

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

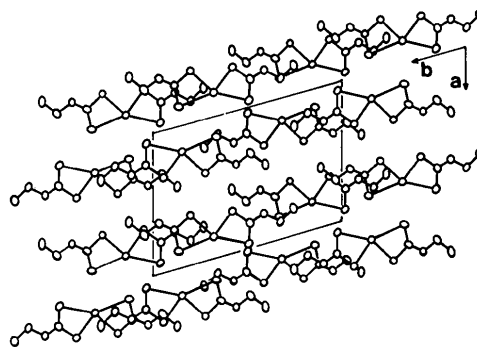


Fig. 2. A *c*-axis projection of triclinic $\text{Te}(\text{S}_2\text{COC}_2\text{H}_5)_2$.

$(\Delta/\sigma)_{\max} \leq 0.002$, $(\Delta\rho)_{\max} = 2.12 \text{ e } \text{\AA}^{-3}$, $(\Delta\rho)_{\min} = -1.96 \text{ e } \text{\AA}^{-3}$; no extinction correction. Scattering factors for H, C, O, S given in *SHELX76* (Sheldrick, 1976) and that for neutral Te corrected for anomalous dispersion (Hamilton & Ibers, 1974). University of Adelaide VAX VMS4.1 computer system with *SHELX76* (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 $\text{Te}(\text{S}_2\text{COC}_2\text{H}_5)_2$ with interatomic

* 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. $[\text{U}(\text{C}_2\text{Cl}_3\text{O}_2)\text{ClO}_2(\text{C}_{18}\text{H}_{15}\text{OP})_2] \cdot \text{CH}_2\text{Cl}_2$, $M_r = 1109.3$, monoclinic, $P2_1/n$, $a = 10.029$ (2), $b = 18.924$ (4), $c = 22.454$ (5) Å, $\beta = 96.72$ (2)°, $U =$

4232 (2) Å³, $Z = 4$, $D_x = 1.74 \text{ g cm}^{-3}$, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $T = 290$ K, $\mu = 41.4 \text{ cm}^{-1}$, $F(000) = 2152$, $R = 0.062$, $wR = 0.077$ for 5182 unique observed [$I/\sigma(I) \geq 3.0$] reflections. The U coordination is pseudo-octahedral, with Cl *trans* to the trichloroacetate. The mean uranyl U–O distance is

* Part 8: Bagnall, Payne, Alcock & Flanders (1985).

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1.71 (1) Å; the U—O(ligand) distances for the trichloroacetate are 2.51 (1) and 2.54 (1) Å, and for the phosphine oxide 2.30 (1) and 2.31 (1) Å. The U—Cl distance is 2.675 (4) Å.

Experimental. During the attempted recrystallization of $U(\text{CCl}_3\text{CO}_2)_4 \cdot 2\text{tppo}$ (tppo = triphenylphosphine oxide) (Bagnall, Lopez & Xing-Fu, 1983), 0.28 g (0.10 mmol) of the complex was dissolved in THF (10 cm³)/CH₂Cl₂ (10 cm³); *n*-pentane was added dropwise until the solution became faintly cloudy. Yellow-green needle crystals separated after the solution stood for 3 d in air; these were washed with CH₂Cl₂ and *n*-pentane, and vacuum dried. The sensitivity of the solution to aerial and photo-induced oxidation was not realized until the results of the structure determination became available.

Table 1. Atomic coordinates ($\times 10^4$) (with standard deviations in parentheses) and average isotropic *U* values

