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Dimethyl *N*,*N*′-(4-methyl-*m*-phenylene)dicarbamate

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Key indicators

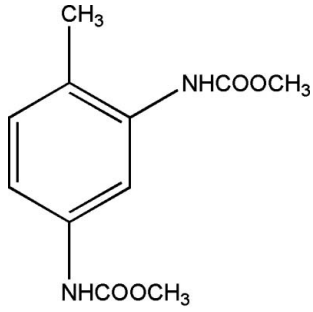
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.052
wR factor = 0.171
Data-to-parameter ratio = 17.1

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

In the crystal structure of the title compound, $C_{11}H_{14}N_2O_4$, which can be used as an intermediate for the syhtnesis of isocyanates, there are two intermolecular N—H···O hydrogen bonds.

Comment

The title compound, (I), can be used as an intermediate for the synthesis of isocyanates (Uriz *et al.*, 2002). Compound (I) was first prepared from toluene 2,4-bis(isocyanate) by reaction with methanol by Siefken (1949), but the single-crystal structure has not been reported until now.



(I)

The molecular structure of (I) is shown in Fig. 1. The crystal packing projected on to the *bc* face is shown in Fig. 2. There are two kinds of intermolecular N—H···O hydrogen bonds (Table 1). The N1—H1*A*···O3 and N2—H2*A*···O1 hydrogen bonds are approximately parallel to the *ab* face.

Experimental

Toluene-2,4-bis(isocyanate) (TDI) was provided by Hebei Cangzhou Dahua Co. Ltd. The raw material was purified twice by a melt crystallization process. Compound (I) was prepared by the reaction of purified TDI with methanol (m.p. 446.5 K by differential scanning calorimetry). Colorless needle-like single crystals suitable for X-ray diffraction were obtained by slow evaporation of a methanol solution at room temperature over a period of 3 d.

Crystal data

$C_{11}H_{14}N_2O_4$
M_r = 238.24
Triclinic, $P\overline{1}$
a = 7.4223 (15) Å
b = 8.1444 (16) Å
c = 10.642 (2) Å
α = 74.01 (3)°
β = 71.64 (3)°
γ = 75.44 (3)°
V = 577.3 (2) Å^3
Z = 2
D_x = 1.371 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5473 reflections
θ = 3.1–27.5°
μ = 0.11 mm^{-1}
T = 293 (2) K
Needle, colorless
0.64 × 0.22 × 0.17 mm

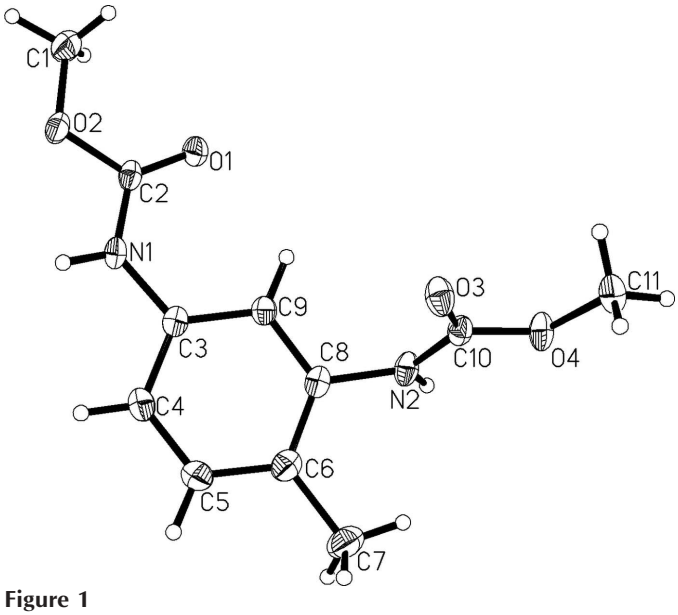


Figure 1
ORTEPII (Johnson, 1976) view of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

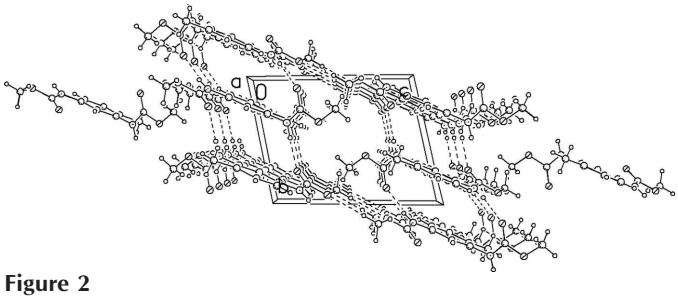


Figure 2
The molecular packing of compound, (I), viewed along the *a* axis. Dashed lines indicate hydrogen bonds.

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer	2630 independent reflections
ω scans	2314 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	$R_{int} = 0.029$
$T_{min} = 0.936$, $T_{max} = 0.982$	$\theta_{max} = 27.5^\circ$
5743 measured reflections	$h = -9 \rightarrow 9$
	$k = -10 \rightarrow 10$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1123P)^2 + 0.3315P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.052$	
$wR(F^2) = 0.171$	
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.036$
2630 reflections	$\Delta\rho_{max} = 0.53$ e Å^{-3}
154 parameters	$\Delta\rho_{min} = -0.57$ e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3^{i}	0.86	2.08	2.9339 (18)	172
N2—H2A···O1^{ii}	0.86	2.00	2.8597 (19)	174

Symmetry codes: (i) $-x, -y+2, -z+2$; (ii) $-x, -y+1, -z+2$.

H atoms were placed in calculated positions and constrained to ride on their parent atoms, with distances C—H = 0.93–0.96 Å and N—H = 0.86 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) and 1.5U_{eq}(C_{methyl})

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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