

A list of structure factors has been deposited with the IUCr (Reference: SE1040). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the (2-Acetamidobenzoyl)-formate Anion as the Benzamidinium Salt

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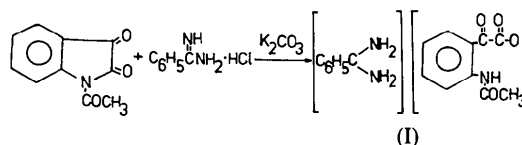
Abstract

The reaction of isatin with benzamidine hydrochloride in methanol yields the title compound, benzamidinium (2-acetamidobenzoyl)formate (C₇H₉N₂⁺·C₁₀H₈NO₄⁻). In the benzamidinium cation the configuration about the exocyclic C atoms is planar, resulting in electron delocalization in the C—N bonds. The C—O bond distances in the carboxylate

moeity in the anion are very similar (average C—O = 1.237 Å), indicating delocalization of the negative charge. Hydrogen bonds are present between the N atoms of the amidinium group and the O atoms of the carboxylate moeity of the anion. The N···O distances vary between 2.819 (5) and 2.901 (5) Å in the hydrogen bonds.

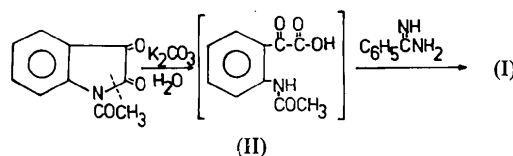
Comment

The reaction of isatin with various amino compounds to form Schiff bases has been widely studied as the products have diverse uses (Joshi, Jain & Sharma, 1986; Joshi, Dandia & Ahmed, 1989; Skiles & McNeil, 1990; Joshi, Jain & Arora, 1992; Joshi, Dandia & Ahmed, 1986). The reaction of *N*-acetyl isatin with benzamidine hydrochloride in refluxing methanolic K₂CO₃ has also been studied. The product obtained thus, (I), crystallized readily as large white needles, indicating the formation of a product other than the expected Schiff base. As a literature survey revealed that benzamidine is readily converted to a cation, resulting in ionic products (Lenhart, Lukehart, Sotiropoulos & Srinivasan, 1984; Beatrice, Kurt, Dieler & Gerhard, 1991), the molecular structure of this compound appeared to be of considerable interest.



The IR spectrum (KBr pellet) of the compound showed the following absorptions: 3400–3240 (broad, NH, NH₂), 1700 (CO), 1450 (C—N), 1320 (C···N) cm⁻¹. The following ¹H NMR parameters confirm the structure of the compound: δ 2.18 (*s*, 3H, CH₃), 3.48 (broad, 4H, NH₂), 6.86–8.38 (*m*, 9H, Ar), 11.88 (*s*, 1H, NH). The mass spectrum showed the molecular ion peak at *m/z* 327 (*M*⁺).

The title compound (I) is composed of two parts: the benzamidinium cation and the (2-acetamidobenzoyl)formate anion. The formation of this salt appears to occur through a ring-opening reaction in which a C—N bond of isatin is cleaved, followed by the addition of a water molecules resulting in the formation of (II) (an intermediate under these conditions), then a simple acid–base reaction of (II) with benzamidine rather than a Schiff base condensation.



The benzamidinium cation has the expected structure. The π electrons are delocalized over the C(7), N(1) and N(2) atoms, which is evident from the average C—N distance of 1.309 (7) Å, indicating considerable double-bond character. The sums of the bond angles about C(7), N(1) and N(2) are 359.9 (14), 360.6 (10) and 360 (13)°, respectively. These data indicate the essentially planar hybridization about these atoms. The distance between C(1) and C(7) [1.479 (7) Å] represents a normal C_{sp^2} — C_{sp^2} single bond.

