Acta Cryst. (1973). B29, 2412

An Unusual Pyridine-Cobalt Configuration: the Crystal Structure of Bis(dimethylglyoximato)(tri-n-butylphosphine)(4-pyridyl)cobalt(I)

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(Received 24 January 1973; accepted 29 May 1973)

Bis(dimethylglyoximato) (tri-n-butylphosphine) (4-pyridyl)cobalt(I), $C_{25}H_{45}N_5O_4PCo$, a 'cobaloxime', includes a pyridine ligand coordinated *via* a Co–C bond. The space group is PI with two molecules per unit cell of dimensions $a = 12 \cdot 109$ (1), $b = 13 \cdot 142$ (2), $c = 11 \cdot 606$ (1) Å, $\alpha = 90 \cdot 317$ (12), $\beta = 122 \cdot 452$ (7), $\gamma = 107 \cdot 117$ (10)°. A full sphere of data, $0 < 2\theta < 65^\circ$ was collected with a four-circle diffractometer using $\theta - 2\theta$ scans and Nb filtered Mo $K\alpha$ radiation. The data were corrected for absorption and averaged to give 10677 independent reflections. Full-matrix least-squares refinement gave a final R of 0.035 based on the 9027 extinction-corrected reflections with $F > 3\sigma(F)$. Bond distances were corrected for molecular libration. The Co–N distances average 1.891 (1) Å, the Co–C coordination distance is 1.983 (1) Å and Co–P is 2.342 (1) Å. Changes are noted in pyridine distances and angles as compared with uncoordinated pyridine and with pyridine coordinated *viu* a Co–N bond.

Introduction

Bis(dimethylglyoximato)cobalt complexes, 'cobaloximes', have been used as model compounds for the more complex cobalamins (Schrauzer, 1968*a*, 1971). Under favorable conditions some cobaloximes undergo reactions similar to the nonenzymatic reactions of coenzyme vitamin B_{12} , a biologically important cobalamin. This paper reports the structure of bis(dimethylglyoximato) (tri-n-butylphosphine) (4-pyridyl) cobalt(1).

The molecular structure of this cobaloxime (Fig. 1) is interesting for two reasons. It includes a pyridyl moiety coordinated through a Co-C bond, a configuration confirmed by the X-ray analysis. The bond distances and angles of the pyridyl moiety attached in this manner differ significantly from the geometry of uncoordinated pyridine (Bak, Hansen & Rastrup-Andersen, 1954) and from pyridine ligands with the usual Co-N coordination as found in other cobaloximes (Lenhert, 1967, 1972). The structure analysis also provides precise geometric data on the dimethylglyoxime ligands and the Co-N coordination which subsequent papers will compare with similar bonds in other cobaloximes, especially those considered to be model compounds for coenzyme B_{12} .

Experimental

Crystals of bis(dimethylglyoximato) (tri-n-butylphosphine) (4-pyridyl)cobalt(1), $C_{25}H_{45}N_5O_4PCo$, were supplied by Professor G. N. Schrauzer. The compound was synthesized by reacting the cobalt(1) nucleophile with 4-bromopyridine; Schrauzer (1968b) has discussed the synthesis of this class of compounds elsewhere. An elemental analysis agreed with the stated formula to within 0.3 percentage points for all elements. Precession photographs indicated a triclinic lattice. The crystal density of 1.294 g cm⁻³, determined by flotation in a carbon tetrachloride-hexane mixture, compares with a calculated value of 1.293 g cm⁻³ based on two molecules (molecular weight, 596.573 daltons) per unit-cell (volume, 1458.7 Å³). The space group assumed, $P\overline{I}$, was confirmed by the subsequent crystal structure analysis.

A large specimen was cleaved to give a data crystal with maximum dimensions of approximately 0.4×0.6 $\times 1.2$ mm and a volume of 0.157 mm³. Eastman 910 cement was used to mount the crystal so that the glass fiber (and the φ axis of the diffractometer) made a small angle with the c^* axis and was roughly perpendicular to the long dimension of the crystal.

Accurate cell constants were determined at 21 °C from 12 reflections $(2\theta > 80^\circ)$ manually centered on an accurately aligned four-circle diffractometer using the Cu $K\alpha_1$ line of the resolved doublet ($\lambda = 1.54051$ Å). The Oak Ridge General Least-Squares program (Busing & Levy, 1962) was used with the observations (positive and negative 2θ values of the 12 Friedel pairs) to give a = 12.109 (1), b = 13.142 (2), c = 11.606 (1)Å, $\alpha = 90.317$ (12)°, $\beta = 122.452$ (12)°, and $\gamma = 107.117$ (10)°.

Intensity data were collected on a four-circle computer-controlled Picker diffractometer (FACS-I system) and a Digital Equipment Corporation PDP-8/I computer with auxiliary disc memory. The computer programs, based on the FACS-I programs, were extensively modified and adapted for use with the disc memory in this laboratory (Lenhert & Henry, 1970). The X-ray source was a standard-focus Mo tube operated at 50 kV (constant potential), 12 mA and a takeoff angle of 1.8° . A 3.75×3.75 mm aperture defined the NaI detector opening and 1.5 mm collimators were used on the incident and diffracted beams. The pulse height analyzer was set to a 95% window centered on

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the Mo $K\alpha$ peak. Attenuation filters (constructed of 1 to 5 thicknesses of 0.0005 in Ni foil) were automatically inserted in the diffracted beam when the count rate exceeded 8000 counts/sec.

The X-ray generator-detector system was monitored by scanning 3 standard reflections ($\overline{335}$, 227, $\overline{174}$) at 2 to 6 h intervals. The intensities dropped by 6, 7 and 4% respectively during the 1100 h X-ray exposure. This probably represents a combination of X-ray damage to the crystal and a drop in X-ray output or detector sensitivity.

Reflections in the range $0 < 2\theta < 10^{\circ}$ were collected with a 0.002 in Nb filter in the incident beam and Zr-Y-Al balanced filters (Young, 1961) in the diffracted beam. In the range $10 < 2\theta < 65^{\circ}$ a 0.001 in Nb incident beam filter was used without balanced filters. The full sphere of reflections out to 65° in 2θ was measured with the θ -2 θ scan technique. The 2θ scan range was given by $A + B \tan \theta$ where $A = 1.1^{\circ}$ and $B = 6.29^{\circ}$ for balanced filters and $B = 0.69^{\circ}$ for β filter measurements. Background was measured at the end points of the scan.

The usual background correction was applied to the scans to give the integrated intensity, I. If the result was negative, I was taken as zero. To obtain the F^2 , the integrated intensities were multiplied by the usual Lorentz-polarization factor, the relative scale factor derived from the standard reflections, the attenuation filter factor, and the X-ray absorption factor.

Crystal shape and orientation for the absorption correction were determined with the crystal mounted on the diffractometer. The χ and φ angles were adjusted to set the crystal face perpendicular to the χ circle and parallel to the ω axis. The distance from the ω axis to the vertical face was then measured with the filar eyepiece of the diffractometer telescope. This distance and the χ and φ angles for each of the 7 crystal faces defined the crystal shape and orientation. The crystal measurements were checked for accuracy by using the coordinates of the crystal corners calculated by ORABS (Wehe, Busing & Levy, 1962) to draw a stereogram of the crystal with ORTEP (Johnson, 1965).

The absorption correction was calculated by Gaussian integration using *ORABS*. The linear absorption coefficient calculated from mass absorption coefficients for Mo K α from *International Tables for X-ray Crystallography* (1962) was 7.04 cm⁻¹. The calculated absorption factors ranged from 0.6118 to 0.8008 with an average of 0.7340.

 $\sigma(I)$ was considered to arise from two sources. The first is the variance resulting from the counting statistics, σ_c^2 . The second σ_x^2 (expressed as a percent), is that from all other sources which affect the value obtained by repeated measurements of the same reflection (e.g., short-term fluctuations in the X-ray source intensity). The combination gives $\sigma^2(I) = \sigma_c^2 + \sigma_x^2 I^2$. σ_x was estimated from repeated measurements of the standard reflections to be 0.00384. The values of $\sigma(I)$ were scaled by the same factor used for I to give $\sigma(F^2)$. Then, for all reflections, including those with $F^2 = 0$, $\sigma(F) = -F + [F^2 + \sigma(F^2)]^{1/2}$. Finally the 21523 observations were averaged and the variances combined to give 10677 independent reflections.

Structure determination and refinement

The crystal structure was solved by the heavy atom method using Co and P coordinates obtained from a sharpened Patterson function. A partial data set $(2\theta < 40^\circ)$ was used to locate and refine the remaining non-hydrogen atoms.

Initial coordinates for the 22 hydrogen atoms fixed by molecular geometry were calculated using a C-H distance of 1.0 Å and appropriate angles. The methyl hydrogen atoms were positioned by a combination of geometrical and electron density considerations as described previously (Miller, Lenhert & Joesten, 1972).



Fig. 1. Stereoscopic view of the molecule. Atom numbers are indicated, and 20% probability ellipsoids are used for all nonhydrogen atoms. The disorder in C(62) and C(63) is not shown; the free rotation in the methyl group C(13) is indicated. The two small arrows show the direction of the principal libration axis for each of the dimethylglyoxime ligands.

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Table 1.

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Anisotropic thermal parameters are of the form $T = \exp \left[-\frac{1}{2} (B_{11} h^2 a^{*2} + B_{23} h^2 b^{*2} + B_{33} h^2 c^{*2} + 2B_{13} h (a^* c^* + 2B_{23} h (b^* c^*))\right]$. *M* is the occupation factor. The first two digits of the hydrogen atom numbers correspond to the number of the adjacent carbon atom except for H(67) and H(85) which are attached to O(6) and O(8) respectively.

