Fig. 1. The atom-numbering scheme of the anion with bond lengths and angles. Where no e.s.d.'s appear they are 0·004 Å for distances and 0·2° for angles.

Fig. 2. An *ORTEP* stereoview of the molecular packing.

atom-numbering scheme of the anion with bond lengths and angles is given in Fig. 1 and an *ORTEP* (Johnson, 1976) stereoview of the molecular packing in Fig. 2.

Related literature. The Ni atom lies at the origin, as in the case of $Ni(S_5C_3)_2NBu_4$ (Mentzafos, Hountas & Terzis 1988), but in the present structure Ni is in formal oxidation state +2. As a result the Ni–S bonds are slightly longer in the present structure, but comparable to those observed in bis(isotrithione-dithiolate)-nickelate (Lindqvist, Sjölin, Sieler, Steimecke & Hoyer,
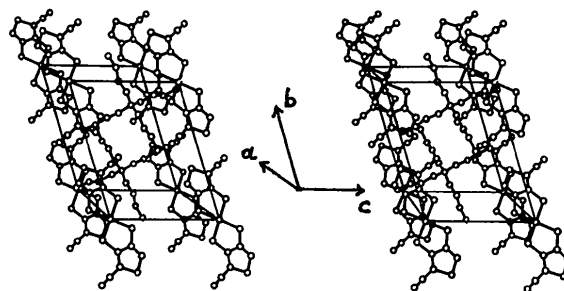
1982) where Ni is in the same formal oxidation state of +2.

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Bis(N-n-propylsalicylideneaminato)nickel(II)

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Abstract. [Ni(C₁₀H₁₂NO)₂], $M_r = 383·14$, monoclinic, $P2_1/c$, $a = 10·026$ (2), $b = 10·067$ (3), $c = 9·167$ (2) Å, $\beta = 100·26$ (3)°, $V/Z = 455·3$ (4) Å³, $Z = 2$, $D_x = 1·397$ (1) g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0·71073$ Å, $\mu = 10·8$ cm⁻¹, $F(000) = 404$, $T = 297$ (2) K, $R = 0·030$ for 1048 reflections. The geometry around the Ni atom is square planar, and, except for the propyl groups, the entire molecule is approximately planar; the plane of the benzene ring in the ligand is tilted 5·4° with respect to the NiN_2O_2 plane. Bond lengths and angles are normal.

Experimental. The title compound was prepared by the method of Sacconi, Paoletti & Del Re (1957). It was

characterized by its melting point (436–437 K), absorption spectra and magnetic properties (Holm, 1961). Plate-shaped crystals suitable for X-ray diffraction were present in the sample. The crystal used measured 0·08 × 0·25 × 0·30 mm. Data were collected on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. 25 reflections with $10 < \theta < 15°$ were used to determine the cell parameters. Systematic extinctions ($0k0$, k odd; $h0l$, l odd) uniquely determined the space group. Data were collected, using ω–2θ scans, in the range $0 < \theta < 24°$ for one quadrant (ranges: $h\,0$ to 11, $k\,0$ to 11, $l\,-10$ to 10). Empirical absorption corrections were applied; correction factors ranged from 0·908 to 1·000. 1431 unique reflections

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{4}{3}\Sigma_i \Sigma_j B_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Ni	0·000	0·000	0·000	2·607 (8)
O	0·1365 (2)	−0·0319 (2)	−0·1033 (2)	3·91 (5)
C1	0·2477 (3)	0·0359 (3)	−0·1097 (3)	2·98 (6)
C2	0·2760 (3)	0·1590 (3)	−0·0418 (3)	2·93 (6)
C3	0·3960 (3)	0·2263 (3)	−0·0563 (4)	4·07 (7)
C4	0·4859 (3)	0·1717 (4)	−0·1350 (3)	4·39 (7)
C5	0·4584 (3)	0·0491 (4)	−0·2013 (3)	4·06 (7)
C6	0·3414 (3)	−0·0183 (3)	−0·1905 (3)	3·74 (7)
C7	0·1831 (3)	0·2211 (3)	0·0389 (3)	3·02 (6)
N	0·0687 (2)	0·1728 (2)	0·0623 (2)	2·68 (5)
C8	−0·0097 (3)	0·2656 (3)	0·1405 (3)	3·11 (6)
C9	−0·1241 (3)	0·3303 (3)	0·0359 (3)	3·59 (7)
C10	−0·2128 (4)	0·4144 (4)	0·1167 (4)	5·34 (9)

