

modifying the method of Livak, *et al.*¹ The over-all yield was increased and the method simplified when the α -amino- γ -hydroxybutyric acid needed for the preparation of the 5-(β -bromoethyl)-hydantoin was not isolated.

α -Bromo- γ -butyrolactone (462 g., 2.8 moles) was added, at such a rate that the temperature did not exceed 25°, with stirring to 985 ml. of concentrated ammonia water. After standing overnight at room temperature, a solution of 157 g. of potassium hydroxide in 2575 ml. of water was added. The solution was concentrated to a volume of *ca.* one liter. A solution of 174 g. (2.15 moles) of potassium cyanate in 334 ml. of water was added to the hot solution and the clear reddish-brown solution which resulted was heated at an internal temperature of 65° for two hours. Fourteen hundred ml. of 48% hydrobromic acid was then added to the chilled solution, which was then heated in a steam-bath (internal temperature was 93°) for two hours. After distilling to dryness *in vacuo*, the residue was suspended in two liters of boiling acetone. The mixture was filtered and the insoluble salts were washed well with hot acetone. The solvent was removed from the filtrate by distillation and the residue was heated with 1350 ml. of 48% hydrobromic acid in a steam-bath for two hours. The solution was taken to dryness *in vacuo* and the crude product remaining was dissolved in 400–500 ml. of boiling water. On chilling in an ice-bath, a tan precipitate appeared. This was filtered, washed with ice-water, and recrystallized from one liter of water. The product melted at 139–141° (uncor.) and weighed 142 g. (24.5%). Livak, *et al.*,¹ obtained their 5-(β -bromoethyl)-hydantoin in an 18.6% over-all yield and reported a melting point of 141.5–142°.

A solution of 41.4 g. (0.2 mole) of 5-(β -bromoethyl)-hydantoin and 16.8 g. (0.22 mole) of thiourea in 75 ml. of ab-

solute ethanol was refluxed for 28.5 hours. After chilling in an ice-bath, the precipitate was separated and washed with ethanol followed by ether. The tan solid melted with decomposition at 191–192° (cor.) and weighed 51.5 g. (91%). When the volume of ethanol was decreased to 50 ml., the yield was increased to 95% but the product then melted at 186.5–189.5°.

Anal. Calcd. for C₈H₁₁BrN₄O₂S: C, 25.45; H, 3.92. Found: C, 25.79; H, 3.98.

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N-(*p*-Dimethaminobenzyl)-aminoethanol

A warm solution of 74.6 g. (0.5 mole) of *p*-dimethylamino-benzaldehyde and 40 g. (0.66 mole) of ethanolamine in 100 ml. of absolute ethanol was hydrogenated at an initial pressure of 58 lb. in the presence of 250 mg. of platinum oxide. About ten hours was required for the reduction. The catalyst and solvent were removed and the residue vacuum distilled. The product, a pale yellow viscous liquid, weighed 139.2 g. (76%), b.p. 157–158° (1.5 mm.).

Anal. Calcd. for C₁₁H₁₈N₂O: N, 14.30. Found: N, 14.28.

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COMMUNICATIONS TO THE EDITOR

ON THE MECHANISM OF THE *IN VIVO* SYNTHESIS OF D-RIBOSE¹

Sir:

The *in vivo* synthesis of D-ribose in the chick has been studied to determine whether the major pathway is by direct conversion from hexose.² Such a conversion would be expected to yield ribose labeled similarly to glycogen after feeding a C¹⁴-labeled compound.

In a representative experiment, 7 chicks 1 month old were fasted for 48 hours and fed 6 mM. of sodium acetate containing 72×10^6 counts/minute/mg. of carboxyl carbon and 15 g. of chick mash per 100 g. body weight. After 18 hours, the animals were sacrificed. Glycogen was isolated from a cold trichloroacetic acid extract of the pooled internal organs and muscle and degraded by the method of Wood, *et al.*³ Ribose was obtained from the purine nucleotide fraction of the trichloroacetic acid insoluble residue. The sodium nucleates were isolated,⁴ the pentose and desoxypentose nucleic acids separated⁵ and the purine ribonucleotides hydrolyzed with dilute acid. Ribose was purified by

two filter paper chromatographic separations using ethyl acetate–acetic acid–water (3:1:3)⁶ and *n*-butanol–ethanol–water (4.5:0.5:5) as solvents. A portion of the pentose was fermented with *Lactobacillus pentosus* 124-2 to acetate and lactate followed by further degradation to give the individual carbons as carbon dioxide. The remainder of the ribose was converted to potassium ribonate⁷ and the salt oxidized with periodate as a check on the fermentation results. The work of Lampen, *et al.*,⁸ has indicated that in this fermentation the α and carboxyl carbons of the acetate arise from pentose carbons 1 and 2, respectively; the carboxyl, α and β carbons of the lactate, from carbons 3, 4 and 5. The data being reported are in agreement with this interpretation.

The values,⁹ in counts/minute/mg. carbon, obtained in this experiment for glycogen and ribose by fermentation were

	C ₁	C ₂	C ₃	C ₄	C ₅	C ₆
Glycogen	39	21	2460	2460	21	39
		C ₁	C ₂	C ₃	C ₄	C ₅
Ribose		45	52	338	10	0

(6) M. A. Jermyn and F. A. Isherwood, *Biochem. J.*, **44**, 402 (1949).

(7) S. Moore and K. P. Link, *J. Biol. Chem.*, **133**, 293 (1940).

(8) J. O. Lampen, H. Gest and J. C. Sowden, *J. Bact.*, **61**, 97 (1951).

(9) Values have been corrected for addition of carrier during degradation. Radioactivity measurements were made on BaCO₃ with an end-window Geiger–Müller counter. Measurements on ribose and ribonate were checked with a gas phase counter at the Brookhaven National Laboratory by Dr. Robert Steele whose assistance is greatly appreciated.

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(2) F. Dickens, *Biochem. J.*, **32**, 1626, 1645 (1938); F. Dickens and G. E. Glock, *Nature*, **166**, 33 (1950); B. L. Horecker and P. Z. Smyrniotis, *Arch. Biochem.*, **29**, 232 (1950); D. B. McNair Scott and S. S. Cohen, *J. Biol. Chem.*, **188**, 509 (1951).

(3) H. G. Wood, N. Lifson and V. Lorber, *ibid.*, **189**, 475 (1945).

(4) J. N. Davidson and C. Waymouth, *Biochem. J.*, **38**, 375 (1944).

(5) G. Schmidt and S. J. Thannhauser, *J. Biol. Chem.*, **161**, 83 (1945).