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# GROWING OF ORGANIC PHOSPHORS FOR SCINTILLATION COUNTERS

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(*Received for publication August 9, 1956*)

**ABSTRACT.** A simple furnace has been designed and constructed for organic phosphor-crystals. The furnace is described and its mode of operation explained. Conditions for good crystal growth are discussed.

## INTRODUCTION

Organic compounds, like anthracene, stilbene, diphenyl, etc. are now commonly used for detection of nuclear radiations when they are used in conjunction with photomultipliers. There are three main features which a phosphor must possess in order to be useful as a radiation detector, viz. (a) the material should be crystallisable in big form so that the mass absorption coefficient for $\gamma$-rays is high, (b) the fluorescent band produced by the phosphor when excited by the incident radiation should correspond to the spectral response of the photomultiplier tube, (c) the scintillation decay time should be very small. A simple arrangement for growing fairly large-size organic crystals which conform to the above three features to a quite satisfactory extent and can be conveniently used for detecting alpha, beta and gamma rays is presented in what follows. A review on the mechanism of crystal growth has been published in the Proceedings of Faraday Society (1949), No. 5.

Growing of organic crystals for scintillation counters is greatly affected by the state of purity of the sample used. It has been found that the same material of different manufacturers exhibits different spectral characteristics. Purity of the sample is also important for growing big single crystals. So when samples of of requisite purity are not readily available they have to be purified by the absorption chromatographic methods. It is also important to remove the suspended impurities. Because of the great importance of these crystals, crystal growing has undergone extensive research, theoretical and experimental in all industrial countries. The review of such works can be found in literature. But in most cases technical details are lacking. In the present work we have presented the details as far as necessary.

*Part of this work was reported at the Indian Science Congress in January 1956.

In general organic cyrstals can be grown from solutions or from melts ; for our purpose the latter is most suitable and can be accomplished in two ways (a) the stationary crucible method (Stockberger, 1936, Huber, 1949) and (b) the dropping crucible method (Leninger, 1952). A furnace was erected earlier following the method (a). The temperature control had to be done manually which is extremely tedious.

Later another furnace has been developed using the method of dropping crucible. The same principle has been employed by Leninger for growing stilbene. The furnace developed here is much more simple and can be employed for growing any organic sample having a melting point less than 250°C. The dropping of the crucible was initially automatically controlled by a water-clock which was later replaced by a motor and gear arrangement.

## DESCRIPTION OF THE FURNACE

The furnace is described in figure 1. (1) is the melting furnace and consists of a copper tube of diameter 2″ and length 13.5″ on which nicrome wire has been evenly wound keeping proper insulation and the whole of it again is wound by asbestos paper. (2) is the annealing furnace made of pyrex tubing of diameter 3″ and length 19.5″ with nicrome wire wound around it. (3) is the crystallising furnace described separately, (4) are soft glass envelopes of diameter 4.8″(4a) is pyrex tube within which the container with the grown crystal rests at the end. (5) is the wheel which supports the crucible through a wire, and the spindle of the wheel is connected to a motor and gear arrangement (6) which controls the rate of fall of the cruicible. The whole arrangement rests on a base plate (7) and cast iron structure (8). The current in the two furnaces are separately controlled by two variacs (9). The indicator lamps (10) show whether a furnace is running and the meter (11) records the current. It has been found that during the process of crystallisation the slightest mechanical shock given to the crystal container disturbs the crystal growth and as such the whole arrangement has to be made shock-proof. This is achieved by placing the furnace on sand kept in an wooden box of proper size, the box itself, in its turn, being placed on shock-absorbing rubber-studs (not shown in the diagram).

The crystallising furnace is shown in figure 2. In the crystallising furnace the spece between the melting furnace and the annealing furnace is enclosed with heat-insulating material like asbestos. There are two thick brass plates (*a*) and (*b*) at top and bottom clampped to syndenio plates (*c*) and (*d*) and with a thin walled copper cylinder (*e*). The whole enclosure is packed with asbestos keeping space for (i) the crystal container to drop vertically at the centre, (ii) a dimetrical hole (*f*) by which the crystal growth can be seen and (iii) three small horizontal holes for inserting thermocouples to measure temperature. In the viewing hole the mica sheets provide heat insulation.

The sample from which the crystal has to be grown is distilled in an arrangement shown in figure 3, under reduced pressure. The crystal container of pyrex-

Fig. 1

tubing also shown in figure 3 is taken out and sealed. The conical shape of the bottom has been found important and necessary as it facilitates the growth of the seed crystal.

