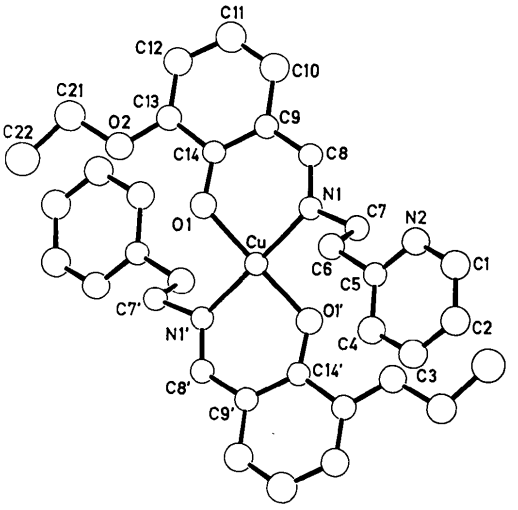

Fig. 1. Plot of the molecule showing the numbering scheme.

1·996 (3) Å, respectively. These bond distances are similar to those found in related salicylaldiminato complexes (Lingafelter & Braun, 1966) and also found in bis{*N*-[2-(2-pyridyl)ethyl]-4,6-dimethoxysalicylideneaminato}copper(II) (Atherton, Fenton, Hewson, McLean, Bastida, Romero, Sousa & Castellano, 1988). In the Schiff base, the N(1)—C(8) bond length of 1·294 (5) Å is in agreement with the value of 1·30 Å proposed for a C—N double bond (Brown, Towns & Trefonas, 1970). The pyridyl N atom is not coordinated to the metal. The bond lengths and angles in this ring are close to the expected values for a heteroaromatic ring.

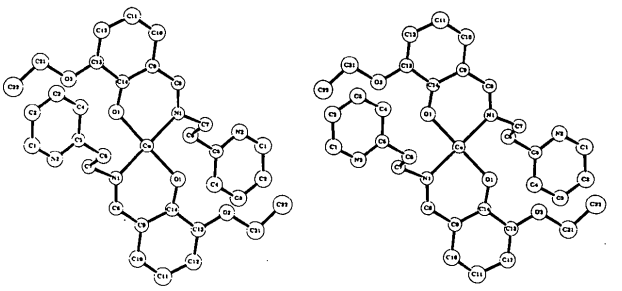

Fig. 2. Stereoscopic view.

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Bis(*N*-isopropyl-5-bromosalicylaldiminato)nickel(II)

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Abstract. Bis[4-bromo-2-(isopropyliminomethyl)-phenolato]nickel(II), $[Ni(C_{10}H_{11}BrNO)_2]$, M_r = 540·90, triclinic, $P\bar{1}$, a = 8·789 (6), b = 10·203 (7), c = 6·075 (4) Å, α = 103·92 (5), β = 80·23 (5), γ = 110·21 (5)°, V = 493·9 (6) Å^3, Z = 1, D_x = 1·818 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 5·01 mm^{-1}, $F(000)$ = 272, T = 293 K, R = 0·055 for 1189 unique observed reflections [$F > 3\sigma(F)$]. The Ni atom at the symmetry centre is *trans*-planar coor-

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dinated by two N and two O atoms. The intraligand O—Ni—N angles are 87·6 and 92·4°; the Ni—O and Ni—N bond lengths are 1·829 and 1·938 Å, respectively.

Introduction. There is only a small energy difference between the diamagnetic and the paramagnetic state of the Ni^{II} ion. Its relation to the coordination of the metal atom could be provided by a series of ring-substituted salicylaldimine chelates of Ni^{II}. Holm & Swaminathan (1963) reported that for 3-substituted bis(*N*-isopropylsalicylaldiminato)nickel compounds the magnetic moments are 3·28, 0 and 3·30 BM (1 BM $\simeq 9{\cdot}274 \times 10^{-24}$ J T^{-1}) for the substituents hydrogen, methyl and ethyl, indicating the coordination configurations tetrahedral (Fox, Orioli, Lingafelter & Sacconi, 1964), planar (Braun & Lingafelter, 1966) and tetrahedral (Braun & Lingafelter, 1967), respectively. In this work we have determined the *N*-isopropyl-5-bromo variant (Vargas, Araya & Costamagna, 1988).

