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219. The Crystal and Molecular Structures of Two Acid-Catalysed Rearrangement Products of Taxochinon

by **Hans Peter Weber**[a]), **Trevor J. Petcher**[a]), **Marcel Hensch**[b])
and **Conrad Hans Eugster**[b])

a) Sandoz Ltd., Pharmaceutical Division, Chemical Research, Basel, Switzerland
b) Institute of Organic Chemistry, University of Zürich, Rämistrasse 76, 8001 Zürich, Switzerland

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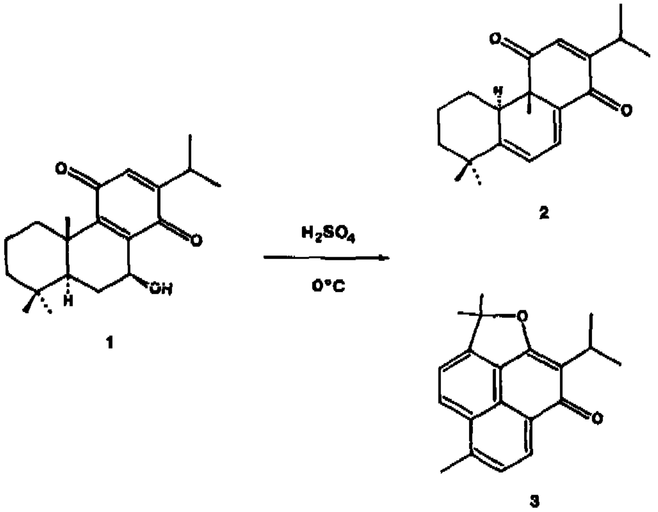
Zusammenfassung. Die Strukturen von zwei säurekatalysierten Umlagerungsprodukten von Taxochinon (**1**) wurden kristallographisch bestimmt: Die eine Verbindung stellte sich als ein 20 (10 → 9) *abeo*-Abietan (**2**) heraus, die andere als ein achirales Phenalenon (**3**). Die Abietanverbindung, $C_{20}H_{26}O_3$, kristallisiert in der Raumgruppe $P2_12_12_1$ mit 4 Molekeln in der Zelle und wurde mit 879 signifikanten Reflexionen bis R = 0,032 verfeinert. Die Phenalenonverbindung, $C_{20}H_{22}O_2$, kristallisiert in der Raumgruppe $P2_1/c$ mit 12 Molekeln in der Zelle und wurde mit 1823 signifikanten Reflektionen bis R = 0,058 verfeinert.

Summary. The structures of two acid catalysed rearrangement products of taxochinone (**1**) have been determined by X-ray analysis. One of the products turned out to be a 20 (10 → 9) *abeo*-abietane (**2**), the other an optically inactive phenalenone (**3**). Crystals of the former compound, $C_{20}H_{26}O_3$, belong to space group $P2_12_12_1$ with four molecules per cell and the structure was refined with 879 significant reflexions to R = 0.032. Crystals of the latter compound, $C_{20}H_{22}O_2$, belong to space group $P2_1/c$ with 12 molecules per cell and the structure was refined with 1823 significant reflexions to R = 0.058.

As described in the preceding paper [1] the treatment of taxochinon (**1**) with sulfuric acid at 0° leads to three rearrangement products of which **2** and **3** (**4** and **5** in [1]) were submitted to X-ray analysis to establish their respective structures. The rearrangement of **1** to **2** was expected from spectral investigations; the formation of the optically inactive compound **3**, however, exhibits an unprecedented ring fusion [1]. The absolute configuration of **2** could not be established by X-ray measurements because of the absence of anomalously scattering atoms [2] but it can be inferred from the known absolute configuration of **1** [3] and the stereochemistry of the 1,2-shift of the methyl group.



1. Crystal Structure of 2. – Crystal Data. (9R, 10R)–20 (10 → 9) *abeo*-12-hydroxy-5,7,12-abietatriene-11,14-dione, $C_{20}H_{26}O_3$, M = 314,425, crystallised from methanol, forms yellow needles with rectangular cross sections. The space group is $P2_12_12_1$, $a = 7.620(3)$, $b = 19.80(1)$, $c = 11.089(5)$ Å, $V = 1762$ Å^3, $Z = 4$ ($d_c = 1.19$ g/cm^3). Intensities were measured on a CAD-4 diffractometer with MoKα radiation (Graphite monochromator; ω: 2θ scan, scan width $\Delta\omega = 0.8 + 0.6\ \mathrm{tg}\theta$, 6000 counts per reflexion with 120 sec max time); 1799 reflexions were in the range $1.5 \leqslant \theta \leqslant 25°$, of which 879 reflexions showed a significant intensity with $I \geqslant 3\sqrt{P+B}$. Data reduction yielded the following statistics: $\bar{B} = 3.6$ Å^2, $<|E|> = 0.804$, $<|E^2 - 1|> = 0.867$, $<|E^2|> = 0.988$. No absorption correction was applied.

