Table 2. *Intramolecular bond distances* (Å) *and angles* (°)

Pt—O(1)	2.056 (5)	Pt—O(2)	2.040 (5)
Pt—P(1)	2.248 (2)	Pt—P(2)	2.246 (2)
O(1)—C(1)	1.375 (8)	O(2)—C(1)	1.385 (8)
C(1)—C(2)	1.533 (12)	C(1)—C(3)	1.539 (12)
C(2)—F(1)	1.349 (10)	C(3)—F(4)	1.341 (10)
C(2)—F(2)	1.328 (10)	C(3)—F(5)	1.331 (9)
C(2)—F(3)	1.322 (11)	C(3)—F(6)	1.324 (9)
P(1)—C(11)	1.815 (7)	P(2)—C(41)	1.819 (7)
P(1)—C(21)	1.825 (7)	P(2)—C(51)	1.818 (7)
P(1)—C(31)	1.830 (7)	P(2)—C(61)	1.826 (7)

P(2)—Pt—P(1)	98.24 (6)	O(1)—Pt—O(2)	66.7 (2)
O(1)—Pt—P(2)	162.26 (14)	O(2)—Pt—P(2)	95.61 (14)
O(1)—Pt—P(1)	99.49 (13)	O(2)—Pt—P(1)	165.41 (14)
Pt—O(1)—C(1)	91.8 (4)	Pt—O(2)—C(1)	92.2 (4)
O(1)—C(1)—O(2)	109.3 (5)	C(2)—C(1)—C(3)	110.5 (7)
O(1)—C(1)—C(2)	109.3 (7)	O(2)—C(1)—C(2)	109.6 (6)
O(1)—C(1)—C(3)	110.5 (6)	O(2)—C(1)—C(3)	107.7 (6)
C(1)—C(2)—F(1)	112.6 (8)	F(1)—C(2)—F(2)	106.1 (8)
C(1)—C(2)—F(2)	113.5 (8)	F(1)—C(2)—F(3)	106.6 (9)
C(1)—C(2)—F(3)	110.2 (8)	F(2)—C(2)—F(3)	107.5 (8)
C(1)—C(3)—F(4)	110.7 (7)	F(4)—C(3)—F(5)	105.5 (7)
C(1)—C(3)—F(5)	114.7 (7)	F(4)—C(3)—F(6)	105.1 (7)
C(1)—C(3)—F(6)	113.8 (7)	F(5)—C(3)—F(6)	106.3 (7)
Pt—P(1)—C(11)	115.4 (2)	C(11)—P(1)—C(21)	108.6 (3)
Pt—P(1)—C(21)	116.3 (2)	C(11)—P(1)—C(31)	100.0 (3)
Pt—P(1)—C(31)	110.8 (2)	C(21)—P(1)—C(31)	104.0 (3)
Pt—P(2)—C(41)	119.7 (2)	C(41)—P(2)—C(51)	109.8 (3)
Pt—P(2)—C(51)	109.6 (2)	C(41)—P(2)—C(61)	102.5 (3)
Pt—P(2)—C(61)	111.7 (2)	C(51)—P(2)—C(61)	102.0 (3)

This paper extends the very limited range of reported structures of complexes in which a chelating dinegative ligand is bonded to platinum through oxygen, and demonstrates again the use of highly fluorinated diols in filling two coordination sites on a metal atom.

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References

Bradford, P., Hynes, R. C., Payne, N. C. & Willis, C. J. (1990). *J. Am. Chem. Soc.* **112**, 2647–2654.

Cahen, D. & Ibers, J. A. (1972). *J. Appl. Cryst.* **5**, 298–299.

Cariati, F., Mason, R., Robertson, G. B. & Ugo, R. (1967). *J. Chem. Soc. Chem. Commun.* p. 408.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Davies, J. A., Eagle, C. T., Pinkerton, A. A. & Syed, R. (1987). *Acta Cryst.* C**43**, 1547–1549.

Enraf-Nonius (1983). *Enraf-Nonius Structure Determination Package*. Delft, The Netherlands.

Enraf-Nonius (1984). *Enraf-Nonius CAD-4 Operators Manual*. Delft, The Netherlands.

Gregg, M. R., Powell, J. & Sawyer, J. F. (1988). *Acta Cryst.* C**44**, 43–46.

Hayward, P. J. & Nyman, C. J. (1971). *J. Am. Chem. Soc.* **93**, 617–619.