a a	D13 D23	1.10 0.62		2.94 (4) 3.24 (0	1.21(4) - 0.88(4)	3.27 (4) 1.69 (5	2.74 (5) 1.08 (5	1.58 (4) 2.37 (5	0.63 (4) 0.11 (4	1.46 (4) 1.07 (4)		4) 04-0 (4) 60-I	0.96 (4) 1.48 (5	1.41 (6) 3.46 (8	0.21 (6) 0.90 (6	0.88 (5) 1.34 (5	0-46 (5) 0-30 (5	3.54 (12) 7.71 (1	0.49 (11) -0.73 (1	2.03 (8) 3.01 (8)	0.76 (13) -0.84 (9	1.08 (4) 0.84 (2	1.27 (5) 0.55 (5	1.70 (6) 1.10 (6	1.10 (5) 1.19 (5	1.22 (4) 0.95 (5	0.91 (4) 0.34 (5	1-36 (5) 0-13 (5	2.03(6) - 0.14(7)	4.22 (10) 0.08 (1-93 (5) 1-35 (1.73 (6) 0.74 ((4.39 (10) 1.05 (9	2.72 (10) -0.96 (1.29 (5) 0.72 (2·04 (22) 1·69 (× • •	1-35 (16) 0-80 (1-35 (16) 0-80 (2-92 (27) 1-57 (
5	D12	0.83	1-31 (I)	2-98 (6)	2·50 (5)	2.33 (5)	4.99 (6)	1-46 (4)	1.23 (4)	0.05 (4)		2-33 (4)	1.58 (5)	0-96 (6)	0.75 (5)	0.37(5)	1.35 (5)	0.95 (11)	0.76(7)	-0.65 (7)	2.64 (11)	0.63 (4)	1-47 (5)	2.12 (5)	1-39 (5)	1.47 (5)	1.20 (5)	0-96 (5)	0.31 (6)	- 0.04 (9)	2·22 (6)	2.71 (7)	3.78 (12)	5.64 (15)	1.50 (5)	2·84 (18)		2.41(17)	2-41 (17) 4-20 (24)
ſ	D_{33}	2.49 (1)	2.52(1)	4·28 (5)	4.17 (5)	4.42 (5)	3.46(5)	3·96 (5)	3.63 (5)	(2) 20 2		(4) 67.7	4.43 (6)	6.63 (9)	6.61 (9)	3.59, (6)	3.01 (6)	12.94 (24)	12.32 (21)	6.67 (10)	5-57 (11)	3.11(5)	3.69(6)	5.27 (8)	3.26 (6)	3.09(5)	2·85 (5)	3.65(6)	4.92(8)	7-45 (12)	3-06 (6)	3.16 (6)	5.02 (10)	4.85 (10)	3.30(6)	5.96 (28)		2.52 (16)	2·52 (16) 4·74 (28)
слу. Б	B_{22}	2.99 (1)	3.67 (2)	11.59 (10)	5-78 (6)	7-35 (7)	8.42 (8)	5.86 (7)	3.55 (5)	(2) 00 C		4-44 (6)	5.18 (7)	5.30 (9)	2.93 (6)	3-83 (6)	3·22 (6)	9-74 (17)	3.51 (8)	6.26(10)	3-93 (10)	2.99 (5)	4.29 (7)	5-11 (8)	5.32 (8)	4.46 (7)	4.15 (6)	4.84 (7)	5.73 (9)	7-96 (14)	5.34 (8)	6.51 (10)	8-96 (15)	13.61 (23)	3.76(6)	4.63 (24)		5.16 (24)	5·16 (24) 6·30 (30)
n U(v) ispura	B_{11}	2.24 (1)	2.69 (1)	4·24 (5)	4.47 (5)	4.68(5)	7.04(7)	2.54(4)	2.69 (4)		(+) 0/.7	$4 \cdot 10$ (5)	2·82 (5)	2·54 (5)	2.55(5)	3.29(5)	4.88 (7)	4.41 (9)	4.36(9)	4.85(8)	12-34 (23)	2·14 (4)	2.94(5)	3.14 (6)	3.30 (6)	3.07 (5)	2.66(5)	2.77(5)	3.52(7)	5.37 (10)	4.43 (7)	4.47 (7)	8-60 (13)	8-05 (13)	3.53(6)	4.46 (20)		4.33 (18)	4-33 (18) 7-23 (30)
allavited to U(U) at	N	0.16200 (2)	0.34788 (3)	0-01217 (11)	0-35061 (11)	0.29524(11)	-0.02807 (11)	0.10670 (12)	0.26080 (12)		(11) 06007.0	0-05093 (10)	-0.22050 (13)	0.16279 (19)	0.25956 (19)	0.14387 (15)	0.05249 (14)	0.12856(36)	0.33905 (31)	0.16459 (20)	-0.03425(29)	0.00551 (12)	0-02519 (15)	-0.08861 (18)	-0.23926 (15)	-0.13294 (14)	0.41043 (14)	0.32263(15)	0-37364 (19)	0.28802 (25)	0.50656(15)	0.60454 (16)	0.72874 (22)	0-83315 (24)	0.30970 (14)	0.41929		0.4779	0.42729 0.38009
	Ń	0.22633 (1)	0.19216 (3)	0.27310 (12)	0-37299 (10)	0.18145(10)	0-08005 (11)	0.31349 (11)	0.36085 (0)		(6) 00140.0	0.09286 (10)	0.29372 (10)	0.41517 (15)	0-44411 (12)	0.03928 (12)	0.01118 (12)	0.49385 (29)	0.55697 (17)	- 0.03783 (15)	-0.09928 (18)	0.25394(10)	0.30960(12)	0.32725(13)	0.23986 (13)	0.21890 (12)	0.28913 (12)	0.28026 (13)	0.37287 (15)	0.36729 (20)	0.18947 (14)	0.29829(15)	0.28803 (21)	0.39214 (27)	0.05870 (12)	0.03116		0.02100	0.02100
	×	0-41405 (2)	0.60504 (3)	0-54042 (11)	0.42397 (11)	0.27136(11)	0.40737 (13)	0.52382 (11)	(11) 20020 0	0.40019 (11)	(01) 66562-0	0-35651 (12)	0.01663(11)	0.58247 (14)	0-55061 (14)	0-22875 (14)	0.26579 (16)	0-67416 (25)	0.60525 (22)	0.12498 (18)	0.20868 (37)	0.25077 (11)	0.17902 (13)	0.06564 (15)	0.08421 (14)	0-19928 (13)	0.77889 (13)	0-83527 (14)	0-97140 (17)	1-02931 (23)	0.60589 (16)	0-67058 (17)	0.66151 (24)	0.73314 (28)	0.60932 (15)	0.73589		0.71060	0.71969
	Μ																																			0.5			0.5 2.5
		Co	Р	0(5)	0(6)							N(18)	N(34)	CUD							C(14)		(22) (22)	(j)) (j))	(32) (33)	(36) (36)	C(30)	C(42)	C(43)	C(44)	C(51)	C(52)	C(53)	C(54)		C(621)			C(622)

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	$B(\Lambda^2)$	5.64	7-45	7-74	10.32	90-6	80·6	5.70	6-45	8.19	6.94	9-43	9-61	06.6	11-68	06-6	6-26	4.87	7.19	8.19	10.06	10-81	9.64	10-44	10-44
	ý	0.2282 (16)	0.4648 (18)	0.3664 (19)	0.1925 (23)	0-3161 (20)	0.2853 (21)	0.4743(15)	0.5497 (17)	0-6390 (19)	0.5590 (17)	0.7014 (22)	0.7768 (21)	0.7934 (23)	0.9144 (24)	0.8753 (22)	0.2818 (16)	0.2299 (15)	0.4482	0.5009	0.3554	0·3084	0.5616 (21)	0-4661 (22)	0.5484 (22)
	ý	0.2802 (12)	0.3786 (14)	0.4403 (15)	0.3699 (18)	0-4282 (17)	0.3009(16)	0.1550 (12)	0.1430 (13)	0.3289 (15)	0.3481 (14)	0.2564 (18)	0.2316 (17)	0.4428 (18)	0.3823 (19)	0.4189 (18)	0.0052 (13)	0.0564 (11)	0.0833	0.0189	-0.1332	-0.0683	-0.0568 (17)	-0·1819 (18)	0·1429 (18)
	×	0-7740 (16)	1.0358 (18)	0-9525 (18)	0-9616 (22)	1.1099 (21)	1.0534 (21)	0-5105 (16)	0.6522 (17)	0-7635 (19)	0.6280 (18)	0.5728 (22)	0.7056 (22)	0-6951 (24)	0-7300 (25)	0-8320 (22)	0-5206 (16)	0.6082 (14)	0.8170	0.7141	0-6407	0.7486	0-9296 (21)	0.8494 (22)	0-8175 (23)
$\overline{\cdot}$		H(422)	H(431)	H(432)	H(441)	H(442)	H(443)	H(511)	H(512)	H(521)	H(522)	H(531)	H(532)	H(541)	H(542)	H(543)	H(611)	H(612)	H(621)	H(622)	H(631)	H(632)	H(641)	H(642)	H(643)
Table 1 (con	$B({ m \AA}^2)$	7-56	6.72	12.70	11-87	13-99	11.88	9-68	12.10	11-00	11.00	11-00	11-00	11.00	11·00	10-92	13.18	11.33	5.10	6-47	5.92	4.76	4.95	4.84	6.24
	м	0.3365 (18)	-0.0116 (18)	0.1424 (31)	0.2072 (25)	0.0640 (33)	0.2807 (25)	0-3977 (25)	0·3886 (24)	0-1152	0.0745	0.1307	0.2276	0.2683	0.2121	-0-0105 (26)	-0.0286 (36)	-0·1226 (24)	0-1157 (15)	-0.0721 (16)	-0·3394 (16)	-0.1543 (14)	0-4136 (14)	0-4989 (15)	0-3250 (16)
	ý	0.2983 (16)	0.1542 (15)	0.5604 (21)	0.5185 (19)	0.4580 (30)	0.6009 (19)	0-5490 (19)	0.5853 (18)	-0.0223	-0.0825	-0.1151	-0.0875	-0.0272	0.0053	-0·1295 (21)	-0·1330 (25)	-0.0959 (19)	0-3350 (11)	0-3611 (13)	0.2152 (12)	0.1838 (11)	0.3588 (12)	0·2824 (11)	0·2100 (13)
	x	0.3647 (19)	0.4612 (19)	0.6615 (28)	0.7678 (25)	0-6547 (34)	0.5647 (25)	0-5743 (24)	0.7031 (25)	0.0340	0.0376	0.1034	0.1655	0.1620	0-0962	0·2655 (26)	0.1575 (35)	0·1578 (25)	0.2041 (14)	0.0162 (16)	0.0481 (15)	0.2482 (14)	0·7744 (14)	0-8407 (14)	0-8558 (16)
	М									0.5	0.5	0.5	0.5	0.5	0.5										
		H(67)	H(85)	H(111)	H(112)	H(113)	H(121)	H(122)	H(123)	H(131)	H(132)	H(133)	H(134)	H(135)	H(136)	H(141)	H(142)	H(143)	H(321)	H(331)	H(351)	H(361)	H(411)	H(412)	H(421)

Final hydrogen coordinates were established by refinement except as noted.

Initial hydrogen-atom temperature factors were taken as 1.2 times the isotropic equivalent of the adjacent carbon atom. At a later stage they were adjusted according to $\Delta B = -2B\Delta\varrho/\varrho_o$ where $\Delta\varrho$ is the difference density and ϱ_o the observed electron density at the hydrogen position and B is the isotropic temperature factor for the hydrogen atom.

The hydrogen atoms located between O(6) and O(7) and between O(5) and O(8) were initially placed equidistant from the oxygen atoms. Refinement moved each hydrogen atom to a different dimethylglyoxime ligand with an O-H distance of about 1.0 Å. The hydrogen atoms on the methyl group, C(13), failed to refine. Examination of the difference electron-density suggested disorder which was approximated by six equally spaced half-hydrogen atoms.

According to chemical evidence (Schrauzer, 1970) the pyridine ligand is attached to cobalt through a Co-C bond rather than the more common Co-N link. The Co-C attachment was confirmed by refining a model with pyridine atoms C(31) and N(34) interchanged. The arrangement with Co-C pyridine coordination gave a smaller residual electron-density difference at the two atomic sites, an R = 0.004 lower, and reasonable thermal parameters for the two atoms, clearly supporting the chemical evidence.