The structure was solved by the multi-solution method [4] and refined to R = 0.032 by least-squares procedures (LS) for 313 parameters[1]. H-positions were located from a difference map and refined isotropically. A final difference map showed a residual electron density of less than 0.1 e/Å^3. Final positional parameters are given in Table 1. A list of observed and calculated structure factors and vibrational parameters can be obtained from the authors.

Results. – The numbering scheme and structural parameters are shown in Fig. 1, a stereoscopic view of the molecule in Fig. 2. The mean of the 10 sp^3-sp^3 bond distances is 1.538 Å with variance $S^2 = 256.10^{-6}$ Å^2. The LS-computed e.s.d. of a C–C bond is about 0.009 Å and may be slightly underestimated (see legend to Fig. 1).

The O(11A) hydroxyl group is approximately in the ring plane, but although the distance O(11A)...O(12A) of 2.62 Å is short enough for an intramolecular hydrogen

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[1] In addition to the positional parameters of all atoms, anisotropic B_{ij} for C and O atoms, isotropic B-values for H-atoms, a scale factor and an isotropic extinction parameter G [4] were introduced. The final value of G = 10.0(3) for an assumed $\bar{T} = 0.02$ cm.

Table 1. *Coordinates of* **2** ($\times 10^4$ for C and O, $\times 10^3$ for H) with LS computed standard deviations in brackets

	X	Y	Z		X	Y	Z
C(1)	4321(6)	4231(2)	- 620(4)	H(2A)	542(5)	497(2)	- 173(3)
C(2)	4585(7)	4969(3)	-1018(5)	H(2B)	514(5)	526(2)	- 39(3)
C(3)	2832(7)	5263(2)	-1357(4)	H(3A)	301(5)	575(2)	- 166(3)
C(4)	1460(6)	5264(2)	- 402(4)	H(3B)	234(6)	495(2)	- 205(4)
C(5)	1367(5)	4573(2)	174(3)	H(4A)	- 14(6)	595(2)	- 128(3)
C(6)	- 129(5)	4340(2)	639(4)	H(4B)	- 66(5)	513(2)	- 153(3)
C(7)	- 187(5)	3766(2)	1384(4)	H(4C)	- 123(5)	550(2)	- 31(3)
C(8)	1183(4)	3351(2)	1548(3)	H(4D)	203(5)	627(2)	15(3)
C(9)	1040(5)	2776(2)	2339(3)	H(4E)	102(5)	579(2)	115(3)
C(10)	2655(5)	2430(2)	2738(3)	H(4F)	314(5)	567(2)	83(3)
C(11)	4224(5)	2682(2)	2384(3)	H(6)	- 122(5)	458(2)	47(3)
C(12)	4398(5)	3246(2)	1564(3)	H(7)	- 132(5)	362(2)	174(3)
C(13)	2843(5)	3452(2)	857(3)	H(10A)	111(5)	176(2)	371(3)
C(14)	3090(5)	4201(2)	415(3)	H(10B)	311(5)	154(2)	523(3)
C(4A)	- 304(7)	5485(2)	- 913(5)	H(10C)	459(5)	204(2)	462(3)
C(4B)	1967(7)	5793(2)	496(5)	H(10D)	267(6)	237(2)	507(4)
O(9A)	- 407(3)	2578(1)	2663(2)	H(10E)	305(5)	76(2)	352(3)
C(10A)	2473(6)	1840(2)	3544(4)	H(10F)	243(5)	108(2)	225(3)
C(10B)	3321(7)	1965(2)	4695(4)	H(10G)	446(5)	123(2)	277(3)
C(10C)	3185(7)	1181(2)	2998(4)	H(11A)	687(6)	249(2)	271(3)
O(11A)	5736(3)	2428(1)	2789(2)	H(13A)	168(5)	305(2)	- 63(3)
O(12A)	5848(3)	3501(1)	1468(3)	H(13B)	388(5)	290(2)	- 58(3)
C(13A)	2721(6)	2924(2)	- 129(3)	H(13C)	254(6)	242(2)	18(4)
H(1A)	554(5)	402(2)	- 40(3)	H(14)	376(5)	448(2)	102(3)
H(1B)	376(5)	393(2)	- 126(3)				

