Table 5. *Torsion angles* (°)

C(14)–O(1)–C(11)–C(12)	–17·9 (5)
O(1)–C(11)–C(12)–C(13)	37·7 (5)
C(11)–C(12)–C(13)–C(14)	–41·2 (4)
C(12)–C(13)–C(14)–O(1)	31·9 (5)
C(13)–C(14)–O(1)–C(11)	–9·1 (5)
O(1)–C(11)–C(10)–C(9)	24·1 (7)
O(2)–C(13)–C(14)–O(1)	–81·7 (4)
O(3)–C(12)–C(11)–O(1)	159·8 (3)
O(3)–C(12)–C(13)–C(14)	–159·4 (4)
O(2)–C(13)–C(12)–C(11)	75·1 (3)

plane so that the sugar pucker is C(12)-*endo*, C(13)-*exo*. From the torsion angles in Table 5 it can be deduced that the furanose ring is in the $^{2}_{3}T$ conformation.

The anomeric configuration can be defined by the atoms bonded to the furanose ring. In the β anomer C(10) is on the opposite side of the mean plane to O(2) and O(3), and these three atoms have deviations of 0·66, –1·65 and –0·32 Å respectively, while the α anomer has C(10), O(2) and O(3) on the same side of the least-squares plane.

Fig. 2 shows the arrangement of the molecules in the unit cell as viewed along [010]. The molecules are linked by hydrogen bonds and van der Waals forces. Each molecule is involved in two strong hydrogen bonds, O(1)···H–O(2) of 2·70 Å and N(2)–H···O(2) of 2·76 Å. Both contacts are between molecules related by twofold screw axes and translated, for the first, along the *bc* diagonal, and, for the second, along the *b* axis. There is a short S···C(14) distance of 3·55 Å between molecules translated along the *bc* diagonal that may indicate some van der Waals

interaction. The effect is to produce a three-dimensional network of molecules.

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1,9-Diacetoxy-5-chloro-12,12-dimethyltricyclo[6.2.2.0^{2,7}]dodeca-4,9-diene-3,6-dione

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Abstract. $C_{18}H_{19}O_6Cl$, triclinic, $P\bar{1}$, $a = 13·280$ (6), $b = 8·638$ (5), $c = 8·164$ (5) Å, $\alpha = 107·6$ (2), $\beta = 82·4$ (2), $\gamma = 97·7$ (2)°, $V = 880·7$ Å^3, $M_r = 366·5$, $D_m = 1·36$, $D_c = 1·38$ g cm^{-3} for $Z = 2$. The structure was solved by direct methods to a final R of 0·073 for 1724 unique X-ray diffractometer data. There are no unusual bond lengths.

Introduction. 1-Methoxycyclohexa-1,3-diene has been shown to undergo regiospecific Diels–Alder addition to

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2-chloro-1,4-benzoquinone, while the related 1,3-dimethoxycyclohexa-1,3-diene behaves quite differently with this dienophile (Giles & Roos, 1976). The reaction between 1,3-diacetoxycyclohexa-1,3-diene, generated *in situ* (Wolinsky & Login, 1970), and 2-chloro-1,4-benzoquinone has now been investigated. It has been found that both possible regioisomers are formed in this reaction, in the ratio of 2:1 as indicated by ¹H NMR spectroscopy. This reaction is, therefore, not of great synthetic importance. However, the major isomer, m.p. 183°C, has been purified chromatographically, and this crystallographic study was undertaken to determine whether Cl bonds to C(4) or C(5).
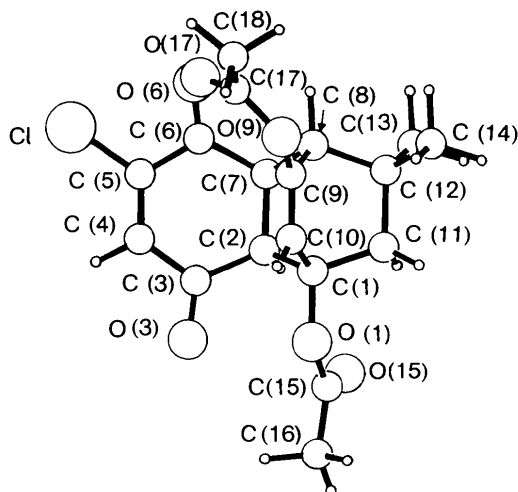
Preliminary oscillation and Weissenberg photographs, taken about the three principal axes, indicated that the space group was either $P1$ or $P\bar{1}$. The latter was chosen from analysis of E statistics, the choice being vindicated by the successful refinement of the structure. The crystal used for data collection had dimensions $0.35 \times 0.1 \times 0.1$ mm and the cell parameters were obtained from least-squares analysis of the settings of 25 reflections measured on a four-circle Philips PW 1100 diffractometer. Intensities were

