was obtained as the first fraction elected from a silica gel column, m.p. 149–150°. Its infrared spectrum was quite different from that of the 152° isomer.

Anal. Calcd. for $C_{18}H_{18}N_2O_3S$: C, 63.14; H, 5.30; N, 8.18. Found: C, 63.07; H, 5.40; N, 7.62.

Reaction of Sodium Ethoxide and Phenylglyoxylonitrile Oxime Tosylate.—A mixture of 5.00 g. (16.7 mmoles) of the tosylate and 100 ml. of absolute ethanol was refluxed while 13.9 ml. of 2.60 *N* sodium ethoxide in ethanol was added dropwise. The mixture was refluxed an additional 2 hr. and then the volume of ethanol was reduced to about 25 ml. at reduced pressure. The residue was diluted with water and the product was partitioned between water and methylene chloride. Evaporation of the methylene chloride left 2.49 g. of residue. Distillation of this residue gave diethyl N-phenylimidocarbonate, 1.73 g., b.p. 100° (5 mm.), $n^{20}D$ 1.5140, infrared spectrum identical with that of an authentic sample.[10]

Diethyl N-(*p*-Chlorophenyl)imidocarbonate.—A solution of 2.0 g. (6 mmoles) of the tosylate of *p*-chlorophenylglyoxylonitrile oxime in 75 ml. of absolute ethanol was stirred at 60° while 13 mmoles of potassium hydroxide in 10 ml. of ethanol was added dropwise. The solution then was stirred overnight at ambient temperature. After the volume of ethanol in the reaction mixture had been concentrated to about 20 ml., water was added, and the mixture was extracted with methylene chloride. The residue obtained on evaporation of the methylene chloride was distilled to give diethyl N-(*p*-chlorophenyl)imidocarbonate, 0.8 g., $n^{20}D$ 1.5295, b.p. 100° (1 mm.), infrared spectrum identical with that of an authentic sample.[10]

Isolation of Urethans and Nitriles.—The procedures used to *obtain the data given in Table II are illustrated.*

(A) **Ethanolic Potassium Hydroxide and *p*-Chlorophenylglvoxylonitrile Oxime Tosylate.**—A solution 3.35 g. (10 mmoles) of the tosylate was treated with 22 mmoles of potassium hydroxide in ethanol as described earlier. The 3.0 g. of crude residue obtained was chromatographed on a silica gel column. Pentane in methylene chloride, methylene chloride, and ethyl acetate in methylene chloride were used as successive eluents. The first fraction characterized was *p*-chlorobenzonitrile, 0.077 g. (5.6%), m.p. 92–93°, infrared spectrum identical with that of an authentic sample. The major fraction, eluted by 5% ethyl acetate in methylene chloride, was recrystallized from hexane and gave *p*-chlorophenylurethan, 1.28 g. (64%), m.p. 68–69° (reported[10] m.p. 68–69°).

(B) **Ethanolic Sodium Ethoxide plus *p*-Methoxyphenylglyoxylonitrile Oxime Tosylate.**—A solution of 4.95 g. (15 mmoles) of the tosylate in 100 ml. of hot absolute ethanol was stirred while 30 ml. of 1.19 *N* sodium ethoxide in ethanol (36 mmoles) was added. The mixture was refluxed for 2 hr. The organic product was isolated by extraction with methylene chloride and was chromatographed on a silica gel column. The first fraction characterized was an isonitrile, 0.127 g. (6.4%), m.p. 61–62°, infrared spectrum identical with that of authentic material. The major fraction, *p*-methoxyphenylurethan, weighed 2.27 g. (76.7%). One recrystallization from hexane gave 1.88 g. of urethan, m.p. 65–66° (reported[14] m.p. 66–67°).

(C) **Ethanolic Potassium Hydroxide and Mesitylglyoxylonitrile Oxime Tosylate.**—When 1.7 g. (5 mmoles) of the tosylate of m.p. 152° (presumably of configuration I) was allowed to react with 12 mmoles of potassium hydroxide in 40 ml. of ethanol for 80 min., the only products characterized after chromatography on silica gel were mesitylglyoxylonitrile oxime, 0.38 g. (40%), m.p. 113–114°, and 0.19 g. of an oil that appeared (infrared and analysis) to be mesitylglyoxylonitrile oxime ethyl ether.

