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Characterization of equimolar VX–water reaction product by gas chromatography–mass spectrometry

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Abstract

One method proposed for the destruction of the chemical warfare nerve agent VX, *O*-ethyl *S*-2-(diisopropylamino)ethyl methylphosphonothiolate, is hydrolysis by the addition of an equimolar amount of water. To better understand this reaction, a method was developed for characterization of the resulting product and for monitoring product stability. Conditions for derivatization of the acidic products to their trimethylsilyl esters, with subsequent analysis by gas chromatography–mass spectrometry using electron (EI) and methane chemical ionization (CI) were optimized to allow nonchromatographable acids and organic products to be detected in a single analysis, without loss of volatile products, and with minimal side reaction. EI and methane CI mass spectra of 23 identified degradation products are provided. Results indicate that the product is relatively stable at ambient temperatures in a closed container with only a minimal amount of thiol secondary reaction being observed. Published by Elsevier Science B.V.

Keywords: Warfare agents; VX

1. Introduction

Detection of chemical agents and their degradation products is important for monitoring the destruction of chemical agents because of safety and environmental concerns, particularly in light of impending plans for agent destruction [1]. One proposed method for the destruction of the nerve agent VX, *O*-ethyl *S*-2-(diisopropylamino)ethyl methylphosphonothiolate (CAS No. 50782-69-0), is hydrolysis by the addition of an equimolar amount of water. A proposed mechanism for this reaction has recently been reported [2]. Several products of this reaction, including the primary product, ethyl methylphosphonic acid (EMPA), are acidic and polar and are not amenable to analysis by gas chromatography–mass spectrometry (GC–MS). A common technique used to analyze these compounds is derivatization to the trimethylsilyl (TMS) ester [3]. One drawback of this technique is that volatile reaction products can be either lost through evaporation or not detected because they coelute with early-eluting derivatizing agent peaks. In this study, conditions are optimized to allow the detection of volatile diisopropylamine during analysis and are also optimized to prevent thiol oxidation, which can lead to erroneous results. A VX–water product was monitored for a period of six months using GC–MS in the chemical ionization (CI) mode with methane as the CI reagent gas to assess product stability.

The identification and detection of VX hydrolysis products are also important for the analysis of

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environmental samples, to provide evidence of non-compliance with the United Nations Chemical Weapons Convention (CWC) [4] treaty banning the development, production, stockpiling and use of scheduled chemical agents, which entered into force in April 1997. The alleged use of nerve agents in the Iran/Iraq conflict [5,6] and the Tokyo subway incident [7] has been well documented. Most studies reported in the recent literature have focused on the detection of VX only or selected signature breakdown products, such as ethyl methylphosphonic acid or pyrophosphonates [8–11], with little information provided on other compounds that may be present. D'Agostino et al. [12] have provided an excellent report on the application of ammonia CI-MS to identify over twenty impurities present in a sample of stored VX.

2. Experimental

2.1. Preparation of the VX–water product

The VX–water product was prepared in two batches by the addition of distilled water to 88% VX (GC–MS purity) to a final concentration of 10% (v/v). This mixture was pumped into a 90°C reactor at a rate of 1 ml/min until the entire mixture had been added. The reaction was continued at 90°C for 3 h after completion of the addition. The VX was obtained from a stored ton container and contained the following impurities, by direct GC–MS analysis: 0.7% diisopropylamine, 0.6% *O*,*O*-diethyl methylphosphonate, 0.4% *O*,*O*-diethyl methylphosphonothioate, 1.4% 2-(diisopropylamino)ethanethiol, 0.6% *O*,*S*-diethyl methylphosphonothioate, 0.1% 2-(diisopropylamino)ethyl vinyl sulfide, 0.2% 2-(diisopropylamino)ethyl ethyl sulfide, 1.6% *O*,*O*-diethyl dimethylpyrophosphonate, 0.3% *O*,*O*-diethyl dimethylmonothionopyrophosphonate, 3.4% dicyclohexylcarbodiimide, 0.04% *O*-ethyl *S*-2-(diisopropylamino)ethyl methylphosphonodithioate, 2.0% bis[2-(diisopropylamino)ethyl] sulfide and 0.5% bis[2-(diisopropylamino)ethyl] disulfide. In addition, 0.8% *S*,*S*-bis[2-(diisopropylamino)ethyl] methylphosphonodithioate and 0.2% *O*-ethyl methylphosphonothioic acid were detected in the sample by ^{31}P NMR.

2.2. Trimethylsilyl derivatization

Derivatization of the VX–water product was accomplished by the addition of 40 μl of bis-(trimethylsilyl)trifluoroacetamide (BSTFA) containing 1% trimethylchlorosilane (TMCS), obtained from Supelco (Bellefonte, PA, USA), to 30 μl of the VX–water product and heating at 60°C for 20 min.

