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Gas chromatography–mass spectrometry analysis of trifluoroacetyl derivatives of precursors of nitrogen and sulfur mustards for verification of chemical weapons convention

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Abstract

Trifluoroacetylation reactions were optimized for gas chromatography–mass spectrometric (GC–MS) analysis of precursors of nitrogen and sulfur mustards using newly developed *N*-trifluoroacetylbenzimidazole (TFABI) and known *N*-trifluoroacetylimidazole (TFAI) and *N*-trifluoroacetylbenzotriazole (TFABT) reagents. TFAI and TFABI gave the best results in terms of yields and reaction time. Trifluoroacetyl derivatives showed clean total ion chromatograms in GC–MS analysis than trimethylsilyl (TMS) derivatives. The negative ion chemical ionization analysis of these derivatives gave pseudo molecular weight information, with low limit of detection.

Keywords: Nitrogen mustard; Sulfur mustard; Precursors; Chemical warfare agents; Chemical weapons convention; Ethanolamines; 2-Hydroxythioethers; Trifluoroacetylations

1. Introduction

Development of analytical methods of chemical warfare (CWAs) and their related chemicals has gained importance in light of verification program of chemical weapon convention (CWC) [1–4]. The CWC prohibits production, storage and use of CWAs [5]; by April 2004, the treaty has been endorsed by 164 countries. The Organization for the Prohibition of Chemical Weapons (OPCW), seated in The Netherlands ensures implementation of CWC by executing its strict verification program [6]. Verification of CWC involves collection and analysis of samples from production, storage and suspected sites during routine or challenge inspections. OPCW inspectors perform on-site analysis of collected samples for CWAs and their degradation products. In case of any ambiguity, the samples are sent to two designated laboratories appointed by OPCW for unequivocal identification of convention related chemicals (CRCs) [3,7–11]. The convention related chemicals are annexed in the CWC text as Schedule 1–3 chemicals [5,6,8]. The CRCs are categorized in three schedules based on their potential use as CWAs or their precursors.

Nitrogen and sulfur mustards (NMs and SMs) are cytotoxic, alkylating and blistering agents, and placed in Schedule 1.4 and 1.6, respectively. Precursors of NMs and SMs are ethanolamines and 2-hydroxyethyl thioethers, respectively, their structures are depicted in Fig. 1.

These nitrogen mustard precursors (NMPs) and sulfur mustard precursors (SMPs) produce corresponding toxic mustards in a single step by nucleophilic substitution of hydroxyl group by chlorine [12], as typically illustrated in Fig. 2.

Hence, these NMPs and SMPs function as important markers of sulfur and nitrogen mustards. Therefore, their detection and identification in a sample submitted for off-site analysis may imply past contamination with mustards. It makes trace analysis of these compounds very important from verification point of view of CWC.

The trace analysis of CRCs can be achieved by employing various chromatographic and spectroscopic techniques

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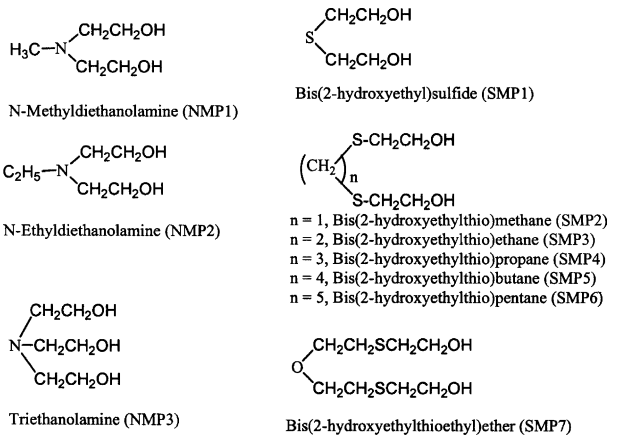

Fig. 1. Precursors of nitrogen and sulfur mustards.

such as gas chromatography coupled with mass spectrometry (GC–MS) nuclear magnetic resonance (NMR) and liquid chromatography coupled to mass spectrometry (LC–MS). Amongst these techniques, GC–MS in electron and chemical ionization modes is the most sought after technique [7,8], as it provides characteristic spectrum and molecular weight information of analytes even at very low concentrations. The requirement of GC–MS analysis is that the analyte should possess sufficient volatility and thermal stability. Hence, polar and non-volatile precursors and degradation products of CWAs need derivatisation prior to GC–MS analysis. Because, underivatized species are sufficiently nonvolatile therefore, their GC–MS is either not feasible or difficult with poor sensitivity.

Derivatisation facilitates chromatographic analysis by imparting required volatility to polar analytes [13]. Black and Muir [14] have recently reviewed derivatisation reactions of CWAs and their degradation products. These reactions include methylation, trimethylsilylation, *tert*-butyldimethyl silylation, pentafluorobenzylation and pentafluorobenzoylation. Recent trends in derivatisation involve conversion of polar analytes into fluorinated derivatives as it enables detection by a very sensitive technique of negative ion chemical ionization mass spectrometry (NICI-MS) [14,15].

