1981). Crystal structures of cyclophanes have been reviewed by Keehn (1983).

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References

Enraf–Nonius (1982). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

KEEHN, P. M. (1983). *Organic Chemistry*, Vol. 45: *Cyclophanes*, edited by P. M. KEEHN & S. M. ROSENFELD, pp. 69–238. New York: Academic Press.

SWEPSTON, P. N., LIN, S.-T., HAWKINS, A., HUMPHREY, S., SIEGEL, S. & CORDES, A. W. (1981). *J. Org. Chem.* **46**, 3754–3756.

SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible.*

Acta Cryst. (1990). C**46**, 172–173

Structure of the zeolite theta-1. Redetermination using single-crystal synchrotron-radiation data.

By M. Z. PAPIZ and S. J. ANDREWS, *SERC Daresbury Laboratory, Warrington WA4 4AD, England*, A. M. DAMAS and MARJORIE M. HARDING, *Chemistry Department, Liverpool University, PO Box 147, Liverpool L69 3BX, England* and RONA M. HIGHCOCK, *BP Research Centre, Chertsey Road, Sunbury on Thames, Middlesex TW16 7LN, England*

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Abstract

Single-crystal synchrotron-radiation data have been recorded, using an area detector, for a crystal of dimensions 4 × 8 × 125 μm, composition SiO_2, and the structure redetermined. $R = 0{\cdot}116$ for 394 reflections with $F > 6\sigma(F)$. The unit cell, and the framework of SiO_4 tetrahedra, sharing corners, with channels parallel to **c**, previously established from powder diffraction data by Highcock, Smith & Wood [*Acta Cryst.* (1985), C**41**, 1391–1394] is confirmed, but atom positions (*z* parameters) are revised by 0·1–2 Å. $M_r = 60{\cdot}1$, $Cmc2_1$, $a = 13{\cdot}836\,(3)$, $b = 17{\cdot}415\,(4)$, $c = 5{\cdot}042\,(1)$ Å, $V = 1215$ Å^3, $Z = 24$, $\lambda = 0{\cdot}914$ Å.

Introduction

For the zeolite theta-1, large enough crystals for single-crystal data measurement with a conventional X-ray source were not obtainable so structure determination was attempted from X-ray powder diffraction data (Highcock, Smith & Wood, 1985). The high intensity of a synchrotron source (SRS) and an area detector diffractometer have now allowed single-crystal data collection, from which the structure has been redetermined.

Experimental

Needle crystals of theta-1 were of uniform size, *ca* 4 × 8 × 125 μm. The Enraf–Nonius 'FAST' area detector diffractometer on workstation 9.6 at SERC Daresbury Laboratory was used, with the SRS running at 2 GeV, 150 mA. The procedures for data collection and processing using *MADNES* software (Messerschmidt & Pflugrath, 1987) were very similar to those described for piperazine silicate (Andrews *et al.*, 1988). 100° of data were recorded about each of two rotation axes in the crystal, at 45° to each other. The first set alone included 785 observations, which merged to 331 independent reflections with a merging *R* (on intensity) of 0·06; the crystal rocking width was 2°. For the second orientation the rocking width appeared to be 10°; addition of these 463 observations to the earlier set yielded 486 independent reflection intensities with a merging *R* of 0·08, to a resolution of 1 Å.

The primitive monoclinic unit cell found initially with $a = 10{\cdot}944\,(20)$, $b = 10{\cdot}970\,(24)$, $c = 4{\cdot}958\,(10)$ Å, $\alpha = \beta = 90°$, $\gamma = 76{\cdot}615\,(86)°$ and assuming $\lambda = 0{\cdot}90\,(1)$ Å, was converted into an orthorhombic cell; the latter is in agreement with that of Highcock, Smith & Wood (1985), $a = 13{\cdot}836\,(3)$, $b = 17{\cdot}415\,(4)$, $c = 5{\cdot}042\,(1)$ Å, assuming $\lambda = 0{\cdot}914$ Å. Since there was some uncertainty in the precise value of the wavelength used on workstation 9.6, the latter cell dimensions and wavelength were used in the analysis. The systematic absences observed for the orthorhombic cell are also in agreement with space group $Cmc2_1$, 24 SiO_2 per unit cell, chosen by Highcock, Smith & Wood.

The structure was solved with the direct-methods facility of *SHELX*86 (Sheldrick, 1986) and refined (*SHELX*76; Sheldrick, 1976) with anisotropic vibration parameters for

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Table 1. *Fractional coordinates of atoms with e.s.d.'s*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}(Å^2)
Si(1)	0·2947 (6)	0·0490 (4)	0·2500	0·065 (4)
Si(2)	0·2056 (5)	0·2116 (4)	0·329 (2)	0·065 (4)
Si(3)	0·5000	0·2257 (6)	0·725 (3)	0·063 (6)
Si(4)	0·5000	0·1234 (6)	0·225 (3)	0·063 (6)
O(11)	0·2722 (15)	0·9815 (12)	0·461 (3)	0·070 (12)
O(12)	0·2285 (13)	0·1203 (10)	0·323 (4)	0·073 (10)
O(14)	0·4059 (12)	0·0731 (10)	0·278 (4)	0·066 (11)
O(22)	0·2722 (14)	0·2580 (14)	0·118 (4)	0·073 (13)
O(23)	0·4075 (12)	0·2780 (10)	0·776 (4)	0·077 (12)
O(34)	0·5000	0·1554 (16)	0·917 (5)	0·069 (16)
O(43)	0·5000	0·1966 (15)	0·423 (4)	0·063 (16)