2.3. Instrumentation

Derivatized samples were analyzed on a Finnigan TSQ-7000 GC–MS–MS system (a subsidiary of ThermoQuest, San Jose, CA, USA), equipped with a 30 m×0.25 mm MSSEL (DB-5) capillary column, with a film thickness of 0.25 μm (J&W Scientific, Folsom, CA, USA). The carrier gas was helium with a flow-rate of 1 ml/min. The oven temperature was ramped from 60–270°C at 15°C/min, with a 5 min hold at 270°C. The injection port temperature was 220°C, the GC–MS interface temperature was 250°C and the source temperature was 150°C. The CI reagent gas was methane, at a source pressure of 4 Torr (=500 Pa). Using a 1-μl syringe, approximately 0.01 μl of sample was injected in the split mode with a split ratio of 50:1. The mass range was scanned from 60–450 μ at 0.7 s/scan. The electron energy was 200 eV and the emission current was 300 μA.

Electron ionization (EI) spectra were also obtained for each of the degradation products, to provide reference spectra. The same conditions as above were used except that the electron energy was 70 eV, the emission current was 400 μA, and the mass range was scanned from 45–450 μ at 0.7 s/scan.

2.4. Product stability study

To determine the thermal stability of the product, the sample was analyzed by GC–CI-MS at intervals over a period of six months. The sample was stored in a capped 1 ml vial in the laboratory at ambient temperature under an air atmosphere.

3. Results and discussion

Twenty-three reaction products were detected and identified by GC–CI-MS in the equimolar VX–water

reaction product. All products are VX degradation products, except for the two ureas and the isothiocyanate, which result from reactions of diisopropylcarbodiimide and dicyclohexylcarbodiimide, which are commonly used as VX stabilizers. No VX was detected (detection limit, approximately 0.05%). When possible, confirmation of compound identification was obtained by comparison to spectra in the literature [12]. Comparison of the spectrum of peak 8, which has a molecular mass of 159, with the spectrum of 2-(diisopropylamino)ethanethiol suggests that the structure may be $HSCH_2CH_2N(i\text{Pr})(C[Me]=CH_2)$. GC retention times ($t_R$), relative molecular masses (M_r) and the structures of the reaction products are listed in Table 1. Results obtained after 10, 42 and 175 days of storage are also provided in Table 1. A typical chromatogram of the mixture is shown in Fig. 1.

The objective of BSTFA derivatization is to convert the acids to TMS esters to allow detection of EMPA and other acids. Two concerns, the detection of diisopropylamine and oxidation of thiols to disulfides, surfaced with this technique. If excess BSTFA is used, diisopropylamine coelutes with the derivatizing agent byproducts. Conditions were optimized so that only a slight excess of the derivatizing agent was added, enough to obtain complete derivatization without masking the diisopropylamine. As shown in Fig. 2, optimum conditions were observed with the addition of 40 μl of BSTFA to 30 μl of the VX–water product. Excess BSTFA was observed using this ratio, while none was observed at lower ratios. Normally, water, which interferes with BSTFA derivatization, is removed by evaporation prior to derivatization. This evaporation step is a potential source of diisopropylamine loss. In this study, because only enough water is added to react on a molar-equivalent basis with VX, the level (if any) of water present is low enough that derivatization of the reaction product can be successfully achieved without prior evaporation.

The second concern surfaced during early analyses of the VX–water product when it was noted that comparison of GC–MS data to NMR data [13]

Table 1
Products identified by GC–CI–MS in the VX–water reaction product [R=-$CH_2CH_2N(i\text{Pr})_2$, see Fig. 1 for peak numbers]

