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1,1'-Diacetyl-2,2'-biimidazole

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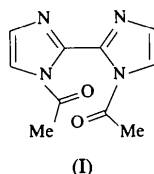
(Received 29 May 1996; accepted 17 June 1996)

Abstract

A crystallographic twofold rotation axis passes through the C—C bond joining the imidazole rings of the title compound, C₁₀H₁₀N₄O₂. The molecule crystallizes in a *cis* disposition. The planar acetyl group is twisted by 5.0 (3)° with respect to the imidazole ring and the two imidazole rings are tilted by 60.53 (5)° in relation to one another.

Comment

First studied for its antiprotozoal activity (Melloni *et al.*, 1975) and later incorporated into hexaaza macrocyclic metal complexes (Kandil & Collier, 1988), 1,1'-diacetyl-2,2'-biimidazole, (I), exhibits a *cis* conformation in the solid state, unlike all previously reported 1,1'-disubstituted 2,2'-biimidazole derivatives (Secondo, Barnett, Collier & Baughman, 1996; Sokal, Baikalova, Domnina & Poria-Koshits, 1992) which are *trans* oriented.



The bond lengths and angles of the 2,2'-biimidazole skeleton do not deviate significantly from those in the free molecule (Cromer, Ryan & Storm, 1987). The unfavorable *cis* conformation [dihedral angle 60.53 (5)°] is stabilized by intermolecular interactions between

(a) the methyl group and an imidazole N atom [C5—H···N1(*x*, −*y*, − $\frac{1}{2}$ + *z*), H···N = 2.521 (2) Å] and (b) an imidazole C—H and an O atom [C3—H···O(− $\frac{1}{2}$ + *x*, $\frac{1}{2}$ − *y*, − $\frac{1}{2}$ + *z*), H···O = 2.415 (2) Å]. The non-H atoms of each half molecule are nearly coplanar, with O1 showing the maximum deviation from the eight-atom least-squares plane [0.056 (1) Å].

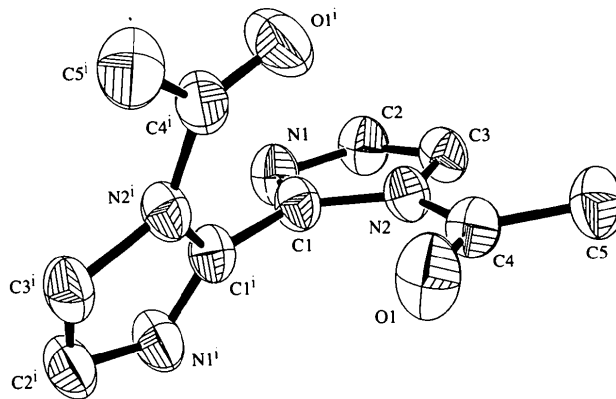


Fig. 1. View of the title molecule with displacement ellipsoids plotted at the 50% probability level.

Experimental

The preparation of the title compound has been reported previously (Melloni *et al.*, 1975). Crystals suitable for diffraction were grown by slow cooling of a hot, saturated acetic anhydride solution. Though the title material is susceptible to hydrolysis both as a solid and in acetic anhydride, none was noted during data collection as evidenced by the low variation of the check-reflection intensities.

Crystal data

C₁₀H₁₀N₄O₂
M_r = 109.11
 Monoclinic
*C*2/*c*
a = 11.987 (2) Å
b = 9.7183 (15) Å
c = 10.2177 (12) Å
 β = 119.889 (9)°
V = 1032.0 (2) Å³
Z = 4
D_x = 1.404 Mg m^{−3}
D_m not measured

Data collection

Siemens P3 diffractometer
 $\theta/2\theta$ scans
 Absorption correction:
 none
 1090 measured reflections
 914 independent reflections
 778 observed reflections
 $[I > 2\sigma(I)]$
R_{int} = 0.0077

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 50 reflections
 θ = 5.5–18.7°
 μ = 0.103 mm^{−1}
T = 293 (2) K
 Block cut from larger crystal
 0.50 × 0.38 × 0.31 mm
 Colorless

θ_{\max} = 25.07°
h = 0 → 14
k = −3 → 11
l = −12 → 10
 3 standard reflections
 monitored every 50 reflections
 intensity decay: 1.1%

RefinementRefinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.0388$ $wR(F^2) = 0.1062$ $S = 1.058$

910 reflections

74 parameters

H atoms riding, C—H

0.96 Å

 $w = 1/[\sigma^2(F_o^2) + (0.0614P)^2 + 0.3167P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.185 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.129 \text{ e } \text{\AA}^{-3}$

Extinction correction:

SHELXL93 (Sheldrick, 1993)

Extinction coefficient:

0.0041 (23)

Atomic scattering factors

from *International Tables for Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Secondo, P. M., Barnett, W. M., Collier, H. L. & Baughman, R. G. (1996). *Acta Cryst.* **C52**, 2636–2638.Sheldrick, G. M. (1990a). *Acta Cryst.* **A46**, 467–473.Sheldrick, G. M. (1990b). *SHELXTL/PC Users Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.Siemens (1991a). *P3/P4/PC Diffractometer Program*. Version 4.27. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.Siemens (1991b). *XDISK. Data Reduction Program*. Version 4.20.2PC. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.Sokal, V. L., Baikalo, L. V., Domnina, E. S. & Poria-Koshits, M. A. (1992). *Izv. Akad. Nauk SSSR Ser. Khim.* **6**, 1376–1380.Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O1	0.58121 (12)	0.25180 (14)	0.67443 (14)	0.0675 (5)
N1	0.36106 (13)	−0.08875 (14)	0.62923 (15)	0.0490 (4)
N2	0.41118 (11)	0.10724 (13)	0.55889 (13)	0.0393 (4)
C1	0.44285 (14)	0.0133 (2)	0.6744 (2)	0.0400 (4)
C2	0.2722 (2)	−0.0609 (2)	0.4803 (2)	0.0498 (5)
C3	0.30004 (14)	0.0569 (2)	0.4352 (2)	0.0452 (4)
C4	0.48132 (15)	0.2270 (2)	0.5628 (2)	0.0441 (4)
C5	0.4223 (2)	0.3111 (2)	0.4235 (2)	0.0558 (5)

Table 2. Selected geometric parameters (\AA , °)

O1—C4	1.196 (2)	N2—C4	1.424 (2)
N1—C1	1.307 (2)	C1—C1'	1.468 (3)
N1—C2	1.381 (2)	C2—C3	1.338 (2)
N2—C1	1.387 (2)	C4—C5	1.481 (2)
N2—C3	1.391 (2)		
C1—N1—C2	105.76 (14)	N2—C1—C1'	127.72 (11)
C1—N2—C3	105.91 (12)	C3—C2—N1	111.07 (14)
C1—N2—C4	127.30 (12)	C2—C3—N2	106.16 (13)
C3—N2—C4	126.71 (13)	O1—C4—N2	119.23 (15)
N1—C1—N2	111.09 (13)	O1—C4—C5	125.2 (2)
N1—C1—C1'	120.96 (11)	N2—C4—C5	115.56 (13)

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

Data collection: *P3/P4/PC Diffractometer Program* (Siemens, 1991a). Cell refinement: *P3/P4/PC Diffractometer Program*. Data reduction: *XDISK* (Siemens, 1991b). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990a). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990b). Software used to prepare material for publication: *SHELXTL/PC*, *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1165). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). **C52**, 2883–2885**Methyl 2-Benzoyloxy-3-oxo-3-phenylpropanoate**

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Abstract

The title compound, $\text{C}_{17}\text{H}_{14}\text{O}_5$, was obtained as a by-product from a procedure to regioselectively hydroxylate homochiral acid derivatives. Structural analysis confirmed its identity and showed it to be achiral.

Comment

The title compound, (I), was investigated as part of a study into the regioselective hydroxylation of 2-hydroxy-3-phenylpropanoic acid derivatives (Shaw & Tan, 1996). This structure determination was undertaken to confirm the structure of an oxidation product of the reaction, which presumably arose from the 2,3-dibromo-3-phenylpropanoate derivative. Compound (I), prepared from a chiral starting material, crystallized from a dichloromethane/hexane mix in the space group $P2_1/n$, thus requiring it to be achiral. An interesting

