STEREOSTRUCTURE OF SLOVANOLIDES: X-RAY CRYSTALLOGRAPHY OF THE 3-OXO-4αH,8α-BENZOYLOXY-10β,11α-DIACETOXYSLOVANOLIDE*

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X-Ray structural analysis of 3-oxo-4αH,8α-benzoyloxy-10β,11α-diacetoxyslovanolide (*XII*) confirmed the correctness of the previously proposed structure of slovanolides. A comparison of the X-ray and ^{1}H NMR data of lactone *XII* showed that the conformation of this substance in crystals and in chloroform or benzene solutions is very similar. The structure of the lactone from *Laserpitium marginatum* has been corrected to formula *XIa*.

In the preceding paper[1] we studied the stereochemistry of 8α-senecioyloxy-10β-hydroxy-11α-acetoxyslov-3-enolide (*I*),** 8α-angeloyloxy-10β-hydroxy-11α-acetoxyslov-3-enolide (*II*)** and 8α-angeloyloxy-10β,11α-diacetoxyslov-3-enolide (*III*),** native lactones, which were isolated from *Laserpitium siler* L. (*Umbelliferae* family, *Laserpitieae* tribe)[2,3]. From the same species we isolated later 8α,11α-diangeloyloxy-10β-hydroxyslov-3-enolide (*IV*)** and 8α-isobutyroyloxy-10β,11α-diacetoxyslov-3-enolide (*V*)** and derived their structures[4]. In a subsequent paper[5] we described the isolation of four further lactones of the type discussed from the same plant material, *i.e.* 8α-(2′-methyl)butyroyloxy-10β-hydroxy-11α-acetoxyslov-3-enolide (*VI*), 8α-isobutyroyloxy-10β-hydroxy-11α-acetoxyslov-3-enolide (*VII*) 8α-hydroxy-10β,11α-diacetoxyslov-3-enolide (*VIII*) and 8α,10β-dihydroxy-11α-aceoxyslov-3-enolide (*IX*), and after correlation with the preceding lactones we proposed their structures. All the mentioned lactones *I*–*IX* are based on the basic 1βH,5βH, 6αH,7αH-guaian-6,12-olide structure (*X*), so far undescribed for other guaianolides, for which we proposed the name slovanolide[5]. Recently a short communication has been published[6], mentioning the results of an X-ray study[7] of the sesquiterpenic

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** Newly proposed names, according to the semisystematic nomenclature from ref.[2]. The original names used in ref.[1,3]: montanolide for lactone *I*, isomontanolide for lactone *II*, acetylisomontanolide for *III*, gradolide for *IV* and polhovolide for *V*.

lactone isolated from *L. marginatum* L. to which structure *XI* has been assigned. The structure of this lactone, isolated from a species of the genus as lactones *I*–*IX*, differs from the structure of the latter lactones by substitution at C(11) and especially by configuration at C(1), which is unexpected* from the point of view of the biogenesis of the otherwise very similar lactones isolated from the species of the same genus. Therefore we considered it necessary to check the structures of lactones *I*–*IX* proposed by us by an X-ray analysis.

