

Structure of 9-Acetoxy-10-phenylanthracene*

BY ALEKSANDER ROSZAK

Institute of Chemistry, Nicolaus Copernicus University, Gagarina 7, 87-100 Toruń, Poland

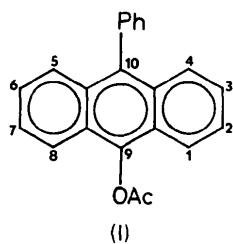
AND TERESA BOROWIAK

Faculty of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-780 Poznań, Poland

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Abstract. $C_{22}H_{16}O_2$, $M_r = 312.37$, monoclinic, $P2_1/c$, $a = 15.045$ (2), $b = 9.214$ (1), $c = 35.703$ (3) Å, $\beta = 96.13$ (1)°, $V = 4921.0$ (9) Å³, $Z = 12$, $D_m = 1.24$ (1), $D_x = 1.265$ Mg m⁻³, $\lambda(Cu K\alpha) = 1.54178$ Å, $\mu = 0.55$ mm⁻¹, $F(000) = 1968$, room temperature, $R = 0.066$ for 4485 unique observed reflections. Three molecules in the asymmetric unit have nearly the same conformation. The acetoxy group is almost perpendicular to the anthracene ring system, while the phenyl ring forms a dihedral angle in the range 73.0 to 79.7°.

Introduction. The structure of the title compound (I) was determined as a reference structure for 9-acetoxy-10-arylanthracenes, which all exhibit a marked fluorescence in organic solvents (Heldt, Heldt & Gronowska, 1975; Gronowska & Heldt, 1977; Gronowska, Aleksandrak & Heldt, 1981) and are studied as active media in dye lasers (Heldt, 1983).



Experimental. Light brown crystals grown from ethanol solution by slow evaporation, m.p. 438.1–439.5 K; space group and approximate cell dimensions determined from Weissenberg and oscillation photographs; D_m by flotation in aqueous KI solution; crystal size: 0.16 × 0.37 × 0.50 mm; Syntex $P2_1$ diffractometer, graphite-monochromated Cu $K\alpha$ radiation, θ – 2θ scan mode, background and intensity of reflections calculated by peak-profile analysis (Lehmann & Larsen, 1974); accurate cell parameters refined from setting angles of 15 reflections with $10 \leq 2\theta \leq 25$ °; max. $(\sin\theta)/\lambda = 0.5461$ Å⁻¹, $-16 \leq h \leq 16$, $0 \leq k \leq 10$, $0 \leq$

$l \leq 38$, two intensity standards [014: 472 (5), 22̄2: 739 (6)], 6230 unique reflections measured, 1745 unobserved reflections ($I < 1.96\sigma_I$); no absorption correction.

Solution by direct methods using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), best set of phases allowed location of 57 of 72 non-H atoms, remainder found by weighted Fourier synthesis. Refinement by block-diagonal least-squares procedures, initially with two molecules alternately in one block and third one in the second block, and finally with each molecule of the asymmetric unit in a single block using F magnitudes with *SHELX76* (Sheldrick, 1976); all H atoms at calculated positions riding with fixed isotropic displacement parameters; because of program limitations only 40 non-H atoms refined anisotropically and 32 (those with least differences between their U_{ii} values) isotropically; final $R = 0.066$, $wR = 0.093$ with $w^{-1} = \sigma^2(F_o) + 0.0007F_o^2$, $S = 2.55$; $(\Delta/\sigma)_{\max} = 0.09$ in final cycle; $(\Delta\rho)_{\max} = 0.20$ and $(\Delta\rho)_{\min} = -0.27$ e Å⁻³ on final difference Fourier map; atomic scattering factors from *SHELX76* (Sheldrick, 1976). Other computer programs used: *PLUTO* (Motherwell & Clegg, 1978) and *CRYPOZ* system (Jaskólski, 1982).

