

FFI RAPPORT

IDENTIFICATION AND QUANTIFICATION BY GC-MS OF SULPHUR MUSTARD AND RELATED COMPOUNDS AFTER LONG TIME STORAGE IN SEA WATER

OPSTAD Aase Mari, TØRNES John Aa

FFI/RAPPORT-2002/03237

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8) ABSTRACT Sulphur mustard in sea water undergoes decomposition to different products over time. After two weeks mustard gas is not detected in the sea water nor in the sediment. Ten decomposition products were identified in sea water and six in sea sediment over a period of six years. The formation of the different decomposition products varies at different time. Thiodiglycol is detected in the samples from day one to six years though with highest concentration between one week and one year. Some hydrolysis products arise after three weeks, like bis(2-chloroethyl) sulphone and 2-chloroethyl vinyl sulphone in sea water. Other arise after eight months like 1,4-dithiane-1,1-dioxide and 1,4-dithiane-1-oxide. The last compound has even increased from 8 months to six years. Most of the other hydrolysis products have decreased during the six years the experiments have been carrying out.		
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IDENTIFICATION AND QUANTIFICATION BY GC-MS OF SULPHUR MUSTARD AND RELATED COMPOUNDS AFTER LONG TIME STORAGE IN SEA WATER

1 INTRODUCTION

After the Second World War, many ships with chemical ammunition were dumped in the Skagerrak, the North Sea and the Atlantic Ocean. The number of shipwrecks is uncertain, but up to 38 ships with more than 250 000 tonnes (gross weight) chemical ammunition may have been dumped. Most of the ammunition was loaded with mustard gas, but also some ammunition with nerve agent (tabun) and arsine compounds (e.g. lewisite, clark I/II) may have been dumped (1). The dumped chemical ammunition in the Baltic Sea (2) has caused a lot of problems for fishermen when mustard gas ammunition have been caught in their fishing nets. Mustard gas attacks the surface of the body followed by severe blisters and contaminates the fishing boat. Mustard gas is also carcinogenic. Polar hydrolysis products from mustard gas are relatively non-toxic and are not considering posing a risk to environment when mustard gas is dumped at sea.

In 1989 FFI was requested by the Ministry of Defence to investigate whether the chemical ammunition in the Skagerrak represented any danger (wrecks at a depth of 600 - 700 m). Many water samples were collected near five of the localised wrecks. The samples were analysed at FFI and the analysis for both mustard gas and tabun were negative. From the video of the wrecks, it was observed that some of the bombs were corroded and some also open to the sea.

FFI recommended that the investigation should be repeated after ten years. In 2002 (3) FFI did a new investigation. Samples both from sea water and sediment were collected nearby some of the wrecks. The analysis of sea water were negative, but some of the sediment samples contained a small amount of mustard gas, hydrolysis products of mustard gas, 2-chloro acetophenon and organoarsenic compounds like clark I, triphenyl arsine and bis(diphenylarsine) oxide. There is no indication of acute toxic effects from the chemical warfare agents in Skagerrak.

Little is known about the behaviour of mustard gas in ammunition dumped at sea other than the fact that it can remain on the seabed for a very long time. It was therefore decided to set up a laboratory experiment that simulates a situation where mustard gas has been dumped in sea water and droplets of the chemical have been spread out on the seabed. The concentration of mustard gas in sea water and sediment was monitored over time. The aim was also to identify and monitor the major decomposition products over time.



Figure 1.1 Dumping areas for chemical ammunition in Scandinavian waters

2 EXPERIMENTAL

2.1 Chemicals

Sea water (pH = 7, salinity = 3 %) from the Kara sea

Mustard gas (HD), CAS no 505-60-2, synthesised at FFI

Thiodiglycol (TDG), CAS no 111-48-8 from Koch-Light

Bis(2-chloroethyl) sulphone, CAS no 471-03-4, synthesised at FFI

Bis(2-chloroethyl) sulphoxide, CAS no 5819-08-9, synthesised at FFI

Tridecane (C13), CAS no 629-50-5, puriss from Koch-Light

Dichloromethane, CAS no 75-09-2, ultra gradient from J T Baker (+uvasol from Merck)

Ammonia reagent gas, grade 4.8 from AGA

Isobutane reagent gas, grade 3.5 from AGA

2.2 Instrumentation

In these experiments we used a Finnigan MAT 95Q hybrid mass spectrometer coupled to a HP 5890 Series II gas chromatograph equipped with a direct inlet (DI) probe and a Fision MD800 mass spectrometer coupled to a Fision 8060 gas chromatograph. The column used for both instruments was 30 m x 0.25 mm with 0.25 μ m DB-5 MS stationary phase from J&W Inc.

The oven programme was 40°C (1 min) – 10 °C/min – 280 °C (10 min) and He (grade 6.0) was used as carrier gas. Direct inlet probe was programmed from 25 °C to 250 °C with ramp 30 °C/min. Other instrumental parameters used are shown in Table 2.1.

Parameter	GC: HP 5890 II MS: Finnigan MAT95Q BEQQ		GC: Fisons GC 8060 MS: Fisons MD800/250	
	EI	CI	EI	CI
Injector (°C)	250	250	220	220
Transfer line (°C)	280	280	260	260
Ion Source temp (°C)	190	150	190	150
Electron energy (eV)	70	150	70	70
Filament current (mA)	1.0	0.2	0.15	0.30
Scan range (amu)	25 - 400	100 - 500	35 - 600	100 - 500
Resolution	1000	1000	unit	unit
Reaction gas		Ammonia or isobutane		Ammonia or isobutane

Table 2.1 Instrumental parameters used for MS analysis

2.3 Sample preparation

To simulate the seabed, samples of 30 g dried soil with high clay content were placed in 100 ml glass bottles with screw caps. 30 ml sea water from Kara sea (pH = 7, salinity 3 %) was poured carefully over the sediment. Two droplets containing 6 mg mustard gas each were carefully placed on top of the sediment by using a 5 µl Hamilton syringe. Control samples without mustard were also prepared. The glass bottles were closed and the stored dark at 25 °C. Several parallels were made and stored for different of time before sample preparation. Figure 2.1 shows a sample stored for six years.

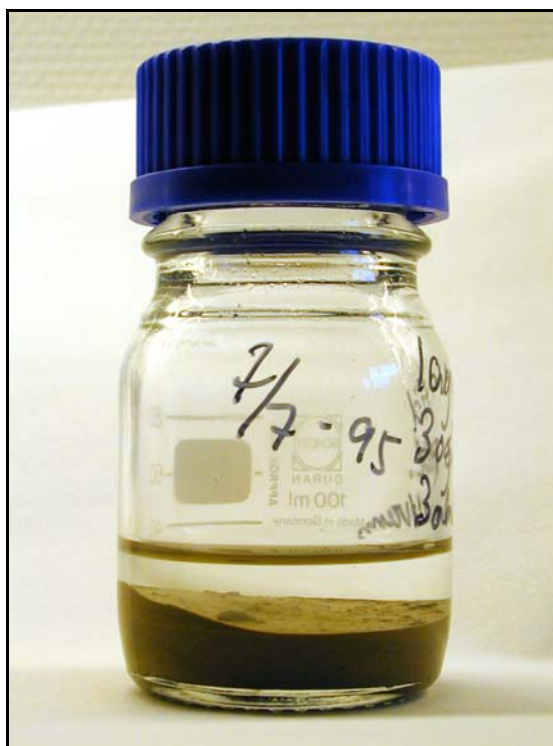


Figure 2.1 A six years old sample with sediment and sea water

When the predetermined time had elapsed, the sea water was carefully separated from the sediment by decanting the water into an Erlenmeyer bottle. Two parallel samples of 10 ml

each were extracted with 2 x 5 ml dichloromethane by shaking for 3 minutes. The extracts were combined and 10 µl of a 0.02 mg/ml solution of C13 was added as internal standard. The extracts were then concentrated to 1 ml with a gentle stream of nitrogen.