Anal. Calcd. for $C_{13}H_{16}N_2O$: C, 72.19; H, 7.45; N, 12.96. Found: C, 72.06; H, 7.69; N, 14.3.

In another experiment, 2.60 g. (7.6 mmoles), of the mixed tosylates of m.p. 132–136° was allowed to react with 15.1 mmoles of potassium hydroxide in 80 ml. of ethanol at 60° for 3 hr. The product mixture was chromatographed on silica gel to give, in order of elution from the column, 0.24 g. of the supposed O-ethyl oxime, 0.11 g. of mesitonitrile, m.p. 49–51° (reported[15] m.p. 50–52°), and 0.79 g. (55%) of the recovered oxime, m.p. 72–76°.

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Synthesis of Di(methylthiomethyl) Polysulfides

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Received January 18, 1963

sym-Dialkyl polysulfides have been known for a long time.[1,2] Some of these have been detected in natural products,[3-5] while others have found industrial applications.[6] Recently, a series of methylthioalkyl isothiocyanates, of which methylthiomethyl isothiocyanate[7] is the lowest member, has been isolated from natural products.[4,8] Symmetrical dialkyl polysulfides, wherein the alkyl moiety contains a sulfur atom, as exemplified by the methylthiomethyl group, have not been described.

$$CH_3—S—CH_2—S_n—CH_2—S—CH_3$$

Methylthiomethyl mercaptan, a key compound in the present investigation, was prepared in good yields by a modification of the method described by Fehér and Vogelbruch.[9] This modification obviated the danger of an explosion. Due to its instability and permeating, garliclike odor, much difficulty was encountered in handling and characterizing this compound. It was found practical not to isolate the mercaptan for preparative work, but to use it in absolute ether solution.

Di(methylthiomethyl) sulfide, included for comparative purposes, was prepared by a new method from chloromethyl methyl sulfide and the hydrate of sodium hydrosulfide (from commercial source) in benzene.

The di- and trisulfides were obtained in substantial amounts as by-products from the reaction used to make the tetrasulfide, namely by treating methylthiomethyl mercaptan and sulfur monochloride in dry benzene in the presence of pyridine at a low temperature—a modification of the general procedure for the synthesis of polysulfides of Patel, *et al.*,[10] who did not obtain the tetrasulfide. The di- and trisulfides were obtained in the pure state by fractional distillation in high vacuum, while the pure tetrasulfide was isolated from the impurities in the residue by extraction with solvents.

In another experiment the same reaction was carried out but in the absence of pyridine. The vacuum fractionation of the reaction product yielded the di- and trisulfides, and an appreciable residue, which could not be distilled. This polysulfidic substance underwent autodesulfuration at room temperature, a behavior

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previously observed to occur in the higher polysulfides.[11,12] In about three weeks, the separation of sulfur ceased and the compound reached the stable state corresponding to an octasulfide. This transformation is shown in Table I. The use of pyridine in this reaction has favored the formation of lower polysulfides.

TABLE I

AUTODESULFURATION OF A DI(METHYLTHIOMETHYL) POLYSULFIDE PRODUCT

Time, days	Found C	Found H	Found S	$(CH_3SCH_2)_2S_n$	Calcd. C	Calcd. H	Calcd. S
0[a]	...	...	...	$n > 10$	...	...	...
3	10.89	2.32	86.79[b]	10	10.85	2.28	86.88
24	13.17	2.97	84.26	8	12.68	2.66	84.66
69	13.20	3.12	84.84	8	12.68	2.66	84.66

[a] A sample could not be taken for analysis at this stage of transformation. [b] Obtained by difference.

Methylthiomethyl pentasulfide was prepared from chloromethyl methyl sulfide and anhydrous potassium pentasulfide substantially by the same procedure used by Riding and Thomas[13] for making di(allyl) pentasulfide.

By analogy to the structure of the dialkyl polysulfides,[14-18] it is assumed that the polysulfidic bridge of the di(methylthiomethyl)polysulfides has an unbranched chain structure.

The physical properties of the di(methylthiomethyl) polysulfides are given in Table II.

