l'Armement (DGA/DRET, France) for support of this work.

Lists of structure factors, anisotropic thermal parameters, interatomic and intermolecular contacts, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55946 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1015]

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Structure of *cis*-4-Cyclohexene-1,2-dicarboxylic Acid

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Abstract

The molecules are connected by two *inter*molecular hydrogen bonds [O· · ·O distances 2.656 (2) and 2.641 (2) Å] forming infinite chains along [100]. No *intra*molecular hydrogen bond is present between the two neighbouring carboxylic groups. This can be explained by geometric considerations.

Comment

There exist several examples of dicarboxylic acids (or their acid salts) where short intramolecular hydrogen bonds are formed between adjacent carboxylic groups. The reasons for the occurrence or non-occurrence of the intramolecular hydrogen bond seem to be partly of geometric and partly of chemical nature (Küppers & Jessen, 1993). Since the title compound is a potential example where an intramolecular hydrogen bond might be found, an X-ray structure analysis was undertaken.

The atomic packing within the unit cell is shown by a stereoscopic *ORTEP*II plot (Johnson, 1976) in Fig. 1. The molecules do *not* form an *intra*molecular hydrogen bond. Instead, the molecules are interconnected by *inter*molecular hydrogen bonds of the 'cyclic dimer type' (Leiserowitz, 1976). Each of the two carboxylic groups of a molecule is linked to an equivalent (through inversion centre $\bar{1}$) carboxylic group of an adjacent molecule. The two intermolecular hydrogen bonds have lengths 2.656 (2) Å (O1—H7· · ·O2) and 2.641 (2) Å (O3· · ·H8—O4). Thus, the molecules build infinite zigzag chains, which extend along the [100] direction. Because of the double bond between C4 and C5, the atoms C3, C4, C5 and C6 roughly define a plane. The maximum deviation from the mean plane is 0.01 Å. As expected, the ring has a half-chair conformation. According to the *cis* configuration, one carboxylic group (C7, O1, O2) is in equatorial position [angle between the C7, O1, O2 plane and the mean plane defined by C3 to C6 is 6.6 (2)°]. The other carboxylic group (C8, O3, O4) is in an axial position; the respective angle is 85.0 (3)°. The torsion angle C7—C1—C2—C8 is 65.2 (2)°.

The fact that no intramolecular hydrogen bond is formed in the present compound can be explained through geometric considerations. Küppers & Jessen (1993) showed for several examples of dicarboxylic acids that the intramolecular hydrogen bond is not formed if the O· · ·O distance between neighbouring —COOH groups (in unstrained molecules with properly rotated —COOH groups) is shorter than approximately 2.2 Å. In the range 2.2-2.4 Å, the molecules turn out to be able to adjust themselves by internal distortion of intramolecular angles, yielding widened O· · ·O distances of about 2.4 Å. The molecules considered in the paper mentioned above are such that the four C atoms, which correspond to the present atoms C7, C1, C2 and C8, are coplanar. This is not the case in the molecule of this study. One can roughly estimate, however, the theoretical distance of neighbouring O atoms in a quasi-planar configuration if the COO groups are rotated around the C—C bond until two confronting O atoms lie within a plane defined by C7, C8 and the midpoint between C1 and C2. Assuming averaged distances

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and angles as used by Küppers & Jessen (1993), an O· · ·O distance of 1.93 Å results. This is, from the experiences mentioned above, too short to build an intramolecular hydrogen bond.

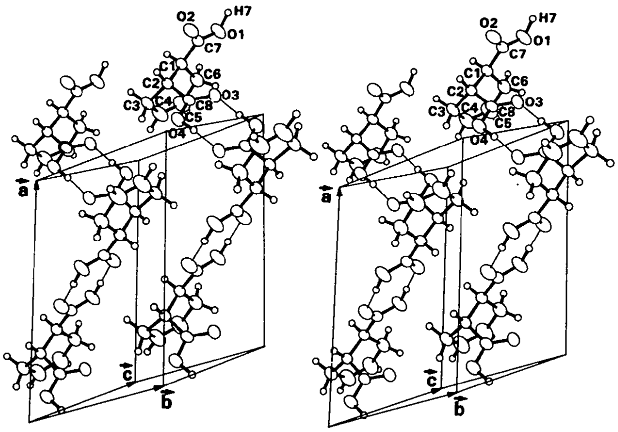

Fig. 1. Stereoscopic view of the crystal structure. Thermal ellipsoids are drawn at 50% probability levels. H atoms have fixed radii. Thin lines indicate hydrogen bonds.

