826. *Stereochemistry of Arsenic. Part XVI.*[1] *Cacodylic Acid*

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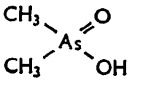
Crystals of cacodylic acid, $(CH_3)_2AsO{\cdot}OH$, are triclinic, $a = 6{\cdot}53$, $b = 6{\cdot}82$, $c = 6{\cdot}61$ Å, $\alpha = 77°\ 30'$, $\beta = 78°\ 45'$, $\gamma = 55°\ 9'$, $Z = 2$, space group $P\bar{1}$. The structure was determined from visual intensity data, and the positional and anisotropic thermal parameters were refined by least-squares. The final R value is 0·149 for 806 observed reflexions.

The structure consists of centrosymmetrical dimers, with O–H · · · O hydrogen bonds of 2·57 Å. The configuration around the arsenic atom is tetrahedral with bond angles in the range 106—115°.

As a part of a series of investigations of arsenic derivatives, we have determined the structure of cacodylic acid (I). The results indicate that the crystal is built up from centrosymmetrical hydrogen-bonded dimers.



(I)

Experimental

Crystals of cacodylic acid are colourless prisms elongated along a, with (100), (010), and (001) developed. The density was measured by flotation in a chloroform–bromoform mixture, and the unit-cell dimensions were determined from various rotation, Weissenberg, and precession photographs.

Crystal data (λ, Cu-K_α = 1·5418 Å; λ, Mo-K_α = 0·7107 Å).—Cacodylic acid, $C_2H_7O_2As$, m. p. 200°, $M = 138{\cdot}0$. Triclinic, $a = 6{\cdot}53 \pm 0{\cdot}01$, $b = 6{\cdot}82 \pm 0{\cdot}01$, $c = 6{\cdot}61 \pm 0{\cdot}01$ Å, $\alpha = 77°\ 30' \pm 5'$, $\beta = 78°\ 45' \pm 5'$, $\gamma = 55°\ 9' \pm 5'$, $U = 234{\cdot}9$ Å^3, $D_m = 1{\cdot}95$, $Z = 2$, $D_x = 1{\cdot}95$. Absorption coefficient for X-rays, λ = 1·5418 Å, $\mu = 95$ cm.$^{-1}$. $F(000) = 136$. No absent reflexions; space group $P1$ or $P\bar{1}$; $P\bar{1}$ from structure analysis.

The intensities of the reflexions were measured visually from Cu-K_α equi-inclination Weissenberg films of the $0kl$—$5kl$ layers; two sets of films were taken for each layer to record the whole of reciprocal space, and the various layers were correlated by carefully timed exposures. The crystal cross-section was 0·2 × 0·2 mm., so that absorption errors are small, and no corrections were applied. The structure amplitudes were derived as usual; 806 reflexions were observed.

Structure Analysis

The arsenic position was determined from the three axial Patterson projections, and the carbon and oxygen atoms were located on a three-dimensional electron-density distribution (Figure 1); the map could be interpreted in terms of space group $P\bar{1}$. Structure factors were

[1] Part XV, *Z. Krist.*, 1965, in the press.

calculated by using standard scattering factors,[2] with $B = 4{\cdot}0$ Å^2; R was 0·270 for the observed reflexions.

The positional and thermal parameters, and an overall scale-factor, were refined by (block-diagonal) least-squares; the function minimised was $\Sigma w(F_o - F_c)^2$, with $\sqrt{w} = |F_o|/18$ when $|F_o| < 18$, and $\sqrt{w} = 18/|F_o|$ when $|F_o| \geqslant 18$. Three cycles with isotropic thermal parameters and three cycles with anisotropic thermal parameters completed the refinement; R, for the 806 observed reflexions, was reduced from 0·270 to 0·149, and $\Sigma w\Delta F^2$ was reduced from $6{\cdot}9 \times 10^3$ to $2{\cdot}9 \times 10^3$. Final measured and calculated structure factors are listed in Table 1.

Co-ordinates and Molecular Dimensions.—The final positional and anisotropic thermal parameters are given in Table 2. x, y, and z are fractional co-ordinates referred to the triclinic

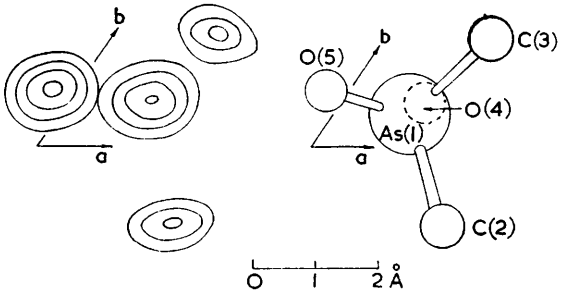


FIGURE 1. Superimposed sections of the three-dimensional electron-density distribution through the atomic centres parallel to (001), contours at arbitrary intervals, arsenic omitted for clarity. A perspective drawing of the molecule is also shown

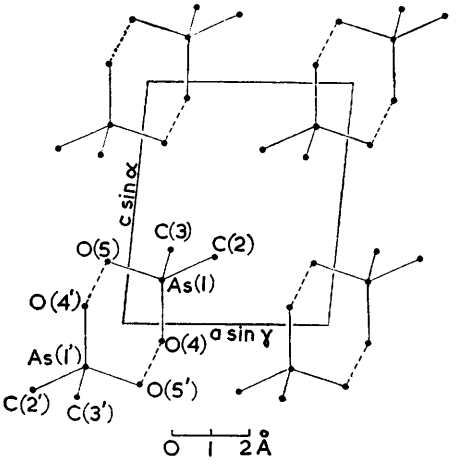


FIGURE 2. Projection of the structure along [010], illustrating the molecular packing. The hydrogen bonds are shown as broken lines

crystal axes; $\sigma(x)$, $\sigma(y)$, and $\sigma(z)$ are their standard deviations (in Å) computed from the least-squares residuals; X', Y', and Z' are co-ordinates (in Å) referred to orthogonal axes a', b, and c^*; and U_{ij} are the components of the mean-square vibration tensors.

