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The Crystal and Molecular Structure of a Tetracyclic Diketone, $C_{13}O_2H_{16}$*

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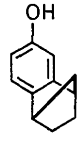
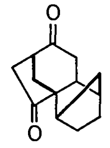
Tetracyclic diketone $C_{13}O_2H_{16}$, m.p. 124°C, was prepared by K. Wiesner, A. Deljac and T.Y.R. Tsai in the process of developing a new stereospecific method for the synthesis of bridged terpenoids. The crystals belong to the space group $C2/c$ with eight molecules in a unit cell of dimensions: $a=16{\cdot}214$ (4), $b=6{\cdot}194$ (4), $c=24{\cdot}198$ (4) Å and $\beta=119{\cdot}06$ (3)°. The data, consisting of 1563 observed reflexions, were collected with a Picker automatic and a General Electric XRD-5 manual diffractometer. The structure was solved by the symbolic addition procedure for phase determination for centrosymmetric space groups, and refined by block-diagonal least-squares cycles to a final R value of 0·042. The molecules are held by the dipole attraction between carbonyl groups.

Introduction

New stereospecific methods for the conversion of phenol (I) to two tetracyclic diketones (II and III) were developed by K. Wiesner and his collaborators. These methods are expected to be useful, not only in the synthesis of songorine (IV), but also in the synthesis of many other bridged terpenoids.



I



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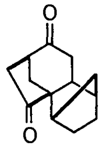

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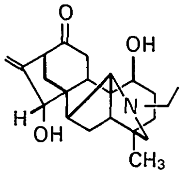

IV

While the stereochemistry of the cyclization of (I) leading to the formation of one or the other tetracyclic diketone was well controlled, it was not known which one corresponded to the structural formula II. The assignment of stereochemistry by a chemical method would be difficult; moreover, a direct comparison with the degradation product of songorine was not possible because of a very limited supply of this alkaloid. In view of the considerable synthetic value of Wiesner's method, an X-ray analysis of one of the diketones was warranted.

The crystals of both tetracyclic diketones were obtained from K. Wiesner. The compound of m.p. 124°C was chosen and was found to have the structure II and its enantiomer. By analogy, the structure of the second diketone, of m.p. 108–109°C, which was characterized by infrared, n.m.r. and mass spectrometry, can be assumed to be III.

The syntheses of the diketones and the result of this investigation have been reported by Wiesner, Deljac, Tsai & Przybylska (1970).

Experimental

Tetracyclic diketone, $C_{13}O_2H_{16}$, m.p. 124°C, F.W.= 204·26.

Monoclinic, $a=16{\cdot}214$ (4), $b=6{\cdot}194$ (4), $c=24{\cdot}198$ (4) Å, $\beta=119{\cdot}06$ (3)°.

$V=2124{\cdot}3$ Å^3, $Z=8$.

$D_m=1{\cdot}26$ g.cm^{-3}, by flotation in a mixture of toluene and carbon tetrachloride;

$D_x=1{\cdot}28$ g.cm^{-3}. $F(000)=880$.

Systematic absences from precession photographs: hkl, when $h+k$ is odd and $h0l$, when l is odd. Space group $C2/c$. $\mu(\mathrm{Cu})=6{\cdot}8$ cm^{-1}.

The crystals were recrystallized from ethyl acetate. They were elongated along the b axis with (001) faces

