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#### Key indicators

Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
R factor = 0.045  
wR factor = 0.135  
Data-to-parameter ratio = 14.3

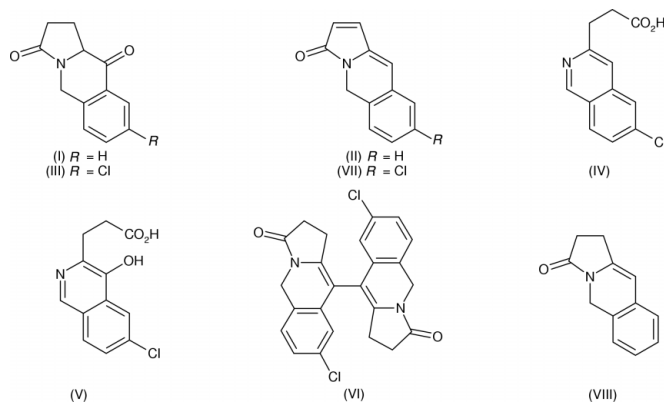
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## 8,8'-Dichloro-1,1',5,5'-tetrahydro-10,10'-bipyrrolo[1,2-*b*]isoquinoline-3,3'(2*H*,2'*H*)-dione

In the course of a study of keto lactams, treatment of 8-chloro-1,10*a*-dihydropyrrolo[1,2-*b*]isoquinoline-3,10(2*H*,5*H*)-dione with concentrated hydrochloric acid was realised. The packing of the resultant dimeric compound,  $\text{C}_{24}\text{H}_{18}\text{N}_2\text{O}_2\text{Cl}_2$ , is governed by the formation of  $\pi$ -stacking interactions.

#### Comment

Cyclized keto lactams of general structure (I) (Rigo *et al.*, 1990) have proved to be of great interest due to the large diversity of rearrangements observed in this family of compounds (Rigo *et al.*, 1991, 1994). In particular, treatment of ketone (I) with polyphosphoric acid led to a dehydration, yielding lactam (II). During attempts to repeat this reaction with ketone (III) and concentrated hydrochloric acid, acid (IV) (19%), acid (V) (31%), dimer (VI) (25%) and diethylenic lactam (VII) (3%) were obtained. NMR spectroscopic measurements easily lead to the structures of (IV), (V) and (VII), and show that compound (VI) seems to be a dimer of the known (Rigo & Kolocouris, 1983) general structure (VIII). The identity of (VI) was confirmed as 8,8'-dichloro-1,1',5,5'-tetrahydro-10,10'-bipyrrolo[1,2-*b*]isoquinoline-3,3'(2*H*,2'*H*)-dione, (VI), by an X-ray analysis of the yellow crystals obtained by slow evaporation of a DMF/benzene solution at room temperature.

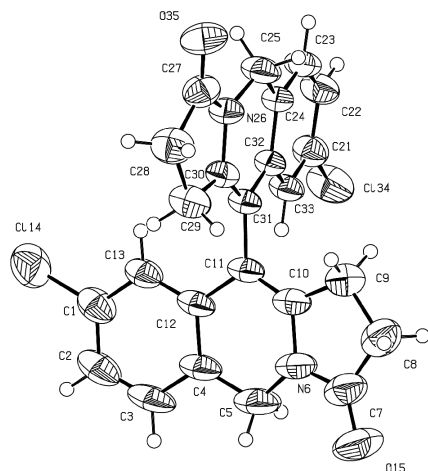


Compound (VI) is composed of two perpendicular tricyclic entities [torsion angle  $\text{C}12-\text{C}11-\text{C}31-\text{C}30 = 95.5(3)^\circ$ ] (Fig. 1). We note the planarity of each tricyclic part of the molecule. The  $sp^2$  hybridization of atoms N6 and N26 is confirmed [the sums of the bond angles around N6 and N26 are  $359.2(2)$  and  $359.4(2)^\circ$ , respectively]. Moreover, the crystal packing is governed by the formation of stacked  $\pi-\pi$  interactions between the aromatic rings (C1-C4/C12/C13) of two neighbouring molecules in the unit cell [(i) and (ii)] (Fig. 2). The distance between the two corresponding centroids is  $3.89(1) \text{ \AA}$ . A similar interaction exists between

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**Figure 1**  
ORTEP (Burnett & Johnson, 1996) representation of compound (VI), with displacement ellipsoids drawn at the 50% probability level.

two other aromatic rings of two other neighbouring molecules [(i) and (iii)] (Fig. 2). In this case, the distance between the two corresponding centroids is 4.04 (1) Å. We found no classical hydrogen bonds.

