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The Conformation of Non-Aromatic Ring Compounds. LXXIX.* The Crystal and Molecular Structure of *N*,*N'*-Dicarbomethoxy-2,3-diaza-5(*exo*)-7(*anti*)- dichlorobicyclo[2,2,1]heptane at −180°C

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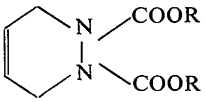
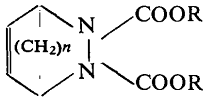
Crystals of *N*,*N'*-dicarbomethoxy-2,3-diaza-5(*exo*)-7(*anti*)-dichlorobicyclo[2,2,1]heptane are monoclinic, space group $C2/c$. The lattice constants are $a=16{\cdot}119$ (6), $b=11{\cdot}107$ (2), $c=13{\cdot}816$ (10) Å, $\beta=103{\cdot}39$ (7)° at −180°C and $Z=8$. The structure was solved by Patterson methods. The molecule, obtained by chlorination of the corresponding 5,6-bicyclo[2,2,1]heptene analogue, turns out to be the 5,7-dichloro derivative with one chlorine atom attached to the methylene bridge. The refinement of the structure using diffractometer data collected at −180°C resulted in a conventional R value of 5·38% (observed reflexions). There appears to be positional disorder of the two carbomethoxy groups; however, different models accounting for this effect and based upon the space groups $C2/c$ or Cc gave no better results. The hybridization of the nitrogen atoms is pyramidal, one carbomethoxy group being in the *exo* and the other in the *endo* position with respect to the diaza-norbornane skeleton.

Introduction

The proton magnetic resonance (p.m.r.) spectra of monocyclic (I) and bicyclic (II) diacylhydrazines are temperature dependent owing to the various conformational exchange processes that become 'frozen in' on the p.m.r. time scale below a certain 'coalescence' temperature.



(I)



(II)

The exact nature of these rate processes is at present a matter of some controversy. Two transitions are often observed and interpreted as follows.

(i) A hindered rotation about the N-COOR bonds. This process is generally associated with barrier heights ΔG^* in the range 13 ± 1 kcal mole^{-1} (Breliere & Lehn, 1965; Anderson & Lehn, 1968*a*). The existence of the relatively high barriers in an amide-like structure is taken to imply a certain amount of double-bond character in the N–C bond.

(ii) A process associated with barrier heights ΔG^* in the range 16–19 kcal mole^{-1} for which two essentially different explanations have been offered.

 (*A*) A conformational transition described as cyclohexene-like ring inversion in (I) or as bridge-flipping in the bicyclo[2,2,2]octane system and its higher homologues (II, $n\neq1$, Anderson & Lehn, 1968*b*; Bittner & Gerig, 1972). This description presupposes an essentially planar ground state geometry about nitrogen, although Price & Sutherland (1967) admit the possibility of some non-planarity.

 (*B*) A hindered nitrogen inversion, as in ammonia, which description requires a pyramidal structure about nitrogen in the ground state and a planar configuration about one or both centres in the transition state.

At first sight the existence of pyramidal (invertible) nitrogens seems incompatible with the existence of an appreciable barrier to rotation about the N–C bond as stated in (i) (Daniels & Roseman, 1966; Allred, Anderson, Miller & Johnson, 1967); however, the possibility of hindered rotation about the N–OR bond has been postulated (Moriarty, Murphy, Druck & May, 1967). Our interest in this field arose when Lehn & Wagner (1969) proposed, on the basis of hypothesis (*A*), a method of conformational fixation using the (assumed) geometrical and energetical properties inherent in the diurethane fragment –N(COOR)–N(COOR) to lock conformations and to slow down conformational rate processes. They were unable to demonstrate the existence of process (ii) in the bicyclo[2,2,1]heptene (III) and heptane (IV) analogues and concluded that the norbornane-like framework is too rigid to allow twisting about the N–N bond (flipping). In contrast, Altona & Sundaralingam (1970) demonstrated that substituted norbornanes can easily adapt to strain by several modes of twist. A subsequent series of molecular mechanics calculations (Altona & Wepster, 1971) of geometries and energies of twisted norbornanes, in which calculations of various artificial barriers about the C(2)–C(3) bond were introduced, strongly suggested that the bicyclic frame work of (IV) should exist in a highly twisted form (presupposing hypothesis (*A*) to be essen-

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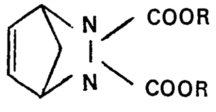
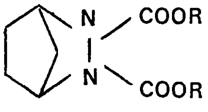
* Part LXXVIII: Braun, Hornstra, Knobler, Rutten & Romers (1972). *Acta Cryst.* B29, 463–469.