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> (Å ² × 10 ³)
U	681.7 (6)	2398.0 (2)	3328.8 (2)	26
P(1)	3409 (4)	1109 (2)	3599 (2)	28
P(2)	-1479 (4)	4049 (2)	3300 (2)	29
Cl(1)	1402 (5)	2961 (2)	4412 (2)	47
Cl(11)	-6327 (6)	7007 (3)	3803 (2)	62
Cl(12)	-4992 (7)	5702 (2)	3580 (2)	67
Cl(13)	-3426 (6)	6929 (3)	3935 (2)	62
O(1)	2173 (10)	1528 (5)	3677 (4)	34
O(2)	-597 (12)	3418 (5)	3219 (4)	40
O(3)	2011 (13)	2820 (7)	3043 (6)	54
O(4)	-621 (13)	1982 (8)	3574 (5)	66
O(5)	-566 (12)	2407 (5)	2279 (4)	44
O(6)	733 (15)	1493 (5)	2517 (4)	53
C(10)	-4876 (20)	6636 (8)	3525 (7)	47
C(17)	-20 (17)	1862 (7)	2138 (7)	37
C(111)	2934 (15)	232 (7)	3334 (6)	29
C(112)	1660 (15)	4 (8)	3397 (6)	36
C(113)	1282 (20)	-692 (9)	3207 (8)	51
C(114)	2165 (19)	-1108 (8)	2964 (7)	46
C(115)	3435 (18)	-889 (8)	2912 (7)	43
C(116)	3849 (17)	-210 (8)	3093 (6)	38
C(121)	4450 (15)	1051 (7)	4305 (6)	29
C(122)	5601 (16)	634 (9)	4381 (7)	41
C(123)	6370 (18)	596 (10)	4930 (8)	50
C(124)	6049 (22)	991 (9)	5397 (8)	55
C(125)	4880 (24)	1399 (9)	5331 (7)	57
C(126)	4057 (18)	1460 (8)	4787 (6)	38
C(131)	4396 (17)	1489 (7)	3070 (6)	34
C(132)	5525 (18)	1886 (7)	3253 (7)	39
C(133)	6262 (19)	2194 (8)	2836 (8)	52
C(134)	5876 (21)	2091 (9)	2221 (7)	53
C(135)	4720 (18)	1702 (9)	2031 (8)	50
C(136)	3986 (17)	1384 (8)	2452 (7)	40
C(211)	-2903 (16)	3807 (7)	3649 (6)	37
C(212)	-2631 (22)	3389 (11)	4198 (8)	59
C(213)	-3674 (21)	3176 (11)	4499 (9)	63
C(214)	-4967 (24)	3357 (12)	4278 (10)	79
C(215)	-5272 (23)	3751 (11)	3722 (11)	69
C(216)	-4170 (19)	3951 (8)	3436 (9)	53
C(221)	-558 (17)	4702 (7)	3762 (6)	34
C(222)	801 (20)	4734 (9)	3749 (8)	54
C(223)	1602 (23)	5241 (10)	4074 (9)	62
C(224)	912 (25)	5714 (10)	4412 (9)	66
C(225)	-451 (25)	5694 (9)	4437 (8)	63
C(226)	-1206 (21)	5170 (8)	4110 (7)	49
C(231)	-2059 (15)	4425 (7)	2585 (6)	30
C(232)	-2175 (16)	3982 (8)	2086 (6)	35
C(233)	-2694 (16)	4252 (10)	1536 (7)	44
C(234)	-3024 (16)	4949 (10)	1476 (8)	47
C(235)	-2906 (20)	-5390 (10)	1947 (9)	55
C(236)	-2427 (20)	5133 (8)	2514 (9)	46
C(30)	-698 (35)	1745 (11)	5001 (9)	93
Cl(31)	107 (8)	924 (4)	5042 (3)	92
Cl(32)	-669 (11)	2127 (4)	5727 (3)	117

The identification as the title compound was confirmed for the bulk sample by microanalysis (calculated values in parentheses): C 41.8 (42.2), H 2.7 (2.9), Cl 19.5 (19.2), P 5.3 (5.6), U 21.0 (21.5)%; a strong $\nu_{\text{as}}(\text{O—U—O})$ absorption was also present in the IR spectrum (930 cm⁻¹). The adventitious chloride ligand must arise from oxidation of either the solvent or the trichloroacetate anion (probably the latter).