Peak No.	t_R (min:s)	M_r	Compound	GC–MS area %		
				10 Days	42 Days	175 Days
1	1:41	101	$i\text{Pr}_2\text{NH}$ [108-18-9]	6.0	6.3	6.5
2	3:59	152	MeP(O)(OEt)_2 [683-08-9]	0.2	0.3	0.3
3	4:31	168	MeP(S)(OEt)_2 [6996-81-2]	0.2	0.2	0.2
4	4:41	196	MeP(O)(OEt)OH [1832-53-7], TMS derivative	57.7	59.8	58.3
5	5:13	161	RSH [5842-07-9]	22.4	18.4	15.2
6	5:15	212	MeP(S)(OEt)OH, TMS derivative	0.3	0.4	0.3
7	5:16	240	MeP(O)(OH)_2 [993-13-5], di-TMS derivative	0.8	0.7	0.8
8	5:23	159	$C_8H_{17}NS$	<0.1	<0.1	<0.1
9	5:37	217	ROH [96-80-0], TMS derivative	–	<0.1	<0.1
10	5:42	168	MeP(O)(SEt)OEt [2511-10-6]	0.3	0.3	0.2
11	6:30	141	Cyclohexyl isothiocyanate	0.2	0.1	0.2
12	6:48	187	$RSCH=CH_2$	<0.1	<0.1	<0.1
13	6:50	189	RSEt	0.2	0.2	0.2
14	8:53	160	*N,N′*-Diisopropylthiourea [2986-17-6]	<0.1	<0.1	<0.1
15	9:02	277	$RSCH_2CH_2OH$, TMS derivative	0.2	0.6	0.4
16	9:24	251	MeP(O)(OEt)OR	<0.1	<0.1	<0.1
17	9:48	221	$RSCH_2CH_2SH$	7.3	8.3	11.4
18	11:22	283	MeP(S)(OEt)SR	<0.1	<0.1	<0.1
19	11:38	288	RSR	2.4	2.5	2.7
20	13:15	320	RSSR	0.8	0.5	0.5
21	13:27	224	*N,N′*-Dicyclohexylurea [2387-23-7]	0.1	<0.1	0.3
22	15:12	348	$RSCH_2CH_2SR$	<0.1	–	<0.1
23	17:19	380	$RSCH_2CH_2SSR$	0.4	0.6	1.8

Numbers given in square brackets are the compounds' CAS numbers.

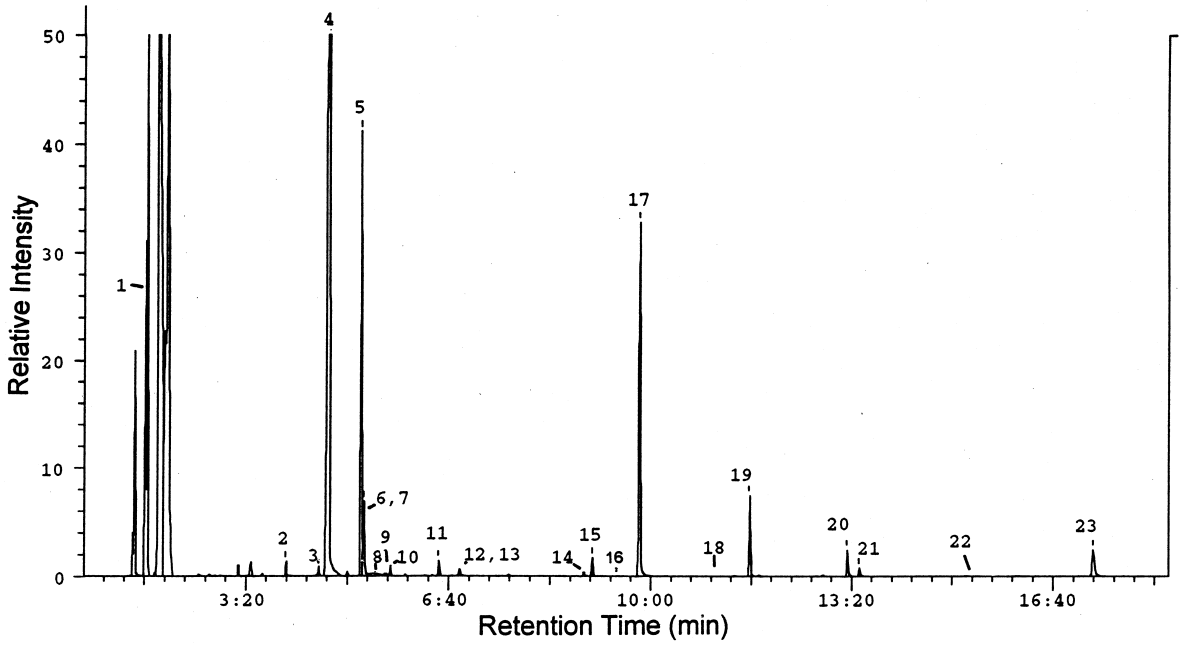

Fig. 1. GC–MS chromatogram of BSTFA-derivatized equimolar VX–water product (30 m×0.25 mm DB-5 column, 60–270°C at 15°C/min, 5 min at 270°C).

revealed abnormally high disulfide concentrations in some samples. As shown in Fig. 3, oxidation of thiols in the mixture to disulfides occurs within days after derivatization. It is well known that thiols oxidize to disulfides via a free radical mechanism in the presence of air, particularly in the presence of base [14]. It is surprising that so little oxidation was observed in the original VX–water product over the six month period. The sample was stored in a small vial, 1 ml, with a 0.1–0.3 ml air space, throughout the study. The vial was kept tightly sealed between sampling, but no attempt was made to replace the air with an inert atmosphere. The label covered most of the vial so there was little exposure to light. The

