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Diethyl 2,2′-(ethylenediimino)di(cyclopentenecarboxylate)

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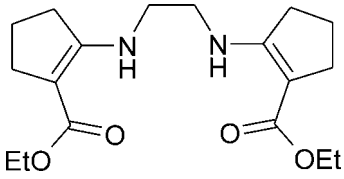
Key indicators: single-crystal X-ray study; T = 183 K; mean σ(C–C) = 0.003 Å; R factor = 0.062; wR factor = 0.178; data-to-parameter ratio = 17.5.

In the title compound, $C_{18}H_{28}N_2O_4$, the molecule displays a partially eclipsed conformation with an N—C—C—N torsion angle of 67.93 **(s.u.?)**° linking the two ethoxycarbonylcyclopentenyl groups. This conformation is different from the staggered conformation observed in the related *N*,*N*′-ethylenediamine with a cycloalkene residue. Molecules are linked by two different N—H···O hydrogen bonds, generating sheets parallel to the (110) plane.

Related literature

For related structure, see: Fernández-G *et al.* (1993); Banares *et al.* (1989); Allen (2002).



Experimental

Crystal data

$C_{18}H_{28}N_2O_4$
M_r = 336.42
Triclinic, $P\overline{1}$
a = 7.2622 (4) Å
b = 10.5603 (9) Å
c = 11.9036 (9) Å
α = 92.908 (4)°
β = 95.723 (5)°
γ = 97.278 (5)°
V = 899.17 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 183 (2) K
0.03 × 0.03 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
6086 measured reflections
3981 independent reflections
2742 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.178
S = 1.01
3981 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N1···O3	0.85 (3)	2.23 (3)	2.833 (2)	128 (2)
N1—H1N1···O3^{i}	0.85 (3)	2.52 (3)	3.175 (3)	135 (2)
N2—H1N2···O1	0.85 (3)	2.23 (3)	2.851 (2)	130 (2)
N2—H1N2···O1^{ii}	0.85 (3)	2.53 (3)	3.195 (2)	136 (2)

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1990); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2230).

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supplementary materials

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Diethyl 2,2'-(ethylenediimino)di(cyclopentenecarboxylate)

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Comment

There is only one crystal structure reported in the literature containing *N*,*N*'-ethylenediamine ligand with a cycloalkene residue, as revealed by a search in the Version 5.27 of the Cambridge Structural Database (Allen, 2002). In this case, the ligand shows a perfectly staggered conformation in the solid state, with a torsion angle of 180° (Fernández-G *et al.*, 1993). The structure of the title compound is shown in Figure 1. Molecules are linked by two diffenerent N—H···O hydrogen bonds involving most of potential donors, generating sheets parallel to the (110) plane, as shown in Fig. 2. The coordination geometry around the N atoms is perhaps best described as trigonal planar, with C11–N1–H1N1, C1–N1–H1N1 and C11–N1–C1 angles of 118 (2), 115 (2) and 125.5 (2)°, and with C3–N2–H1N2, C2–N2–H1N2 and C3–N2–C2 angles of 116 (2), 117 (2) and 125.2 (2)°, respectively. In contrast to the cyclohexylen subsituted *N*,*N*'-ethylenediamine ligand (Fernández-G *et al.*, 1993), (I) has no centre of symmetry and with a N1–C1–C2–N2 dihedral of −67.93° it displays a partially eclipsed conformation.

Experimental

The synthesis was carried out according to a published procedure (Banares *et al.*, 1989). Ethyl-α-ketocyclopentylcarboxylate (two equivalents) was added to a solution of ethylenediamine (one equivalent) in dry ethanol. After standing for several hours, the ethanol was partially removed until precipitation commenced. The title compound was isolated in good yield. Crystals suitable for X-ray analysis were obtained directly from the reaction mixture.

Refinement

The hydrogen atoms of the two amine groups at N1 and N2 were located by difference Fourier synthesis and refined isotropically. All other hydrogen atoms wereintroduced in calculated positions and treated as riding on their parent atoms with C—H= 0.98Å (methyl) or 0.99Å (methylene) and with U_{iso}(H) = 1.5U_{eq}(C).

Figures

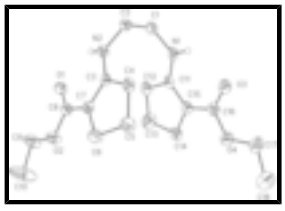

Fig. 1. Molecular structure of **1**. Displacement ellipsoids are drawn at the 40% probability level.