Crystal character: yellow blocks. Data collected with a Syntex *P2*₁ four-circle diffractometer. Maximum 2θ 50°, with scan range $\pm 1.1^\circ$ (2θ) around the $K\alpha_1$ – $K\alpha_2$ angles, scan speed 2–29° min⁻¹, depending on the intensity of a 2 s pre-scan; backgrounds measured at each end of the scan for 0.25 of the scan time. *hkl* ranges: 0 to 11, 0 to 22, -26 to 26. Three standard reflections monitored every 200 reflections, and showed slight changes during data collection: data rescaled to correct for this. Unit-cell dimensions and standard deviations obtained by least-squares fit to 15 reflections ($20 < 2\theta < 25^\circ$). 8180 reflections collected; 5182 considered observed [$I/\sigma(I) \geq 3.0$] and used in refinement; corrections for Lorentz, polarization and absorption effects, the last with *ABSCOR* (Alcock, 1970); max. and min. transmission factors 0.50 and 0.37. Crystal

Table 2. Bond lengths (Å) and bond angles (°) with standard deviations in parentheses

(i) Around U atom			
U—O(1)	2.30 (1)	U—O(5)	2.54 (1)
U—O(2)	2.31 (1)	U—O(6)	2.51 (1)
U—O(3)	1.74 (1)	U—Cl(1)	2.675 (4)
U—O(4)	1.67 (1)		
O(3)—U—O(4)	177.5 (6)	O(1)—U—O(2)	164.6 (3)
O(3)—U—O(1)	87.5 (5)	O(1)—U—O(5)	123.4 (3)
O(3)—U—O(2)	91.0 (5)	O(1)—U—O(6)	71.9 (3)
O(3)—U—O(5)	88.3 (5)	O(1)—U—Cl(1)	83.1 (2)
O(3)—U—O(6)	87.9 (5)	O(2)—U—O(5)	71.9 (3)
O(3)—U—Cl(1)	90.9 (4)	O(2)—U—O(6)	123.4 (4)
O(4)—U—O(1)	93.0 (5)	O(2)—U—Cl(1)	81.5 (3)
O(4)—U—O(2)	89.1 (6)	O(5)—U—O(6)	51.5 (4)
O(4)—U—O(5)	89.4 (5)	O(5)—U—Cl(1)	153.4 (3)
O(4)—U—O(6)	90.0 (5)	O(6)—U—Cl(1)	155.0 (3)
O(4)—U—Cl(1)	91.6 (4)		
(ii) Trichloroacetate group			
C(11)—O(5)	1.22 (2)	C(10)—Cl(11)	1.79 (2)
C(11)—O(6)	1.28 (2)	C(10)—Cl(12)	1.78 (2)
C(11)—C(10)	1.54 (2)	C(10)—Cl(13)	1.72 (2)
U—O(5)—C(11)	92.8 (9)	C(11)—C(10)—Cl(12)	110 (1)
U—O(6)—C(11)	92.9 (9)	C(11)—C(10)—Cl(13)	113 (1)
O(5)—C(11)—O(6)	122 (1)	Cl(11)—C(10)—Cl(12)	108 (1)
O(5)—C(11)—C(10)	120 (1)	Cl(11)—C(10)—Cl(13)	111 (1)
O(6)—C(11)—C(10)	117 (1)	Cl(12)—C(10)—Cl(13)	110 (1)
C(11)—C(10)—Cl(11)	105 (1)		
(iii) Phosphine oxide groups			
		(1)	(2)
O—P		1.50 (1)	1.51 (1)
P—C(11)		1.81 (1)	1.77 (2)
P—C(21)		1.80 (1)	1.80 (1)
P—C(31)		1.78 (2)	1.79 (1)
U—O—P		148.6 (5)	166.3 (6)
O—P—C(11)		109.5 (6)	111.4 (7)
O—P—C(21)		109.4 (6)	110.1 (7)
O—P—C(31)		113.3 (6)	109.8 (6)
P—C(11)—C(12)		118 (1)	116 (1)
P—C(11)—C(16)		121 (1)	125 (1)
P—C(21)—C(22)		122 (1)	118 (1)
P—C(21)—C(26)		117 (1)	121 (1)
P—C(31)—C(32)		121 (1)	118 (1)
P—C(31)—C(36)		118 (1)	123 (1)

dimensions 0.56 × 0.36 × 0.30 mm. Systematic absences $h0l$, $h + l \neq 2n$; $0k0$, $k \neq 2n$ indicating space group $P2_1/n$ (non-standard setting of $P2_1/c$).

