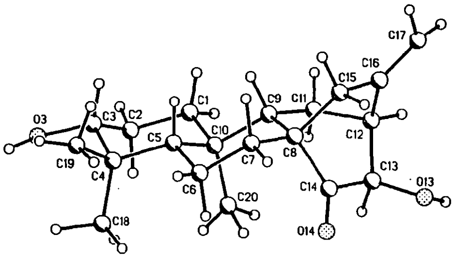

Fig. 1. Perspective view of the neriifolene molecule with atomic numbering.

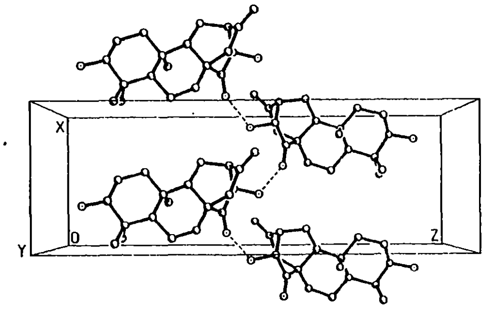

Fig. 2. Crystal packing of neriifolene viewed along the *b* axis.

$\sigma^2(F) + 0.0009F^2$ was used. The maximum $\Delta/\sigma = 0.167$ and goodness of fit = 1.33. The largest difference peak was 0.17 e Å^{-3} and deepest difference hole was −0.18 e Å^{-3}. All calculations were performed with *SHELXTL-Plus* (Siemens Analytical X-ray Instruments Inc., 1990) on a MicroVAX 2000 computer. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vo. IV). Figures were also prepared by *SHELXTL-Plus* graphics programs. The structure of neriifolene is shown in Fig. 1, which also shows the atomic numbering. Atomic coordinates of non-H atoms are given in Table 1.* Bond lengths and bond angles are given in Table 2. The packing of the crystal as viewed along the *b* axis is shown in Fig. 2. Neighbouring molecules are connected through hydrogen bonds between O(13) and O(14)′ with O(13)···O(14)′ distance of 2.806 Å.

Related literature. For isolation of neriifolene and investigation of *Euphorbia neriifolia* see Ng (1990). Isolation of compounds of the same molecular formula from other species of *Euphorbia* plants has also been reported (Cambie, Clark, Lal, Rickard, Rutledge & Woodgate, 1990; Lal, Cambie, Rutledge & Woodgate, 1990; Jia, Ding, Wang & Liu, 1990).

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* 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 54689 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cambie, R. C., Clark, G. R., Lal, A. R., Rickard, C. E. F., Rutledge, P. S. & Woodgate, P. D. (1990). *Acta Cryst.* **C46**, 2387–2389.

Jia, Z., Ding, Y, Wang, Q. & Liu, T. (1990). *Phytochemistry*, **29**, 7, 2343–2345.

Lal, A. R., Cambie, R. C., Rutledge P. S. & Woodgate, P. D. (1990). *Phytochemistry*, **29**, 1925–1935.

Ng, A. S. (1990). *Phytochemistry*, **29**, 662–664.

Siemens Analytical X-ray Instruments Inc. (1990). *SHELXTL-Plus*. Release 4.0. Madison, Wisconsin, USA.

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Structure of *N*-[(4-Cyanophenyl)(phenyl)methylene]aniline

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Abstract. 4-[α-(Phenylimino)benzyl]benzonitrile, $C_{20}H_{14}N_2$, $M_r = 282.34$, monoclinic, $C2$, $a = 15.796$ (4), $b = 7.332$ (2), $c = 14.498$ (4) Å, $\beta = 113.30$ (2)°, $V = 1542.2$ (8) Å^3, $Z = 4$, $D_x = 1.216$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.07$ mm^{-1}, $F(000) = 592$, $T = 291$ (1) K, final $R = 0.054$ for 1567 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. There are no short contacts between the molecules. All bond lengths, bond angles and dihedral angles are normal. Because of the con-

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jugation between the —C≡N group and the C=N group they are situated in one plane. The molecule almost has mirror symmetry with the two phenyl groups nearly perpendicular to the mirror plane and the remaining parts of the molecule in the mirror plane.

