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The Crystal and Molecular Structure of N, N'-Bissalicylidene-1, 5-diamino-3-azapentanedioxouranium(VI)

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The ligand in the title compound is quinquedentate, and gives rise to a 7-coordinate (approximately pentagonal bipyramidal) uranyl complex. The crystal structure is orthorhombic, *Pnma*, with a = 10.50, b = 21.76, and c = 8.025 Å; 716 unique reflexions were observed and the structure was refined to R = 0.069. The deviations of the uranium environment from strict D_{sh} symmetry are discussed, and the ligand geometry is compared with that of its quadridentate (lower) homologue.

Introduction

N,N'-Bissalicylidene-1,5-diamino-3-azapentane (referred to hereafter as saldienH₂) has been shown to act as a planar quinquedentate ligand in its complex with uranium(VI) (Akhtar, McKenzie, Paine & Smith, 1969), although it is only quadridentate with first-row transition metals (McKenzie & Paine, 1969). We now report full details of the crystal and molecular structure of UO₂saldien.

Experimental

UO₂saldien, prepared as described previously (Akhtar et al., 1969; Augustin, Kerrinnes & Langenbeck, 1964), formed orange-yellow prismatic crystals, many of which showed obvious signs of twinning. A single crystal of approximately $0.05 \times 0.15 \times 0.15$ mm was used for obtaining all the following data. Unit cell a =10.50 (1), b = 21.76 (2), c = 8.025 (8) Å; U = 1833.7 Å³; $\rho_o = 2.08 \text{ gcm}^{-3}$, Z=4, $\rho_c = 2.09 \text{ gcm}^{-3}$; space group $Pn2_1a$ or Pnma from absences (k+l=2n+1 for 0kl and lh=2n+1 for hk0); Pnma chosen and confirmed by subsequent successful refinement. 716 unique non-zero reflexions were measured visually from precession films hk0 to hk3 and h0l to h6l obtained with Mo Ka radiation ($\mu = 108.5 \text{ cm}^{-1}$). The usual Lorentz and polarization corrections were applied as were absorption corrections by the method of Busing & Levy (1957).

The structure was solved by normal heavy-atom Patterson and difference Fourier methods, and refined by block-diagonal and later by full-matrix least-squares calculations. All atoms except the hydrogens were located and refined with isotropic thermal vibration

Table 1. Atomic fractional coordinates $(\times 10^4)$ and isotropic thermal parameters (Å²) with e.s.ds. in parentheses

	<i>x</i> / <i>a</i>	y/b	z/c	В
U	327 (2)	2500 (0)	651 (2)	3.13 (4)
O(1)	-45 (27)	2500 (d)	2740 (41)	6.5 (8)
O(2)	643 (29)	2500 (0)	- 1348 (43)	7.4 (9)
O(3)	1831 (18)	1786 (10)	1108 (28)	6.1 (5)
N(1)	-2009(28)	2500 (0)	134 (43)	4.5 (7)
N(2)	-589(22)	1384 (12)	84 (33)	6.1 (6)
C(1)	-2550 (35)	1965 (16)	- 692 (54)	8.7 (10)
C(2)	- 2045 (28)	1376 (16)	98 (44)	6.3 (8)
C(3)	19 (26)	950 (13)	- 337 (36)	5.1 (7)
C(4)	1353 (28)	904 (15)	-417 (42)	6.0 (7)
C(5)	1884 (27)	345 (13)	-1119 (37)	5.1 (7)
C(6)	3139 (28)	253 (14)	-1263 (39)	5.6 (7)
C(7)	4039 (32)	650 (15)	- 629 (46)	6.7 (8)
C(8)	3593 (32)	1192 (15)	201 (41)	6.0 (8)
C(9)	2268 (27)	1289 (13)	264 (37)	4.6 (6)
H(1)	- 2270	2500	1345	6.5
H(2)	-2291	1974	- 1940	10.7
H(3)	- 3568	1977	- 579	10.7
H(4)	-2377	988	- 604	8.3
H(5)	-2380	1342	1357	8.3
H(6)	- 510	551	- 692	7.1
H(7)	1218	15	- 1634	7.1
H(8)	3486	-159	- 1869	7.6
H(9)	5050	555	- 707	8.7
H(10)	4259	1523	716	8·0

