Structure of 2'-(*N*-Isopropyloxamoyl)-acetanilide, $C_{13}H_{16}N_2O_3$

J. Zukerman-Schpector†

Instituto de Física e Química de São Carlos,
Universidade de São Paulo, Caixa Postal 369,
13560-970 São Carlos SP, Brazil

Angelo Da C. Pinto, J. F. M. Da Silva and
Rosangela B. Da Silva

Instituto de Química, Universidade Federal de Rio de
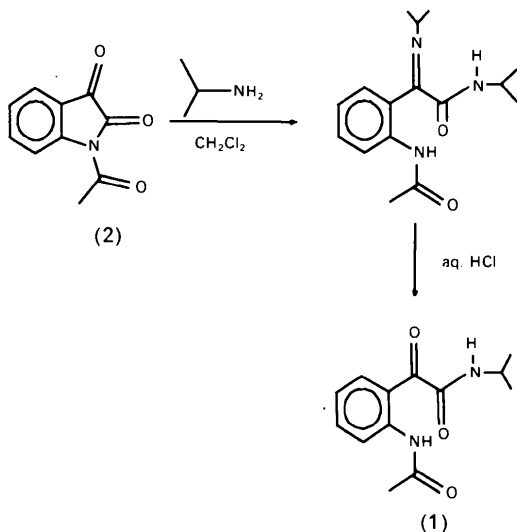*Janeiro, Cidade Universitária, 21910 Rio de Janeiro RJ,*
Brazil

Abstract

The dihedral angle between the acetyl moiety and the phenyl ring is 33.7 (3)°. The C(2)—C(3) distance is 1.530 (5) Å. The molecules are linked through hydrogen bonds.

Comment

The title compound (1) was obtained from the reaction of 1-acetylindole-2,3(2*H*,3*H*)-dione (2) with isopropylamine followed by work-up with aqueous HCl to hydrolyse the intermediate (Popp & Piccirilli, 1971), as is shown in the scheme below. Compounds possessing a structure consisting of a two-carbon bridge linking an aryl group to a nitrogen moiety, as in (1), became of great pharmacological interest when 2-phenylethylamines [*e.g.* (*R*)-(−)-norepinephrine] were seen to exhibit adrenergic activity (Korolkovas & Burckhalter, 1982). In this class of compounds, the average N—O distance is 2.9 Å and the average distance betwen the N atom and the centre of the aromatic ring is 5.4 Å (Pullman, Coubeils, Courriére & Gerbois, 1972); in the present case the values are 2.726 (4) [N(2)—O(2)] and 5.08 Å, respectively. The C(2)—C(3) distance of 1.530 (5) Å is longer than expected, the characteristic value for a $C(sp^2)$—$C(sp^2)$ bond being 1.50 Å. The O(1)—C(2)—C(3)—O(2) torsional angle is 162.4 (3) . The molecules are linked through hydrogen bonds: $N(1)\cdots O(3^i) = 2.930 (4)$, $H(N1)\cdots O(3^i) = 1.902 (2)$ Å, $N(1)—H(N1)\cdots O(3^i) = 171.0 (2)$ [symmetry code: (i) $\frac{1}{2} + x$, $\frac{1}{2} - y$, z]; $N(2)\cdots O(1^{ii}) = 2.824 (4)$, $H(N2)\cdots O(1^{ii}) = 1.891 (3)$ Å, $N(2)—H(N2)\cdots O(1^{ii}) = 157.3 (2)$ [symmetry code: (ii) $x - \frac{1}{2}$, $\frac{1}{2} - y$, z].
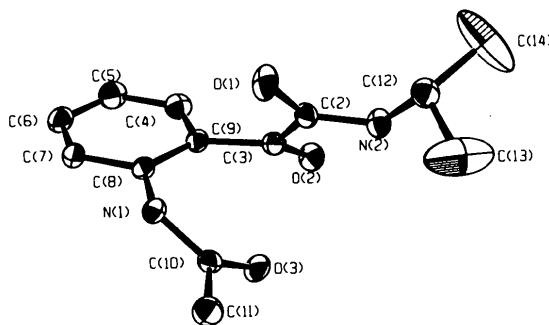

Fig. 1. The molecular structure of $C_{13}H_{16}N_2O_3$ showing the atom labelling; 20% probability thermal ellipsoids are shown for non-H atoms.