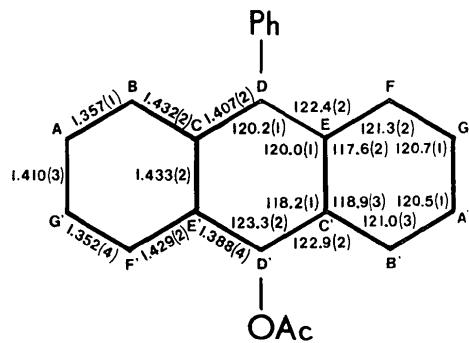
Discussion. Final positional parameters and U_{eq} or U_{iso} for non-H atoms are given in Table 1.† The projection on the plane of the anthracene ring system for each of the three molecules in the asymmetric unit with the atom labelling is shown in Fig. 1. Bond lengths are listed in Table 2.

The anthracene ring systems are not strictly planar (r.m.s. deviation 0.032, 0.029 and 0.040 Å for molecules *A*, *B* and *C*, respectively) and slightly bent along the C(9)–C(10) line. Outer rings, which are more

† Lists of structure factors, anisotropic displacement parameters, H-atom parameters, bond angles and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43422 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

* IUPAC name: 10-phenyl-9-anthryl acetate.

planar than the central ring, form dihedral angles of 3.1 (4) (*A*), 2.5 (4) (*B*) and 3.9 (4) $^{\circ}$ (*C*). These deformations are similar to those for the anthracene moiety in 9-acetoxy-10-(2-acetoxy-5-bromophenyl)-anthracene (*II*) (Roszak & Skrzat, 1985) and 9-acetoxy-10-(2,4-diacetoxyphenyl)anthracene (*III*) (Roszak & Borowiak, 1986).



The average values for the anthracene parameters for the three molecules in the asymmetric unit, assuming $mm2$ symmetry, are all very similar to the corresponding values for compound (III) [Roszak & Borowiak (1986); see Table 3 and discussion therein]. The influence of the 9- and 10-substituents is very clear when these values are compared with the 'average unsubstituted anthracene' (Harlow, Loghry, Williams & Simonsen, 1975) for which $CDE = C'D'E' = 121.0^\circ$ and $CD = C'D' = 1.399 \text{ \AA}$. The phenyl group, having σ -electron-releasing character (Domenicano & Murray-Rust, 1979) causes the angle CDE to decrease by about $0.8(1)^\circ$ and the bond CD to lengthen by $0.008(2) \text{ \AA}$, while the acetoxy group, having σ -electron-withdrawing character (Roszak & Borowiak, 1986) generates the opposite effect: increase of angle $C'D'E'$ by $2.3(2)^\circ$ and contraction of the $C'D'$ bond by $0.011(4) \text{ \AA}$. These changes agree well with the general theory of such substitution effects (Domenicano, Vaciago & Coulson, 1975). The differences between the individual bond lengths and angles for the phenyl rings in the three molecules are greater, which is due to the higher displacement parameters for these atoms. However, substitution effects on the internal angles of the ring are also visible; in particular the average value of $117.6(4)^\circ$ for the $C(16)-C(15)-C(20)$ angle agrees well with the expected value of 117.8° calculated on the basis of substituent angular parameters for the 9-acetoxy-10-anthryl group (Roszak & Borowiak, 1986).

The phenyl rings form dihedral angles of 74.0 (4), 79.7 (4) and 73.0 (4) $^{\circ}$ with their anthracene planes in molecules *A*, *B* and *C*, respectively. This angle is 69.9 (4) and 64.8 (4) $^{\circ}$ for compound (II) (for two molecules in the asymmetric unit), 68.2 (3) $^{\circ}$ for compound (III) and 84.4 (1) $^{\circ}$ for 9-acetoxy-10-(4-acetoxyphenyl)anthracene (IV) (Roszak & Duax,

Table 1. *Fractional coordinates and isotropic displacement parameters for the three molecules in the asymmetric unit with e.s.d.'s in parentheses*

