Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.103$
$S = 1.081$
1262 reflections
97 parameters
H atoms: amido and hydroxyl H atoms refined, others riding; fixed U
$w = 1/[\sigma^2(F_o^2) + (0.0470P)^2 + 0.9955P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.18$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 4. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
O1	0.97411 (11)	0.1539 (2)	0.33620 (8)	0.0489 (4)
C1	1.0873 (2)	0.2071 (2)	0.34994 (9)	0.0347 (4)
N	1.19818 (13)	0.1292 (2)	0.34957 (8)	0.0350 (3)
C2	1.31517 (15)	0.2334 (2)	0.36361 (9)	0.0314 (4)
C3	1.2574 (2)	0.3919 (2)	0.38714 (11)	0.0431 (4)
O2	1.11566 (12)	0.36531 (15)	0.37009 (8)	0.0473 (4)
C4	1.3572 (2)	0.2524 (2)	0.28779 (10)	0.0399 (4)
O3	1.25899 (14)	0.3286 (2)	0.22437 (8)	0.0522 (4)
C5	1.4297 (2)	0.1625 (2)	0.43090 (10)	0.0440 (4)
C6	1.5566 (2)	0.2643 (3)	0.45442 (14)	0.0667 (6)

Table 5. *Selected geometric parameters (Å, °) for (2)*

O1—C1	1.218 (2)	C2—C5	1.530 (2)
C1—N	1.324 (2)	C2—C3	1.538 (2)
C1—O2	1.354 (2)	C3—O2	1.440 (2)
N—C2	1.453 (2)	C4—O3	1.420 (2)
C2—C4	1.522 (2)	C5—C6	1.519 (3)
O1—C1—N	128.8 (2)	N—C2—C3	99.55 (12)
O1—C1—O2	121.40 (15)	C4—C2—C3	112.45 (14)
N—C1—O2	109.75 (14)	C5—C2—C3	113.29 (14)
C1—N—C2	114.18 (14)	O2—C3—C2	106.04 (13)
N—C2—C4	110.75 (13)	C1—O2—C3	109.46 (12)
N—C2—C5	109.80 (13)	O3—C4—C2	113.59 (14)
C4—C2—C5	110.52 (13)	C6—C5—C2	115.2 (2)

Table 6. *Hydrogen-bonding geometry (Å, °) for (2)*

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N—H1$A\cdots$O3^{i}	0.84 (2)	2.06 (2)	2.878 (2)	164 (2)
O3—H3$C\cdots$O1^{ii}	0.89 (2)	1.87 (2)	2.759 (2)	174 (2)

Symmetry codes: (i) $\frac{5}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $2 - x, y, \frac{1}{2} - z$.

For both compounds, data collection: *P*3 (Siemens, 1990*b*); cell refinement: *P*3; data reduction: *SHELXTL* (Siemens, 1994); program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1985); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *XP* in *SHELXTL*; software used to prepare material for publication: *XCIF* in *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1395). Services for accessing these data are described at the back of the journal.

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N-(3-Nitrobenzylidene)-*p*-phenylenediamine

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(*Received 24 July 1997; accepted 1 April 1998*)

Abstract

The molecule of the title compound, $C_{13}H_{11}N_3O_2$, is nearly planar. There is an extended series of π bonds through the whole molecule. The molecules pack in a columnar manner with a 'ring-double-bond overlap' mode, in which the interplanar spacings are alternately 3.459 (5) and 3.526 (5) Å.

Comment

Conjugated organic molecules containing both donor and acceptor groups are of great interest for molecular electronics devices. The title compound, (I), was designed as a medium for high-density data storage. As an aid to understanding its optical and electronic properties, the crystal structure of (I) has been determined.

