Triclinic
$P\bar{1}$
$a = 6.8829\ (2)$ Å
$b = 6.9979\ (3)$ Å
$c = 7.8655\ (3)$ Å
$\alpha = 111.529\ (1)°$
$\beta = 95.615\ (2)°$
$\gamma = 106.786\ (2)°$
$V = 328.45\ (2)$ Å^3
$Z = 2$
$D_x = 1.316$ Mg m^{-3}
D_m not measured

Cell parameters from 3259 reflections
$\theta = 2.86$–$26.44°$
$\mu = 0.105$ mm^{-1}
$T = 153\ (2)$ K
Block
0.54 × 0.46 × 0.39 mm
Colourless

Data collection

Siemens SMART CCD diffractometer
Exposures over 0.3° φ or ω rotation scans
Absorption correction: none
3259 measured reflections
1258 independent reflections
1152 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.050$
$\theta_{\rm max} = 26.44°$
$h = -8 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -9 \rightarrow 9$
no standard reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.116$
$S = 1.135$
1258 reflections
92 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.117P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.001$
$\Delta\rho_{\rm max} = 0.223$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.234$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C1	1.227 (2)	C3—C6	1.491 (2)
O2—C2	1.350 (2)	C3—C4	1.499 (2)
O2—H21	0.86 (2)	C4—C5	1.531 (2)
C1—C2	1.456 (2)	O3—H31	0.88 (3)
C1—C5	1.499 (2)	O3—H32	0.86 (3)
C2—C3	1.342 (2)		
O1—C1—C2	124.30 (14)	C2—C3—C6	127.21 (15)
O1—C1—C5	127.38 (14)	C2—C3—C4	110.99 (14)
C2—C1—C5	108.32 (13)	C6—C3—C4	121.80 (13)
C3—C2—O2	132.23 (14)	C3—C4—C5	105.34 (13)
C3—C2—C1	110.82 (14)	C1—C5—C4	104.53 (13)
O2—C2—C1	116.94 (13)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H21···O3	0.86 (2)	1.78 (2)	2.618 (2)	165 (1)
O3—H31···O1$^{\rm i}$	0.88 (2)	2.02 (2)	2.888 (2)	166 (1)
O3—H32···O1$^{\rm ii}$	0.86 (2)	1.91 (2)	2.760 (2)	167 (1)

Symmetry codes: (i) $1 - x, 2 - y, 2 - z$; (ii) $x, y, z - 1$.

Crystal decay was monitored by measurement of duplicate reflections. The three oxygen-bonded H atoms were located by difference Fourier calculations and refined isotropically; other H atoms were placed geometrically and refined with a riding model (including free rotation of the methyl group).

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SAINT* (Siemens, 1995). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXL*93. Software used to prepare material for publication: *SHELXL*93.

We thank Dr D. K. Weerasinghe (Firmenich Inc., NJ, USA) for a generous supply of the title compound.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1197). Services for accessing these data are described at the back of the journal.

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5,7-Dimethyl-3-phenyl-1,2,4-benzotriazine

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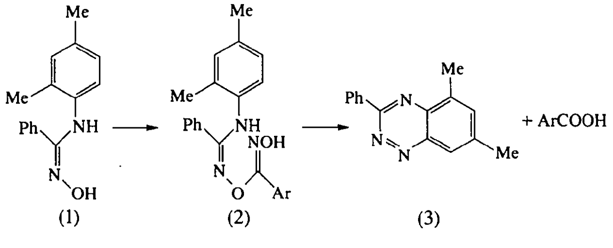
Abstract

The crystal structure of the title compound, $C_{15}H_{13}N_3$, has been determined by X-ray diffraction. The molecule is planar and the crystal packing is determined by van der Waals and graphitic interactions.

Comment

N-Substituted benzamidoximes show a complex behaviour in reactions with nitrile oxides depending on how the initially formed non-isolated *O*-benzoyloximic intermediate (2) evolves (Risitano *et al.*, 1981). Benzotriazines are the unexpected products in these reactions and their formation occurs through an unusual [3,5]-sigmatropic rearrangement (Risitano *et al.*, 1997). The title compound, (3), was prepared from arylnitrile oxide and *N*-(2,4-dimethylphenyl)benzamidoxime, (1).



