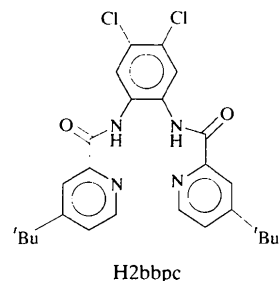


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*Acta Cryst.* (1999). **C55**, 99–100

### *N,N'*-(4,5-Dichloro-*o*-phenylene)bis(4-*tert*-butylpyridine-2-carboxamide)

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(Received 29 June 1998; accepted 1 September 1998)

#### Abstract

In the title compound, C<sub>26</sub>H<sub>28</sub>Cl<sub>2</sub>N<sub>4</sub>O<sub>2</sub>, one of the pyrimidine rings is nearly coplanar with the benzene ring whereas the other forms a dihedral angle of 50.9 (1)°.

#### Comment

The design of new metal catalysts to mimic the oxidative activities of metalloporphyrins has continued to be an active area of research. Previous work revealed that diamide tetradentate compounds, such as 1,2-bis-(pyridine-2-carboxamido)benzene (H2bpb) and 4,5-dichloro-1,2-bis(pyridine-2-carboxamido)benzene (H2bpc), form stable complexes with transition metal ions such as Mn<sup>III</sup> and Cr<sup>III</sup>, and found them to be active catalysts for the oxidation of alkenes and alkanes (Chapman *et al.*, 1981). However, attempts to prepare the related ruthenium complexes have failed. This may be due to the low solubility of the amides and their metal complexes in organic solvents. To circumvent the problem, a soluble derivative of H2bpc [H2bbpc] was prepared by introducing a *tert*-butyl group on the pyridine ring (Ko *et al.*, 1995).

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The mean C—N length in the pyridine rings of H2bbpc is 1.336 (4) Å. The N2—C11 and N3—C16 bond distances are longer than the N2—C10 and N3—C17 distances. The C—C lengths observed in the structure show normal values (Allen *et al.*, 1987). One of the pyridine rings (N1) is nearly coplanar with the benzene ring [dihedral angle 2.42 (9)°] and this coplanarity is stabilized by a C—H···O intramolecular hydrogen bond between C12 and O1. The other pyridine ring forms a dihedral angle of 50.9 (1)° with the benzene ring. The crystal structure is stabilized by van der Waals interactions.

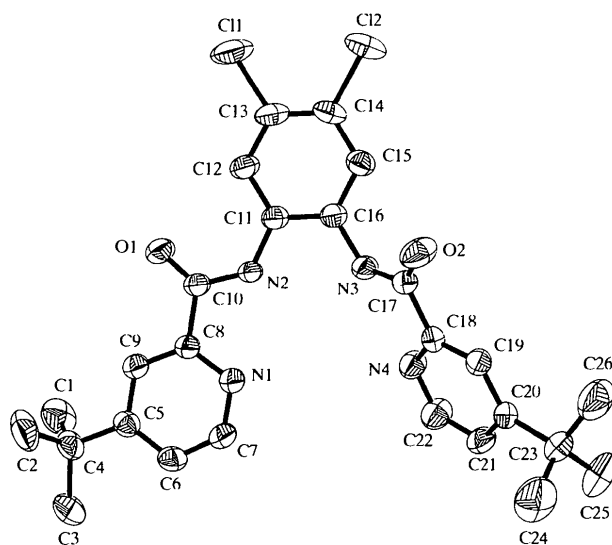


Fig. 1. The structure of the H2bbpc showing 50% probability displacement ellipsoids and the atom-numbering scheme.

#### Experimental

The title compound was synthesized according to the method of Ko *et al.* (1995) and crystallized by slow evaporation of the compound from acetonitrile at room temperature. Only rather small weakly diffracting crystals could be obtained.