After further refinement of all heavy-atom parameters and hydrogen atom coordinates, an extinction correction (Zachariasen, 1968) was made. An extinction parameter, r^* , was calculated for each of the 25 most intense reflections using an average value of \overline{T} (0.04392 cm), the effective crystal thickness, F_o (uncorrected) and F_c for each reflection. The average value of r^* , 1.663 × 10⁻⁵ was then used to correct all the F_o and the corrected values were used in all subsequent calculations.

A difference synthesis indicated disorder in the region of C(62) and C(63) along one butyl chain. As an expedient, the large apparent motion perpendicular to the chain was approximated according to Kartha & Ahmed (1960) by replacing C(62) and C(63) with half atoms separated by about 0.25 Å. The anisotropic thermal parameters but not the coordinates were refined in subsequent least-squares calculations. Hydrogen atom positions were calculated from the average positions of the half atoms and were not subsequently refined.

The atomic scattering factors for neutral Co, P, O, N and C tabulated by Cromer & Mann (1968) and H scattering factors calculated by Stewart, Davidson & Simpson (1965) were used. Co and P were corrected for the real part of the anomalous dispersion.

The final cycles of full-matrix least-squares were performed on blocks of parameters corresponding to groups of atoms (butyl chains, dimethylglyoxime ligands and pyridine) with the scale factor, Co and P included in each group. This reduced R to 4.15% for

Table 2. Observed and calculated structure factors

The running index is l. F_o and F_c have been multiplied by 10. The extinction correction has been applied to F_o and all values changed by more than 0.5% are marked with an X.

-18.2 10 7 -18.3 8 51	L 7 84 -11 8 67 -60 9 26 -17 L 10 12 16 -65 11 5 1 -11 12 16	9 60 -56 10 39 -37 81 6 -10 12 24 19 13 51 48	-15,1,1 1 8 -1 2 42 -37 3 20 -21 4 46 -48	9 8 11 10 22 19 11 53 53 12 5 3 13 39 -39	6 76 -79 7 87 -91 8 30 -27 9 121 114 10 109 103	12 12 -12 13 14 20 14 21 19 -14.12.L	15 19 -22 16 4 -7 -13,-1,t 1 88 -05	13 55 -50 14 11 -13 15 40 35 -13,9+L	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	-12.4.L 1 140 140	8 32 -34 9 55 -55 10 23 24 11 31 28 12 4 -3	9 56 55 10 119 -120 11 191 -199 12 5 1 13 21 17	2 69 -70 3 57 -53 4 88 95 5 22 21 6 246 249
10 27 11 7 -18.4	26 9 -17,11,L 5 18 -14 L 6 17 -19 -60 7 39 -33	-16,8,1 1 12 -16 2 77 -73 3 0 0	6 105 101 7 6 6 8 19 -20 9 87 -87 10 60 -60	-15+12+L 1 30 -39 2 56 52 3 64 60 4 11 10	12 14 -8 13 55 -54 14 17 -14 15 53 53 16 45 40	2 26 -28 1 54 -53 4 68 -70 5 57 -53 6 65 69	3 86 82 6 16 -14 5 59 -04 6 13 -11 7 177 -179	2 22 18 3 96 98 4 24 -25 5 194 -195 6 35 -33	6 84 -85 7 39 35 8 68 65 9 41 43	2 91 -92 3 97 -97 4 38 -37 5 45 49 6 8 13 7 103 90	-12,15,L 1 13 -1 2 0 -9 3 10 19 4 53 55	15 20 23 16 34 -35 17 58 -58 -11,-2,L	8 119 -124 9 33 -26 10 5 -2 11 4 1 12 76 76
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7 35 8 49 9 8 10 30 11 34	32 3 30 -28 47 4 3 6 -1 5 23 -23 -24 6 67 -64 -30 7 59 55	11 31 -32 17 17 -19 13 23 -22 14 19 14 15 51 43	2 44 -42 3 73 -67 4 5 -10 5 30 29 6 63 61	1 42 -41 2 35 -32 3 69 -64 4 33 29 5 61 54	5 13 8 6 84 -88 7 13 -15 8 0 -9 9 53 52	10 29 -27 11 34 -28 12 39 37 13 80 84 14 6 5	8 48 -43 9 24 -24 10 43 42 11 55 54 12 21 13	10 13 -6 11 24 29 12 4 6 13 13 -11 14 21 -23	12 132 -128 13 86 -85 14 41 39 15 16 17 16 45 43	-12.13.0 1 21 17 2 31 32 3 7 -11 4 22 -20	4 74 77 5 117 -110 6 92 -91 7 25 25 8 25 26	16 13 12 17 13 -18 -11+6+L 1 25% 253	6 9 -11 7 16 -17 8 50 -48 -11+18+L
12 10 13 14 -17,9, 4 26 5 28	-10 0 76 78 10 9 6 7 20 31 -26 11 52 -47 -26 12 45 -46 27 13 21 -27	-15,0.6 1 +5 +6 2 26 24 3 0 -5 4 51 -4#	8 42 -39 9 43 -38 10 38 -33 11 0 5 12 61 54	• •2 -42 7 53 -53 8 146 -147 9 60 -63 10 42 42 11 27 30	10 00 05 11 3 -11 12 37 -38 13 11 -10 14 6 -6	17 12 -15 10 31 -21 -13,-2,L 1 5 6 2 116 -116	13 18 19 14 41 -34 15 25 -19 16 17 17	-12,-7,L 1 35 -33 2 19 14 3 51 49 6 62 34	17 26 27 -12,3,1 1 56 55 2 196 192 3 47 47	5 49 40 6 43 41 7 62 58 8 28 24 9 62 -63 6 43 -1	161 163 10 102 102 11 76 -80 12 61 -60 13 24 20	2 213 213 3 10° -102 4 23 -16 5 158 -159 6 18 -15 7 327 15	1 6 2 2 8 7 3 58 57 4 20 21 5 22 -21
6 37 7 52 6 39 10 20	37 14 28 24 50 15 12 13 -4 -40 -16,7,1 -18 1 79 74	5 95 -91 6 12 8 7 77 78 8 69 69 9 24 17	13 0 -3 14 15 -12 -15-11-1 1 53 -51	12 40 34 13 22 -19 14 67 -69 15 26 -27 16 20 19	$\begin{array}{c} -16.11.1\\ 1 & 21 & 16\\ 2 & 66 & 61\\ 3 & 12 & 12\\ 4 & 13 & -16 \end{array}$	3 53 -52 6 81 78 5 24 17 6 95 97 7 6 5	1 C -8 2 4 -2 3 36 36 4 161 156 5 34 -32	5 0 2 6 57 -55 7 33 -32 8 0 5 9 17 22	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1 6 11 2 18 12 3 28 25	15 21 24 16 47 42 -11,-3,L 1 99 97	8 105 100 9 21 -15 10 37 -22 11 81 -72 12 29 -24	-10,-12,L 6 17 11 5 32 28 6 36 32
12 10 -17,10 5 33	13 3 12 -11 13 3 12 -11 4 8 9 14 5 41 -43 52 6 26 25 32 7 58 53	11 35 -39 12 18 17 13 11 2 14 46 94 15 9 10	3 20 21 4 75 77 5 61 58 6 45 40 7 74 -72	-14,1,1 1 9 6 2 40 -40 3 28 28 4 141 134		9 61 -59 10 22 22 11 11 13 12 35 30 13 25 -31	6 144 -141 7 37 -35 8 42 -37 9 78 71 10 167 104 11 12 -17	10 28 31 11 11 7 12 38 -41 13 10 -11 14 13 -14 15 28 20	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	L 16 -18 2 40 -40 3 47 -49 4 2 4 5 68 82 6 4 -3	3 10 -0 3 10 -70 5 132 -128 6 17 10 7 53 53	13 78 75 14 47 38 15 10 -10 16 15 -11 -11.7.L	7 5 -18 8 72 -74 9 47 -45 10 10 7 11 28 26
• •	2 8 28 -27	16 29 -28	0 13 -2	5 12 10	11 59 -03	24 37 -34	12 19 -17		15 28 25	7 40 -35	8 73 69	1 83 82	

1900000	e muldrending datum interverding barrending burnending burnending burnending burnending and medanizziel Address 2000-2014 - 2015 2000-2014 - 2014 - 2015 2014-2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 2014-2014 - 201 2014 - 201 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014 - 2014		<pre> Contractions</pre>	21, rutirelstration internation of the second of the second and the second second of the	ини поментных поментных оним оним оним вольных поментных поментных поментных поментных поментных поментных поме поментных изволять польках польках полькаха, поментных поментных поментных полькахахах поментных поментных помен поментных поментных п поментных поментных по поментных поме поментных поментных поментных поментных поментных поментных поментных поментных поментн поментных поментных по	1999		
	2.2	- 100	$\begin{array}{cccccccccccccccccccccccccccccccccccc$					7.1 . 139 . 144 . 265 . 145 . 159 . 144 . 265 . 159 . 144 . 265 . 159 . 144 . 265 . 149 . 149

Table 2 (cont.)