Hynes, R. C., Payne, N. C. & Willis, C. J. (1990). *J. Chem. Soc. Chem. Commun.* pp. 744–745.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Mitchell, C. M. & Stone, F. G. A. (1971). *J. Chem. Soc. A*, pp. 102–107.

Modinos, A. & Woodward, P. J. (1975). *J. Chem. Soc. Dalton Trans.* pp. 2134–2140.

Robertson, G. B. & Tucker, P. A. (1983). *Acta Cryst.* C**39**, 858–860.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1992). C**48**, 45–48

A Chiral Aminophosphine Complex of Platinum: Structure of *cis*-Dichloro[(1*S*)-*N*,*N*-dimethyl-1-{2-[(*S*)-*tert*-butylphenylphosphino]phenyl}ethylamine-*N*,*P*]platinum(II)

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Abstract. $[Pt(C_{20}H_{28}NP)Cl_2]$, $M_r = 579.42$, orthorhombic, $P2_12_12_1$, $a = 13.482(1)$, $b = 15.926(4)$, $c = 9.827(2)$ Å, $V = 2110(1)$ Å^3, $Z = 4$, $D_m = 1.830(3)$, $D_x = 1.823$ Mg m^{-3}, $F(000) = 1128$, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 7.05$ mm^{-1}, $T = 295$ K, $R = 0.025$, $wR = 0.029$ for 3719 unique reflections with $I > 3\sigma(I)$ and 227 variables refined on F. The four-coordinate Pt atom displays a distorted square-planar geometry with Pt—P and Pt—N distances of 2.236 (1) and 2.106 (5) Å respectively. The Pt—Cl(1) distance of 2.360 (2) Å is longer than Pt—Cl(2) at 2.297 (2) Å due to the greater *trans* influence of P relative to N. The six-membered chelate ring adopts a λ twist-boat conformation, with *S* absolute configuration both at backbone carbon C(7) and at the coordinated P atom.

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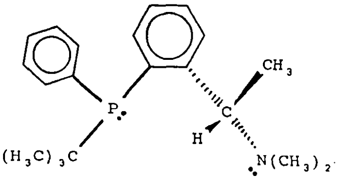
Introduction. In our investigations of the asymmetric hydrosilylation of prochiral ketones catalyzed by chiral Rh–aminophosphine complexes, we have been interested in the stereochemistry of the chiral ligand. Previous studies have focused on complexes incorporating a chiral C atom in the backbone of aminophosphine ligands (Payne & Stephan, 1982; McKay & Payne, 1986*a*,*b*) while our current efforts are aimed at adding a second asymmetric site at the coordinating P atom. A new diastereotopic aminophosphine ligand, terphenphos, (1), has been synthesized by reaction of *tert*-butylphenylchlorophosphine with *ortho*-lithiated (*S*)-*N*,*N*-dimethylphenylethylamine (Payne & Stephan, 1982; Tobin & Payne, 1992). We describe herein the determination of the molecular structure and absolute configuration of a Pt complex of this new ligand, [$PtCl_2$(terphenphos)], (I), which underwent a partial, spontaneous resolution.



(1)

Table 1. *Fractional atomic coordinates* (× 10^4) *and thermal parameters* (Å^2 × 10^4) *with e.s.d.'s in parentheses*