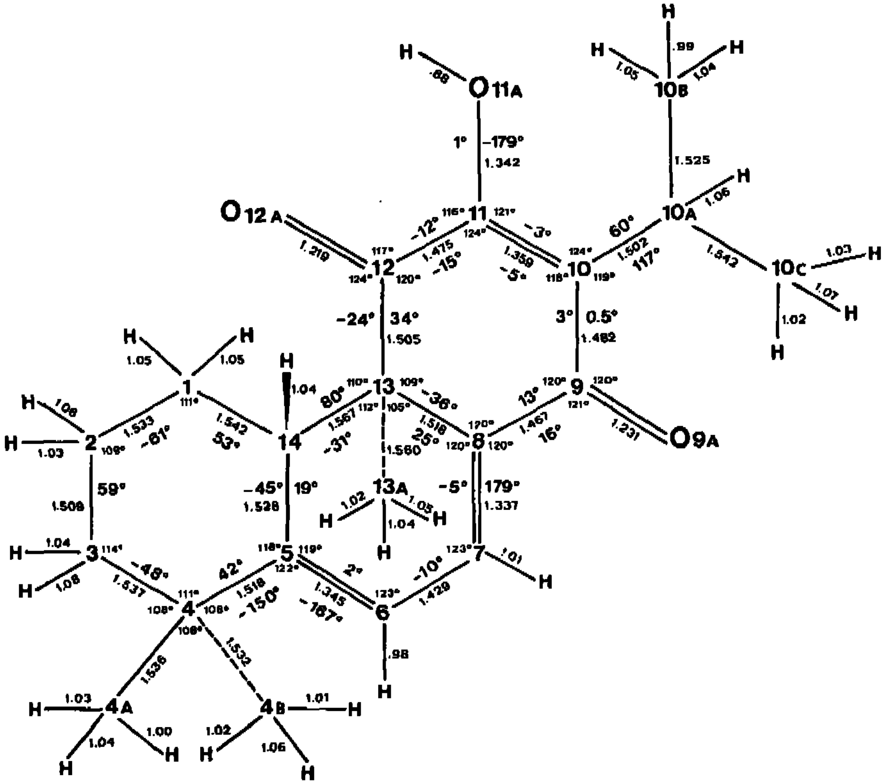

Fig. 1. *Bond lengths, bond angles and some torsion angles of* **2**. Standard deviations for parameters involving C and O atoms are ≈ 0.012 Å for bond lengths, ≈ 1.0° for bond angles, and ≈ 1.5° for torsion angles, σ(C···H) ≈ 0.1 Å

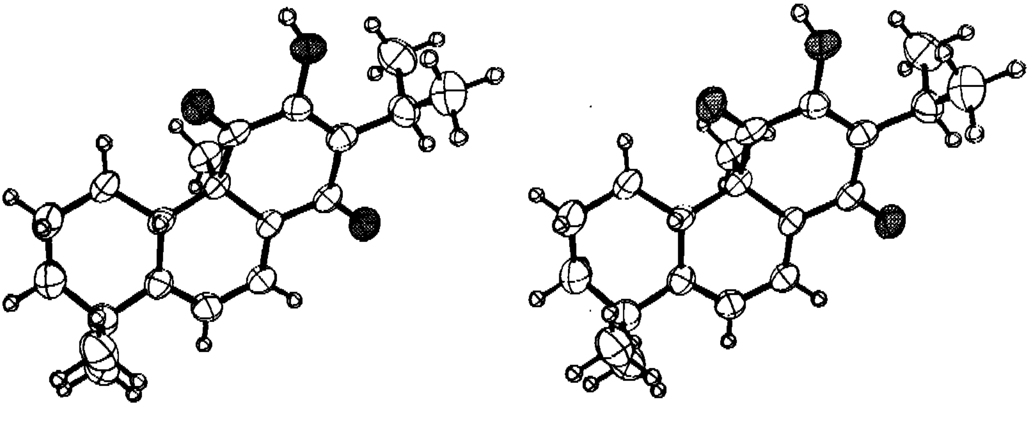

Fig. 2. *Stereoscopic projection of* **2** (oxygen atoms shaded). Anisotropic vibration ellipsoids at the 50% probability level, hydrogens with uniform, isotropic B of 1.0 Å².

