Crystal Structure of Maturin acetate from *Psacalium peltatum* (Kunth) (*matarique*)

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Maturin acetate is a furanoeremophilane natural product compound (C₁₈H₁₆O₅) (4-formyl-9-meth-oxy-5-methyl-naphtho[2,3-b]furan-3-yl)methyl acetate (**I**), was isolated from *Psacalium peltatum* (Kunth), named *matarique*. The crystal has a monoclinic system, space group *P*2₁/*c*, *Z* = 4; the unit cell dimensions are: *a* = 19.205(3) Å, *b* = 10.356(2) Å, *c* = 7.695(1) Å, β = 97.236(3)°. The structure is essentially planar; the molecules in the crystal are joined by a weak interaction C–H–O and π - π stacking.

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Psacalium peltatum (Kunth) Cass. is an endemic medicinal plant member of the *matarique* complex, widely distributed in the central part of Mexico. The roots of *P. peltatum* have been employed as alcoholic maceration to treat conditions that induce inflammation from such as wounds, skin ulcers and rheumatism.¹ Our research has shown that furanoeremophilane-type sesquiterpene, maturin acetate (**I**) is the main constituent of this resin. Although maturin acetate has been isolated as the most abundant natural product in of Mexican species, such as *P. beamanii*,² *Roldana angulifolia*,³ and *Trichilia cuneata*,⁴ no report on the crystal structure determination of this compound has appeared. Therefore, due to this lack of data and x-ray studies, the crystal structure determination of maturin acetate was undertaken.

Roots of *Psacalium peltatum* (Kunth) Cass., were collected from a pine-oak forest of Mineral del Chico, Hidalgo, Mexico, [20°09'55" N and 98°45'08" W]. A voucher specimen was deposited at the National Herbarium (MEXU 1138692) of the Institute of Biology, UNAM, Mexico. Air-dried and powdered roots (4.381 kg) of *P. peltatum* were sequentially extracted with hexane by exhaustive maceration (3 times $\times$ 2 L), at room temperature. Hexane extract of roots from *P. peltatum*, was separated in a column chromatographic process and eluted

with hexane-ethyl acetate in a mixture gradient, from fraction (98:2) eluted of hexane-ethyl acetate was isolated Maturin acetate and their spectroscopic features were compare with the described data.^{5,6}

A yellow crystal prism was mounted on a glass fiber. The X-ray intensity data were measured at 298 K on a Bruker Smart

Table 1 Crystal and experimental data

Chemical formula	C ₁₈ H ₁₆ O ₅
Formula weight	312.31
Temperature	298(2)K
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>c</i>
Cell dimensions	<i>a</i> = 19.205(3) Å <i>b</i> = 10.356(2) Å <i>c</i> = 7.695(1) Å β = 97.236(3)°
Volume	1518.3(4) Å ³
<i>Z</i>	4
<i>D</i> _x	1.366 Mg/m ³
Radiation	0.71073 Å
μ (Mo <i>K</i> α)	0.100 mm ⁻¹
<i>F</i> (0 0 0)	656
Crystal size	0.31 $\times$ 0.12 $\times$ 0.05 mm
No. of reflections collected	12272
No. of Independent reflections	2800
θ range for data collection	2.14 to 25.39°
Data/restraints/parameters	2800/0/211
Goodness-of-fit on <i>F</i> ²	0.821
Final <i>R</i> indices [<i>I</i> > 2 σ (<i>I</i>)]	<i>R</i> 1 = 0.0466, <i>wR</i> 2 = 0.0891
<i>R</i> indices (all data)	<i>R</i> 1 = 0.1129, <i>wR</i> 2 = 0.1008
(Δ / σ) _{max}	0.036
($\Delta\rho$) _{max}	0.192
($\Delta\rho$) _{min}	-0.219 e. Å ⁻³
Measurement	Bruker Smart APEX AXS CCD area detector/
Program System	Smart
Structure Determination	SHELXS-97
Refinement	SHELXL-97
CCDC deposition number	882591

Fig. 1 Scheme diagram.

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Fig. 2 Maturin acetate structure showing the atomic numbering scheme. The thermal ellipsoids are drawing at 40% of probability.

Apex diffractometer with a CCD area detector system equipped with Mo K_{α} radiation ($\lambda = 0.71073$ Å). The Maturin acetate ($C_{18}H_{16}O_5$) crystallized in a monoclinic system. A total of 1800 frames were collected with a scan width of 0.3° in ω and an exposure time of 10 s/frame. The frames were integrated with the Bruker Saint software package,⁷ using a monoclinic unit cell. A total of 12272 reflections were collected in a range of $2.14 \geq \theta > 25.39^\circ$, of which 2800 ($R_{\text{int}} = 0.074$) reflections with $I > 2\sigma(I)$ were independent. The structure was solved by direct methods using the SHELXS-97 program.⁸ A least-squares refinement was based on the full matrix was carried out using the SHELXL-97 program.⁸ The non-hydrogen atoms were refined with anisotropic thermal parameters. Hydrogen atoms were input at calculated positions, and allowed to ride on the atoms to which they became attached. The isotropic thermal parameters were refined for H-atoms using a $U_{\text{eq}} = 1.2$ times the atom to which they are attached. The goodness-of-fit on F^2 was 0.821. The final indices were $R_1 = 0.0466$, $wR_2 = 0.089$ [$I > 2\sigma(I)$]. Crystallographic data (excluding structure factors) for maturin acetate were deposited with the Cambridge Crystallographic Data Center as supplementary publication No. CCDC 882591.

Figure 2 shows an ORTEP drawing of Maturin acetate with thermal ellipsoids drawn at 40% probability. Details of the crystal and refinement are given in Table 1. The final atomic

coordinates and equivalent thermal parameters for all non-hydrogen atoms are given in Table S1. The bond distances and bond angles for all non-hydrogen atoms are given in Table S2. Hydrogen coordinates are given in Table S3.

Maturin acetate has a furanoeremophilane skeleton (Fig. 1) and the structure is essentially planar [O1–C9 atoms 0.0126(21) Å]; the acetate group rotates around the C10–O2 bond by $15.4(1)^\circ$ and the formyl group forms a dihedral angle of $9.4(2)^\circ$ with a planar structure. The bond lengths and angles in the maturin acetate exhibit normal values.⁹ The compound in the crystal is stabilized by weak inter-molecular interaction C–H–O (C=O–H–C) and type π – π .

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