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

Molecule A	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> or <i>U</i> _{eq} (Å ²)
C(1)	0.0951 (2)	-0.2573 (5)	0.3035 (1)	0.064 (1)†
C(2)	0.0826 (3)	-0.3997 (5)	0.2950 (1)	0.073 (2)†
C(3)	0.1567 (3)	-0.4926 (5)	0.2927 (1)	0.071 (1)
C(4)	0.2416 (3)	-0.4405 (4)	0.2994 (1)	0.063 (1)
C(5)	0.4458 (3)	-0.0210 (4)	0.3260 (1)	0.067 (1)†
C(6)	0.4575 (3)	0.1233 (5)	0.3325 (1)	0.075 (1)
C(7)	0.3825 (3)	0.2147 (5)	0.3348 (1)	0.071 (1)
C(8)	0.3000 (3)	0.1593 (4)	0.3306 (1)	0.064 (1)
C(9)	0.1989 (2)	-0.0509 (4)	0.3181 (1)	0.053 (1)
C(10)	0.3456 (2)	-0.2341 (4)	0.3139 (1)	0.054 (1)
C(11)	0.3592 (2)	-0.0854 (4)	0.3209 (1)	0.055 (1)
C(12)	0.2834 (2)	-0.0083 (4)	0.3234 (1)	0.054 (1)
C(13)	0.1830 (2)	-0.1961 (4)	0.3100 (1)	0.056 (1)†
C(14)	0.2584 (2)	-0.2904 (4)	0.3079 (1)	0.054 (1)
C(15)	0.4239 (2)	-0.3330 (4)	-0.3129 (1)	0.058 (1)†
C(16)	0.4478 (3)	-0.3884 (5)	0.2795 (1)	0.070 (1)†
C(17)	0.5198 (3)	-0.4825 (5)	0.2793 (1)	0.083 (2)†
C(18)	0.5687 (3)	-0.5220 (5)	0.3119 (1)	0.084 (2)†
C(19)	0.5465 (3)	-0.4654 (5)	0.3457 (1)	0.084 (2)†
C(20)	0.4747 (3)	-0.3742 (5)	0.3462 (1)	0.070 (1)†
C(21)	0.0821 (2)	0.1051 (4)	0.2931 (1)	0.063 (1)†
C(22)	-0.0003 (3)	0.1772 (5)	0.3030 (1)	0.083 (2)†
O(1)	0.1249 (2)	0.0359 (3)	0.3236 (1)	0.064 (1)†
O(2)	0.1110 (2)	0.1066 (4)	0.2632 (1)	0.087 (1)†
Molecule B				
C(1)	0.7588 (3)	-0.3997 (5)	0.5944 (1)	0.069 (1)†
C(2)	0.8365 (3)	-0.4675 (5)	0.6054 (1)	0.081 (2)†
C(3)	0.9186 (3)	-0.3919 (5)	0.6054 (1)	0.080 (1)
C(4)	0.9202 (3)	-0.2507 (5)	0.5946 (1)	0.069 (1)
C(5)	0.7535 (3)	0.1971 (4)	0.5532 (1)	0.065 (1)
C(6)	0.6740 (3)	0.2640 (5)	0.5438 (1)	0.076 (1)
C(7)	0.5930 (3)	0.1883 (6)	0.5447 (1)	0.081 (1)
C(8)	0.5922 (3)	0.0458 (5)	0.5543 (1)	0.073 (2)†
C(9)	0.6783 (2)	-0.1775 (4)	0.5729 (1)	0.061 (1)†
C(10)	0.8387 (2)	-0.0252 (4)	0.5736 (1)	0.055 (1)†
C(11)	0.7571 (2)	0.0462 (4)	0.5637 (1)	0.056 (1)
