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An automated air sampling system comprising an
OPTIC GC injector II. Quantification of VX
vapour

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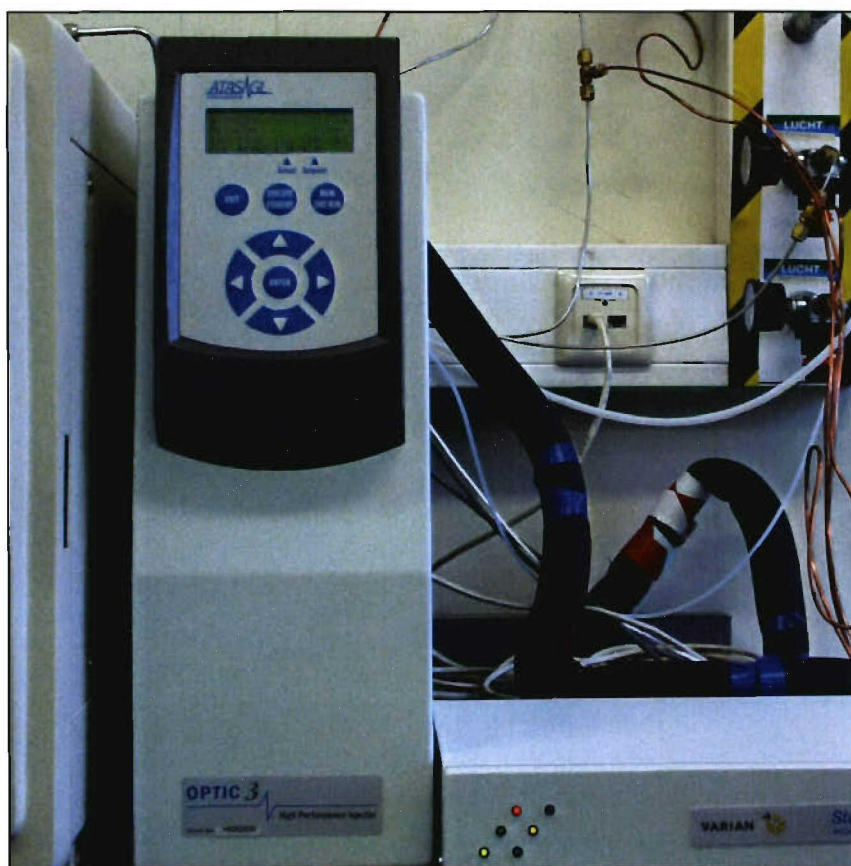
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Een geautomatiseerd luchtmonstername-systeem met een OPTIC gaschromatografie-injector II. Concentratiebepaling van uitstromende VX damp



Probleemstelling

In het kader van het project Detectieapparatuur chemische strijdmiddelen beschikt het werkgebied Detectie Chemische Strijdmiddelen sinds jaar en dag over een zogenaamde dampgeneratieopstelling. Met deze opstelling kunnen dampen met bekende concentratie worden gegenereerd van alle bekende strijdmiddelen. In het ideale geval is de concentratie van het strijdgas in het uitstromende gas te allen tijde bekend (in 'real time'). Hiertoe werd in het verleden

een Meloy-vlamfotometer ingezet die op fosfor danwel zwavel analyseert. In de loop van de tijd ontstond de behoefte om het uitstromende gas ook op samenstelling te analyseren zodat meer zekerheid bestaat over de aard van de verbinding en eventuele verontreinigingen c.q. afbraakproducten. Daartoe werd een gaschromatograaf met een ingebouwde thermodesorptie-injector aangeschaft. Na enkele jaren werd de vaste thermodesorptie-injector vervangen door een meer flexibel thermodesorptiesysteem dat onder meer bestaat uit een OPTIC GC-

injector. Deze optie biedt de mogelijkheid tot automatiseren van de verificatie van de dampconcentratie. Het vorige rapport in deze serie beschreef de bepaling van soman-damp met dit systeem. Dit rapport beschrijft de bepaling van VX damp met de OPTIC. VX is een veel lastiger te bepalen verbinding vanwege het hoge kookpunt en de kans op ontleding. Een goede kwantitatieve analyse van VX in de uitstromende lucht zal niet eenvoudig zijn.

Beschrijving van de werkzaamheden

Het OPTIC-systeem werd getest met het zenuwgas VX, dat qua gasgeneratie een relatief moeilijke stof is. Verschillende concentraties werden gemaakt zowel in droge als in zeer vochtige lucht. De resultaten werden vergeleken met een 'solvent desorption'-methode die de referentie is.

Resultaten en conclusies

Het OPTIC-systeem is in staat VX-damp te bepalen op een concentratieniveau van 0.1 mg/m³. Het duurt ongeveer 2 uur voordat het sampling-systeem in evenwicht is met de VX-damp.

Toepasbaarheid

Het systeem is toepasbaar voor het volgen van de concentratie VX-damp op een niveau van 0.1 mg/m³, hetgeen het zeer geschikt maakt voor het testen van detectoren.

