

N,N'-Bis(4-biphenyl)urea

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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$

R factor = 0.043

wR factor = 0.133

Data-to-parameter ratio = 17.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The crystal structure of the title compound, $\text{C}_{25}\text{H}_{20}\text{N}_2\text{O}$, has the $\text{N}-\text{H}\cdots\text{O}$ α -network typical of diaryl ureas.

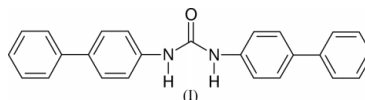
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Comment

The title compound, (I) (Fig. 1), was synthesized as part of our ongoing study of the crystal packing in urea structures (George *et al.*, 2001; George & Nangia, 2001). Symmetrical disubstituted ureas generally form a polar hydrogen-bond chain, with *anti* NH donors and carbonyl O-atom acceptors in a bifurcated motif. Compound (I) crystallizes in the polar space group $Pna2_1$. Phenyl rings *A* and *C*, as well as *B* and *D* (see Fig. 1 for labelling), are twisted with respect to each other by -25.9 (5) and 24.2 (5) $^\circ$, respectively (Table 1). Phenyl rings *A* and *B* are tilted with respect to the urea plane by 57.1 (5) and -37.1 (5) $^\circ$ (Table 1). The α -network is formed by *a*-glide-related molecules through $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2 and Fig. 2). The outer phenyl rings (*C* and *D*) of *c*-translated molecules are close-packed, with no specific interactions. Screw-axis-related layers, viewed down the *b* axis, are arranged with a slight offset of hydrogen-bonded chains. Compound (I) crystallizes in the same space group as *N,N'*-diphenylurea [Dannecker *et al.*, 1979; $a = 9.091$ (8), $b = 10.535$ (9) and $c = 11.768$ (10) \AA], with similar *a* and *b* axes, but a longer *c* axis because of the biphenyl group.



Experimental

Compound (I) was prepared by condensation of an aryl amine with triphosgene (Corbin *et al.*, 2001). A solution of triphosgene (300 mg, 1 mmol) in dichloromethane (3 ml) was added dropwise over 1 h to a solution of 4-biphenylamine (857 mg, 5 mmol) and 4-dimethylaminopyridine (773 mg, 6 mmol) in dichloromethane (7 ml). The

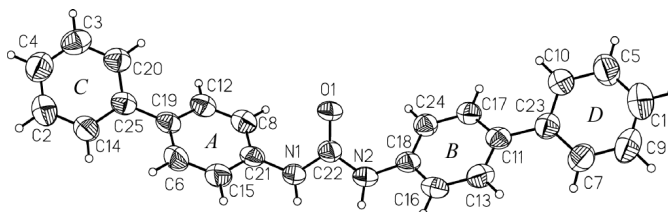


Figure 1

ORTEP II (Johnson, 1976) diagram and the atom-numbering scheme for (I); displacement ellipsoids are drawn at the 50% probability level for non-H atoms.

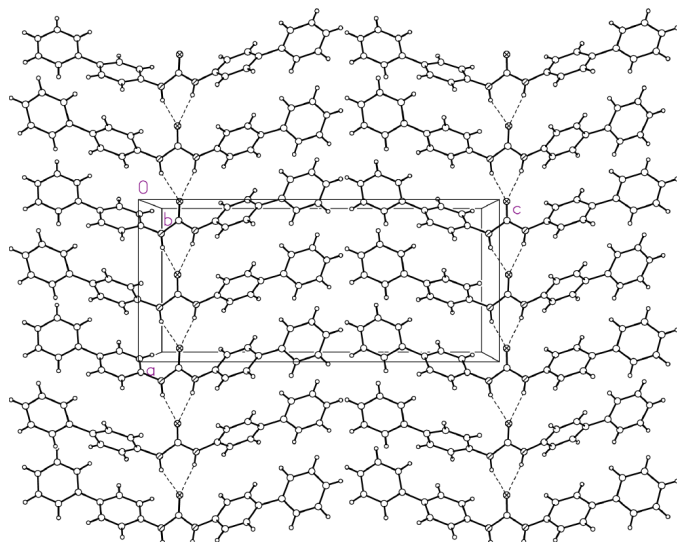


Figure 2
Packing diagram of (I), showing the N—H···O hydrogen-bonded α -network along the *a* axis.

resulting solution was stirred for 27 h or until no starting material remained (thin-layer chromatography). Nitrogen was bubbled through the reaction mixture to displace any unreacted phosgene and the solvent was removed under vacuum. The compound was recrystallized from dimethyl sulfoxide (m.p. above 538 K)

Crystal data

$C_{25}H_{20}N_2O$
 $M_r = 364.43$
Orthorhombic, $Pna2_1$
 $a = 8.9673$ (18) Å
 $b = 10.528$ (2) Å
 $c = 19.971$ (4) Å
 $V = 1885.5$ (7) Å³
 $Z = 4$
 $D_x = 1.284$ Mg m⁻³

Data collection

Enraf–Nonius CAD-4
diffractometer
 ω scans
4948 measured reflections
4296 independent reflections
2077 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.00$

Mo $K\alpha$ radiation
Cell parameters from 25
reflections
 $\theta = 9.6$ – 10.7°
 $\mu = 0.08$ mm⁻¹
 $T = 293$ (2) K
Plate, brown
 $0.15 \times 0.14 \times 0.10$ mm

$\theta_{max} = 27.4^\circ$
 $h = 0 \rightarrow 11$
 $k = 0 \rightarrow 13$
 $l = -25 \rightarrow 25$
3 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.133$
 $S = 0.84$
4296 reflections
253 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2)]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.11$ e Å⁻³
 $\Delta\rho_{min} = -0.13$ e Å⁻³

Table 1

Selected geometric parameters ($^\circ$).

C12—C19—C25—C20	−25.9 (5)	C22—N1—C21—C8	57.1 (5)
C17—C11—C23—C10	24.2 (5)	C22—N2—C18—C24	−37.1 (5)

Table 2

Hydrogen-bonding geometry (Å, $^\circ$).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···O1 ⁱ	0.86	2.14	2.918 (3)	149
N2—H2A···O1 ⁱ	0.86	2.07	2.878 (3)	156

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *Xtal3.5* (Hall *et al.*, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLUTON* (Spek, 1997); software used to prepare material for publication: *SHELXL97*.

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