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Table 2. Observed and calculated structure factors

<u>ь</u> к		₽_I	٩,									3	13403	141.4	0.301
4 44	3•7	191.3	0. 366	ş	30.7 54.3	59.3	0.140		ι. . δ			10	104.5	114.3	-3.906
6 130	8.5 9.6	375+1 207-5	-3.964		1 0		8		0 C2.1	i €?•0 5 47•5	-2.834	11	80.0	86.4	0.303
10 23	3.4	\$27.5	-2.948	i.	77.9	77.9	- a. e 21 c. 26 s		a 50+1	55+4	0.384	*3	87.0	1 05 . 5	- 2.696
14 17	3•δ	174.9	0.347 -2.937	4	25.3 68.6	30.5 68.6	-2.938		3 <u>3</u> 1.2	1 33.9 59.0	-2.789	14	40.7	76.1	C.310
15 14	7•4 7•6	142.2	0.340		1 7				6 56.1 3 0	53+7	0.370	16	67+5	71.6	0.291
20 7		79.3	0.343	1	24.1	17.0			1 33.	1 27. 8	0. ; ; ;	18	60.1	69.0	-2.673
24 6	0.0	57•5	-2.705	1 4	75-5	78.0	0+345 -3-815		ن 146 °	7 150.0	0.341	19 20	59 • 1 48 • D	53.3	0.309
26 4	7.0 4.7	50.4	-3.001	0	64,1	73.6	0 • 3 4 7		1 166.4	1 164.0	-2.682	91 2.2	48.y	56.1	-2.848
30 a.	3•7 1	34.5	-2.647	4	30.1	4.E	C. 1 38		1 1 3 5 •	1 34-9	C.186	23	25.7	40.6	0.353
1 11	3. 2	120.1	-2.972	0	40.1	40.8	-2+737		164.	162.6	-2.993		1 1	3396	••••
7 13	5.3	114.6	0.164	4	36.2	42.1	-2.759		134-1	136.5	0+180	о 1	31.9	30.3	-1.059
11 9	5.6	79•7 93•0	-2-917 0-179	•	44.8	41.7	0.371		5 152.0 57.0) 155+4) 01+j	0.309 -3.772	3	37+3	33.6	0.403
13 5 15 5	5.6	61.0	-1.887	1	34.0	30.4	0.408	1	D 103.0	1 03.5 0.0	-2.844	4	46.5	47+7	-3.868
17 50	•••	47+3	-3.872	•	1 40.1	141.3	0.332	1	97.	97.1	0. 19 5	6	\$8.7	69.8	C. 180
0 430	5.3	43 4 • 7	0.077	-	\$37.1	339.3	-1.930	1	84.9	65.3	-2.833	ž	43+4	44-1	-3.868
10 11	7•9	117.5	-1.697	3 4	137.1 131.8	156.0	0.120	1	5 C9.0	71.1	0.367 9.325	9 10	61.1 45.9	58.5 45.4	-2-957
13 8/	• 7 • 3	81.0 77.1	C+333 -2+621	5	108.0	116.4	- 2.976	1	58.5	53+3	-2.643	11	47.3	52.9	0.193
16 81	.6	84.4	0. 26 3	1	55.9	58.1	0.323	19	38.8	45+4	0.313	13	33+4	36.5	-3.883
10 51	. 8	50.7	0.349	ş	315.7	203.0	0.205 -3.447	21	5 39.6 1 39.9	56.3	0.335 -2.86 t	14	35.6	34.9	0.395
1 16	3	179 • 3	-2.697	3 O 1 I	\$ 73.5	181.4 67.7	-3.925	a:	40.9	49 - 5	-2.811				
3 31ê 5 60		54.0	0.190	1.2	178.8	183.5	C.199		۰ <u>،</u> ۰	44+3	-2.686	1	82.1	86.1	-1.854