The bond lengths and valency angles in the molecule are given, with their standard deviations, in Table 3. The shorter intermolecular contacts are listed in Table 4, and the packing of the molecules is shown in Figure 2.

DISCUSSION

The analysis has established that the arsenic atom in cacodylic acid has the expected tetrahedral configuration. The As–C bond lengths do not differ significantly, and the mean value of 1·91 ± 0·04 Å is not significantly different from the usual As^V–C distance [*e.g.*,[3] 1·955 ± 0·018 Å in $(Me_2AsS)_2$]. The As–O bond lengths are also, surprisingly, equal within experimental error, the mean value being 1·62 ± 0·03 Å.

The crystal is built up from centrosymmetrical hydrogen-bonded dimers (Figure 2), with O–H · · · O = 2·57 Å, so that the structure is similar to that of carboxylic acids, rather than to arsonic acids, $RAsO(OH)_2$, where the two replaceable hydrogens give rise to more complex hydrogen-bonding schemes.[4] All the other intermolecular contacts correspond to van der Waals interactions, the shortest involving oxygen atoms (Table 4); the shortest As · · · As and C · · · C contacts are 3·98 and 3·88 Å.

[2] " International Tables for X-Ray Crystallography," vol. III, Kynoch Press, Birmingham, 1962.
[3] N. Camerman and J. Trotter, *J.*, 1964, 219.
[4] A. Shimada, *Bull. Chem. Soc. Japan*, 1959, **32**, 309; 1960, **33**, 301; 1961, **34**, 639; 1962, **35**, 1600.

