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A technique for the preparation of low-temperature powder samples of noxious materials*

The determination of crystal structures by neutron powder profile analysis is a widely used technique. To ensure that experimental intensities (and hence the final crystal parameters) are reliable, it is essential to use a polycrystalline sample that is homogeneous, *i.e.* is random and does not contain macroscopic crystalline grains (see, for example, Will, Parrish & Huang, 1983). For materials which are liquid or gaseous at room temperature the preparation of such a sample (typically several cm^3 in volume) is a major experimental problem. In this laboratory we use routinely a technique of mechanically grinding the condensed solid *in situ* at low temperatures (Powell, Dolling, Evans & Nieman, 1980). The method requires access to the powder sample and so condensation of the gas or liquid cannot be done in a totally closed system. This has drawbacks in the case of noxious or toxic samples. We have recently improved the system by the addition of a 'condenser assembly' and this has greatly facilitated the safe handling of even the most unpleasant of samples.

The system is shown schematically in Fig. 1. It consists of two connected coils (condenser and flushing) immersed in a cold bath (liquid–dry ice mixture). The assembly is constructed of glass to allow visual inspection of the condensation process. The condenser coil exits through the base of the bath in the form of a short tip. The entire assembly rests in the neck of the sample chamber (a welded vanadium can) and a seal is made between the tip and the neck by a Tygon gasket. The sample chamber is immersed in liquid nitrogen (or other cryogen) and the entire assembly is contained in an exhaustible filtered fume hood. The temperature of the cold bath can be adjusted by suitable choice of the cooling mixture. The temperature should be chosen so that the condensed sample exits from the condenser tip just above its freezing temperature.

To prepare a sample, the room-temperature gas (from a low-pressure regulator) or liquid is passed through the condenser coil, cooled and then solidifies in the sample chamber. Even when the room-temperature gas cannot be liquified in the condenser coil the gasket prevents leakage of the cooled gas prior to its solidification. When a quantity of solid has been condensed into the chamber the flow of gas (or liquid) is stopped, the flow of nitrogen gas through the flushing coil is started and the assembly is raised from the neck of the sample chamber and moved aside. The condensed solid is then mechanically ground in the sample chamber in the usual manner (Powell *et al.*, 1980). The atmosphere of cold nitrogen around the sample chamber and the flow of cooled nitrogen gas through the tip prevent unwanted condensation of ice in the tip or the chamber. After the cryogrinding procedure the condensing assembly is replaced in the neck of the sample chamber, the flow of cooled nitrogen gas is stopped and condensation of the sample is resumed.

This cycle of condensation followed by cryogrinding is continued until the sample chamber is full of powder. The chamber is then sealed while cold and is maintained cold while being transferred to the cryostat for the neutron diffraction measurements.

With liquid nitrogen as the cryogen we have used this method to prepare excellent powder samples of chlorine and of deuterium bromide. We feel that with the sample chamber immersed in liquid helium samples with very low freezing points, *e.g.* carbon monoxide and nitrogen, may also be prepared in a similar manner. When used with care the technique appears capable of handling most noxious gases or liquids.

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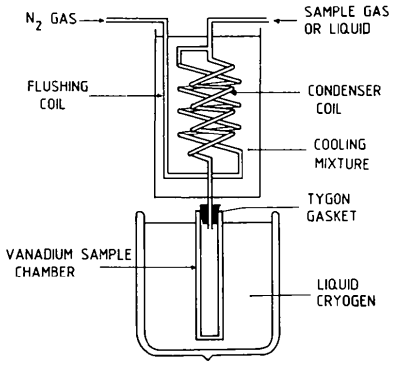

Fig. 1. Schematic drawing of powder preparation system.

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Meeting Report

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The 50th anniversary of the pepsin X-ray photograph

The meeting to celebrate the 50th anniversary of the X-ray diffraction pattern of pepsin obtained by J. D. Bernal and Dorothy Crowfoot (now Hodgkin) was held in Cambridge on Friday 13th May under the auspices of the British Crystallographic Association. The lectures were held in the Austin wing of the Clarendon Laboratory, the site but not the building where the experiments were carried out. There were lectures by Dorothy Hodgkin, Max Perutz and Aaron Klug. These covered the events that led to the experiments and the early protein research (DCH), the early days of haemoglobin studies (MFP) and a review of virus structure, water structure and Bernal's role in their development (AK). Fortunately, many concerned with the pepsin experiment and with the early investigations into other proteins and viruses were able to come to the meeting. The contributions in particular of Bill Pirie, John Philpot and Helen Megaw filled in many gaps and, on occasion, corrected commonly accepted versions of history. There were also some important scientific contributions: the detailed crystal structure of tobacco mosaic virus was discussed by Aaron Klug and the 2·8 Å resolution tomato bushy stunt virus structure, begun by Bernal and Fankuchen, was reported by Steven Harrison from Harvard University. There were many happy impressions from the day: most obvious perhaps was how the excitement produced by the first X-ray protein studies, so vividly brought to life in the lectures and discussions, was jus-

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tified by the spectacular achievements in the field since 1934.

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Crystallographers

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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, 5 Abbey Square, Chester CH1 2HU, England).