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prominently developed. The unit-cell dimensions were derived from the 2θ measurements of the high-order axial reflexions using Cu radiation ($K\alpha_1$ and $K\alpha_2$). The data were collected at room temperature with a Picker four-circle automatic diffractometer and supplemented with the General Electric XRD-5 manual diffractometer. The crystal used for reflexions of $2\theta \leq 130°$ had the dimensions $0{\cdot}24 \times 0{\cdot}24 \times 0{\cdot}14$ mm, and a larger one ($0{\cdot}36 \times 0{\cdot}47 \times 0{\cdot}36$ mm) was mounted for the G.E. data, $130 \leq 2\theta \leq 162°$. Both crystals had **b** along the φ axis. Of the 2310 accessible reflexions, 1563 (68%) had significant counts. The threshold was taken as a net count of 150 or 10% of the background, whichever was higher. The $\theta/2\theta$ scanning method was used with Ni-filtered Cu radiation at a take-off angle of 6°. The scan ranges varied from 2 to 4° in 2θ and the speed was 2° per min. Background measurements for 20 sec were taken at each end of the scans. High intensities were remeasured with lower current settings and if necessary attenuated with additional thickness of Ni foil. Four different standard reflexions were used for scaling; they were measured after every 30–50 reflexions. There was no evidence of crystal deterioration during the collection of the data.

The intensities were corrected for Lorentz and polarization factors but absorption corrections were considered unnecessary, the difference between the maximum and the minimum transmission coefficients $e^{-\mu t}$ being 0·06 for each crystal.

Solution of the structure and its refinement

The structure was solved by the symbolic addition method for centrosymmetric space groups (Hauptman & Karle, 1959; Karle & Karle, 1966). The scale factor and the overall isotropic temperature factor ($B = 4{\cdot}0$ Å^2) were evaluated by Wilson's method (1942). The statistics of the E's for this compound agreed well with the theoretical values, given in parentheses, for centrosymmetric space groups (Karle, Dragonette & Brenner, 1965):

$\langle\lvert E\rvert\rangle = 0{\cdot}81$	(0·80),
$\langle\lvert E\rvert^2\rangle = 1{\cdot}05$	(1·00),
$\langle\lvert E^2-1\rvert\rangle = 1{\cdot}03$	(0·97);
$\lvert E\rvert > 3$ 0·7%	(0·3%),
$\lvert E\rvert > 2$ 5·0%	(5·0%),
$\lvert E\rvert > 1$ 31·9%	(32·0%).

In addition to two origin defining reflexions (937 and 2,2,17), two other reflexions (12,0,0 and 3,3,15) had to have their phases assigned before the program could be successfully applied. Four different combinations were therefore tried and the results examined. The best set led to 283 phase assignments with E values $> 1{\cdot}5$. These reflexions were used to evaluate a three-dimensional E map which revealed the complete structure of the molecule. At this stage all the atoms were treated as carbon atoms in the calculation of structure factors and an R value of 0·33 was obtained.

One least-squares cycle reduced the R value to 0·22. A Fourier synthesis was then carried out to confirm the placement of the oxygen atoms, and the introduction of the oxygen scattering factors and of the anisotropic refinement of the thermal parameters for all the fifteen non-hydrogen atoms resulted in an R of 0·11 after three more cycles of refinement. The coordinates of the hydrogen atoms were then calculated assuming tetrahedral angles and C–H bonds of 1·07 Å. Eight additional cycles of refinement, in which the hydrogen atoms were refined isotropically, reduced the reliability index $R = \sum|\Delta F|/\sum|F_o|$, to the final value of 0·042, which was calculated omitting the unobserved reflexions. The final shifts for the non-hydrogen atoms did not exceed $\frac{1}{5}$ of their e.s.d. values, whereas those for the hydrogen atoms were generally larger but none greater than $\frac{1}{3}$ of the corresponding e.s.d.'s.

The scattering factors of Hanson, Herman, Lea & Skillman (1964) were used for carbon and oxygen atoms and those of Stewart, Davidson & Simpson (1965) for hydrogen atoms. The refinement was carried out by block-diagonal approximation of the least-squares method, minimizing the expression $\sum\omega(|F_o| - |F_c|)^2$. Total shifts were used throughout, except for the last few cycles, when shift factors of 0·5 and 0·7 were applied.

