Miskin, 1974) and $Na_6Mo_{10}O_{33}$ (Bolger & Gatehouse, 1973) is probably due to the size of the alkali metal ion; the larger Rb–O and Cs–O coordination polyhedra are able to accommodate a more extensive Mo–O framework.

In conclusion it may be noted that the $A_2Mo_nO_{3n+1}$ series reported here is in some ways analogous to the $M_xB_{2n}O_{4n+3}$ series reported by Galy & Lavaud (1971).

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The Crystal and Molecular Structure of [4,4,2]Propella-3,8-diene-11,12-dione

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The crystal structure of the title compound has been determined and refined by three-dimensional least-squares calculations to a final R value of 0·057 using 1870 counter data. The compound crystallizes in the monoclinic space group $P2_1/c$ with $a=7·438$, $b=11·539$, $c=12·935$ Å and $\beta=116·22°$. There are four $C_{12}H_{12}O_2$ molecules in the unit cell. The molecule possesses nearly *mm* symmetry with the two six-membered rings in the boat form and folded toward the diketone.

Introduction

The structure of [4,4,2]propella-3,8-diene-11,12-dione was determined as part of a study of the effects of π electrons on $n\rightarrow\pi^*$ transition energies of *cis*-α-diketones (Neely, Fink, van der Helm & Bloomfield, 1971). The visible and ultraviolet solution absorption spectra of this compound as well as its di- and tetrahydro derivatives have been reported (Bloomfield & Moser, 1968). The title compound and the dihydro derivative show $n\rightarrow\pi^*$ absorption at unusually long wavelengths compared with other unsubstituted α-diketones (Birnbaum, Cookson & Lewin, 1961; Leonard & Mader, 1950; Alder, Schafer, Esser, Krieger & Reubke, 1955). Theoretical studies show that the transition energies depend on the molecular conformations (Fink, 1973; Neely, Fink, van der Helm & Bloomfield, 1971). The structure of the title compound was determined to obtain bond-length and bond-angle data necessary for the calculations and possible clues about preferred conformations of these molecules in solution.

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Experimental results

A sample of [4,4,2]propella-3,8-diene-11,12-dione was supplied by Bloomfield (1968) for the structure determination. Crystals suitable for the X-ray work were grown by slowly cooling a solution of the compound in redistilled n-hexane from 25 to 0°C over a two-day period. Plate-like red crystals formed in this manner.

Preliminary studies showed that the compound is quite stable at temperatures below 0°C when protected from light but undergoes significant decomposition at room temperature in the presence of light. The crystal edges turned noticeably white after only a few hours exposure and diffraction intensities monitored for a few reflections decreased approximately 1% per hour. Decomposition was retarded significantly when crystals were coated with a thin layer of hair spray and kept only in subdued light. Diffraction-intensity decreases of several reflections were reduced to about 0·03% per hour for a new crystal handled in this manner. Decomposition of the data crystals was also satisfactorily controlled in this manner.

All X-ray measurements were made with a General Electric XRD-5 diffractometer using Cu $K\alpha(\lambda = 1{\cdot}5418$ Å) radiation with Ni filters. The diffraction pattern showed that the compound crystallized in the monoclinic system. The systematic absences of $h0l$ ($l \neq 2n$) and $0k0$ ($k \neq 2n$) showed the space group to be $P2_1/c$. The 2θ values of 38 reflections were measured accurately at room temperature (23°C) and used in a least-squares program to calculate the unit-cell dimensions.

The diffraction intensities of all $h\bar{k}l$ and $h\bar{k}\bar{l}$ reflections with $2\theta \leq 140°$ were measured as integrated intensities using a θ–2θ scan. The scan rate for all reflections was 2° min^{-1}. This encompassed 1870 reflections. The intensities were corrected for background radiation and crystal decomposition by means of an hourly monitor reflection. The intensities of 131 reflections were not appreciably above the background radiation intensities. A net count of 70 was assigned to these reflections for the purpose of least-squares refinement. Lorentz, polarization and absorption corrections were also applied. Crystallographic data are given in Table 1.

Table 1. *Crystallographic data*

Formula	$C_{12}H_{12}O_2$
F.W.	188·23
$F(000)$	400
Space group	$P2_1/c$
D_m*	1·251 g cm^{-3}
D_c	1·255 g cm^{-3}
Z	4
μ(Cu $K\alpha$)	6·91 cm^{-1}
a	7·438 (3) Å
b	11·539 (5)
c	12·935 (5)
β	116·22 (2)°
Intensity data	1870 unique reflections

* Measured by flotation in aqueous KI solution.