The average value of the C(15)—O(2) and C(15)—O(4) bond distances in the anion is 1.238 (6) Å, indicating that the negative charge is delocalized over these atoms. The N(3)—H3*n* bond length is 1.13 (8) Å indicating normal single-bond character.

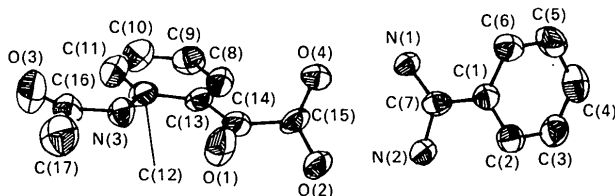


Fig. 1. An ORTEP (Johnson, 1965) plot of the title compound showing the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level.

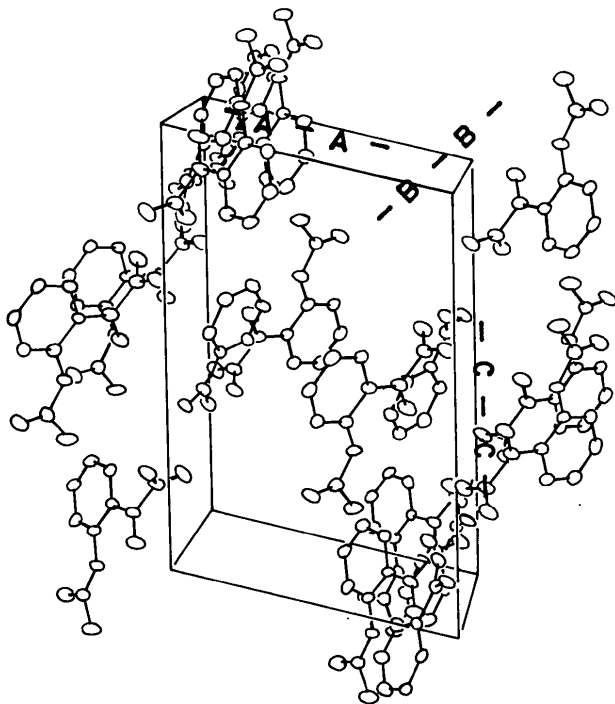


Fig. 2. The unit cell of the title compound.

The dihedral angle between the plane defined by C(14), O(1), C(15) and C(13) and that defined by C(8), C(9), C(10), C(11) and C(12) is $7.33 \pm 1.19^\circ$, which is a measure of the non-coplanarity of the phenyl ring of the indole and the carbonyl groups. The dihedral angle between the plane defined by C(7), N(1), N(2) and H1*na* and H1*nb* and that defined by C(1), C(2), C(3), C(4), C(5) and C(6) is $16.25 \pm 2.74^\circ$.

The amidinium and carboxylic parts of the molecule show intramolecular hydrogen bonding between the N and the O atoms. The N...O distances in the hydrogen bonds vary between 2.819 (5) and 2.901 (5) Å.

Experimental

A mixture of *N*-acetyl isatin (0.01 mol), benzamidine hydrochloride (0.01 mol) and K_2CO_3 (1 g) was refluxed in methanol (50 ml) for 3 h. A white product was obtained on cooling, which was then recrystallized from methanol (yield 2.25 g, 69%); m.p. 469 K. Analysis: calculated for $C_{17}H_{17}N_3O_4$, C 62.38, H 5.19, N, 12.84%; found, C 62.10, H 5.15, N 12.78%.