Experimental. The title compound was prepared by reaction of 3.2 g of *p*-CN-benzophenone and 3 ml of aniline in 120 ml boiling toluene with 20 mg of *p*-toluenesulfonic acid as a catalyst (yield 91%). In order to keep the reaction water free a watertrap was used. Yellow crystals (m.p. 413–414 K) were obtained by multiple recrystallization from anhydrous ethanol. The vibrational spectrum shows a C=N band at 1629 cm^{-1} and a —C≡N band at 2228 cm^{-1}. A crystal platelet of approximate size 0.06 × 0.32 × 0.51 mm was used. The crystal was mounted on a glass fibre. D_m was not determined. Intensity data were collected with $\omega/2\theta$ scans, variable scan speed 1.5–15.0° min^{-1} in θ, scan width 1.2° + dispersion. A Nicolet *R3m*/*V* diffractometer with graphite-monochromated Mo $K\alpha$ radiation was used for preliminary examinations and data collection. The lattice parameters were determined from a symmetry-constrained least-squares fit of 29 reflections with $2\theta_{\max} = 26.9°$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (200, 020, 002, $\bar{2}00$, $0\bar{2}0$, $00\bar{2}$) were recorded every 300 reflections, only random deviations were detected during 87.02 h of X-ray exposure; 4399 reflections with $2.0 \le 2\theta \le 50.0°$, $-19 \le h \le 19$, $-9 \le k \le 3$,

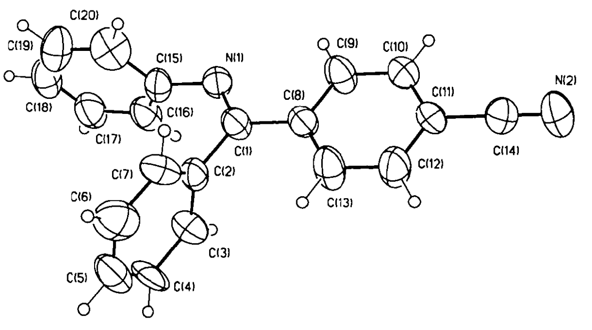

Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of the molecule showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

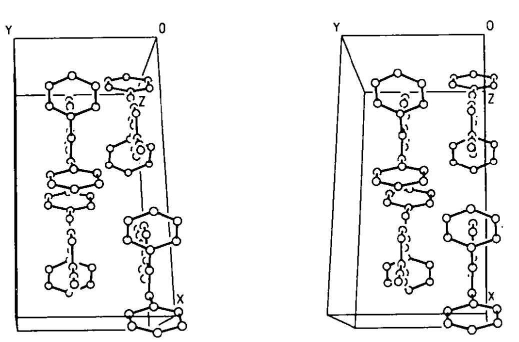

Fig. 2. Stereoscopic view (*SHELXTL-Plus*; Sheldrick, 1987) of the unit cell. The molecule almost has mirror symmetry with the mirror plane perpendicular to *b*.

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

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ The *y* coordinate of atom N(1) was not refined in order to fix the molecule in the cell.

	x	y	z	U_{eq}
N(1)	0.0899 (1)	0.0974	0.6605 (1)	607
N(2)	0.3673 (2)	0.0984 (5)	0.3412 (2)	845
C(1)	0.1767 (1)	0.0918 (5)	0.6911 (1)	498
C(2)	0.2398 (1)	0.0983 (5)	0.8007 (1)	481
C(3)	0.2652 (3)	−0.0712 (4)	0.8510 (3)	739
C(4)	0.3235 (3)	−0.0657 (4)	0.9527 (2)	711
C(5)	0.3523 (2)	0.0962 (5)	1.0029 (2)	707
C(6)	0.3265 (3)	0.2564 (5)	0.9539 (3)	802
C(7)	0.2703 (2)	0.2535 (4)	0.8506 (3)	583
C(8)	0.2181 (1)	0.0925 (5)	0.6150 (1)	488
C(9)	0.1620 (2)	0.0990 (5)	0.5131 (2)	605
C(10)	0.2010 (2)	0.0991 (5)	0.4429 (2)	622
C(11)	0.2951 (2)	0.0958 (5)	0.4730 (2)	539
C(12)	0.3512 (2)	0.0920 (5)	0.5734 (2)	738
C(13)	0.3123 (2)	0.0917 (5)	0.6439 (2)	717
C(14)	0.3354 (2)	0.0967 (5)	0.3991 (2)	634
C(15)	0.0472 (1)	0.0844 (5)	0.7308 (2)	561
C(16)	0.0265 (3)	−0.0719 (5)	0.7624 (3)	719
C(17)	−0.0233 (3)	−0.0755 (5)	0.8267 (3)	776
C(18)	−0.0458 (2)	0.0924 (5)	0.8573 (2)	723
C(19)	−0.0224 (3)	0.2483 (4)	0.8256 (3)	724
C(20)	0.0208 (3)	0.2542 (4)	0.7631 (3)	718

