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On the experimental technique of X-ray diffraction in liquid crystals

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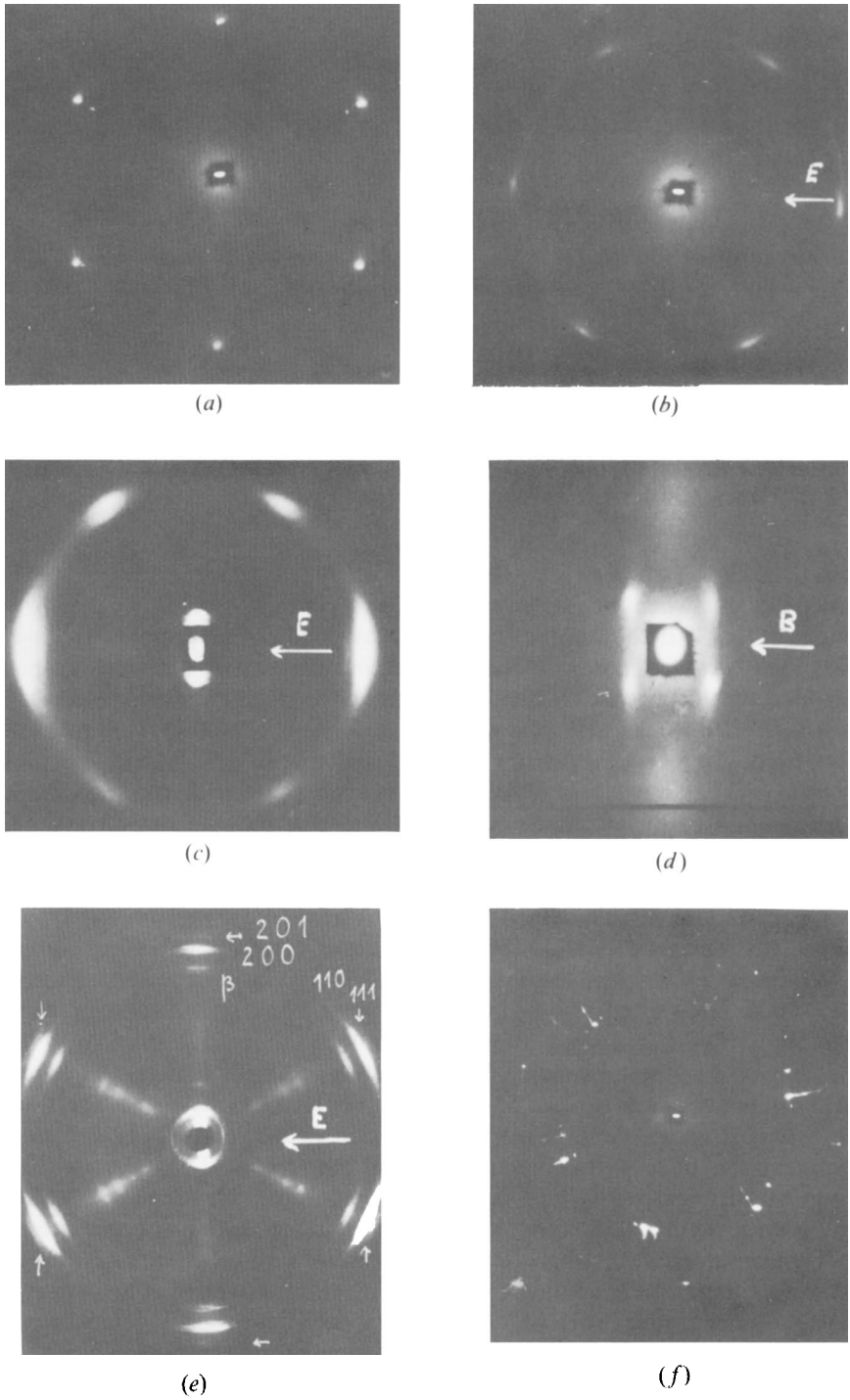
A modified freely suspended film method is described. Sample alignment can be achieved either by spontaneous ordering or by applying an electric or a magnetic field. Conventional X-ray equipment can be used.

Since the time when the free suspended film method was published by Moncton and Pindak [1] in 1979 these authors and other investigators have applied this method to study the structure of different liquid-crystalline phases [2–9]. In the free suspended film method the thickness of the film is between two to several hundred molecules and so it is necessary to use high power X-ray equipment (20–50 kW). Liquid crystal samples for X-ray investigations are prepared customarily by using either capillaries made from Lindemann glass or different kinds of containers with beryllium windows. The preparation of these samples is cumbersome and they do not always lead to satisfactory X-ray photographs.

