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Octahedral Nickel Dioxime Iodides: Bis(oxamide oxime)(oxamide oximium)nickel(II) Triiodide Hydrate and *cis*-Diiodobis(oxamide oxime)nickel(II)

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Abstract. Room temperature, λ(Mo $K\alpha$) = 0·7107 Å. $[Ni(C_2H_7N_4O_2)(C_2H_6N_4O_2)_2]I_3.H_2O$: $M_r = 812·73$, trigonal, $R\bar{3}c$, $a = 16·218$ (3), $c = 14·562$ (3) Å, $V = 3317$ Å^3, $Z = 6$, $D_x = 2·44$ g cm^{-3}, $\mu = 48$ cm^{-1}, $F(000) = 2304$, final $R = 0·045$ for 860 observed independent reflections. In the tripositive complex cation Ni^{II}, site symmetry 32, is octahedrally coordinated by three oxamide oxime chelate ligands [Ni–N, 2·055 (5) Å], one of which is protonated to a monopositive species. $[NiI_2(C_2H_6N_4O_2)_2]$: $M_r = 548·71$, monoclinic, $P2_1/a$, $a = 13·444$ (2), $b = 7·830$ (2), $c = 15·222$ (3) Å, $\beta = 115·42$ (1)°, $V = 1447·2$ Å^3, $Z = 4$, $D_x = 2·52$ g cm^{-3}, $\mu = 55·9$ cm^{-1}, $F(000) = 1032$, final $R = 0·032$ for 3037 observed independent reflections. The crystals consist of neutral octahedral *cis*-diiodobis(oxamide oxime)nickel(II) complexes, Ni–N, 2·034 (4)–2·061 (4) Å, Ni–I, 2·806 (1), 2·851 (1) Å.

Introduction. Iodine oxidation of stacked square-planar metal complexes, especially complexes of the Ni triad with dioximato or tetraazamacrocyclic ligands, may lead to solids with enhanced electrical conductivity and with interesting structural properties (Endres, Keller, Mégnamisi-Bélombé, Moroni, Pritzkow, Weiss & Comès, 1976; Cowie, Gleizes, Grynkewich, Kalina, McClure, Scaringe, Teitelbaum, Ruby, Ibers, Kannewurf & Marks, 1979; Hunziker, Hilti & Rihs, 1981; Hoffman & Ibers, 1983). Having synthesized new molecular conductors with oxamide oxime ['oaoH$_2$', HON=C(NH$_2$)–C(NH$_2$)=NOH] metal complex cations and organic radical anions (Endres, 1982, 1984*a*; Endres, Bongart, Nöthe, Hennig, Schweitzer, Schäfer, Helberg & Flandrois, 1985), we also tried to prepare partially oxidized oxamide oxime metal complexes by iodine oxidation. As in the case of Pt, where the unoxidized salt [Pt(oaoH)(oaoH$_2$)]I.2H$_2$O was obtained (Endres, 1985), no oxidation took place with the Ni complex. The two title species were formed instead.

Experimental. The two compounds were obtained together in varying ratios. Mainly [Ni(oaoH$_3$)(oaoH$_2$)$_2$]I$_3$.H$_2$O obtained on dissolving [Ni(oaoH$_2$)$_3$]Cl$_2$ (Tschugaeff & Surenjanz, 1907; Endres & Jannack, 1980) in H$_2$O, adding NaI and I$_2$ and stirring at room temperature. Filtered, green filtrate allowed to evaporate, turning blue after some days. Dark-violet rhombohedrally shaped crystals of the triiodide formed together with small amount of green {most likely [NiI$_2$(oaoH$_2$)$_2$]} and blue (starting complex) material. Triiodide also forms on dissolving [Ni(oaoH)$_2$] in

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aqueous HI and letting the solution evaporate. Larger yields of emerald-green $[NiI_2(oaoH_2)_2]$ are obtained when basic nickel carbonate is dissolved in hot aqueous HI, aqueous $oaoH_2$ solution is added, the mixture filtered, and the dark-green filtrate evaporated.