Structure determination

The structure was solved by direct methods using N.R.C. programs written by Hall & Ahmed (1968). The 230 largest normalized structure-factor amplitudes, those exceeding 1·50 in magnitude, were used in the sign-determining procedure. This yielded 100 signed structure factors from which an E map was calculated. The positions of all carbon and oxygen atoms were easily discernible among the larger peaks and gave a trial structure that refined readily.

The carbon and oxygen coordinates taken from the E map were refined by three-dimensional block-diagonal least-squares calculations, minimizing the quantity $\sum w(k|F_o| - |F_c|)^2$. The weighting scheme $\sqrt{w} = |kF_o|/P$ if $|kF_o| \leq P$ and $\sqrt{w} = P/|kF_o|$ if $|kF_o| > P$ (where $P = 16$ electrons) was used throughout the refinement. This scheme maximized w for those reflection intensities which could be measured most accurately. Atomic scattering factors of carbon and oxygen to calculate

Table 2. *Parameters for the carbon and oxygen atoms* ($\times 10^4$)

The anisotropic temperature factors are expressed in the form:

$$\exp\left[-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{23}kl + b_{13}hl + b_{12}hk)\right].$$

Standard deviations for the last digit are given in parentheses.

	x	y	z	b_{11}	b_{22}	b_{33}	b_{23}	b_{13}	b_{12}
C(1)	1295 (2)	3860 (1)	2293 (2)	203 (5)	57 (2)	63 (2)	−8 (3)	118 (4)	−11 (5)
C(2)	2896 (3)	2942 (2)	2493 (2)	241 (5)	81 (2)	92 (2)	−1 (3)	154 (5)	35 (6)
C(3)	4342 (3)	2839 (2)	3748 (2)	218 (5)	91 (2)	101 (2)	32 (4)	103 (5)	41 (6)
C(4)	3720 (3)	2911 (2)	4555 (2)	271 (6)	98 (2)	86 (2)	43 (4)	80 (5)	30 (6)
C(5)	1566 (3)	3096 (2)	4261 (2)	324 (6)	105 (2)	76 (2)	34 (4)	176 (6)	31 (6)
C(6)	530 (2)	3937 (2)	3246 (1)	205 (5)	63 (2)	67 (2)	3 (3)	129 (4)	−8 (5)
C(7)	−1747 (3)	3958 (2)	2794 (2)	210 (5)	96 (2)	103 (2)	−17 (4)	168 (5)	−31 (6)
C(8)	−2746 (3)	4660 (2)	1712 (2)	187 (5)	92 (2)	114 (2)	−14 (4)	90 (5)	6 (5)
C(9)	−2112 (3)	4602 (2)	914 (2)	247 (6)	88 (2)	85 (2)	6 (3)	55 (5)	1 (6)
C(10)	−390 (3)	3836 (2)	1050 (2)	285 (6)	86 (2)	66 (2)	6 (3)	103 (5)	32 (6)
C(11)	2192 (3)	5078 (2)	2623 (2)	213 (5)	71 (2)	89 (2)	18 (3)	135 (5)	−23 (5)
C(12)	1413 (3)	5158 (2)	3553 (2)	206 (5)	76 (2)	74 (2)	−28 (3)	79 (5)	24 (5)
O(1)	3094 (2)	5703 (1)	2283 (1)	453 (5)	103 (2)	173 (2)	20 (3)	358 (5)	−117 (5)
O(2)	1399 (2)	5884 (1)	4203 (1)	366 (4)	110 (2)	124 (2)	−100 (3)	192 (4)	−34 (4)

F_c were taken from *International Tables for X-ray Crystallography* (1962). After six refinements on the coordinates and isotropic temperature factors of carbon and oxygen atoms, the R index $(=\sum||kF_o|-|F_c||/\sum|kF_o|)$ dropped from an initial value of 0·316 to 0·169. Anisotropic thermal parameters were then introduced and additional refinements carried out. A difference Fourier map was calculated to locate the hydrogen atoms after R had dropped to 0·134. 12 of the 19 most prominent peaks in the difference map showed the expected positions of all hydrogen atoms. Further refinement added the coordinates and isotropic temperature factors of the 12 hydrogens as parameters. Significant shifts were obtained in these parameters during refinement. The least-squares matrix was blocked in 9×9 blocks for anisotropic atoms and in 4×4 blocks for the isotropic atoms. All reflections were included in the refinement. Least-squares calculations were continued until the shifts in all parameters were less than $\frac{1}{7}$ of their standard deviation. At this point R was 0·057 when all reflections were included in the summation and was equal to 0·052 when only reflections with a net count above 70 were included. A final difference Fourier map, using the refined parameters of all atoms, showed no peaks (positive or negative) greater than 0·19 e Å^{-3}. The final positional and thermal parameters and their estimated standard deviations, as calculated from the inverse of the least-squares matrix, are given in Tables 2 and 3.*

