Thanks are also due to Dr. M. R. Deshpande for discussions. The computational help by Mr. K. C. Patel and Miss Chhaya Shah is also thankfully acknowledged.

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PREPARATION OF THIN UNIFORM BETA-RAY SOURCE*

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ABSTRACT

An improved technique of electrospraying method for preparation of thin uniform beta-ray source deposit is described. The resolution of the F-conversion line of ThB source prepared by this method is comparable to that of the ion collection method. The arrangement is simple and found more efficient than the other methods.

CSEVERAL methods for preparing a thin betasource have been suggested by Yaffe¹. Among them, the electrospraying method was preferred by many workers²⁻⁶ for preparing β -source. The advantage of this technique is that the loss of the solute is small and thin substrates do not get overheated. The electrospraying method was first proposed by Carswell and Milsted². Bruninx and Rudstem³ used this technique and investigated a number of variables. Vardingh and Lauer preferred hypodermic needle connected to the glass container which allowed a

* The work was done at Bedford College, London University, U.K.

constant flow rate. Michelson and Richardson's used a straight capillary, whereas Michelson⁶ found pyrex capillary bent twice at right angles more suitable. This short communication describes an improved experimental arrangement for preparing thin uniform beta-source by electrospraying method.

EXPERIMENTAL

Fig. 1 shows the experimental arrangement for preparing a thin uniform beta-source. The apparatus consists of a circular brass plate (1) covered with plastic sheet in order to protect the metallic surface against containination. A thin aluminised film of VYNS resin¹ was fixed to an aluminium ring which was placed

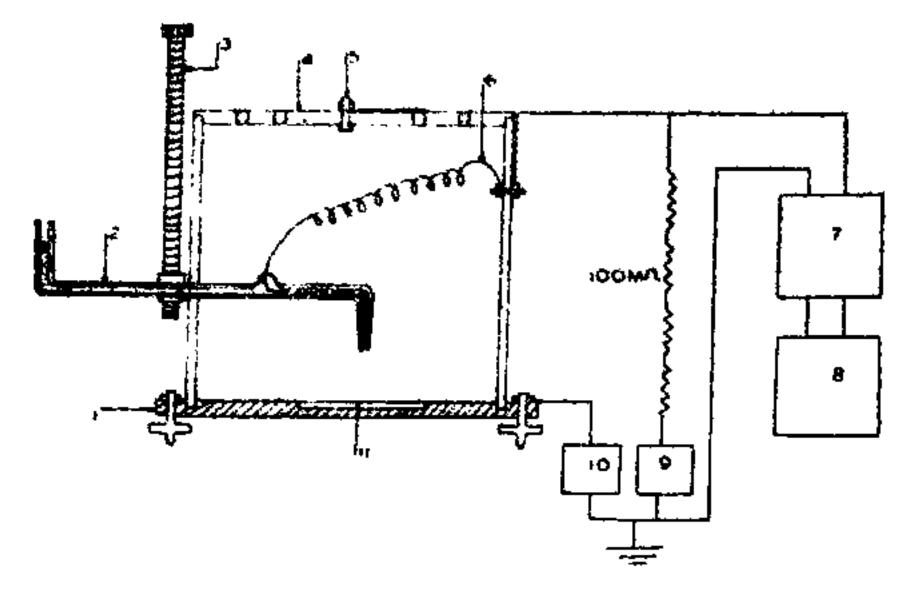


Fig. 1. Arrangement for preparing thin uniform beta source by electrospraying method. 1. Brass plate covered with plastic sheet, 2. Pyrex capillary, 3. Adjustable brass thread, 4. Perspex box, 5. Handle to take out the lid, 6. Pt-Ir wire, 7. High frequecy oscillator (output 0-8 kV), 8. Power supply, 9. Micro-ammeter, 10. Galvanometer, 11. Thin film fixed to Al-ring.

