

CHROM. 18 061

## CAPILLARY COLUMN GAS CHROMATOGRAPHY-MASS SPECTROMETRY OF TABUN

P. A. D'AGOSTINO\*, A. S. HANSEN, P. A. LOCKWOOD and L. R. PROVOST

*Defence Research Establishment Suffield, Ralston, Alberta T0J 2N0 (Canada)*

(Received July 22nd, 1985)

---

### SUMMARY

A sample of munitions grade tabun, known to contain several impurities, was studied by combined capillary column gas chromatography-mass spectrometry under both electron-impact and chemical ionization conditions. Five impurities, three of which were previously unreported in tabun, were identified and characterized based on their mass spectral data. Ammonia chemical ionization was particularly useful since this technique provided molecular ion information for all the organophosphorous impurities.

---

### INTRODUCTION

The alleged use of the chemical warfare agent tabun in the Iran/Iraq conflict emphasizes the need for specific detection and identification methods for this and other nerve agents<sup>1-3</sup>. Recent gas chromatographic (GC) analysis of a munitions grade tabun sample in our laboratory indicated the presence of a number of impurities. The identity of these compounds, estimated to comprise about 25% of the organic content, would be valuable for future chemical event analyses.

A capillary column GC-mass spectroscopic (MS) study using both electron-impact (EI) and chemical ionization (CI) conditions was initiated with the principle objective being the identification of the tabun impurities. Initial EI investigation led to the positive identification of only one impurity, triethyl phosphate. As a result CI, using methane and ammonia as reagent gases, was employed in order to resolve the ambiguous EI data.

CI-MS using methane, isobutane, ethylene and methanol has been used recently for the analysis of organophosphorus chemical warfare agents<sup>4</sup> and pesticides<sup>5-7</sup>. Both the unique character of the CI data and the molecular ion information often afforded have been exploited for identification and characterization purposes.

Methane CI and in particular ammonia CI, an ionization technique previously unreported for chemical warfare agent determination and used sparingly in pesticide study<sup>5</sup>, provided the molecular ion and fragmentation information necessary for the identification of the five tabun impurities. The mass spectral data acquired, along with GC retention index data, are reported for tabun and the other organophosphorus compounds identified in the munitions grade sample.

## EXPERIMENTAL

*Standards*

All tabun samples were prepared in HPLC-grade chloroform (Fisher Scientific, Edmonton, Canada) and stored in PTFE-lined screw-capped glass vials prior to GC analysis. The munitions grade tabun, used for impurity identification, was taken from a sample which had been in a steel container for at least 30 years.

*Distillation of munitions grade tabun*

Munitions grade tabun was fractionally distilled in an all glass apparatus at 3 Torr using a Vigreux column (20 cm  $\times$  1.8 cm I.D.) and the fraction boiling at 97–97.5°C (3 Torr) was analysed by combined GC–MS.

*Tabun synthesis*

Tabun was prepared according to the method of Holmstedt<sup>8</sup> using benzene instead of chlorobenzene as the solvent. Crude tabun was purified by fractional distillation at 0.6 Torr using a vacuum-jacketted and silvered Hempel column (30 cm  $\times$  1.8 cm I.D.) filled with glass rings (5  $\times$  5 mm). The fraction boiling at 57°C (0.6 Torr) was collected as pure tabun and used for combined GC–MS analysis.

*Thermal decomposition of pure tabun*

Pure tabun (9 ml) was heated in glass at 200° for 20 min. After cooling a sample of the resulting dark material was used for capillary column GC–MS analysis.

*Ethyl isopropyl dimethylphosphoramidate synthesis*

Ethyl dimethylphosphoramidochloridate (8.24 g, 0.048 mole) was added to a solution of sodium isopropoxide (made from 1.2 g of sodium) in 2-propanol (75 ml). After the addition was complete the reaction mixture was heated for 30 min at 60°C. Excess 2-propanol was removed *in vacuo*, water (50 ml) was added and the aqueous layer was extracted with dichloromethane (3  $\times$  50 ml). The organic layers were combined, dried over anhydrous sodium sulphate, filtered and the excess solvent removed leaving a clear, colourless oil. Distillation afforded 7.8 g (83%) of product b.p. 56–57°C/0.2 mm.

