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# Diethyl 4,8-dioxo-2,6-di-*p*-chlorophenyl-1,3,5,7-tetrahydro-2,3a,4a,6,7a,8a-hexaazacyclopenta-[*def*]fluorene-8b,8c-dicarboxylate

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**Key indicators**

Single-crystal X-ray study
*T* = 292 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
*R* factor = 0.041
*wR* factor = 0.112
Data-to-parameter ratio = 12.6

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

In the title compound, $C_{28}H_{32}Cl_2N_6O_6$, the dihedral angle between the two adjacent five-membered rings in the glycoluril unit is 70.18 (9)° and molecules are connected mainly by C—H···O intermolecular interactions.

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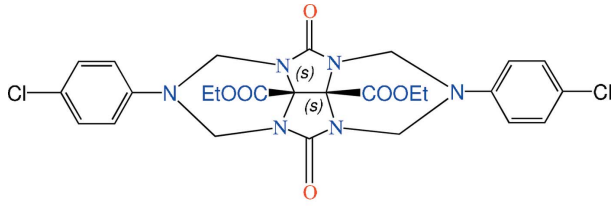
## Comment

Since Mock and co-workers characterized the chemical nature and the structure of cucurbit[6]uril (CB[6]; Freeman *et al.*, 1981), many receptors based on glycoluril have been reported (Rowan *et al.*, 1999; Hof *et al.*, 2002; Lee *et al.*, 2003; Lagona *et al.*, 2003). We report here the crystal structure of the title compound, (I) (Fig. 1), a new receptor based on glycoluril. Selected bond lengths and angles are listed in Table 1. The crystal packing is mainly dictated by intermolecular C—H···O interactions (Table 2 and Fig. 2). The dihedral angle between the two adjacent five-membered rings in the glycoluril unit is 70.18 (9)°.




(I)

## Experimental

*p*-Chloroaniline (2.54 g, 20 mmol) and formaldehyde (4.8 g, 80 mmol) were added to a stirred solution of diethoxycarbonyl glycoluril (2.86 g, 10 mmol) in dimethylformamide (50 ml) under a nitrogen atmosphere. The mixture was stirred overnight and the solvent was

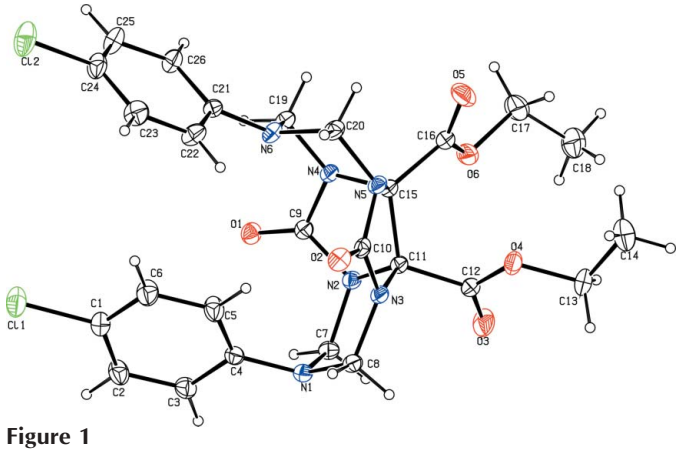



**Figure 1**
View of (I), showing the atom-labeling scheme, with displacement ellipsoids drawn at the 50% probability level.

evaporated to dryness and purified by column chromatography (hexane–EtOAc = 4:1 *v*/*v*) to obtain the title compound (yield 2.36 g, 40%) as a white solid. Crystals suitable for X-ray diffraction were grown by slow evaporation of a $CH_2Cl_2$–MeOH (2:1) solution under ambient conditions.