11 143	• 5	39 . 5	0.258	14	94.0	112.0	-2.840		. 04.0 : 56.5	57•3	-2.798	3	98.8	90.9	-2.916
15 113	• 3	113.7	0. 269	15	47 • 5	53.2	0.263	:	101.2	100.8	0.373	1	74.8	70.6	0.186
17 97	• 3	60.1	-2.832	17	31.8	30.1	-2.717		. 3 3 . 1	1 27 - 5	-2.933	ş	54.6	s8.3	-2.806
21 55 23 41	• 4	54-8	-3.749	19	46.0	12.1	0.363		127.4	1 2 3 - 3	0.207	2	97•1 76•7	98.6 76.7	0.307
· · · ·	4			21	34.0	20.4	-3.650	8	1 87+1 2 95+5	86.1 101.6	-3.937	9	83.3	79 - 4	-1.898
4 78		88.1	-3-110	23	34.9	50.7	- 1.783 P. 395	1	64.7	70.1	0. 337		61.7	68.1	0. 207
	5	3000	3.033	24	4 . 0 2 1	44.0	0.409		41.5	46.6	-1.807	13	71.9	68.3	- 2. 896
3 45	• 7 1	47+4	0.336	0 1	49-1	46.4	-3.973	1	5 53+3	54+3	-3.777	14	47•9 53•1	45-1 54-6	-3.809
5 134	ê 1	20.3	-3.868	2	34.0	29.3	0.371	1	5 57.5	58.3 30.3	C. 331 - 2. 706	16	35-9	40.5	0.337
• 76 2 85	• 5	79.3	-2.810	1	36.5	32.4	-2.903		50.	46.3	-2.765		3		
4 91	-9 1	07.4	-2.906	7	69.6	66.4	0.354	19	53-4	49-7	0.309	1	100.7	95+3	-1.860
	7	••••	0.307		52.0	89.4 47.4	-2.904		3 51.3 5 61.3	66.5	0.180	3	93-5	\$3.9 \$5.7	0.3c6 0.310
5 30	• 7	24.7	-2.808	11	30+7	36.1	-1.901	1	1 31.8 51.6	33•4	-2.887	4	109.4	117+4	-1.903
• 57	8 .0	54.8		15	43.4	40.5	0.313		ag.e	25.6	0.330	é	1 26 . 3	1 28 . 3	0. 21 1
71	.0	65+4	0.364		2 2	10.2	- 3.0 40		64.3	54+4	-2.993	8	93.5	93.5	-2.870
	.7	68.7	0.328		87.8 31.4	93•3 *5•5	0.317 -2.007	1) 41.4 > 19.9	43.1	-2.873	9	91 + 7 1 01 • 0	\$7.1	-3.873
1 29	9 •1	33.8	Q. 449	3	77 .8 91.1	83.7	-2.774	11	14.3	23.4	0.314	11	88.4	82.6	0.266
\$ 43 1	• 1 •	30.0	0.333	\$	104.3	\$13.7	0.350	1	29.0	33-9	- 3.9 5 2	13	55.3	59 • 2	-3.797
° 37	.8	19.7	-2.674		93.3	8	0.334	••	3 4	3/.0	- 1.907	14	48.7	53.3	0.335
° 347	.9 3	51.0	0.130	11	25.9	3 5+ 5	0.305	1	136.6	05.1	-2.681	16	50.4	54.6	-2.794
184	-3 -1	78.3	0.251	13	51.3	90.1 57.5	-1.970	1	91.6	93.5	0.346	18	53.9	40.7	0.348
\$ a36	., s	31.0	-3.946	14	32.5	67.9 23.5	-2.817		101.6	101.7	-1.910	30	39.7	44-3	-3.803
7 il 8 339	.9 . 9 3	10.0	C.507 0.187	10	47.9	40.4	0.430	ä	75-7	78.1	0.381	•	93•5	95.0	-3.907
10 175	.6 .3 1	18:3	-2.904	19	29 - 3	25.7	0.281			40.8	-1.809	1	81.0 79.4	77.0	-3.871
12 163	•5 •	62.3	0.333		3		01310	1	37+3	38.0	0.250	3	69 . 5	69.9	0. 19 5
