

## Laboratory Notes

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### A modified diffusion apparatus for the growth of single crystals

Single crystals of insoluble compounds can sometimes be grown by diffusion of two solutions which interact to form the solid by metathesis. Minimization of convection and removal of crystals without further mixing of the solutions are often difficult. These two problems have been avoided by a modification of the gel-diffusion apparatus of Armington & O'Connor (1968).

The apparatus (Fig. 1) consists of two 125 ml round-bottom flasks which serve as reservoirs for the reactant solutions. Medium-porosity fritted-glass disks are mounted in the side arms, and the flasks are connected, through spherical joints, by a diffusion tube. The diffusion tube is held in place by the use of clamps and the entire apparatus is supported with tripods or ring stands.

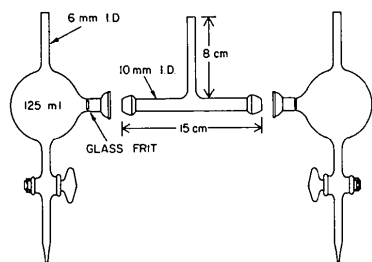


Fig. 1. Diagram of the apparatus.

In use, the apparatus is assembled, and the reservoirs are filled to the level of the bottom of the diffusion tube with the reagent solutions. The center tube is then filled with water to prevent rapid mixing of the reagents. After the formation of the crystals in the diffusion tube, the reservoirs are slowly drained simultaneously through the stopcocks until the levels are below the center tube. The apparatus is disconnected and the crystals removed.

The apparatus has been used to grow single crystals of yttrium, zinc, cobalt and cadmium anthranilates with a solution of sodium anthranilate in one reservoir and a solution of metal salt in the other (Proding, 1940). These crystals are comparable in quality to the single crystals used to determine the structure of the copper anthranilate (Lange & Haendler, 1975). Growth requires 2–3 weeks for well-shaped crystals suitable for X-ray

work. Structural studies of the yttrium and zinc anthranilates are currently in progress.

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### A simple back-reflexion camera

The camera consists essentially of two components: 1, a truncated metal hypodermic needle and 2, a block of metal in which is set a countersunk hole (see Fig. 1). The assembly acts as a film punch, film holder and X-ray collimator.

A suitable-sized hypodermic needle is cut such that the end which normally seats on the syringe forms a stop of the minimum practical diameter; the other end is cut about 15 mm away, then sharpened conically to provide a circular knife edge. The hole in the metal is a clearance fit for the hypodermic tubing at

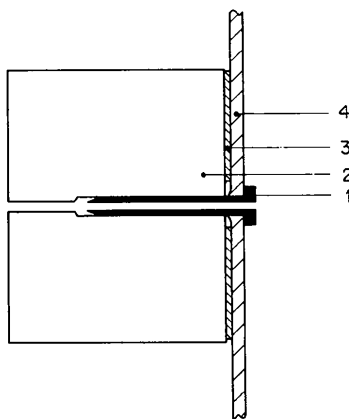


Fig. 1. The back-reflexion camera.

one end and is slightly narrower at the other. This is to prevent X-ray fogging of the film and to minimize secondary scattering.

In operation, the hypodermic needle is made to perforate a sheet of wrapped X-ray film (4 in the figure) by a light hammer-blow against a Perspex block. Light-sealing is excellent when using Kodak Polysoft dental X-ray film. The punchings can be cleared from the bore using a fine needle or stiff wire of the appropriate diameter and the needle/film assembly may be fitted into the solid block of metal. A coating of double-sided adhesive tape on the block of metal, 3, ensures that the X-ray film cannot rotate during the exposure and that the film is held at right angles to the X-ray beam. However, if the hypodermic needle is a sufficiently tight fit in the hole, this precaution is unnecessary.

Many useful exposures have been made at this laboratory using cameras of this type.

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## Crystallographers

*This section is intended to be a series of short paragraphs dealing with the activities of crystallographers, such as their changes of position, promotions, assumption of significant new duties, honours, etc. Items for inclusion, subject to the approval of the Editorial Board, should be sent to the Executive Secretary of the International Union of Crystallography (J. N. King, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England).*

Professor P. P. Ewald's 90th birthday is being marked by a special symposium at the March 1978 meeting of the American Crystallographic Association. The symposium will be held on 23 March and the first speaker will be Professor Ewald, who will present a review of his work on the dynamical theory.

Sir Peter Hirsch, Isaac Wolfson professor of metallurgy at the University of Oxford, and Dr H. E. Huxley, MRC Laboratory of Molecular Biology, Cambridge, have been awarded Royal Medals for 1977. Three Royal Medals are awarded annually by Queen Elizabeth upon the recommendations of the Royal Society. The award to Sir Peter Hirsch is for his studies of the struc-

tures and properties of imperfect crystals, and his determination of the atomic and crystallographic processes which enable strong alloys to be hardened by plastic working. The award to Dr Huxley is for his research on the structure of muscle and on the molecular mechanisms of the contractile process.

Dr **M. L. Huggins** celebrated his 80th birthday in 1977. This occasion was marked by a special symposium held in his honour at the Polytechnic Institute of New York on 15 October 1977. Speakers included Professor **P. J. Flory**, Professor **L. Pauling**, Professor **P. P. Ewald**, Dr **A. Weissberger**, Professor **H. Mark** and Dr Huggins.

Professor **W. Klyne**, Professor of Chemistry, Westfield College, University of London, died on 13 November at the age of 64. It was through his efforts that the MRC Steroid Reference Collection was based at Westfield College. His publications included *The Chemistry of Steroids* (1957) and the multi-volume *Atlas of Stereochemical Correlations* (1974).

Professor **R. Mason**, who has been Professor of Chemistry at the University of Sussex, Brighton, England, since 1971, has been appointed to succeed Sir Hermann Bondi as Chief Scientific Advisor in the UK Ministry of Defence.

Professor **Herbert O'Daniel** died on 15 August 1977. From 1947 until his retirement in 1971 he was Director of the Mineralogisches Institut der Johann Wolfgang Goethe-Universität in Frankfurt am Main. His interests in crystallography were widespread and included structure analysis, by means of X-ray and neutron diffraction, in crystal chemistry and in mineralogy.

He completed his studies for his doctorate in Heidelberg in 1930, and in 1935 he obtained the degree Dr. habil. at the Technische Hochschule München. From 1938 to 1944 he was Head of the X-ray Section of the Kaiser Wilhelm-Institut, later the Max-Planck-Institut für Silikatforschung, in Berlin-Dahlem. In 1947 he was appointed to the full professorship of mineralogy at the University of Frankfurt. There he re-established the Deutsche Mineralogische Gesellschaft (DMG) and initiated the 'Sektion für Kristallkunde', the national organisation for crystallographers in his country.

In these early post-war years he helped establish contacts with the IUCr. From 1950 to 1959 and from 1967 to 1970 he was Secretary of the National Committee for Crystallography of the Federal Republic

of Germany and was Chairman of the delegation to the Eighth IUCr General Assembly at Stony Brook in 1969. He was also a member of the Union's Commission on *Structure Reports* from 1951 to 1957. With the death of Professor O'Daniel a devoted leader in crystallography and mineralogy has been lost.

Dr **F. Sanger**, MRC Laboratory of Molecular Biology, Cambridge, has been awarded the Copley Medal of the Royal Society for his research on the chemical nature of proteins and nucleic acids.

## International Union of Crystallography

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