TABLE 1

Measured and calculated structure factors

h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c
0	0	1	27·4	34·3	0	3	−7	4·3	6·5	1	5	5	12·5	−13·1	1	−6	−2	8·8	−13·2
0	0	2	45·3	−49·5	1	0	1	20·0	−26·2	1	5	6	14·7	−15·0	1	−6	−3	1·5	−3·5
0	0	3	23·8	−30·7	1	0	2	21·7	−37·6	1	5	7	2·0	−3·5	2	0	0	27·0	−30·5
0	0	4	1·7	−0·4	1	0	3	1·7	−4·4	1	6	−3	16·0	14·0	2	0	1	25·6	−36·5
0	0	5	9·8	11·9	1	0	4	29·0	26·9	1	6	−2	10·2	9·8	2	0	2	10·3	−14·5
0	0	6	9·4	11·1	1	0	5	24·9	26·2	1	6	−1	12·5	−11·6	2	0	3	14·9	17·9
0	0	7	0·0	−0·2	1	0	6	0·0	1·4	1	6	0	21·7	−18·0	2	0	4	33·0	32·3
0	0	8	6·6	−7·9	1	0	7	10·4	−12·9	1	6	1	0·0	0·2	2	0	5	12·7	11·5
0	1	−7	2·9	3·0	1	0	8	6·6	−7·5	1	6	2	16·1	16·8	2	0	6	15·3	−12·3
0	1	−6	9·7	11·4	1	0	−8	0·0	0·2	1	6	3	14·1	15·6	2	0	7	14·9	−11·0
0	1	−5	5·6	7·8	1	0	−7	9·6	10·0	1	6	4	2·3	0·2	2	0	8	2·7	1·8
0	1	−4	13·3	−16·4	1	0	−6	10·8	9·0	1	6	5	12·7	−14·0	2	0	−7	11·0	9·3
0	1	−3	55·6	−44·6	1	0	−5	6·1	−7·8	1	6	6	9·1	−11·4	2	0	−6	0·0	−0·9
0	1	−2	18·1	−23·2	1	0	−4	27·2	−26·9	1	7	−2	1·9	2·2	2	0	−5	18·4	−18·2
0	1	−1	48·4	52·7	1	0	−3	27·5	−31·1	1	7	−1	10·0	−10·1	2	0	−4	18·2	−18·2
0	1	0	74·5	74·4	1	0	−2	0·0	3·0	1	7	0	9·2	−11·4	2	0	−3	9·2	8·6
0	1	1	13·3	7·8	1	0	−1	57·1	58·2	1	7	1	3·5	3·8	2	0	−2	24·7	28·9
0	1	7	8·4	−5·7	1	0	0	35·9	40·5	1	7	2	13·7	16·5	2	0	−1	2·6	6·8
0	1	8	8·5	−9·1	1	1	−7	9·2	10·4	1	7	3	10·4	10·9	2	1	−7	7·2	5·8
0	2	−7	3·2	4·5	1	1	−6	5·6	4·8	1	7	4	4·3	−5·0	2	1	−6	3·4	−3·7
0	2	−6	9·8	11·1	1	1	−5	9·0	−10·2	1	−1	8	0·0	−7·7	2	1	−5	17·5	−14·9
0	2	−5	5·0	5·3	1	1	−4	25·3	−24·2	1	−1	7	9·2	−8·7	2	1	−4	10·6	−10·4
0	2	−4	27·6	−25·3	1	1	−3	23·0	−24·7	1	−1	6	3·6	3·9	2	1	−3	22·9	19·8
0	2	−3	37·9	−41·9	1	1	−2	5·6	10·2	1	−1	5	20·5	21·6	2	1	−2	30·3	36·3
0	2	−2	2·5	3·4	1	1	−1	43·1	52·3	1	−1	4	11·5	15·4	2	1	−1	10·1	−8·3
0	2	−1	45·6	50·3	1	1	0	10·2	13·9	1	−1	3	28·0	−25·9	2	1	0	41·6	−53·8
0	2	0	17·6	23·1	1	1	1	51·0	−56·2	1	−1	2	37·2	−39·3	2	1	1	35·0	−39·2
0	2	1	20·7	−18·6	1	1	2	44·7	−46·6	1	−1	1	22·3	17·6	2	1	2	7·6	−10·1
0	2	2	37·8	−29·4	1	1	3	10·5	12·1	1	−1	0	48·8	55·5	2	1	3	21·8	22·0
0	2	3	17·4	−12·0	1	1	4	35·8	42·3	1	−1	−1	19·7	30·5	2	1	4	34·1	31·6
0	2	4	22·1	18·9	1	1	5	21·5	23·5	1	−1	−2	1·6	2·8	2	1	5	0·0	1·6
0	2	5	32·0	25·6	1	1	6	8·1	−6·7	1	−1	−3	16·4	−20·8	2	1	6	18·5	−19·3
0	2	6	9·3	7·1	1	1	7	12·0	−12·9	1	−1	−4	21·2	−26·9	2	1	7	8·2	−7·4
0	2	7	10·2	−8·0	1	1	8	3·7	−3·7	1	−1	−5	3·5	−2·7	2	1	8	7·3	7·0
0	2	8	9·0	−8·5	1	2	−7	8·7	9·0	1	−1	−6	12·9	15·0	2	2	−7	2·8	2·7
0	3	−6	8·4	8·6	1	2	−6	4·2	4·0	1	−1	−7	6·0	7·1	2	2	−6	8·6	−7·8
0	3	−5	2·0	−2·0	1	2	−5	11·5	−12·8	1	−1	−8	3·2	−4·6	2	2	−5	15·1	−12·3
0	3	−4	19·3	−20·5	1	2	−4	16·5	−21·6	1	−2	7	3·6	−3·6	2	2	−4	5·2	5·0
0	3	−3	23·9	−21·4	1	2	−3	0·0	−1·0	1	−2	6	5·1	6·3	2	2	−3	26·9	27·2
0	3	−2	8·8	9·4	1	2	−2	19·2	25·3	1	−2	5	14·7	14·5	2	2	−2	19·7	22·5
0	3	−1	42·5	34·5	1	2	−1	15·4	21·1	1	−2	4	0·0	1·0	2	2	−1	6·0	−6·5
0	3	0	12·8	10·1	1	2	0	6·7	−11·0	1	−2	3	30·1	−30·3	2	2	0	34·8	−39·1
0	3	1	40·4	−34·4	1	2	1	46·8	−56·4	1	−2	2	25·0	−28·1	2	2	1	56·6	−50·7
0	3	2	45·1	−35·8	1	2	2	54·9	−50·3	1	−2	1	18·4	21·3	2	2	2	5·1	−5·8
0	3	3	1·7	1·6	1	2	3	16·5	21·6	1	−2	0	39·2	44·5	2	2	3	38·5	39·9