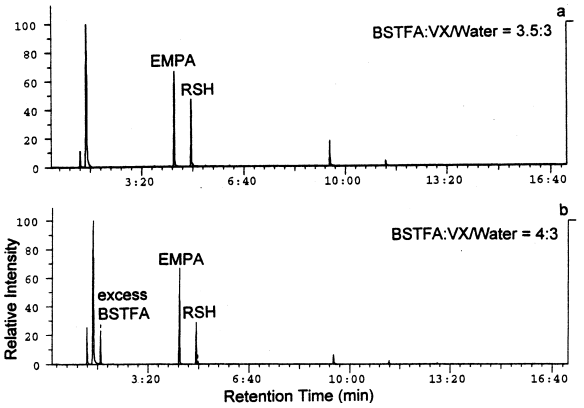

Fig. 2. GC–MS chromatograms of BSTFA–(VX–water) products at ratios of (a) 4:3 and (b) 3.5:3.

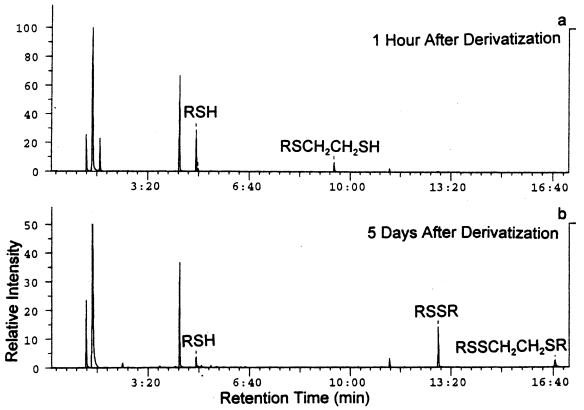

Fig. 3. GC–MS chromatograms of the VX–water product (a) 1 h and (b) five days after BSTFA derivatization.

derivatized product contained a larger air space (0.23 ml of air to 0.07 ml of product) with greater light exposure. It is conceivable the enhanced reactivity of the derivatized solution could be due to catalysis by derivatizing agent byproducts or to greater exposure to air and/or light. Further studies are required to determine the primary source of this oxidation. It is important to note that the derivatized product should be analyzed immediately after derivatization or stored under an oxygen-free atmosphere and/or away from light, to achieve accurate results. It should also be noted that 2-(diisopropylamino)ethanethiol does not derivatize under the conditions used here. Partial derivatization does occur when excess derivatizing agent is used and when the heated reaction time is increased.

Correlation with recent NMR data [13] provides support that all major (>1%) equimolar VX–water hydrolysis products are detected by this method. It should be noted, however, that one VX hydrolysis product, *S*-2-(diisopropylamino)ethyl methylphosphonothioic acid, because of its zwitterionic character, is not derivatized by BSTFA and, therefore, is not detected under these conditions. This acid forms primarily under alkaline conditions. Analysis of equimolar VX–water reaction products by ^{31}P NMR indicates that this acid is usually present at levels below 1%. It is rationalized that this acid is formed during reaction, not from the hydrolysis of VX, but from the hydrolysis of *S*,*S*-bis[2-(diisopropylamino)ethyl] methylphosphonodithioate, an impurity present in the VX [13]. Detection of this acid is very important because its toxicity approaches that of VX and its hydrolysis to methylphosphonic acid is slow. This acid can be detected by NMR [15] and by liquid chromatography (LC)–MS–MS with atmospheric pressure chemical ionization (APCI) [16]. Although this compound resists most derivatization attempts, one method, using trimethylphenyl ammonium hydroxide (TMPAH) to induce methylation in a heated GC injection port, has been reported, but with a detection limit of only 100 parts per million (ppm) in decontamination matrices [16].