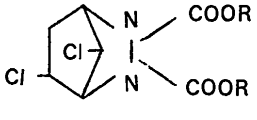
tially correct). For lack of information case (*B*) (invertible nitrogen) was not considered in these calculations. Obviously, an X-ray analysis was necessary to solve the uncertainties. Unfortunately, none of the esters of type (IV) we synthesized (R = methyl, ethyl, isopropyl) afforded crystals of sufficient quality for a single-crystal analysis. Therefore, the double bond of (III) ($R = CH_3$) was chlorinated to yield a new compound (V), which appeared promising.



(III)



(IV)



(V)

The results of our diffraction analysis turn out to be quite interesting in two respects:

(i) The chlorination product (V) is not one of the isomers expected for normal addition to the double bond. One of the chlorine atoms (Fig. 1) is situated on the methylene bridge. Crystals of the corresponding bromination products (DBH) turn out to have nearly the same lattice parameters (Table 1) and the same space group (*C*2/*c*). We may, therefore, conclude that in the dibromo adduct one of the bromine atoms is connected to the methylene bridge. A similar rearrangement is known to occur in the addition of chlorine and bromine to bicyclo-[2,2,1]heptene (Roberts, Johnson & Carboni, 1954; Kwart & Kaplan, 1954), but rearrangement reactions of the diaza-analogues do not seem to have been reported before.

(ii) We observe a pronounced pyramidal structure about the nitrogen atoms. The ester groups are at an angle of about 90° to one another, and the C–N–N–C fragment of the ring system shows perfect planarity.

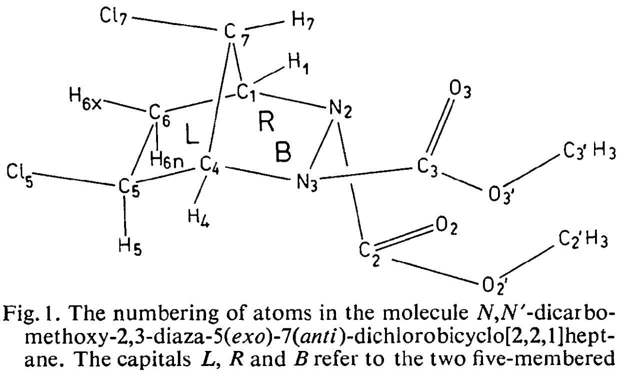
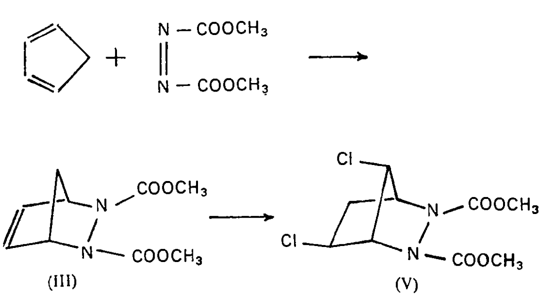


Fig. 1. The numbering of atoms in the molecule *N*,*N*′-dicarbomethoxy-2,3-diaza-5(*exo*)-7(*anti*)-dichlorobicyclo[2,2,1]heptane. The capitals *L*, *R* and *B* refer to the two five-membered rings and to one six-membered ring.

The systematic name of compound (V) is *N*,*N*′-dicarbomethoxy-2,3-diaza-5(*exo*)-7(*anti*)-dichlorobicyclo[2,2,1]heptane (hereafter DCH). The atomic numbering is given in Fig. 1.

Experimental

The title compound DCH (V) was prepared according to the following reaction scheme.



The Diels–Alder reaction step has been described by McKenzie, Rodgman & Wright (1952); the chlorination of (III) was carried out both by means of sulphuryl chloride under reflux condition in carbon tetrachloride solution (Kharash & Brown, 1939) and by slow addition of chlorine to a cold (0°) solution of (III) in the same solvent. Apparently, both reactions yield (V) as the sole product as evidenced by vapour phase and thin-layer chromatography of the crude reaction mixture.

