

absorption correction was necessary for any of the structures. Details of the data collection are given in Table 1. Scattering factors were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates for (I), (II) and (III) are contained in Table 2.\* Important bond lengths and angles are given in Table 3. Fig. 1 illustrates the molecules with the numbering scheme employed. Fig. 2 illustrates the projected packing of the molecules.

**Related literature.** For additional information on related *N*-acylpyridinium salt reactions and chemistry see Battye, Ihsan & Moodie (1980), Hassner, Krepski & Alexanian (1978), Sheinkman, Suminov &

\* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55216 (56 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0409]

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### *anti*-2-Methyl-1,3-dithiane 1,1,3-Trioxide

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**Abstract.**  $C_5H_{10}O_3S_2$ ,  $M_r = 182.25$ , orthorhombic,  $P2_12_12_1$ ,  $a = 9.019$  (1),  $b = 14.522$  (1),  $c = 6.059$  (1) Å,  $V = 793.7$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.525$  g cm<sup>-3</sup>, Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å,  $\mu = 5.93$  cm<sup>-1</sup>,  $F(000) = 384$ , room temperature,  $R = 0.031$  for 753 reflections with  $I > 3\sigma(I)$ . The S=O bond lengths in —SO<sub>2</sub><sup>-</sup> are 1.426 (3) and 1.432 (3) Å while in the —SO— group the S=O bond is substantially longer, 1.497 (3) Å.

**Experimental.** Colourless crystal, prepared by sequential asymmetric oxidation and deacylation of 2-acetyl-2-methyl-1,3-dithiane (Page & Namwindwa, 1991).

Crystal  $0.38 \times 0.38 \times 0.28$  mm, Rigaku AFC-6S diffractometer,  $\omega/2\theta$  scans, unit cell from 25 reflections with  $15 < 2\theta < 27^\circ$ , Lp corrections, no absorption correction,  $2\theta_{\max} = 50^\circ$ ,  $0 < h < 10$ ,  $0 < k < 16$ ,  $0 < l < 7$  and Friedel equivalents, three standard reflections showed no significant change, 1710 reflections measured, 1410 unique,  $R_{\text{int}} = 0.009$ , 753

with  $I > 3\sigma(I)$  used for refinement. Structure solution by direct methods (*SHELXS86*; Sheldrick, 1986) and refinement on  $F$ , all within *TEXSAN* (Molecular Structure Corporation, 1985); anisotropic vibration parameters for non-H atoms, H atoms located in difference map and their isotropic  $U$  values, but not their coordinates, refined; 91 parameters,  $w^{-1} = [\sigma^2(F) + 0.0009F^2]$ ,  $R = 0.031$ ,  $wR = 0.040$ ,  $S = 1.67$ , max. shift/e.s.d. 0.003, max. and min. peaks in final difference map 0.14,  $-0.11$  e Å<sup>-3</sup>. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1\* gives the atomic coordinates and Table 2 selected bond lengths and angles. Fig. 1 shows the atom numbering.

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55267 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0123]

Table 1. Fractional coordinates and  $B_{eq}$  values with their standard deviations
$$B_{eq} = 8\pi^2 U_{eq} = 8\pi^2/3 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j.$$

	x	y	z	$B_{eq}$ ( $\text{\AA}^2$ )
S(1)	0.5775 (1)	0.34572 (7)	0.5608 (2)	2.81 (4)
S(2)	0.4468 (1)	0.47063 (7)	0.8954 (2)	2.99 (5)
O(1)	0.6043 (4)	0.3354 (2)	0.3292 (5)	4.3 (2)
C(1)	0.3193 (5)	0.3753 (3)	0.9216 (8)	3.4 (2)
C(2)	0.4880 (4)	0.4551 (3)	0.6013 (7)	2.7 (2)
C(3)	0.3932 (5)	0.2817 (3)	0.8824 (8)	3.3 (2)
C(4)	0.5853 (6)	0.5319 (3)	0.5186 (9)	4.4 (2)
C(6)	0.4409 (5)	0.2671 (3)	0.6444 (8)	3.6 (2)
O(2)	0.7012 (3)	0.3396 (2)	0.7066 (6)	4.0 (1)
O(3)	0.3544 (3)	0.5562 (2)	0.9034 (6)	4.1 (1)