The derivatization reactions of NMPs and SMPs for GC–MS analysis involve their conversion into trimethylsilyl (TMS) and *tert*-butyldimethylsilyl (TBDMS) ethers [14]. These reactions are used by most of the laboratories involved in off-site analysis and participating in OPCW proficiency tests [7–11]. The versatile nature of silylation is a drawback for environmental samples like soil and water. Silylation of extracted samples from these matrices leads to derivatization of analytes (present in trace level) together with carboxylic

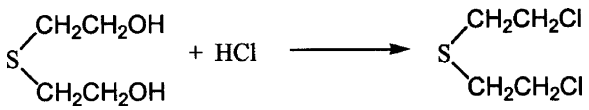

Fig. 2. Formation of sulfur mustard from SMP1.

and alcoholic function bearing impurities (present in larger amount), as silylation of acids and alcohols takes place with equal efficiency. It results into masking of analyte peaks by high background. Therefore use of derivatizing agent that reacts with only hydroxyl function, leaving carboxylic groups can reduce background to a greater extent.

Another problem with silylating agents such as *N*,*O*-bis (trimethylsilyl)trifluoroacetamide (BSTFA) and *N*-(*tert*-butyldimethylsilyl)–*N*-methyl-trifluoroacetamide (MTBSTFA) is that they themselves also give rise to additional peaks in total ion chromatogram (TIC). These background peaks can mask other important CRCs present in a given sample. One more limitation of silylated derivatives is that they can only be analyzed in positive mode by GC–MS, where universal nature of electron ionization (EI) in positive mode also gives several unwanted peaks in TIC. Whereas, NICI-MS can be used to alleviate these problems, since it is applicable only to elctrophilic molecules. It makes negative ion chemical ionization (NICI) more sensitive and selective for determination of small amounts of analytes.

Thus, it was very strongly desirable to find alternative procedures that would derivatize the hydroxyl functionality selectively, thereby producing a 'clean' total ion chromatogram in the EI mode while enhancing the limits of detection in the NICI mode. For that reason, we explored trifluoroacetylation of precursor diols of NMs and SMs. To the best of our information, no systematic trifluoroacetyl derivatization of NMPs and SMPs with their positive EI and NICI GC–MS analysis is reported.

In this communication, we are reporting trifluoroacetyl (TFA) derivatives of NMPs and SMPs, which are found to be better than silyl derivatives for GC–MS analysis in EI and NICI mode. In EI mode, these TFA derivatives were detected with minimum background in TIC. We are also reporting the optimized NICI conditions where we could achieve higher sensitivity with pseudo molecular weight information.

Trifluoroacetylation can be performed by the use of any of reagents like trifluoroacetic anhydride (TFAA), *S*-ethyl trifluorothioacetate (*S*-ETFA), ethyl trifluoroacetate (ETFA), 2-(trifluoroacetoxy)pyridine (TFAP), trifluoroacetyl triflate (TFAT) and *N*-(trifluoroacetoxy)succinimide (TFAS) [16]. These trifluoroacetylating agents have disadvantages as derivatising agents. TFAA and TFAT produce strong acids after reaction that can damage a gas chromatographic column. ETFA and TFAP do not posses sufficient reactivity; *S*-ETFA produces interfering thiols; and TFAS is highly unstable [16].

For the present investigation, three reagents namely *N*-trifluoroacetylimidazole (TFAI), *N*-trifluoroacetylbenzimidazole (TFABI) and *N*-trifluoroacetylbenzotriazole (TFABT) were evaluated for trifluoroacetylation of NMPs and SMPs (Fig. 3). These reagents posses adequate reactivity and do not produce acid after reaction; hence, they do not require additional base for derivatization. TFAI is commercially available trifluoroacetylating agent [17], TFABI was prepared in our laboratory and TFABT was prepared as reported by Katritzky [16].

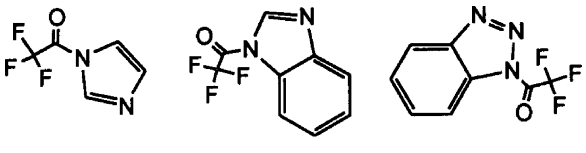

Fig. 3. The derivatizing reagents.

