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Study of Some Variables which Influence the Labelling Iodoantipyrine ^{131}I

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Introduction

Several methods have been published for the preparation of 4-iodo 2–3 dimethyl phenyl pyrazolone (iodoantipyrine) [1, 2, 3] but none of them mention the experimental procedures involved nor the yield obtained, with the exception of ref. [3], which employs the Conway camera microdiffusion method, with a yield of 30%.

We have studied the influence of some variables on the yield of labelled iodoantipyrine, obtained by isotopic exchange according to the method of MITTA and CAMIN [4]. In particular we have studied the influence of p_H, concentration, temperature and illumination.

Experimental

The labelling method we used was the following:

Of a buffer solution of sodium acetate and acetic acid (p_H 4.6) with a sodium acetate concentration of 0.5–1 M, 1 ml, containing 0.4 mg of iodoantipyrine, was placed in a glass stoppered tube. To this we added 0.5–1.0 mCi Na ^{131}I (carrier-free, concentration about 50 mCi/ml). The mixture was heated in a water bath at 100 °C for about 10 min under a 100–150 W lamp at a distance of 10 cm. After cooling we added 1–2 drops of $Na_2S_2O_3$ (1 M). Radiochemical yields were about 95–97%, measured as the fraction of the total activity recovered in the labelled material.

The radiochemical purity of the product was checked by the following tests.

a) Paper chromatography [1]: The solvent is prepared by mixing equal volumes of ethanol and of toluene (Iodides R_f: 0.00; Iodantipyrine R_f: 0.95).

b) Thin layer chromatography [5]: The solvent is prepared by mixing 80 volumes of chloroform and 20 volumes of toluene (Iodides R_f: 0.00; Iodoantipyrine R_f: 0.86).

In both cases Draggendorf's reagent is used to detect iodoantipyrine and 10% lead acetate to detect iodides.

c) Electrophoresis on paper strips moistened with ammonium carbonate solution at p_H 9 with 300 volts during 30 min.

The radioactivity of the iodide was measured by scanning. It was always less than 5% of the total activity.

In normal practice the product is tested for sterility presence of pyrogenous substances and inorganic radioiodides.

In our experiments we found red light to be quite inefficient in promoting the labelling of iodoantipyrine. Yields obtained with an intensity of 32 Lux did not exceed 8 percent. Light from an ordinary electric bulb and violet light gave somewhat better results up to 50 percent for the former and 25 percent for the latter (both at an intensity of 45 Lux). By far the best results were obtained by ultra violeta light (roughly above 300 mμ and 67 Lux) with yields up to 95 percent.

The fact that fairly good yields were obtained with an ordinary light bulb was possibly due to the fact that it produces a certain amount of ultra violet light.

A p_H of 4.6 was found to be optimal with an ordinary electric light bulb and 20 min reaction time. We obtained yields up to about 60 percent at p_H 4.6 with corresponding yields of only 30 percent at p_H 4.1 and only 4 percent at p_H 5.6. (The sodium acetate concentration was 0.25 M.)

High temperatures are most beneficial. With p_H 4.6, a sodiumacetate concentration of 0.25 M, a reaction time of 5 min and an ordinary incandescent lamp, we obtained a yield of 58 percent at 35 °C, 84 percent at 81 °C and 96 percent at 100 °C. If an oxidant was added to the solution, either H_2O_2 or choramine T, the reaction rate was increased by 10% or 20% at 35 °C, but no improvement was observed at 100 °C.

The influence of the total acetic acid + acetate concentration was also studied. It was found that at p_H 4.6 the labelling yield increases with acetic acid + acetate concentration up to an optimal value of 0,25 M. Above this value it decreases and becomes constant above 0.35 M.

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1. P. J. TALSO, T. N. LAHR, N. SPAFFORD, G. FERENZI and H. R. O. JACKSON, J. Lab. clin. Med. **46**, 619 (1955).
2. W. H. STRAUB, J. M. SULLIVAN and J. C. ROSE, Georgetown Med. Bull. **16**, 20 (1962); C. A. 837 (1963).
3. E. ENGELSTEIN, B. STRUFALDI, J. C. BARBERIO and R. R. PIERONI, Presented at the Study Group Meeting on the Utilization of Research Reactors at Sao Paolo, Brasil (I.A.E.A. 1963, unpublished).
4. A. E. A. MITTA and L. L. CAMÍN, Informe N° 112 C.N.E.A. (1964).
5. A. E. A. MITTA, L. L. CAMÍN and M. L. P. TROPAREVSKY, Radiochim. Acta **6**, 111 (1966).