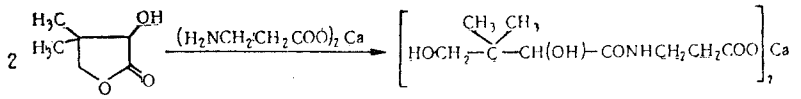
SYNTHESIS OF CALCIUM DL-PANTOTHENATE

B. M. Gavrilov, V. P. Chekhun, and A. A. Karepov

UDC 615.739.127-012

The calcium salt of β-alanine is prepared and used for the synthesis of calcium DL-pantothenate.

The synthesis of calcium DL-pantothenate is accomplished with almost quantitative yield by the reaction of the calcium salt of β-alanine with α-hydroxy-β,β-dimethyl-γ-butyrolactone [1]:



Calcium DL-pantothenate is precipitated from alcohol solution by acetone [2]. We prepared the calcium salt of β-alanine from β-alanine sulfate by treatment with calcium oxide in aqueous medium. β-Alanine sulfate is prepared by the hydrolysis of β-phthalimidopropionitrile.

EXPERIMENTAL

Calcium Salt of β-Alanine. A mixture of 50 g of β-phthalimidopropionitrile with mp 152-154°C and 160 g of 42% sulfuric acid was heated at 114-115° for 3 h. After cooling to 14-15° phthalic acid was filtered off and washed with 150 ml of water and the filtrate and water washings stirred with 50 g of calcium oxide. The calcium sulfate formed was separated and washed with 150 ml of water. Then the solution was stirred with a further 20 g of calcium oxide and heated until the ammonia evolution ceased, and the precipitate was again filtered off and washed with 20 ml of water. The solution was evaporated in vacuum to 1/20 volume, filtered from inorganic impurities with activated carbon, and evaporated to dryness. The yield was 21.9 g (80%).

Calcium DL-Pantothenate. A mixture of 250 ml of methanol, 33 g of α-hydroxy-β,β-dimethyl-γ-butyrolactone, and 25 g of the calcium salt of β-alanine was heated at 64-65° for 3 h. Then 5 g of activated carbon was added and the mixture was cooled to 20-25° over a half hour and filtered. The solid was washed with 50 ml of methanol and the filtrate and alcohol washings evaporated to $d = 0.98\ g/cm^3$. The residue was poured into acetone, taken in the ratio 8 : 1, and calcium DL-pantothenate was filtered off, washed with 100 ml of acetone, and dried in vacuum for 5 h at 50°. The yield was 54.0 g (98.1%). Found %: C 45.48; H 6.81; N 5.71. $C_{18}H_{32}N_2O_{10}Ca$. Calculated %: C 45.35; H 6.77; N. 5.88.

LITERATURE CITED

1. F. Kogan, R. V. Heinzelman, and D. T. Weisblat, J. Am. Chem. Soc., 79, 3545 (1957).
2. J. J. Rogers, J. Am. Chem. Soc., 62, 1784 (1940).

Biochemical and Vitamin Preparation Combine, Krasnodar. Translated from Khimiko-Farmatsevticheskii Zhurnal, No. 1, p. 36, January, 1967. Original article submitted July 16, 1966.