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Crystallographic data for some acenaphthene derivatives. By T. C. W. Mak and J. Trotter, *Department of Chemistry, University of British Columbia, Vancouver* 8, *Canada*

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Tautomeric forms in which nitroxy groups are directly linked have been postulated to explain the properties of organic molecules containing NO, NO_2 and ONO_2 groups (*e.g.* Hetman, 1960). The only X-ray work on organic nitrate esters is that on pentaerythritol tetranitrate (Booth & Llewellyn, 1947), which can hardly be regarded as a typical example.

cis-1,2-Acenaphthenediol dinitrate, synthesized by Csizmadia & Hayward (1962), provides a test of the possibility of intramolecular bonding between nitroxy groups, and we are investigating its crystal structure. In the course of the analysis some other derivatives of acenaphthene were also examined, and X-ray data for all the crystals are presented in Table 1 ($\lambda(\text{Cu } K\alpha) = 1{\cdot}5418$ Å, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å).

Although our chief interest is in the dinitrates, analyses of acenaphthenequinone and *cis*-1,2-acenaphthenediol are also being carried out. The crystals of *trans*-1,2-acenaphthenediol are pseudo-tetragonal, and while it is possible to visualize the general arrangement of molecules in the unit cell, detailed analysis would probably be quite difficult.

We are indebted to Mr I. G. Csizmadia and Dr L. D. Hayward for suggesting the problem, for suitable crystalline samples, and for many helpful discussions of their chemical investigations in advance of publication. We thank the National Research Council of Canada for financial support and for the award of a research studentship (to T. C. W. M.) during the tenure of which this work was carried out.

References

Booth, A. D. & Llewellyn, F. J. (1947). *J. Chem. Soc.*, p. 837.
Csizmadia, I. G. & Hayward, L. D. (1962). (In preparation.)
Hetman, J. S. (1960). *Talanta*, **5**, 267.

Table 1. *Crystallographic data*

Crystal system	Acenaphthene-quinone $C_{12}H_6O_2$ Orthorhombic	*cis*-1,2-Acenaphthenediol $C_{12}H_{10}O_2$ Monoclinic	*trans*-1,2-Acenaphthenediol $C_{12}H_{10}O_2$ Orthorhombic	*cis*-1,2-Acenaphthenediol dinitrate $C_{12}H_8O_6N_2$ Monoclinic	*trans*-1,2-Acenaphthenediol dinitrate $C_{12}H_8O_6N_2$ Monoclinic
a (Å)	7·81	12·77	11·37	17·10	10·56
b (Å)	27·0	4·845	11·37	4·242	7·77
c (Å)	3·851	15·74	28·94	19·18	7·85
β (°)	90°	111° 50′	90°	122° 12′	113° 14′
U (Å³)	812	904·0	3741·4	1177·3	591·9
Z	4	4	16	4	2
M	182·17	186·20	186·20	276·20	276·20
d (calc.)	1·49	1·368	1·322	1·557	1·548
d (meas.)	1·48	1·35	1·29	1·53	1·53
Space group	$P2_12_12_1$	$P2_1/c$	*Cmcm*	$P2_1/c$	$P2_1$

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International Union of Crystallography

Commission on Crystallographic Apparatus

Radiation Hazards Associated with X-ray Diffraction Techniques

The currently accepted maximum permissible total body dosage of radiation is equivalent to approximately 100 milliröntgens per week (*Recommendations of the International Commission on Radiological Protection*, 1959). The primary beam at the window of an X-ray tube may have a radiation level of the order of 10^5 röntgens per minute, and serious permanent skin burns (Fig. 1) can occur with only a few seconds exposure at this proximity.