11, 130. 16 55.	6 1 9	19:7 0 t . 4	-3.886	Ĩ	133.7	130:3	-3:8:7	3	31.6	31.5	-2.843	ş	82.4	74+1	-3.073
18 100	6 1	03.2	-2.881	3	170.3	105.1	0.234	2	27.3	\$4.3	0.363	¥	17.4	70.1	0.177
/3	•			4	154.7	70.7 103.0	-2.925 -2.912	•	3 6	57•4	0.341	0 1	36.0 45.8	49 • 3 46 • 7	-2.818
1 167	8 .	61 • 3	-2.069 -2.924	6 1 0	46.3 56.0	47 • 9 5 7 • 7	0.339	1	47.0	55•4 50•0	-1.846 0.339	2 1	57.3	54-4	0. 19 0
1 130. J 156.	9 1	40.5 59.5	0.051	11	103.4	94-4	0.343	:	40.0	\$3.0	0.305	•	51 . 4	54-4	-2.857
9 121	1 1	16.5		13	93.3		-2.833	3	42.3	43.6	-3.778	5 6	43.0	43.7	0.379
13 86.	4	6.5		15	77.4	39.5	0.315		3 7	35+7	0.307		39.6	16.2	- 2. 811
16 47.		35.1	-3.000	10	51 · 0 67 • 7	44.0	-2.593	0 1	55.0	63.0 48.1	-3.797	s ,	33.7	11.0	- 3 . 7 41
19 51.	9	75•4 50•1	-2.851 0.393	18 19	36.3	45+3 65-5	0.401	-	65.1	66.3	0.319	• 7	39.8	37.3	- 2. 745
c 103.	7 1	D6.0	0 . 1 41	21	\$6.0	51 . 7	- 2. 771	4	56.2	61.3	-2.803	2	48.3	41.8	0.375
1 19. 2 Ch	:	16.6	-0.190	•	1 4. 2	19.7	-0.137	\$ \$	45-3	41 • 2 57 • 8	0.353 0.345	3 4	44+4 43-8	40.8	-2.763
3 = 5	1	33.0	3.007	2	41.4	49.9	0.023	4	3 8 30.5	2	0.908	5	49.1	35.4	0. 429
13 51.		8.3	0.350	8	37.3	12.9 25.2	3.001 0.094		3 9	74.0		្ថ័រ			405
16 50.	4 3	8.6	-2.911	° *	5 31 • 7	37.0	- 2. 779	i	\$2.9	34.6	0-371	1	96.1	103.1	0.135 -3.811
• 49		8.c ·	-3.962	1	131.4	117.3	-2.882	2	39.1 31.2	34•5 33•4	0.372 -2.765	3	85•5 170•1	91 . B 170 . 5	-3.920
1 177• 2 55•	, 10 4 5	8.3	-3.914 0.147	3	135.9	141.0	0.311	6	39.6	-9.9	0.409	į	50.5	51.8	0.391
3 193.	8 <u>-</u> 9	1.Č	3.825	ž	104.1	1 66.3	-1.000	:	144.4	34.8	0 . 39 4	6	80.3	84.4	- 2.913
3 455.	o 17	c.6 -	- 2.906	;	6			•	4 0	40.0	- 40 7 27	7 8	66.5	132+1 72+2	0.256
o 61.	4 5	8.0	2.645	, i	41.0	40.5	C. 31 6	с 1	191.0	185.5	0.170 -2.881	9	120.0	116.9	-2.888
3 71.	1 7	3.3	0. 320	3	38.0	80.5 31 • 7	9.287	:	189.0	198.8	-3.984	11	1 09 . 8	112.3	0. 147
, 78.		·•: -	2.927	\$	74 • 5 74 • 1	70.0	-2.846		\$ 37 . 7	145.0	0. 11 3	13	86.0	84.9	-1.839
				-				9	132.0	1 29 . 9	-2.912	14 15	53+4 75+3	\$ 5 • 0 7 7 • 5	-2.871 C.101
								7	193.3	19208	0.168	16	55.2	51.8	0.163

