

larger average Cu—Br...Br angle. The data point for $n = 5$ lies further from the line than would be predicted from its geometrical parameters and it appears that the experimental value of $|J_{2h}|$ may have been overestimated for this latter case.

Straatman *et al.* (1984) have also shown that J_{2h} depends strongly upon the lengths of the Cu—X bonds involved in the two-halide bridges with $|J_{2h}|$ increasing as the Cu—X length shortens. For the bromide series, there is little variation in these lengths, so it would not appear to be an important factor. For the chlorides, in contrast, there are significant differences which need to be taken into account when discussing those correlations.

The authors acknowledge the support of NSF grant DMR-8219430. In addition, the X-ray diffraction facility was established through funds supplied by NSF grant CHE-8408407 and by The Boeing Company. Appreciation is expressed to Professor J. K. Garland for sharing results prior to publication.

References

- ARAMUBURU, J. A. & MORENO, M. (1985). *J. Chem. Phys.* **83**, 6071–6077.
- AREND, H., HUBER, W., MISCKOVSKY, F. H. & VAN LEEUWEN, G. R. K. (1978). *J. Cryst. Growth*, **43**, 213–223.
- BARENDREGT, F. & SCHENK, H. (1970). *Physica (Utrecht)*, **49**, 465–468.
- BLOCK, R. & JANSEN, L. (1982). *Phys. Rev. B*, **26**, 148–153.
- CAMPANA, C. F., SHEPHERD, D. F. & LITCHMAN, W. N. (1981). *Inorg. Chem.* **20**, 4039–4044.
- CHOW, C., CHANG, K. & WILLETT, R. D. (1973). *J. Chem. Phys.* **59**, 2629–2640.
- FERGUSON, G. L. & ZASLOW, B. (1971). *Acta Cryst.* **B27**, 849–852.
- GARLAND, J. K., EMERSON, K. & PRESSPRICH, M. (1989). Submitted.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht, The Netherlands.)
- JONGH, L. J. DE & MIEDEMA, A. (1974). *Adv. Phys.* **23**, 1–260.
- LANDEE, C. P., HALVORSON, K. & WILLETT, R. D. (1987). *J. Appl. Phys.* **61**, 3295–3297.
- LARSEN, K. P. (1974). *Acta Chem. Scand. Ser. A*, **28**, 194–200.
- PHELPS, D. W., LOSEE, D. B., HATFIELD, W. E. & HODGSON, D. J. (1976). *Inorg. Chem.* **15**, 3147–3152.
- RUBENACKER, G. V., WAPLAK, S., HUTTON, S. L., HAINES, D. N. & DRUMHELLER, J. E. (1985). *J. Appl. Phys.* **57**, 3341–3342.
- SCOTT, B. & WILLETT, R. D. (1987). *J. Appl. Phys.* **61**, 3289–3291.
- SHELDRIK, G. M. (1985). *SHELXTL*. Version 5.1. Nicolet Instrument Corporation, Madison, WI, USA.
- SNIVELY, L. O., HAINES, D. N., EMERSON, K. & DRUMHELLER, J. E. (1982). *Phys. Rev. B*, **26**, 5245–5247.
- SNIVELY, L. O., SEIFERT, P. L., EMERSON, K. & DRUMHELLER, J. E. (1979). *Phys. Rev. B*, **20**, 2101–2104.
- SNIVELY, L. O., TUTHILL, G. & DRUMHELLER, J. E. (1981). *Phys. Rev. B*, **24**, 5349–5355.
- STEADMAN, J. P. & WILLETT, R. D. (1970). *Inorg. Chim. Acta*, **4**, 367–371.
- STRAATMAN, P., BLOCK, R. & JANSEN, L. (1984). *Phys. Rev. B*, **29**, 1415–1418.
- TICHY, K., BENES, J., HÄLG, W. & AREND, H. (1978). *Acta Cryst.* **B34**, 2970–2981.
- WILLETT, R. D. (1964). *J. Chem. Phys.* **41**, 2243–2244.
- WILLETT, R. D. (1986). *Inorg. Chem.* **25**, 1918–1920.
- WILLETT, R. D., GATTESCHI, D. & KAHN, O. (1985). Editors. *Magnetostructural Correlations in Exchange Coupled Systems*. Dordrecht: Reidel.
- WILLETT, R. D., PLACE, H. & MIDDLETON, M. (1988). *J. Am. Chem. Soc.* Accepted.

