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3-Aminopyrazine-2-carboxylic Acid

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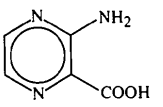
Abstract

3-Aminopyrazine-2-carboxylic acid, $C_5H_5N_3O_2$, displays an extensive network of intra- and intermolecular hydrogen bonds which are undoubtedly responsible for the modest values of the displacement parameters. H-atom transfer to the ring N atoms did not occur and the carboxy and amino H atoms are ordered. The virtually planar molecules lie very nearly in planes parallel to $(10\bar{2})$ and are stacked along the *a* direction with separations of 3.324 (2) Å indicating π–π interactions.

Comment

As pyridinium pamoate afforded numerous hydrogen-bonding possibilities (Blackburn, Dobson & Gerkin, 1996), so too does the title compound, (I). Its structure was therefore of interest and was found to exhibit extensive hydrogen bonding.



(I)

An *ORTEP*II diagram (Johnson, 1976) of (I) showing the atomic numbering scheme is presented in Fig. 1 and a stereoview of a portion of the structure, including a unit cell and with hydrogen bonds depicted, is given in Fig. 2. In the structure of (I), both intra- and intermolecular hydrogen bonds are significant (Fig. 2, Table 3). The following points may be noted: (i) each N and O atom is involved in at least one substantial hydrogen bond, (ii) the H1 and H3 atoms are involved in bifurcated hydrogen bonds, each of which comprises one intramolecular and one intermolecular bond, and (iii) each molecule participates in six intermolecular as well as in two intramolecular hydrogen bonds. The extensive hydrogen-bonding network is undoubtedly responsible for the modest size of the displacement parameters in this structure.

In contrast to the case of pyridinium pamoate (Blackburn *et al.*, 1996), H-atom transfer does not occur in the present case. The carboxy H atom is ordered, as are the amino H atoms. This behavior is, however, similar to that reported for pyrazine-2-carboxylic acid (Takusagawa, Higuchi, Shimada, Tamura & Sasada, 1974), in which the carboxy H atom is retained and is ordered, and the acceptor atom is a ring N atom.

Excluding atoms within hydrogen bonds, only the approach of the C6 and $H2^i$ atoms [symmetry code: (i) $-1+x, \frac{1}{2}-y, -\frac{1}{2}+z$] falls short of the corresponding sum of the van der Waals radii (Bondi, 1964), by as much as 0.1 Å. Although the approaches of the H5 and H6 atoms to neighboring O1 and O2 atoms are seen prominently in Fig. 2, they are of normal value.

The pyrazine core is planar, the average distance of its atoms from the best least-squares plane through them being 0.002 Å, with a mean e.s.d. of 0.002 Å. The angle between the core plane and the carboxy group plane is 1.60 (7)°, that between the core plane and the amino group plane is 3 (2)° and that between the carboxy and amino planes is 2 (2)°. The molecule as a whole is thus virtually planar. Moreover, all the molecules lie in planes parallel to $(10\bar{2})$ to within a dihedral angle of ~5°. [Strictly, there are two equally occupied orientations of the pyrazine core planes, the dihedral angle between them being 10.02 (6)°, and along any chain of hydrogen-bonded molecules within one plane, the pyrazine core planes alternate between these two orientations.] Each plane of molecules is separated by ~1.34 Å from the nearer of the two adjacent planes of

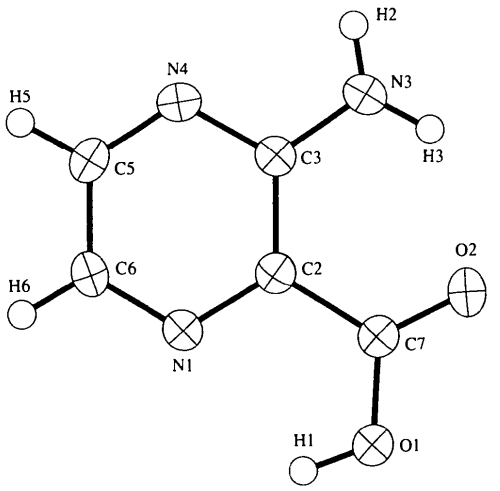


Fig. 1. An *ORTEP*II drawing (Johnson, 1976) of 3-aminopyrazine-2-carboxylic acid showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level for all atoms except H, for which they have been set artificially small.