Dr **Gopinath Kartha** died of a heart attack at the age of fifty-seven on 18 June 1984. Dr David Harker writes that Kartha had been a member of the staff of the Biophysics Department of Roswell Park Memorial Institute in Buffalo, New York, since November 1959, except for a six-month stay at the University of Madras in 1961. After receiving his PhD in physics at the University of Madras in 1953 and holding postdoctoral positions there, at the Cavendish Laboratory and at the National Research Council of Canada, he joined the Protein Structure Project at the Polytechnic Institute of Brooklyn early in 1959 and moved with it to Roswell Park in Buffalo late that same year. G. Kartha is author or co-author of eighty-nine publications and has presented eighty-two papers at scientific meetings. All dealt with one or other aspect of structural crystallography and the vast majority with the structures of molecules of biological interest. Of greatest importance is his determination of the molecular structure of the enzyme ribonuclease in 1967. This was the first protein structure elucidated and published in the United States. Of almost equal value is Kartha's work with G. N. Ramachandran on the structure of the fibrous protein collagen published in 1954 and 1955. Several of his papers dealt with methods of solving crystal structures using data furnished by X-ray diffraction. Pre-eminent among these is his use of anomalous dispersion to facilitate the location of heavy atoms in crystal structures containing large numbers of light atoms, and of applying this knowledge to finding the phases of the coefficients in the Fourier series representing these structures. This method was of great value in solving the structure of ribonuclease and has since been widely used in solving the structures of proteins and other macromolecules. G. Kartha was a member of Sigma Xi, of the New York Academy of Sciences, and a Fellow of the Indian Academy of Sciences. His election to these honorary societies attests to the importance and originality of his contributions to science. His association with the Protein Structure Project, of which the author of this obituary was director, ensured the accomplishment of its mission. All structural crystallographers and especially those interested in biologically significant substances will keenly regret that Gopinath Kartha is no longer with us.

Dr **W. H. Taylor**, emeritus Reader in Crystallography in the University of Cambridge, died on 14 May 1984 at the age of 79. Dr. R. C. Evans writes that after graduating in physics in W. L. Bragg's laboratory at Manchester University in 1926, W. H. Taylor remained there as a research student and assistant lecturer until 1934 and in these eight years of exceptionally fruitful work published some dozen papers on the crystal structures of silicate minerals. In 1934 the award of a Leverhulme Research Fellowship enabled Taylor to spend two years working first in Cambridge, under J. D. Bernal, and then at the Davy Faraday Laboratory in London under Sir William Bragg. In these years he extended his interests into the field of organic structures but he resumed the study of silicates on his return to Manchester in 1936 as Head of the Physics Department of the College of Technology (now UMIST). He remained in this post until in 1945 he moved to Cambridge as Reader in Crystallography in the Cavendish Laboratory, an appointment he held until his retirement in 1971. It was a challenging task to re-establish the Crystallographic Laboratory after the war but Taylor's reputation attracted research workers in many fields and from many countries. He, himself, was able, with collaborators, to continue his work on feldspars and zeolites and to extend his interests into the field of metals and alloys.

Taylor's scientific talents were combined with exceptional ability as an administrator and diplomatist, and his services outside the laboratory were understandaby in constant demand. He was a member of a number of governmental and other scientific committees, Chairman (1950–52) of the X-ray Analysis Group of the Institute of Physics and later (1955–64) a Vice-President of that Institute. He was actively associated with the organization of the international conference in London in 1946 that led to the foundation of the International Union of Crystallography and he served the Union as Chairman of the Organizing Committee for the Fifth Congress in Cambridge in 1960 and as Chairman of the Programme Committee for the Sixth Congress in 1963.

Taylor's activities did not cease with his retirement: for five years thereafter he edited the *Philosophical Magazine* and simultaneously maintained his interest in feldspar studies in collaboration with an Italian group under Professor Quareni in Padua. In 1979, in recognition of over fifty years of research in mineralogy, he was honoured by the award of the Roebling Medal of the Mineralogical Society of America.

Professor **James A. Ibers** and Professor **Michael G. Rossmann** have been elected members of the National Academy of Sciences. Professor Ibers is leaving the Department of Chemistry at Northwestern University, Evanston, Illinois; he has recently accepted a new position at Santa Barbara.

Dr **Isabella Karle** has been selected by the American Institute of Chemists as one of the 1984 Chemical Pioneers for her work on the symbolic addition procedure for crystal structure determination.

Notes and News

Announcements and other items of crystallographic interest will be published under this heading at the discretion of the Editorial Board. The notes (in duplicate) should be sent to the Executive Secretary of the International Union of Crystallography (J. N.King, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England).

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The M. J. Buerger Award

The first award in the name of Professor Martin J. Buerger is scheduled for presentation at the August 1985 meeting of the American Crystallographic Association in Stanford, California. The purpose of the award is to recognize a mature scientist who has made contributions of exceptional distinction in areas of interest to the American Crystallographic Association. These criteria were deliberately made very broad to reflect the wide range of contributions made by Professor Buerger, which included areas of crystal growth, morphology, structure analysis, phase transitions and instrumentation as well as education through his teaching and many classic text and reference books. Candidates are not restricted as to nationality, race, sex, religion or membership in ACA.