X-Ray Structural Analysis of Lactone XII

For the X-ray structural analysis we used 3-oxo-4αH,8α-benzoyloxy-10β,11α-diacetoxyslovanolide (*XII*) the preparation of which has been described in one of the preceding papers[1]. The results of X-ray analysis (positional parameters for carbon, oxygen and hydrogen atoms) of lactone *XII* are presented in Tables I and II, and a perspective view of the molecule of this substance, including the numbering of the atoms, is shown in Fig. 1. According to the X-ray diffraction pattern lactone *XII* contains a *cis*-annelated five-membered homocycle, a *cis*-annelated γ-lactone ring fused with the seven-membered ring. Two acetate groups are bound to C(10) and C(11) and the benzoyloxy group to C(8). The configuration of all chiral centres corresponds to the formula *XII* proposed earlier[1]. The seven-membered ring is present in the most stable, twist-chair conformation[8]. The approximate two-fold axis passes through C(7) and the mid-point of the C(1)–C(10) bond. Such conformation with C(7) as a unique axis-carbon gives rise to several features of interest. It enables the formation of the fully equatorial, *cis* fused γ-lactone at position C(7), C(6) and the presence of the *trans*-oriented equatorial substituent at C(8) (with respect to the substituent at C(7)). Furthermore, in this conformation, the strain energy associated with the two axial substituents at C(1) and C(10) is reduced to its lowest value, a value comparable to an axial position in cyclohexane[9]. However, grilactone[10], another guaianolide with the *cis,cis* configuration at both five-membered ring junctions, contains the seven-membered ring in a twist-boat conformation.** The favouring in grilactone of the twist-boat conformation over the twist-chair can be attributed to the different hybridisation state of the C(10) carbon atom. In such a case, the twist-boat conformation, although energetically less favourable by itself, allows both *cis* fused five-membered rings to be diequatorial. *cis*-Fusion of the cyclopentanone at C(1),

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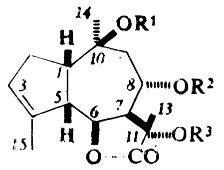
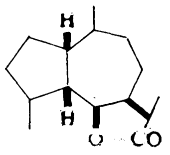
* As for the configuration at C(8) in lactone *XI*, an error is evidently present in ref.[6] concerning the indication of the substitution 8αH, since the next ref.[7] gives the name 1αH,5βH,6αH,7αH,8α-angeloyloxy-10β-acetoxyguaian-6,12-olide. This name is not correct either, because it does not consider the double bond between C(3) and C(4).

** Originally the authors assigned the twist-chair form for the seven-membered ring in grilactone. However, we think — from the endocyclic torsion angles quoted in paper[10] — that it follows the twist-boat conformation.

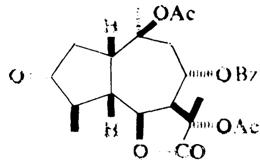
C(5) introduces considerable strain due to the junction at a dihedral angle of 58·8° and causes significant puckering of the five-membered ring. The average of the five endocyclic torsion angle moduli is 28·1° and the ring adopts a distorted half-chair conformation.



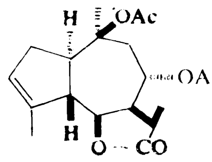
I: $R^1 = H$; $R^2 =$ Sen; $R^3 =$ Ac
II: $R^1 = H$; $R^2 =$ Ang; $R^3 =$ Ac
III: $R^1, R^3 =$ Ac; $R^2 =$ Ang
IV: $R^1 = H$; $R^2, R^3 =$ Ang
V: $R^1, R^3 =$ Ac; $R^2 =$ i-But
VI: $R^1 = H$; $R^2 =$ 2-Mebut; $R^3 =$ Ac
VII: $R^1 = H$; $R^2 =$ i-But; $R^3 =$ Ac
VIII, $R^1, R^3 =$ Ac; $R^2 = H$
IX, $R^1, R^2 = H$; $R^3 =$Ac



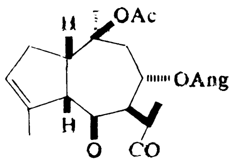
X



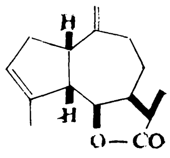
XII



XI



XIa



XIII

Sen = $OCCH = C(CH_3)_2$; Ac = $OCCH_3$; Ang = $OCC(CH_3) = CHCH_3(Z)$; *i*-But = = $OCCH(CH_3)_2$; 2-Mebut = $OCCH(CH_3)C_2H_5$; Ben = OCC_6H_5.

The γ-lactone ring is less puckered. The average of torsion angle moduli is 18·3° and the ring approximates an envelope conformation with C(7) as the flap.