2. Experimental

2.1. Materials

N-Trifluoroacetylimidazole and *N*,*O*-bis(trimethylsilyl)-trifluoroacetamide (BSTFA) were obtained from Lancaster synthesis Inc., Windham, NH, USA. *N*-Methyldiethanolamine, *N*-ethyldiethanolamine, triethanolamine and bis(2-hydroxyethyl)sulfide were obtained from Fluka (Sigma–Aldrich, Powai, Mumbai, India). All other precursors of sulfur mustards (SMP2–SMP7) were synthesized according to an open literature procedure [18]. The products were characterized by NMR, IR and MS analysis and determined to be >96% pure. TFABI was prepared by treating 0.1 moles benzimidazole with 0.18 moles trifluoroacetic anhydride at 0–5 °C in absence of solvent. After keeping for 3 h at room temperature, the TFABI was extracted with dry *n*-hexane. TFABT was prepared as per reported procedure [16].

2.2. Derivatization procedure

Stock solutions of NMPs and SMPs were prepared in dry acetonitrile (CH_3CN) at the concentration of 1 mg/mL. These solutions were diluted to 20 μg/mL with dry CH_3CN in different sealed vials. Derivatisation involved carefully adding measured quantities of 10% solutions of BSTFA, TFAI, TFABI and TFABT in dry acetonitrile to give final concentration of 10 μg/mL of analytes. Reactions were performed at room temperature and/or heating blocks at different temperatures for definite time. Reaction mixtures were cooled and volume was adjusted to a definite value to maintain the required concentration. Volumetric measurements were made by using Qualigens calibrated Qualipette, reactions were carried out in screw cap glass vials with PTFE silicon septa (Supelco, Bellefonte, PA, USA). Derivatizations did not include any extraction or clean-up procedures.

2.3. GC–MS analysis

The GC–MS analyses were performed in EI mode (70 eV) with an Agilent 6890 GC system, equipped with model 5973 mass selective detector (Agilent Technologies, USA). SGE BPX5 fused silica capillary columns (30 m × 0.32 mm i.d., 0.25 μm film thickness) were employed for both EI and NICI determinations. In both cases, the column oven temperature was raised linearly from 50 °C (hold for 2 min) to 280 °C (hold for 5 min) at 10 °C/min. Helium was used as carrier gas at constant flow of 1.2 ml/min. The samples were analyzed in splitless mode at injection temperature of 250 °C, EI source temperature 230 °C and quadrupole analyzer at 150 °C.

GC–MS analysis in NICI mode was performed on an Agilent 6890N GC system, equipped with model 5973N mass selective detector (Agilent Technologies, USA). The samples were analyzed in splitless mode at injection temperature of 250 °C, CI source temperature 150 °C, quadrupole analyzer at 150 °C and ionization current at 235 eV. The methane was used as buffer gas at a flow rate of 2 mL/min.

3. Results and discussion

Trifluoroacetylation reactions are typically illustrated in Fig. 4 with examples of SMPs. Unlike reactions with trifluoroacetic anhydride, these reactions do not require additional base to drive the reaction to completion. Our first aim was to optimize the reaction conditions for trifluoroacetylations of NMPs and SMPs with TFAI, TFABI and TFABT. The parameters that have been optimized were solvent, time and temperature of reaction. To select the best solvent, reactions were carried out in aprotic solvents of different polarity at room temperature for 30 min. To optimize the reaction time and temperature, derivatizations were done with best solvent. During optimization of reaction temperature, formation of side products was also studied. Concentrations of NMPs and SMPs were kept approximately 10 ng/μL, and peak areas of trifluoroacetylated products were used for comparison purposes.

Various solvents tested for reactions were *n*-hexane, *n*-heptane, dichloromethane, tetrahydrofuran (THF), diethyl ether and acetonitrile. With TFAI and TFABI the derivatizations took place smoothly in all of the tested solvents. TFABT gave poor yields (less than 10%) in all of the solvents. Amongst these solvents *n*-heptane showed TIC without any background. Even the bases (imidazole, benzimidazol and benzotriazole; Fig. 4) produced during reactions were not

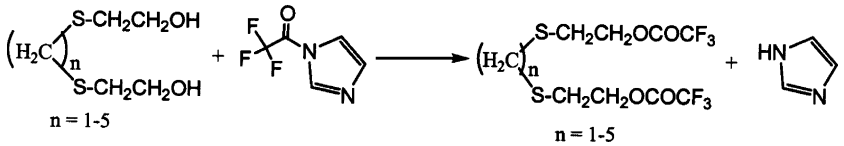

Fig. 4. Trifluoroacetylation of bis(2-hydroxyethylthio)alkane with TFAI.

observed in TIC, which could be due to insolubility of these bases in *n*-heptane. In all of the other solvents including *n*-hexane broad peaks of bases were observed with varying intensities depending on their solubility. Hence, *n*-heptane was selected as solvent of choice for these trifluoroacetylations.

