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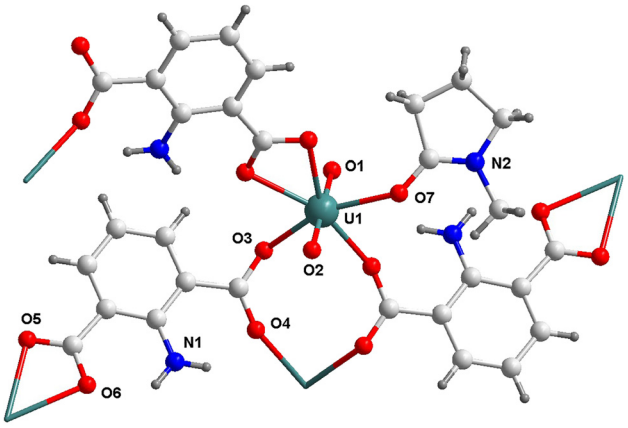
The crystal structure of poly[(μ_2-2-aminoisophthalato-κ^4O,O′:O″:O‴)-(*N*-methylpyrrolidone κ^1O)-dioxido-uranium(VI)], $C_{13}H_{14}N_2O_7U$



https://doi.org/10.1515/ncrs-2024-0309
Received July 17, 2024; accepted October 10, 2024;
published online October 23, 2024

Abstract

$C_{13}H_{14}N_2O_7U$, monoclinic, $P2_1/n$ (no. 14), a = 8.6855(14) Å, b = 11.982(2) Å, c = 14.953(2) Å, β = 94.557(5)°, V = 1,551.3(4) Å^3, Z = 4, $R_{gt}(F)$ = 0.0559, wR_{ref} (F^2) = 0.1081, T = 150(2) K

CCDC no.: 2389946

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

All reagents are obtained commercially and used as received. The crystals of the title uranyl complex were synthesized solvothermally. A mixture of 0.1 mmol $UO_2(NO_3)_2{\cdot}6H_2O$ (0.0502 g), 0.1 mmol 2-aminoisophthalic acid (0.0181 g), and 1 mmol boric acid (0.0618 g) was added to 2 mL N-methylpyrrolidone, stirred for 10 min, and transferred to a 25 mL Teflon-lined stainless steel autoclave, which was placed in an oven and heated at 373 K for 72 h, then cooled, washed with distilled water, and dried in air, yielding 68 % (based on 2-aminoisophthalic acid).

Table 1: Data collection and handling.

Crystal:	Yellow needle
Size:	0.20 × 0.15 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	10.5 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.2°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	22,418, 2,754, 0.099
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2,155
$N(param)_{refined}$:	209
Programs:	Bruker,[1] Olex2,[2] SHELX[3,4]

2 Experimental details

The structure was solved by Direct Methods with the SHELXS-2018 program. All H-atoms from C and N atoms were positioned with idealized geometry and refined isotropically (U_{iso}(H) = 1.2U_{eq}(C) or 1.2U_{eq}(N)) using a riding model with C–H = 0.949, 0.950 for 2-aminoisophthalate 0.980, 0.989, 0.991 and 0.992 Å for N-methylpyrrolidone, and N–H = 0.880 Å, respectively.

3 Comment

Many uranyl complexes based on isophthalic acid and its derivatives have been synthesized with diversity of skeleton charge, structure dimension, and nuclear number due to the modifiability and coordination variousness of the isophthalic acid.[5–17] Herein, we report the single crystal structure of a

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4707 (15)	0.4837 (10)	0.6381 (8)	0.020 (2)
C2	0.4297 (14)	0.4734 (10)	0.7324 (8)	0.0190 (19)
C3	0.4777 (15)	0.3759 (11)	0.7798 (8)	0.021 (2)
H3	0.531970	0.319709	0.750456	0.025*
C4	0.4470 (14)	0.3610 (11)	0.8682 (8)	0.0226 (19)
H4	0.480125	0.295061	0.899402	0.027*
C5	0.3677 (15)	0.4425 (11)	0.9110 (9)	0.022 (2)
H5	0.348517	0.433122	0.972168	0.026*
C6	0.3163 (15)	0.5372 (10)	0.8658 (8)	0.0193 (19)
C7	0.3456 (14)	0.5567 (10)	0.7747 (8)	0.0180 (18)
C8	0.2321 (14)	0.6200 (10)	0.9163 (8)	0.020 (2)
C9	0.3777 (19)	0.1487 (15)	0.2839 (11)	0.045 (3)
C10	0.396 (2)	0.0251 (14)	0.3179 (11)	0.046 (3)
H10A	0.495590	0.013717	0.353613	0.056*
H10B	0.310306	0.003345	0.354274	0.056*
C11	0.390 (2)	−0.0379 (15)	0.2316 (11)	0.050 (3)
H11A	0.286272	−0.071166	0.218283	0.060*
H11B	0.467373	−0.098641	0.235321	0.060*
C12	0.426 (2)	0.0488 (14)	0.1554 (11)	0.048 (3)
H12A	0.535543	0.045262	0.141242	0.057*
H12B	0.358005	0.037621	0.099848	0.057*
C13	0.379 (2)	0.2565 (14)	0.1493 (12)	0.049 (4)
H13A	0.291034	0.252010	0.104002	0.073*
H13B	0.473882	0.269343	0.119675	0.073*
H13C	0.362620	0.318205	0.190505	0.073*
N1	0.2900 (13)	0.6486 (9)	0.7304 (7)	0.027 (3)
H1A	0.305839	0.658068	0.673514	0.032*
H1B	0.238157	0.698986	0.758590	0.032*
N2	0.3920 (16)	0.1510 (12)	0.2000 (9)	0.048 (4)
O1	0.5870 (9)	0.2077 (8)	0.4865 (5)	0.0235 (19)
O2	0.2433 (11)	0.3651 (8)	0.4798 (6)	0.032 (2)
O3	0.5246 (11)	0.3984 (7)	0.5998 (6)	0.025 (2)
O4	0.4592 (12)	0.5780 (7)	0.6005 (6)	0.030 (2)
O5	0.2301 (11)	0.6122 (7)	1.0010 (6)	0.025 (2)
O6	0.1581 (11)	0.7021 (8)	0.8783 (5)	0.026 (2)
O7	0.3483 (11)	0.2300 (8)	0.3340 (6)	0.031 (2)
U1	0.41517 (6)	0.28693 (4)	0.48354 (3)	0.01970 (15)

uranyl complex based on 2-aminoisophthalate with coordinated N-methylpyrrolidone.

As shown in the figure, the asymmetric unit is made of one uranyl cation, one completely deprotonated 2-aminoisophthalic acid, and one coordinated N-methylpyrrolidone molecule. It can be seen that the U1 is seven-coordinated and coordinates with seven oxygen atoms to form a pentagonal bipyramid polyhedron: two oxygen atoms from the uranyl cation, two oxygen atoms from the same carboxyl groups in the ligand chelating with U1, two oxygen atoms from two carboxyl groups of two different ligands, and the last oxygen atom from the solvent molecule N-methyl pyrrolidone. Two adjacent uranyl cations are bridged by four oxygen atoms from two ligands to generate a two-dimensional structure. The bond lengths of U–O are in the range of 1.759–2.465 Å, which are similar to other reported uranyl pentagonal bipyramids.[5–25]

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: Postgraduate Education Reform and Quality Improvement Project of Henan Province (YJS2023JD65).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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