Phytochemistry, Vol. 28, No. 7, pp. 1973–1974, 1989.
Printed in Great Britain.

0031–9422/89 \$3.00+0.00
Pergamon Press plc.

SHORT REPORTS

EUDESMANE DERIVATIVES FROM *EPALTES MEXICANA*

EMMA MALDONADO

Instituto de Quimica, Universidad Nacional Autonoma de Mexico, Circuito Exterior, Cd. Universitaria, Coyoacan 04510, Mexico, D.F.

(*Received in revised form* 16 *November* 1988)

Key Word Index—*Epaltes mexicana*; Compositae; Inulae; eudesmane derivatives; sesquiterpenoids.

Abstract—Investigation of *Epaltes mexicana* afforded several eudesmane derivatives, all of them closely related to cuauhtemone. The structures of two new compounds were elucidated by spectroscopic methods.

INTRODUCTION

Investigation of *Epaltes* species has shown that eudesmane derivatives are common in this genus [1, 2]. In continuation of our work on the chemistry of the Compositae we have isolated from *E. mexicana*, the new compounds **4** and **7** together with the previously described β-sitosterol, stigmasterol and the cuauhtemone derivatives **1**, **2** [3], **3** [4], **5** [5] and **6** [6].

RESULTS AND DISCUSSION

The spectral data of compound **4** were similar to those of the known compounds **1** and **2** [3]. The only difference between these substances was the nature of the ester residue on C-3, which in the case of **4** corresponds to the 2′,3′-dihydroxy-2′-methylbutyrate, characterized by the broad quartet at δ 3.95 ($J = 7$ Hz, sharp *q* upon D_2O equilibration) assigned to H-3′, as well as by the doublet at δ 1.23 ($J = 7$ Hz) and the singlet at δ 1.34 attributed to the protons at C-4′ and C-5′, respectively.

The structure of compound **7** was established as the angeloyl analogue of **6**. Since the ^1H NMR data of **7** only differ from those of **6** [6] in the signals corresponding to the ester group. The ^{13}C NMR data (see Experimental) also supports structure **7**.

Compound **7** undoubtedly is a natural constituent of *E. mexicana*, while compound **4** could be produced from **1** during the isolation process, since a benthonitic earth, which promotes epoxide ring opening [7], was used. In order to clarify this, a $CHCl_3$ solution of **1** was treated with this earth and compound **4** was formed, confirming the above assumption.

The chemistry of *E. mexicana* showed a close relationship with previously analysed *Epaltes* species and supports the placement of this genus in the *Pluchea* group.

EXPERIMENTAL

Ground dried aerial parts of *E. mexicana* Less (682 g) collected in Catazaja, Chiapas State, Mexico (specimen in deposit in the National Herbarium, AOH-208), were extracted with hexane (5 l), Me_2CO (5 l) and MeOH (3 l) affording 35.4, 14.2 and 9.5 g of residue, respectively. The hexane extract was fractionated on a silica gel column eluted with a hexane–EtOAc gradient. The less polar frs were discarded. The remaining frs were combined with the Me_2CO and MeOH extract, decolorized with benthonitic earth ('Tonsil' [7]) and subsequently with activated charcoal to obtain 31.2 g of extract, which after successive CC (silica gel Merck G) afforded 5.52 g of **1** [3], 2.25 g of **2** [3], 1.29 g of **3** [4], 62 mg of **4**, 21.7 mg of **5** [5], 72 mg of **6** [6] and 96.4 mg of **7**.

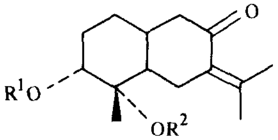
3-O-[*2,3-dihydroxy-2-methylbutyroyl*]*Cuauhtemone* (**4**). Pale yellow oil, IR $\nu_{\max}^{CHCl_3}$ cm^{-1}: 3510, 1730, 1675, 1595, 1450, 1390, 1195, 1130, 1080, 1020, 980, 908. CIMS *m/z* (rel. int.): 369 $[M+1]^+$ ($C_{20}H_{32}O_6$, 6); 351 $[M+1-H_2O]^+$ (87); 235 $[M+1-C_5H_{10}O_4]^+$ (55); 217 $[351-C_5H_{10}O_4]^+$.

