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Diimidazo[1,5-*a*;1′,5′-*d*]pyrazine-5,10-dione

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Key indicators

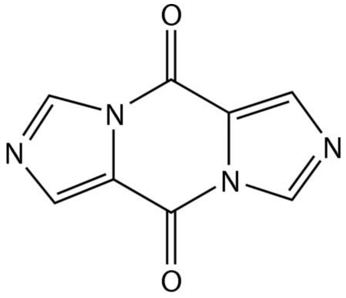
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.104
Data-to-parameter ratio = 6.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_8H_4N_4O_2$, was prepared to explore the synthesis of some 4-substituted imidazoles. The molecule is centrosymmetric and almost planar. In the crystal structure, centrosymmetric dimers are formed *via* C—H···N hydrogen bonds. The dimers are linked by further C—H···N hydrogen bonds, forming a three-dimensional network.

Comment

As noted by Kasina & Nematollahi (1975), alkyl 4-imidazolecarboxylates and their derivatives are tedious to synthesize. They reported a simple synthesis based on the reaction of diimidazo[1,5-*a*;1′,5′-d]pyrazine-5,10-dione, (I), with a number of reagents such as methanol, hydrazine, methylhydrazine and 1,1-dimethylhydrazine. Compound (I) (Fig. 1) was synthesized by the reaction of 4,5-imidazole-dicarboxylic acid with acetic anhydride.



(I)

The molecule is centrosymmetric and almost planar. The maximum deviation from the least-squares plane defined by the entire molecule is 0.010 (2) Å for atom N1. A search of the Cambridge Structural Database (Version 1.8, last update November 2006; Allen, 2002) revealed only one other compound containing the diimidazo[1,5-*a*;1′,5′-*d*]pyrazine-5,10-dione unit, namely 3,8-bis(*p*-methylphenylamino)-1,6-dimethyldiimidazo[1,5-*a*;1′,5′-*d*]pyrazine-5,10-dione, (II) (Burak *et al.*, 1992). A comparison of the bond distances and angles indicates some differences. For example, bond C4═O1 is 0.028 Å shorter, while bonds C3—C4 and N1—C3 are 0.024 and 0.026 Å, respectively, longer in (I) than in (II). For the remainder of the molecule the differences are between 1 and 4 s.u.

In the crystal structure of (I), centrosymmetric dimers are formed *via* C2—H2···N2^{ii} hydrogen bonds. These dimers are futher linked by C1—H1···N2^{i} hydrogen bonds, forming a three-dimensional network. Details of the hydrogen bonding and symmetry codes are given in Table 1 and Fig. 2.

Experimental

Compound (I) was synthesized according to the procedure described by Kasina & Nematollahi (1975). Imidazole-4,5-dicarboxylic acid (2.0 g, 12.82 mmol) was introduced, under an atmosphere of nitrogen, into a 50 ml three-necked flask equipped with a magetic stirring bar. An excess of acetic anhydride (20 ml) was added and the mixture stirred and heated under reflux at 433 K for 3 d. The dark solution obtained was evaporated under vacuum, giving a sticky solid which was washed with CCl_4. When all the acetic acid had been eliminated, the residue was sublimed on heating at 463 K, using an oil bath and under vacuum. A pure yellow solid was obtained on the refrigerated finger. On recrystallization from $CHCl_3$, pale-yellow rod-like X-ray-quality crystals of (I) were obtained (yield 0.42 g, 35%).

Crystal data

$C_8H_4N_4O_2$
$M_r = 188.15$
Monoclinic, $P2_1/c$
$a = 5.4047$ (19) Å
$b = 6.9777$ (18) Å
$c = 9.678$ (4) Å
$\beta = 96.23$ (3)°
$V = 362.8$ (2) Å^3
$Z = 2$
$D_x = 1.722$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.13$ mm^{-1}
$T = 173$ (2) K
Rod, pale yellow
0.23 × 0.13 × 0.10 mm