<sup>1</sup>H NMR (C<sup>2</sup>HCl<sub>3</sub>).  $\delta$  1.3 (3H, t, CH<sub>3</sub>),  $\delta$  1.3 (6H, d,  $J = 12\text{H}_z$ , CH<sub>3</sub>),  $\delta$  2.6 (6H, d,  $J_{\text{PNCH}_3} = 20\text{H}_z$ ),  $\delta$  4.0 (2H, m,  $J = 14\text{H}_z$ , CH<sub>2</sub>),  $\delta$  4.6 (1H, m,  $J = 12\text{H}_z$ , CH).

<sup>13</sup>C NMR (C<sup>2</sup>HCl<sub>3</sub>).  $\delta$  15.43 (1C, d,  $J_{\text{POCC}} = 6.7\text{H}_z$ , CH<sub>3</sub>),  $\delta$  22.92 (2C, d,  $J_{\text{POCC}} = 2.7\text{H}_z$ , CH<sub>3</sub>),  $\delta$  35.84 (2C, d,  $J_{\text{PNC}} = 3.4\text{H}_z$ , CH<sub>3</sub>),  $\delta$  60.98 (1C, d,  $J_{\text{POC}} = 5.6\text{H}_z$ , CH<sub>2</sub>),  $\delta$  69.62 (1C, d,  $J_{\text{POC}} = 5.7\text{H}_z$ , CH).

IR (*thin film*). 1460, 1387, 1310, 1260, 1180, 1112, 1054, 1000, 960, 891, 784, 702 cm<sup>-1</sup>.

*Analysis*. Calculated: C<sub>7</sub>H<sub>18</sub>NO<sub>3</sub>P; C, 43.10%; H, 9.30%; N, 7.20%. Found: C, 43.10%; H, 9.20%; N, 6.95%.

*EI mass spectrum* [ $m/z$  (% relative intensity)]. 39 (4); 41 (7); 42 (13); 43 (11); 44 (100); 45 (13); 82 (2); 108 (18); 109 (2); 110 (4); 124 (15); 125 (4); 126 (4); 136 (3); 152 (3); 153 (8); 154 (3); 195 (3).

*Ethyl n-propyl dimethylphosphoroamidate synthesis*

Ethyl dimethylphosphoramidochloridate (15 g, 0.09 mole) was added to a solution of sodium propoxide (made from 2.3 g of sodium) in 1-propanol (100 ml). After the addition was complete the reaction mixture was heated for 30 min at 60°C. Excess 1-propanol was removed *in vacuo*, water (100 ml) was added and the aqueous layer was extracted with dichloromethane (3 × 75 ml). The organic layers were combined, dried over anhydrous sodium sulphate, filtered and the excess solvent removed leaving a clear, colourless oil. Distillation afforded 17 g (74%) of product b.p. 62–63°C/0.2 mm.

$^1\text{H NMR}$  ( $\text{C}^2\text{HCl}_3$ ).  $\delta$  0.95 (3H, t,  $J = 7\text{H}_z$ ,  $\text{CH}_3$ ),  $\delta$  1.30 (3H, t,  $J = 7\text{H}_z$ ,  $\text{CH}_3$ ),  $\delta$  1.64 (2H, m,  $J = 7\text{H}_z$ ,  $\text{CH}_2$ ),  $\delta$  2.70 (6H, d,  $J_{\text{PNC}} = 9\text{H}_z$ ,  $\text{CH}_3$ ),  $\delta$  3.73–4.30 (4H, m).

$^{13}\text{C NMR}$  ( $\text{C}^2\text{HCl}_3$ )  $\delta$  9.39 (1C, s,  $\text{CH}_3$ ),  $\delta$  15.48 (1C, d,  $J_{\text{POCC}} = 6.5\text{H}_z$ ),  $\delta$  23.09 (1C, d,  $J_{\text{POCC}} = 6.8\text{H}_z$ ,  $\text{CH}_2$ ),  $\delta$  35.97 (2C, d,  $J_{\text{PNC}} = 3.2\text{H}_z$ ,  $\text{CH}_3$ ),  $\delta$  61.39 (1C, d,  $J_{\text{POC}} = 5.6\text{H}_z$ ,  $\text{CH}_2$ ),  $\delta$  66.92 (1C, d,  $J_{\text{POC}} = 5.6\text{H}_z$ ,  $\text{CH}_2$ ).

