industrially. The main markets appear to be in the production of low volume, high value elements: precious metals, rare earths with valuable optical properties and raw materials for electronic devices. Applications in lower value fields such as waste management or by-product recovery should benefit from novel resin formats designed for improved resin/liquid contacting.

See also: **II/Ion Exchange:** Historical Development; Novel Layered Materials: Non-Phosphates; Novel Layered Materials: Phosphates; Organic Ion Exchanger; Organic Membranes; Theory of Ion Exchange.

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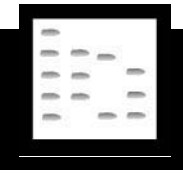
CHEMICAL WARFARE AGENTS: CHROMATOGRAPHY



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Introduction

The development and application of analytical methods for the analysis of chemical warfare agents have received increased attention, due in large part to the recently ratified Convention on the Prohibition of the Development, Production, Stockpiling and Use of Chemical Weapons and their Destruction (commonly referred to as the Chemical Weapons Convention or CWC). After considerable effort the CWC was opened to signature in 1993, with the treaty coming into force on 29 April 1997. The treaty has been ratified by 120 states, all of which agree not to develop, produce, stockpile, transfer or use chemical weapons and agree to destroy their own chemical weapons and production facilities. A strong compliance-monitoring regime involving site inspections was built into the CWC to ensure that the treaty remains verifiable. The Organisation for the Prohibition of Chemical Weapons, or OPCW, based in The Hague, has responsibility for implementation of the treaty. Routine OPCW inspections have or will take place at declared sites, including small scale production, storage and destruction sites, and challenge inspections will take place at sites suspected of noncompliance.

An analytical capability will be required to help verify compliance with the treaty, since inspectors will have the option to take and analyse suspect samples to help establish compliance or noncompliance. Gas chromatography is the current method of choice for the separation and analysis of chemical warfare agents, with this technique being employed in the field-portable gas chromatographic–mass spectrometric (GC-MS) instrumentation in use by the OPCW inspectorate.

Chemical warfare agents are a group of toxic chemicals that have been defined in the CWC as 'any chemical which through its chemical effect on life processes can cause death, temporary incapacitation or permanent harm to humans or animals …'. Poisonous or toxic compounds have been utilized in an effort to gain military superiority throughout history but it is only during the past century that chemical warfare agents have been produced and used on a large scale. Tear gas grenades were used in 1914 by the French at the outbreak of World War I, but it was not until the Germans first used chlorine near Ypres in 1915 that the world entered the modern era of chemical warfare. Other chemical warfare agents such as phosgene and mustard were used by both sides in World War I.

The use and development of chemical warfare agents continued after World War I despite the signing of the 1925 Geneva Protocol, which bans the first use of chemical weapons. Mustard was used by the Italians against the Abyssinians (Ethiopia) during 1936–1937, and just prior to World War II, the Germans discovered and produced the first nerve agent, tabun. Nerve agents were made into weapons by the Germans but neither side made use of their chemical weapons stock. More effective nerve agents, such as VX, were developed in the 1950s, mustard was used in the Yemen War (1963–1967), and allegations of chemical warfare agent use were reported in South-East Asian conflicts. Nerve and mustard agents were used by Iraq in the 1980s war between Iran and Iraq, and were considered a real threat to United Nations armed forces during their action against Iraq (1990–1991). More recently, sarin was used against the population of a Kurdish village in 1993 and again in 1995 by terrorists in the Tokyo underground transit system. Proliferation of chemical weapons and their use will hopefully decrease over the coming years as the CWC proceeds towards its goal of worldwide chemical weapons destruction.

Chemical Warfare Agent Categories

Chemical warfare agents have been classified into nerve, blister, choking, vomiting, blood, tear and incapacitating agent categories based on their effect on humans. The most significant chemical warfare agents in terms of military capacity and past use are the nerve and blister agents. For these reasons the analysis of these compounds will be emphasized over the other groups. The choking, blood and vomiting agents are for the most part obsolete chemical agents that were employed during World War I. The tear agents were used during the Vietnam War but their primary use, because of their inability to produce high casualties, remains in riot control and for the training of military personnel in chemical defence. Incapacitating agents have been included as the USA did develop an agent in this category.

The compounds listed in **Table 1** represent the most common chemical warfare agents, grouped by category with their Chemical Abstracts registry numbers, and is not intended to be exhaustive. It has been estimated that more than 7000 compounds are controlled under the CWC, although in practical terms the actual number of chemical warfare agents, precursors, degradation products and related compounds that are contained in the OPCW GC-MS database is in the hundreds. The structures of some common nerve and blister chemical warfare agents are illustrated in **Figure 1**.

Gas Chromatographic Methods

Chemical warfare agents have often been referred to as warfare gases and, in the military, the phrase 'gas, gas, gas' has become synonymous with attack by chemical warfare agents. In fact, many chemical warfare agents exist as liquids at ambient temperatures but have varying degrees of volatility and pose a significant vapour hazard as well as a liquid contact hazard. This physical characteristic has made the analysis of chemical warfare agents amenable to gas chromatography (GC) with a variety of detectors including mass spectrometry (MS).