The efficiency of VX destruction during the equimolar VX–water reaction has been shown to be >99.99% after 4 h at 90°C [13]. A comparison of various decontamination processes is discussed in two review articles [17,18]. Although the objective of this study was the detection of degradation products at concentrations >0.1%, a discussion of low level detection of VX is warranted. The presence of BSTFA does not appear to interfere with the detection of VX down to 0.1%. The use of GC–MS for the detection of VX at low ppm concentrations in decontamination matrices, however, is severely limited by background interference and the potential for agent reformation. The interference occurs because of the large number of degradation products containing the diisopropylaminoethyl group at the ppm level that have spectra similar to that of VX. This problem can be minimized by taking advantage of the increased specificity offered by tandem mass spectrometry, particularly in combination with CI, which gives enhanced formation of the protonated molecular ion at *m*/*z* 268, which can be used as the precursor ion. The problem of agent reformation is more difficult to deal with. Our experience has shown that VX at low ppm levels is often detected in decontamination solutions, both in the BSTFA-derivatized equimolar VX–water products and in chlorinated solvent extracts of VX–caustic products by GC–MS at elevated levels that are inconsistent with NMR data. The source of this discrepancy is not clear and warrants further investigation. LC–MS–MS, a technique that allows the direct analysis of the decontamination solution without extraction or work-up, may have application for the detection of VX at part per billion (ppb) levels. Because of the presence of nitrogen, VX is ideally suited for the application of APCI.

EI and methane CI spectra for the major products (greater than 0.1%) are provided in Figs. 4 and 5. As shown, EI spectra of the higher-molecular-mass sulfides and disulfides containing the diisopropylaminoethyl group are dominated by the ion at *m*/*z* 114 ($[i\mathrm{Pr}_2\mathrm{N}{=}\mathrm{CH}_2]^+$). Because of the spectral similarities and the absence of molecular ion information, compound identification based on EI spectra alone is difficult. Methane CI was a valuable technique and provided the molecular ion and fragmentation information necessary to identify these compounds. Structure assignments for the major fragment ions of VX and VX impurities have been reported previously [12]. Table 2 lists the eight most