*IR* (*thin film*). 1460, 1392, 1311, 1258, 1194, 1168, 1071, 1048, 1000, 963, 855, 787, 754, 700  $\text{cm}^{-1}$ .

*Analysis*. Calculated:  $\text{C}_7\text{H}_{18}\text{NO}_3\text{P}$ ; C, 43.10%; H, 9.30%; N, 7.20%. Found: C, 42.85%; H, 9.20%; N, 7.06%.

*EI mass spectrum* [ $m/z$  (% relative intensity)]. 39 (2); 41 (4); 42 (10); 43 (11); 44 (100); 45 (14); 82 (3); 83 (3); 108 (21); 109 (2); 110 (5); 111 (3); 124 (20); 125 (3); 126 (15); 136 (3); 152 (3); 153 (3); 154 (17); 195 (3).

*Instrumental analysis*

A Varian (Georgetown, Canada) Model 3700 gas chromatograph equipped with a flame ionization detector was used for all capillary column GC-flame ionization detection (FID) analyses. Data were recorded on a Varian 4270 integrator.

Capillary column GC-MS analyses were performed with a VG Micromass 70/70H double focusing mass spectrometer (VG Analytical, Wythenshawe, U.K.).

TABLE I

CAPILLARY COLUMN GC-MS OPERATING CONDITIONS

<i>Operating parameters</i>	<i>EI</i>	<i>Methane* CI</i>	<i>Ammonia** CI</i>
Accelerating voltage (kV)	4	4	4
Electron energy (eV)	70	70	70
Emission ( $\mu\text{A}$ )	200	1000	1000
Source temperature ( $^{\circ}\text{C}$ )	200	110	100
Source pressure (Torr)	$2 \cdot 10^{-6}$	ca. 0.1–0.5***	ca. 0.1–0.5***
Resolution	500	500	500
(10% valley definition)			
GC-MS interface	direct (230°C)	direct (230°C)	direct (230°C)
Scan function and rate	350 to 20 a.m.u., exponential down, 1 s/decade.		

\* Pure grade (99.5%) (Liquid Carbonic, Scarborough, Canada).

\*\* Anhydrous grade (99.99%) (Liquid Carbonic).

\*\*\* Pressure inside the source was estimated from a pressure reading of  $5 \cdot 10^{-5}$  Torr near the source, since the VG 70/70H does not read the pressure within the source.

The EI and CI operating conditions used during GC-MS study are listed in Table I. Details of CI optimization have been reported<sup>9</sup>.

GC analyses were performed on three J + W 15 m × 0.32 mm I.D. capillary columns (J + W Scientific, Rancho Cordova, CA, U.S.A.) coated with 0.25 μm DB-1 (100% dimethylpolysiloxane), DB-5 (95% methyl-(5%)-diphenylpolysiloxane) and DB-1701 (86% dimethyl-(14%)-cyanopropylphenylpolysiloxane) films. An on-column injector of our own design was used for sample introduction<sup>10</sup>. Injections were made at 50°C. This temperature was maintained for 2 min and followed by a 10°C/min temperature program to a maximum of 300°C. High-purity helium, at a linear velocity of 35 cm/s (methane injection at 50°C), was used as the carrier gas.

## RESULTS AND DISCUSSIONS

### GC-MS analysis

Fig. 1 illustrates the ammonia CI capillary column GC-MS chromatogram obtained for the munitions grade tabun sample. Similar total-ion-current chromatograms were obtained under EI and methane CI conditions. Tabun and five other organophosphorus compounds, listed in Table II, were identified in the sample based on mass spectral interpretation. Tabun, the major sample component, accounted for 76% of the organic content while diethyl dimethylphosphoramidate and triethyl phosphate contributed 12% and 1%, respectively.

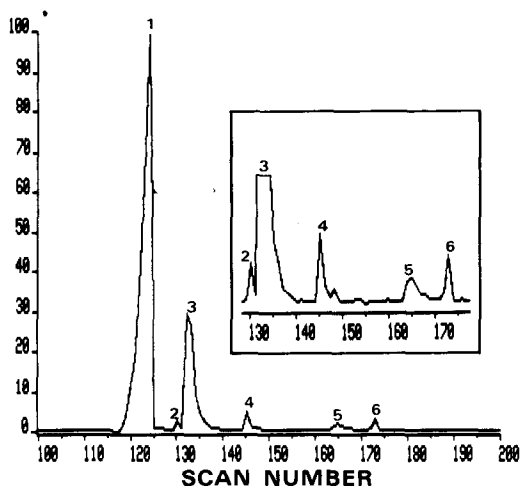


Fig. 1. Ammonia chemical ionization capillary column GC-MS chromatogram of munitions grade tabun. Compounds are identified in Table II (15 m × 0.32 mm I.D. J + W DB-5 column).

