Triclinic Modification of Bis(*O*-ethyl dithiocarbonato)tellurium(II)

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Abstract. [Te($C_3H_5OS_2$)$_2$], $M_r = 370\cdot0$, triclinic, $P\bar{1}$, $a = 11\cdot182$ (2), $b = 15\cdot707$ (2), $c = 7\cdot889$ (1) Å, $\alpha = 102\cdot11$ (1), $\beta = 106\cdot41$ (1), $\gamma = 74\cdot85$ (1)°, $U = 1269$ (2) Å^3, $Z = 4$, $D_m = 1\cdot96$ (2), $D_x = 1\cdot937$ Mg m^{-3}, graphite-monochromatized Mo $K\bar{\alpha}$ radiation, $\lambda = 0\cdot7107$ Å, μ(Mo $K\bar{\alpha}$) $= 29\cdot16$ mm^{-1}, $F(000) = 712$, $T = 293$ (2) K, $R = 0\cdot067$ for 2870 reflections with $I \geq 3\cdot0\sigma(I)$. The immediate environment about each Te atom of the asymmetric unit is planar and comprised of four S atoms derived from two asymmetrically chelating xanthate ligands; two such molecules are related so as to form loosely associated dimers *via* weak intermolecular Te···S interactions.

Experimental. Triclinic Te($S_2COC_2H_5$)$_2$ prepared as in the literature (Hoskins, Tiekink & Winter, 1985). Crystal $0\cdot13 \times 0\cdot25 \times 0\cdot28$ mm grown from CHCl$_3$. Density measured in aq. ZnBr$_2$. Enraf–Nonius CAD-4F diffractometer controlled by PDP8/A computer;

ω:2θ scan technique. Cell parameters from least-squares procedure on 25 reflections ($13 \leq \theta \leq 16°$). Max. and min. transmission factors for absorption correction: $0\cdot7189$ and $0\cdot4344$. 4829 reflections measured in range $1° \leq \theta \leq 25°$, $-13 \leq h \leq 13$, $-18 \leq k \leq 18$, $0 \leq l \leq 9$. Three standard reflections ($\bar{1},11,1$, $\bar{7}41$, $\bar{7}43$) decreased, on average, to 90% of initial values and correction applied. 4474 unique reflections ($R_{int} = 0\cdot019$); 2870 satisfied $I \geq 3\cdot0\sigma(I)$. Structure solved from Patterson map and refinement of 236 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms; H-atoms included at their tetrahedral estimates. $R = 0\cdot067$, $wR = 0\cdot07$, $S = 1\cdot01$ for $w = [\sigma^2(F) + 0\cdot011|F|^2]^{-1}$,