**Een geautomatiseerd luchtmonsternamesysteem met
een OPTIC gaschromatografie-injector II.
Concentratiebepaling van uitstromende VX damp**

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1 Introduction

1.1 Automated thermodesorption of VX

In a previous report [1] in this series, the OPTIC GC injection system (further called OPTIC) was described. The injector itself as well as the air sampling and analysis system in which the OPTIC is implemented were described in detail. Following this technical description the performance of the system was demonstrated by the quantification of soman vapour.

The OPTIC proved to be very successful in the rapid quantification of soman vapour. The concentrations were in accordance with the values as determined with a solvent desorption method. Several conditions were examined like the humidity of the sampled air and the effect of the air flow rate through the Tenax tube during sampling. The OPTIC was also successfully employed for the quantification of sulphur mustard vapour. Even though the chromatographic conditions were different from that for soman, the thermodesorption conditions were the same. Sarin and tabun have not yet been investigated but it is expected that a similar experimental approach should give satisfactory results. This is still to be determined experimentally.

More problems are to be expected with VX, mainly because of its low volatility. For comparison: the saturated vapour pressure of VX at 20°C is only 0.0007 mm Hg, that of soman is 0.3 mm Hg, tabun is 0.07 and that of sarin is 2.9 mm Hg. This extremely low volatility and the specific properties of the P-S bond in VX make VX a 'sticky agent' that adsorbs on all kind of surfaces (see paragraph 1.2). Attempts at TNO Defence, Security and Safety to thermodesorb VX were not promising [2]. Amounts of 25 ng of VX in a solvent were transferred onto standard Tenax tubes, followed by removal of the solvent by flushing the tubes with nitrogen gas. These Tenax tubes were thermodesorbed at 220°C and the volatile products were trapped in a cold trap at -100°C. No or little VX was found. Next the cold trap was replaced by a piece of deactivated silica and the whole system was deactivated by injecting a silylating agent. Now an average recovery of 30% was obtained. This work was done within the framework of UN Chemical Weapons Convention in 1989. In 1990 a Canadian research institute reported their work on the thermodesorption of VX using Tenax minitubes [3]. They obtained best results with silanized tubes and silanized glass wool plugs. The peak area was more than doubled as compared to standard metal plugs. This supports the theory that VX decomposes on hot metal. Calibration curves were linear from 35 pg to 440 ng leading to the conclusion that decomposition and/or desorption were not significant and that the recovery was approximately 100% with these treated minitubes. An alternative approach to the detection of VX vapour is to convert VX to a volatile derivative which is then trapped on Tenax and easily desorbed. This approach is used by Robert B. Walton [4] in combination with the Hapsite mobile GC mass spectrometer. The VX is converted to a G analog by reaction with AgF. This is done on-line by inserting the AgF reaction tube in the sampling hose. Next the volatile analog is trapped on Tenax and subsequently thermodesorbed on the GC column. They were able to measure levels of 0.001 mg/m³ with a 20 minutes sampling time. With the Hapsite as well as with the abovementioned minitubes no cold trap is used. Instead the thermally desorbed VX is trapped on the top of the GC column and then released by temperature programming the column.

The OPTIC combines thermodesorption with automated sampling. This might introduce even more problems because VX can adsorb onto the walls of the sampling hose. In principle this should only be a matter of time before the walls of the tubing become in equilibrium with the vapour phase concentration of VX. However, desorption from the walls might occur when the concentration is lowered thereby overestimating the actual concentration (memory effect).

The OPTIC method will be compared with solvent extraction. This last method serves as a reference method because it has proven to give accurate results. Parameters as trap flow rate, trap time, vapour concentration and solvent volume are not critical.

A lot of parameters from the OPTIC system have to be examined like trap time, air humidity, and the response time of the OPTIC i.e. is the OPTIC able to follow the concentration of the vapour in reasonable time? In order to evaluate this last parameter a Chemical Agent Monitor (CAM) was used which serves here as a real time (continuous) monitor.

1.2 Properties of VX

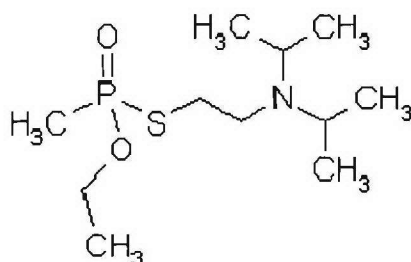


Figure 1 Molecular structure of VX.

VX has the chemical name methylphosphonic acid, *S*-[2-[bis(1-methylethyl)amino]ethyl]-*O*-ethyl ester and it is also known as *O*-Ethyl *S*-2-diisopropylaminoethyl methylphosphonothiolate (Figure.1). Molecular formula of VX is $C_{11}H_{26}NO_2PS$, with a formula weight of 267.37. Its Chemical Abstracts Service registry number is 50782-69-9. At room temperature it appears as a colourless to straw coloured liquid and odourless, similar in appearance to motor oil. It has a very low vapour pressure. The vapour is readily absorbed on most surfaces. Some physical data of VX has been listed in Table 1.

