We thank NSERC Canada for an Operating Grant (to GF), and the Government of Pakistan for a scholarship (to AK).

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Structure of Tetraaquatrinitratoneodymium–4,4′-Bipyridyl–Water (1/2/1)

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(*Received* 20 *November* 1986; *accepted* 29 *June* 1987)

Abstract. $[Nd(NO_3)_3(H_2O)_4].2C_{10}H_8N_2.H_2O$, $M_r = 732{\cdot}71$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}137\,(7)$, $b = 15{\cdot}954\,(14)$, $c = 24{\cdot}688\,(27)$ Å, $U = 2811{\cdot}1$ Å^3, $Z = 4$, $D_m = 1{\cdot}70$, $D_x = 1{\cdot}73$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 19{\cdot}40$ cm^{-1}, $F(000) = 1468$, room temperature, final $R = 0{\cdot}044$ for 2146 $[I > 4\sigma(I)]$ observed reflections. The asymmetric unit contains one molecule of tetraaquatrinitratoneodymium(III), two molecules of 4,4-bipyridyl and one molecule of water. These latter three solvent molecules remain unconnected to the metal complex but all molecules form a three-dimensional network of hydrogen bonds. In the tetraaquatrinitratoneodymium(III) complex, the metal atom is ten coordinate being bonded to three bidentate nitrate ions and four water molecules. The geometry is of a standard type for molecules of the type *M*(bidentate)$_3$(monodentate)$_4$; that is a 4*A*,6*B*-expanded dodecahedron. Nd—O bond lengths to the nitrate ligands are consistently longer [2·558 (8)–2·716 (9) Å] than those to the water molecules [2·419 (8)–2·462 (8) Å].

Introduction. Numerous compounds of the type $Ln(NO_3)_3(4\text{-bipy})_x(H_2O)_n$ (4-bipy = 4,4′-bipyridyl) have been reported (Czakis-Sulikowska & Radwanska-Doczckalska, 1975); seven classes of adducts have so far been characterized and are listed in Table 1. At least one crystal structure in each category has been determined and all of these compounds have been found to be isomorphous with at least one or more lanthanide element.

In five of the seven categories, 4-bipy remains uncoordinated, the metal-coordination sphere being solely made up of nitrate ions and water molecules with

Table 1. *Summary of crystal structures of type* $Ln(NO_3)_y(H_2O)_x$ *with* (4-*bipy*)

Structure type	Compound	Space group	Coordination number	Cell dimensions (Å; °)	Reference
(1)	$[La_2(NO_3)_6(H_2O)_7].4(4\text{-bipy})$	*Cmc*2$_1$	10,11	24·52, 16·67, 13·25	(*a*)
(2)	$[Ho(NO_3)_3(H_2O)_3].2(4\text{-bipy})$	*Pbca*	9	7·73, 43·77, 15·67	(*a*)
(3)	$[Y(NO_3)_3(H_2O)_3].2(4\text{-bipy})$	*P*2$_1$/*c*	9	7·91, 20·81, 16·56	(*a*)
(4)	$[4\text{-bipyH}]^+.[Ce(NO_3)_4(H_2O)_2(4\text{-bipy})]$	*P*2$_1$2$_1$2$_1$	11	7·31, 18·59, 19·95	(*d*)
(4)	$[4\text{-bipyH}]^+.[Nd(NO_3)_4(H_2O)_2(4\text{-bipy})]$	*P*2$_1$2$_1$2$_1$	11	7·21, 18·51, 19·93	(*b*)
(5)	$2[4\text{-bipyH}]^+.[Nd_2(NO_3)_8(H_2O)_4(4\text{-bipy})]^{2-}.3(4\text{-bipy})$	*P*2$_1$/*c*	10	18·72, 10·72, 18·03; 94·4	(*c*)
(6)	$[Ho(NO_3)_3(H_2O)_3].2(4\text{-bipy})$	*P*2$_1$/*c*	9	15·83, 21·44, 15·70; 100·4	(*c*)
(7)	$[Y(NO_3)_3(H_2O)_4].2(4\text{-bipy})$	$P\bar{1}$	10	15·18, 12·75, 8·16; 79·7, 78·5, 95·1	(*b*)
(8)	$[Nd(NO_3)_3(H_2O)_4].2(4\text{-bipy}).H_2O$	*P*2$_1$2$_1$2$_1$	10	7·14, 15·95, 24·69	(*e*)

References: (*a*) Weakley (1982), (*b*) Al-Rasoul & Weakley (1982), (*c*) Weakley (1984), (*d*) Bukowska-Strzyzewska & Tasik (1978), (*e*) this work.