Table 2. Selected bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

S(1)	O(1)	1.432 (3)	S(2)	C(2)	1.834 (4)		
S(1)	C(2)	1.798 (4)	S(2)	O(3)	1.497 (3)		
S(1)	C(6)	1.754 (5)	C(1)	C(3)	1.533 (6)		
S(1)	O(2)	1.426 (3)	C(3)	C(6)	1.520 (7)		
S(2)	C(1)	1.807 (4)	C(2)	C(4)	1.505 (6)		
O(1)	S(1)	C(2)	107.6 (2)	S(2)	C(1)	C(3)	112.9 (3)
O(1)	S(1)	C(6)	109.5 (2)	S(2)	C(2)	C(4)	110.5 (3)
O(1)	S(1)	O(2)	117.9 (2)	C(1)	C(3)	C(6)	113.2 (4)
C(2)	S(1)	C(6)	102.7 (2)	S(1)	C(2)	S(2)	109.4 (2)
C(2)	S(1)	O(2)	108.8 (2)	S(1)	C(2)	C(4)	110.3 (3)
C(6)	S(1)	O(2)	109.3 (2)	S(1)	C(6)	C(3)	112.5 (3)
C(1)	S(2)	C(2)	96.9 (2)				
C(1)	S(2)	O(3)	106.2 (2)				
C(2)	S(2)	O(3)	104.3 (2)				

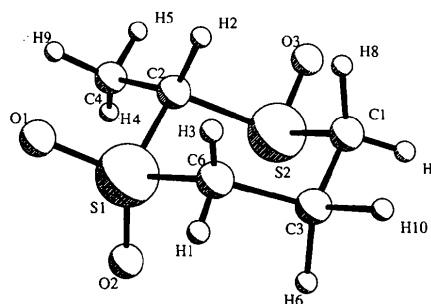


Fig. 1. One molecule of the title compound, illustrating the structure and the atom numbering (PLUTO; Motherwell &amp; Clegg, 1978).

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## Structure of Tetrafluoroisophthalonitrile\*

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**Abstract.**  $C_6F_4(CN)_2$ ,  $M_r = 200.10$ , orthorhombic,  $Pbca$ ,  $a = 13.343$  (7),  $b = 21.991$  (5),  $c = 10.462$  (3)  $\text{\AA}$ ,  $Z = 16$  (two molecules in the asymmetric unit),  $V = 3070$  (4)  $\text{\AA}^3$ ,  $D_x = 1.732$  (2)  $\text{g cm}^{-3}$ , Mo  $K\alpha$  radiation,  $\lambda = 0.71073$   $\text{\AA}$ ,  $\mu = 1.68$   $\text{cm}^{-1}$ ,  $F(000) = 1568$ ,  $T = 172$  (3) K,  $R = 0.056$  for 2671 unique observed reflections with  $I > \sigma(I)$ . The bond lengths and angles are normal, except that both rings are slightly puckered and in both molecules both nitrile groups are bent out of the mean plane of the ring by 2 to 3°. Five short intermolecular distances can be seen in the packing.

**Experimental.** The compound was obtained from Dr Robert Battershell of the Diamond Shamrock Corp.; crystals suitable for X-ray diffraction were found in

the original sample. A colorless crystal  $0.40 \times 0.45 \times 0.45$  mm, mounted in air, was used for the data collection. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 25 reflections with  $11 < \theta < 27^\circ$  were used to determine the cell parameters. Systematic extinctions ( $0kl$ ,  $k$  odd;  $h0l$ ,  $l$  odd;  $hk0$ ,  $h$  odd) uniquely determined the space group. Data were collected, using  $\omega$  scans, in the range  $0 < \theta < 28^\circ$  for one quadrant (ranges:  $h$ , 0 to 17;  $k$ , 0 to 29;  $l$ , –12 to 12). The intensities of 7174 different reflections were measured. Three check reflections measured every 4500 s of exposure time showed no systematic change over the course of the data collection. The structure was solved by direct methods (*MITHRIL*; Gilmore, 1984) and refined with isotropic thermal parameters. After absorption corrections were made, using the program *DIFABS* (Walker & Stuart, 1983; transmis-

\* 2,4,5,6-Tetrafluoro-1,3-benzenedicarbonitrile