Experimental

Crystal data

$C_{13}H_{16}N_2O_3$	Mo $K\alpha$ radiation
$M_r = 248.28$	$\lambda = 0.71073$ Å
Monoclinic	Cell parameters from 23
$P2_1/a$	reflections
$a = 9.892 (2)$ Å	$\theta = 9-18°$
$b = 9.846 (1)$ Å	$\mu = 0.84$ mm^{-1}
$c = 14.169 (2)$ Å	$T = 292$ K
$\beta = 107.52 (1)°$	Irregular
$V = 1315.9 (7)$ Å^3	$0.25 \times 0.20 \times 0.18$ mm
$Z = 4$	Colourless
$D_x = 1.25$ Mg m^{-3}	Crystal source: ethyl acetate

Data collection

Enraf-Nonius CAD-4	$R_{int} = 0.016$
diffractometer	$\theta_{max} = 25°$
$\omega/2\theta$ scans	

† Present address: Departamento de Química, Universidade Federal de São Carlos, Brazil.

Absorption correction:
none
2075 measured reflections
1993 independent reflections
1361 observed reflections
$[I > 3\sigma(I)]$

$h = -11 \rightarrow 11$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 16$
1 standard reflection
  frequency: 30 min
  intensity variation: $\pm 1.0\%$

Refinement

Refinement on F
$R = 0.053$
$wR = 0.059$
$S = 1.69$
1361 reflections
164 parameters
$w = [\sigma^2(|F_o|) + 0.0006|F_o|^2]^{-1}$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Atomic scattering factors from *SHELX*76
(Sheldrick, 1976)

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

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
N(1)	0.4467 (3)	0.1739 (3)	0.1267 (2)	3.84 (9)
N(2)	0.2664 (3)	0.2795 (3)	0.3601 (2)	4.3 (1)
O(1)	0.4750 (2)	0.2108 (3)	0.3442 (2)	5.6 (1)
O(2)	0.1529 (2)	0.0485 (3)	0.2619 (2)	4.55 (8)
O(3)	0.2218 (2)	0.2385 (3)	0.1103 (2)	4.99 (9)
C(2)	0.3469 (3)	0.1995 (3)	0.3256 (2)	3.6 (1)
C(3)	0.2711 (3)	0.0805 (3)	0.2619 (2)	3.6 (1)
C(4)	0.3565 (4)	−0.1446 (4)	0.2373 (3)	4.8 (1)
C(5)	0.4402 (5)	−0.2364 (4)	0.2062 (3)	5.8 (1)
C(6)	0.5213 (4)	−0.1908 (5)	0.1486 (3)	5.6 (2)
C(7)	0.5216 (4)	−0.0553 (4)	0.1250 (3)	4.6 (1)
C(8)	0.4395 (3)	0.0386 (4)	0.1564 (2)	3.6 (1)
C(9)	0.3567 (3)	−0.0056 (3)	0.2154 (2)	3.6 (1)
C(10)	0.3394 (3)	0.2652 (4)	0.1023 (2)	3.8 (1)
C(11)	0.3712 (4)	0.3969 (4)	0.0643 (3)	5.0 (1)
C(12)	0.3256 (4)	0.3931 (4)	0.4256 (3)	4.5 (1)
C(13)	0.2808 (9)	0.5206 (6)	0.3764 (5)	14.5 (3)
C(14)	0.293 (1)	0.3835 (8)	0.5173 (5)	15.5 (4)