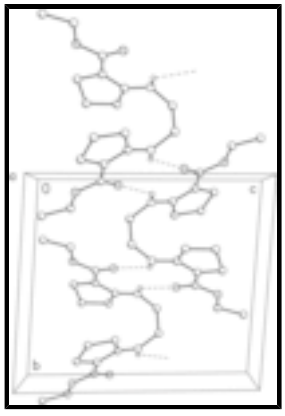

Fig. 2. The packing of (I), viewed down the *c* axis, showing one layer of molecules connected by N—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

Diethyl 2,2'-(ethylenediimino)di(cyclopentenecarboxylate)

Crystal data

$C_{18}H_{28}N_2O_4$	V = 899.17 (11) Å^3
M_r = 336.42	Z = 2
Triclinic, $P\bar{1}$	F_{000} = 364
Hall symbol: -P1	D_x = 1.243 Mg m^{-3}
a = 7.2622 (4) Å	Mo $K\alpha$ radiation λ = 0.71073 Å
b = 10.5603 (9) Å	μ = 0.09 mm^{-1}
c = 11.9036 (9) Å	T = 183 (2) K
α = 92.908 (4)º	Prism, colourless
β = 95.723 (5)º	0.03 × 0.03 × 0.02 mm
γ = 97.278 (5)º	

Data collection

Nonius KappaCCD diffractometer	2742 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.026
Monochromator: graphite	θ_{max} = 27.4º
T = 183(2) K	θ_{min} = 2.5º
φ and ω scans	h = −9→9
Absorption correction: none	k = −13→12
6086 measured reflections	l = −15→15
3981 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.062	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.178$	$w = 1/[\sigma^2(F_o^2) + (0.0888P)^2 + 0.3965P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3981 reflections	$\Delta\rho_{max} = 0.63$ e Å^{-3}
227 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.0548 (2)	0.54795 (15)	0.64357 (12)	0.0394 (4)
O2	0.1410 (2)	0.58230 (16)	0.83181 (12)	0.0427 (4)
O3	0.4505 (2)	−0.04109 (16)	0.63702 (14)	0.0432 (4)
O4	0.3582 (2)	−0.07723 (17)	0.80960 (14)	0.0455 (4)
N1	0.2322 (3)	0.11600 (17)	0.50903 (16)	0.0350 (4)
N2	0.2955 (3)	0.40689 (17)	0.52720 (14)	0.0312 (4)
C1	0.2004 (3)	0.1990 (2)	0.41747 (18)	0.0359 (5)
H1A	0.0697	0.2174	0.4128	0.043*
H1B	0.2180	0.1539	0.3451	0.043*
C2	0.3316 (3)	0.3252 (2)	0.43306 (17)	0.0333 (5)
H2A	0.4621	0.3065	0.4451	0.040*
H2B	0.3183	0.3713	0.3629	0.040*
C3	0.3759 (3)	0.40687 (18)	0.63412 (17)	0.0289 (4)
C4	0.5496 (3)	0.3452 (2)	0.66374 (18)	0.0346 (5)
H4A	0.6602	0.3943	0.6368	0.042*
H4B	0.5350	0.2562	0.6305	0.042*
C5	0.5669 (4)	0.3486 (3)	0.7932 (2)	0.0467 (6)
H5A	0.6994	0.3691	0.8246	0.056*
H5B	0.5168	0.2646	0.8187	0.056*
C6	0.4537 (3)	0.4526 (2)	0.83228 (19)	0.0405 (5)
H6A	0.3841	0.4256	0.8963	0.049*
H6B	0.5356	0.5339	0.8553	0.049*
C7	0.3224 (3)	0.46606 (19)	0.72838 (17)	0.0309 (5)
C8	0.1630 (3)	0.5333 (2)	0.72661 (18)	0.0327 (5)
C9	−0.0083 (4)	0.6581 (3)	0.8407 (2)	0.0553 (7)

