NOTES

# PREPARATION OF NICOTINOHYDROXAMIC ACID-CARBOXYL- $^{14}C$.

Received on February 5, 1973.

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

2 ml of thionyl chloride was added to 210 mg of nicotinic acid-carboxyl-$^{14}C$ (4 mCi, obtained from Daiichi Pure Chemicals Co., Tokyo) and refluxed for 2.5 hours in oil bath at 80°C. After the excess of thionyl chloride was evaporated under reduced pressure, 2 ml of methanol was added to the residue. After standing for 15 minutes at room temperature, the solvent was evaporated 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 in vacuo. Crude $^{14}C$-NHA was recrystallized from 1.5 ml of water. 87.2 mg of $^{14}C$-NHA was obtained in a yield of 35.5 % on the basis of nicotinic acid-carboxyl-$^{14}C$, 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.

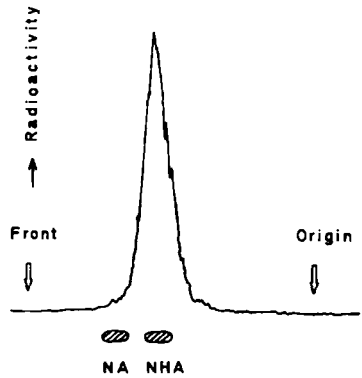


Fig. 1. Thin layer chromatogram of $^{14}$C-NHA, NHA and nicotinic acid (NA).
Kieselgel $GF_{254}$ (250 μ in thickness),
water, Aloka thin layer chromatogram
scanner Model TLC-2B.

The authors thank Mr. T. Naito, Director, Eisai Research Laboratories, for his valuable advices.

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## REFERENCES

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