Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Diisopropyl hydrazocarboxylate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.069
wR factor = 0.143
Data-to-parameter ratio = 14.8

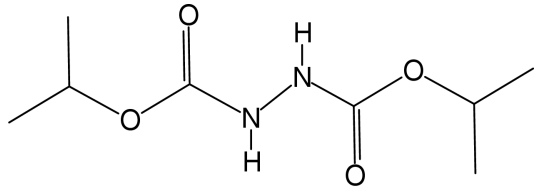
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 20 July 2001
Accepted 25 July 2001
Online 27 July 2001

The title compound, $C_8H_{16}N_2O_4$, is the product of a Mitsunobu coupling using the diisopropylazodicarboxylate. It forms hydrogen-bonded chains similar to those in its previously reported diethyl analogue.

Comment

The title compound, (I), was obtained as a byproduct of a Mitsunobu coupling reaction using triphenylphosphine and diisopropylazodicarboxylate as reagents. The N1—N2 bond is 1.380 (3) Å, and the two C═O bond distances are 1.216 (3) and 1.217 (4) Å. These values are in agreement with the parameters of 1.381 and 1.202/1.206 Å, respectively, reported for the 1:1 adduct of (I) with triphenylphosphine oxide (Héroux & Brisse, 1997). The torsion angle around the central N—N bond is 73.2° in the latter structure, compared with 100.4° in the title compound.



(I)

Each molecule of (I) is connected to two adjacent molecules by four N—H···O hydrogen bonds (Fig. 2 and Table 1). The hydrogen-bond linkage between two neighbouring molecules *A* and *B* involves one H-atom donor site and one acceptor site in each of them, N—H(*A*)···O(*B*) and N—H(*B*)···O(*A*). The hydrogen-bonded chains propagate parallel to [100], with N···O distances of 2.853 (3) and 2.829 (3) Å and N—H···O bond angles of 157 and 174°. A similar hydrogen-bonded one-dimensional network occurs in the diethyl analogue of (I) (Linke & Kalker, 1977).

In contrast, in the 1:1 adduct of diisopropyl hydrazocarboxylate, (I), with triphenylphosphine oxide, (II), the carbonyl O atoms do not participate in hydrogen bonding. Instead, each phosphoryl O atom of (II) is a bifurcated acceptor to the NH groups of two molecules of (I). The result is a cyclic arrangement of four molecules, two (I) + two (II) (Héroux & Brisse, 1997).

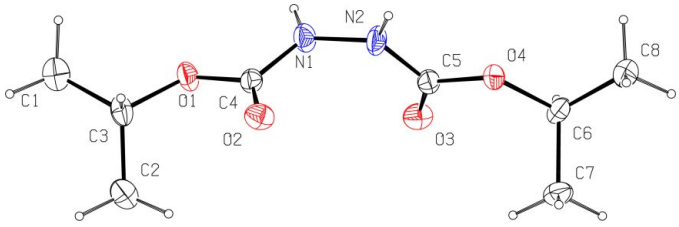
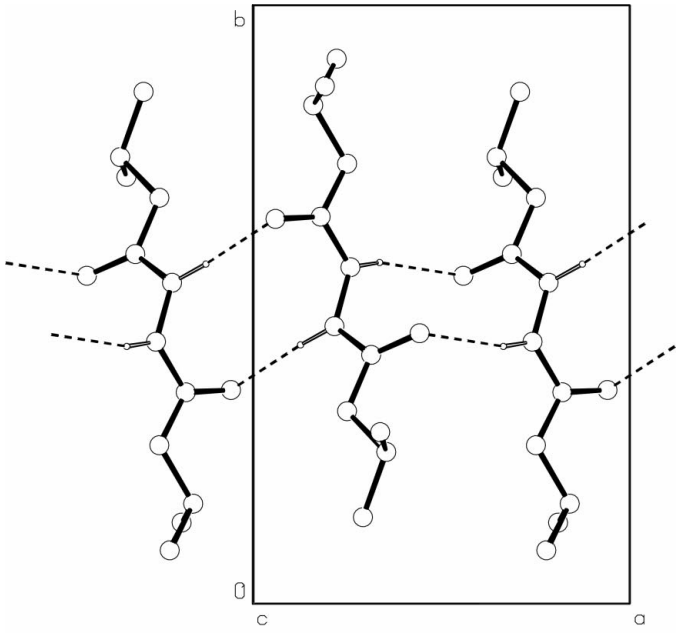


Figure 1
The structure of (I) showing 50% probability displacement ellipsoids.



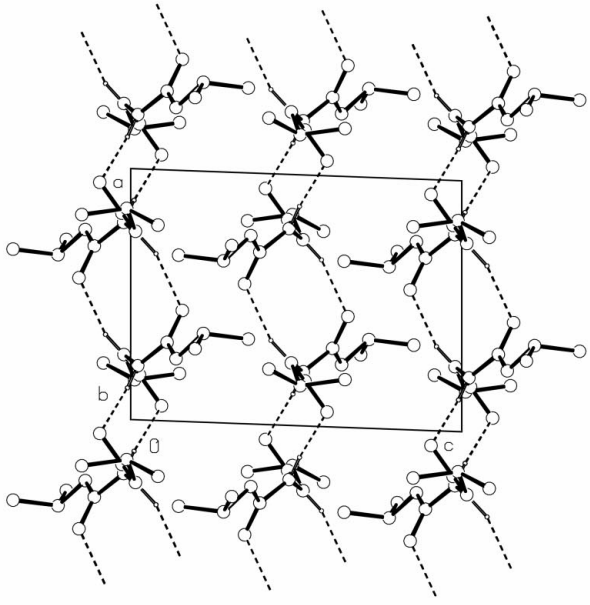


Figure 2
Projections of the crystal structure viewed along *c* (top) and along *b* showing hydrogen-bonded chains parallel to [100] (bottom).

Experimental

Crystals of (I) were isolated as colourless needles from $CHCl_3$.

Crystal data

$C_8H_{16}N_2O_4$
M_r = 204.23
Monoclinic, $P2_1/c$
a = 8.0299 (16) Å
b = 12.765 (3) Å
c = 10.587 (2) Å
β = 92.11 (3)°
V = 1084.4 (4) Å^3
Z = 4
D_x = 1.251 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4438 reflections
θ = 3.0–25.0°
μ = 0.10 mm^{-1}
T = 120 (2) K
Needle, colourless
0.12 × 0.05 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans to fill Ewald sphere
Absorption correction: multi-scan (Blessing, 1997)
T_{min} = 0.988, T_{max} = 0.995
4768 measured reflections
1890 independent reflections
1289 reflections with $I > 2\sigma(I)$
R_{int} = 0.099
θ_{max} = 25.0°
$h = -9 \rightarrow 9$
$k = -15 \rightarrow 15$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.143
S = 1.06
1890 reflections
128 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0001P)^2 + 1.7000P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.014 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.88	2.02	2.853 (3)	157
N2—H2···O2^{ii}	0.88	1.95	2.829 (3)	174

Symmetry codes: (i) $1-x, -y, -z$; (ii) $-x, -y, -z$.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1990).

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