H9A	−0.1290	0.6082	0.8098	0.066*
H9B	0.0124	0.7355	0.7977	0.066*
C10	−0.0100 (6)	0.6945 (5)	0.9631 (3)	0.1147 (18)
H10A	−0.1145	0.7429	0.9731	0.172*
H10B	0.1076	0.7476	0.9916	0.172*
H10C	−0.0244	0.6171	1.0052	0.172*
C11	0.1436 (3)	0.11193 (19)	0.60260 (18)	0.0321 (5)
C12	−0.0269 (3)	0.1780 (2)	0.6155 (2)	0.0382 (5)
H12A	−0.1329	0.1401	0.5602	0.046*
H12B	−0.0001	0.2706	0.6046	0.046*
C13	−0.0699 (4)	0.1559 (3)	0.7355 (3)	0.0665 (9)
H13A	−0.2024	0.1194	0.7355	0.080*
H13B	−0.0483	0.2380	0.7818	0.080*
C14	0.0571 (3)	0.0636 (2)	0.7851 (2)	0.0405 (5)
H14A	0.1269	0.1001	0.8576	0.049*
H14B	−0.0158	−0.0192	0.7979	0.049*
C15	0.1879 (3)	0.0473 (2)	0.69595 (18)	0.0337 (5)
C16	0.3416 (3)	−0.0251 (2)	0.70755 (18)	0.0351 (5)
C17	0.5103 (4)	−0.1500 (3)	0.8320 (2)	0.0519 (7)
H17A	0.6303	−0.0979	0.8223	0.062*
H17B	0.4946	−0.2272	0.7794	0.062*
C18	0.5081 (6)	−0.1870 (4)	0.9510 (3)	0.0802 (10)
H18A	0.6131	−0.2344	0.9708	0.120*
H18B	0.3906	−0.2411	0.9587	0.120*
H18C	0.5192	−0.1099	1.0019	0.120*
H1N1	0.328 (4)	0.078 (3)	0.507 (2)	0.047 (7)*
H1N2	0.195 (4)	0.440 (3)	0.518 (2)	0.049 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0418 (9)	0.0456 (9)	0.0326 (8)	0.0212 (7)	−0.0044 (7)	−0.0017 (7)
O2	0.0425 (9)	0.0581 (10)	0.0312 (8)	0.0258 (8)	0.0008 (7)	−0.0043 (7)
O3	0.0447 (9)	0.0448 (9)	0.0456 (9)	0.0215 (7)	0.0105 (7)	0.0078 (7)
O4	0.0471 (9)	0.0554 (10)	0.0398 (9)	0.0243 (8)	0.0076 (7)	0.0094 (8)
N1	0.0373 (10)	0.0286 (9)	0.0410 (10)	0.0133 (8)	0.0039 (8)	−0.0003 (8)
N2	0.0338 (9)	0.0308 (9)	0.0303 (9)	0.0133 (8)	0.0005 (7)	−0.0015 (7)
C1	0.0427 (12)	0.0332 (11)	0.0321 (11)	0.0131 (9)	−0.0027 (9)	−0.0031 (9)
C2	0.0391 (11)	0.0331 (11)	0.0294 (10)	0.0126 (9)	0.0029 (9)	0.0000 (8)
C3	0.0294 (10)	0.0238 (9)	0.0336 (11)	0.0063 (8)	−0.0002 (8)	0.0028 (8)
C4	0.0330 (11)	0.0327 (11)	0.0390 (12)	0.0125 (9)	−0.0011 (9)	0.0000 (9)
C5	0.0507 (14)	0.0527 (14)	0.0392 (13)	0.0273 (12)	−0.0071 (11)	−0.0015 (11)
C6	0.0400 (12)	0.0476 (13)	0.0352 (12)	0.0188 (10)	−0.0033 (9)	−0.0021 (10)
C7	0.0332 (10)	0.0295 (10)	0.0302 (10)	0.0087 (8)	−0.0015 (8)	0.0007 (8)
C8	0.0355 (11)	0.0327 (11)	0.0304 (10)	0.0083 (9)	0.0022 (9)	−0.0005 (8)
C9	0.0505 (15)	0.081 (2)	0.0395 (13)	0.0388 (14)	0.0015 (11)	−0.0127 (13)
C10	0.104 (3)	0.204 (5)	0.0504 (19)	0.099 (3)	0.0024 (18)	−0.032 (2)
C11	0.0314 (10)	0.0230 (9)	0.0409 (12)	0.0053 (8)	0.0016 (9)	−0.0069 (8)

C12	0.0336 (11)	0.0303 (11)	0.0514 (14)	0.0100 (9)	0.0026 (10)	−0.0028 (10)
C13	0.0704 (18)	0.0708 (19)	0.075 (2)	0.0453 (16)	0.0351 (16)	0.0245 (16)
C14	0.0396 (12)	0.0420 (13)	0.0413 (13)	0.0120 (10)	0.0057 (10)	−0.0035 (10)
C15	0.0326 (10)	0.0312 (11)	0.0376 (11)	0.0073 (9)	0.0039 (9)	−0.0033 (9)
C16	0.0356 (11)	0.0342 (11)	0.0369 (12)	0.0105 (9)	0.0037 (9)	0.0008 (9)
C17	0.0528 (15)	0.0580 (16)	0.0511 (15)	0.0280 (13)	0.0047 (12)	0.0150 (12)
C18	0.096 (3)	0.081 (2)	0.067 (2)	0.028 (2)	−0.0014 (18)	0.0152 (18)

