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Studies on the Chemistry of Lichens

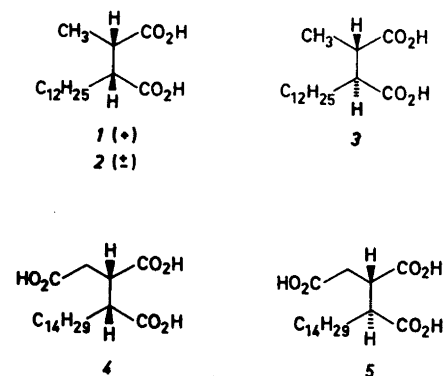
28. *Additional Evidence for the erythro Configuration of Roccellic Acid

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In order to establish beyond doubt the absolute configuration of roccellic acid (1), erythro-2-methyl-3-dodecylsuccinic acid (2) was synthesised from erythro-2-allyl-3-methylsuccinic acid.¹ Since the melting points of the synthetic racemic acid (2) and

the optically active roccellic acid differed by a few degrees, we tried to resolve the erythro-allylmethylsuccinic acid. Unfortunately, the early attempts were completely unsuccessful. However, the IR-spectra of the erythro acid (2) (Fig. 1:1) and roccellic acid were superimposable.¹ The work with nor-rangiformic acid (5) had shown that while the IR-spectra of the racemic and optically active forms of (5) were superimposable, as were the spectra of active and inactive erythro forms (4), there were substantial differences between the spectra of



the threo (5) and erythro (4) forms (Fig. 1: 3,4).^{2,3} It therefore seemed safe to conclude that roccellic acid had the erythro configuration. However, some additional evidence seemed to be desirable.

Roccellic acid was therefore epimerised with concentrated sulfuric acid to give one isomer, m.p. 136–138°, identical with synthetic erythro-2-methyl-3-dodecylsuccinic acid (2), and one isomer, m.p. 81–83°, which must be threo-2-methyl-3-dodecylsuccinic acid (3). The IR-spectrum of the threo acid (3) was different from that of roccellic acid, particularly in the region 1150–1300 cm⁻¹, (Fig. 1:1,2). Roccellic acid therefore most probably has the erythro configuration and, consequently, the (2*S*:3*R*) configuration as suggested earlier.^{1,4} The erythro forms (2) and (4) of roccellic and nor-rangiformic acid both have two fairly distinct IR absorption peaks at about 1200 and 1260 cm⁻¹ (presumably C–O stretching of the carboxyls; Fig. 1: 1, 3). By contrast the threo forms have one broad band with some fine structure at about 1200 cm⁻¹ (Fig. 1: 2,4). The 1,2-diisopropyl, di-*t*-butyl and dicyclohex-

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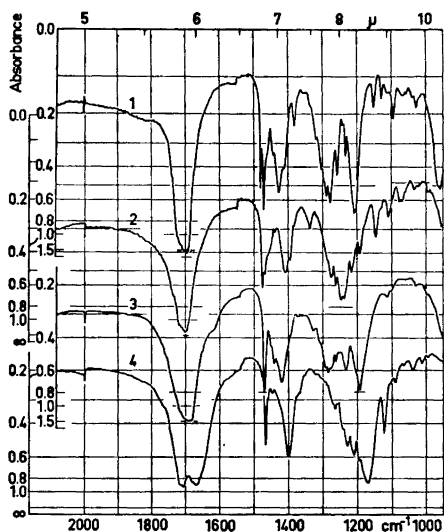


Fig. 1. IR-spectra of the *erythro* and *threo* forms of roccellic and norrangiformic acid. 1. *erythro*-2-Methyl-3-dodecylsuccinic acid (2) ((±)-roccellic acid). 2. *threo*-2-Methyl-3-dodecylsuccinic acid (3). 3. *erythro*-1,2,3-Heptadecanetricarboxylic acid (4). 4. *threo*-1,2,3-Heptadecanetricarboxylic acid (5) ((±)-norrangiformic acid).

ylsuccinic acids show a similar absorption pattern, although with minor deviations.⁴ The difference between the IR-spectra of the *erythro* and *threo* 1,2-dialkylsuccinic acids may therefore be used to distinguish between the two forms. The reason for the difference is an interesting question. At present, it can only be concluded that this difference must be associated with the manner in which the alkyl groups affect the relative orientation of the carboxyls in the crystal.

Experimental. Roccellic acid (1) 0.350 g was heated in concentrated sulfuric acid (10 ml) at 140° until the solution turned light brown (4 min). Ice was added and the product isolated in the usual way. Recrystallisation from aqueous ethanol gave the crude *erythro* acid (0.160 g). Repeated crystallisations from ethanol gave *erythro*-2-methyl-3-dodecylsuccinic acid (2), (0.075 g) m.p. 136–138° (lit.⁵ 131–132.5°), identical with a synthetic sample.¹ The mother liquor from the first crystallisation was evaporated to give a mixture of the crude

threo acid and the corresponding anhydride. The crude product (0.130 g) was treated with alkali, isolated in the usual way and recrystallised from cyclohexane-light petroleum 1: 2 to give *threo*-2-methyl-3-dodecylsuccinic acid (3) (0.046 g), m.p. 81–83° (lit.⁵ 81–82°).

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The Crystal Structure of Rh_2Ga_9 and Ir_2Ga_9

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In the course of phase analysis and crystal structure studies on platinum metal-gallium systems the phases Rh_2Ga_9 and Ir_2Ga_9 have been synthesized and they were found to be isomorphous with Co_2Al_9 .

An alloy of the composition $\text{RhGa}_{4.5}$ was prepared from rhodium powder (L. Light & Co., about 99.98 %) and gallium lump (Johnson, Matthey Chemicals Limited 4N) by heating of a mixture of the elements in an evacuated silica tube at 900°C. The reaction was accelerated by shaking the tube; the components reacted violently with an increase in temperature. The alloy was then annealed at 550°C for 2 days. The heat-treatment was discontinued by quenching in water. The product thus obtained was grey, porous and crystalline. However, no single crystals suitable for a single crystal investigation were found.