1990). The folding angles of the fulvene ligands in the above complexes vary from 8 to 22°. A correlation with the ability to stabilize a formal charge on the metal of the $(CO)_3Fe$, (cod)Ni, (indenyl)Rh and CpCo fragments is not, however, apparent. Hence, dipolar resonance structures do not seem to contribute very significantly to the electronic structure of these compounds. There is, however, a clear distinction between the η^4-coordination mode of the fulvenes donating four π electrons in these complexes and the above mentioned η^5- or η^6-coordination found for fulvenes functioning as six π-electron donors.

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Structure of Octaaquasaccharinatoneodymium Disaccharinate Hydrate*

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Abstract. $[Nd(C_7H_4NO_3S)(H_2O)_8]^{2+}.2C_7H_4NO_3S^-.H_2O$, $M_r = 852{\cdot}9$, monoclinic, $P2_1/c$, $a = 7{\cdot}730\ (4)$, $b = 13{\cdot}408\ (9)$, $c = 30{\cdot}59\ (3)$ Å, $\beta = 94{\cdot}90\ (7)°$, $V = 3159\ (4)$ Å^3, $Z = 4$, $D_m = 1{\cdot}79$, $D_x = 1{\cdot}793\ (2)$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}93$ mm^{-1}, $F(000) = 1716$, $T = 296\ (1)$ K, final $R = 0{\cdot}0482$ for 6059 reflections. The neodymium ions adopt a tricapped trigonal prismatic coordination defined by eight water oxygen atoms and the carbonyl oxygen atom of one of the three independent saccharinate anions.

Introduction. Saccharin and its metal complexes and salts have been the subject of rather extensive structural investigations (*e.g.* Hergold-Brundić, Kamenar & Jovanovski, 1989; Cotton, Falvello, Llusar, Libby, Murillo & Schwotzer, 1986, and references therein). We decided to investigate its neodymium complex as part of our study on lanthanide complexes with N-donor ligands.

Experimental. The title compound was prepared by adding freshly precipitated neodymium hydroxide to a warm aqueous suspension of saccharin. After a few days long violet needles were formed. A specimen, 0·6 × 0·45 × 0·2 mm, was cut from a larger crystal. D_m by flotation in $C_2H_4Br_2/CHCl_3$. Oscillation and Weissenberg photographs allowed the crystal system and space group to be established. Syntex $P2_1$ diffractometer, Mo $K\alpha$ radiation for lattice parameters (15 reflections, $23 < 2\theta < 28°$), variable $\omega/2\theta$ scan, $5 < 2\theta < 57°$. Two standards every 50 reflections, mean relative e.s.d. of the control reflections 5·6%. 6147 of the 8206 intensities measured had $I \geq 3\sigma(I)$,

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* Saccharin is 1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide.

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and were merged to produce 6059 unique observed intensities. $R_{\rm int}$ after the correction was 0·046. Index range h 0→10, k 0→18, l −41→40, absorption corrections with locally modified *ABSORB* (Ugozzoli, 1987) program between 0·807 and 1·227. The structure was solved and refined with locally modified *SHELX76* (Sheldrick, 1976). Neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV), real and imaginary components of anomalous dispersion included for all non-H atoms. The Nd atoms were located from a Patterson map; the remaining non-H atoms from subsequent difference syntheses; C-bonded H atoms placed geometrically, water H atoms from difference syntheses, not all found. Final full-matrix least-squares refinement based on F [non-H atoms anisotropic, C-bonded H atoms with constrained positional parameters and a common isotropic displacement parameter $U = 0{\cdot}068\ (7)$ Å^2, O-bonded H atoms with fixed positions and a common isotropic displacement parameter $U = 0{\cdot}14\ (1)$ Å^2]. $R = 0{\cdot}0482$, $wR = 0{\cdot}0457$, maximum shift/e.s.d. ratio 0·17, $\Delta\rho$ between −1·49 and 1·65 e Å^{-3}, highest peaks around Nd atoms, $w = 1/\sigma^2(F_o)$.