The compound was crystallized from cyclohexane, melting point 99–100 °C. The crystals are monoclinic with faces {010}, {101} and $\{10\bar{1}\}$. The lattice constants (Table 1) were determined at room temperature and at − 180 °C with a three-circle diffractometer using Cu $K\alpha$ radiation ($\lambda = 1{\cdot}54178$ Å). The measured density (flotation method) is 1·52 g cm^{-3}, in agreement with the density calculated for eight molecules per unit cell. The systematic extinctions are *hkl*, $h+k$ odd, and *h*0*l*, (*h*), *l* odd, indicating the space groups *C*2/*c* or *Cc*. The former was chosen on grounds of statistical tests and was corroborated by Patterson analysis, but the latter was also considered during some stages of the refinement (see below).

Table 1. *Crystal data of DCH and DBH*

N,*N*′-Dicarbomethoxy-2,3-diaza-5(*exo*)-7(*anti*)-dichlorobicyclo[2,2,1]heptane $C_9H_{12}N_2O_4Cl_2$

$M = 284{\cdot}0$ g $mole^{-1}$, m.p. 99–100 °C

Space group *C*2/*c*, $Z = 8$ molecules per unit cell

DCH, 20 °C	DCH, − 180 °C	DBH, 20 °C
$a = 16{\cdot}073$ (8) Å	$a = 16{\cdot}119$ (8) Å	$a = 16{\cdot}4$ Å
$b = 11{\cdot}152$ (2)	$b = 11{\cdot}107$ (2)	$b = 11{\cdot}4$
$c = 13{\cdot}997$ (10)	$c = 13{\cdot}816$ (10)	$c = 14{\cdot}2^5$
$\beta = 102{\cdot}85$ (7)°	$\beta = 103{\cdot}39$ (7)°	$\beta = 103$° (assumed)

Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu(\text{Mo } K\alpha) = 5{\cdot}30$ cm^{-1}

$d_{obs} = 1{\cdot}52$ g cm^{-3}, $d_x = 1{\cdot}544$ g cm^{-3}

A crystal with dimensions 0·5 × 0·6 × 0·7 mm was used for measurements of reflexion intensities at − 180 °C using a cooling system designed by van Bolhuis (1971). The crystal was mounted about the reciprocal lattice direction [101]* on the φ-axis of a goniometer head of a three-circle diffractometer. The crystal was irradiated with Mo $K\alpha$ rays monochromated with a graphite crystal. The ω-scan was applied between a minimum glancing angle $\theta = 4°$ and a maximum $\theta = 30°$. One of the standard reflexions $\bar{2}4\bar{1}$, $\bar{2}25$ or $\bar{5}10$ was measured after each series of 20 reflexions.

3497 symmetry-independent reflexions were recorded. Included in this number are, however, 466 reflexions having counts smaller than twice the background count. The intensities were corrected for the decrease in scattering power of the crystal by means of a fifth-order polynomial function of the exposure time. The fairly large size of the crystal and the relatively large value of the linear absorption coefficient necessitated an absorption correction which was carried out by means of the Monte Carlo method described by De Graaff (1973). The calculated transmission factors varied between 0·82 and 0·87. Finally, the intensities were reduced to structure-factor moduli in the usual way.

Refinement

The positions of the chlorine atoms Cl(5) and Cl(7) were deduced from a sharpened Patterson function assuming the centrosymmetric space group $C2/c$. The scanning of a minimum function applied at the symmetry-related positions of Cl(5) enabled us to locate all atoms except hydrogen.

An isotropic least-squares refinement was carried out in the usual way. The scattering factors for carbon, nitrogen and oxygen were taken from *International Tables for X-ray Crystallography* (1962), those for chlorine and hydrogen from Cromer & Waber (1965) and from Stewart, Davidson & Simpson (1965), respectively.

