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Application of the Microwave Discharge Modification of the Wilzbach Technique for the Tritium Labelling of some Organics of Biological Interest

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APPLICATION OF THE MICROWAVE DISCHARGE MODIFICATION OF THE WILZBACH TECHNIQUE FOR THE TRITIUM LABELLING OF SOME ORGANICS OF BIOLOGICAL INTEREST

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The modification of the Wilzbach technique using microwave discharge [1] has been routinely used in our laboratory for rapid tritium labellings. The applicability of the method and the effects of some of the reaction parameters were studied and published previously [2]. The main advantage of the method is its simplicity and rapidity and the low extent of decomposition of the compound to be labelled during the reaction. Specific activities obtained, however, are not high enough for some investigations.

In this paper the labelling of dihydrostreptomycine, tetracycline and antipyrine will be described.

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CONTENTS

EXPERIMENTAL

The apparatus used was essentially the same as published earlier [2].

Dihydrostreptomycine - ^{3}H

Dihydrostreptomycine trisulfate (102 mg) was placed into the reaction vessel and degassed thoroughly. Tritium gas was then admitted up to a pressure of 3.6 mm Hg, which corresponded to an activity of 380 mCi

Microwave discharge (180 W) was performed for three minutes. After pumping off the remaining tritium, the product was dissolved in water and precipitated by adding alcohol to the solution. This precipitation was repeated twice more and finally the product was dried in vacuum at room temperature.

Yield: 72 mg (70 %)

Specific activity: 85 μCi/mg; 75 mCi/mM

Radiochemical yield based on tritium gas: 1.6 %

The radiochemical purity of the product was checked by paper chromatography on Whatman No. 1 paper. The chromatogram was developed by using the ascendent technique in the following solvent system:

400 ml	n-butanol saturated with water
8 g	p-toluenesulfonic acid
10 ml	piperidine

The dihydrostreptomycine was localized as a pink spot by spraying the chromatogram with a reagent obtained by mixing equal volumes of the following solutions:

0.1 % diacetyl in water

2.5 % α-naphtol in alcohol

20 % w/v KOH in water

The radiochromatogram of the labelled dihydrostreptomycine (scanned by using a Packard M7200 scanner) showed a well defined single radioactive peak which corresponded to the dihydrostreptomycine spot obtained by the spray reagent (Fig. 1).

Tetracycline - ^{3}H

The labelling procedure was essentially the same as described above; 100 mg of tetracycline hydrochloride was labelled at a tritium pressure of 3.2 mm Hg (340 mCi). Labile tritium was removed by dissolving the product three times in methanol followed by evaporation to dryness under reduced pressure. The crude product was precipitated from water solution, the pH of which was adjusted to 4.5 by the dropwise addition of 0.1 N hydrochloric acid, and finally dried in vacuum at 50 ^{o}C.

Yield: 86.5 mg (86.5 %)

Specific activity: 165 μCi/mg; 79 mCi/mM

Radiochemical yield based on tritium gas: 4.2 %

The radiochemical purity of the product was checked by thin-layer chromatography on Eastman-Kodak Silicagel-G TLC sheet using 10 % aqueous citric acid solution saturated with n-butanol as developing solvent system. The tetracycline was localized as a yellow spot by spraying the chromatogram with 1N hydrochloric acid followed by heating at 50 °C for a few minutes. The chromatogram is shown on Fig. 2.

Antipyrine - ^{3}H

150 mg of antipyrine was labelled by the procedure described above at a tritium pressure of 3.8 mm Hg (400 mCi). After removing labile tritium by dissolving the product three times in methanol followed by evaporation to dryness under reduced pressure, antipyrine was found to be pure without any further purification.

Yield: 90 mg (60 %)

Melting point: 109 - 110 °C (uncorr.)

Specific activity: 350 μCi/mg; 66 mCi/mM

Radiochemical yield based on tritium gas: 7.1 %

Thin-layer chromatography on Eastman-Kodak Silicagel-G sheet, using methanol as developing solvent and Dragendorff sprayreagent to localize antipyrine, showed a single radioactive peak. No decomposition could be observed after keeping the product at room temperature for five months in powder form. (Fig. 3).

ACKNOWLEDGEMENT

The author´s thanks are due to Mr. H. Tovedahl for the radioactivity measurements.

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Legends to the figures

Fig. 1 Radio paper chromatogram of dihydrostreptomycine - ^{3}H - trisulfate.

Solvent: 400 ml n-butanol saturated with water.
8 g p-toluenesulfonic acid.
10 ml piperidine.

Paper: Whatman No. 1.

Fig. 2 Radio thin-layer chromatogram of tetracycline - ^{3}H - hydrochloride.

Solvent: 10 % aqueous citric acid solution saturated with n-butanol.

Thin layer: Eastman-Kodak Silicagel-G.

Fig. 3 Radio thin-layer chromatogram of antipyrine - ^{3}H.

a) immediately after preparation.

b) after five months' storage at room temperature.

Solvent: methanol.

Thin layer: Eastman-Kodak Silicagel-G.

