3697 reflections	$\Delta\rho_{max}$ = 0.204 e Å^{-3}
231 parameters	$\Delta\rho_{min}$ = −0.183 e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Extinction correction: none
	Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1002). Services for accessing these data are described at the back of the journal.

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N-(*p*-Chlorobenzoyl)-*N*-methylaniline

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Abstract

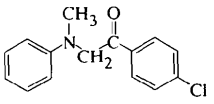
The crystal and molecular structure of the title compound [alternative name: *p*-chlorophenyl (*N*-methylanilino)methyl ketone], $C_{15}H_{14}ClNO$, has been determined by X-ray crystallography. There are two independent molecules in the asymmetric unit. The packing shows that it is unlikely for there to be intermolecular charge transfer in the crystal.

Comment

As some indoles are of pharmacological interest as potential neuro-active drugs, a method of synthesizing 3- and 2-functionalized derivatives was required (Smith & Visnick, 1985; Walkup & Linder, 1985). An alternative methodology uses phenacylanilines as starting products (Brown & Mann, 1948): we used *N*-methyl-*p*-chlorophenacylaniline which transforms to 1-methyl-3- or 2-*p*-chlorophenylindole or a mixture of both under different acid catalysts (Rodríguez & Martín-Villamil, 1997, unpublished results). The molecular structure of the phenacyl precursor was determined to investigate the catalytic reaction behaviour and to look into possible charge–transfer complexation (Abdulla *et al.*, 1985).

The structure of the title compound, (I), consists essentially of planar and almost perpendicular *N*-methylaniline and *p*-chlorobenzoyl fragments, linked through a methylene bridge between the aniline-N and the carbonyl-C atoms. There are two independent molecules in the asymmetric unit (Fig. 1). The two molecules differ slightly in their torsion and dihedral angles. The dihedral angles between the C2–C7 and C12–C17 rings are 76.05 (1) for molecule *A* and 86.43 (1)° for molecule *B*. The torsion angle C5—C8—C10—N11 is −179.0 (2) in *A* and −162.4 (2)° in *B*. The chloro substituent is essentially coplanar with the ring to which it is attached in both molecules. Significant intermolecular interactions occur between the phenyl rings; the crystal packing involves T-shaped contacts between the aniline

ring of molecule *A* and the *p*-chlorobenzoyl ring of *B* at $(\frac{1}{2}-x, y-\frac{1}{2}, z-\frac{1}{2})$, and the aniline ring of molecule *B* and the *p*-chlorobenzoyl rings of *A* at $(-x, -y, 1-z)$ and $(1-x, -y, 1-z)$. These contacts define two different herring-bone patterns along the *a* axis, linking the two independent molecules in the asymmetric unit and forming layers in the *ac* plane. Isolated stacking interactions between the *p*-chlorobenzoyl rings link the layers along the *b* axis (Fig. 2) (Albert & Cano, 1991). The herring-bone packing is expected for an aromatic compound with a low C:H ratio (Desiraju, 1989).



(I)

The findings of the X-ray structure determination eliminate the possibility of enolic forms contributing to the yellow colour. They also rule out an intramolecular interaction between the electron-accepting *p*-chlorobenzoyl group and the electron-donating *N*-methylaniline part of the molecule, thus suggesting that it is unlikely for there to be intermolecular charge transfer in the crystal.

