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Dimethyl 5-oxo-4,5-dihydro-1*H*-tetrazole-1,4-dicarboxylate

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A notable feature of the structure of the title compound, $C_5H_6N_4O_5$, is the presence of four crystallographically independent and essentially planar but otherwise unremarkable molecules in the asymmetric unit. The molecules are distributed as two pairs which differ in conformation from one pair to the other in the orientation of one of the carbomethoxy substituents relative to the tetrazole ring.

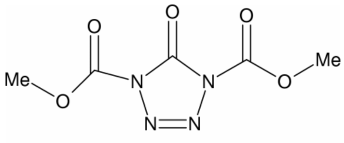
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(N–C) = 0.013 Å
R factor = 0.085
wR factor = 0.247
Data-to-parameter ratio = 8.2

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

Comment

The asymmetric unit of the title compound, (I), contains four crystallographically independent molecules. The constituent atoms of all four molecules have been labelled in an identical manner except that the individual molecules are identified by the suffix *A*, *B*, *C* or *D*. The molecules can be considered in pairs as *A* with *B* (Fig. 1) and *C* with *D* (Fig. 2), which clearly differ in the orientation of one carbomethoxy group relative to the tetrazole ring. As can be seen in Table 1, which compares the geometric parameters for all four molecules, the only values, aside from the torsion angles about the C3—N4 bond, which are sensitive to the change in conformation are the C3—N4—C5 and C3—N4—N3 angles. Aside from this the bond lengths and angles are fully consistent with the connectivity and bond types indicated in the *Scheme* below.



(I)

Experimental

Compound (I) was prepared by saturating a vigorously stirred ice-cold solution of tetrazol-5-one (Haines *et al.*, 1982) (1.72 g, 0.02 mol) and dry pyridine (3.96 g, 0.05 mol) in anhydrous diethyl ether (100 ml) with gaseous phosgene ($COCl_2$), allowing the solution to come to room temperature (298 K) and continuing stirring for a further 10 min before the addition of methanol (10 ml) and further pyridine (3.96 g, 0.05 mol). After filtration removal of solvent from the filtrate furnished solid (I), which was recrystallized from diethyl ether to provide crystals suitable for analysis.

Crystal data

$C_5H_6N_4O_5$
M_r = 202.14
Monoclinic, $P2_1$
a = 10.023 (17) Å
b = 15.92 (3) Å
c = 12.123 (15) Å
β = 115.67 (11)°
V = 1744 (5) Å^3
Z = 8

D_x = 1.540 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 reflections
θ = 8.0–11.5°
μ = 0.14 mm^{-1}
T = 298 (2) K
Block, colourless
0.64 × 0.50 × 0.34 mm

Data collection

Nicolet *P*3 diffractometer
θ–2*θ* scans
4147 measured reflections
4147 independent reflections
2144 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 30.0°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 22$
$l = -17 \rightarrow 15$
2 standard reflections
 every 50 reflections
 intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.085$
$wR(F^2) = 0.247$
$S = 1.00$
4147 reflections
505 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1438P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.60$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}

Table 1

Comparison of bond lengths and angles (Å, °) for molecules *A*, *B*, *C* and *D* of (I).