In various countries a number of regulations already exist, which usually apply to the use of equipment in factories, educational establishments *etc*, and to which individuals are required, or advised, to adhere. The International Commission on Radiological Protection has, in its broad survey (1959), made recommendations relevant to X-ray analysis. There is, also, an article on Protection against Radiation Injury in Volume 3 of *International Tables for X-ray Crystallography* (1962): this deals with X-ray, neutron, and electron diffraction. Whilst the Commission on Crystallographic Apparatus of the International Union of Crystallography believes that it is therefore inappropriate for it to formulate further regulations, there are certain matters, not necessarily relevant for inclusion in regulations, which

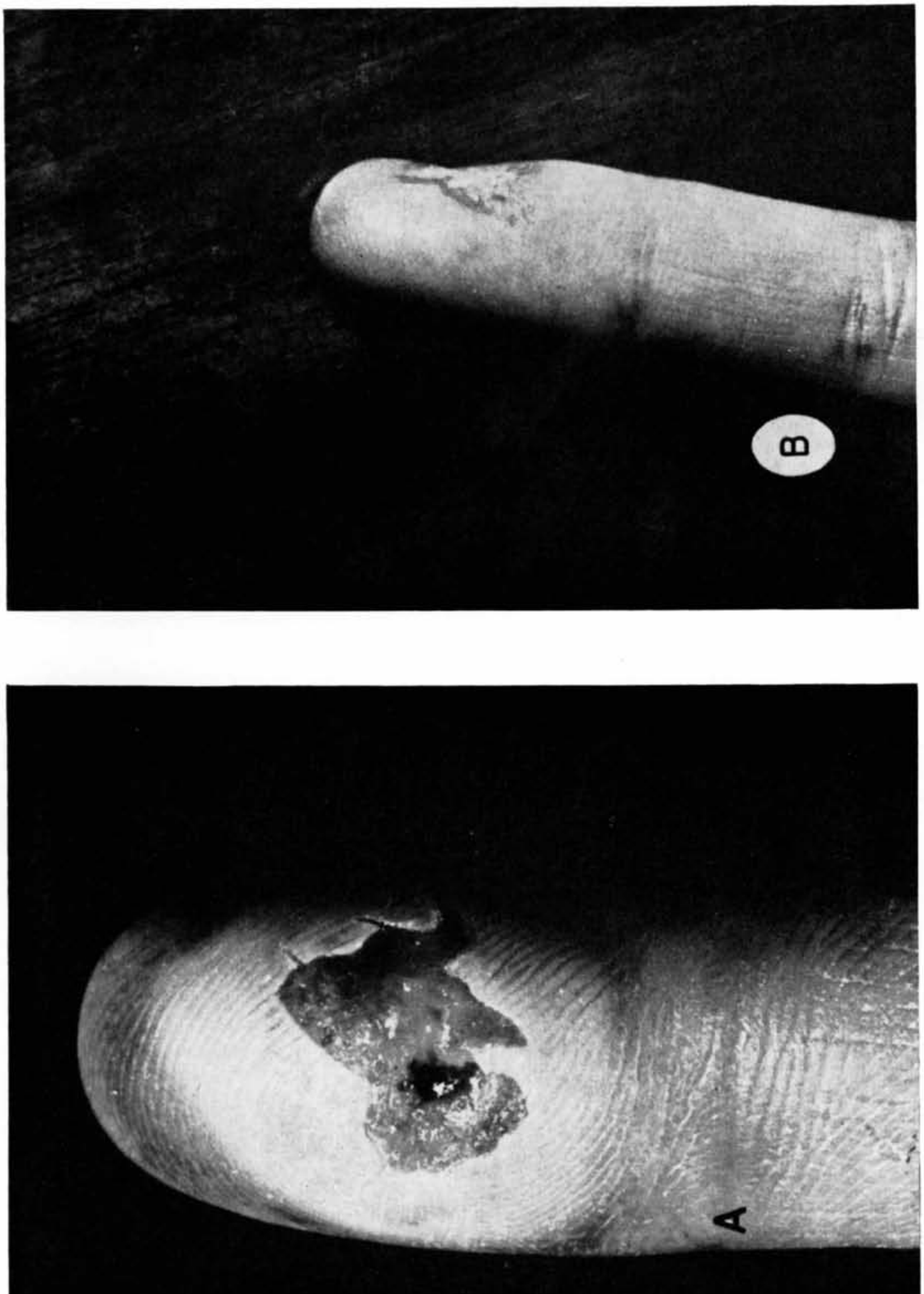

Fig. 1. Lesion from radiation burn on finger (Watrous, Hodges & McAndrew, 1952).
(*A*) 7 days after exposure, (*B*) 2 years after exposure. (Reproduced by kind permission of the American Medical Association).

