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A Salophene–Uranium–Water Complex

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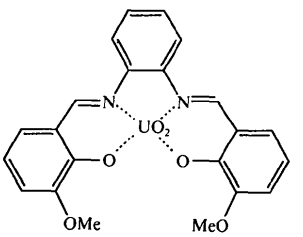
Abstract

In dioxo{2,2′-[1,2-phenylenebis(nitrilomethylidyne)]-bis(6-methoxyphenolato)(2−)-*N*,*N*′,*O*,*O*′]uranium–water (2/3), $2[UO_2(C_{22}H_{18}N_2O_4)].3H_2O$, the two independent uranium cations have pentagonal bipyramidal coordination.

Comment

Normally, metal cations (Cu^{2+}, Ni^{2+}, Pd^{2+}, Zn^{2+}) in a salophene moiety have square-planar coordination (Holm & O'Connor, 1971; van Staveren, van Eerden, van Veggel, Harkema & Reinhoudt, 1988). As a result of the large radius of the uranium cation, the salophene moiety in the title compound (I) is distorted and pentagonal bipyramidal coordination is found. The bowl-like shape of the salophene moiety is also found in related solid-state structures with angles of the adjacent aromatic rings (50.8–53.0° and 50.2–52.6°) comparable to those reported previously (van Staveren, van Eerden, van Veggel, Harkema & Reinhoudt, 1988; van Doorn, Bos, Harkema, van Eerden, Verboom & Reinhoudt, 1991; van Doorn, Schaafstra, Bos, Harkema, van Eerden, Verboom & Reinhoudt, 1991). The water molecule at the fifth coordination site can be exchanged for other neutral molecules, which is of critical importance for molecular recognition by metalloreceptors.



(I)

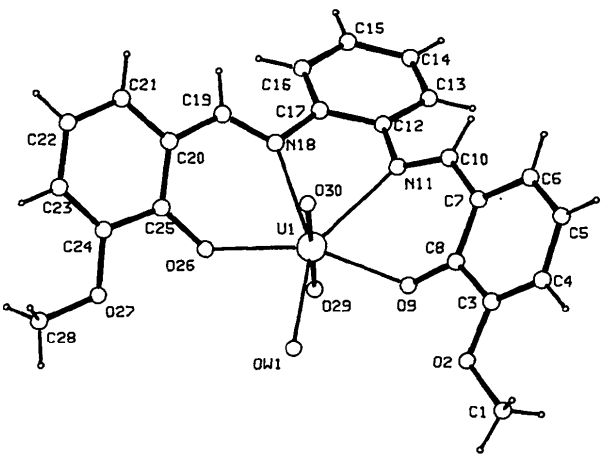


Fig. 1. View of the title complex including the atom numbering. The numbering of the second independent molecule is obtained by adding 30 to the numbers given.

Experimental

The title complex was synthesized according to the procedures of van Doorn, Bos, Harkema, van Eerden, Verboom & Reinhoudt (1991), to investigate its structure and coordination. A crystal suitable for X-ray analysis was obtained by slow evaporation of the solvent from a solution in $CHCl_3$.

Crystal data

$2[UO_2(C_{22}H_{18}N_2O_4)].3H_2O$
M_r = 1342.9
Triclinic
$P\bar{1}$
a = 12.056 (6) Å
b = 12.293 (5) Å
c = 15.843 (6) Å
α = 84.01 (4)°
β = 74.91 (6)°
γ = 86.29 (5)°
V = 2253 (2) Å^3
Z = 2
D_x = 1.98 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 23 reflections
θ = 6–12°
μ = 6.88 mm^{-1}
T = 293 K
Parallelepiped
0.7 × 0.4 × 0.1 mm
Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans [width (1.0 + 0.34tanθ)°, speed 0.3° s^{-1}]
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
T_{min} = 0.78, T_{max} = 1.24
15 016 measured reflections
7875 independent reflections
5771 observed reflections [$F_o^2 > 3\sigma(F_o^2)$]
R_{int} = 0.021
θ_{max} = 25°
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity variation: 1.6%