Details of X-ray investigations are in Table 1. Heavy-atom positions from Patterson maps, light atoms from Fourier maps, H positions from difference Fourier maps. Disordered H sites in the triiodide not considered. Refinement with anisotropic temperature factors for non-H atoms, individual isotropic thermal parameters for H, based on *F*. Full-matrix least squares for triiodide, *STRUCSY* (1984) program system on an Eclipse computer. $[NiI_2(oaoH_2)_2]$ refined with cascade-matrix least squares, *SHELXTL* program (Sheldrick, 1983) on a Nova 3 computer. Scattering factors including anomalous dispersion from *International Tables for X-ray Crystallography* (1974). Plots on a Tektronix plotter using *SHELXTL*.

Discussion. Atom coordinates are listed in Tables 2 and 3.* Views of the complex cation in the triiodide and of the $[NiI_2(oaoH_2)_2]$ molecule, together with the numbering schemes, are given in Figs. 1 and 2, bond lengths and angles in Tables 4 and 5. The Ni atom in $[Ni(oaoH_3)(oaoH_2)_2]I_3.H_2O$ lies on a site with crystallographic symmetry 32. The asymmetric part of the complex cation consists of $\frac{1}{6}$ Ni and one half of a chelate ligand. At first the composition of the compound suggested the presence of Ni^{III}, but this oxidation state seemed unlikely. Like the dichloride $[Ni(oaoH_2)_3]Cl_2$ (Tschugaeff & Surenjanz, 1907; Endres & Jannack, 1980), the triiodide yields $[Ni(oaoH)_2]$ when treated with base; free iodine cannot be detected when this neutral Ni^{II} complex is precipitated. Static magnetic measurements of the triiodide salt† yielded a room-temperature effective magnetic moment of 3·57 (3) μ_B ($1\mu_B \equiv 9{\cdot}274 \times 10^{-24}$ JT^{-1}), typical for octahedral Ni^{II} with strong orbital contribution. Hence, to account for the third positive charge, one of the ligand molecules has to be protonated to form a monopositive cation (an alternative explanation, that the crystal 'water' might be H_3O^+, seems improbable). That oxamide oxime can actually be obtained as a protonated species has been observed in two squaric acid salts (Endres & Schendzielorz, 1984). In these salts the protons are attached to the oxime N atoms, impossible in the Ni complex cation. Earlier publications discuss the protonation of the amino groups (Wenger, Monnier &

Table 1. *Experimental details*

	$[Ni(oaoH_3)$- $(oaoH_2)_2]I_3.H_2O$	$[NiI_2(oaoH_2)_2]$
Crystal shape	Rhombohedral	Irregular prism
Crystal dimensions (mm)	0·23 × 0·15 × 0·07	0·15 × 0·08 × 0·04
Diffractometer	Siemens–Stoe AED2	Syntex *R*3
Reflections for lattice constants	50	25
Scan mode	θ–2θ	θ–2θ
$2\theta_{max}$(°)	60	60
Range of *hkl*	−22,0,0 to 19,22,20	−18,0,0 to 16,11,21
Check reflections	2	2
Measured at intervals	3h	200 reflections
Intensity variation	11% drop	±8·5%
Reflections measured	3223	4221
Observed unique reflections $[I > 2{\cdot}5\sigma(I)]$	860	3037
R_{int}	0·045	0·018
Absorption corrections:		
Reflections for ψ scans	9	9
Min. transmission (max. = 1)	0·64	0·67
Refinement:		
Number of parameters	58	220
Weighting scheme	Unit weights	$w = 1/\sigma^2(F)$
wR, *R*, *S*	0·045, 0·045, 7·34	0·030, 0·032, 1·63
$(\Delta/\sigma)_{max}$	1·12	0·24
$\Delta\rho$ max. (min.) (e Å^{-3})	+2·14 (−0·74) close to I	+0·82(−0·92)

Table 2. *Atom coordinates* (×10⁴) *and equivalent isotropic temperature factors* (Å² × 10³) *of* $[Ni(oaoH_3)(oaoH_2)_2]I_3.H_2O$

$$U_{eq} = (U_{11} + U_{22} + U_{33} + 2U_{12}\cos\gamma + 2U_{13}\cos\beta + 2U_{23}\cos\alpha)/3.$$

	x	*y*	*z*	U_{eq}
I	4506 (1)	4506 (1)	2500	29·5 (3)
Ni	0	0	2500	16 (1)
N(1)	1242 (4)	755 (4)	1769 (3)	19 (2)
N(2)	2640 (5)	2210 (5)	1623 (5)	30 (3)
O(1)	1449 (4)	571 (4)	873 (4)	25 (3)
C	1854 (4)	1603 (4)	2045 (4)	19 (3)
O(2)	0	0	0	4 (3)