From the sediment sample two parallel samples of 10 g each were taken out and extracted with 2 x 5 ml dichloromethane by sonication for 5 minutes. The solvent was decanted from the sediment and combined. No filtration or centrifugation was necessary. 10 µl of a 0.02 mg/ml solution of C13 was added as internal standard and the samples concentrated to about 1 ml with a gentle stream of nitrogen.

3 RESULTS AND DISCUSSION

Several decomposition products from mustard gas were identified both in sea water samples and in sediment samples. A list of the identified compounds is given in Table 3.1

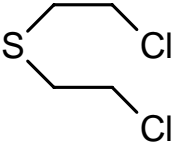
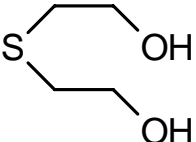
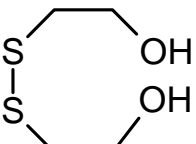
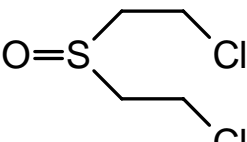
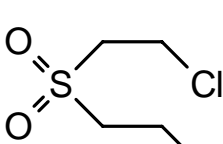
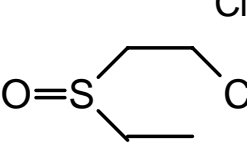
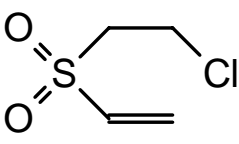
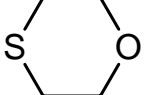

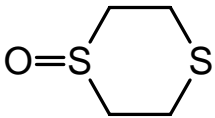
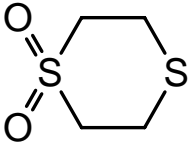
Compound	Structure	CAS#	MW	RI	Identified compound	
					Water	Sediment
Sulphur mustard (1)		505-60-2	158	1173	✓	✓
Thiodiglycol (TDG) (2)		111-48-8	122	1184	✓	✓
2,2'-dithiobisethanol (3)		1892-29-1	154	1428	✓	✓
Bis(2-chloroethyl) sulphoxide (4)		5819-08-9	174	1452	✓	
Bis(2-chloroethyl) sulfone (5)		471-03-4	190	1431	✓	
2-chloroethyl vinyl sulphoxide (6)		40709-82-8	138	1161	✓	
2-chloroethyl vinyl sulphone (7)		7327-58-4	154	1212	✓	
1,4-thioxane (8)		15980-15-1	104	890	✓	✓
1,4-dithiane (9)		505-29-3	120	1079	✓	✓
1,4-dithiane-1-oxide (10)		19087-70-8	136	1396	✓	✓
1,4-dithiane-1,1- dioxide (11)		139408-38-1	152	1447	✓	✓

Table 3.1 Compounds related to sulphur mustard identified in sea water and/or sediment samples

In cases where authentic standard were not accessible EI, CI mass spectral data and GC retention indices (4) was compared with published data. (Table 3.2).

	Retentions index (RI) ¹			CI	References
	FFI	Canada ²	Verifin ³		
Sulphur mustard (HD)	1173	1173	1178	Not recorded	Authentic standard
Thiodiglycol (TDG)	1184	1182	1184	i-C ₄ H ₁₀ /NH ₃	Authentic standard
2,2'-dithiobisethanol	1428		1425	i-C ₄ H ₁₀ /NH ₃	(8)
Bis(2-chloroethyl) sulphoxide	1452	1443	1455	i-C ₄ H ₁₀ /NH ₃	(9)(10)
Bis(2-chloroethyl) sulphone	1431		1433	NH ₃ (i-C ₄ H ₁₀ not recorded)	(9)(10)
2-chloroethyl vinyl sulphoxide	1161			i-C ₄ H ₁₀ /NH ₃	(9)
2-chloroethyl vinyl sulphone	1212			i-C ₄ H ₁₀ /NH ₃	(9)(9)
1,4-thioxane	890	880	877	i-C ₄ H ₁₀	(10)
1,4-dithiane	1079	1060	1068	i-C ₄ H ₁₀ /NH ₃	(10)(11)
1,4-dithiane-1-oxide	1396			i-C ₄ H ₁₀ /NH ₃	(8)(9)(11)
1,4-dithiane-1,1-dioxide	1447			i-C ₄ H ₁₀ /NH ₃	(11)

Table 3.2 Retention indices, reagent gases used for CI and literature references of the detected decomposition products from mustard gas

¹RI is based on calculation using n-alkanes as index standards and GC-columns with stationary phase 95 % dimethyl/5 % phenyl silicone; such as SE-54, CP-Sil 8 and DB-5

²Reference (6)

³Reference (7)

3.1 Decomposition of sulphur mustard

Hydrolysis is an important degradation pathway in aquatic environments, but the low solubility in water prevents hydrolysis. Hydrolysis in sea water is two to three times slower than in fresh water, due to the chloride ions in sea water affecting the equilibrium between mustard and the intermediate sulphonium ion (5). In addition, the rates of hydrolysis are slowed down at low temperatures, (5) as table 3.3 shows.

T (°C)	t _{1/2} (min)	k (min ⁻¹)
5	175	0.004
15	49	0.0141
25	15	0.046

Table 3.3 Mustard gas half-lives in sea water with the corresponding rate constant

The laboratory experiments were carried out at room temperature, while the temperature at the seabed of Skagerrak would be near constant all year around, i.e. 4-6 °C at 700 m depth (1).

It is well known that the chemical warfare agent sulphur mustard (H, **1**) hydrolyses in water to give hemimustard which then hydrolyses to TDG (**2**) (Figure 3.1). Both reactions take place through a rearrangement of the reactant to an intermediate sulphonium chloride salt (neighbouring group assistance) followed by attack of water to give hemimustard and TDG, respectively. In an alternative reaction, 1,4-thioxane (**8**) is formed through an internal reaction of the intermediate sulphonium chloride salt of the hemimustard (**5**).