	A	*B*	*C*	*D*
N1—N2	1.392 (10)	1.366 (11)	1.370 (11)	1.403 (11)
N3—N4	1.376 (11)	1.391 (9)	1.496 (13)	1.410 (11)
N2—N3	1.257 (12)	1.273 (11)	1.182 (11)	1.235 (12)
N1—C5	1.387 (12)	1.372 (10)	1.455 (12)	1.395 (11)
N4—C5	1.404 (11)	1.382 (12)	1.322 (12)	1.370 (12)
N1—C1	1.419 (12)	1.409 (12)	1.377 (12)	1.418 (11)
N4—C3	1.416 (13)	1.402 (11)	1.397 (12)	1.463 (14)
C5—O5	1.187 (10)	1.191 (10)	1.146 (11)	1.177 (10)
C1—O1	1.197 (11)	1.174 (12)	1.237 (13)	1.176 (10)
C3—O3	1.197 (12)	1.196 (10)	1.177 (12)	1.145 (12)
C1—O2	1.313 (12)	1.315 (11)	1.232 (13)	1.307 (11)
C3—O4	1.300 (11)	1.295 (11)	1.317 (12)	1.309 (11)
O2—C2	1.428 (13)	1.484 (13)	1.563 (13)	1.449 (13)
O4—C4	1.439 (12)	1.455 (12)	1.462 (13)	1.515 (18)
C5—N1—N2	112.0 (8)	110.2 (7)	107.7 (7)	110.2 (8)
N1—N2—N3	108.6 (8)	110.7 (7)	115.0 (9)	108.9 (8)
N2—N3—N4	107.9 (7)	105.8 (7)	104.5 (7)	108.6 (8)
N3—N4—C5	112.6 (5)	111.6 (6)	111.7 (8)	110.9 (8)
N1—C5—N4	99.0 (7)	101.7 (7)	101.1 (8)	101.4 (7)
O5—C5—N1	130.0 (8)	128.6 (9)	128.3 (8)	128.6 (9)
O5—C5—N4	130.9 (9)	129.5 (8)	130.7 (9)	130.0 (10)
C1—N1—C5	126.3 (8)	126.6 (7)	127.3 (8)	127.9 (7)
C1—N1—N2	121.6 (8)	123.2 (7)	124.9 (8)	120.7 (7)
O1—C1—N1	121.8 (10)	121.8 (8)	120.4 (10)	121.5 (8)
O2—C1—N1	110.4 (8)	109.2 (8)	110.1 (10)	108.0 (8)
O1—C1—O2	127.8 (10)	128.9 (10)	129.4 (11)	130.5 (9)
C2—O2—C1	115.8 (9)	112.9 (8)	113.5 (10)	116.4 (9)
C3—N4—C5	124.3 (7)	127.0 (7)	134.3 (9)	137.1 (9)
C3—N4—N3	123.1 (7)	121.3 (7)	113.9 (8)	111.7 (8)
O3—C3—N4	122.7 (8)	122.4 (8)	124.4 (10)	122.9 (10)
O4—C3—N4	110.2 (8)	110.7 (7)	107.1 (9)	104.0 (10)
O3—C3—O4	126.9 (10)	126.9 (9)	128.5 (10)	133.0 (12)
C4—O4—C3	116.3 (8)	115.6 (8)	113.0 (9)	107.2 (10)
O1—C1—N1—C5	−1.3 (13)	−1.6 (13)	4.9 (13)	13.6 (13)
O1—C1—N1—N2	173.3 (8)	−178.9 (8)	−177.0 (8)	−180.0 (8)
O2—C1—N1—C5	176.1 (8)	−178.4 (7)	−177.7 (7)	−168.6 (8)
O2—C1—N1—N2	−9.3 (11)	4.2 (11)	0.4 (12)	−2.2 (10)
O3—C3—N4—C5	−8.2 (13)	6.4 (12)	174.3 (9)	173.9 (10)
O3—C3—N4—N3	173.2 (9)	−176.7 (8)	−3.4 (13)	2.2 (12)
O4—C3—N4—C5	176.9 (7)	−176.0 (7)	−5.1 (13)	−5.1 (12)
O4—C3—N4—N3	−1.6 (11)	0.8 (10)	177.2 (7)	−176.9 (7)

In the final stages of refinement, the H atoms of the methyl groups were introduced in calculated positions, with C—H = 0.96 Å, and allowed for as riding, with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. The approximate orientation of the methyl groups was initially determined but not further refined by employing the *SHELXL*97 (Sheldrick, 1997) AFIX 133 instruction.

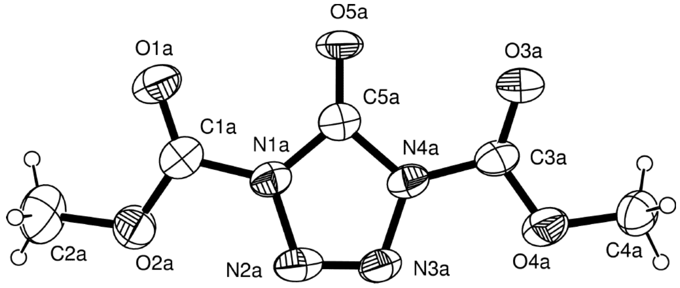


Figure 1

Molecule *A* of (I), showing the labelling scheme and the conformation of the molecule, both of which apply also to molecule *B*. Non-H atoms are shown as 30% probability displacement ellipsoids and H atoms as small circles.

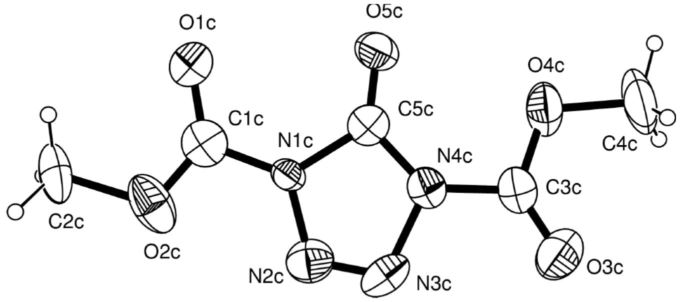


Figure 2

The labelling scheme and molecular conformation of molecules *C* and *D* of (I). The example shown is molecule *C* and the representation is otherwise the same as in Fig. 1.

Data collection: *Nicolet P*3 *Software* (Nicolet, 1980); cell refinement: *Nicolet P*3 *Software*; data reduction: *RDNIC* (Howie, 1980); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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