Table 1. *Fractional atomic coordinates and B_{eq} (Å^2) values*

$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
Te	$-0\cdot13917$ (7)	$0\cdot16133$ (5)	$0\cdot22228$ (12)	$4\cdot39$
Te'	$0\cdot22034$ (7)	$0\cdot32862$ (6)	$0\cdot27289$ (13)	$4\cdot71$
S(1)	$-0\cdot3350$ (3)	$0\cdot2823$ (2)	$0\cdot2146$ (5)	$5\cdot02$
S(2)	$-0\cdot1044$ (3)	$0\cdot3398$ (3)	$0\cdot2128$ (7)	$6\cdot30$
S(3)	$-0\cdot2799$ (3)	$0\cdot0687$ (2)	$0\cdot2497$ (6)	$5\cdot50$
S(4)	$-0\cdot0127$ (3)	$-0\cdot0208$ (2)	$0\cdot2378$ (6)	$5\cdot57$
S(1')	$0\cdot4182$ (3)	$0\cdot2083$ (2)	$0\cdot2867$ (7)	$6\cdot66$
S(2')	$0\cdot1841$ (3)	$0\cdot1461$ (3)	$0\cdot2477$ (7)	$6\cdot29$
S(3')	$0\cdot3698$ (3)	$0\cdot4265$ (2)	$0\cdot3041$ (7)	$6\cdot32$
S(4')	$0\cdot0984$ (4)	$0\cdot5122$ (3)	$0\cdot2721$ (7)	$6\cdot24$
C(1)	$-0\cdot2578$ (12)	$0\cdot3637$ (9)	$0\cdot2091$ (17)	$4\cdot50$
O(1)	$-0\cdot3337$ (8)	$0\cdot4415$ (5)	$0\cdot2139$ (14)	$4\cdot94$
C(2)	$-0\cdot2864$ (15)	$0\cdot5196$ (10)	$0\cdot2070$ (22)	$5\cdot94$
C(3)	$-0\cdot4002$ (17)	$0\cdot5961$ (10)	$0\cdot1916$ (28)	$6\cdot92$
C(4)	$-0\cdot1568$ (11)	$-0\cdot0234$ (9)	$0\cdot2500$ (18)	$5\cdot15$
O(2)	$-0\cdot1955$ (9)	$-0\cdot0954$ (6)	$0\cdot2594$ (16)	$6\cdot41$
C(5)	$-0\cdot1057$ (14)	$-0\cdot1794$ (9)	$0\cdot2592$ (22)	$5\cdot33$
C(6)	$-0\cdot1732$ (17)	$-0\cdot2501$ (10)	$0\cdot2631$ (22)	$6\cdot19$
C(1')	$0\cdot3423$ (11)	$0\cdot1247$ (8)	$0\cdot2682$ (17)	$3\cdot98$
O(1')	$0\cdot4174$ (9)	$0\cdot0476$ (6)	$0\cdot2719$ (17)	$6\cdot53$
C(2')	$0\cdot3666$ (16)	$-0\cdot0316$ (10)	$0\cdot2578$ (22)	$6\cdot24$
C(3')	$0\cdot4774$ (17)	$-0\cdot1108$ (10)	$0\cdot2304$ (29)	$7\cdot62$
C(4')	$0\cdot2484$ (15)	$0\cdot5188$ (10)	$0\cdot2961$ (22)	$6\cdot02$
O(2')	$0\cdot2866$ (9)	$0\cdot5907$ (6)	$0\cdot2969$ (17)	$6\cdot69$
C(5')	$0\cdot1956$ (14)	$0\cdot6767$ (9)	$0\cdot2924$ (22)	$5\cdot32$
C(6')	$0\cdot2620$ (17)	$0\cdot7468$ (10)	$0\cdot2873$ (25)	$6\cdot83$

Table 2. *Interatomic distances (Å) and bond angles (°)*

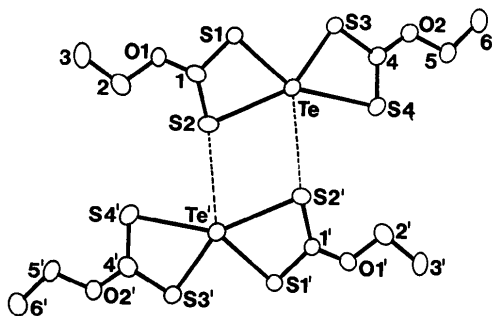|	Unprimed	Primed
Te–S(1)	$2\cdot491$ (3)	$2\cdot499$ (3)
Te–S(2)	$2\cdot948$ (4)	$2\cdot958$ (4)
Te–S(3)	$2\cdot481$ (3)	$2\cdot483$ (4)
Te–S(4)	$2\cdot852$ (3)	$2\cdot850$ (4)
S(1)–C(1)	$1\cdot734$ (12)	$1\cdot703$ (12)
S(2)–C(1)	$1\cdot650$ (13)	$1\cdot678$ (11)
S(3)–C(4)	$1\cdot716$ (13)	$1\cdot706$ (16)
S(4)–C(4)	$1\cdot653$ (13)	$1\cdot662$ (15)
C(1)–O(1)	$1\cdot293$ (16)	$1\cdot281$ (15)
C(4)–O(2)	$1\cdot334$ (16)	$1\cdot307$ (16)
O(1)–C(2)	$1\cdot472$ (15)	$1\cdot469$ (17)
C(2)–C(3)	$1\cdot501$ (23)	$1\cdot536$ (23)
O(2)–C(5)	$1\cdot434$ (16)	$1\cdot463$ (15)
C(5)–C(6)	$1\cdot506$ (21)	$1\cdot492$ (19)
Te···S(2)	$3\cdot509$ (3)	$3\cdot490$ (3)
S(1)–Te–S(2)	$65\cdot1$ (1)	$64\cdot6$ (1)
S(1)–Te–S(3)	$83\cdot3$ (1)	$83\cdot2$ (1)
S(1)–Te–S(4)	$150\cdot5$ (1)	$149\cdot7$ (1)
S(2)–Te–S(3)	$148\cdot2$ (1)	$147\cdot8$ (1)
S(2)–Te–S(4)	$144\cdot4$ (1)	$145\cdot6$ (1)
S(3)–Te–S(4)	$67\cdot3$ (1)	$66\cdot6$ (1)
Te–S(1)–C(1)	$93\cdot4$ (5)	$94\cdot5$ (4)
Te–S(2)–C(1)	$80\cdot0$ (5)	$79\cdot9$ (4)
Te–S(3)–C(4)	$89\cdot9$ (5)	$91\cdot4$ (5)
Te–S(4)–C(4)	$79\cdot1$ (5)	$80\cdot2$ (2)
S(1)–C(1)–S(2)	$121\cdot4$ (8)	$121\cdot0$ (7)
S(3)–C(4)–S(4)	$123\cdot6$ (8)	$121\cdot8$ (8)
S(1)–C(1)–O(1)	$111\cdot0$ (9)	$113\cdot4$ (8)
S(2)–C(1)–O(1)	$127\cdot5$ (9)	$125\cdot7$ (9)
C(1)–O(1)–C(2)	$119\cdot2$ (10)	$120\cdot1$ (10)
O(1)–C(2)–C(3)	$105\cdot9$ (12)	$104\cdot6$ (12)
S(3)–C(4)–O(2)	$110\cdot3$ (9)	$112\cdot2$ (11)
S(4)–C(4)–O(2)	$126\cdot1$ (10)	$125\cdot8$ (12)
C(4)–O(2)–C(5)	$118\cdot4$ (10)	$119\cdot9$ (11)
O(2)–C(5)–C(6)	$108\cdot2$ (12)	$109\cdot2$ (12)