In order to optimize the temperature and time, the reactions were carried out at 15, 30 and 70 °C and analyzed at different time intervals. The quantities produced with TFAI at 70 °C after heating for 4 h were taken as 100% conversion for comparison purposes. The reactions were run in triplicate and average value of peak areas of three runs were taken as yields; relative standard deviations (R.S.D.) were within ±3%. The 100% conversion took place with TFABI and TFAI within 5 min even at 15 °C. Further heating of NMPs and SMPs with TFAI and TFABI for extended periods did not increase the yields, which ensured the completion of reaction. Reactions with TFABT were slower, even on heating the reaction mixture for more than 120 min at 70 °C in various solvents did not enhanced the amount of products. Reaction mixtures were not heated beyond 70 °C as it could degrade other important CWC related analytes present in a given sample. Thus for trifluoacetylations of SMPs and NMPs, TFAI and TFABI are found to be best-suited derivatising reagents. Higher reactivity of the imidazole ring (pK_a 7.03) compare to that of triazole (pK_a 8.3) may be due to the lower basicity of former. This lower basicity facilitated release of the ttrifluoroacetyl group when attacked by the nucleophile, which was the hydroxyl functionality of the precursors.

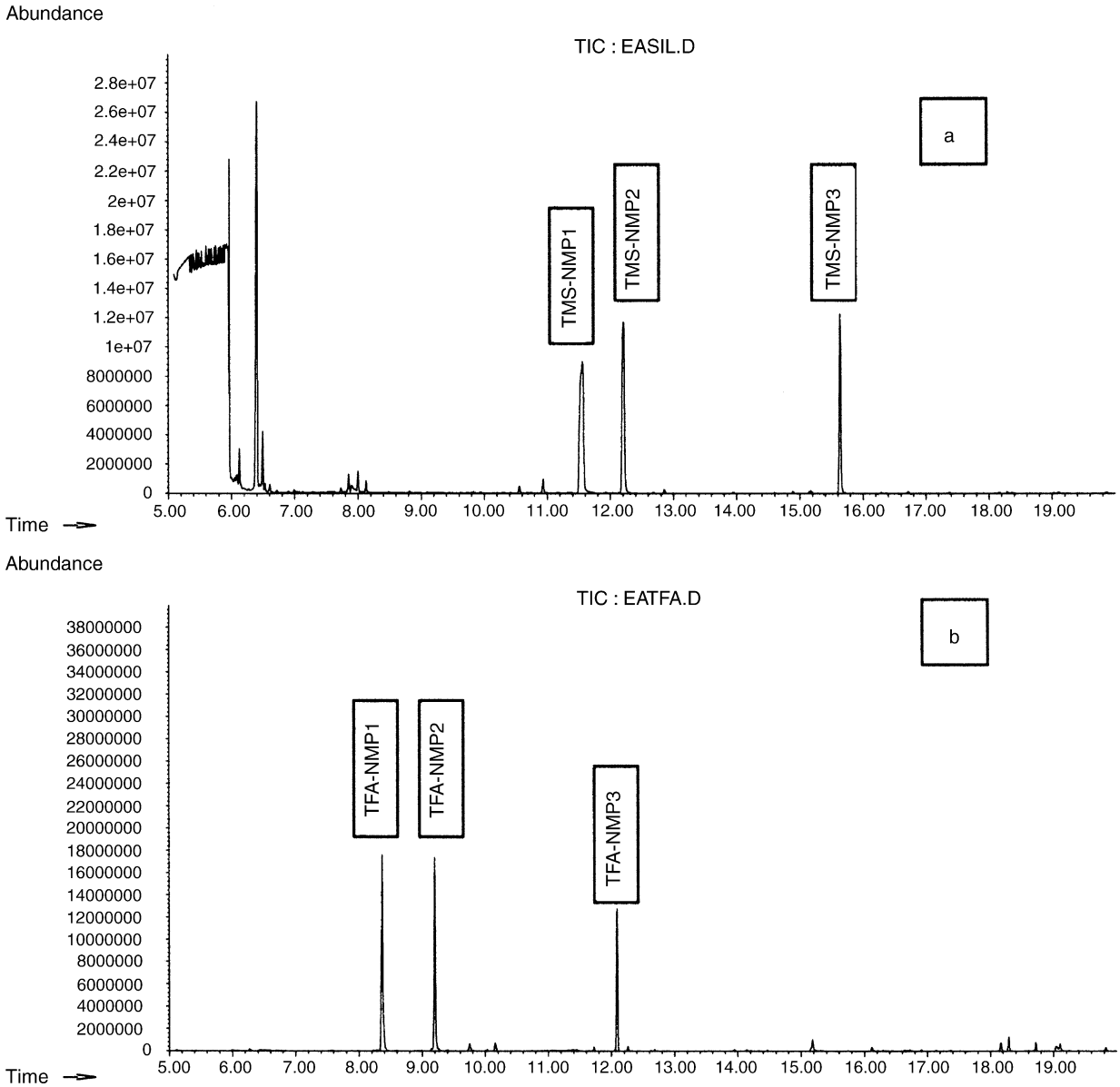

Fig. 5. TIC of GC–EI-MS analysis of (a) TMS derivatives of NMPs (10 μg/mL) and (b) TFA derivatives of NMPs (10 μg/mL) formed with TFAI.