-1,-1,-1 2 262 -270 3 68 -64 4 84 -74 5 233 236	$\begin{array}{c} -1, 11 + 1 \\ 1 & 45 & 43 \\ 2 & 101 & -97 \\ 3 & 209 & -212 \\ 4 & 23 & 16 \end{array}$	0,-10,L 1 46 47 2 395 397 3 96 97 4 323 -328	6 111 112 7 325 -329 8 219 -224 9 96 -93 10 1 -5	0,14,1 0 44 43 1 38 -34 2 152 -150 3 57 -56	11 26 23 12 27 25 13 72 -70	10 130 -139 11 60 56 12 57 59 13 10 13	0 14 -16 2+-19+1 0 11 -39	5 274 275 4 42 -39 5 143 -137 6 36 -36 7 216 -217	2,6,L 0 40 36 1 217 -222 2 68 -69 3 136 -138	9 19 -10 9 54 -54 10 25 -28	10 53 -55 11 72 -72 12 57 -53 3,-1,-L	6 00 -55 5 7 13 6 33 32 3,13,1	6 116 113 7 63 -64 8 32 -37
7 202 -204 8 91 85 9 49 -48 10 61 57 11 156 152	6 56 57 7 47 42 8 86 -87 9 54 -53 10 1 -4	5 111 -113 6 34 32 7 14 -12 8 155 155 9 16 12 10 91 -89	12 51 -55 13 37 -38 14 17 -11 0,2,1	5 6 -1 6 0 2 7 0 -7 0,15,L	1 86 -91 2 195 203 3 268 278 4 34 31 5 192 -190	0 487K 485 1 91 85 2 508X -488 3 639X -628 4 80 -77	2 15 19 3 20 19 4 16 11 5 28 -27	9 141 144 10 54 49 11 46 43 12 43 -37 13 71 -67	5 230 228 6 50 -56 7 34 -28 8 12 -6 9 98 -95	1 127 -122 2 84 -77 3 54 -54 4 126 122 5 210 205	1 24 31 2 326 -332 3 352 -364 4 126 126 5 17 11	1 73 -76 2 21 -21 3 21 -20 4 12 11 5 71 89	10 27 -28 11 44 41 12 37 35 46.L
13 107 -104 14 53 -51 15 2 -6 -1.0.L	-1,12,1 1 106 -113 2 171 -169 3 58 59 4 128 134	12 26 -27 13 23 -20 0,-9,L 1 260 262	1 230 -213 2 10 -1 3 91 91 4 124 144 5 29 -30	1 60 -55 2 19 -22 3 39 40 4 62 42 5 21 -16	7 205 -205 8 199 197 9 156 156 10 26 -26 11 40 -37	6 166 154 7 2 6 8 64 -63 9 201 -200 10 38 -42	0 18 18 1 35 36 2 40 39 3 26 32 4 17 -17	2,-5,L 0 19 14 1 363 -353 2 346 355 3 180 181	2+7+L 0 491 -496 1 29 -16	7 108 -108 8 41 -42 9 37 -35 10 49 47 11 32 31	7 185 180 8 186 -186 9 109 -112 10 57 -57 11 32 -33	3,14,1 0 94 -91 1 8 -13 2 12 4 3 0 -3	1 316 -320 2 93 85 3 94 92 4 262 259 5 193 199
1 119 -124 2 608 × 816 3 487 × -488 4 15 17 5 67 -80 6 452 -45	5 20 26 6 13 -7 7 39 -44 8 40 -46 9 11 3 10 29 28	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	6 307 -357 7 214 -218 6 51 -59 9 64 65 10 56 49 11 21 18	6 5 -10 0.16.L 0 3 -3 1 7 -8 2 32 32	12 42 -85 13 24 -26 1,-7,L 0 89 100 1 175 181	11 03 02 12 40 40 13 23 27 1.4.L 0 36 -41	5 57 -52 6 8 2 7 36 36 2+-17+1 0 88 82	4 168 -165 5 48 -57 6 175 -173 7 78 74 8 248 243 9 21 20	2 123 -130 3 24 -30 4 170 173 5 16 -23 6 115 -116 7 16 -11	3,-12,L 0 154 -150 1 24 -23 2 148 148 3 160 162	12 51 47 3.0.1 0 227 223 1 05 -74 2 344 -358	4 57 54 3,15,L 0 34 -37 1 43 40 2 14 9	6 119 -118 7 109 -111 8 51 -47 9 50 -45 10 52 51 11 45 40
7 14 20 8 207 209 9 119 121 10 3 0 11 66 -66 12 127 -128	-1,13+L 1 52 -51 2 27 31 3 194 198 4 67 70	6 56 56 9 146 -145 10 113 -117 11 49 52 12 14 14 13 23 16	12 83 -76 13 18 -11 14 8 -9 0,3,L 0 327 319	3 75 73 4 0 0 5 37 -36 0.17.L 0 11	2 37 40 3 147 -133 4 352 -356 5 117 -120 6 48 45 7 184 185	1 361 -355 2 443 -418 3 5 5 4 210 206 5 179 179 6 200 -208	1 45 46 2 46 -47 3 37 -34 4 49 -49 5 11 13 6 69 68	10 37 -37 11 11 11 12 48 -44 13 27 28 21-4-L	8 11 -9 9 20 21 10 72 70 2+8-L 0 18C -176	4 67 66 5 53 -51 6 118 -115 7 33 35 8 24 25 9 37 37	3 59 -50 4 334 329 5 224 223 6 78 81 7 155 -160 8 233 -232	3 23 20 3.16.L 0 57 54 1 38 41	12 39 -39 4,-5,L 0 208 -216 1 145 -130 2 128 130
13 12 11 14 17 19 15 23 24	5 49 -49 6 16 -17 7 16 -13 8 8 6 9 33 32	14 9 3 0,-8,L 1 34 -19 2 175 -175 1 112 -118	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1 20 13 2 24 22 3 27 -26	8 127 130 9 77 -76 10 147 -140 11 91 -95 12 66 -64	7 112 -108 8 173 -171 9 59 -56 10 115 118 11 33 36 12 29 -28	7 10 -3 8 5 -10 21-16-L 0 48 49 1 105 -105	0 175 -172 1 201 204 2 344 -331 3 0503 -054 4 17 -15	1 206 208 2 35 -37 3 105 99 4 30 -33 5 193 -198	10 60 56 11 46 -46 311.L 0 39 46	9 4 -1 10 67 65 11 50 46 12 23 20	4,-19,L 0 23 -20 1 16 -13 2 29 -31 3 34 35	3 507 508 4 277 269 5 63 -57 6 169 -168 7 28 23
2 G07X 916 3 555X -543 4 171 -175 5 338 -346 6 81 75	-1,14+L 1 34 33 2 109 106 3 11 11 4 98 -95	4 205 208 5 266 267 6 14 10 7 0 -12 8 120 -111	1 274 279 6 121 123 9 25 25 10 14 20 11 86 -87	1 3 4 1,-19,1 0 30 31 1 15 -14	14 42 41 1,-6,L 0 375 366 1 35 40	1,5,L 0 387 -382 1 24 -32 2 149 162	2 69 -68 3 21 -20 4 45 -50 5 44 40 6 0 -3	6 55 -52 7 110 115 8 31 -7 9 120 -116 10 19 -5	7 40 39 8 53 51 9 74 74 10 21 19	2 124 124 3 39 36 4 64 -62 5 140 -136 0 13 -15	0 195 -199 1 44 -51 2 51 49 3 454 452 4 369 364	5 4 8 418.1 0 35 -30 1 20 -26	9 21 20 10 58 56 11 52 -50 12 65 -66
7 332 321 8 68 7G 9 12 11 10 99 -96 11 122 -116 12 30 -33	5 11 -18 6 33 31 7 0 -6 8 26 23 -1.15.L	9 101 -104 10 102 105 11 100 103 12 23 20 13 11 21 14 36 -13	0,4,1 0 282 279 1 673x 656	2 10 -21 3 27 -25 4 4 -10 5 8 11 1,-18,L	2 106 103 3 140 -134 4 256 -237 5 296 304 6 107 101 7 184 186	5 124 129 4 55 57 5 132 -130 6 260 -262 7 18 -11 8 5 -3	7 57 -56 8 32 -28 9 10 -13 2+-15+1 0 128 -129	11 10 22 12 0 C 13 122 120 23.L 0 53 -54	2 49.L 0 262 260 1 89 85 2 66 69 3 8 1 4 197 -199	7 140 140 8 55 57 9 54 54 10 28 -29 11 93 -92 12 16 13	5 15 9 6 34 -21 7 141 -142 8 40 39 9 133 133 10 83 82	2 38 36 3 64 60 4 5 -11 5 28 -29 6 4 -5	4,-4,L 0 47 -54 2 554X 570 2 922K 920 3 151 147 4 350 -344
13 56 48 14 29 22 -1.2.L 1 140 141 2 273 -265	1 24 24 2 6 C 3 66 -67 4 61 -62 5 25 26 6 21 27	0+-7+L 1 80 -93 2 99 -91 3 84 86 4 455 474	2 135 -128 3 472 -470 4 270 -256 5 89 -102 6 232 229 7 222 225	0 12 -17 1 39 -36 2 11 2 3 19 21 4 36 34 5 32 26	6 167 -165 9 232 -237 10 31 -33 11 20 26 12 52 53 13 72 73	9 88 87 10 103 102 11 44 -43 12 71 -72 1+6.L	1 93 -92 2 77 78 3 19 20 4 7 -11 5 13 11 6 93 -90	1 330 326 2 1497x-1465 3 684¥ -662 4 252 251 5 146 -145 6 200 202	5 69 -72 6 83 81 7 61 62 8 42 41 9 0 -3	3,-10,L 0 258 265 1 30 24 2 162 -166 3 78 -79	11 28 31 12 42 -42 3,2,t 0 438 431 1 255 -252	4,-17,L 0 24 20 1 48 47 2 61 60 3 46 -50 4 66 -62	5 241 -243 6 40 -41 7 158 156 8 69 49 9 94 90 10 9 -6
3 568 X -562 4 271 -256 5 31 -26 6 309 307 7 244 229	7 7 13 -1,10,4L 1 22 -18 2 15 -11 3 13 -5	5 142 139 6 306 -316 7 172 -175 8 136 -136 9 138 140 10 236 239	8 33 -29 9 6 3 10 119 -121 11 82 -84 12 104 100	6 10 -20 7 55 -55	14 34 -37 15.L 0 334 -320 1 20 -1 3 115 -119	0 18 18 1 359 304 2 125 130 3 50 45 4 55 -58 5 241 -251	7 11 -10 6 16 18 9 28 28 10 28 26	7 42 -45 8 281 -282 9 55 -61 10 75 73 11 42 44	2,10,L 0 58 61 1 9 9 2 129 -130 3 112 -112	4 137 -139 5 31 -33 6 152 147 7 73 72 8 8 -13	2 297 301 3 270 277 4 351 -356 5 298 -299 6 86 -80 7 51 51	5 9 13 6 12 13 7 11 7	11 128 -128 12 13 6 4,-3,L 0 667X 644
9 194 -132 10 21 -17 11 27 -27 12 40 34 13 0 -3	4 35 38 5 65 68 6 8 4 -1,17,L	11 15 -11 12 12 -7 13 49 -48 14 38 -14	0.5+L 0 161 170 1 156 -144 2 779x -758	2 79 76 3 63 61 4 30 32 5 6 6 6 62 -65	3 218 202 4 270 278 5 207 207 6 20 -20 7 67 -69	6 37 42 7 96 88 8 8 -2 9 38 37 10 37 -44	0 110 -115 1 114 119 2 72 71 3 12 13 4 43 43	13 50 46 22.L 0 668×-657 1 2376×-2525	5 107 110 6 116 115 7 14 -9 8 8 -6 9 17 -18	10 88 -87 11 3 -5 12 85 83	8 172 170 9 64 64 10 48 -51 11 46 -47 12 54 -55	1 48 47 2 14 12 3 63 -61 4 8 -5 5 57 56	2 116 -127 3 434 -440 4 212 -217 5 36 -38 6 193 195
