catalyst the residue distilled at 130−131° and 10 mm Hg. Yield 4.7 g.

Hydrogenation of 3-methylphthalide. When hydrogenating 3-methylphthalide as described above, the same product in the same yield was obtained. The infra-red spectra were identical in the two cases.

Separation of the two products of hydrogenation. The hydrogenation product (3 g) dissolved in 50 ml ether was shaken for 5 min with 20 ml 2 N sodium carbonate solution. From the ethereal solution, which had been dried with sodium sulphate 1.7 g oil was isolated which distilled at 130−131°C, 10 mm Hg. The equivalent weight was determined by heating samples dissolved in ethanol for some time with 0.1 N sodium hydroxide and back-titrating with hydrochloric acid. (Found: C 69.76; H 8.84; M 154, 155. Calc. for $C_9H_{14}O_2$: C 70.09; H 9.15; M 154.2). n_D^{20} 1.4748, d_D^{20} 1.0595, $[MR]_{found}$ 40.98. Hexahydro-3-methylphthalide $[MR]_{calc}$ 41.03.

The aqueous solution was acidified with hydrochloric acid and extracted with ether. An oil was obtained, which after distillation at 134−135°, 10 mm Hg, crystallized. M.p. 35−37°C. Yield 1.1 g. The substance could be titrated as an acid.

(Found C 68.94; H 10.13; M 156, 153. Calc. for $C_9H_{16}O_2$: C 69.18; H 10.33; M 156.2). n_D^{20} 1.4648, d^{20}_4 1.015, $[MR]_{found}$ 42.85. Ethylcyclohexanecarboxylic acid $[MR]_{calc}$ 43.12.

For further identification of the acid its *p*-bromophenacyl ester was prepared in the usual way. After recrystallization from ethanol m.p. 70−71°C. (Found: C 57.74; H 5.90; Br 22.26. Calc. for $C_{17}H_{21}O_3Br$: C 57.80; H 5.99; Br 22.61).

The m.p. was not depressed on mixing with the *p*-bromophenacyl ester of the acid obtained by hydrogenation of 2-ethylbenzoic acid.

Oxidation of the lactone (part A) with sodium hypobromite. To an ice-cold solution of 9.5 g sodium hydroxide in 150 ml water 12.5 g bromine were added in small portions. The lactone (2.5 g) was dissolved in 15 ml 2 N sodium hydroxide and the two solutions mixed and left at room temperature until the next day. The bromoform formed was extracted with ether and the solution after adding hydrochloric acid again extracted with ether. After removing excess of bromine with sodium sulphite and drying with sodium sulphate the ether was distilled off. The remaining yellow oil (0.5 g) crystallized partly. After washing with benzene and recrystallization from water the substance had m.p. 189−193°C. The *cis*-cyclohexane-1,2-dicarboxylic acid has, according to the literature, m.p. 192°.

Reduction of the lactone with hydrogen iodide. The lactone $C_9H_{14}O_2$ (7.25 g), 25 ml hydrogen iodide (1.7) and 2.9 g of yellow phosphorus were refluxed for 10 h. A yellow layer which formed on top of the solution was isolated, washed with water saturated with sulphur dioxide and dried in a desiccator. After distilling at 49−52°C at 0.15 mm Hg the liquid weighed 6.6 g.

367.0 and 313.7 mg subst. gave 362.1 and 308.7 mg AgI, respectively.
(Found I 53.33, 53.20 %. Calc. for $C_8H_{15}I$ I 53.30 %).
n_D^{20} 1.5300, d^{20}_4 1.462, $[MR]_{found}$ 50.09, $[MR]_{calc}$ 49.76.