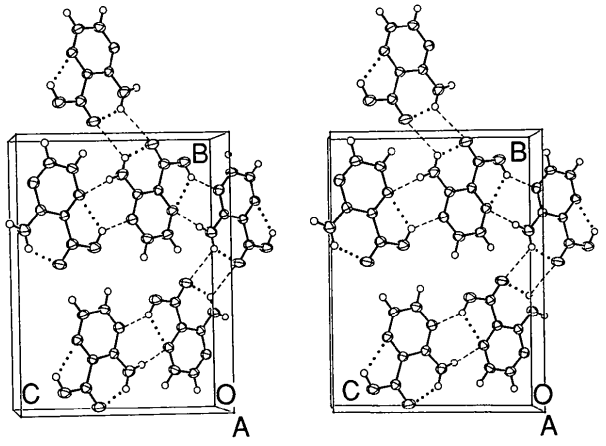


Fig. 2. A stereoview of a portion of the title structure including a unit cell (*ORTEP*II; Johnson, 1976). Displacement ellipsoids are drawn at the 50% probability level for all atoms except H, for which they have been set artificially small. Intramolecular hydrogen bonds are represented by dotted lines and intermolecular hydrogen bonds by dashed lines.

molecules and by ~1.98 Å from the farther adjacent plane. Molecules from every second plane form stacks in the *a* direction, with stack separations of 3.324 (2) Å, a distance indicative of significant π–π interactions (see, for example, Beeson, Fitzgerald, Gallucci, Gerkin, Rademacher & Czarnik, 1994, and references therein).

Intramolecular bond distances and angles are in general accord with those reported for both pyrazine at 184 K (de With, Harkema & Feil, 1976) and pyrazine-2-carboxylic acid (Takusagawa *et al.*, 1974), though some corresponding values differ by statistically significant amounts. From Takusagawa *et al.* (1974), the average distance of the pyrazine atoms from the best plane through them in pyrazine-2-carboxylic acid is 0.023 (7) Å, a value more than ten times as great as in the present pyrazine core. The manifold hydrogen bonds and π–π interactions result in a rather high density for a carboxylic acid, *i.e.* 1.609 Mg m^{-3}.

Experimental

3-Aminopyrazine-2-carboxylic acid (Aldrich Chemical Company) was dissolved in absolute ethanol. Solvent evaporation from the room-temperature solution produced clusters of plate-like crystals from one of which the experimental sample was cut. The cut crystal was mounted on a glass fiber with epoxy cement.

Crystal data

$C_5H_5N_3O_2$
M_r = 139.11
Monoclinic
$P2_1/c$
a = 3.756 (1) Å
b = 14.191 (1) Å
c = 10.911 (1) Å
β = 99.18 (1)°
V = 574.1 (1) Å^3
Z = 4
D_x = 1.609 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 11.7–16.5°
μ = 0.120 mm^{-1}
T = 296 K
Cut plate
0.35 × 0.31 × 0.16 mm
Colorless

Data collection

Rigaku AFC-5*S* diffractometer
ω scans
Absorption correction: none
1582 measured reflections
1381 independent reflections
936 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.013
θ_{max} = 27.5°
h = 0 → 4
k = 0 → 18
l = −14 → 14
6 standard reflections monitored every 150 reflections
intensity decay: 2.70%

Refinement

Refinement on F
R = 0.045
wR = 0.053
S = 1.80
936 reflections
104 parameters
H atoms: C—H riding (0.98 Å) and all parameters refined for O—H and N—H
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max}$ = <0.01
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: Zachariasen (1963, 1968)
Extinction coefficient: 2.2 (2) × 10^{-6}
Atomic scattering factors from Stewart, Davidson & Simpson (1965) for H atoms and from Cromer & Waber (1974) for C, O and N atoms