C(12)	0.6747 (2)	-0.0302 (4)	0.5635 (1)	0.059 (1)†
C(13)	0.7567 (2)	-0.2516 (4)	0.5831 (1)	0.057 (1)
C(14)	0.8393 (2)	-0.1741 (4)	0.5834 (1)	0.055 (1)
C(15)	0.9252 (2)	0.0553 (4)	0.5750 (1)	0.056 (1)†
C(16)	0.9798 (3)	0.0468 (6)	0.5469 (1)	0.103 (2)†
C(17)	1.0613 (3)	0.1184 (7)	0.5497 (2)	0.119 (2)†
C(18)	1.0879 (3)	0.1999 (6)	0.5798 (2)	0.091 (2)†
C(19)	1.0344 (4)	0.2131 (7)	0.6076 (1)	0.116 (2)†
C(20)	0.9527 (3)	0.1419 (6)	0.6048 (1)	0.104 (2)†
C(21)	0.5597 (3)	-0.3189 (4)	0.5430 (1)	0.064 (1)
C(22)	0.4733 (3)	-0.3871 (6)	0.5496 (1)	0.090 (2)†
O(1)	0.5966 (2)	-0.2506 (3)	0.5745 (1)	0.071 (1)†
O(2)	0.5934 (2)	-0.3197 (3)	0.5145 (1)	0.081 (1)†
Molecule C				
C(1)	-0.3380 (3)	-0.1137 (4)	0.3884 (1)	0.065 (1)
C(2)	-0.3539 (3)	-0.1513 (5)	0.4238 (1)	0.070 (1)
C(3)	-0.3298 (3)	-0.0577 (5)	0.4540 (1)	0.073 (1)
C(4)	-0.2895 (3)	0.0710 (4)	0.4488 (1)	0.065 (1)
C(5)	-0.1800 (2)	0.4317 (4)	0.3603 (1)	0.061 (1)
C(6)	-0.1711 (2)	0.4717 (5)	0.3244 (1)	0.066 (1)†
C(7)	-0.1980 (3)	0.3785 (5)	0.2942 (1)	0.066 (1)†
C(8)	-0.2336 (2)	0.2461 (4)	0.3003 (1)	0.057 (1)
C(9)	-0.2815 (2)	0.0658 (4)	0.3452 (1)	0.054 (1)
C(10)	-0.2310 (2)	0.2516 (4)	0.4056 (1)	0.055 (1)
C(11)	-0.2186 (2)	0.2947 (4)	0.3686 (1)	0.053 (1)
C(12)	-0.2446 (2)	0.2005 (4)	0.3377 (1)	0.051 (1)
C(13)	-0.2968 (2)	0.0216 (4)	0.3812 (1)	0.054 (1)
C(14)	-0.2710 (2)	0.1177 (4)	0.4121 (1)	0.054 (1)
C(15)	-0.2009 (2)	0.3480 (4)	0.4383 (1)	0.057 (1)†
C(16)	-0.1258 (3)	0.3113 (5)	0.4629 (1)	0.073 (1)†
C(17)	-0.1003 (3)	0.3989 (6)	0.4940 (1)	0.085 (2)†
C(18)	-0.1474 (3)	0.5205 (6)	0.5004 (1)	0.081 (2)†
C(19)	-0.2205 (3)	0.5589 (5)	0.4770 (1)	0.076 (2)†
C(20)	-0.2468 (3)	0.4717 (5)	0.4455 (1)	0.068 (1)†
C(21)	-0.2523 (3)	-0.1193 (5)	0.3020 (1)	0.069 (1)
C(22)	-0.2975 (3)	-0.2161 (5)	0.2726 (1)	0.079 (2)†
O(1)	-0.3127 (2)	-0.0253 (3)	0.3148 (1)	0.064 (1)†
O(2)	-0.1753 (2)	-0.1171 (4)	0.3136 (1)	0.099 (1)†