The weighting scheme used originally was that of Mills & Rollett (1961), $\sqrt{\omega} = 1/\{1 + [(|F_o| - P_2)/P_1]^2\}^{1/2}$ where P_1 and P_2 were taken as 3·00 and 4·50 respectively. When the anisotropic refinement was introduced a different weighting procedure was adopted to obtain the most uniform values of mean $\omega(\Delta F)^2$ over various ranges of observed structure factors and $\sin^2\theta$. It was of the form

$$\sqrt{\omega} = 1/[P_1 + |F_o| + P_2|F_o|^2]^{1/2}$$

with

$$P_1 = 7{\cdot}40 \quad \text{and} \quad P_2 = 0{\cdot}01\,.$$

In Table 1 the final fractional coordinates of the carbon and oxygen atoms and in Table 2 their vibrational parameters are listed. The positional parameters and temperature factors of the hydrogen atoms are presented in Table 3.

Table 1. *Final fractional coordinates* ($\times 10^4$) *and their estimated standard deviations*

	x/a	y/b	z/c
C(1)	−2046 (1)	9406 (3)	3265 (1)
C(2)	−1274 (1)	9635 (3)	3096 (1)
C(3)	−315 (1)	8947 (3)	3642 (1)
C(4)	−375 (1)	6765 (3)	3964 (1)
C(5)	−1360 (1)	5748 (3)	3586 (1)
C(6)	−1978 (1)	7447 (3)	3660 (1)
C(7)	−1419 (1)	7969 (3)	4372 (1)
C(8)	−400 (1)	7414 (3)	4564 (1)
C(9)	436 (1)	5434 (3)	3986 (1)
C(10)	1395 (2)	6458 (4)	4421 (1)
C(11)	1410 (2)	8486 (5)	4054 (1)
C(12)	446 (2)	8394 (4)	3454 (1)
C(13)	318 (2)	5971 (4)	3330 (1)
O(14)	242 (1)	7496 (2)	5097 (1)
O(15)	−2682 (1)	10704 (3)	3094 (1)

Table 2. *Vibration tensor components and their e.s.d.'s* ($Å^2 \times 10^3$)

$$\text{T.F.} = \exp\{-2\pi^2(U_{11}a^{*2}h^2 + U_{22}b^{*2}k^2 + U_{33}c^{*2}l^2 + 2U_{23}b^*c^*kl + 2U_{13}a^*c^*hl + 2U_{12}a^*b^*hk)\}.$$

	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{13}$	$2U_{12}$
C(1)	46 (1)	57 (1)	41 (1)	3 (2)	24 (2)	8 (2)
C(2)	60 (1)	49 (1)	42 (1)	18 (2)	40 (2)	1 (2)
C(3)	50 (1)	45 (1)	38 (1)	1 (1)	41 (2)	−14 (2)
C(4)	43 (1)	38 (1)	33 (1)	−2 (1)	32 (1)	−5 (1)
C(5)	50 (1)	41 (1)	42 (1)	−7 (2)	34 (2)	−18 (2)
C(6)	40 (1)	54 (1)	49 (1)	3 (2)	36 (2)	−9 (2)
C(7)	55 (1)	60 (1)	46 (1)	4 (2)	57 (2)	0 (2)
C(8)	49 (1)	35 (1)	35 (1)	4 (1)	39 (1)	−4 (1)
C(9)	53 (1)	49 (1)	51 (1)	−1 (2)	49 (2)	9 (2)
C(10)	46 (1)	77 (1)	60 (1)	10 (2)	48 (2)	9 (2)
C(11)	51 (1)	90 (2)	69 (1)	14 (3)	59 (2)	−19 (2)
C(12)	55 (1)	77 (1)	47 (1)	15 (2)	55 (2)	−11 (2)
C(13)	63 (1)	87 (2)	54 (1)	−23 (2)	69 (2)	7 (2)
O(14)	62 (1)	50 (1)	33 (1)	1 (1)	29 (1)	−1 (1)
O(15)	71 (1)	89 (1)	81 (1)	60 (2)	68 (2)	68 (2)