-1.3.1 1.7478-713 2.179-174 3.265-269	-1,18+L	1 216 205 2 228 -203 3 48 47 4 123 -123 5 558 -568	4 121 -115 5 50 39 6 357 357 7 100 -107 6 129 -131	8 44 43 9 30 34 110.L 0 5 -7	9 15 9 10 93 92 11 10 37 12 38 37 13 31 -30	12 10 12 1,7,1 0 400 401 1 12 -4	6 113 -111 7 61 60 8 67 67 9 39 40 10 6 5	3 638# 671 4 240 241 5 109 -114 6 151 150 7 168 -162	2.11+L 0 41 -44 1 111 -110 2 88 -86 3 15 19	1 298 -295 2 281 -284 3 107 99 4 71 -81 5 199 204	3.3.L 0 37 -10 1 241 -231 2 173 174 3 386 -389	7 43 44 8 2 0 4,-15,L 0 47 -47	8 80 -79 9 48 -45 10 82 -85 11 31 -31 12 77 78
4 297 241 5 125 130 6 50 87 7 376 -378 8 140 -132 9 45 45	1 15 16 2 26 26 3 28 23 019.1 2 42 40	6 237 -243 7 80 -86 8 89 83 9 161 165 10 47 -45 11 117 -119	9 33 -31 10 95 -93 11 98 102 12 90 92 13 27 -28	1 108 107 2 79 79 3 54 -54 4 42 -42 5 27 -23 6 28 -26	14 100 -95 14+L 0 420X -400 1 384 381 2 91 -80	2 169 -164 3 88 -94 4 83 -86 5 23 17 6 130 133 7 5 -1	11 28 -26 2,-13,1 C 91 9C 1 126 121 2 20 -16	8 156 -156 9 130 132 10 131 134 11 24 27 12 36 40 13 68 -62	4 04 93 5 97 97 6 25 -27 7 19 -20 8 7 13	6 165 165 7 200 -200 8 139 -138 9 67 -68 10 16 -15 11 110 109	4 227 -228 5 36 37 6 101 -102 7 123 119 8 46 42 9 82 -80	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	42.L 0 217 216 1 692X -677 2 344 -352 3 151 152
10 12 -4 11 27 19 12 26 26 13 86 -85 14 51 -48	3 6 4 4 14 -1t 0,-18,L 1 30 30 2 6 -13	12 57 -57 13 71 -74 14 50 53 05.1 1 311 -125	0.6+L 0 2+3 -225 1 700× -691 2 189 184 3 121 121 4 15 14	7 64 61 6 18 11 9 10 -12 10 28 -25	3 210 230 4 108 120 5 214 -214 6 105 -109 7 258 -272 8 59 63	8 38 -37 9 61 -56 10 62 -63 11 0 -1	3 116 -119 4 180 -160 5 160 -159 6 96 95 7 103 102 8 5 -5	21.L 0 718 × -097 1 30 -27 2 053 × 058 3 249 254	2.12.L 0 119 -118 1 1 0 2 83 83 3 83 79 4 37 33	17 61 61 3+-8+L 0 443 -440 1 571 -568 2 168 167	10 59 -56 11 32 -31 3,4,1 0 170 -147 1 103 102	7 46 -47 8 63 -65 9 51 -49 4,-14,L 0 121 -119	4 119 -119 5 120 119 6 76 77 7 199 -199 8 86 -89 9 91 -92
-1.4.6 1 255 -251 2 482 X 463 3 403 411 4 362 220 5 120 101	3 39 -40 4 31 -25 5 29 -75 6 11 9 7 47 47	2 142 -129 3 89 -92 4 561 x -567 5 217 -219 6 231 234 7 206 212	5 118 120 6 115 -116 7 193 -190 8 28 -19 9 11 16 10 101	C 63 61 1 74 74 2 90 -91 3 77 -76 4 3 1 5 35 -35	9 196 187 10 7 -2 11 80 -77 12 35 -32 13 87 -83	0 294 102 1 430 -432 2 125 -124 3 12 -3 4 63 -59 5 143 139	9 5 -50 10 32 -30 11 47 -46 21-12-1 0 70 67	4 14 13 5 321 - 326 6 178 - 173 7 122 - 126 8 230 232 9 226 225	3 45 -41 6 6C -63 7 27 25 2.13.L	3 63 66 4 121 -122 5 180 176 6 207 -202 7 138 -134 8 25 27	2 302 -314 3 208 -213 4 233 241 5 152 153 6 109 111 7 26 30	1 27 -29 2 51 -52 3 53 49 4 153 152 5 64 -63 6 122 -123	10 15 -17 11 77 79 12 23 19 4,-1+L 0 194 -202
6 167 -161 7 315 -324 8 120 115 9 101 100 10 74 -68	0+-17+1 1 3 -5 2 62 -62 3 33 -30 4 62 61 5 33	8 139 139 9 115 -118 10 230 -233 11 37 -35 12 31 28	11 89 89 12 34 - 34 0,7,1 0 273 - 282	0 28 23 7 26 21 8 57 -57 9 50 -50 10 40 -38	13.L 0 549X 539 1 353 -345 2 143 148 3 180 183	6 17 23 7 73 -68 8 39 -32 9 41 -35 10 24 30	1 78 -84 2 135 -133 3 266 -27C 4 21 -17 5 121 122 0 175	10 47 -42 11 38 -34 12 13 -8 13 40 -33 2.0.1	1 151 150 2 47 46 3 2 1 4 6 -7 5 56 -55 6 11 6	9 70 68 10 62 63 11 42 44 12 60 -58	8 105 -104 9 25 -22 10 41 44 11 46 44	7 90 -86 8 10 -8 9 40 36 10 21 17 413-1	1 322 -333 2 19 -26 3 176 188 4 32 34 5 160 157 6 295 -300
12 63 -84 13 71 -72 14 32 30	6 44 44 7 6 C 5 59 -55 9 34 -27	14 92 87 04.1 1 4 -6 2 119 -120	2 275 284 3 122 124 4 88 88 5 9 -2 6 161 -161	114.L 0 39 38 1 63 -64 2 121 -119	4 466 -478 5 98 -103 6 64 60 7 158 -157 8 95 94	1.9.1 0 307 -294 1 124 -124 2 150 151	7 28 -25 8 92 -92 9 41 -37 10 35 -32 11 21 19	0 69 -76 1 328 33C 2 76 -66 3 45 -5C 4 269 -282 5 100 -282	2+14+L 0 125 123 1 37 38 2 26 -29	0 548 x - 546 1 24C 232 2 273 264 3 110 -111 4 69 -65	0 39 -35 1 40 -28 2 147 +161 3 238 233 4 203 205	0 67 72 1 79 82 2 80 79 3 52 48 4 100 -102	7 275 -277 8 51 52 9 68 69 10 65 62 11 33 30
2 875× 840 3 40 45 4 278 -279 5 50 -51 6 190 -185	1 71 -68 2 33 -28 3 81 77 4 60 6C 5 17 17	4 5 -7 5 443 450 6 91 84 7 177 178 8 11 4	8 49 50 9 72 65 10 44 43 11 14 -33 12 46 -47	4 42 42 5 16 -21 6 41 40 7 73 -69 8 44 -38	10 60 -66 11 66 -00 12 20 -14 13 11 -3 14 89 87	4 67 68 5 16 12 6 144 -140 7 70 -69 8 16 19	2,-11,1 0 13 7 1 271 -273 2 52 -43	6 46 41 7 28 -24 8 190 194 9 32 -28 10 133 -134	4 31 -27 5 2 5 2,15,1 0 36 35	6 271 -267 7 156 155 8 129 130 9 86 84 10 32 30	6 91 91 7 39 -33 8 53 -49 9 61 57 10 60 54	6 28 30 7 17 12 8 61 56 9 31 27 10 32 -33	4.0.L 0 92 -98 1 110 108 2 319 318
8 285 270 9 44 -41 10 47 -48 11 57 -61 12 105 -127	6 10 16 7 73 -71 8 31 -26 9 66 67 10 53 51	V 101 - 47 10 33 34 11 85 78 12 69 70 13 53 54 14 24 -23	0+8+L 0 96 -86 1 279 275 2 177 184 3 192 -192	10 16 14 11 31 27	12.L 0 791x 754 1 276 206 2 180 176 3 681X -640	10 40 39 1,10+L 0 154 -159 1 122 121	4 162 163 5 95 91 6 21 -18 7 112 -109 8 43 44	12 33 -31 13 31 28 2.1.1 0 775 X - 771	2 22 -22 3 2 0 4 0 10 2.16.L	11 34 -32 12 60 -58 3+-6+L 0 162 166 1 626# 621	3.6.L 0 21 -14 1 220 -219 2 228 234	4,-12,L 0 195 189 1 134 136 2 22 28 3 12 -3	5 67 -64 5 83 -78 6 259 -260 7 95 97 8 106 104
-1.6.t -1.6.t 1.870x 845 2.257 -20C 3.103 -174	0,-15,L 1 18 -23 2 95 92 3 68 68 4 37 -35 5 30 -38	0,-3,L 1 195 175 2 21 -30 3 4 25 4 178 189	4 23 -27 5 26 -20 6 11 10 7 132 126 8 69 66 9 10 -3	1 07 -08 2 58 56 3 125 124 4 58 63 5 12 8 6 111 -107	4 348 -388 5 369 360 6 225 237 7 136 137 8 114 114 9 220 -218	2 158 162 3 89 87 4 33 -31 5 98 -107 6 86 -93 7 54 51	9 34 37 10 23 22 11 65 67 12 24 -24 7,-10,1	1 27 32 2 60 61 3 432 -432 4 130 -143 5 458 476 6 267 267	0 24 -24 1 54 -62 2 14 12 3 19 22 2.17.L	2 125 -119 3 59 -69 4 83 67 5 97 -102 6 129 121 7 174 173	3 221 219 4 81 -86 5 122 -125 6 12 -12 7 34 -34 6 37 37	4 135 -135 5 39 40 6 112 114 7 25 25 8 64 64 9 70 -71	0 62 63 10 15 17 11 57 -55 4.1.L 0 141 -118
4 40 -39 5 55 -54 6 101 92 7 329 333 8 26 19 9 108 -111	6 20 -19 7 37 -31 8 52 49 9 47 44 10 5 -2 11 29 -20	5 484 501 6 344 -355 7 174 -167 8 143 -145 9 23 -25 10 142 138	10 81 -81 11 33 -34 0.9.1 0 169 159 1 145 149	7 112 -112 8 44 44 9 63 61 10 27 26 11 22 20 12 28 -29	10 95 -91 11 92 93 12 79 78 13 72 71 14 50 49	8 47 48 9 17 20 10 17 16 1,11,L 0 101 102	0 164 -172 1 12 -21 2 264 260 3 80 73 4 16 15 5 17 -15	7 146 152 8 148 -145 9 174 -175 10 35 33 11 57 57 12 14 16	0 31 -29 1 32 28 3+-19+L 0 34 30 1 5 8	8 10 10 9 29 -28 10 29 -27 11 18 -12 12 46 44 13 44 41	9 70 65 10 20 -23 3+7+L 0 228 224 1 248 245	10 63 -62 11 28 26 4+-11+L 0 7 2 1 107 -105	1 191 187 2 102 100 3 276 -282 4 357 -357 5 168 -172 6 62 62