The packing of (3) is mainly determined by normal van der Waals interactions and two intermolecular hydrogen bonds involving the N3 and N2 atoms [N3···H13^i 2.79 Å, N3···H13^i—C13^i 146° and N3···C13^i 3.603 (3) Å; N2···H12^i 2.98 Å, N2···H12^i—N2^i 132° and N2···C12^i 3.659 (4) Å; symmetry code: (i) $-x$, $-y$, $-z-1$]. The molecule is planar [maximum deviation from the mean molecular plane calculated for all non-H atoms is 0.041 (3) Å for C13]. The values of the dihedral angle between the plane through the two fused rings and the phenyl ring, and of the torsion angle N1—C1—C11—C12 are 178.06 (7) and 1.5 (3)°, respectively. The molecular planarity and the endocyclic bond distances (Table 1) show the presence of a large π delocalization which involves all non-H atoms.

The most important contacts are between N2 and N10 with the H atoms linked to C12, C16 and C17. The bond distances and angles for these hydrogen bonds are: N2···H12 2.49 Å, N2···H12—C12 100° and N2···C12 2.812 (3) Å; N10···H16 2.50 Å, N10···H16—C16 100° and N10···C16 2.817 (2) Å; N10···H17*F* 2.45 Å, N10···H17*F*—C17 106° and N10···C17 2.871 (2) Å.

In the heterocyclic rings, the bond distances around the N atoms agree with the corresponding values reported in the literature for similar compounds. The slight difference found is a result of the presence of two opposing factors, namely, the $-I$ effect of the N atoms which attracts electronic density towards them and the hyperconjugation effect of the two methyl groups at C6 and C8. Propagation of these effects is favoured by the planar structure of the molecule. The major influence is the $-I$ effect of the N atoms, which is also responsible for the increase of the electronic charge on these atoms (particularly on N10). The angles at the three N atoms in the triazinic ring (mean value 118.06°) are smaller than those of a benzene ring. This can be explained by the fact than an N-atom lone pair needs more space than a C—C bonding electron pair.

The crystal packing is determined by van der Waals interactions, as suggested by a number of non-bonded contacts: C1···C7 3.612 (2), C8···C11 3.581 (2), C5···C5^{ii} 3.616 (2) and C15···C18^{iii} 3.639 (4) Å [symmetry codes: (ii) $-x$, $-y$, $-z$; (iii) $1-x$, $-y$, $-z$]. The packing is also stabilized by graphitic interactions between the triazinic rings, whose planes are parallel (related by an inversion centre and disposed in a typical head-to-tail fashion) and separated by 3.54 Å, and, to a lesser extent, by the intermolecular hydrogen bonds.

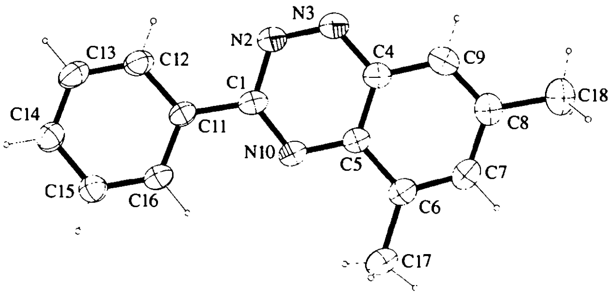


Fig. 1. A view of compound (3) showing the atomic numbering scheme and displacement ellipsoids at the 30% probability level for non-H atoms.

Experimental

Yellow crystals of (3) suitable for X-ray analysis were obtained by recrystallization from methanol.