Acta Cryst. (1988). **C44**, 2076–2079

The Synthesis and Structure of (*N*-{2-[2-(2-Ammonioethylamino)ethylamino]ethyl}-salicylideneaminato-*O,N,N',N''*)nickel(II) Perchlorate

BY G. BRUNO, F. CUSMANO PRIOLO, F. NICOLÒ AND E. ROTONDO

Dipartimento di Chimica Inorganica e Struttura Molecolare, Università di Messina, Vill. S. Agata, salita Sperone 31, 98100 Messina, Italy

(Received 5 November 1987; accepted 13 May 1988)

Abstract. [Ni(C₁₃H₂₂N₄O)](ClO₄)₂, $M_r = 507.9$, triclinic, $P\bar{1}$, $a = 8.699$ (1), $b = 10.029$ (1), $c = 11.923$ (1) Å, $\alpha = 94.34$ (3), $\beta = 108.63$ (4), $\gamma = 96.10$ (3)°, $V = 973.4$ (4) Å³, $Z = 2$, $D_x = 1.73$ Mg m⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 12.5$ cm⁻¹, $F(000) = 524$, $T = 298$ K, final $R = 0.055$ and $wR = 0.061$ for 2900 independent reflections [$I > 3\sigma(I)$]. The coordination polyhedron around Ni is an irregular square pyramid with the protonated saltrien [saltrien = 2-O⁻-C₆H₄CH=N(CH₂)₂NH(CH₂)₂NH(CH₂)₂NH₂]

acting as tetradentate ligand through one O and three N atoms.

Introduction. Structural, electronic and magnetic properties of Schiff-base coordination compounds have been measured in depth and form a significant part of our knowledge of the inorganic chemistry of chelate systems (Holm & O'Connor, 1971). In recent years Schiff-base complexes have been proposed as a model to describe energy transfer in naturally occurring systems,

and the role of the coordination sphere about the metal ion in determining the nature of the model system has been greatly emphasized (Wilkins, 1971). A more recent application of Schiff bases arose from the observation that the metal ion can act as a template in the condensation of carbonyl derivatives with diprimary polyfunctional amines (Casellato, Vigato, Fenton & Vidali, 1979). In this case suitable experimental conditions can lead to the synthesis of diimine complexes still containing a primary NH_2 group, and therefore capable of condensation with a second carbonyl derivative. Thus the 1:1 condensation product could be seen as an isolable intermediate ('half unit') of a two-step process for the synthesis of non-symmetrical Schiff bases (Costes, Cross, Darbien & Laurent, 1982; Bett, Fenton & Tate, 1981). The interest in this kind of compound is related to the possibility which they offer for a sharper modulation of the electronic and steric factors governing their known 'oxygen transfer' ability (Costa, Mestroni, Puxeddu & Reisenhofer, 1970). In this context we have previously described the nickel(II) and copper(II) 1:1 template synthesis of diethylenetriamine and salicylaldehyde. The tetradentate behaviour of the 'half unit' was demonstrated by a crystal structure determination of these complexes (Cusmano Priolo, Rotondo, Rizzardi, Bruno & Bombieri, 1983; Rotondo, Cusmano Priolo, Romeo, Bruno & Bombieri, 1983). In the present paper we describe the nickel(II) template condensation of one molecule of salicylaldehyde and one molecule of diethylenetriamine which leads to a potentially pentadentate ligand; the crystal structure determination demonstrated that in this case also the 'half unit' acts as a tetradentate ligand.

Experimental. 5 mmol of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was suspended in methanol (20 cm^3), 5 mmol of salicylaldehyde in methanol (10 cm^3) was added and the resulting solution added dropwise to 5 mmol of triethylenetetramine (trien) in methanol (30 cm^3). The reaction mixture was allowed to stand for 2 d at room temperature and then 10 cm^3 of water saturated with NaClO_4 added. Brown needle crystals, not fully characterized, separated from the solution and slow evaporation of part of the solvent yielded red crystals of the title compound. Analytical data (wt%), calculated values in parentheses, and the characteristic IR bands (cm^{-1}): $\text{C}_{13}\text{H}_{22}\text{Cl}_2\text{N}_4\text{NiO}_6$, C 30.72 (30.95), H 4.33 (4.30), N 11.03 (10.79); $\nu(\text{NH})$ 3200 (*br*), $\nu(\text{C}=\text{N})$ 1640 (*vs*).