bond, the C(11)–O(11A)–H angle, 139(5)°, and the H...O(12A) distance 2.6(1) Å, exclude this possibility. Instead, there is a linear intermolecular hydrogen bond from the hydoxyl O(11A) to the keto oxygen O(9A) of a molecule one **a**-translation removed, with O(9A)...O(11A) = 2.96(1) Å, H...O(9A) = 2.1(1) Å and ∢(O...H–O) = 178(5)°. The isopropyl group is symmetrical with respect to the ring plane, with H(10A) syn-planar to the C(9) carbonyl group. The packing of the molecules is shown in Fig. 3. Apart from the hydrogen bond mentioned above there are no close intermolecular contacts.

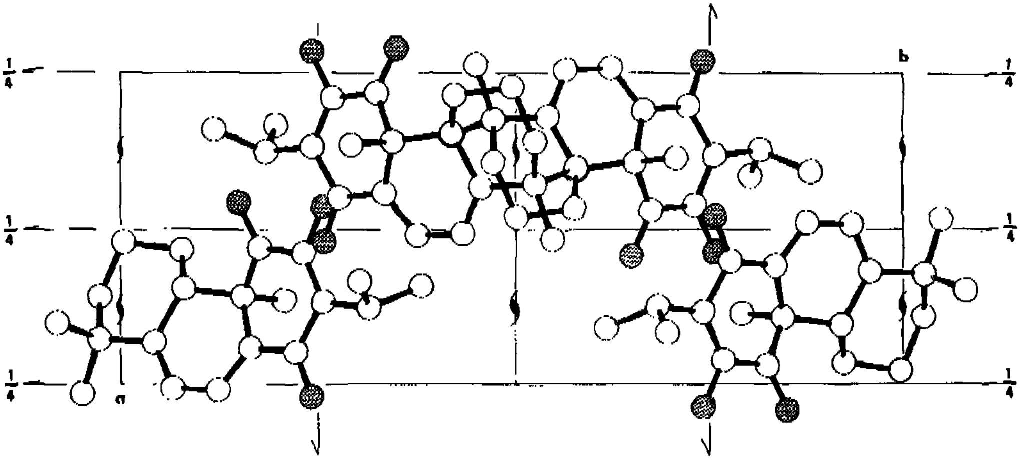

Fig. 3. *Packing diagram of* **2**. Shaded circles denote oxygen atoms, hydrogen atoms are omitted from the drawing

II. Crystal Structure of 3. – Crystal Data. 2-Isopropyl-4,4,7-trimethyl-1H-phenaleno-[1,9-bc]-furane-1-one, $C_{20}H_{20}O_2$, **M** = 292,378, crystallised from ethanol, forms yellow, irregular shaped plates. The space group is **P2$_1$/c**, **a** = 19.73(1), **b** = 28.51(2), **c** = 8.973(4) Å, β = 95.18(5)°; **V** = 5027 Å^3, **Z** = 12, **d$_c$** = 1.15 g/cm^3, **d$_x$** = 1.13(3) g/cm^3[2]). Intensities were measured on a CAD-4 diffractometer with Mo*K*α-radiation (Graphite monochromator, ω: *2θ* scan, scand width $\Delta\omega$ = 0.9° + 0.6° tgθ, 6000 counts per reflexion with 120 sec max time); 7268 reflexions were scanned in the range $1.5° \leqslant \theta \leqslant 22.5°$, of which 1825 reflexions showed a significant intensity with $I \geqslant 3\sigma(I)$. Data reduction yielded the following statistics: $\bar{B} = 3.4$ Å^2, $<|E|> = 0.722$, $<|E^2 - 1|> = 1.092$, $<|E|^2> = 1.039$. No absorption correction was applied.

The structure was solved by an automated symbolic addition procedure [5] and refined to R = 0.058 by least-squares procedures (LS) for 776 parameters. C- and O-atoms were refined with anisotropic temperature factors, H-positions were calculated[3]), and included in the final stages of refinement with a fixed isotropic B = 3.0 Å^2.

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[2]) The experimental determination of the density by the flotation method was extremely difficult because the hydrophobic crystals dissolved immediately in most organic solvents. The density could finally be measured in glycerin/water with the addition of a wetting agent.