Refinement

Refinement on F
R = 0.040
wR = 0.051
S = 1.83
5571 reflections
591 parameters
$w = 4F_o/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max}$ = 0.02
$\Delta\rho_{max}$ = 1.8 e Å^{-3} (close to U)
$\Delta\rho_{min}$ = −1.92 e Å^{-3}
Extinction correction: Zachariasen (1968)
Extinction coefficient: 10 (1) × 10^{-8}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

B_{iso} for OW4; $B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for other atoms.

	x	y	z	B_{iso}/B_{eq}
U1	0.05450 (3)	0.12389 (3)	0.13452 (2)	2.264 (7)
U31	0.38346 (3)	0.59297 (3)	0.34489 (2)	2.239 (7)
OW1	−0.0751 (6)	0.1284 (6)	0.0358 (5)	4.1 (2)
OW31	0.3746 (8)	0.5235 (7)	0.5004 (5)	5.1 (2)
OW3	0.9726 (8)	0.7824 (7)	0.1169 (5)	5.6 (2)
OW4	0.419 (2)	0.319 (2)	0.504 (2)	22.2 (9)
O2	−0.2614 (6)	−0.0831 (7)	0.1881 (6)	5.0 (2)
O9	−0.0655 (6)	−0.0136 (6)	0.1888 (4)	3.4 (2)
O26	0.1394 (6)	0.2584 (5)	0.0360 (4)	2.8 (1)
O27	0.1189 (6)	0.4093 (6)	−0.0897 (5)	3.6 (2)
O29	0.1517 (6)	0.0307 (6)	0.0689 (4)	3.8 (2)
O30	−0.0399 (6)	0.2185 (6)	0.1996 (4)	3.7 (2)
O32	0.0660 (7)	0.6340 (7)	0.5666 (5)	4.2 (2)
O39	0.2234 (5)	0.6730 (6)	0.4243 (4)	2.8 (1)
O56	0.5440 (5)	0.4816 (6)	0.3274 (4)	3.2 (1)
O57	0.6477 (6)	0.3069 (6)	0.3857 (5)	4.0 (2)
O59	0.4645 (6)	0.7063 (5)	0.3537 (5)	3.1 (2)
O60	0.3033 (6)	0.4812 (6)	0.3336 (5)	3.1 (2)
N11	0.0849 (6)	0.0356 (6)	0.2803 (5)	2.3 (2)
N18	0.2098 (6)	0.2013 (7)	0.1902 (5)	2.6 (2)
N41	0.2848 (7)	0.7111 (6)	0.2422 (5)	2.5 (2)
N48	0.4780 (6)	0.5947 (6)	0.1822 (5)	2.4 (2)
C1	−0.356 (1)	−0.142 (1)	0.180 (1)	8.3 (4)
C3	−0.2459 (9)	−0.0779 (9)	0.2703 (8)	3.7 (2)
C4	−0.320 (1)	−0.107 (1)	0.3472 (9)	4.2 (3)
C5	−0.292 (1)	−0.095 (1)	0.4263 (9)	4.6 (3)
C6	−0.1877 (9)	−0.0606 (9)	0.4248 (8)	3.7 (3)
