

Chemistry. — "*Optical resolution of bromosulphoacetic acid*". By H. J. BACKER and H. W. MOOK. (Communicated by Prof. F. M. JAEGER.)

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The resolution of chlorosulphoacetic acid, described in the preceding communication, led us to attempt the application of the same method to an analogous asymmetric compound, namely bromosulphoacetic acid, $\text{SO}_3\text{H} \cdot \text{CHBr} \cdot \text{CO}_2\text{H}$.

Sodium bromosulphoacetate gives in the cold crystalline salts with various alkaloids, dissolved in dilute acetic acid. These salts were examined polarimetrically after careful decomposition with ammonia and elimination of the alkaloid.

Strychnine and cinchonidine gave a dextrorotatory ammonium salt, whilst brucine, chinidine and yohimbine led to the laevocompound. Cinchonine gave variable results.

Strychnine and brucine proved to be the most suitable alkaloids for preparing both enantiomorphs in a pure state.

The *dextrorotatory acid* was prepared from 40 m.mol. sodium bromosulphoacetate and 80 m.mol. acetate of strychnine in a dilution of 2100 c.c. After a day one fifth of the total amount had crystallised.

The product was decomposed with ammonia and treated with strychnine in acetic solution in such dilution, that again only a part crystallised. The rotatory power of the ammonium salt had reached its maximum and was not changed by two further crystallisations.

Another method of controlling the optical purity of the strychnine salt consists in washing the finely powdered substance with water. The pure product did not change by this treatment, whilst the rotation of a less pure specimen, examined as ammonium salt, rose to the value of the pure salt.

As even a small excess of a strong base immediately causes racemisation, the d-barium salt was not prepared from the strychnine salt with baryta, but with barium iodide. The dextrorotatory acid, prepared both from the d-barium salt and from the d-ammonium salt with sulphuric acid, had double the rotation of the ammonium salt in the same dilution.

Brucine served for the separation of the *laevorotatory acid*.

As the solubilities of the brucine salts of the two enantiomorphs do not differ much, it appeared desirable to eliminate first a part of the dextro-compound by means of strychnine.

Thus from 20 m.mol. of *r*-bromosulphoacetic acid a fifth part was

separated as strychnine salt, and the mother liquor, decomposed by ammonia, was treated with the acetate of brucine in a dilution of 2 liters. One tenth part of the remaining acid crystallised as brucine salt.

This product, washed twice with 50 c.c. cold water, was pure and showed as ammonium salt the same rotation as the enantiomorph, in opposite direction.

The rotatory power of the l-acid, prepared from this ammonium salt with sulphuric acid, corresponded to that of the d-acid.

The highest constant values, found for the molecular rotation of the two active bromosulphoacetic acids and their neutral ammonium salts, are reproduced in next table :

λ (μ)	589	560	533	510	486
acid [M]	31°	37°	42°	47°	56°
salt [M]	16°	18°	21°	24°	27°.

Racemisation of acid and salt takes place slowly at room temperature. A dilute solution of the acid may be boiled for a few minutes without appreciable alteration.

Traces of strong bases greatly accelerate racemisation of the salts.

Experimental details of this research will be published elsewhere.

Organic Chemical Laboratory of the University.

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