Discussion. Final atomic parameters are given in Table 1,* the geometry of the coordination environment of the Nd ions is shown in Table 2 and an *ORTEP* (Johnson, 1976) view of the complex cation is given in Fig. 1. All distances are within the usually observed ranges. The Nd ion is bonded to only one of the saccharinate residues *via* its carbonyl O atom. The ligand–metal coordination pattern is similar to that found in magnesium saccharinate (Jovanovski & Kamenar, 1982). To assess the role of electrostatic interactions in such a coordination mode the charge distributions for the free ligand anions have been calculated with the CNDO/2 method (Pople & Beveridge, 1970), and the averaged results are presented in Table 1 for the relevant atoms of saccharinate (1). The H-atom charges were between 0·00 and −0·02 e. The carbonyl O atom is the most negatively charged atom in the ligand and is perhaps therefore the atom most likely to be coordinated to the metal cation. The coordination polyhedron of Nd may be best described as a tricapped trigonal prism with $\Delta = 0{\cdot}013$ Å^2 ($\Delta = \sum d_i^2/9$; d_i is the distance between the real position of the ith atom, and the relevant vertex in the ideal, least-squares-fitted polyhedron; Drew, 1977), with O12, OW2, OW3, and OW5, OW6, OW7 as the

Table 1. *Final atomic coordinates, equivalent isotropic displacement parameters* (Å^2) *and the saccharinate anion electrostatic charges q* (e)

$$U_{\rm eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$	q
Nd	0·20291 (4)	0·10612 (2)	0·19808 (1)	0·0302 (1)	
S1	−0·2946 (2)	0·3119 (1)	0·08220 (5)	0·0378 (5)	+0·32
S2	−0·3591 (2)	−0·0500 (1)	0·08921 (5)	0·0398 (5)	
S3	0·2549 (2)	0·6933 (1)	0·16527 (6)	0·0412 (5)	
O11	−0·4291 (6)	0·4075 (3)	0·1857 (2)	0·053 (2)	−0·46
O21	−0·1352 (6)	0·2538 (3)	0·0840 (2)	0·052 (2)	−0·36
O31	−0·4197 (6)	0·2898 (3)	0·0457 (2)	0·054 (2)	−0·36
O12	0·0766 (5)	0·0291 (3)	0·1312 (2)	0·047 (2)	
O22	−0·4331 (6)	−0·1354 (4)	0·1097 (2)	0·065 (2)	
O32	−0·4848 (5)	0·0211 (4)	0·0705 (2)	0·058 (2)	
O13	0·0963 (6)	0·4439 (3)	0·1873 (2)	0·047 (2)	
O23	0·4226 (5)	0·7211 (3)	0·1861 (2)	0·051 (2)	
O33	0·1381 (6)	0·7750 (3)	0·1559 (2)	0·059 (2)	
OW1	0·3522 (6)	−0·0537 (3)	0·1824 (2)	0·057 (2)	
OW2	0·0753 (7)	0·2501 (3)	0·1611 (2)	0·062 (2)	
OW3	0·4086 (6)	0·1404 (3)	0·1434 (2)	0·057 (2)	
OW4	0·3852 (7)	0·2585 (4)	0·2200 (2)	0·066 (2)	
OW5	0·1101 (7)	0·1867 (4)	0·2638 (2)	0·065 (2)	
OW6	0·1073 (9)	−0·0386 (4)	0·2437 (2)	0·091 (3)	
OW7	0·4500 (8)	0·0652 (4)	0·2531 (2)	0·082 (2)	
OW8	−0·1307 (8)	0·0967 (6)	0·2009 (2)	0·113 (3)	
OW9	0·7318 (7)	0·8566 (4)	0·2019 (2)	0·071 (2)	
N1	−0·3800 (6)	0·3062 (4)	0·1283 (2)	0·040 (2)	−0·36
N2	−0·2170 (6)	0·0024 (4)	0·1233 (2)	0·045 (2)	
N3	0·1658 (6)	0·6080 (4)	0·1933 (2)	0·042 (2)	
C11	−0·3734 (7)	0·3925 (5)	0·1489 (2)	0·038 (2)	+0·35
C21	−0·2944 (7)	0·4752 (4)	0·1242 (2)	0·034 (2)	−0·02
C31	−0·2720 (8)	0·5738 (5)	0·1346 (2)	0·046 (2)	−0·01
C41	−0·1955 (9)	0·6367 (5)	0·1058 (3)	0·053 (2)	−0·01
C51	−0·1462 (9)	0·6009 (6)	0·0661 (3)	0·056 (3)	−0·02
C61	−0·1666 (8)	0·4996 (5)	0·0551 (2)	0·044 (2)	−0·00
C71	−0·2423 (7)	0·4402 (4)	0·0850 (2)	0·035 (2)	−0·02
C12	−0·0568 (7)	−0·0049 (4)	0·1099 (2)	0·037 (2)	
C22	−0·0514 (7)	−0·0607 (4)	0·0678 (2)	0·033 (2)	
C32	0·0924 (8)	−0·0860 (5)	0·0457 (2)	0·045 (2)	
C42	0·0647 (9)	−0·1372 (6)	0·0073 (2)	0·052 (2)	
C52	−0·0990 (10)	−0·1644 (5)	−0·0091 (2)	0·052 (2)	
C62	−0·2443 (9)	−0·1403 (5)	0·0125 (2)	0·046 (2)	
C72	−0·2124 (7)	−0·0896 (4)	0·0510 (2)	0·036 (2)	
C13	0·1602 (7)	0·5207 (4)	0·1718 (2)	0·034 (2)	
C23	0·2305 (7)	0·5246 (4)	0·1285 (2)	0·036 (2)	
C33	0·2415 (8)	0·4506 (5)	0·0972 (2)	0·041 (2)	
C43	0·3067 (8)	0·4744 (6)	0·0580 (2)	0·049 (2)	
C53	0·3606 (9)	0·5710 (6)	0·0491 (2)	0·053 (3)	
C63	0·3454 (8)	0·6447 (6)	0·0792 (2)	0·052 (2)	
C73	0·2815 (7)	0·6203 (5)	0·1191 (2)	0·039 (2)	