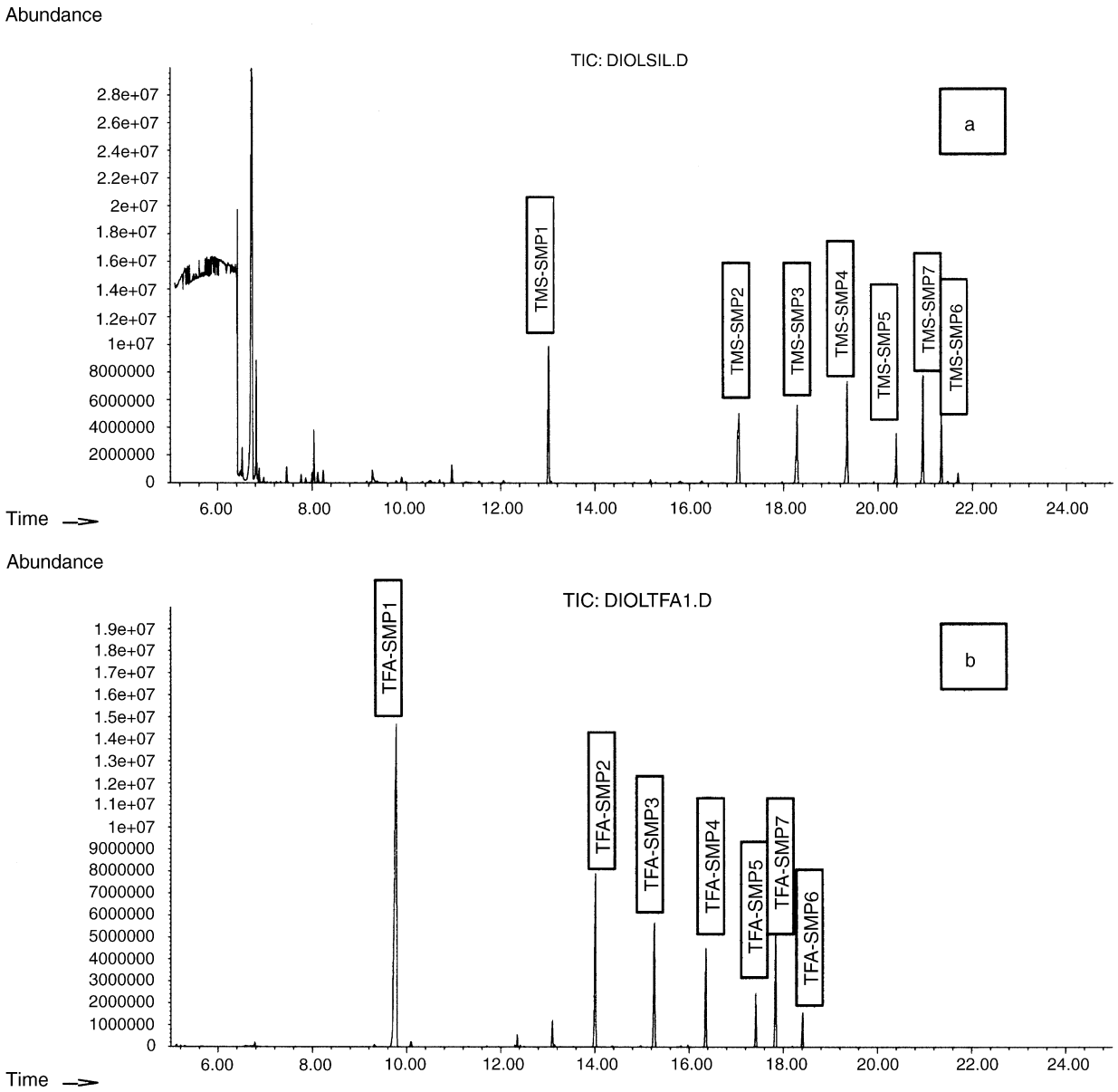

Fig. 6. TIC of GC–EI-MS analysis of (a) TMS derivatives of SMPs (10 μg/mL) and (b) TFA derivatives of SMPs (10 μg/mL) formed with TFAI.