The OPCW, an important end-user of analytical techniques for chemical warfare agents, requires the use of two or more spectrometric techniques and the availability of authentic reference standards for the unambiguous identification of these controlled compounds. For this reason, the combined use of GC–Fourier transform infrared spectrometry (FTIR) has received increased attention as newer technologies have led to detection limits approaching those routinely reported during GC-MS analysis. For analyses involving low levels of chemical warfare agents in the presence of high levels of interfering chemical background, tandem mass spectrometry (MS-MS) is receiving increased attention.

GC Retention Behaviour

Packed column GC was routinely employed in the past for the analysis of chemical warfare agents, but with the advent of fused silica capillaries this technology has been used less frequently. Capillary column GC has become the most frequently employed analytical separation method for the screening of samples contaminated with chemical warfare agents. Separation of chemical warfare agents may be achieved with fused silica columns coated with poly-

Table 1 Common chemical warfare agents

Full name (trival name(s))	*Chemical Abstracts no.*
(a) *Nerve* (reacts irreversibly with cholinesterase; this results in acetylcholine accumulation, continual stimulation of the body's nervous system and eventual death)	
O-Isopropyl methylphosphonofluoridate (sarin, GB)	107-44-8
O-Pinacolyl methylphosphonofluoridate (soman, GD)	96-64-0
O-Cyclohexyl methylphosphonofluoridate (GF)	329-99-7
O-Ethyl *N,N*-dimethylphosphoramidocyanidate (tabun, GA)	77-81-6
O-Ethyl *S*-2-diisopropylaminoethyl methylphosphonothiolate (VX)	50782-69-9
(b) *Blister* (affects the lungs, eyes and produces skin blistering)	
Bis(2-chloroethyl)sulfide (mustard, H)	505-60-2
Bis(2-chloroethylthio)ethane (sesquimustard, Q)	3563-36-8
Bis(2-chloroethylthioethyl)ether (T)	63918-89-8
Tris(2-chloroethyl)amine (HN-3)	555-77-1
2-Chlorovinyldichloroarsine (lewisite, L)	541-25-3
(c) *Choking* (affects respiratory tract and lungs)	
Chlorine	7782-50-5
Carbonyl dichloride (phosgene, CG)	75-44-5
(d) *Vomiting* (causes acute pain, nausea and vomiting in victims)	
Diphenylarsinous chloride (DA)	712-48-1
10-Chloro-5,10-dihydrophenarsazine (adamsite, DM)	578-94-9
Diphenylarsinous cyanide (DC)	23525-22-6
(e) *Blood* (prevents transfer of oxygen to the body's tissues)	
Hydrogen cyanide (HCN, AC)	74-90-8
(F) *Tear* (Causes tearing and irritation of the skin)	
[(2-chlorophenyl)methylene]propanedinitrile (CS)	2698-41-1
2-Chloro-1-phenylethanone (CN)	532-27-4
Dibenz[b,f][1,4]oxazepin (CR)	257-07-8
(g) *Incapacitating* (prevents normal activity by producing mental or physiological effects)	
3-Quinuclidinyl benzilate (BZ)	6581-06-2

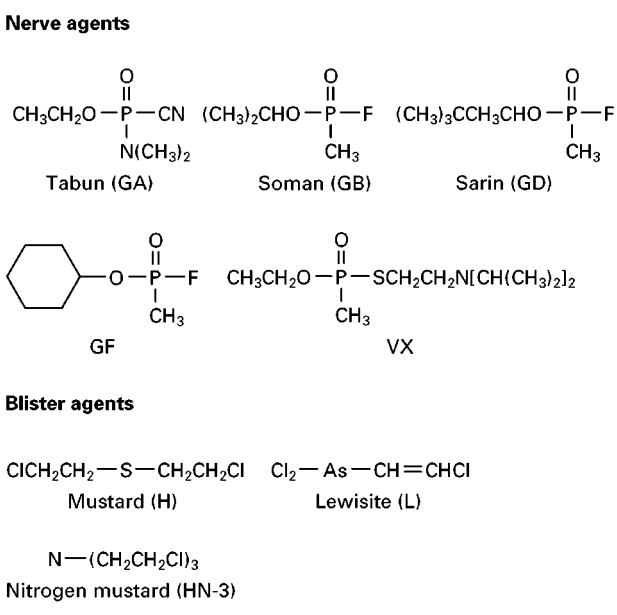

Figure 1 Structures of common chemical warfare agents.

siloxane or other films and retention index data relative to *n*-alkanes and *n*-alkylbis(trifluoromethyl)phosphine sulfides (M-series) have been reported for many chemical warfare agents and related compounds under temperature programming conditions. Retention indices relative to *n*-alkanes have been reported for 100% dimethyl-polysiloxane (e.g. J&W DB-1), (95%)-methyl-(5%)-diphenyl-polysiloxane (e.g. J&W DB-5), (86%)-dimethyl-(14%)-cyanopropylphenyl-polysiloxane (e.g. J&W DB-1701) and other films. In general, the best separations have been achieved with a moderately polar film such as (86%)-dimethyl-(14%)-cyanopropylphenyl-polysiloxane. **Table 2** lists typical retention index data for several common chemical warfare agents on three different liquid phases under temperature programming conditions.