Table 1 Some physical data of VX [5].

Boiling point @ 760 mm Hg	298°C
Vapour pressure	0.0007 mm Hg @ 20°C
Vapour density (Air= 1 STP)	9.2 @ 25°C.
Volatility	9 mg/m ³ @ 25°C
Specific gravity (H ₂ O=1g/mL @ 25°C)	1.0113
Liquid density	1.0083 g/mL @ 25°C
Viscosity (Centistokes)	9.958 @ 25 °C
Solubility (g/100g solvent)	5.0 @ 21.5°C and 3.0 @ 25°C and ∞ in water < 9.5 °C Soluble in organic solvents
Freezing/Melting point (°C)	-50°C

VX has the unexpected property of being soluble in cold water but sparingly soluble in warm water ($> 9.5^{\circ}\text{C}$) [5]. VX is a lethal nerve agent. Among lethal CW agents, the nerve agents have had an entirely dominant role since the Second World War. They acquired their name because they affect the transmission of nerve impulses in the nervous system. All nerve agents belong chemically to the group of organophosphorous compounds. They are stable and easily dispersed, highly toxic and have rapid effects both when absorbed through the skin and via respiration. Nerve agents can be manufactured by means of fairly simple chemical techniques. The phosphorylthiocholine class of compounds has been discovered during the early 50 s. Thereafter, during the mid 50 s VX has been developed and stockpiled by the US (see Figure.1 for the molecular structure of VX).

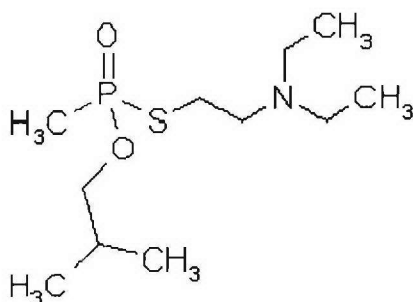


Figure 2 Molecular structure of V-gas.

The Soviet Union manufactured and stockpiled a closely related compound referred to as 'VR'. The molecular structure is depicted in Figure 2. The chemical name of V-gas is methylphosphonothionic acid, S-[2-(diethylamino)ethyl] O-2-methylpropylester. Its molecular formula is $\text{C}_{11}\text{H}_{26}\text{NO}_2\text{PS}$ and the CAS registry number is 159939-87-4.

Today, VX is still one of the most toxic chemical warfare agents (CWAs). VX is a lethal cholinesterase inhibitor. Doses which are potentially life-threatening may be only slightly larger than those producing least effects. Death usually occurs within 15 minutes after absorption of an effective fatal dosage. An effective inhaled dosage of less than 0.1 mg-min/m^3 results in a runny nose while less than 30 mg-min/m^3 might be enough for a fatality of 50%. These effective dosages for vapour are estimated for exposure durations of 2-10 minutes. The (in the US) permissible airborne exposure concentration for VX for an 8-hour workday of a 40 hour work week is an 8 hour time weighted average (TWA) of 0.00001 mg/m^3 (= Airborne Exposure Limit = AEL). In recent years this limit has been lowered several times.

Because of its high toxicity in combination with its low volatility and relatively high stability in the field (among the classical nerve agents such as sarin and soman) VX has the military potential to be used as a very persistent chemical warfare agent. *VX is much more persistent than G agents.* If VX is used in a military chemical attack it will be posing a (toxic) threat for a long period ranging from several hours up to several days. *The persistency of VX depends on the ammunition used and weather conditions. Heavily splashed liquid can persist for long periods under normal weather conditions. In very cold weather, VX can persist for months.*

Unstabilised VX of 95% purity decomposes at a rate of 5% a month at 71°C [5]. Carbodiimide stabilizers can be added to VX, especially when large scale production and/or long storage time is involved. Most routes of synthesis for the preparation of VX will lead to products with an original purity of 95-98% by weight. It has proven difficult - if not impossible - to obtain a much more purified VX ($>99\%$) as a product of

any more or less direct route of synthesis. Often, with other CWAs distillation is used as a means of purification. In the case of VX the results of distillation can be disappointing. Indeed, the final result may be of a lower purity due to decomposition reactions taking place during the distillation process. Typical impurities found in a VX product are summarised in Table 2.

Table 2 Common impurities of VX.

Compound
O,O-diethyl methylthiophosphonate
O,S-diethyl methylphosphonothionate
2-diisopropylaminoethyl hydrogen methylphoshinate
2-diisopropylaminoethyl ethyl methylphosphonate
O-ethylO-hydrogen methylphosphonothioate

Hydrolysis of VX is reported to be slowly in a neutral environment and the rate and composition of the hydrolysis products mixture is strongly depending on the acidity (see Table 2 for details). In alkaline circumstances the speed of hydrolysis rises sharply. Among the basic hydrolysis products is a compound known as EA 2192, diisopropylaminoethyl methylphosphonothionic acid, which is very long-lived and almost as toxic as VX itself. Its speed of hydrolysis can be about 1000 times more slowly than that of VX under comparable circumstances.