0	3	4	29·5	24·5	1	2	4	35·3	47·0	1	−2	−1	15·9	23·5	2	2	4	18·0	19·7
0	3	5	20·2	18·2	1	2	5	13·8	11·1	1	−2	−2	1·9	−2·7	2	2	5	23·2	−19·7
0	3	6	3·4	2·7	1	2	6	18·0	−14·2	1	−2	−3	18·4	−22·3	2	2	6	21·3	−21·1
0	3	7	8·2	−7·7	1	2	7	10·5	−9·3	1	−2	−4	12·2	−16·1	2	2	7	0·0	−0·8
0	4	−6	3·5	5·3	1	2	8	0·0	0·1	1	−2	−5	5·7	8·8	2	2	8	10·2	9·5
0	4	−5	5·3	−6·7	1	3	−7	5·0	5·9	1	−2	−6	11·8	15·3	2	3	−6	10·8	−9·7
0	4	−4	13·8	−14·0	1	3	−6	2·2	0·6	1	−2	−7	0·0	2·3	2	3	−5	11·6	−9·3
0	4	−3	4·6	−3·5	1	3	−5	10·7	−11·2	1	−3	6	8·2	9·3	2	3	−4	14·8	12·4
0	4	−2	16·1	14·9	1	3	−4	12·1	−12·8	1	−3	5	9·1	7·9	2	3	−3	28·3	28·1
0	4	−1	19·4	20·6	1	3	−3	9·7	9·7	1	−3	4	9·2	−11·1	2	3	−2	12·8	12·9
0	4	0	0·0	−2·1	1	3	−2	29·2	32·5	1	−3	3	21·6	−23·2	2	3	−1	8·0	−9·2
0	4	1	37·5	−33·0	1	3	−1	15·4	17·8	1	−3	2	9·2	−10·6	2	3	0	25·2	−28·7
0	4	2	30·5	−26·1	1	3	0	31·5	−31·5	1	−3	1	10·0	11·6	2	3	1	37·4	−31·7
0	4	3	6·2	6·5	1	3	1	65·2	−59·8	1	−3	0	23·1	26·5	2	3	2	18·7	14·9
0	4	4	25·6	19·8	1	3	2	25·5	−21·2	1	−3	−1	19·7	18·8	2	3	3	50·5	41·0
0	4	5	16·3	12·1	1	3	3	45·4	32·8	1	−3	−2	8·2	−12·3	2	3	4	0·0	1·5
0	4	6	2·5	−1·2	1	3	4	36·6	28·1	1	−3	−3	18·4	−24·6	2	3	5	32·2	−29·8
0	4	7	10·3	−9·6	1	3	5	0·0	−3·6	1	−3	−4	2·5	−1·9	2	3	6	21·3	−19·0
0	5	−5	4·7	−5·7	1	3	6	14·6	−14·1	1	−3	−5	12·1	17·0	2	3	7	4·5	3·1
0	5	−4	15·5	−9·6	1	3	7	8·6	−7·1	1	−3	−6	7·1	12·0	2	3	8	13·1	10·8
0	5	−3	2·6	2·6	1	3	8	0·0	1·7	1	−4	5	3·6	3·7	2	4	−6	9·1	−7·8
0	5	−2	18·1	19·6	1	4	−6	2·8	−4·2	1	−4	4	10·1	−11·5	2	4	−5	5·1	−3·9
0	5	−1	16·6	13·1	1	4	−5	8·8	−8·3	1	−4	3	12·0	−16·8	2	4	−4	13·7	9·6
0	5	0	14·4	−13·0	1	4	−4	3·6	−2·5	1	−4	2	2·6	−2·8	2	4	−3	22·0	20·6
0	5	1	31·4	−23·7	1	4	−3	11·3	11·9	1	−4	1	11·4	12·7	2	4	−2	13·4	11·9
0	5	2	11·7	−10·3	1	4	−2	29·8	27·8	1	−4	0	14·4	16·2	2	4	−1	17·2	−17·3
0	5	3	6·4	6·2	1	4	−1	14·1	14·4	1	−4	−1	3·6	2·2	2	4	0	34·2	−30·1
0	5	4	16·8	13·7	1	4	0	35·5	−34·8	1	−4	−2	11·9	−17·8	2	4	1	0·0	1·3
0	5	5	8·9	8·0	1	4	1	52·0	−45·0	1	−4	−3	11·5	−15·9	2	4	2	46·4	35·8
0	5	6	6·9	−6·5	1	4	2	8·3	6·3	1	−4	−4	3·5	5·2	2	4	3	32·2	25·1
0	6	−3	3·5	4·8	1	4	3	37·2	32·0	1	−4	−5	10·1	16·4	2	4	4	14·3	−11·6
0	6	−2	13·3	14·8	1	4	4	11·1	9·3	1	−4	−6	5·6	9·1	2	4	5	29·9	−28·1
0	6	−1	7·9	6·6	1	4	5	10·9	−11·0	1	−5	4	5·6	−6·9	2	4	6	15·2	−14·6
0	6	0	14·8	−12·2	1	4	6	12·4	−13·6	1	−5	3	8·8	−9·9	2	4	7	7·3	6·4
0	6	1	16·7	−15·0	1	4	7	6·2	−6·6	1	−5	2	0·0	0·1	2	4	8	13·2	10·9
0	6	2	3·1	−2·8	1	4	8	0·5	4·0	1	−5	1	11·3	13·9	2	5	−5	0·0	0·4
0	6	3	8·7	8·5	1	5	−5	5·5	−7·5	1	−5	0	9·6	9·9	2	5	−4	8·5	8·2
0	6	4	8·6	11·4	1	5	−4	0·0	1·6	1	−5	−1	6·1	−9·0	2	5	−3	15·0	10·9
0	7	0	5·9	−7·5	1	5	−3	17·5	15·5	1	−5	−2	11·7	−17·2	2	5	−2	5·0	2·0
0	7	1	7·7	−8·4	1	5	−2	19·6	18·2	1	−5	−3	4·6	−7·4	2	5	−1	22·7	−18·6
0	1	2	27·5	−37·1	1	5	−1	0·0	−2·0	1	−5	−4	5·1	7·5	2	5	0	22·9	−20·0
0	1	3	15·7	−22·2	1	5	0	31·1	−26·3	1	−5	−5	8·7	13·2	2	5	1	16·7	14·7
0	1	4	6·4	9·8	1	5	1	20·0	−16·8	1	−6	2	2·1	4·0	2	5	2	48·0	37·1
0	1	5	18·3	22·7	1	5	2	14·3	15·0	1	−6	1	7·8	9·6	2	5	3	16·1	15·8
0	1	6	9·4	10·5	1	5	3	23·0	22·4	1	−6	0	3·1	3·8	2	5	4	18·3	−16·7
0	1	−8	2·6	−4·5	1	5	4	5·7	3·8	1	−6	−1	6·6	−9·2	2	5	5	27·9	−24·7