Crystal data

$C_7H_9N_2^+ \cdot C_{10}H_8NO_4^-$

$M_r = 327.34$

Monoclinic

$P2_1/c$

$a = 10.831 (1) \text{ \AA}$

$b = 9.723 (2) \text{ \AA}$

$c = 16.087 (2) \text{ \AA}$

$\beta = 104.07 (1)^\circ$

$V = 1643.4 (12) \text{ \AA}^3$

$Z = 4$

$D_x = 1.323 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 4-12^\circ$

$\mu = 0.090 \text{ mm}^{-1}$

$T = 295 \text{ K}$

Plate

$0.30 \times 0.15 \times 0.05 \text{ mm}$

Colourless

Data collection

Enraf-Nonius CAD-4

diffractometer

$\omega/2\theta$ scans

Absorption correction:

none

3243 measured reflections

3079 independent reflections

1097 observed reflections

$[I > 3\sigma(I)]$

$R_{int} = 0.087$

$\theta_{max} = 25^\circ$

$h = 0 \rightarrow 12$

$k = 0 \rightarrow 11$

$l = -19 \rightarrow 19$

2 standard reflections

frequency: 30 min

intensity variation: none

Refinement

Refinement on F

$R = 0.062$

$wR = 0.060$

$S = 0.68$

1097 reflections

285 parameters

All H-atom parameters

refined

$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = 0.21$

$\Delta\rho_{max} = 0.209 \text{ e \AA}^{-3}$

$\Delta\rho_{min} = -0.172 \text{ e \AA}^{-3}$

Extinction correction: none

Atomic scattering factors

from *SDP-Plus* (B. A.

Frenz & Associates, Inc.,

1983)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)
$$B_{eq} = (4/3)\sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	B _{eq}
O(1)	0.1810 (4)	0.1459 (4)	0.5221 (3)	6.6 (1)
O(2)	0.1229 (4)	0.2049 (4)	0.3375 (2)	5.7 (1)
O(3)	0.4900 (4)	-0.1155 (5)	0.7342 (2)	7.2 (1)
O(4)	0.0254 (4)	0.0089 (4)	0.3500 (2)	5.9 (1)
N(1)	-0.0671 (4)	-0.0094 (4)	0.1707 (3)	4.6 (1)
N(2)	-0.0184 (4)	0.2144 (5)	0.1595 (3)	5.3 (1)
N(3)	0.3510 (4)	-0.0089 (5)	0.6250 (3)	4.4 (1)
C(1)	-0.1308 (5)	0.0963 (6)	0.0307 (3)	3.9 (1)
C(2)	-0.1078 (5)	0.1981 (6)	-0.0232 (3)	4.4 (1)
C(3)	-0.1666 (6)	0.1922 (7)	-0.1094 (4)	5.7 (2)
C(4)	-0.2489 (6)	0.0895 (7)	-0.1418 (4)	6.2 (2)
C(5)	-0.2705 (6)	-0.0161 (7)	-0.0888 (4)	5.6 (2)
C(6)	-0.2133 (5)	-0.0109 (6)	-0.0033 (3)	4.9 (2)
C(7)	-0.0695 (5)	0.0982 (5)	0.1235 (3)	4.1 (1)
C(8)	0.3102 (5)	-0.1007 (6)	0.3989 (3)	4.6 (2)
C(9)	0.3981 (5)	-0.2018 (6)	0.4032 (3)	4.9 (2)
C(10)	0.4721 (5)	-0.2398 (6)	0.4812 (4)	5.5 (2)
C(11)	0.4578 (5)	-0.1788 (6)	0.5557 (4)	4.8 (2)
C(12)	0.3705 (5)	-0.0745 (5)	0.5532 (3)	3.8 (1)
C(13)	0.2934 (5)	-0.0345 (5)	0.4727 (3)	3.6 (1)
C(14)	0.1978 (5)	0.0747 (5)	0.4623 (3)	4.4 (1)
C(15)	0.1082 (5)	0.0951 (5)	0.3747 (3)	4.4 (1)
C(16)	0.4099 (5)	-0.0335 (6)	0.7092 (3)	4.7 (2)
C(17)	0.3611 (7)	0.0659 (8)	0.7687 (4)	7.5 (2)

Table 2. Selected geometric parameters (Å, °)