Thus hydrolysis-based decontamination schemes are not effective against VX.

Oxidation is the method of choice for VX decontamination. Common bleach (Na^+OCl^-) and superchlorinated bleach ($\text{Ca}^{+2}(\text{COCl}^-)_2$) react with VX at the *N* and *S* atoms. Once the *S* atom is oxidised, hydrolysis is very rapid. If a sufficient amount (and concentration) of bleach is used, all VX will be oxidised and no EA 2192 will be formed. For a better miscibility of the VX/decon mixture about 10% by weight of denaturated ethanol can be added. Because the ethanol can react with the hypochlorite, it has to be added after mixing of the VX with the decon solution.

Table 3 Important hydrolysis products of VX.

Compound	MW
EA2192, diisopropylaminoethyl methylphosphonothionic acid	239.32
Methylphosphonic acid, ethyl ester	124.08
Ethanol	46.07
Diisopropylaminoethanethiol	161.31
Methylphosphonothionic acid, ethyl ester	140.14
Diisopropylaminoethanol	145.25
Methylphosphonic acid	96.02

In addition, the carbodiimide stabilizers added to VX produce the following compounds under hydrolytic circumstances: N,N-diisopropylurea and N,N-dicyclohexylurea.

The incompatibility of VX to brass, steel and aluminium is reported to be negligible. On the other hand previous experience at PML indicates that bare metal surfaces like i.e. untreated stainless steel do indeed catalyse the decomposition (through oxidation or otherwise) of VX. VX also has a high absorptive coefficient towards various materials. The vapour readily adsorbs on bare metals, glass and most plastic material. Of course this process will speed up rapidly when the temperature of the metal is well above room temperature.

VX and other compounds having the phosphonothiolate structure $R-S-P(=O)(R)(OR)$ are known to undergo reversible photoisomerisation to the corresponding phosphonothionates $R-O-P(=S)(R)(OR)$. Other than this there are no reports about any important photochemical processes in sunlight.

This process of photoisomerisation can be driven thermally. Other than that there are no important thermal degradation processes according to some literature. Previous experience at PML on the other hand indicates a thermally driven degradation even in the presence of dry nitrogen at temperatures above 40-60°C.

Table 4 Overview of some decomposition products of V-agents.

Compound (name)
Hydrogen S-2-diisopropylaminoethyl methylphosphonothionate
Ethyl hydrogen methylphosphate
Diethyl methylphosphonate
Diethyl dimethylphosphonate
S,S-bis(2-diethylaminoethyl) methylphosphonodithionate
S,S-bis(2-diisopropylaminoethyl) methylphosphonodithionate
2-diethylaminoethanethiol
Bis(2-diethylaminoethyl)sulphide
Bis(2-diethylaminoethyl)disulphide
2-diisopropylaminoethanethiol
Bis(2-diisopropylaminoethyl)sulphide
Bis(2-diisopropylaminoethyl)disulphide

By now, it can be concluded that during the VX vapour generation and/or VX analysis one has to be aware of the presence or formation of one of the described impurities or decomposition and hydrolysis products of VX. Knowing their specific chemical characteristics, these products can affect, interfere or obstruct a correct sampling, monitoring and analysis of the VX product. They are always present in the starting material, but additional amounts or other products can just as easily be formed while generating VX vapours or in the process of analysing standard solutions of VX. Finally, it seems a good approach that every result has to be distrusted and not taken for granted too easily. This may help to ensure the right conclusions will be derived from the experimental results.

2 Experimental

2.1 Generation of VX vapour

The gas/vapour generation system is described in detail in the previous report [1]. The 'Fortuin' primary generation vessel was equipped with a large evaporation tube containing 2 ml of liquid VX. The generation system was configured as a one stage dilution set up. The flow rates were controlled with mass flow controllers (Bronhorst High Tec). The generation was started with the following flow rates:

- Primary nitrogen flow over liquid VX in the generation vessel was 100 ml/min. It was diluted in 7.5 l/min dry air.
- The sampling flow rate was 120 ml/min.
- The sample inlet line was a 1.5 meter piece of PEEK tubing with an internal diameter of 0.5 mm. The inlet end of this tubing was allowed to sample from the exhaust of the generation system with the above mentioned flow rate of 120 ml/min.

2.2 Calibration of Flame Ionisation Detector (FID)

The calibration was done by injection of solutions of VX in hexane in the OPTIC injector using the same column as in the thermodesorption conditions. The tenax tube however was replaced by a glass liner. This simple action and a small adaptation in the OPTIC temperature program changes the OPTIC from a thermodesorption unit in a normal temperature programmable solvent injector.

A stock solution of 20 mg VX in 50 ml hexane was made (400 ng/ μ l). Several dilutions in hexane were made in the concentration range from 10 to 400 ng/ μ l. A volume of 1 μ l was injected with a Hamilton 1 μ l plunger in needle syringe. At least three injections were done at each concentration.