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coordination number varying from 9 to 11. However, in two of the examples, 4-bipy is bonded to the metal. In type 4, 4-bipy acts as a monodentate ligand, and in type 5 it acts as a centrosymmetric bridge between two metal atoms.

Previous experimental work shows that the nature of the product obtained in the reaction of the nitrate with 4,4′-bipyridyl is sensitive to the presence of traces of acid and to the solvent ($EtOH/H_2O$) composition.

We have now prepared an eighth type of complex of formula $[Nd(NO_3)_3(H_2O)_4].2(4\text{-bipy}).H_2O$ (1) and determined its crystal structure. This complex is probably isomorphous with a praseodymium complex of the same formula (Weakley, 1982) (though the number of water molecules is said to be 3–4) for which similar cell dimensions though a different orthorhombic space group were reported.

Experimental. Well formed crystals of (1) were produced by mixing a hot solution of neodymium nitrate hydrate (prepared from Nd_2O_3 dissolved in nitric acid) with hot 4-bipy in 75% ethanol; the mixture was heated up to boiling for 20 min and crystals separated on cooling.

Density measured by flotation in CCl_4/CH_2Cl_2, preliminary cell constants established from precession photographs. A crystal of approximate size $0{\cdot}30 \times 0{\cdot}20 \times 0{\cdot}20$ mm was mounted to rotate around the *a* axis on a Stoe Stadi-2 diffractometer and data collected *via* variable-width ω scans. Background counts were 20 s and the scan rate of $0{\cdot}033°\ s^{-1}$ was applied to a width of $(1{\cdot}5 + \sin\mu/\tan\theta)$. Standard reflections $h22$ were measured every 20 reflections for each layer: no significant variation in intensity. 2946 independent reflections were measured with $2\theta < 50°$ ($0 \leq h \leq 8$, $0 \leq k \leq 18$, $0 \leq l \leq 29$); 2146 with $I > 4\sigma(I)$ were used in subsequent refinement. An empirical absorption correction was applied (Walker & Stuart, 1983), transmission factors 0·74–1·33. The structure was determined by the heavy-atom method. The metal atom and the O, N, C atoms were refined with anisotropic thermal parameters. The H atoms in the solvent 4-bipy were placed in trigonal positions and refined with a common thermal parameter in each ligand. The water H atoms were located in a difference Fourier map and their coordinates fixed. Their thermal parameters were however allowed to refine. The structure was refined (on F) to $R = 0{\cdot}044$ ($wR = 0{\cdot}056$). The opposite (and rejected) enantiomorph refined to $R = 0{\cdot}049$. Weighting scheme $w = 1/[\sigma^2(F) + 0{\cdot}003F^2]$, $\sigma(F)$ taken from counting statistics. In final difference map, max. and min. peaks 0·51 and $-0{\cdot}54$ e $Å^{-3}$. Final shifts/e.s.d.'s all <0·20. Scattering factors from *International Tables for X-ray Crystallography* (1974). Calculations were carried out using *SHELX*76 (Sheldrick, 1976) together with some of our own programs on the Amdahl V7 at