TABLE II

DI(METHYLTHIOMETHYL) POLYSULFIDES

$CH_3SCH_2 \cdot S_n \cdot CH_2SCH_3$

Di(methylthiomethyl)	n	B.p., °C. (mm.)	Formula	$n^{t°}_{D}$	Mol. wt. Calcd.	Mol. wt. Found	Calcd. C	Calcd. H	Calcd. S	Found C	Found H	Found S
Monosulfide	1	110.8–111.8 (13.5)[a]	$C_4H_{10}S_3$	1.5811 (20)[a]	154	157[a]	31.13	6.53	62.34	30.71, 30.77	6.64, 6.57	62.13, 61.84[a]
Disulfide	2	83.2–85.8 (0.021)	$C_4H_{10}S_4$	1.6280 (20)	186	188	25.77	5.41	68.82	25.63, 25.51	5.58, 5.49	68.97, 69.24
Trisulfide	3	118–119.8 (0.021)	$C_4H_{10}S_5$	1.6628 (20)	218	212	21.99	4.61	73.39	22.36, 22.29	4.85, 4.69	73.66, 73.75
Tetrasulfide	4	...	$C_4H_{10}S_6$	1.7072 (30)[b]	251	250	19.18	4.02	76.80	19.09, 19.10	4.09, 3.96	77.03, 76.47
Pentasulfide	5	...	$C_4H_{10}S_7$	1.7077 (78.6)[b]	283	268	17.00	3.57	79.43	16.56, 16.64	3.46, 3.59	80.15, 80.39

[a] Fehér and Vogelbruch[9] reported: b.p. 113° (13 mm.); n^{20}_{D} 1.5818; mol. wt., 149; S, 61.58%. [b] The value at 20° was >1.71.

Experimental[19]

Methylthiomethyl Mercaptan.—*S*-(Methylthiomethyl)isothiuronium chloride[7] (19.6 g., 0.114 mole) was hydrolyzed by stirring with 24.6 g. of 5 *N* sodium hydroxide solution at room temperature for 4 hr. The oil that separated as an upper layer was extracted with several portions of peroxide-free ether. The combined ether solutions were washed with water and dried over anhydrous sodium sulfate. After filtration, the solvent was removed by distillation through a Vigreux column in a nitrogen atmosphere at a bath temperature of 45–50°. The residue was subjected to a vacuum of 100 mm. for 5 min. A vapor phase chromatogram of this concentrate showed two peaks, one for the thiol and one for ether. A rough estimate of the amount of mercaptan present was made from the ratio of the areas of the mercaptan and ether peaks. The retention times for the thiol and ether were 2.30 and 0.74 min., respectively, when a 0.1-μl. sample was used with a Perkin-Elmer "Q" column (Apiezon "L" grease) at 165° and 15-p.s.i. helium gas pressure. The best yield of pure mercaptan was 7.49 g. (69.0%, lit.[9] 58%) from 20.0 g. of starting material. Instead of the pure mercaptan, a thiol–ether concentrate, freshly prepared, was preferred for the reaction purposes. The concentrate could be conveniently stored in the presence of nitrogen at about 3°. The pure product was obtained by vacuum distillation under nitrogen at 47 mm. It was a clear, water-white, mobile liquid with a very sharp, penetrating, garliclike odor; b.p. 54–55.3° [lit.[9] b.p. 60° (47 mm.)]. An elemental analysis and infrared spectral data, now reported for the first time, are given subsequently.

Anal. Calcd. for $C_2H_6S_2$: C, 25.50; H, 6.42; S, 68.08. Found: C, 26.14; H, 6.83; S, 67.87.

The infrared spectrum showed, in addition to the characteristic weak band for the thiol group at 2500 cm.$^{-1}$, strong absorption bands at 2830, 1430–1420 (doublet), 1205, 986, and 699 cm.$^{-1}$.

Methylthiomethyl mercaptan, in the pure state or in ether solution, slowly decomposed and deposited a white solid on standing. From related work (unpublished results) on the previous thiol, a solid of this nature was found to be a mixture of thioformaldehyde polymers. Trithiane was not identified as a component of this mixture.

Di(methylthiomethyl) Sulfide.—In an attempt to prepare methylthiomethyl mercaptan by the addition of chloromethyl methyl sulfide to a dry benzene suspension of the solid (previously formed *in situ* by the distillation of the water of hydration as an azeotrope with benzene from commercial sodium hydrosulfide) and by the reflux of the resultant mixture for 10 hr., the sulfide was obtained instead as the major product in the form of a light yellow, mobile oil with a peppery odor. Ten grams of chloromethyl methyl sulfide gave approximately 2.0 g. of di(methylthiomethyl)sulfide, the physical constants (Table II) of which agreed with those of Fehér and Vogelbruch[9] for the same compound prepared by a different method.