[†] Atom refined anisotropically.

1987). The acetoxy groups are planar and almost perpendicular to their anthracene planes with dihedral angles of 86.5 (4) (A), 89.7 (4) (B) and 82.7 (4)° (C), similar to the other 9-acetoxy-10-aryl-anthracenes (II, III, IV). Their geometry is very similar to the 'average acetoxy group' (Roszak, 1986; Hummel, Roszak & Bürgi, 1986) evaluated on the basis of 102 OAc groups bonded to aromatic six-membered carbon rings retrieved from the Cambridge Structural Database

Table 2. Bond lengths (Å) with mean values for the three molecules in the asymmetric unit

	Molecule A	Molecule B	Molecule C	Mean
C(1)—C(2)	1.354 (6)	1.346 (6)	1.357 (6)	1.352 (3)
C(1)—C(13)	1.433 (5)	1.421 (6)	1.427 (5)	1.427 (4)
C(2)—C(3)	1.414 (6)	1.419 (7)	1.398 (6)	1.410 (6)
C(3)—C(4)	1.362 (6)	1.358 (6)	1.354 (6)	1.358 (2)
C(4)—C(14)	1.432 (5)	1.426 (5)	1.433 (5)	1.430 (2)
C(5)—C(11)	1.425 (5)	1.439 (5)	1.434 (5)	1.433 (4)
C(5)—C(6)	1.358 (6)	1.356 (6)	1.353 (6)	1.356 (1)
C(6)—C(7)	1.417 (6)	1.407 (6)	1.405 (6)	1.410 (4)
C(7)—C(8)	1.336 (6)	1.357 (7)	1.359 (6)	1.351 (7)
C(8)—C(12)	1.433 (5)	1.433 (5)	1.425 (5)	1.430 (3)
C(9)—C(12)	1.378 (5)	1.397 (6)	1.397 (5)	1.391 (6)
C(9)—C(13)	1.384 (5)	1.378 (5)	1.392 (5)	1.385 (4)
C(9)—O(1)	1.401 (4)	1.408 (5)	1.412 (4)	1.407 (3)
C(10)—C(11)	1.404 (5)	1.403 (5)	1.408 (5)	1.405 (2)
C(10)—C(14)	1.406 (5)	1.416 (5)	1.404 (5)	1.409 (4)
C(10)—C(15)	1.493 (5)	1.494 (5)	1.500 (5)	1.496 (2)
C(11)—C(12)	1.441 (5)	1.425 (5)	1.427 (5)	1.431 (5)
C(13)—C(14)	1.437 (5)	1.434 (5)	1.435 (5)	1.435 (1)
C(15)—C(16)	1.378 (6)	1.366 (6)	1.395 (5)	1.380 (8)
C(16)—C(17)	1.389 (6)	1.386 (7)	1.394 (6)	1.390 (2)
C(17)—C(18)	1.357 (7)	1.337 (8)	1.357 (7)	1.350 (7)
C(18)—C(19)	1.389 (8)	1.347 (8)	1.355 (6)	1.364 (13)
C(19)—C(20)	1.370 (6)	1.388 (8)	1.403 (6)	1.387 (10)
C(20)—C(15)	1.395 (5)	1.357 (6)	1.372 (6)	1.375 (11)
C(21)—C(22)	1.481 (6)	1.485 (6)	1.487 (6)	1.484 (2)
C(21)—O(1)	1.363 (5)	1.357 (5)	1.369 (5)	1.363 (3)
C(21)—O(2)	1.194 (5)	1.183 (5)	1.188 (5)	1.188 (3)

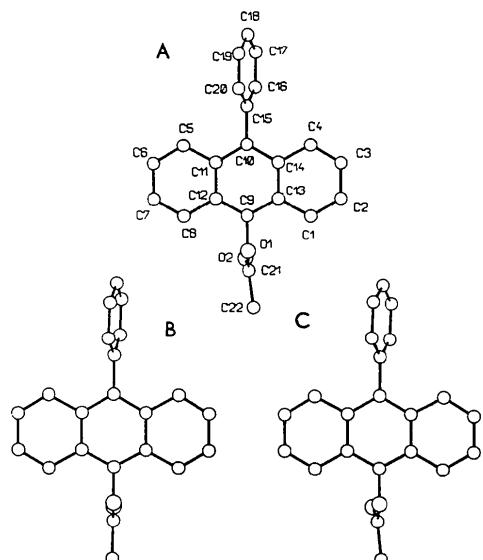


Fig. 1. View perpendicular to the plane of the anthracene ring system for the three molecules in the asymmetric unit.

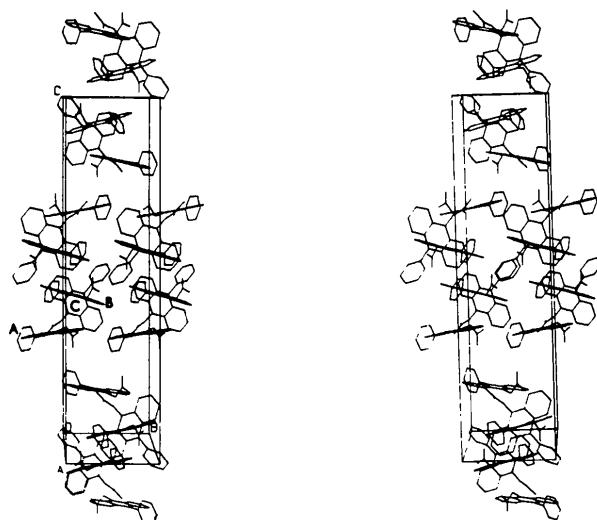


Fig. 2. Stereoscopic drawing of the packing of the molecules A, B and C in the unit cell.