The use of retention indices relative to *n*-alkanes by the OPCW during on-site inspections is anticipated in the near future to differentiate between controlled compounds that exhibit similar electron impact mass spectrometric (EI-MS) data during GC-MS analysis,

Table 2 GC retention index data for common chemical warfare agents (relative to *n*-alkanes)

Compound name	*GC retention index*[a]		
	DB-1	*DB-5*	*DB-1701*
O-Isopropyl methylphosphonofluoridate (sarin, GB)	792	824	966
O-Pinacolyl methylphosphonofluoridate (soman, GD)[b]	1008 1013	1045 1049	1188 1193
O-Ethyl *N*,*N*-dimethylphosphoramidocyanidate (tabun, GA)	1078	1132	1340
O-Ethyl *S*-2-diisopropylaminoethyl methylphosphonothiolate (VX)	1664	1710	1881
Bis(2-chloroethyl)sulfide (mustard, H)	1124	1173	1326
Bis(2-chloroethylthio)ethane (sesquimustard, Q)	1623	1689	1923
Bis(2-chloroethylthioethyl)-ether (T)	1910	1983	2241

[a]GC retention indices determined with three J&W 0.25 μm films with the following temperature program: 50°C (2 min), then 10°C min^{-1} to 300°C (5 min).
[b]Retention data for both enantiomer pairs.

but different GC retention behaviour. Application of GC retention indices during the analysis of VX-contaminated samples is likely since the EI-MS data for VX and a number of VX-related compounds are remarkably similar. The EI data for these compounds lack a molecular ion and contain a base ion at *m/z* 114 due to $(CH_2N(iPr)_2)^+$ and additional ions related to the $-SC_2H_4N(iPr)_2$ substituent.

Chiral stationary phases have been developed for the resolution of stereoisomers of several chiral nerve agents, most notably soman. The use of multiple columns of differing polarity during one analysis has also been successfully employed during chemical warfare agent analysis and the term 'retention spectrometry' was coined to describe this technique.

Conventional GC Detectors

Most of the GC detectors commonly applied to pesticide residue analysis have also been applied to the screening of samples for chemical warfare agents. Detection limits are typically in the nanogram to picogram range. Flame ionization detection (FID) is routinely used for preliminary analyses, as this technique provides a good indication of the complexity of a sample extract. **Figure 2** illustrates GC-FID chromatograms obtained using a J&W DB-1 capillary column for three different munitions grade mustard formulations, HT, HS and HQ. HT is typically 60% distilled mustard (H) and 40% bis(2-chloroethylthioethyl)ether (T) by weight, while HQ is usually 75% distilled mustard and 25% sesquimustard (Q) by weight. HS is crude mustard-containing 15% carbon tetrachloride. Munitions grade samples typically contain additional sample components that may provide synthetic procedure or source information. These samples contained several other sulfur-containing impurities, including 1,4-thioxane, 1,4-dithiane (two common mustard degradation products) and several longer chain blister agents (chromatographic peaks 8, 9 and 10 in **Figure 2**).

Figure 3 illustrates the application of capillary GC-FID for the characterization of tabun and related impurities in a munitions grade sample used for military chemical agent detector evaluation. The volatile organic content of this munitions grade tabun was estimated on the basis of the FID response of the individual components. Tabun accounted for 81% of the sample. The impurities, isopropyl *N*,*N*-dimethylphosphoramidocyanidate (isopropyl analogue of tabun), ethyl *N*,*N*,*N*′,*N*′-tetramethylphosphorodiamidate, *N*,*N*,*N*′,*N*′-tetramethylphosphorodiamidic cyanide and the cluster of pyrophosphates, represented approximately 5%, 4%, 2% and 5%.

