

1584 reflections
243 parameters
H atoms idealized with
riding models

$\Delta\rho_{\max} = 0.34 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$
Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

C1—O1	1.384 (3)	C4—O4	1.437 (3)
C1—O5	1.413 (3)	O4—C1'	1.387 (3)
O1—C7	1.420 (4)	C1'—O5'	1.425 (3)
C1—O1—C7	113.7 (2)	C1—O5—C5	112.0 (2)
C1'—O4—C4	116.2 (2)	C1'—O5'—C5'	112.3 (2)
O5—C1—O1—C7	-77.4 (3)	O5—C5—C6—O6	-54.6 (2)
C1'—O4—C4—C5	-161.3 (2)	O5'—C1'—O4—C4	-88.4 (2)
C1'—O4—C4—C3	78.4 (2)	O5'—C5'—C6'—O6'	57.3 (2)

Table 2. Comparison of methyl β -lactoside (I) with related structures ($^\circ$)

Compound	φ	ψ	ω	ω'
Methyl β -lactoside- CH_3OH^a	-88.4	-161.3	-54.6	57.3
Methyl β -cellobioside- CH_3OH^b	-91.1	-160.7	-55.1	52.4
β -Lactose ^c	-70.9	-131.5	72.6	50.5
α -Lactose- H_2O^d	-94.2	-142.8	63.2	59.4
α -Lactose- $\text{CaCl}_2 \cdot 7\text{H}_2\text{O}^e$	-76.9	-136.7	63.8	59.8
α -Lactose- $\text{CaBr}_2 \cdot 7\text{H}_2\text{O}^f$	-76.0	-134.9	61.9	62.4
<i>N</i> -Acetyl α -lactosamine- H_2O^g	-88.1	-139.5	-56.0	66.8

Notes: (a) this work; (b) Ham & Williams (1970); (c) Hirotsu & Shimada (1974); (d) Fries *et al.* (1971); (e) Cook & Bugg (1973); (f) Bugg (1973); (g) Longchambon *et al.* (1981).

Table 3. Hydrogen-bonding geometry (\AA , $^\circ$)

D—H...A	H...A	D...A	D—H...A
O4'—H4'O...O11 ⁱ	1.87	2.686 (3)	171
O11—H11O...O6	1.93	2.727 (3)	164
O6—H6O...O2 ⁱⁱ	1.94	2.748 (2)	169
O2—H2O...O2 ⁱⁱⁱ	1.96	2.757 (3)	163
O2'—H2'O...O3 ^{iv}	1.96	2.775 (3)	175
O3—H3O...O5'	2.08	2.764 (2)	141
O3'—H3'O...O6 ^{iv}	1.96	2.740 (2)	160
O6'—H6'O...O3 ⁱⁱⁱ	1.84	2.662 (2)	175

Symmetry codes: (i) $2-x, y-\frac{1}{2}, 2-z$; (ii) $1+x, y, 1+z$; (iii) $x-1, y, z-1$; (iv) $x, y, 1+z$.

Data collection: *CAD-4 ARGUS* (Enraf–Nonius, 1994). Cell refinement: *CAD-4 ARGUS*. Data reduction: *Chi90s* (Boyle, 1997), *XCAD4* (Harms, 1997) and *SHELXTL/PC* (Sheldrick, 1994). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997). Molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997). Software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1465). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 1721–1725

4-Methylbicyclo[6.3.0]undecane-2,6-dione, (I), 7-bromo-4-methylbicyclo[6.3.0]undecane-2,6-dione, (II), 7-acetonyl-4-methylbicyclo[6.3.0]undecane-2,6-dione, (III), and 8-methyltricyclo[9.3.0.0^{2,6}]tetradec-5-ene-4,10-dione, (IV)