10 10 -30 11 47 -45 12 29 27 13 69 68	014.L 1 34 28 2 53 46 3 27 -26 4 88 -85	11 62 56 12 7C -70 13 68 -69 14 88 -87	2 04 -61 3 174 -108 4 45 47 5 12 5 6 78 77 7 76 9	1,-12,-L 0 91 -97 1 53 59 2 210 208	1,-1,L 0 294,X 287 1 410X -400 2 1314X-1293 3 365 -365 4 273 225	1 130 134 2 19 -13 3 53 -01 4 109 -111 5 38 -37 6 81 63	6 204 -204 7 41 -41 8 151 151 9 80 79 10 40 40	13 42 35 2,2,1 0 189 -194 1 48 43 2 244 -247	2 7 2 3 15 -15 4 54 -52 5 12 10	3,-5,L 0 215 217 1 222 -215 2 135 -145 3 73 -74	2 93 93 3 99 -96 4 136 -135 5 42 -39 6 49 47 7 33 32	2 153 -151 3 29 -25 4 4 0 5 143 139 6 15 -12 7 26 -28	7 223 226 8 17 13 9 71 -72 10 64 -65 11 67 -67
1 196 194 2 280 -290 3 34 -34 4 204 202 5 84 87	5 37 41 6 12 -14 7 64 58 8 56 56 9 37 -37	1 46 59 2 546# 545 3 19 14 4 33 -31 5 209 -200	8 66 -62 9 62 -58 10 31 -31 11 4 -5	4 121 -124 5 208 -210 6 147 -144 7 22 23 8 92 89	5 297 305 6 65 65 7 40 37 6 17 11 9 126 -122	7 91 93 8 16 -17 9 4 -1	12 102 -102 2+-9+1 0 76 -78 1 249 252	3 41 45 4 570 550 5 201 202 6 12 -2 7 59 62	0 9 -2 1 16 -14 2 46 -48 3 43 -42 4 34 33	4 118 123 5 98 98 6 220 221 7 76 -71 8 121 -122	8 78 79 9 5 -8 10 86 -81 3.8-L	8 63 -64 9 121 -120 10 12 1 11 81 76	4.2.L 0 22 33 1 46 43 2 424 -436 3 156 -168
7 32 -30 8 189 -194 9 42 -46 10 6 3 11 55 57	11 33 -32 12 7 -17 0,-13,L 1 31 32	7 52 47 8 131 -128 9 45 -45 10 33 23 11 115 -116	0 77 82 1 62 -67 2 140 -139 3 39 44 4 117 124	10 16 -16 11 14 -13 12 54 -56	11 160 157 12 18 17 13 40 42 14 21 -16	1 47 -45 2 119 -119 3 57 -55 4 4 -1 5 57 57	3 127 -130 4 34 -41 5 93 -95 6 151 -156 7 275 276	9 6 -4 10 165 103 11 41 39 12 31 32	6 13 -2 7 5 7 3,-17,1 0 87 -86	10 35 -31 11 6 -2 12 83 80 13 40 -37	1 93 101 2 12 -9 3 137 -137 4 65 -67 5 114 119	0 257 -256 1 82 -79 2 123 -123 3 29 31 4 130 130	5 58 -64 6 74 73 7 40 -43 8 100 -99 9 71 -69
12 51 49 13 20 -22 -1,6,1 1 192 -192 2 123 -116	2 18 -16 3 50 -50 4 13 9 5 101 100 6 10 11 7 53 51	12 142 -141 13 53 -58 14 10 -13 0,-1,L 1 1076 x 1064	5 14 19 7 107 -110 8 96 -97 9 10 -10 10 0 6	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1,0,6 0 1215X-1217 1 376X -369 2 482X -483 3 142 140 4 337 347	7 31 -27 8 28 -24 1.13.L 0 44 -47	6 108 100 9 9 -8 10 42 -40 11 66 -72 12 23 -23 13 61 63	0 332 314 1 74 68 2 32 -33 3 658 × 663 4 333 323	1 40 -38 2 53 -56 3 12 6 4 62 60 5 7 -8 6 47 -44	0 126 127 1 861X -842 2 299 -296 3 46 18 4 495 487	8 9 -13 9 66 -69 3,9,L	5 27 31 6 187 -186 7 86 -91 8 126 -128 9 20 16 10 106 106	10 30 -37 11 9 11 4,3,1 0 257 252 1 330 -351
3 247 252 4 181 180 5 59 -56 6 54 50 7 143 -135 8 51 -51	8 43 -40 9 47 -48 10 7 11 11 18 16 12 13 8	2 498 x 492 3 111 118 4 322 307 5 389 -375 6 158 154 7 322 326	0,11+1 0 41 -33 1 207 -204 2 114 118 3 176 184	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	5 39 -26 6 347 -350 7 240 -232 8 156 -159 9 105 104 10 208 208	$ \begin{array}{rrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrrr$	2+-8+L 0 245 249 1 146 147 2 208 -215 3 69 -75	5 205 -201 6 121 -121 7 170 -169 8 36 37 9 151 154 10 46 43	7 8 9 8 21 -16 3,-16,2 0 64 -66 1 65 67	5 169 159 6 73 -73 7 203 -205 8 58 -62 9 64 63 10 11 12	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	11 26 27 4,-9,L 0 340 -335 1 335 327 2 87 94	2 89 -63 3 232 226 4 126 133 5 112 113 6 10 12 7 177 -176
9 71 72 10 59 58 11 31 28 12 15 -15	0,-12,L 1 00 56 2 83 -85 3 43 47 4 218 217 5 109 104	8 65 62 9 24 22 10 95 -94 11 183 -185 12 30 31 13 21 23	4 31 33 5 14 -17 6 95 -97 7 96 -97 8 23 24 9 15 14	12 2 0 13 83 81 1,-10,t 0 157 156 1 19 -19	11 30 -38 12 33 -31 13 15 14 1,1,L 0 280 -274	7 63 -68 1,14,L 0 117 -117 1 20 -16 2 100 97	4 99 99 5 86 84 6 232 232 7 282 285 8 120 -118 9 124 -122	11 24 -25 12 45 -51 2,4,1 0 99 -93 1 299 293	2 21 24 3 50 47 4 22 -21 5 68 -68 6 4 14 7 32 30	11 42 37 12 5 -4 13 100 -99 3,-3,L 0 298 -291	6 19 -18 7 38 -39 8 79 -79 9 10 -6 3,10,L	3 161 160 4 76 78 5 166 -160 6 61 -52 7 70 69 8 40 38	8 29 -25 9 55 54 10 17 17 11 43 41
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	6 17 16 7 76 -75 8 88 -87 9 34 36 10 62 62 11 20 18	14 42 38 0,0,1 1 531× 523 2 382× -379 3 262 200	10 9 -6 0,12,L 0 93 -97 1 93 92 2 914 222	2 311 -318 3 160 -162 4 129 138 5 10 3 6 117 118 7 8 11	1 74 73 2 32 29 3 104 103 4 216 -204 5 352 -356 6 129 -128	3 19 16 4 13 -13 5 3 -9 6 61 -63 1+15+1	10 71 -72 11 15 -14 12 84 82 13 63 66 24-74	2 379 387 3 21 -11 4 200 -205 5 119 -120 6 55 55 7 29 -10	8 21 17 9 36 37 3,-15+L 0 113 114 1 98 99	1 226 -221 2 1157% 1151 3 706% 731 4 184 177 5 24 27 6 114 -118	0 104 -103 1 28 -30 2 12 -4 3 86 89 4 95 93 5 61 -59	9 86 87 10 32 32 11 99 -95 12 46 -46	0 181 -189 1 80 -83 2 359 365 3 79 75 4 46 49 5 78 81
7 30 -30 8 138 140 9 59 62 10 8 2 11 18 -20	12 18 18 13 34 -30 0,-11,t 1 123 -125	4 184 -190 5 173 168 6 515 506 7 139 138 8 41 39	3 2 5 4 52 -55 5 67 -72 6 52 -55 7 32 32	8 149 -149 9 23 -21 10 69 69 11 14 3 12 67 64	7 16 21 8 52 48 9 151 144 10 17 8 11 95 -92	0 6 6 1 63 61 2 49 51 3 16 -11 4 26 -29 5 15	0 161 160 1 185 -183 2 286 -281 3 14 13 4 146 144	8 138 131 9 75 72 10 51 -53 11 77 -74 12 29 -26	2 49 53 3 45 46 4 36 -31 5 10 -51 6 76 73	7 44 -44 8 167 164 9 74 71 10 38 38 11 29 29	6 58 -58 7 22 -21 8 6 -2 3,11,L	0 413 416 1 491 491 2 59 -56 3 139 134 4 127 -121	6 93 -67 7 8 -4 8 72 71 9 33 25 10 13 9
-1,10,L 1 139 130 2 106 101 3 100 -96	2 18 13 3 474 48C 4 210 210 5 104 -104 6 59 -58 7 129 -134	v 162 -140 10 130 -131 11 15 -16 12 94 88 13 5 2 14 19 18	0,13.L 0,13.L 0,13.L 1,147,148	1,-9,L 0 17 -17 1 139 -140 2 39 -34	1.2.L 0 166 -192 1 367 377	1, 10, L 0 0 4 1 23 20 2 29 -32	6 168 167 7 174 -169 8 172 -174 9 15 10 10 56 55	2,5,1 0 368 376 1 56 -45 2 141 134 3 77 -75	8 47 48 9 10 3 10 26 -28 3,-14,L	3,-2,L 0 284 -293 1 1317X 1305	1 86 88 2 69 70 3 24 26 4 88 -88 5 86 -86	6 225 225 7 77 76 8 80 80 9 20 16 10 79 -78	4,5,1 0 32 -29 1 316 326 2 140 139 3 122 -125
5 7 -1 6 21 17 7 93 86 8 77 74 9 37 -43	0 9 12 9 95 91 10 47 42 11 30 -35 12 3 -14 13 64 -64	0.1.L 0 292x 302 1 773x -765 2 582x 588 3 186 -179	$\begin{array}{c} 30 & -29 \\ 3 & 129 & -127 \\ 4 & 54 & -50 \\ 5 & 9 & 10 \\ 6 & 6 & -5 \\ 7 & 16 & 25 \end{array}$	6 195 196 5 68 -69 6 18 -14 7 213 -213 8 71 -73	3 42 -58 4 403 -406 5 176 -168 6 282 285 7 151 150	1.17.L 0 9 -3 1 37 -36	241 41 13 33 -29 24-64L 0 228 -232	- cc 236 5 102 102 6 140 141 7 35 33 8 57 54 9 62 - 59	1 75 -72 2 43 -44 3 64 -69 4 50 -47 5 118 122	3 77 -78 4 107 -115 5 61 -65 6 92 -96 7 304 305	3,12,4 0 130 126 1 61 03	4,-7,L 0 505 491 1 131 -130	5 78 -60 6 36 -37 7 97 98 8 37 35 9 58 -62
10 49 -48 11 24 -25		5 407 473	• 7 -10	10 69 86	9 39 -36	2 -8 -51	2 23 34	11 10 18	7 26 25	d 17V 180 9 24 -22	3 74 -72	5 32 -37	