Similar percentage contributions were found in an alleged liquid chemical warfare sample analysed in Sweden<sup>2</sup>. Their analyses indicated the presence of tabun, diethyl dimethylphosphoramidate and triethyl phosphate at 75%, 3–10% and, 1–4% levels, respectively. Chlorobenzene, often used as a solvent during tabun synthesis, was also found. Two phosphorus-containing compounds remained unidentified.

Our application of CI-MS with both ammonia and methane as reagent gases enabled the identification of three previously unreported organophosphorus com-

## COMPOUNDS IDENTIFIED IN MUNITIONS GRADE TABUN

Compound No.	Compound identity*	Structure	Molecular weight	% of sample**	CA registry No.
1	Ethyl dimethylphosphoramidocyanidate (Tabun)	$\begin{array}{c} \text{O} \\ \parallel \\ \text{H}_5\text{C}_2\text{O}-\text{P}-\text{N}(\text{CH}_3)_2 \\   \\ \text{CN} \end{array}$	162	76	77-81-6
2	Triethyl phosphate	$\begin{array}{c} \text{O} \\ \parallel \\ \text{H}_5\text{C}_2\text{O}-\text{P}-\text{OC}_2\text{H}_5 \\   \\ \text{OC}_2\text{H}_5 \end{array}$	182	1	78-40-0
3	Diethyl dimethylphosphoramidate	$\begin{array}{c} \text{O} \\ \parallel \\ \text{H}_5\text{C}_2\text{O}-\text{P}-\text{N}(\text{CH}_3)_2 \\   \\ \text{OC}_2\text{H}_5 \end{array}$	181	12	2404-03-7
4	Ethyl isopropyl dimethylphosphoramidate	$\begin{array}{c} \text{O} \\ \parallel \\ \text{H}_5\text{C}_2\text{O}-\text{P}-\text{N}(\text{CH}_3)_2 \\   \\ \text{OCH}(\text{CH}_3)_2 \end{array}$	195	5	
5	Ethyl tetramethylphosphorodiamidate	$\begin{array}{c} \text{O} \\ \parallel \\ \text{H}_5\text{C}_2\text{O}-\text{P}-\text{N}(\text{CH}_3)_2 \\   \\ \text{N}(\text{CH}_3)_2 \end{array}$	180	5	2404-65-1
6	Tetramethylphosphorodiamidic cyanide	$\begin{array}{c} \text{O} \\ \parallel \\ (\text{CH}_3)_2\text{N}-\text{P}-\text{N}(\text{CH}_3)_2 \\   \\ \text{CN} \end{array}$	161	1	14445-60-4

\* Based on EI and CI mass spectral data.

\*\* Based on capillary column GC-FID chromatogram peak areas.