Experimental

Crystal data

$C_8H_{10}O_4$
M_r = 170.16
Triclinic
$P\bar{1}$
a = 10.897 (1) Å
b = 6.941 (1) Å
c = 6.345 (1) Å
α = 64.28 (1)°
β = 74.32 (1)°
γ = 74.81 (1)°
V = 410.47 (4) $Å^3$
Z = 2

D_x = 1.377 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 64 reflections
θ = 3.32–31.16°
μ = 0.10 mm^{-1}
T = 293 K
0.49 × 0.42 × 0.17 mm
Colourless
Crystal source: aqueous solution

Data collection

Philips PW1100 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2389 measured reflections
2389 independent reflections
2186 observed reflections $[|F|^2 > 0]$

θ_{max} = 30°
h = −14 → 15
k = −8 → 9
l = 0 → 8
3 standard reflections frequency: 60 min intensity variation: none

Refinement

Refinement on F
Final R = 0.066
wR = 0.049
2186 reflections
151 parameters
All H-atom parameters refined
$w = 1/\sigma^2$

$(\Delta/\sigma)_{max}$ = 0.054
$\Delta\rho_{max}$ = 0.25 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.25 e $Å^{-3}$
Extinction correction: $f = F(1-gF^2/\sin\theta)$ (*SHELX*76)
Extinction coefficient: g = 0.58 (2) × 10^{-5}

Data collection: Philips PW1100. Program(s) used to solve structure: *MITHRIL* (Gilmore, 1983). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: ORFFE (Busing, Martin & Levy, 1964); *PLATON* (Spek, 1982). Atomic scattering factors from Cromer & Mann (1968) and Cromer & Liberman (1970) for C and O; from Stewart, Davidson & Simpson (1965) for H atoms.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* ($Å^2$)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
C1	0.6579 (2)	0.4326 (3)	0.2869 (3)	0.0336 (5)
C2	0.7409 (2)	0.4127 (3)	0.4579 (3)	0.0315 (5)
C3	0.7855 (2)	0.6290 (3)	0.3837 (3)	0.0413 (6)
C4	0.8301 (2)	0.7293 (3)	0.1250 (4)	0.0599 (8)
C5	0.8070 (2)	0.6714 (4)	−0.0298 (4)	0.0627 (9)
C6	0.7298 (2)	0.4991 (3)	0.0311 (3)	0.0431 (6)
C7	0.5939 (1)	0.2367 (3)	0.3765 (3)	0.0349 (5)
C8	0.8535 (1)	0.2304 (3)	0.4748 (3)	0.0320 (5)
O1	0.5668 (1)	0.1955 (2)	0.2152 (2)	0.0533 (5)
O2	0.5649 (1)	0.1315 (2)	0.5910 (2)	0.0510 (5)
O3	0.8682 (1)	0.1019 (2)	0.3810 (2)	0.0430 (5)
O4	0.9330 (1)	0.2148 (2)	0.6058 (2)	0.0469 (5)

Table 2. *Geometric parameters* (Å, °)

C1—C2	1.532 (3)	C7—O1	1.295 (3)
C2—C3	1.536 (3)	C7—O2	1.229 (2)
C3—C4	1.473 (3)	C8—O3	1.229 (3)
C4—C5	1.311 (4)	C8—O4	1.310 (3)
C5—C6	1.496 (4)	O1—H7	1.10 (3)
C6—C1	1.521 (2)	O4—H8	1.00 (2)
C1—C7	1.507 (3)	O1—O2[i]	2.656 (2)
C2—C8	1.503 (2)	O3[ii]—O4	2.641 (2)
C1—C2—C3	110.5 (1)	C6—C1—C7	114.8 (2)
C2—C3—C4	111.9 (2)	C1—C2—C8	111.4 (2)
C3—C4—C5	124.2 (2)	C3—C2—C8	111.5 (1)
C4—C5—C6	124.8 (2)	O1—C7—O2	123.4 (2)
C5—C6—C1	111.0 (2)	O3—C8—O4	122.5 (1)
C6—C1—C2	112.4 (2)	O1—H7—O2[i]	177.3 (29)
C2—C1—C7	111.9 (1)	O4—H8—O3[ii]	175.0 (28)

Symmetry code: (i) $1 - x, -y, 1 - z$; (ii) $2 - x, -y, 1 - z$.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55993 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1025]

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N,N'-Dimethylcryptand[2.2.2] Diiodide

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Abstract

The bisquaternary ammonium cation in 1,10-dimethyl-4,7,13,16,21,24-hexaoxa-1,10-diazoniabicyclo[8.8.8]-hexacosane diiodide displays an *exo–exo* conformation at the bridgehead N atoms and has a twofold crystallographic axis. The packing arrangement is closely related to that of the bisborohydride of cryptand[2.2.2].