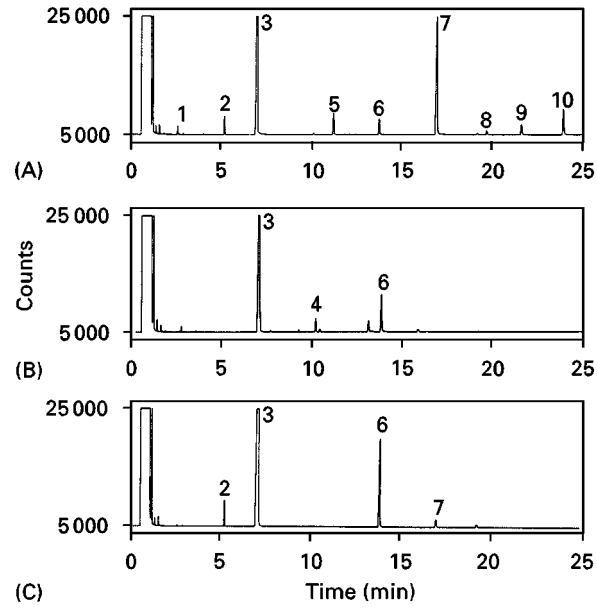

Figure 2 Capillary column GC-FID chromatograms of three munitions grade mustard samples: (A) HT, (B) HS and (C) HQ. Identified compounds include: 1, 1,4-thioxane; 2, 1,4-dithiane; 3, mustard (H); 4, bis(2-chloroethyl)disulfide; 5, 2-chloroethyl (2-chloroethoxy)ethyl sulfide; 6, sesquimustard (Q); 7, bis (2-chloroethylthioethyl)ether (T); 8, 1,14-dichloro-3,9-dithia-6,12-dioxatetradecane; 9, 1,14-dichloro-3,6,12-trithia-9-oxatetradecane; and 10, 1,16-dichloro-3,9,15-trithia-6,12-dioxaheptadecane. (GC column: 15 m × 0.32 mm ID J&W DB-1, temperature programme: 50°C (2 min), then 10°C min^{-1} to 280°C (5 min), 2×10^{-10} A full scale.)

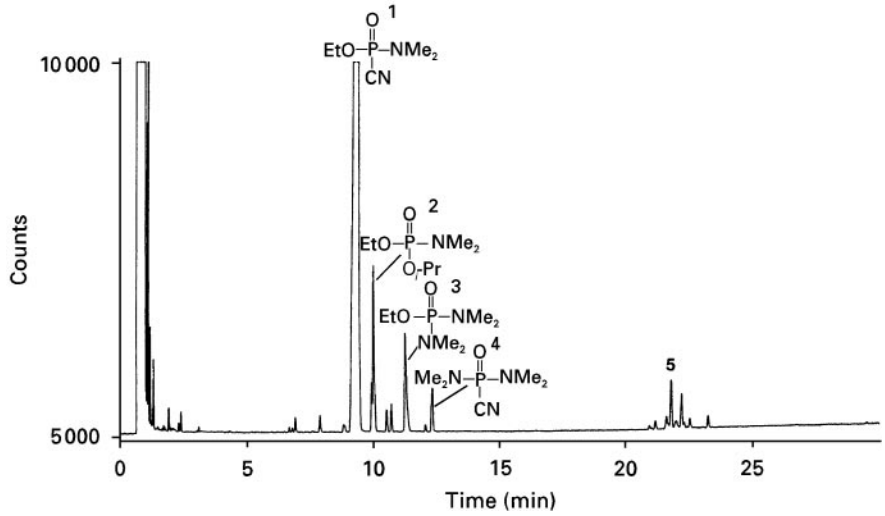

Figure 3 Capillary column GC-FID chromatogram of munitions grade tabun (GA) sample. Compounds identified include: 1, tabun (GA); 2, ethyl isopropyl *N,N*-dimethylphosphoramidocyanidate; 3, ethyl *N,N,N′,N′*-tetramethylphosphorodiamidate; 4, *N,N,N′,N′*-tetramethylphosphorodiamidic cyanide; and 5, pyrophosphates. (GC-column: 15 m × 0.32 mm ID J&W DB-5, temperature programme: 50°C (2 min), then 10°C min^{-1} to 280°C (5 min), 5×10^{-11} A full scale.)

The need for higher specificity and sensitivity has led to the application of element-specific detectors such as flame photometric detection (FPD), thermionic detection (TID), atomic emission detection (AED) and electron capture detection (ECD). The simultaneous use of FID with one or more element-specific detectors has also been demonstrated during dual or tri-channel GC analysis using conventional and thermal desorption sample introduction. While these detectors may provide strong collaborative evidence for the presence of chemical warfare agents, they cannot be used for full confirmation. Use of GC with one or more spectrometric technique such as MS is required to confirm the presence of chemical warfare agents.

Mass Spectrometry

Mass spectrometry is the method of choice for the detection and characterization of chemical warfare agents, their precursors, degradation products and related compounds. Extensive use has been made of GC-MS and the mass spectra of numerous chemical warfare agents and related compounds have been published, with the most common chemical warfare agent mass spectra being available in the OPCW, commercial or defence community databases.