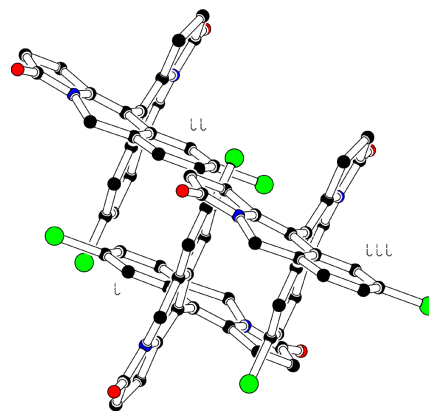
## Experimental

A stirred solution of ketone (III) (1.0 g, mmol) in concentrated hydrochloric acid (10 ml) was refluxed for 36 h. After cooling, the yellow crystals were filtered off, giving (VI), 25%, m.p. > 493 K (after recrystallization from DMF/benzene). <sup>1</sup>H NMR ( $\delta$  p.p.m.): 2.59 (*s*, 8H), 4.92 (*d*,  $J = 16.9$  Hz, 2H), 5.04 (*d*,  $J = 16.9$  Hz, 2H), 6.80 (*d*,  $J = 1.9$  Hz, 2H), 7.04 (*d*,  $J = 8.2$  Hz, 2H), 7.11 (*dd*,  $J = 8.2, 1.9$  Hz, 2H); <sup>13</sup>C NMR ( $\delta$  p.p.m.): 22.5, 28.4, 43.3, 102.4, 121.9, 125.0, 126.4, 127.7, 133.3, 134.0, 141.8, 175.0. The filtrate after removal of crystalline (VI) was extracted with methylene dichloride, giving lactam (VII), 3%, m.p. 481–482 K (recrystallized from CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR ( $\delta$  p.p.m.): 4.93 (*s*, 2H), 6.15 (*s*, 1H), 6.35 (*d*,  $J = 5.6$  Hz, 1H), 7.16 (*d*,  $J = 8.9$  Hz, 1H), 7.17 (*d*,  $J = 5.6$  Hz, 1H), 7.23 (*s*, 1H), 7.26 (*d*,  $J = 8.6$  Hz, 1H); <sup>13</sup>C NMR ( $\delta$  p.p.m.): 42.2, 108.8, 125.0, 127.6, 128.0, 128.4, 128.7, 131.6, 133.3, 133.4, 138.4, 169.1. The aqueous solution was evaporated in part, giving acid (V), 31%, m.p. 458–460 K (recrystallized from H<sub>2</sub>O). <sup>1</sup>H NMR:  $\delta$  2.47 (*t*, 8.8 Hz, 2H), 3.09 (*t*, 8.8 Hz, 2H), 7.38 (*dd*,  $J = 8.8, 2.2$  Hz, 1H), 7.77 (*d*,  $J = 8.8$  Hz, 1H), 8.13 (*d*,  $J = 2.2$  Hz, 1H), 8.22 (*s*, 1H); <sup>13</sup>C NMR:  $\delta$  31.9, 39.9, 124.0, 129.1, 130.0, 131.5, 134.6, 135.7, 136.8, 142.8, 158.0, 186.2. Evaporation of the filtrate gave (IV) hydrochloride, 19%. Treatment of this salt with 1,2-epoxypropane led to acid (IV), m.p. 450–452 K (recrystallized from CH<sub>2</sub>Cl<sub>2</sub>). <sup>1</sup>H NMR ( $\delta$  p.p.m.): 2.58 (*t*,  $J = 7.9$  Hz, 2H), 3.03 (*t*,  $J = 7.9$  Hz, 2H), 7.17 (*s*, 1H), 7.31 (*d*,  $J = 8.5$  Hz, 1H), 7.44 (*s*, 1H), 7.63 (*d*,  $J = 8.5$  Hz, 1H), 8.72 (*s*, 1H); <sup>13</sup>C NMR ( $\delta$  p.p.m.): 31.9, 39.9, 124.1, 129.1, 130.0, 131.5, 134.7, 135.7, 136.8, 142.8, 158.0, 186.2.

### Crystal data

C<sub>24</sub>H<sub>18</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>2</sub>  
 $M_r = 437.30$   
 Monoclinic,  $P2_1/c$   
 $a = 8.1130$  (10) Å  
 $b = 12.0890$  (10) Å  
 $c = 20.181$  (2) Å  
 $\beta = 92.79$  (3)°  
 $V = 1977.0$  (4) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.469$  Mg m<sup>-3</sup>  
 Cu  $K\alpha$  radiation  
 Cell parameters from 24 reflections  
 $\theta = 32$ – $45^\circ$   
 $\mu = 3.16$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Prism, yellow  
 $0.32 \times 0.16 \times 0.10$  mm



**Figure 2**  
Crystal packing, showing the  $\pi$ - $\pi$  stacking interactions [symmetry code: (i)  $x, y, z$ ; (ii)  $-x, 1 - y, -z$ ; (iii)  $1 - x, 2 - y, -z$ ].

### Data collection

Nonius CAD-4 diffractometer	$\theta_{\max} = 71.9^\circ$
$\theta/2\theta$ scans	$h = -10 \rightarrow 2$
Absorption correction: analytical (Alcock, 1970)	$k = 0 \rightarrow 14$
$T_{\min} = 0.432, T_{\max} = 0.743$	$l = -24 \rightarrow 24$
6211 measured reflections	3 standard reflections
3864 independent reflections	every 200 reflections
3055 reflections with $I > 2\sigma(I)$	frequency: 60 min
$R_{\text{int}} = 0.014$	intensity decay: 4%

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 0.791P]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.135$	$(\Delta/\sigma)_{\max} = 0.001$
$S = 1.04$	$\Delta\rho_{\max} = 0.64 \text{ e \AA}^{-3}$
3864 reflections	$\Delta\rho_{\min} = -0.44 \text{ e \AA}^{-3}$
271 parameters	
H-atom parameters constrained	

H atoms were located in a Fourier difference synthesis and were fixed to the corresponding carrier atom.

Data collection: *CAD-4 EXPRESS* (Enraf-Nonius, 1992); cell refinement: *CAD-4 EXPRESS*; data reduction: *PLATON* (Spek, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON*; software used to prepare material for publication: *SHELXL97*.

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