In the seven-membered ring the valency angles C(5), C(1), C(10) and C(7), C(6), C(5) (120·5°(5) and 119·4°(5)), respectively are larger than expected and presumably reflect a deformation which may be due to the strain caused by the *cis* fusion of both five-membered rings.

X-Ray structural analysis of lactone *XII* confirmed the correctness of its structure, proposed earlier[1]. This also confirmed the structure of the starting original substance, 8α-angleloyloxy-10β,11α-diacetoxyslov-3-enolide[1] (*III*) from which compound *XII* was prepared by a multistep reaction sequence in which an inversion at some of the

TABLE I

Positional parameters (. 10^4) and isotropic thermal parameters (Å . 10^4) for carbon and oxygen atoms in keto lactone *XII*

Atom	*X*	*Y*	*Z*	UEQ
C(1)	5 497(7)	607(5)	6 418(3)	534(23)
C(2)	5 369(8)	−212(5)	5 905(3)	582(25)
C(3)	6 946(9)	−444(6)	5 762(3)	635(27)
C(4)	7 923(8)	422(5)	6 012(3)	617(25)
C(5)	6 836(7)	1 290(5)	6 226(3)	510(22)
C(6)	6 535(7)	2 057(5)	5 702(3)	504(22)
C(7)	5 717(7)	3 090(5)	5 820(3)	500(22)
C(8)	4 520(7)	3 031(5)	6 300(3)	512(22)
C(9)	3 530(7)	2 053(5)	6 242(3)	569(23)
C(10)	4 064(8)	1 130(5)	6 625(3)	573(25)
C(11)	6 950(8)	3 895(5)	5 911(3)	522(23)
C(12)	8 211(9)	3 439(6)	5 549(3)	572(26)
C(13)	7 503(9)	4 032(6)	6 563(3)	619(26)
C(14)	2 864(9)	286(6)	6 694(4)	768(32)
C(15)	8 874(9)	−30(6)	6 528(4)	805(33)
C(16)	6 363(10)	5 156(6)	5 146(4)	721(31)
C(17)	6 022(12)	6 297(6)	5 042(4)	946(40)
C(18)	3 344(8)	4 587(6)	6 688(3)	646(28)
C(19)	2 296(8)	5 431(5)	6 539(3)	599(26)
C(20)	1 568(9)	5 480(6)	6 001(4)	757(31)
C(21)	513(10)	6 270(7)	5 893(4)	934(37)
C(22)	212(10)	6 981(7)	6 336(5)	890(38)
C(23)	928(11)	6 949(8)	6 871(4)	1 053(43)
C(24)	1 957(11)	6 172(6)	6 977(4)	898(35)
C(25)	3 498(11)	2 018(7)	7 585(4)	822(35)
C(26)	4 201(12)	2 277(8)	8 164(3)	976(42)
O(1)	7 981(5)	2 395(3)	5 459(2)	607(17)
O(2)	9 296(6)	3 870(4)	5 373(2)	746(19)
O(3)	7 385(7)	−1 228(4)	5 507(2)	901(23)
O(4)	6 576(6)	4 960(3)	5 737(2)	627(18)
O(5)	6 483(6)	4 483(4)	4 774(2)	755(21)
O(6)	3 619(5)	3 954(3)	6 208(2)	585(16)
O(7)	3 930(7)	4 453(4)	7 160(2)	862(23)
O(8)	4 491(5)	1 514(4)	7 225(2)	624(17)
O(9)	2 261(7)	2 215(6)	7 441(3)	1 028(27)

chiral centres of the starting lactone *III* should not take place. In view of the chemical correlation or the correlation carried out by means of ^{1}H NMR spectra of native lactones *I*, *II*, *IV–IX* with 8α-angeloyloxy-10β,11α-diacetoxyslov-3-enolide[1,4,5] (*III*), the originally proposed structures of lactones *I*, *II*, *IV–IX* (including absolute configurations) should be considered proved.