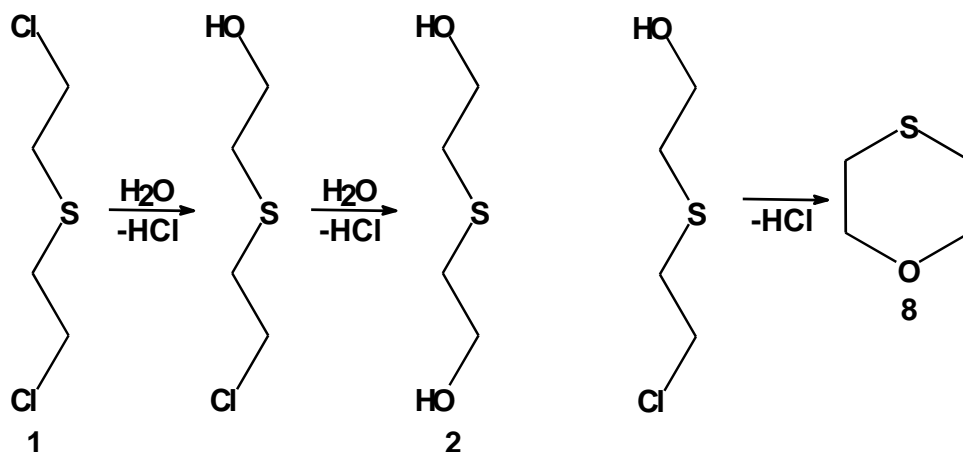


Figure 3.1 Hydrolysis of sulphur mustard (H,1)

Sulphur mustard can itself act as a nucleophile and attack the sulphonium chloride salt of another sulphur mustard molecule. This gives another sulphonium chloride salt, which upon attack of the salt's chloride ion gives sesquimustard (Q) (**5**). Q was not detected in the samples. It has been proposed that Q through an internal reaction can form a 6-ring sulphonium chloride, which upon attack by the chloride ion forms 1,4-dithiane (**9**) (**12**).

If undisturbed, a layer of TDG will build up on the interface between sulphur mustard and water. At this interface, sulphur mustard can also react with TDG, giving stable sulphonium chloride salts. Sesquimustard (Q) may also form sulphonium salts that react with water, mustard, TDG and Q molecules, forming higher homologues of sulphonium salts. The sulphonium chloride salts create a thicker boundary layer. These effects reduce the dissolution and the rate of hydrolysis of sulphur mustard (**5**).

Sulphur mustard and a number of its hydrolysis products are oxidized (environmental degradation) to give sulfoxide (**4** and **6**) and sulphone (**5** and **7**) analogs.

Table 3.3 and 3.4 show the decomposition of mustard gas after different time in sea water and sediment. The concentration of mustard gas and TDG is calculated by using the internal standard method. For the other identified compounds, no quantification was carried out.

Compound	Amounts recovered per sample after exposure time (days)								
	1	7	14	24	39	48	247	360	2180
Mustard gas	28 (0.67)	0.15 (0.0036)	—	—	—	—	—	—	—
Thiodiglycol (TDG)	3.7 (0.56)	100 (15)	93 (14)	100 (15)	—	170 (26)	130 (20)	34 (8)	11 (2.1)
2,2'-dithiobisethanol	—	—	—	—	—	—	3.8	0.2	4.1
Bis(2-chloroethyl) sulphoxide	2.5	8.8	5.5	3.0	2.0	5.6	3.4	3.8	0.5
Bis(2-chloroethyl) sulphone	—	—	—	1.4	1.7	6.7	2.7	3.2	—
2-chloroethyl vinyl sulphoxide	0.2	0.3	0.3	0.3	0.3	0.6	0.4	0.2	0.05
2-chloroethyl vinyl sulphone	—	—	—	30	0.93	1.7	8.8	3.2	0.03
1,4-thioxane	0.02	0.01	0.03	0.02	0.01	0.02	0.01	0.07	0.004
1,4-dithiane	3.4	5.9	5.5	5.2	0.5	2.9	1.2	0.5	0.2
1,4-dithiane-1-oxide	—	—	—	—	—	—	0.1	0.03	0.5
1,4-dithiane-1,1-dioxide	—	—	—	—	—	—	0.07	0.1	0.04

Table 3.4 Recovered amounts from sea water (peak areas of compound relative to the peak areas of internal standard). Blue text: concentration (μg compound/ml water)

Compound	Amounts recovered per sample after exposure time (days)								
	1	7	14	24	39	48	247	360	2180
Mustard gas	100 (2.4)	45 (1.1)	—	—	—	Not measured	—	—	—
Thiodiglycol (TDG)	89 (14)	4.8 (0.7)	10 (1.5)	18 (2.7)	—		77 (12)	33 (5)	0.8 (1.6)
2,2'-dithiobisethanol	—	—	—	—	—		0.5	0.01	0.2
1,4-thioxane	0.07	—	—	—	—		0.09	0.07	—
1,4-dithiane	0.01	—	—	—	—		0.5	0.07	0.05
1,4-dithiane-1-oxide	—	—	—	—	—		—	—	0.09
1,4-dithiane-1,1-dioxide	—	—	—	—	—		0.7	0.1	0.04

Table 3.5 Recovered amounts from sediments (peak areas of compound relative to the peak areas of internal standard). Blue text: concentration (μg compound/g wet sediment)

Some water samples were evaporated to dryness in a vacuum concentrator. The dry samples including salt from sea water were diluted in 1 ml acetonitrile and then pushed through a cation exchange (SCX) cartridge before silylation with methyl-N-tert(butyltrimethylsilyl)trifluoroacetamide (MTBSTFA). Only the thiodiglycol-derivative was detected in the samples (after six years). The sensitivity of the instrument decreases with number of samples, because the ion source becomes coated with salt. Further experiments have to be done to remove the salt from the samples before analyses on the mass spectrometer.

4 CONCLUSIONS

The hydrolysis gives about 1000 times more of thiodiglycol than 1,4-thioxane in sea water and about 100 times more in the sediment. (In (5) four times more).

Already after one day mustard gas in sea water is oxidized to sulphoxide. Forming of the sulphone products are slower in sea water and they were not detected before 24 days. The open chain sulphoxides and sulphones were not detected in the sediment.

The formation of 2,2'-dithiobisethanol, 1,4-dithiane-1-oxide and 1,4-dithiane -1,1-dioxide from mustard gas both in sea water and sediment takes about 8 months at room temperature. All the decompositions products have decreased from one year to six years, except 2,2'-dithiobisethanol and 1,4-dithiane-1-oxide. 1,4-dithiane-1-oxide was detected for first time in sediment samples at six years, and the relative amount increased from 8 months (first time detected) to six years in sediment samples.

APPENDIX**A MASS SPECTRA OF SULPHUR MUSTARD AND SOME RELATED COMPOUNDS**

1,4-thioxane

1,4-dithiane

2-chloroethyl vinyl sulphoxide

Sulphur mustard (HD)

Thiodiglycol (TDG)

