Polarographic analysis[11] is most convenient, but determination of absorptivity at 420–430 mμ using pure 9,10-diketostearic acid as the reference standard can also be employed.[24] Absorption at about 270–280 mμ may be unreliable because of interference from ketohydroxy- and dihydroxystearic acids.[24]

In ketol-forming experiments in which a small excess (12.5%) of potassium permanganate is used, 5–15% of the reaction products cannot be accounted for analytically as ketohydroxy-, dihydroxy- and diketostearic acids or as unoxidized or saturated acids. Separation of products yields a small fraction with a high acid number (270–300) and the characteristic odor of pelargonic acid. A minor amount of chain cleavage must also be occurring during the reaction. On the other hand, when exactly equivalent amounts of oleic acid and potassium permanganate are used the yield of cleavage products is negligible but about 10% of the oleic acid remains unoxidized.

Application of the ketol reaction with *p*H control to linoleic acid (*cis,cis*-9,12-octadecadienoic acid) yields a reaction product in which 90% of the unsaturation has disappeared. The combined hydroxyl and carbonyl oxygen content (5.7%) of the product, however, indicates that on the average only one oxygen atom has been introduced into the chain, whereas a total of four would be anticipated. The reaction product is a viscous liquid and, since the carboxyl function is still present, it is assumed that a substantial amount of polymerization involving the unsaturation must have occurred. *trans,trans*-9,11-Octadecadienoic acid gives similar results.

Acknowledgment.—We wish to thank George C. Nutting and Ann M. Smith for ultraviolet absorption measurements, and Howard Teeter for a sample of *trans,trans*-9,11-octadecadienoic acid.

PHILADELPHIA 18, PENNA.

[24] R. T. Holman, W. O. Lundberg, W. M. Lauer and G. O. Burr, THIS JOURNAL, **67**, 1285 (1945).

[CONTRIBUTION FROM THE ORGANIC CHEMICALS DIVISION, NITRO RESERACH DEPARTMENT, MONSANTO CHEMICAL CO.]

Derivatives of 4,5-Dihydronaphtho[1,2]thiazole-2-thiol and -*s*-Triazines

BY JOHN J. D'AMICO AND MARION W. HARMAN

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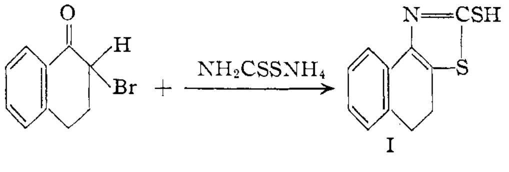
Ammonium dithiocarbamate and 2-bromo-1-tetralone reacted to give 4,5-dihydronaphtho[1,2]thiazole-2-thiol. Seventeen derivatives of this thiazolethiol were prepared. Three interesting results encountered were: (1) the reaction of cyanuric chloride with sodium dimethyldithiocarbamate or dimethylammonium dimethyldithiocarbamate gave 2-dimethylamino-4,6-bis-(dimethylthiocarbamoylthio)-*S*-triazine and 2-dimethylamino-4,6-bis-(dimethylthiocarbamoyl)-*S*-triazine, respectively; (2) potassium diethyl- or diisopropyldithiocarbamate and cyanuric chloride reacted to give 2,4,6-tris-(diethyl- or diisopropylthiocarbamoylthio)-*S*-triazine; and (3) the reaction of diethylammonium diethyldithiocarbamate or diisopropylammonium diisopropyldithiocarbamate with cyanuric chloride gave 2-diethylamino-4,6-bis-(diethylthiocarbamoylthio)-*S*-triazine and 2-diisopropylamino-4,6-bis-(diisopropylthiocarbamoylthio)-*S*-triazine, respectively.

Since 2-mercaptobenzothiazole and some of its derivatives have long been established as important accelerators for the vulcanization of rubber with sulfur, it was desirable to prepare the unknown 4,5-dihydronaphtho[1,2]thiazole-2-thiol (I) and its derivatives, and to compare their accelerator activity with that of 2-mercaptobenzothiazole and its corresponding derivatives. This evaluation will be reported elsewhere. A second objective was the preparation of cyanuryl derivatives of dialkyldithiocarbamic acids.