C7	−0.1090 (9)	−0.0318 (8)	0.3449 (7)	3.0 (2)
C8	−0.1372 (8)	−0.0363 (8)	0.2650 (7)	2.9 (2)
C10	0.0070 (9)	−0.0038 (8)	0.3452 (7)	3.1 (2)
C12	0.1970 (8)	0.0426 (8)	0.2931 (7)	2.7 (2)
C13	0.2431 (9)	−0.0358 (9)	0.3456 (7)	3.2 (2)
C14	0.3527 (9)	−0.023 (1)	0.3542 (7)	4.0 (3)
C15	0.4132 (9)	0.067 (1)	0.3127 (8)	4.5 (3)
C16	0.3676 (9)	0.143 (1)	0.2593 (8)	3.8 (3)
C17	0.2590 (7)	0.1306 (8)	0.2486 (6)	2.5 (2)
C19	0.2336 (8)	0.3049 (8)	0.1782 (6)	2.4 (2)
C20	0.2021 (8)	0.3864 (8)	0.1162 (6)	2.4 (2)
C21	0.2210 (9)	0.4958 (9)	0.1238 (7)	3.3 (2)
C22	0.2005 (9)	0.5764 (9)	0.0623 (7)	3.6 (2)
C23	0.1644 (9)	0.5505 (9)	−0.0078 (7)	3.3 (2)
C24	0.1497 (8)	0.4436 (8)	−0.0195 (6)	2.8 (2)
C25	0.1613 (8)	0.3599 (7)	0.0453 (6)	2.2 (2)
C28	0.114 (1)	0.488 (1)	−0.1608 (7)	5.0 (3)
C31	−0.019 (1)	0.620 (1)	0.6476 (8)	5.7 (4)
C33	0.0287 (9)	0.6507 (9)	0.4905 (7)	3.2 (2)
C34	−0.0802 (9)	0.646 (1)	0.4859 (8)	4.0 (3)
C35	−0.1094 (9)	0.665 (1)	0.4070 (8)	4.4 (3)
C36	−0.0271 (8)	0.6916 (9)	0.3322 (7)	3.3 (2)
C37	0.0903 (8)	0.6950 (8)	0.3349 (7)	2.9 (2)
C38	0.1188 (8)	0.6732 (8)	0.4152 (6)	2.5 (2)
C40	0.1727 (8)	0.7231 (8)	0.2541 (6)	2.6 (2)
C42	0.3551 (8)	0.7498 (8)	0.1605 (6)	2.3 (2)
C43	0.3316 (9)	0.8455 (8)	0.1123 (7)	3.3 (2)
C44	0.409 (1)	0.8816 (9)	0.0334 (7)	3.6 (2)
C45	0.508 (1)	0.8218 (9)	0.0020 (7)	3.7 (3)
C46	0.5318 (9)	0.7246 (9)	0.0511 (7)	3.2 (2)
C47	0.4561 (8)	0.6890 (8)	0.1292 (6)	2.6 (2)
C49	0.5271 (8)	0.5094 (9)	0.1453 (7)	3.1 (2)
C50	0.5682 (8)	0.4104 (8)	0.1869 (6)	2.5 (2)
C51	0.6072 (9)	0.3227 (8)	0.1344 (7)	3.3 (2)
C52	0.6577 (9)	0.2300 (9)	0.1660 (8)	3.8 (3)
C53	0.6720 (9)	0.2236 (8)	0.2501 (8)	3.7 (2)
C54	0.6361 (8)	0.3094 (8)	0.3016 (7)	3.1 (2)
C55	0.5780 (7)	0.4023 (8)	0.2733 (7)	2.7 (2)
C58	0.708 (1)	0.214 (1)	0.4169 (9)	6.6 (3)