2-chloroethyl vinyl sulphone

1,4-dithiane-1-oxide

2,2'-dithiobisethanol

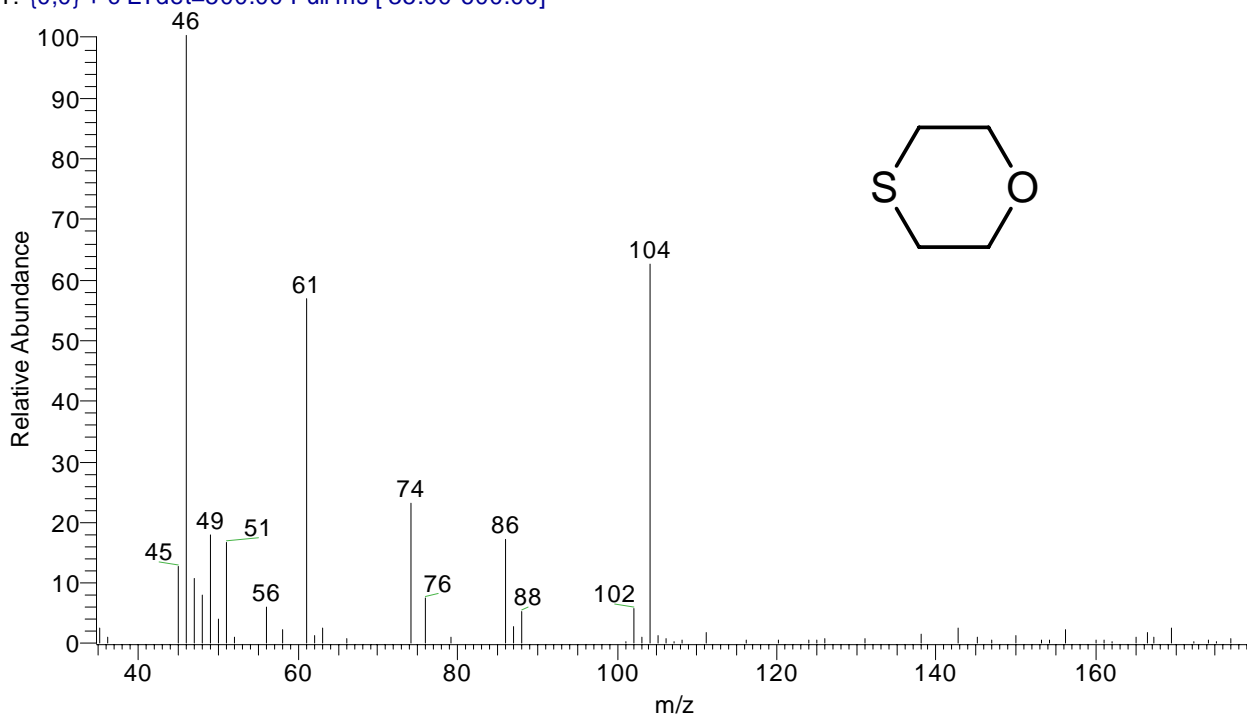
Bis(2-chloroethyl) sulphone

1,4-dithiane-1,1-dioxide

Bis(2-chloroethyl) sulphoxide

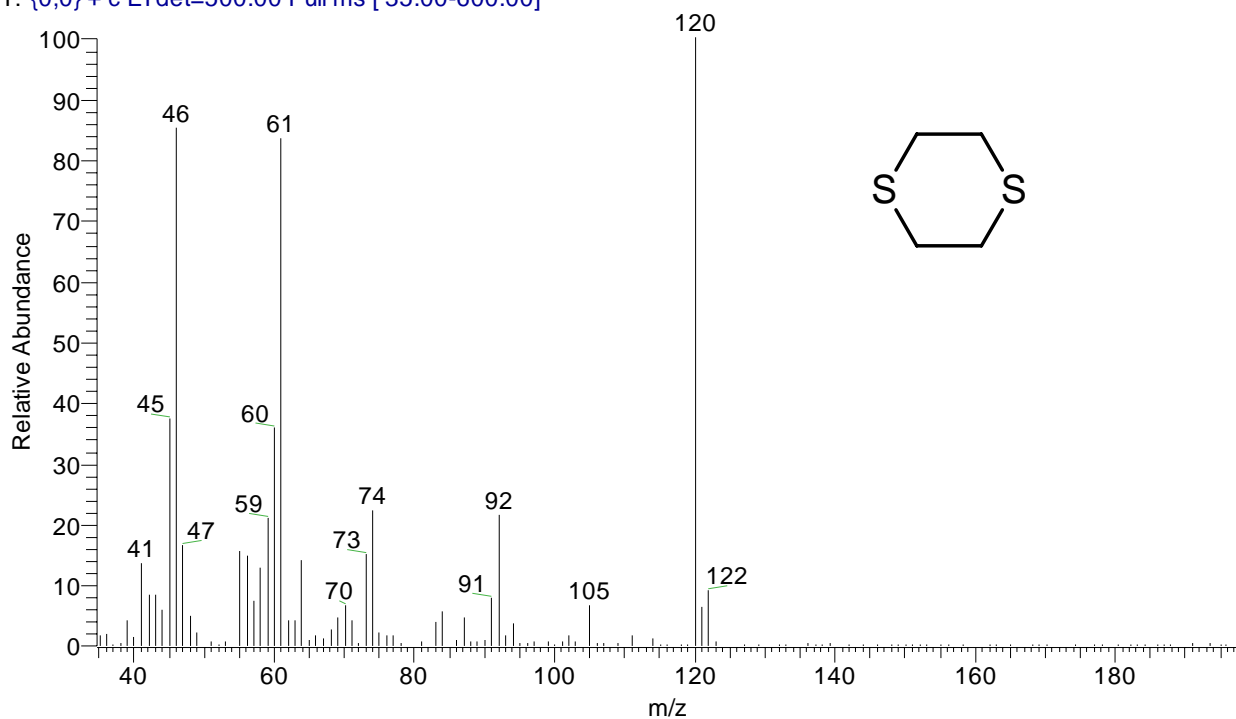
1,4-thioxane (MW 104)

01030725 #330-339 RT: 5.83-5.92 AV: 10 SB: 30 5.58-5.71, 6.04-6.19 NL: 4.38E3
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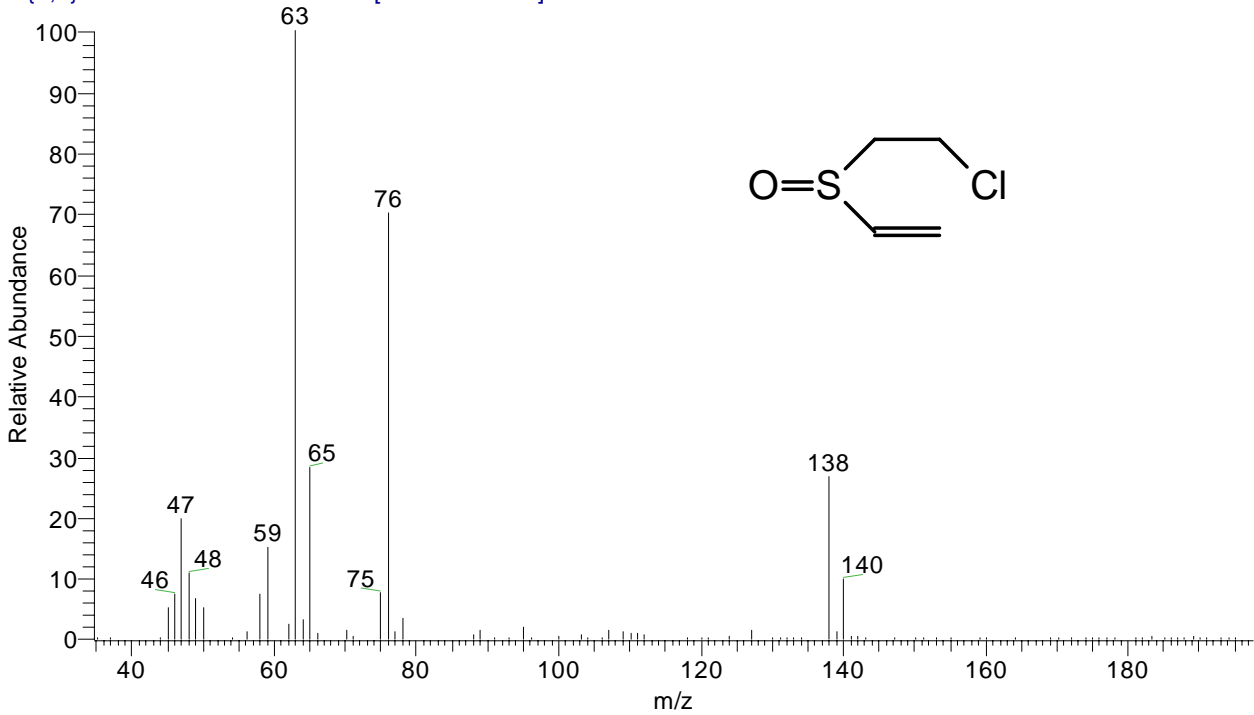
1,4-dithiane (MW 120)