Ammonium dithiocarbamate reacts with 2-bromo-1-tetralone to form I. The reaction may be represented as



I

2,2-Dithiobis-(4,5-dihydronaphtho[1,2]thiazole) was prepared by the reaction of I with a 30% aqueous solution of ammonium persulfate.

The potassium salt of I reacted with N,N-diethylthiocarbamoyl chloride to give 4,5-dihydronaphtho[1,2]thiazol-2-yl diethyldithiocarbamate.

N-Cyclohexyl-4,5-dihydronaphtho[1,2]-2-thiazolesulfenamide was prepared by the oxidative condensation of I with cyclohexylamine.

Employing the elegant procedure described by Newby,[1] 4,5-(dihydronaphtho[1,2]thiazol-2-ylthio)-hydroquinone was prepared by the reaction of I with *p*-benzoquinone.

The reaction of the potassium salt of I with cyanuric chloride furnished the desired 2,4,6-tris-(4,5-dihydronaphtho)[1,2]thiazol-2-ylthio-*S*-triazine.

The zinc salt of I was obtained by treating the sodium salt of I with zinc chloride.

An acetone solution of the potassium salt of I reacted with 2,4-dinitrochlorobenzene to form 2-(2,4-dinitrophenylthio)-4,5-dihydronaphtho[1,2]-thiazole.

The reaction of an aqueous solution of the sodium salt of I with β-dimethylaminoethyl chloride gave the desired 4,5-dihydro-2-(2-dimethylaminoethylthio)-naphtho[1,2]thiazole.

2,2′-(2-Butynylenedithio)-bis-(4,5-dihydronaphtho[1,2] thiazole and 2,2′-(2-butenylenedithio)-bis-(4,5-dihydronaphtho[1,2]thiazole were prepared by the reaction of the potassium salt of I with 1,4-dichloro-2-butyne and 1,4-dichloro-2-butene, respectively.

3-(4,5-Dihydronaphtho[1,2]thiazol-2-ylthio)-pro-

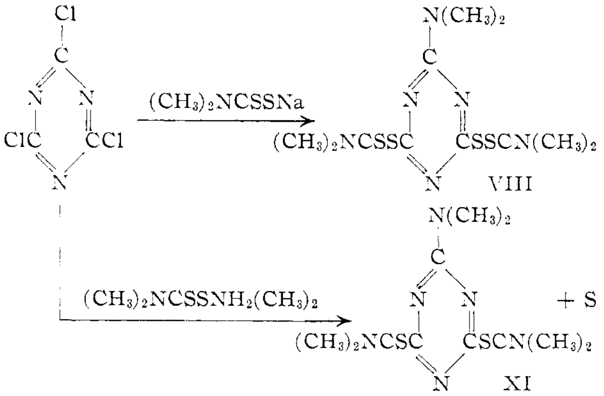
[1] T. H. Newby, U. S. Patent 2,616,871.

pionitrile was prepared by the reaction of the sodium salt of I with 2-chloropropionitrile.

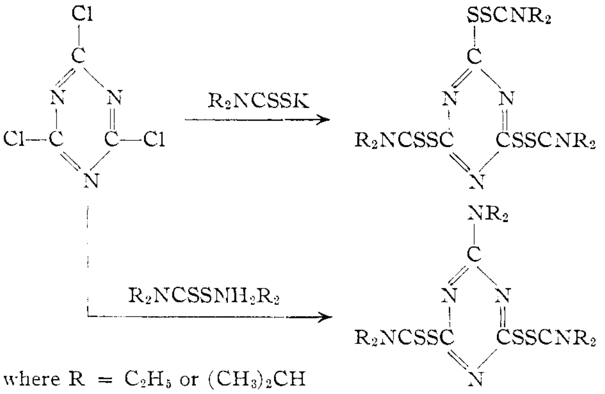
The reaction of an acetone solution of the potassium salt of I with 3-chloro-2,4-pentanedione, chloroacetone or ethyl α-chloroacetoacetate gave 3-(4,5-dihydronaphtho[1,2]thiazol-2-ylthio)-2,4-pentanedione, 2-(acetonylthio)-4,5-dihydronaphtho-[1,2]thiazole and ethyl α-(4,5-dihydronaphtho-[1,2]thiazol-2-ylthio)-acetoacetate, respectively.