Table 3. *Parameters of the hydrogen atoms*

Standard deviations for the last digit are given in parentheses.

	x	y	z	B
H(C2)1	0·352 (3)	0·316 (2)	0·198 (2)	7·4 (5)
H(C2)2	0·221 (2)	0·215 (2)	0·221 (1)	5·3 (4)
H(C3)	0·586 (2)	0·265 (2)	0·394 (1)	4·9 (4)
H(C4)	0·481 (3)	0·282 (2)	0·547 (2)	8·4 (6)
H(C5)1	0·149 (3)	0·342 (1)	0·494 (2)	5·9 (5)
H(C5)2	0·069 (2)	0·234 (2)	0·402 (2)	7·2 (5)
H(C7)1	−0·197 (2)	0·431 (1)	0·347 (1)	5·8 (4)
H(C7)2	−0·215 (3)	0·309 (2)	0·264 (2)	6·7 (5)
H(C8)	−0·391 (3)	0·513 (2)	0·166 (2)	6·4 (5)
H(C9)	−0·284 (3)	0·507 (2)	0·017 (2)	6·2 (5)
H(C10)1	0·018 (3)	0·411 (2)	0·049 (2)	6·9 (5)
H(C10)2	−0·088 (2)	0·298 (2)	0·085 (1)	6·1 (5)

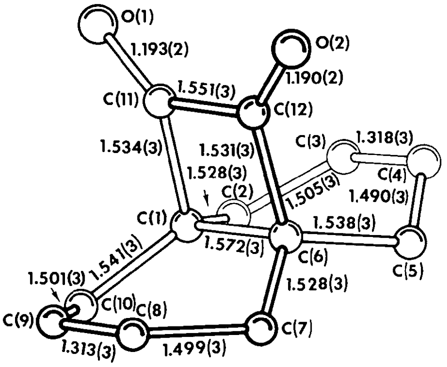

Fig. 1. Bond distances (Å).

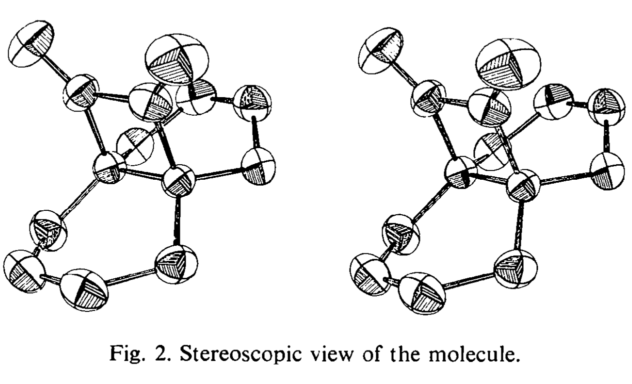

Fig. 2. Stereoscopic view of the molecule.

All computations were carried out on an IBM 360–40 computer. The structure-factor least-squares and Fourier calculations were made with the programs written by Ahmed (1966*a*, *b*). Fig. 3 was drawn with Johnson's (1965) *ORTEP* program. The hydrogen atom scattering factors were those of Stewart, Davidson & Simpson (1965).

Description and discussion of the structure

The bond lengths and angles calculated for the structure are shown in Fig. 1 and Tables 4 and 5. The four bonds to the cyclobutane ring range from 1·528 (3) to 1·541 (3) Å in length with an average value of 1·534 Å. The four bonds in the cyclohexane rings adjacent to the double bonds show the expected shortening from a single-bond length. They range from 1·490 (3) to 1·505 (3) Å with an average length of 1·499 Å. The two double bonds in the six-membered rings are identical within experimental error with lengths of 1·318 (3) and 1·313 (3) Å. The cyclobutane ring shows a small increase in some bond lengths, compared to a normal C–C single bond (Sutton, 1965), as is often found for four-membered rings (Dunitz, 1949; Margulis & Fisher, 1967; Greenberg & Post, 1968; Benedetti, Corradini & Pedone, 1970; Barnett & Davis, 1970). The longest bond in this ring connects the bridgehead carbon atoms and has a length of 1·572 (3) Å while the bond between the carbonyl carbons is 1·551 (3) Å. The two remaining bonds in this ring are near the normal single-bond length with values 1·534 (3) and 1·531 (3) Å. The two carbonyl bonds are identical within experimental error with values of 1·193 (2) and 1·190 (2) Å. The C–H bond lengths fall in the range 0·98 to 1·10 Å with standard deviations of 0·02 Å. The bond angles in the four-membered ring are all within 0·5° of 90·0°, with 89·6 (1)° at both of the bridgehead carbons and 90·3 (1)° and

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30852 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

90·5 (1)° at the carbonyl carbons. The remaining angles about the bridgehead carbons range from 111·1 (2) to 115·6 (2)°.

