PREPARATION AND PROPERTIES OF SOME β-(THIO LACTONES)

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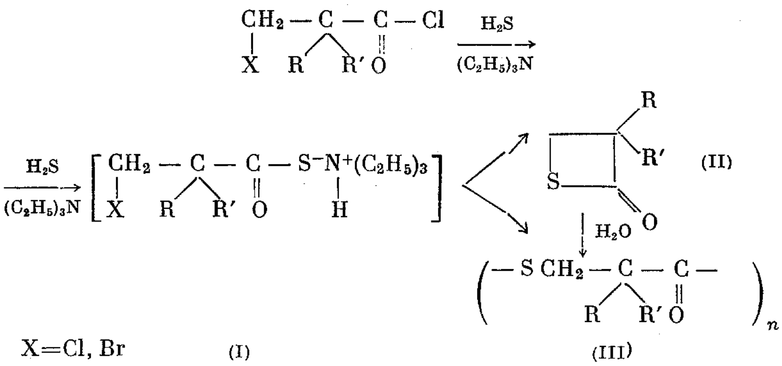
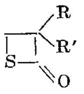
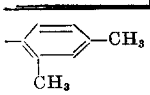
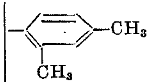
We have previously described the preparation of β-(thio lactones) from α-substituted β-halopropionyl chlorides [1]. The further application of this method led to the synthesis of a number of compounds of this class with different substituents in the α-position:



The yields of the β-(thio lactones) obtained, their physical constants, and their elementary analysis figures are given in the table.

The yields of the β-(thio lactones) are related to their stabilities, which depend on the character of the substituents in the α-position. Thus, α,α-di-2,4-xylyl-β-propiothiolactone [3-mercapto-2,2-di-2,4-xylylpropionic acid β-(thio lactone)] was prepared by this method from 3-chloro-2,2-di-2,4-xylylpropionyl chloride in 84.5% yield. In this case the reaction was usually accompanied by side reactions. The main by-product was the poly(thio ester) (III), which was probably formed both by the polymerization of the already formed β-(thio lactone) (II) and from the intermediate salt (I), which could either undergo cyclization with formation of (II) or react with another similar



R	R′	Yield, %	B.p., °C (p, mm)	n_D^{20}	d_4^{20}	MR		Found, %			Calc., %		
						Found	Cal.	C	H	S	C	H	S
		84,5	117—119 *					77,18	6,85	10,80	77,00	6,75	10,81
CH_3	SCH_3	55,7	56—58 (0,015)	1,5247	1,175	38,58	39,53	41,28	5,86		40,54	5,40	
H	CH_3	68	43—46 (10)	1,4975	1,127	26,50	26,94	46,28	5,99	29,51	47,05	5,88	31,37
CH_3	Cl†	55,5	49 (6)	1,5198	1,29	32,10	31,80	34,56	3,70	22,53	35,16	3,66	23,44
CH_3	Br	25,6	47—50 (3)	1,5478	1,64	35,05	34,70	27,25	3,03		26,51	2,76	

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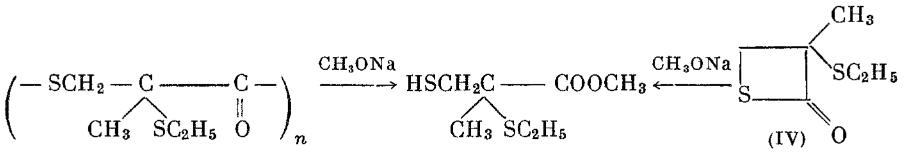
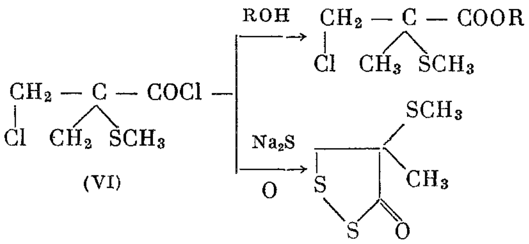
* M.p.