Table 2. *Bond distances* (Å), *bond angles* (°), *torsion angles* (°), *least-squares planes and dihedral angles* (°)

N(1)—C(1)	1.264 (3)	C(8)—C(13)	1.377 (3)
N(1)—C(15)	1.431 (3)	C(9)—C(10)	1.383 (4)
N(2)—C(14)	1.136 (4)	C(10)—C(11)	1.374 (4)
C(1)—C(2)	1.505 (2)	C(11)—C(12)	1.373 (3)
C(1)—C(8)	1.489 (4)	C(11)—C(14)	1.447 (4)
C(2)—C(3)	1.416 (5)	C(12)—C(13)	1.386 (4)
C(2)—C(7)	1.332 (4)	C(15)—C(16)	1.322 (5)
C(3)—C(4)	1.397 (5)	C(15)—C(20)	1.449 (5)
C(4)—C(5)	1.372 (5)	C(16)—C(17)	1.437 (7)
C(5)—C(6)	1.349 (5)	C(17)—C(18)	1.402 (5)
C(6)—C(7)	1.407 (5)	C(18)—C(19)	1.338 (5)
C(8)—C(9)	1.391 (3)	C(19)—C(20)	1.333 (7)

C(1)—N(1)—C(15)	119.9 (2)	C(9)—C(10)—C(11)	120.5 (2)
N(1)—C(1)—C(8)	118.3 (2)	C(10)—C(11)—C(14)	120.2 (2)
N(1)—C(1)—C(2)	122.9 (2)	C(10)—C(11)—C(12)	120.1 (3)
C(2)—C(1)—C(8)	118.7 (2)	C(12)—C(11)—C(14)	119.8 (2)
C(1)—C(2)—C(7)	123.1 (3)	C(11)—C(12)—C(13)	119.6 (3)
C(1)—C(2)—C(3)	116.8 (2)	C(8)—C(13)—C(12)	121.1 (2)
C(3)—C(2)—C(7)	120.1 (3)	N(2)—C(14)—C(11)	179.6 (4)
C(2)—C(3)—C(4)	116.9 (3)	N(1)—C(15)—C(20)	116.7 (2)
C(3)—C(4)—C(5)	121.8 (3)	N(1)—C(15)—C(16)	123.8 (2)
C(4)—C(5)—C(6)	120.4 (3)	C(16)—C(15)—C(20)	119.4 (3)
C(5)—C(6)—C(7)	118.7 (3)	C(15)—C(16)—C(17)	120.9 (3)
C(2)—C(7)—C(6)	122.1 (3)	C(16)—C(17)—C(18)	117.5 (3)
C(1)—C(8)—C(13)	120.9 (2)	C(17)—C(18)—C(19)	120.1 (3)
C(1)—C(8)—C(9)	120.3 (2)	C(18)—C(19)—C(20)	123.2 (3)
C(9)—C(8)—C(13)	118.8 (2)	C(15)—C(20)—C(19)	118.8 (3)
C(8)—C(9)—C(10)	119.9 (2)		