Table 3. *Atom coordinates* (×10⁴; I ×10⁵) *and equivalent isotropic temperature factors* (Å² ×10³) *of* $[NiI_2(oaoH_2)_2]$

U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
I(1)	34206 (3)	14788 (4)	34152 (2)	29 (1)
I(2)	47993 (3)	50688 (5)	22328 (3)	33 (1)
Ni	2799 (1)	4604 (1)	2426 (1)	23 (1)
N(1)	3453 (3)	6017 (5)	3691 (3)	28 (2)
N(2)	1562 (3)	4687 (5)	2873 (3)	26 (2)
N(3)	1978 (4)	3458 (5)	1096 (3)	31 (2)
N(4)	2151 (4)	6596 (5)	1496 (3)	30 (2)
N(5)	3308 (5)	6653 (8)	5116 (4)	48 (2)
N(6)	1026 (4)	5778 (6)	4036 (3)	37 (2)
N(7)	987 (5)	3902 (7)	−564 (4)	48 (2)
N(8)	1093 (7)	7446 (8)	−99 (5)	60 (3)
O(1)	4529 (3)	6563 (6)	4212 (3)	42 (2)
O(2)	453 (3)	4191 (6)	2373 (3)	36 (2)
O(3)	1886 (4)	1721 (5)	841 (3)	48 (2)
O(4)	2214 (4)	8347 (5)	1698 (3)	51 (2)
C(1)	2902 (4)	6091 (6)	4199 (3)	26 (2)
C(2)	1744 (4)	5493 (6)	3670 (3)	24 (2)
C(3)	1509 (4)	4428 (6)	350 (4)	28 (2)
C(4)	1587 (4)	6280 (7)	584 (4)	30 (2)

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42285 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Kapétanidis, 1957; Pearse & Pflaum, 1959), but experience with the structural chemistry of oaoH$_2$ complexes suggests that protonation of an oxime O atom is more likely. As the symmetry of the cation implies disorder of this additional proton over six sites, it cannot be detected in the X-ray structure analysis. The disordered H atoms of the water molecule, which lies on a site of symmetry $\bar{3}$, could also not be found. An impression of the packing is given by Fig. 3.

In the neutral [NiI$_2$(oaoH$_2$)$_2$] the I atoms are arranged *cis* to each other. *cis*-Addition of acids H*X* to [Ni(oaoH)$_2$] to give [Ni*X*$_2$(oaoH$_2$)$_2$], *e.g.* [NiCl$_2$(oaoH$_2$)$_2$], is a general reaction (Endres, 1984*b*), whereas *trans*-octahedral complexes have never been found. Thus, contrary to other dioximato nickel complexes (Endres *et al.*, 1976; Cowie *et al.*, 1979), no partial oxidation to a conducting molecular solid took place in the above cases.

Table 4. *Bond lengths* (Å) *and angles* (°) *in* [Ni(oaoH$_3$)(oaoH$_2$)$_2$]I$_3$.H$_2$O

Ni–N(1)	2·055 (5)	N(2)–C	1·310 (9)
N(1)–O(1)	1·416 (7)	C–C′	1·502 (8)
N(1)–C	1·293 (8)		

N(1)–Ni–N(1′)	76·0 (2)	N(1)–C–N(2)	126·5 (9)
Ni–N(1)–O(1)	128·1 (4)	N(1)–C–C′	113·3 (6)
Ni–N(1)–C	118·4 (4)	N(2)–C–C′	120·2 (7)
O(1)–N(1)–C	112·0 (6)		

(′) denotes equivalent position *y*, *x*, $\frac{1}{2} - z$.

