

## NOTE.

*D-Ribobenziminazole, A Correction.* By GEOFFREY R. BARKER, KATHLEEN R. COOKE, and J. MASSON GULLAND.

THE properties of *d*-ribobenziminazole reported by two of us (J., 1943, 625) were at variance with those previously published by Richtmeyer and Hudson (*J. Amer. Chem. Soc.*, 1942, **64**, 1612) and we were unable to account for the discrepancy. After our paper had gone to press, Dimler and Link (*J. Biol. Chem.*, 1943, **150**, 345) described the preparation of *d*-ribobenziminazole and recorded the m. p. and specific rotation of the compound, both of which were in agreement with those recorded by Richtmeyer and Hudson, and also the m. p.'s of the picrate and the hydrochloride. We have now repeated the preparation of *d*-ribobenziminazole from synthetic *d*-ribonic acid, characterised as before by means of the phenylhydrazide, and we are now in agreement with the American authors. The reason for the previous results and the implications regarding the pentose of the nucleic acid of yeast are being investigated and will be reported upon shortly.

*d-Ribobenziminazole.*—Cadmium ions were removed, by passage of hydrogen sulphide, from a solution in water (200 c.c.) of cadmium *d*-ribonate (5.5 g.), prepared by Steiger's method (*Helv. Chim. Acta*, 1936, **19**, 189), and the cadmium sulphide was coagulated by boiling and removed by filtration through charcoal. The filtrate, after removal of hydrogen sulphide by aeration, was concentrated under diminished pressure to a syrup, which was heated with *o*-phenylenediamine (3.6 g.), concentrated hydrochloric acid (5.5 c.c.), glacial phosphoric acid (2 c.c.), and ethyl alcohol (5 c.c.) for 2 hours at 130–140°. The resulting syrup was dissolved while hot in water (20 c.c.), and after being decolorised with charcoal the solution was made just alkaline to litmus with 4*N*-sodium hydroxide, then just acid to litmus with 2*N*-hydrochloric acid, and finally aqueous ammonia (*d* 0.880) (0.2 c.c.) was added. A small quantity of insoluble material was filtered off, the excess of *o*-phenylenediamine removed by extraction with ether, and the resulting solution evaporated to dryness under diminished pressure. Water (10 c.c.) was added to the residue and, after standing at 0° for 3 hours, the *d*-ribobenziminazole was collected, washed with water, crystallised twice from water (6 c.c.), and separated in rosettes of fine needles (1 g.), m. p. 191° (Found: N, 11.6. Calc. for  $C_{11}H_{14}O_4N_2$ : N, 11.7%). In 5% citric acid solution (*c* = 2),  $[\alpha]_D$  was +23.5°. The picrate crystallised from water in long yellow needles, m. p. 184–186° (Found: N, 15.0. Calc. for  $C_{11}H_{14}O_4N_2 \cdot C_6H_3O_7N_3$ : N, 15.0%). The hydrochloride separated from absolute alcohol in rosettes of fine needles, m. p. 201–203° (Found: N, 9.8. Calc. for  $C_{11}H_{14}O_4N_2 \cdot HCl$ : N, 10.2%).

*d-Ribonic Phenylhydrazide.*—*d*-Ribonic acid, obtained from synthetic cadmium *d*-ribonate (1 g.) as described above, was heated with phenylhydrazine (0.3 c.c.) on a boiling water-bath for 1 hour. The residue, when cold, was dissolved in ethyl alcohol (5 c.c.); ribonic phenylhydrazide separated in long colourless needles, which, after being crystallised three times from ethyl alcohol, had m. p. 162–164° (Found: N, 10.6. Calc. for  $C_{11}H_{16}O_5N_2$ : N, 10.9%).—UNIVERSITY COLLEGE, NOTTINGHAM. [Received, March 15th, 1944.]