were taken from *International Tables for X-ray Crystallography* (1962). Atomic parameters are given in Tables 1 and 2. Bond distances, angles and conformation angles are shown in Table 3.

Table 4. *Least-squares planes and deviations of atoms* (Å)

An asterisk indicates atoms not included in the plane.

The plane through the nitro group

$0{\cdot}6396X-0{\cdot}3981Y+0{\cdot}6602Z=6{\cdot}6715$

C(9)	0·000	O(7)	0·000
O(6)	0·000	N	0·000

The plane through the benzene ring

$0{\cdot}5879X-0{\cdot}1341Y+0{\cdot}7977Z=8{\cdot}0217$

C(6)	0·001	C(9)	0·005
C(7)	0·011	C(10)	0·007
C(8)	−0·014	C(11)	−0·010
O(1)*	0·051	O(6)*	−0·303
N*	0·016	O(7)*	0·338

The dihedral angle between the two planes is 17·4°.

Discussion. The xyloside group is in the C_1 chair conformation. The bond distances, angles and conformation angles in the xyloside group are in agreement with those observed in other *β*-D-xyloside derivatives (Brown, Cox & Llewellyn, 1966; Moran & Richards, 1973). The bond distances and angles in the *p*-nitrophenyl group are also in agreement with those in *p*-nitrophenol (Coppens & Schmidt, 1965*a*,*b*) and its derivatives (Guttormson & Robertson, 1972; Brehm & Moult, 1975). The benzene ring shows good planarity (Table 4). The plane of the nitro group makes an angle of 17·4° with the benzene plane. The valence angle of the oxygen atom linking the *p*-nitrophenyl group with the xyloside group is similar to that of *p*-nitrophenyl-*β*-D-*N*-acetylglucosaminide (Brehm & Moult, 1975). The O(1)–C(6) bond is *gauche* to the O(5)–C(1) bond and *trans* to the C(2)–C(1) bond. The C(1)–O(1) bond is twisted by 23·6° against the benzene plane. The conformation around the C(1)–O(1)–C(6) linkage (Fig. 1) is very similar to that observed in *p*-nitrophenyl-*β*-D-*N*-acetylglucosaminide. Only one hydrogen bond with the O···O distance of 2·832 Å is observed between O(2) and O(4) $(1-x, -\frac{1}{2}+y, \frac{1}{2}-z)$ (Fig. 2).

The author wishes to thank Dr H. Uedaira for supporting this work and for helpful discussions. The program *FMLS* (Ashida, 1973) was used with some modifications for the full-matrix least-squares calculations. The computation was done on a HITAC 8450 computer in our laboratory.

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Dinitrosylbis(triphenylphosphine)cobalt Hexafluorophosphate

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(*Received* 29 *January* 1976; *accepted* 18 *February* 1976)

Abstract. $C_{36}H_{30}N_2O_2F_6P_3Co$, $M=788{\cdot}49$, monoclinic, $C2/c$, $a=17{\cdot}431$ (9), $b=12{\cdot}352$ (6), $c=20{\cdot}256$ (10) Å, $\beta=125{\cdot}27°$, $U=3560{\cdot}7$ Å^3, $Z=4$, $D_c=1{\cdot}70$ g cm^{-3}. The structure consists of discrete cations and anions each possessing crystallographic twofold axes. The coordination about Co in the cation is tetrahedral distorted towards square planar, the N–Co–N angle being 136·7 (4)°.

Introduction. Cell dimensions were obtained from a least-squares fit to the settings for 25 reflexions on a Philips PW 1100 automated four-circle diffractometer

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(Mo $K\alpha$, 0·71069 Å). Intensities from a crystal, 0·40 × 0·25 × 0·15 mm, were collected on the diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Measurements were carried out for $4° \leq 2\theta \leq 60°$ at a scan rate of 0·05° s^{-1} over a symmetric scan range of ±0·5° from the calculated angle. Three standard reflexions measured at intervals throughout data collection did not indicate any significant decomposition. Lorentz and polarization corrections were applied but no account was taken of absorption [μ(Mo $K\alpha$) = 6·24 cm^{-1}]. Of the 3310 unique reflexions recorded, only the 3271 with $F_o \geq 4\sigma(F_o)$ were used in the structure solution and refinement.

