

From the Rb–Zn distance value (3.615 Å) a contraction greater than 10% in the atomic radius of Rb metal can be evaluated. This fact and the expansion of part of the Zn bonds indicate that the stability of this type of structure is actually coming down, although the existence of RbZn<sub>13</sub> is beyond the suggestion of Sanderson & Baenziger (1953).

The author wishes to thank Professor A. Iandelli for his constant interest in the work and for the valuable discussions.

## References

- MATJUŠENKO, N. N., VERKHOROBIN, L. F. & KAREV, V. N. (1964). *Kristallografija, SSSR*, **9**, 273.  
 SANDERSON, M. J. & BAENZIGER, N. C. (1953). *Acta Cryst.* **6**, 627.  
 SHOEMAKER, D. P., MARSH, R. E., EWING, F. J. & PAULING, L. (1952). *Acta Cryst.* **5**, 637.

*Acta Cryst.* (1971). B27, 863

### The crystallography of nitramine–solvent complexes. I. Crystallographic data on 1,7-diacetoxy-2,4,6-trinitro-2,4,6-triazaheptane (BSX) and on its complexes with acetone, nitromethane, acetonitrile and acetophenone.

By G. F. CLARINGBULL, *British Museum (Natural History), London S.W.7, England* and R. W. H. SMALL, *The University, Lancaster, England*

(Received 24 November 1970)

Pure BSX crystallizes from acetone at room temperature in tablets, space group *C2*, *Cm* or *C2/m* with  $a=26.24$ ,  $b=9.17$ ,  $c=6.14$  Å,  $\beta=91.5^\circ$ ,  $Z=4$ . At 0°C acicular crystals in *P2<sub>1</sub>/n* are obtained with  $a=14.54$ ,  $b=20.47$ ,  $c=6.38$  Å,  $\beta=91.5^\circ$ . Similar acicular crystals are obtained from the three other solvents. Those from acetone, nitromethane and acetonitrile appear to be 1:1 solvent/BSX complexes, from acetophenone 1:2 solvent/BSX. Optical data are given.

This paper describes the results of investigations carried out some time ago, elsewhere, which have not been previously published; it is presented now because of current interest in these complexes (Hall, 1970) and to serve as an introduction to some later papers describing the structure determination of some complexes of this type.

1,7-Diacetoxy-2,4,6-trinitro-2,4,6-triazaheptane, which has the trivial name BSX, may be crystallized from acetone at room temperature to give flat monoclinic crystals tabular on {100} and showing the forms {110} and {011}. The cell dimensions and space group of these crystals were determined by means of oscillation photographs using Cu *K* $\alpha$  ( $\lambda=1.5418$  Å) radiation; these data and the crystal density are given in Table 1.

Table 1. *Crystallographic data for pure BSX (tabular crystals)*

$a=26.24$ (2) Å*	Space group <i>C2</i> or <i>Cm</i> or <i>C2/m</i>
$b=9.17$ (1) Å	Density: observed 1.590 (4)
$c=6.14$ (1) Å	calculated 1.592, for four
$\beta=91.5$ (2)°	molecules of C <sub>8</sub> H <sub>14</sub> N <sub>6</sub> O <sub>10</sub>
	( $M=354$ ).

\* Figures in parenthesis show the standard deviation in the last figure.

Crystallization of BSX from acetone at 0°C gave monoclinic crystals acicular along [c] and exhibiting the forms {010}, {110} and {101}. These crystals were observed to transform to normal tabular BSX if the temperature was raised to 15°C while they were in contact with saturated solution. When in the dry condition at 15°C the crystals gradually became opaque (pseudomorphic) and suffered a weight loss corresponding to one molecule of acetone per molecule of BSX. Similar acicular crystals were grown from nitromethane and acetonitrile at 0°C and from acetophenone at 25°C. Other solvents including ethyl acetate and ethylidene chloride yielded crystals of normal tabular BSX at 0°C. The acicular crystals from different solvents were found to have similar cell dimensions and the same space group. Agreement between the density calculated from the cell dimensions and the observed density could only be obtained on the assumption of a molecular weight based on a 1:1 solvent/BSX complex in the case of the crystals grown from acetone, nitromethane and acetonitrile and a 1:2 solvent/BSX complex for the crystals from acetophenone. The results are summarized in Table 2. It was noted that the space group *P2<sub>1</sub>/n* is incompatible with an ordered structure containing two molecules of acetophenone per unit cell.