With the exception of those bound to the methyl carbon atoms C(2′) and C(3′) the hydrogen atoms were located in a difference Fourier map. The hydrogen atoms belonging to the methyl groups were introduced at calculated positions assuming the C–H bonds to be staggered with respect to the bonds C(2)–O(2′) and C(3)–O(3′). After introduction of weights $w(F_o)$ as described by Portheine, Romers & Rutten (1972), the refinement was continued with anisotropic temperature factors for all heavy atoms using 3 × 3 and 6 × 6 blocks and keeping the hydrogen atoms at fixed positions with fixed isotropic B's. The positions and isotropic temperature parameters of the hydrogen atoms were next refined separately with 3 × 3 and 1 × 1 blocks. Finally, all atoms were refined simultaneously with one full matrix. Defining the reliability indices R and R_w as

$$R = \sum |F_o - F_c/S| / \sum F_o$$

and

$$R_w = [\sum w(F_o)\,(F_o - F_c/S)^2 / \sum w(F_o)F_o^2]^{1/2},$$

where $1/S$ reduces F_c to the scale of F_o, the final reliability indices are $R = 5{\cdot}38$ and $R_w = 7{\cdot}03\,\%$ (observed reflexions only). The positional parameters and isotropic B values of hydrogen atoms are listed in Table 2 and the vibrational parameters U_{ij} of the heavy atoms are to be found in Table 3.*

Table 2. *Positional parameters (fractions of cell edges) of DCH*

Estimated standard deviations (in 10^{-3} Å units for C, N and O, in 10^{-4} Å units for Cl and in 10^{-2} Å units for H) are given in parentheses. B values (Å^2 units) of hydrogen atoms and their e.s.d.'s are included in the list.

	x	y	z	B
C(1)	0·0853 (2)	0·3266 (2)	0·5016 (2)	—
N(2)	0·1291 (2)	0·3158 (2)	0·4187 (2)	—
N(3)	0·1060 (2)	0·1987 (2)	0·3760 (2)	—
C(4)	0·0488 (2)	0·1463 (2)	0·4359 (2)	—
C(5)	−0·0338 (2)	0·2201 (2)	0·4033 (2)	—
C(6)	−0·0100 (2)	0·3419 (2)	0·4576 (2)	—
C(7)	0·0932 (2)	0·1973 (2)	0·5374 (2)	—
C(2)	0·1266 (2)	0·4069 (2)	0·3520 (2)	—
C(3)	0·1750 (2)	0·1340 (2)	0·3580 (2)	—
O(2)	0·1197 (2)	0·3961 (2)	0·2649 (2)	—
O(2′)	0·1355 (2)	0·5130 (2)	0·4000 (2)	—
O(3)	0·2393 (2)	0·1791 (2)	0·3454 (2)	—
O(3′)	0·1556 (2)	0·0171 (2)	0·3499 (2)	—
C(2′)	0·1380 (3)	0·6164 (3)	0·3398 (3)	—
C(3′)	0·2210 (3)	−0·0589 (3)	0·3268 (4)	—
Cl(5)	−0·12338 (6)	0·14795 (7)	0·43503 (7)	—
Cl(7)	0·04125 (6)	0·16790 (6)	0·63522 (6)	—
H(1)	0·112 (3)	0·384 (3)	0·552 (3)	0·59 (50)
H(4)	0·048 (2)	0·059 (2)	0·431 (2)	0·10 (44)
H(5)	−0·047 (2)	0·225 (2)	0·333 (2)	0·08 (44)
H(6*n*)	−0·025 (3)	0·411 (3)	0·411 (3)	0·85 (51)
H(6*x*)	−0·038 (3)	0·351 (3)	0·510 (4)	2·21 (69)
H(7)	0·147 (3)	0·175 (3)	0·562 (3)	1·21 (59)
H(2′1)	0·183 (4)	0·599 (4)	0·306 (4)	4·23 (88)
H(2′2)	0·129 (4)	0·691 (3)	0·377 (4)	3·23 (79)
H(2′3)	0·086 (4)	0·609 (4)	0·281 (3)	3·31 (78)
H(3′1)	0·195 (5)	−0·127 (5)	0·306 (5)	6·70 (126)
H(3′2)	0·275 (4)	−0·058 (4)	0·371 (4)	4·61 (98)
H(3′3)	0·231 (4)	−0·022 (4)	0·265 (4)	4·24 (90)

The estimated standard errors in positional coordinates are ∼0·03 Å for hydrogen, ∼0·002 Å for carbon, nitrogen and oxygen and ∼0·0006^5 Å for chlorine. In view of the suggested accuracy of the bond lengths between the heavy atoms (a conservative estimate would be ∼0·006 Å) the observed differences between chemically equivalent bonds are surprisingly large and exceed the e.s.d. of the difference (∼0·008 Å) by a factor of 2·4 and 3·7 in two cases (see Table 4). The unexpected differences mainly occur in the two ester groups whose atoms show conspicuously large U_{ij} values (Table 3).