Heavy atoms located from a Patterson synthesis and the light atoms then found on successive Fourier syntheses, including one molecule of solvent CH₂Cl₂. Anisotropic temperature factors for all non-H atoms. H atoms given fixed isotropic temperature factors, $B = 5.0 \text{ \AA}^2$, inserted at calculated positions and not refined. Final refinement on F by large-block least-squares methods. Largest positive and negative peaks on a final difference Fourier synthesis of height $\pm 5 \text{ e \AA}^{-3}$ produced by diffraction ripples from U. A weighting scheme of the form $W = X.Y$ applied, where $X = 1.0$ for $(\sin\theta/\lambda) \geq 0.35 \text{ \AA}^{-1}$ and $Y = 1.0$ or $110.0/F_{\text{obs}}$ if $F_{\text{obs}} \geq 110.0$, and shown to be satisfactory by a weighting analysis. Max. Δ/σ in final cycle 0.9. Computing with XRAY76 (Stewart, 1976) on Burroughs B6800. Scattering factors in the analytical form and anomalous-dispersion factors taken from *International Tables for X-ray Crystallography* (1974). Final atomic coordinates are given in Table 1, and bond lengths and angles in Table 2.* Fig. 1 shows a view of the molecule.

Related literature. [UO₂(OPPh₃)₂(NO₃)₂] (Alcock, Roberts & Brown, 1982); [UO₂(OPPh₃)₂Cl₂] (Bombieri, Forsellini, Day & Azeez, 1978).

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* Lists of structure factors, anisotropic thermal parameters and phenyl-group dimensions have been deposited with British Library Lending Division as Supplementary Publication No. SUP 42707 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 1,3:2,5:4,6-Tri-*O*-methylene-D-mannitol*

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Abstract. C₉H₁₄O₆, $M_r = 218.2$, orthorhombic, $P2_12_12_1$, $a = 14.323(2)$, $b = 15.754(2)$, $c = 4.292(1) \text{ \AA}$, $V = 968.5 \text{ \AA}^3$, $Z = 4$, $D_m = 1.50(1)$, $D_x = 1.496 \text{ g cm}^{-3}$, $\text{Cu K}\alpha$ ($\lambda_{\alpha_1} = 1.54056 \text{ \AA}$), $\mu = 10.4 \text{ cm}^{-1}$, $F(000) = 464$, $T = 296 \text{ K}$, final $R = 0.040$

* NRCC No. 25327.

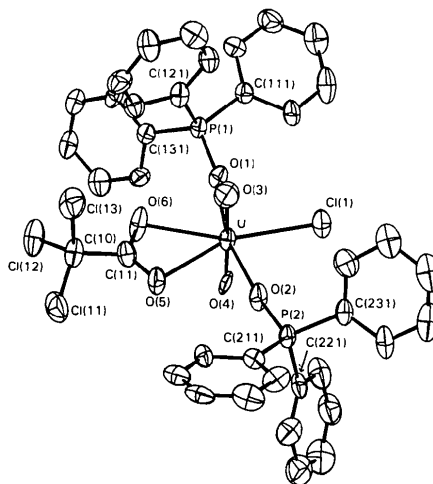


Fig. 1. View of the molecule, showing the atomic numbering (H atoms omitted for clarity).

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for 538 unique observed reflections. The analysis confirms the formulation. The molecule has a non-crystallographic twofold rotation axis passing through the 2,5-*O* methylene C atom and the mid-point of the C(3)–C(4) bond. The torsion angle $\phi[C(2)C(3)C(4)-C(5)]$ of this bond is $48.8(6)^\circ$; the dioxepane ring is in the twist-chair conformation.