01030725 #628-632 RT: 8.85-8.89 AV: 5 SB: 14 8.56-8.62, 8.95-9.01 NL: 5.59E4
T: {0,0} + c EI det=500.00 Full ms [35.00-600.00]



2-chloroethyl vinyl sulfoxide (MW 138)

01030725 #773-781 RT: 10.31-10.39 AV: 9 SB: 9 10.25-10.28, 10.40-10.44 NL: 4.59E4
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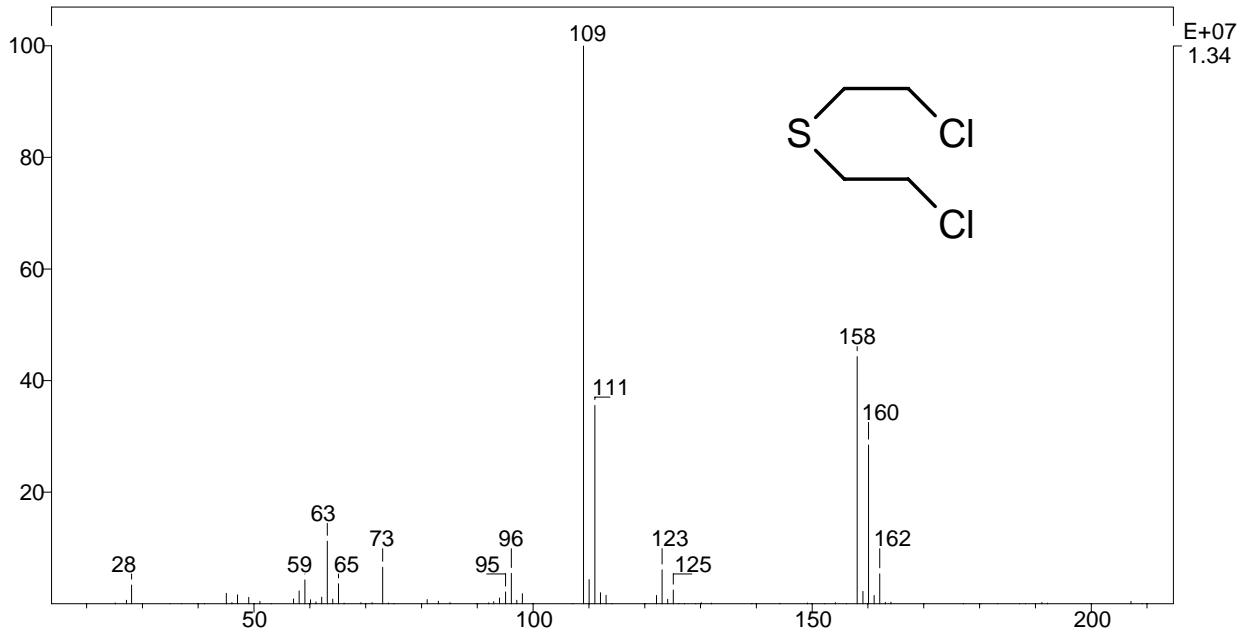


Sulfur mustard (HD) (MW 158)

SPEC: 950901-05

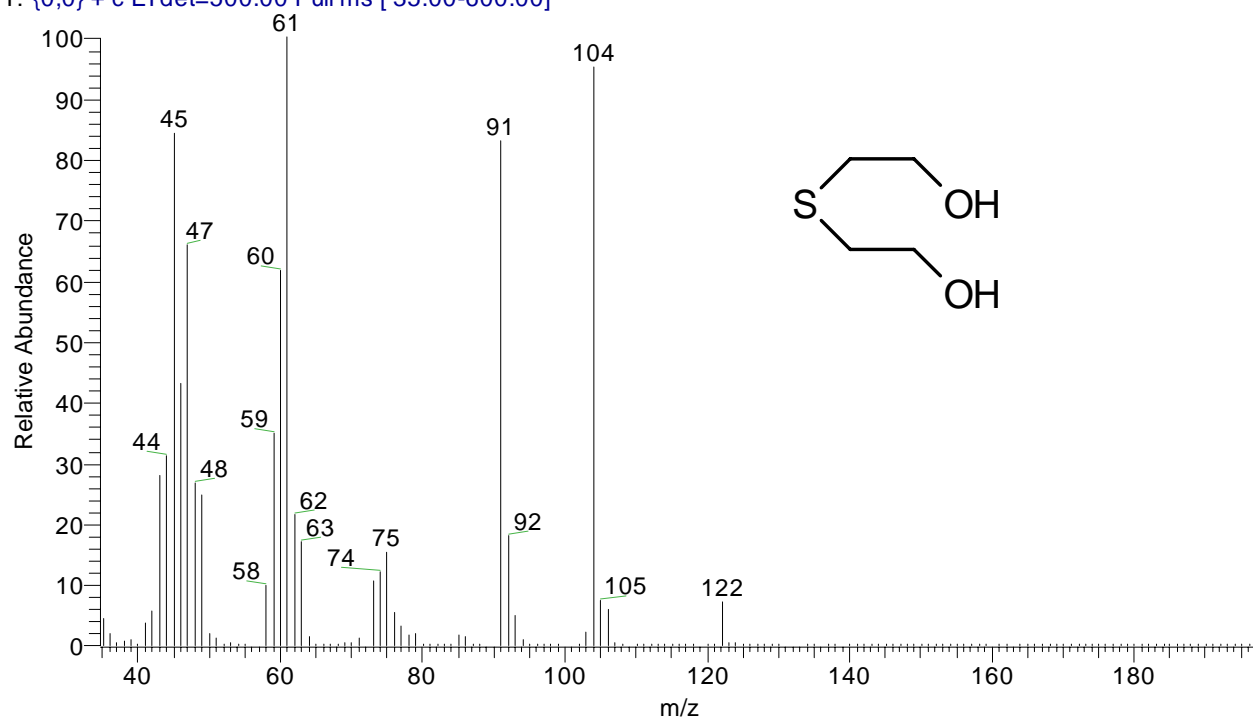
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01-Sep-95 DERIVED SPECTRUM#9