† Found: Cl 26.23%; calculated: Cl 26.00%.

molecule and so start a polymer chain. Hence, in the preparation of β-(thio lactones) it is necessary to work in absence of moisture and at high dilution. By the action of sodium methoxide on the polymer formed in the course of the preparation of α-(ethylthio)-α-methyl-β-propiothiolactone we obtained methyl 2-(ethylthio)-3-mercapto-2-methylpropionate, identical to the ester formed by the cleavage of (IV):

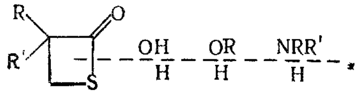


We have previously obtained a quantiative yield of α,α-diphenyl-β-propiothiolactone by the action of sodium sulfide in alcohol on 3-halo-2,2-diphenylpropionic acids [1]. However, an attempt to use a solution of sodium sulfide in alcohol in the preparation of α-methyl-α-(methylthio)-β-propiothiolactone led to 3-chloro-2-methyl-2-(methylthio)propionic ester and a substance which was probably 4-methyl-4-(methylthio)-1,2-dithiolan-3-one:



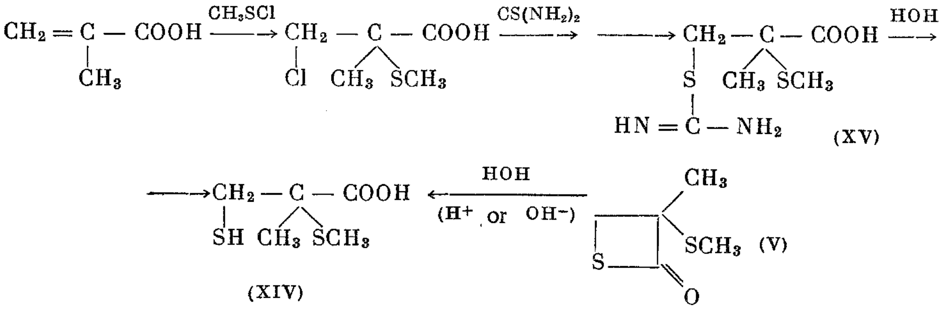
The use of sodium sulfide as a suspension in acetone instead of hydrogen sulfide gave lower yields of β-(thio lactones). α-Methyl- and α-halo-α-methyl-β-propiothiolactones readily polymerize even when kept for a short time in the cold. Caustic alkalis, alkoxides, and other alkaline agents bring about instantaneous exothermic polymerization. α-(Alkylthio)-α-methyl-β-propiothiolactones are relatively more stable and may be kept indefinitely. However, to avoid polymerization in the course of the cleavage of these β-(thio lactones) with alkoxides it is necessary to add the β-(thio lactone) to a highly diluted solution of the sodium alkoxide in the corresponding alcohol with cooling.

Further study of the properties of β-(thio lactones) again confirmed that they react with nucleophilic reagents only with opening of the four-membered ring at the S-carbonyl bond [1-7] in accordance with the scheme



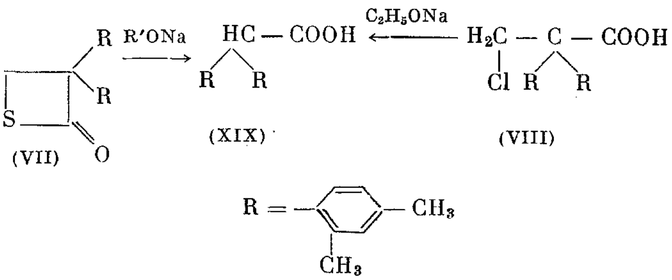
The aminolysis of β-(thio lactones) leads to β-mercapto carboxamides, their alcoholysis to β-mercapto esters, and their hydrolysis to β-mercapto acids.