Table 2. *Selected geometric parameters* (Å, °)

U1—OW1	2.477 (8)	C5—C6	1.34 (2)
U1—O9	2.249 (7)	C6—C7	1.40 (1)
U1—O26	2.251 (6)	C7—C8	1.41 (2)
U1—O29	1.791 (6)	C7—C10	1.46 (2)
U1—O30	1.782 (7)	C12—C13	1.40 (1)
U1—N11	2.552 (8)	C12—C17	1.38 (1)
U1—N18	2.541 (9)	C13—C14	1.38 (2)
U31—OW31	2.500 (8)	C14—C15	1.37 (2)
U31—O39	2.242 (6)	C15—C16	1.38 (2)
U31—O56	2.271 (6)	C16—C17	1.38 (1)
U31—O59	1.790 (7)	C19—C20	1.43 (1)
U31—O60	1.780 (7)	C20—C21	1.40 (1)
U31—N41	2.543 (9)	C20—C25	1.41 (1)
U31—N48	2.532 (7)	C21—C22	1.37 (2)
O2—C1	1.43 (2)	C22—C23	1.37 (2)
O2—C3	1.37 (2)	C23—C24	1.37 (2)
O9—C8	1.30 (2)	C24—C25	1.40 (1)
O26—C25	1.32 (1)	C33—C34	1.34 (2)
O27—C24	1.37 (1)	C33—C38	1.41 (1)
O27—C28	1.42 (1)	C34—C35	1.38 (2)
O32—C31	1.42 (1)	C35—C36	1.36 (1)
O32—C33	1.39 (1)	C36—C37	1.44 (1)
O39—C38	1.31 (1)	C37—C38	1.40 (2)
O56—C55	1.34 (1)	C37—C40	1.43 (1)
O57—C54	1.37 (1)	C42—C43	1.39 (1)
O57—C58	1.43 (1)	C42—C47	1.40 (1)
N11—C10	1.27 (1)	C43—C44	1.40 (1)
N11—C12	1.43 (1)	C44—C45	1.38 (1)
N18—C17	1.42 (1)	C45—C46	1.41 (1)
N18—C19	1.31 (1)	C46—C47	1.39 (1)
N41—C40	1.32 (1)	C49—C50	1.44 (1)
N41—C42	1.40 (2)	C50—C51	1.41 (1)
N48—C47	1.42 (1)	C50—C55	1.40 (2)
N48—C49	1.29 (1)	C51—C52	1.37 (2)
C3—C4	1.34 (2)	C52—C53	1.39 (2)
C3—C8	1.42 (2)	C53—C54	1.38 (2)
C4—C5	1.40 (3)	C54—C55	1.40 (1)
OW1—U1—O9	74.3 (3)	O60—U31—N41	88.3 (3)
OW1—U1—O26	82.8 (3)	O60—U31—N48	87.5 (3)
OW1—U1—O29	90.2 (3)	N41—U31—N48	63.3 (2)
OW1—U1—O30	90.4 (3)	C1—O2—C3	119 (1)
OW1—U1—N11	142.8 (2)	C24—O27—C28	117.9 (9)
OW1—U1—N18	153.1 (2)	C31—O32—C33	117.8 (9)
O9—U1—O26	156.6 (3)	C54—O57—C58	117.2 (9)
O9—U1—O29	89.1 (3)	C10—N11—C12	117.4 (9)
O9—U1—O30	92.0 (3)	C17—N18—C19	119.0 (9)
O9—U1—N11	69.0 (3)	C40—N41—C42	118.0 (8)