TABLE 1 (*Continued*)

h	k	l	$\|F_o\|$	F_c	h	k	l	$\|F_o\|$	F_c	h	k	l	$\|F_o\|$	F_c	h	k	l	$\|F_o\|$	F_c
2	5	6	6·4	−5·6	3	1	−3	32·7	32·2	3	8	1	7·2	7·0	4	2	−2	21·4	−20·4
2	5	7	10·4	11·1	3	1	−2	8·6	8·2	3	8	2	9·1	−8·8	4	2	−3	0·0	0·6
2	6	−4	10·0	8·8	3	1	−1	33·8	−34·5	3	8	3	15·4	−16·5	4	2	−4	14·4	12·9
2	6	−3	5·7	5·1	3	1	0	25·8	−29·1	3	−1	7	1·7	−3·1	4	2	−5	5·6	5·9
2	6	−2	9·0	−9·3	3	1	1	4·5	0·7	3	−1	6	7·3	−7·4	4	3	7	8·0	8·2
2	6	−1	21·1	−16·6	3	1	2	12·9	14·7	3	−1	5	4·8	−3·1	4	3	6	9·5	9·5
2	6	0	5·6	−5·6	3	1	3	12·3	16·0	3	−1	4	5·3	7·7	4	3	5	2·4	2·1
2	6	1	21·3	15·9	3	1	4	4·2	−2·4	3	−1	3	18·4	19·9	4	3	4	14·3	−15·1
2	6	2	30·1	26·3	3	1	5	22·5	−20·5	3	−1	2	11·3	13·1	4	3	3	15·3	−21·8
2	6	3	9·4	8·2	3	1	6	12·1	−13·0	3	−1	1	21·0	−22·5	4	3	2	6·1	9·3
2	6	4	23·0	−18·7	3	1	7	5·3	4·8	3	−1	0	32·6	−36·2	4	3	1	32·2	40·7
2	6	5	21·1	−17·3	3	1	8	8·2	9·6	3	−2	0	15·8	−16·9	4	3	0	14·5	21·3
2	6	6	3·1	4·5	3	2	−6	9·6	−8·4	3	−2	1	19·3	−18·8	4	3	−1	13·1	−15·6
2	7	−2	11·7	−10·6	3	2	−5	4·5	−2·5	3	−2	2	0·0	0·3	4	3	−2	20·2	−21·6
2	7	−1	15·0	−13·8	3	2	−4	16·8	16·9	3	−2	3	15·1	16·6	4	3	−3	5·0	−5·7
2	7	0	0·0	0·3	3	2	−3	29·6	26·8	3	−2	4	10·1	12·6	4	3	−4	6·5	6·5
2	7	1	19·2	16·5	3	2	−2	0·0	−0·2	3	−2	5	0·0	−0·4	4	3	−5	6·3	6·8
2	7	2	17·8	16·1	3	2	−1	32·2	−32·2	3	−2	6	6·1	−6·4	4	4	8	5·4	−4·1
2	7	3	3·7	−3·5	3	2	0	13·7	−18·7	3	−3	4	12·0	11·3	4	4	7	8·2	9·1
2	7	4	17·4	−17·3	3	2	1	21·9	16·5	3	−3	3	8·6	10·3	4	4	6	16·1	14·7
2	7	5	6·2	−6·2	3	2	2	20·0	28·3	3	−3	2	5·7	−5·6	4	4	5	7·7	7·4
2	8	3	7·8	−10·1	3	2	3	8·0	11·3	3	−3	1	14·1	−14·9	4	4	4	9·9	−9·4
2	8	2	7·2	7·2	3	2	4	18·5	−16·0	3	−3	0	4·7	−5·2	4	4	3	23·0	−21·6
2	8	1	13·8	13·9	3	2	5	26·7	−25·0	3	−3	−1	8·1	10·4	4	4	2	4·3	−3·2
2	−1	−7	9·3	10·0	3	2	6	7·2	−10·1	3	−3	−2	10·6	13·6	4	4	1	20·9	27·8
2	−1	−6	3·3	3·1	3	2	7	7·5	7·3	3	−3	−3	0·0	1·6	4	4	0	17·9	22·7
2	−1	−5	16·8	−16·9	3	2	8	7·9	10·0	3	−3	−4	8·0	−10·4	4	4	−1	6·6	−6·3
2	−1	−4	20·8	−21·9	3	3	−6	9·1	−7·9	3	−3	−5	6·5	−8·3	4	4	−2	20·5	−19·3
2	−1	−3	2·1	−2·6	3	3	−5	1·8	3·6	3	−4	−4	5·1	−8·5	4	4	−3	11·1	−12·3
2	−1	−2	22·4	20·0	3	3	−4	16·1	17·0	3	−4	−3	2·6	−3·3	4	4	−4	1·7	1·6
2	−1	−1	21·2	25·5	3	3	−3	11·1	10·9	3	−4	−2	7·3	9·9	4	4	−5	7·9	7·7
2	−1	0	5·5	−1·7	3	3	−2	9·2	−9·1	3	−4	−1	12·3	12·8	4	5	7	4·4	6·1
2	−1	1	28·6	−33·3	3	3	−1	22·2	−20·3	3	−4	0	2·3	−0·3	4	5	6	18·8	18·5
2	−1	2	14·5	−18·0	3	3	0	5·8	−9·6	3	−4	1	10·0	−10·4	4	5	5	11·2	11·1
2	−1	3	15·1	16·3	3	3	1	21·9	25·5	3	−4	2	5·3	−5·8	4	5	4	8·3	−7·0
2	−1	4	28·3	25·0	3	3	2	38·4	40·4	3	−4	3	3·2	3·5	4	5	3	18·6	−17·5
2	−1	5	11·8	11·0	3	3	3	3·9	7·0	3	−1	−1	2·2	−4·3	4	5	2	11·2	−13·3
2	−1	6	8·8	−5·1	3	3	4	23·5	−24·9	3	−1	−2	20·1	20·8	4	5	1	7·8	7·5
2	−1	7	12·6	−10·0	3	3	5	22·7	−23·4	3	−1	−3	10·7	13·1	4	5	0	21·1	22·5
2	−2	−7	6·1	7·2	3	3	6	1·9	−4·7	3	−1	−4	2·7	−3·8	4	5	−1	4·0	6·0
2	−2	−6	8·1	7·8	3	3	7	11·7	10·3	3	−1	−5	8·1	−10·6	4	5	−2	17·5	−16·9
2	−2	−5	6·0	−8·5	3	3	8	7·8	9·1	3	−1	−6	2·9	−5·2	4	5	−3	14·3	−14·5
2	−2	−4	21·1	−24·6	3	4	−6	5·3	−4·9	3	−2	−6	0·0	−2·1	4	5	−4	0·0	0·7
2	−2	−3	11·6	−11·0	3	4	−5	4·4	5·1	3	−2	−5	7·9	−11·8	4	5	−5	6·9	6·6
2	−2	−2	20·8	20·9	3	4	−4	13·0	13·9	3	−2	−4	5·2	−8·9	4	6	7	0·0	0·3
