

## A New Xanthanolide from *Carpesium longifolium*

Chao YANG, Meng Chun YE, Zhong Jian JIA\*

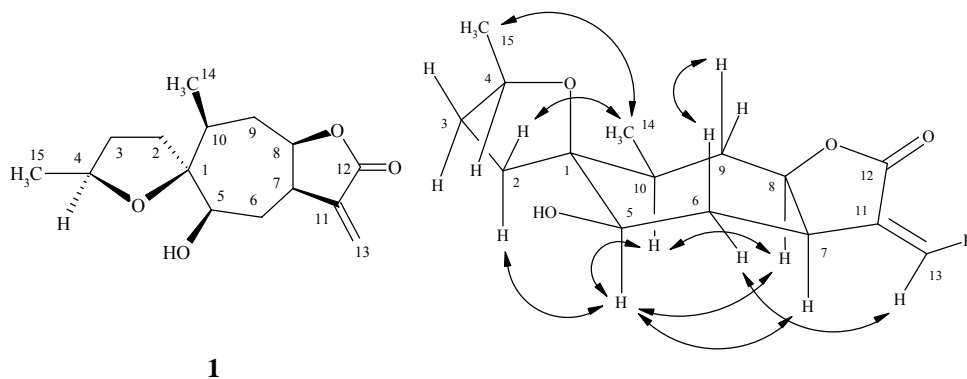
Department of Chemistry, National Laboratory of Applied Organic Chemistry,  
Lanzhou University, Lanzhou 730000

**Abstract:** A new xanthanolide was isolated from the aerial parts of *Carpesium longifolium*. Its structure was elucidated as 1 $\beta$ , 4 $\beta$ -epoxy-5 $\beta$ -hydroxy-10 $\alpha$ H-xantha-11(13)-en-12, 8 $\beta$ -olide by spectral methods (HRMS, 1D and 2D NMR).

**Keywords:** *Carpesium longifolium*, Compositae, xanthanolide.

No previous work on the xanthane-type sesquiterpene of the genus *Carpesium* has been found up to now. A new xanthanolide **1** was isolated from *Carpesium longifolium* Chen et C. M. Hu. Here we report the structure elucidation of it.

**Figure 1** Key NOE correlation of compound **1**



Compound **1**, C<sub>15</sub>H<sub>22</sub>O<sub>4</sub> (HRMS: revealed [M+H]<sup>+</sup> = 267.1590, requires 267.1591), was isolated as colorless oil, [ $\alpha$ ]<sub>D</sub><sup>25</sup> +23.0 (*c* 1.71, CHCl<sub>3</sub>). Its IR spectrum exhibited strong absorptions at 3400, 1759, 1659, 1086 cm<sup>-1</sup>. The <sup>1</sup>H and <sup>13</sup>C NMR spectra of **1** (**Table 1**) indicated the presence of a  $\alpha$ -methylene- $\gamma$ -lactone moiety, one oxygenated quaternary carbon, two >CH-O- units and two >CH-CH<sub>3</sub> units. Further <sup>1</sup>H-<sup>1</sup>H COSY experiments revealed two partial structures of compound **1**: CH<sub>3</sub>-CH(-O)-CH<sub>2</sub>-CH<sub>2</sub>- and CH(CH<sub>3</sub>)-CH<sub>2</sub>-CH(-O)-CH(-)-CH(-O)-. The C-C interconnectivity of all the

\*E-mail: jiazj@lzu.edu.cn

fragments was established through cross peaks in HMBC experiment (see **Table 1**). The above information suggested the basic structure of **1** was a xanthanolide. An epoxy bridge was required for the molecular formula  $C_{15}H_{22}O_4$ , and the typical furan ring chemical shift of C-1 at  $\delta$  91.0 and C-4 at  $\delta$  78.0 indicated the epoxy bridge was between C-1 and C-4<sup>1</sup>. Finally, the stereochemistry of **1** was established by <sup>1</sup>H-<sup>1</sup>H NOESY spectra. Clear NOE correlations (shown by arrows in **Figure 1**) between H-2a and H-5, H-2b and H-14, H-6 $\alpha$  and H-13', H-6 $\beta$  and H-9 $\beta$ , H-8 and H-10, and H-5 and H-7, H-8, H-10 indicated that H-5, H-7, H-8 and H-10 were  $\alpha$ -configuration, the hydroxy at C-5 and the epoxy bridge at C-1 were in  $\beta$ -orientation. The stereochemistry at C-4 was deduced from correlation between H-14 and H-15. Hence, compound **1** was identified as 1 $\beta$ , 4 $\beta$ -epoxy-5 $\beta$ -hydroxy-10 $\alpha$ H-xantha-11(13)-en-12, 8 $\beta$ -olide.

**Table 1** <sup>1</sup>H, <sup>13</sup>C NMR (DEPT) and HMBC data of **1** (CDCl<sub>3</sub>, TMS,  $\delta$  ppm)<sup>a, b</sup>

No.	<sup>1</sup> H ( $\alpha$ / $\beta$ )	<sup>13</sup> C	DEPT	HMBC (C / H)
1	-	91.0	C	C-1 / H-3, 6, 9, 14
2	2.11 (brdd, 12.2, 8.6) 1.71 (brdd, 12.2, 4.0)	24.9	CH <sub>2</sub>	C-2 / H-5, 10
3	2.01 (m) 1.47* (m)	35.5	CH <sub>2</sub>	C-3 / H-15
4	4.18 (ddq, 5.8)	78.0	CH	C-4 / H-2
5	3.59 (br.d, 10.0)	77.5	CH	C-5 / H-2, 10
6	2.03 (dd, 15.0, 5.6) 1.65 (ddd, 15.0, 12.0, 10.0)	33.3	CH <sub>2</sub>	-
7	3.34 (dddd, 12, 9, 5.6, 3.2)	38.8	CH	C-7 / H-5, 9, 13
8	4.64 (ddd, 12.0, 9.0, 3.3)	80.5	CH	C-8 / H-6, 10
9	1.79 (brdd, 14.0, 3.3) 1.50* (ddd, 14.0, 12.0, 11.0)	35.3	CH <sub>2</sub>	C-9 / H-14
10	1.58* (brdd, 11.0, 6.7)	37.3	CH	C-10 / H-2, 5
11	-	138.9	C	C-11 / H-6
12	-	169.6	C	C-12 / H-13
13	6.28 (d, 2.9) 5.59 (d, 2.9)	122.8	CH <sub>2</sub>	-
14	1.03 (d, 6.7)	18.4	CH <sub>3</sub>	C-14 / H-9
15	1.29 (d, 5.8)	20.5	CH <sub>3</sub>	C-15 / H-3

a. Signal multiplicity and coupling constants (Hz) are in parentheses;

b. \*Overlapping signals.

### Acknowledgment

We are grateful for the NNSFC (No.29972017).

### References

1. Ahmed A. Mahmoud, *Planta Medica*, **1998**, *64*, 724.

Received 10 June, 2002