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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, NO₂ and ONO₂ 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 (λ (Cu K α) = 1.5418 Å, λ (Mo K α) = 0.7107 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

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Crystal system	$\begin{array}{c} Acenaphthene-\\quinone\\ C_{12}H_6O_2\\ Orthorhombic \end{array}$	cis-1,2- Acenaphthenediol $\mathrm{C_{12}H_{10}O_2}$ 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 ₈ O ₆ N 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 (Å ³)	812	904 ·0	3741.4	1177-3	591.9
Z	4	4	16	4	2
M	$182 \cdot 17$	$186 \cdot 20$	186.20	$276 \cdot 20$	$276 \cdot 20$
d (cale.)	1.49	1.368	1.322	1.557	1.548
d (meas.)	1.48	1.35	1.29	1.53	1.53
Space group	$P2_{1}2_{1}2_{1}$	$P2_1/c$	Cmcm	$P2_1/c$	$P2_1$

Table 1. Crystallographic data

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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

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