

8-Quinolinyurea

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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.044

wR factor = 0.098

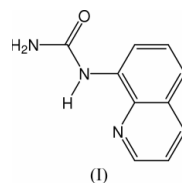
Data-to-parameter ratio = 11.2

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

The crystal structure of 8-quinolinyurea, $\text{C}_{10}\text{H}_9\text{N}_3\text{O}$, shows the urea moiety to be close to coplanar with the quinoline ring, due to intramolecular hydrogen bonding between the quinoline-N atom and the H atom on the nearest urea N, and between the urea O atom and a quinoline-ring H atom. The molecules associate, through hydrogen bonds involving all potential donor and acceptor sites, to form a two-dimensional sheet structure.

Comment

We have previously reported the synthesis and crystal structures of unsymmetrically substituted ureas together with their adducts with a number of carboxylic acids. These ureas included phenylurea (Kashino & Haisa, 1977; Bott *et al.*, 2000) and 1,1-diethylurea (Smith & Kennard, 2000; Smith *et al.*, 2000). These unsymmetrically substituted ureas are of interest because of their potential herbicidal properties, *e.g.* monuron [3-(4-chlorophenyl)-1,1-dimethylurea; Baughman, Hembre *et al.*, 1980] and diuron [3-(3,4-dichlorophenyl) 1,1-dimethylurea; Baughman, Sams *et al.*, 1980] are commercial herbicides. The structure of the 1:1 proton-transfer compound of the unsymmetrical Lewis base-substituted urea (8-quinoliny)urea with 3,5-dinitrosalicylic acid has previously been reported (Smith *et al.*, 2001) and we report here the crystal structure of the parent urea compound, (8-quinoliny)urea, (I), which has also been investigated for its phytotoxic properties (Pagani *et al.*, 1983; Smith *et al.*, 1997).



This determination shows only minor deviations from planarity in the overall molecule (Fig. 1), with the torsion angles $\text{C7}-\text{C8}-\text{N11}-\text{C21}$ and $\text{C8}-\text{N11}-\text{C21}-\text{N31}$ being $8.6(3)$ and $171.0(1)^\circ$, respectively. This is largely due to the presence of intramolecular hydrogen bonds, on one side between the hetero-N atom of the quinoline residue and the H atom on the nearest urea-N atom [$\text{N11}\cdots\text{N1} = 2.685(2) \text{ \AA}$], and on the other side between the urea O atom and a quinoline ring H atom [$\text{O21}\cdots\text{C7} = 2.895(2) \text{ \AA}$]. The (8-quinolinium)urea cations in the 1:1 proton-transfer compound with 3,5-dinitrosalicylic acid are considerably different conformationally, with the urea side chain inverted and non-coplanar with the quinoline ring and with no intramolecular

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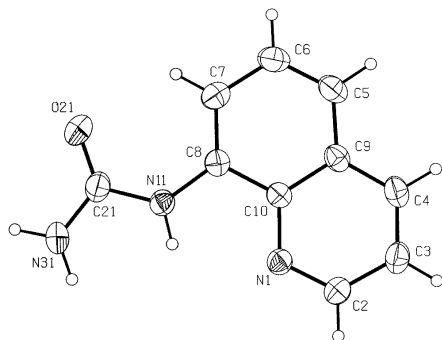


Figure 1
The molecular configuration and atom-labelling scheme for (I), with displacement ellipsoids drawn at the 30% probability level.

hydrogen bonds. The molecules of (I) also form a cyclic hydrogen-bonding association through the urea functional groups and the hetero-N atom of the quinoline ring (Fig. 2 and Table 1). These dimers are then extended into a convoluted two-dimensional sheet structure *via n*-glide-related molecules, with a separation of *ca* 3.8 Å [$Cg1 \cdots Cg1 = 3.838(3)$ Å ($Cg1$ is the centroid of the N11-containing ring) and $\alpha = 0.02(2)^\circ$] between the sheets.

Experimental

The title compound, (I), was synthesized using the Vogel (1989) procedure. 8-Aminoquinoline (10 g, 0.069 mmol) was dissolved in 10 ml of hot glacial acetic acid and a solution of NaCNO (4.51 g, 0.069 mmol) in 50 ml of warm water was added with stirring, and the warmed solution stirred for a further 30 min. The mixture was cooled on an ice bath for 30 min and the precipitate of (I) removed by vacuum filtration and vacuum dried, giving 10 g of off-white product (77% yield). Recrystallization from absolute ethanol gave colourless crystals (m.p. 476–477 K) suitable for X-ray diffraction.

Crystal data

$C_{10}H_9N_3O$	$D_x = 1.402 \text{ Mg m}^{-3}$
$M_r = 187.20$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 978 reflections
$a = 7.1436(8) \text{ \AA}$	$\theta = 2.7\text{--}22.1^\circ$
$b = 8.1394(9) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 15.2836(17) \text{ \AA}$	$T = 293(2) \text{ K}$
$\beta = 93.333(2)^\circ$	Block, colourless
$V = 887.16(17) \text{ \AA}^3$	$0.10 \times 0.16 \times 0.32 \text{ mm}$
$Z = 4$	

Data collection

Bruker CCD area-detector diffractometer	1133 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.050$
Absorption correction: none	$\theta_{\text{max}} = 25.0^\circ$
4542 measured reflections	$h = -8 \rightarrow 6$
1566 independent reflections	$k = -9 \rightarrow 9$
	$l = -15 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.044$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.098$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.97$	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
1566 reflections	$\Delta\rho_{\text{min}} = -0.12 \text{ e \AA}^{-3}$
140 parameters	Extinction correction: <i>SHELXTL97</i>
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.0062 (18)

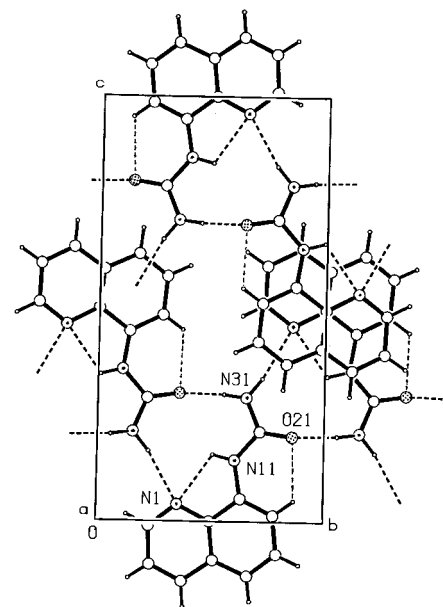


Figure 2
Packing in the unit cell, viewed down *a*, with hydrogen-bonding interactions shown as dashed lines.

Table 1

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N11-H11 \cdots N1$	0.859 (17)	2.227 (17)	2.685 (2)	113.3 (14)
$N31-H31A \cdots O21^i$	0.91 (2)	1.94 (2)	2.836 (3)	171 (2)
$N31-H31B \cdots N1^{ii}$	0.90 (2)	2.19 (2)	3.072 (2)	166.1 (18)
$C7-H7 \cdots O21$	0.93	2.30	2.895 (2)	122

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

H atoms attached to N11 and N31 (H11, H31A and H31B) were located in difference syntheses and their positional and isotropic displacement parameters were refined. Other H atoms were included in the refinement in the riding-model approximation.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXTL97* (Bruker, 1997); program(s) used to refine structure: *SHELXTL97*; molecular graphics: *PLATON for Windows* (Spek, 1999); software used to prepare material for publication: *SHELXTL97*.

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