The hydrolysis of α-methyl-α-(methylthio)-β-propiothiolactone (V) in presence of hydrochloric acid requires more severe conditions than its alkaline hydrolysis. The 3-mercapto-2-methyl-2-(methylthio)propionic acid obtained by the hydrolysis of (V) was prepared also as follows:



This confirms that alkanesulfenyl chlorides add to α,β-unsaturated acids with formation mainly of α-(alkylthio) β-halo carboxylic acids [8, 9]. When an attempt was made to bring about the cleavage of (V) with thiourea, the starting substances were recovered unchanged.

The reaction of α,α-di-2,4-xylyl-β-propiothiolactone with sodium ethoxide or sodium butoxide led to the previously described [10] di-2,4-xylylacetic acid, which we prepared also by the reaction of 3-chloro-,2,2-di-2,4-xylylpropionic acid with sodium ethoxide:



Analogous cleavage of a carbon-carbon bond with formation of diphenylacetic acid was observed in the reaction of sodium methoxide with α,α-diphenyl-β-propiolactone, with 3-halo-2,2-diphenylpropionic acids, and with 2,2-diphenylhydracrylic esters [11].

EXPERIMENTAL

General Method for the Preparation of β-(Thio Lactones). 0.2 mole of the β-chloro* or β-bromo† acid chloride was dissolved in 640 ml of methylene chloride, and dry hydrogen sulfide was passed in for one hour at −20°. Then, in a stream of hydrogen sulfide at the same temperature 40.4 g of triethylamine in 100 ml of dry ether was added dropwise. The mixture was left for one hour at room temperature and poured into 800 ml of ether. The precipitated triethylamine hydrochloride was filtered off, and the solution was washed with water and dried with magnesium sulfate. Solvent was evaporated, and the residue was vacuum-distilled. For yields, physical constants, and elementary analysis figures see the table.

The reaction of 3-chloro-2-methyl-2-(methylthio)propionyl chloride (VI) (for preparation see [9] with $Na_2S \cdot 9H_2O$ in acetone gave α-methyl-α-(methylthio)-β-propiothiolactone in 30% yield. α-Chloro-α-methyl-β-propiothiolactone was preparared analogously in 8.8% yield.

In the reaction of (VI) in CH_2Cl_2 with an equivalent amount of $Na_2S \cdot 9H_2O$ in methanol, (V) was not isolated. After the addition of water ether extraction gave methyl 3-chloro-2-methyl-2-(methylthio)propionate, apparently containing traces of (V), in 60% yield; b.p. 52-53° (3-4 mm); n_D^{20} 1.4973. Found: C 39.45; H 6.02%. $C_6H_{11}ClO_2S$. Calculated: C 40.82; H 6.17%.

On acidification of the aqueous layer a chlorine-free oil, b.p. 70-90° (0.06 mm), separated. When kept in a refrigerator the oil was converted into a crystalline substance which did not contain a mercapto group and was probably 4-methyl-4-(methylthio)-1,2-dithiolan-3-one, m.p. 48-50° (from absolute methanol). Found: C 33.71; H 4.44; S 53.11%. $C_5H_8OS_3$. Calculated: C 33.33; H 4.44; S 53.44%.

α,α-Di-2,4-xylyl-β-propiothiolactone (VII). 6.26 g of PCl_5 was added to a solution of 9.5 g of 3-chloro-2,2-di-2,4-xylylpropionic acid (VIII) in 80 ml of dry benzene, and the mixture was heated for 90 min at 70°; benzene and $POCl_3$ were then vacuum-distilled off. The residual acid chloride was dissolved in 80 ml of CH_2Cl_2, H_2S was passed for one hour at −20°, and then 8.73 ml of triethylamine in 30 ml of dry ether was added dropwise in a stream of H_2S at the same temperature. The mixture was left for two hours at room temperature, 200 ml of ether was added, and triethylamine hydrochloride was filtered off. The solution was washed with water, solvent was removed in a vacuum, and the residue was recrystallized from absolute alcohol or heptane. For the yield, melting point, and results of elementary analysis see the table.