3.1. Comparison of total ion chromatograms (TICs) in GC–EI-MS analysis of silylated and trifluoroacetylated derivatives

GC–EI-MS analysis of trimethylsilyl and TFA derivatives of NMPs and SMPs are respectively shown in Figs. 5 and 6. It is evident from Fig. 5a versus Fig. 5b and Fig. 6a versus Fig. 6b that TICs of TFA derivatives had no background as compared to TMS derivatives. The TMS derivatives showed huge background of un-reacted BSTFA and side product trifluoroacetamide. These background peaks can mask other important analytes if present in a given sample. Whereas, TIC of TFA derivatives is absolutely without any background peaks, which is of particular importance for detection of CWC related volatile analytes like pinacolyl alcohol (Sched-

Table 1
Negative ion chemical ionization (NICI) mass spectral data of TFA derivatives of NMPs and SMPs

S. no.	TFA derivatives	*m/z* values (relative abundances)	
		$[M + CF_3COO]^-$	Fragment ions
1	NMP1	424 (24)	227 (5), 113 (100)
2	NMP2	438 (23)	227 (5), 113 (100)
3	NMP3	550 (21)	227 (43), 113 (100)
4	SMP1	427 (16)	227 (100), 113 (67)
5	SMP2	473 (25)	332 (4), 227 (4), 113 (100)
6	SMP3	487 (14)	227 (100), 113 (38)
7	SMP4	501 (20)	360 (4), 227 (6), 113 (100)
8	SMP5	515 (9)	374 (4), 227 (8), 113 (100)
9	SMP6	529 (2)	227 (21), 113 (100)
10	SMP7	531 (6)	227 (100), 113 (45)

Table 2
Electron ionization mass spectral data of TFA derivatives of NMPs and SMPs

S. no.	TFA derivatives	*m/z* values (relative abundances)	
		$[M]^{\bullet+}$	Fragment ions
1	NMP1	311 (2)	198 (9), 184 (100), 141 (68), 113 (4), 69 (49), 42 (43)
2	NMP2	325 (2)	310 (4), 212 (10), 198 (100), 141 (55), 113 (4), 69 (49), 42 (26)
3	NMP3	437 (1)	324 (16), 310 (100), 141 (69), 113 (2), 69 (39), 42 (15)
4	SMP1	314 (2)	200 (31), 141 (91), 113 (9), 87 (73), 69 (100), 45 (40)
5	SMP2	360 (17)	246 (2), 219 (2), 187 (86), 141 (100), 113 (4), 69 (8), 47 (21)
6	SMP3	374 (11)	260 (3), 233 (5), 201 (50), 141 (100), 113 (5), 91 (7)
7	SMP4	388 (15)	274 (1), 247 (84), 215 (3), 187 (5), 141 (100), 100 (14), 69 (48)
8	SMP5	402 (10)	288 (2), 261 (52), 229 (19), 187 (30), 141 (100), 115 (21), 87 (3), 69 (59)
9	SMP6	416 (<1)	302 (4), 275 (12), 243 (17), 187 (19), 141 (58), 129 (21), 101 (37), 69 (100)
10	SMP7	–	304 (2), 277 (4), 244 (1), 201 (36), 141 (100), 113 (4), 87 (31), 69 (43), 45 (29)

ule 2B.14), phosphites (Schedule 3B.8-11) and several phosphonates (Schedule 2B.4), that elute in this region only. Thus, if such early eluting volatile analytes are present along with SMPs and NMPs these might get masked by the background peaks of TMS derivatives; while with TFA derivatives no such possibility exists. This aspect of trifluoroacetylation reactions is of particular importance for proficiency testing and off-site analysis. When spiked samples are spiked with volatile an-