A gas–liquid chromatogram showed a retention time of 15.2 min. for the sulfide when a 0.25-μl. sample was used on a Perkin-Elmer "Q" column at a column temperature of 166° and a helium gas pressure of 20 p.s.i.

Di(methylthiomethyl) Disulfide.—One of the fractions from the fractional distillation of the crude tetrasulfide, *vide infra*, was pure di(methylthiomethyl) disulfide. It was a yellow, mobile liquid with a soft, garliclike odor. From 5.2 g. of methylthiomethyl mercaptan, 1.54 g. of the pure disulfide was obtained. The physical constants of this compound and the other polysulfides are given in Table II.

Di(methylthiomethyl) Trisulfide.—This trisulfide was obtained from the same source as the disulfide by vacuum fractional redistillation of a higher boiling fraction of the crude product. It was a liquid, resembling the disulfide closely in appearance and odor. The same amount of mercaptan gave 2.0 g. of this substance.

Di(methylthiomethyl) Tetrasulfide.[20]—To a solution of 5.2 g. (0.056 mole, 5.7% excess) of methylthiomethyl mercaptan, as an

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(20) This reaction should be performed under a well ventilated hood.

ether concentrate,[21] and 5 ml. of dry pyridine in 10 ml. of dry benzene was added dropwise at 5–10° with agitation a solution of 3.5 g. (0.026 mole) of sulfur monochloride in 10 ml. of dry benzene over a period of 1.25 hr. White, crystalline pyridine hydrochloride soon precipitated. The addition of more benzene (10 ml.) was necessary to loosen the crystalline mass. After the reaction mixture had come to room temperature, it was stoppered and allowed to stand overnight. Water (50 ml.) was added and the mixture was shaken for 15 min. The benzene layer was separated and the aqueous layer extracted with 10 ml. of benzene. The benzene layers were combined and washed successively with 40 ml. of 1 *N* sulfuric acid, 40 ml. of 1 *N* sodium bicarbonate, and thrice with 40-ml. portions of water. After the benzene solution was filtered and dried over anhydrous sodium sulfate, the solvent was removed *in vacuo* at 55–60° under nitrogen. When the solvent ceased to distil, the pressure slowly was reduced to 34 mm. The crude product (7.9 g.) was fractionated at 0.021 mm. under nitrogen. The portion that distilled at 83.2–88.5° was essentially pure di(methylthiomethyl) disulfide and that which distilled at 90.2–111° was impure trisulfide. Redistillation of the latter fraction under the same conditions gave pure di(methylthiomethyl) trisulfide. Distillation was discontinued to avoid decomposition when the residue did not distil upon raising the bath temperature to 165°. The residue (2.3 g., 35.4%) was impure tetrasulfide. The method of solvent extraction[5,10,22] previously employed for the isolation and purification of higher polysulfides was used for this compound. Extraction with absolute alcohol, filtration of the alcohol solution, and removal of the solvent at room temperature *in vacuo* and under nitrogen gave the alcohol-soluble tetrasulfide. From this substance, after extraction with absolute chloroform and removal of the solvent under the same conditions, the di(methylthiomethyl) tetrasulfide was obtained as a pale yellow, moderately viscous oil with the usual garliclike odor. The yield was 1.39 g. (21.4%).

Di(methylthiomethyl) Pentasulfide.—To a suspension of 12.1 g. (0.051 mole) of potassium pentasulfide in 100 ml. of anhydrous ether, protected from moisture and previously flushed with dry nitrogen, was added a solution of 9.8 g. (0.101 mole) of chloromethyl methyl sulfide in 50 ml. of anhydrous ether over a period of 0.5 hr. at room temperature, with magnetic stirring. The reaction mixture was flushed again with dry nitrogen, stoppered, and allowed to react at room temperature for 13 days. During this time, the mixture was stirred 8 hr. daily for 9 days. The solid component changed from a coarse, orange-red powder to a pale yellow crystalline material. The reaction mixture was filtered under slight vacuum, the solid residue was washed with several 10-ml. portions of ether, and the washings were combined with the filtrate. The resultant ether solution of the pentasulfide was washed with three 50-ml. portions of water, dried over anhydrous sodium sulfate, filtered, and the ether removed by distillation at 34–40° under a slight vacuum and in a nitrogen atmosphere. The residue was taken to, and kept at, 50° and about 35 mm. for 10 min. The yield of crude pentasulfide was 10 g. (70%). By extracting the crude product with absolute ethanol (distilled over magnesium) to remove the alcohol-soluble impurities, and by subjecting the residue to a high vacuum at room temperature to eliminate the last traces of solvent, the pentasulfide was obtained as a pale yellow, slightly viscous oil with the usual garliclike odor. The yield was 3.9 g. (27.3%). The pentasulfide was stable with respect to autodesulfuration on standing at room temperature.