A possible explanation of this effect might be a dis-

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* A list of structure factors has been deposited with the National Lending Library, England, as Supplementary Publication No. SUP 30141. Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

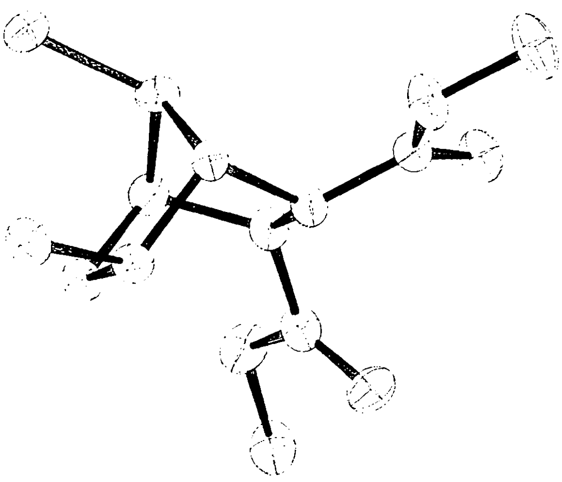

Fig. 2. A perspective view of DCH. The orientations of vibrational ellipsoids are scaled to include 50% probability.

order of one or two carbomethoxy groups over *endo* and *exo* positions with respect to N(2) and N(3). However, a careful survey of difference Fourier maps showed no such irregularity in the expected positions. A second explanation might be a random distribution of the side-chain atoms over slightly different positions. In such case the thermal ellipsoids are largely an artifact due to the apparent disorder. For this reason we tried to apply the procedure described by de Wolf, Verschoor & Romers (1972). The shape of the thermal ellipsoids enabled us to estimate the distances between the separate atoms according to the method of Vos & Smits (1961). Since, however, the separations are very small ($<0{\cdot}4$ Å) the least-squares procedure to refine the structure failed. Although we tried several models, including those based upon the acentric space group *Cc*, the resulting structures were worse than the ordered centrosymmetric structure. For this reason we only present a description of the centric structure.

Discussion of the structure and packing

The bond lengths, valency angles and a number of torsion angles are listed in Tables 4, 5 and 6. A perspective view of the molecule is depicted in Fig. 2, which clearly illustrates the pyramidal hybridization about the nitrogen atoms. It can also be noted that one carbomethoxy group bound to N(2) occupies an *endo* position while the other one is bound to N(3) in *exo* position. Finally, it can be seen that the terminal carbomethoxy atoms O(2′), O(3′), C(2′) and C(3′) display rather large vibrational amplitudes due to the presumed disorder effect. In view of the unknown standard errors in geometrical entities the values of bond lengths and valency angles need no comment.

Table 3. *Vibrational parameters* $U_{ij}(10^{-4}\ \text{Å}^2)$ *in the temperature factor exp* $[-2\pi^2 \sum_{ij} h_i h_j a_i^* a_j^* U_{ij}]$

Numbers in parentheses are e.s.d.'s.

	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{13}$
C(1)	327 (11)	262 (10)	301 (11)	−62 (16)	−84 (16)	131 (17)
N(2)	343 (10)	207 (8)	356 (10)	−88 (14)	−77 (14)	227 (16)
N(3)	290 (9)	200 (8)	352 (10)	−75 (14)	−67 (14)	217 (15)
C(4)	276 (10)	235 (10)	333 (11)	−66 (15)	−49 (17)	191 (17)
C(5)	253 (10)	325 (11)	320 (11)	−68 (17)	11 (18)	56 (16)
C(6)	323 (11)	273 (11)	370 (12)	14 (17)	−24 (18)	174 (18)
C(7)	278 (10)	276 (10)	287 (11)	−78 (16)	48 (17)	1 (16)
C(2)	279 (10)	251 (10)	416 (12)	−110 (16)	−50 (18)	208 (18)
C(3)	276 (10)	257 (10)	309 (11)	−39 (16)	−62 (17)	113 (16)
O(2)	489 (10)	366 (9)	345 (9)	−179 (16)	25 (15)	209 (15)
O(2′)	638 (12)	220 (8)	465 (10)	−133 (15)	−34 (14)	457 (18)
O(3)	277 (8)	344 (9)	559 (11)	−125 (13)	−136 (16)	260 (15)
O(3′)	388 (9)	235 (8)	677 (12)	−81 (14)	−157 (16)	531 (18)
C(2′)	573 (16)	244 (11)	689 (18)	−73 (22)	128 (23)	696 (27)
C(3′)	426 (15)	319 (13)	949 (24)	−8 (22)	−302 (28)	613 (30)
Cl(5)	259 (3)	399 (3)	510 (4)	−107 (4)	−21 (5)	157 (5)
Cl(7)	439 (3)	360 (3)	284 (3)	−58 (5)	19 (4)	141 (5)