Table 4. *Bond angles* (°)

The standard deviations are 0·2° for all angles.

C(2)—C(1)—C(6)	115·6	C(1)—C(2)—C(3)	111·7
C(5)—C(6)—C(1)	114·6	C(1)—C(10)-C(9)	111·8
C(10)-C(1)—C(6)	114·2	C(6)—C(5)—C(4)	112·6
C(7)—C(6)—C(1)	115·1	C(6)—C(7)—C(8)	112·0
C(2)—C(1)—C(10)	112·2	C(2)—C(3)—C(4)	121·1
C(5)—C(6)—C(7)	112·9	C(10)-C(9)—C(8)	121·4
C(2)—C(1)—C(11)	112·2	C(5)—C(4)—C(3)	121·4
C(10)-C(1)—C(11)	111·1	C(7)—C(8)—C(9)	120·3
C(5)—C(6)—C(12)	111·3	C(1)—C(11)-O(1)	134·2
C(7)—C(6)—C(12)	111·1	C(6)—C(12)-O(2)	134·0
C(6)—C(1)—C(11)	89·6	C(12)-C(11)-O(1)	135·5
C(1)—C(6)—C(12)	89·6	C(11)-C(12)-O(2)	135·4
C(1)—C(11)-C(12)	90·3		
C(6)—C(12)-C(11)	90·5		

Table 5. *Carbon–hydrogen bond lengths* (Å)

The standard deviations in the bond lengths are 0·02 Å.

C(2)-H(C2)1	0·99	C(7)—H(C7)1	1·04
C(2)-H(C2)2	1·03	C(7)—H(C7)2	1·04
C(3)-H(C3)	1·07	C(8)—H(C8)	1·00
C(4)-H(C4)	1·10	C(9)—H(C9)	1·03
C(5)-H(C5)1	0·98	C(10)-H(C10)1	1·04
C(5)-H(C5)2	1·05	C(10)-H(C10)2	1·05

A stereoscopic view of the molecule with thermal ellipsoids is shown in Fig. 2. The molecule has almost *mm* symmetry with both cyclohexene rings in the boat form folded toward the cyclobutadione segment. The four-membered ring is quite flat. A least-squares plane fitted to the atoms in this ring shows all atoms 0·004 Å removed from the plane with C(1) and C(12) on one side and C(6) and C(11) on the other side. One oxygen, O(1), is 0·002 Å removed in the direction of C(11) while O(2) is 0·065 Å removed from the plane in the direction of C(12). The latter is a very significant distortion of the planarity of the cyclobutadione segment of the molecule, breaking the possibility of *m*, *mm*, or *C*2 symmetry. Least-squares planes through each of the carbonyl carbons and the three atoms bonded to them show that C(11) is in a virtually planar environment while C(12) is 0·018 Å removed from a plane fitted to C(6), C(11), C(12) and O(2) (Table 6).

The destruction of the *mm* symmetry of the molecule is not just due to O(2). In Fig. 1 one can see that bond distances equivalent for *mm* symmetry differ by as much as 5σ while equivalent bond angles, as grouped in Table 4, differ by as much as 7σ. One might consider these differences not significant; however, they are more clearly reflected in intramolecular distances, as shown in Table 7. Distances of the type C(1)···C(3) vary from 2·509 to 2·519 Å and those of the type C(3)···C(11) from 3·010 to 3·063 Å, which are significant differences. The molecule as a whole is skewed away from *mm* symmetry therefore.

