cis-DICHLORO(DIMETHYL SULFOXIDE)(PYRIDINE)PLATINUM(II)

The Pt-Cl bond lengths [2.316 (4) trans to S and 2.309 (3) Å trans to N] are normal for this type of complex (Sotman et al., 1988). The Pt-S distance is 2.209 (4) Å. The results of X-ray structure determinations of the cis-[Pt(dmso)LCl₂] complexes show that Pt-S bond lengths decrease following the ligand L sequence: dmso [av. 2.234 (6) Å (Melanson & Rochon, 1975) and 2.248 (5) Å (Shibaeva, 1985)] > MeCN [av. 2.220 (6) Å for two crystallographically independent molecules (Moiseev et al., 1989)] > PhCH₂CN [2.215 (1) Å (Belsky, Konovalov & Kukushkin, 1990)] > py [2.209 (4) Å] > 2Me C_5H_4N [2.200 (3) Å (Melanson & Rochon, 1977)] > NH₃ [2·186 (4) Å (Melanson & Rochon, 1978a)]. The observed variations in the Pt-S bond lengths provide a rare example of the *cis* influence of the ligands.

The Pt—N distance [2.027 (10) Å] is normal and agrees with Pt—N bond lengths in (Ph₃-PCH₂Ph)[PtpyCl₃] [2.028 (6) Å (Belsky, Kukushkin, Konovalov, Moiseev & Yakovlev, 1990)] and (Et₄N)[PtpyCl₃] [2.018 (6) Å (Kukushkin *et al.*, 1990)]. The pyridine ring is planar. The dihedral angle between the py plane and the PtNSCl(1)Cl(2) plane is 56.8°. The value of the dihedral angle in *cis*-[Pt(dmso)(2Me-C₅H₄N)Cl₂] with the more sterically hindered 2-methylpyridine ligand is 87.1° (Melanson & Rochon, 1977).

References

- BELSKY, V. K., KONOVALOV, V. E. & KUKUSHKIN, V. YU. (1990). Inorg. Chim. Acta. In the press.
- BELSKY, V. K., KUKUSHKIN, V. YU., KONOVALOV, V. E., MOISEEV, A. I. & YAKOVLEV, V. N. (1990). Zh. Obshch. Khim. In the press.
- CARUSO, F., SPAGNA, R. & ZAMBONELLI, L. (1980). Acta Cryst. B36, 713-715.
- DAVIES, J. A. (1981). Adv. Inorg. Chem. Radiochem. 24, 115-187.
- ELDING, L. I. & OSKARSSON, Å. (1987). Inorg. Chim. Acta, 130, 209–213.
- KUKUSHKIN, YU. N. (1985). The Chemistry of the Coordination Compounds. Moscow: VS.
- KUKUSHKIN, V. YU., BELSKY, V. K., ALEKSANDROVA, E. A., PANKOVA, E. YU., KONOVALOV, V. E., YAKOVLEV, V. N. & MOISEEV, A. I. (1990). *Zh. Obshch. Khim.* In the press.
- KUKUSHKIN, V. YU., MOISEEV, A. I. & SIDOROV, E. O. (1989). Zh. Obshch. Khim. 59, 1958–1966.
- MELANSON, R. & ROCHON, F. D. (1975). Can. J. Chem. 53, 2371–2374.
- MELANSON, R. & ROCHON, F. D. (1977). Acta Cryst. B33, 3571-3573.
- MELANSON, R. & ROCHON, F. D. (1978a). Acta Cryst. B34, 941–943.
- MELANSON, R. & ROCHON, F. D. (1978b). Acta Cryst. B34, 1125-1127.
- MOISEEV, A. I., KUKUSHKIN, V. YU., SIMANOVA, S. A., BELSKY, V. K. & KONOVALOV, V. E. (1989). XIV Chernyaev Conference, Vol. 1, p. 55. Novosibirsk, USSR.
- SHELDRICK, G. M. (1984). SHELXTL Users Manual. Revision 4.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.
- SHIBAEVA, R. P. (1985). Koord. Khim. 11, 129-131.
- SOTMAN, S. S., FUNDAMENSKY, V. S., KUKUSHKIN, V. YU. & PANKOVA, E. YU. (1988). Zh. Obshch. Khim. 58, 2297–2304.