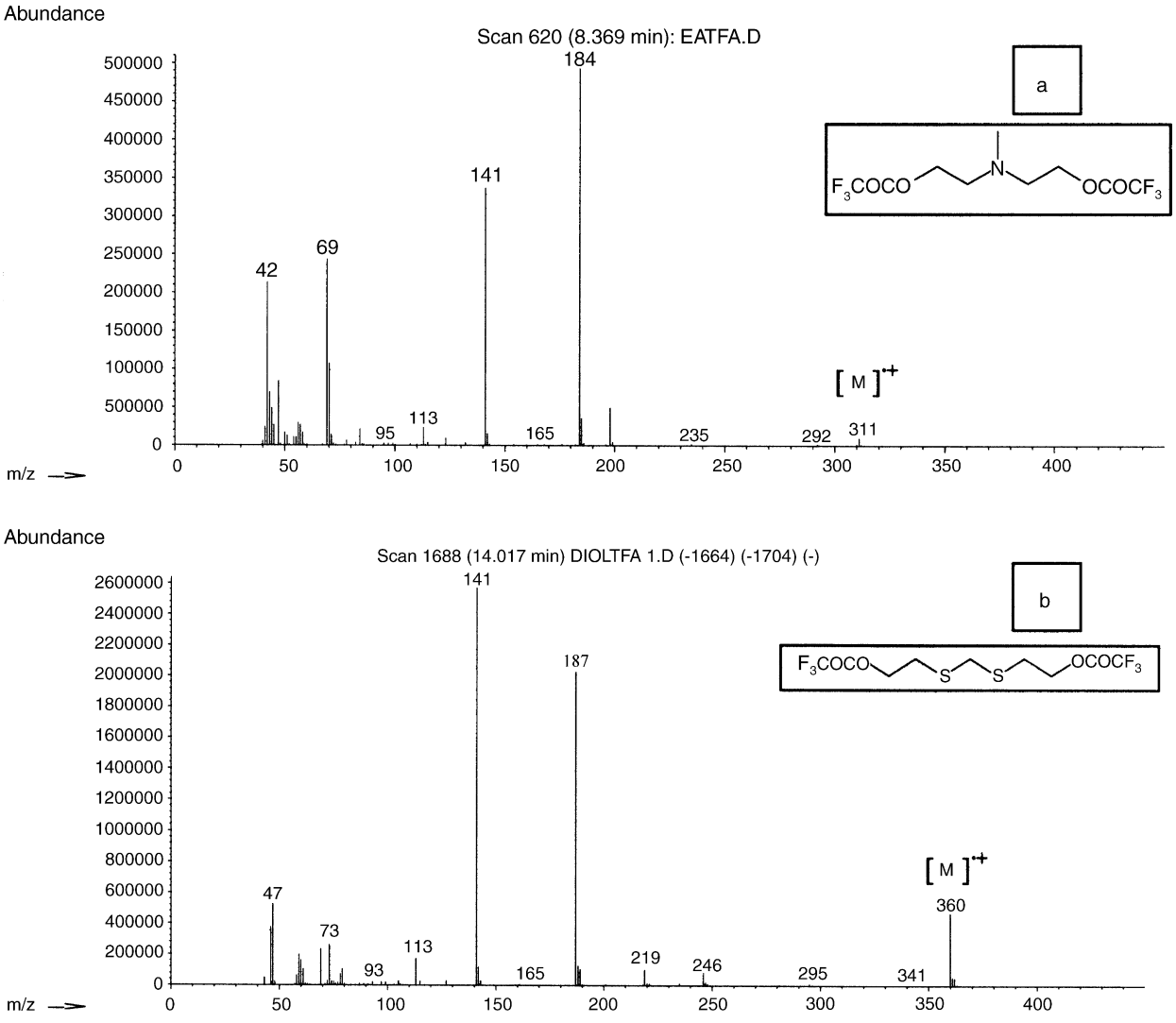

Fig. 7. EI-MS of TFA derivatives of (a) NMP1 and (b) SMP2.

alytes, trifluoroacetylation with the reported reaction conditions would be advantageous because the derivatization reaction added no background to TIC chromatogram.

3.2. Negative ion chemical ionization (NICI) analysis of TFA derivatives

Due to electrophilic nature, the TFA derivatives were expected to impart higher sensitivity in NICI mode; and indeed, it was found to be so. The NICI conditions were optimized with one example of NMPs and SMPs, to get the quasi molecular ion and higher detection limit. The optimized conditions were then subsequently applied to all the other TFA derivatives. Optimization was performed by observing pseudo molecular ion and signal intensity of 10 μg/mL solution of tris(trifluoroacetylethyl)amine and bis(trifluoroacetylethylthio)ethane. For this, methane was used as moderating gas and clues were taken from our earlier work [15]. The NICI mass spectral data of TFA derivatives are given in Table 1, which exhibited pseudo molecular ion $[M + CF_3COO]^-$ with 227 $[CF_3COOHOOCCF_3]^-$ and 113 $[CF_3COO]^-$ fragment ions.

The limit of detection (LOD) is an important parameter of any developed analytical technique. The LOD of TFA derivatives of NMPs and SMPs in EI with full scan mode was found to be 5 ng, which is comparable to their TMS derivatives. The LOD in EI under selected ion monitoring was found to be 500 pg having signal-to-noise ratio of 10:1. The limit of detection of these derivatives in NICI full scan mode could reach down to 100 pg/μL with signal-to-noise ratio of 10:1.

Since, ultimate goal of this investigation was to employ these derivatives and GC–MS analysis conditions during official proficiency tests conducted by OPCW, where spiking level of compounds is between 1 and 10 μg [7–11]. This method will prove valuable as it provided molecular weight information and characteristic spectra of TFA derivatives at concentrations of 0.1 and 0.5 μg, respectively, which is much below the spiking level.