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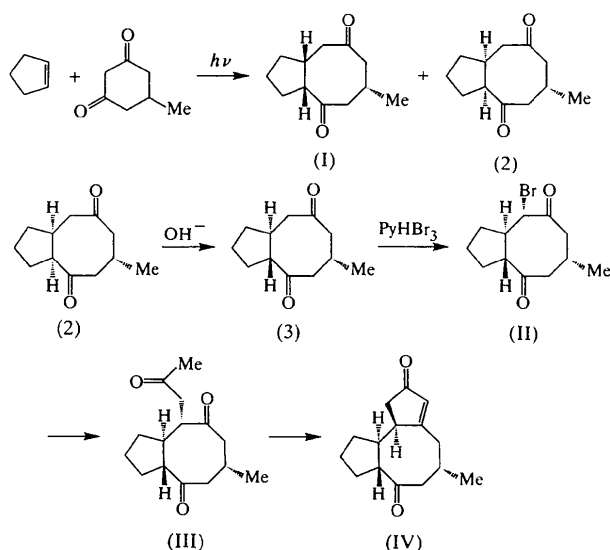
Abstract

The crystal structures of the four title compounds, $\text{C}_{12}\text{H}_{18}\text{O}_2$, (I), $\text{C}_{12}\text{H}_{17}\text{BrO}_2$, (II), $\text{C}_{15}\text{H}_{22}\text{O}_3$, (III), and $\text{C}_{15}\text{H}_{20}\text{O}_2$, (IV), which were obtained during studies of the preparation of C_5 – C_8 – C_5 fused-ring compounds,

have been determined. The eight-membered rings in all four compounds take the boat–chair form.

Comment

(±)-7,8-Epoxy-4-basmen-6-one is a diterpenoid tobacco isolate (Wahlberg *et al.*, 1983), and the first synthesis was achieved by a transannular cyclization strategy (Myers & Condroski, 1995). We have investigated the synthesis of C₅–C₈–C₅ fused-ring compounds by intramolecular aldol reaction (Umehara *et al.*, 1993; Ohba & Umehara, 1997). The present paper reports the preparation and structure of C₅–C₈–C₅ fused-ring compound (IV), which has a basmane-like skeleton with methyl at the 4-position. Structures and relative stereochemistry of compounds (I)–(III), obtained in the course of synthesis, were also verified by X-ray crystallography.



Epimerization of (III) seems to occur under basic conditions. A semi-empirical molecular orbital calculation by MOPAC/PM3 method (CACHe Scientific, 1994) indi-

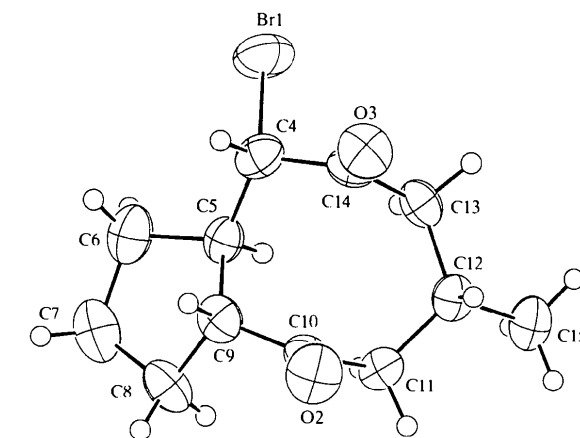


Fig. 2. The molecular structure of (II), with displacement ellipsoids at the 50% probability level. H atoms are represented by circles of an arbitrary radius.

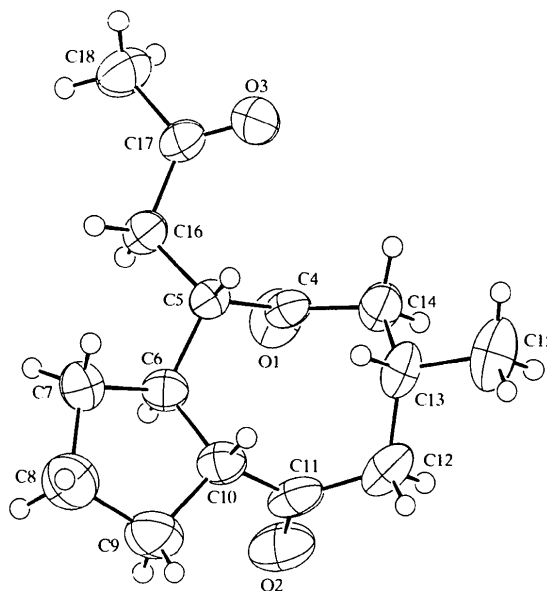


Fig. 3. The molecular structure of (III), with displacement ellipsoids at the 50% probability level. H atoms are represented by circles of an arbitrary radius.

