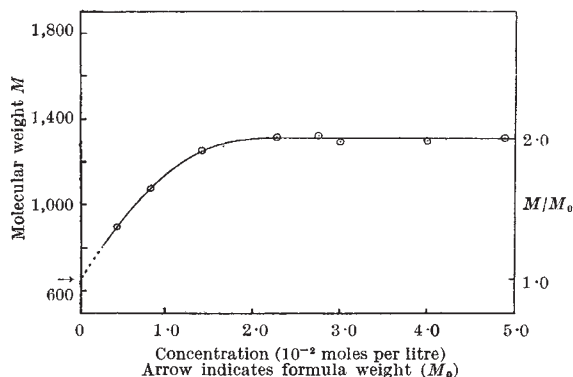


Ferric laurate has been found to belong to neither of these two main types. This trivalent soap not only fails to exhibit the characteristic properties of the aluminium soaps, but is actually dispersed as single molecules in sufficiently dilute solutions in toluene. In stronger solutions, aggregation to the dimer occurs. Molecular weights of the soap determined by the Cottrell boiling-point method are shown in the accompanying graph.



Analysis gave the iron content of the soap as 8.75 per cent (calculated for $\text{Fe}(\text{C}_{12}\text{H}_{23}\text{O}_2)_3$, 8.54 per cent), indicating that the substance obtained was mainly the neutral tri-soap. In the absence of basic hydroxyl groups, the ferric tri-soap would not be expected to polymerize like the aluminium mono- and di-soaps. The explanation of its limited aggregation compared with other metal soaps appears to lie in the increased solubilizing power of its three hydrocarbon chains, as against two or one in the divalent and mono-valent soaps. Steric hindrance to further aggregation may also be provided by the six hydrocarbon chains of the dimer.

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Preparation of Chenodeoxycholic Acid from Cholic Acid

KAWAI¹ converted cholic acid to chenodeoxycholic acid ($3\alpha:7\alpha$ -dihydroxycholanolic acid, anthropodeoxycholic acid) by Kishner-Wolff reduction of the derived 3 : 7-dihydroxy-12-keto cholanolic acid. Plattner and Heusser² similarly reduced methyl 3 : 7-diacetoxy-12-keto cholanoate.

Recently, publication of improved methods of preparation of 3 : 7-dihydroxy-12-keto cholanolic acid and its derivatives (see, for example, Fieser *et al.*³), together with the Huang-Minlon⁴ method of reduction of ketones, have led us to re-investigate the production of chenodeoxycholic acid by this route on the preparative scale. We have needed this acid and its derivatives for comparison with material isolated from bile.

We have found, indeed, that Huang-Minlon reduction of 3 : 7-diacetoxy-12-keto cholanolic acid proceeds smoothly on the 5-gm. scale to give chenodeoxycholic acid in approximately 70 per cent yield (compare Fieser and Rajogopalan³). The product can be 'crystallized' from ethyl acetate, and a (new) crystalline methyl ester (melting point 85–87°) was prepared from it by treatment at room temperature for 48 hr. with 1 per cent v/v sulphuric acid/methanol. This ester and the diacetyl ethyl ester (melting point 106–108°) were identical with material from chenodeoxycholic acid isolated from natural sources^{5,6}.

Full details of this work will be recorded elsewhere.

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Preparation of Grey Tin

It is well known that most commercial forms of tin can be transformed to the grey (α) allotropic modification in an ordinary refrigerator by contact with particles of previously prepared grey tin. The difficulty, of course, is to get some grey tin to start with, and we know of many laboratories both in Britain and abroad in which attempts to make grey tin have been unsuccessful. We have worked on various aspects of grey tin from time to time, and, although we are not ready to give a connected account of this work, we wish to record a few of our observations in the hope that they may be helpful to other investigators.

The principle on which we make grey tin is to deform pure tin (99.99 per cent) by *cold work* and allow it to recrystallize at a temperature below the $\alpha \rightleftharpoons \beta$ transformation temperature (13.2° C.). The recrystallization temperature of tin is so low that deformation at room temperature is the equivalent of *hot work*. Tin may be conveniently cold worked by compression while surrounded by solid carbon dioxide. We generally compress under a pressure of 4 tons a cylinder 0.25 in. in diameter and 0.25 in. high. We then place the cold-worked tin in a glass tube and store it in solid carbon dioxide. It is almost completely transformed into grey tin powder within twelve to twenty-four hours, the rate of transformation increasing with the purity of the tin.

If a few grains of the grey tin thus produced are sprinkled on commercially pure white tin kept at a low temperature, the whole lot is transformed into grey tin, again at a rate which increases with the purity of the tin; for example, we have transformed a kilogram of 'Chempur' tin into grey tin in about forty-eight hours.

While the foregoing is a fair statement of our general experience, we must report that one batch of 'spectroscopically pure' tin could not be trans-