Fig. 1. The numbering scheme used for triclinic Te(S$_2$COC$_2$H$_5$)$_2$, highlighting the loose association of dimers. Atoms otherwise not indicated are C atoms (Johnson, 1971).
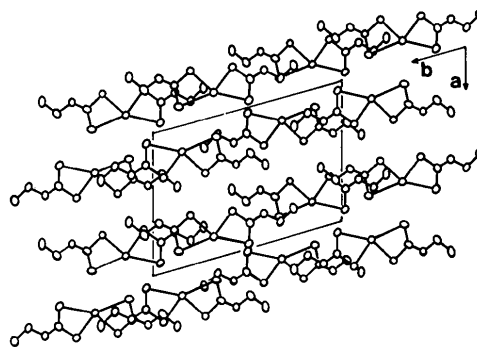

Fig. 2. A c-axis projection of triclinic Te(S$_2$COC$_2$H$_5$)$_2$.

$(\Delta/\sigma)_{max} \leq 0.002$, $(\Delta\rho)_{max} = 2.12$ e Å^{-3}, $(\Delta\rho)_{min} = -1.96$ e Å^{-3}; no extinction correction. Scattering factors for H, C, O, S given in *SHELX*76 (Sheldrick, 1976) and that for neutral Te corrected for anomalous dispersion (Hamilton & Ibers, 1974). University of Adelaide VAX VMS4.1 computer system with *SHELX*76 (Sheldrick, 1976). Crystallographic results* are summarized in Tables 1 and 2 and Figs. 1 and 2.

Related literature. The structure represents a second modification of Te(S$_2$COC$_2$H$_5$)$_2$ with interatomic

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* Lists of structure factors, anisotropic thermal parameters, and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42683 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

parameters in essential agreement with those of the previously reported monoclinic, $P2_1/c$, modification (Husebye, 1967).

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Actinide Structural Studies. 9.* Chlorodioxo(trichloroacetato)bis(triphenylphosphine oxide)uranium Dichloromethane Solvate

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Abstract. [U(C$_2$Cl$_3$O$_2$)ClO$_2$(C$_{18}$H$_{15}$OP)$_2$].CH$_2$Cl$_2$, M_r = 1109.3, monoclinic, $P2_1/n$, $a = 10.029$ (2), $b = 18.924$ (4), $c = 22.454$ (5) Å, $\beta = 96.72$ (2)°, $U = 4232$ (2) Å^3, $Z = 4$, $D_x = 1.74$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $T = 290$ K, $\mu = 41.4$ cm^{-1}, $F(000) = 2152$, $R = 0.062$, $wR = 0.077$ for 5182 unique observed [$I/\sigma(I) \geq 3.0$] reflections. The U coordination is pseudo-octahedral, with Cl *trans* to the trichloracetate. The mean uranyl U–O distance is

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