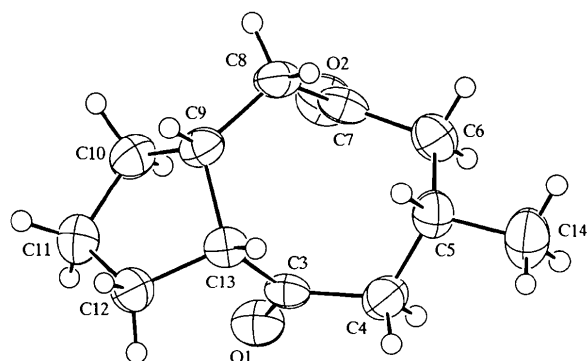


Fig. 1. The molecular structure of (I), with displacement ellipsoids at the 50% probability level. H atoms are represented by circles of an arbitrary radius.

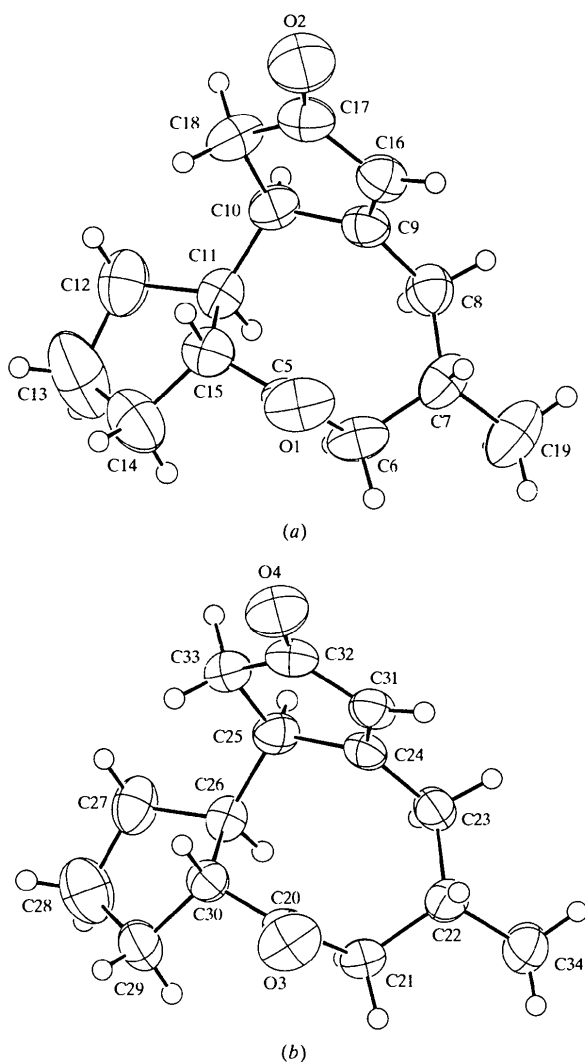


Fig. 4. The structures of the two independent molecules, (a) and (b), of (IV), with displacement ellipsoids at the 50% probability level. H atoms are represented by circles of an arbitrary radius.

Experimental

Two isomers of *cis*-4-methylbicyclo[6.3.0]undecane-2,6-dione, (I) and (2), were synthesized directly by the photocycloaddition which was carried out in a methanol solution of 5-methylcyclohexane-1,3-dione and cyclopentene with a high-pressure mercury lamp. The photoadduct (2) was isomerized to *trans*-isomer (3) under basic conditions (3% KOH-ethanol) at room temperature. The bromination of (3) with pyridinium bromide perbromide in acetic acid gave the 7-bromide (II), the 5-bromide, the 3-bromide and the 3,5-dibromide. On the other hand, bromination of (3) in the solid phase by the same reagent yielded the three monobromides and the 1,7-dibromide; to a ground mixture of (3) and pyridinium bromide perbromide in a mortar, two drops of acetic acid were added and mixed with a pestle, then bromination occurred at room temperature. Crystal structures of the mono- and dibromides except (II) will be reported separately (Umehara *et al.*, 1999). 7-Bromide (II)

was allylated with allyltributyltin in the presence of azobisisobutyronitrile, and the 7-allyl derivative obtained was oxidized to the 7-acetyl compound (III) with palladium(II) chloride and copper(I) chloride in *N,N*-dimethylformamide under an O₂ atmosphere. The intramolecular aldol reaction of (III) under basic conditions (3% KOH-ethanol-H₂O) gave the desired C₅-C₈-C₅ fused ring compound (IV). Crystals were grown from a hexane/ether solution for (I) and (II), and from an ethanol/ether solution for (III) and (IV). The melting points of compounds (I)-(IV) are 360, 382, 387 and 423 K, respectively.