Crystal data

$C_{15}H_{13}N_3$
$M_r = 235.28$
Triclinic
$P\bar{1}$
$a = 7.089\ (1)$ Å
$b = 9.648\ (2)$ Å
$c = 9.798\ (1)$ Å
$\alpha = 94.12\ (1)°$
$\beta = 108.04\ (1)°$
$\gamma = 101.22\ (2)°$
$V = 618.7\ (2)$ Å^3
$Z = 2$
$D_x = 1.263$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7–14°$
$\mu = 0.077$ mm^{-1}
$T = 293\ (2)$ K
Prismatic
0.4 × 0.2 × 0.2 mm
Yellow

Data collection

Siemens *R*3*m*/*V* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (Kopfmann & Huber, 1968)
$T_{min} = 0.943$, $T_{max} = 0.965$
2869 measured reflections
2199 independent reflections

1038 reflections with $I > 2\sigma(I)$
$R_{int} = 0.011$
$\theta_{max} = 25.05°$
$h = -1 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -11 \rightarrow 11$
3 standard reflections every 197 reflections intensity decay: 1.32%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.081$
$S = 0.748$
2199 reflections
166 parameters
H atoms riding with a fixed and unique $U_{\rm eq}$
$w = 1/[\sigma^2(F_o^2) + (0.042P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.106$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.117$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.013 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

C1—N10	1.322 (2)	N3—C4	1.363 (2)
C1—N2	1.374 (2)	C4—C5	1.407 (2)
N2—N3	1.313 (2)	C5—N10	1.355 (2)
N10—C1—N2	125.15 (15)	N3—C4—C9	118.5 (2)
N10—C1—C11	118.88 (14)	C5—C4—C9	120.7 (2)
N2—C1—C11	115.97 (13)	N10—C5—C4	119.85 (14)
N3—N2—C1	119.36 (13)	N10—C5—C6	120.73 (15)
N2—N3—C4	118.61 (14)	C4—C5—C6	119.4 (2)
N3—C4—C5	120.82 (15)	C1—N10—C5	116.20 (14)

Reflection intensities were evaluated by profile fitting of a 96-step peak scan among a 2θ shells procedure (Diamond, 1969) and then corrected for Lorentz–polarization effects. S.u.'s $\sigma(I)$ were estimated from counting statistics. The structure was solved by direct methods and completed by a combination of full-matrix least squares and Fourier map. All non-H atoms were refined anisotropically. H atoms were located at idealized positions and allowed to ride on their parent C atoms, with a common isotropic displacement parameter ($U_{\rm iso} = 0.07$ Å^2). The two methyl groups show disordered H atoms which have been treated in an ideal manner considering them in two positions rotated by 60° with respect to each other. All calculations were performed on a μ-VAX 3400 and on a AXP DecStation 3000/400.

Data collection: *P3/V* (Siemens, 1989). Cell refinement: *P3/V*. Data reduction: *SHELXTL-Plus* (Sheldrick, 1990*a*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*b*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *XPW* (Siemens, 1996). Software used to prepare material for publication: *PARST*95 (Nardelli, 1995) and *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1306). Services for accessing these data are described at the back of the journal.

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Ambroxol Theophylline-7-acetate Salt Monohydrate†

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Abstract

In the title monohydrate salt of ambroxol (AMB) with theophylline-7-acetic acid (TAA), $C_{13}H_{19}Br_2N_2O^+.-C_9H_9N_4O_4^-.H_2O$, protonation of the secondary amino group of AMB takes place. An N^+—H···O^- hydrogen bond is established between the protonated N atom and an O atom of the carboxylate anion, the same atom as is involved in an N—H···O interaction with the aromatic amino group of the cation. The other O atom of the carboxylate anion participates in an N^+—H···O^- hydrogen bond with a symmetry-related salt molecule, giving rise to a dimeric arrangement. The water molecule is linked to a carbonyl O atom of TAA and the aromatic amino group of AMB, and connects the salt molecules in the crystal at the level of the OH group of AMB.

Comment

Ambroxol (AMB) is reported to form a 1:1 salt with theophylline-7-acetic acid (TAA) in aprotic solvents

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† Systematic name: [(2-amino-3,5-dibromophenyl)methyl](*trans*-4-hydroxycyclohexyl)ammonium 1,2,3,6-tetrahydro-1,3-dimethyl-2,6-dioxo-7*H*-purine-7-acetate hydrate.

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