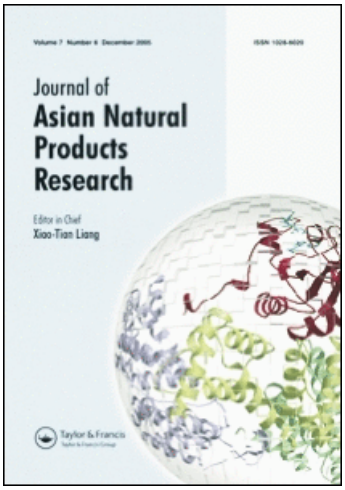


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# A new diterpene from the processed roots of *Euphorbia Kansui*

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# A new diterpene from the processed roots of *Euphorbia Kansui*

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A new diterpene has been isolated from the processed roots of *Euphorbia Kansui*. By means of physico-chemical evidences and spectral analysis, the structure was identified as 4-*O*-acetyl-5-*O*-benzoyl-3$\beta$-hydroxy-20-deoxyingenol (**1**).

*Keywords*: *Euphorbia Kansui*; Diterpene; Ingenol; Deoxyingenol

## 1. Introduction

The toxicity of *Euphorbia Kansui* has been known for a long time. The plant was recorded in *Sheng Nung's Herbal* as a low-grade drug and has been used as a herbal remedy for edema, ascites [1,2] and cancer [3] in China. The toxicity could be reduced greatly by roasting it with rice vinegar [4]. We studied the processed *kansui* in order to investigate its chemical constituents. A new diterpene was isolated as 4-*O*-acetyl-5-*O*-benzoyl-3$\beta$-hydroxy-20-deoxyingenol (**1**). In this paper, we mainly report the isolation and structural elucidation of compound **1**.

## 2. Results and discussion

Compound **1** was obtained as colourless oil. The molecular formula of **1** was deduced as $C_{29}H_{36}O_6$ by HRESIMS at *m/z* 480.2503$[M]^+$. The $^1$H NMR spectrum contained signals at $\delta_H$ 6.14 (s, H-1), 5.89 (d, $J = 3.1$ Hz, H-7), 5.05 (s, H-3), 4.27(m, H-8), 5.54(s, H-5), 3.30(s, OH-3), 2.34 (m, H-12) and 1.75 (m, H-12), 2.54(m, H-11), 1.56(s, H-20), 0.95 (m, H-14), 1.07 (s), 1.13(s), 1.01(d, $J = 7.1$ Hz), 1.78(s)(Me-16, 17, 18, 19), which were very similar to those of 20-deoxyingenol [5]. Analysis of the HSQC and HMBC spectra indicated that compound **1** contained two ester groups at $\delta_H$ 2.06 (3H, s), $\delta_C$ 172.6, 21.2 (acetyl); $\delta_H$ 8.14

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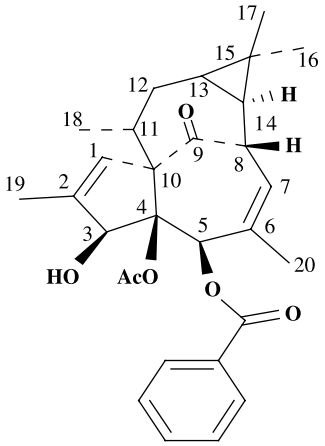


Figure 1. Structure of **1**.



(2H, d, $J = 7.8$ Hz), 7.60 (1H, t, $J = 7.3$ Hz), 7.49(2H, t, $J = 7.6$ Hz), $\delta_C$ 166.4, 129.2, 130.1, 130.1, 128.5, 128.5, 133.5 (benzoyl). The HMBC correlation between $\delta_H$ 5.54 and $\delta_C$ 166.4 showed the benzoyl was attached to 5-OH. The HMBC correlation between $\delta_H$ 3.30 and $\delta_C$ 83.0 indicated that the $-$OH attach to C-3. The acetyl carbonyl signal at $\delta_C$ 172.6 had no HMBC correlation with proton signal on the skeleton, which indicated that the acetyl was attached to the tertiary OH at C-4. The relative configurations of 3-OH was determined to be $\beta$ equatorial on the basis of the NOESY correlation between H-3 and H-5, while H-5 has been ascertained to be $\alpha$ configuration proton by comparison with the literature [5–8]. But no correlation between acetyl and H-3 or H-5 was observed in the NOESY spectrum. So we ascertained that these two ester groups were determined to be $\beta$ equatorial. Thus, compound **1** was elucidated as 4-*O*-acetyl-5-*O*-benzoyl-3$\beta$-hydroxy-20-deoxyingenol (figures 1 and 2).