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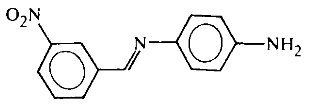

(I)

The molecule of (I) is nearly planar. The dihedral angles between its three planar fragments NO_2 ($p1$), C_6H_4–C ($p2$) and N–C_6H_4–N ($p3$) are 4.2 (4) ($p1/p2$) and 15.9 (1)° ($p2/p3$).

Examination of the bond lengths suggests that there is an extended series of π bonds through the whole molecule. Except for the N1—C15 bond connecting the nitro and phenyl groups, all the other bonds between non-H atoms show $\pi + \sigma$ character; the C—C bond lengths in the phenyl groups range from 1.368 (3) to 1.397 (3) Å. The C10—C11, C10—N2, C21—N2 and C24—N3 bond lengths are shorter than typical single σ bonds. The two phenyl groups in the molecule adopt a *trans* configuration.

There are weak N—H···N intermolecular hydrogen bonds in the crystal. The most interesting structural feature is that the molecules pack in a columnar manner (Fig. 2), similar to the ring-double-bond overlap of molecular planes reported in organic cation–TCNQ structures (Chasseau *et al.*, 1971; Fritchie, 1966; Goldberg & Shmueli, 1973; Hanson, 1965; Kistenmacher *et al.*, 1974). The zigzag C—C—N—C linkage in the molecule is sandwiched between two phenyl groups of its two nearest-neighbour molecules, with interplanar spacings of 3.459 (5) and 3.526 (5) Å (calculated as the spacings from the centres of the phenyl groups to the C11—C10—N2—C21 plane).

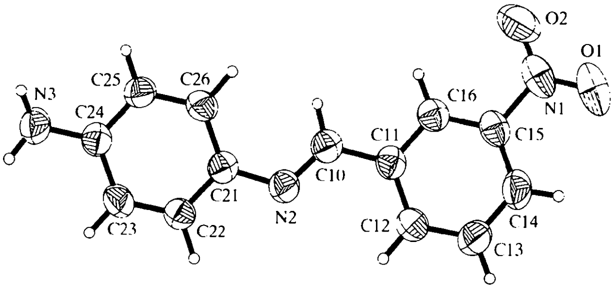

Fig. 1. The molecular structure of (I) with 50% probability ellipsoids.

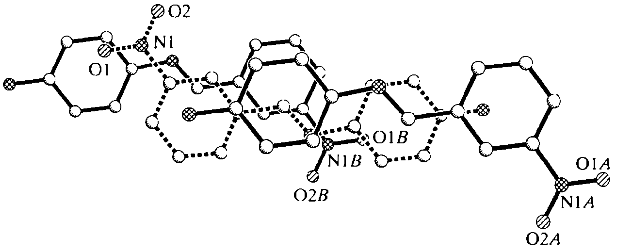

Fig. 2. The nearest-neighbour packing. The direction of view is normal to the molecular plane. [Symmetry codes: (*A*) $-x, -y, 1-z$; (*B*) $1-x, -y, 1-z$.]

Experimental

A mixture of 1,4-phenylenediamine (0.1 mmol, 0.108 g) and 3-nitrobenzaldehyde (0.1 mmol, 0.151 g) in dichloromethane (10 ml) was cooled to 273 K for 48 h. A deep-red precipitate was obtained and it was recrystallized from dichloromethane/heptane solution.

Crystal data

$C_{13}H_{11}N_3O_2$
$M_r = 241.25$
Monoclinic
$P2_1/n$
$a = 9.617 (2)$ Å
$b = 10.209 (2)$ Å
$c = 12.721 (3)$ Å
$\beta = 110.55 (3)°$
$V = 1169.5 (4)$ Å^3
$Z = 4$
$D_x = 1.370$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9.66–12.24°$
$\mu = 0.096$ mm^{-1}
$T = 293 (2)$ K
Prism
0.50 × 0.40 × 0.40 mm
Red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2148 measured reflections
2051 independent reflections
1216 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.033$
$\theta_{\rm max} = 24.96°$
$h = -11 \rightarrow 10$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 15$
3 standard reflections frequency: 60 min intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.159$
$S = 1.088$
2043 reflections
208 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.1014P)^2 + 0.3550P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.007$
$\Delta\rho_{\rm max} = 0.186$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.183$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.002 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—N1	1.216 (3)	N2—C21	1.413 (3)
O2—N1	1.218 (3)	N3—C24	1.366 (3)
N1—C15	1.475 (3)	C10—C11	1.459 (3)
N2—C10	1.269 (3)		
C10—N2—C21	121.2 (2)	N2—C10—C11	123.3 (2)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H32···N2$^{\rm i}$	0.85 (3)	2.46 (3)	3.302 (5)	167 (3)