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Structure of Ethylenediammonium *catena*-Poly[μ -(2-pyridinecarboxylato- $\kappa N, \kappa O: \kappa O'$)-tris(2-pyridinecarboxylato- $\kappa N, \kappa O$)neodymate] Hexahydrate

BY P. STARYNOWICZ

Institute of Chemistry, The University, 14 F. Joliot-Curie Str., 50-383 Wroclaw, Poland

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Abstract. $C_2H_{10}N_2^{2+}[\{Nd(C_6H_4NO_2)_4\}_2]^{2-}.6H_2O, M_r = 1435\cdot5, monoclinic, Cc, a = 13.654 (9), b = 21.813 (11), c = 20.136 (12) Å, \beta = 99.36 (4)^\circ, V = 5917 (7) Å^3, Z = 4, D_m = 1.60, D_x = 1.611 (2) Mg m^{-3}, \lambda(Mo K\alpha) = 0.71069 Å, \mu = 1.83 mm^{-1}, F(000) = 2880, T = 304 (1) K, final R = 0.0395 for 7450 reflections. The Nd ions are surrounded by four N and five carboxylic O atoms. The structure consists of a polymeric chain of complex anions, ethylenediammonium cations and water of hydration.$

Introduction. In a previous paper (Starynowicz, 1991) the crystal structure of barium neodymium α -picoli-

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nate has been reported. It seemed interesting to investigate neodymium coordination in another (α -picolinato)neodymate system. This work is a part of our study on the structures and properties of complexes with lanthanide-nitrogen bonds.

Experimental. The title compound was prepared by adding ethylenediamine and 2-pyridinecarboxylic (α -picolinic) acid to a warm aqueous suspension of freshly precipitated neodymium hydroxide. After two days, parallelepiped pink-violet crystals were formed. A specimen $0.7 \times 0.35 \times 0.2$ mm was cut from a larger crystal. D_m by flotation in C₂H₄Br₂/CHCl₃. Oscillation and Weissenberg photographs indicated

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Ndl

Nd2

011 021

012 022

013

O23 O14

O24 O15

025

O16 O26 O17

O27 O18

O28 OW1

0*W*2

OW3

OW5

0W6 N1 N2

N3 N4

N5 N6 N7

N8

N9

N10

C1 C2

C11 C21 C31 C41

C5I

C61 C12 C22

C32 C42 C52 C62 C13 C23 C33 C43 C53 C63

C14 C24 C34

C44 C54 C64 C15

C25 C35 C45

C55

C65 C16

C26 C36

C46

C56 C66

C17 C27

C37 C47 C57

C67

C18

C28 C38

C48

C58

C68

the monoclinic crystal system and space group Cc or C2/c. Syntex P2₁ diffractometer, Mo K α radiation for lattice parameters (15 reflections, $21 < 2\theta < 29^{\circ}$), variable $\omega/2\theta$ scan, $4 < 2\theta < 60^\circ$, two standards every 50 reflections, mean relative e.s.d. of the control reflections 3.5%. 8673 intensities measured, 8672 unique, out of which 7449 had $I \ge 3\sigma(I)$, index range $h \to 19$, $k \to 30$, $l - 28 \to 27$, absorption corrections with locally modified program ABSORB (Ugozzoli, 1987), transmission factors between 0.722 and 1.043. Structure was solved with SHELXS86 (Sheldrick, 1986) and refined with locally modified SHELX76 (Sheldrick, 1976) in the noncentrosymmetric space group Cc. 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 hydrogens placed geometrically, water and N-bonded hydrogens from difference syntheses, not all found. The structure orientation with respect to the polar axes (Jones, 1986) with better R was chosen. Final full-matrix least-squares refinement based on F[non-H atoms anisotropic, hydrogens isotropic with constrained positional parameters and common temperature factors, separately for hydrogens bonded to C(ethylenediamine), C(picolinate), N and O, the constraints refined as additional parameters, and two hydrogens were geometrically fixed], R = 0.0395, wR = 0.0369, the maximum shift/e.s.d. ratio was 0.20, $\Delta \rho$ between -1.37 and 1.49 e Å⁻³, the highest peaks around Nd atoms and $w = 1/\sigma^2(F)$. 903 parameters refined.

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 the ORTEP (Johnson, 1976) view of the complex anion fragment is given in Fig. 1.