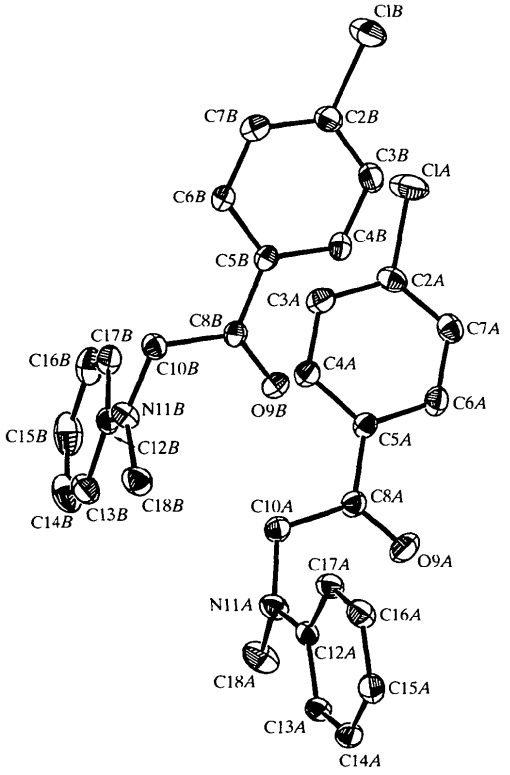


Fig. 1. *ORTEP* (Johnson, 1965) perspective view of the two independent molecules with atomic numbering. Displacement ellipsoids are drawn at the 30% probability level and H atoms are omitted for clarity.

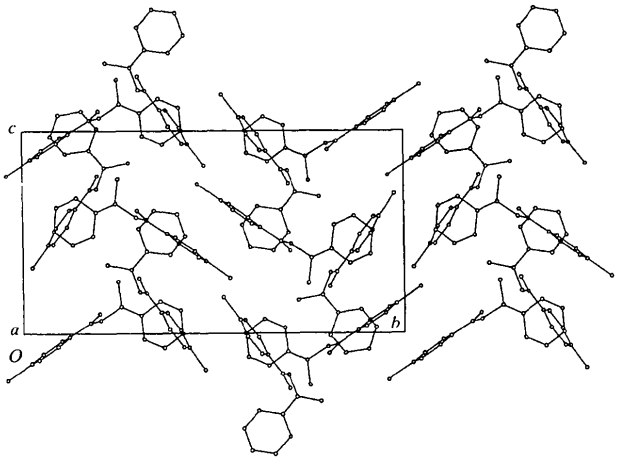


Fig. 2. Packing of the molecules in the crystal viewed down the *a* axis.

Experimental

The title compound was obtained by the reaction of *p*-chlorophenyl bromomethyl ketone and *N*-methylaniline in ethanol. The mixture was warmed under reflux for 1 h and, after cooling, a yellow solid precipitated which was filtered off, washed with water and ethanol, and recrystallized from ethanol; m.p. 382 K. Crystals for X-ray diffraction were prepared by slow evaporation of a dilute ethyl acetate solution.

Crystal data

$C_{15}H_{14}ClNO$
$M_r = 259.72$
Monoclinic
$P2_1/n$
$a = 10.2360\,(10)$ Å
$b = 22.433\,(3)$ Å
$c = 12.188\,(2)$ Å
$\beta = 104.890\,(10)°$
$V = 2704.7\,(6)$ Å^3
$Z = 8$
$D_x = 1.276$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54180$ Å
Cell parameters from 66 reflections
$\theta = 4$–$32°$
$\mu = 2.388$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.56 × 0.30 × 0.20 mm
Yellow

Data collection

Seifert XRD 3000*S* diffractometer
ω–2θ scans
Absorption correction: none
4385 measured reflections
4385 independent reflections
2807 reflections with $I > 2\sigma(I)$

$\theta_{max} = 64.89°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 26$
$l = -14 \rightarrow 13$
2 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.142$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.349$ e Å^{-3}
$\Delta\rho_{min} = -0.336$ e Å^{-3}