Table 4. *Bond lengths* (Å) *in DCH*

C(1)–N(2)	1·484	N(2)–C(2)	1·364	C(5)–H(5)	0·94
N(2)–N(3)	1·441	N(3)–C(3)	1·394	C(6)–H(6*x*)	1·00
N(3)–C(4)	1·493	C(2)–O(2)	1·188	C(6)–H(6*n*)	0·94
C(4)–C(5)	1·539	C(3)–O(3)	1·199	C(7)–H(7)	0·89
C(5)–C(6)	1·551	C(2)–O(2′)	1·343	C(2′)–H(2′1)	0·97
C(6)–C(1)	1·525	C(3)–O(3′)	1·335	C(2′)–H(2′2)	1·00
C(4)–C(7)	1·528	O(2′)–C(2′)	1·424	C(2′)–H(2′3)	1·03
C(1)–C(7)	1·515	O(3′)–C(3′)	1·443	C(3′)–H(3′1)	0·88
C(5)–Cl(5)	1·793	C(1)–H(1)	0·96	C(3′)–H(3′2)	0·94
C(7)–Cl(7)	1·778	C(4)–H(4)	0·98	C(3′)–H(3′3)	1·00

Table 5. *Valency angles in DCH* (°)

C(1)–N(2)–N(3)	105·3	C(4)–N(3)–N(2)	105·0
C(1)–N(2)–C(2)	121·1	C(4)–N(3)–C(3)	121·3
N(3)–N(2)–C(2)	115·4	N(2)–N(3)–C(3)	113·4
N(2)–C(2)–O(2)	126·2	N(3)–C(3)–O(3)	124·3
N(2)–C(2)–O(2′)	109·5	N(3)–C(3)–O(3′)	109·5
O(2)–C(2)–O(2′)	124·3	O(3)–C(3)–O(3′)	126·0
C(2)–O(2′)–C(2′)	115·7	C(3)–O(3′)–C(3′)	114·7
N(2)–C(1)–C(6)	108·5	N(3)–C(4)–C(5)	103·5
C(1)–C(6)–C(5)	102·1	C(4)–C(5)–C(6)	102·9
N(2)–C(1)–C(7)	99·0	N(3)–C(4)–C(7)	98·4
C(6)–C(1)–C(7)	103·7	C(5)–C(4)–C(7)	105·1
C(6)–C(5)–Cl(5)	113·2	C(1)–C(7)–Cl(7)	113·8
C(4)–C(5)–Cl(5)	112·4	C(4)–C(7)–Cl(7)	115·2
C(4)–C(7)–C(1)	93·8		

Inspection of Table 6 reveals that ring *B* is a slightly distorted tub-boat. Ring *R* has an ideal envelope form but ring *L* is a rather distorted envelope. Apparently the asymmetrical introduction of chlorine atoms into the molecule rather than the different orientation (*exo* and *endo*) of the ester groups has a large impact on the shape of the norbornane-like ring system. The projection along the line C(4)···C(1) [Fig. 3(*a*)] shows that the distortion is C(+, −) according to the nomenclature of Altona & Sundaralingam (1970). We also note that the orientation of the ester groups is symmetrical with respect to the plane going through C(1), N(2), N(3) and C(4).

The Newman projections, Fig. 3(*b*), (*c*), (*d*) and (*e*), show that the carbomethoxy groups are nearly planar.

