

plates give a distinct advantage, uniformity of dose rate being found within 10 per cent over  $0.7d$ ; this is again to be compared with  $0.5\delta$  in the case of the corresponding cylinder.

#### A comparison of the irradiation facilities offered by two parallel rectangular plates and by a hollow cylinder

The uniformity of the dose distribution within an irradiation enclosure is one of its most important characteristics; the greater the volume of uniform exposure, the greater the productivity of the unit. For special purposes such as the irradiation of rabbits and dogs, whose bodies are roughly cylindrical in shape, the cylindrical enclosure finds a natural application. However, for flat objects the arrangement with two parallel rectangles is preferable on account of its greater capacity in the  $y$  direction. This may be illustrated by comparing the outputs of the two alternative installations in a case where it is desired to irradiate, uniformly within 10 per cent, an object having the shape of a right rectangular parallelepiped. Suppose the enclosure is 1 metre long and the time taken to pass through is one minute. We consider a cylinder of diameter  $\delta = 25.44$  cm; the allowed transverse dimension is then  $0.5\delta$ , i.e. 12.72 cm. Putting the density  $\rho = 1$  for simplicity, the capacity is

$$P = 0.5\delta \times 0.5\delta \times L\rho = 12.72 \\ \times 12.72 \times 100 \times 1 = 16.18 \text{ kg}$$

If the total activity of the cylinder is  $Q = 1000$  mg equiv. of radium, the dose rate at the centre will be  $0.291 \text{ r min}^{-1}$ . The corresponding rectangular array is of spacing  $H = 25.44$  cm and has the same surface area as the cylinder; the plates are therefore of width  $d = \frac{1}{2}\pi\delta = 40$  cm. The required uniformity of dose can be obtained over a cross-section of  $0.5H \times 0.7d$ ; the capacity is accordingly

$$P = 0.5H \times 0.7d \times L\rho = 35.616 \text{ kg}$$

with a dose rate at the centre of  $0.21 \text{ r min}^{-1}$ . The productivity of the rectangular array is seen to be 1.6 times that of the equivalent hollow cylinder.

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#### References

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### All-glass Apparatus for Micro-synthesis of Radioactive Solids

(Received 6 January 1958)

THE preparation of milligram amounts of radioactive organic compounds frequently calls for elaborate apparatus and shielding devices which are not always readily available in small laboratories. Syntheses resulting in 15–30 mg of radioactive solids of high specific activity, using relatively short lived radio-isotopes, and involving reaction with continuous stirring, precipitation, washing and drying of the resultant compound in a short period of time, usually involve several pieces of apparatus, several manipulations, or a reaction train. We have recently developed an inexpensive all-glass apparatus to handle such preparations. The preparation of radio-iodinated 2-amino-3,4-di-iodobenzoic acid (specific activity  $400 \mu\text{c per mg}$ ) is described as an example of the use of this apparatus.

#### Materials and methods

##### Construction of apparatus

Fig. 1 is an exploded view of the apparatus. The reaction chamber consists of a filter stick (Corning No. 39570–10F) cut down to  $2\frac{1}{2}$  in. length and having a capacity of about 5 ml. It is sealed below the sintered glass disc to a 14/20 male standard taper joint, which fits into a 14/20 female joint at the bottom of a small glass cup. The latter has a male 24/40 joint at its lower extremity, and serves as a receptacle for circulating hot or cold water, or for crushed ice. The entire assembly fits into the neck (24/40) of a 50 ml filtration flask. The assembled apparatus is  $6\frac{1}{2}$  in. high, which allows efficient shielding with a minimum number of lead bricks.

The sintered glass plate in the reaction chamber serves as a floor for reacting solutions. This plate will not allow the passage of liquids by gravity over a period 2–3 hr. Continuous stirring is performed by slight positive pressure (air or inert gas) through the side of the filtration flask; slight positive pressure is necessary to eliminate passage of liquids through the filter plate over periods longer than 3 hr. Waste liquors or reaction products in solution are drawn off into the flask by suction via the same route. Washing and drying of precipitates is readily performed by the same means. Glass hooks on the apparatus allow the use of spring clips between components, to permit the use of positive pressure.