TABLE II

Positional parameters (. 10^3) and isotropic temperature factors (. 10^3) for hydrogen atoms in ketolactone *XII*

Atom	*X*	*Y*	*Z*	*U*
H(11)	571	26	685	70
H(21)	480	12	553	70
H(22)	480	−92	605	70
H(41)	869	74	569	70
H(51)	720	180	658	70
H(61)	582	159	542	70
H(71)	502	332	545	70
H(81)	503	299	673	70
H(91)	352	181	578	70
H(92)	243	226	638	70
H(131)	791	336	681	127
H(132)	661	439	681	127
H(133)	838	460	650	127
H(141)	244	−2	628	127
H(142)	199	69	693	127
H(143)	327	−36	696	127
H(151)	950	59	674	127
H(152)	962	−59	632	127
H(153)	820	−43	685	127
H(171)	616	645	457	127
H(172)	672	682	529	127
H(173)	489	642	517	127
H(201)	181	491	566	154
H(211)	−5	631	547	154
H(221)	−61	758	626	154
H(231)	69	753	721	154
H(241)	250	614	740	154
H(261)	333	260	844	127
H(262)	500	289	807	127
H(263)	473	164	840	127

Comparison of X-*Ray Data of Lactones* XII *and* XI

The result which we obtained on X-ray structural analysis of lactone *XII* led us to a detailed analysis of the data published in connection with the X-ray analysis of lactone *XI* from *L. marginatum*[7]. We came to the surprising conclusion that the torsion angles (dihedral) for lactone *XI* given in the paper, comparable from the point of view of the structures of both lactones *XI* and *XII*, are in agreement with the dihedral angles for lactone *XII* found by us, although they should differ distinctly in view of the proposed structure with 1αH as follows from an analysis of Dreiding models. From the published values [7] for lactone *XI* we calculated the values of further dihedral angles which were very close to the values for lactone *XII* if comparable angles were taken into consideration. In Table III we present the comparable values for dihedral angles, characteristic of the differentiation of the structures with 1αH and with 1βH, calculated from X-ray data for lactones *XI* and *XII* and approximately estimated on the basis of Dreiding models. For the structure with 1αH proposed[6,7] for lactone *XI* it follows clearly that the lactone from *L. marginatum* cannot have the structure *XI*, but that formula *XIa* is consistent with the data (without absolute