Table 2 (cont.)

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---|---|--|---
---|--|--|
| | 00.0 | 03.3 | -3.007
 | 18 | 24.9 | ÷4.8

 | -3.833 | 12 | 75+1

 | 73.3 | -2.868 | | 10.6
 | 11.1 | |
| 10 | 40.3 | 30.0 | -3.034
 | 19 | 49 • 5 | 49 • 3

 | 0.435 | :4 | 19.4

 | \$1.3 | C. 161 | 2 | 16
 | | |
| 19 | 41 • 4 | 50.0 | 0.375
 | 20 | 24.9 | 18.1

 | 0.368 | 16 | \$7.6

 | \$1.6 | -2.8.4 | · · · |
 | 34.4 | |
| | 5 2 | |
 | 31 | 49 . 2 | 45.0

 | -2.712 | 18 |

 | 3 2 . 9 | | ~ ~ ~ | · · · ·
 | | |
| • | 130.6 | 112.3 | -2.911
 | 23 | \$4.3 | 14.6

 | -3.730 | | 7 1

 | 33.9 | | | 33.7
 | 30.3 | 779 |
| 1 | 82.3 | 84.4 | -2.957
 | 23 | 40.0 | 42.5

 | 0.178 | 0 | 11.6

 | 5.0 | 2.80.0 | | 73.9
 | 03.0 | -2.043 |
| 3 | 80.6 | 61.1 | 0.320
 | | 6 1 |

 | | - |

 | | | | 39.0
 | 30.9 | 0.200 |
| 3 | 75.6 | 79 • 4 | 0.194
 | c | \$1.2 | 61.0

 | -2-9.00 | |

 | | -2.503 | 3 | 71.5
 | 75.1 | 0.335 |
| 4 | 96.3 | 104.0 | -2.699
 | 1 | 41.8 | 44.8

 | -1.073 | ; |

 | 43+3 | 0.339 | 4 | 30.3
 | 39 • 4 | -3.901 |
| 5 | 31 . 7 | 39.1 | -2.750
 | 2 | 55.7 |

 | 0.170 | |

 | 21.03 | 0.007 | 5 | 50.0
 | 00.00 | ~ 2. 770 |
| 6 | 67.7 | 92.5 | 0.265
 | 1 | 46.8 | 45.0

 | 0.111 | 2 | 30.0

 | 34+9 | -2.030 | 9 | 2
 | | |
| 7 | 41.5 | 40.3 | 0.115
 | 1 | 46.3 | 47.1

 | | | 27.1

 | 10.9 | -3.000 | ٥ | 73+6
 | 70.6 | - 2. 863 |
| 8 | 61.0 | 81.1 | - 2.861
 | | 46.4 | 1.1

 | | 7 | 49+3

 | 30.0 | 0.203 | 3 | 55-2
 | 52.7 | 0.375 |
| ۵ | 47.3 | | To. Sal
 | | |

 | 0.334 | | 74

 | | | 4 | 56.3
 | 67.4 | -2.857 |
| 1 Ó | 67.3 | 70.5 | 0.114
 | á | 33.1 |

 | 0.301 | 6 | 55.9

 | 90.4 | -2.829 | 6 | 56.8
 | 56.9 | 0.328 |
| 11 | 11.4 | 43.4 | 0.100
 | | 40.9 | 44-3

 | -3.037 | 2 | 95+5

 | 93+1 | 0.322 | 8 | 57•3
 | 55+B | -2.823 |
| | | 2002 |
 | | 10.3 | 32.1

 | 0.300 | 3 | 29.8

 | 27 • 4 | 0.144 | 10 | 58.3
 | 52.4 | 0.319 |
| | | | -3.004
 | 11 | 30.9 | 20.9

 | 0.1 49 | 4 | 96.2

 | 93.4 | -2.827 | 13 | 42.6
 | 36.7 | - 2 . 71 1 |
| | 23.1 | 49.3 | -2.901
 | | °, |