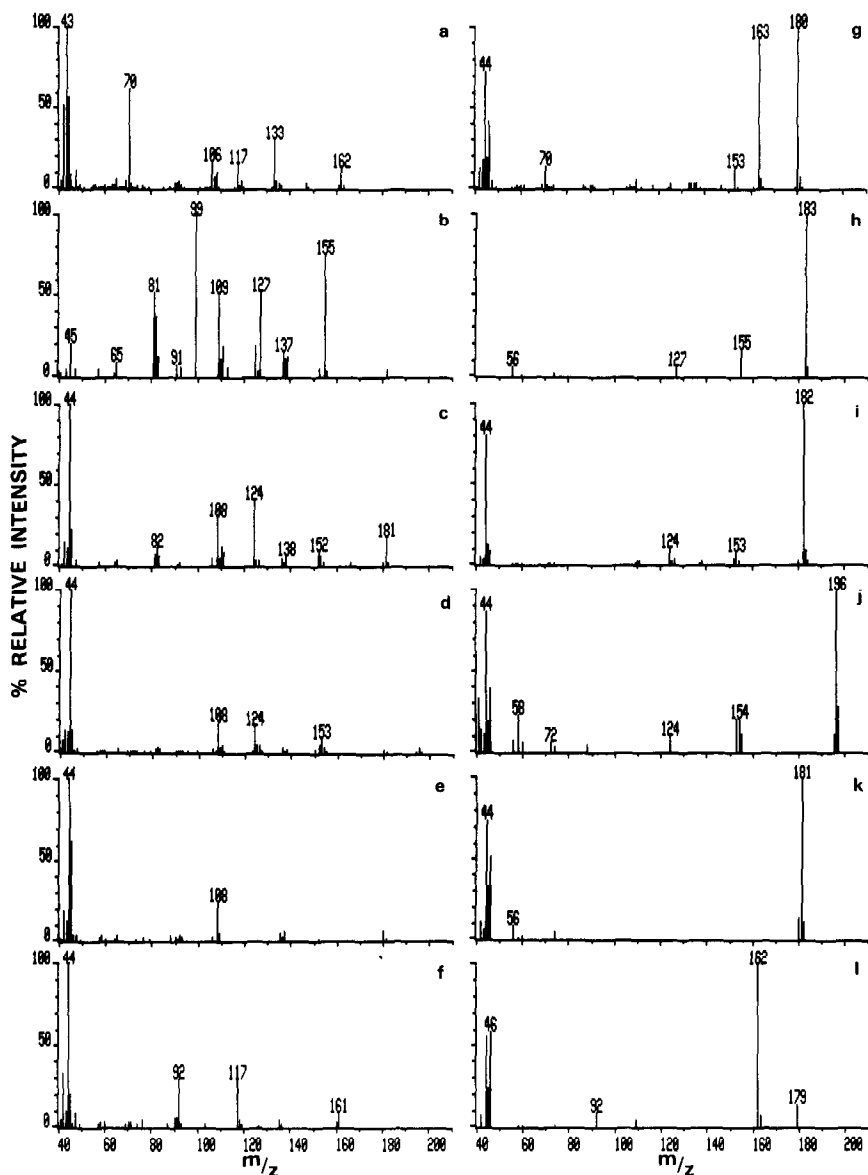


Fig. 2. (a) to (f) EI mass spectra of compounds 1 to 6 respectively. (g) to (l) Ammonia CI mass spectra of compounds 1 to 6, respectively. Compounds are identified in Table II.

pounds in munitions grade tabun. CI, a much softer ionization technique than EI, allowed us to positively identify the molecular ions of the tabun impurities. Pseudo-molecular ( $M + H$ )<sup>+</sup> ions were found for all components under both ammonia and methane CI conditions. In addition the ( $M + NH_4$ )<sup>+</sup> complex ion was found for tabun and another component under ammonia CI conditions. This molecular ion information was used with the acquired EI fragmentation data to identify the organophosphorus impurities.

Both the EI and ammonia CI mass spectra of the six organophosphorus compounds are shown in Fig. 2. The considerable fragmentation observed during EI analysis, due primarily to simple homolytic or heterolytic cleavage(s) and one or two hydrogen rearrangements (*e.g.*, McLafferty rearrangement), was not observed during ammonia CI operation.

Those compounds containing an ethoxy substituent were generally characterized by  $(M - C_2H_3)^+$ ,  $(M - C_2H_5)^+$  or  $(M - OC_2H_5)^+$  EI fragmentation ions. Multiple ethoxy substitution may lead to ions with the following possible ion structures:  $(M - C_4H_7)^+$ ,  $(M - C_4H_9)^+$ ,  $(M - OC_4H_9)^+$ ,  $(M - C_6H_{11})^+$  or  $(M - OC_6H_{13})^+$ . Ethyl isopropyl dimethylphosphoramidate produced fragmentation ions with possible structures  $(M - C_3H_6)^+$ ,  $(M - OC_3H_6)^+$ ,  $(M - C_5H_{11})^+$  and  $(M - OC_5H_{11})^+$  due to the presence of both an ethoxy and isopropoxy substituent.

Ethyl isopropyl dimethylphosphoramidate was tentatively identified based on similarities in EI fragmentation to diethyl dimethylphosphoramidate. Concern arose as to the presence of a isopropoxy substituted compound since the synthesis scheme specifies the use of ethanol. It was thought that the ethanol, used in the synthesis, could be contaminated with trace levels of isopropanol. The presence of this impurity could and probably did result in production of ethyl isopropyl dimethylphosphoramidate during synthesis. This compound and the *n*-propyl analogue, previously unreported in the literature, were synthesized and used to confirm the presence of ethyl isopropyl dimethylphosphoramidate in the munitions grade tabun.