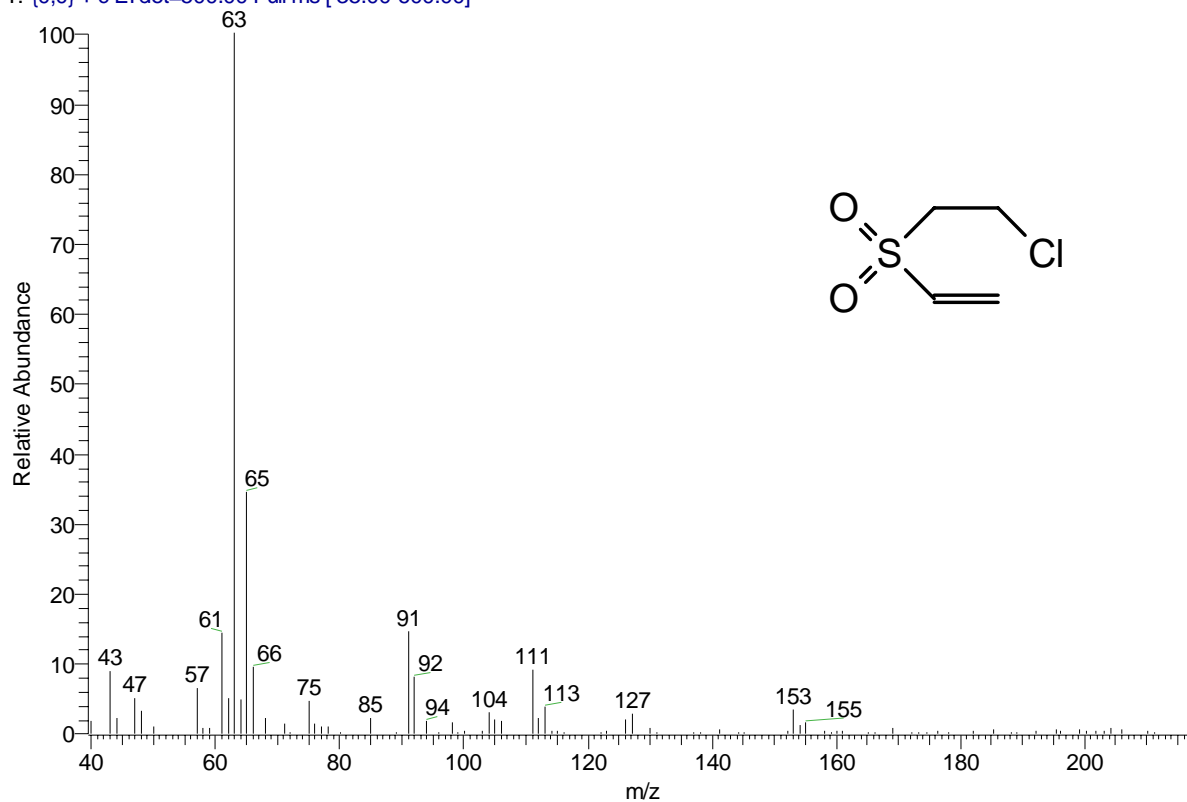
Thiodiglycol (TDG) (MW 122)

01030725 #795-798 RT: 10.53-10.56 AV: 4 SB: 6 10.46-10.48, 10.60-10.63 NL: 2.95E6
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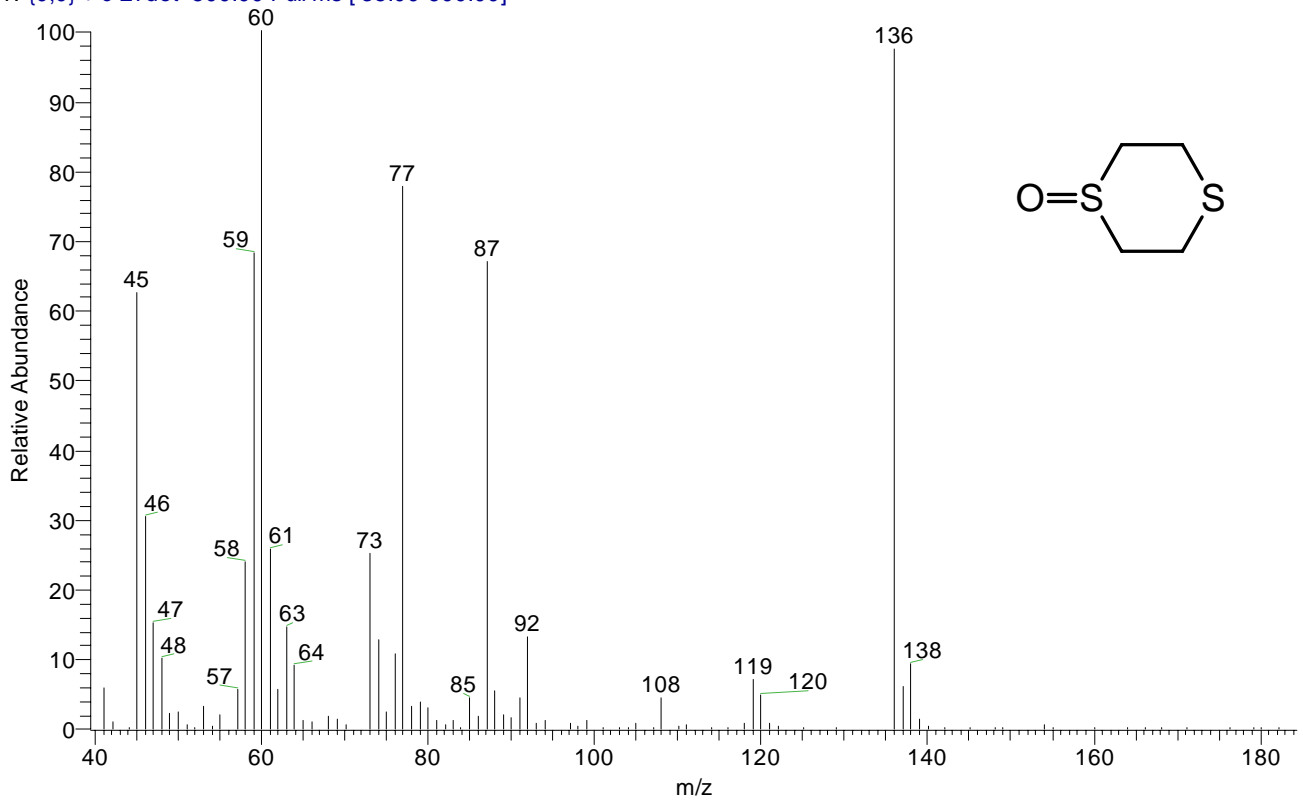
2-chloroethyl vinyl sulfone (MW 154)