Compound (I)

Crystal data

C₁₂H₁₈O₂
M_r = 194.27
 Triclinic
P $\bar{1}$
a = 6.629 (1) Å
b = 16.350 (1) Å
c = 5.328 (1) Å
 α = 95.78 (2)°
 β = 109.90 (2)°
 γ = 80.30 (2)°
V = 534.6 (2) Å³
Z = 2
D_s = 1.207 Mg m⁻³
D_m not measured

Data collection

Rigaku AFC-5 diffractometer
 ω scans
 Absorption correction: none
 2056 measured reflections
 1882 independent reflections
 1384 reflections with *I* > 2 σ (*I*)

Mo *K* α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 13.8–15.0°
 μ = 0.080 mm⁻¹
T = 297 K
 Plate-like
 0.5 × 0.5 × 0.1 mm
 Colourless

*R*_{int} = 0.019
 θ _{max} = 25°
h = 0 → 8
k = -19 → 19
l = -6 → 6
 3 standard reflections every 100 reflections
 intensity decay: none

Refinement

Refinement on *F*²
R = 0.058
wR = 0.069
S = 1.05
 1685 reflections
 145 parameters
 H-atom parameters constrained

w = 1/[$\sigma^2(F) + 0.0009F^2$]
 $(\Delta/\sigma)_{\max}$ = 0.003
 $\Delta\rho_{\max}$ = 0.22 e Å⁻³
 $\Delta\rho_{\min}$ = -0.28 e Å⁻³
 Extinction correction: none
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected bond lengths (Å) for (I)

O1—C3	1.203 (2)	C7—C8	1.507 (2)
O2—C7	1.211 (2)	C8—C9	1.533 (2)
C3—C4	1.509 (2)	C9—C10	1.519 (2)
C3—C13	1.524 (2)	C9—C13	1.559 (2)
C4—C5	1.518 (2)	C10—C11	1.535 (2)
C5—C6	1.531 (2)	C11—C12	1.538 (2)
C5—C14	1.525 (2)	C12—C13	1.527 (2)
C6—C7	1.506 (2)		

Compound (II)

Crystal data

C₁₂H₁₇BrO₂
M_r = 273.17

Mo *K* α radiation
 λ = 0.71073 Å

Triclinic

$P\bar{1}$
 $a = 7.442 (1) \text{ \AA}$
 $b = 14.747 (3) \text{ \AA}$
 $c = 5.586 (2) \text{ \AA}$
 $\alpha = 92.84 (2)^\circ$
 $\beta = 101.39 (2)^\circ$
 $\gamma = 87.87 (2)^\circ$
 $V = 600.1 (3) \text{ \AA}^3$
 $Z = 2$
 $D_x = 1.512 \text{ Mg m}^{-3}$
 D_m not measured

Data collection

Rigaku AFC-5 diffractometer
 θ - 2θ scans
 Absorption correction:
 by integration (Coppens *et al.*, 1965)
 $T_{\min} = 0.175$, $T_{\max} = 0.605$
 2286 measured reflections
 2107 independent reflections

Refinement

Refinement on F
 $R = 0.042$
 $wR = 0.049$
 $S = 1.07$
 1908 reflections
 153 parameters
 H-atom parameters constrained

Table 2. Selected bond lengths (Å) for (II)

Br1—C4	1.966 (3)	C7—C8	1.531 (6)
O2—C10	1.210 (4)	C8—C9	1.531 (5)
O3—C14	1.214 (4)	C9—C10	1.504 (5)
C4—C5	1.502 (4)	C10—C11	1.504 (4)
C4—C14	1.508 (5)	C11—C12	1.533 (4)
C5—C6	1.530 (5)	C12—C13	1.529 (4)
C5—C9	1.533 (4)	C12—C15	1.528 (5)
C6—C7	1.544 (5)	C13—C14	1.498 (4)