*GC conditions: Injector temperature: 30 to 200 °C/min, 16°C/second.
Column: 8 m CPsil5CB, Df=1 μ m. Flow = 4 ml/min.
Oven: 0.5 min 80°C, 20 °C/min to 250°C.*

2.3 OPTIC program for thermodesorption experiments

The parameter programming for the thermodesorption experiments is depicted in Figure 1. At t=0 the sampling starts by switching the valve (brown curve). During this period the air from the generation system is sucked through the Tenax tube in the OPTIC injector. The injector temperature is 40°C during sampling which is low enough to trap the non-volatile VX. The column flow is blocked during sampling and the split flow or purge flow is also blocked. After 2 minutes the sampling is stopped by switching the six port valve back to injection position. The column flow and split flow are now flowing through the Tenax tube while this tube is still held at 40°C. The split flow serves to replace the humid air in the void volume of the Tenax tube by clean dry helium. After 30 seconds of purging the split flow is switched off and the thermodesorption starts by heating the injector to 250°C with a heating rate of 16°C/s. The trapped VX is transferred to the GC column which is held at 80°C. VX is focussed at the top of the column at this low temperature. Next the column is programmed to 250°C.

*GC conditions: Injector temperature: 40 to 200°C/min, 16°C/second.
 Column: 8 m CPsil5CB, Df=1 µm. Flow = 4 ml/min.
 Oven: 2.5 min 80°C, 30°C/min to 250°C.*

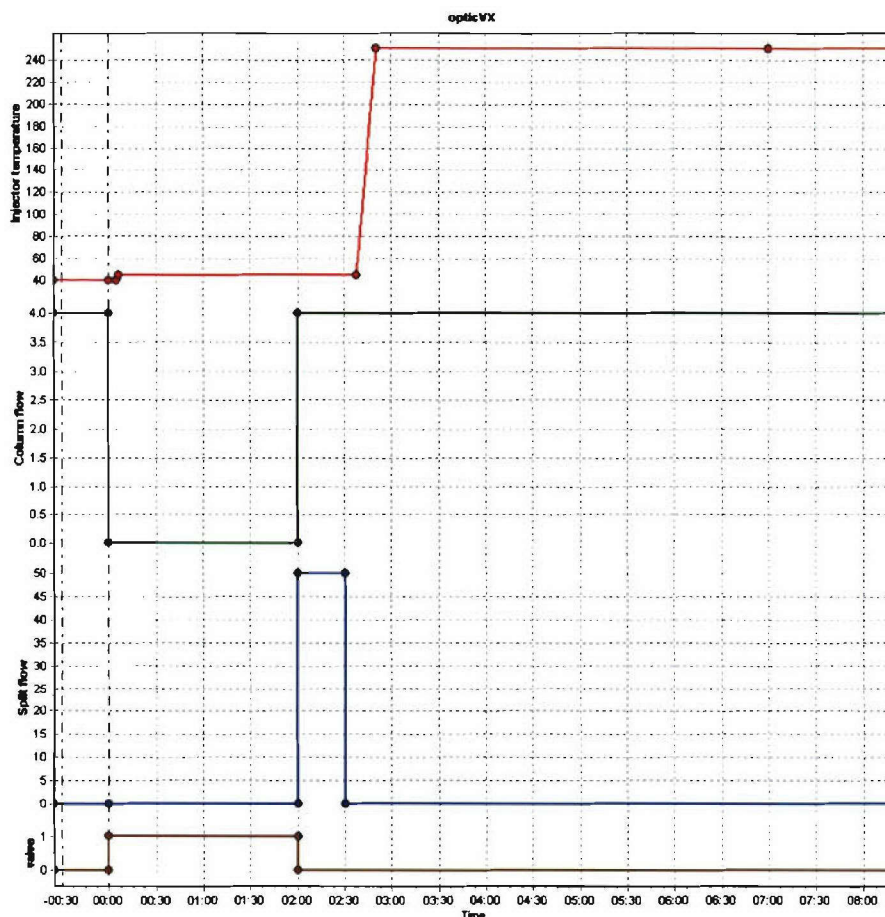


Figure 3 Optic program for thermodesorption GC experiments with VX. Parameters are injector temperature (red curve), column flow (green curve), split flow (blue curve) and position of the sampling valve (brown curve).

2.4 Effect of traptime

The trap time was adjusted by programming the six port valve (Figure. 1). As a consequence the other parameters in figure 1 had to be reprogrammed as well. The GC column oven had to be adjusted as well. So, if the trap time changed from 2 to 4 minutes all parameters had to be lengthened by 2 minutes.

The trap times examined were 2 minutes (standard program), 4 minutes and 6 minutes.

2.5 Effect of humidity

The humidity of the main air stream from the vapour generation system was changed from about 50% to dry (< 1% RH) by doubling the dry air stream and closing the 100% humidified air stream. In this way the total air flow is kept constant keeping the VX concentration unchanged.