01030726 #834-837 RT: 10.94-10.98 AV: 4 SB: 5 10.88-10.90, 11.12-11.13 NL: 2.46E4
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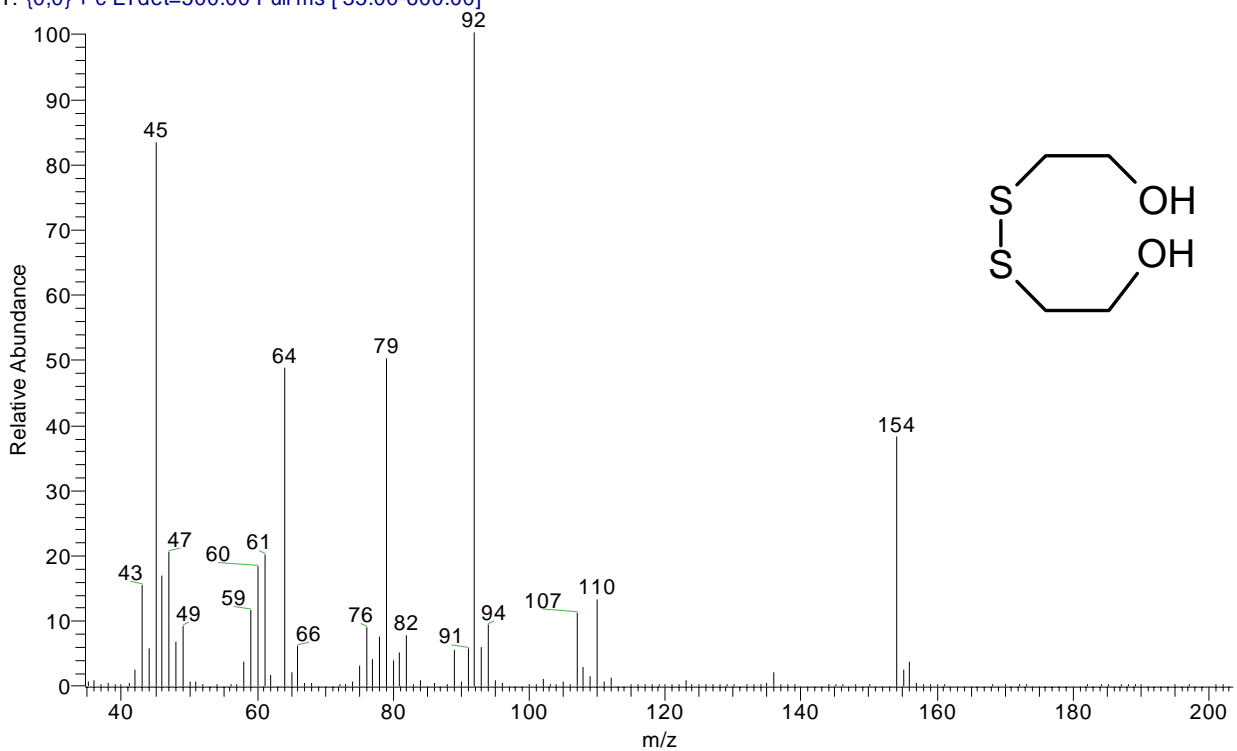
1,4-dithiane-1-oxide (MW 136)

01030726 #1089-1093 RT: 13.52-13.57 AV: 5 SB: 22 13.33-13.42, 13.72-13.83 NL: 1.69E5
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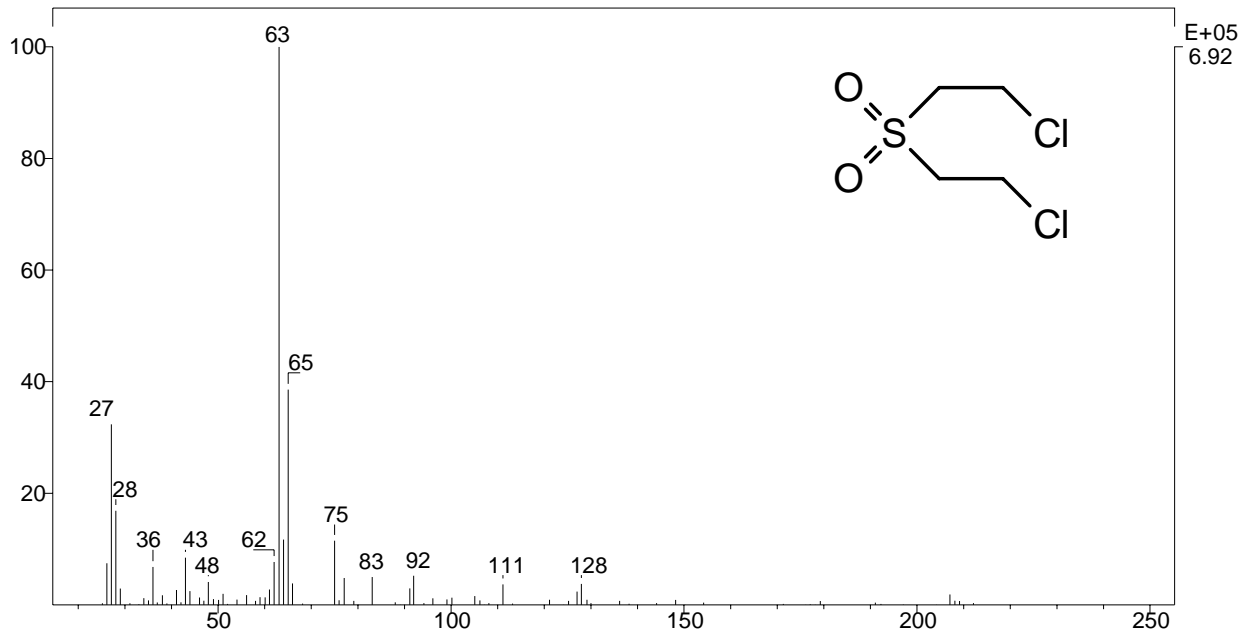


2,2'-dithiobisethanol (MW 154)

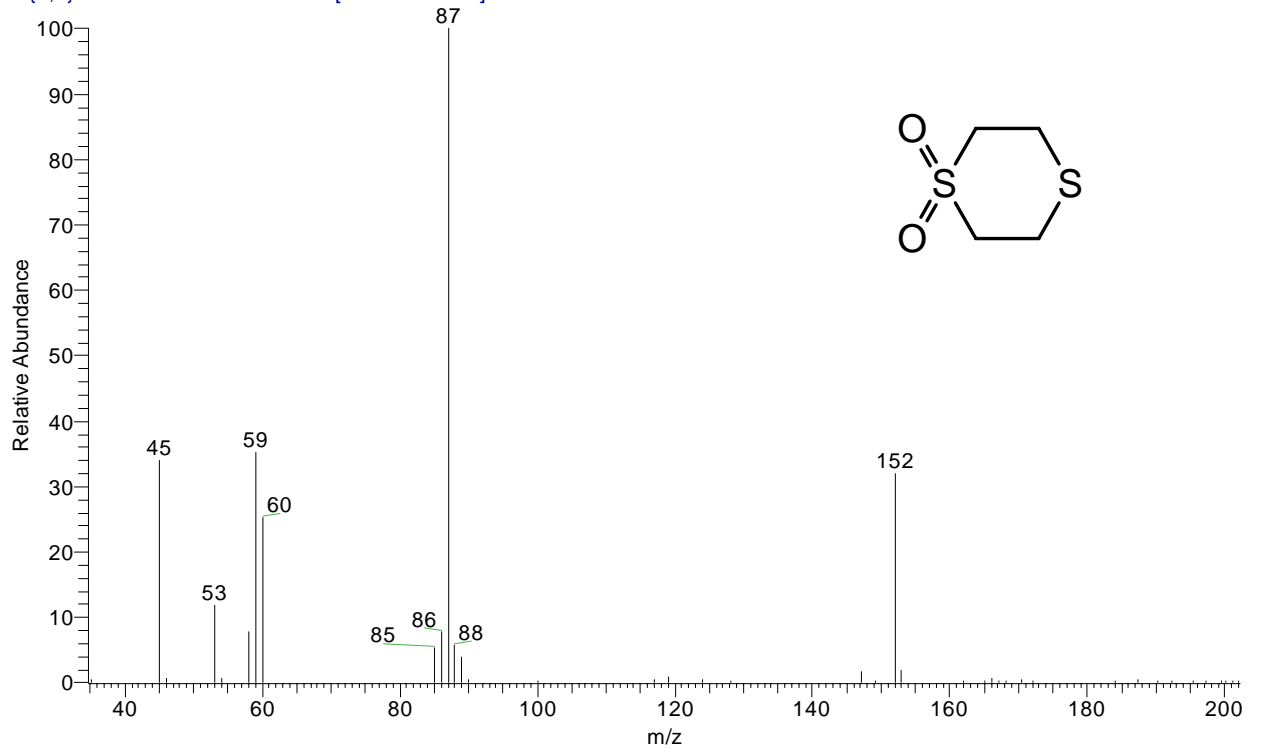
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T: {0,0} + c EI det=500.00 Full ms [35.00-600.00]