The 2-allylthio-, 2-(3-chloro-2-butenylthio)- or 2-(2-chloroallylthio)-4,5-dihydronaphtho[1,2]thiazole were prepared by treating the potassium salt of I with the following compounds: allyl chloride, 1,3-dichloro-2-butene and 2,3-dichloro-1-propene.

Orthner and co-workers[2] postulated that 2-dimethylamino-4,6-bis-(dimethyl thiocarbamoylthio)-*S*-triazine (VIII) would be obtained when either sodium dimethyldithiocarbamate or dimethylammonium dimethyldithiocarbamate was treated with cyanuric chloride. Their view was not substantiated by our work. While the reaction of cyanuric chloride with sodium dimethyldithiocarbamate gave the expected VIII, 2-dimethylamino-4,6-bis-(dimethylthiocarbamoyl)-*S*-triazine (XI) was isolated as the major product when the sodium dimethyldithiocarbamate was replaced by dimethylammonium dimethyldithiocarbamate in this reaction. Molecular weight determination, sulfur, hydrogen and nitrogen analysis agreed for the proposed structure of XI. The reaction may be represented as



It was anticipated that the reaction of cyanuric chloride with potassium diethyldithiocarbamate or diisopropyldithiocarbamate would give the corresponding bis derivatives but instead the 2,4,6-tris-(diethyl- and diisopropylthiocarbamoylthio)-*S*-triazines were obtained. Substituting the above potassium salts of dialkyldithiocarbamates with diethylammonium diethyldithiocarbamate and diisopropylammonium diisopropyldithiocarbamate gave 2-diethylamino-4,6-bis-(diethylthiocarbamoylthio)-*S*-triazine and 2-diisopropylamino-4,6-bis-(diisopropylthiocarbamoylthio)-*S*-triazine, respectively, as shown in the diagram.



where R = C_2H_5 or $(CH_3)_2CH$

Experimental[3]

4,5-Dihydronaphtho[1,2]thiazole-2-thiol (I).—To a stirred slurry containing 330 g. (3.0 moles) of ammonium dithiocarbamate and 1500 ml. of ethyl alcohol was added 642 g. (2.85 moles) of 2-bromo-1-tetralone[4]; the temperature rose immediately from 26 to 73°. The reaction mixture was stirred for 18 hours and then heated at 75–78° for two hours. After cooling to 10°, the precipitate was filtered, washed with water until free of chloride and air-dried at 50°. The product, a tan colored solid, m.p. 200–204°, was obtained in 80.5% yield, m.p. after recrystallization from ethyl alcohol 206–208°.

Anal. Calcd. for $C_{11}H_9NS_2$: N, 6.39; S, 29.24. Found: N, 6.43; S, 29.30.

2,2'-Dithiobis-(4,5-dihydronaphtho[1,2]thiazole).—To an agitated suspension of 25 g. (0.114 mole) of I in 200 ml. of water was added dropwise a solution containing 28.6 g. (0.125 mole) of ammonium persulfate in 70 ml. of water over a 40-minute period at 25–30°. The reaction mixture was stirred for two additional hours. The precipitate was filtered, washed with water until the wash water was neutral to litmus, and air-dried at 50°. The product, a tan colored solid, m.p. 120–124°, was obtained in 96.5% yield. After recrystallization from ethyl alcohol it melted at 125–126°.

Anal. Calcd. for $C_{22}H_{16}N_2S_4$: N, 6.42; S, 29.38. Found: N, 6.41; S, 29.37.

4,5-Dihydronaphtho[1,2]thiazol-2-yl Diethyldithiocarbamate.—To a stirred solution at 20° containing 43.9 g. (0.2 mole) of I, 11.2 g. (0.2 mole) of potassium hydroxide and 600 ml. of acetone was added dropwise a solution containing 30.3 g. (0.2 mole) of N,N-diethylthiocarbamoyl chloride[5] in 150 ml. of acetone. The reaction mixture was stirred for 5 hours and was filtered to remove the potassium chloride. The filtrate was added to 700 g. of ice-water and stirred for 15 minutes. The resulting solid was filtered, washed with water until free of chloride and air-dried at 50°. The product, a tan-colored solid, m.p. 108–112°, was obtained in 98.5% yield. After recrystallization from ethyl alcohol it melted at 117–118°.