*Crystal data*

| | |
|---|---|
| $C_{26}H_{26}Cl_2N_6O_6$ | $Z = 2$ |
| $M_r = 589.43$ | $D_x = 1.444$ Mg m$^{-3}$ |
| Triclinic, $P\overline{1}$ | Mo $K\alpha$ radiation |
| $a = 8.300$ (2) Å | Cell parameters from 4340 reflections |
| $b = 12.698$ (3) Å | $\theta = 2.3$–$25.9°$ |
| $c = 14.428$ (4) Å | $\mu = 0.29$ mm$^{-1}$ |
| $\alpha = 103.836$ (17)° | $T = 292$ (2) K |
| $\beta = 103.21$ (2)° | Block, colorless |
| $\gamma = 105.087$ (19)° | 0.30 × 0.30 × 0.20 mm |
| $V = 1355.2$ (6) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 4737 independent reflections |
| | 3709 reflections with $I > 2\sigma$ |
| $\varphi$ and $\omega$ scans | $R_{int} = 0.052$ |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997) | $\theta_{max} = 25.0°$ |
| | $h = -9 \rightarrow 9$ |
| $T_{min} = 0.917$, $T_{max} = 0.944$ | $k = -15 \rightarrow 15$ |
| 10962 measured reflections | $l = -17 \rightarrow 17$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0603P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.041$ | |
| $wR(F^2) = 0.113$ | $(\Delta/\sigma)_{max} = 0.001$ |
| $S = 1.08$ | $\Delta\rho_{max} = 0.22$ e Å$^{-3}$ |
| 4737 reflections | $\Delta\rho_{min} = -0.28$ e Å$^{-3}$ |
| 375 parameters | Extinction correction: *SHELXL97* |
| H-atom parameters constrained | Extinction coefficient: 0.0066 (17) |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| C7—N2 | 1.477 (2) | C11—N2 | 1.446 (2) |
| C8—N3 | 1.478 (2) | C11—N3 | 1.457 (2) |
| C9—N4 | 1.387 (2) | C15—N5 | 1.456 (2) |
| C9—N2 | 1.396 (2) | C15—N4 | 1.458 (2) |
| C10—N3 | 1.368 (2) | C19—N4 | 1.459 (2) |
| C10—N5 | 1.390 (2) | C20—N5 | 1.462 (2) |
| | | | |
| N2—C11—N3 | 111.66 (13) | N5—C15—N4 | 112.95 (13) |
| | | | |
| N1—C7—N2—C11 | 53.0 (2) | N6—C19—N4—C15 | −49.18 (19) |
| N1—C8—N3—C11 | −46.0 (2) | N6—C20—N5—C15 | 52.37 (19) |

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5···O2 | 0.93 | 2.40 | 3.311 (2) | 167 |
| C20—H20*A*···O5$^{i}$ | 0.97 | 2.57 | 3.245 (2) | 127 |
| C17—H17*A*···O1$^{ii}$ | 0.97 | 2.55 | 3.278 (3) | 132 |
| C8—H8*A*···O2$^{iii}$ | 0.97 | 2.56 | 3.361 (2) | 140 |
| C3—H3···O2$^{iv}$ | 0.93 | 2.58 | 3.404 (3) | 148 |

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $-x+2, -y+1, -z+2$; (iii) $-x+1, -y, -z+2$; (iv) $x+1, y, z$.

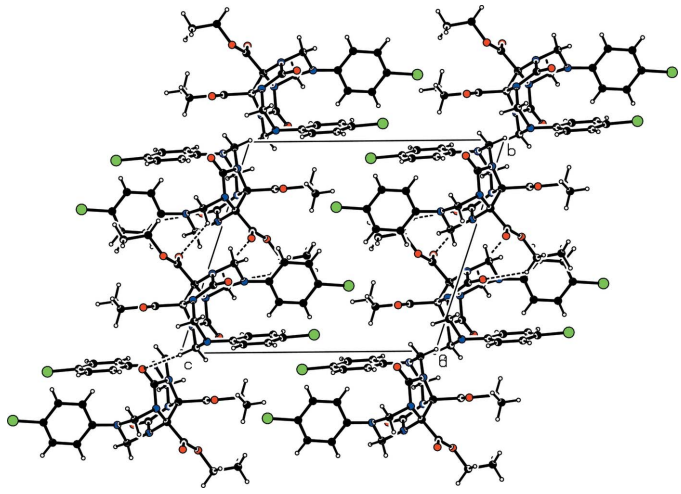



**Figure 2**
Packing diagram of compound (I).

One of the ethyl groups (C18) was found to be disordered over two orientations. The occupancies of the disordered positions C18/C18′ refined to 0.58 (2):0.42 (2). All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding, allowing for free rotation of the methyl groups. The constraint $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C) was applied.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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