(Allen *et al.*, 1979). All bond lengths, bond angles and torsion angles agree with the average values to within 1/3 of the standard deviation of the distribution in the sample.

In spite of significant differences [according to the Cruickshank & Robertson (1953) test] for some of their geometric parameters, the three molecules in the asymmetric unit have nearly the same conformation (see Fig. 1). Fig. 2 illustrates their arrangement in the unit cell. The anthracene planes of molecules A and B are almost perpendicular [83.1 (4) and 82.3 (4)°] to the anthracene plane of molecule C. The dihedral angle between the anthracene planes of molecules A and B is 25.9 (4)°. There are no unusually short intermolecular contacts in the structure.

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Acta Cryst. (1987). **C43**, 501–503

Hydroxy-1 Tricyclo[7.6.0.0^{2,7}]pentadécatriène-2,4,6 One-15 Ethylène-acétal*

PAR A. COURTOIS

Laboratoire de Minéralogie–Cristallographie, UA CNRS 809, Université de Nancy I, BP 239, 54506 Vandoeuvre CEDEX, France

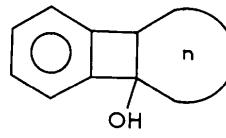
ET M. C. CARRÉ, P. CAUBÈRE ET B. GRÉGOIRE

Laboratoire de Chimie Organique I, UA CNRS 457, Université de Nancy I, BP 239, 54506 Vandoeuvre CEDEX, France

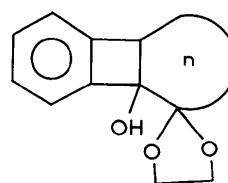
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Abstract. $C_{17}H_{22}O_3$, $M_r = 274 \cdot 3$, tetragonal, $P4_2/n$, $a = 20 \cdot 410(5)$, $c = 7 \cdot 176(3) \text{ \AA}$, $V = 2989 \text{ \AA}^3$, $Z = 8$, $D_x = 1 \cdot 22 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0 \cdot 71073 \text{ \AA}$, $\mu = 0 \cdot 77 \text{ cm}^{-1}$, $F(000) = 1184$, $T = 295 \text{ K}$, $R = 0 \cdot 071$, $wR = 0 \cdot 066$ for 1016 observed reflections. The title compound possesses *trans* geometry at the saturated ring–butenic ring junction. The characteristics of the benzocyclobutene part are identical to those of the other previously studied benzocyclobutenols with *cis* or *trans* geometry at the junction. Hydrogen bonds between the alcohol function and one oxygen of the ketal ring link two molecules to each other around centres of symmetry. Hydrogen CH...O bonds involving the second oxygen of the ketal ring connect these pairs of molecules in the crystal. This study and a previous one concerning the analogous benzocyclobutanol with a twelve-carbon saturated ring show that *trans* geometry exists for saturated rings of 9 to 12 carbons, contrary to the *cis* geometry found in previous works concerning benzocyclobutenols with smaller saturated rings.

Introduction. Les benzocyclobuténols (I) et des alcools analogues comportant un substituant sur le noyau aromatique sont des substrats intéressants tant du point de vue chimique (Caubère, 1978) que pharmacologique (Trockle, Catau, Barberi, Jacque, Carré & Caubère, 1981; Carré, Youlassani, Caubère, Saint-Aubin-Floch, Blanc & Advenier, 1984). Par condensation arynique d'énolates de cétones cycliques ces alcools n'ont été obtenus qu'avec un cycle saturé à $n = 5$, 6 ou 7 chainons et présentent tous une stéréochimie *cis* au niveau de la jonction du cycle buténique et du cycle saturé (Courtois, Protas, Guillaumet & Caubère, 1973a,b; Courtois, Protas, Mourad & Caubère, 1975, 1977).



(I)



(II)

Grâce à l'emploi d'énolates de cétones α -acétal, Grégoire, Carré & Caubère (1986) ont pu étendre la

* IUPAC: hydroxy-1 tricyclo[7.6.0.0^{10,15}]pentadécatriène-10,12,14 one-2 éthylène-acétal.