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The Crystal and Molecular Structure of Tetracyclo[6.4.3.0.0^{2,7}]-pentadec-4-en-13-one

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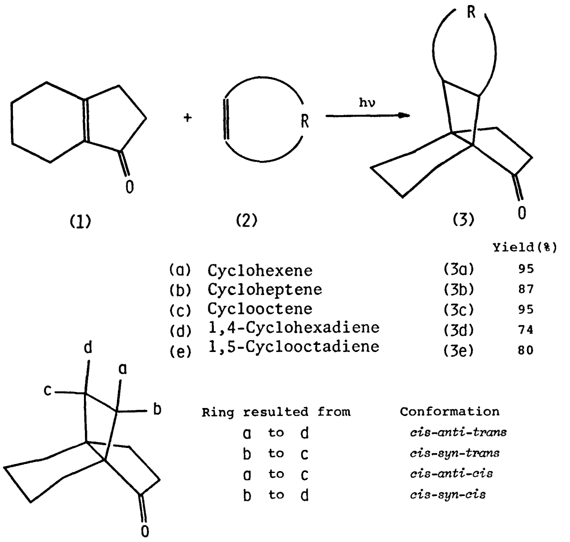
The X-ray molecular structure of the photocycloadduct of bicyclo[4.3.0]non-1(6)-en-7-one with 1,4-cyclohexadiene has been determined. The crystals belong to the monoclinic system, space group $P2_1/c$, $Z=4$, $a=8.108(1)$, $b=8.836(1)$, $c=17.095(1)$ Å, and $\beta=95.38(1)°$ at 22 °C; $a=7.975(1)$, $b=8.760(1)$, $c=16.949(2)$ Å, and $\beta=94.85(1)°$ at −160 °C. The structure was solved by the direct method and refined by the least-squares procedure to $R=0.052$ (22 °C) and 0.053 (−160 °C) for 1777 and 2349 non-zero reflections respectively. The ring junction between the four- and six-membered rings is *trans*. The molecule has the *cis-anti-trans* structure. No essential difference is observed between the molecular structure at 22 °C and −160 °C except for the thermal motions.

Odaira and his coworkers[1)] have found that a photoreaction between bicyclo[4.3.0]non-1(6)-en-7-one (**1**) and aliphatic cycloolefins (**2**) gave a single cycloaddition product (**3**) although four types of isomers were expected to be formed.



			Yield(%)
(a)	Cyclohexene	(3a)	95
(b)	Cycloheptene	(3b)	87
(c)	Cyclooctene	(3c)	95
(d)	1,4-Cyclohexadiene	(3d)	74
(e)	1,5-Cyclooctadiene	(3e)	80

Ring resulted from	Conformation
a to d	*cis-anti-trans*
b to c	*cis-syn-trans*
a to c	*cis-anti-cis*
b to d	*cis-syn-cis*

In order to determine the stereochemistry of the photocycloadducts, X-ray analyses of **3a**,[2)] **3d**, and **3e**[3)] have been carried out. This paper will deal with the molecular structure of **3d** at 22 °C and −160 °C. Interest in the effect of the ring size on the detailed structure of the photocycloadducts also prompted this study.

Experimental

Colorless, square-prismatic crystals were kindly supplied by Professor Y. Odaira and his coworkers of this University.

A Rigaku automated, four-circle diffractometer was used for the measurement of the unit-cell dimensions and the integrated intensities. Graphite-monochromatized Cu *K*α and Mo *K*α radiations were used at 22 °C and −160 °C respectively. The low temperature was attained by the gas-flow method of liquid nitrogen. Crystals with approximate dimensions of 0.25×0.25×0.3 mm (22 °C) and 0.3×0.3×0.3 mm (−160 °C) were used; they were coated with collodion to prevent sublimation. The crystal data are given in Table 1.