Geometric parameters (Å, °)

O1—C8	1.229 (2)	C6—H6B	0.9900
O2—C8	1.362 (3)	C7—C8	1.432 (3)
O2—C9	1.436 (3)	C9—C10	1.491 (4)
O3—C16	1.229 (3)	C9—H9A	0.9900
O4—C16	1.360 (3)	C9—H9B	0.9900
O4—C17	1.435 (3)	C10—H10A	0.9800
N1—C11	1.340 (3)	C10—H10B	0.9800
N1—C1	1.452 (3)	C10—H10C	0.9800
N1—H1N1	0.85 (3)	C11—C15	1.366 (3)
N2—C3	1.346 (3)	C11—C12	1.514 (3)
N2—C2	1.445 (3)	C12—C13	1.515 (4)
N2—H1N2	0.85 (3)	C12—H12A	0.9900
C1—C2	1.528 (3)	C12—H12B	0.9900
C1—H1A	0.9900	C13—C14	1.526 (4)
C1—H1B	0.9900	C13—H13A	0.9900
C2—H2A	0.9900	C13—H13B	0.9900
C2—H2B	0.9900	C14—C15	1.511 (3)
C3—C7	1.371 (3)	C14—H14A	0.9900
C3—C4	1.510 (3)	C14—H14B	0.9900
C4—C5	1.531 (3)	C15—C16	1.430 (3)
C4—H4A	0.9900	C17—C18	1.490 (4)
C4—H4B	0.9900	C17—H17A	0.9900
C5—C6	1.534 (3)	C17—H17B	0.9900
C5—H5A	0.9900	C18—H18A	0.9800
C5—H5B	0.9900	C18—H18B	0.9800
C6—C7	1.509 (3)	C18—H18C	0.9800
C6—H6A	0.9900		
C8—O2—C9	117.05 (17)	C10—C9—H9A	110.3
C16—O4—C17	117.03 (18)	O2—C9—H9B	110.3
C11—N1—C1	125.48 (19)	C10—C9—H9B	110.3
C11—N1—H1N1	118.2 (18)	H9A—C9—H9B	108.6
C1—N1—H1N1	115.3 (18)	C9—C10—H10A	109.5
C3—N2—C2	125.21 (18)	C9—C10—H10B	109.5
C3—N2—H1N2	116.6 (18)	H10A—C10—H10B	109.5
C2—N2—H1N2	115.6 (18)	C9—C10—H10C	109.5
N1—C1—C2	112.60 (17)	H10A—C10—H10C	109.5
N1—C1—H1A	109.1	H10B—C10—H10C	109.5
C2—C1—H1A	109.1	N1—C11—C15	126.27 (19)
N1—C1—H1B	109.1	N1—C11—C12	122.7 (2)

C2—C1—H1B	109.1	C15—C11—C12	111.01 (19)
H1A—C1—H1B	107.8	C11—C12—C13	104.29 (19)
N2—C2—C1	113.00 (18)	C11—C12—H12A	110.9
N2—C2—H2A	109.0	C13—C12—H12A	110.9
C1—C2—H2A	109.0	C11—C12—H12B	110.9
N2—C2—H2B	109.0	C13—C12—H12B	110.9
C1—C2—H2B	109.0	H12A—C12—H12B	108.9
H2A—C2—H2B	107.8	C12—C13—C14	108.6 (2)
N2—C3—C7	126.52 (19)	C12—C13—H13A	110.0
N2—C3—C4	122.58 (18)	C14—C13—H13A	110.0
C7—C3—C4	110.81 (18)	C12—C13—H13B	110.0
C3—C4—C5	103.39 (17)	C14—C13—H13B	110.0
C3—C4—H4A	111.1	H13A—C13—H13B	108.4
C5—C4—H4A	111.1	C15—C14—C13	103.8 (2)
C3—C4—H4B	111.1	C15—C14—H14A	111.0
C5—C4—H4B	111.1	C13—C14—H14A	111.0
H4A—C4—H4B	109.0	C15—C14—H14B	111.0
C4—C5—C6	106.65 (18)	C13—C14—H14B	111.0
C4—C5—H5A	110.4	H14A—C14—H14B	109.0
C6—C5—H5A	110.4	C11—C15—C16	123.6 (2)
C4—C5—H5B	110.4	C11—C15—C14	111.79 (19)
C6—C5—H5B	110.4	C16—C15—C14	124.6 (2)
H5A—C5—H5B	108.6	O3—C16—O4	121.46 (19)
C7—C6—C5	103.00 (18)	O3—C16—C15	126.6 (2)
C7—C6—H6A	111.2	O4—C16—C15	111.94 (18)
C5—C6—H6A	111.2	O4—C17—C18	106.8 (2)
C7—C6—H6B	111.2	O4—C17—H17A	110.4
C5—C6—H6B	111.2	C18—C17—H17A	110.4
H6A—C6—H6B	109.1	O4—C17—H17B	110.4
C3—C7—C8	123.61 (19)	C18—C17—H17B	110.4
C3—C7—C6	111.42 (18)	H17A—C17—H17B	108.6
C8—C7—C6	124.96 (19)	C17—C18—H18A	109.5
O1—C8—O2	121.69 (19)	C17—C18—H18B	109.5
O1—C8—C7	126.8 (2)	H18A—C18—H18B	109.5
O2—C8—C7	111.52 (18)	C17—C18—H18C	109.5
O2—C9—C10	107.0 (2)	H18A—C18—H18C	109.5
O2—C9—H9A	110.3	H18B—C18—H18C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O3	0.85 (3)	2.23 (3)	2.833 (2)	128 (2)
N1—H1N1···O3^{i}	0.85 (3)	2.52 (3)	3.175 (3)	135 (2)
N2—H1N2···O1	0.85 (3)	2.23 (3)	2.851 (2)	130 (2)
N2—H1N2···O1^{ii}	0.85 (3)	2.53 (3)	3.195 (2)	136 (2)

