

***N,N'*-Dicyclohexyl-*N*-(2-pyrazinoyl)urea**

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**Key indicators**

Single-crystal X-ray study  
 $T = 153\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.028  
 $wR$  factor = 0.058  
Data-to-parameter ratio = 10.8

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

The title compound,  $\text{C}_{18}\text{H}_{26}\text{N}_4\text{O}_2$ , is obtainable from 1,3-dicyclohexylcarbodiimide and pyrazine-2-carboxylic acid in dimethylformamide in the presence of catalytic amounts of  $[\text{H}_3\text{Ru}_4(\text{C}_6\text{H}_6)_4\text{OH}]\text{Cl}_2$ . The intermolecular  $\text{N}-\text{H}\cdots\text{O}=\text{C}$  hydrogen bond is responsible for the formation of infinite chains stretching along the  $a$  axis of the crystal.

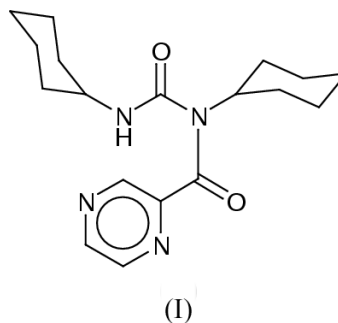
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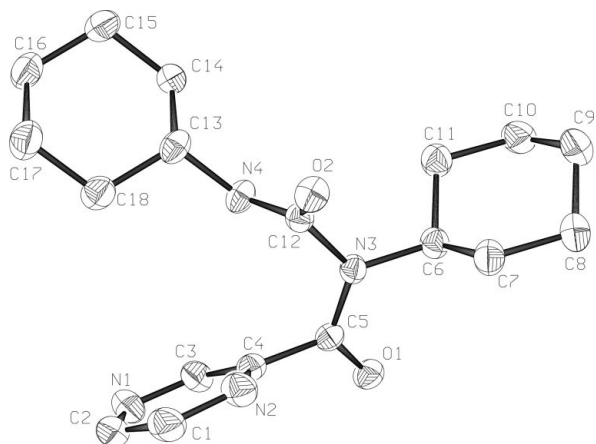
**Comment**

*N,N'*-Dicyclohexyl-*N*-(2-pyrazinoyl)urea, (I), is formed quantitatively from 1,3-dicyclohexylcarbodiimide and pyrazine-2-carboxylic acid in dimethylformamide at room temperature, if small amounts of  $[\text{H}_3\text{Ru}_4(\text{C}_6\text{H}_6)_4\text{OH}]\text{Cl}_2$  are present as catalyst; (I) crystallizes directly from the reaction solution. In the absence of the catalyst, (I) seems to be formed as well, although much more slowly; no crystallization occurs under these conditions. The catalyst,  $[\text{H}_3\text{Ru}_4(\text{C}_6\text{H}_6)_4\text{OH}]\text{Cl}_2$ , is readily available according to the published methods (Chérioux *et al.*, 2001). The precise role of the catalyst in this reaction is not well established, but it can be recovered unchanged after the reaction. Only in the presence of the catalyst, the reaction, being complete after 2 h, gives (I) as an air-stable colorless block-shaped crystalline product.



Compound (I) is analogous to *N,N'*-dicyclohexyl-*N*-nicotinoylurea, (II), which has been synthesized in an obscure reaction from *L*-phenylalanine-*L*-leucine ethyl ester, nicotinic acid, 1-hydroxybenzotriazole and 1,3-dicyclohexylcarbodiimide in dichloromethane (Gallagher *et al.*, 1999).

In the molecular structure of (I) (Fig. 1), the average values of the  $\text{C}-\text{C}-\text{C}$  bond angles in the two cyclohexane rings ( $111.0$  and  $111.2^\circ$ ) and the absolute values of torsion angles (average  $55.9$  and  $55.5^\circ$ ), agree with the theoretically predicted values of  $111.5$  and  $54.7^\circ$  for a chair conformation (Bixon & Lifson, 1967). The bond lengths and angles are in accordance with those in related structures (Orpen *et al.*, 1994).