Samples collected for chemical warfare agent analysis typically fall into one of the following general categories: (a) munitions or munition fragments (e.g. neat liquid or artillery shell casing); (b) environmental (e.g. soil, water, vegetation or air samples); (c) artificial materials (e.g. paint or rubber); and (d) biological media (e.g. blood or urine). The ease of analysis depends on the amount of preparation required to obtain a suitable sample or extract for GC analysis. In the simplest case where neat liquid can be obtained the sample requires dilution with a suitable solvent prior to GC-MS analysis. Environmental and other samples generally require (at a minimum) solvent extraction and concentration prior to analysis, with an exception being direct thermal desorption analysis of samples using a GC equipped with a thermal desorption device that may be loaded with the actual sample. Recommended methods for sample preparation have been published by The Ministry of Foreign Affairs of Finland as part of their contribution to Chemical Disarmament.

Figure 4 illustrates the capillary column GC-MS chromatogram obtained for the extract of a soil sample containing VX and several related compounds. Seven components were identified in the sample following ultrasonic extraction of 1 g of soil with dichloromethane. Extraction of chemical warfare agents from biological samples is generally more difficult. Bonierbale, Debordes and Coppet recently demonstrated a method for the extraction of VX from rat blood as part of a study designed to follow the hydrolysis of VX. **Figure 5** illustrates a typical mass chromatogram for the blood extract containing VX, malathion and diisopropylaminoethanethiol.

The data acquired in Figures 4 and 5 and most MS data obtained by OPCW inspectors during inspections have been obtained under EI-MS conditions. However, many of the chemical warfare agents in particular the organophosphorus nerve agents such as VX, and the longer chain blister agents related to

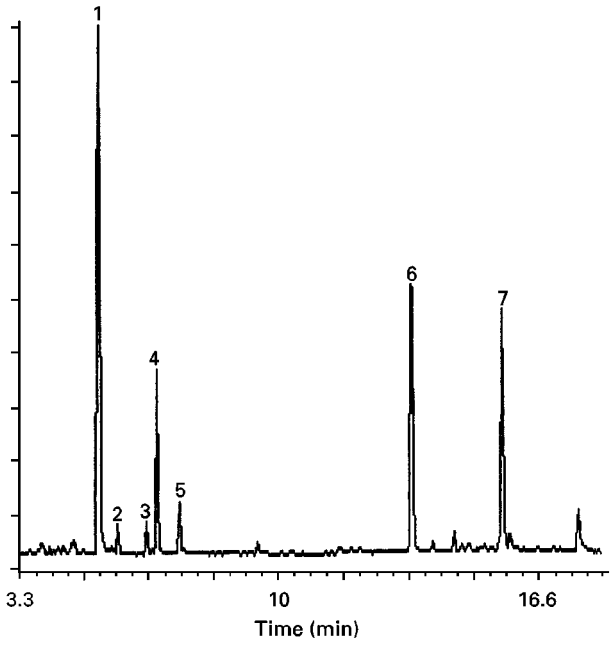

Figure 4 Capillary column GC-MS (EI) chromatogram of dichloromethane extract of soil sample contaminated with VX. VX and six other compounds were identified: 1, diethyl methylphosphonate; 2, *N,N*-diisopropylethylamine; 3, 2-(diisopropylamino)ethyl methyl sulfide; 4, *O,S*-ethyl methyl methylphosphonothiolate; 5, *O,S*-diethyl methylphosphonothiolate; 6, VX; and 7, bis[2-(diisopropylamino)ethyl] disulfide. (GC column: 15 m × 0.32 mm ID J&W DB-1701, temperature programme: 50°C (2 min) then 10°C min^{-1} to 280°C (5 min).)

mustard, such as Q and T, do not provide molecular ion information during EI-MS. This hinders confirmation of these compounds and makes identification of novel chemical warfare agents or related impurities difficult.

Considerable effort has been devoted to the use of chemical ionization (CI) as a complementary ionization technique. This milder form of ionization generally affords molecular ion information for the chemical warfare agents and has been used extensively for the identification of related compounds or impurities in chemical warfare agent munitions samples and environmental sample extracts. The identity of these related compounds is important because the origin of samples, synthetic process information or degree of degradation (weathering) may be deduced based on this 'fingerprint' data.

Isobutane, ethylene and methane gases were initially demonstrated as suitable CI gases for the acquisition of organophosphorus nerve agent CI-MS data. More recently, the efficacy of ammonia CI-MS for organophosphorus nerve agents and related compounds has been demonstrated and many laboratories now employ this complementary confirmation technique. Ammonia CI not only offers abundant molecular ion data, but also affords a high degree of specificity as less basic sample components are not ionized by the ammonium ion.

Capillary column GC-MS-MS offers the analyst the potential for highly specific, sensitive detection of chemical warfare agents as this technique significantly reduces the chemical noise associated with complex sample extracts. The specificity of product scanning with moderate sector resolution, as well as the specificity of ammonia CI, were demonstrated with a hybrid tandem mass spectrometer during analysis of painted panel samples circulated during an international round robin verification exercise.