Di(methylthiomethyl) Polysulfide Products Higher than Pentasulfide.[20]—To a solution of 7.4 g. (0.079 mole) of methylthiomethyl mercaptan in 25 ml. of dry benzene was added a solution of 5.3 g. (0.039 mole) of sulfur monochloride in 15 ml. of dry benzene according to the procedure described earlier for the tetrasulfide. No pyridine was used. Copious evolution of hydrogen chloride gas took place at the beginning of the reaction. When the reaction mixture had reached room temperature, however, only a little hydrogen chloride gas was evolved which gave a weak test with blue litmus paper and with ammonia. With the exception that the benzene solution had to be washed six times with 50-ml. portions of ice–water to obtain a negative test for chloride ion, the procedure for working up the reaction mixture and removing the solvent was the same as that used for the tetrasulfide. The weight of the crude oil was 7.6 g. An attempt to distil it under vacuum (0.08 mm.) in a nitrogen atmosphere gave evidence of decomposition, accompanied by an increase in pressure to 0.1 mm. Two small quantities of oil, 1.4 g. and 1.3 g., presumably impure di- and trisulfide, were collected. When the bath temperature had reached 185° and the vapor 123°, distillation ceased and the pressure dropped to 0.08 mm. The residue was a clear, yellow-orange, moderately viscous oil (3.5 g.) with a mild odor not resembling the previous polysulfides. The oil soon became opalescent and, on standing at room temperature, slowly underwent autodesulfuration with the separation of well defined crystals of rhombic sulfur, identified by melting point (109.1–111.5°) and molecular weight (found, 256, 257; calcd. for S_8, 256). Shaking the polysulfidic material with acetone produced the same transformation in much less time. The autodesulfuration which took place on standing at room temperature was observed over a period of time. At intervals, the clear, supernatant oil was carefully removed from the crystals of sulfur, and its composition determined by elemental analysis. The results are given in Table I. At the end of 3 days, the composition of the polysulfide corresponded to that of di(methylthiomethyl) decasulfide. It corresponded to that of an octasulfide at the end of 24 days, and thereafter remained constant.

Acknowledgment.—The authors are grateful to Dr. D. Stanley Tarbell of the University of Rochester for his helpful suggestions; to Dr. W. Davidson for the molecular weight determinations; to Mr. F. Bissett of our laboratory for the gas chromatography and spectrophotometric determinations; to Mr. C. DiPietro of the Analytical Laboratory for the microanalyses; and to Mr. A. Deacutis for the preparation of potassium pentasulfide.

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The Formation of Thiachroman as a Major Product in the Claisen Rearrangement of Allyl Phenyl Sulfide[1]

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Received March 12, 1963

Under conditions effecting the Claisen rearrangement of allyl phenyl ethers, thia analogs afford mainly the corresponding propenyl phenyl sulfides (prototropic isomerization).[2] Only recently, however, the observations of Kwart and Hackett[3] suggested that, through amine catalysis, the thia analogs also may be induced to follow the path of the Claisen rearrangement; *viz.*, allyl phenyl sulfide (I), dissolved in a high-boiling amine, was converted (15–20%) into 2-methyl-2,3-dihydrobenzothiophene (II) by a single distillation (atmospheric pressure).

During the course of our studies[1,4] a convenient preparation of II was sought and we followed, essentially, the method of Kwart and Hackett. In addition to isolating the desired compound we found for the

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[1] This study is part of a series dealing with the nature of organic sulfur groups and is supported by grants from the Petroleum Research Fund. The preceding paper is by C. Y. Meyers, G. Lombardini, and L. Bonoli, *J. Am. Chem. Soc.*, **84**, 4603 (1962). (a) Inquiries should be directed to C. Y. Meyers, Department of Chemistry, University of Southern California, Los Angeles 7, Calif.

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