Table 2. *Distances* (Å) *and angles* (°) *for the* Nd *coordination sphere*

Nd—O12	2·422 (3)	Nd—OW1	2·500 (4)
Nd—OW2	2·407 (5)	Nd—OW3	2·448 (5)
Nd—OW4	2·539 (5)	Nd—OW5	2·443 (6)
Nd—OW6	2·536 (6)	Nd—OW7	2·496 (6)
Nd—OW8	2·591 (6)		
OW1—Nd—O12	68·4 (2)		
OW2—Nd—O12	79·9 (2)	OW2—Nd—OW1	140·8 (2)
OW3—Nd—O12	75·0 (2)	OW3—Nd—OW1	72·1 (2)
OW3—Nd—OW2	78·0 (2)		
OW4—Nd—O12	137·8 (2)	OW4—Nd—OW1	119·1 (2)
OW4—Nd—OW2	71·0 (2)	OW4—Nd—OW3	69·6 (2)
OW5—Nd—O12	139·3 (2)	OW5—Nd—OW1	135·2 (2)
OW5—Nd—OW2	84·0 (2)	OW5—Nd—OW3	137·1 (2)
OW5—Nd—OW4	67·8 (2)		
OW6—Nd—O12	91·3 (2)	OW6—Nd—OW1	67·1 (2)
OW6—Nd—OW2	138·2 (2)	OW6—Nd—OW3	139·2 (2)
OW6—Nd—OW4	130·7 (2)	OW6—Nd—OW5	76·5 (2)
OW7—Nd—O12	134·9 (2)	OW7—Nd—OW1	66·5 (2)
OW7—Nd—OW2	138·8 (2)	OW7—Nd—OW3	89·9 (2)
OW7—Nd—OW4	67·9 (2)	OW7—Nd—OW5	78·7 (2)
OW7—Nd—OW6	72·7 (2)		
OW8—Nd—O12	71·1 (2)	OW8—Nd—OW1	116·2 (2)
OW8—Nd—OW2	71·7 (2)	OW8—Nd—OW3	137·6 (2)
OW8—Nd—OW4	124·2 (2)	OW8—Nd—OW5	68·4 (2)
OW8—Nd—OW6	66·9 (3)	OW8—Nd—OW7	132·3 (2)