Table 1. Crystal data of **3d**

$C_{15}H_{20}O$, monoclinic	*F.W.* 216.3, mp 80—81 °C, space group $P2_1/c$	
	22 °C	−160 °C
a/Å	8.108(1)	7.975(1)
b/Å	8.836(1)	8.760(1)
c/Å	17.095(1)	16.949(2)
β/°	95.38(1)	94.85(1)
V/Å^3	1219.3(1)	1179.8(3)
D_m/g cm^{-3}	1.18	
Z	4	4
D_c/g cm^{-3}	1.18	1.218
μ/cm^{-1}	5.56 (for Cu *K*α)	0.795 (for Mo *K*α)

The unit-cell dimensions were determined by the least-squares fit of 2θ values of high-angle reflections.

The intensity data were collected by the θ-2θ scan technique at a 2θ rate of 2° min^{-1}. The scan widths were $\Delta 2\theta = (2.0+0.28\tan\theta)°$ at 22 °C for Cu *K*α and $(2.0+0.70\tan\theta)°$ at −160 °C for Mo *K*α. The backgrounds were counted for 10 s before and after the scan of each peak. Totals of 1808 ($\theta \leqq 120°$ at 22 °C, Cu *K*α) and 2730 ($\theta \leqq 55°$ at −160 °C, Mo *K*α) unique data were collected, of which 83 (22 °C) and 381 (−160 °C) less than $3\sigma(F)$ were classed as unobserved. Three standard reflections (400, $1,1,\overline{10}$, and 040) were measured at every 60 reflections at both temperatures; no intensity decrease was observed during the data collection. Corrections for the usual Lp effect was carried out, but not for absorption [μ(Cu *K*α)=5.56 and μ(Mo *K*α)=0.795 cm^{-1}].

Structure Solution and Refinement

The structure was solved by the direct method with the *MULTAN* program,[4)] using intensity data obtained at 22 °C. All the non-hydrogen atoms appeared clearly in the E map based on the set of signs with the highest figure of merit. All the hydrogen atoms were located by difference synthesis. The structure was refined

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TABLE 2. FINAL ATOMIC PARAMETERS, WITH e.s.d.'s IN PARENTHESES

(a) Non-hydrogen atoms (×10^4).

Anisotropic temperature factor (×10^4) of the form: $\exp[-\{\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+\beta_{12}hk+\beta_{13}hl+\beta_{23}kl\}]$.