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

	x	*y*	*z*	U_{eq}
Pt	5248.03 (14)	4678.24 (11)	2115.30 (18)	261.0 (3)
Cl(1)	5177.0 (16)	4172.4 (11)	4370.2 (14)	569 (4)
Cl(2)	6162.7 (12)	3493.8 (9)	1667.5 (16)	410 (3)
P	5182.9 (10)	4888.9 (7)	−133.0 (12)	247 (2)
N	4371 (4)	5724 (3)	2636 (4)	382 (11)
C(1)	4138 (4)	6421 (3)	323 (6)	349 (13)
C(2)	4355 (4)	5766 (3)	−587 (5)	304 (11)
C(3)	3945 (4)	5768 (4)	−1895 (6)	403 (14)
C(4)	3351 (5)	6427 (4)	−2306 (7)	550 (18)
C(5)	3167 (5)	7085 (5)	−1441 (9)	608 (20)
C(6)	3537 (5)	7069 (4)	−143 (8)	528 (18)
C(7)	4487 (4)	6505 (3)	1776 (6)	367 (13)
C(8)	5513 (5)	6880 (4)	1916 (7)	502 (17)
C(9)	4530 (7)	6016 (5)	4074 (6)	683 (24)
C(10)	3313 (5)	5438 (5)	2577 (8)	621 (20)
C(11)	4534 (4)	3979 (3)	−865 (5)	303 (12)
C(12)	3864 (4)	3559 (4)	−48 (6)	342 (13)
C(13)	3305 (5)	2897 (4)	−538 (7)	432 (15)
C(14)	3414 (4)	2647 (4)	−1871 (7)	452 (16)
C(15)	4085 (6)	3045 (4)	−2678 (7)	556 (18)
C(16)	4639 (5)	3720 (4)	−2201 (6)	472 (14)
C(17)	6370 (4)	5085 (3)	−1100 (5)	335 (12)
C(18)	6975 (5)	5728 (5)	−306 (7)	510 (17)
C(19)	6208 (5)	5435 (4)	−2533 (6)	441 (14)
C(20)	6994 (5)	4284 (4)	−1236 (8)	591 (19)

Experimental. A sample of the complex was prepared by adding one equivalent of a diasteromeric mixture of (*R*,1*S*)- and (*S*,1*S*)-terphenphos in ethanol to K_2PtCl_4 in H_2O. Addition of further ethanol gave equal amounts of the title compound and its diastereomer. Crystallization from a dichloromethane solution layered with diethyl ether yielded first the title compound Pt[*o*-{(*S*)-(*tert*-C_4H_9)(C_6H_5)P}C_6H_4-(*S*)-$CHCH_3N(CH_3)_2$]Cl_2, (I), as clear pale-yellow equidimensional blocks. Crystal density by neutral buoyancy in 1,2-dibromoethane and hexane. Laue symmetry *mmm*, systematic absences *h* odd for *h*00, *k* odd for 0*k*0, *l* odd for 00*l*, defined space group $P2_12_12_1$ (*International Tables for Crystallography*, 1983, Vol. A). Crystal used for recording intensity data of approximate dimensions 0.25 × 0.26 × 0.27 mm, $V = 0.016$ mm^3, eight crystal faces, forms {100}, {010} and {101}. Enraf–Nonius CAD-4F diffractometer (Enraf–Nonius, 1984); cell parameters from 24 reflections with $20 < 2\theta < 55°$; Mo radiation monochromated by graphite. ω scans had an average width at half height of 0.11°. A total of 5017 observations by the θ–2θ scan method, variable-speed scans of width $(0.75 + 0.35\tan\theta)°$, 25% extensions at both ends for backgrounds. Maximum time per datum 60 s, maximum 2θ 65°, over a period of 88 h. Index ranges −1 to 21, −1 to 23 and −1 to 14. Standard reflections $41\bar{1}$, $\bar{5}\bar{5}4$ and $1\bar{8}2$, monitored every 3 h of X-ray exposure time, deviations random, no decay correction necessary. Background, monochromator and crystal polarization, and Lorentz corrections applied; Enraf–Nonius (1983) *Structure Determination Package* running on a DEC PDP 11/23+ computer. Analytical absorption correction (*AGNOST*; Cahen & Ibers, 1972), transmission factors from 0.185 to 0.274. *R* factor for averaging on *F* 0.009 (315 data) after correction. 4514 unique reflections with $I > 0$. Solution by Patterson and Fourier techniques. The chirality of the model was that expected from the synthesis. Parameters refined by full-matrix least-squares methods, minimizing $\sum w(|F_o| - |F_c|)^2$, weight $w = 4F_o^2/\sigma^2(F_o^2)$. Scattering factors for neutral atoms from Cromer & Liberman (1970), those for H atoms from Stewart, Davidson & Simpson (1965). Anomalous-dispersion corrections applied for all atoms, values from *International Tables for X-ray Crystallography* (1974, Vol. IV). With all 25 non-H atoms refined with anisotropic thermal parameters, refinement gave agreement factors $R = 0.036$ and $wR = 0.049$. 22 of 28 H atoms located in a difference Fourier synthesis, peak heights from 0.49 (13) to 0.24 (13) e Å^{-3}. All methyl H atoms located using a tilted Fourier routine (Enraf–Nonius, 1983). Rotational disorder found for *N*-methyl C(10), six H atoms of multiplicity 0.5 present. All included with idealized sp^2 and sp^3 geometries, isotropic Debye parameters 110% of those of bonded atoms. Methyl groups fitted by a least-squares procedure, positions optimized as refinement progressed, but not refined. 3719 unique observations with $I > 3\sigma(I)$, $p = 0.04$ (Busing & Levy, 1957), used to refine 227 variables. Evidence for secondary extinction, parameter refined to 1.17 (6) × 10^{-7} (Larsen, 1967). Convergence with updated H-atom positions at $R = 0.025$ and $wR = 0.029$, $S = 0.92$, $(\Delta/\sigma)_{\max} = 0.02$. Residual electron density 1.14 (14) e Å^{-3} (0.520, 0.457, 0.305) at 0.93 Å from the Pt atom; no chemical significance. Statistical analysis in terms of wR, F_o, Miller indices and diffractometer setting angles showed no unusual trends. The inverted model gave a poorer agreement, see below. Final atomic positional parameters are given in Table 1.