Table 2. *Crystallographic data for the acicular crystals*

Solvent	Acetone	Nitromethane	Acetonitrile	Acetophenone
$a$ (Å)	14.54 (1)	14.23 (1)	14.15 (1)	14.40 (1)
$b$ (Å)	20.47 (2)	20.24 (2)	20.01 (2)	20.05 (2)
$c$ (Å)	6.38 (1)	6.38 (1)	6.31 (1)	6.38 (1)
$\beta$ (°)	91.5 (2)	92.5 (2)	93.5 (2)	92.5 (2)
Space group	<i>P2<sub>1</sub>/n</i>	<i>P2<sub>1</sub>/n</i>	<i>P2<sub>1</sub>/n</i>	<i>P2<sub>1</sub>/n</i>
Density observed (g.ml <sup>-3</sup> )	1.418 (4)	1.519 (4)	1.454 (4)	1.482 (4)
Molecular weight (assumed)	412	415	395	414
Density, calculated (g.ml <sup>-3</sup> )	1.441	1.501	1.471	1.494

Table 3. *Optical data for the acicular crystals*

Solvent		Acetone	Nitromethane	Acetonitrile	Acetophenone
Refractive indices	$\alpha_D$	1.491	1.493	1.499	1.532
	$\beta_D$	1.506	1.508	1.509	1.550
	$\gamma_D$	1.526	1.543	1.525	1.556
Optic axial angle (meas.)	$2V_D$	79°	64°	78°	60°
Extinction angle	$\alpha: [c]$	36°	43°	42°	(63° red), (58° blue) 19° blue, 21° red
Orientation		$\gamma = b$	$\gamma = b$	$\gamma = b$	$\gamma = b$

In order to distinguish further between the acicular crystals from different solvents, their optical properties were measured with parallel and convergent polarized light. These data are summarized in Table 3. Only crystals from acetophenone showed marked dispersion of the refractive indices with respect to different wavelengths of light. This is often shown by compounds involving an aromatic nucleus.

These results are all consistent with the existence, in the crystalline state, of complexes between molecules of BSX and certain solvents.

#### Reference

HALL, P. G. (1970). *J. Chem. Soc. (A)*, p. 3137.

### 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, 13 White Friars, Chester CH1 1NZ, England).*

#### Professor Kathleen Lonsdale 1903-1971

Professor Dame Kathleen Lonsdale died on 1 April 1971. She was Professor of Chemistry and Head of the Department of Crystallography, University College, London from 1949 to 1968. Her many contributions to crystallography included her work as General Editor of the three volumes of *International Tables for X-ray Crystallography*. She was one of the first women to be elected Fellow of the Royal Society and the first woman President of the British Association for the Advancement of Science. As President of the International Union of Crystallography she chaired the sessions of the Seventh General Assembly in Moscow, 1966.

A full obituary will be published in *Acta Crystallographica*, Section A in due course.

#### Conference on Framework Silicates and Metals Cambridge (England), 10 December 1971

The Crystallography Group of The Institute of Physics and The Physical Society and The Mineralogical Society are jointly holding a one-day meeting at the Cavendish Laboratory, Cambridge, in honour of Dr W. H. Taylor, who will retire from the position of Reader in Crystallography in September 1971. The meeting will have two sessions on topics which have been of particular interest to Dr Taylor; in the morning the session will be devoted to *Framework Silicates* and in the afternoon the topic will be *Metals*. A

Conference Dinner will be held in St John's College on the evening of 10 December.

Further information and registration forms will be available in due course through the two societies. Accommodation for the nights of 9 and 10 December (if required) will be provided in a College. In the meantime, the Local Secretary (Dr P. Gay, Department of Mineralogy and Petrology, Downing Place, Cambridge, England) will be pleased to give advice to prospective participants.

#### *Diffusion des Rayons X aux Petits Angles* (Bibliography)

The fourth and last bibliography in the recent series prepared under the auspices of the Commission on Crystallographic Apparatus of the International Union of Crystallography, *Diffusion des Rayons X aux Petits Angles*, by A. J. Renouprez, has been published. Copies have been distributed free of charge to all subscribers in 1970 to *Acta Crystallographica* or the *Journal of Applied Crystallography*. Additional copies can be obtained from A. Oosthoek's Uitgevers Mij N.V., Doomstraat 5-13, Utrecht, The Netherlands, at the price of 10 Netherlands Guilders (U.S. \$3.00 or £1.25 at the present rates of exchange) per copy, including postage. In the event of foreign exchange difficulties, UNESCO coupons will be accepted. Orders may also be placed with Polycrystal Book Service, P.O. Box 11567, Pittsburgh, Pa. 15238, U.S.A., or with any bookseller.