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
22 °C									
O	5131(2)	2205(2)	2171.1(9)	326(4)	247(3)	409(6)	68(6)	−85(3)	−56(2)
C(1)	6915(2)	2498.8(19)	3374.6(9)	133(3)	100(2)	240(6)	1(4)	8(2)	−4.8(19)
C(2)	6823(2)	1185.9(19)	3992.7(10)	128(3)	97(2)	299(6)	−9(4)	4(2)	0.7(20)
C(3)	7734(2)	−307(2)	4060.6(12)	167(3)	101(3)	461(9)	18(5)	−10(3)	−2(2)
C(4)	7838(2)	−737(2)	4918.3(13)	149(3)	126(3)	544(10)	−27(5)	−26(3)	52(3)
C(5)	7511(2)	158(3)	5502.3(12)	166(4)	177(3)	393(8)	−94(6)	−19(3)	73(3)
C(6)	7043(3)	1816(3)	5432.4(11)	210(4)	170(3)	254(7)	−77(6)	12(3)	16(2)
C(7)	7450(2)	2303.9(20)	4623.9(10)	144(3)	112(3)	252(6)	−35(4)	11(2)	3.5(20)
C(8)	6754(2)	3602.2(19)	4082.8(9)	135(3)	92(2)	254(6)	−19(4)	12(2)	−6.5(19)
C(9)	7707(3)	5086(2)	4077.5(12)	216(4)	111(3)	385(8)	−80(5)	−10(3)	−5(2)
C(10)	9319(3)	4970(3)	3692.9(14)	206(4)	185(4)	523(10)	−180(7)	0(3)	49(3)
C(11)	9055(3)	4226(3)	2893.5(13)	196(4)	232(4)	456(9)	−62(7)	56(3)	60(3)
C(12)	8489(3)	2606(3)	2964.5(12)	180(4)	196(4)	316(7)	30(6)	52(3)	8(3)
C(13)	5345(3)	2667(2)	2840.8(11)	191(4)	116(3)	318(7)	−25(5)	−28(2)	−4(2)
C(14)	4088(2)	3523(2)	3253.9(13)	143(3)	152(3)	525(10)	38(5)	−21(3)	16(3)
C(15)	4862(2)	3857(2)	4077.7(12)	157(3)	126(3)	448(8)	46(5)	38(3)	−11(3)
−160 °C									
O	5136.8(17)	2247.6(15)	2148.8(7)	127(2)	95.1(19)	16.3(4)	20(3)	−32.2(15)	−17.3(15)
C(1)	6915.8(18)	2555.3(16)	3370.0(8)	53(2)	39.4(17)	9.5(5)	−0(3)	1.3(15)	−2.6(14)
C(2)	6819.4(18)	1218.1(17)	3985.8(9)	51(2)	41.1(18)	13.1(5)	−2(3)	1.1(16)	2.1(15)
C(3)	7768.7(19)	−282.6(18)	4043.4(10)	61(2)	42.4(18)	19.0(6)	4(3)	−5.3(18)	−3.4(17)
C(4)	7869.9(19)	−734.3(19)	4911.5(10)	51(2)	54.0(19)	22.8(6)	−14(3)	−13.7(19)	21.6(18)
C(5)	7495.5(20)	154(2)	5508.4(10)	63(2)	76(2)	15.5(5)	−40(4)	−9.0(18)	28.9(18)
C(6)	6985(2)	1820.6(19)	5441.3(9)	79(2)	67(2)	11.2(5)	−32(4)	3.7(17)	−0.7(17)
C(7)	7426.6(18)	2341.1(17)	4634.0(9)	56(2)	45.1(18)	11.2(5)	−11(3)	1.5(16)	1.3(15)
C(8)	6715.0(18)	3653.1(17)	4090.3(8)	54(2)	39.9(17)	10.4(5)	1(3)	5.2(15)	−0.6(15)
C(9)	7663(2)	5169.5(18)	4095.3(9)	83(2)	45.0(19)	14.6(5)	−22(4)	−5.0(18)	−3.4(16)
C(10)	9321(2)	5085.7(20)	3709.6(10)	76(2)	65(2)	20.4(6)	−54(4)	−1.4(20)	15.6(19)
C(11)	9074(2)	4348(2)	2893.0(10)	78(3)	82(2)	17.7(6)	−21(4)	19.7(20)	19.8(19)
C(12)	8532.7(20)	2684.8(19)	2962.7(9)	69(2)	70(2)	13.1(5)	13(4)	15.7(17)	3.8(17)
C(13)	5331.5(20)	2714.3(18)	2822.8(9)	72(2)	48.2(19)	14.1(5)	−16(3)	−9.7(17)	5.3(16)
C(14)	4026.1(19)	3584.3(19)	3243.2(10)	55(2)	60(2)	21.3(6)	4(4)	−10.9(18)	1.5(18)
C(15)	4781.6(19)	3879.1(18)	4088.7(10)	57(2)	52.2(19)	18.2(6)	16(3)	12.6(18)	−4.5(17)

(b) Hydrogen atoms (×10^3).