should be drawn to the attention of crystallographers and others for their guidance in the use, or the consideration of design details, of X-ray diffraction equipment.

The following notes deal only with X-ray diffraction. Neutron and electron diffraction, X-ray fluorescence spectroscopy, micro-probes, microradiography, radiography, and the use of radio-isotopes, *etc*, are not included.

(1) Sources of Unwanted Radiation

X-ray crystallographic techniques involve the use of an X-ray generator to which is coupled some form of apparatus such as a photographic camera or counter diffractometer. In operation, dangerous leakage and scattering of radiation can occur in the following ways:

(*A*) Escape of primary X-rays when a tube-housing window is open and apparatus is not in position at that window.

(*B*) By scattering of the primary X-ray beam at interfaces between an open window of a tube-housing and the diffraction apparatus being used at that window.

(*C*) Escape of the residual primary X-ray beam from the diffraction apparatus.

(*D*) Leakage, from openings in the diffraction apparatus or through the walls of the apparatus, of radiation (including unwanted fluorescent radiation) scattered by the air or by mechanical parts of the apparatus.

(*E*) Leakage of radiation generated by valve rectifiers in the high-voltage power-units of X-ray generators.

(*F*) Penetration of radiation through the walls and (closed) window-shutters of an X-ray tube-housing.

(2) Detection of Stray Radiation: Personnel Monitoring

It is essential that any experimental arrangement should be surveyed with a counter-tube sensitive to the soft X-rays normally used in X-ray diffraction. The use of portable survey meters employing counter-tubes with low inherent filtration (*i.e.* low-absorption windows and high quantum efficiency in the low-energy X-ray region) is recommended for surveying installations. If a thin sheet of lead long enough to cover the counter tube length and extended about 10 cm beyond the window is wrapped around the tube, the counter will be directionally sensitive and simplify the location of radiation leaks. Survey meters for this purpose are commercially available. A procedure for the approximate conversion of quantum counting rates to röntgens has been described (Kohler & Parrish, 1956). (See also Section 5 concerning accurate calibration of secondary standards.) Large pieces of film placed around the apparatus may be used to locate narrowly defined leaks, particularly if exposed a sufficiently long time (*e.g.* an hour or more).

Film badges worn on the chest, wrist or finger may be used for personnel monitoring, but it should be noted that they are liable to provide a false sense of security because the stray radiation which may occur in X-ray diffraction apparatus is often in the form of narrow low-energy beams.

Medical opinion (*U.S. National Commission on Radiation Protection*, 1954) suggests that blood counts are not a desirable or effective method of personnel monitoring.

The problems involved in complying with local and national safety codes and regulations, insurance *etc*, should be taken into consideration in setting up a code of laboratory practice. The appointment of a person responsible for the proper instruction of all personnel, and the keeping of inspection records and checking of X-ray units at regular intervals are, however, most important. Some statutory regulations specifically require the appointment of a Radiation Officer responsible for these activities. *No one should be permitted to use X-ray apparatus until they have received proper instruction in safe practices.* When new apparatus is being mounted, or existing apparatus modified or re-aligned, it is necessary to survey the apparatus. Regular departmental meetings on safety practices, reviewing the safety of existing apparatus, *etc*, are useful and often necessary to emphasize the importance of the problem.

(3) Factors Responsible for the Hazards

Each of the various possible sources of radiation hazard listed in Section 1 results from one or more of the following:

(*a*) Negligence by the user.