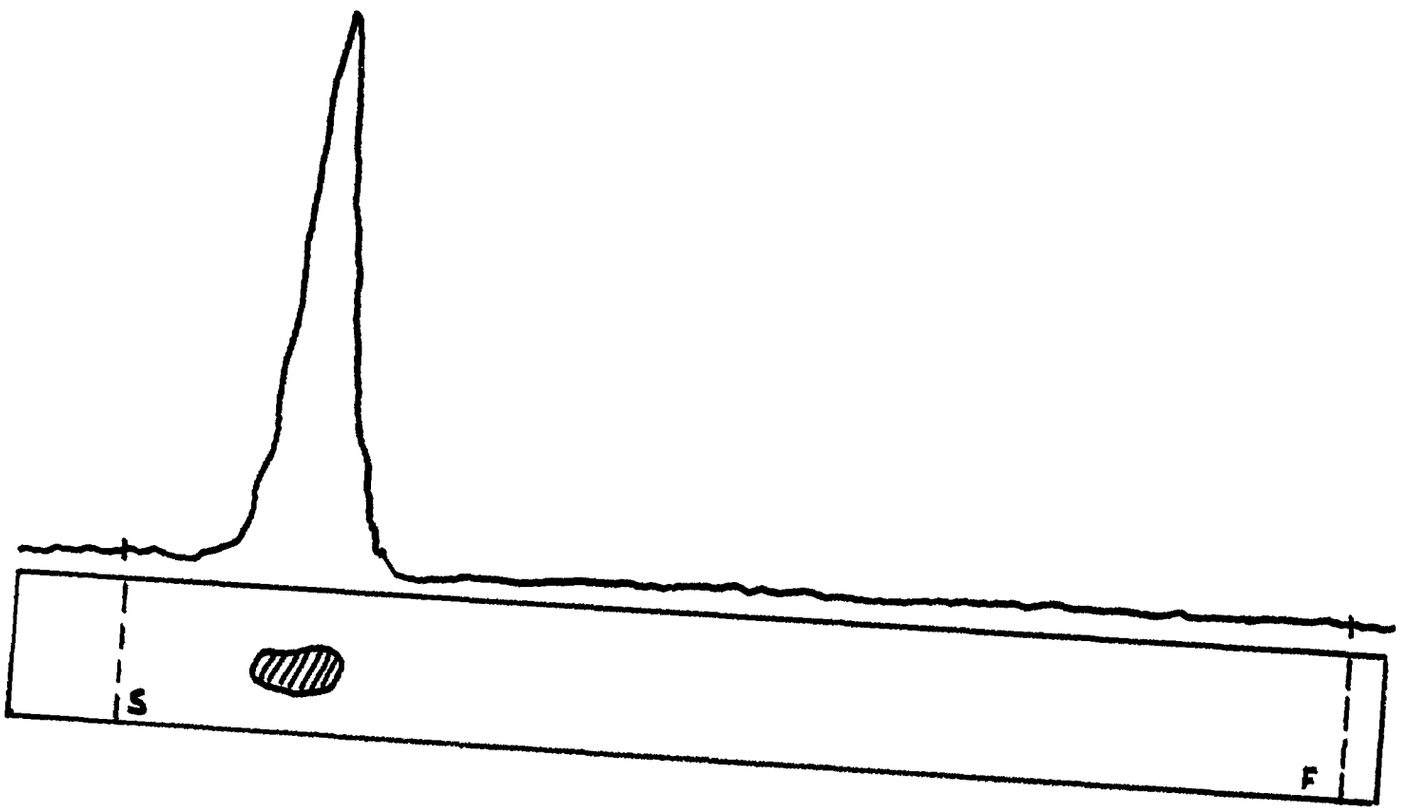


FIG. 1

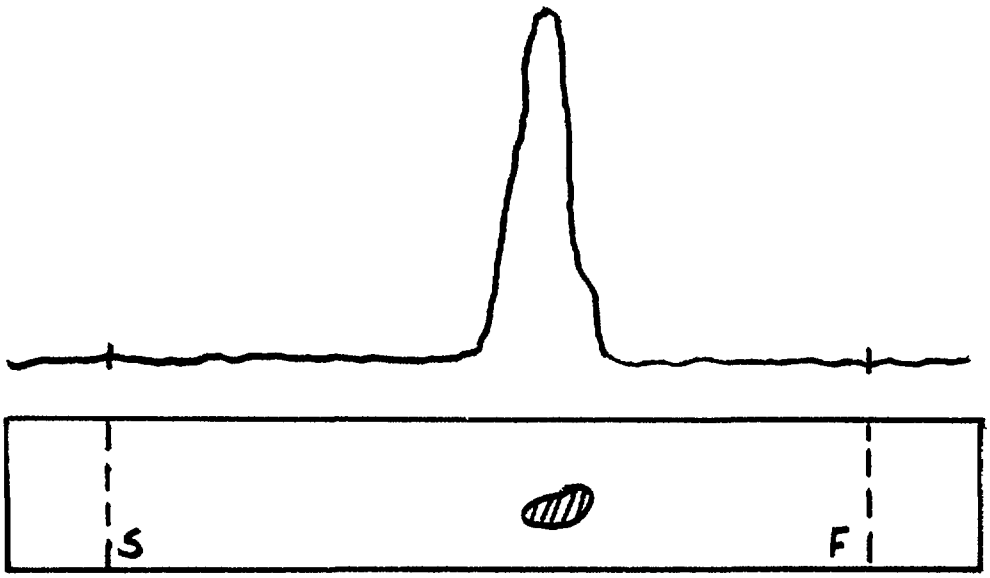
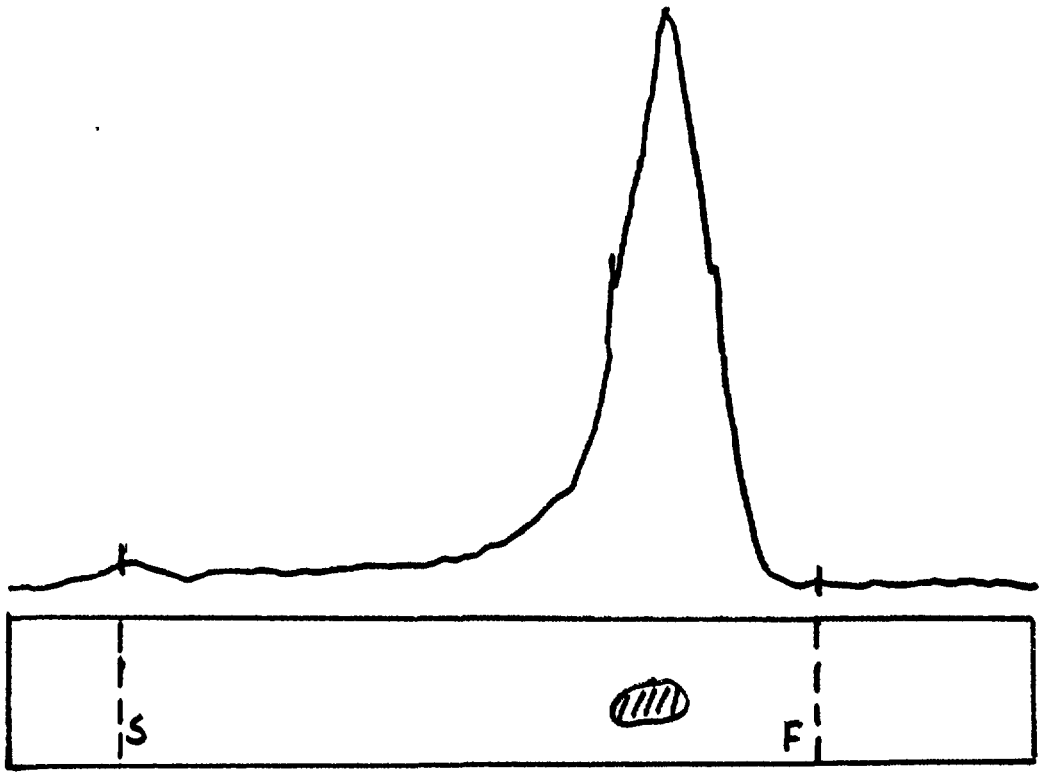