2	−2	−1	27·6	29·5	3	4	−3	1·8	2·6	3	−2	−3	5·4	8·2	4	6	6	13·5	14·8
2	−2	0	3·5	6·4	3	4	−2	20·3	−19·6	3	−2	−2	13·3	17·0	4	6	5	15·1	15·2
2	−2	1	23·3	−17·2	3	4	−1	22·2	−20·1	3	−2	−1	2·6	3·4	4	6	4	1·7	−2·0
2	−2	2	16·3	−16·0	3	4	0	6·3	6·8	3	2	−7	3·6	−4·0	4	6	3	19·5	−18·5
2	−2	3	4·1	2·5	3	4	1	36·8	31·9	3	0	−7	0·0	0·5	4	6	2	18·4	−18·5
2	−2	4	17·6	15·1	3	4	2	30·6	28·5	3	0	−6	7·5	−7·4	4	6	1	0·0	0·3
2	−2	5	9·7	11·0	3	4	3	0·0	1·9	3	0	−5	8·0	−8·3	4	6	0	18·1	17·7
2	−2	6	0·0	−1·4	3	4	4	25·4	−21·3	3	0	−4	3·8	4·6	4	6	−1	9·2	9·4
2	−2	7	7·4	−8·0	3	4	5	20·8	−19·8	3	0	−3	20·8	22·0	4	6	−2	9·4	−8·7
2	−3	−6	7·7	8·9	3	4	6	2·6	2·1	3	0	−2	18·4	18·0	4	6	−3	12·7	−10·5
2	−3	−5	0·0	−0·0	3	4	7	17·6	15·7	3	0	−1	15·2	−18·3	4	6	−4	0·0	−1·7
2	−3	−4	15·2	−18·7	3	4	8	4·3	5·7	3	1	−6	7·8	−8·1	4	7	6	6·1	7·9
2	−3	−3	15·1	−15·1	3	5	−5	5·1	5·0	4	−3	−3	6·8	6·9	4	7	5	15·1	14 9
2	−3	−2	12·8	13·3	3	5	−4	8·7	7·9	4	−3	−2	4·0	5·5	4	7	4	4·8	3 9
2	−3	−1	30·8	25·2	3	5	−3	0·0	−1·6	4	−3	−1	2·5	−3·9	4	7	3	17·0	−15·7
2	−3	0	11·3	11·2	3	5	−2	21·7	−20·7	4	−3	0	9·9	−11·1	4	7	2	17·9	−18 0
2	−3	1	8·1	−5·9	3	5	−1	18·9	−18·6	4	−3	1	6·1	−6·3	4	7	1	2·4	−2·0
2	−3	2	16·7	−15·1	3	5	0	13·3	13·0	4	−3	2	4·6	5·7	4	7	0	9·8	10·5
2	−3	3	6·1	−8·2	3	5	1	33·2	30·1	4	−3	3	8·6	9·2	4	7	−1	10·2	9·6
2	−3	4	9·2	7·6	3	5	2	17·0	15·9	4	0	0	11·7	−14·2	4	7	−2	0·0	0·7
2	−3	5	14·0	10·1	3	5	3	7·3	−5·3	4	0	1	13·1	15·2	4	7	−3	6·8	−6·4
2	−3	6	0·0	0·3	3	5	4	22·4	−20·0	4	0	2	19·2	20·1	4	8	5	7·0	11·0
2	−4	−5	3·3	4·4	3	5	5	11·0	−11·8	4	0	3	2·3	−0·1	4	8	4	5·9	6·6
2	−4	−4	7·3	−8·2	3	5	6	12·7	12·7	4	0	4	12·9	−14·9	4	8	3	5·5	−6·9
2	−4	−3	12·8	−14·4	3	5	7	15·9	16·9	4	0	5	10·9	−12·6	4	8	2	14·6	−14·0
2	−4	−2	0·0	0·1	3	6	−4	3·9	3·4	4	0	6	0·0	−1·1	4	8	1	5·7	−6·6
2	−4	−1	18·2	18·8	3	6	−3	6·6	−7·3	4	0	7	5·8	6·3	4	8	0	6·1	5·9
2	−4	0	17·4	15·7	3	6	−2	16·3	−14·9	4	1	7	7·8	7·5	4	8	−1	9·7	10·7
2	−4	1	3·5	−3·1	3	6	−1	8·8	−8·2	4	1	6	5·2	3·6	4	−1	0	14·4	−17·2
2	−4	2	15·9	−14·0	3	6	0	9·8	9·5	4	1	5	12·1	−13·5	4	−1	1	2·4	2·1
2	−4	3	8·4	−8·5	3	6	1	22·6	20·0	4	1	4	22·7	−23·3	4	−1	2	13·6	14·8
2	−4	4	3·6	2·4	3	6	2	10·8	9·5	4	1	3	2·5	−4·2	4	−1	3	5·2	7·1
2	−5	−3	10·7	−11·0	3	6	3	15·0	−13·9	4	1	2	19·1	23·8	4	−1	4	6·6	−6·6
2	−5	−2	6·6	−6·3	3	6	4	21·0	−21·3	4	1	1	16·7	23·5	4	−1	5	9·4	−10·2
2	−5	−1	9·1	9·8	3	6	5	0·0	0·6	4	1	0	0·0	−1·4	4	−1	6	2·9	−3·0
2	−5	0	14·6	13·3	3	6	6	17·7	19·7	4	1	−1	19·2	−18·8	4	−2	1	6·0	−6·2
2	−5	1	0·0	−0·1	3	6	7	7·2	11·1	4	1	−2	8·2	−10·6	4	−2	2	9·9	10·3
2	−5	2	10·1	−9·1	3	7	−3	9·4	−8·7	4	1	−3	7·5	8·1	4	−2	3	10·3	11·5
3	0	0	46·2	−43·3	3	7	−2	10·6	−10·5	4	1	−4	12·5	13·5	4	−2	4	0·0	−1·3
3	0	1	11·5	−17·4	3	7	−1	0·0	0·1	4	2	7	7·7	7·4	4	−2	5	7·8	−7·2
3	0	2	15·5	17·4	3	7	0	10·2	9·8	4	2	6	8·2	7·2	4	0	−6	3·1	−4·1
3	0	3	16·9	19·7	3	7	1	11·7	11·7	4	2	5	6·2	−7·5	4	0	−5	2·6	2·0
3	0	4	1·7	3·1	3	7	2	1·8	−0·1	4	2	4	24·1	−23·7	4	0	−4	7·9	10·5
3	0	5	11·0	−10·6	3	7	3	16·1	−18·4	4	2	3	12·2	−11·7	4	0	−3	10·8	13·4
3	0	6	9·9	−10·4	3	7	4	12·9	−14·6	4	2	2	16·6	20·2	4	0	−2	0·0	−1·4
3	0	7	2·0	0·2	3	7	5	7·0	8·9	4	2	1	33·8	33·3	4	0	−1	16·5	−22·5
3	1	−5	8·3	−7·2	3	7	6	12·5	17·0	4	2	0	11·7	14·1	4	1	−5	3·9	4·5
3	1	−4	12·2	12·8	3	8	0	11·5	11·3	4	2	−1	17·3	−15·4	4	1	−6	4·1	−3·9

