

DMDS Derivatisation Procedure

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1 Risk Assessment

Table 1: Reagents (Figure 1) and associated hazards.

Substances used	Hazards identified
Hexane	Flammable, harmful by inhalation, irritant, toxic to aquatic life
Dimethyl disulfide (DMDS)	Flammable, toxic, irritant, very toxic to aquatic life
5% iodine in diethyl ether	Toxic, irritant, toxic to aquatic life
Sodium thiosulfate solution	Irritant
Silica powder	Lung irritant

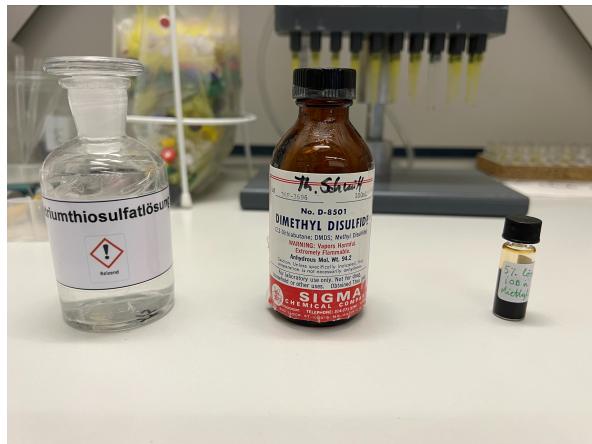


Figure 1: Reagents for derivatisation.

2 Method

2.1 Purification of CHC extract - Column Chromatography

2.1.1 Column preparation

A pipette column was prepared by packing a bit of glass wool to the bottom of the pipette with a glass rod and tweezers to avoid the silica from falling through but loose enough to allow liquid to pass. Silica gel 60 (0.063-0.200 mm, 70-230 mesh ASTM) was added to approximately half of the pipette shown in Figure 2. A beaker was positioned beneath the column and eluent (**hexane**) was washed through the column five times making use of a syringe to gently push the eluent through the column, stopping when the eluent reached approximately 5 cm above the silica section as the silica section must **stay wet throughout the process**. With eluent ~5 cm above the saturated silica column, the column was ready for the sample.



Figure 2: Pipette column.

2.1.2 Sample addition and collection

The sample was loaded into the column directly above the silica section. The waste beaker was replaced with a smaller beaker and the column was filled with eluent, ensuring the column stayed wet. Two fractions were obtained, concentrated, then combined and the column loaded with the polar substances was tossed into the derivative waste.¹

2.2 DMDS Derivatisation

Equal parts of DMDS ($500 \mu\text{l}$) to the cleaned extract were combined along with three drops of 5 % iodine in diethyl ether solution. The solution was left overnight at 70° in the oven and sodium thiosulfate was added in excess. The aqueous and organic layers were separated (aqueous on bottom and organic on top) and the organic layer was concentrated.

2.3 Data Analysis

The ion chromatogram was extracted and filtered to $\text{m/z} = 61$ to show peaks containing the methyl sulfide ion peak ($\text{CH}_2\text{SCH}_3^+$). The double bond positions could be determined by observing the major fragment ion peaks which add up to the molecular ion peak (M^+).¹ Depending on the number of double bonds, the number of fragments varies (i.e. two double bonds would yield four major fragments).

References

- [1] D. A. Carlson, C. S. Roan, R. A. Yost and J. Hector, *Analytical Chemistry*, 1989, **61**, 1564–1571.

¹The polar substances could be collected with dichloromethane as the eluent after collection of the nonpolar substances in hexane.