Anal. Calcd. for $C_{16}H_{18}N_2S_3$: N, 8.38; S, 28.76. Found: N, 8.16; S, 28.67.

N-Cyclohexyl-4,5-dihydronaphtho[1,2]-2-thiazolesulfenamide.—A solution of 32.4 g. of iodine and 34.8 g. of potassium iodide in 420 ml. of water was added dropwise at 25–30° in 1.5 hours to a stirred solution containing 24 g. (0.11 mole) of I, 35.2 g. (0.22 mole) of 25% aqueous sodium hydroxide, 100 g. of water and 108.9 g. (1.1 moles) of cyclohexylamine. The reaction mixture was stirred for an additional hour and then extracted with 500 ml. of ethyl ether. The ether solution was washed with water until the washings were neutral to litmus and dried over sodium sulfate. The ether was removed *in vacuo*. The product, m.p. 63–66°, was obtained in 86% yield, m.p. after recrystallization from ethyl alcohol 74–76°.

Anal. Calcd. for $C_{17}H_{20}N_2S_2$: N, 8.85; S, 20.26. Found: N, 8.35; S, 20.35.

(4,5-Dihydronaphtho[1,2]thiazol-2-ylthio)-hydroquinone.—A suspension of 27 g. (0.25 mole) of benzoquinone and 185 ml. of methyl alcohol was heated to 50°. The resulting solution was cooled to 0° and to the recrystallized benzoquinone, a suspension containing 54.9 g. (0.25 mole) of I and 300 ml. of methyl alcohol was added in one portion.

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(2) L. Orthner, L. G. Werk and M. Bogemann, U. S. Patent 2,061,-520.

(3) All melting points were taken upon a Fisher–Johns block and are uncorrected.

(4) A. L. Wilds and W. J. Close, THIS JOURNAL, **68**, 87 (1946).

(5) Kindly supplied by Sharples Chemicals, Inc., Philadelphia, Pa.

The stirred reaction mixture was held at 5–10° for two hours and then heated to 60°. The reaction mixture was added to 1000 ml. of hot water. After stirring for 30 minutes, the solid was filtered, washed with 500 ml. of hot water and air-dried at 50°. The product, m.p. 225–230°, was obtained in 90.5% yield, m.p. after recrystallization from chloroform 236–238°.

Anal. Calcd. for $C_{17}H_{13}NO_2S_2$: N, 4.28; S, 19.59. Found: N, 4.53; S, 19.54.

2,4,6-Tris-(4,5-dihydronaphtho[1,2]thiazol-2-ylthio)-*S*-triazine.—To a solution containing 20 g. (0.0914 mole) of I, 300 ml. of acetone and 5.1 g. (0.0914 mole) of potassium hydroxide was added dropwise 5.6 g. (0.0305 mole) of cyanuric chloride[6] dissolved in 400 ml. of acetone. After heating at 50–55° for five hours and cooling to 25°, the precipitate was filtered, washed with water until free of chloride and air-dried at 50°. The product, m.p. 230–235°, was obtained in 98.5% yield. After recrystallization from benzene, it melted at 238–239°.

Anal. Calcd. for $C_{36}H_{24}N_6S_6$: N, 11.47; S, 26.25. Found: N, 11.16; S, 25.93.

Zinc Salt of I.—To an agitated solution at 80° containing 20 g. (0.0914 mole) of I, 14.6 g. (0.0914 mole) of 25% aqueous sodium hydroxide and 2000 ml. of water was added in one portion 6.2 g. (0.0457 mole) of zinc chloride dissolved in 100 ml. of water. External heating was discontinued and the reaction mixture was stirred for two additional hours. The resulting solid was filtered, washed with water until the wash water was neutral to litmus and air-dried at 50°. The product, m.p. 180–190° dec., was obtained in 99% yield.

Anal. Calcd. for $C_{22}H_{16}N_2S_4Zn$: Zn, 13.02. Found: Zn, 13.41.

2-(2,4-Dinitrophenylthio)-4,5-dihydronaphtho[1,2]thiazole.—To a stirred solution containing 21.9 g. (0.1 mole) of I, 5.6 g. (0.1 mole) of potassium hydroxide and 300 ml. of acetone was added in one portion 20.3 g. (0.1 mole) of 2,4-dinitrochlorobenzene. The stirred reaction mixture was heated at 50–56° for 5 hours. After cooling to room temperature, the reaction mixture was poured onto 700 g. of crushed ice. After stirring for one hour, the solid was filtered, washed with water until the washings were neutral to litmus and air-dried at 50°. The product, m.p. 169–172°, was obtained in 80.5% yield, m.p. after recrystallization from ethyl acetate 173–174°.