Compound (III)

Crystal data

C₁₅H₂₂O₃
 $M_r = 250.34$
 Monoclinic
 $P2_1/n$
 $a = 7.874 (2) \text{ \AA}$
 $b = 21.134 (3) \text{ \AA}$
 $c = 9.220 (3) \text{ \AA}$
 $\beta = 114.00 (2)^\circ$
 $V = 1401.7 (7) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.186 \text{ Mg m}^{-3}$
 D_m not measured

Data collection

Rigaku AFC-5 diffractometer

Cell parameters from 25 reflections
 $\theta = 14.7$ – 15.0°
 $\mu = 3.405 \text{ mm}^{-1}$
 $T = 298 \text{ K}$
 Plate-like
 $0.75 \times 0.55 \times 0.15 \text{ mm}$
 Colourless

1542 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.015$
 $\theta_{\text{max}} = 25^\circ$
 $h = 0 \rightarrow 9$
 $k = -18 \rightarrow 18$
 $l = -7 \rightarrow 7$
 3 standard reflections every 100 reflections intensity decay: none

$w = 1/[\sigma^2(F) + 0.0009F^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.59 \text{ e \AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 13.8$ – 14.9°
 $\mu = 0.081 \text{ mm}^{-1}$
 $T = 297 \text{ K}$
 Prism
 $0.6 \times 0.4 \times 0.3 \text{ mm}$
 Colourless

$R_{\text{int}} = 0.017$
 $\theta_{\text{max}} = 26^\circ$

 θ - 2θ scans

Absorption correction: none
 2961 measured reflections
 2760 independent reflections
 1707 reflections with $I > 2\sigma(I)$

Refinement

Refinement on F
 $R = 0.053$
 $wR = 0.063$
 $S = 1.07$
 2343 reflections
 185 parameters
 H-atom parameters constrained

Table 3. Selected bond lengths (Å) for (III)

O1—C4	1.215 (2)	C8—C9	1.518 (4)
O2—C11	1.221 (3)	C9—C10	1.530 (3)
O3—C17	1.216 (2)	C10—C11	1.511 (3)
C4—C5	1.523 (3)	C11—C12	1.493 (3)
C4—C14	1.505 (3)	C12—C13	1.531 (3)
C5—C6	1.534 (3)	C13—C14	1.529 (3)
C5—C16	1.530 (3)	C13—C15	1.530 (3)
C6—C7	1.537 (3)	C16—C17	1.496 (3)
C6—C10	1.545 (3)	C17—C18	1.502 (3)
C7—C8	1.528 (3)		

Compound (IV)

Crystal data

C₁₅H₂₀O₂
 $M_r = 232.32$
 Triclinic
 $P\bar{1}$
 $a = 11.331 (4) \text{ \AA}$
 $b = 13.315 (2) \text{ \AA}$
 $c = 9.298 (2) \text{ \AA}$
 $\alpha = 101.44 (1)^\circ$
 $\beta = 105.67 (2)^\circ$
 $\gamma = 77.15 (2)^\circ$
 $V = 1302.5 (6) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.185 \text{ Mg m}^{-3}$
 D_m not measured

Data collection

Rigaku AFC-5 diffractometer
 θ - 2θ scans
 Absorption correction: none
 5385 measured reflections
 5112 independent reflections
 3234 reflections with $I > 2\sigma(I)$

Refinement

Refinement on F
 $R = 0.052$
 $wR = 0.058$
 $S = 1.00$

$h = 0 \rightarrow 10$

$k = 0 \rightarrow 26$

$l = -11 \rightarrow 11$

3 standard reflections every 100 reflections intensity decay: none

$w = 1/[\sigma^2(F) + 0.0009F^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.003$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

Extinction correction: none
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 14.4$ – 15.0°

$\mu = 0.077 \text{ mm}^{-1}$

$T = 297 \text{ K}$

Prism

$0.7 \times 0.4 \times 0.2 \text{ mm}$

Colourless

$R_{\text{int}} = 0.011$

$\theta_{\text{max}} = 26^\circ$

$h = 0 \rightarrow 14$

$k = -16 \rightarrow 16$

$l = -11 \rightarrow 11$

3 standard reflections every 100 reflections intensity decay: 4.0%

$w = 1/[\sigma^2(F) + 0.0009F^2]$
 $(\Delta/\sigma)_{\text{max}} = 0.003$
 $\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$

