SHORT COMMUNICATIONS

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X-ray refinement of the structure of cubic telluric acid.

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$Te(OH)_6$, cubic, $Fd3c$, $a = 15\cdot705$ (3) Å, $Z = 32$, $D_x = 3\cdot16$ g cm^{-3}. The final $R = 0\cdot025$ for 139 independent diffractometer data. The structure is built up of $Te(OH)_6$ octahedra connected *via* hydrogen bonds. The Te–O distances are $1\cdot913$ (6) Å (6×). The O–H$\cdots$O bond lengths are $2\cdot639$ (12) Å (6×) and $2\cdot773$ (13) Å (6×).

Introduction

The crystal structure of cubic telluric acid has previously been studied by Cohen-Addad (1971). Mahalaxmi (1968) solved the structure from film data and obtained a Te–O coordination distance of $1\cdot88$ (3) Å. Cohen-Addad also tried to resolve the H-atom positions from a neutron powder investigation, but her data indicated disorder in the structure. A Te–O bond distance of $1\cdot93$ Å was given without any indication of standard deviation. The present refinement has been undertaken to resolve the oxygen coordination of Te^{VI} in cubic $Te(OH)_6$ to an accuracy comparable with that of monoclinic $Te(OH)_6$ (Lindqvist, 1970; Lindqvist & Lehmann, 1973).

The intensities were collected on a four-circle Syntex $P2_1$ diffractometer with graphite-monochromated Mo $K\alpha$ radiation. The crystal had the form of an octahedron with an edge of $0\cdot12$ mm. An $\omega/2\theta$ scan was used with scan intervals of $2\cdot4$–$3\cdot2°$ ($\Delta2\theta$) and a scan speed of $2\cdot0$–$12\cdot0°$ min^{-1}. The intensities were collected out to $2\theta = 54°$ and a total of 1363 independent reflections were measured; reflections with $I/\sigma(I) > 3$ were used for the structure refinement. The background and integrated intensities were calculated with the Lehmann & Larsen (1974) profile-analysis method (local program; Lindqvist & Ljungström, 1978). The data were corrected for Lorentz, polarization and absorption effects (program *DATAPH*; Coppens, Leiserowitz & Rabinovich, 1965); μ(Mo $K\alpha$) = $62\cdot08$ cm^{-1} (*International Tables for X-ray Crystallography*, 1962). The determination of the structure was started with the atomic positional parameters given by Mahalaxmi (1968). The final refinement included anisotropic temperature factors and an isotropic extinction parameter (*LINUS*; Coppens & Hamilton, 1970) and yielded an R of $0\cdot025$. The resulting positional parameters are given in Table 1.*

Discussion

Interatomic distances and angles are given in Table 2. The structure of this phase of telluric acid is built up of discrete $Te(OH)_6$ octahedra. These are regular within the standard deviations and are held together by hydrogen bonds. The Te–O bond length within the octahedron agrees well with values found in the monoclinic phase: $1\cdot916$ (5) Å (X-ray diffraction study; Lindqvist, 1970) and $1\cdot909$ (1) Å (neutron diffraction study; Lindqvist & Lehmann, 1973), but the angles in monoclinic $Te(OH)_6$ vary significantly from the ideal value, *i.e.* in the range $87\cdot60$ (6)–$92\cdot08$ (7)°. The intermolecular O$\cdots$O distances in the cubic phase indicate that each $Te(OH)_6$ entity participates in six relatively strong hydrogen bonds [$2\cdot639$ (12) Å] and six weaker bonds [$2\cdot773$ (13) Å]. The average volume of each $Te(OH)_6$ molecule is $121\cdot40$ Å^3 in the cubic phase and $124\cdot25$ Å^3 in monoclinic $Te(OH)_6$. Preparation of suitable cubic $Te(OH)_6$ crystals for neutron diffraction work has been commenced in cooperation with Dr Jacques Moret, Montpellier, in order to be able to study the hydrogen bonds in detail.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33625 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final positional parameters*

	x	y	z
Te	0	0	0
O	$0\cdot1402$ (4)	$0\cdot5425$ (4)	$0\cdot2809$ (4)

Table 2. *Bond distances (Å) and angles (°) in cubic $Te(OH)_6$*

Te–O	O$\cdots$O	O–Te–O
$1\cdot913$ (6) (6×)	$2\cdot639$ (12) (6×)	$90\cdot2$ (3)
	$2\cdot773$ (13) (6×)	$89\cdot2$ (3)

References

COHEN-ADDAD, C. (1971).*Bull. Soc. Fr. Minéral. Cristallogr.* **94**, 171–172.

COPPENS, P. & HAMILTON, W. C. (1970). *Acta Cryst.* A**26**, 71–83.