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Determination of the Configuration of β-Amino and β-Hydroxy Acids by Means of Optical Rotatory Dispersion

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It has recently been shown by Sjöberg, Fredga and Djerassi [1], that the absolute configuration of α-amino and α-hydroxy acids can be readily derived from the sign of the Cotton effect in the anomalous rotatory dispersion curves of the dithio-

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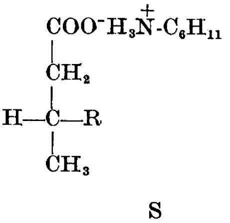
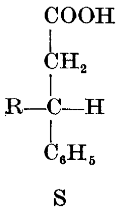
carbamate and xanthate derivatives, respectively. Other thiono derivatives have also been employed for similar purposes [2,3].

Some β-amino and β-hydroxy acids have recently been isolated from various natural sources. Although the configuration of a number of β-amino and β-hydroxy acids have been determined by chemical means, no well established simple method for the intercorrelation of β-amino and β-hydroxy acids or for the determination of their absolute configuration seems to exist. In fact, even in recent studies [4,5] there have been put forward contradictory interpretations of the steric course of the deamination of β-amino acids. It was therefore interesting to see if dithiocarbamates and xanthates of β-amino and β-hydroxy acids, respectively, would give anomalous rotatory dispersion curves, and if the sign of the Cotton effects could be used in stereochemical work in the same way as has been done in the alpha-series [1].

As model substances in the present study we chose β-aminobutyric acid and β-amino-β-phenylpropionic acid and the corresponding β-hydroxy acids. The absolute configuration of the optically active forms of these acids seems to be well established [6-9]. We used the most readily available antipodes of the four acids; the methyl dithiocarbamates of D-(−)-β-aminobutyric acid (I) and of L-(+)-β-amino-β-phenylpropionic acid (III) as well as the methyl dithiocarbonates (xanthates) of D-(−)-β-hydroxybutyric acid (II) and of L-(+)-β-hydroxy-β-phenylpropionic acid (IV) were prepared in the usual way by the action of carbon disulphide and methyl iodide on the appropriate carboxylic acids. The thiono derivatives of the two substituted butyric acids (I and II) were isolated as the cyclohexylammonium salts.





I. R = $-\mathrm{NH}\overset{\overset{S}{\|}}{\mathrm{C}}\mathrm{SCH_3}$ III. R = $-\mathrm{NH}\overset{\overset{S}{\|}}{\mathrm{C}}\mathrm{SCH_3}$

II. R = $-\mathrm{O}\underset{\underset{S}{\|}}{\mathrm{C}}\mathrm{SCH_3}$ IV. R = $-\mathrm{O}\underset{\underset{S}{\|}}{\mathrm{C}}\mathrm{SCH_3}$

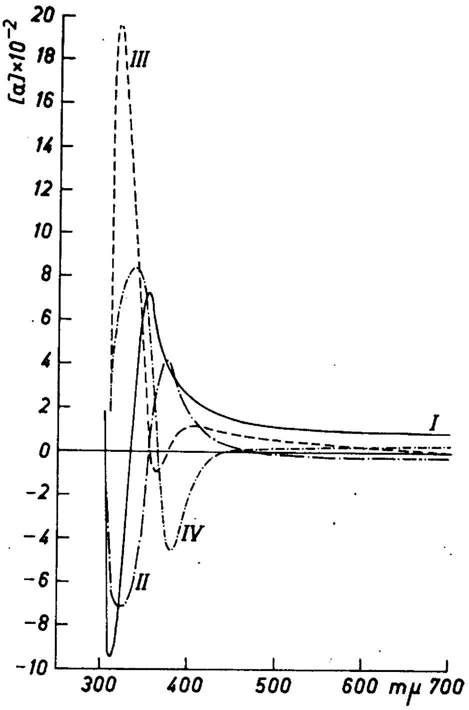


Fig. 1. Optical rotatory dispersion curves of N-dithiocarbomethoxy-D-(−)-β-aminobutyric acid cyclohexylammonium salt (I), O-dithiocarbomethoxy-D-(−)-β-hydroxybutyric acid cyclohexylammonium salt (II), N-dithiocarbomethoxy-L-(+)-β-amino-β-phenylpropionic acid (III) and O-dithiocarbomethoxy-L-(+)-β-hydroxy-β-phenylpropionic acid (IV).