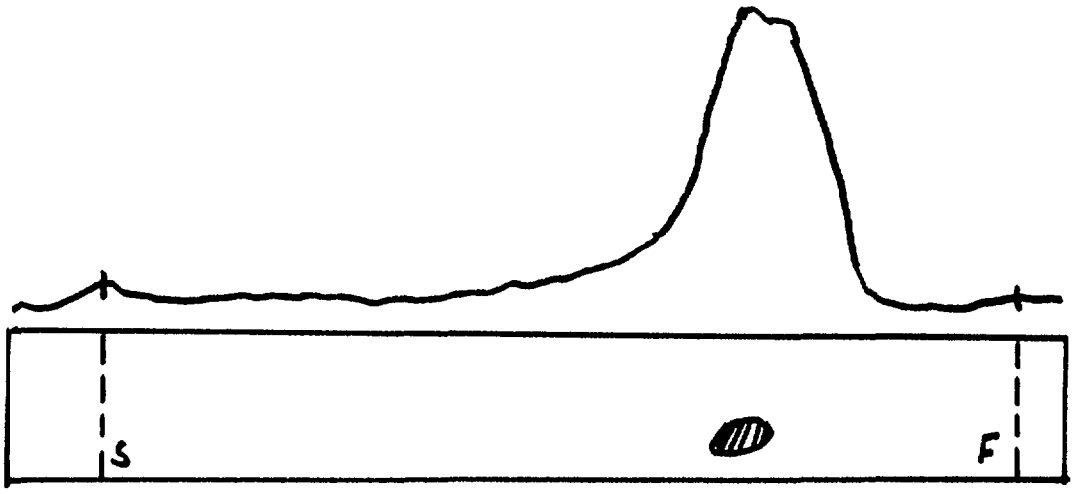


FIG. 2.



a.)



b.)

FIG. 3.

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