

TRITIUM LABELLING OF MORPHOLINE ON GAMMA-IRRADIATED SILICA GEL

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SUMMARY

The use of gamma-irradiated silica gel to promote exchange between tritium gas and carbon-bound hydrogen has been previously described.^(1,2,3) The suitability of this method for labelling molecules containing labile as well as carbon-bound hydrogen has not hitherto been reported.

The labelling of morpholine to facilitate its use as a weak base indicator as part of a study of intracellular pH⁽⁴⁾ afforded an opportunity to test the suitability of gamma-irradiated silica gel as a labelling reagent.

EXPERIMENTAL

The preparation of gamma-irradiated silica gel and the sorption of tritium gas on to it have been described elsewhere.^(1,2,3)

One ml. of redistilled morpholine (B.D.H.) was vapour transferred in vacuo onto 2 gm of silica gel which had been degassed and irradiated with 1 Mrad of ⁶⁰Co radiation and on to which 150 mCi of tritium gas had been absorbed. The mixture
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was held at room temperature for one hour and the product transferred in the vapour phase using mild heating.

Radio-gas-liquid-chromatography showed only one peak in both mass and activity traces with impurities less than one per cent abundance.

A test sample washed in water and re-submitted to radio-gas-liquid chromatography showed that more than 90% of the activity was labile and presumably associated with the nitrogen atom.

The difficulty of separating morpholine from water led us to choose dry ammonia gas as the reagent for washing out labile tritium from the labelled morpholine. Gaseous ammonia was dried by passing it through a 15 cm column containing pre-dried cotton wool, through a trap at 195 K and then bubbled through the labelled morpholine. The emergent gas was monitored by liquid scintillation counting and after two hours the activity fell to background levels. Bubbling was continued for a further two hours and aliquots of the morpholine counted at regular intervals but they showed no change in activity throughout this period.

A sample of the morpholine was then mixed with water and radio-gas-liquid chromatography of this mixture showed only one activity peak indicating that all the labile tritium had been removed.

Scintillation counting of the morpholine then showed an activity yield of 20 μCi - an activity sufficient for its purpose!

ACKNOWLEDGEMENTS

We thank Messrs. K.M. Matthews and J.M. Seakins for helpful discussions.

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