$S = 0.932$
4385 reflections
438 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0917P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.0069 (5)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Cl1*A*—C2*A*	1.735 (3)	Cl1*B*—C2*B*	1.738 (3)
C8*A*—O9*A*	1.214 (3)	C8*B*—O9*B*	1.223 (3)
C8*A*—C10*A*	1.521 (4)	C8*B*—C10*B*	1.510 (4)
C10*A*—N11*A*	1.446 (3)	C10*B*—N11*B*	1.441 (4)
N11*A*—C12*A*	1.377 (3)	N11*B*—C12*B*	1.377 (3)
N11*A*—C1*A*	1.455 (4)	N11*B*—C18*B*	1.448 (4)
C7*A*—C2*A*—Cl1*A*	119.1 (2)	C7*B*—C2*B*—Cl1*B*	119.5 (3)
C3*A*—C2*A*—Cl1*A*	119.2 (2)	C3*B*—C2*B*—Cl1*B*	119.5 (2)
C4*A*—C5*A*—C8*A*	122.6 (2)	C4*B*—C5*B*—C8*B*	119.2 (2)
C6*A*—C5*A*—C8*A*	118.7 (2)	C6*B*—C5*B*—C8*B*	122.1 (2)
O9*A*—C8*A*—C5*A*	120.6 (2)	O9*B*—C8*B*—C5*B*	119.9 (2)
O9*A*—C8*A*—C10*A*	120.0 (3)	O9*B*—C8*B*—C10*B*	120.6 (2)
C5*A*—C8*A*—C10*A*	119.4 (2)	C5*B*—C8*B*—C10*B*	119.6 (2)
N11*A*—C10*A*—C8*A*	114.1 (2)	N11*B*—C10*B*—C8*B*	113.7 (2)
C12*A*—N11*A*—C10*A*	120.0 (2)	C12*B*—N11*B*—C10*B*	119.6 (2)
C12*A*—N11*A*—C18*A*	119.0 (3)	C12*B*—N11*B*—C18*B*	120.2 (3)
C10*A*—N11*A*—C18*A*	116.8 (3)	C10*B*—N11*B*—C18*B*	117.9 (3)
N11*A*—C12*A*—C13*A*	121.8 (3)	N11*B*—C12*B*—C17*B*	122.1 (3)
N11*A*—C12*A*—C17*A*	121.9 (2)	N11*B*—C12*B*—C13*B*	121.0 (3)

Scattering factors, dispersion corrections and absorption coefficients were taken from *International Tables for Crystallography*, Vol. C, Tables 6.1.1.4, 4.2.6.8 and 4.2.4.2, respectively.

Data collection: Seifert diffractometer software. Cell refinement: *LSUCRE* (Appleman, 1971). Data reduction: *XRAY80* (Stewart *et al.*, 1980). Program(s) used to solve structure: *MULTAN80* (Main *et al.*, 1980). Program(s) used to refine structure: *XRAY80*; *SHELXL97* (Sheldrick, 1997). Molecular graphics: *Xtal3.0* (Hall & Stewart, 1990). Software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: KA1278). Services for accessing these data are described at the back of the journal.

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2-Salicylideneamino-4,5-bis(2-furyl)furan-3-carbonitrile

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Abstract

There are two independent molecules in the asymmetric unit of the title compound, $C_{20}H_{12}N_2O_4$. The main difference between the independent molecules is that while one of them is nearly planar, the other has the furyl substituents at 24.1 (1) and 16.1 (1)° to the central furan ring. The near planarity of the molecules introduces steric strain and distortions of the exocyclic bond angles around the central furan ring. Each molecule contains a strong intramolecular O—H···N hydrogen bond.

Comment

Although Schiff bases have been widely used as ligands in the formation of transition metal complexes and structurally characterized, a relatively small number of the free Schiff bases have been similarly characterized (Calligaris & Randaccio, 1987). Schiff base compounds show photochromism and thermochromism in the solid state by proton transfer from the hydroxyl O atom to the imine N atom (Hadjoudis *et al.*, 1987).

The title compound, (I), has two crystallographically independent molecules in the asymmetric unit. The extent of the planarity differs; in one the angles between the planar moiety *A* (O1, O2, N1, N2, C1–C9, C19, C20) and the planar moieties *B* (O3, C11–C13) and *C* (O4, C15–C18) are 2.3 (1) and 7.1 (1)°, respectively, whereas in the other molecule they are 21.4 (1) and 16.1 (1)°. The near planarity of the molecules re-