The conductivity was measured with a Radiometer CDM 3 conductivity bridge, the electronic spectrum recorded on a Cary 219 double-beam spectrophotometer, and the vibrational spectrum recorded on a Perkin-Elmer spectrophotometer. Single crystals, crystallized from a methanol solution; a red prism with approximate dimensions $0.15 \times 0.15 \times 0.10 \text{ mm}$ was used for intensity-data collection. Accurate unit-cell

dimensions and crystal-orientation matrices were obtained from least-squares refinement of 2θ , ω , χ and ψ values of 20 strong reflections in the range $14^\circ < 2\theta < 28^\circ$. Siemens-Stoe four-circle diffractometer using graphite-monochromated $\text{Mo } K\alpha$ ($\lambda = 0.71069 \text{ \AA}$) radiation, $\omega/2\theta$ mode, $2\theta_{\text{max}} = 50^\circ$ ($0 \leq h \leq 13$, $-14 \leq k \leq 14$, $-16 \leq l \leq 16$). Three standard reflections ($\bar{5}33$, $\bar{4}\bar{3}\bar{4}$ and $43\bar{3}$), monitored every 3600 s, showed no significant intensity variation over the total exposure time. Lorentz and polarization corrections were applied to the intensity data but no correction for absorption was considered.

The structure was solved by using standard Patterson methods, successive least-squares refinements, and difference Fourier maps. All non-hydrogen atoms were refined anisotropically, while hydrogen atoms were added at calculated positions and included in the structure factor calculations with a common thermal parameter ($U = 0.07 \text{ \AA}^2$). Of 3800 measured independent reflections, 2900 having $I > 3\sigma(I)$ were used to refine 274 parameters to final residuals of $R = 0.055$ and $wR = 0.061$, $w = 3.2823/[\sigma^2(F_o) + 0.00023F_o^2]$, $S = 2.146$, $(\Delta/\sigma)_{\text{max}} < 0.1$, largest peak = 0.88 e \AA^{-3} near the oxygen atoms of the perchlorate anion. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed with *SHELX76* (Sheldrick, 1976) and the *PARST* (Nardelli, 1983) set of programs on the IBM 4341 computer at the Centro di Calcolo dell'Università di Messina. The refined structure was plotted with the use of *PLUTO* (Motherwell & Clegg, 1978) (Figs. 1 and 2).

Discussion. Final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1; bond lengths and angles are listed in Table 2.* A view of the molecule is shown in Fig. 1. Fig. 2 shows the packing diagram. In the cation the nickel atom is four coordinate with three nitrogen and one oxygen atoms of the saltrien as ligands. The weighted mean plane through the four basal donor atoms shows a lack of planarity with N1 and N3 deviating by 0.128 (5) and 0.124 (5) \AA , respectively, while N2 and O1 are -0.057 (5) and -0.022 (3) \AA on the opposite side, indicating a distortion of the basal plane towards the tetrahedral configuration; the nickel atom is -0.008 (1) \AA out of this plane. This distortion is similar to that found in the parent compound $\text{Cu}(\text{saltrien})$ (Rotondo, Cusmano Priolo, Bombieri & Bruno, 1984) while in $\text{Cu}(\text{saldien})$ (Cusmano Priolo, Rotondo, Rizzardi, Bruno & Bombieri, 1983) and in $\text{Ni}(\text{saldien})$