3,3'-Dithiobis [N-isobutyl-2-methylpropionamide] (IX). A solution of 1.27 g of α-methyl-β-propiothiolactone (X) in 20 ml of acetonitrile was added dropwise to a solution of 1.82 g of isobutylamine in 20 ml of acetoni-

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* For the preparation of α-methyl-α-(methylthio)- and α-chloro-α-methyl-β-propiothiolactones.

† For the preparation of α-methyl- and α-bromo-α-methyl-β-propiothiolactones.

trile, the mixture was boiled for five hours, solvent was evaporated in air, and the residue, which did not contain a mercapto group, was washed with dilute hydrochloric acid and recrystallized from acetonitrile. The yield of (IX) was 1.89 g (87.1%); m.p. 139-141°. Found: C 55.35; H 9.27; N 8.11; S 18.37%. $C_{16}H_{32}N_2O_2S_2$. Calculated: C 55.17; H 9.19; N 8.04; S 18.39%.

Analogously, from 1.36 g of (VII) and 0.79 ml of isobutylamine in 70 ml of acetonitrile we obtained 2.62 g (88.7%) of 3,3'-dithiobis[N-isobutyl-2,2-di-2,4-xylylpropionamide], m.p. 156-158.5° (from acetonitrile). Found: C 74.93; H 8.12; N 3.74; S 8.84%. $C_{46}H_{60}N_2O_2S$. Calculated: C 75.00; H 8.15; N 3.80; S 8.70%.

From 1.02 g of (X) and 1.37 g of anthranilic acid in 30 ml of acetonitrile we obtained 2'-carboxy-3-mercapto-2-methylpropionanilide, m.p. 134-135° (from water). Found: C 55.10; H 5.41; N 5.99; S 13.50%. $C_{11}H_{13}NO_3S$. Calculated: C 55.23; H 5.43; N 5.85; S 13.39%.

N-2-Hydroxyethyl-3-mercapto-2,2-diphenylpropionamide. 10 ml of 2-aminoethanol was added to a solution of 0.96 g of α,α-diphenyl-β-propiothiolactone (for preparation see [1]) in 10 ml of acetonitrile, and after six days 30 ml of water was added; when a precipitate formed which contained a mercapto group (test with iodine), it was recrystallized from alcohol. Yield 1 g (80.5%); m.p. 156-158°. Found: C 67.85; H 6.31; N 4.85; S 10.52%. $C_{17}H_{19}NO_2S$. Calculated: C 67.77; H 6.31; N 4.65; S 10.63%.

3,3'-Dithiobis[N-cyclohexyl-2-(ethylthio)-2-methylpropionamide]. 1 g of cyclohexylamine in 10 ml of dry ether was added to a solution of 0.81 g of α-(ethylthio)-α-methyl-β-propiothiolactone (IV) in 10 ml of dry ether. After 30 min the solution gave a positive test for the mercapto group. After seven days ether was evaporated in air, and the residue, which did not contain the mercapto group, was washed with dilute hydrochloric acid and recrystallized from ether; m.p. 104-106°. Found: C 55.38; H 8.46; N 5.38; S 24.60%. $C_{24}H_{44}N_2O_2S_4$. Calculated: C 55.54; H 8.76; N 5.49; S 23.78%.

Analogously, we obtained 3,3'-dithiobis[N-cyclohexyl-2-methylpropionamide], m.p. 203-204° (from acetonitrile). Found: C 59.91; H 8.99; N 7.18; S 15.98%. $C_{20}H_{36}N_2O_2S_2$. Calculated: C 60.00; H 9.00; N 7.00; S 16.00%.

From 1.35 g of (X) and excess of benzylamine in dry ether we obtained 3,3'-dithiobis[N-benzyl-2-methylpropionamide], m.p. 143-144.5° (from acetonitrile). Found: C 63.53; H 6.78; N 6.76; S 15.51%. $C_{22}H_{28}N_2O_2S_2$. Calculated: C 63.46; H 6.73; N 6.73; S 15.38%.