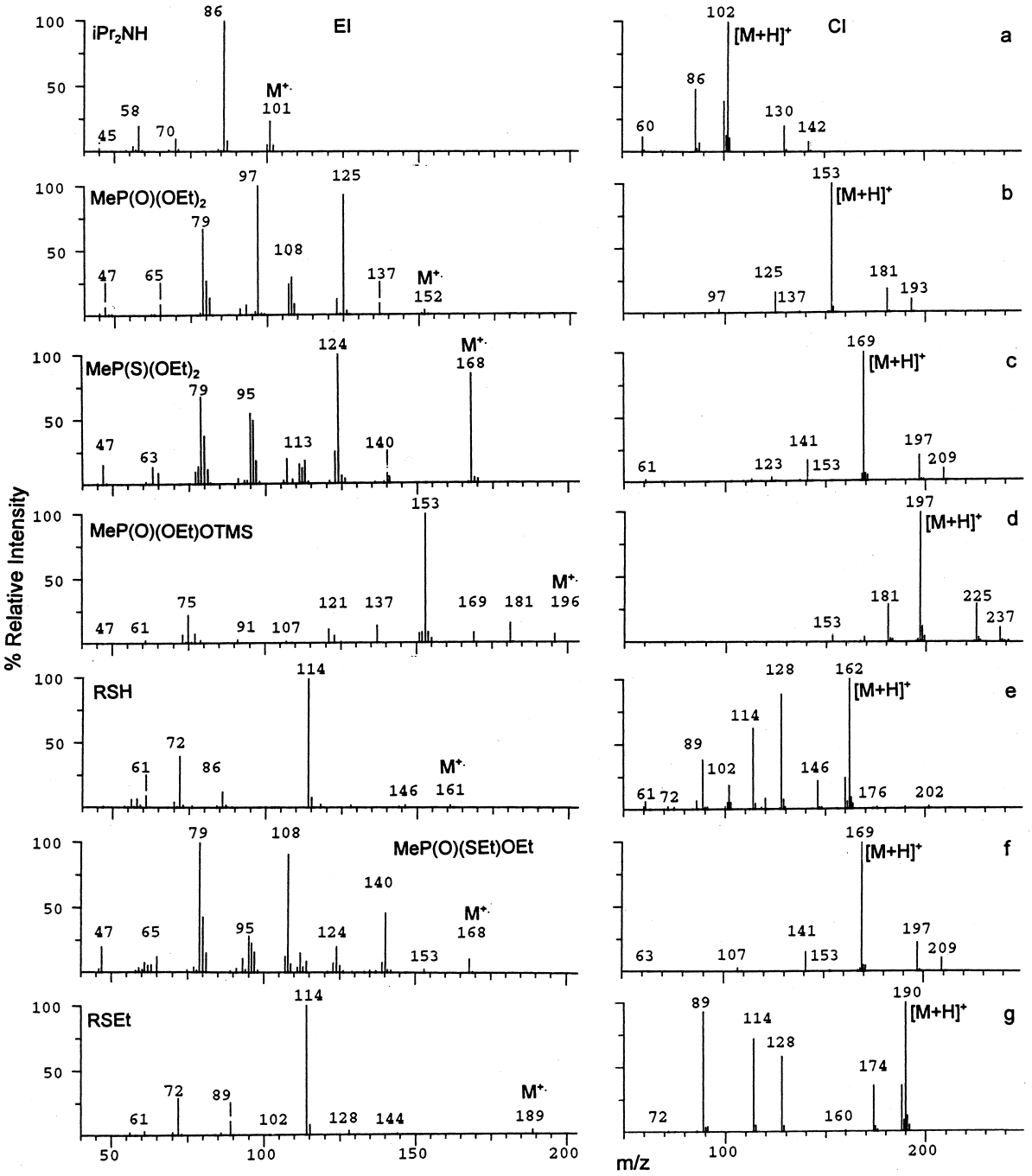

Fig. 4. EI and methane CI mass spectra of (a) diisopropylamine, (b) *O,O*-diethyl methylphosphonate, (c) *O,O*-diethyl methylphosphonothioate, (d) ethyl methylphosphonic acid, TMS derivative, (e) 2-(diisopropylamino)ethanethiol, (f) *O,S*-diethyl methylphosphonothioate and (g) 2-(diisopropylamino)ethyl ethyl sulfide [R=-$CH_2CH_2N(iPr)_2$].