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51038 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^2$) for non-hydrogen atoms, with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Ni	1380 (1)	2023 (1)	2470 (1)	4.10 (2)
C11	7330 (2)	2924 (1)	10235 (1)	4.81 (4)
O2	5948 (6)	2678 (5)	9210 (4)	8.20 (17)
O3	8679 (6)	2572 (7)	9938 (6)	10.20 (25)
O4	7638 (10)	4252 (5)	10623 (5)	12.45 (29)
O5	7172 (9)	2160 (8)	11109 (6)	13.55 (34)
C12	5735 (2)	2532 (1)	5051 (1)	4.84 (4)
O6	5098 (12)	3617 (9)	5446 (11)	18.42 (48)
O7	6413 (8)	3041 (8)	4260 (6)	11.86 (33)
O8	4376 (8)	1611 (8)	4524 (8)	13.90 (32)
O9	6799 (11)	2316 (9)	6037 (7)	20.16 (42)
O1	775 (4)	204 (5)	2101 (3)	4.44 (12)
C1	-281 (6)	-551 (5)	2472 (4)	4.15 (15)
C2	-671 (7)	-1914 (6)	2027 (5)	4.80 (17)
C3	-1793 (7)	-2731 (5)	2337 (6)	5.45 (20)
C4	-2503 (6)	-2232 (7)	3124 (6)	5.50 (20)
C5	-2103 (6)	-942 (6)	3587 (6)	5.24 (20)
C6	-997 (6)	-54 (5)	3272 (5)	4.34 (15)
C7	-630 (7)	1313 (6)	3804 (5)	4.98 (17)
N1	306 (6)	2222 (4)	3546 (4)	4.70 (14)
C8	529 (8)	3600 (6)	4129 (6)	5.94 (19)
C9	897 (7)	4499 (6)	3268 (6)	5.84 (21)
N2	2102 (6)	3899 (4)	2855 (4)	5.04 (15)
C10	2456 (9)	4443 (6)	1851 (6)	6.01 (22)
C11	3310 (8)	3427 (6)	1359 (5)	5.71 (20)
N3	2325 (6)	2107 (5)	1203 (4)	4.75 (15)
C12	3081 (7)	1006 (6)	791 (5)	5.03 (19)
C13	4415 (7)	561 (6)	1761 (5)	5.28 (19)
N4	3823 (5)	-426 (4)	2429 (4)	4.76 (14)

Table 2. Bond lengths (\AA) and bond angles ($^\circ$)

Ni—O1	1.824 (3)	Ni—N1	1.823 (6)
Ni—N2	1.891 (4)	Ni—N3	1.940 (6)
C1—C2	1.394 (7)	C1—C6	1.386 (8)
C2—C3	1.366 (9)	C3—C4	1.371 (10)
C4—C5	1.332 (9)	C5—C6	1.399 (9)
C6—C7	1.425 (8)	C7—N1	1.278 (8)
N1—C8	1.464 (7)	C8—C9	1.502 (10)
C9—N2	1.457 (9)	N2—C10	1.458 (9)
C10—C11	1.510 (10)	C11—N3	1.463 (8)
N3—C12	1.474 (9)	C12—C13	1.488 (8)
C13—N4	1.467 (8)	C11—O2	1.397 (4)
C11—O3	1.400 (7)	C11—O4	1.346 (5)
C11—O5	1.372 (8)	C12—O6	1.389 (11)
C12—O7	1.365 (8)	C12—O8	1.360 (7)
C12—O9	1.293 (8)	O1—C1	1.332 (7)

N1—Ni—N2	86.0 (2)	N2—Ni—N3	86.8 (2)
N3—Ni—O1	91.6 (2)	O1—Ni—N1	95.8 (2)
Ni—O1—C1	126.1 (3)	O1—C1—C2	117.5 (5)
O1—C1—C6	123.6 (5)	C1—C2—C3	120.4 (5)
C2—C3—C4	120.5 (5)	C3—C4—C5	119.8 (6)
C4—C5—C6	121.9 (6)	C5—C6—C1	118.5 (5)
C5—C6—C7	118.7 (5)	C7—C6—C1	122.8 (5)
C6—C7—N1	124.1 (5)	C7—N1—Ni	127.2 (4)
C7—N1—C8	118.4 (5)	C8—N1—Ni	114.4 (4)
N1—C8—C9	105.3 (5)	C8—C9—N2	105.9 (5)
C9—N2—Ni	107.6 (3)	C9—N2—C10	116.8 (5)
N2—C10—C11	106.9 (5)	C10—C11—N3	106.9 (5)
C11—N3—Ni	109.0 (3)	C11—N3—C12	112.4 (5)
N3—C12—C13	113.5 (5)	C12—C13—N4	113.5 (5)
O2—C11—O3	108.6 (3)	O2—C11—O4	109.4 (3)
O2—C11—O5	112.2 (4)	O3—C11—O4	107.6 (4)
O3—C11—O5	107.2 (4)	O4—C11—O5	111.6 (4)
O6—C12—O7	103.7 (5)	O6—C12—O8	102.6 (6)
O6—C12—O9	100.8 (6)	O7—C12—O8	113.3 (5)
O7—C12—O9	113.8 (5)	O8—C12—O9	119.6 (5)
C2—C1—C6	118.9 (5)	Ni—N2—C10	109.5 (4)

(Rotondo, Cusmano Priolo, Romeo, Bruno & Bombieri, 1983) the coordination around each metal atom is essentially planar.