**Figure 1**  
The molecular structure of (I) showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. H atoms have been omitted for clarity.

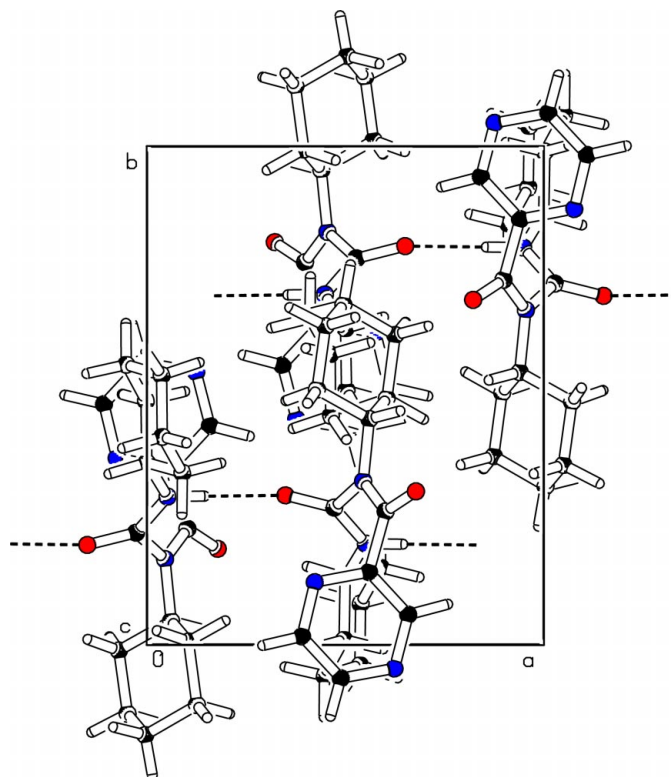
Intermolecular hydrogen bonding involving the N4 amide H atom and carbonyl atom O2 leads to the formation of infinite chains running parallel to the *a* axis of the crystal (Fig. 2). Similar intermolecular (amide)N–H···O=C hydrogen bonding leading to the formation of infinite chains is observed in the structure of *N,N'*-dicyclohexylurea, (III) (Govindasamy & Subramanian, 1997). The H···O distance of N–H···O=C in (I) [2.929 (2) Å] is slightly shorter than that in (III) [2.962 (5) Å], which may be due to the fact that the carbonyl O atom in (III), in contrast to that in (I), participates in two hydrogen bonds involving both ‘active’ H atoms of the next molecule in the chain. In the structure of (II), however, with two independent molecules, there are three symmetry-independent hydrogen bonds of two different kinds, namely (amide)N–H···N(py) and (py)C–H···O=C(amide), which leads to the formation of a totally different, much more complex, hydrogen-bonded system.

## Experimental

*N,N'*-Dicyclohexyl-*N*-(2-pyrazinoyl)urea was formed quantitatively when pyrazine-2-carboxylic acid (1.0 g, 8 mmol), 1,3-dicyclohexylcarbodiimide (2.1 g, 10 mmol) and [H<sub>3</sub>Ru<sub>4</sub>(C<sub>6</sub>H<sub>6</sub>)<sub>4</sub>OH]Cl<sub>2</sub> (7 mg, 0.008 mmol) were dissolved under N<sub>2</sub> in dimethylformamide (20 ml). The red solution was stirred for 2 h at 293 K and filtered. The product crystallized at room temperature from the filtrate. Crystals were collected by filtration. <sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>): 1.22 (*m*, 10 H), 1.55 (*m*, 10 H), 3.55 (quint-*d*, 1 H), 4.23 (quint, 1 H), 5.94 (*d*, 1 H), 8.51 (*dd*, 1 H), 8.66 (*d*, 1 H), 8.98 (*d*, 1 H); IR (KBr, cm<sup>-1</sup>): 3278 (NH, sharp), 1703 (CO, amide) and 1681 (CO, urea); MS (ESI, *m/z*): 330.