Atom	x	y	z	B/Å^2	Atom	x	y	z	B/Å^2
22 °C					−160 °C				
H(2)	560(2)	97.0(19)	405.0(9)	3.1(4)	H(2)	558.2(19)	98.4(18)	404.0(9)	0.6(3)
H(3A)	715(3)	−109(2)	372.7(12)	5.8(5)	H(3A)	719(2)	−112(2)	371.2(10)	1.5(3)
H(3B)	889(2)	−27(2)	391.1(12)	5.0(5)	H(3B)	894(2)	−18(2)	387.9(11)	2.1(4)
H(4)	823(3)	−183(3)	506.7(12)	6.2(5)	H(4)	826(2)	−180(2)	503.3(11)	2.1(4)
H(5)	762(3)	−22(3)	602.7(12)	5.9(5)	H(5)	758(2)	−25(2)	603.6(11)	2.0(4)
H(6A)	585(3)	191(3)	548.5(12)	6.0(5)	H(6A)	576(2)	192(2)	549.7(10)	1.5(4)
H(6B)	766(3)	240(3)	587.1(13)	6.5(6)	H(6B)	760(2)	238(2)	588.4(11)	1.6(4)
H(7)	873(2)	232(2)	463.6(10)	3.9(4)	H(7)	869(2)	238(2)	463.8(11)	1.5(4)
H(9A)	698(3)	591(2)	378.0(12)	5.8(5)	H(9A)	694(2)	596(2)	380.1(11)	2.3(4)
H(9B)	789(3)	544(2)	461.9(12)	5.7(5)	H(9B)	787(2)	552(2)	465.0(10)	1.5(4)
H(10A)	979(3)	600(3)	366.0(14)	7.0(6)	H(10A)	978(2)	614(2)	366.6(11)	2.3(4)
H(10B)	1021(3)	431(3)	403.1(13)	6.5(6)	H(10B)	1019(2)	447(2)	404.2(11)	2.0(4)
H(11A)	810(3)	483(2)	254.2(12)	5.7(5)	H(11A)	817(2)	493(2)	255.0(12)	2.4(4)
H(11B)	1011(3)	417(3)	261.4(14)	7.5(6)	H(11B)	1012(3)	438(2)	259.6(12)	2.5(4)
H(12A)	828(3)	212(2)	244.3(12)	5.3(5)	H(12A)	837(2)	218(2)	243.4(11)	2.1(4)
H(12B)	945(3)	198(3)	327.9(12)	5.6(5)	H(12B)	948(2)	210(2)	328.2(11)	1.8(4)
H(14A)	382(3)	441(3)	297.4(14)	7.2(6)	H(14A)	383(3)	452(3)	295.6(12)	3.0(5)
H(14B)	300(3)	292(3)	322.1(14)	7.4(6)	H(14B)	296(2)	302(2)	321.4(11)	2.1(4)
H(15A)	457(3)	488(2)	420.7(12)	5.2(5)	H(15A)	451(2)	490(2)	426.9(11)	1.7(4)
H(15B)	439(3)	320(2)	448.0(12)	5.3(5)	H(15B)	432(2)	316(2)	447.1(11)	1.8(4)

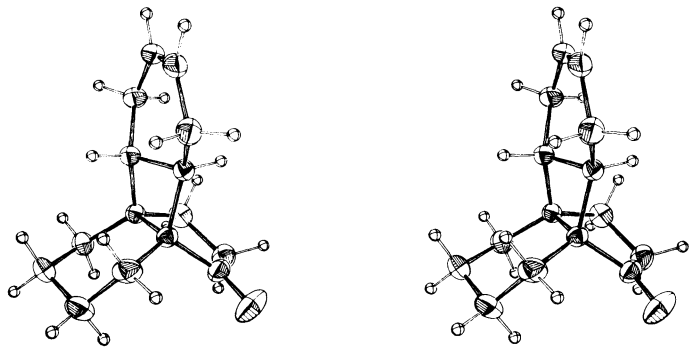

Fig. 1. An *ORTEP* drawing[9] of the molecule. Non-hydrogen atoms are represented by thermal ellipsoids at 20% probability level.