[3]) —CH_3 groups were put in staggered conformation, > CH groups with C–H bisecting the C—C—C angle. For the C(22)-methyl group the difference Fourier map indicated that one hydrogen is roughly *syn*-planar to C(11).

Table 2. *Coordinates of* **3** (× 10^4 for C and O, × 10^3 for H) with LS computed standard deviations in brackets

	X	Y	Z		X	Y	Z
C(101)	2070(4)	39(3)	777(10)	H(117C)	218(3)	92(2)	- 319(8)
C(102)	1911(5)	375(3)	- 296(10)	H(118)	374(3)	216(2)	328(7)
C(103)	2174(4)	831(3)	- 116(10)	H(119A)	416(3)	247(2)	106(7)
C(104)	2114(5)	1271(3)	-1084(10)	H(119B)	439(3)	191(2)	137(7)
O(105)	2544(3)	1609(2)	- 145(6)	H(119C)	373(3)	205(2)	8(7)
C(106)	2792(4)	1403(3)	1163(9)	H(120A)	307(3)	279(2)	215(7)
C(107)	3206(5)	1584(3)	2292(10)	H(120C)	259(3)	240(2)	109(8)
C(108)	3398(4)	1267(4)	3525(9)	H(120B)	256(3)	240(2)	300(8)
C(109)	3164(4)	768(3)	3539(10)	H(122A)	267(3)	- 85(2)	414(8)
C(110)	3353(4)	444(3)	4650(9)	H(122B)	195(3)	- 70(2)	312(8)
C(111)	3112(5)	- 14(3)	4506(10)	H(122C)	266(3)	- 84(2)	228(7)
C(112)	2712(4)	- 174(3)	3282(11)	C(201)	1180(5)	379(3)	4647(11)
C(113)	2507(4)	148(3)	2086(9)	C(202)	1145(5)	792(4)	3899(11)
C(114)	2747(4)	615(3)	2290(9)	C(203)	681(5)	1132(4)	4318(10)
C(115)	2574(4)	924(3)	1141(9)	C(204)	481(6)	1619(4)	3758(12)
C(116)	1409(5)	1480(4)	-1254(12)	O(205)	- 44(3)	1770(2)	4785(7)
C(117)	2420(5)	1214(4)	-2549(10)	C(206)	- 140(5)	1420(3)	5755(10)
C(118)	3473(6)	2095(3)	2287(12)	C(207)	- 581(4)	1405(3)	6835(10)
C(119)	3973(6)	2148(4)	1094(16)	C(208)	- 562(5)	967(4)	7715(10)
C(120)	2912(7)	2457(4)	2099(16)	C(209)	- 111(4)	566(3)	7380(10)
O(121)	3797(3)	1406(2)	4617(7)	C(210)	- 108(5)	150(3)	8118(10)
C(122)	2489(5)	- 687(3)	3196(12)	C(211)	348(5)	- 211(3)	7767(11)
H(101)	186(3)	- 30(2)	62(8)	C(212)	793(5)	- 148(3)	6682(10)
H(102)	159(3)	29(2)	- 125(8)	C(213)	774(4)	271(3)	5865(9)
H(110)	366(3)	55(2)	558(8)	C(214)	314(4)	621(3)	6234(10)
H(111)	325(3)	25(2)	538(8)	C(215)	290(4)	1039(3)	5466(9)
H(116A)	138(3)	177(2)	- 197(8)	C(216)	133(7)	1622(5)	2174(13)
H(116B)	103(3)	124(2)	- 176(8)	C(217)	1045(6)	1981(4)	3948(15)
H(116C)	123(3)	157(2)	- 23(8)	C(218)	-1069(6)	1800(4)	7139(14)
H(117A)	238(3)	150(2)	- 320(7)	C(219)	-1611(6)	1876(4)	5991(16)
H(117B)	294(3)	113(2)	- 237(7)	C(220)	- 764(8)	2175(5)	7953(21)