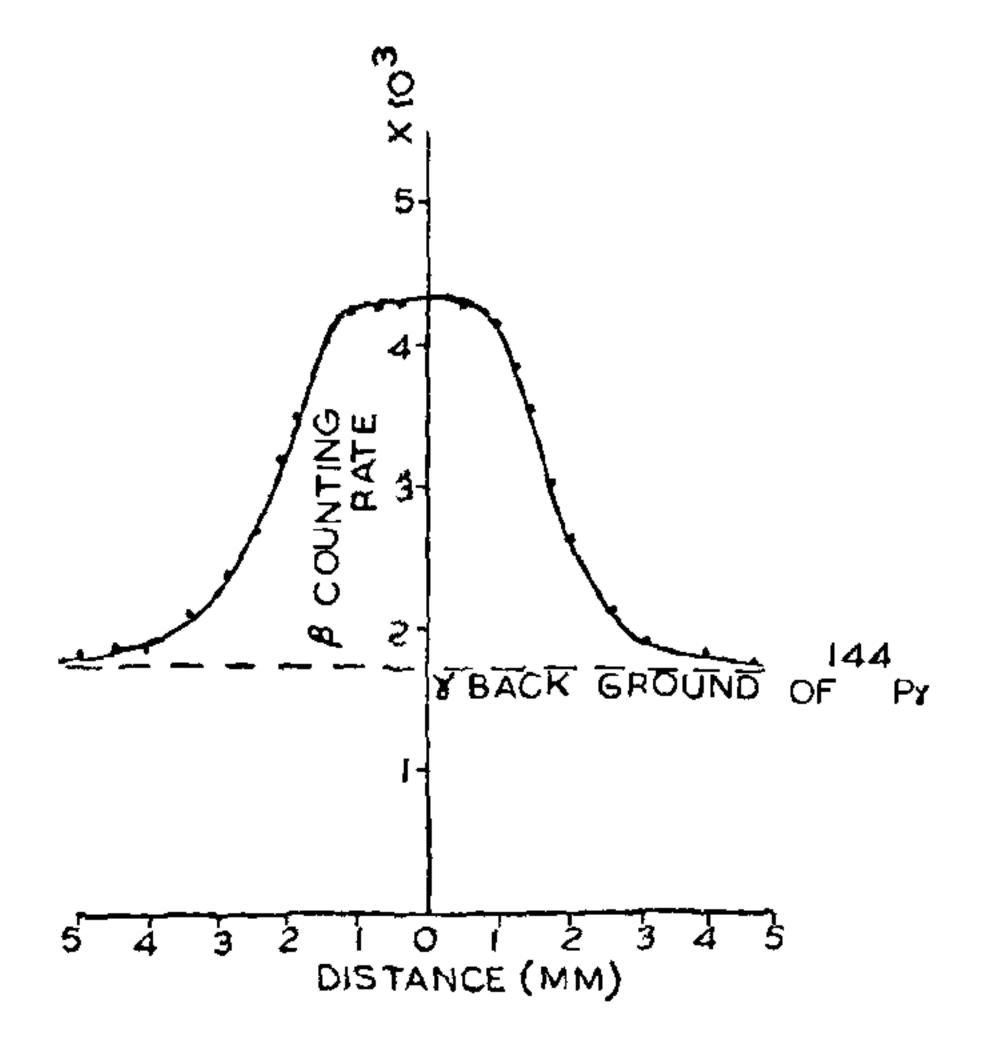


Fig. 2. Uniformity plot of 141Ce-source depostit7,

at the centre of the plate. The walls and top of the box are made of perspex. The lid is provided with a few holes for ventilation. The whole system stands on leavelling screws. A capillary jet of pyrex tube of 0.4 mm I.D. was drawn, the optimum length of the capillary jet being 2 cm. The tube was bent twice at right angles. The horizontal position of the tube keeps the hydrostatic pressure constant. The diameter of the hole at the end of the capillary jet was 0.23 mm whereas that of the central wire was 0.224 mm. The end of the wire was kept nearly 1 mm above

the tip of the tube. A central wire of Pt-Ir was preferred as it is not attacked by acid nor does it undergo corrosion. The tube can be moved up and down by adjusting the brass thread. The horizontal position can be adjusted by sliding the tube through the hole. The discharge phenomena and formation of drops at the tip can be seen through a microscope placed in front of the box and using a lamp at the back. The anode is connected to the central wire while the metallic base was earthed. The EHT up to 8 kV was employed from the high frequency oscillator.

For obtaining a good spray, it was essential to degrease the wire and the capillary with chromic acid and wash them with distilled water. A mixture of alcohol and water in the ratio of 2:1 was found to be a good solvent for the material.

Preparation of the Beta-ray Sources

The ThB-source was deposited on the platinum spiral by ion collection method. The active deposit from pt-wire was dissolved in a few drops of N/10 HCl and then dried under an infrared lamp. It was redissolved in the mixture of distilled water and alcohol. Nearly 50% of the active deposit was taken out of the wire. The radioactive solution was taken into the capillary and sprayed on the thin film. The resolution of the ThB-F line with 7 mm diameter source deposit measured with the beta ray spectrometer was comparable to that obtained by ion collection on 5 mm diameter metallic screw?. This reveals an indirect evidence for the uniformity of the source prepared by electrospraying method.