The painted panel extract was contaminated with numerous hydrocarbons. Only two of the three longer chain blister agents, sesquimustard (Q) and bis(2-chloroethylthioethyl)ether (T), could be identified during capillary column GC-MS (EI) analysis (**Figure 6**A). The arrow indicates the chromatographic retention time of the third blister agent, 2-chloroethyl (2-chloroethoxy)ethyl sulfide (O). The specificity of ammonia CI (Figure 6B) was clearly demonstrated during this analysis. All three longer chain blister agents were determined in the presence of high levels

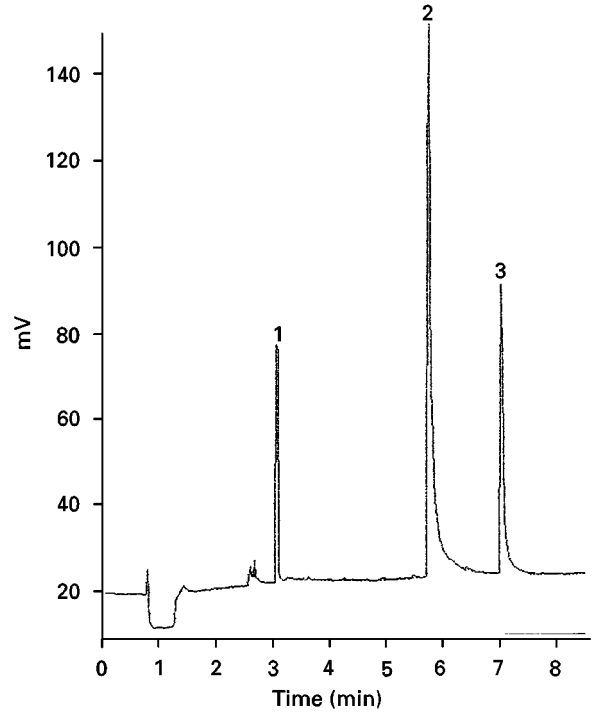

Figure 5 Capillary column GC-MS (EI) chromatogram of 374 μmol L^{-1} VX incubated for 2 h in the presence of rat plasma. Extracted sample components: 1, 3 μmol L^{-1} malathion; 2, VX; and 3, diisopropylaminoethanethiol. (GC column: 25 m × 0.32 mm ID CP 8CB; temperature programme: 100°C (0.5 min), then 30°C min^{-1} to 210°C (1 min) followed by 30°C min^{-1} to 300°C (3 min).)