Recently we have modified the Moncton–Pindak method of preparing liquid crystal samples [10]. The modified method is useful for X-ray photographic and diffractometric methods; in addition it only requires conventional X-ray equipment (1·5 kW). In our experiment the liquid crystal is placed inside a hole of diameter of about 1 mm which is usually bored in a copper plate. The hole is filled with the liquid crystal sample in powder form at room temperature and pressed by hand with the use of a piston of a suitable diameter. This usually guarantees that the liquid crystal remains in the hole while heated. Since, during the heating, the crystal can flow out of the hole it is necessary to ensure, using a microscope, that it is still there. The thickness of the plate determines that of the liquid crystal layer to within the accuracy of the meniscus on both surfaces of the sample. The cleanness of the two outer surfaces of the plate close to the hole is indispensable for the success of mounting the sample in the hole. Otherwise contact with other surfaces causes the liquid crystal to creep. The liquid crystal surfaces are uncovered. The copper plate together with the sample is placed inside a heater with a precisely regulated temperature. The liquid crystal sample is directly illuminated with X-rays. The diffracted radiation is recorded on a film within the period of time of about 60 min for thin samples, and about 30 min for thick samples. The modified Moncton–Pindak method, which perhaps may be called the free standing sample method for liquid crystal studies, has some advantages depending on the thickness of the liquid crystal sample. For layer thicknesses of about 0·1 mm the diffraction pattern is analogous to that



(*a*) (*b*) (*c*) (*d*) (*e*) (*f*)

X-ray photographs of: (*a*) thin layer of 4-(2′-methylbutyl)phenyl-4′-*n*-octylbiphenyl-4-carboxylate (+2M4P8BC), S_C^* self alignment, (*b*) thin layer of +2M4P8BC, S_I^* aligned by an electric field, (*c*) thick layer of +2M4P8BC, S_I^* aligned by an electric field, (*d*) thick layer of a nematic phase of 4,4′-di-*n*-heptyloxyazoxybenzene with cybotactic groups aligned by a magnetic field, (*e*) thick layer of +2M4P8BC, S_C^* aligned by an electric field (CrKα, β radiation), (*f*) thin layer of the smectic E phase of 4,4′-di-*n*-pentylbiphenyl badly aligned.

obtained with the Moncton–Pindak method. For example, our photographs of three dimensionally ordered crystals have sharp reflections, which indicate long range spontaneous ordering of the liquid crystal molecules (i.e. a single domain sample); see figure (*a*). To obtain a good spontaneously aligned monodomain sample the material should be cooled slowly from either the nematic or the isotropic phase down to the smectic phase. The cooling speed was about 1°C in 6 hours for several systems. However, this procedure does not guarantee success for every liquid crystal, see figure (*f*). The reflections become more diffuse ending up with the formation of a continuous ring (see figure (*b*)) when the three dimensional order ceases to exist.

For thick liquid crystal samples (that is with a thickness of about 1 mm) the molecules in the sample will not align spontaneously. An alignment is then obtained by applying an electric or a magnetic field as in the capillary method (see figures (*c*), (*d*), and (*e*)). In our case the strength of the magnetic field was of about 1·2 T. When an electric field is applied the sample should be mounted in a plate made from an insulating material in which electrodes are embedded. An electric field of about 300–2000 V/mm d.c. is used. The direction of the electric field should be perpendicular to the X-ray beam, which follows from the ∞m symmetry of the electric field. It seems to us that our free standing sample method of preparing the sample combines the advantages of both the Moncton–Pindak method for thin liquid crystal samples and of the capillary method for thick samples. In addition our method allows us to avoid the technical problems inherent in these two methods.

The free standing sample method is relatively simple and more sensitive, it allows a detailed study of the reciprocal lattice by using the technique of single crystal X-ray crystallography, and also for microscopic studies of textures.

Chemical decomposition was not taken into account in our experiments since the sample can be changed for every new photograph. For temperatures of about 150°C–200°C we found degradation of the compounds.

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References

[1] Moncton, D. E., and Pindak, R., 1979, *Phys. Rev. Lett.*, **43**, 701.
[2] Collet, J., Sorensen, L. B., Pershan, P. S., Litster, J. D., Birgeneau, R. J., and Als-Nielsen, J., 1982, *Phys. Rev. Lett.*, **49**, 553.
[3] Moncton, D. E., Pindak, R., Davey, S. C., and Brown, G. S., 1982, *Phys. Rev. Lett.*, **49**, 1865.
[4] Collet, J., Pershan, P. S., Sirota, E. B., and Sorensen, L. B., 1984, *Phys. Rev. Lett.*, **52**, 356.
[5] Budai, J., Pindak, R., Davey, S. C., and Goodby, J. W., 1984, *J. Phys. Lett., Paris*, **45**, L-1053.
[6] Davey, S. C., Budai, J., Goodby, J. W., Pindak, R., and Moncton, D. E., 1984, *Phys. Rev. Lett.*, **53**, 2129.
[7] Sirota, E. B., Pershan, P. S., Sorensen, L. B., and Collet, J., 1985, *Phys. Rev. Lett.*, **55**, 2039.
[8] Smith, G. S., Safinya, C. R., Roux, D., and Clark, N. A., 1987, *Molec. Crystals liq. Crystals*, **144**, 235.
[9] Sirota, E. B., Pershan, P. S., and Amador, S., 1987, *Phys. Rev.* A, **35**, 2283.
[10] Przedmojski, J., Dąbrowski, R., Pura, B., Zickert, K., and Gierlotka, S., 1988, *Molec. Crystals liq. Crystals* (in the press).