Table 2. *Atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses*

$$\bar{U} = \textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$\bar{U}(Å^2 \times 10^3)$
Nd	2041 (1)	571 (0)	2492 (0)	41 (1)
N(10)	−566 (16)	873 (6)	1532 (4)	60 (11)
O(11)	1155 (12)	926 (7)	1515 (3)	73 (9)
O(12)	−1270 (15)	869 (7)	1985 (4)	74 (11)
O(13)	−1575 (14)	864 (6)	1149 (3)	74 (11)
N(20)	2084 (13)	2453 (6)	2494 (4)	56 (12)
O(21)	684 (14)	2051 (5)	2639 (4)	82 (10)
O(22)	3449 (13)	2029 (5)	2310 (4)	75 (10)
O(23)	2159 (15)	3204 (5)	2502 (4)	81 (13)
N(30)	4666 (19)	908 (6)	3457 (4)	59 (12)
O(31)	2967 (14)	969 (6)	3480 (3)	66 (10)
O(32)	5365 (14)	832 (6)	2980 (4)	73 (11)
O(33)	5794 (15)	978 (7)	3822 (3)	79 (12)
W(1)	4736 (12)	327 (5)	1919 (3)	53 (9)
W(2)	2944 (14)	−695 (5)	2972 (3)	66 (9)
W(3)	805 (16)	−685 (5)	2043 (3)	71 (10)
W(4)	−721 (12)	324 (6)	3068 (3)	80 (10)
W(5)	7039 (11)	2766 (7)	2496 (5)	80 (15)
N(41)	4787 (17)	1398 (7)	6056 (5)	67 (12)
C(42)	4520 (23)	2199 (9)	6144 (4)	75 (15)
C(43)	4598 (21)	2768 (9)	5732 (4)	68 (13)
C(44)	5034 (18)	2538 (7)	5203 (4)	53 (11)
C(45)	5351 (20)	1652 (9)	5139 (7)	81 (16)
C(46)	5208 (20)	1159 (8)	5542 (4)	54 (13)
C(47)	5096 (18)	3104 (8)	4739 (5)	63 (15)
C(48)	5659 (20)	3947 (8)	4832 (5)	71 (14)
C(49)	5661 (24)	4474 (9)	4395 (6)	105 (17)
N(410)	5146 (15)	4275 (9)	3906 (5)	115 (15)
C(411)	4623 (27)	3474 (9)	3816 (6)	96 (18)
C(412)	4586 (24)	2864 (9)	4238 (5)	71 (15)
N(71)	−900 (15)	−840 (8)	3897 (4)	63 (12)
C(72)	−454 (22)	−1582 (12)	3806 (6)	66 (18)
C(73)	−394 (18)	−2194 (8)	4208 (4)	55 (12)
C(74)	−948 (15)	−1983 (8)	4734 (4)	55 (12)
C(75)	−1529 (17)	−1176 (7)	4808 (6)	58 (12)
C(76)	−1516 (23)	−625 (9)	4352 (5)	70 (15)
C(77)	−927 (17)	−2599 (7)	5167 (4)	49 (11)
C(78)	−1251 (16)	−3444 (8)	5058 (4)	53 (12)
C(79)	−1287 (17)	−3996 (8)	5512 (5)	58 (12)
N(710)	−835 (16)	−3766 (7)	6010 (3)	62 (11)
C(711)	−486 (19)	−2965 (9)	6101 (5)	60 (13)
C(712)	−490 (16)	−2348 (7)	5691 (5)	52 (12)

Table 3. *Molecular dimensions in the coordination sphere* (*distances*, Å; *angles*, °)

Nd—O(11)	2·558 (8)	Nd—O(32)	2·693 (10)
Nd—O(12)	2·716 (10)	Nd—*W*(1)	2·419 (8)
Nd—O(21)	2·578 (9)	Nd—*W*(2)	2·428 (8)
Nd—O(22)	2·573 (9)	Nd—*W*(3)	2·454 (8)
Nd—O(31)	2·606 (9)	Nd—*W*(4)	2·462 (8)