Table 2. *Interatomic distances (Å) and angles (°)*

Ni–O	1·826 (2)	O–Ni–N	92·9 (1)
Ni–N	1·920 (2)	Ni–O–C1	130·6 (2)
O–C1	1·318 (3)	O–C1–C2	123·2 (2)
C1–C2	1·393 (4)	O–C1–C6	118·2 (3)
C2–C3	1·407 (4)	C6–C1–C2	118·6 (3)
C3–C4	1·366 (4)	C1–C2–C3	119·7 (3)
C4–C5	1·383 (5)	C2–C3–C4	120·8 (3)
C5–C6	1·373 (4)	C3–C4–C5	119·5 (3)
C6–C1	1·406 (4)	C4–C5–C6	121·0 (3)
C2–C7	1·432 (3)	C5–C6–C1	120·4 (3)
C7–N	1·298 (3)	C7–C2–C1	121·3 (2)
N–C8	1·485 (3)	C7–C2–C3	119·0 (3)
C8–C9	1·506 (4)	N–C7–C2	126·7 (3)
C9–C10	1·514 (4)	Ni–N–C7	124·8 (2)
		Ni–N–C8	121·4 (2)
		C7–N–C8	113·8 (2)
		N–C8–C9	111·9 (2)
		C8–C9–C10	112·1 (2)
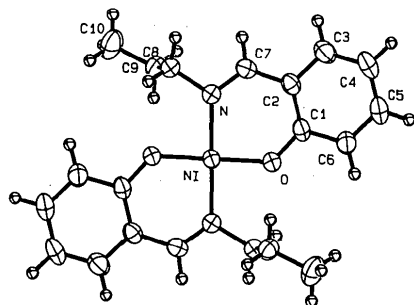
Fig. 1. View of the title compound. The plane of the page is the plane of the NiN$_2$O$_2$ group. The benzene ring is tilted 5·4° out of this plane. Thermal ellipsoids are shown at the 50% probability level.

were measured, of which the 1048 with $I > \sigma(I)$ were used in the calculations. Three check reflections measured every 4000 s of exposure time showed no systematic change with time. With $Z = 2$, the molecule is required to have a crystallographic center of symmetry so that the Ni atom could be placed at the origin. The remaining non-H atoms were located from successive electron density maps. All atoms except H were given anisotropic thermal parameters; H atoms were included at idealized positions with fixed isotropic

thermal parameters. All parameters were refined by full-matrix least squares on F's. Refinement converged with $R = 0·030$, $wR = 0·035$ and $S = 1·217$; $w = 1/\sigma^2(F)$ was calculated from $\sigma^2(I) = \sigma^2(I)_c + (0·04I)^2$, where $\sigma(I)_c$ is the standard deviation in I based on counting statistics alone. $(\Delta/\sigma)_{max}$ in the final cycle was 0·01. The largest difference from zero in the final difference electron density map was a peak of height 0·208 e Å^{-3} near the middle of a C–C bond. Atomic scattering factors and anomalous-dispersion corrections for all atoms were taken from *International Tables for X-ray Crystallography* (1974). The computer programs used were all from the *Enraf–Nonius Structure Determination Package* and have been described by Frenz (1978).

Atomic coordinates are given in Table 1, and interatomic distances and angles in Table 2.* The atom labelling and thermal ellipsoids are shown in Fig. 1.

Related literature. The structures of several related compounds, where the propyl group has been replaced by another group, R, have been reported. For $R = H$, see Stewart & Lingafelter (1959). For $R = CH_3$, see Frasson, Panattoni & Sacconi (1959), Fox & Lingafelter (1967), and Steurer & Adlhart (1983). For $R = C_2H_5$, see Shkol'nikova, Knyazeva & Voblikova (1967). For $R = C_3H_7$ (isopropyl), see Fox, Orioli, Lingafelter & Sacconi (1964). For $R = C_3H_5$ (allyl), see Bhatia, Bindlish, Saini & Jain (1981). For $R = C_6H_{11}$ (cyclohexyl), see Bhatia, Syal, Kashyap, Jain & Brown (1983). All of these structures show planar geometry around Ni except the isopropyl derivative, which has a tetrahedral geometry.

* Lists of anisotropic thermal parameters, H-atom parameters, deviations from planarity, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51589 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Studies on Antifungal Agents. 29. Structure of N-[1-(4-Chlorophenyl)-2-(1H-1,2,4-triazol-1-yl)ethylidene]methylamine N-Oxide

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Abstract. $C_{11}H_{11}ClN_4O$, $M_r = 250.69$, monoclinic, $P2_1/c$, $a = 8.999$ (2), $b = 15.171$ (1), $c = 8.933$ (1) Å, $\beta = 93.78$ (1)°, $V = 1216.8$ Å^3, $Z = 4$, $D_x = 1.368$ g cm^{-3}, $\lambda(Cu K\alpha) = 1.54184$ Å, $\mu = 27.3$ cm^{-1}, $F(000) = 520$, $T = 297$ K, final $R = 0.044$ for 1768 unique observed reflections. The title compound is an important intermediate in the synthesis of 3-aryl-5-(aryloxyalkyl)-3-(1H-imidazol-1-ylmethyl)-2-methyl-isoxazolidines; the results of the X-ray structure determination confirm the E configuration of this intermediate.