Anal. Calcd. for $C_{17}H_{11}N_3O_4S_2$: S, 16.64. Found: S, 16.78.

4,5-Dihydro-2-(2-dimethylaminoethylthio)-naphtho[1,2]-thiazole.—To a stirred solution containing 54.9 g. (0.25 mole) of I, 40 g. (0.25 mole) of 25% aqueous sodium hydroxide and 500 ml. of water was added 36 g. (0.25 mole) of β-dimethylaminoethyl chloride hydrochloride and 40 g. (0.25 mole) of 25% aqueous sodium hydroxide. The stirred reaction mixture was heated at 50–60° for 5 hours. After cooling to 25° the reaction mixture was extracted with 500 ml. of ether. The ether extract was washed with water until the washings were neutral to litmus and dried over sodium sulfate. The ether was removed *in vacuo*. The product, an amber colored oil, was obtained in 82.5% yield.

Anal. Calcd. for $C_{15}H_{18}N_2S_2$: N, 9.65; S, 22.08. Found: N, 9.42; S, 22.14.

2,2′-(2-Butynylenedithio)-bis-(4,5-dihydronaphtho[1,2]-thiazole) and 2,2′-(2-Butenylenedithio)-bis-(4,5-dihydronaphtho[1,2]thiazole).—A solution of the potassium salt of I was prepared by mixing 43.8 g. (0.2 mole) of I, 11.2 g. (0.2 mole) of potassium hydroxide and 500 ml. of acetone. To this solution at room temperature 12.3 g. (0.1 mole) of 1,4-dichloro-2-butyne[7] or 12.5 g. (0.1 mole) of 1,4-dichloro-2-butene[8] was added dropwise. An exothermic reaction set in causing the temperature to rise from 25 to 38°. The stirred reaction mixture was heated at 50–56° for 24 hours. The reaction mixture was poured onto 500 g. of crushed ice. After stirring for 10 minutes, the solid was filtered, washed with water until the washings were neutral to litmus and air-dried at room temperature. The former compound m.p. 145–146° after recrystallization from ethyl acetate, and the latter compound, m.p. 113–114° after recrystallization from ethyl alcohol, were obtained in yields of 98.2 and 98.5%, respectively.

Anal. Calcd. for $C_{26}H_{20}N_2S_4$: N, 5.73; S, 26.24. Found: N, 5.71; S, 25.99. Calcd. for $C_{26}H_{22}N_2S_4$: N, 5.71; S, 26.14. Found: N, 5.62; S, 25.78.

3-(4,5-Dihydronaphtho[1,2]thiazol-2-ylthio)-propionitrile.—To a stirred solution containing 109.6 g. (0.5 mole) of I, 500 ml. of water and 80 g. (0.5 mole) of 25% aqueous sodium hydroxide was added 44.8 g. (0.5 mole) of 2-chloropropionitrile. The reaction mixture was heated at 50–60° for two hours and maintained at 25–30° for three hours. To the stirred reaction mixture 400 ml. of ethyl ether was added. After stirring for 15 minutes the reaction mixture was filtered. The solid was air-dried at room temperature. This product (56 g.), m.p. 206–208° after recrystallization from ethyl alcohol, was identified as unreacted I. A mixed melting point with an authentic sample gave no depression.

The filtrate was washed with water until the washings were neutral to litmus, and dried over sodium sulfate. The ether was removed *in vacuo*. The desired product, an amber colored oil, was obtained in 85.2% yield (conversion 38.3%).

Anal. Calcd. for $C_{14}H_{12}N_2S_2$: N, 10.28; S, 23.54. Found: N, 9.95; S, 24.10.