 | | 6 | 85.0

 | 92.3 | 0.308 | 9 |
 | | |
| :: | 30.4 | 20.1 | 0.325
 | ů | 20.8 | 30.4

 | 0.334 | | 7 S

 | | | | 43.0
 | 18.4 | - 2.0 31 |
| 12 | 30.0 | 37-1 | 0.293
 | 1 | 80.0 | 84.1

 | -3.846 | 1 | 56.0

 | 50.6 | 0.341 | |
 | | |
| 10 | 37-1 | 45.1 | -3.700
 | 3 | 69.4 | 89 • 4

 | 0.274 | 1 | 42.5

 | 14.4 | -2.784 | • • • • | 68.0
 | 70.8 | - 2.811 |
| 17 | 30 • 3 | 35-4 | -2.866
 | 4 | 39.7 | 25.8

 | ••333 | 5 | 11.1

 | 11.5 | 0.356 | | 67.4
 | 66.0 | 4. 16 1 |
| 10 | 39.0 | 40.1 | 0.318
 | ş | 91.1 | 89+1

 | -2.874 | | 7 6

 | | | Ā | 67.0
 | 6 | - 2. 78 1 |
| | 5 3 | |
 | 0 | 36.6 | 37.0

 | -2.923 | • | 41.5

 | 44.0 | - 2. 799 | õ |
 | 60.7 | 0.164 |
| • | 50+3 | 03.0 | 801.0
 | 7 | 78.6 | 75+5

 | 0.304 | 1 | 27.7

 | 11.3 | 4.457 | - a |
 | •••• | ** 3*4 |
| 1 | 30.9 | 26.3 | -2.705
 | 9 | 71.1 | 68.9

 | -2.825 | Á. | 41.1

 | 48.0 | -2.8.8 | , ' | 12.8
 | | 0.118 |
| 1 | 20.1 | 29.0 | -2.909
 | 11 | 48.4 | 56.3

 | 0.365 | 6 | 44.0

 | 47.0 | 0. 116 | | ,
 | 3344 | •• ; •• |
| 3 | 48.0 | 49.6 | 0.333
 | 13 | 51.0 | 56.6

 | - 2.808 | |

 | , | | , 9 |
 | • • • | |
| 4 | 30.8 | 23.7 | 0. 29 4
 | 4 5 | 66.1 | 2.07

 | 0. 288 | |

 | | | 1 | 34+3
 | 34+4 | 0 . 444 |
| 5 | 41.8 | 45.6 | - 2.905
 | 17 | 19.4 | 43.0

 | -2.771 | : |

 | 33.0 | 0.303 | 10 |
 | | |
| ð | 19.7 | 43.0 | -2.001
 | • | 6 |

 | | 3 | 34.0

 | 24.9 | - 2. 777 | • | 30.0
 | 24.9 | -3.050 |
| 8 | 47.43 | 49.8 | 0.133
 | 0 | 110.7 | 1 14-6

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parameters to give an R value of 0.070. Allowing the uranium atom ellipsoidal vibrations resulted in no significant improvement in R and indicated negligible anisotropy. At this stage the difference Fourier map was examined for evidence of hydrogen atoms. Well defined peaks were not observed, but the density at the calculated hydrogen positions was everywhere positive. Hydrogen atoms, placed at the calculated positions, could not be refined; so the group refinement facilities of the *GENSFLS* program were used. Each hydrogen was grouped with its corresponding carbon or nitrogen, and translations of the group were refined. One thermal parameter was refined for each group; the hydrogen vibration was constrained to be $2\cdot0$ Å²

greater than the carbon or nitrogen one. Final parameter shifts were all less than 0.1 of the corresponding e.s.ds. A final difference Fourier synthesis showed a single peak of about 1.25 $e^{A^{-3}}$ close to the uranium position, but otherwise nothing greater than 0.5 $e^{A^{-3}}$. Analysis of the final $|F_o - F_c|$ showed no significant variation either with $|F_o|$ or with $\sin \theta/\lambda$. This final refinement led to an *R* value of 0.069, and to the positional and vibrational parameters listed in Table 1 and the structure factors in Table 2.