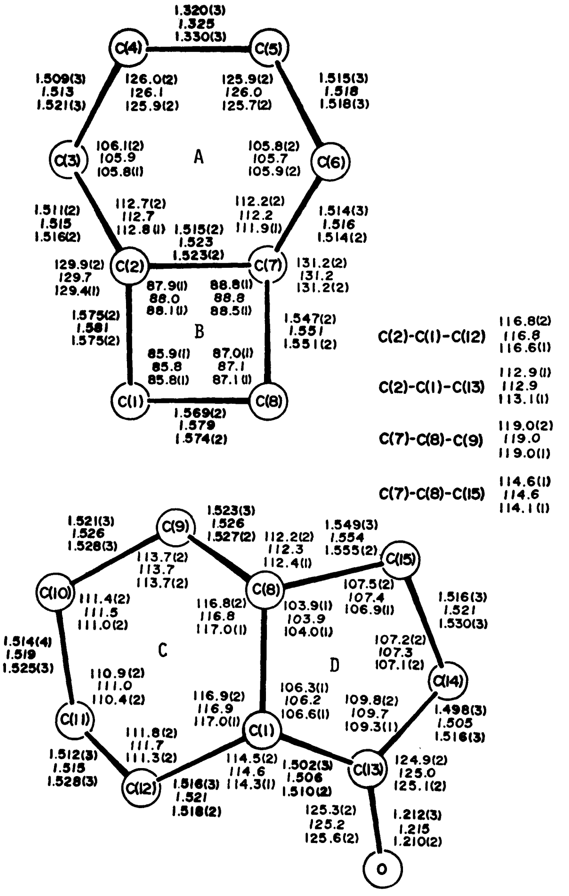

Fig. 2. Bond lengths and bond angles with e.s.d.'s in parentheses.
Upper figure: room temperature structure (22 °C). Middle figure: as above, corrected for the thermal motion by the rigid body approximation.[8] Lower figure: low temperature structure (−160 °C).

with the weight unity by means of the block-diagonal least-squares procedure (*HBLS*-V program[5]). Anisotropic and isotropic thermal parameters were applied for non-hydrogen and hydrogen atoms respectively. The final R value was 0.052 for 1725 non-zero (0.055 for all 1808) reflections.

The anisotropic refinement of the low-temperature structure was started by using the atomic parameters obtained by the use of the room-temperature data. The locations of all the hydrogen atoms were confirmed by the difference systhesis, the parameters of which were refined isotropically. The final R value was 0.053 for 2349 non-zero (0.070 for all 2730) reflections. The weighting schemes used were $w=(\sigma^2(F_o)+a|F_o|+b|F_o|^2)^{-1}$ for $|F_o|>0$ and $w=c$ for $|F_o|=0$. In the case of $a|F_o|+b|F_o|^2<0$, $w=\sigma^{-2}(F_o)$ was used instead. The values of a, b, and c used at the final refinements were 0.026, −0.000, and 0.564. The computations were carried out on a NEAC 2200-700 computer at Osaka University.

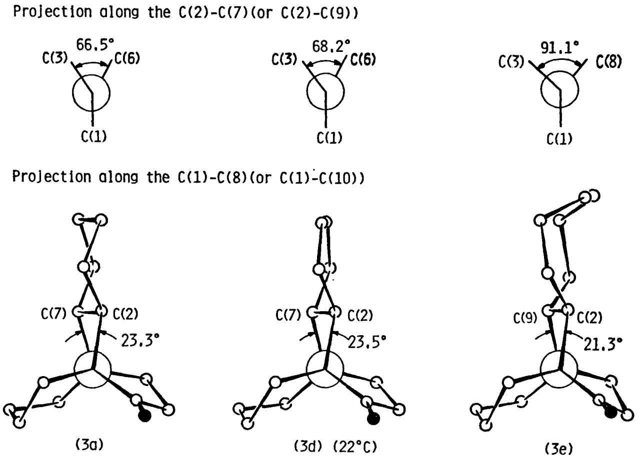

Fig. 3. A view of the three related molecules projected along the C(1)–C(8) or the corresponding C(1)–C(10) bond, and Newman projections along the C(2)–C(7) or the corresponding C(2)–C(9) bond. Oxygen atoms are expressed as dark circles.

The atomic scattering factors for non-hydrogen atoms were taken from Hanson *et al.*,[6] and for hydrogen, from Stewart *et al.*[7] The final atomic parameters are listed in Table 2.[†††]

Results and Discussion

Molecular Structure. A perspective view of the molecule is shown in Fig. 1, while the bond lengths and bond angles are given in Fig. 2.

No essential difference is observed between the molecular structures at 22 °C and −160 °C except for the

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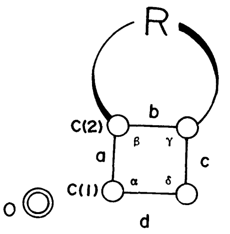
[†††] The tables of the structure factors (22 °C and −160 °C) are kept as Document No. 8005 at the Chemical Society of Japan.

Table 3. Structure of the cyclobutane ring moieties in three similar compounds
(e.s.d.'s in parentheses) Lower figures are those corrected for thermal vibrations.