O9—U1—N18	132.2 (3)	C47—N48—C49	119.2 (8)
O26—U1—O29	86.7 (3)	O2—C3—C4	127 (1)
O26—U1—O30	92.4 (3)	C42—C43—C44	121 (1)
O26—U1—N11	134.3 (3)	C43—C44—C45	121 (1)
O26—U1—N18	71.1 (3)	C44—C45—C46	119.0 (9)
O29—U1—O30	178.8 (3)	C45—C46—C47	121.1 (9)
O29—U1—N11	94.9 (3)	N48—C47—C42	117.1 (7)
O29—U1—N18	94.1 (3)	N48—C47—C46	123.1 (8)
O30—U1—N11	85.1 (3)	C42—C47—C46	119.8 (9)
O30—U1—N18	84.8 (3)	N48—C49—C50	127 (1)
N11—U1—N18	63.2 (2)	C49—C50—C51	117.1 (9)
OW31—U31—O39	75.2 (2)	C49—C50—C55	122.6 (9)
OW31—U31—O56	78.8 (2)	O2—C3—C8	110.9 (9)
OW31—U31—O59	90.6 (3)	C4—C3—C8	122 (1)
OW31—U31—O60	90.8 (4)	C3—C4—C5	120 (1)
OW31—U31—N41	146.0 (2)	C4—C5—C6	120 (2)
OW31—U31—N48	150.7 (3)	C5—C6—C7	120 (1)
O39—U31—O56	153.9 (2)	C6—C7—C8	122 (2)
O39—U31—O59	90.0 (3)	C6—C7—C10	119 (2)
O39—U31—O60	90.5 (3)	C8—C7—C10	119.9 (8)
O39—U31—N41	70.8 (2)	O9—C8—C3	120 (2)
O39—U31—N48	134.1 (2)	O9—C8—C7	123.3 (9)
O56—U31—O59	90.4 (3)	C3—C8—C7	116.2 (9)
O56—U31—O60	89.7 (3)	N11—C10—C7	127 (2)
O56—U31—N41	135.2 (2)	N11—C12—C13	122.5 (8)
O56—U31—N48	72.0 (2)	N11—C12—C17	116.2 (9)
O59—U31—O60	178.8 (3)	C13—C12—C17	121 (1)
O59—U31—N41	90.7 (3)	C12—C13—C14	118.6 (9)
O59—U31—N48	91.3 (3)	C13—C14—C15	120 (2)
C14—C15—C16	121 (1)	C34—C35—C36	120 (2)
C15—C16—C17	120 (1)	C35—C36—C37	121 (1)
N18—C17—C12	117.7 (9)	C36—C37—C38	119.4 (8)
N18—C17—C16	123.1 (8)	C36—C37—C40	117 (1)
C12—C17—C16	119.1 (9)	C38—C37—C40	123.3 (9)
N18—C19—C20	127 (1)	O39—C38—C33	118.3 (9)
C19—C20—C21	117 (1)	O39—C38—C37	124.1 (8)
C19—C20—C25	122.8 (9)	C33—C38—C37	117.5 (9)
C21—C20—C25	119.6 (9)	N41—C40—C37	124.7 (9)
C20—C21—C22	120 (2)	N41—C42—C43	124.2 (8)
C21—C22—C23	121 (2)	N41—C42—C47	116.5 (8)
C22—C23—C24	121 (1)	C43—C42—C47	119.3 (8)
O27—C24—C23	125.4 (9)	C51—C50—C55	120.1 (9)
O27—C24—C25	114.6 (9)	C50—C51—C52	121 (2)
C23—C24—C25	120 (2)	C51—C52—C53	120 (2)
O26—C25—C20	122.2 (8)	C52—C53—C54	121 (1)
O26—C25—C24	119.3 (9)	O57—C54—C53	122.9 (9)
C20—C25—C24	118.5 (9)	O57—C54—C55	115.4 (9)
O32—C33—C34	125.6 (9)	C53—C54—C55	122 (2)
O32—C33—C38	112.7 (9)	O56—C55—C50	124.1 (8)
C34—C33—C38	122 (2)	O56—C55—C54	118.3 (9)
C33—C34—C35	121 (1)	C50—C55—C54	117.3 (9)

Data were corrected for Lorentz and polarization effects. The unit cell contains two independent salophene–uranium–water complexes (1:1:1) and an isolated water molecule. The uranium cations were located by the Patterson method and the rest of the non-H atoms by successive Fourier syntheses. Weights for each reflection were calculated from $w = 4F_o/\sigma^2(F_o^2)$ and $\sigma^2(F_o^2) = \sigma^2(I) + (pF_o^2)^2$; the value of the instability factor p was determined to be 0.04. In the difference Fourier map two peaks were found which were attributed to water O atoms. One of the O atoms (*OW3*) gave a reasonable displacement parameter; the displacement parameter for the other water O atom (*OW4*) became very large ($B > 20$) on refinement. From the large value of B, we concluded that the position of the second water molecule is only partially occupied (the latter has not been included in the formula). *OW4* was refined isotropically, all other non-H atoms were refined anisotropically. H atoms were treated as riding on their parent atom at a distance of 0.95 Å, except for the water H atoms which were not located and therefore not included in the refinement. Calculations were performed using *SDP-Plus* (B. A. Frenz & Associates, Inc., 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HU1037). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Racemic Chloro[tris(2-aminoethyl)amine]zinc(II) Chloride Trihydrate, $[ZnCl(C_6H_{18}N_4)]Cl.3H_2O$

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Abstract

The synthesis and the crystal and molecular structure of the five-coordinate racemic $[Zn(tren)Cl]Cl.3H_2O$ complex [tren = tris(2-aminoethyl)amine, $C_6H_{18}N_4$] are reported. The coordination polyhedron around