3-(4,5-Dihydronaphtho[1,2]thiazol-2-ylthio)-2,4-pentanedione (II), 2-(Acetonylthio)-4,5-dihydronaphtho[1,2]thiazole (III) and Ethyl α-4,5-Dihydronaphtho[1,2]thiazol-2-ylthio)-acetoacetate (IV).—To a stirred solution containing 33 g. (0.15 mole) of I, 8.4 g. (0.15 mole) of potassium hydroxide and 300 ml. of acetone was added in one portion 20.2 g. (0.15 mole) of 3-chloro-2,4-pentanedione,[9] 14 g. (0.1 mole) of chloroacetone, or 24.8 g. (0.15 mole) of ethyl α-chloroacetoacetate.[10] An exothermic reaction set in causing the temperature to rise from 20 to about 50°. The reaction mixture was stirred for five hours and the potassium chloride removed by filtration. For II and III the filtrate was added to 500 g. of crushed ice. After stirring for 20 minutes the solid was filtered, washed with water until the wash water was neutral to litmus and air-dried at room temperature.

For IV the acetone of the filtrate was removed *in vacuo*. The resulting residue was dissolved in 500 ml. of ethyl ether. The ether solution was washed with water until the washings were neutral to litmus and dried over sodium sulfate. Upon removal of the ether *in vacuo* the product, a viscous amber oil, was obtained in 90.3% yield. The data are summarized in Table I.

2-Allylthio-4,5-dihydronaphtho[1,2]thiazole (V), 2-(3-Chloro-2-butenylthio)-4,5-dihydronaphtho[1,2]thiazole (VI) and 2-(2-Chloroallylthio)-4,5-dihydronaphtho[1,2]-thiazole (VII).—To a stirred solution containing 21.9 g. (0.1 mole) of I, 5.6 g. (0.1 mole) of potassium hydroxide and 300 ml. of acetone was added 0.1 mole of allyl chloride, 1,3-dichloro-2-butene[11] or 2,3-dichloro-1-propene.[12] The stirred reaction mixture was heated at 55–56° for five hours and filtered to remove potassium chloride. For V, the acetone was removed *in vacuo* at maximum temperature of 90°.

For VI and VII, the filtrate was added to 500 g. of crushed ice. After stirring for 15 minutes, the resulting solid was filtered, washed with water until the washings were neutral to litmus and air-dried at room temperature. The data are summarized in Table II.

Cyanuryl Derivatives of Dialkyldithiocarbamic Acids (VIII–XIII).—The reaction was carried out in a manner similar to that described by Orthner and co-workers except with the following modifications. One mole of either the sodium, potassium or amine salt of the dialkyldithiocarbamic acid (Table III) was dissolved in 1000 ml. of acetone at 56°. External heating was removed and to the stirred solution was added dropwise a solution containing 61.5 g. (0.33 mole) of cyanuric chloride in 500 ml. of acetone. The stirred reaction mixture was heated at 55–56° for four hours. For

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(6) Kindly supplied by American Cyanamid Company, New York, N. Y.

(7) Kindly supplied by General Aniline and Film Corp., New York, N. Y.

(8) Kindly furnished by Carbide and Carbon Chemicals Co., New York, N. Y.

(9) E. R. Buchman and E. M. Richardson, THIS JOURNAL, **67**, 395 (1945).

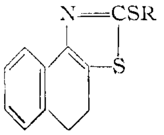
(10) E. R. Buchman and E. M. Richardson, *ibid.*, **61**, 891 (1939).

(11) Kindly furnished by E. I. du Pont de Nemours and Co., Wilmington, Del.

(12) Kindly supplied by Shell Chemical Corp., Emeryville, Calif.

TABLE I

3-(4,5-DIHYDRONAPHTHO[1,2]THIAZOL-2-YLTHIO)-2,4-PENTANEDIONE, 2-(ACETONYLTHIO)-4,5-DIHYDRONAPHTHO[1,2]THIAZOLE AND ETHYL α-(4,5-DIHYDRONAPHTHO[1,2]THIAZOLE-2-YLTHIO)-ACETOACETATE

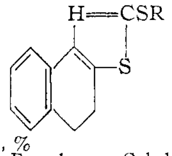


No.	R	Yield, % crude	M.p., °C.	Empirical formula	Nitrogen, %		Sulfur, %	
					Calcd.	Found	Calcd.	Found
II	CH(COCH$_3$)$_2$	90.5	105–106[a]	$C_{16}H_{15}NO_2S_3$	4.41	4.36	20.20	20.08
III	CH$_2$COCH$_3$	91.1	68–69[a]	$C_{14}H_{13}NOS_2$	5.09	5.06	23.29	23.11
IV	CHCOCH$_3$ | COOC$_2$H$_5$	90.3	Oil	$C_{17}H_{17}NO_3S_3$	4.03	3.97	18.46	18.57

[a] Recrystallization from ethyl alcohol.