N(1)—C(7)	1.289 (7)	O(4)—C(15)	1.222 (6)
N(2)—C(7)	1.329 (7)	N(3)—C(12)	1.379 (7)
C(1)—C(2)	1.378 (8)	N(3)—C(16)	1.371 (6)
C(1)—C(6)	1.396 (7)	C(8)—C(9)	1.358 (8)
C(1)—C(7)	1.479 (7)	C(8)—C(13)	1.401 (8)
C(2)—C(3)	1.380 (7)	C(9)—C(10)	1.366 (8)
C(3)—C(4)	1.356 (9)	C(10)—C(11)	1.379 (8)
C(4)—C(5)	1.390 (9)	C(11)—C(12)	1.381 (8)
C(5)—C(6)	1.365 (7)	C(12)—C(13)	1.415 (6)
O(1)—C(14)	1.234 (7)	C(13)—C(14)	1.464 (7)
O(2)—C(15)	1.254 (6)	C(14)—C(15)	1.517 (7)
O(3)—C(16)	1.176 (7)	C(16)—C(17)	1.541 (9)
C(2)—C(1)—C(6)	119.1 (5)	N(3)—C(12)—C(11)	123.9 (4)
C(2)—C(1)—C(7)	121.4 (5)	N(3)—C(12)—C(13)	117.6 (5)
C(6)—C(1)—C(7)	119.5 (5)	C(11)—C(12)—C(13)	118.4 (5)
C(1)—C(2)—C(3)	119.6 (5)	C(8)—C(13)—C(12)	118.7 (5)
C(2)—C(3)—C(4)	121.1 (6)	C(8)—C(13)—C(14)	118.1 (4)
C(3)—C(4)—C(5)	120.1 (6)	C(12)—C(13)—C(14)	123.2 (5)
C(4)—C(5)—C(6)	119.2 (5)	O(1)—C(14)—C(13)	123.5 (4)
C(1)—C(6)—C(5)	120.8 (5)	O(1)—C(14)—C(15)	117.9 (5)
N(1)—C(7)—N(2)	119.1 (5)	C(13)—C(14)—C(15)	118.4 (5)
N(1)—C(7)—C(1)	121.6 (4)	O(2)—C(15)—O(4)	126.7 (5)
N(2)—C(7)—C(1)	119.2 (5)	O(2)—C(15)—C(14)	115.4 (4)
C(12)—N(3)—C(11)	128.2 (5)	O(4)—C(15)—C(14)	117.9 (5)
C(9)—C(8)—C(13)	121.5 (5)	O(3)—C(16)—N(3)	125.7 (5)
C(8)—C(9)—C(10)	119.4 (5)	O(3)—C(16)—C(17)	123.4 (5)
C(9)—C(10)—C(11)	121.1 (5)	N(3)—C(16)—C(17)	110.9 (5)
C(10)—C(11)—C(12)	120.9 (5)		

Table 3. Hydrogen bonds (Å)

N(1)···O(4)	2.819 (5)	N(2)···O(4 ⁱⁱ)	2.868 (6)
N(1)···O(2 ⁱ)	2.839 (5)	N(2)···O(2)	2.901 (5)

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

The data were corrected for Lorentz and polarization effects. There was no crystal decay and no absorption correction was made. The structure was solved by routine direct methods and refined using a full-matrix least-squares method with non-H atoms anisotropic. H atoms were located on a difference map

and refined with isotropic temperature factors. Programs from the *SDP-Plus* package (B. A. Frenz & Associates, Inc., 1983) were used. All calculations were carried out on a PDP11/73 computer.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the IUCr (Reference: AL1059). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Salvireptanolide

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Abstract

The whole molecule of (7*R*,8*S*,9*R*)-5,6-*seco-neo-clerodane*-1,3,5(10),13(16),14-pentaene-12-oxo-15,16-epoxy-18,19;20(7)-diolide, C₂₀H₁₈O₆, adopts a T-

† Contribution No. 1223 of Instituto de Química, UNAM.