3.3. Electron ionization mass spectra (EI-MS) of TFA derivatives of NMPs and SMPs

Electron ionization mass spectral data of TFA derivatives of NMPs and SMPs are given in Table 2. The EI-MS of representative TFA derivatives of NMPs and SMPs are depicted in Fig. 7. The genesis for the formation of various fragment ions are typically illustrated in Figs. 8 and 9. The fragmentation mode is illustrated by taking one example of each category. By same analogy the mass spectra of all other compounds can also be explained. Straight forward fragmentation routes can justify formation of most of the ions. Molecular ion was observed in all the TFA derivatives of SMPs except SMP7. The ions of *m/z* value 69 $([CF_3]^+)$ and 141 $([CF_3COOC_2H_4]^+)$ with high relative abundances were present in all the derivatives. Loss of trifluoroacetic acid from molecular ion itself (*M*-114) was characteristic fragmentation observed in all the

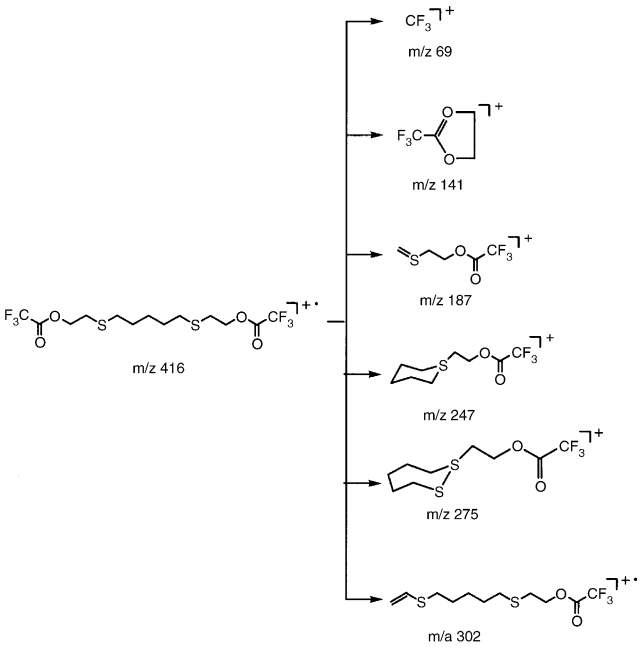

Fig. 8. Genesis of prominent fragment ions in EI-MS of TFA derivatives of SMP6.

derivatives. In addition, alpha-cleavage from sulfide linkages could lead to formation of other typical fragment ions as shown in Fig. 8. The simple fragmentation pattern and occurrence of molecular ion in EI-MS is of great diagnostic value for identification of these derivatives.

Similarly the EI-MS of TFA derivatives of NMPs can also be explained by simple fragmentation routes. Molecular ions were observed with weak relative abundances. Three prominent ions with *m/z* values of 42 $([CH_2{=}N{=}CH_2]^+)$, 69 $([CF_3]^+)$ and 141 $([CF_3COOC_2H_4]^+)$ were present in all the derivatives. The characteristic (beta-cleavage from molecu-

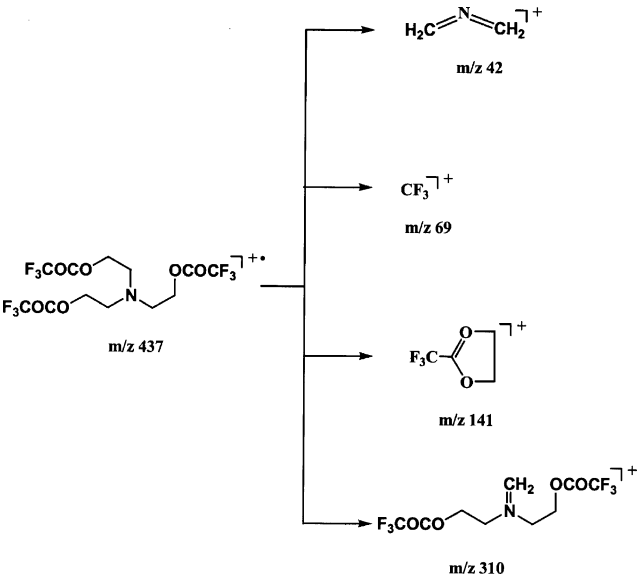

Fig. 9. Genesis of prominent fragment ions in EI-MS of TFA derivatives of NMP3.

lar ion produced $[M–CF_3OCOCH_2]^+$ ion with 100% relative abundance. Fragmentation routes of these derivatives are proposed in Fig. 9 taking tris(trifluoroacetoxyethyl) amine as typical example.

4. Conclusion

Precursors of nitrogen and sulfur mustards are important markers of these toxic chemical warfare agents, hence their detection and identification is of high importance from verification point of view of CWC. Derivatisation of polar analytes is a prerequisite for their GC–MS analysis, and being polar compounds these precursors also require derivatisation. Trifluoroacetylation of NMPs and SMPs was efficiently performed with reagents namely trifluoroacetylimidazole and trifluoroacetylbenzimidazole. Both the reagents efficiently derivatized these precursors. The TFABI is a new reagent tested by us that derivatized the NMPs and SMPs within the 5 min without heating. Derivatization conditions were optimized in such a way that afforded clean TICs for TFA derivatives. EI-MS of these derivatives were very simple and showed molecular ions. In NICI the pseudo molecular ions were observed with low limit of detection.

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