	X	Y	Z		X	Y	Z
O(221)	- 920(3)	935(2)	8776(7)	C(311)	5924(5)	- 543(4)	10074(11)
C(222)	1282(5)	- 542(3)	6362(12)	C(312)	5515(5)	- 433(4)	8804(11)
H(201)	153(3)	11(2)	439(8)	C(313)	5494(4)	32(3)	8247(10)
H(202)	146(3)	84(2)	302(8)	C(314)	5900(4)	368(3)	9089(9)
H(210)	- 43(3)	11(2)	897(8)	C(315)	5901(5)	824(3)	8612(10)
H(211)	34(3)	- 53(2)	837(8)	C(316)	5000(7)	1783(4)	7592(15)
H(216A)	0(3)	196(2)	183(7)	C(317)	5936(7)	1658(4)	5876(13)
H(216B)	43(3)	147(2)	141(7)	C(318)	7105(6)	1499(4)	11446(13)
H(216C)	- 32(3)	141(2)	214(8)	C(319)	7709(8)	1639(6)	10724(17)
H(217A)	90(3)	231(2)	357(8)	C(320)	6709(8)	1886(6)	11941(17)
H(217B)	124(3)	201(2)	509(7)	O(321)	7079(3)	544(2)	12378(7)
H(217C)	148(3)	186(2)	336(8)	C(322)	5105(5)	- 808(4)	7968(12)
H(218)	- 140(3)	168(2)	793(7)	H(301)	478(3)	- 5(2)	631(8)
H(219A)	- 196(3)	213(2)	621(8)	H(302)	481(3)	76(2)	557(7)
H(219B)	- 140(3)	196(2)	495(8)	H(310)	662(3)	- 30(2)	1184(8)
H(219C)	- 189(3)	155(2)	572(7)	H(311)	592(3)	- 88(2)	1045(7)
H(220A)	- 105(3)	243(2)	827(7)	H(316A)	508(3)	214(2)	759(8)
H(220B)	- 50(3)	202(2)	898(8)	H(316B)	458(3)	170(2)	685(7)
H(220C)	- 36(3)	230(2)	738(8)	H(316C)	483(3)	171(2)	865(7)
H(222A)	121(3)	- 82(2)	705(8)	H(317A)	601(3)	201(2)	587(8)
H(222B)	120(3)	- 65(2)	525(8)	H(317B)	640(3)	149(2)	579(8)
H(222C)	178(3)	- 43(2)	654(8)	H(317C)	562(3)	157(2)	492(7)
C(301)	5096(5)	190(4)	6970(11)	H(318)	733(3)	133(2)	1249(7)
C(302)	5096(5)	650(4)	6545(10)	H(319A)	800(3)	188(2)	1137(8)
C(303)	5507(5)	980(3)	7364(10)	H(319B)	803(3)	134(2)	1059(7)
C(304)	5627(6)	1511(4)	7296(12)	H(319C)	756(3)	177(2)	967(7)
O(305)	6153(3)	1598(2)	8578(7)	H(320A)	700(3)	214(2)	1257(8)
C(306)	6279(5)	1194(3)	9355(10)	H(320B)	647(3)	205(2)	1099(8)
C(307)	6693(5)	1114(3)	10634(10)	H(320C)	631(3)	177(2)	1259(7)
C(308)	6719(5)	640(4)	11178(11)	H(322A)	518(3)	- 114(2)	851(7)
C(309)	6327(4)	247(3)	10415(10)	H(322B)	458(3)	- 73(2)	792(8)
C(310)	6320(5)	- 204(3)	10887(10)	H(322C)	522(3)	- 84(2)	687(7)