TABLE 1 (*Continued*)

h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c	h	k	l	$\lvert F_o\rvert$	F_c
4	−1	−5	0·0	−0·7	5	2	−3	17·3	−11·9	5	5	1	9·0	−10·2	5	−1	5	4·8	−4·8
4	−1	−4	6·8	8·5	5	2	−4	4·2	−1·3	5	5	0	9·9	9·9	5	−1	4	10·9	−10·1
4	−1	−3	9·0	12·2	5	3	7	0·0	−0·6	5	5	−1	13·5	13·4	5	−1	3	4·0	−4·0
4	−1	−2	0·0	1·4	5	3	6	13·5	11·2	5	5	−2	0·0	2·4	5	−1	2	6·4	7·4
4	−1	−1	12·8	−16·3	5	3	5	16·2	16·2	5	5	−3	8·8	−7·3	5	−1	1	10·9	9·9
4	−2	0	12·4	−15·2	5	3	4	0·0	1·8	5	5	−4	6·9	−6·2	5	−1	0	3·8	1·3
4	−2	−5	1·4	−3·2	5	3	3	18·1	−18·1	5	6	7	8·9	−8·9	5	−1	−1	6·5	−7·0
4	−2	−4	7·3	5·2	5	3	2	9·3	−15·1	5	6	6	3·6	−3·1	5	−1	−2	9·4	−6·5
4	−2	−3	9·0	9·1	5	3	0	17·2	19·7	5	6	5	12·9	11·8	5	−1	−3	3·2	0·8
4	−2	−2	4·1	2·9	5	3	−1	4·5	6·5	5	6	4	14·2	13·4	5	−1	−4	8·3	6·2
4	−2	−1	6·7	−8·2	5	3	−2	12·2	−10·8	5	6	3	4·4	−4·3	5	−2	3	0·0	−1·1
5	0	1	15·0	14·9	5	3	−3	15·5	−13·0	5	6	2	18·1	−19·2	5	−2	2	9·6	7·0
5	0	2	4·4	5·1	5	3	−4	5·2	−3·1	5	6	1	15·8	−15·1	5	−2	1	7·6	5·9
5	0	3	6·6	−7·6	5	3	−5	6·0	4·3	5	6	0	4·9	3·5	5	−2	0	0·0	−3·2
5	0	4	11·1	−11·8	5	4	7	4·7	−3·4	5	6	−1	17·7	15·5	5	−2	−1	8·6	−7·5
5	0	5	2·6	−3·6	5	4	6	10·3	9·0	5	6	−2	7·7	7·5	5	−2	−2	4·7	−3·7
5	0	6	6·1	5·6	5	4	5	21·0	17·7	5	6	−3	6·2	−5·2	5	0	0	10·5	9·9
5	1	6	11·5	9·0	5	4	4	7·7	8·9	5	6	−4	6·4	−5·6	5	0	−5	6·1	4·8
5	1	5	4·3	3·1	5	4	3	12·1	−15·3	5	7	6	7·5	−6·8	5	0	−4	5·5	5·7
5	1	4	11·9	−11·7	5	4	2	25·3	−26·1	5	7	5	4·4	4·3	5	0	−3	3·7	−1·7
5	1	3	12·4	−14·0	5	4	1	0·0	−2·5	5	7	4	15·3	14·5	5	0	−2	12·1	−10·7
5	1	2	3·9	3·4	5	4	0	17·1	18·9	5	7	3	4·8	5·1	5	0	−1	3·8	−5·7
5	1	1	15·1	19·4	5	4	−1	7·9	9·5	5	7	2	16·9	−13·9	5	1	−1	0·0	−2·9
5	1	0	8·2	14·5	5	4	−2	7·1	−7·0	5	7	1	16·4	−15·0	5	1	−2	12·4	−11·9
5	2	7	2·1	0·5	5	4	−3	10·4	−9·7	5	7	0	3·0	1·6	5	1	−3	8·6	−6·3
5	2	6	14·4	11·3	5	4	−4	5·4	−4·4	5	7	−1	14·1	11·9	5	1	−4	0·0	2·6
5	2	5	12·3	11·6	5	4	−5	2·1	1·8	5	7	−2	9·8	7·1	5	1	−5	7·2	5·2
5	2	4	8·0	−7·4	5	5	7	9·9	−7·5	5	8	4	10·8	11·4	5	2	0	12·8	15·7
5	2	3	18·5	−18·9	5	5	6	4·5	3·7	5	8	3	8·6	9·3	5	2	−5	10·2	5·7
5	2	2	1·5	−1·4	5	5	5	21·4	17·2	5	8	2	6·2	−5·7					
5	2	1	10·5	18·3	5	5	4	12·4	11·3	5	8	1	12·3	−11·4					
5	2	−1	1·8	2·0	5	5	3	13·2	−12·1	5	8	0	2·8	−2·5					
5	2	−2	10·7	−10·6	5	5	2	24·1	−24·1	5	8	−1	7·4	6·0					