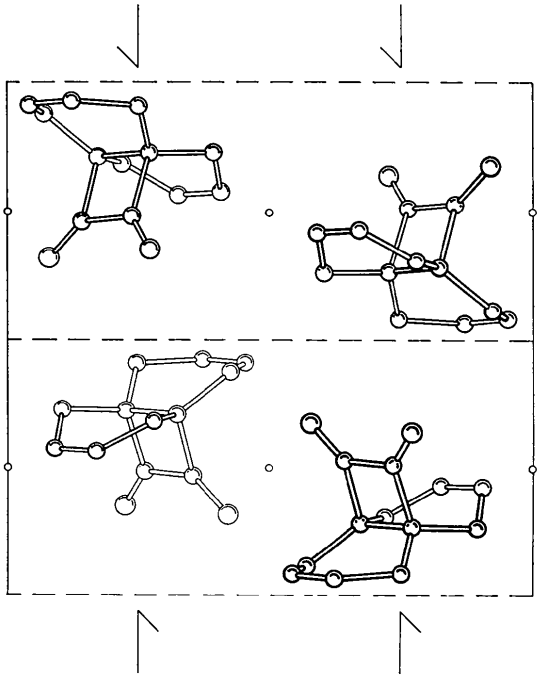

Fig. 3. A view of the molecular packing down the *a* axis.

Table 6. *Least-squares planes*

The planes are described by the expression: $Ax+By+Cz=D$, in which x. y and z are fractional coordinates, and D is the distance of the plane from the origin. The standard deviations in the atom locations are 0·002 Å.

	Planes				A	B	C	D
(1)	C(1)	C(6)	C(11)	C(12)	−4·676	4·510	−4·209	0·166
(2)	C(1)	C(11)	C(12)	O(1)	−4·659	4·532	−4·234	0·174
(3)	C(6)	C(11)	C(12)	O(2)	−4·615	4·277	−4·474	−0·018

Distances from planes (Å)

	$\Delta(1)$	$\Delta(2)$	$\Delta(3)$
C(1)	0·004	0·001	
C(6)	−0·004		0·005
C(11)	−0·004	−0·004	0·005
C(12)	0·004	0·001	−0·018
O(1)	−0·002	0·002	
O(2)	0·065		0·008

The only cause for this destruction of molecular symmetry can be the molecular packing, which is shown in Fig. 3. Some close intermolecular contacts are given in Table 7. Atom O(2) in comparison with O(1) has more close contacts.

Table 7. *Intramolecular and intermolecular distances* (Å)

Intramolecular distances

C(1)–C(3)	2·509	C(11)–C(3)	3·049	O(1)–C(3)	3·717
C(6)–C(4)	2·519	C(12)–C(4)	3·063	O(2)–C(4)	3·777
C(1)–C(9)	2·518	C(11)–C(9)	3·030	O(1)–C(9)	3·699
C(6)–C(8)	2·509	C(12)–C(8)	3·010	O(2)–C(8)	3·620

All intermolecular distances less than 3·90 (C–C), 3·70 (C–O), 2·90 (O–H) and 2·6 Å (H–H)

C(8)–C(3)^{a}	3·821	O(1)——H(C7)2^{a}	2·854
C(3)–C(7)^{b}	3·850	O(2)——H(C2)2^{a}	2·887
O(2)–C(10)^{a}	3·472	O(2)——H(C10)2^{a}	2·441
C(3)–O(2)^{c}	3·437	O(2)——H(C7)1^{d}	2·858
C(4)–O(2)^{c}	3·541	O(1)——H(C8)^{b}	2·768
O(1)–C(8)^{b}	3·686	H(C8)——H(C9)^{e}	2·537
		H(C10)1–H(C10)1^{b}	2·368

Symmetry operations applied to coordinates given in Tables 2 and 3

(*a*)	$-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$	(*d*)	$-x$	$1-y$	$1-z$
(*b*)	$1+x$	y	z	(*e*)	$-1-x$	$1-y$	$-z$
(*c*)	$1-x$	$1-y$	$1-z$				

It is also interesting to note that the O(2)···H(C10)2 separation between molecules related by twofold screw axes is only 2·441 Å, about 0·16 Å less than the sum of a hydrogen and oxygen van der Waals radius (Pauling, 1960; Hamilton & Ibers, 1968). Atom O(2) is bent out of the plane of the four-membered ring by about 0·03 Å in the direction which shortens this distance, suggesting perhaps some hydrogen bonding. For molecules where hydrogen bonding appears to exist between keto oxygens and alkyl hydrogens the most favorable geometrics have an H···O distance of 2·27 Å and a C–H···O angle of 164° (Hamilton & Ibers, 1968). The values in this structure are 2·441 Å and 136°, respectively. Any existing hydrogen-bond interaction in this crystal would have to be considered as quite weak.

The theoretical studies of the electronic energy states of this molecule, based on the conformation determined here, have been reported elsewhere. The most interesting result of these studies was the fact that most of the interaction between the diene π systems and the diketone π system is apparently transmitted through σ bonds rather than through spatial interaction of the π orbitals (Neely *et al.*, 1971).

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