Experimental. A crystal of dimensions 0·2 × 0·25 × 0·1 mm was used on a Nicolet *R3m/V* diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, unit-cell dimensions were from 20 centred reflections $3 < \theta < 7{\cdot}5°$. Wyckoff scan was used for the data collection of 1757 unique reflections of which 1189 were observed with $F > 3\sigma(F)$. According to the pre-scan intensity the Wyckoff-scan speed ranged from 1·5 to 19·5° min^{-1}. Absorption correction was based on ψ scan of 11 reflections (max. and min. transmission factors 0·544 and 0·441 respectively). Diffraction intensities were measured up to $\sin\theta/\lambda = 0{\cdot}60$ $Å^{-1}$ in the index range $h = -10$–10, $k = -12$–11 and $l =$ 0–7. Three standard reflections 211, $2\bar{3}\bar{1}$ and $\bar{1}01$ varied less than 2·1% over 57·5 h of data collection. Solved by direct phase determination, $E_{min} = 1{\cdot}2$. Full-matrix least squares minimized $w(\Delta F)^2$; H-atom positions were calculated geometrically and considered isotropically with $U = 1{\cdot}2U$ of bonded C. All other atoms refined anisotropically for 124 variables. $R = 0{\cdot}055$, $wR = 0{\cdot}041$, $S = 1{\cdot}60$, where $w^{-1} = \sigma^2(F)$. Final $(\Delta/\sigma)_{max} = 0{\cdot}001$, $\Delta\rho_{max} = 0{\cdot}8$ and $\Delta\rho_{min} = -0{\cdot}5$ e $Å^{-3}$ on final difference Fourier map. Atomic scattering factors were taken from *SHELXTL-PLUS* program (Sheldrick, 1988). Table 1 gives the atom parameters and Fig. 1 shows the molecule with the atom labelling.*

* Lists of bond lengths and angles in the title compound and in differently substituted complexes, atomic coordinates of H atoms, anisotropic thermal parameters, distances from the best plane of the salylaldimine residue and structure factors, and a stereoview of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52557 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic parameters* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

	x	*y*	*z*	U_{eq}*
Ni	0	0	0	37 (1)
Br	−2810 (1)	5419 (1)	−2343 (2)	58 (1)
O	−871 (6)	1397 (5)	1505 (8)	47 (2)
N	1122 (7)	1052 (6)	−2386 (9)	36 (3)
C(1)	−611 (9)	2558 (7)	−1561 (12)	38 (3)
C(2)	−1286 (9)	2255 (7)	603 (12)	37 (3)
C(3)	−2393 (9)	2933 (7)	1856 (12)	42 (3)
C(4)	−2830 (9)	3857 (7)	976 (12)	43 (4)
C(5)	−2172 (10)	4130 (7)	−1189 (13)	43 (3)
C(6)	−1090 (10)	3519 (8)	−2409 (13)	43 (4)
C(7)	638 (9)	2015 (7)	−2829 (12)	40 (3)
C(8)	2578 (9)	747 (8)	−3786 (12)	41 (3)
C(9)	3155 (10)	1409 (9)	−5892 (12)	56 (4)
C(10)	3934 (9)	1248 (9)	−2237 (13)	59 (34)

* Equivalent isotropic *U* defined as one third of the trace of the orthoganalized U_{ij} tensor.

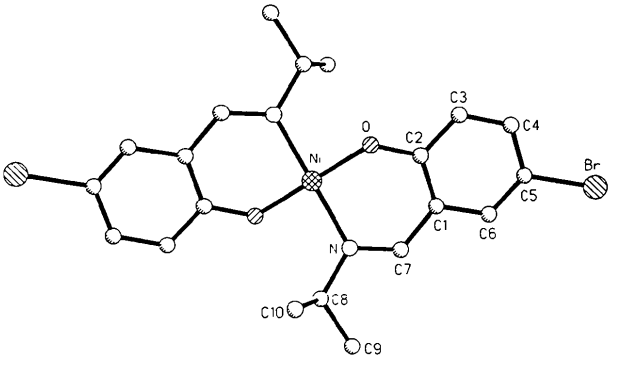

Fig. 1. Perspective drawing of the complex with the atoms labelled according to the table.

Discussion. The crystal structure consists of discrete molecules with fourfold coordination of the Ni^{II} ion.