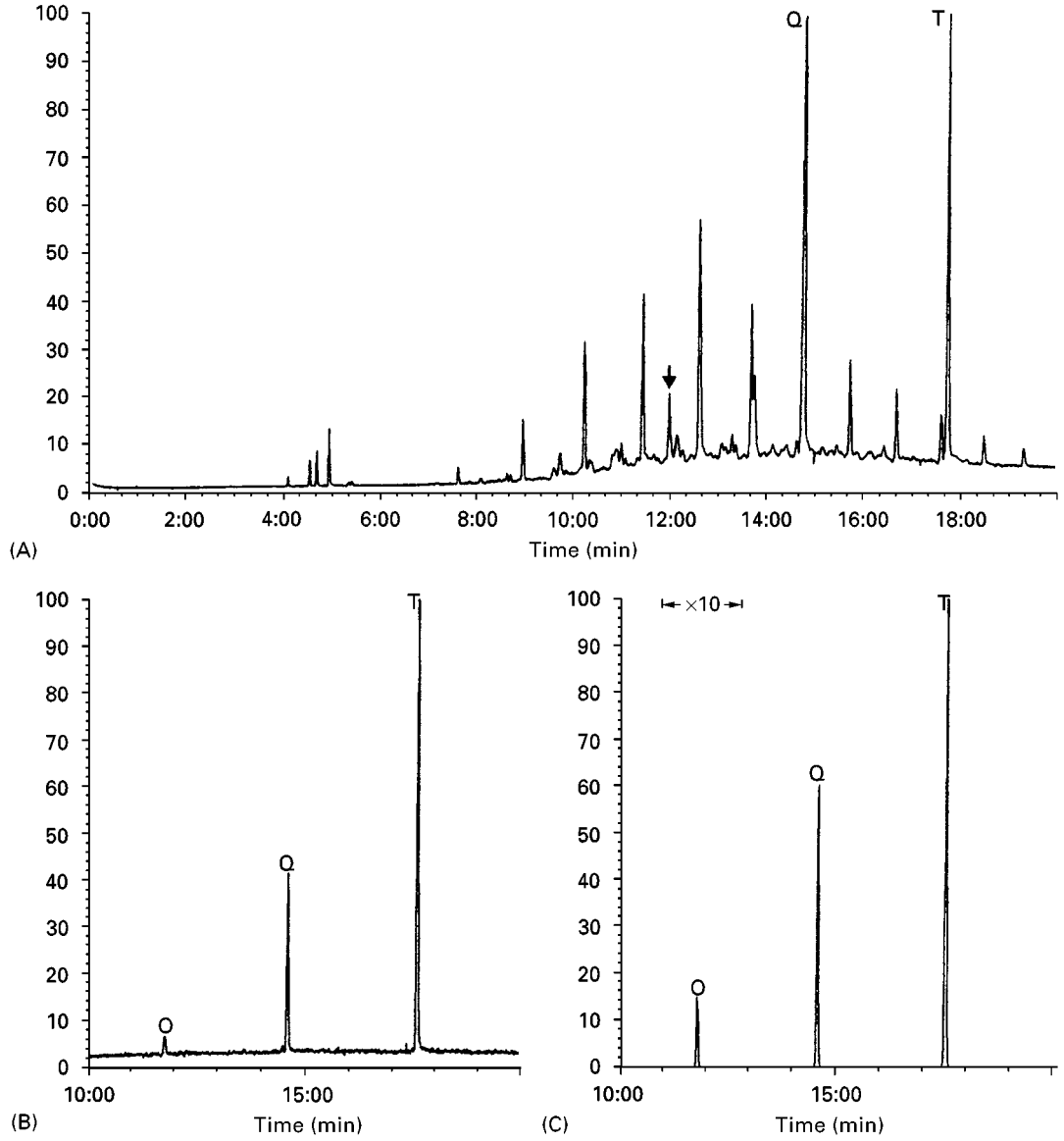

Figure 6 Capillary column (A) GC-MS (EI), (B) GC-MS (ammonia CI) and (C) GC-MS/MS (EI) chromatograms obtained during analysis of international round robin painted panel extracts. Sequimustard (Q) and bis(2-chloroethylthioethyl)ether (T) were detected during EI analysis. The downward arrow in (A) indicates the retention time of 2-chloroethyl (2-chloroethoxy)ethyl sulfide (O). This compound (O) was masked by the organic content of the sample during EI analysis and only detected following (B) ammonia CI and (C) MS-MS analysis. (GC column: 15 m × 0.32 mm ID J&W DB-1701, temperature programme: 40°C (2 min), then 10°C min^{-1} to 280°C (5 min).)

of interfering hydrocarbons, as the hydrocarbons were not sufficiently basic to ionize. Similarly, it was possible to use the resolution of hybrid tandem mass spectrometry to discriminate between ions at m/z 123 arising from the longer chain blister agents from those ions at m/z 123 arising from the hydrocarbon background. The resultant GC-MS-MS chromatogram (Figure 6C), where only m/z 123 ions due to the blister agents were transmitted into the collisional activated dissociation cell, was virtually free of chemical noise and all three components were detected. The three longer chain blister agents were well resolved with the J&W DB-1701 capillary column, with all three components exhibiting similar product spectra during GC-MS-MS analysis.

Hydrolysis Products of Chemical Warfare Agents

Both the nerve and blister agents undergo hydrolysis in the environment and methods are required for retrospective detection and confirmation of these hydrolysis products. Hydrolysis products are significant as they are generally compounds that would not be

routinely detected in environmental samples and their presence strongly suggests the prior presence of chemical warfare agents. The degradation products of the chemical warfare agents, in particular the nerve agents, are nonvolatile hydrolysis products that must be derivatized prior to GC analysis. A variety of derivatization techniques including methylation, *t*-butyldimethylsilylation and trimethylsilylation have been investigated to allow GC analysis of, in particular, the organophosphorus acids related to the nerve agents (e.g. alkyl methylphosphonic acids and methylphosphonic acid).

Mustard and longer chain blister agents hydrolyse to their corresponding diols, with thiodiglycol being the product formed following hydrolysis of mustard. These compounds may be analysed by GC-MS directly provided the sample is loaded onto the column using 'cool' on-column injection. **Figure 7** illustrates a typical GC-MS(EI) chromatogram obtained for products formed following hydrolysis of a sample containing both HT and HQ. The principal hydrolysis products of H, Q and T – thiodiglycol, bis(2-hydroxyethylthio)ethane, and bis[(2-hydroxyethylthio)ethyl] ether, respectively – are well resolved with the J&W DB-1701 capillary column. Molecular ion information for the longer chain hydrolysis products, were obtained under ammonia CI conditions.