Table 2. *Geometric parameters* ($\text{Å}, \degree$)

N(1)—C(8)	1.405 (5)	C(4)—C(5)	1.384 (6)
N(1)—C(10)	1.354 (5)	C(4)—C(9)	1.403 (5)
N(2)—C(2)	1.314 (5)	C(5)—C(6)	1.380 (6)
N(2)—C(12)	1.459 (5)	C(6)—C(7)	1.376 (6)
O(1)—C(2)	1.219 (4)	C(7)—C(8)	1.389 (5)
O(2)—C(3)	1.211 (4)	C(8)—C(9)	1.404 (5)
O(3)—C(10)	1.231 (4)	C(10)—C(11)	1.474 (5)
C(2)—C(3)	1.530 (5)	C(12)—C(13)	1.440 (7)
C(3)—C(9)	1.485 (5)	C(12)—C(14)	1.433 (9)
C(8)—N(1)—C(10)	126.8 (3)	N(1)—C(8)—C(7)	116.6 (3)
C(2)—N(2)—C(12)	121.6 (3)	N(1)—C(8)—C(9)	124.3 (3)
N(2)—C(2)—O(1)	124.9 (3)	C(7)—C(8)—C(9)	119.1 (3)
N(2)—C(2)—C(3)	115.7 (3)	C(3)—C(9)—C(4)	114.8 (3)
O(1)—C(2)—C(3)	119.4 (3)	C(3)—C(9)—C(8)	126.9 (3)
O(2)—C(3)—C(2)	120.1 (3)	C(4)—C(9)—C(8)	118.3 (3)
O(2)—C(3)—C(9)	122.1 (3)	N(1)—C(10)—O(3)	121.6 (3)
C(2)—C(3)—C(9)	117.1 (3)	N(1)—C(10)—C(11)	116.1 (3)
C(5)—C(4)—C(9)	121.7 (4)	O(3)—C(10)—C(11)	122.2 (3)
C(4)—C(5)—C(6)	119.0 (4)	N(2)—C(12)—C(13)	110.8 (4)
C(5)—C(6)—C(7)	120.2 (4)	N(2)—C(12)—C(14)	111.7 (4)
C(6)—C(7)—C(8)	121.6 (4)	C(13)—C(12)—C(14)	112.1 (5)

Data were corrected for Lorentz and polarization. The structure was solved by direct methods. H atoms were found in difference synthesis and included as fixed contributors with an overall isotropic temperature factor that refined to $U_{iso} = 0.137$ (5) Å^2. Programs used were: *SHELXS*86 (Sheldrick, 1985), *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965). The refinement

was by full-matrix least-squares methods. Most of the calculations were performed on a VAX 6420 computer at the Instituto de Fisica e Quimica de São Carlos.

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Structure of Two 1,3,5-Substituted 2-Pyrazolines, $C_{20}H_{18}N_2O_5$ and $C_{20}H_{20}N_2O_5$

T. Pilati and G. Casalone

CNR-CSRSRC, c/o Dipartimento di Chimica Fisica ed Elettrochimica, Università degli Studi di Milano, Via Golgi 19, I-20133 Milano, Italy

Abstract

The structures of methyl 5-benzoyl-1-(2-methoxycarbonylphenyl)-2-pyrazoline-3-carboxylate (1) and methyl (±)-5-(α-hydroxybenzyl)-1-(2-methoxycarbonylphenyl)-2-pyrazoline-3-carboxylate (2a) are compared. In (1), there is an extensive conjugation involving the N—N=C chain of the pyrazoline ring and the phenyl and carboxy groups bonded at N1 and C3, respectively. In (2a), the presence of a strong hydrogen bond between the hydroxyl group and the methoxycarbonylphenyl group causes the rotation of the latter with respect to the pyrazoline ring by 47°, with a partial loss of conjugation and with the elongation of the N1–phenyl bond.