The structure was solved by automated multisolution direct methods and subsequent difference syntheses, and refined by full-matrix least squares with *SHEL-X* (G. M. Sheldrick). Anisotropic temperature factors were employed for all non-hydrogen atoms. H atoms were placed in idealized positions (C–H = 1·08 Å) and constrained to remain 1·08 (1) Å from the adjacent C during refinement. The H atoms were given an overall isotropic temperature factor which was refined. Two reflexions, 110 and $\bar{1}11$, displayed inordinately large discrepancies between F_c and F_o and were omitted from the final refinement.

In the refinement $\sum w\Delta^2$ was minimized and the final value of $R_G = [\sum w\Delta^2/\sum wF_o^2]^{1/2}$ was 0·066, with R = 0·048 and $R_w = \sum w^{1/2}\Delta/\sum w^{1/2}F_o$ = 0·054. Final weights employed are given by $w = 1{\cdot}1896/[\sigma^2(F_o) + 0{\cdot}0001F_o^2]$. Complex neutral atom scattering factors (*International Tables for X-ray Crystallography*, 1974) were employed.

Atom coordinates and temperature factors are given in Tables 1–3. A final difference synthesis displayed no features greater than 0·62 e Å^{-3}.*

Discussion. The complex cations $[M(NO)_2(PPh_3)_2]^+$ (M=Rh, Ir) undergo a nitrogen coupling reaction on treatment with CO, *viz.*

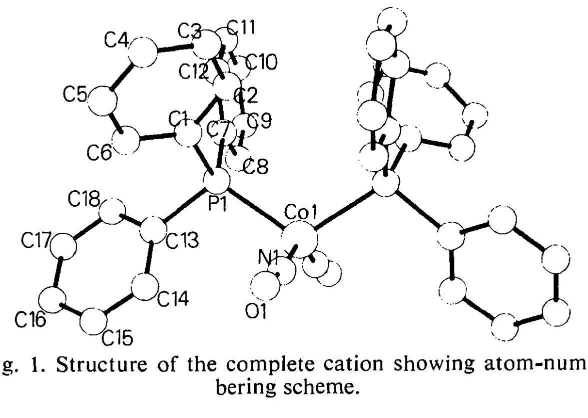

Fig. 1. Structure of the complete cation showing atom-numbering scheme.

Table 1. *Atom coordinates* (× 10^4)

	x	*y*	*z*
Co(1)	0	2854	2500
N(1)	1071 (3)	2362 (3)	3086 (2)
O(1)	1871 (4)	2150 (5)	3542 (3)
P(1)	−46 (1)	3860 (1)	3412
C(2)	1250 (3)	5460 (3)	3758 (2)
C(3)	2053 (3)	6084 (3)	4254 (3)
C(4)	2599 (3)	5911 (3)	5084 (3)
C(5)	2343 (3)	5142 (3)	5413 (2)
C(6)	1540 (2)	4516 (3)	4920 (2)
C(1)	996 (2)	4669 (2)	4085 (2)
C(8)	−1944 (2)	4285 (3)	2517 (2)
C(9)	−2742 (3)	4928 (4)	2172 (3)
C(10)	−2649 (3)	6036 (4)	2287 (3)
C(11)	−1777 (3)	6495 (3)	2754 (3)
C(12)	−980 (3)	5857 (3)	3116 (2)
C(7)	−1059 (2)	4740 (2)	2998 (2)
C(14)	376 (3)	1987 (3)	4330 (2)
C(15)	397 (3)	1360 (3)	4905 (3)
C(16)	−69 (3)	1679 (3)	5237 (2)
C(17)	−563 (3)	2616 (3)	4991 (2)
C(18)	−588 (2)	3269 (3)	4423 (2)
C(13)	−113 (2)	2961 (2)	4091 (2)
P(2)	0	1091 (1)	7500
F(1)	258 (3)	223 (3)	8153 (2)
F(2)	1043 (2)	1135 (4)	7800 (2)
F(3)	250 (3)	1966 (3)	8155 (2)

Table 2. *Anisotropic temperature factors* (Å^2 × 10^3)