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) −*x*, −*y*+1, −*z*+1.

Fig. 1

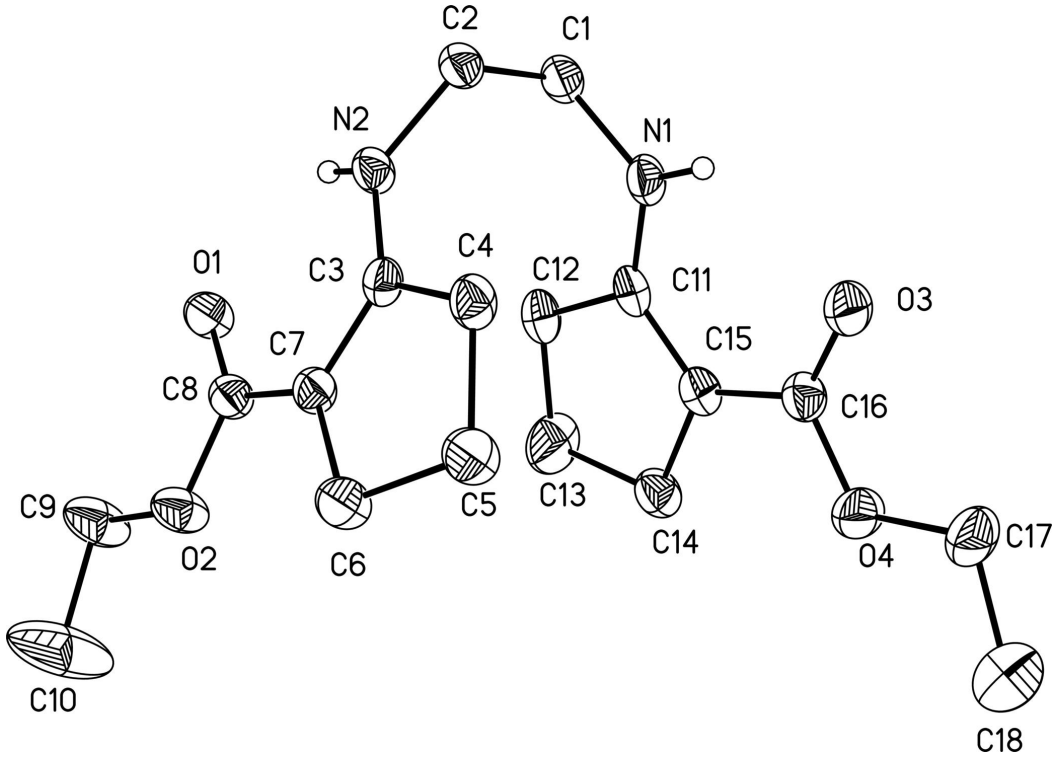
C2
C1
N2
N1
O1
C3
C4
C12
C11
O3
C7
C15
C8
C16
C9
O2
C6
C5
C13
C14
O4
C17
C10
C18

Fig. 2