3,3"-Dithiobis[4'-bromo-2-methylpropionanilide] (XI). 1.53 g of (X) in 10 ml of acetonitrile was added to a solution of 3.44 g of p-bromoaniline in 10 ml of acetonitrile. The mixture was boiled for six hours, solvent was removed, and the oily residue was washed with dilute hydrochloric acid and then oxidized with iodine in ethereal solution. After the removal of ether we obtained (XI); yield 2.55 g (62.1%); m.p. 191.5-193° (from acetonitrile). Found: C 44.37; H 4.27; Br 28.49; N 5.66; S 12.00%. $C_{20}H_{22}Br_2N_2O_2S_2$. Calculated: C 43.95; H 4.03; Br 29.33; N 5.13; S 11.72%.

Analogously, from 1.48 g of (V) and 2.21 ml of cyclohexylamine we obtained 3,3'-dithiobis[N-cyclohexyl-2-methyl-2-(methylthio)propionamide]. Yield 2.3 g (96%); m.p. 118-120° (from acetonitrile). Found: C 53.16; H 8.01; N 5.94; S 25.54%. $C_{22}H_{20}N_2O_2S_4$. Calculated: C 53.65; H 8.13; N 5.69; S 26.01%.

3-Mercapto-2-methyl-p-propionotoluidide (XII). 1.86 g of (X) in 25 ml of dry ether was added dropwise to a solution of 3.23 g of p-toluidine in 30 ml of dry ether. On the next day the solution was boiled for five hours, and the precipitated 3,3'-dithiobis[2-methyl-p-propionotoluidide] was recrystallized from acetonitrile; yield 0.29 g (7.7%); m.p. 193-194°. Found: C 63.17; H 6.66; N 6.78; S 15.54%. $C_{22}H_{28}N_2O_2S_2$. Calculated: C 63.46; H 6.73; N 6.73; S 15.38%.

After the removal of ether we obtained 2.52 g (66.2%) of (XII), m.p. 91-92° (from a mixture of petroleum ether and methylene chloride). Found: C 63.03; H 6.97; N 6.70; S 15.14%. $C_{11}H_{15}NOS$. Calculated: C 63.15; H 7.17; N 6.69; S 15.31%.

3-Mercapto-4'-methoxy-2-methylpropionanilide (XIII). A solution of 2.46 g of p-anisidine in 20 ml of acetonitrile was added to a solution of 1.53 g of (X) in 10 ml of acetonitrile. After five days solvent was removed, and the residue was washed with dilute HCl and dissolved in 1 N NaOH. The solution was washed with chloroform, and acidification then gave 2.53 g of (XIII), m.p. 96° (from a mixture of petroleum ether and methylene chloride). Found: C 58.63; H 6.68; N 6.25; S 14.22%. $C_{11}H_{15}NO_2S$. Calculated: C 58.66; H 6.66; N 6.28; S 14.22%.

From the chloroform solution we isolated the corresponding disulfide, m.p. 171-173.5° (from acetonirile). Found: C 58.10; H 6.32; N 6.27; S 15.46%. $C_{22}H_{28}N_2O_4S_2$. Calculated: C 58.92; H 6.25; N 6.25; S 14.28%. The total yield of (XIII) and the disulfide was 75%.

3-Mercapto-2-methyl-2-(methylthio)propionic Acid (XIV). a) 4.4 g of (V) in 15 ml of methanol was added dropwise in a stream of nitrogen to a solution of 3.6 g of NaOH in a mixture of 45 ml of water and 10 ml of methanol. After 5 min the solution gave a positive test for the mercapto group. The mixture was heated for one hour at 70°. On the next day water was added and the mixture was acidified with HCl; the oil that separated was extracted with ether. Ether was removed, and we obtained 3.95 g (7.94%) of (XIV); b.p. 100° (0.4 mm); m.p. 71-72° (from benzene). Found: C 36.25; H 6.04; S 38.34%. $C_5H_{10}O_2S_2$. Calculated: C 36.14; H 6.02; S 38.55%.