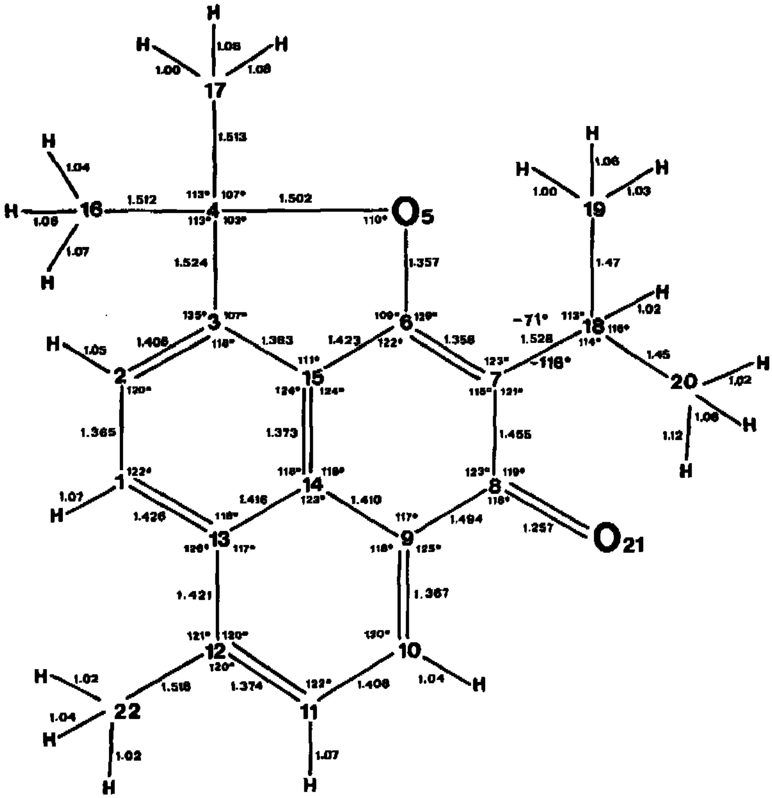

Fig. 4. *Bond lengths, bond angles and some torsion angles of* **3**, *averaged over the three independent molecules.* Standard deviations for averaged parameters involving C and O atoms are ≈ 0.008 Å for bond lengths, ≈ 0.7° for bond angles, and ≈ 1.0° for torsion angles.

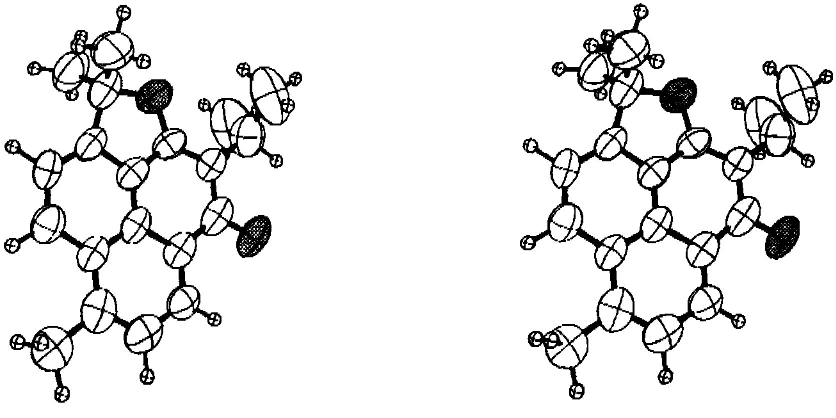

Fig. 5. *Stereoscopic projection of* **3** (oxygen atoms shaded). Molecule II is shown, but I and III are very similar. Anisotropic vibration ellipsoids at the 50% probability level, hydrogens with uniform, isotropic B = 1 Å².

In addition to the scale factor an isotropic extinction coefficient G [4] was introduced (final value G = 0.8 (1.8), $\overline{T}$ = 0.02 cm) resulting in a total of 776 parameters⁴). The final difference Fourier map showed no residual electron density higher than 0.2 e/Å³. Final positional parameters are given in Tab. 2.

Results. The numbering scheme and structural parameters averaged over the three independent molecules, are shown in Fig. 4, and a stereoscopic view showing the molecular conformation is given in Fig. 5. The presence of three independent molecules in the asymmetric unit allows an internal check of the accuracy of the structure determination. For this purpose the variances calculated for each set of three equivalent bond lengths have been averaged over the N=23 sets of bonds⁵).

$$< S^2 > \sum_{n=1}^{N} \left[\sum_{i=1}^{3} (d_{in} - \overline{d}_n)^2/2 \right] /N.$$

A value of $< S^2 >$ = 181.10⁻⁶ Å² was determined, corresponding to a mean e.s.d. of 0.013 Å for an individual bond, approximately equal to the mean LS-computed e.s.d. of 0.015 Å. For standard deviations of molecular parameters see legend to Fig. 4.