The packing of the molecules is illustrated in Fig. 4 in a projection of the structure along [010]. Taking into consideration intermolecular distances between heavy atoms being less than 4·0 Å the molecule i at x, y, z is surrounded by 14 neighbours at positions

ii	x,	$1+y$,	z	ix	$\frac{1}{2}-x$,	$\frac{1}{2}-y$,	$1-z$
iii	x,	$-1+y$,	z	x	$-x$,	y,	$\frac{1}{2}-z$
iv	$-x$,	y,	$\frac{3}{2}-z$	xi	$\frac{1}{2}-x$,	$\frac{1}{2}+y$,	$\frac{1}{2}-z$
v	$\frac{1}{2}+x$,	$\frac{1}{2}+y$,	z	xii	$\frac{1}{2}-x$,	$-\frac{1}{2}+y$,	$\frac{1}{2}-z$
vi	$-x$,	$1-y$,	$1-z$	xiii	x,	$1-y$,	$-\frac{1}{2}+z$
vii	x,	$-y$,	$\frac{1}{2}+z$	xiv	$-x$,	$-y$,	$1-z$
viii	x,	$1-y$,	$\frac{1}{2}+z$	xv	x,	$-y$,	$-\frac{1}{2}+z$

The shortest intermolecular distances are contacts between carbonyl oxygen atoms and carbon atoms. These contacts, shorter than 3·22 Å, are indicated by dashed lines in Fig. 4. Similar very short distances between oxygen atoms and carbon atoms in the range between 2·96 Å and 3·17 Å were observed in the structure of *β*-chloroglutaric anhydride (Koer, De Kok & Romers, 1972). Similar short contacts were also found in the structure of *cis*-2-chloro-4-t-butylcyclohexanone (De Graaff, Giesen, Rutten & Romers, 1972).

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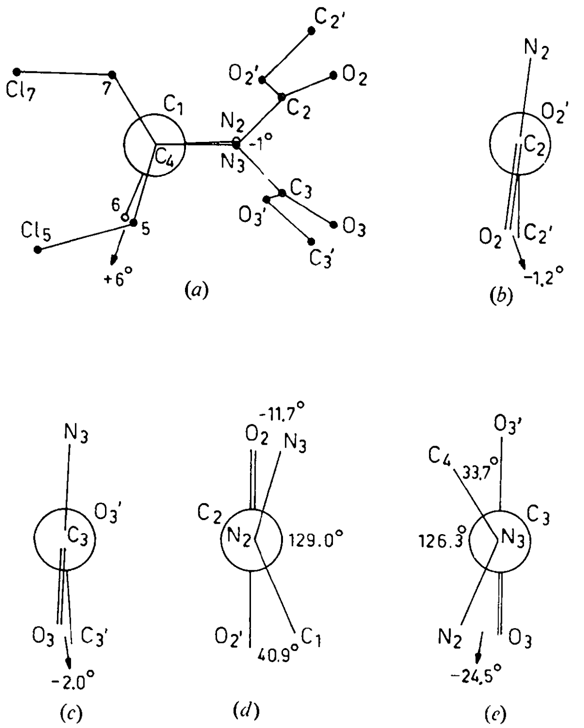

Fig. 3. Newman projections along (*a*) C(4)···C(1), (*b*) C(2)–O(2′), (*c*) C(3)–O(3′), (*d*) N(2)–C(2) and (*e*) N(3)–C(3).

Table 6. *Torsion angles in DCH* (°)

Ring *B*		Exocyclic torsion angles	
C(1)–N(2)–N(3)–C(4)	0·4	Cl(7)–C(7)–C(4)–N(3)	175·1
N(2)–N(3)–C(4)–C(5)	71·6	Cl(7)–C(7)–C(1)–N(2)	−176·3
N(3)–C(4)–C(5)–C(6)	−75·4	Cl(7)–C(7)–C(4)–C(5)	68·6
C(4)–C(5)–C(6)–C(1)	7·3	Cl(7)–C(7)–C(1)–C(6)	−64·7
C(5)–C(6)–C(1)–N(2)	64·3	Cl(5)–C(5)–C(6)–C(1)	128·9
C(6)–C(1)–N(2)–N(3)	−71·6	Cl(5)–C(5)–C(4)–N(3)	162·5
Ring *R*		Ring *L*	
C(7)–C(1)–N(2)–N(3)	36·2	C(7)–C(1)–C(6)–C(5)	−40·2
N(2)–N(3)–C(4)–C(7)	−36·3	C(7)–C(4)–C(5)–C(6)	27·3
N(3)–C(4)–C(7)–C(1)	56·6	C(1)–C(7)–C(4)–C(5)	−49·3
C(4)–C(7)–C(1)–N(2)	−56·6	C(4)–C(7)–C(1)–C(6)	55·0

