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Chemistry. — *"On the preparation of α-sulphopropionic acid."* By H. J. BACKER and J. V. DUBSKY. (Communicated by Prof. F. M. JAEGER.)

(Communicated in the meeting of September 27, 1919).

The only practical method for the preparation of the α-sulphopropionic acid has hitherto been the one found by FRANCHIMONT [1]) i. e, the action of sulphuric acid with propionic acid anhydride.

Besides FRANCHIMONT's general method, an analogical method has been used by MELSENS [2]) for the preparation of the sulphonacetic acid, the first term of the sulphocarbonic acids, namely the action of sulphuric acid anhydride with acetic acid.

These two methods show much similarity. In both cases the mixed anhydride, the acetylsulphuric acid, is formed as an intermediary product, as FRANCHIMONT [3]) had already supposed and STILLICH [4]) afterwards established, and as VAN PESKI [5]) proved for the reaction of MELSENS.

With a view to comparing it with the method of FRANCHIMONT we have applied MELSENS' method also to propionic acid, by treating it with sulphurtrioxide.

In both cases the reaction turned out, just as in the case of the acetic acid, to be indirect, while the mixed anhydride, the propionylsulphuric acid, must be taken to be the intermediary product.

$$\left.\begin{array}{l} CH_3.CH_2.CO_2H + SO_3 = CH_3.CH_2.CO_2SO_3H = \\ \qquad = CH_3.CH(SO_3H).CO_2H. \end{array}\right\} \text{I.}$$

$$\left.\begin{array}{l} (CH_3.CH_2.CO)_2O + H_2SO_4 = \\ = CH_3.CH_2.CO_2H + CH_3.CH_2.CO_2SO_3H \rightarrow CH_3.CH(SO_3H).CO_2H. \end{array}\right\} \text{II.}$$

When the substances are mixed carefully whilst cooling, a colourless, very viscous liquid is formed, which yields the sulphopropionic acid only at a higher temperature, with development of heat and brown coloration.

We were able to follow the process of this reaction by titration,

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1) Recueil trav. chim. **7**, 27 (1888).
2) Ann. der Chemie **52**, 276 (1844).
3) Versl. dezer Akad. **16**, 373 (1881).
4) Ber. d. dtsch. chem. Ges. **38**, 1241 (1905).
5) Versl. dezer Akad. **22**, 996 (1914).

as the propionylsulphuric acid, when hydrolyzed, neutralizes three equivalents of a basis, and the sulphonic acid only two equivalents.

The sulphopropionic acid was separated and weighed in the form of its baryumsalt. From 1 molecule propionic acid with sulphurtrioxide we got an average yield of 0.35 mol. sulphopropionic acid, and from 1 mol. propionic acid anhydride with sulphuric acid (monohydrate) 0.55 mol.

Compared with FRANCHIMONT's method, the reaction with sulphurtrioxide is experimentally less simple. The reaction is violent, the product is more coloured, the baryumsalt likewise, and the yield is various. Only for the preparation of large quantities may the reaction be recommended.

FRANCHIMONT's reaction, however, is very easy to carry out; but it is a drawback, that only half the propionic acid anhydride can be transformed into the sulphonic acid, the other half returning to propionic acid.

We have therefore tried to combine the advantages of both methods, the better yield of the first and the greater purity of the product obtained by the second.

For this purpose, it is possible to mix propionic acid anhydride with sulphuric acid, and to treat the product, containing one molecule free propionic acid, with the equimolecular quantity of sulphurtrioxide.

However, it is simpler to let the propionic acid anhydride react directly with *pyrosulphuric acid.*

$$(CH_3 . CO)_2O + H_2S_2O_7 = 2\ CH_2 . CH(SO_3H) . CO_2H.$$

By this method we got an average yield of 0.75 mol. sulphonic acid from 1 mol. propionic acid anhydride.

As the pyrosulphuric acid may be added in the crystallized state to the propionic acid, the method is easier than the reaction with sulphurtrioxyde. The reaction proceeds more quietly and the product is purer.

We have applied this method to acetic acid anhydride also, but for such an easily accessible substance, the original method of FRANCHIMONT is simpler.

For the sulphuration of precious carbonic acid anhydrides, the method just described may, however, be recommended for its higher yield.

Groningen, Sept. 8, 1919. *Org. Chem Lab. of the Univ.*