The mean values of the bond lengths and angles around the metal atom are in good agreement with those found previously in the Ni(saldien) complex; the only significant difference involves the Ni—O1 and Ni—N3 lengths which are 1.824 (3) and 1.940 (6) \AA in the present compound while the corresponding values of the analogous copper compound were 1.795 (5) and 1.915 (6) \AA . The ring O1—Ni—N1 is nearly planar, the deviations of the atoms from their weighted mean plane are: Ni 0.005 (1), N1 -0.050 (5), O1 -0.044 (3), C7 -0.022 (6), C6 0.057 (6) and C1 0.025 (5) \AA . The phenyl ring makes an angle of 4.1 (2) $^\circ$ with this plane.

The conformations of the five-membered rings of the saltrien ligand are very close to those adopted in the analogous copper derivative (Rotondo *et al.*, 1984): ring N1—Ni—N2 envelope [C8 and C9 deviating by -0.014 (7) and 0.612 (7) \AA , respectively] and N2—Ni—N3 half-chair [C10 and C11 0.415 (7) and

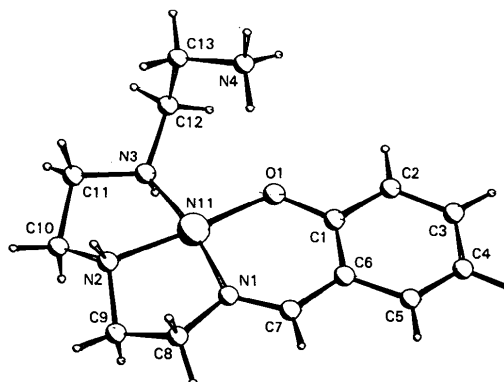


Fig. 1. A view of the molecule.

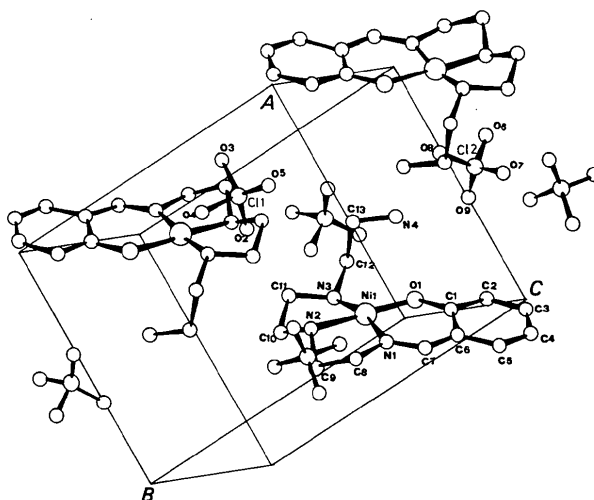


Fig. 2. Packing diagram.

0.253 (7) Å]. The torsion angles of the two ethylenic bridges are $-45.6(6)^\circ$ for N1–C8–C9–N2 and $49.4(6)^\circ$ for N2–C10–C11–N3 and the corresponding C–C bond lengths are 1.502 (10) and 1.510 (10) Å, respectively, in good agreement with the corresponding values found in the Cu(saltren) complex [both 1.498 (9) Å]. It seems likely that such values are the real ones for the C–C distances while the apparently shorter values found in various salen complexes can be attributed to thermal motion of the ethylene bridges in the crystals (Rotondo *et al.*, 1984; Bresciani-Pahor *et al.*, 1976).

This structure confirms that the presence of a hydrogen bond from the phenoxy oxygen to the terminal nitrogen is usual in these kinds of complexes. The final difference Fourier map, as in the analogous copper compound, shows some electron-density residues of the order $0.9 \text{ e } \text{Å}^{-3}$ near to the non-coordinated nitrogen (N4) in a position characteristic for three hydrogen atoms bound to sp^3 -hybridized nitrogen atoms. One of these hydrogens (H43) forms a short contact of 1.983 (3) Å with the oxygen of the saltren ligand; the separation N4...O1 is 2.705 (6) Å, characteristic of a hydrogen bond. The molecular packing (Fig. 2) is mainly determined by normal van der Waals interactions and by some hydrogen bonds involving the oxygen atoms of the perchlorate anions; one of them makes a long-range interaction with the metal atom [Ni...O8 3.044 (7) Å] in the apical position.