Determination of absolute configuration. The model was inverted and refined to convergence with updated H-atom positions, giving $R = 0.048$ and $wR = 0.069$, $S = 1.67$, and $(\Delta/\sigma)_{\max} = 0.02$, a significantly poorer result. The absolute configuration of (S) at C(7), that expected from the enantiomer used in the ligand synthesis, was confirmed by a comparison of Friedel pairs. Structure factors based on the final model and its enantiomer, differing by more than 10%, have been deposited.* The F_o values without exception indicate that the assignment of the absolute configuration (S,1S) to the complexed ligand is correct.

Discussion. A stereoview of the molecule showing the atomic numbering scheme is given in Fig. 1. The crystals consist of discrete molecules, since the shortest intermolecular contacts are 2.80 Å for HC(7)···Cl(2) at $(1 - x, \frac{1}{2} + y, \frac{1}{2} - z)$, and 2.83 Å for HC(14)···Cl(2) at $(x - \frac{1}{2}, \frac{1}{2} - y, -z)$. Intramolecular distances and angles are given in Table 2.

Table 2. *Intramolecular bond distances* (Å) *and angles* (°)

Pt—Cl(1)	2.3599 (15)	C(3)—C(4)	1.381 (9)
Pt—Cl(2)	2.2966 (15)	C(4)—C(5)	1.372 (12)
Pt—P	2.2365 (13)	C(5)—C(6)	1.370 (12)
Pt—N	2.106 (5)	C(7)—C(8)	1.512 (10)
P—C(2)	1.844 (6)	C(11)—C(12)	1.382 (8)
P—C(11)	1.840 (6)	C(11)—C(16)	1.383 (8)
P—C(17)	1.888 (6)	C(12)—C(13)	1.382 (9)
N—C(7)	1.512 (8)	C(13)—C(14)	1.377 (11)
N—C(9)	1.503 (8)	C(14)—C(15)	1.359 (10)
N—C(10)	1.498 (10)	C(15)—C(16)	1.391 (9)
C(1)—C(2)	1.406 (8)	C(17)—C(18)	1.525 (9)
C(1)—C(6)	1.389 (9)	C(17)—C(19)	1.531 (8)
C(1)—C(7)	1.510 (9)	C(17)—C(20)	1.534 (9)
C(2)—C(3)	1.399 (8)		