O(11)—Nd—O(12)	46·50 (28)	O(21)—Nd—*W*(2)	142·44 (27)
O(11)—Nd—O(21)	80·63 (30)	O(22)—Nd—*W*(2)	137·17 (31)
O(12)—Nd—O(21)	65·02 (31)	O(31)—Nd—*W*(2)	71·25 (29)
O(11)—Nd—O(22)	74·43 (31)	O(32)—Nd—*W*(2)	71·12 (31)
O(12)—Nd—O(22)	95·80 (31)	*W*(1)—Nd—*W*(2)	86·59 (30)
O(21)—Nd—O(22)	48·95 (30)	O(11)—Nd—*W*(3)	70·49 (30)
O(11)—Nd—O(31)	153·11 (32)	O(12)—Nd—*W*(3)	67·80 (33)
O(12)—Nd—O(31)	127·53 (30)	O(21)—Nd—*W*(3)	132·58 (33)
O(21)—Nd—O(31)	74·95 (30)	O(22)—Nd—*W*(3)	143·10 (29)
O(22)—Nd—O(31)	81·04 (31)	O(31)—Nd—*W*(3)	135·46 (29)
O(11)—Nd—O(32)	127·27 (28)	O(32)—Nd—*W*(3)	130·14 (33)
O(12)—Nd—O(32)	161·02 (31)	*W*(1)—Nd—*W*(3)	83·71 (32)
O(21)—Nd—O(32)	97·27 (31)	*W*(2)—Nd—*W*(3)	68·68 (29)
O(22)—Nd—O(32)	66·09 (30)	O(11)—Nd—*W*(4)	112·47 (27)
O(31)—Nd—O(32)	47·18 (29)	O(12)—Nd—*W*(4)	66·25 (28)
O(11)—Nd—*W*(1)	71·36 (27)	O(21)—Nd—*W*(4)	76·39 (30)
O(12)—Nd—*W*(1)	116·78 (28)	O(22)—Nd—*W*(4)	123·93 (30)
O(21)—Nd—*W*(1)	121·90 (30)	O(31)—Nd—*W*(4)	72·63 (29)
O(22)—Nd—*W*(1)	74·51 (30)	O(32)—Nd—*W*(4)	118·15 (28)
O(31)—Nd—*W*(1)	112·65 (28)	*W*(1)—Nd—*W*(4)	161·52 (30)
O(32)—Nd—*W*(1)	65·55 (28)	*W*(2)—Nd—*W*(4)	78·32 (31)
O(11)—Nd—*W*(2)	135·23 (30)	*W*(3)—Nd—*W*(4)	80·91 (31)
O(12)—Nd—*W*(2)	126·95 (32)		

the University of Reading. Positional parameters are given in Table 2* and selected dimensions in Table 3.

Discussion. The structure of the $[Nd(NO_3)_3(H_2O)_4]$ complex is shown in Fig. 1, together with the atomic numbering scheme. The metal atom is ten coordinate being bonded to three bidentate nitrate anions [Nd—O 2·558 (8)–2·716 (10) Å] and four water molecules [Nd—W 2·419 (8)–2·462 (8) Å]. These values are similar to those found in the other Nd complexes of this series (Al-Rasoul & Weakley, 1982; Weakley, 1982, 1984). The geometry of the Nd metal is a common ten-coordinate environment for compounds of the type M(bidentate)$_3$(monodentate)$_4$. It can be described as a $4A,6B$ extended dodecahedron of symmetry C_{2v} (Drew, 1977). The three bidentate ligands numbered 2,1,3 occupy respectively the a,b',b' faces of this polyhedron. There are two other structures (types 1 and 7) which contain ten-coordinate metal atoms. Of these type 1 has an equivalent geometry to (1). We used our method (Drew, 1977) for calculating r.m.s. deviations from ideal geometries and found values of 0·071 Å for type 1 (Table 1) and 0·062 Å for (1). On the other hand type 7 (Table 1) has an irregular (1:6:3) geometry, with one water molecule in the unique capping position and with three others in the three-face. The Yb atom lies close to the mean planes of the three NO_3 ligands. That structure is totally different from (1) because as is apparent from Fig. 1 all four water molecules are to one side of the metal atom in that structure.

In the Nd complex, the two solvent 4-bipy molecules and the solvent water molecules are all held together by an intricate network of hydrogen bonds. The unit cell of (1) in the a projection is shown in Fig. 2. Details of hydrogen bonds are shown in Table 4. As can be seen, each of the ten H atoms of the five water molecules is involved in intramolecular hydrogen bonds. Four of them form hydrogen bonds to the four N atoms of the two 4-bipy ligands; one to the water molecule, three to the nitrate numbered 2, and one each to nitrates 1 and 3. The two longest Nd—O distances are to O(12) and O(32), both of which participate in intermolecular hydrogen bonds to W(1) and W(4) respectively. The Nd—O bonds to nitrate 2 are not so long, despite the O atoms being involved in hydrogen bonds and this may be due to the fact that the a edge of the coordination polyhedron is less crowded than the b' edges.

