X-ray Structure Analysis Online

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

Nadia ROJANO-VILCHIS, Simón HERNÁNDEZ-ORTEGA,† Manuel JIMENEZ-ESTRADA, and Armando TORRES-AVILEZ

Instituto de Química,Universidad Nacional Autónoma de México circuito exterior, ciudad universitaria, México 04510, México, D.F, México

Maturin acetate is a furanoeremophilane natural product compound ($C_{18}H_{16}O_5$) (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 $P2_1/c$, $Z = 4$; 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.

(Received May 26, 2012; Accepted June 30, 2012; Published on web October 10, 2012)

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 × 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) elueted 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 fibber. The X-ray intensity data were measured at 298 K on a Bruker Smart

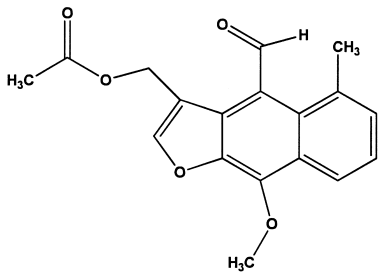

Fig. 1 Scheme diagram.

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	$P2_1/c$
Cell dimensions	a = 19.205(3)Å
	b = 10.356(2)Å
	c = 7.695(1)Å
	β = 97.236(3)˚
Volume	1518.3(4)Å^3
Z	4
D_x	1.366 Mg/m^3
Radiation	0.71073 Å
μ(Mo K_α)	0.100 mm^{-1}
F(0 0 0)	656
Crystal size	0.31 × 0.12 × 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 F^2	0.821
Final R indices [$I > 2\sigma(I)$]	$R1$ = 0.0466, $wR2$ = 0.0891
R indices (all data)	$R1$ = 0.1129, $wR2$ = 0.1008
$(\Delta/\sigma)_{max}$	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

† To whom correspondence should be addressed.
E-mail: shernandezortega@gmail.com

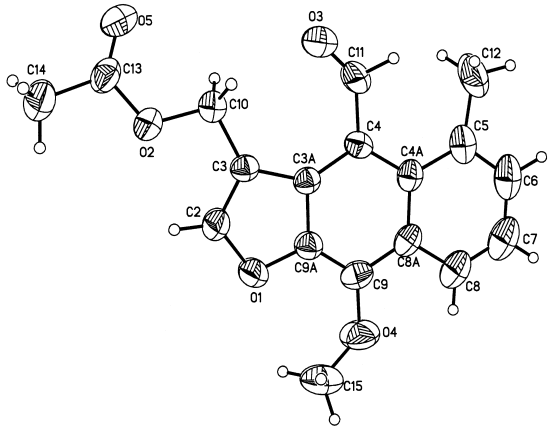

Fig. 2 Maturin acetate structure showing the atomic numbering scheme. The thermal ellipsoids are drawing at 40% of probability.

Apex diffractometter with a CCD area detector system equipped with Mo K_α radiation (λ = 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 $2.14 \geq \theta > 25.39$˚, of which 2800 (R_{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 SHEXL-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_{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)˚ and the formyl group forms a dihedral angle of 9.4(2)˚ 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.

Acknowlegments

N. A. Rojano-Vilchis acknowledges scholarship and financial support provided by the Consejo Nacional de Ciencia y Tecnologia (CONACyT: 101038), and Programa en Ciencias Biológicas of the Universidad Nacional Autónoma de México (UNAM). Thanks are given to the Consejo Superior de Investigaciones Cientificas (CSIC) of Spain for the award of a licence for the use of the Cambridge Crystallographic Data Base (CSD).

References

1. R. Bye, E. Linares, and H. Estrada, *Phytochemistry of Medicinal Plants.*, **1995**, Plenum Press New York., p. 65.
2. A. L. Perez, A. Arciniega, J. L. Villaseñor, and A. Romo de Vivar, *Rev. Soc. Quím. Méx.*, **2004**, *48*, 21.
3. A. Arciniegas, A. L. Pérez, J. L. Villaseñor, and A. Romo de Vivar, *J. Nat. Prod.*, **2006**, *69*, 1826.
4. M. Doe, Y. Hirai, T. Kinoshita, K. Shibata, H. Haraguchi, and Y. Morimoto, *Chem. Lett.*, **2004**, *33*, 714.
5. J. Correa and J. Romo, *Tetrahedron*, **1966**, *22*, 685.
6. F. Bohlmann, C. Zdero, and M. Grenz, *Chem. Ber.*, **1977**, *110*, 474.
7. Bruker (2007). SAINT & SMART. Bruker AXS Inc. Madison, Wisconsin, USA.
8. G. M. Sheldrick, *Acta Cryst.*, **2008**, *A64*, 112.
9. F. H. Allen, O. Kennard, D. G. Watson, L. Brammer, A. G. Orpen, and R. Taylor, *J. Chem. Soc. Perkin Trans.*, **1987**, *2*, p. S1.