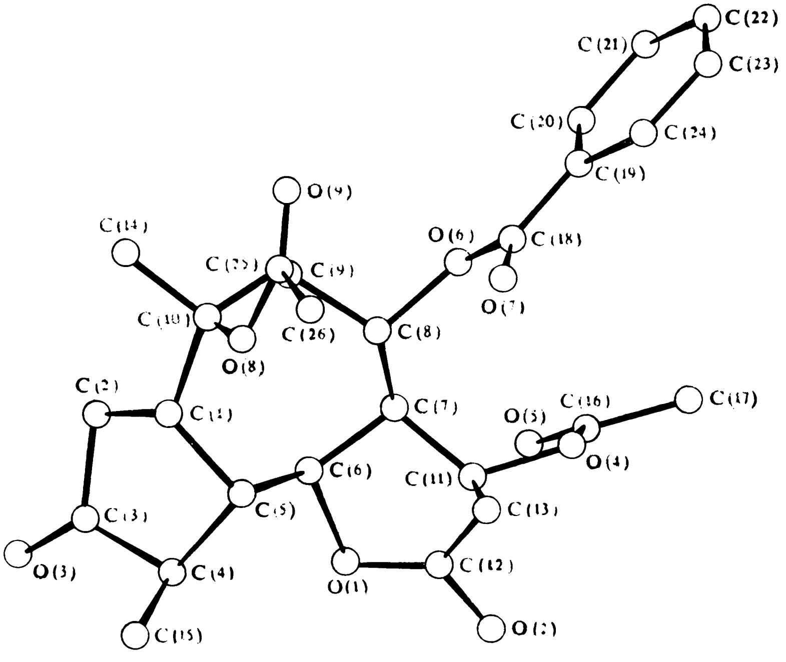


FIG. 1

Numbering of the atoms of lactone *XII*

configuration). Hence, this lactone contains – the same as slovanolides (1βH,5βH, 6αH,7αH-guaian-6,12-olides[5]; for example lactones *I*–*IX*) – a *cis*-annellated five-membered and seven-membered homocycle, a *cis*-annellated γ-lactone ring, the same position of the double bond and the substitution and the steric arrangement at C(8) and C(10), and it differs from the slovanolides described so far only by the absence of the oxygen-containing function at C(11). In view of the structure and the parent species of lactone *XIa* it is possible to assume that the determination of the absolute configuration will prove it to belong to slovanolides.

NMR *Spectra of Lactone* XII *and Its Conformation in Solution*

The structure *XII* for the keto lactone *XII* investigated was in full accord with its ^{13}C NMR spectrum. The proton decoupled ^{13}C NMR spectrum (Table IV) contained 24 signals of which two correspond to two symmetrically equivalent benzoate carbons. The multiplicity of the signals and thus the character of the carbons were determined from the so-called "attached proton test" experiment[11] (modulation of the transversal magnetisation by spin–spin ^{13}C – ^{1}H coupling, using the spin echo experiment with a gated ^{1}H decoupling). The carbon atoms of the same type were then assigned on the basis of chemical shifts and comparison with ^{13}C data in structurally related substances. However, for conformational analysis all the ^{13}C chemical shifts are hardly utilisable. For this purpose ^{1}H NMR spectra were measured in which all the signals were assigned structurally (Table V). In view of the difficult analysis of the partly superimposed multiplets of hydrogens H(2′) and H(5) in $CDCl_3$ solution the measurement was also carried out in $C_6{}^2H_6$, where the signals of hydro-

TABLE III

Comparable dihedral angles obtained from X-ray analysis of lactones *XIa* and *XII* and estimated from Dreiding models for structure *XI*

Dihedral angles	*XII*	*XIa*	*XI*
C(10)—C(1)—C(2)—C(3)	+172·5	+170·5	−160
C(5)—C(1)—C(2)—C(3)	+ 38·1	+33·0	− 30
C(1)—C(2)—C(3)—C(4)	− 17·9	− 17·8	+ 20
C(2)—C(3)—C(4)—C(5)	− 9·0	− 5·8	0
C(3)—C(4)—C(5)—C(1)	+ 32·1	+ 26·9	− 20
C(3)—C(4)—C(5)—C(6)	− 89·2	− 91·0	−140
C(4)—C(5)—C(1)—C(2)	− 43·3	− 36·6	+ 40
C(4)—C(5)—C(1)—C(10)	−175·5	−170·6	+150
C(6)—C(5)—C(1)—C(2)	+ 73·4	+ 76·8	+150
C(6)—C(5)—C(1)—C(10)	− 58·8	− 57·1	− 90

gens H(1) and H(4) are superimposed. The data from both solvents permitted the expression of the values of the solvent effect caused by the aromatic solvent (ASIS). Among the generally negative values of ASIS the practically zero effects on hydrogens H(7) and H(8) are of interest, while the effect on H(6) is very distinct (−0·59 ppm). This behaviour is characteristic of C(6)-lactones and different from that of C(8)-lactones[12]. The information on the conformation of compound *XII* in solution can be obtained from the vicinal coupling constants of the hydrogens of the five-membered and the seven-membered homocycle. The values of the constants in both solvents (and thus also the conformation) are practically the same and therefore the lacking data may be completed by the data from the second solvent. The set of ${}^3J_{H,H}$ (Table VI) was then used for the calculation of the dihedral angles $\Phi_{i,j}$ (NMR) according to the Karplus-like relation from ref.[13], and their values were compared with the $\Phi_{i,j}$ values (X-ray) calculated from the coordinates of hydrogens, determined by X-ray diffraction analysis in the crystal. In spite of certain differences, evidently caused by the not quite adequate relation $J_{H,H} = f(\Phi)$ derived for the six-membered ring and used here for the five-membered and seven-membered ring, it may be generally observed that the values $\Phi_{i,j}$ (X-ray) and $\Phi_{i,j}$ (NMR) agree well. The conformation of compound *XII* in solution should thus be very similar to the conformation found in the crystal and represented in Fig. 1.

