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NOTE

Sodium [1,2-$^{13}C_2$,$^{2}H_3$]Acetate

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Sodium [1,2-$^{13}C_2$,$^{2}H_3$]acetate has been prepared on a small scale suitable for biosynthetic investigations.

Keyword: Sodium [1,2-$^{13}C_2$,$^{2}H_3$]acetate.

Introduction

Sodium [1,2-$^{13}C_2$,$^{2}H_3$]acetate was required for biosynthetic studies on fungal polyketides. This substance is not readily available commercially and is expensive, so we aimed to prepare it from sodium [1,2-$^{13}C_2$]acetate. Surprisingly, the literature methods for preparing sodium[$^{2}H_3$]acetate were not readily adaptable. The classical method[1] involves hydrolysis of carbon suboxide to malonic acid, followed by decarboxylation, clearly a wasteful method when ^{13}C-labelled material is required. Of the methods involving exchange of hydrogen atoms with D_2O, two were developed in order to study the kinetics of exchange.[2,3] Sealed glass vessels were used and the deuterated acetate obtained was of unspecified isotopic purity. Two reports of preparations of isotopically pure sodium [$^{2}H_3$]acetate gave few experimental details[4,5] and could not in our hands be successfully reproduced. We required a convenient, reproducible

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method of preparing small quantities of sodium [1,2-$^{13}C_2$,$^{2}H_3$]acetate of high isotopic purity. This has been achieved by exchanging [1,2-$^{13}C_2$]acetate with deuterated lithium hydroxide solution, generated *in situ* in a screw top PTFE centrifuge tube, which serves as a chemically inert, reusable sealed tube.

Experimental

Lithium metal (0.25mmole, 1.74mg) was dissolved in deuterium oxide (Sigma) (2ml) in a PTFE screw top centrifuge tube (Nalgene). Sodium [1,2-$^{13}C_2$]acetate (Amersham International)(2.5mmole, 210mg) was added, the tube closed and the reaction mixture stirred at 150°C for 24 hours. The D_2O (containing HOD) was removed *in vacuo* by placing the PTFE tube inside a round-bottomed flask which was placed on a rotary evaporator. Fresh D_2O (2ml) was added and the mixture was again heated at 150° for 24 hours. This procedure was repeated a total of four times. 100µl of the solution was removed and mixed with phenylalanine (12.5µmole in D_2O). The ^{1}H NMR (Perkin Elmer R12 spectrometer operating at 60MHz) signal intensities were compared and the acetate was found to be 99% deuterated.

After neutralisation with dilute hydrochloric acid and evaporation of the solvent for the last time, the labelled sodium acetate, contaminated with a very small amount of lithium chloride was immediately usable for biosynthetic studies.[6]

Acknowledgements

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