

to 25 cc. gave $\alpha_{5893}^{20} + 0.95^\circ$ in a 2-dc. tube; $[\alpha]_{5893}^{20} + 288^\circ$. The rearrangement of the incompletely resolved *l*-amide, $[\alpha]_{5893}^{20} - 13.8^\circ$, was also studied. The corresponding crystalline amine so produced melted at 201–206°; 0.0870 g. dissolved in ethyl alcohol (95%) and made up to a total volume of 25 cc. gave $\alpha_{5893}^{20} - 0.61^\circ$ in a 2-dc. tube; $[\alpha]_{5893}^{20} - 87.7^\circ$.

Anal. Calcd.: N, 13.6. Found: N, 13.8.

Conclusions

The Hofmann rearrangement of the amide of an optically active 2,2'-disubstituted 6-carboxydiphenyl has been studied. The corresponding amine so produced was found to be optically active, and to have the same sign for the rotatory power as the amide from which it was prepared. It was also found that the rearrangement took place in such a manner as to produce no appreciable racemization.

These results preclude the possibility of migration of the optically active group in any free form, either as a positive, negative or neutral free radical.

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Notes

A New Color Reaction with Sucrose

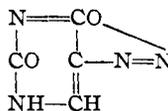
BY HARRY W. RAYBIN

In studying the chemical properties of diazo-uracil¹ it was observed that in alkaline sucrose solution it produced a blue-green color. Raffinose was the only other sugar found to give a similar but weaker color.

Fructose, glucose, maltose, lactose did not give this test.

Preliminary experiments were also negative (mostly yellow to brown-reds) with inulin, melezitose, turanose, as well as

for starch, glycogen, pneumococcus polysaccharide Type I, cellobiose, galactose, mannose, arabinose, lyxose, xylose, rhamnose, *l*-fucose, *d*-mannoheptose, mannoketoheptose, *d*- α -glucoheptose, *d*- α -glucoheptulose, lactulose, erythritol, adonitol, dulcitol, *d*-arabitol, perseitol, mannitol, glycerin, formaldehyde, acetone, pyruvic acid, acetoacetic ester, acetone-dicarboxylic acid.



Experimental Procedure.—Forty to 50 mg. of sucrose in 5 cc. of *N*/20 sodium hydroxide (10°) is shaken in a corked test-tube with 7 to 10 mg. of diazouracil until the latter dissolves. The blue-green color develops within a few minutes, the solution being kept cold. The addition of magnesium ion gives a stable blue precipitate.

An interesting reaction product with indicator properties has been isolated by salting out from neutral solution.

(1) See *Proc. Nat. Acad. Sci.*, **18**, No. 3, p. 220; *THIS JOURNAL*, **55**, 1667 (1933).

Work is being continued on the reaction of diazo oxides with carbohydrates. The author would be glad to hear of the results of others, especially with the uncommon and not readily available sugars.²

(2) The writer is greatly indebted for a number of samples to: Professor C. S. Hudson of the U. S. Public Health Service; Mr. F. Bates of the U. S. Bureau of Standards; Professor W. C. Austin of Loyola Medical School; Professors H. T. Clarke and M. Heidelberger of College of Physicians and Surgeons, Columbia University.

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The Preparation of Racemic Tartaric Acid

BY ALAN NEWTON CAMPBELL, LOUIS SLOTIN AND STEWART A. JOHNSTON

In preparations of racemic acid by the standard method,¹ we have found that calcium sulfate is an inevitable impurity, despite frequent recrystallizations. This situation is due to the well-known tendency of calcium sulfate to form supersaturated solutions. In previous work² it was found that the racemization of active tartrate could be brought about by much lower concentrations of alkali than are used in the standard preparation. A method designed to yield a product free from calcium sulfate was worked out on this basis as follows: 360 g. of *d*-tartaric acid is dissolved in two liters of 4 *N* sodium hydroxide. This solution is gently boiled in a copper flask under reflux for a week. The solution is made distinctly acid with strong hydrochloric acid, and evaporated to a bulk of 500 cc. It is then filtered hot from precipitated sodium chloride and 200 cc. of concentrated hydrochloric acid added to the filtrate. More sodium chloride separates which is again filtered. A liter of 95% alcohol is then added and the precipitated sodium chloride removed. The alcohol is removed by distillation from the steam-bath and the residue allowed to evaporate in an open dish on the steam-bath until the liquid portion has fallen to 150 cc. The thin sirupy liquid obtained is inoculated with a few crystals of racemic acid and allowed to stand overnight. This treatment causes the liquid to set solid. It is macerated with 100 cc. of cold water and filtered. The crude racemic acid thus obtained is dissolved in hot water, boiled with animal charcoal, filtered and allowed to crystallize. The crystals are dried in an oven at 80° to get rid of traces of hydrogen chloride. Further recrystallization from water is necessary to obtain a perfectly pure product. The final product has a melting point, when dehydrated, of 203–204°, is quite inactive and free from chloride. The yield is 150 g. of crystallized product.

(1) Cohen, "Practical Organic Chemistry," 1928 ed., p. 145; Holleman, *Rec. trav. chim.*, **17**, 66 (1898).

(2) Campbell and Campbell, *THIS JOURNAL*, **54**, 3834 (1932).