Comment

The title compound was prepared and its structure determined in the course of an investigation of bisquaternary ammonium salts with wide separation of the two positive charges. *N,N'*-Dimethylcryptand[2.2.2] diiodide has already been reported by Pietraszkiewicz, Salanski & Jurczak (1985), who synthesized it *via* the high-pressure reaction of *N,N'*-dimethyldiaza-18-crown-6 with 1,2-bis(2-iodoethoxy)ethane. We chose to prepare the compound by methylating commercially available cryptand[2.2.2] with methyl iodide in methanol.

The bisquaternary ammonium cation has a crystallographic twofold axis and displays the expected *exo–exo* conformation (Fig. 1). A similar molecular structure has been determined for the bisborohydride of cryptand-[2.2.2], $(C_{18}H_{36}N_2O_6)(BH_3)_2$, by Metz, Moras & Weiss (1976). Given the different chemical nature of the bisborohydride compared with the bisquaternary diiodide, the parallels between the two crystal structures are rather

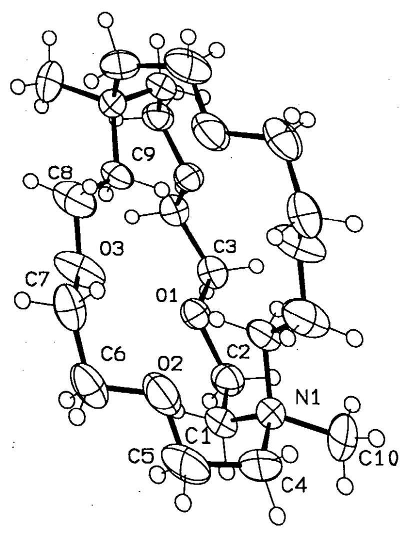

Fig. 1. View of $C_{20}H_{42}N_2O_6^{2+}$ showing the labelling of the non-H atoms. Thermal ellipsoids are shown at 50% probability levels; H atoms are drawn as small circles of arbitrary radii.

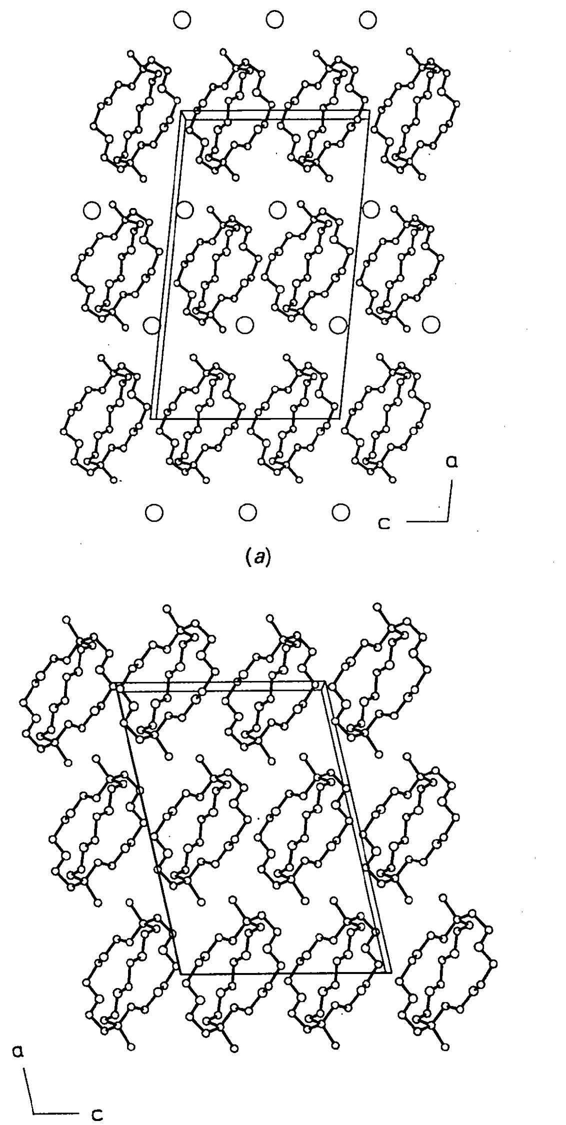

Fig. 2. Packing arrangement of (*a*) $C_{20}H_{42}N_2O_6^{2+}.2I^-$ and (*b*) $(C_{18}H_{36}N_2O_6)(BH_3)_2$.

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