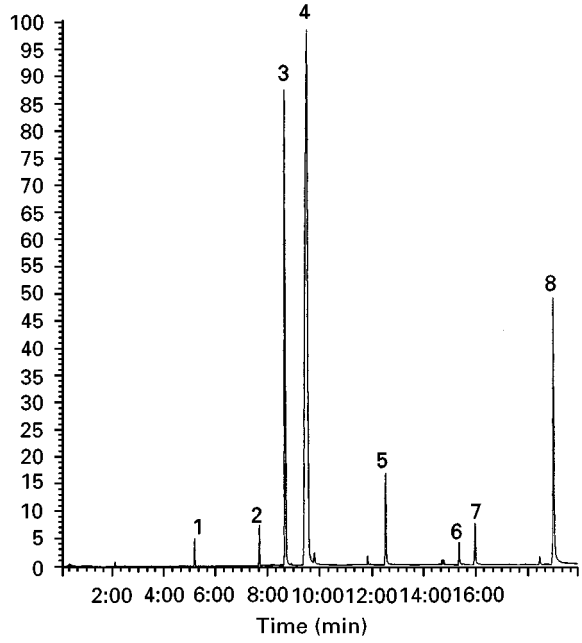

Figure 7 Capillary column GC-MS(EI) chromatogram of the hydrolysis products of a munitions grade mustard sample containing HT and HQ. Eight compounds were identified: 1, 1,4-dithiane; 2, mustard (H); 3, hemisulfur mustard; 4, thiodiglycol (hydrolysis product of H); 5, 2-chloroethyl (2-hydroxyethylthio)ethyl ether; 6, 2-chloroethyl (2-hydroxyethylthio)ethyl sulfide; 7, bis(2-hydroxyethylthio)ethane (hydrolysis product of Q); and 8, bis[(2-hydroxyethylthio)ethyl] ether (hydrolysis product of T). (GC column: 15 m × 0.32 mm ID J&W DB-1701, temperature programme: 40°C (2 min), then 10°C min^{-1} to 280°C (5 min).)

Use of thermospray mass spectrometry and, more recently, the use of atmospheric pressure ionization techniques (e.g. electrospray (ESI), ionspray and atmospheric pressure CI), has enabled the direct mass spectrometric analysis of the hydrolysis products of chemical warfare agents. Both techniques may be interfaced to liquid chromatography for component separation, with thermospray having been largely superceded by atmospheric pressure ionization (API) for most LC-MS applications. Examples of LC-ESI-MS methods for the direct analysis of chemical warfare agent hydrolysis products are relatively few, and only recently has this technique been demonstrated for the analysis of the nerve agents as well. These new LC-ESI-MS methods complement existing GC-MS methods for the analysis of chemical warfare agents and their hydrolysis products and will likely replace some GC-MS methods currently in use for the analysis of contaminated aqueous samples.

Safety and Disposal

Chemical warfare agents are extremely hazardous and lethal compounds. They can only be used in designated laboratories by personnel trained in safe-handling and decontamination procedures and with immediate access to medical support. Safety and standard operating procedures must be developed and approved before any chemical warfare agents are handled. Chemical warfare agents can only be used in laboratory chemical hoods with a minimum face velocity of 100 linear feet per minute equipped with emission control devices that limit exhaust concentration to below 0.0001 mg m^{-3}. Personnel handling chemical warfare agents should wear rubber gloves, lab coats and full faceshields, and a respirator (gas mask) must be kept within easy reach. Sufficient decontaminants to destroy all chemical warfare agent being handled must be on hand before commencing operations.

Blister agents such as mustard can be destroyed with 10% hypochlorite or strong bleach solutions. The nerve agents can be destroyed using saturated methanolic solutions of sodium or potassium hydroxide. Decontaminated chemical warfare agents must be disposed of in an environmentally approved method according to local legislation.

Conclusion

Capillary column GC is the chromatographic method of choice for the separation and analysis of chemical warfare agents. This technology is employed in the

field-portable GC-MS instrumentation currently in use by the OPCW inspectorate. Use of GC with one or more spectrometric technique such as mass spectrometry is required to confirm the presence of chemical warfare agents. For this reason many analyses are carried out by GC-MS under electron impact or chemical ionization conditions. For analyses involving low levels of chemical warfare agents in the presence of high levels of interfering chemical background, GC-MS-MS is receiving increased attention.

Atmospheric pressure ionization (e.g. electrospray (ESI), ionspray and atmospheric pressure CI) techniques may be used for the direct mass spectrometric analysis of the hydrolysis products of chemical warfare agents in aqueous samples. Examples of LC-ESI-MS methods for these analyses are relatively few, and only recently has this technique been demonstrated for the analysis of the nerve agents as well. These new LC-ESI-MS methods complement existing GC-MS methods for the analysis of chemical warfare agents and their hydrolysis products and will likely replace some GC-MS methods currently in use for the analysis of contaminated aqueous samples.

See also: **II/Chromatography: Gas:** Column Technology; Detectors: Mass Spectrometry; Detectors: Selective. **Chromatography: Liquid:** Detectors: Mass Spectrometry.

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CHIRAL SEPARATIONS

Amino Acids and Derivatives

See **III / AMINO ACIDS AND DERIVATIVES: CHIRAL SEPARATIONS**

Capillary Electrophoresis

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Background

Since its commercial inception in 1988 capillary electrophoresis (CE) has increasingly offered the separation scientist a very wide range of application (biopolymers to cations), with ultra-high efficiency. It is generally cost-effective, easy in use, with low sample and buffer requirements and has the capability for automation. For these reasons CE has expanded considerably and is now used as an orthogonal and complementary technique alongside well-established analytical procedures. Of all its