Dithiocarbamates of α-amino and xanthates of α-hydroxy acids have long wave length absorption bands near 330 and 350 mμ, respectively, which are strongly anisotropic, and it has been found that the absolute configuration of the α-asymmetric center can be established from the sign of the Cotton effect: members of the L-series show a positive Cotton effect and members of the D-series exhibit a negative effect [1].

The dithiocarbamates and xanthates (I—IV) of the β-amino and β-hydroxy acids absorb around 330 and 350 mμ. From the optical rotatory dispersion curves of these compounds (Fig. 1) it can be seen that they exhibit strong Cotton effects. The dispersion curves of the cyclohexylammonium salts of N-dithiocarbomethoxy-D-(−)-β-aminobutyric acid (I) and of O-dithiocarbomethoxy-D-(−)-β-hydroxybutyric acid (II) have positive Cotton effects centered around the 330 and 350 mμ absorption bands, and the dispersion curves of N-dithiocarbomethoxy-L-(+)-β-amino-β-phenylpropionic acid (III) and of O-dithiocarbomethoxy-L-(+)-β-hydroxy-β-phenylpropionic acid (IV) have negative Cotton effects. Although the study of this problem has so far been limited to the investigation of only four structurally varied compounds, it seems as though the sign of the Cotton effect would indicate the absolute configuration of the β-asymmetric center in β-amino and β-hydroxy acids. Since, however, the derivatives of the α- and β-substituted acids of the same configuration have opposite sign of the Cotton effect, the following extension of the previously established relationship [1] is suggested: *If a dithiocarbamate of an amino acid, or a xanthate of a hydroxy acid, has an optical rotatory dispersion curve with a positive Cotton effect, then in the case of an α-amino or an α-hydroxy acid we have the* L*-configuration and in the case of a β-amino or a β-hydroxy acid the* D*-configuration.*

A full report of this work will be published later.

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Separation of α- and β-Glucometasaccharinic Acids by Means of Anion Exchange Chromatography

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In previous papers [1-3] it has been shown that hydroxy acids which are formed during the alkaline degradation of cellulose can be separated chromatographically by means of anion exchange resins. Several hydroxy acids containing 4 – 6 carbon atoms can be effectively separated by elution with borate solution, whereas with acids such as glycolic and lactic acid the separation is simpler and more efficient in acetate medium [4]. In connection with further work on the separation and identification of hydroxy acids which are formed in various technical processes, it has been found desirable to investigate the possibility of separating the stereoisomeric forms of glucometasaccharinic acids by means of anion exchange chromatography.

As shown by Kenner and Richards [5], β-glucometasaccharinic acid can easily be obtained in a pure form by alkaline degradation of 3-*O*-methylglucose. Large amounts of α-glucometasaccharinic acid are also formed in this reaction. In the present work (Figs. 1 and 3) it is shown that these stereoisomers can be effectively separated by chromatographic elution with 0.07 M sodium tetraborate solution under conditions which have been described in earlier papers for the separation of other saccharinic acids. The fractions were analyzed using the Technicon AutoAnalyzer [6] which means a considerable saving in time compared with the manual dichromate titration used earlier. The elution band corresponding to β-glucometasaccharinic acid appears before that of α-glucometasaccharinic acid and a quantitative separation can be obtained on a fairly short column.

Alkaline degradation of 3-O-methylglucose. The alkaline degradation of 3-*O*-methylglucose and the isolation of β-glucometasaccharinic acid from the reaction mixture has, with small changes, been performed according to the procedure given by Kenner and Richards [5]. A solution of 5.13 g of 3-*O*-methylglucose in