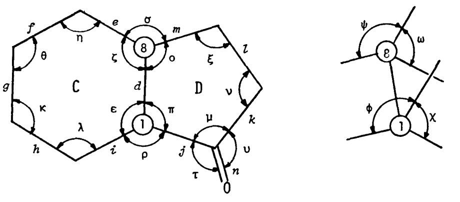


	$C_{15}H_{22}O$ (**3a**)	$C_{15}H_{20}O$ (**3d**, 22 °C) (Present study)	$C_{17}H_{24}O$ (**3e**)
Length [l/Å]			
a	1.571(2) *1.576*	1.575(2) *1.581*	1.581(3) *1.587*
b	1.524(2) *1.531*	1.547(2) *1.523*	1.550(3) *1.555*
c	1.552(2) *1.557*	1.547(2) *1.551*	1.550(3) *1.556*
d	1.573(2) *1.581*	1.569(2) *1.579*	1.549(3) *1.559*
Angle [ϕ/°]			
α	86.1(1) *86.2*	85.9(1) *85.8*	86.9(1) *87.0*
β	88.1(1) *88.1*	87.9(1) *88.0*	88.0(1) *87.9*
γ	88.5(1) *88.6*	88.8(1) *88.8*	88.2(1) *88.2*
δ	87.0(1) *87.0*	87.0(1) *87.1*	88.9(1) *88.9*
Dihedral angle [ϕ/°]	33.9	34.3	30.1

thermal motions. The room-temperature structure shows a shortening of the bond lengths far from the center of the molecule due to the thermal motion of atoms. This shortening could be rather well corrected by the rigid body approximation of the molecule,[8] and the corrected values can be compared with those of the low-temperature structure (Fig. 2). However, some discrepancies are seen in the bond lenths of C(11)–C(12), C(13)–C(14), and C(14)–C(15). This may be due to the non-rigidity of the molecule.

The most distinctive feature of the molecular structure is that the A/B ring junction is *trans*, while the photocycloadduct has the *cis-anti-trans* structure (Scheme). The present compound has a structure very similar to those of **3a** and **3e** except for the local structure of the A ring. The similarity and difference in the room-temperature structures of the three related molecules may clearly be seen in Fig. 3. The torsional angle about the C(2)–C(7) bond in the present molecule [68.2°] is similar to that in **3a** [66.5°], but they are much smaller than that about the corresponding C(2)–C(9) bond in **3e** [91.1°]. This can be interpreted as being due to the size difference of the A ring. The torsional angle about the C(1)–C(8) bond [23.5°] is equal to that in **3a** [23.3°]; these values are slightly larger than that about the corresponding C(1)–C(10) bond in **3e** [21.3°].

Table 4. Structure of the C and D ring moieties in three similar compounds
(e.s.d.'s in parentheses)



	$C_{15}H_{22}O$ (**3a**)	$C_{15}H_{20}O$ (**3d**, 22 °C) (Present study)	$C_{17}H_{24}O$ (**3e**)
Length [l/Å]			
e	1.522(2)	1.523(3)	1.523(3)
f	1.518(3)	1.521(3)	1.496(4)
g	1.523(3)	1.514(4)	1.506(4)
h	1.517(3)	1.512(3)	1.518(4)
i	1.522(2)	1.516(3)	1.515(3)
j	1.506(2)	1.502(3)	1.507(3)
k	1.511(2)	1.498(3)	1.487(3)
l	1.523(3)	1.516(3)	1.509(4)
m	1.553(2)	1.549(3)	1.557(3)
n	1.210(1)	1.212(3)	1.213(3)
Angle [ϕ/°]			
ε	117.1(1)	116.9(2)	116.2(2)
ζ	116.0(1)	116.8(2)	117.6(2)
η	113.4(2)	113.7(2)	113.8(2)
θ	110.9(2)	111.4(2)	111.1(2)
κ	110.7(2)	110.9(2)	110.6(2)
λ	111.6(1)	111.8(2)	112.1(2)
μ	109.2(1)	109.8(2)	109.0(2)
ν	106.9(1)	107.2(2)	106.5(2)
ξ	106.5(3)	107.5(2)	106.0(2)
o	103.6(1)	103.9(1)	103.1(2)
π	106.4(1)	106.3(1)	107.1(2)
ρ	114.7(1)	114.5(2)	114.3(2)
σ	112.5(2)	112.2(2)	111.7(2)
τ	125.7(1)	125.3(2)	125.9(2)
υ	126.1(2)	124.9(2)	125.1(2)
ϕ	115.2(1)	116.8(2)	116.0(2)
χ	113.8(1)	112.9(1)	113.2(2)
ψ	120.0(1)	119.0(2)	118.8(2)
ω	114.1(1)	114.6(1)	114.0(2)