Data collection

Stoe IPDS-2 diffractometer
ω and φ scans
Absorption correction: none
1582 measured reflections
471 independent reflections
338 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.051$
$\theta_{\rm max} = 25.6°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.104$
$S = 0.97$
471 reflections
73 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0657P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.19$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.065 (16)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···N2$^{\rm i}$	0.97 (3)	2.49 (3)	3.440 (4)	166 (3)
C2—H2···N2$^{\rm ii}$	0.96 (3)	2.57 (3)	3.362 (4)	140 (3)

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

Crystals of compound (I) were non-merohedral twins (matrix 1.00406, 0.00693, 0.00563, −0.00918 − 1.00824, −0.00523, −0.38756, 0.01207, −1.00098). The TWIN integration routine (*X-AREA*; Stoe & Cie, 2005) was used to obtain the combined data file. Approximately 32% of the reflections were overlapped and were eliminated from the final data used here. The H atoms were located in difference Fourier maps and refined isotropically; the C—H bond lengths are 0.96 (3) and 0.97 (3) Å.

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

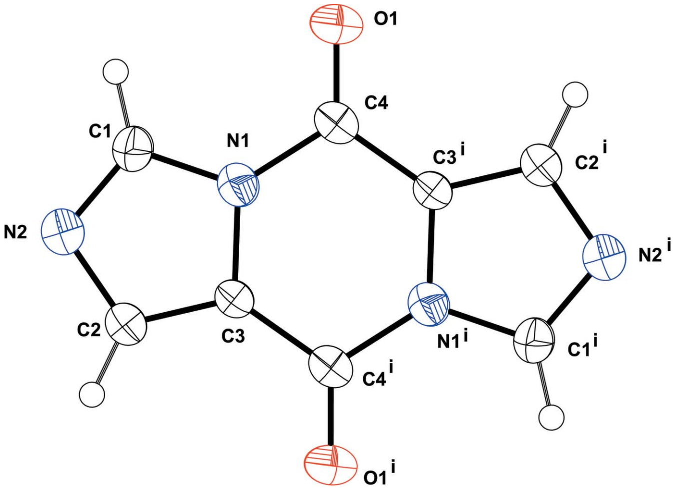


Figure 1
The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level (arbitrary spheres for the H atoms). Atoms labelled with the suffix i are generated by the symmetry operation $(2-x, -y, 1-z)$.

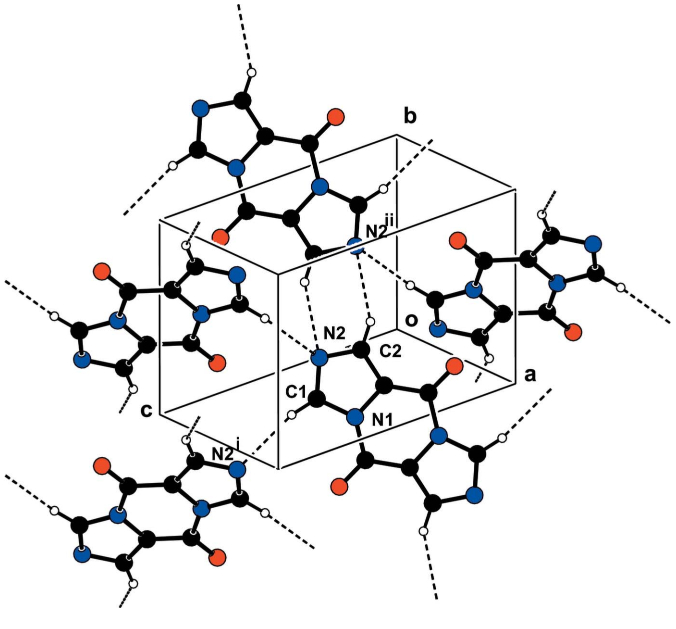


Figure 2
The crystal packing of (I), viewed perpendicular to (011). The C—H···N hydrogen bonds are shown as dashed lines; symmetry codes correspond to those in Table 2.

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References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Burak, K., Machon, Z. & Glowiak, T. (1992). *J. Crystallogr. Spectrosc. Res.* **22**, 665–671.
Kasina, S. & Nematollahi, J. (1975). *Synthesis*, pp. 162–163.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie. (2005). *X-AREA* (Version 1.26) and *X-RED32* (Version 1.26). Stoe & Cie GmbH, Darmstadt, Germany.