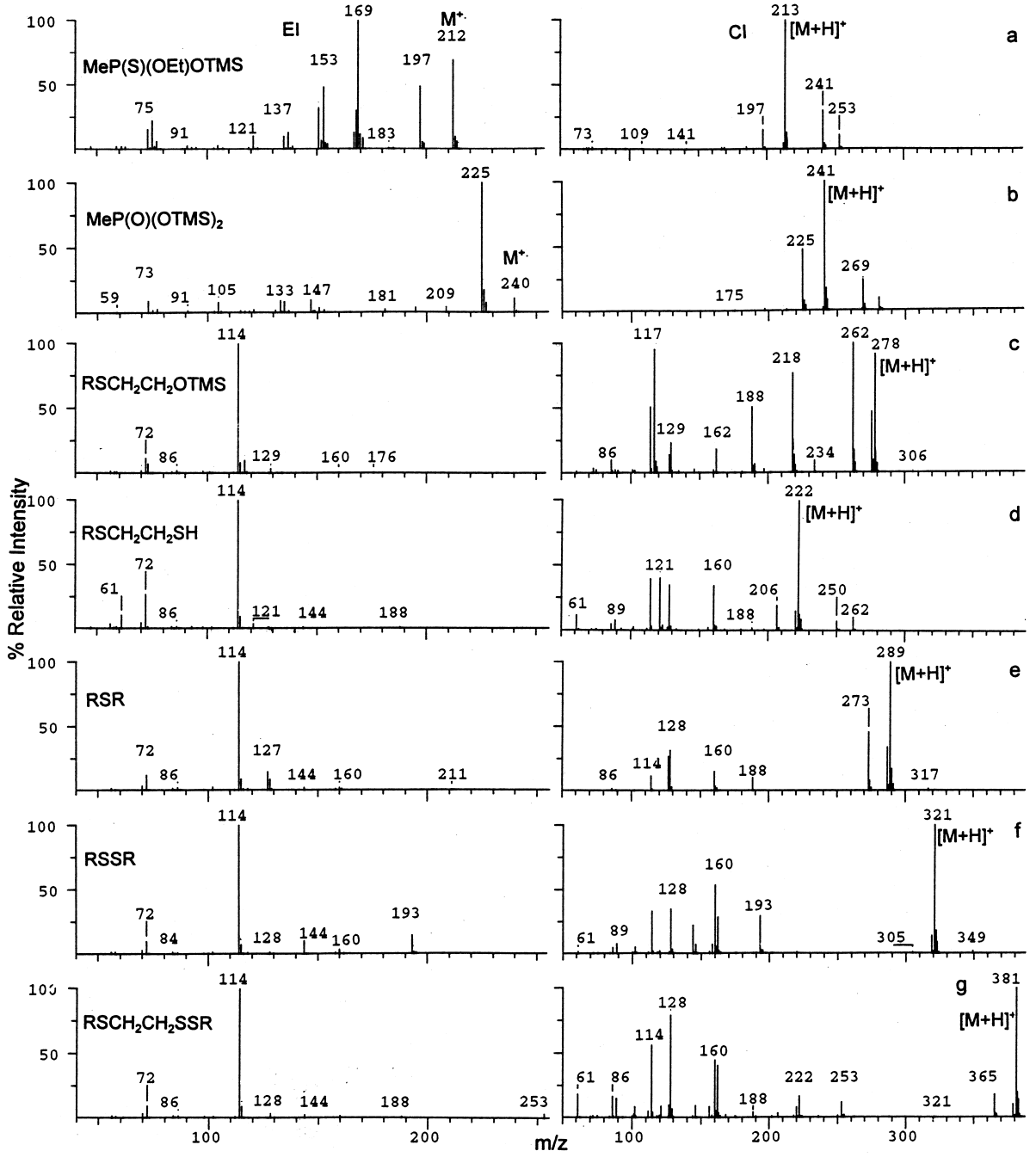

Fig. 5. EI and methane CI mass spectra of (a) *O*-ethyl methylphosphonothioic acid, TMS derivative, (b) methylphosphonic acid, di-TMS derivative, (c) 2-[2-(diisopropylamino)ethylthio]ethanol, TMS derivative, (d) 2-[2-(diisopropylamino)ethylthio]ethanethiol, (e) bis[2-(diisopropylamino)ethyl] sulfide, (f) bis[2-(diisopropylamino)ethyl] disulfide and (g) 1,9-bis(diisopropylamino)-3,6,7-trithianonane [R= $CH_2CH_2N(iPr)_2$].

Table 2
EI and methane CI mass spectral data for minor degradation products observed in BSTFA-derivatized equimolar VX–water [R=-$CH_2CH_2N(iPr)_2$]

Peak number	Compound	M_r	*m/z* (*% base peak*)										
8	$C_8H_{17}NS$	159	EI	159	144	112	102	101	98	84	70		
				37	*100*	*22*	*52*	*13*	*34*	*41*	*37*		
			CI	161	160	159	158	144	101	100	89		
				8	*100*	*22*	*13*	*16*	*10*	*7*	*14*		
9	ROTMS	217	EI	144	115	114	73	72	86	70	59	(202)	(217)
				4	*8*	*100*	*5*	*15*	*2*	*2*	*3*	*1*	*0.6*
			CI	219	218	217	216	203	202	128	114		
				12	*63*	*38*	*20*	*16*	*100*	*13*	*15*		
11	Cyclohexyl isothiocyanate	141	EI	142	141	83	82	72	67	55	53		
				9	*100*	*77*	*32*	*7*	*23*	*87*	*7*		
			CI	144	143	142	141	100	88	84	83		
				3	*7*	*93*	*8*	*13*	*33*	*6*	*100*		
12	$RSCH{=}CH_2$	187	EI	187	115	114	87	72	70	59	56		
				2	*7*	*100*	*15*	*32*	*4*	*2*	*3*		
			CI	188	186	172	128	114	95	87	69		
				100	*19*	*28*	*54*	*81*	*6*	*34*	*5*		
14	*N,N′*-Diisopropylthiourea	160	EI	160	159	117	85	69	60	59	58		
				42	*5*	*5*	*5*	*1*	*9*	*2*	*100*		
			CI	201	189	163	162	161	160	127	60		
				10	*42*	*4*	*8*	*100*	*7*	*4*	*4*		
16	MeP(O)(OEt)OR	251	EI	127	114	112	85	84	72	70	44	(208)	(236)
				36	*100*	*34*	*18*	*32*	*26*	*36*	*9*	*5*	*3*
			CI	253	252	250	236	129	128	127	114		
				10	*93*	*16*	*21*	*7*	*100*	*15*	*3*		
18	MeP(S)(OEt)SR	283	EI	155	127	115	114	95	72	70	47		
				2	*11*	*8*	*100*	*7*	*13*	*4*	*3*		
			CI	286	285	284	282	268	183	128	114		
				8	*14*	*100*	*9*	*15*	*5*	*42*	*11*		
21	*N,N′*-Dicyclohexylurea	224	EI	224	143	100	99	98	70	61	56		
				58	*57*	*23*	*83*	*46*	*28*	*37*	*100*		
			CI	265	253	226	225	223	100	98	83		
				6	*30*	*13*	*100*	*18*	*9*	*7*	*3*		
22	$RSCH_2CH_2SR$	348	EI	188	160	135	128	115	114	72	70		
				2	*2*	*4*	*9*	*9*	*100*	*10*	*2*		
			CI	351	350	349	347	333	188	128	114		
				11	*23*	*100*	*17*	*21*	*6*	*24*	*16*		

abundant peaks and their ratios as well as other significant peaks (in parentheses) observed for the nine less abundant degradation products.

