

PREPARATION OF NICOTINOHYDROXAMIC ACID-CARBOXYL-¹⁴C

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Nicotinohydroxamic acid (NHA) has been found to have the antiurease activity⁽¹⁾. We have tried to label NHA with ¹⁴C for metabolic studies. This paper is concerned with the synthesis of nicotinohydroxamic acid-carboxyl-¹⁴C (¹⁴C-NHA).

2 ml of thionyl chloride was added to 210 mg of nicotinic acid-carboxyl-¹⁴C (4 mCi, obtained from Daiichi Pure Chemicals Co., Tokyo) and refluxed for 2.5 hours on oil bath at 80°C. 2 ml of benzene was added to the residue obtained following removal of thionyl chloride and then evaporated in vacuo.

A mixture of nicotinyl chloride-carbonyl-¹⁴C obtained and 2 ml of methanol was allowed to stand for 15 min. at room temperature and thereafter solvent was removed by evaporation in vacuo. 1.5 ml of a solution of 3 g of hydroxylamine hydrochloride in 13 ml of 23 % NaOH aqueous solution was added to the oily residue and stirred for 2.5 hours at room temperature. The reaction mixture was adjusted to pH 7.5 by adding conc. HCl and then evaporated to dryness. Crude ¹⁴C-NHA was recrystallized from 1.5 ml of water. 87.2 mg of ¹⁴C-NHA was obtained in a yield of 35.5 % based on nicotinic acid-carboxyl-¹⁴C, m.p. 161-162°C, 16.27 µCi/mg. The melting point of an admixture with the authentic sample, NHA, was 162-164°C. The product was also confirmed to be pure on thin layer chromatogram, as shown in Fig. 1.

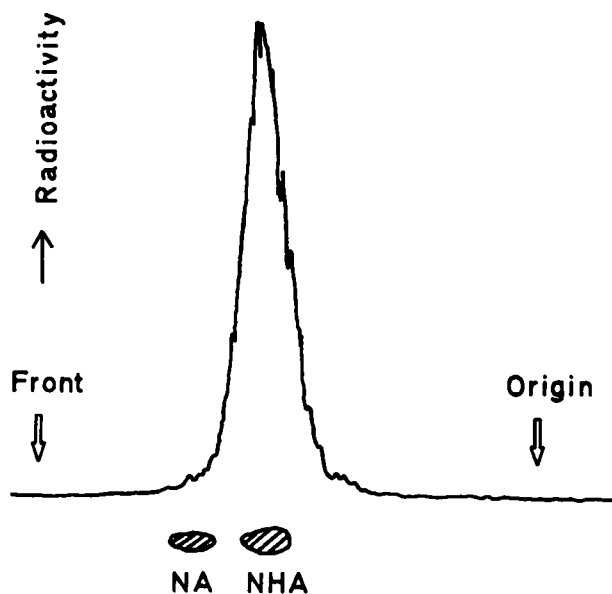


Fig. 1. Thin layer chromatogram of ^{14}C -NHA, NHA and nicotinic acid (NA).

Kieselgel GF₂₅₄ (250 μ in thickness),
water, Aloka thin layer chromatogram
scanner Model TLC-2B.

References

- 1) Hase, J. and Kobashi, K. — J. Biochem., 62 : 293 (1967).

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