Experimental. Crystal $0.12 \times 0.25 \times 0.30$ mm, Enraf–Nonius CAD-4 diffractometer with Ni-filtered Cu $K\alpha$ radiation. Lattice parameters from 22 reflections with $48 \leq 2\theta \leq 65°$. 2310 reflections measured using the ω–2θ scan technique within ranges $6 \leq 2\theta \leq 130°$, $-10 \leq h \leq 0$, $-17 \leq k \leq 0$, $-10 \leq l \leq 10$. Intensities of three standard reflections ($\bar{1}\bar{1}2$, $2\bar{1}\bar{1}$, $\bar{1}02$) recorded every 3500 s of X-ray exposure showed no significant decay. 1768 unique observed reflections [$I > 3\sigma(I)$, $R_{int} = 0.017$]. Data corrected for Lorentz and polarization effects but not for absorption. Structure solved by *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H atoms found from subsequent difference Fourier syntheses. Refinement by full-matrix least squares to minimize $\sum w(|F_o| - |F_c|)^2$ led to $R = 0.044$ and $wR = 0.062$ for 154 variables with $w = 1/\sigma^2(F_o)$ and $S = 2.12$. Non-H atoms refined anisotropically; H atoms not refined but included as constant contributions to structure factors. Maximum least-squares shift to e.s.d. ratio 0.02 in the final refinement cycle. Largest residual electron densities in the final difference

Fourier synthesis 0.23 and -0.45 e Å^{-3}. Atomic scattering factors from Cromer & Waber (1974); *SDP* package (Frenz, 1978).† Positional parameters and equivalent isotropic thermal parameters are given in Table 1.

Related literature. Previously (Mullen, Swift, Maryniak, Allen, Mitchell, Kinsolving & Georgiev, 1988), we described the synthesis of a novel series of systemic antifungal agents, the 3-aryl-5-(aryloxyalkyl)-3-(1H-imidazol-1-ylmethyl)-2-methylisoxazolidines (3) *via* a 1,3-dipolar cycloaddition reaction of α-substituted

† Lists of anisotropic thermal parameters, H-atom positional parameters, bond distances and angles, and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51591 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Refined positional parameters*

$B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab\cos\gamma + \beta_{13}ac\cos\beta + \beta_{23}bc\cos\alpha)$.

	x	y	z	B_{eq} (Å^2)
Cl	0.44963 (6)	0.10235 (4)	0.17459 (6)	5.79 (1)
C(1)	0.3297 (2)	0.1184 (1)	0.0155 (2)	4.08 (4)
C(2)	0.2468 (3)	0.0486 (1)	−0.0434 (3)	4.89 (5)
C(3)	0.1582 (3)	0.0606 (1)	−0.1740 (3)	4.57 (4)
C(4)	0.1520 (2)	0.1420 (1)	−0.2452 (2)	3.40 (3)
C(5)	0.2311 (2)	0.2126 (1)	−0.1797 (2)	3.60 (4)
C(6)	0.3217 (2)	0.2006 (1)	−0.0491 (2)	3.89 (4)
C(7)	0.0611 (2)	0.1572 (1)	−0.3866 (2)	3.40 (3)
C(8)	−0.0571 (2)	0.2276 (1)	−0.3978 (2)	3.83 (4)
C(9)	−0.3142 (2)	0.1755 (2)	−0.4879 (3)	4.86 (5)
C(10)	−0.3817 (2)	0.1307 (2)	−0.2837 (3)	5.26 (5)
C(11)	0.2019 (2)	0.0466 (1)	−0.5190 (3)	4.48 (4)
N(1)	0.0794 (2)	0.11086 (9)	−0.5075 (2)	3.41 (3)
N(2)	−0.2052 (2)	0.1903 (1)	−0.3829 (2)	3.71 (4)
N(3)	−0.4291 (2)	0.1383 (1)	−0.4299 (3)	5.69 (5)
N(4)	−0.2459 (2)	0.1608 (1)	−0.2483 (2)	4.98 (4)
O(1)	−0.0073 (2)	0.11985 (9)	−0.6288 (2)	4.26 (3)

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