All the distances are within the range observed by Sinha (1976). It is, however, interesting to note that the bonds between Nd ions and the bridging O atoms (O17 or O28) are not longer than other neodymium-carboxylic oxygen bonds, contrary to what has been observed elsewhere (*e.g.* Dào, 1987). The coordination figure of Nd1 may best be described as a capped square antiprism (CSA) with Δ = 0.086 Å² ($\Delta = \sum d_i^2/9$, d_i is the distance between the real position of the *i*th atom, and the relevant vertex

Table 1. Final atomic coordinates and equivalent isotropic thermal U factors

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

x	v	Z	$U_{rr}(\text{\AA}^2)$
0	0.21370 (2)	0	0.0318 (1)
0.19194 (4)	0.47612 (2)	- 0.02744 (3)	0·0306 (1)
0.0676 (5)	0.2168 (3)	- 0.1045 (3)	0.048(2)
0.1511 (8)	0.1855(3) 0.5291(3)	-0.1834(4) 0.0777(3)	0.045 (2)
0.1032(5)	0.5684(3)	0.1563 (4)	0.065(2)
0.0309 (4)	0.1386 (3)	0.0898 (3)	0.042 (2)
0.1314 (6)	0.0860 (4)	0.1672 (4)	0.071(3)
0.2666 (5)	0.3905(3) 0.3123(3)	-0.0803(3) -0.0801(4)	0.047(2) 0.066(3)
-0.1484(4)	0.2478(3)	0.0432 (3)	0.049 (2)
- 0.3102 (5)	0.2688 (3)	0.0290 (3)	0.058 (2)
0.0654 (4)	0.4578 (3)	-0.1229(3)	0.045 (2)
-0.0723(5) 0.1760(5)	0.4796(4) 0.3888(3)	0.0445 (4)	0.070(3) 0.054(2)
0.0774 (5)	0.3135 (3)	-0.0024 (3)	0.047 (2)
0.3007 (4)	0.5646 (3)	- 0.0134 (3)	0.041 (2)
0.3687 (5)	0.6480(3)	-0.0493(3) -0.7548(3)	0.049(2)
-0.7451(7)	-0.1476(4)	-0.7808(4)	0.076 (3)
- 0.7489 (6)	-0.0144 (3)	- 0.7748 (4)	0.063 (3)
- 0.1691 (6)	-0.1177(3)	- 0.3666 (4)	0.070 (3)
-0.0252(12) -0.0330(13)	-0.0800(8) -0.1771(11)	-0.2659(8) -0.1915(10)	0.189(9) 0.258(13)
0.0702 (6)	0.1051(3)	- 0.0509 (4)	0.044 (2)
0.0134 (5)	0.4725 (3)	0.0184 (4)	0.043 (2)
0.1860 (5)	0.1931(4)	0.0466 (4)	0.045(2)
-0.1359(5)	0.4382(3) 0.2804(3)	-0.0817(4)	0.037(2) 0.046(2)
0.0802 (5)	0.5718 (3)	- 0.0731 (4)	0.041 (2)
0.0482 (5)	0.2735 (3)	0.1171 (4)	0.041 (2)
0.2621(6)	0.5186(3) 0.7338(4)	-0.1369(3) -0.8410(4)	0.042 (2)
-0.3418(6)	0.1805 (3)	-0.6834(4)	0.049 (3)
- 0.3287 (7)	0.2127 (4)	- 0.7985 (4)	0.046 (3)
-0.2741(8)	0.1900 (5)	-0.7323(5) -0.0277(5)	0.054(3)
0.0910 (13)	-0.0028(5)	-0.0581(8)	0.095 (7)
0.1250 (12)	0.0039 (5)	-0.1186 (8)	0.090 (6)
0.1371 (11)	0.0627 (5)	-0.1431(7)	0.078(5)
0.1003(7) 0.1101(7)	0.1762(4)	-0.1341(5)	0.047 (3)
- 0.0655 (7)	0.4389 (5)	- 0.0074 (6)	0-054 (3)
- 0.1453 (8)	0.4306 (6)	0.0253 (7)	0.074 (5)
-0.1403(9) -0.0653(9)	0.4382(7) 0.4928(5)	0.1125 (6)	0.067 (4)
0.0146 (7)	0.4982 (4)	0.0786 (5)	0.047 (3)
0.1050 (6)	0.5348 (4)	0.1070 (4)	0.059 (4)
0.3620 (8)	0.2208(3) 0.2078(7)	0.0230(5) 0.0482(6)	0.059(4) 0.069(4)
0.3794 (8)	0.1620 (7)	0.0964 (7)	0.088 (5)
0.3022 (7)	0.1346 (6)	0.1206 (5)	0.067(4)
0.2059 (7)	0.1311(4) 0.1231(4)	0.0931(4) 0.1197(4)	0.043(3) 0.042(3)
0.4253 (8)	0.4640 (5)	0.0865 (5)	0.055 (3)
0.5123 (8)	0.4387 (5)	0.1170 (6)	0.062 (4)
0.4969 (7)	0.3871(3) 0.3612(4)	0.0352 (5)	0.059(4) 0.050(3)
0.4071 (6)	0.3885 (4)	0.0052 (4)	0.037 (2)
0.3459 (6)	0.3607 (4)	- 0.0569 (4)	0.037 (3)
-0.1263(8) -0.2039(9)	0.3003(5) 0.3265(7)	- 0.1441 (6)	0.079 (5)
-0.2917(9)	0.3309 (6)	- 0.1661 (6)	0.076 (5)
- 0.3036 (8)	0.3122 (5)	-0.1032 (6)	0.055 (4)
-0.2223(7)	0.2871 (4)	-0.0096 (4)	0.044(3) 0.041(3)
0.0882 (7)	0.6295 (4)	- 0.0506 (5)	0.049 (3)
0.0222 (9)	0.6748 (4)	-0.0707 (6)	0.060 (4)
- 0.0579 (9)	0.6612 (5)	-0.1176 (6)	0.070 (4)
0.0028 (7)	0.5588(4)	-0.1202(5)	0.004 (4)
- 0.0025 (6)	0.4938 (4)	-0.1481 (4)	0.045 (3)
0.0279 (7)	0.2555 (5)	0.1781 (5)	0.052 (3)
0.0242 (11) 0.1016 (11)	0.2897 (6)	0.2322 (7)	0.079 (5)
0.1215 (10)	0.3639 (5)	0.1708 (6)	0.071 (4)
0.0960 (6)	0.3266 (4)	0.1150 (4)	0.030 (3)
0.1180 (0)	0.3442 (3)	-0.1983 (5)	0.059 (3)
0.2883 (9)	0.5164 (6)	-0.2513 (5)	0.066 (4)
0.3424 (10)	0.5696 (6)	- 0.2423 (6)	0.076 (5)
0.3335 (8)	0.5709 (4)	-0.1286(4)	0.030 (4)
0.3300 (6)	0 5965 (4)	-0.0585 (4)	0.036 (2)