Table 3. *Fractional coordinates* ($\times 10^3$) *with e.s.d.'s and temperature factors for the hydrogen atoms*

The number of the parent atom is obtained by omitting the last digit.

	x/a	y/b	z/c	B ($Å^2$)
H(21)	−125 (1)	1117 (3)	297 (1)	4·1
H(22)	−143 (1)	865 (4)	273 (1)	4·3
H(31)	−10 (1)	1016 (3)	398 (1)	3·3
H(51)	−137 (1)	435 (3)	378 (1)	4·8
H(52)	−155 (1)	547 (3)	314 (1)	3·3
H(61)	−260 (1)	697 (4)	353 (1)	4·7
H(71)	−162 (2)	698 (4)	463 (1)	5·8
H(72)	−146 (2)	949 (3)	447 (1)	4·8
H(91)	38 (2)	385 (4)	407 (1)	5·3
H(101)	189 (2)	544 (4)	448 (1)	6·4
H(102)	146 (2)	677 (4)	484 (1)	5·2
H(111)	193 (2)	837 (4)	394 (1)	6·1
H(112)	146 (2)	990 (4)	428 (1)	5·5
H(121)	41 (2)	923 (4)	312 (1)	6·1
H(131)	81 (2)	535 (4)	329 (1)	6·1
H(132)	−30 (1)	557 (3)	296 (1)	4·0

The observed and calculated structure factors are shown in Table 4. The structure factors for the unobserved reflexions were calculated, but they were excluded from the refinement procedure. The agreement summary is given in Table 5.

All the computations were carried out on the IBM/360 system with programs written in Fortran IV by Ahmed, Hall, Pippy & Huber (1966).

Description and discussion of the structure

The molecular structure showing thermal ellipsoids is depicted in Fig. 1. It was drawn using Johnson's (1965) *ORTEP* program, enclosing 50% probability. The examination of these ellipsoids and of Table 2 indicated that the rigid-body approximation as the basis for bond-length corrections may not be appropriate. The atoms C(5) and C(13) vibrate more than the others in the direction of the bridging bonds, also O(15) and the atoms of the ring *C* display comparatively larger thermal motion.

The application of the Schomaker & Trueblood (1968) thermal analysis confirmed this, as the differences for some of the atoms between the observed

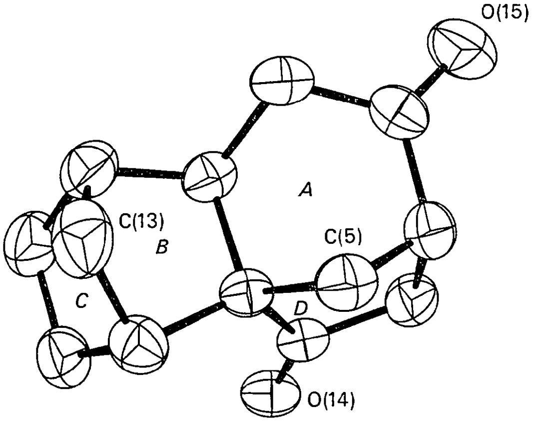

Fig. 1. Perspective view of the molecule of tetracyclic diketone showing thermal ellipsoids.