4443 reflections
347 parameters
H-atom parameters
constrained

Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

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7-Phenyl-2,3,4,5-tetrahydro-1H-1,4-diazepin-5-one

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Abstract

The geometry of the title diazepinone, C₁₁H₁₂N₂O, reflects the presence of cross-conjugated amide and vinylogous amide functions. The packing is dominated by hydrogen-bonding interactions which link the molecules into corrugated sheets.

Comment

2,3-Dihydrodiazepines, (1), have been extensively characterized both in the form of salts (1*s*) and free bases (1*b*) by X-ray crystallography (Lloyd & McNab, 1993; Brisander *et al.*, 1998), and we now extend this work by reporting the first structure of a related diazepinone, (2). While the path of conjugation in (1) is endocyclic, it is exocyclic in (2), and we discuss here the structural implications of this difference. Of the three dihydrodiazepine bases, (1*b*), whose structures have been reported (Brisander *et al.*, 1998; Jordan *et al.*, 1998), those of (3) and (5) are disordered, and so the present discussion will focus on (4).

Table 4. Selected bond lengths (Å) for (IV)

O1—C5	1.218 (2)	C16—C17	1.446 (3)
O2—C17	1.220 (2)	C17—C18	1.499 (3)
O3—C20	1.220 (2)	C20—C21	1.509 (2)
O4—C32	1.221 (2)	C20—C30	1.506 (2)
C5—C6	1.501 (2)	C21—C22	1.534 (2)
C5—C15	1.501 (3)	C22—C23	1.537 (2)
C6—C7	1.532 (3)	C22—C34	1.520 (3)
C7—C8	1.528 (2)	C23—C24	1.502 (2)
C7—C19	1.519 (3)	C24—C25	1.512 (2)
C8—C9	1.501 (3)	C24—C31	1.336 (2)
C9—C10	1.500 (3)	C25—C26	1.530 (2)
C9—C16	1.337 (2)	C25—C33	1.531 (2)
C10—C11	1.529 (2)	C26—C27	1.529 (2)
C10—C18	1.527 (3)	C26—C30	1.531 (2)
C11—C12	1.522 (3)	C27—C28	1.498 (3)
C11—C15	1.532 (2)	C28—C29	1.527 (3)
C12—C13	1.532 (4)	C29—C30	1.541 (2)
C13—C14	1.513 (4)	C31—C32	1.456 (2)
C14—C15	1.541 (3)	C32—C33	1.506 (3)

The refinements were carried out based on all the reflections with $|F_o| \neq 0$. The threshold, $I > 2\sigma(I)$, was used only for calculation of the *R* factor. The positions of all the H atoms were calculated geometrically and a riding model was used in their refinement (C—H 0.96 Å). $U_{iso}(H)$ values were refined.

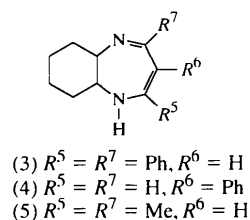
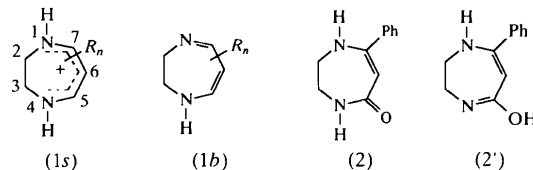
For all compounds, data collection: *AFCIMSC Diffractometer Control System* (Rigaku Corporation, 1993); cell refinement: *AFCIMSC Diffractometer Control System*; data reduction: local programs; program(s) used to solve structure: *CRYSTAN-GM* (Edwards *et al.*, 1996); program(s) used to refine structure: *CRYSTAN-GM*; molecular graphics: *CRYSTAN-GM*; software used to prepare material for publication: *CRYSTAN-GM*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1088). Services for accessing these data are described at the back of the journal.

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Compound (2) exists exclusively in the solid state in the diazepinone form rather than the tautomeric diazepinol form, (2'). With the exception of the N4—C5=O51 fragment, the bond lengths of the conjugated