The 4-bipy ligands remain unbonded to the metal but are all connected through hydrogen bonds as is shown in Fig. 2. The individual pyridyl rings are planar but in each 4-bipy molecule, the plane of one ring is rotated with respect to the other about the central C—C bond by 7·7 (1) and 5·1 (1)° in the two ligands.

It is interesting that the present structure is the only one of the eight types (Table 1) in which there is water of crystallization. In all other structures, all water molecules are bonded to the metal atoms.

As is apparent from Fig. 2, there are pseudo-twofold axes along the y direction; at $x = 0·20$ and $z = 0·25$ and related positions. There is a close correspondence

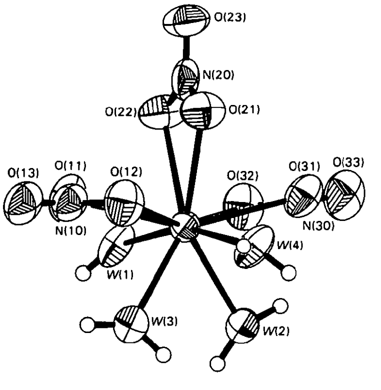

Fig. 1. The structure of $[Nd(NO_3)_3(H_2O)_4]$.

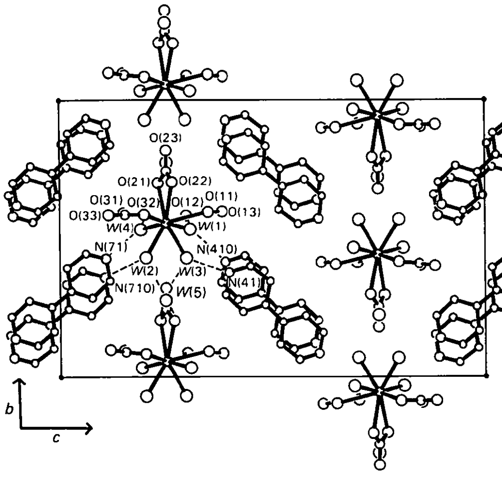

Fig. 2. The unit cell of $[Nd(NO_3)_4(H_2O)_4].2(4\text{-bipy}).H_2O$ in the a projection. Hydrogen bonds around one-molecule are shown as broken lines.

Table 4. *Intermolecular hydrogen bonds (distances, Å; angles, °)*

W—H···X	Symmetry operation on X	H···X	W···X	W—H···X
W(1)—HW(11)···N(410)	$1-x, -\frac{1}{2}+y, \frac{1}{2}-z$	1·81	2·64 (1)	154
W(1)—HW(12)···O(12)	$-1+x, y, z$	2·09	2·98 (1)	165
W(2)—HW(21)···W(5)	$1-x, -\frac{1}{2}+y, \frac{1}{2}-z$	1·82	2·71 (1)	168
W(2)—HW(22)···N(710)	$\frac{1}{2}+x, -\frac{1}{2}-y, 1-z$	2·05	2·80 (1)	138
W(3)—HW(31)···N(41)	$\frac{1}{2}-x, -y, -\frac{1}{2}+z$	1·88	2·72 (1)	145
W(3)—HW(32)···O(23)	$-x, -\frac{1}{2}+y, \frac{1}{2}-z$	2·22	2·98 (1)	140
W(4)—HW(41)···N(71)	x, y, z	1·87	2·77 (1)	174
W(4)—HW(42)···O(32)	$-1+x, y, z$	2·05	2·92 (1)	157
W(5)—HW(51)···O(21)	$1+x, y, z$	2·07	2·86 (1)	143
W(5)—HW(52)···O(22)	x, y, z	2·05	2·86 (1)	141

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44149 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

between pairs of positions in the neodymium complex and between the two 4-bipy ligands.

We thank A. W. Johans for his assistance with the crystallographic investigations and SERC for provision for the diffractometer.