^{*} 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 53371 (52 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

Nd1-011	2.432 (6)	Nd1-013	2.425 (6)
015	2·447 (6)		2.424 (7)
	2.382 (7)	—N1	2.811 (7)
—N3	2.599 (7)	—N5	2.697 (7)
—N7	2.681 (8)		
Nd2-012	2.440 (6)	Nd1-014	2.454 (7)
-O16	2.400 (6)	017	2.424 (7)
-O18	2.424 (6)	N2	2.745 (7)
—N4	2.670 (7)	N6	2.659 (7)
—N8	2.706 (7)		. ,
O13—Nd1—O11	128.3 (3)		
O15-Nd1-O11	138.7 (3)	O15-Nd1-O13	89.7 (2)
O27-Nd1-O11	74.3 (3)	O27-Nd1-O13	126.7 (3)
O27-Nd1-015	97.1 (3)		
O28 ⁱ -Nd1-O11	91-3 (3)	O28 ⁱ —Nd1—O13	86.1 (3)
O28 ⁱ —Nd1—O15	73.8 (3)	O28 ⁱ —Nd1—O27	146.4 (3)
N1-Nd1-011	60.5 (3)	N1-Nd1-013	71.0 (3)
N1-Nd1-015	137.5 (3)	N1-Nd1-027	125·0 (3)
N1-Nd1-O28 ⁱ	67.6 (3)		()
N3-Nd1-011	80.3 (3)	N3-Nd1-013	64.7 (3)
N3-Nd1-015	137.8 (3)	N3-Nd1-027	76.1 (3)
N3-Nd1-028 ⁱ	132.2 (3)	N3-Nd1N1	67.4 (3)
N5-Nd1-011	77.2 (3)	N5-Nd1-013	146.9 (3)
N5-Nd1-015	61.7 (3)	N5-Nd1-027	76.3 (3)
N5-Nd1-028 ⁱ	71·0 (3)	N5-Nd1-N1	118.3 (3)
N5-Nd1-N3	148.0 (3)		
N7—Nd1—O11	132.4 (3)	N7Nd1013	71.6 (3)
N7-Nd1-015	68·8 (3)	N7Nd1O27	62.3 (3)
N7—Nd1—O28 ⁱ	136-0 (3)	N7—Nd1—N1	132.9 (3)
N7—Nd1—N3	71.4 (3)		
N7Nd1N5	108.7 (3)		
O14-Nd2-012	146.0 (3)		
O16-Nd2-O12	129.3 (2)	O16-Nd2-O14	79·7 (3)
O17—Nd2—O12	80.1 (3)	O17-Nd2-O14	75-3 (3)
017—Nd2—016	103-0 (3)		
O18—Nd2—O12	69.3 (2)	O18-Nd2-O14	111.5 (3)
018—Nd2—016	125.0 (2)	O18-Nd2-O17	132-0 (3)
N2-Nd2-012	62.3 (3)	N2-Nd2-014	125-4 (3)
N2-Nd2-016	72.6 (3)	N2-Nd2-017	66.8 (3)
N2	123.0 (3)		
N4—Nd2—O12	87.7 (3)	N4—Nd2—O14	62.1 (3)
N4-Nd2-016	141.7 (3)	N4-Nd2-017	70-5 (3)
N4—Nd2—O18	72.1 (3)	N4—Nd2—N2	130-8 (3)
N6-Nd2-012	79.3 (3)	N6—Nd2—O14	134.6 (3)
N6-Nd2-016	63-6 (3)	N6-Nd2-017	136-1 (3)
No-Nd2-018	73-8 (3)	N6-Nd2-N2	69.4 (3)
IND	145.9 (3)		
No-Na2-012	128-8 (3)	N8-Nd2-014	71.3 (3)
No-Na2-016	/3-1 (3)	N8—Nd2—O17	146.5 (3)
1N0-IN02-018	62-0 (3)	N8—Nd2—N2	137.3 (3)
N8_Nd2_N6	72.1 (2)		
110-1102-1100	() () ()		

