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NOTES

Preparation of Phenylacetic acid-4-^{3}H

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A simple and rapid catalytic dehalogenation process has been used for the preparation of phenylacetic acid -4-^{3}H. Ethyl *p*-bromophenylacetate was hydrogenated in absolute ethanol solution in the presence of palladium on charcoal catalyst and triethylamine. The ethyl phenylacetate formed was hydrolyzed by refluxing it with a sodium hydroxide solution. The free acid was precipitated by acidifying the alkaline solution and recrystallised after dilution with inactive phenylacetic acid. The radiochemical purity of the product was checked by radiothinlayer chromatography.

Ethyl *p*-bromophenylacetate (106 mg; 0.43 mM) was dissolved in 1 ml of absolute ethanol. Triethylamine (0,05 ml; 0.5 mM) and 10 % palladium on charcoal catalyst (20 mg) was added to the solution. The mixture wad stirred in a hydrogen atmosphere containing 8 Ci of tritium. The calculates amount of hydrogen was absorbed within four hours keeping the pressure higher than 450 mm Hg.

The catalyst was filtered off and washed with ethanol. The alcohol was distilled under reduced pressure. The residue was mixed with 1 ml of 2 N sodium hydroxide and boiled gently under reflux for two hours. The cold reaction mixture was then extracted with ether in order to remove triethylamine, and the alkaline solution was acidified by the dropwise addition of concentrated hydrochloric acid. The acid precipitated was recrystallised from water after diluting it with pure inactive phenylacetic acid (260 mg). The pure product was dried in a vacuum dessicator over phosphorus pentoxide.
Yield : 270 mg (84 %) phenylacetic acid -4-^{3}H.
Melting point : 75-75.5 °C (uncorrected).
Specific activity : 2.8 mCi/mg; 380 mCi/mM.
Radiochemical yield based on tritium gas : 9.4 %.

The radiochemical purity of the product was checked by thinlayer chromatography on Eastman-Kodak TLC sheet in ethanol, water, concentrated ammonia (100/12/16) developing solvent system. The chromatogram showed only a single radioactive peak corresponding to phenylacetic acid ($R_F \approx 0.6$).

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