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)*

U_{iso} for H atoms; $U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for all other atoms.

	x	y	z	U_{eq}/U_{iso}
O1	−0.4940 (4)	0.90582 (9)	0.1769 (1)	0.0466 (4)
O2	−0.1979 (4)	0.96414 (8)	0.3498 (1)	0.0473 (4)
N1	−0.2933 (4)	0.72727 (9)	0.2512 (1)	0.0315 (4)

N4	0.1547 (4)	0.69254 (9)	0.4750 (1)	0.0343 (4)
N3	0.1909 (5)	0.8494 (1)	0.5206 (1)	0.0426 (5)
C2	−0.1726 (4)	0.7984 (1)	0.3263 (2)	0.0277 (4)
C3	0.0569 (4)	0.7819 (1)	0.4413 (1)	0.0285 (4)
C5	0.0320 (5)	0.6240 (1)	0.3979 (2)	0.0360 (5)
C6	−0.1923 (5)	0.6401 (1)	0.2860 (2)	0.0353 (5)
C7	−0.2862 (5)	0.8949 (1)	0.2865 (2)	0.0334 (5)
H1	−0.549 (7)	0.850 (2)	0.136 (2)	0.085 (9)
H2	0.319 (6)	0.830 (1)	0.589 (2)	0.054 (6)
H3	0.128 (7)	0.910 (2)	0.500 (2)	0.062 (7)

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

O1—C7	1.328 (2)	N4—C5	1.319 (2)
O2—C7	1.216 (2)	N3—C3	1.336 (2)
N1—C2	1.333 (2)	C2—C3	1.423 (2)
N1—C6	1.331 (2)	C2—C7	1.480 (2)
N4—C3	1.354 (2)	C5—C6	1.388 (2)
C2—N1—C6	118.3 (1)	N3—C3—C2	124.5 (2)
C3—N4—C5	117.7 (1)	N4—C5—C6	122.7 (1)
N1—C2—C3	121.1 (1)	N1—C6—C5	120.6 (1)
N1—C2—C7	117.8 (1)	O1—C7—O2	119.0 (2)
C3—C2—C7	121.1 (1)	O1—C7—C2	118.2 (1)
N4—C3—N3	116.0 (1)	O2—C7—C2	122.8 (1)
N4—C3—C2	119.6 (1)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.92 (2)	2.27 (2)	2.730 (2)	111 (2)
N3—H3···O2	0.91 (2)	2.04 (2)	2.718 (2)	131 (2)
O1—H1···N4^{i}	0.92 (2)	2.02 (2)	2.760 (2)	137 (2)
N3—H2···N1^{ii}	0.86 (2)	2.26 (2)	3.116 (2)	173 (2)
N3—H3···O2^{iii}	0.91 (2)	2.41 (2)	2.998 (2)	123 (2)

Symmetry codes: (i) $x-1, \frac{3}{2}-y, z-\frac{1}{2}$; (ii) $1+x, \frac{3}{2}-y, \frac{1}{2}+z$; (iii) $-x, 2-y, 1-z$.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: FG1125). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Flurtamone

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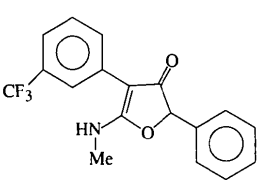
Abstract

Flurtamone, 5-methylamino-2-phenyl-4-(3-trifluoromethylphenyl)furan-3(2*H*)-one, $C_{18}H_{14}F_3NO_2$, is a weed killer. The phenyl substituents are rotated by about 30 and 72° from the mean plane of the central five-membered ring, while the methylamino substituent lies almost within this plane.

Comment

Flurtamone, (I), is an active material used as a weed killer. It is a carotenoid synthesis inhibitor and is applied before planting (pre-emergence or post-emergence) in order to control many grass and broad-leaved weeds. It is selective for cotton, peanuts, sorghum and sunflowers, but is not yet a commercial product. Its identification code is CAS RN [96525-23-4].



(I)