Table 5. *Bond lengths* (Å) *and angles* (°) *in* [NiI$_2$(oaoH$_2$)$_2$]

I(1)–Ni	2·806 (1)	I(2)–Ni	2·851 (1)
Ni–N(1)	2·061 (4)	Ni–N(2)	2·050 (5)
Ni–N(3)	2·051 (4)	Ni–N(4)	2·034 (4)
N(1)–O(1)	1·385 (6)	N(1)–C(1)	1·282 (8)
N(2)–O(2)	1·407 (5)	N(2)–C(2)	1·294 (7)
N(3)–O(3)	1·405 (5)	N(3)–C(3)	1·284 (6)
N(4)–O(4)	1·400 (5)	N(4)–C(4)	1·290 (6)
N(5)–C(1)	1·335 (7)	N(6)–C(2)	1·325 (8)
N(7)–C(3)	1·327 (7)	N(8)–C(4)	1·327 (8)
C(1)–C(2)	1·489 (7)	C(3)–C(4)	1·486 (7)

I(1)–Ni–I(2)	95·2 (1)	I(1)–Ni–N(1)	93·3 (1)
I(2)–Ni–N(1)	89·1 (1)	I(1)–Ni–N(2)	87·0 (1)
I(2)–Ni–N(2)	164·9 (1)	N(1)–Ni–N(2)	75·8 (2)
I(1)–Ni–N(3)	93·3 (1)	I(2)–Ni–N(3)	94·8 (2)
N(1)–Ni–N(3)	171·9 (2)	N(2)–Ni–N(3)	99·9 (2)
I(1)–Ni–N(4)	169·2 (1)	I(2)–Ni–N(4)	88·4 (2)
N(1)–Ni–N(4)	97·0 (2)	N(2)–Ni–N(4)	92·3 (2)
N(3)–Ni–N(4)	76·1 (2)	Ni–N(1)–O(1)	128·5 (4)
Ni–N(1)–C(1)	117·6 (3)	O(1)–N(1)–C(1)	111·9 (4)
Ni–N(2)–O(2)	129·4 (3)	Ni–N(2)–C(2)	118·2 (3)
O(2)–N(2)–C(2)	111·8 (5)	Ni–N(3)–O(3)	129·9 (3)
Ni–N(3)–C(3)	117·8 (3)	O(3)–N(3)–C(3)	112·2 (4)
Ni–N(4)–O(4)	129·2 (3)	Ni–N(4)–C(4)	118·7 (3)
O(4)–N(4)–C(4)	112·1 (4)	N(1)–C(1)–N(5)	124·2 (5)
N(1)–C(1)–C(2)	113·6 (4)	N(5)–C(1)–C(2)	122·2 (6)
N(2)–C(2)–N(6)	126·8 (4)	N(2)–C(2)–C(1)	113·2 (5)
N(6)–C(2)–C(1)	120·0 (4)	N(3)–C(3)–N(7)	125·6 (5)
N(3)–C(3)–C(4)	114·1 (4)	N(7)–C(3)–C(4)	120·3 (5)
N(4)–C(4)–N(8)	125·2 (5)	N(4)–C(4)–C(3)	113·2 (4)
N(8)--C(4)–C(3)	121·6 (5)		

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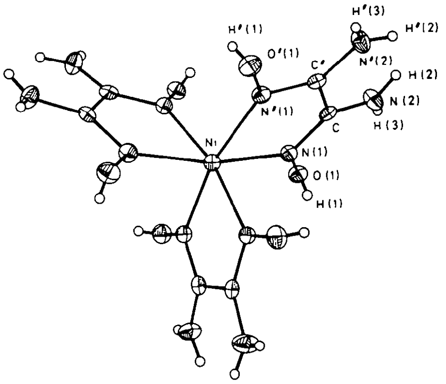

Fig. 1. The complex cation in [Ni(oaoH$_3$)(oaoH$_2$)$_2$]I$_3$.H$_2$O. Thermal contours at 30% probability, H atoms as spheres with arbitrary radius.

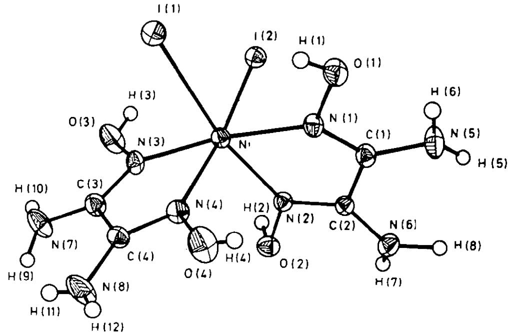

Fig. 2. The [NiI$_2$(oaoH$_2$)$_2$] molecule, analogous to Fig. 1.