*Use of apparatus in the preparation of radio-iodinated 2-amino-3,4-di-iodobenzoic acid*

In a separate container, 130 mc of  $\text{NaI}^{131}$  (carrier free) was evaporated to about 0.2 ml at  $110^\circ\text{C}$ ; a

gentle flow of nitrogen was allowed to impinge on the liquid surface, to eliminate boiling. When cool, 0.45 ml of a solution of iodine monochloride in glacial acetic acid (0.04 ml ICl in 0.75 ml acetic acid) was introduced and mixed;  $I^{131}Cl$  was rapidly formed by exchange. 0.25 ml of a solution of anthranilic acid in glacial acetic acid (30 mg anthranilic acid in 0.25 ml acetic acid) was placed in the reaction chamber, and stirred with a gentle air current. 0.45 ml  $I^{131}Cl$  in glacial acetic acid was added dropwise, (using a distance pipette) over a 5 min period, after which the  $I^{131}Cl$  container was washed out twice with 0.5 ml volumes of glacial acetic acid, these washes being combined in the reaction chamber. After 20 min of continuous stirring, 3 ml water were added, mixed thoroughly, air stirring stopped, and negative pressure applied to draw the precipitate down on the filter disc. The sides of the reaction vessel were washed down with a thin stream of water, and the washed precipitate allowed to dry over a 20 min period, by suction. The crude radioactive 2-amino-3,5-di-iodobenzoic acid can be purified by solution in ammonium hydroxide and reprecipitation with hydrochloric acid, the process being repeated as often as necessary. The method used is a modification of the original method of WHEELER and JOHNS<sup>(1)</sup>.

### Results

By the method described, 20 to 25 mg of radioactive 2-amino-3,5-di-iodobenzoic acid can be prepared and purified in a total time of a little over an hour, in a single reaction vessel. The specific activity of the compound was 400  $\mu$ c/mg.

### Discussion

The preparation described above is an example of a multistage microsynthesis which can be performed rapidly and with a minimum of manipulation in the apparatus described.

The flexibility of the apparatus is considerable. The material prepared can be subjected to several further reactions yielding a solid product, or a liquid form or solution of a further reaction product, in the same reaction vessel, the product in the latter instance being collected merely by substituting a clean 50 ml flask. For example, the substituted benzoic acid could be diazotized after drying and coupled with proteins for immunological studies. Replicate units can easily be set up simultaneously, or reaction vessels can be interchanged indefinitely by substituting fresh reaction chambers. Conversion for use with volatile solvents, involving positive pressure, is accomplished by modification of the top of the reaction vessel to accommodate a female 19/38

socket, engaging with a lead-in line ending in a male 19/38 plug (not shown in the illustration).

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## Micro Counter Tubes for Beta-gamma Measurements

(Received 30 December 1957)

BETA-GAMMA sensitive micro counter tubes of the Maze type have been developed for radioisotope tracer work. The tubes can be sterilized and used in humans for medical diagnosis by radioisotope uptake and for biological research. Flexible lead wires are hermetically sealed to the metal-encased glass tubes. Volumes range from 2 mm<sup>3</sup> to 850 mm<sup>3</sup>, and wall thicknesses range from 0.4 MeV to 0.2 MeV. Thinner windows have been made in some types. Life is  $>10^7$  counts, with helium filling. Practical beta detection sensitivity is 0.001 to 0.01  $\mu$ c at 1.0 cm distance in air. Similar tubes 1.0 mm o.d., 6 mm to 30 mm long have been made for interchangeable use in needle-type probes. Techniques of construction and typical  $\beta$  response characteristics to Sr- $Y^{90}$ , Co $^{60}$ , Y $^{90}$ , and Ra  $\gamma$ -rays are given.

### 1. Introduction

An obvious requirement for the measurement of the weakest radioactivity is that the sensing element respond to individual particles or quanta. Some form of counter that gives digital indication is accordingly required, rather than a macroscopic device such as an ionization chamber or semiconductor. Detectors for internal use in medical diagnosis by radioisotope uptake in humans, or for biological experiments in animals, must be as small and mechanically flexible as possible. Scintillation detectors have limitations due to the size of the multiplier tube and the necessity for use of a rigid probe; otherwise they would be ideal for this work.

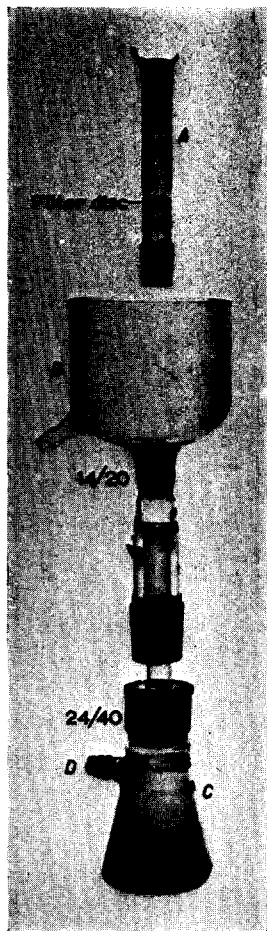


FIG. 1. Exploded view of apparatus

- A. Reaction chamber with 14/20 standard taper joint.
- B. Bath with 14/20 joint and 24/40 joint.
- C. 50 ml Erlenmeyer filtration flask with 24/40 joint.
- D. Side arm for application of positive or negative pressure.