The central cyclobutane ring (B Ring) is puckered, and such a puckering is also observed in the other two compounds; these structures are compared in Table 3. The longest C–C bond, *a* [1.575(2) Å], is much longer than the normal C–C single bond. The *c* bond [1.547(2) Å], the length of which is equal through the three compounds, is slightly longer than the usual length. No significant difference is observed between the corresponding bond lengths nor bond angles in **3a** and **3d**. However, some deviations from these are seen in **3e**; the *b* bond [1.544(3) Å] is longer, *d* [1.549(3) Å] is shorter, and the α [86.9(1)°] and γ

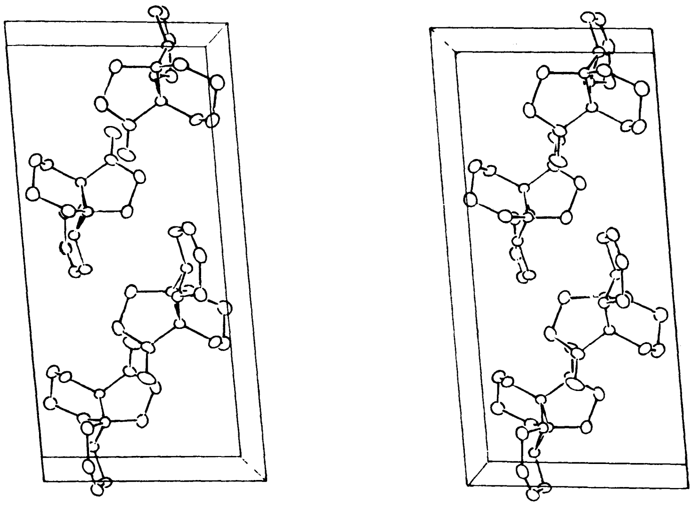
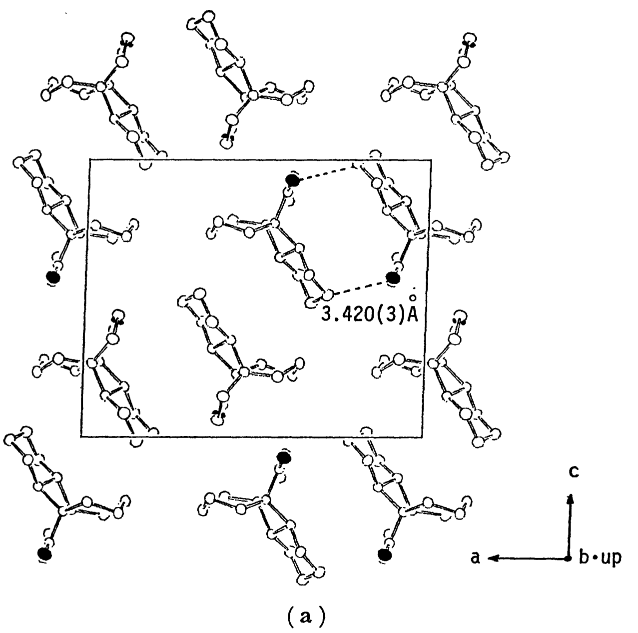
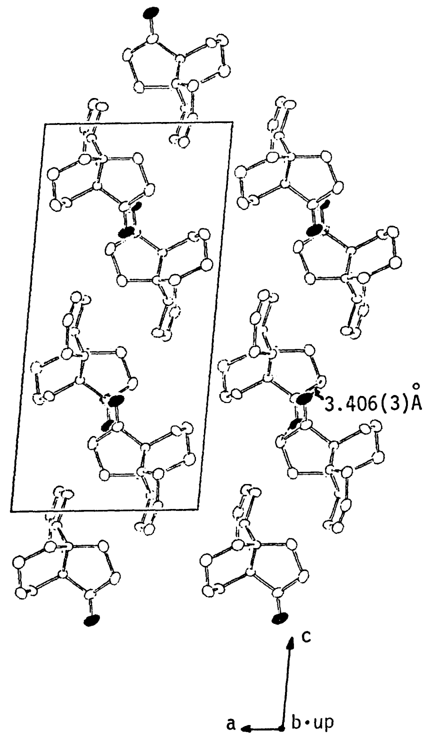