Cl(1)—Pt—Cl(2)	85.48 (7)	C(1)—C(2)—C(3)	120.1 (5)
Cl(1)—Pt—P	167.77 (6)	C(2)—C(3)—C(4)	119.9 (7)
Cl(1)—Pt—N	91.10 (14)	C(3)—C(4)—C(5)	120.3 (7)
Cl(2)—Pt—P	87.42 (5)	C(4)—C(5)—C(6)	119.8 (7)
Cl(2)—Pt—N	176.30 (14)	C(1)—C(6)—C(5)	122.2 (8)
P—Pt—N	95.70 (13)	N—C(7)—C(1)	115.0 (5)
Pt—P—C(2)	112.1 (2)	N—C(7)—C(8)	111.6 (5)
Pt—P—C(11)	106.7 (2)	C(1)—C(7)—C(8)	114.0 (6)
Pt—P—C(17)	119.3 (2)	P—C(11)—C(12)	117.8 (4)
C(2)—P—C(11)	102.4 (3)	P—C(11)—C(16)	123.8 (4)
C(2)—P—C(17)	105.5 (3)	C(12)—C(11)—C(16)	118.4 (6)
C(11)—P—C(17)	109.7 (3)	C(11)—C(12)—C(13)	121.6 (6)
Pt—N—C(7)	117.2 (4)	C(12)—C(13)—C(14)	119.6 (6)
Pt—N—C(9)	113.1 (5)	C(13)—C(14)—C(15)	119.4 (6)
Pt—N—C(10)	106.5 (4)	C(14)—C(15)—C(16)	121.4 (6)
C(7)—N—C(9)	104.9 (5)	C(11)—C(16)—C(15)	119.6 (6)
C(7)—N—C(10)	109.1 (5)	P—C(17)—C(18)	107.9 (4)
C(9)—N—C(10)	105.4 (6)	P—C(17)—C(19)	113.7 (4)
C(2)—C(1)—C(6)	117.6 (6)	P—C(17)—C(20)	111.9 (4)
C(2)—C(1)—C(7)	127.1 (5)	C(18)—C(17)—C(19)	107.6 (5)
C(6)—C(1)—C(7)	115.4 (6)	C(18)—C(17)—C(20)	108.1 (6)
P—C(2)—C(1)	122.3 (4)	C(19)—C(17)—C(20)	107.5 (6)
P—C(2)—C(3)	117.6 (5)		

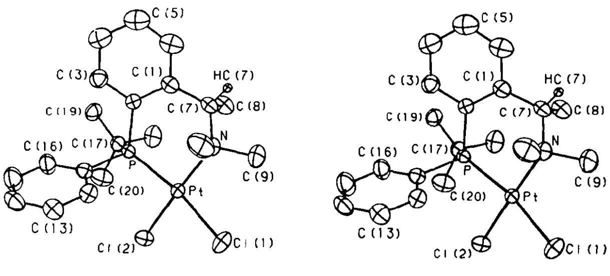


Fig. 1. A stereoview of the molecule, showing the atomic numbering scheme.

The four-coordinate Pt atom displays a small distortion from square-planar geometry. The chloride ligands Cl(1) and Cl(2) are displaced from the plane formed by P, Pt and N by 0.417 (2) and 0.080 (2) Å respectively, giving a dihedral angle between the Pt—Pt—N and Cl(1)—Pt—Cl(2) planes of 10.3 (5)°. The Pt—Cl(2) length of 2.297 (2) Å *trans* to N is comparable, for example, to the mean value of 2.303 (5) Å in the *cis*-diammine complex $PtCl_2\{(CH_3)NHCH_2CH(CH_3)NH_2\}$ (Ball, Bowman & Payne, 1976). As expected, the Pt—Cl(1) distance of 2.360 (2) Å, *trans* to P, is significantly longer (30σ), consistent with the greater *trans* influence of P relative to N. A similar variation in Pt—Cl distances, 2.390 (8) Å *trans* to P, and 2.314 (10) Å *trans* to N, was found by Cullen, Evans, Han & Trotter (1987) in their study of a platinum complex containing a (P—N) ferrocenylphosphine ligand.

Complexes of Rh and Pd containing other closely related ligands have been studied. These included the diphenylphosphino salt $[Rh\{(R)\text{-}o\text{-}(C_6H_5)_2PC_6H_4CHCH_3N(CH_3)_2\}(C_7H_8)]ClO_4$, (II), (McKay, 1986), the di-*tert*-butylphosphino species $[Rh\{(R)\text{-}o\text{-}(tert\text{-}C_4H_9)_2PC_6H_4CHCH_3N(CH_3)_2\}(C_7H_8)]ClO_4$, (III), (McKay & Payne, 1986*a*), and an *n*-butylphenylphosphino analog $Pd[o\text{-}\{(R)\text{-}(n\text{-}C_4H_9)(C_6H_5)P\}\text{-}C_6H_4\text{-}(S)\text{-}CHCH_3N(CH_3)_2]Cl_2$, (IV), (Takenaka, Sasada, Yamamoto & Tsuji, 1977). Dimensions in the ligand in (I) agree well with those found for (II)–(IV). The chelate backbone phenylene ring has an average C—C length of 1.390 (4) Å and C—C—C angles ranging from 117.6 (6) to 122.3 (8)°. The phenyl ring is planar within experimental error, with an average C—C distance of 1.380 (4) Å and angles ranging from 118.4 (6) to 121.5 (6)°. The average

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* Lists of anisotropic thermal parameters, H-atom parameters, Friedel pairs, root-mean-square amplitudes of vibration, selected torsion angles, weighted least-squares planes, and structure amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54553 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0057]

C—C length in the *tert*-butyl group, 1.530 (5) Å, is in excellent agreement with that in (III), 1.527 (5) Å.