Table 2 (cont.)

|--|

all 10677 reflections and 3.48% for the 9027 reflections with $F_o > 3\sigma(F_o)$. The final weighted R index, $R' = \{ [\sum w(|F_o| - |F_c|)^2] / \sum wF_o^2 \}^{1/2}$ was 0.034 for all reflections. For nonhydrogen atoms, the average shift to error ratio was less than 0.05 with a maximum ratio of 0.45 for the x parameter of C(14). The quantity minimized in the least-squares calculation was $\sum w(|F_o| - |F_c|)^2$ where the weights, which ranged from 0.13 to 3.5, were proportional to $1/\sigma^2(F_o)$.

A final difference synthesis calculated with all reflections showed a maximum residual electron density of 0.34 e Å⁻³ in the region of the disordered butyl moiety and a minimum of -0.32 e Å⁻³ near the cobalt atom.

The atomic parameters and their standard deviations (calculated from the inverse matrix of the normal equations) are displayed in Table 1. Table 2 shows the observed and calculated structure factors.

Structure factor, Fourier, bond distance and angle, and least-squares calculations were carried out with the X-RAY 67 program system (Stewart, 1967).

Description and discussion of the structure

Non-bonded contacts

Fig. 2 shows the contents and immediate surroundings of a unit cell. The molecule nearest the origin (a)is shown in greater detail in Fig. 1. The molecules are packed fairly tightly in all directions with no significant voids, but there are no hydrogen bonds between molecules. Van der Waals forces maintain the intermolecular integrity of the system.

There are no remarkably short intermolecular distances as examination of the 35 contacts listed in Table 3 will show. Most of the intermolecular contacts with the dimethylglyoxime ligands tend to reduce motion normal to the plane of these ligands but to allow rotation about the C(34)-Co-P axis. The principal points of contact involve the methyl groups C(11) and C(13)on opposite sides of the molecule, the pyridine, and the butyl chains. Two of the butyl chains have their orientation stabilized by intramolecular contacts with the equatorial ligands; the third, $C(61) \cdots C(64)$ suffers some disorder in the C(62), C(63) region in spite of 7 contacts with adjacent molecules.

Table 3. Intermolecular and selected intramolecular contacts

All intermolecular contacts with contact radii less than 1.3 Å for H, 1.55 Å for N, 1.45 Å for O and 1.75 Å for aromatic carbon are shown.

		operation
$H(321) \cdots N(16)$	2.60 Å	. 1
$H(321) \cdots H(67)$	2.41	ī
$H(321) \cdots O(6)$	2.49	i
$H(361) \cdots O(8)$	2.47	ī
$H(361) \cdots N(18)$	2.54	1
$H(361) \cdots H(85)$	2.36	1
$H(411) \cdots C(1)$	2.86	1
$H(411) \cdots C(2)$	2.90	1
$H(422) \cdots O(5)$	2.56	1
$H(422) \cdots N(15)$	2.75	1
$H(432) \cdots H(112)$	2.53	1
$H(511) \cdots O(7)$	2.64	1
H(511)···N(17)	2.74	1
$H(522) \cdots O(6)$	2.48	1
$H(611) \cdots N(18)$	2.85	1
$H(611)\cdots C(4)$	2.82	1
$H(612) \cdots N(18)$	2.81	1
$H(412) \cdots N(34)$	2.74	2
$H(621) \cdots H(351)$	2.57	2
$H(521) \cdots N(34)$	2.78	2
$H(543) \cdots H(331)$	2.34	2
$H(132) \cdots H(131)$	2.49	3
$C(33) \cdots H(133)$	2.79	3
$C(3) \cdots H(131)$	2.92	3
$H(131) \cdots H(131)$	2.47	3
$C(32) \cdots H(132)$	3.04	3
$H(361) \cdots H(632)$	2.36	4
$H(351) \cdots H(642)$	2.48	4
$O(8) \cdots H(632)$	2.72	4
$C(35) \cdots H(111)$	3.02	2
$C(30) \cdots H(111)$ $C(25) \cdots H(112)$	2.93	2
$U(125) \cdots H(112)$ $U(125) \cdots U(622)$	3.03	5
$H(133) \cdots H(022)$ $H(531) \cdots H(621)$	2-24	0
$\Omega(7) \cdots \Pi(031)$	2.33	6
O(7) = I(045)	4.22	0

Symmetry operations: 1, (x, y, z); 2, (x+1, y, z+1); 3, (-x, -y, -z); 4, (-x+1, -y, -z); 5, (-x+1, -y+1, -z); 6, (-x+1, -y+1, -z+1).



Fig. 2. Stereogram of intermolecular packing. The molecules shown correspond to the atomic coordinates of Table 1 as follows: (a) (x, y, z); (b) (1-x, 1-y, 1-z); (c) (1-x, 1-y, -z); (d) (x, y, z+1); (e) (1-x, -y, 1-z); (f) (x+1, y, z+1); (g) (1-x, -y, -z).

Bond distances and angles

The bond distances have been corrected for thermal motion using the computer program ORSBA (Johnson, 1970a) which fits either a rigid body or a segmented body to the molecular motion described by the anisotropic thermal parameters. In this instance, the complete Schomaker & Trueblood (1968) description was used including screw, translation and libration motions. The molecule does not behave as a single rigid body. However, the 4 groups consisting of the cobalt taken together with each of the 2 dimethylglyoxime ligands, the pyridine ligand and the 4 atoms of the tributylphosphine ligand nearest to it, behave, to a good approximation, as rigid bodies. The reaction center (Johnson, 1970b) for each of the 4 groups is near the cobalt atom showing that, to a good approximation, each group behaves as if it were pivoted at cobalt. Table 4 gives the parameters describing the motion along with the standard error of U(i,j) for the four molecular segments treated here as rigid bodies. In Fig. 1 the left and right arrows show the principal libration axes for the right and left dimethylglyoxime ligands, respectively. The fit deteriorates significantly when both dimethylglyoxime ligands are treated as a single rigid unit.

The corrected bond distances and angles obtained from the rigid-body treatment are shown in Fig. 3. They are listed in Tables 5 and 6 along with the uncorrected values. Standard deviations as calculated from the least-squares refinement are included in the tables. Chemically equivalent bond distance averages (corrected for libration) are: Co-N, 1.891 Å; C-N, 1.305; N-O(H), 1.343; N-O, 1.355; C-C(Me), 1.511; C-C, 1.455; P-C, 1.836. Pyridine distances and angles are discussed below. The average C-H distances (for those hydrogen positions which were refined) are: methyl groups, 0.906 Å; methylene groups, 0.943 and pyridine, 0.965.

The phosphine ligand

The phosphine ligand coordinates to the cobalt center through a Co-P bond of 2.342 (1) Å. The bonding geometry of the phosphorus atom is distorted tetrahedral. The close proximity of the equatorial ligands to the α and β carbon atoms of the butyl triad forces the three chains away from the equatorial plane, increasing the Co-P-C angles by about 5° and decreasing the C-P-C angles by a like amount.

The n-butyl groups are planar to within an r.m.s. deviation of 0.05 Å due to the steric effects of the hydrogen atoms on adjacent carbon atoms. It is interesting to note the angles between each of the three planes and the equatorial plane: C(40)'s plane, 27°; C(50)'s plane, 23°; C(60)'s plane, 88°. The two n-butyl groups which make acute angles with the equatorial plane are in contact at the α and β carbon atoms. The C(60)'s chain where C(62) and C(63) show disorder has intramolecular contacts, especially C(62) and C(63),

are with points to one side of the chain, allowing greater freedom of motion than in any other part of the molecule. These contact distances are listed in Table 3.

The pyridine ligand

Coordination of pyridine through a Co-C bond effectively replaces the hydrogen atom on C(31) with cobalt. This contrasts with the usual Co-N coordination where the unpaired electrons of the free pyridine



Fig. 3. Intramolecular bond distances and angles. All distances and angles shown have been corrected for librational motion as discussed in the text.

molecule are shared with cobalt. The effects of this 'substitution' are seen by comparing the pyridine bond distances and angles for the most accurate determinations to date on uncoordinated pyridine, pyridine with Co-C coordination and pyridine with Co-N coordination. These results are shown in Table 7. Comparison of uncoordinated and Co-C coordinated pyridine supports the conclusion that Co-C coordination has no effect on the C-C pyridine distances but probably a small effect on the C-N distances. Co-N coordination shortens the C-C pyridine bonds (as compared with uncoordinated pyridine) but has no effect on the C-N distances.

Coordination clearly has an effect on the pyridine bond angles. In the Co-C case, the largest effect is on the C-C-C angle at the coordination site which decreases nearly 3° . The adjacent C-C-C angles increase by 1.5° . These changes are consistent with the idea that they are produced by distortion resulting from contacts between the equatorial ligands and adjacent pyridine atoms.

Pyridine ligands with Co-N coordination which are without intramolecular contacts near the C-C-C angles show these angles within about 0.5° of those in uncoordinated pyridine. The nitrogen end shows C-N-C and N-C-C angles which also differ somewhat from those in the uncoordinated pyridine. In this case the C-N-C angle increases somewhat with a corresponding decrease in the N-C-C angles. Here the strains introduced by coordination are presumably smaller since the Co-N distance is longer (2.043 Å) than the Co-C(4) distance (1.983 Å).

The heavy atoms of the pyridine ligand are planar to within 0.005 Å and the pyridine plane makes an

Atoms in rigid unit	Standard error of $U(I,J)$.	Reaction X	center coor Y	dinates* <i>Z</i>	Principal valu of libration	ie Di of	rection cos libration a	sines P .xes*	rincipal axes of translation
Co, N(15), N(16) O(5), O(6), C(1) C(2), C(11), C(12)	0.0027 Ų	−0·081 Å	0·153 Å	0·231 Å	6·3° 3·9 3·3	0·845 0·293 0·447	- 0.096 - 0.739 0.667	0.525 - 0.607 - 0.597	0·205 Å (r.m.s. 0·189 0·164
Co, N(17), N(18) O(7), O(8), C(3) C(4), C(13), C(14)	0.0035	-0.378	-0.518	-0.209	6·3 4·5 3·3	$ \begin{array}{r} 0.133 \\ -0.882 \\ 0.452 \end{array} $	-0.438 -0.462 -0.772	0·889 - 0·096 - 0·447	0·197 0·192 0·154
Co, N(34), C(31) C(32), C(33) C(35), C(36)	0.0024	-0.356	0.118	-0.486	6·6 2·7 2·4	0·537 0·840 0·076	- 0·297 - 0·272 - 0·915	0·790 0·469 0·396	0·207 0·186 0·170
Co, P, C(41) C(51), C(61)	0.0017	0.678	0.622	-0·518	3·5 2·6 1·3	0·384 - 0·031 0·923	-0.418 -0.897 0.143	0.824 - 0.440 - 0.357	0·200 0·188 0·158

Table 4. Rigid-body analysis of molecular libration

* In terms of a Cartesian system with the origin at the cobalt atom, x along a, y along $c^* \times a$ and z along c^* .