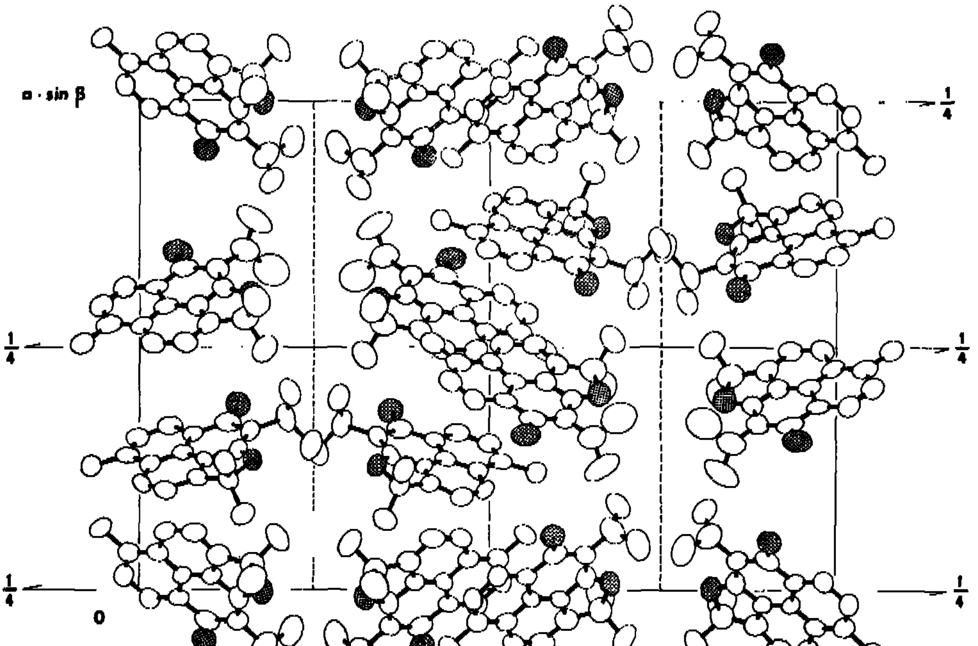

Fig. 6. *Packing diagram of* **3**. Atoms are represented by the envelope of the anisotropic vibration ellipsoid at the 50% probability level. Shaded atoms denote oxygen atoms, hydrogen atoms are omitted from the drawing.

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[4]) A list of structure factors (observed and calculated) and vibrational parameters can be obtained by the authors.

[5]) Bonds C(18)–C(19) and C(18)=C(20) have been omitted from these calculations because they appear to be severely affected by anisotropic vibration (see Fig. 4).

The conformations of the three molecules are very similar. In all three molecules the methine hydrogen H(18) is approximately syn-planar to C(8) = O(21) and the methyl group C(22) has one hydrogen atom syn-planar to C(11). The pattern of bond lengths in the pseudo-aromatic ring systems and of bond angle deformations induced by the fused furane ring are also similar in all three molecules. The tetracyclic phenalenone system is planar; the mean displacement of atoms from the LS-plane through the four rings including C(18), O(21) and C(22) is 0.025, 0.023, and 0.014 Å, respectively, for the three molecules. There is, however, no consistent pattern in the small but significant atomic displacement from planarity among the three molecules. There are no short intermolecular contacts (see packing diagram, Fig. 6).

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220. Aza-2-diènes-1,3

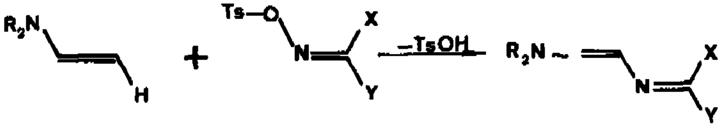
2. Synthèse et structure d'un ylure d'azométhine stable

par **Jean-Pierre Fleury, Jean-Paul Schoeni** et **Daniel Clerin**
Laboratoire de Chimie Organique Générale, Ecole Supérieure de Chimie, 68200–Mulhouse–F
et **Hans Fritz**
Laboratoire de Spectroscopie, Ciba-Geigy, Bale

(10. VI. 75)

2-Aza-1,3-dienes. 2. Synthesis and Structure of a Stable Azomethine-ylid. *Summary.* Thermal treatment of 2-(2′-cyano-2′-methoxycarbonyl)methylide-amino-1-morpholino-cyclohexene results in the formation of a stable isomere. Its azomethine-ylid structure is demonstrated from the spectroscopic data (UV., IR., ^{1}H- and ^{13}C-NMR.). 1,3-dipolar cycloaddition reactions of this ylid leads to spiropyrrolines.

Nous avons récemment décrit [1] une nouvelle synthèse d'aza-2-diènes **2**, basée sur la substitution du groupement sulfonyloxy de dérivés isonitrosomaloniques sulfonylés **1** par des énamines. Les azadiènes **2** sont des synthones intéressantes qui ouvrent des voies d'accès originales à différents systèmes hétérocycliques [2] [3].



1 **2**

(Ts = CH_3—C_6H_4—SO_2—*p*; X, Y = CN, COOR)

Dans ce mémoire, nous décrirons l'isomérisation de l'un de ces azadiènes en un ylure d'azométhine acyclique non substitué sur l'azote, qui représente à notre connaissance le premier exemple isolé et stable d'un tel groupement. Sa structure sera discutée sur la base des résultats spectroscopiques et nous donnerons un aperçu de sa réactivité.