The cyano and dimethylamino substituted compounds often produced  $(M - CN)^+$  and  $(M - C_2H_6N)^+$  EI fragmentation ions, respectively. Significant *m/z* 44 ions, observed for compounds containing the dimethylamino substituent, were probably due to  $(C_2H_6N)^+$  ions.

Fragmentation was more extensive under methane CI than ammonia CI for all the compounds studied. This was due to the more energetic ion-molecule reactions occurring under methane CI. The fragmentation ions were similar to those observed under EI operation with the exceptions being the presence of  $(M + H)^+$  and in some cases  $(M + C_2H_5)^+$  pseudo molecular ions. Table III summarizes the principle methane CI ions observed for the tabun components.

### GC retention indices

The GC retention indices of the compounds identified, relative to a homologous series of *n*-alkane standards under temperature programming conditions, were determined using capillary columns coated with DB-1, DB-5 and DB-1701 films according to a previously described method<sup>10</sup>. These data (Table IV), along with the mass spectra acquired should aid other researchers confronted with the analysis of samples alleged to contain tabun.

Retention indices were found to increase with column polarity. Small index changes ( $\Delta RI$ ) of 45 to 66 units were observed between the DB-1 and DB-5 stationary phases. However, the relative ordering of the six components remained the same. A different retention order and considerably larger  $\Delta RI$  values (142 to 270) were observed between the DB-5 and the more polar DB-1701 column. Most notable were the large  $\Delta RI$  values of 208 and 270 obtained for tabun and tetramethylphosphorodiamidic cyanide. It appears that the cyano functional group of the DB-1701 film exerted considerable influence on these, the only two compounds, with a cyano substituent.

*Distillation of the munitions grade tabun*

The munitions grade tabun was distilled and the fraction boiling at 97–97.5°C was collected in an unsuccessful attempt to obtain pure tabun. Tabun still accounted for about 75% of the GC–FID chromatogram obtained. Components 2 through 5 (refer to Table II) were present at 1%, 12%, 6% and 6%, respectively. The sixth component, tetramethylphosphorodiamidic cyanide, was not detected during capillary column GC–FID analysis.

TABLE III

METHANE CHEMICAL IONIZATION MASS SPECTRA OF COMPOUNDS IDENTIFIED IN MUNITIONS GRADE TABUN

<i>Compound name</i>	<i>Molecular weight</i>	<i>m/z</i>	<i>Relative intensity</i>
Ethyl dimethylphosphoramidocyanidate (tabun)	162	163	34
		136	100
		135	56
		108	61
		70	40
Triethyl phosphate	182	183	73
		155	100
		127	51
		113	24
Diethyl dimethylphosphoramidate	181	99	40
		210	4
		182	100
		154	40
		140	15
		126	20
Ethyl isopropyl dimethylphosphoramidate	195	124	20
		108	20
		196	10
		195	13
		182	11
		154	100
		153	26
		140	10
Ethyl tetramethylphosphorodiamidate	180	126	14
		124	13
		108	16
		209	5
		181	100
		180	44
		153	25
		137	23
Tetramethylphosphorodiamidic cyanide	161	135	21
		108	20
		162	63
		135	100
		117	11
		92	19



TABLE IV

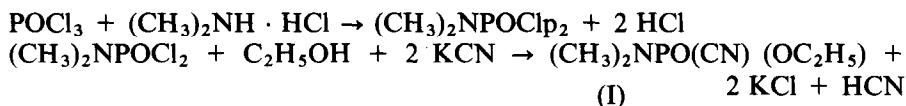
GC RETENTION INDICES FOR COMPOUNDS IDENTIFIED IN MUNITIONS GRADE TABUN ON DB-1, DB-5 AND DB-1701 FUSED-SILICA CAPILLARY COLUMNS