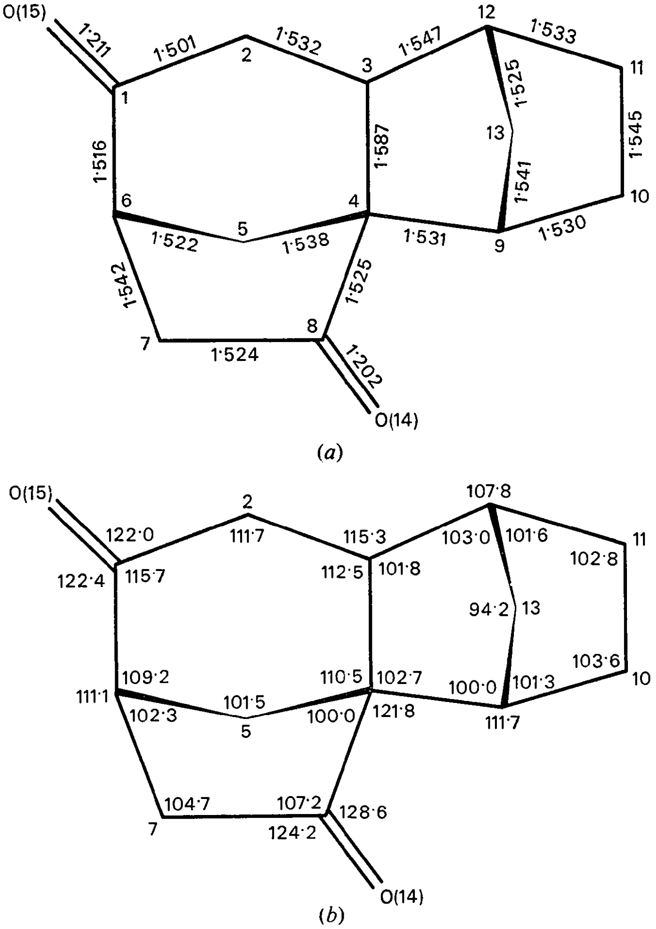

Fig. 2. (*a*) Bond lengths. (*b*) Valency angles, C(3)–C(4)–C(8) = 106·2, C(5)–C(4)–C(9) = 115·3°.

Table 4. *Observed and calculated structure factors* (×10)

Reflexions too weak to be measured are marked with an asterisk.

Table 5. *Agreement summary*

1563 observed reflexions, $|F_o \text{ max}| = 191{\cdot}6$.

Limits	Number
$\|\Delta F\|/\|F_o\| \leq 2R$, or $\|\Delta F\| \leq 1\|F_{\text{th}}\|$	1559
$2R < \|\Delta F\|/\|F_o\| \leq 3R$, or $1\|F_{\text{th}}\| < \|\Delta F\| \leq 2\|F_{\text{th}}\|$	4
747 unobserved reflexions, $\|F_c \text{ max}\| = 6{\cdot}2$	
$\|F_c\| \leq 1{\cdot}0\|F_{\text{th}}\|$	703
$1{\cdot}0\|F_{\text{th}}\| < \|F_c\| \leq 1{\cdot}5\|F_{\text{th}}\|$	44

$R = 0{\cdot}042$, $|F_{\text{th}}|$ = threshold amplitude = 2·2 to 5·3.

U_{ij}'s and those calculated from rigid-body parameters were more than twice the e.s.d.'s of observed U_{ij} values. Moreover, none of the bond corrections exceeded 0·008 Å. On the basis of these results the corrections were not applied.

The bond lengths for the non-hydrogen atoms are listed in Fig. 2(*a*) and the valency angles are shown in Fig. 2(*b*). The estimated standard deviation for bond lengths is 0·003 Å except for C(8)–O(14), for which it is 0·002 Å, and for C(10)–C(11) and C(12)–C(13), for which it is 0·004 Å. The estimated standard deviations for the angles do not exceed 0·20°. The e.s.d.'s were derived from the inverse of the least-squares matrix and since interactions between atoms were not taken into account, and the unobserved reflexions were omitted, they should be considered as underestimated. An increase of 50% would give more realistic values.

The average length for the twelve Csp^3–Csp^3 bonds is 1·539 Å; however, on excluding the large value for the C(3)–C(4) bond, the average of 1·535 Å is obtained. The value given by Sutton (1965) is 1·537 ± 0·005 Å.

