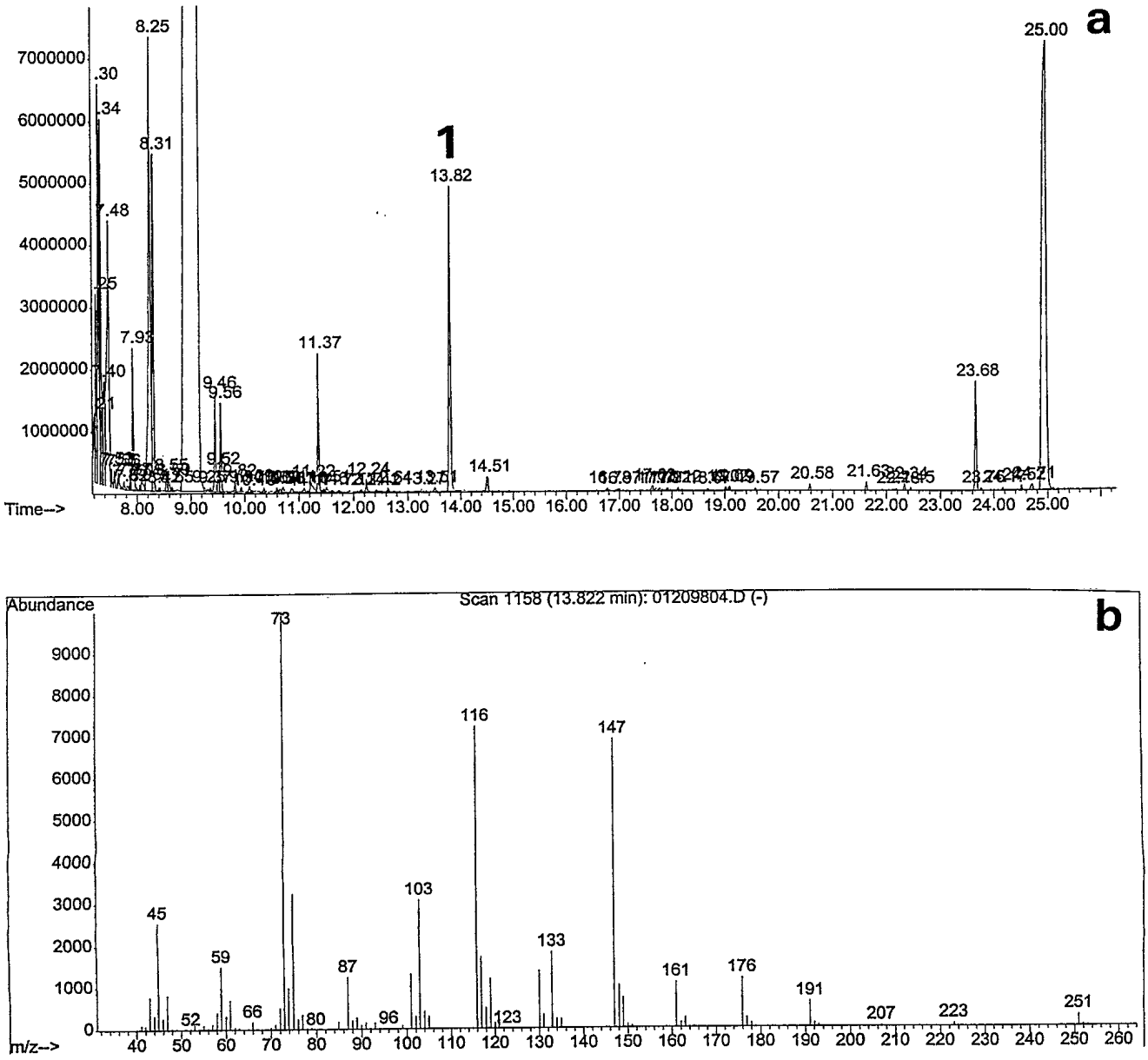

Figure 2: a) GC-MS total-ion-current (400 to 41 u) chromatogram obtained for aqueous sample #2 following silylanation. b) EI mass spectrum obtained for bis(TMS) derivative of thiodiglycol (1).

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CA and US participated in a joint TTCP E-AG-42 analytical exercise involving the analysis of aqueous field samples for the presence of mustand degradation products. The laboratories reported consistent results that clearly indicated the prior presence of mustard in a destruction site more than twenty years after the fact. Both laboratories used mass spectrometry for confirmation of the hydrolysis product of mustard, thiodiglycol. The US confirmed thiodiglycol as its bis(TMS) derivative during GC-MS and CA as the intact compound during GC-MS and LC-ESI-MS analysis. In addition both laboratories reported the presence of 1,4-thioxane and 1,4-dithiane, two compounds commonly associated with the degradation of mustard. CA also detected 6-oxa-3,9-dithia-1,11-undecanediol, the hydrolysis product of the longer chain sulfur vesicant, bis[(2-chloroethylthio)ethyl] ether (T) during LC-ESI-MS analysis.

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