X-ray scattering factor curves were taken from International Tables for X-ray Crystallography (1962), $\Delta f'$ and $\Delta f''$ for uranium from Roof (1961), and μ_a for uranium from Roof (1959). Computing was carried out, using the Sheffield X-ray programs, on the University of Sheffield's ICL 1907 and the S.R.C. Atlas computers.

Results and discussion

The structure of the UO_2 saldien molecule is illustrated in Figs. 1 and 2; the bond lengths and angles are given in Table 3, and details of certain least-squares planes are given in Table 4. The molecules lie across symmetry planes, which contain the uranyl group and the central NH group of the ligand.

 Table 3. Principal bond lengths and angles with e.s.ds.

 in parentheses

U-O(1)	1·72 (2)Å	C(1)-N(1)-C(1')	106 (2)°
U-O(2)	1.64 (3)	U - N(1) - C(1)	117 (1)
U-O(3)	2.25(2)	N(1)-C(1)-C(2)	110 (3)
U-N(1)	2.49 (3)	C(1)-C(2)-N(2)	110 (2)
U-N(2)	2.65 (3)	C(2) - N(2) - U	112 (2)
N(1)-C(1)	1.46 (5)	C(2) - N(2) - C(3)	122 (3)
C(1) - C(2)	1.52 (5)	U - N(2) - C(3)	126 (2)
C(2) - N(2)	1.53 (4)	N(2)-C(3)-C(4)	127 (3)
N(2)-C(3)	1.19 (4)	C(3) - C(4) - C(9)	129 (3)
C(3) - C(4)	1.41 (4)	C(4) - C(9) - O(3)	116 (3)
C(4) - C(5)	1.45 (4)	C(9)-O(3)-U	135 (2)
C(4) - C(9)	1.39 (4)	O(3) - C(9) - C(8)	118 (3)
C(5) - C(6)	1.34 (4)	C(3) - C(4) - C(5)	117 (3)
C(6) - C(7)	1.38 (5)	C(9) - C(4) - C(5)	113 (3)
C(7) - C(8)	1.43 (5)	C(4) - C(5) - C(6)	123 (3)
C(8) - C(9)	1.41 (4)	C(5) - C(6) - C(7)	123 (3)
C(9) - O(3)	1.35 (4)	C(6) - C(7) - C(8)	118 (3)
N(1)-U-N(2)	67.3 (0.7)	C(7) - C(8) - C(9)	118 (3)
N(2)-U-O(3)	69.5 (0.8)	C(8) - C(9) - C(4)	125 (3)
O(3)–U–O(3')	87.6 (1.0)	O(1)-UO(2)	178.6 (1.2)

The uranium environment is a slightly distorted pentagonal bipyramid. The uranyl group, which defines the axis of the bipyramid, is, within experimental error, linear and perpendicular to the least-squares plane through the five equatorial atoms. These form a slightly puckered pentagon, but the largest deviation from D_{5h} symmetry is the rather large O(3)–U–O(3') angle. The inequality of the uranyl U–O bond lengths, though not great, is rather unusual (but see *e.g.* Nguyen Quy Dao, 1972); they each differ by 2σ from their mean of 1-68 Å, itself a perfectly normal value. The difference may arise sterically, as the uranium atom is displaced from the mean plane of the pentagon by 0-06 Å towards O(2), *i.e.* in the direction of the shorter U–O bond. The shape of the pentagonal bipyramid, idealized to exact D_{5h} symmetry, may be defined by the ratio of axial to equatorial bond lengths, observed in this structure at $r_a/r_e = 0.71$. This is distinctly larger than the 'ideal' value of 0.62 (obtained when ligand-ligand distances are equal for the equatorial and the pyramid edges), but is rather smaller than is found for most uranyl compounds (*ca.* 0.72 to 0.79). (Pentagonal bipyramids not containing an actinide MO₂ group show ratios of about 0.9 to 1.0.)

As this is the first reported structure of a saldien complex, no direct comparisons of ligand geometry can be made. However, the present complex may be usefully compared with complexes of the quadridentate ligand salen (Calligaris, Nardin & Randaccio, 1972; Bailey, Higson & McKenzie, 1972). The saldien ligand shows some signs of strain in achieving 5-coordination (such as the inequality of the U–N distances and other features noted below), but otherwise it seems closely similar to what would be expected from salen complexes, except for the unusually short C(3)-N(2) distance of 1·19 Å.