Table 1. ^1H NMR spectral data of compounds **4** and **7** (80 MHz, $CDCl_3$, TMS int. standard)

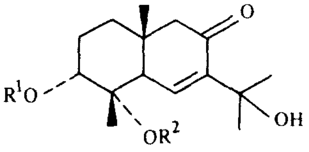
H	**4**	**7**
3	4.96 *dd*	4.96 *dd*
	3.5, 2.5	3.5, 3
6	2.92 *br d*	7.03 *d*
	12	2.5
9	2.22 *s*	2.33 *s*
12	2.05 *s*	1.46 *s*
13	1.83 *s*	1.45 *s*
14	0.96 *s*	1.01 *s*
15	1.34 *s*	1.30 *s*
3′	3.95 *br q*	6.12 *qq*
	7	7, 1.5
4′	1.23 *d*	2.03 *dq*
	7	7, 1.5
5′	1.28 *s*	1.99 *br s*

---

*Contribution No. 955 of Instituto de Química, UNAM.



1 R^1 = 2′,3′ – epoxy – 2′ – methylbutyroyl; R^2 = H
2 R^1 = Tigloyl; R^2 = H
3 R^1 = 2′,3′ – epoxy – 2′ – methylbutyroyl; R^2 = Ac
4 R^1 = 2′,3′ – dihydroxy – 2′ – methylbutyroyl; R^2 = H



5 R^1 = 2′,3′ – epoxy – 2′ – methylbutyroyl; R^2 = Ac
6 R^1 = 2′,3′ – epoxy – 2′ – methylbutyroyl; R^2 = H
7 R^1 = Angeloyl; R^2 = H

3α-*Angeloyloxy*-4α,11-*dihydroxyeudesm*-6-*en*-8-*one* (**7**). Colourless oil, $[\alpha]_D + 80.12$ ($CHCl_3$; *c* 6:64); IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 3510, 1720, 1660, 1453, 1394, 1350, 1240, 1150, 1080, 1010, 980. CIMS *m/z* (rel. int.): 350 $[M]^+$ ($C_{20}H_{30}O_5$); 333 $[M+1-H_2O]^+$ (24); 315 $[M+1-2H_2O]^+$ (100); 251 $[M+1-AngOH]^+$ (40); 215 $[315-AngOH]^+$ (19). ^{13}C NMR (20 MHz, $CDCl_3$, δ in ppm): 33.17 *t*. (C-1); 24.09 *t* (C-2); 75.73 *d* (C-3); 71.92* *s* (C-4); 50.61 *d* (C-5); 142.56 *d* (C-6); 145.12 *s* (C-7); 200.76 *s* (C-8); 58.61 *t* (C-9); 39.17 *s* (C-10); 71.23 *s* (C-11); 29.40 *q* (C-12); 28.81 *q* (C-13); 17.89 *q* (C-14); 22.71 *q* (C-15); 167.29 *s* (C-1′); 127.28 *s* (C-2′); 138.93 *d* (C-3′); 20.77 *q* (C-4′); 15.95 *q* (C-5′).

Preparation of **4** *from* **1**. Benthonitic earth (‘Tonsil’, 3 g) [7], was added to a soln of **1** (194.4 mg) in $CHCl_3$ (15 ml). The suspension was left to stand for 4 days. After filtration and solvent elimination, 198 mg of residue was obtained, which was purified by CC (silica gel Merck G, EtOAc–hexane, 2:3) to give 70.3 mg of **4**.

Acknowledgements—We are indebted to M. Sc. Jose Luis Villaseñor, National Herbarium, UNAM, for the identification of plant material. We also thank Messrs Jorge A. Cárdenas, R. Gaviño, R. Villena, M. Torres and L. Velasco for technical assistance.

REFERENCES

1. Bohlmann, F., Borthakur, N., Robinson, H. and King, R. M. (1982) *Phytochemistry* **21**, 1795.
2. Rojatkar, S. R., Puranik, V. G., Tavale, S. S., Guru Row, T. N. and Nagasampagi, B. A. (1987) *Phytochemistry* **26**, 569.
3. Bohlmann, F. and Zdero, C. (1976) *Chem. Ber.* **109**, 2563.
4. Nakanishi, K., Crouch, R., Miura, I., Dominguez, X., Zamudio, A. and Villarreal, R. (1974) *J. Am. Chem. Soc.* **96**, 609.
5. Bohlmann, F. and Mahanta, P. K. (1978) *Phytochemistry* **17**, 1189.
6. Arriaga-Giner, F. J., Borges-del-Castillo, J., Manresa-Ferrero, M. T., Vázquez-Bueno, P., Rodríguez-Luis, F. and Valdés-Iraheta, S. (1983) *Phytochemistry* **22**, 1767.
7. Salmón, M., Penieres, G., Miranda, R. and Alvarez, C. (1981) *J. Heterocyc. Chem.* **18**, 1475.