The shortening of bonds C(5)–C(6) and C(12)–C(13) to 1·522 and 1·525 Å respectively, is possibly significant, whereas the lengthening of the C(3)–C(4) bond, which is 1·587 Å, is definitely significant. Unusually long bonds between highly substituted carbon atoms are not uncommon, however, and several examples have been cited by Birnbaum & Ferguson (1969).

The C–C bonds adjoining carbonyl groups vary from 1·501 to 1·525 Å, giving an average value of 1·517 Å. The C=O bonds are 1·202 and 1·211 Å and they are in good agreement with Sutton's value of 1·215 ± 0·005 Å.

The angles of the six-membered ring have an average value of 110·2°. The largest angle of 115·7° is at the carbonyl group and the smallest of 101·5° is associated with the five-membered ring. The angles of the five-membered rings average 101·9°. The smallest angle (94·2°) was found, as expected, at the bridging atom C(13) of the bicycloheptane (norbornane) skeleton.

Of the C–C=O angles, one deserves a special comment. The angle C(4)–C(8)=O(14) is considerably larger than others. The increase to 128·6° appears to be due to the repulsion by the hydrogen atom attached to C(10), especially as the distance O(14)···H(102) of 2·39 Å is somewhat shorter than the sum of the van der Waals radii of the two atoms.

The heights of the hydrogen atoms on the electron density difference maps varied from 0·55 to 0·70 e.Å^{-3}. The C–H bond lengths range from 0·93 to 1·04 Å and their e.s.d.'s from 0·02 to 0·03 Å. The least-squares refinement of the hydrogen parameters led to values for C–H bonds consistently lower than the originally assumed 1·07 Å. The average value for CCH and HCH angles is 111·1° and they range from 106·7 to 116·2°.

The six-membered ring is in the boat form, which is considerably flattened at one end (see Fig. 4). The atoms C(2) and C(5) are at 0·52 and 0·84 Å respectively from the plane of the remaining four atoms of the ring. The atoms C(1) and C(4) are situated at 0·045 Å from that plane and C(6) with C(3) are at the same distance on the other side of it. The deviations from the ideal values of torsional angles for the boat form of 60 and 0° are the largest for the C(1)–C(2) and C(1)–C(6) bonds, which are 38·1 and 21·2° respectively.

A plane was also calculated for the atoms C(1), C(6), C(3), C(4), C(10) and C(11). It was found that C(1), C(3) and C(11) lie close to that plane, but the atoms C(6) and C(10) lie at 0·08 and 0·05 Å respectively below it. Atom C(4), on the other hand, is situated at 0·12 Å above the plane. There is therefore a marked puckering of the skeleton at C(4), which is a fully substituted atom.

The three five-membered rings are envelope shaped. Their bond and torsional angles are listed in Table 6 for comparison with the values calculated for two theoretical models. The signs of the torsional angles were determined using the definition of Klyne & Prelog (1960). Their e.s.d.'s are 0·2° (Huber, 1961). The torsional angles for the rings *B* and *C* of the norbornane system are closer to the values for the maximally puckered envelope model for fused ring systems of Brutcher Jr & Bauer Jr (1962). The ring *D* values, however, match better the Pitzer & Donath (1959) form. The greatest distortions from ideal conformations are expected to be present in ring *D*, which is fused to a six-membered ring and which is characterized by the presence of a fully substituted C(4) atom and the carbonyl group.

A close examination of the torsional angles of the norbornane system revealed the presence of *synchro*-twist (Altona & Sundaralingam, 1970), which appears to be caused by the substitution at atoms C(3) and C(4). The torsional angle for C(4)–C(9) is larger than for C(12)–C(3), whereas the corresponding angle for C(9)–C(10) is smaller than for C(11)–C(12). The differences are 10·8 and 1·8° respectively.