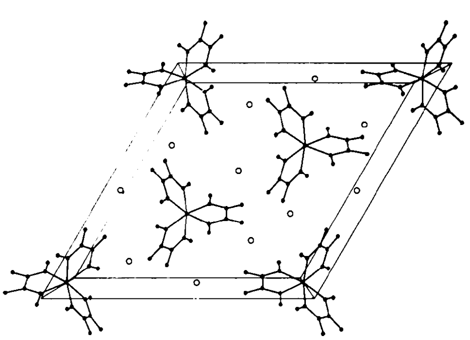

Fig. 3. Projection of the structure of [Ni(oaoH$_3$)(oaoH$_2$)$_2$]I$_3$.H$_2$O along the trigonal *c* axis. Open circles are iodide ions. The water O(2) atom is exactly below Ni and not visible.

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Structure of 1,1,2-Tricarbonyl-μ-(dimethyl 2-butynedioate-μ-C^2;μ-C^3)-(2-3-η-dimethyl 2-butynedioate)-μ-(1–5:1′–5′-η-fulvalene)-dimolybdenum(*Mo*–*Mo*), $[Mo_2(C_{10}H_8)(C_6H_6O_4)_2(CO)_3]$

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Abstract. $M_r = 687·9$, monoclinic, $P2_1/c$, $a = 9·6984\ (6)$, $b = 15·4379\ (21)$, $c = 16·4863\ (17)$ Å, $\beta = 95·880\ (7)°$, $V = 2455·4\ (8)$ Å^3, $Z = 4$, $D_x = 1·86$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 10·6$ cm^{-1}, $F(000) = 1368$, $T = 298$ K, final $R = 0·022$, $wR = 0·029$ for 2692 $[I > 3\sigma(I)]$ unique reflections. Important features include the lack of multiple alkyne coupling, a contracted Mo—Mo distance of 2·906 (1) Å, a bridging (4e$^-$-donating) alkyne C—C distance of 1·374 (4) Å and a singly coordinating (2e$^-$-donating) alkyne C—C distance of 1·265 (4) Å. The bridging alkyne is slightly displaced towards the singly coordinating alkyne ligand, in the more sterically hindered direction.

Introduction. The unusual structural constraints imposed by a fulvalene ligand anchored to a molybdenum dinuclear center should result in interesting structural and chemical properties relative to analogous η^5-C_5H_5Mo dimers. The multiple alkyne coupling in η^5-C_5H_5Mo dimers (Beck, Knox, Stansfield, Stone, Winter & Woodward, 1982; Green, Norman & Orpen, 1981; Knox, Stansfield, Stone, Winter & Woodward, 1978) is nearly always present with rare exceptions (Cotton, Schwortzer & Shamshoum, 1983; Davidson, 1983; Boag, Green, Howard, Spencer, Stansfield, Thomas, Stone & Woodward, 1980) and is especially noteworthy. The study of this class of compounds by Drage and co-workers led to the discovery of novel modes of ligand binding, most notably with respect to alkyne coupling.

Synthetic techniques used are described elsewhere (Drage, Tilset, Voldhardt & Weidman, 1985).

Experimental. Long, thin red-orange tabular single crystal (0·17 × 0·40 × 0·05 mm idealized dimensions), mounted with epoxy glue on a glass fiber. Cell parameters and orientation matrix by least-squares refinement using 24 reflections, $28 \leq 2\theta \leq 33°$, measured on an Enraf–Nonius CAD-4 diffractometer (controlled by a DEC PDP 8/a with an RK05 disk), with graphite-monochromatized Mo $K\alpha$ radiation. Total of 3494 reflections measured using θ–2θ scans for 2θ from 3 to 45° (h −10 to 10, k 0 to 15, l 0 to 15); scan range $(0·6 + 0·35\tan\theta)°$, scan speeds min. 0·9, max. 6·6° min^{-1}; corrections for Lorentz–polarization and absorption effects (max. transmission 94%, min. 66%, average 91%); intensities of three standard

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