### Crystal data

C <sub>18</sub> H <sub>26</sub> N <sub>4</sub> O <sub>2</sub>	Mo Kα radiation
<i>M<sub>r</sub></i> = 330.43	Cell parameters from 8000 reflections
Orthorhombic, <i>P</i> 2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>	$\theta$ = 2.5–26.0°
<i>a</i> = 9.4745 (8) Å	$\mu$ = 0.08 mm <sup>-1</sup>
<i>b</i> = 11.9101 (7) Å	<i>T</i> = 153 (2) K
<i>c</i> = 15.8212 (10) Å	Block, colourless
<i>V</i> = 1785.3 (2) Å <sup>3</sup>	0.50 × 0.40 × 0.35 mm
<i>Z</i> = 4	
<i>D<sub>x</sub></i> = 1.229 Mg m <sup>-3</sup>	



**Figure 2**  
Packing diagram of (I) showing the intermolecular hydrogen bonds in the crystal.

### Data collection

Stoe IPDS diffractometer	<i>R</i> <sub>int</sub> = 0.039
$\varphi$ oscillation scans	$\theta_{\max}$ = 26.0°
Absorption correction: none	<i>h</i> = -11 → 11
14 006 measured reflections	<i>k</i> = -14 → 14
3477 independent reflections	<i>l</i> = -19 → 19
2753 reflections with <i>I</i> > 2σ( <i>I</i> )	

### Refinement

Refinement on <i>F</i> <sup>2</sup>	( $\Delta/\sigma$ ) <sub>max</sub> < 0.001
<i>R</i> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )] = 0.028	$\Delta\rho_{\max}$ = 0.13 e Å <sup>-3</sup>
<i>wR</i> ( <i>F</i> <sup>2</sup> ) = 0.058	$\Delta\rho_{\min}$ = -0.13 e Å <sup>-3</sup>
<i>S</i> = 0.90	Extinction correction: <i>SHELXL97</i>
3477 reflections	Extinction coefficient: 0.0134 (14)
322 parameters	Absolute structure: Flack (1983)
All H-atom parameters refined	Flack parameter = 0.8 (9)
$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2]$	
where $P = (F_o^2 + 2F_c^2)/3$	

**Table 1**

Selected geometric parameters (Å, °).

N3–C5	1.3617 (17)	N4–C13	1.4618 (18)
N3–C6	1.4902 (17)	O1–C5	1.2243 (16)
N3–C12	1.4386 (16)	O2–C12	1.2274 (15)
N4–C12	1.3287 (17)	C4–C5	1.5061 (19)
C5–N3–C12	121.63 (10)	O1–C5–C4	119.11 (12)
C5–N3–C6	120.19 (11)	N3–C5–C4	117.17 (11)
C12–N3–C6	118.17 (11)	O2–C12–N4	125.62 (12)
C12–N4–C13	123.64 (12)	O2–C12–N3	121.00 (11)
O1–C5–N3	123.62 (12)	N4–C12–N3	113.35 (11)

**Table 2**  
Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N4-H4N \cdots O2^i$	0.905 (17)	2.025 (17)	2.9287 (15)	176.1 (14)

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

Image plate experiment parameters: image plate distance 70 mm;  $\varphi$  oscillation scans: 0–200°; step:  $\Delta\varphi = 1.5^\circ$ ;  $2\theta$  range: 3.27–52.1°;  $d_{\max}$ – $d_{\min} = 12.45$ – $0.81$  Å. The H atoms were located from Fourier difference maps and refined isotropically [N4–H4N 0.91 (2) and C–H 0.95–1.04 Å]. The absolute configuration could not be determined because of the lack of anomalous scatterers.

Data collection: *EXPOSE* (Stoe & Cie, 2000); cell refinement: *CELL* (Stoe & Cie, 2000); data reduction: *INTEGRATE* (Stoe & Cie, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON99* (Spek, 1990); software used to prepare material for publication: *SHELXL97*.

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