Table 1. ^{13}C NMR spectral data of compounds **5** and **6** (75 MHz, Me_2CO-d_6)

C	**5**	mult.	**6**	mult.
1	129.92	CH	130.11	CH
2	26.35	CH_2	26.81	CH_2
3	34.49	CH_2	34.92	CH_2
4	144.66	C	144.98	C
5	128.59	CH	128.63	CH
6	76.92	CH	77.08	CH
7	52.95	CH	52.91	CH
8	73.21	CH	73.14	CH
9	48.72	CH_2	49.21	CH_2
10	132.55	C	133.27	C
11	136.95	C	137.01	C
12	169.72	C	170.25	C
13	124.32	CH_2	128.82	CH_2
14	16.50	Me	16.77	Me
15	60.40*	CH_2	60.61	CH_2
1′	165.15	C	174.81	C
2′	141.48	C	43.40	CH
3′	60.75*	CH_2	64.34	CH_2
4′	123.72	CH_2	13.95	Me

*Interchangeable values.

EXPERIMENTAL

Plant material was collected in May at La Rabita, Granada, Spain, and identified by Professor F. Valle, Department of Botany, University of Granada. A voucher specimen is available for inspection at the herbarium of the Faculty of Sciences of the University of Granada. The plant, once air-dried (1 kg), was cut up and extracted with refluxing Et_2O (4 l). The Et_2O extract (22 g, 2.2% of the plant material) was chromatographed on a silica gel column with $CHCl_3$–Me_2CO mixtures giving salonitenolide (**4**) (0.6 g), onopordopicrin (**5**), (8.3 g) and arctiopicrin (**6**) (1.8 g).

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A DITERPENE, DISTANOL, FROM *SIDERITIS DISTANS*

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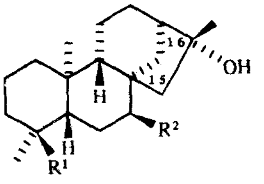
Key Word Index—*Sideritis distans*; Labiatae; *ent*-kauran-7α,16β,18-triol.

Abstract—A new diterpene, distanol, has been isolated from the petrol extract of the aerial part of *Sideritis distans* Wild. Its structure and stereochemistry has been assigned by spectroscopic methods.

From the aerial part of *Sideritis distans* Wild, a species growing in Greece, we have previously [1] isolated four tetracyclic isokaurene diterpenes: siderol (*ent*-7α-acetoxykaur-15-ene-18-ol) [2], sideridiol (*ent*-kaur-15-ene-7α,18-diol) [2], sideroxol (*ent*-15β,16β-epoxykaurane- 7α,18-diol) [3], epoxysiderol (ent-15β,16β-epoxykauran-7α-acetoxy-18-ol) [4].

Further investigation of the petrol extract of this plant led us to the isolation (trace amounts) of a new diterpenoid of the *ent*-kaurane series which was named distanol (**1**).

Distanol (**1**), mp 260–265° has molecular formula $C_{20}H_{34}O_3$ (*m/z* 322 M^+) determined by mass spec-



	R^1	R^2
1	CH_2OH	OH
2	CH_2OAc	OAc

troscopy. Its IR spectrum shows characteristic absorptions for hydroxyl groups. Acetylation of **1** gave a diacetate (**2**) whose IR spectrum shows hydroxyl absorption for a tertiary hydroxyl group at 3330 cm^{-1}. The ^{1}H NMR spectrum of **1** shows an AB quartet (δ3.24 and 3.60, J = 11.0 Hz) assigned to the equatorial CH_2OH on C-4 of a tetracyclic diterpene backbone a signal at δ3.70 ($W_{1/2}$ = 6.0 Hz) indicative of a secondary hydroxyl group (**2**), two tertiary methyl groups (δ0.75 and 1.06) and one methyl group at low field (δ1.32).

The strong deshielding of the latter is consistent with the presence of a methyl group geminal to a hydroxyl group and this is sustained by the absence of an oxymethyne proton.

These data suggest that **1** is *ent*-kauran-7α,16β,18-triol. The configuration of the tertiary hydroxyl in distanol (**1**) is α as observed in other diterpenes [5]. It is interesting to note that some diterpenoids belonging to the *ent*-kaurane group with a hydroxyl function on C-16 occur both as fungal metabolites and in higher plants [6].

EXPERIMENTAL

^{1}H NMR 360 MHz, DMSO, TMS as internal reference. MS: 75 eV. IR: nujol mull. CC: silica gel (Merck) (0.063–0.200). TLC: silica gel G. (Merck), eluent cyclohexane–EtOAc (3:7).

Isolation of the diterpene. General experimental details of extraction and separation on the diterpenes of the genus *Sideritis* have been described previously [1, 2, 7]. Air-dried aerial parts of *S. distans* (320 g) were extracted for 48 hr with petrol in a Soxhlet. From the petrol extract the major diterpenic constituents were isolated and worked-up as reported earlier. The mother liquor gave a sticky residue which was repeatedly chromatographed on a silica gel column. Elution with Et_2O–EtOAc (1:3) gave compound **1**.

Distanol (**1**). Mp 262–265° (from EtOAc); R_f 0.35 (cyclohexane–EtOAc (3:7); negative TNM test; IR ν_{max} cm^{-1} 3350 (*br*, OH); MS *m*/*z* 322 (M^+); ^{1}H NMR ($CDCl_3$ + DMSO): δ0.75 (3H, *s*, 4α-Me), 1.06 (3H, *s*, 10α-Me); 1.32 (3H, *s*, 16-Me), 3.24 and 3.60 (2H, AB*q*, J = 11.0 Hz, 4β-CH_2OH); 3.70 (1H, *t*, $W_{1/2}$ = 6.0 Hz, 7α-H).

Treatment of **1** with Ac_2O–pyridine as usual, gave **2** as an oil which could not be crystallized. IR ν_{max} cm^{-1}: 3330 (OH), 1738 and 1250 (OAc); ^{1}H NMR ($CDCl_3$): δ0.82, 1.06 and 1.32 (9H, 3*s*, 3Me); 3.72 (2H, ABq, J = 11.0 Hz, 4β-CH_2OAc); 2.05 and 2.08 (6H, 2*s*, 2OAc); 4.75 (1H, *t*, $W_{1/2}$ = 6.0 Hz, 7αH).

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