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Key indicators

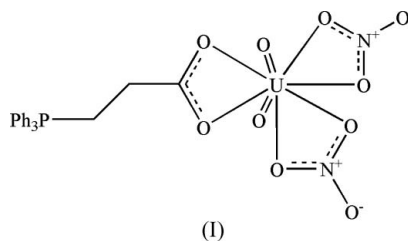
Single-crystal X-ray study
 $T = 291\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.010\text{ \AA}$
 R factor = 0.042
 wR factor = 0.081
Data-to-parameter ratio = 15.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

Dinitratodioxo[3-(triphenylphosphonio)-propionato]uranium(VI)

The U atom in the crystal structure of the title compound, $[\text{U}(\text{NO}_3)_2\text{O}_2(\text{C}_{21}\text{H}_{19}\text{O}_2\text{P})]$, shows hexagonal-bipyramidal coordination, as the nitrate and carboxylate groups function as chelates. The oxo O atoms occupy the axial sites of this uncommon coordination geometry.

Comment

The uranyl ion, UO_2^{2+} , is the most stable form of uranium that exists in nuclear fuel processing (Weigel, 1986). The UO_2^{2+} cation has a linear skeleton in square, pentagonal and hexagonal bipyramidal geometries when connected to carboxylate groups. The two axial O atoms ('uranyl O atoms') typically show no tendency to bridge (Borkowski & Cahill, 2003). The crystal chemistry of uranyl carboxylates is a relatively well developed subject (Leciejewicz *et al.*, 1995).



One strategy for synthesizing uranyl carboxylates involves the use of zwitterionic phosphobetaine in the synthesis. A previous study of triphenylphosphoniopropionate, $\text{Ph}_3\text{P}^+\text{CH}_2\text{CH}_2\text{CO}_2^-$, has described its binding to metal salts (Li & Mak, 1997). The negatively charged carboxylate end binds to dioxouranium dinitrate to yield the title compound, (I) (Fig. 1).

The U atom in (I) is eight-coordinate in a hexagonal-bipyramidal coordination geometry and, as expected, the dioxouranium unit is nearly linear. The bond dimensions compare well with values found in other compounds (Borkowski & Cahill, 2003; Zhang *et al.*, 2002). The six equatorial positions have three pairs of O atoms that come from the chelating nitrate and carboxylate groups; the hexagon is distorted and the angles range from $50.8(2)$ to $69.9(2)^\circ$.

Experimental

Caution: Although the uranium oxynitrate contains depleted uranium, standard precautions for handling radioactive substances should be followed. Triphenylphosphoniopropionate was synthesized according to the literature procedure of Denny & Smith (1962). Uranium oxynitrate hexahydrate (502 mg, 1 mmol) and triphenylphosphoniopropionate (338 mg, 1 mmol) were dissolved in warm

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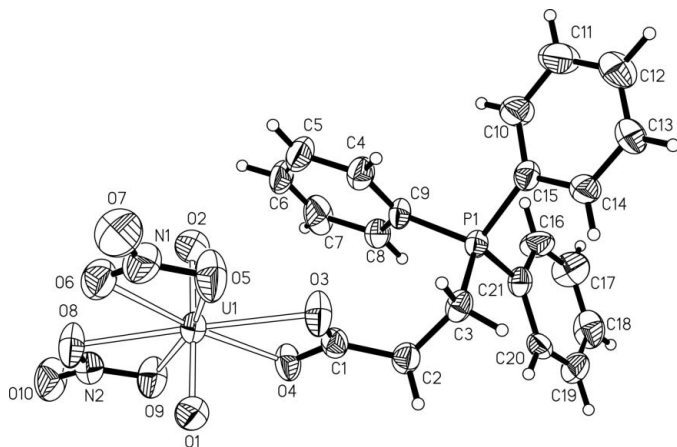


Figure 1

A view of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

water (5 ml) to give a clear solution. A yellow crystalline product formed after a few days in about 90% yield.

Crystal data

[U(NO₃)₂O₂(C₂₁H₁₉O₂P)]
 $M_r = 728.38$
 Monoclinic, $P2_1/c$
 $a = 13.880$ (3) Å
 $b = 9.860$ (5) Å
 $c = 17.962$ (4) Å
 $\beta = 95.42$ (1)°
 $V = 2447$ (2) Å³
 $Z = 4$

$D_x = 1.977$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 20 reflections
 $\theta = 3\text{--}25^\circ$
 $\mu = 6.76$ mm⁻¹
 $T = 291$ (2) K
 Block, yellow
 $0.23 \times 0.20 \times 0.18$ mm

Data collection

Siemens P4 diffractometer
 ω scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.241$, $T_{\max} = 0.296$
 10703 measured reflections
 4808 independent reflections
 3239 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.091$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -17 \rightarrow 17$
 $k = -1 \rightarrow 12$
 $l = -22 \rightarrow 22$
 3 standard reflections
 every 120 reflections
 intensity decay: 5%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.081$
 $S = 1.00$
 4808 reflections
 316 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0124P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.06$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.86$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

U1—O1	1.754 (5)	U1—O5	2.485 (6)
U1—O2	1.743 (5)	U1—O6	2.496 (5)
U1—O3	2.430 (4)	U1—O8	2.498 (5)
U1—O4	2.435 (5)	U1—O9	2.507 (5)
O1—U1—O2	178.3 (2)	O3—U1—O5	69.02 (18)
O1—U1—O3	91.2 (2)	O3—U1—O6	119.37 (17)
O1—U1—O4	89.3 (2)	O3—U1—O8	174.18 (19)
O1—U1—O5	87.4 (2)	O3—U1—O9	123.17 (17)
O1—U1—O6	92.7 (2)	O4—U1—O5	122.12 (16)
O1—U1—O8	89.7 (2)	O4—U1—O6	172.47 (16)
O1—U1—O9	89.4 (2)	O4—U1—O8	120.97 (18)
O2—U1—O3	90.4 (2)	O4—U1—O9	69.90 (16)
O2—U1—O4	92.1 (2)	O5—U1—O6	50.80 (16)
O2—U1—O5	92.7 (3)	O5—U1—O8	116.78 (18)
O2—U1—O6	86.1 (2)	O5—U1—O9	167.49 (18)
O2—U1—O8	88.8 (2)	O6—U1—O8	66.33 (18)
O2—U1—O9	90.2 (2)	O6—U1—O9	117.36 (17)
O3—U1—O4	53.29 (16)	O8—U1—O9	51.08 (17)

The H atoms were positioned geometrically and refined as riding, with C—H = 0.93 and 0.97 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The largest feature in the difference map was 1.11 Å from atom U1.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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