A Chemical Agent Monitor (CAM) from Smiths Detection UK was employed to monitor the VX concentration in real time. The CAM was allowed to sample from the same vapour exit as the OPTIC. CAM spectra were recorded with the CAM evaluation software and the net peak height of the peak at 10400 μ s was used as CAM output signal.

2.6 OPTIC Response time

This experiment was performed to test the OPTIC's capability to follow a change in concentration. At a certain timepoint the concentration was raised by doubling the flow in the vapour generation vessel. The vapour was again monitored with the CAM which was allowed to sample from the same vapour as the OPTIC. Again CAM spectra were recorded with the CAM evaluation software and the net peak height of the peak at 10400 μ s was used as CAM output signal.

2.7 OPTIC Response time with heated transfer line

For this purpose a 2 meter piece of ultimet tubing (Varian methyl deactivated metal tubing) of 0.76 mm internal diameter. This tubing is made for transfer lines and retention gaps. A temperature control unit was made by the electronic construction department of our laboratory. The ultimet tubing was equipped with heating wire and connected to a specially adapted low voltage control unit which powers the heating wire. A thermocouple was also integrated and the temperature was fed back to the control unit. The control unit was equipped with a West thermocontroller. In order to get a fast and accurate temperature control of the transferline, the controller was set and left in autotune mode. The temperature was set at 60°C and after a few cycles the temperature was raised to 60°C without overshoot and kept stable without any problem. First VX was sampled with the non heated PEEK tubing and two days later the PEEK tubing was replaced by the heated transfer line. The settings from the generation system were the same for both experiments. The concentration VX was assumed to be unchanged as was also confirmed by the CAM. In both experiments 3 bar in G mode was measured.

2.8 Comparison with solvent extraction

Chrompack glass adsorption tubes filled with about 100 mg of *Tenax* TA 20-35 mesh was used. A *Bronkhorst* mass flow controller connected to the vacuum line at the outlet side and to the *Tenax* tube at the inlet side controlled the sampling flow rate. The flow rate was adjusted to 1 l/min. The inlet side of the *Tenax* tube was held in the exhaust of the vapour generation system for a period of 144 minutes.

After sampling the tube was detached from the mass flow controller and adsorbed vapour was desorbed with hexane (*Lichrosolv* from *Merck*, Germany). The effluent was collected in a 4 ml screw neck glass vial. The volume of collected hexane effluent was determined by weight (density of hexane is 0.66 g/ml). Two fractions of 1 ml are usually collected. It is learned by experience that almost all recovered component is collected in fraction 1.

1 μ l of the hexane solution was injected in a Chrompack CP9001 gas chromatograph equipped with a Flame Photometric Detector (FPD). This detector was calibrated with the same VX solutions as was used for the calibration of the FID (par. 2.2).

3 Results

3.1 Calibration of Flame Ionisation Detector (FID)

The peak shape was not as good as compared to the peak shape obtained with thermodesorption of VX from Tenax. A lot of solvent has to be eluted from a short column. This leads to larger uncertainty in the sensitivity which amounts to 1000 +/- 200 counts/ng.

3.2 OPTIC program for thermodesorption experiments

In Figure 4, a chromatogram is presented as obtained from the thermodesorption of approximately 16 ng of VX sampled from 200 ml dry (< 1% RH) air. The chromatogram shows an excellent sharp peak at 5.1 min. Obviously the sample is well focussed on the top of the GC-column which eliminates the need for a cryogenic focussing unit. Such a unit might even be the source of errors in case of VX.

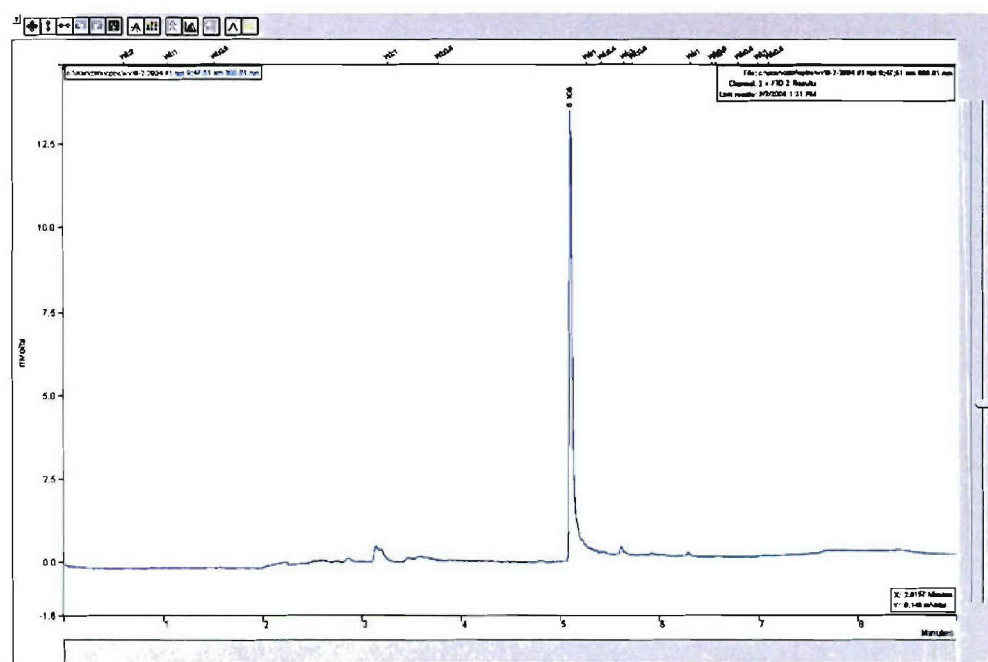


Figure 4 Typical chromatogram from the thermodesorption of VX. The peak represents 16 ng of trapped VX.