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Acta Cryst. (1987). C**43**, 2084–2088

Low-Temperature X-ray and Neutron Diffraction Studies of Tetra-*n*-butylammonium Octadecacarbonyl-μ_3-hydrido-*octahedro*-hexaosmium*

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(*Received* 29 *April* 1987; *accepted* 23 *June* 1987)

Abstract. $[NBu^n_4][Os_6(\mu_3\text{-}H)(CO)_{18}]$, $C_{16}H_{36}N^+.[Os_6(CO)_{18}H]^-$, $M_r = 1888{\cdot}86$, monoclinic, $I2$; at 200 K (X-ray), $a = 13{\cdot}759\ (11)$, $b = 15{\cdot}034\ (9)$, $c = 22{\cdot}093\ (9)$ Å, $\beta = 93{\cdot}16\ (5)°$, $V = 4563\ (5)$ Å^3, $Z = 4$, $D_x = 2{\cdot}75$ Mg m^{-3}, Mo $K\alpha$, $\bar{\lambda} = 0{\cdot}71069$ Å, $\mu = 16{\cdot}7$ mm^{-1}, $F(000) = 3392$, $R = 0{\cdot}049$ for 2847 unique observed reflections with $(\sin\theta)/\lambda \le 0{\cdot}60$ Å^{-1}; at 20 K (neutron), $a = 13{\cdot}743\ (4)$, $b = 14{\cdot}780\ (4)$, $c = 21{\cdot}430\ (8)$ Å, $\beta = 93{\cdot}15\ (3)°$, $V = 4346\ (3)$ Å^3, $Z = 4$, $D_x = 2{\cdot}89$ Mg m^{-3}, $\lambda_N = 1{\cdot}1617\ (1)$ Å, $\mu = 0{\cdot}154$ mm^{-1}, $F(000) = 1062{\cdot}5$ fm, $R = 0{\cdot}123$ for 2780 unique observed reflections with $(\sin\theta)/\lambda \le 0{\cdot}56$ Å^{-1}. The neutron diffraction study of the title compound confirms the μ_3 site proposed for the hydride ligand in the $[Os_6(\mu_3\text{-}H)(CO)_{18}]^-$ anion. The average Os—H distance is 1·93 (2) Å, and is the first such distance to be determined; the mean Os—H—Os angle is 100·4 (7)°.

Introduction. The title compound was prepared by protonation of the $[Os_6(CO)_{18}]^{2-}$ dianion as described by Eady, Johnson & Lewis (1976). A brief report of the X-ray crystal structure of the $[(PPh_3)_2N]$ salt of $[Os_6(\mu_3\text{-}H)(CO)_{18}]$ was published in 1976 (McPartlin, Eady, Johnson & Lewis, 1976). In order to confirm the assignment of a μ_3 site for the hydride ligand we have undertaken a neutron diffraction study of this anion, as its $[NBu^n_4]$ salt. The dimensions of the Os—(μ_3-H) unit, which has not previously been characterized by neutron diffraction, are of interest to allow comparison with the more widely studied Os—(μ_2-H) and Os—H moieties. Further, as a class, very few μ_3-H complexes have been studied by neutron diffraction.

Experimental. *X-ray study:* red/orange irregular block (approx. $0{\cdot}15 \times 0{\cdot}25 \times 0{\cdot}30$ mm); Nicolet *P*3*m* diffractometer, 12 reflections centred ($17 < 2\theta < 25°$), graphite-monochromated Mo $K\alpha$; temperature 200 K by LT-1 N_2 gas-flow apparatus; for data collection $3{\cdot}5 < 2\theta < 50°$, ω–2θ scans, width in $2\theta = 2{\cdot}4° + \Delta_{\alpha_1\alpha_2}$, 2 s prescan after which reflections with >30 counts remeasured at rates between 1·5 and 29·3° min^{-1}, 2 octants of data (*hkl*, *hk*–*l*, 4094 reflections, *h* 0→16, *k* 0→17, *l* –26→26) measured over 110 X-ray hours, corrected for *ca* 10% decay from check reflections; systematic absences *hkl*: $h+k+l=2n+1$, space group $I2$ (No. 5) determined by Laue symmetry, solution and refinement. 3038 unique data, $R_{\rm int} = 0{\cdot}035$ (186 pairs of intensities averaged); for structure solution and refinement 2847 with $F > 4\sigma(F)$ retained, absorption correction by 238 azimuthal scan

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* Carried out, in part, under contract DE-AC02-76CH00016 with the US Department of Energy, Office of Basic Energy Sciences.

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0108-2701/87/112084-05$01.50