TABLE IV

Carbon-13 chemical shifts of compound *XII* in C^2HCl_3[a]

Carbon	δ_C (mult.)	Carbon	δ_C (mult.)
C(1)	48·35 (d)	C(14)	20·64 (q)[b]
C(2)	37·65 (t)	C(15)	16·24 (q)
C(3)	216·07 (s)	2 × $COCH_3$	
C(4)	46·72 (d)	C(16)	170·15 (s)[c]
C(5)	44·69 (d)	C(17)	22·48 (q)
C(6)	75·20 (d)	C(23)	169·81 (s)[c]
C(7)	47·86 (d)	C(24)	20·54 (q)[b]
C(8)	64·97 (d)	COC_6H_5	
C(9)	40·03 (t)	C(18)	164·71 (s)
C(10)	78·03 (s)	C(19)	133·48 (s)
C(11)	81·79 (s)	C(20), C(24)	129·57 (d)
C(12)	173·28 (s)	C(21), C(23)	128·46 (d)
C(13)	25·72 (q)	C(22)	139·41 (d)

[a] Referenced to C^2HCl_3 and chemical shifts recalculated to tetramethylsilane, $\delta(C^2HCl_3) = 77$; [b,c] the assignment of signals can be interchanged.

EXPERIMENTAL

X-Ray Analysis of Keto Lactone *XII*

Crystal data: Molecular formula: $C_{26}H_{30}O_9$, $F(000) = 1\,032$; molecular weight: 486·52 g . mol^{-1}; space group: orthorhombic, $P2_12_12_1$, $Z = 4$; cell dimensions: $a = 9{\cdot}069(1)$ Å, $b = 12{\cdot}656(3)$ Å, $c = 22{\cdot}461(3)$ Å, $u = 2\,578{\cdot}0(8)$ Å^3; density: $D_c = 1{\cdot}25$ g cm^{-1}, $D_m = 1{\cdot}24$ g cm^{-1}; radiation: CuK$\bar{\alpha}$, $\lambda = 1{\cdot}5418$ Å, μ(Cu—K$\bar{\alpha}$) = 0·80 mm^{-1}; final R-value 0·054 for 1 580 reflections.

Crystallographic measurements: A crystal of 0·1 × 0·2 × 0·4 mm dimensions, grown from the mixture of ethyl acetate and diisopropyl ether, was used to collect intensity data on a Syntex $P2_1$ diffractometer by $\Theta - 2\Theta$ scanning technique using a variable scan speed and graphite-monochromated CuK$\bar{\alpha}$ radiation. Lattice parameters were refined by a least-squares procedure utilizing 15 reflections the angles of which were measured by a centering routine associated with the diffractometer. The background and integrated intensity for each reflection were evaluated from a profile analysis according to Lehmann and Larsen[14] using the PRAN program[15]. Those 1 585 reflections for which $I/\sigma(I) > 1{\cdot}96$ were regarded as observed. Lorenz and polarization