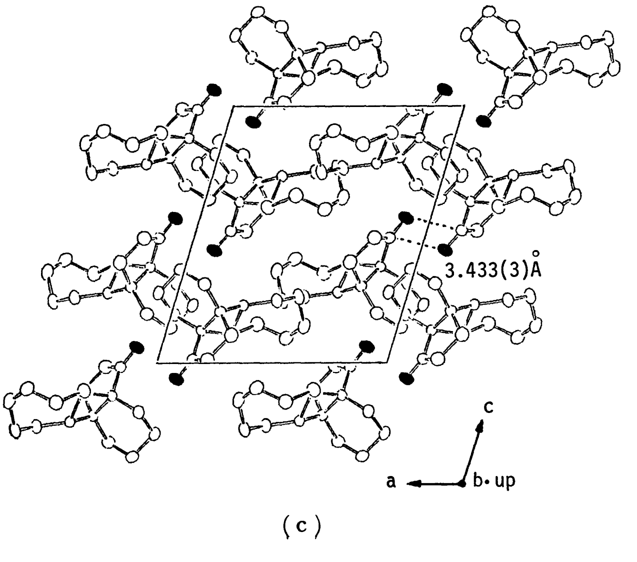
Fig. 4. A stereoscopic drawing of the crystal structure of **3d** (Present study).



(a)



(b)



(c)

Fig. 5. Crystal structure of the three related molecules viewed down along the b axis.
(a) **3a**, (b) **3d**(Present study), (c) **3e**.
Oxygen atoms are expressed as dark ellipses.

[88.9(1)°] angles are larger than the corresponding bond lengths and angles in the other two compounds.

Almost all the bond lengths and bond angles in Rings C and D in the present compound (**3d**) are equal to the corresponding lengths and angles in **3a** and **3e**, within the limits of error, except for the bond lengths, f and k, and bond angles involving the bridgehead carbon atoms, ζ, ϕ, and χ (Table 4).

Crystal Structure. A stereoscopic drawing[9] of the molecular packing is given in Fig. 4. Some close atomic contacts are observed between molecules related

Table 5. Short intermolecular atomic contacts [l/Å] (less than 3.6 Å), with e.s.d.'s in parentheses

	22 °C		−160 °C
3d (Present study)			
C(6)···O[a]	3.584(3)		3.455(2)
O···C(9)[b]	3.526(3)		3.457(2)
O···C(14)[b]	3.406(3)		3.356(2)
key: a)	x,	$0.5-y$,	$0.5+z$
b)	$1-x$,	$-0.5+y$,	$0.5-z$
3a			
O···C(4)[a]	3.420(3)		
O···C(14)[b]	3.535(3)		
key: a)	$0.5-x$,	$-0.5+y$,	$1.5-z$
b)	$1-x$,	$-y$,	$2-z$
3e			
O···C(16)[a]	3.433(3)		
key: a)	$-x$,	$-y$,	$1-z$

by a two-fold screw axis. The carbon atom next to the carbonyl group of the D ring, C(6), has the closest contact between the carbonyl oxygen atoms of the adjacent molecules along the 2_1 axis [3.406(3) Å at 22 °C]. The other close contacts are listed in Table 5.

The packings of the **3a** and **3d** molecules are compared with that of the present crystal in Fig. 5.[9] In the **3a** crystal, the carbonyl oxygen atoms have close atomic contacts between atoms in the adjacent molecules related by the 2_1 axis and the center of symmetry (Table 5). In the **3e** crystal, close intermolecular atomic distances are noticed between the two adjacent molecules related by the center of symmetry (Fig. 5(c) and Table 5). Throughout the three related compounds, it can be concluded that the carbonyl oxygen atoms of the D ring play an important role in the packing of molecules.

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