## XC.—Note on Pyromucylhydroxamic Acid.

By ROBERT HOWSON PICKARD, D.Sc., Ph.D., and ALLEN NEVILLE, B.Sc.

THE reactions of pyromucylhydroxamic acid have been studied with the view of obtaining the furfuran-carbamides and -carbamates by Thiele and Pickard's method (*Annalen*, 1899, 309, 189). These are, however, uncrystallisable oils, which decompose on hydrolysis (compare Freundler, *Bull. Soc. Chim.*, 1897, [iii], 17, 419).

Pyromucylhydroxamic acid, C<sub>4</sub>H<sub>3</sub>O·CO·NH·OH, is formed when ethyl pyromucate \* is hydrolysed with an anhydrous alcoholic solution of hydroxylamine. By precipitation with a solution of copper acetate, the grass-green copper salt is obtained, which is then suspended in alcohol and decomposed by hydrogen sulphide; on evaporation, the filtered solution yields the hydroxamic acid, which crystallises from water in lustrous, white needles and melts at 124°.

<sup>\*</sup> In preparing pyromucic acid by Frankland and Aston's method (Trans., 1901, 79, 511), a better yield is obtained if the solution of the pyromucates and lime is just rendered acid (to litmus) and then concentrated before the pyromucic acid is liberated by the addition of more sulphuric acid.

0·1351 gave 0·2329  $CO_2$  and 0·0467  $H_2O$ . C = 47.01; H = 3.84. 0·1686 ,, 16·8 c.c. moist nitrogen at 14° and 727 mm. N = 11.20.  $C_5H_5O_3N$  requires C = 47.24; H = 3.93; N = 11.02 per cent.

It gives the usual cherry-red coloration with ferric chloride, is comparatively stable towards boiling hydrochloric acid, and at first the compound appeared to have an analogous constitution to the 5-phenyl-3-isoxazolone obtained by Ruhemann and Stapleton (Trans., 1900, 77, 239) by the action of hydroxylamine on ethyl phenylpropiolate. It was, however, proved to be a hydroxamic acid by comparing the properties of its benzoyl derivative with those of 5-phenyl-3-benzoyl-isoxazolone.

Benzoylpyromucylhydroxamic acid, C<sub>4</sub>H<sub>3</sub>O·C(OH):NO·CO·C<sub>6</sub>H<sub>5</sub>, is precipitated when an aqueous solution of the hydroxamic acid is shaken with the calculated quantity of benzoyl chloride and sodium acetate. It crystallises from alcohol in needles and melts at 134°.

0.2025 gave 10.9 c.c. moist nitrogen at 15° and 763 mm. N=6.32.  $C_{12}H_9O_4N$  requires N=6.06 per cent.

It has an acid reaction and dissolves in a solution of sodium carbonate, whilst the monobenzoyl derivative of 5-phenyl-3-isoxazolone, which melts at 106°, is insoluble in the reagent.

The sodium and ammonium salts are precipitated when ether is added to their alcoholic solutions. An aqueous solution of the sodium salt, when boiled with water, evolves carbon dioxide, and an oil (containing nitrogen) is obtained when the solution is evaporated.

This oil is presumably the difurfurancarbamide, but decomposes completely when hydrolysed. No better success was attained on attempting to prepare the carbamates by boiling the sodium salt with alcohols.

MUNICIPAL TECHNICAL SCHOOL, BLACKBURN.