COPPENS, P., LEISEROWITZ, L. & RABINOVICH, D. (1965). *Acta Cryst.* **18**, 1035–1038.

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

LEHMANN, M. S. & LARSEN, F. K. (1974). *Acta Cryst.* A**30**, 580–584.

LINDQVIST, O. (1970). *Acta Chem. Scand.* **24**, 3178–3188.

LINDQVIST, O. & LEHMANN, M. S. (1973). *Acta Chem. Scand.* **27**, 85–95.

LINDQVIST, O. & LJUNGSTRÖM, E. (1978). To be published.

MAHALAXMI, V. (1968). Thesis. Rensselaer Polytechnic Institute, Troy, New York. (No. 69-2487, Ann Arbor, Michigan.)

Some molecular packing studies of crystalline hydrocarbons.

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Packing analysis of some alkyne structures shows that short C⋯C contacts are more numerous in these than in other hydrocarbon structures, and that the structures are well described by currently used potential-energy functions. Calculations of the lattice energy of the two crystalline modifications of benzene using various proposed energy functions correctly predict that benzene I is the more stable, only when interactions arising from C—H bond polarity are included.

Packing analysis of alkyne structures

Potential-function parameters for non-bonded interactions for molecular-packing analysis may be deduced by least-squares fitting of calculated to experimental crystal data. These are most commonly derivatives of lattice energy with respect to cell parameters or rigid-body structural parameters, which should be zero for the observed structure; other non-zero data are heats of sublimation or elastic constants, which are often of lower accuracy, but at least some of which must be included for scaling purposes (Williams, 1970, 1974). Some difficulty can arise because of parameter interaction, which in the case of hydrocarbon crystals derives particularly from the fact that the peripheral atoms of the molecule are invariably hydrogen, and consequently as short contacts between carbon atoms occur infrequently the repulsive C⋯C coefficient may not be well defined (Williams, 1970). For this reason it has been considered desirable to use data sets based on a variety of hydrocarbon types, which should include some aromatic hydrocarbons wherein the carbon atoms are relatively exposed. The structures of alkynes, particularly longer-chain poly-ynes, might be expected to be more dependent upon C⋯C interaction, and we have analysed the packing of a number of such structures to investigate this and to establish the effectiveness of currently used energy functions to describe such structures. Energy functions calculated have been both conventional lattice energy, E, using the potential parameters given by Williams (1974), and repulsive lattice energy, E_R, as defined by Williams (1969) and using the potential parameters given therein. In each calculation the molecule was defined by the observed carbon atom positions, with hydrogen atoms calculated in the expected geometry for a C—H bond length of 1·027 Å (Williams, 1965), and the cell parameters assumed as observed. Lattice energy was minimized with respect to all variable cell and rigid-body orientational parameters, and repulsive lattice energy with regard to orientational parameters only. For all structures the molecular centre is a centre of inversion, and thus there are no translational variables.

The results of these calculations are given in Table 1, wherein θ and θ_R represent the changes in molecular orientation during the two different energy refinements. For comparison, comparable results are listed for *n*-hexane, benzene and naphthalene. The types of intermolecular contacts which are shorter than the distance of the minimum of the appropriate non-bonded energy function (C⋯C 3·93; C⋯H 3·56; H⋯H 3·18 Å), *i.e.* the repulsive interactions, are listed in Table 2 for *n*-hexane, naphthalene and 1,10-diphenyldecapentayne. For the aliphatic hydrocarbon these are mainly C⋯H and H⋯H, for the aromatic hydrocarbon C⋯H predominate, but in neither structure are C⋯C interactions at all numerous. By contrast, in the poly-yne more than half the contacts are between carbon atoms. Notwithstanding this very different distribution, the lattice-energy minima for the alkyne structures correspond well with the observed structures, and the relative weighting of the terms in the potential-energy function between the different types of contact then appears to be confirmed.

As a further check, the repulsive lattice-energy space was explored more extensively by systematically varying the orientational parameters used for the starting model and minimizing from each (Williams, 1969, 1973), for 1,8-diphenyloctatetrayne and for 1,10-diphenyldecapentayne. For both a number of energy minima were located, but that corresponding to the observed structure was the global minimum, being at least 10 kJ mol^{-1} lower than any other.

Polarity of the C—H bond in potential-energy models

The majority of potential-energy models that have been proposed for packing studies of hydrocarbons have implicitly or explicitly assumed Coulombic interactions arising from C—H bond polarity to be negligible, but it has been shown that agreement between the calculated energy minimum and the observed structure can be markedly improved by inclusion of a Coulombic term, and for a point-charge model optimal fit was obtained with a charge separation of 0·36 e (Williams, 1974). Improvement was particularly notable for