Bond distances associated with the P atom reflect the steric bulk of its substituents. Thus the P—C(2) length in (I) of 1.844 (6) Å, from P to the backbone phenylene ring, falls directly between the P—C distances in (II) and (III) of 1.821 (4) and 1.864 (7) Å respectively. Similarly, the P—phenyl-C(11) distance of 1.840 (6) Å in (I) is significantly longer than that of 1.818 (5) Å in (IV), and the mean value of 1.827 (3) Å in (II). The bond length P—*tert*-butyl-C(17) of 1.888 (6) Å in (I) is considerably shorter than the mean value of 1.917 (6) Å in the di-*tert*-butyl complex (III), while the P—*n*-butyl-C(17) distance in (IV) is only 1.817 (5) Å.

The six-membered chelate ring adopts a twist-boat conformation of absolute configuration λ, in which atoms C(2), C(1) and C(7) are displaced by 0.149 (5), -0.250 (6) and -0.789 (6) Å respectively from the Pt—P—N plane. The C(8) methyl group is axially disposed, as is the C(10) *N*-methyl group, while C(9) is equatorial. The sterically uncrowded position of C(10) is consistent with the twofold disorder observed in a difference Fourier synthesis. The *tert*-butyl and phenyl substituents on the P atom bracket the plane of the chelate ring almost equally, showing no tendency to occupy pseudo-axial and pseudo-equatorial positions.

In conclusion, the absolute configuration of the less-soluble diastereomer of *cis*-$PtCl_2$(terphenphos) crystallized from methylene chloride–diethyl ether mixtures has been shown to be (*S*,1*S*); that of the free ligand is therefore (*R*,1*S*). Studies of the efficacy of complexes of this and related ligands in asymmetric synthesis are in progress.

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References

Ball, R. G., Bowman, N. J. & Payne, N. C. (1976). *Inorg. Chem.* **15**, 1704–1707.

Busing, W. R. & Levy, H. A. (1957). *J. Chem. Phys.* **26**, 563–568.

Cahen, D. & Ibers, J. A. (1972). *J. Appl. Cryst.* **5**, 298–299.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cullen, W. S., Evans, S. V., Han, N. F. & Trotter, J. (1987). *Inorg. Chem.* **26**, 514–519.

Enraf–Nonius (1983). *Enraf–Nonius Structure Determination Package*. Version 3.0. Enraf–Nonius, Delft, The Netherlands.

Enraf–Nonius (1984). *Enraf–Nonius CAD-4 Operators Manual.* Enraf–Nonius, Delft, The Netherlands.

Larsen, A. C. (1967). *Acta Cryst.* **23**, 664–665.

McKay, I. D. (1986). PhD thesis, Univ. of Western Ontario, Canada.

McKay, I. D. & Payne, N. C. (1986*a*). *Can. J. Chem.* **64**, 1930–1935.

McKay, I. D. & Payne, N. C. (1986*b*). *Acta Cryst.* C**42**, 304–306.

Payne, N. C. & Stephan, D. W. (1982). *Inorg. Chem.* **21**, 182–188.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Takenaka, A., Sasada, Y., Yamamoto, K. & Tsuji, J. (1977). *Bull. Chem. Soc. Jpn*, **50**, 3177–3181.

Tobin, G. R. & Payne, N. C. (1992). In preparation.

Acta Cryst. (1992). C**48**, 48–50

Structure of Sodium 4-(4-Hydroxyphenylazo)benzoate Tetrahydrate

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Abstract. $Na^+.C_{13}H_9N_2O_3^-.4H_2O$, $M_r = 336.28$, $P\bar{1}$, $Z = 2$, $a = 7.297$ (1), $b = 17.704$ (2), $c = 6.343$ (1) Å, $\alpha = 94.03$ (1), $\beta = 107.71$ (1), $\gamma = 83.40$ (1)°, $V = 774.9$ Å^3, $D_x = 1.441$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, graphite monochromator, $\mu = 0.132$ mm^{-1}, $F(000) = 352$, $T = 173$ K, $R = 0.037$,

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