3.3 Effect of trap time

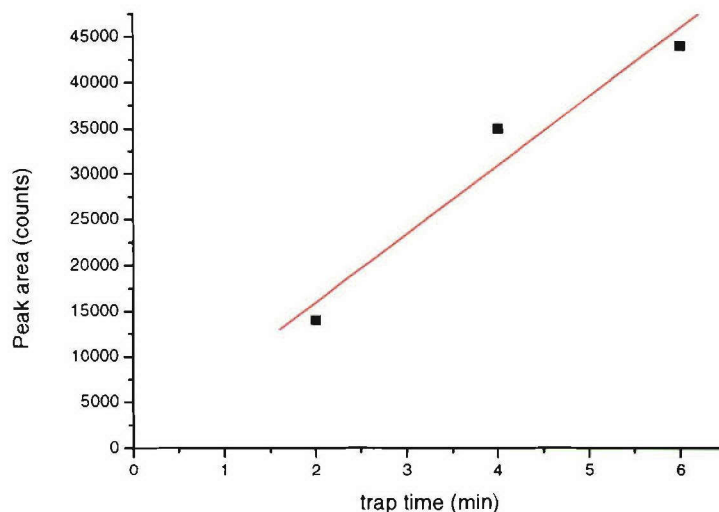


Figure 5 Area of VX peak as a function of traptime.

Figure 5 shows a linear relationship between amount of recovered VX and traptime. Although only 3 datapoints are available one can say that no strange effects are present which make the trap time critical. Most important is that the fitted line, if extrapolated, crosses the Y-axis at only 1000 counts, near the origin. This means that no big errors are made using short trap times.

3.4 Effect of humidity

The black curve shows that the OPTIC needs 1.5 to 2 hours to reach a plateau value. When the humidity is changed the OPTIC as well as the CAM show a steep response. This is caused by the abrupt closing of the humid air and doubling the dry air which disturbs the air flows in the vapour generation system. The OPTIC then returns to its original value after about 1.5 hour. The CAM also returns to its original value. It can be concluded that the OPTIC is not sensitive to humidity in this range. It would be interesting to test at high humidity (90%).

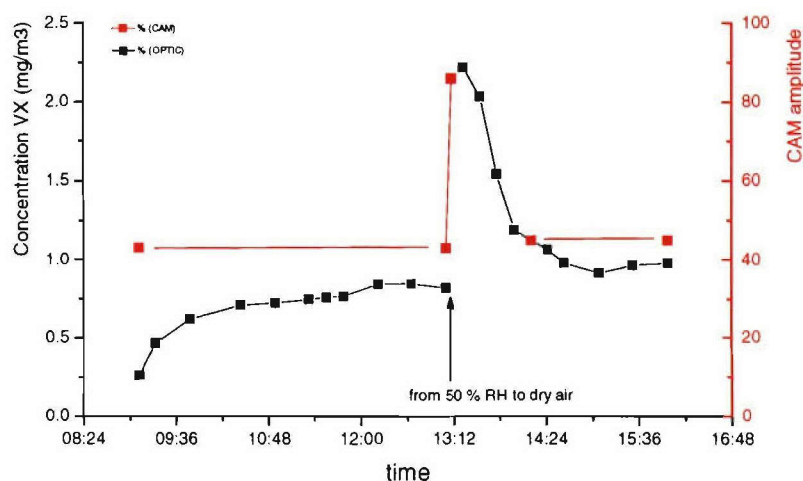


Figure 6 Response of OPTIC and CAM when air humidity changes from $\approx 50\%$ to $< 1\%$. At the timepoint indicated by the arrow the humid air is disconnected.

3.5 OPTIC response time

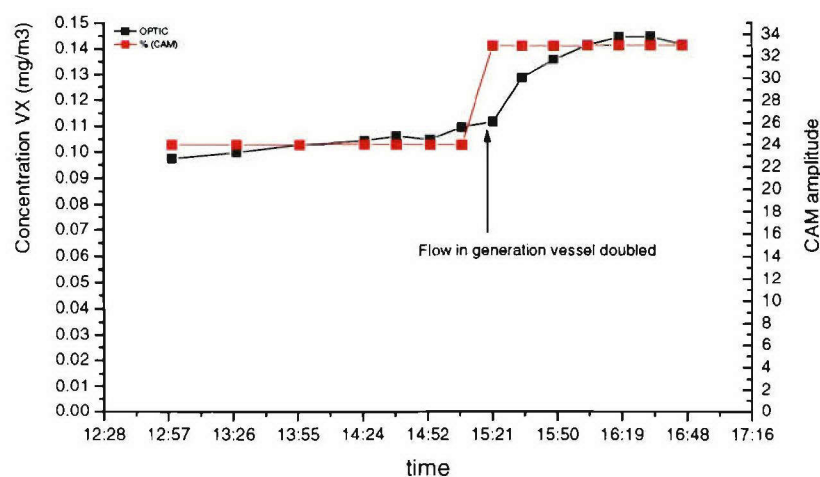


Figure 7 Response of OPTIC and CAM when doubling the flow in the generation vessel.