TABLE 2

Final parameters

Atom	x	y	z	$\sigma(x)$ (Å)	$\sigma(y)$ (Å)	$\sigma(z)$ (Å)	X' (Å)	Y' (Å)	Z' (Å)
As(1)	0·1667	0·0602	0·1785	0·007	0·006	0·006	0·997	1·288	1·147
C(2)	0·4072	−0·2732	0·2794	0·066	0·056	0·068	2·345	0·059	1·795
C(3)	0·1953	0·2660	0·3049	0·069	0·057	0·054	1·223	2·978	1·959
O(4)	0·1879	0·1290	−0·0726	0·033	0·030	0·030	0·966	1·477	−0·466
O(5)	−0·1089	0·1157	0·2543	0·060	0·047	0·043	−0·437	0·746	1·634

Atom	U_{11}	U_{12}	U_{13}	U_{22}	U_{23}	$U_{33} \times 10^2$ (Å^2)
As(1)	5·09	−2·00	−0·71	2·85	−1·06	4·00
C(2)	6·53	−3·20	−0·95	3·89	1·45	7·97
C(3)	9·10	−3·10	−0·99	4·98	−2·46	5·24
O(4)	5·75	−2·68	−1·21	4·23	−0·64	4·18
O(5)	10·12	−3·15	−2·02	4·73	−2·37	4·63

TABLE 3

Bond distances (Å) and valency angles, and standard deviations

Bond distances		Valency angles	
As(1)–C(2)	1·937 ± 0·06	C(2)–As(1)–C(3)	109·9° ± 2·5°
As(1)–C(3)	1·890 ± 0·06	C(2)–As(1)–O(4)	114·8 ± 2·0
As(1)–O(4)	1·625 ± 0·03	C(2)–As(1)–O(5)	107·8 ± 2·3
As(1)–O(5)	1·609 ± 0·05	C(3)–As(1)–O(4)	109·0 ± 2·0
O(4) · · · (5′)	2·567 ± 0·06	C(3)–As(1)–O(5)	106·1 ± 2·3
		O(4)–As(1)–O(5)	108·9 ± 1·8

TABLE 4

Shorter intermolecular distances

(All distances ≤4 Å between molecule 1 and neighbouring molecules were calculated; only contacts <3·5 Å are listed.)

Atom (in molecule 1)	To atom	In molecule	d (Å)	Atom (in molecule 1)	To atom	In molecule	d (Å)
As(1)	O(4)	2	3·46	O(4)	O(5)	2	2·57
As(1)	O(5)	2	3·49	O(5)	O(4)	2	2·57
C(2)	O(4)	3	1·29	O(5)	O(5)	4	3·48
C(2)	O(5)	4	3·34				

Molecule		x	y	z
	1 at	x	y	z
	2 at	$-x$	$-y$	$-z$
	3 at	$1-x$	$-y$	$-z$
	4 at	$-x$	$-y$	$1-z$

The authors are indebted to Dr. W. R. Cullen for the crystal sample, to the University of British Columbia Computing Centre for assistance, and to the National Research Council of Canada and to the Committee on Research, Faculty of Graduate Studies, University of British Columbia, for financial support.

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