Fig. 1. Perspective view of the molecule with atomic nomenclature.

Table 2. *Fractional atomic coordinates of the hydrogen atoms* $(\times 10^3)$

	x	y	z
H(7)	290	556	1128
H(2)	426	448	1006
H(4)	400	554	545
H(9)	111	421	1042
H(10)	265	97	609
H(111)	285	41	992
H(112)	355	215	1117
H(131)	270	438	1295
H(132)	216	273	1372
H(133)	137	407	1339
H(141)	94	41	964
H(142)	42	182	1150
H(143)	119	39	1170
H(161)	657	41	749
H(162)	599	86	590
H(163)	553	−88	656
H(181)	−83	344	574
H(182)	−112	192	681
H(183)	−36	152	481

Table 1. *Fractional atomic coordinates of the non-hydrogen atoms* $(\times 10^4)$

	x	y	z
Cl	2414 (1)	7531 (2)	6651 (2)
C(5)	2918 (4)	6262 (6)	7527 (8)
C(6)	2472 (4)	6248 (7)	9274 (8)
C(7)	2727 (4)	4919 (6)	9973 (7)
C(2)	3707 (4)	4081 (6)	9110 (7)
C(3)	4238 (5)	4575 (6)	7554 (7)
C(4)	3723 (4)	5494 (7)	6740 (7)
C(8)	1799 (4)	3611 (6)	9934 (7)
C(9)	1756 (4)	2531 (7)	8105 (8)
C(10)	2582 (4)	1787 (6)	7392 (7)
C(1)	3407 (4)	2206 (6)	8589 (7)
C(11)	2993 (4)	1721 (6)	10234 (7)
C(12)	1978 (4)	2496 (6)	11029 (7)
C(13)	2055 (5)	3474 (7)	12919 (7)
C(14)	1071 (4)	1158 (7)	10931 (9)
C(15)	5130 (4)	1380 (7)	8307 (9)
C(16)	5858 (5)	364 (8)	6981 (8)
C(17)	373 (5)	3187 (10)	6954 (9)
C(18)	−545 (4)	2441 (8)	6011 (8)
O(6)	1932 (4)	7269 (5)	10140 (6)
O(3)	5107 (3)	4245 (5)	7022 (5)
O(1)	4233 (3)	1266 (4)	7619 (4)
O(15)	4695 (3)	7829 (5)	10266 (5)
O(9)	853 (3)	2065 (5)	7244 (5)
O(17)	676 (4)	4601 (6)	7383 (7)

Table 3. *Bond lengths* (Å)

C(2)–C(3)	1·517 (9)	C(12)–C(13)	1·529 (9)
C(3)–C(4)	1·457 (9)	C(12)–C(14)	1·546 (9)
C(4)–C(5)	1·321 (9)	C(15)–C(16)	1·502 (9)
C(5)–C(6)	1·474 (9)	C(17)–C(18)	1·495 (10)
C(6)–C(7)	1·520 (9)	C(1)–O(1)	1·452 (8)
C(7)–C(2)	1·555 (8)	O(1)–C(15)	1·362 (8)
C(7)–C(8)	1·553 (9)	C(15)–O(15)	1·195 (8)
C(8)–C(9)	1·507 (9)	C(9)–O(9)	1·420 (8)
C(9)–C(10)	1·323 (9)	O(9)–C(17)	1·321 (9)
C(10)–C(1)	1·500 (9)	C(17)–O(17)	1·193 (8)
C(1)–C(2)	1·555 (9)	C(3)–O(3)	1·218 (7)
C(1)–C(11)	1·537 (9)	C(6)–O(6)	1·204 (7)
C(11)–C(12)	1·560 (9)	C(5)–Cl	1·719 (8)
C(12)–C(8)	1·556 (8)		