The bond lengths and bond angles have been deposited.* The Ni—O and Ni—N distances of 1·829 (5) and 1·938 (6) Å, respectively, agree well with the corresponding averaged distances of 1·837 and 1·920 Å in the related planar chelate bis(*N*-isopropyl-3-methylsalicylaldiminato)nickel. These bond distances in planar complexes are shorter than the corresponding distances of 1·894 and 1·990 Å in the related tetrahedral complex bis(*N*-isopropylsalicylaldiminato)nickel.* The shortening of the nickel coordination bond distances upon changing from tetrahedral to planar coordination may be partially due to an increased amount of d_π–p_π interaction in the Ni—O and Ni—N bonds of the planar complex. The bond lengths and angles of the salicylaldimine residue agree with the averaged values for salicylaldiminate complexes determined by X-ray structure analysis. The salicylaldimine group is essentially planar with the Ni atom 0·54 Å out of the plane, as in related complexes.

* See deposition footnote.

It has been suggested that the tendency towards formation of tetrahedrally coordinated species is due to the steric effects when *sec* alkyl groups are substituted on the N atoms of salicylaldimine (Sacconi, Paoletti & Ciampolini, 1963). However, substituents in the 3 and 5 positions of salicylaldimine inexplicably restore the planar coordination of the Ni^{II} ion.

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Structure of Tetramesityldistibane

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Abstract. Tetra(2,4,6-trimethylphenyl)distibane, $C_{36}H_{44}Sb_2$, $M_r = 720{\cdot}3$, monoclinic, $P2_1/n$, $a = 12{\cdot}057$ (2), $b = 15{\cdot}992$ (2), $c = 17{\cdot}700$ (2) Å, $\beta = 99{\cdot}80$ (1)°, $V = 3362{\cdot}9$ Å^3, $Z = 4$, $D_x = 1{\cdot}42$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 16{\cdot}4$ cm^{-1}, $F(000) = 1448$, $T = 296$ (1) K, $R = 0{\cdot}040$ for 3396 unique observed reflections. The crystal structure comprises discrete molecules which adopt an *anti* conformation of the non-bonding electron pairs. Molecular packing considerations preclude any extended interaction between Sb atoms of neighbouring molecules.

Introduction. In the course of our investigation into the reaction between mes_2SbLi (mes = mesityl = 2,4,6-trimethylphenyl) with group 11 and 12 chlorides we repeatedly obtained tetramesityldistibane together with reduction of the metal. In the light of previous structural studies of distibanes and their exhibition of thermochromic effects (Ashe, Butler & Diephouse, 1981), a study of the solid state structure and packing for this complex was carried out.

Experimental. Tetramesityldistibane was prepared by the oxidation of dimesitylstibido anion in the presence of CuCl. Yellow crystals were grown from hexane. A prism with approximate dimensions 0·25 × 0·30 × 0·30 mm was mounted under argon in a Lindemann capillary, and placed on an Enraf–Nonius CAD-4 diffractometer, using graphite-monochromated Mo $K\alpha$ X-radiation. Cell parameters were refined for 25 reflections within the range $26 \le 2\theta \le 30°$, measured by the diagonal slit method of centring. Data were collected at 296 (1) K, using ω–2θ scan mode for $3 \le 2\theta \le 48°$, variable scan rate 2–10° min^{-1} in ω, with scan width = (0·8 + 0·35tanθ)°. Range of hkl from 0, 0, −20 to 13, 18, 19. Two standard reflections were remeasured every 3600 s of X-ray exposure (no significant variation), and two orientation standards after every 100 reflections collected with a recalculation of the orientation matrix after any significant angular deviation. Empirical absorption correction applied to data, transmission coefficient range 0·94–1·00 (av. 0·98). Total reflections measured 5536, 5260 unique (averaged for observed and accepted reflections; 1·5 and 1·4% based on I and F_o respectively). 3396

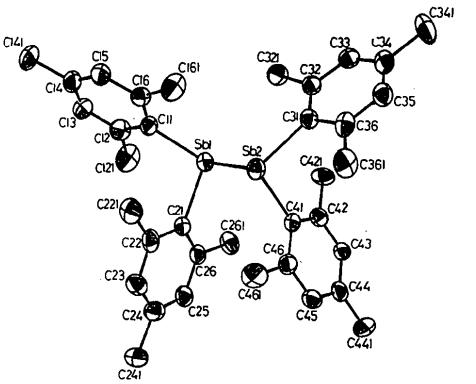

Fig. 1. The structure of tetramesityldistibane. Thermal motion is shown by 30% probability ellipsoids.

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