The atoms C(3), C(4), C(9) and C(12) of the ring *B* were found to be situated at ±0·04 Å from the plane calculated for these atoms. The corresponding four atoms of the ring *C*, however, C(9), C(10), C(11) and C(12) can be considered as planar, as $\chi^2 = 10{\cdot}3$ and their distances do not exceed 0·005 Å. The atoms C(4), C(6), C(7) and C(8) of the ring *D* are not planar and their distances vary from 0·01 to 0·03 Å.

The atoms C(1), C(2), C(6) and O(15) lie on a plane:

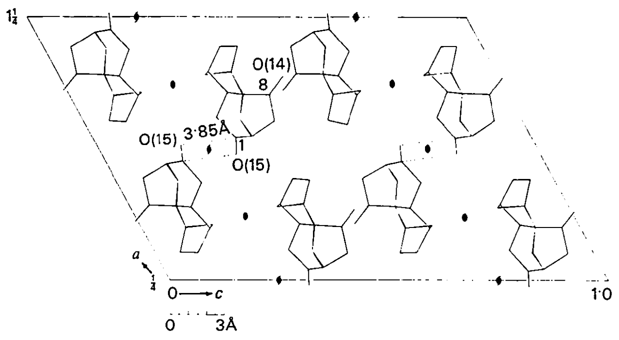

Fig. 3. Packing of the molecules projected down the *b* axis.

$$-0{\cdot}153X'-0{\cdot}531Y-0{\cdot}834Z'=-7{\cdot}754,\quad \chi^2=0{\cdot}26,$$
$$\text{r.m.s.d.}=0{\cdot}0005\,.$$

The atoms C(7), C(8), C(4) and O(14) are also planar. The equation of their plane is:

$$0{\cdot}270X'+0{\cdot}952Y'-0{\cdot}143Z'=1{\cdot}371,\quad \chi^2=0{\cdot}06,$$
$$\text{r.m.s.d.}=0{\cdot}0002\,.$$

X', Y, Z' in the above equations are orthogonal coordinates in Å, where $X'=X-Z\cos\beta^*$ and $Z'=Z\sin\beta^*$.

The structure of tetracyclic diketone projected along the *b* axis is shown in Fig. 3. There are no hydrogen bonds, but the molecules are held together by the electrostatic attraction between carbonyl groups. The packing can be described as consisting of sheets of molecules perpendicular to the *a* axis.

There are two types of dipole–dipole interactions, those between C(8)–O(14) groups related by the centres of symmetry and those between C(1)–O(15) carbonyl groups related to each other by the twofold screw axis parallel to the *b* axis. The O(15)···C(1) distance, shown in Fig. 3, is 3·854 Å (e.s.d. 0·003 Å) and the angles O(15)···C(1)–O(15) and C(1)–O(15)···C(1) are 112·2 and 114·3° respectively (e.s.d. 0·15°).

The anti-parallel arrangement of C(8)–O(14) carbonyl groups is shown in greater detail in Fig. 4. The C···O distances are considerably shorter, being 3·128 and 3·237 Å. The oxygen atoms have contacts of 3·168 and 3·179 Å and the C···C distances are greater than 3·5 Å. The estimated standard deviation for these intermolecular distances is 0·002 Å. The C=O···C angles are close to 100°, but the O···C=O angles are appreciably smaller (80·9 and 76·5°). None of the C···O contacts is smaller than the expected minimum van der Waals distance of 3·1 Å.

A number of interactions between non-hydrogen-bonded carbonyl oxygen atoms and a ring carbonyl carbon atoms with the C=O···C angles close to 157° were described by Bolton (1964). In the case of tetracyclic diketone, the attraction of the dipole which is strongest along the dipole axis is not utilized, probably owing to other aspects of the molecular packing. A similar anti-parallel arrangement between pairs of C≡N groups, related by a centre of symmetry, was found in the structure of 4-cyanopyridine (Laing, Sparrow & Sommerville, 1971).