#### Crystal data

C<sub>26</sub>H<sub>28</sub>Cl<sub>2</sub>N<sub>4</sub>O<sub>2</sub>  
*M<sub>r</sub>* = 499.42

Mo *K*α radiation  
 $\lambda$  = 0.71073 Å

## Triclinic

$P\bar{1}$   
 $a = 9.3876$  (1) Å  
 $b = 12.4039$  (2) Å  
 $c = 12.7550$  (3) Å  
 $\alpha = 104.807$  (1)°  
 $\beta = 99.431$  (1)°  
 $\gamma = 110.927$  (1)°  
 $V = 1286.54$  (4) Å<sup>3</sup>  
 $Z = 2$   
 $D_x = 1.289$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Cell parameters from 5053 reflections  
 $\theta = 1.72$ – $27.05$ °  
 $\mu = 0.282$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Parallelepiped  
 $0.32 \times 0.22 \times 0.10$  mm  
 Yellow

## Data collection

Siemens SMART CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: empirical (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.915$ ,  $T_{\max} = 0.972$   
 9032 measured reflections

5102 independent reflections  
 3148 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.054$   
 $\theta_{\max} = 27.05$ °  
 $h = -11 \rightarrow 10$   
 $k = -15 \rightarrow 15$   
 $l = 0 \rightarrow 16$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.075$   
 $wR(F^2) = 0.176$   
 $S = 1.074$   
 5102 reflections  
 314 parameters  
 H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.426P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.376$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.273$  e Å<sup>-3</sup>  
 Extinction correction: SHELXTL  
 Extinction coefficient: 0.008 (3)  
 Scattering factors from International Tables for Crystallography (Vol. C)

Data collection: SMART (Siemens, 1996). Cell refinement: SAINT (Siemens, 1996). Data reduction: SAINT. Program(s) used to solve structure: SHELXTL (Sheldrick, 1997). Program(s) used to refine structure: SHELXTL. Molecular graphics: SHELXTL. Software used to prepare material for publication: SHELXTL and PARST (Nardelli, 1995).

The authors would like to thank the Malaysian Government for research grant R&D No. 190-9609-2801. This work was also supported by a grant for the Key Research Project from the State Science and Technology Commission and the National Nature Science Foundation of China. KC thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1233). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1999). **C55**, 100–102

## 1-Isobutyl-2,6,6-trimethyl-4-oxo-4,5,6,7-tetrahydro-3-indoleacetic acid

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(Received 23 February 1998; accepted 30 July 1998)

## Abstract

The title compound (C<sub>17</sub>H<sub>25</sub>NO<sub>3</sub>) is a perhydro-indoleacetic acid which exhibits hypoglycemic activity. The cyclohexyl ring adopts a sofa conformation. The molecule contains an intramolecular O—H···O hydrogen bond.

Table 1. Selected geometric parameters (Å, °)

O1—C10	1.213 (4)	N2—C11	1.401 (4)
O2—C17	1.213 (4)	N3—C17	1.357 (4)
N1—C7	1.333 (4)	N3—C16	1.416 (4)
N1—C8	1.341 (4)	N4—C22	1.331 (4)
N2—C10	1.350 (4)	N4—C18	1.337 (4)
C11—N2—C10—C8	177.3 (3)	C17—N3—C16—C15	51.7 (5)
N1—C8—C10—N2	-1.8 (5)	C16—N3—C17—C18	-178.6 (3)
C10—N2—C11—C12	6.0 (6)	N3—C17—C18—N4	-1.6 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12A···O1	0.93	2.276	2.880 (4)	122
C25—H25A···O1 <sup>i</sup>	0.96	2.501	3.449 (5)	170

Symmetry code: (i)  $1 + x, y, 1 + z$ .

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different  $\varphi$  angle (0, 88 and 180°) for the crystal and each exposure of 60 s covered 0.3° in  $\omega$ . The crystal-to-detector distance was 3 cm and the detector swing angle was -20°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible. The structure was solved by direct methods and refined by full-matrix least-squares techniques. All H atoms were geometrically fixed and allowed to ride on their attached atoms.