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Crystal data of 5,6-dehydrocavain and derivatives.*

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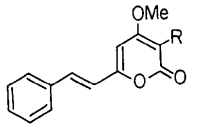
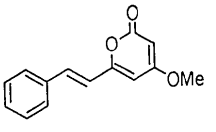
Crystals of 5,6-dehydrocavain (4-methoxy-6-*trans*-styryl-2-pyrone) are monoclinic, space group $P2_1/c$, with 4 molecules in a unit cell defined by $a=15{\cdot}11$, $b=4{\cdot}11$, $c=19{\cdot}92$ Å, $\beta=80°45'$. Crystals of 3-bromo-4-methoxy-6-*trans*-styryl-2-pyrone are triclinic, space group $P1$ or $P\bar{1}$, with 4 molecules in a unit cell defined by $a=14{\cdot}07$, $b=8{\cdot}20$, $c=13{\cdot}99$ Å, $\alpha=55°24'$, $\beta=89°15'$, $\gamma=83°16'$. Crystals of aniba-dimer A (a photodimer of dehydrocavain) are orthorhombic, space group $Pc2_1n$ with 4 molecules in a unit cell defined by $a=20{\cdot}662$, $b=10{\cdot}309$, $c=11{\cdot}048$ Å.

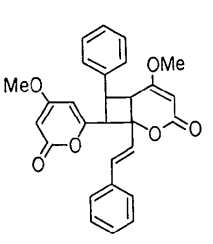
5,6-Dehydrocavain, usually represented as (I*a*), is a constituent of the leaves of *Aniba gardneri* (Meissn.) Mez, where it occurs accompanied by aniba-dimer A (II). The dimer can be synthesized by photo-irradiation of the monomer either in solution or in the solid state (von Bulow & Gottlieb, 1968; Andrade de Mata Rezende, von Bulow, Gottlieb, Lamêgo Vieiro Pinho & da Rocha 1971). The unsolved structural problems inherent in these compounds refer to the geometry of dehydrocavain (I*a* or III) and the stereochemistry of aniba-dimer A (II). To aid the solution of these problems, it was necessary to obtain the crystal data of the compounds. The data were obtained using a Weissenberg camera; rotation, oscillation and equi-inclination photographs of all samples were taken with filtered Cu $K\alpha$ radiation ($\lambda=1{\cdot}5418$ Å).



(Ia R = H)
(Ib R = Br)



(III)



(II)

(1) Dehydrocavain ($C_{14}H_{12}O_3$) (I*a*), m.p. 139–140° (recrystallized from benzene), consists of greenish-yellow needles, a few tenths of a millimetre in width and about 2 to 3 mm in length. The crystals, always twinned on the (100) plane, are characterized by the following data:

System: monoclinic	Space group: $P2_1/c$
$a=15{\cdot}11(2)$ Å	$d_{obs}=1{\cdot}24(2)$ g cm^{-3}
$b=4{\cdot}11(2)$	$d_{calc}=1{\cdot}24(1)$ g cm^{-3}
$c=19{\cdot}92(2)$	$Z=4$.
$\beta=80°45'$	

(2) 3-Bromo-4-methoxy-6-*trans*-styryl-2-pyrone ($C_{14}H_{11}BrO_3$) (I*b*), m.p. 210-212° (recrystallized from benzene), was prepared by direct bromination of (I*a*) in $CHCl_3$ at 0°. The reaction product was separated from unchanged starting material by silica column chromatography, using benzene as eluent. Its structure was determined by mass [*M* 308 (100%) and 306 (98%)] and p.m.r. [$CDCl_3$, τ: 2·3–2·7 (multiplet, $C_6\mathbf{H}_5$), 2·38 (doublet, *J* 16·0 Hz, C_6H_5 **CH**), 3·28 (doublet, *J* 16·0 Hz C_6H_5 CH**CH**), 3·78 (singlet, pyrone **CH**), 5·93 (singlet OCH_3)] spectroscopy. The crystals, in external morphology similar to dehydrocavain, are characterized by the following data:

System: triclinic	Space group: $P1$ or $P\bar{1}$
$a=14{\cdot}07(4)$ Å	$d_{obs}=1{\cdot}58(2)$ g cm^{-3}
$b=8{\cdot}20(3)$	$d_{calc}=1{\cdot}55(1)$ g cm^{-3}
$c=13{\cdot}99(3)$	$Z=4$.
$\alpha=55°24'$	
$\beta=89°15'$	
$\gamma=83°16'$	

(3) Aniba-dimer A ($C_{28}H_{24}O_6$) (II), m.p. 178-179° (recrystallized from benzene), consists of crystals which exhibit a tabular habit, with length of the order of 2·5 to 3·0mm and width of about 0·5 by 1mm. They are orthorhombic and the systematic extinctions are compatible with both *Pcmn* and $Pc2_1n$. The former may be discarded, since it requires the compound to be exactly planar. The cell dimensions of this compound were measured using an automatic single crystal diffractometer of type $P\bar{1}$ from Syntex Analytical Instruments.

System: orthorhombic	Space group: $Pc2_1n$
$a=20{\cdot}662(4)$ Å	$d_{obs}=1{\cdot}28(2)$ g cm^{-3}
$b=10{\cdot}309(6)$	$d_{calc}=1{\cdot}220(1)$ g cm^{-3}
$c=11{\cdot}048(7)$	$Z=4$.

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References

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