b) 2.1 g of (V) was dissolved in 45 ml of methanol, and 15 ml of concentrated HCl was added. The mixture was boiled for 18 h. It was then extracted with ether, and the combined ether extracts were extracted with sodium bicarbonate solution. The bicarbonate extract was acidified with 1 N HCl and extracted with ether; the ether extract was dried with $MgSO_4$. Ether was removed and we obtained 0.36 g (13.6%) of (XIV), m.p. 67-70°, undepressed by admixture of (XIV) prepared by method (a).

c) A mixture of 5 g of 3-chloro-2methyl-2-(methylthio)propionic acid (for preparation see [9]) and 2.3 g of thiourea in 25 ml of alcohol was boiled for five hours. The crystals that came down after a few days were filtered off, dissolved in water, and treated with sodium bicarbonate solution. The 3-(amidinothio)-2-methyl-2-(methylthio)propionic acid (XV) then isolated was recrystallized from alcohol; m.p. 172-173°. Found: C 33.95; H 6.23; S 29.35%. $C_6H_{12}N_2O_2S_2$. Calculated: C 34.61; H 5.77; S 30.76%.

(XV) was dissolved in alkali with slight warming. On acidification of the alkaline solution (XIV) separated as an oil, which solidified on standing; m.p. 71° (from water). Found: C 36.30; H 6.09; S 37.56%. $C_5H_{10}O_2S_2$. Calculated: C 36.14; H 6.02; S 38.55%. Mixtures with samples of (XIV) prepared by methods (a) and (b) melted without depression.

From the mother liquors after treatment with iodine we isolated the disulfide corresponding to (XIV); m.p. 139-142° (from water). Found: C 36.18; H 5.28; S 38.78%. $C_{10}H_{18}O_4S_4$. Calculated: C 36.36; H 5.45; S 38.78%.

Methyl 3-Mercapto-2-methylpropionate (XVI). 3.36 g of (X) in 50 ml of dry methanol was added dropwise in a stream of nitrogen to a solution of sodium methoxide (from 0.76 of sodium) in 250 ml of absolute methanol at −20°. The mixture, which gave a positive test for the mercapto group, was left to the next day. Methanol was removed in a vacuum with heating, water was added to the residue, the mixture was acidified with 1 N HCl, the oil that separated was extracted with ether, and the extract was dried with $MgSO_4$. Vacuum distillation gave (XVI)*; b.p. 63.5-64° (14 mm); n_D^{20} 1.4600; d_4^{20} 1.06. Found: C 44.73; H 7.41; S 23.93%; MR 34.62. $C_5H_{10}O_2S$. Calculated: C 44.77; H 7.46; S 23.88%; MR 34.64.

Methyl 3-Mercapto-2-methyl-2-(methylthio)propionate (XVII). A solution of 4.4 g of (V) in 50 ml of absolute methanol was added dropwise in a stream of nitrogen to a solution of sodium methoxide (from 0.76 g of sodium) in 150 ml of absolute methanol. The mixture was heated for 90 min at 50°. Methanol was removed in a vacuum, water was added, and the mixture was acidified with HCl and extracted with ether. The extract was dried with $MgSO_4$, and vacuum distillation gave 3.4 g (63.7%) of (XVII); b.p. 64-68° (3 mm); n_D^{20} 1.5177; d_4^{20} 1.16. Found: C 39.99; H 6.64%; MR 46.99. $C_6H_{12}O_2S_2$. Calculated: C 40.00; H 6.66%; MR 47.23.

By the analogous cleavage of (IV) with sodium ethoxide we obtained ethyl 2-(ethylthio)-3-mercapto-2-methylpropionate; b.p. 58-60° (0.07 mm); n_D^{20} 1.5078; d_4^{20} 1.085. Found: C 46.40; H 7.76; S 30.20%; MR 57.12. $C_8H_{16}O_2S_2$. Calculated: C 46.15; H 7.69; S 30.76%; MR 56.47.