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54127 (41 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

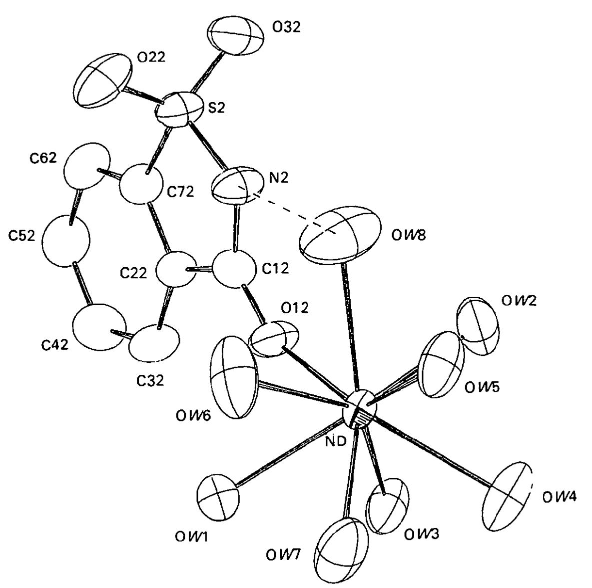

Fig. 1. Complex cation. The intramolecular hydrogen bond is marked with a dashed line. The numbering scheme for saccharinate residues 1 and 3 follows that shown here for residue 2.

vertices of the bases, and O*W*1, O*W*4 and O*W*8 as the caps. The structure is held together by a network of intermolecular hydrogen bonds, and electrostatic and van der Waals forces. The crystal packing is shown in Fig. 2.

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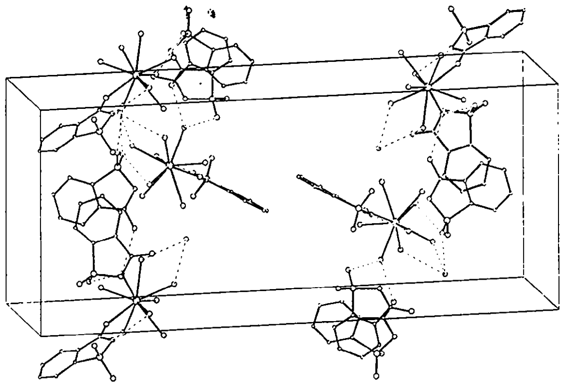

Fig. 2. View of the crystal structure. **a** is into the plane of the paper, **b** is vertical and **c** is horizontal. The dashed lines represent the hydrogen bonds.

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Structure of Bis(dipicolinato)dioxouranium Monopicolinic Acid Hexahydrate

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Abstract. Bis[2,6-pyridinedicarboxylato(1 −)]dioxouranium–2-pyridinecarboxylic acid (1/1) hexahydrate, $[U(C_7H_4NO_4)_2O_2].C_6H_5NO_2.6H_2O$, $M_r = 833{\cdot}45$, triclinic, $P\bar{1}$, $a = 14{\cdot}195\ (6)$, $b = 15{\cdot}208\ (8)$, $c = 6{\cdot}801\ (3)$ Å, $\alpha = 91{\cdot}63\ (3)$, $\beta = 93{\cdot}80\ (3)$, $\gamma = 117{\cdot}19\ (4)°$, $V = 1300{\cdot}2$ Å^3, $Z = 2$, $D_x = 2{\cdot}13$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 19{\cdot}82$ mm^{-1}, $F(000) = 804$, $T = 290$ K, $R = 0{\cdot}036$,