The temperature characteristic of the crystallising furnace is first studied for various values of the currents flowing through the melting furnace and the annealing furnace. It has been found that the temperatures become steady in about four hours and any steady temperature gradient can be maintained inside the crystallising furnace and that is not much affected by the daily variation of the room temperature. The rate at which the container is lowered is also measured

and this rate is controlled by the rate of water drops falling at (6) of figure 1. It is not difficult to obtain a rate of falling of the container equal to 1/8" per hour.

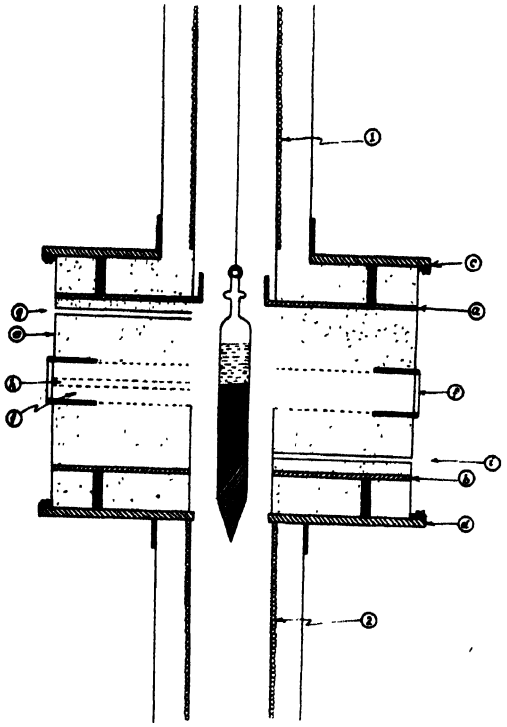

Fig. 2

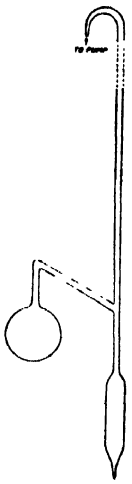

Fig. 3

*Growing of the crystal*: The sample sealed in the container is hung by means of a fine wire from the wheel above (5) in figure 1, through a fine hole at the centre of the syndenio plate at the top of the melting furnace. The furnaces are mounted vertically so that the container can reach the bottom without touching the sides anywhere. Initial position of the container in the melting furnace is adjusted by putting requisite amount of water on the water-float bath. The furnaces are then started. The melting furnace should be set at about 10°C above the melting point of the sample so that the sample is brought into the liquid state. At a temperature about 3°C above the crystallising temperature the molten mass comes to a viscous state and it has been found that good crystals can not be grown if we start from this state. In the case of diphenyl it is kept at 85°C (melting point 71°C). The temperature at the top of the crystallising furnace has to be kept at about 75°C, 69°C at the middle and about 60° at the bottom. The correspond-temperatures for stilbene are 130°C, 120°C and 103°C respectively. In our case copper-constantan thermocouples have been used to measure these temperatures. Occasionally, during the growth of the crystal these temperatures have to be checked. The thermo-e.m.f. measurements have been made with a Tinsley potentiometer within an accuracy of $\pm 0.5$°C.

With the help of this apparatus single crystals of stilbene and diphenyl have been grown. Fairly large crystals of diameter upto 1″ and length 3″ have been grown. After cutting and proper polishing they have been used in conjunction

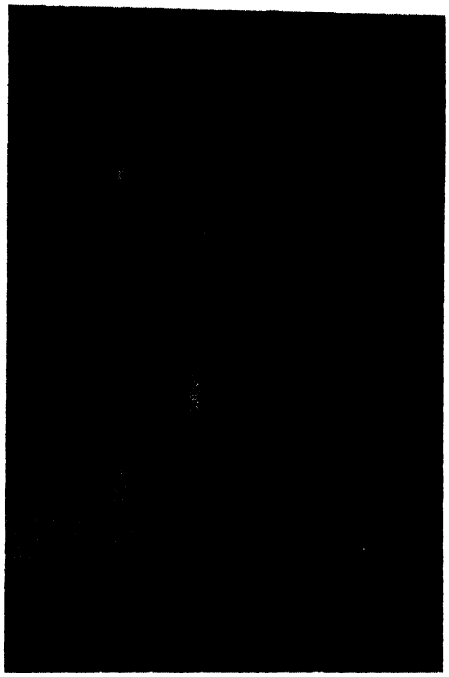

Fig. 4. Photograph of an unpolished stilbene phosphor. The scale is in inches.

with photomultipliers where they have given satisfactory service. A photograph of the crystal is shown in figure 4.

ACKNOWLEDGMENT

The authors are indebted to Late Prof. M. N. Saha, F.R.S. for his constant encouragement and to Prof. A. K. Saha for very helpful suggestions during the progress of the work.

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