(*b*) Failure by the user to recognize and deal with incompatibility of design when coupling diffraction apparatus to X-ray generators of different manufacturing origin.

(*c*) Faulty equipment.

(*d*) Unsatisfactory design of equipment.

(4) Comments Concerning the Hazards

Hazard (*A*)

If, while an X-ray tube is energized, a tube-window is open when apparatus is not in the operating position at that window, there can be an extremely dangerous escape of the primary beam. It is essential that this must not occur and that some safety interlock device between tube-housing and diffraction apparatus be used. Safety measures adopted should (*a*) have a fail-safe characteristic and (*b*) not restrict the responsible operator from making those adjustments recognized as an essential part of the procedure associated with the particular apparatus involved.

Various designs have been described (*American Crystallographic Association Project*; Barnes & Franks, 1962; Chambers, Okrasinski & Cole, 1961; Hughes, 1962) to eliminate this hazard.

Hazard (*B*)

Leakage and scattering of X-rays, from the region of coupling between an X-ray tube window and the apparatus in use at that window, is one of the most serious hazards, and is likely to give rise to narrow but intense beams of stray radiation. Separate consideration is necessary according to whether or not the tube-housing and apparatus (camera, diffractometer *etc*) are made by the same manufacturer.

(i) *Equipment from same manufacturer*. Much equipment exists in which radiation is strongly scattered from the region where the camera or diffractometer *etc* locates against the window of a tube-housing. The user should therefore check his apparatus and, if necessary, make modifications (see below).

(ii) *Components from different manufacturers*. Here, it is the user's responsibility to recognize and deal with the problems which arise. Designs have been published for labyrinths (Abrahams & Blackmore, 1953; Kennard, Martin & Woodget, 1959; Hughes & Taylor, 1961) and some of the devices referred to under *Hazard* (*A*) also incorporate this feature.

From a design point of view this hazard should be considered in conjunction with Hazard (*A*). As far as possible, the primary beam leaving the tube window should be limited at the window, to the cross-section to be used. The process of adjusting diffraction apparatus at a tube window presents a potential danger to the operator: suitable flexibility of alignment with maintenance, at the same time, of full protection is therefore essential in any design, and the use of X-rays only in the final stages of alignment, when this condition obtains, is advocated. When fluorescent screens are used for viewing a beam, they should be mounted on long supports to avoid accidental insertion of the fingers into the beam. To minimize unnecessary repetition of alignment procedures, the design of film cameras should, when practicable, allow the separate removal of the film cassette; if however, it is necessary to remove the whole camera for each exposure, then the design should be such as to ensure high accuracy in re-positioning the camera at the tube window and thus make re-alignment unnecessary.

Hazards (*C*) *and* (*D*)

These are largely determined by the basic design of the apparatus, and thus it is hoped that manufacturers will ensure that all future designs will eliminate these hazards. Apparatus should be carefully monitored by the user.

Hazard (*E*)

Thermionic rectifiers used in X-ray generators can act as powerful sources of penetrating X-rays. Causes include under-running of rectifier filaments, and, in a faulty valve, the passage of some inverse current at high voltage. Walls of high-voltage circuit tanks have not always been adequate to prevent this radiation from escaping. Monitoring of equipment should include this region of the generator.

Hazard (*F*)

With most modern X-ray generators using sealed tubes in safety shields, leakage of radiation through the tube-housing and window-shutters does not appear to present any hazard. There may be exceptions, and it is hoped that all commercial designs will give complete safety rather than be based on an arbitrary 'permissible' leakage.

(5) General Recommendations

In experimental work, operators need to be able to make adjustments *etc* when apparatus is in operation. Stray radiation must therefore be minimized at origin even where safety regulations depend on the entire generator and apparatus being placed in an enclosure fitted with door safety-interlocks. No method is completely fool-proof, and appropriate scientific training and awareness of responsibility are the eventual requirements of personnel.