TABLE V

Proton NMR Parameters of compound *XII*

Proton	Chemical shifts (coupling constants)		ASIS[a]
	in C^2HCl_3	in $C_6{}^2H_6$	
H(1)	3·17 m (13·3; 8; 5·3; 1)	2·72[b]	−0·45
H(2)	2·40 dd (17·7; 8·0)	1·85 bdd (18·0; 7·9; ≠ 0)	−0·55
H(2′)	2·21[c]	1·61 bdd (18·0; 13·3; 1)	−0·60
H(4)	2·85 bq (7·8; 1; 1)	2·72[b]	−0·13
H(5)	2·33[c]	2·09 bddd (12·2; 5·3; 1·6; ≠ 0)	−0·24
H(6)	4·75 dd (12·4; 10·2)	4·16 dd (12·2; 10·1)	−0·59
H(7)	3·91 t (10·6; 10·2)	3·89 t (10·7; 10·1)	−0·02
H(8)	5·90 dt (10·6; 10·4; 1·1)	5·90 dt (10·7; 10·5; 1·1)	0·00
H(9)	2·73 dt (15·1; 1·1; 1)	2·56 dt (15·0; 1·1; 1)	−0·17
H(9′)	2·11 dd (15·1; 10·4)	1·53 dd (15·0; 10·5)	−0·58
H(13)	1·57 s	1·16 s	−0·41
H(14)	1·62 s	1·56 s	−0·06
H(15)	1·23 d (7·8)	0·93 d (7·6)	−0·30
OAc:	2·22 s	1·95 s	−0·27
	1·86 s	1·52 s	−0·34
OBzl: *o*-	8·00 m	8·03 m	
m-	7·45 m	7·01—7·15 m	
p-	7·60 m	7·01—7·15 m	

[a] Defined as: δ_H (in $C_6{}^2H_6$) − δ_H (in C^2HCl_3); [b] overlapping of H(1) and H(4) multiplets; [c] partially overlapped by other signals.

corrections were applied but no absorption corrections were made [$\mu(Cu-K\bar{\alpha}) = 0{\cdot}80\ mm^{-1}$]. Anomalous dispersion corrections were made to the scattering factors of the oxygen and carbon atoms.

Structure analysis: MULTAN (ref.[16]) was used to calculate phases and the E map revealed the positions of 32 non-hydrogen atoms. Further calculations were performed with the SHELX program system[17]. The difference Fourier map revealed the remaining non-hydrogen atom positions and refinement first with isotropic and later with anisotropic temperature factors gave $R = 0{\cdot}090$. At this stage most of the H-atoms could be located from difference maps; however for simplicity, they were treated as follows: Methyl groups were set up and refined as rigid groups. All these H-atoms were assigned a common isotropic temperature factor which refined to $U = 0{\cdot}127(9)$ Å^2. The remaining H-atoms were placed in calculated positions and refined subject to the constraint that the C—H vectors were constant in magnitude and direction but not in position (*i.e.* a rigid model). The phenyl hydrogens were given a common isotropic temperature factor which refined to $U = 0{\cdot}153(17)$ Å^2; a common isotropic temperature factor for the remaining H-atoms converged at $U = 0{\cdot}070(6)$ Å^2. The quantity minimized in the least-squares calculations was $\sum(|F_0| - |F_c|)^2$. Unit weights were used throughout the refinement. Five reflections were omitted in the last cycle of refinement. The final R value was 0·054 for 1 580 reflections.

NMR Spectrometry

The ^{1}H NMR spectra were measured on a Varian XL-200 spectrometer at 200 MHz and ^{13}C NMR spectra on the same instrument at 50·3 MHz. Solutions containing about 20 mg of lactone *XII* in 0·4 ml C^2HCl_3 (or $C_6{}^2H_6$) were used for both measurements at room temperature (about 22°C).

TABLE VI

Comparison of X-ray and ^{1}H NMR conformational data of Compound *XII*

H_i, H_j	$\phi_{i,j}$(X-ray)	$J_{i,j}$	$\phi_{i,j}{}^a$ (NMR)
1,2	160·5	13·3	180
1,2′	38·3	7·9	27
1,5	− 46·8	5·3	45
4,5	− 84·0	1·6	71
5,6	−160·1	12·4	167
6,7	− 31·1	10·2	0
7,8	−166·5	10·6	154
8,9	−147·0	10·4	153
8,9′	93·9	1·1	103

[a] Only absolute values of the angles are obtainable. In the cases where J value corresponds to two possible angles (due to the periodicity of Karplus curve) only one, which is closer to $\phi_{i,j}$ (X-ray) is given.

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