The five-membered chelate rings have the expected *gauche* conformation, but ligand strain causes them to be somewhat unsymmetrical. In this they resemble the five-membered rings in salen complexes, though the torsion angle about the C-C bond in the present compound is much higher (54.5°) than those observed for salen (mostly 30° to 47°). The six-membered chelate ring is essentially coplanar with the benzene ring, but



Fig. 1. Projection of the molecule along [001].

Table 4. Equations of least-squares planes

Equations are given in the form pX+qY+rZ=s, where X, Y, and Z are coordinates in Å referred to the cell axes and origin. The deviations of certain atoms from these planes are given in square brackets.

Plane	Defined by	р	q	r	s	
1	N(1), N(2), O(3), N(2'), O(3')	-0.2272	0.0000	0.9738	0.3709	
-	$[U \ 0.06, \ N(1) \ 0.21, \ N(2) \ -0.16, \ O(3)]$	3) 0·06]				
2	U, N(1), N(2)	-0.1656	-0.1180	0.9791	-0.1870	
•	[C(1) - 0.42, C(2) 0.27]					
3	N(2), O(3), C(3)-(9)	0.0496	0.4929	0.8687	1.3077	
	[N(2) 0.09, C(3) - 0.05, C(4) 0.02, C(4) 0.02]	C(6) 0.01, C(7)	0.04, C(8) 0.02,	C(9) 0.01,	O(3) = 0.07, U	0.941



Fig. 2. Edge view of the molecule projected along [100].

with the uranium atom lying 0.94 Å out of the plane. (This is much further than would have been predicted from any salen complexes.) The other nine atoms, N(2), O(3), and C(3) to (9), show a mean deviation of 0.05 Å from their least-squares plane, but this figure contains rather large contributions from N(2) and O(3), which are twisted from this plane by being shared with the pentagonal coordination plane of the uranium.

The large dihedral angle $(31\cdot0^{\circ})$ between the salicylideneimine plane and the coordination plane is possibly another sign of strain in this molecule, as the values for salen complexes rarely exceed 15°, except in highly strained systems such as Cosalen benzoylacetonate (Bailey *et al.*, 1972). The two salicylideneimine planes of the molecule meet at a dihedral angle of 59·1° and each is at 60·5° to the mirror plane. So these three planes intersect one another (at almost exactly 60°) in a common line, which is nearly parallel to the direction of the U–N(1) bond, as can be seen from the edge-on view of the molecule in Fig. 2. We are grateful to the Government of Pakistan for a scholarship awarded to one of us (M.N.A.).

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An X-ray and Infrared Study of Bis(isocyanurato)diamminecopper(II)

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A three-dimensional structural analysis and an infrared study of an ammine isocyanuric acid copper complex are reported. The complex, which has the formula $[Cu(C_3N_3O_3H_2)_2(NH_3)_2]$, crystallizes in space group $P\overline{1}$, with unit-cell dimensions a=7.03, b=9.05, c=6.91 Å, $\alpha=42.19^{\circ}$, $\beta=90.39^{\circ}$, $\gamma=$ 95.56°. Least-squares refinement led to a final agreement index of 10% on 837 reflexions. The complex consists of ammine groups and isocyanuric acid rings, bonded through nitrogen to copper atoms, forming square-planar arrangements cross-linked by hydrogen bonds. Some infrared bands are assigned by comparison of the spectra of $[Cu(N_3C_3O_3H_2)_2(NH_3)_2]$ with the following: $[Cu(N_3C_3O_3H_2)_2(H_2O)_2]$, the deuterated analogues, cyanuric acid and diammine isocyanurato complexes of other metals.

Introduction

Work in this laboratory (Taylor, 1972) has shown that some metal oxides and salts react directly with heated urea to form cyanurato complexes which have received little attention from chemists during the last 50 years, and have not yet been structurally defined.

Cyanurato complexes of copper were originally re-