The temperature factor exponent takes the form: $-2\pi^2(U_{11}h^2a^{*2} + \ldots + 2U_{12}hka^*b^*)$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Co(1)	81	31	49	0	47	0
N(1)	139 (4)	98 (3)	99 (3)	62 (2)	98 (3)	78 (3)
O(1)	175 (5)	275 (7)	185 (5)	159 (5)	142 (4)	167 (5)
P(1)	39	32	35	−3	22	−3
C(2)	60 (2)	54 (2)	54 (2)	−13 (2)	37 (2)	−18 (2)
C(3)	66 (2)	65 (2)	76 (3)	−19 (2)	46 (2)	−27 (2)
C(4)	45 (2)	61 (2)	74 (3)	−14 (2)	24 (2)	−15 (2)
C(5)	57 (2)	64 (2)	54 (2)	5 (2)	5 (2)	−9 (2)
C(6)	49 (2)	53 (2)	48 (2)	6 (2)	14 (2)	−7 (2)
C(1)	38 (1)	36 (1)	43 (2)	−5 (1)	23 (1)	−3 (1)
C(8)	46 (2)	52 (2)	66 (2)	−3 (2)	25 (2)	−8 (2)
C(9)	43 (2)	84 (3)	71 (3)	−1 (2)	25 (2)	−4 (2)
C(10)	56 (2)	81 (3)	89 (3)	1 (2)	38 (2)	23 (2)
C(11)	70 (3)	53 (2)	116 (4)	−13 (2)	48 (3)	13 (2)
C(12)	49 (2)	46 (2)	75 (2)	−17 (2)	31 (2)	1 (1)
C(7)	37 (1)	42 (2)	40 (1)	−2 (1)	23 (1)	−2 (1)
C(14)	79 (3)	46 (2)	63 (2)	9 (2)	51 (2)	3 (2)
C(15)	101 (3)	53 (2)	80 (3)	21 (2)	65 (3)	11 (2)
C(16)	89 (3)	69 (2)	57 (2)	4 (2)	49 (2)	−17 (2)
C(17)	76 (3)	71 (2)	55 (2)	−1 (2)	48 (2)	−7 (2)
C(18)	63 (2)	55 (2)	48 (2)	−4 (2)	39 (2)	−2 (2)
C(13)	48 (2)	41 (1)	35 (1)	−3 (1)	25 (1)	−7 (1)
P(2)	49 (1)	63 (1)	42 (1)	0	23 (1)	0
F(1)	174 (3)	122 (3)	100 (2)	47 (2)	46 (2)	−26 (2)
F(2)	65 (2)	249 (5)	111 (2)	16 (3)	48 (2)	24 (2)
F(3)	135 (3)	135 (3)	92 (2)	−51 (2)	48 (2)	−4 (2)

$$[M(NO)_2(PPh_3)_2]^+ + 4CO \rightarrow [M(CO)_3(PPh_3)_2]^+ + N_2O + CO_2$$

(Johnson & Bhaduri, 1973; Bhaduri, Johnson, Savory, Segal & Walter, 1974) whereas the Co analogue does not. Therefore the structure determination of $[Co(NO)_2(PPh_3)_2]$ $[PF_6]$ was undertaken to find whether the lack of reactivity correlated with any structural

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31691 (21 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 3. *Hydrogen-atom coordinates* ($\times 10^4$) *and isotropic temperature factors* ($Å^2 \times 10^3$)

	x	y	z	U
H(2)	766 (23)	5603 (33)	3121 (8)	92 (4)
H(3)	2252 (28)	6726 (24)	4016 (23)	92 (4)
H(4)	3211 (18)	6423 (27)	5428 (22)	92 (4)
H(5)	2711 (25)	4963 (36)	6046 (9)	92 (4)
H(6)	1356 (27)	3862 (23)	5156 (23)	92 (4)
H(8)	−1993 (29)	3446 (13)	2350 (24)	92 (4)
H(9)	−3352 (18)	4408 (27)	1833 (22)	92 (4)
H(10)	−3256 (18)	6555 (27)	1954 (22)	92 (4)
H(11)	−1655 (28)	7354 (11)	2872 (25)	92 (4)
H(12)	−362 (17)	6310 (28)	3558 (19)	92 (4)
H(14)	745 (26)	1822 (34)	4063 (23)	92 (4)
H(15)	750 (26)	600 (17)	5114 (25)	92 (4)
H(16)	41 (29)	1232 (29)	5738 (17)	92 (4)
H(17)	−922 (26)	2943 (31)	5236 (24)	92 (4)
H(18)	−1009 (24)	3983 (20)	4201 (24)	92 (4)

features by comparison with the known structure of the Ir compound (Mingos & Ibers, 1970).