The closest intermolecular approaches are between the oxygen atoms and hydrogen atoms of the neighbouring molecules and they are 2·55 and 2·57 Å. The closest H···H contact is 2·39 Å. None of these values deviates significantly from the normal van der Waals distances of 2·6 and 2·4 Å, as their e.s.d. values are high (0·02 to 0·03 Å).

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Table 6. *Conformation of cyclopentanes*

The numbering starts at the out-of-plane carbon atom. Calculated values, I, are for the envelope form of Pitzer & Donath (1959) and values II are for the maximally puckered envelope model of Brutcher Jr & Bauer Jr (1961).

		Calc. I	Calc. II	Ring *B*		Ring *C*		Ring *D*	
Bond angles (°)	ω_1	100·4	96·6	94·2		94·2		101·5	
	$\omega_2=\omega_5$	102·4	96·6	103·0,	100·0	101·3,	101·6	102·3,	100·0
	$\omega_3=\omega_4$	105·6	104·3	101·8,	102·7	103·6,	102·8	104·7,	107·2
Torsional angles (°)	$\theta_{1,2}=\theta_{5,1}$	46·1	60·0	−55·2,	+59·1	−55·6,	+56·3	−45·6,	+47·5
	$\theta_{2,3}=\theta_{4,5}$	28·6	36·5	+30·5,	−41·3	+34·5,	−36·3	+25·4,	−31·7
	$\theta_{3,4}$	0·0	0·0	+6·9		+0·8		+4·3	
Distance of atom 1 from the plane of 2–3–4–5 (Å)		0·75	0·91	0·89		0·87		0·72	

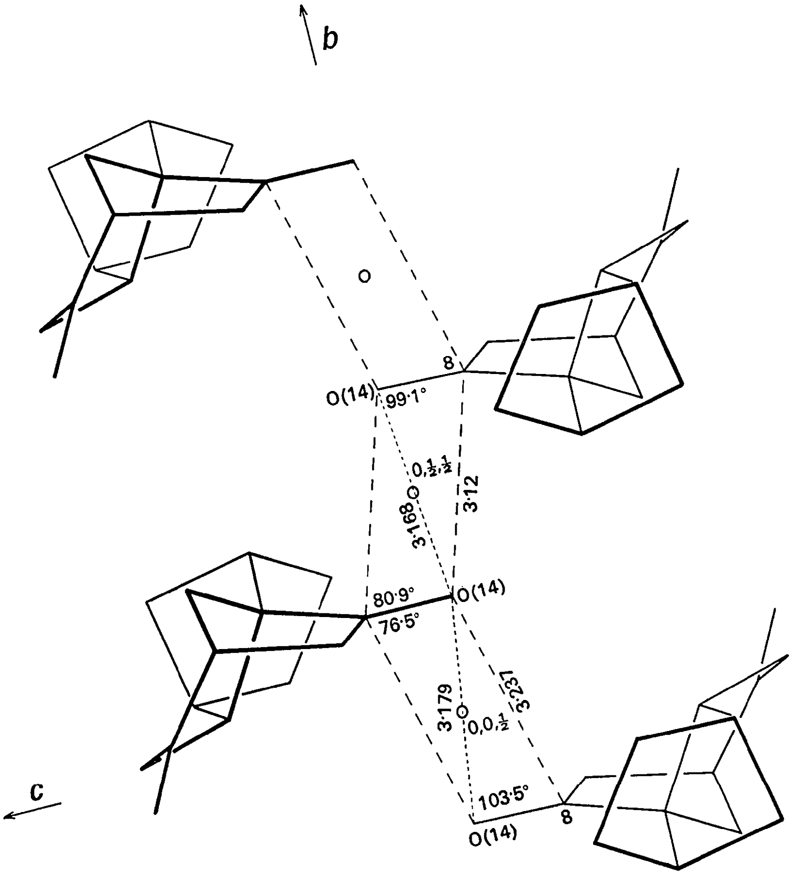

Fig. 4. Parallel arrangement of carbonyl groups.

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