The flow in the generation vessel, also called the primary air flow, was doubled at the point indicated by the arrow in Figure 7. The CAM being a real time monitor immediately shows a sharp increase in peak height. The OPTIC follows within three cycles (sampling, GC analysis and cooling of GC and OPTIC) which takes about 40 minutes. According to the OPTIC, the concentration of VX was raised from 0.10 to 0.15 mg/m³ which means that the vapour output from the 'Fortuin' vessel was not limited by diffusion. In that case the increase in primary air flow would have little or no effect.

3.6 OPTIC Response time with heated transfer line

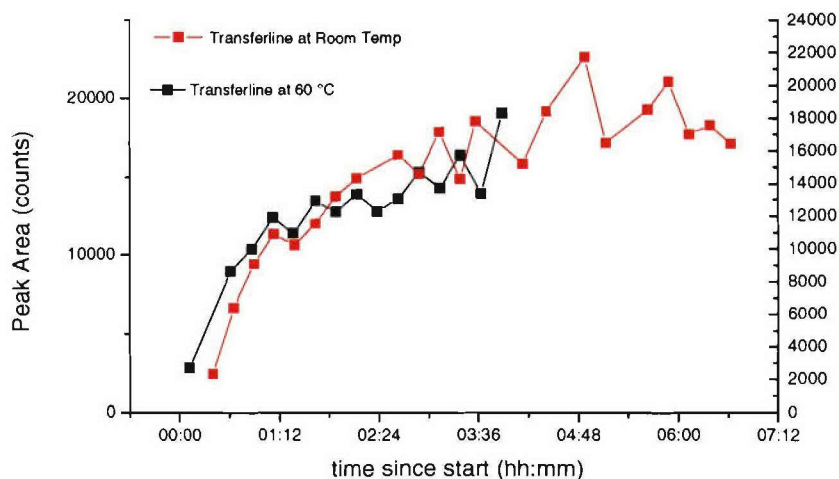


Figure 8 Reponse of optic equipped with ultimet tubing, either heated (black curve) or at room tempature (red curve).

As is clear from Figure 8 there is no advantage from the use of a heated transfer line at 60 °C. A higher temperature might help. In general one can say that it takes at least two hours to get a stable output from the OPTIC after switching from blank air to VX in air with the same humidity. Best practical solution might be a short transferline.

3.7 Comparison with solvent extraction

Solvent extraction:

Air sampled: 144 minutes x 1 l/min = 144 liter.
 Air was sampled on 1-9-04 from 12:00 h to 14:24 h.
 Volume of hexane effluent was 1.07 ml.
 Calibartion factor = 1700 counts/ng VX.
 Average peak area = 30000 counts.

Concentration VX in hexane effluent = $(30000/1700) = 17.6 \text{ ng/}\mu\text{l} = 17.6 \mu\text{g/ml}$.

Vapour Concentration VX = $(17.6 \mu\text{g/ml} \times 1.07 \text{ ml}) / 144 \text{ l} = 0.13 \mu\text{g/l} = 0.13 \text{ mg/m}^3$.

OPTIC automated thermodesorption :

Average of samples taken on 1-9-04 : 17962 counts +/- 11%.
 Measured trap flow was 90 ml/min. Trap time was 2 minutes.

Vapour concentration VX: $(17962/1000)/180 = 0.10 \text{ mg/m}^3$.

Considering the errors that can be made in both methods the results are in agreement.

4 Conclusions and Discussion

The OPTIC automated sampling system is able to analyse VX vapour in the exhaust air from the vapour generation system. The conformity with the solvent desorption method is quite good.

Sampling via a piece of tubing takes about 2 hours to reach a stable value of VX peak area either with PEEK tubing or with a heated (60 °C) methyl deactivated tubing. No influence of air humidity until 50 % RH was detected. Higher air humidities might introduce problems.

All experiments were performed at a concentration level of 0.1 mg/m³ VX. Lower concentrations as well as higher air humidities remain to be investigated.

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6 Signature

Rijswijk, December 2005

TNO Defence, Security and Safety

A handwritten signature in black ink, appearing to be 'M.S. Nieuwenhuizen', written in a cursive style.

Dr M.S. Nieuwenhuizen, MSc
Head of Department

A handwritten signature in blue ink, appearing to be 'J.L.N. Harteveld', written in a cursive style.

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