The use of X-ray 'warning' signs at the entrance to an X-ray laboratory, small signs attached to the X-ray units, lights indicating when apparatus is in operation *etc*, are useful in minimizing the risk of accidental exposure. Wherever possible it is preferable to place X-ray units in a room separate from where personnel do other work.

Various publications deal with safety, and reference has been made to the existence in some countries, of regulations which are enforced (*e.g.* in factories). No attempt is made here to criticize or override such publications.

The latest (1959) revision of the Recommendations of the International Commission on Radiological Protection (I.C.R.P.) suggests that with installations for X-ray analysis, 'the leakage radiation at any accessible point at a distance of 5 cm from the surface of the tube-housing shall not exceed 25 mr in 1 hour at every specified rating'. The Commission on Crystallographic Apparatus suggests that a similar criterion should be applied to the combined assembly of tube-housing and apparatus, in operation. For two reasons the figure of 25 mr in 1 hour needs reduction when applied to the whole assembly. First the existing I.C.R.P. figure for tube-housing alone is based on only 3 hours exposure time per week at the indicated proximity: in counter-diffractometry for example, the operator may be located near to a working assembly for a considerably longer period than this each week. Secondly, the combined assembly represents a much larger 'source' and attenuation due to the inverse-square law is less marked. For these reasons it is suggested that the leakage radiation, at any accessible point at a distance of 25 cm from the effective bounding surface of the combined working assembly of tube-housing and diffraction apparatus, should be less than 2 mr in 1 hour at every specified tube-rating. Whilst this may be taken as an upper limit, it should not be regarded as a satisfactory working-level. Every effort should be made to reduce the amount of stray radiation to a level not sensibly detectable with a monitor appropriate for the energy range involved. This applies particularly in locating those sharply defined beams of scattered radiation which are likely to escape from inadequately shielded apparatus.

If each apparatus (camera or other device as previously defined), and the tube-housing at which it is operating, is regarded as a single 'combined assembly' and made safe according to the above recommendations, then hazards will not arise (*a*) through multiplication of apparatus operating in a similar way at other windows of the same generator, (*b*) through increasing the number of generators in the same room.

High-voltage generator units containing thermionic rectifiers should be subjected to the same criteria, for reasons given under *Hazard* (*E*).

It is hoped that manufacturers will use the above criteria of safety and give the user some quantitative reassurance as to the safety-levels achieved in each type of equipment marketed. Facilities for calibration of counter-tubes, for use as secondary standards by manufacturers or other organisations, make this quite feasible.*

* For example, the National Physical Laboratory, Teddington, England, undertakes the calibration of instruments to measure dose-rates down to 0·5 mr per hour for X-rays generated over the range 50 to 7·5 kV. Calibration is restricted to instruments which are to be used as secondary standards either by manufacturers of such instruments or by a limited number of other organisations which have need for a secondary standard.

(6) Bibliography

Extensive bibliographies have been published (*International Tables for X-ray Crystallography*, 1962; Braestrup & Wyckoff, 1958). In addition, bibliographies have been included in many of the local, government and other, Regulations formulated in various countries.

References

ABRAHAMS, S. C. & BLACKMORE, W. R. (1953). *Rev. Sci. Instrum.* **24**, 885.

American Crystallographic Association Project: Safety considerations in the design of X-ray tube and collimator couplings on X-ray diffraction equipment. (Details from BEU, K. E., Chairman of the A.C.A. Apparatus and Standards Committee (Goodyear Atomic Corporation, P.O. Box 628, Piketon, Ohio, U.S.A.)).

BARNES, D. C. & FRANKS, A. (1962). *J. Sci. Instrum.* **39**, 648.

BRAESTRUP, C. B. & WYCKOFF, H. O. (1958). *Radiation Protection.* Springfield: Chas. C. Thomas.

CHAMBERS, F., OKRASINSKI, M. & COLE, H. (1961). *I.B.M. Journal*, **5**, 69.