Table 4. *Selected bond angles* (°)

C(11)–C(1)–C(2)	108·2 (5)
C(7)–C(2)–C(1)	107·4 (4)
C(8)–C(7)–C(2)	110·0 (4)
C(7)–C(8)–C(12)	109·1 (5)
C(8)–C(9)–C(10)	116·0 (5)
C(1)–C(10)–C(9)	112·2 (6)
C(12)–C(11)–C(1)	110·3 (5)
C(11)–C(12)–C(8)	107·0 (5)

measured with graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.7017$ Å), and the ω–2θ scan mode [scan width $1.2°$ (θ), scan speed $0.04°$ (θ) s^{-1}]. 2162 reflections were collected in the 2θ range $6°$ to $44°$ and, using the criterion $I_{rel} > 2\sigma(I_{rel})$, 1724 reflections were considered 'observed'. A Lorentz–polarization correction was applied; no correction was made for absorption.

The structure was solved by the automatic centrosymmetric routine of the SHELX program system (Sheldrick, 1976), in which an E map yielded 22 of the 25 heavy atoms, and subsequent least-squares refinement, followed by a difference map, showed the remaining heavy atoms. The final refinement was carried out with the heavy atoms treated anisotropically, the methyl H atoms refined as rigid groups and the remaining H atoms constrained at 1.08 Å from their respective C atoms, their positions being dictated by the geometry of the molecule. The isotropic temperature factors of the H atoms, treated as three single parameters, refined to $U = 0.072$ Å^2 (aromatic H), $U = 0.053$ Å^2 (methine, methylene H) and $U = 0.137$ Å^2 (methyl H). The refinement converged to $R = 0.073$ and $R_w = \Sigma w^{1/2}|F_o - F_c|/\Sigma w^{1/2}|F_o| = 0.062$ with $w = 1/\sigma^2$.*

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32929 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. The analysis shows that the major isomer, m.p. 183 °C, formed in the Diels–Alder reaction is the title compound. Tables 1 and 2 show the final coordinates. Fig. 1 shows a perspective view of the molecule with the atomic nomenclature. Principal bond lengths and angles are listed in Tables 3 and 4. All bond lengths are in good agreement with accepted values (Kennard, 1962) and there are no intermolecular close contacts less than 3.5 Å.

All calculations were carried out on a Univac 1106 computer at the University of Cape Town.

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The Monoadduct between [4.4.3]Propella-2,4-diene and 4-Phenyl-1,2,4-triazoline-3,5-dione*

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Abstract. $C_{21}H_{23}N_3O_2$, monoclinic, $P2_1/c$, $a = 14.142$ (2), $b = 7.228$ (1), $c = 18.014$ (3) Å, $\beta = 108.27$ (1)°, $M_r = 349.43$, $Z = 4$, $D_x = 1.328$ g cm^{-3}. The two six-membered rings are in the boat form, both folded towards the central five-membered ring. The 1,2,4-triazoline-3,5-dione ring is *anti* with respect to the latter. The bonding around the two vicinal atoms N(1) and N(3) is pyramidal, while around the third, N(2), it is planar. The cyclopentane ring has an envelope shape with C(12) flapping towards the unsubstituted six-membered ring.

Introduction. The crystal structure of the title compound is of interest in connexion with stereochemical studies of Diels–Alder adducts of [4.4 X]propella-2,4-dienes ($X \equiv$ substituted rings) (Korat, Tatarsky & Ginsburg, 1972; Kalo, Vogel & Ginsburg, 1977).

Intensities from a colourless crystal, $0.4 \times 0.2 \times 0.2$ mm, were collected on a semi-automatic Stoe–Weissenberg diffractometer with graphite-mono-

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* Configuration of Diels–Alder Adducts. I.