By the cleavage of (V) with sodium butoxide we obtained butyl 3-mercapto-2-methyl-2-(methylthio) propionate; yield 52.4%; b.p. 75° (0.08 mm); n_D^{20} 1.5011; d_4^{20} 1.08. Found: C 47.49; H 8.12; S 29.22%; MR 60.59. $C_9H_{18}O_2S_2$. Calculated: C 48.64; H 8.10; S 28.82%; MR 60.39.

Analogously, from 3.24 g of (IV) we obtained 2.03 g (52.5%) of methyl 2-(ethylthio)-3-mercapto-2-methylpropionate (XVIII); b.p. 60-61° (0.1 mm); n_D^{20} 1.5139; d_4^{20} 1.11. Found: C 43.27; H 7.28; S 31.86%; MR 52.49. $C_7H_{14}O_2S_2$. Calculated: C 43.29; H 7.21; S 32.98%; MR 51.85.

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* This has been prepared previously by the addition of hydrogen sulfide to methyl methacrylate, but the constants are not given [12].

From 4.05 g of the polymer formed in the course of the preparation of (IV) by analogous treatment we obtained 1.27 g of (XVIII); b.p. 63-67° (0.12 mm); n_D^{20} 1.5161.

Di-2,4-xylylacetic Acid (XIX). a) A solution of 1.48 g of (VII) in 50 ml of ethanol was added to a warm solution of sodium ethoxide (from 0.69 g of sodium) in 10 ml of ethanol. The mixture was boiled for three hours. Alcohol was removed in a vacuum, and the oily residue was dissolved in water, acidified, and extracted with ether. We obtained (XIX) in quantitative yield; m.p. 183-184° (the literature [10] gives m.p. 181-182°). Found: C 80.66; H 7.51%. $C_{18}H_{20}O_2$. Calculated: C 80.59; H 7.46%.

When sodium butoxide in butyl alcohol was used instead of sodium ethoxide, (XIX) was again obtained in quantitative yield.

b) A solution of (VIII) in absolute ethanol was added to a solution of four times the amount of sodium ethoxide in absolute ethanol. The mixture was boiled for three hours, alcohol was vacuum-evaporated with heating, and the residue was washed with ether, dissolved in water, and acidified. The crystals of (XIX) that separated were washed with water and recrystallized from a mixture of petroleum ether and methylene chloride; m.p. 182-183°, undepressed by admixture of a sample of (XIX) prepared by method (a).

3-Chloro-2,2-di-2,4-xylylpropionic acid (VIII). 20.3 ml of sulfuryl chloride was added dropwise with stirring to 17.3 ml of pyruvic acid at −5°, and stirring was then continued at room temperature for two hours and at 40° until no more gas was liberated (three hours). The mixture was cooled to −10°, 160 ml of cooled (−10°) 90% H_2SO_4 was added, and 53 g of m-xylene was added dropwise with stirring; the mixture was then stirred further for two hours and poured onto ice. The precipitate formed was recrystallized from alcohol; yield 49 g (62%); m.p. 174-175°. Found: C 71.71; H 6.63; Cl 10.84%. $C_{19}H_{21}ClO_2$. Calculated: C 72.03; H 6.63; Cl 11.21%.

3-Bromo-2-methylpropionyl Chloride (XX). A mixture of 50.1 g of 3-bromo-2-methylpropionic acid and 63.7 ml of thionyl chloride was kept for 15 h and then boiled until no more gas was liberated (90 min). Vacuum distillation gave 48.1 g (86.4%) of (XX); b.p. 51° (10 mm); n_D^{20} 1.4837; d_4^{20} 1.59. Found: C 25.87; H 3.23; Br + Cl 62.25%; MR 33.36. C_4H_6BrClO. Calculated: C 25.79; H 3.18; Br + Cl 62.43%; MR 33.31.

SUMMARY

1. Some new β-(thio lactones) containing halogen, alkylthio, aryl, and alkyl substituents in the α-position were synthesized.

2. Irrespective of the character of the α-substituents and the order in which the reactants are mixed, β-(thio lactones), unlike their oxygen analogs, undergo cleavage with formation of the corresponding 2-substituted 3-mercaptopropionic acids and their derivatives.

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