Symmetry code: (i) $x - \frac{1}{2}$, $y - \frac{1}{2}$, z.

in the ideal, least-squares-fitted polyhedron; Drew, 1977) for N1 as the cap, O28, O11, N3 and O13 as the capped base, and O15, N5, O27 and N7 as the non-capped base, no particular symmetry around the metal ion being displayed. The best-fitted tricapped trigonal prism (TTP; O13, O28, O15 and N3, O11, O27 as the bases, N1, N5, N7 as the caps, approximate point symmetry $C_{2\nu}$ has been found to have Δ $= 0.111 \text{ Å}^2$. The coordination figure of Nd2 is one intermediate between CSA (the non-capped base -O14, O16, N2, O17, the capped base - N8, N6, O12, N4, the cap – O18, $\Delta = 0.093 \text{ Å}^2$) and TTP (the bases - O12, N4, O17 and N6, N8, O16, the caps - O18, O14, N2, $\Delta = 0.094 \text{ Å}^2$), in both cases without symmetry. The coordination environments of Nd1 and Nd2 are essentially the same as that of Nd1 in neodymium barium α -picolinate, which in the case

of Nd1 (of the present structure) may be shown by taking O11, N5, O28 and N3, N7, O13 as the bases, and O17, O15, N1 as the caps of the TTP, Δ being then 0.196 Å². The tetrakis(α -picolinato)neodymate subunits accept bridging oxygens from neighbouring subunits to the ninth coordination site of the Nd ions so as to form infinite chains along the [110] and [110] directions. The tendency towards polymerization in Nd- α -picolinate ligand systems is demonstrated both in this compound and in neodymium barium α -picolinate. The structure is held together by an elaborate network of intermolecular hydrogen bonds, some of them bifurcated. The crystal packing is shown in Fig. 2.

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Fig. 1. Molecular diagram of a fragment of the chain showing the complex anion. The numbering patterns of the C atoms follow that for the C14 to C64 atoms.



Fig. 2. View of the crystal structure. The dashed lines represent hydrogen bonds.

References

Dào, C. N. (1987). PhD Dissertation, Univ. of Wrocław, Poland.
DREW, M. G. B. (1977). Coord. Chem. Rev. 24, 179-275.
JOHNSON, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
JONES, P. G. (1986). Acta Cryst. A42, 57.

Ugozzoli, F. (1987). Comput. Chem. 11(2), 109–120.