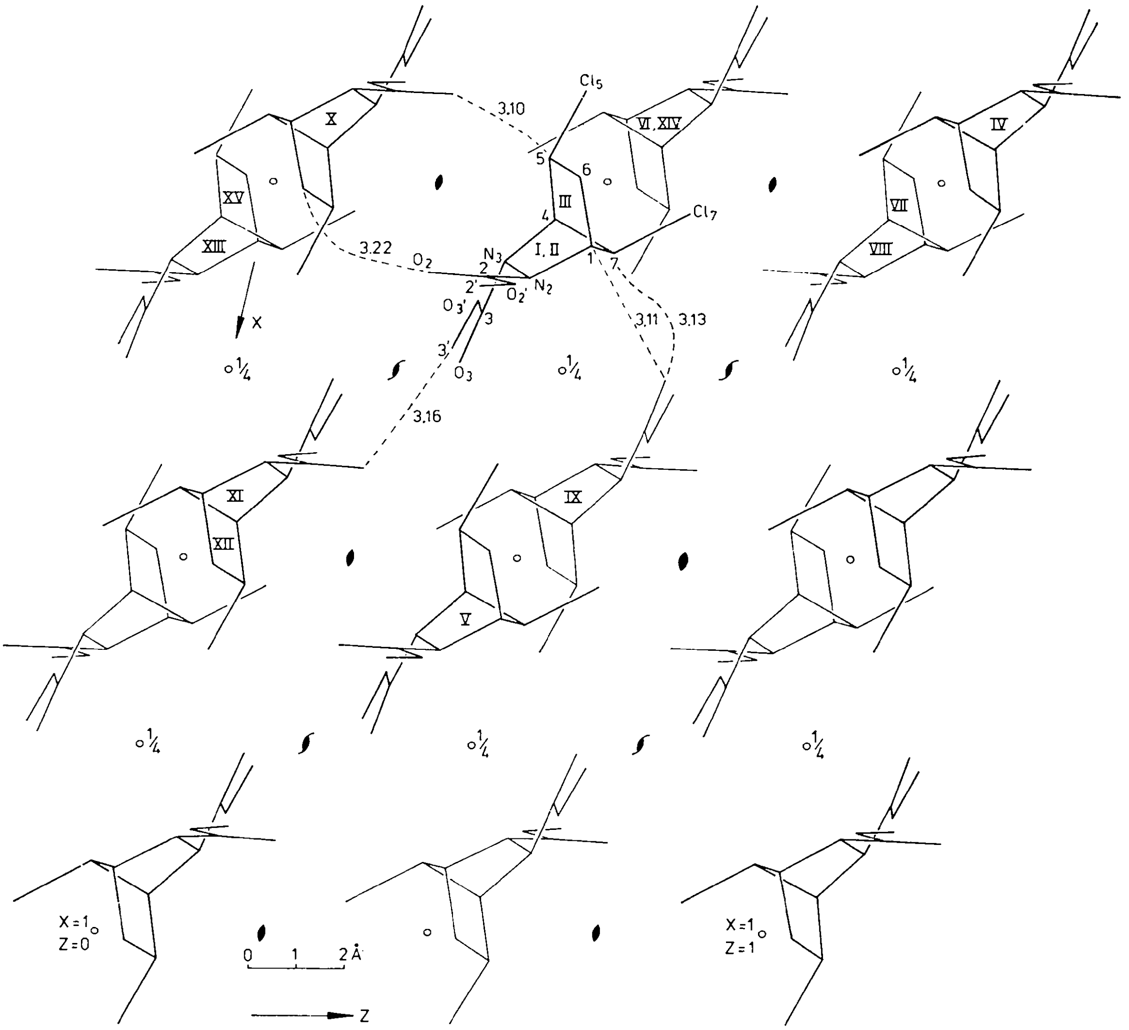

Fig. 4. The structure of DCH in a projection along [010]. The dashed lines indicate intermolecular contacts. The Roman numbers are explained in the text.

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