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Si(1)—O(11)	1·61 (2)	Si(3)—O(23)	1·59 (2)
Si(1)—O(11^{iv})	1·58 (2)	Si(3)—O(23^{v})	1·59 (2)
Si(1)—O(12)	1·59 (2)	Si(3)—O(34)	1·56 (2)
Si(1)—O(14)	1·60 (2)	Si(3)—O(43)	1·61 (2)
Si(2)—O(12)	1·62 (2)	Si(4)—O(14)	1·59 (2)
Si(2)—O(22)	1·62 (2)	Si(4)—O(14^{v})	1·65 (2)
Si(2)—O(22^{ii})	1·59 (2)	Si(4)—O(34^{vi})	1·62 (2)
Si(2)—O(23)	1·60 (2)	Si(4)—O(43)	1·65 (2)
Si(1)—O(11)—Si(1^{i})	144·3 (1·3)	Si(2)—O(22^{ii})—Si(2^{ii})	151·7 (1·5)
Si(1)—O(12)—Si(2)	152·2 (1·4)	Si(2^{ii})—O(23)—Si(3)	151·5 (1·4)
Si(1)—O(14)—Si(4)	156·1 (1·3)	Si(3)—O(34)—Si(4^{iii})	148·1 (1·5)
		Si(3)—O(43)—Si(4)	146·4 (1·4)

Symmetry code: (i) $x, -y, \frac{1}{2}+z$; (ii) $\frac{1}{2}-x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $x, y, 1+z$; (iv) $x, -y, -\frac{1}{2}+z$; (v) $1-x, y, z$; (vi) $x, y, -1+z$.

all atoms, and weights $1/[\sigma^2(F) + 0{\cdot}00012F^2]$, giving, for the larger data set, *i.e.* the merged data for two orientations, $R = 0{\cdot}116$ ($wR = 0{\cdot}13$) for 394 reflections with $F > 6\sigma(F)$. Atom parameters are listed in Table 1.* An alternative refinement, using the first data set, *i.e.* for the crystal of lower rocking width only, gave $R = 0{\cdot}080$ ($wR = 0{\cdot}09$), but the atom parameters were not significantly different and the e.s.d.'s were larger.

The relatively high R values can be explained by a number of factors, including the large mosaic spread and the camera geometry (Andrews *et al.*, 1988).

Results, comparison with powder diffraction study

Our results are in agreement, within 0·1–0·15 Å, with the structure as illustrated in the c-axis projection by Highcock, Smith & Wood (1985), but indicate substantial revision of some of the z coordinates, Si(3) and Si(4) by 0·5 Å, O(22), O(34) and O(43) by up to 1·5 Å. This does not alter the description of the structure as a three-dimensional network of SiO_4 tetrahedra, sharing all corners, in such a way that there are large channels parallel to **c**. The revised Si and O atom positions do not differ significantly from those derived in a single-crystal study of silica-ZSM-22, $24SiO_2(C_2H_5)_2NH$ (Marler, 1987), but the present material contains no organic guest molecules. Selected bond lengths and angles are given in Table 2.

We are grateful to SERC Daresbury Laboratory for the provision of synchrotron-radiation facilities, and to Dr J. R. Helliwell for helpful advice.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52102 (5 pp.). Copies may be obtained through the Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Andrews, S. J., Papiz, M. Z., McMeeking, R., Blake, A. J., Lowe, B. M., Franklin, K. R., Helliwell, J. R. & Harding, M. M. (1988). *Acta Cryst.* **B44**, 73–77.

Highcock, R. M., Smith, G. W. & Wood, D. (1985). *Acta Cryst.* **C41**, 1391–1394.

Marler, B. (1987). *Zeolites*, **7**, 393–397.

Messerschmidt, A. & Pflugrath, J. W. (1987). *J. Appl. Cryst.* **20**, 306–315.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1986). *SHELX86*. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

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Structure of *α-trans*-cinnamic acid. Addendum.

By Derk A. Wierda, Timothy L. Feng and Andrew R. Barron,* *Department of Chemistry, Harvard University, Cambridge, Massachusetts* 02138, *USA*

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Abstract

An addendum [Wierda, Feng & Barron (1989). *Acta Cryst.* **C45**, 838] of a recent report of a determination of the structure of the title compound [Wierda, Feng & Barron (1989). *Acta Cryst.* **C45**, 338–339] drew attention to a reference [Bryan & Freyberg (1975). *J. Chem. Soc. Perkin Trans.* 2, pp. 1835–1840] overlooked by the authors in their original report. The addendum failed to make clear that this reference contained a report of a complete room-temperature determination of the crystal structure in question. The two structures are in good agreement, except for variations attributed to differences in temperature.

All relevant information is contained in the *Abstract.*

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* To whom correspondence should be addressed.