Compound name	Retention index*		
	DB-1	DB-5	DB-1701
Ethyl dimethylphosphoramido-cyanidate (tabun)	1077.9 ± 0.3	1131.6 ± 0.5	1339.8 ± 0.2
Triethyl phosphate	1090.5 ± 0.1	1137.2 ± 0.4	1308.5 ± 0.8
Diethyl dimethylphororamidate	1096.6 ± 0.6	1145.2 ± 0.1	1295.6 ± 0.2
Ethyl isopropyl dimethyl-phosphoramidate	1121.4 ± 0.4	1166.6 ± 0.5	1308.5 ± 0.8
Ethyl tetramethylphosphoro-diamidate	1158.7 ± 0.3	1216.6 ± 0.6	1392.8 ± 0.6
Tetramethylphosphorodiamidic cyanide	1179.5 ± 0.6	1245.7 ± 0.3	1514.7 ± 0.5

\* Retention indices expressed as a mean ± S.D. ( $n = 3$ ).

#### Tabun synthesis

In order to obtain pure material, tabun (compound I) was synthesized<sup>8</sup> according to the following scheme.



Tabun was isolated by fractional distillation and unlike the munitions grade material was found to constitute 100% of the organic content based on capillary column GC-FID analysis.

The impurities identified in the munitions grade tabun, probably resulted from the original large scale synthesis or from decomposition on storage. In order to investigate this possibility pure tabun was heated in glass at 200°C for 20 min. This material was cooled and examined by capillary column GC-MS. All the tabun impurities, except ethyl isopropyl dimethylphosphoramidate, thought to be due to the presence of isopropanol in the ethanol used during munitions synthesis, were observed.

Tabun accounted for 80% of the organic content in the GC-FID chromatogram and, compounds 2, 3, 5 and 6 contributed 0.4%, 5%, 0.2% and, 3%, respectively in the thermally treated sample. The remaining FID signal was due to two components of molecular weight 269 and 288. Pseudo molecular ( $M + H$ )<sup>+</sup> ions at  $m/z$  270 and 289, obtained during ammonia CI analysis, confirmed the molecular weights of these thermolysis products. These two compounds, as yet unidentified, are probably two sets of diastereomers. The EI and CI data were identical for both contributing GC peaks observed for each component. Pyrophosphate structures are being considered.

Based on the results described above it appears that a proportion of the components found in the munitions grade tabun resulted from disproportionation of the tabun either during synthesis or in storage.

## CONCLUSIONS

We have reported the application of ammonia CI for the detection and identification of organophosphorus chemical warfare agents. This technique provides excellent molecular ion information with minimal fragmentation and aided in the identification of five impurities, three of which were previously unreported, in munitions grade tabun.

The GC and mass spectral data provided are sufficient for the identification of tabun and its impurities. The relative proportions of tabun impurities may also provide analysts with information on origin or synthesis method when dealing with samples contaminated with this chemical warfare agent.

Tabun was found to disproportionate upon heating. All the impurities observed except ethyl isopropyl dimethylphosphoramidate can be accounted for by this mechanism. This compound, thought to be formed due to isopropanol impurities during origin synthesis of the munitions grade tabun, was synthesized along with the *n*-propyl analogue for confirmation purposes.

## REFERENCES

- 1 *Chem. Eng. News*, 62, No. 14 (1984) 4.
- 2 S. Budiansky, *Nature (London)*, 308 (1984) 483.
- 3 *Report of the Specialists appointed by the Secretary-General to Investigate Allegations by the Islamic Republic of Iran Concerning the Use of Chemical Weapons, Report S/16433*, United Nations, New York, March 26, 1984.
- 4 S. Sass and T. L. Fisher, *Org. Mass Spectrom.*, 14 (1979) 257-264.
- 5 R. L. Holmstead and J. E. Casida, *J. Assoc. Off. Anal. Chem.*, 57 (1974) 1050-1055.
- 6 H.-J. Stan, *Fresenius Z. Anal. Chem.*, 287 (1977) 104-111.
- 7 M. R. Bauer and C. G. Enke, *Proc. of the 32nd Annual Conference on Mass Spectrometry and Allied Topics, San Antonio, TX, May 27-June 1, 1984*, pp. 16-17.
- 8 B. Homlstedt, *Acta Phys. Scand.*, 25 (1951) 1-120.
- 9 P. A. D'Agostino and L. R. Provost, *33rd Annual Conference on Mass Spectrometry and Allied Topics, San Diego, CA, May 26-May 31, 1985*.
- 10 P. A. D'Agostino and L. R. Provost, *J. Chromatogr.*, 331 (1985) 47-54.