The structure consists of discrete cations and PF_6^- anions, each lying on crystallographic twofold axes so that the unique asymmetric unit consists of half of each ion. The complete cation is depicted in Fig. 1 and the bond lengths and more important angles (with standard deviations from the least-squares covariance matrix) are given in Tables 4 and 5.

Table 4. *Bond lengths* (Å)

P(1)—Co(1)	2·266 (3)	C(11)–C(10)	1·365 (7)
N(1)—Co(1)	1·645 (6)	C(12)–C(11)	1·383 (6)
O(1)—N(1)	1·174 (6)	C(7)—C(12)	1·394 (5)
C(1)—P(1)	1·815 (5)	C(13)–P(1)	1·823 (5)
C(1)—C(2)	1·388 (5)	C(13)–C(14)	1·391 (5)
C(3)—C(2)	1·392 (6)	C(15)–C(14)	1·382 (6)
C(4)—C(3)	1·389 (7)	C(16)–C(15)	1·376 (6)
C(5)—C(4)	1·375 (7)	C(17)–C(16)	1·354 (7)
C(6)—C(5)	1·393 (6)	C(18)–C(17)	1·384 (6)
C(1)—C(6)	1·395 (5)	C(13)–C(18)	1·387 (5)
C(7)—P(1)	1·815 (5)	F(1)—P(2)	1·553 (5)
C(7)—C(8)	1·381 (5)	F(2)—P(2)	1·549 (5)
C(9)—C(8)	1·391 (6)	F(3)—P(2)	1·566 (5)
C(10)–C(9)	1·382 (7)		

Table 5. *Selected bond angles* (°)

N(1)-Co(1)-N(1′)	136·7 (4)		
P(1)-Co(1)-P(1′)	113·5 (2)	C(7)—P(1)-Co(1)	115·7 (2)
N(1)-Co(1)-P(1)	100·0 (2)	C(13)-P(1)-Co(1)	109·2 (2)
N(1)-Co(1)-P(1′)	103·3 (2)	C(7)—P(1)-C(1)	107·6 (2)
O(1)-N(1)—Co(1)	171·0 (5)	C(13)-P(1)-C(1)	104·0 (2)
C(1)-P(1)—Co(1)	115·0 (2)	C(13)-P(1)-C(7)	104·1 (2)

During the course of this work some results of another structure determination for this cation have become available (Huffman, 1975), but two independent nitrosyls and phosphines are indicated so that the cation has no crystallographically imposed symmetry, and virtually no details of the structure or the salt employed have been revealed. The structure of $[Rh(NO)_2(PPh_3)_2][ClO_4]$ has also been published (Kaduk & Ibers, 1975) enabling comparison of structure parameters for the whole series $[M(NO)_2(PPh_3)_2]^+$.

The coordination about the metal in all these cations is that of a tetrahedron flattened towards square planar. The Co complex has the least distorted tetrahedral structure with a N–Co–N angle of 136·7 (4)° compared with 157·5 (3) and 154·2 (7)° for the Rh and Ir analogues respectively, indicating that the Co is the closest to a classical tetrahedral d^{10} ion. Furthermore the Co–N–O angle of 171·0 (5)° indicates virtually linear geometry for this unit, while in the Rh and Ir compounds the angles are 158·9 (4) and 163·5 (10)° respectively, suggesting more NO^+ character in the Co compound.

The different stereochemistry of the Co complex would appear to reflect the different electronic structure which causes its lack of reactivity toward CO.

Thanks are due to Drs P. G. Owston and E. M. McPartlin (Polytechnic of North London) for use of the diffractometer and assistance with data collection. The help of Dr K. R. Grundy (Cambridge) in supplying a sample of the compound and subsequent useful discussions, and financial support from ICI Ltd are also gratefully acknowledged.

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