TABLE II

2-(ALLYLTHIO OR CHLOROALKENYLTHIO)-4,5-DIHYDRONAPHTHO[1,2]THIAZOLE

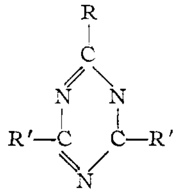


No.	R	Yield, % crude	M.p., °C.	Empirical formula	Nitrogen, %		Sulfur, %	
					Calcd.	Found	Calcd.	Found
V	CH$_2$CH=CH$_2$	96.5	Oil	$C_{14}H_{13}NS_2$	5.40	5.03	...	...
VI	CH$_2$CH=CClCH$_3$	88.0	63–65	$C_{15}H_{14}ClNS_2$	4.55	4.59	20.83	20.87
VII	CH$_2$CCl=CH$_2$	98.7	60–61[a]	$C_{14}H_{12}ClNS_2$	4.77	4.79	21.83	21.63

[a] Recrystallization from ethyl alcohol.

TABLE III

CYANURYL DERIVATIVES OF DIALKYLDITHIOCARBAMIC ACIDS



No.	Alkali or amine salt of dialkyldithiocarbamic acid	R	R′	M.p., °C.	Yield, % crude	Empirical formula	Nitrogen, %		Sulfur, %	
							Calcd.	Found	Calcd.	Found
VIII	(CH$_3$)$_2$NCSSNa	(CH$_3$)$_2$N	(CH$_3$)$_2$NCSS	172–173[a]	48.0	$C_{11}H_{18}N_6S_4$	23.21	22.90	35.43	35.39
IX	(C$_2$H$_5$)$_2$NCSSK	(C$_2$H$_5$)$_2$NCSS	(C$_2$H$_5$)$_2$NCSS	136–137	39.5	$C_{18}H_{30}N_6S_6$	16.08	15.78	36.79	36.72
X	[(CH$_3$)$_2$CH]$_2$NCSSK	[(CH$_3$)$_2$CH]$_2$NCSS	[(CH$_3$)$_2$CH]$_2$NCSS	119–120[a]	31.4	$C_{24}H_{42}N_6S_6$	13.85	13.65	31.70	31.64
XI	(CH$_3$)$_2$NCSSNH$_2$(CH$_3$)$_2$	(CH$_3$)$_2$N	(CH$_3$)$_2$NCS	163–164[b]	84.3	$C_{11}H_{18}N_6S_2$[d]	28.16	28.91	21.49	21.56
XII	(C$_2$H$_5$)$_2$NCSSNH$_2$(C$_2$H$_5$)$_2$	(C$_2$H$_5$)$_2$N	(C$_2$H$_5$)$_2$NCSS	115–116[c]	56.9	$C_{17}H_{30}N_6S_4$	18.81	18.42	28.71	28.69
XIII	[(CH$_3$)$_2$CH]$_2$NCSSNH$_2$[CH-(CH$_3$)$_2$]$_2$	[(CH$_3$)$_2$CH]$_2$N	[(CH$_2$)$_2$CH]$_2$NCSS	218[a]	39.9	$C_{23}H_{42}N_6S_4$	15.83	15.64	24.16	23.88

[a] Recrystallization from dioxane. [b] Recrystallization from benzene. [c] Recrystallization from ethyl alcohol. [d] Calcd. H, 6.08; mol. wt., 298.4. Found: H, 6.36; mol. wt., 301.0.

VIII–XII the reaction mixture was filtered at 50–56° to remove either the alkali chloride or the amine hydrochloride. The volume of the filtrate was concentrated to about 100 ml. by vacuum distillation. To the stirred residue 200 ml. of heptane was added. After stirring the mixture for 20 minutes the solid was collected by filtration, washed with water until the washings were free of chloride and air-dried at 50°.

For XIII, after cooling to 25°, the reaction mixture was filtered, washed with water until the wash water was neutral to litmus and air-dried at 50°. The data are summarized in Table III.

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