The molar conductivity of the complex in methanol at 298 K is $188 \text{ } \Omega \text{ cm}^2 \text{ mol}^{-1}$; this value is in the range for a 2:1 electrolyte type and suggests that the complex, analogously to the copper derivative, acts in methanol as a dipositively charged species.

Magnetic susceptibilities measured by the Evans (1959) method revealed that the complex is dia-

magnetic when dissolved in methanol. The electronic spectrum in the same solvent shows a very intense band at 380 nm which is typical for all deprotonated salicylaldehyde derivatives; in the tail of this intense band, a shoulder can be seen at 445 nm, attributable to a $d-d$ transition of a d^8 Ni atom in an approximately square-planar arrangement. The diffuse reflectance spectrum of the solid powdered compound recorded in the same region shows a well resolved maximum at 448 nm.

References

- BETT, G., FENTON, D. E. & TATE, J. R. (1981). *Inorg. Chim. Acta*, **54**, 101–102.
- BRESCIANI-PAHOR, N., CALLIGARIS, M., DELISE, P., NARDIN, G., RANDACCIO, L., ZOTTI, E., FACCHINETTI, G. & FLORIANI, C. (1976). *J. Chem. Soc. Dalton Trans.* pp. 2310–2316.
- CASELLATO, V., VIGATO, P. A., FENTON, D. E. & VIDALI, M. (1979). *Chem. Soc. Rev.* **8**, 199–220.
- COSTA, G., MESTRONI, G., PUXEDDU, A. & REISENHOFER, E. (1970). *J. Chem. Soc. A*, pp. 2870–2874.
- COSTES, J. P., CROSS, G., DARBIEN, M. H. & LAURENT, J. P. (1982). *Inorg. Chim. Acta*, **60**, 111–114.
- CUSMANO PRIOLO, F., ROTONDO, E., RIZZARDI, G., BRUNO, G. & BOMBIERI, G. (1983). *Acta Cryst.* **C39**, 550–552.
- EVANS, D. F. (1959). *J. Chem. Soc.* pp. 2003–2005.
- HOLM, R. H. & O'CONNOR, M. J. (1971). *Prog. Inorg. Chem.* **14**, 241–401.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
- ROTONDO, E., CUSMANO PRIOLO, F., BOMBIERI, G. & BRUNO, G. (1984). *Acta Cryst.* **C40**, 960–962.
- ROTONDO, E., CUSMANO PRIOLO, F., ROMEO, M., BRUNO, G. & BOMBIERI, G. (1983). *Acta Cryst.* **C39**, 1525–1527.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- WILKINS, R. G. (1971). *Adv. Chem. Ser.* No. 100, pp. 111–126.

Acta Cryst. (1988). **C44**, 2079–2082

Structure of a *nido*-Metalla-Monocarbon Carbaborane, [5-(PPh₃)-9,9,9-(H)(PPh₃)₂-9,6-IrCB₈H₁₀]

BY NATHANIEL W. ALCOCK, JOHN G. TAYLOR AND MALCOLM G. H. WALLBRIDGE

Department of Chemistry, University of Warwick, Coventry CV4 7AL, England

(Received 9 May 1988; accepted 25 July 1988)

Abstract. 5,9,9-Tris(triphenylphosphine)-6,9-carbaborane(14), C₅₅H₅₆B₈IrP₃ (excluding solvent), $M_r = 1088.71$, orthorhombic, $Pna2_1$, $a = 32.796(10)$, $b = 17.945(7)$, $c = 10.656(4)$ Å, $U =$

$6271(3)$ Å³, $Z = 4$, $D_x = 1.15 \text{ g cm}^{-3}$, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu(\text{Mo } K\alpha) = 22.2 \text{ cm}^{-1}$, $F(000) = 2192$, $T = 290 \text{ K}$, $R = 0.069$ for 2206 unique observed [$I/\sigma(I) \geq 3.0$] reflections. A minor product from