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Structure of 1,1,1,2,2-Pentacarbonyl- μ -hydrido- μ - 2η -[(Z,s-cis,Z)-N-isopropylcinnamylideneamino- μ -N, μ - C^{γ}]-diruthenium(Ru-Ru), HRu₂(CO)₅(C₁₂H₁₄N)

BY ANTHONY L. SPEK* AND ALBERT J. M. DUISENBERG

Vakgroep Algemene Chemie, afdeling Kristal- en Structuurchemie, Universiteit te Utrecht, Padualaan 8, 3548 CH Utrecht, The Netherlands

AND WILHELMUS P. MUL, OLAF C. P. BEERS AND CORNELIS J. ELSEVIER*

Anorganisch Chemisch Laboratorium, Universiteit van Amsterdam, J. H. van't Hoff Instituut, Nieuwe Achtergracht 166, 1018 WV Amsterdam, The Netherlands

(Received 12 May 1990; accepted 20 July 1990)

Abstract. $C_{17}H_{15}NO_5Ru_2$, $M_r = 515.45$, orthorhombic, *Pcab*, a = 12.828 (1), b = 16.813 (1), c = 34.324 (2) Å, V = 7402.9 (8) Å³, Z = 16, $D_x = 1.85$ g cm⁻³, λ (Mo K α) = 0.71073 Å, $\mu = 16.3$ cm⁻¹, F(000) = 4032, T = 295 K, R = 0.038 for 5117 reflections with $I > 2.5\sigma(I)$. The two crystallographically independent molecules are essentially identical except for one torsion angle. The Ru—Ru bond is bridged asymmetrically by both a hydride atom and an *N*-isopropyl cinnamaldimin-4-yl ligand.

Introduction. Transition metal hydrido complexes are believed to be the key intermediates in important catalytic processes like hydrogenation, hydroformylation, hydrocyanation, and double-bond migration of olefins (see *e.g.* Crabtree, 1988). The presence of hydrides in combination with unsaturated ligands on transition metal carbonyl moieties affords an opportunity to study the chemical and structural properties of such crucial species in detail.

In the course of our study on the coordination and reactivity of monoazadienes ($R^1CH=CHCH=NR^2$) on transition metal carbonyls we recently reported the preparation of a series of linear tetranuclear clusters, $Ru_4(CO)_{10}[R^1C=CHCH=NR^2]_2$ (1*a*: $R^1 =$ Me, $R^2 = i$ -Pr; 1*b*: $R^1 =$ Me, $R^2 = c$ -Hex; 1*c*: $R^1 =$ Me, $R^2 = t$ -Bu; 1*d*: $R^1 =$ Ph, $R^2 = i$ -Pr; 1*e*: $R^1 =$ Ph, $R^2 = t$ -Bu) (Polm, Mul, Elsevier, Vrieze, Christophersen & Stam, 1988; Mul, Elsevier, Polm, Vrieze, Zoutberg, Heijdenrijk & Stam, 1990). These clusters are formed in high yield by thermolysis of $Ru_2(CO)_6[R^1C=CHCH_2NR^2]$. In one case an intermediate, a dinuclear hydrido complex, $HRu_2(CO)_5[Ph=C=CHCH=N-i-Pr]$ (2*d*), could be isolated. We now report the crystal structure of this dinuclear hydrido complex.

Experimental. The air-sensitive title compound was prepared by thermolysis of Ru₂(CO)₆[Ph-C= CHCH₂N-*i*-Pr] in refluxing heptane and purified by chromatography on silica employing hexane/ dichloromethane (9/1) as the eluent (Mul et al., 1990). Crystals suitable for X-ray diffraction work were grown by cooling a concentrated hexane solution at 243 K. X-ray data for a light-brown crystal $(0.12 \times 0.25 \times 0.63 \text{ mm})$, sealed in a Lindemannglass capillary, were collected on an Enraf-Nonius CAD-4F diffractometer using Zr-filtered Mo $K\alpha$ radiation. Lattice parameters and their e.s.d.'s were derived from the setting angles of 25 SET4 reflections $(14 < \theta < 18^{\circ})$. The space group was determined from the observed systematic absences. A total of 9711 reflections $\left[\theta < 27.5^\circ\right]$; $\omega/2\theta$ scan; $\Delta \omega = 0.60 +$ $0.35 \tan(\theta)^{\circ}$; 0 < h < 16, 0 < k < 21, 0 < l < 44] were scanned. Three reference reflections (204, 220, 012) showed a small linear decay of 1% during the 132 h of X-ray exposure time. The intensities were corrected for Lp for the small decay and for absoprtion (Gaussian integration; grid $8 \times 8 \times 8$; corrections 1.22 to 1.49), resulting in a set of 5117 unique

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^{*} Authors to whom correspondence should be addressed.