No.	Plane through atoms	Equation of the plane	χ^2
1	C(2),C(3),C(4),C(5),C(6),C(7)	$0.9743x - 0.025y - 0.224z = -3.19$ Å	26.6
2	C(8),C(9),C(10),C(11),C(12),C(13)	$-0.011x - 0.99973y - 0.021z = -0.86$ Å	14.6
3	C(15),C(16),C(17),C(18),C(19),C(20)	$-0.6091x - 0.011y - 0.7930z = -5.63$ Å	35.8

	Dihedral angle
1,2	88.9 (1)
1,3	114.56 (8)
2,3	88.1 (1)

$-18 \leq l \leq 18$ were measured. The data were corrected for Lorentz–polarization but not for absorption effects because of the small absorption coefficient, and averaged ($R_{\text{int}} = 0.105$) to 2090 unique reflections, 1567 of which had $F \geq 4.0\sigma(F)$. The systematic absences (hkl) $h + k = 2n + 1$ conform to the space groups $C2$, Cm and $C2/m$. The structure was solved *via* direct methods and $\Delta\rho$ maps in space group $C2$. It was refined (on F) using full-matrix least squares with anisotropic displacement parameters for all non-H atoms and a common isotropic displacement parameter for the H atoms, which were placed in geometrically calculated positions (C—H 0.96 Å). 200 parameters were refined. Weights $w = 1.0/[\sigma^2(F) + (0.0040F^2)]$ led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. The refinement (damping factor damp = 1000 on the L.S. card of *SHELX76*; Sheldrick, 1976) converged to $S = 0.97$, $R = 0.054$, $wR = 0.073$, $(\Delta/\sigma)_{\max} = 0.23$ (no extinction correction). The largest peaks in the final $\Delta\rho$ map were $\pm 0.2\,(1)\ \text{e}\,\text{Å}^{-3}$. The correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987) and the extra symmetries m (at $y = 0.097$) and $\bar{1}$ were indicated. A refinement in space group $C2/m$ led to unreasonable results ($R = 0.165$, $wR = 0.258$, $S = 3.1$, unreasonable atomic displacement parameters) and this space group was discarded. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The programs used were *PARST* (Nardelli, 1983), *SHELX76* (Sheldrick, 1976), *SHELXTL-Plus* (Sheldrick, 1987), *PCK83* (Williams, 1984), *PLATON* (Spek, 1982). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit-cell contents is in Fig. 2. Positional parameters and the equivalent values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles, least-squares planes and dihedral angles are given in Table 2.

Related literature. Appropriately substituted aniles of p-CN-benzophenone have potential use as complexation agents for the spectrophotometric determination of some metals. The general method for synthesis of aniles has been reported (Layer, 1963).

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* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54687 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL0501]

References

Layer, R. W. (1963). *Chem. Rev.* **63**, 489–510.

Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

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

Sheldrick, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet *R3mV* crystallographic systems. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Spek, A. L. (1982). *The EUCLID Package*. In *Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.

Williams, D. E. (1984). *PCK83*. A crystal and molecular packing analysis program. Univ. of Louisville; Quantum Chemistry Program Exchange (QCPE), program No. 481. Indiana Univ., Indiana, USA.

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Structure of a Novel Twist-Boat Spiroketal

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Abstract. 10-(*tert*-Butyldiphenylsilyloxy)-2-methoxy-2,3,8-trimethyl-1,7-dioxaspiro[5.5]undecan-5-one, $C_{29}H_{40}O_5Si$, $M_r = 496{\cdot}72$, monoclinic, $C2/c$, $a = 38{\cdot}29\,(5)$, $b = 7{\cdot}774\,(2)$, $c = 20{\cdot}65\,(2)$ Å, $\beta = 109{\cdot}05\,(8)^\circ$, $V = 5810\,(8)$ Å^3, $Z = 8$, $D_x = 1{\cdot}136$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}09$ cm^{-1}, $F(000) = 2144$, $T = 298$ K, $R = 0{\cdot}075$ for 1469 observed reflections. The structure consists of two substituted and spiro-fused tetrahydropyran rings. One tetrahydropyran ring is in a chair conformation with intra-ring torsion angles alternating ± in the range 55–58°; both its methyl and *tert*-

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