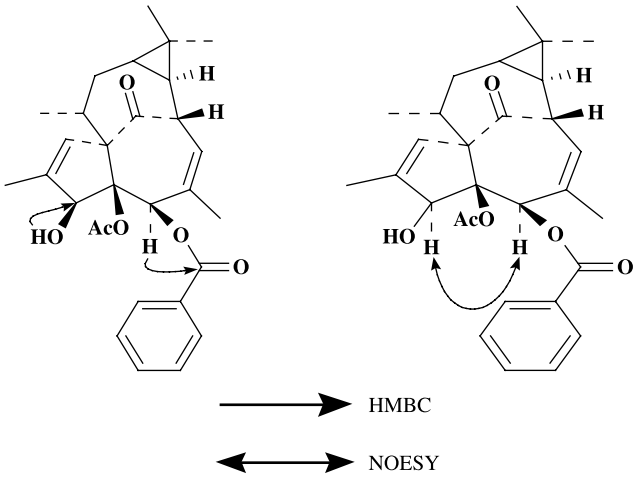


Figure 2. Key HMBC and NOESY correlations for **1**.

## 3. Experimental

### *3.1 General experimental procedures*

Melting points were determined on a Yanaco MP-S3 Micro-hot stage and are uncorrected. The UV spectrum was recorded on a Shimadzu UV-2201 spectrometer and the IR spectrum was obtained with an IFS-55 spectrum instrument. ESIMS was performed on a QSTAR LCQ mass spectrometer. HRMS was performed on QSTAR LCQ mass spectrometer. NMR spectra were taken in $CDCl_3$ on a Bruker ARX-300 spectrometer. Silica gel for chromatography was produced by the Qingdao Ocean Chemical Group Co. of China.

### *3.2 Plant material and procession*

The material of *Euphorbia Kansui* was collected from Xian city of Shanxi province, China, and identified by Professor Qishi Shun (School of Traditional Chinese Materia Medica, Shenyang Pharmaceutical University). A voucher specimen (20040917) has been deposited at Shenyang Pharmaceutical University. The cleaned *Euphorbia Kansui* (20 kg) was soaked with rice vinegar (6 kg). When the rice vinegar had been absorbed completely, the material was heated with a small fire in a pot. Then taken out to dry naturally, and crushed [4].

Table 1. $^{1}$H NMR (600 MHz, $CDCl_3$) and $^{13}$C NMR(150 MHz, $CDCl_3$) data for compound **1**.

| *Position* | *$^{1}$H NMR* | *$^{13}$C NMR* |
|---|---|---|
| 1 | 6.14(1H, s) | 132.6 |
| 2 | | 135.5 |
| 3 | 5.05(1H, s) | 83.0 |
| 4 | | 86.1 |
| 5 | 5.54(1H, s) | 77.9 |
| 6 | | 134.6 |
| 7 | 5.89 (1H, d, $J = 3.1$ Hz) | 126.2 |
| 8 | 4.27 (1H, m) | 43.5 |
| 9 | | 206.0 |
| 10 | | 72.0 |
| 11 | 2.54(1H, m) | 38.6 |
| 12 | 2.34(1H, m) | 31.4 |
| | 1.75(1H, m) | |
| 13 | 0.70(1H, d.d, $J = 15.6$, 8.6 Hz) | 22.9 |
| 14 | 0.95(1H, m) | 23.2 |
| 15 | | 24.2 |
| 16 | 1.07(3H, s) | 28.5 |
| 17 | 1.13(3H, s) | 15.7 |
| 18 | 1.01(3H, d, $J = 7.1$ Hz) | 16.9 |
| 19 | 1.78(3H, s) | 15.5 |
| 20 | 1.56(3H, s) | 21.4 |
| Acetyl | | |
| CO | | 172.6 |
| COMe | 2.06(3H, s) | 21.2 |
| Benzoyl | | |
| CO | | 166.4 |
| COPh 1 | | 129.1 |
| 2,6 | 8.14(2H, d, $J = 7.8$ Hz) | 130.1 |
| 3,5 | 7.49(2H, t, $J = 7.6$ Hz) | 128.5 |
| 4 | 7.60(1H, t, $J = 7.3$ Hz) | 133.5 |
| OH-3 | 3.30brs | |

### 3.3 Extraction and isolation

The dried processed roots of *Euphorbia Kansui* (20 kg) were extracted twice with 95% and 50% ethanol under reflux, respectively. After evaporating the solvent under reflux. The 95% EtOH extract (1342 g) and 50% EtOH extract (895 g) were obtained. The 95% EtOH extract (250 g) was subjected to silica gel column chromatography, eluted with petroleum ether–acetone with increasing polarity. The elution fractions (500 ml each) were combined into 53 portions according to TLC monitoring. Portion 17, eluted with petroleum ether–acetone (50:1), was isolated and further purified by silica gel column chromatography to give compound **1** (44 mg).

**3.3.1 4-*O*-acetyl-5-*O*-benzoyl-3$\beta$-hydroxy-20-deoxyingenol (1)**. Colourless oil, HRESIMS *m/z* 480.2503[M]$^+$ (calcd for $C_{29}H_{36}O_6$, 480.2512); $^1$H and $^{13}$C NMR data in $CDCl_3$ see table 1.



## Acknowledgements

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