Table 5. Intramolecular distances (Å) before and after correction for molecular libration

2.339(1)	2.342†	O(7) - H(67)	1.501 (18)	
1.876 (2)	1.890	O(6)H(67)	0.981 (19)	
1.880 (1)	1.894	O(5)O(8)	2.470 (2)	
1.874 (1)	1.888	O(6) - O(7)	2·477 (2)	
1.875 (1)	1.892	C(31) - C(32)	1.388 (3)	1.397†
1.979 (1)	1.983	C(32) - C(33)	1.389 (2)	1.391
1 284 (2)	1.294	C(33) - N(34)	1.324 (2)	1.332
1.301 (2)	1.311	N(34) - C(35)	1.322(3)	1.330
1.297 (2)	1.308	C(35) - C(36)	1.387 (2)	1.390
1.299 (2)	1.309	C(36) - C(31)	1.389 (2)	1.397
1.349 (2)	1.359	PC(41)	1.831(1)	1.835
1.334 (2)	1.344	C(41) - C(42)	1.520 (3)	
1.340 (2)	1.351	C(42) - C(43)	1.521 (2)	
1.333 (2)	1.343	C(43) - C(44)	1.504 (4)	
1.504 (4)	1.516	PC(51)	1.837 (2)	1.841
1.497 (3)	1.509	C(51) - C(52)	1.523 (2)	
1.497 (3)	1.508	C(52) - C(53)	1.507 (4)	
1.498 (3)	1.512	C(53) - C(54)	1.499 (4)	
1.450 (3)	1.461	PC(61)	1.829 (2)	1.833
1.437 (3)	1.449	C(61) - C(62)	1.523*	
1.506 (18)		C(62) - C(63)	1.450*	
0.968 (18)		C(63) - C(64)	1.491*	
	$\begin{array}{c} 2\cdot 339 \ (1) \\ 1\cdot 876 \ (2) \\ 1\cdot 880 \ (1) \\ 1\cdot 877 \ (1) \\ 1\cdot 877 \ (1) \\ 1\cdot 9779 \ (1) \\ 1\cdot 284 \ (2) \\ 1\cdot 301 \ (2) \\ 1\cdot 297 \ (2) \\ 1\cdot 297 \ (2) \\ 1\cdot 297 \ (2) \\ 1\cdot 349 \ (2) \\ 1\cdot 344 \ (2) \\ 1\cdot 334 \ (2) \\ 1\cdot 344 \ (2) \\ 1\cdot 334 \ (2) \\ 1\cdot 344 \ (3) \\ 1\cdot 497 \ (3) \\ 1\cdot 498 \ (3) \\ 1\cdot 497 \ (3) \\ 1\cdot 497 \ (3) \\ 1\cdot 506 \ (18) \\ 0\cdot 968 \ (18) \end{array}$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\begin{array}{cccccccccccccccccccccccccccccccccccc$

* Not included in final least-squares cycles, distances calculated from the average position of C(621), C(622) and C(631), C(632).

† This column is corrected for molecular libration.

Table 6. Intramolecular angles (°) and standard deviations before and after correction for molecular libration

N(15)-CoN(16)	81.64 (6)	81.63†	C(13)-C(3)C(4)	124.7 (2)	124.7†
N(16) - Co - N(17)	98.54 (6)		N(18) - C(4) - C(14)	121.6 (3)	121.6
$N(17) - C_0 - N(18)$	81.46 (6)	81.44	N(18) - C(4) - C(3)	113.0(2)	112.9
N(18)-Co-N(15)	98.26(6)		C(14) - C(4) - C(3)	$125 \cdot 4(3)$	125.5
$P = C_0 = C(31)$	179.2(19)		$C_0 - N(18) - O(8)$	$123 \cdot 3(1)$	123.4
$N(15) = C_0 = C(31)$	88.38 (6)		$C_0 N(18) - C(4)$	116.2(2)	116.3
$N(16) - C_0 - C(31)$	89.80 (5)		C(4) = -N(18) - O(8)	120.4(2)	120.3
$N(17) = C_0 =C(31)$	87.81 (5)		N(18) - O(8) - H(85)	100.3(16)	
$N(18) - C_0 - C(31)$	88.85(5)		$C_0 = -C(31) C(32)$	122.8(1)	122.7
$N(15) = C_0 =P$	92.43(4)		$C(36) - C(31) - C_0$	122.3(2)	122.1
$N(16) = C_0 =P$	90.32(3)		C(36) - C(31) - C(32)	114.9(2)	115.2
$N(17) - C_0 P$	91.38 (3)		C(31) - C(32) - C(33)	120.3(2)	120.2
$N(18) = C_0 = P$	91.03(4)		C(32) - C(33) - N(34)	124.7(2)	124.6
$C_0 = N(15) = O(5)$	122.5(1)	122.5	C(33) = N(34) = C(35)	114.9(2)	115.2
$C_0 - N(15) - C(1)$	122.5(1) 116.5(2)	116.5	N(34) - C(35) - C(36)	124.9(2)	124.8
C(1) = N(15) = O(5)	121.0(2)	121.0	C(35) - C(36) - C(31)	120.3(2)	120.1
N(15) - C(1) - C(11)	121.0(2) 121.9(3)	121.9	$C_0 - P_{$	116.66 (6)	116.66
N(15) = C(1) = -C(2)	113.2(2)	113.2	$C_0 - P_{}C(51)$	113.97 (6)	113.92
C(1) = C(1) = C(2)	124.8(2)	124.8	$C_0 P_{} C(61)$	112.86 (4)	112.85
N(16) - C(2) - C(12)	1240(2) 123.3(3)	123.3	C(41) - P C(51)	$103 \cdot 16(7)$	103.21
N(16) = C(2) = C(12)	112.5(3)	112.5	C(51) - P C(61)	103.40(9)	103.45
C(12) - C(2) - C(1)	124.2(2)	124.2	C(61) - P C(41)	105.41(8)	105.40
$C_0 = -N(16) = O(6)$	123.2(1)	123.2	$P_{}C(41) - C(42)$	116.9(1)	
$C_0 = N(16) - C(2)$	1252(1) 116(1(2))	116.1	C(41) - C(42) - C(43)	112.5(2)	
C(2) = N(16) - O(6)	120.8(2)	120.7	C(42) = C(43) = C(44)	113.9(2)	
N(16) = O(6) = H(67)	$102 \cdot 0$ (16)	120 /	$P_{}C(51) - C(52)$	116.8(2)	
$C_0 = N(17) = O(7)$	122.5(10)	122.4	C(51) - C(52) - C(53)	112.8(2)	
$C_0 = N(17) - C(3)$	116.5(2)	116.5	C(52) - C(53) - C(54)	114.5(3)	
C(3) = N(17) = O(7)	121.0(2)	121.1	$P_{}C(61) = C(62)$	118.0*	
N(17) C(3) - C(13)	1210(2) 122.5(2)	122.5	C(61) = C(62) = C(63)	115.6*	
N(17) = C(3) = -C(13)	$122^{\circ}3(2)$ 112.8(2)	112.9	C(62) = C(62) = C(63)	118.0*	
N(1/) = C(3) = -C(4)	112.0 (2)	112.0	C(02) - C(03) - C(04)	1100	

* Not included in final least-squares cycles, angles calculated from the average position of C(621), C(622) and C(631), C(632). † This column is corrected for molecular libration.

Table 7.	Comparison of	average	pyridine	distances
	and	angles		

parallel to the N–O bonds of the e	equatorial	ligands,	is
characteristic of the cobaloximes ((Lenhert,	1972).	

The equatorial ligand	İs
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The cobalt coordination is distorted from square planar geometry by the five-membered rings formed by the dimethylglyoxime ligands. The inter-ring N-Co-N angles of about 81.5° (Table 6) and the extra-ring angles of 98.5° are characteristic of the cobaloximes. Intramolecular O-H···O hydrogen bonds of 2.47 Å connect the two ligands. Both hydrogen atoms were located unambiguously; each has a clear association with only one of the two dimethylglyoxime ligands (Table 5) as shown by least-squares refinement of the hydrogen atom positions and the final difference synthesis.

All atoms of the equatorial ligands are planar to within an r.m.s. deviation of 0.06 Å as shown in Table 8. If only the 5 atoms of each ligand ring are included in the plane calculations, the dihedral angle between the planes of the two five-membered ligand rings, C_2N_2Co , is 4.4°. This bending as well as the individual deviations of several of the atoms apparently results from intramolecular contacts with the axial ligands. The effects of these contacts (listed in Table 3) can best be seen by examining the stereopair, Fig. 1. Contacts with the pyridyl moiety especially between H(361), N(18) and O(8) tilt one ligand, and contacts between H(321), N(16) and O(6) tilt the other ligand, but in the

	<i>(a)</i>	<i>(b)</i>	(<i>c</i>)
	Uncoordinated	Co-C(4)	Co-N
Co-C(4) Co-N		1·983 (1) Å	2·043 (1) Å
N(1)-C(2) N(1)-C(6)	1·342 (5) Å	1.331 (2)	1.340 (1)
C(2)-C(3) C(5)-C(6)	1.391 (5)	1.390 (2)	1.378 (1)
C(3)–C(4) C(5)–C(4)	1.398 (5)	1.397 (2)	1.372 (1)
C(6) - N(1) - C(2)) 116·7°	115·2 (2)°	117·47 (8)°
N(1)-C(2)-C(3 N(1)-C(6)-C(5) 124·0	124.7 (1)	122.69 (5)
C(2)-C(3)-C(4) C(6)-C(5) $C(4)$	·) 118·6	120.1 (1)	119-28 (5)
C(3) - C(4) - C(5)) 118:1	115-2 (2)	118.60 (8)

- (a) Microwave results of Bak, Hansen & Rastrup-Andersen (1954) for uncoordinated pyridine.
- (b) This work. Pyridine coordinated via Co-C(4).
- (c) Average values for 3 independent structure determinations, each of comparable accuracy with the present study. Pyridine coordinated via Co-N (Lenhert, 1972). Corrections for molecular libration not applied. Estimated corrections about 0.005 Å or less.

angle of 89.0° with the plane of the equatorial ligands. The orientation of the pyridine plane, approximately opposite direction. The tilt axis bisects the N-Co-N inner ring angle. Contacts between the tributylphosphine ligand, especially between H(411), C(1) and C(2) on one ligand and H(611) and C(4) on the other tilt each dimethylglyoxime ligand toward the pyridine side.

Table 8.	Deviations	from	least-squares	planes
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	2DMG	DMG1	DMG2
Co	0·077* Å	0·006* Å	-0·012* Å
N(15)	-0.006*	-0.006*	
N(16)	0.042*	-0.007*	
N(17)	0.035*		0.014*
N(18)	0.067*		0.012*
$\mathbf{C}(\mathbf{i})$	-0.027*	0.002*	
$\tilde{C}(2)$	0.003*	0.004*	
$\tilde{C}(3)$	-0.022*		-0.008*
$\mathbf{C}(4)$	0.001*		-0.005*
ciii	-0.082*	0.006	
$\tilde{C}(12)$	-0.013*	0.012	
$\tilde{C}(13)$	-0.119*		-0.051
C(14)	-0.047*		-0.023
O(5)	-0.054*	-0.033	
0(6)	0.028*	-0.053	
$\tilde{O}(7)$	0.012*		0.001
$\tilde{O}(8)$	0.104*		0.024
H(67)	0.057	- 0.058	
H(85)	0.087		-0.058

Dihedral angles between the planes: 2DMG-DMG1, 2.2°; 2DMG-DMG2, 2.2°; DMG1-DMG2, 4.4°.

* Denotes atoms included in the least-squares fit.

The authors thank G. N. Schrauzer for the crystals used in this analysis. Special thanks are due to Dr C. K. Johnson for helpful discussions and assistance with the rigid-body analysis, which was carried out at the Oak Ridge National Laboratory. Financial support from the National Institutes of Health via grants AM 09085 to Vanderbilt University, FR 00254 to the Common Research Computer Facility in Houston, Texas and from Vanderbilt University for computing are gratefully acknowledged. One of us (WWA) was supported by an AEC special fellowship and by a U.S. Air Force active duty assignment.

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