The 144 Ce-Source

The ¹⁴⁴Ce-source was obtained from the Radiochemical Centre at Amersham as a solution in 1 NHCl acid. A few drops of the solution were evaporated under an infrared lamp and then dissolved in dil. N/10 HNO₃. After drying the nitrate was redissolved in the solvent and sprayed on the film as before.

The current value for obtaining a thin uniform source was 10 μ A.

Uniformity of the Source Deposit

The uniformity of the source deposit was tested by counting the β -particles with a plastic scintillator. A typical plot is shown in Fig. 2. The flat top of the curve shows the uniform deposit of the source. The diameter of the source deposit was estimated to be 7 mm^7 . With this source, the decay scheme of ¹⁴¹Ce has been studied and many low energy lines were successfully resolved.

ACKNOWLEDGEMENT

The valuable discussions of Prof. H. O. W. Richardson of Bedford College, London University, are gratefully acknowledged.

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SYNTHESIS OF PROSOGERIN-B

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ABSTRACT

Constitution assigned to prosogerin-B isolated from Prosopis spicigera as 2', 4-'dihydroxy-5'methoxy-3, 4-methylenedioxychalkone (I) has been confirmed by its synthesis.

ROSOGERIN-B isolated¹ from *Prosopis spicigera* was proposed its constitution as 2', 4'-dihydroxy-5'methoxy-3, 4-methylenedioxychalkone (I) on the basis of colour reactions, spectral data, degradative studies and its derivatives. This communication reports the confirmation of the proposed structure by synthesis. For this purpose, the chalkone (I) and its isomeric 5'-dihydroxy-4'-methoxy-3, 4-methylenedioxychalkone (iso-prosogerin-B) (II) were made using 2-hydroxy-4-benzyloxy-5-methoxyacetophenone² and 2-hydroxy-4methoxy-5-benzyloxyacetophenone³ as the starting compounds respectively. 2-Hydroxy-4-methoxy-5benzyloxyacetophenone on condensation with piperonal gave 2'-hydroxy-4'-methoxy-5'-benzyloxy-3, 4-methylenedioxychalkone (III) that was debenzylated to obtain the chalkone (II). Similarly 2-hydroxy-4-benzyloxy-5methoxyacetophenone was used to obtain 2'-hydroxy-4'benzyloxy-5'-methoxy-3, 4-methylenedioxychalkone(IV) which was debenzylated to obtain the required chalkone (I). Identity of prosogerin-B with the synthetic chalkone(I) but not with the isomeric chalkone(II), has now unequivocally confirmed its proposed constitution(I).

Further, prosogerin-A, 6-methoxy-7-hydroxy-3', 4'methylenedioxyflavone(V) also isolated from *Prosopis* spicigera has been obtained using 2'-hydroxy-4'-benzyloxy 5'-methoxy-3, 4-methylenedioxychalkone (IV). For this purpose, the chalkone (IV) when treated with selenium dioxide, underwent cyclodehydrogenation4-6 to give б-methoxy-7-benzyloxy-3','4'-methylenedioxyflavone (VI). On debenzylation, the flavone (VI) gave 6-methoxy-7-hydroxy-3', 4'-methylenedioxyssavone (V) which agreed with the authentic sample of prosogerin-A.1, 8.

(I) $R_1 = H$; $R_2 = CH_3$ (II) $R_1 = CH_3$; $R_2 = H$

(VI) $R = C_1H_1$

(III) $R_1 = CH_3$; $R_2 = C_7H_7$

(IV) $R_1 = C_7H_7$; $R_2 = CH_3$

EXPERIMENTAL

2'-Hydroxy-4'-methoxy-5'-benzyloxy-3, 4-methylenedioxychalkone (III):

A solution of 2-hydroxy-4-methoxy-5-benzyloxy acetophenone³ (1 g) and piperonal (1.6 g) in ethanol (40 ml) was treated with aqueous potassium hydroxide (1.8 g in 3 ml) and then left at room temperature for 48 hr. The reaction mixture when acidified with hydrochloric acid in cold, gave the chalkone(III) as a yellow solid that was filtered, washed and died. It crystallised from ethyl acetate-petroleum ether as yellow needles (0.9 g), m.p. 145-47° (Found: C, 71.5; 11, 5.3. $C_{24}H_{20}O_{6}$ requires C, 71.28; H, 4.99%). It gave brown colouration with alcoholic ferric chloride,

2', 5'-Dihydroxy-4'-methoxy-3, 4-methylenedioxychalkone (iso-prosogerin-B)(II):

A solution of chalkone (III) (0.8 g) in glacial acetic acid (15 ml) was treated with hydrochloric acid (2 ml) and then heated on a boiling water-bath for 6 hr. The reagents were removed by distillation under reduced pressure and the reaction product thus obtained when treat d with water, yielded the required chalkone (II)