Symmetry code: (i) $x - \frac{1}{2}, \frac{1}{2} - y, z - \frac{1}{2}$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP-Plus* (B. A. Frenz & Associates Inc., 1983). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1208). Services for accessing these data are described at the back of the journal.

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1,2,3,4,5,6,7,8,9,10-Decahydro-3,3,6,6-tetramethyl-1,8-dioxo-10-vinylacridin-9-ylmethyl Acetate

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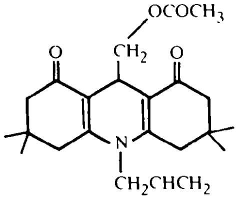
Abstract

In the title compound, $C_{23}H_{31}NO_4$, the central piperidine ring adopts a sofa conformation, while that of the outer rings is half-chair. The molecule is folded about the line passing through the central C and N atoms. The puckering amplitude of the piperidine ring is small, due to π conjugation. The methyl acetate substituent occupies an axial position. The packing is stabilized by C—H···O hydrogen bonds.

Comment

The potency of acridines as antiviral and antibacterial agents is due to their ability to bind with DNA by intercalation (Neidle, 1979). Acridine diones are laser active and fluoresce well in alcohol solvents (Selladurai *et al.*, 1990). With a view to determining the conformation of this class of compounds, the title compound, (I), was considered for crystallographic study.



(I)

The bond distances of the keto groups of the acridine moiety [C6—O6 = 1.232 (5) and C4—O4 = 1.224 (5) Å] are longer than that of the carbonyl group of the acetoxy substituent [C17—O18 = 1.189 (7) Å], and this behaviour agrees with the literature (Gunasekaran *et al.*, 1996). The angles around N10 sum to 359.4 (5)°, which is indicative of sp^2 hybridization. The acridine moiety is folded about the line passing through C5 and N10, as seen from the dihedral angle of 3.10 (8)° between the two halves (C1–C5, C4a, C10, N10 and C5–C9, C6a, C9a, N10).

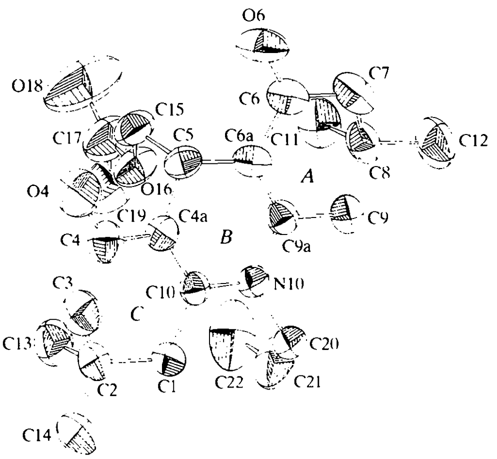


Fig. 1. *ORTEP*II (Johnson, 1976) plot, showing the molecular structure of the title compound and the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level.

The atoms of the acetoxy substituent, which occupies an axial position, lie in a plane making a dihedral angle of 80.0 (2)° with the plane through the central ring. The total puckering amplitudes (Cremer & Pople, 1975) of the rings give a quantitative evaluation of puckering [Q_T = 0.492 (5), 0.168 (4), 0.486 (5) for rings *A*, *B* and

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