HUGHES, W. (1962). *J. Sci. Instrum.* **39**, 93.

HUGHES, W. & TAYLOR, C. A. (1961). *J. Sci. Instrum.* **38**, 493.

International Tables for X-ray Crystallography (1962). Vol. III. Birmingham: The Kynoch Press.

KENNARD, O., MARTIN, A. J. P. & WOODGET, L. (1959). *J. Sci. Instrum.* **36**, 48.

KOHLER, T. R. & PARRISH, W. (1956). *Rev. Sci. Instrum.* **27**, 705.

Recommendations of the International Commission on Radiological Protection (1959). London: Pergamon Press.

U.S. National Commission on Radiation Protection (1954). *Radiology*, **63**, 428.

WATROUS, R. M., HODGES, H. W. & MCANDREW, M. J. (1952). *J. Amer. Med. Assoc.* **152**, 513.

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 General Secretary of the International Union of Crystallography (D. W. Smits, Mathematisch Instituut, University of Groningen, Reitdiepskade 4, Groningen, The Netherlands).

1963 Summer Schools on Crystallography

Brooklyn (U. S. A.): 3-15 June

Summer School on X-ray Diffraction. The lectures and laboratory work of this two-week session cover the equivalent of a six-credit lecture and laboratory graduate course. No previous X-ray experience is assumed, but those with prior experience may make arrangements to undertake advanced work. At the completion of the course, registrants should be able to do most routine X-ray powder and single-crystal work.

Attendance limited to twenty-five registrants. Fee: $ 275.

Information: Mrs Doris Cattell, Special Courses, Polytechnic Institute of Brooklyn, Brooklyn 1, N. Y., U. S. A.

Mol (Belgium): 12-31 August

International summer course on solid-state physics with special reference to (i) radiation damage, and (ii) neutron diffusion and diffraction in solids and liquids.

Attendance limited to 70 registrants. Fee 5000 FB.

Information: Cours International d'Eté, Département de Physique de l'Etat Solide, C. E. N., Mol, Belgium.

International Colloquium on Neutron Diffusion and Diffraction

An international colloquium on neutron diffusion and diffraction will be held at Grenoble, France, 3-5 September 1963, under the presidency of Prof. Louis Neel. The responsibility for the programme is in the hands of a committee consisting of Drs E. F. Bertaut (Grenoble) and A. Herpin (C. E. A., Saclay, S. & O.). Abstracts of papers to be presented at this meeting should be sent in duplicate to Dr Bertaut (Institut Fourier, Place du Doyen-Gosse, Grenoble) before 15 April. Further information and application forms can be obtained from Mr H. Dubedout (Relations extérieures, Centre d'Etudes Nucléaires, C. E. N. G., Chemin des Martyrs, Grenoble).

International Union of Crystallography

Commission on Crystallographic Apparatus

An exhibition of non-commercial crystallographic apparatus, sponsored by the Commission on Crystallographic Apparatus, will be held during the Sixth General Assembly of the International Union of Crystallography at the Palazzo dei Congressi EUR in Rome, Italy. Research equipment such as models, teaching aids, optical devices, goniometer heads and similar devices not available from commercial sources will be welcome. Each exhibit must not exceed $80 \times 60 \times 40$ cm in size or weigh more than 20 kg. Electrical outlets requiring European male plugs supplying 125 V, 50 cycles, will be provided but no water or gas will be available. Exhibitors will be required to prepare their own labels and signs. Those wishing space (for which there is no charge) to display their apparatus are requested to write immediately either to Dr Vladimiro Scatturin, Istituto di Chimica Generale, Universita Degli Studi, Bari, Italy, or to the Chairman of the Commission (Dr William Parrish, Philips Laboratories, Irvington-on-Hudson, N. Y., U. S. A.) to obtain a copy of the rules and regulations and an application form. The form must be completed and received by Dr Scatturin no later than 15 July to be considered.