Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

anti-2,19-Diethoxycarbonyl-2,19-diformylamino[3.2.3.2]paracyclophane

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.067
wR factor = 0.179
Data-to-parameter ratio = 20.1

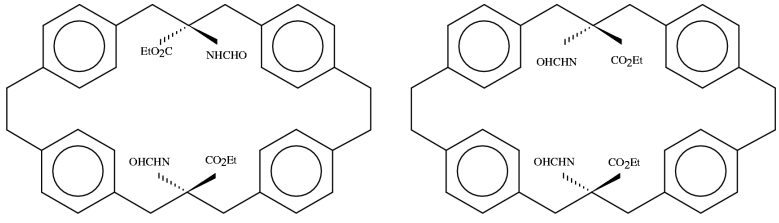
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 7 August 2002
Accepted 27 August 2002
Online 31 August 2002

The title compound, $C_{42}H_{46}N_2O_6$, crystallizes with half a molecule in the asymmetric unit, the molecule being centrosymmetric. The ethyl ester and *N*-formyl side chains attached to the C_α atom of the molecule adopt a *trans* and *cis* configuration, respectively. The crystal structure is stabilized by C—H···O, N—H···O and C—H···π interactions and herring-bone-type packing is observed.

Comment

Macrocyclic molecules act as synthetic receptors in molecular recognition (Keehn & Rosenfeld, 1983). Incorporation of the unusual amino acid Aib (α-aminoisobutyric acid) with a paracyclophane unit resulted in the title compound, (I) (Kotha *et al.*, 2002). This synthesis yielded a mixture of isomers. The crystal structure of the *trans* isomer, (I), is



Trans Isomer (I)

Cis Isomer

reported here. The compound crystallized from a mixture of CH_2Cl_2 and petroleum ether in space group *Pbca*, with one

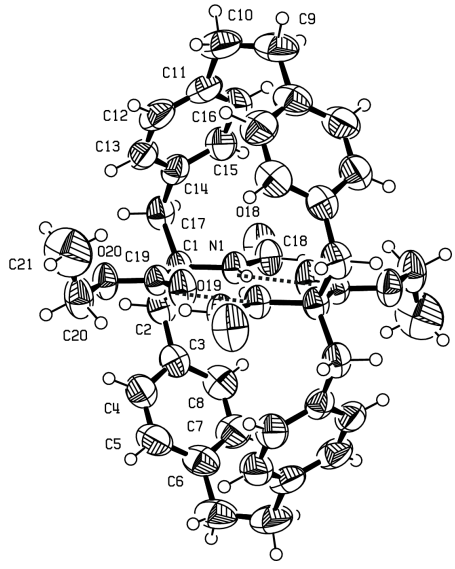


Figure 1
The molecular structure of the title molecule with 50% probability displacement ellipsoids and the atomic numbering scheme.

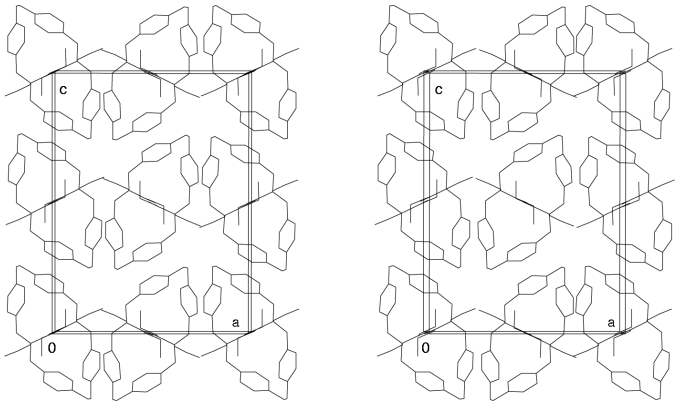


Figure 2
Stereoview of the herring-bone-type packing of the molecules in the *ac* plane.

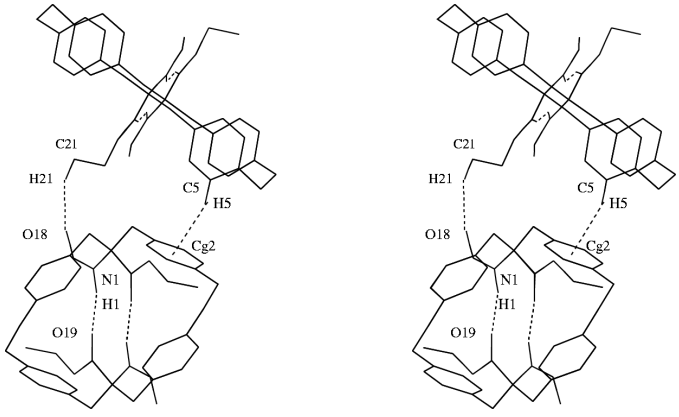


Figure 3
Stereoview of the molecule of (I), showing the N—H···O, C—H···O and C—H···π interactions. For clarity, only H atoms involved in hydrogen bonding are shown.

half molecule in the asymmetric unit, the molecules lying on inversion centres.

An *ORTEP*-3 diagram (Farrugia, 1997) of (I) is shown in Fig. 1. The conformation of the ethyl acetate side chain is *trans* [C1—C19—O20—C20 = 176.4 (2)°], while that of the *N*-formyl side chain is *cis* [C1—N1—C18—O18 = 1.0 (4)°] with respect to the C_α atom.

One half of the molecule is linked to the other half through a pair of transannular N—H···O hydrogen bonds. Herring-bone-type of packing is stabilized by van der Waals forces, and by C—H···O and C—H···π-type intermolecular interactions (Figs. 2 and 3).

Experimental

The title unusual macrocyclic cyclophane-based α-amino acid derivative has been synthesized by coupling of ethyl isocyanoacetate with 1,2-bis(4-bromomethylphenyl)ethane under phase-tranfer-catalysis conditions.

Crystal data

$C_{42}H_{46}N_2O_6$
M_r = 674.81
Orthorhombic, *Pbca*
a = 15.890 (2) Å
b = 11.2944 (16) Å
c = 21.125 (3) Å
V = 3791.1 (9) Å^3
Z = 4
D_x = 1.182 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4538 reflections
θ = 1.9–28.0°
μ = 0.08 mm^{-1}
T = 293 (2) K
Rectangular, colourless
0.54 × 0.45 × 0.45 mm

Data collection

Bruker CCD area detector diffractometer
φ and ω scans
31 175 measured reflections
4538 independent reflections
3019 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 28.0°
$h = -20 \rightarrow 20$
$k = -12 \rightarrow 14$
$l = -27 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.179
S = 1.09
4538 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0764P)^2 + 0.7790P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Table 1
Hydrogen-bonding geometry (Å, °).

*Cg*2 is the centroid of **[please provide ring/plane details]**.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O19^{i}	0.86	2.14	2.9272 (19)	151
C21—H21A···O18^{ii}	0.96	2.42	3.317 (4)	155
C5—H5···$Cg2^{iii}$	0.93	3.07	3.921 (1)	153

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

The H atoms were fixed geometrically at idealized positions and refined as riding on the heavier atoms to which they were bonded.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

The work was supported by Council of Scientific and Industrial Research, India, and the DST.

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