Bis(2-chloroethyl) sulfone (MW 190)

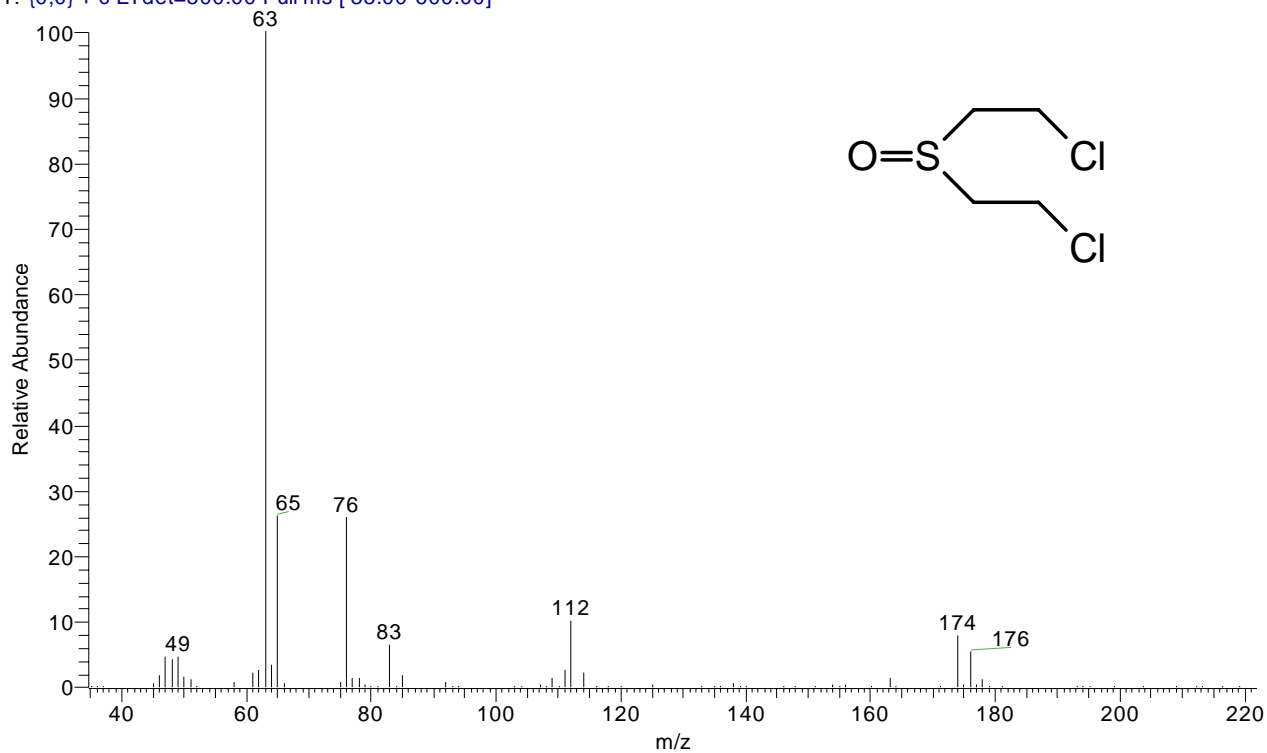
SPEC: 950731-04
Data: +/772>773 - /766>770,775>778 31-Jul-95 DERIVED SPECTRUM#9

1,4-dithiane-1,1-dioxide (MW 152)

01030726 #1153-1155 RT: 14.17-14.19 AV: 3 SB: 11 14.07-14.11, 14.26-14.31 NL: 9.45E4
T: {0,0} + c EI det=500.00 Full ms [35.00-600.00]

Bis(2-chloroethyl) sulfoxide (MW 174)

01030726 #1162-1167 RT: 14.26-14.31 AV: 6 SB: 30 14.10-14.21, 14.53-14.70 NL: 2.80E5
T: {0,0} + c EI det=500.00 Full ms [35.00-600.00]



References

- (1) Tørnes John Aa, Blanch Jan H, Wedervang Tor I, Andersen Arne G, Opstad Aase M (1989): Undersøkelse av skipsvrak inneholdende kjemisk ammunisjon senket i norske farvann etter annen verdenskrig, FFI/RAPPORT-89/6007, Forsvarets forskningsinstitutt, Unclassified
- (2) Mariusz Mazurek, Zygfryd Witkiewck, Stanislaw Popiel, Maciej Sliwakowski (2001): Capillary gas chromatography-atomic emission spectroscopy-mass spectrometry analysis of sulphur mustard and transformation products in a block recovered from Baltic Sea, *Journal of Chromatography A*, 919, 133-145
- (3) Tørnes, John Aa, Voie, Øyvind A, Ljønes, Marita, Opstad, Aase M, Bjerkeseth, Leif H, Hussain. Fatima (2002): Investigation and risk assessment of ships loaded with chemical ammunition scuttled in Skagerrak, FFI/RAPPORT-2002/04951
- (4) Tørnes John Aa, Opstad Aase M (1996): Research report on verification of chemical weapons convention-part XIII- Evaluation of Recommended Operating Procedures for sampling and Analysis, FFI/RAPPORT-96/05379, Forsvarets forskningsinstitutt, Unclassified
- (5) Background on chemical warfare, Mitretek Systems, Mc Lean, VA, USA (2000). Internet: <http://www.mitretek.org/home.nsf/homelandsecurity/Mustard>
- (6) James R Hancock and Clifford E Lough (1991): Canadian Contribution to SICA Retention Index Data Base of Compounds of Chemical Defence Interest (Presentation: Nato AC/225 (Panel VII/SICA)N/205 (NATO Unclassified).
- (7) The Ministry for Foreign Affairs of Finland (1994): Recommended Operating Procedures for Sampling and Analysis in the Verification of Chemical Disarmament (ROP).
- (8) (1998): NIST-library.
- (9) FFI-CWA/MS-library.
- (10) (1995): NATO Handbook for Sampling & Identification of Chemical Warfare Agents, Volume 3, Spectrometric and Chromatographic Data for CWA and Related Compounds, Book 4, (Edition 4) (NATO Restricted).
- (11) The Ministry for Foreign Affairs of Finland (1994): Interlaboratory Comparison Test Coordinated by the Provisional Technical Secretariat for the Prerartory Commission for the Organisation for the Prohibition of Chemical Weapons, H.1 First Interlaboratory Comparison Test.
- (12) Rohrbaugh D K, Yang Y C (): Liquid Chromatography/Electrospray Mass Spectrometry of Mustard-related Sulfonium Ions, *J. Mass Spectrom* **32**, 1247-1258.