The stability data in Table 1 suggests that the VX–water product, stored at ambient temperature in a closed container, is relatively stable over a period of six months, with only small amounts of thiol secondary reaction (i.e. 2 $RSH \rightarrow RSCH_2CH_2SH + iPr_2NH$) and minimal oxidation (i.e. 2 $RSH \rightarrow RSSR$) observed [$R=(iPr)_2NCH_2CH_2$-]. The proposed mechanism for the thiol secondary reaction involves decomposition of the thiol via the formation of a zwitterion intermediate ($^-SCH_2CH_2N^+HiPr_2$) and neighboring sulfur assistance to produce thiirane and

diisopropylamine [2]. Thiirane then can react with another molecule of RSH to give $RSCH_2CH_2SH$. Further polymerization to give $RS(CH_2CH_2S)_nH$ might be expected, but was not detected.

4. Conclusions

BSTFA derivatization followed by electron and chemical ionization GC–MS analysis was successfully applied to detect and identify 23 products in an equimolar VX–water reaction product. The major products were ethyl methylphosphonic acid and the thiols 2-(diisopropylamino)ethanethiol and 2-[2-(diisopropylamino)ethylthio]ethanethiol. Methane chemical ionization provided molecular ion information that was not provided by electron ionization and was, therefore, particularly useful for the identification of the higher-molecular-mass products. Derivatization with 40 μl of BSTFA to 30 μl of reaction product provided complete derivatization and at the same time allowed one to detect diisopropylamine in the presence of derivatizing agent. It is imperative that the derivatized product be analyzed as soon as possible after derivatization or that it is stored under an inert atmosphere and/or in the absence of light to prevent thiol oxidation side reactions. The VX–water reaction product, when stored at ambient temperature in a sealed container, is relatively stable over a period of six months, with only a small amount of thiol secondary reaction and minimal oxidation being observed.

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References

[1] Recommendations for Disposal of Chemical Agents and Munitions, Committee on Review and Evaluation of the Army Chemical Stockpile Disposal System, National Research Council, National Academy Press, Washington, DC, 1994.
[2] Y.-C. Yang, L.L. Szafraniec, W.T. Beaudry, D.K. Rohrbaugh, L.R. Procell, J.B. Samuel, J. Org. Chem. 61 (1996) 8407–8413.
[3] G. Bauer, W. Vogt, Anal. Chem. 53 (1981) 917–919.
[4] Conference on Disarmament, The Convention on the Prohibition of the Development, Production, Stockpiling and Use of Chemical Weapons and on their Destruction, CD/1170, Geneva, August 1992.
[5] Report of the Mission Dispatched by the Secretary-General to Investigate the Use of Chemical Weapons in the Conflict Between the Islamic Republic of Iran and Iraq, S/20060, United Nations Security Council, New York, 20 July, 1988.
[6] L. Ember, C&E News 71 (1993) 8–9.
[7] M. Nagao, T. Takatori, Y. Matsuda, M. Nakajima, H. Iwase, K. Iwadate, Toxicol. Appl. Pharmacol. 144 (1997) 198–203.
[8] W.R. Creasy, J.R. Stuff, B. Williams, K. Morrissey, J. Mays, R. Duevel, H.D. Durst, J. Chromatogr. A 774 (1997) 253–263.
[9] M. Katagi, M. Nishikawa, M. Tatsuno, H. Tsuchihashi, J. Chromatogr. B 689 (1997) 327–333.
[10] H. Gu, L. Zhou, G. Cai, Y. Zhong, Sepu 14 (1996) 41–44.
[11] C. Boulet, P.A. D'Agostino, Phosphorus, Sulfur, Silicon, Relat. Elem. 104 (1995) 93–101.
[12] P.A. D'Agostino, L.R. Provost, L. Visentini, J. Chromatogr. 402 (1987) 221–232.
[13] J.B. Samuel, L.L. Szafraniec, W.T. Beaudry, S.H. Hong, D.K. Rohrbaugh, Y.-C. Yang, Hydrolysis of VX With Equimolar Water at Elevated Temperatures, ERDEC-TR-426, US Army Edgewood, Research, Development and Engineering Center Technical Report, Aberdeen Proving Ground, MD, July 1997.
[14] T.J. Wallace, A. Schriesheim, W. Bartok, J. Org. Chem. 28 (1963) 1311–1314.
[15] Y.-C. Yang, L.L. Szafraniec, W.T. Beaudry, D.K. Rohrbaugh, J. Am. Chem. Soc. 112 (1990) 6621–6627.
[16] W.R. Creasy, J. Mays, R. Deuvel, T.R. Connell, B.R. Williams, Proceedings of the 1996 Conference on Chemical and Biological Defense Research, 19–22 November 1996, Aberdeen Proving Ground, MD, October 1997, pp. 653–659.
[17] Y.-C. Yang, Chem. Ind. 9 (1995) 334–337.
[18] Y.-C. Yang, J.A. Baker, J.R. Ward, Chem. Rev. 92 (1992) 1729–1743.