

233 (12), C₃H₅, 41 (100). CD (MeCN): $\epsilon_{316} + 0.92$; $\epsilon_{307} + 1.3$; $\epsilon_{297} + 1.0$; $\epsilon_{193} + 16.6$.

$$[\alpha]_D^{25} = \frac{589}{+142.6} + \frac{578}{+149} + \frac{546}{+171.6} + \frac{436}{+318}$$

$c = 0.82$, CHCl₃.

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DEACETOXYBRACHYCARPONE, A TRINORTRITERPENOID FROM *CLEOME BRACHYCARPA*

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Key Word Index—*Cleome brachycarpa*; Capparidaceae; trinortriterpenoid; deacetoxybrachycarpone.

Abstract—A new trinortriterpenoid dilactone, deacetoxybrachycarpone, has been isolated from *Cleome brachycarpa* and its structure determined mainly by spectroscopic studies. Cabralealactone and ursolic acid were also isolated.

INTRODUCTION

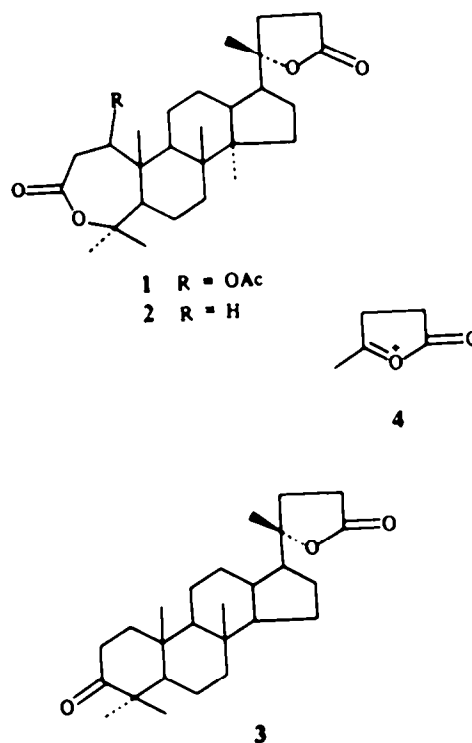
Recently we reported [1] the isolation of a new trinortriterpenoid, brachycarpone (1), from *Cleome brachycarpa* [L.] DC., a medicinal plant of Pakistan [2]. The structure of brachycarpone was confirmed through X-ray crystallographic studies. We have now isolated another closely related compound, deacetoxybrachycarpone (2), as well as the known compounds cabralealactone (3) and ursolic acid from the same plant.

RESULTS AND DISCUSSION

Deacetoxybrachycarpone (2) analysed for C₂₇H₄₂O₄. Its UV spectrum showed only an end absorption at 208 nm, indicating the absence of conjugated double bonds in the compound. Its IR spectrum showed no hydroxyl absorption but strong peaks at 1765 (γ-lactone) and 1730 cm⁻¹ (seven-membered lactone) were present as in brachycarpone. The peak at 1720 cm⁻¹ attributed to the acetoxy group in 1 was missing in 2.

The ¹H NMR (300 MHz) spectrum of 2 showed the presence of six tertiary methyl singlets (δ 0.88, 1.02, 1.07, 1.36, 1.40 and 1.48), each integrating for 3H. There was no signal near δ 2.0 due to an acetate methyl group. A multiplet centred at δ 2.59 (4H) was assigned to two methylene groups in the γ- and seven-membered lactone rings.

The mass spectrum of 2 showed a weak [M]⁺ peak at



m/z 430.3084 (calculated 430.3082). Other important peaks in the spectrum were at m/z 372 [$M - \text{acetone}$]⁺ and m/z 99 (base peak, 4). The ^{13}C NMR spectrum (Table 1) showed the absence of olefinic carbon atoms and the presence of two lactone carbonyls (δ 174.78 and 176.53). The assignments in Table 1 were made on the basis of a polarization transfer experiment (DEPT) [3].

Cabralealactone (3) was first isolated from *Cabralea polytricha* A. Juss [4]. Its isolation from *C. brachycarpa* and its ^{13}C NMR spectrum are reported here for the first time. Ursolic acid was also isolated and characterized as its acetate. Both the known triterpenoids were unambiguously identified (see Experimental).

EXPERIMENTAL

The DEPT expts were carried out at $\theta = 45^\circ$, 90° and 135° ; the quaternary carbons were determined by subtraction of these spectra from the broad-band ^{13}C NMR spectrum. The plant material was collected in Karachi and a voucher specimen has been deposited at the herbarium of the Department of Botany, University of Karachi.

Extraction and isolation. The plant material (30 kg) was cut into small pieces and finely divided in an Ultra-Turrax homogeniser under 95° . EtOH. The extract was filtered and concentrated at reduced pressure to yield a gum, which was partitioned between H_2O and hexane. The hexane layer was evaporated and 30 g of the residue chromatographed on a column of silica gel. Elution was carried out with solvent gradients of increasing polarity. The fraction eluted with hexane-EtOAc (3:2) was rechromatographed on another column using hexane- Me_2CO (9:1) as eluant to give compounds 2 (15 mg) and 3 (50 mg), and ursolic acid (20 mg), which were further purified by crystallization (MeOH).

Deacetoxybrachycarpone (2). Colourless crystals, mp $185-186^\circ$, $[\alpha]_D^{25} + 47^\circ$ (CHCl_3). (Found: C, 74.43; H, 9.74; calc. for $\text{C}_{27}\text{H}_{42}\text{O}_4$: C, 75.31; H, 9.83%). IR $\nu_{\text{max}}^{\text{KBr}}$ cm^{-1} : 1762, 1730; ^1H NMR (300 MHz, CDCl_3): δ 0.88 (s, 3H), 1.02 (s, 3H), 1.07 (s, 3H), 1.36 (s, 3H), 1.40 (s, 3H), 1.48 (s, 3H), 2.59 (m, 4H); EIMS m/z (rel. int.): 430 (2), 414 (6), 372 (20), 301 (6), 273 (16), 195 (15), 163 (20), 99 (4, 199); ^{13}C NMR: see Table 1.

Cabralealactone (3). Colourless crystals, mp $180-182^\circ$ (lit. [4] mp $182-183^\circ$). IR, MS and ^1H NMR spectra identical to those reported for cabralealactone [4]; ^{13}C NMR: see Table 1.

Ursolic acid. Colourless crystals, mp 289° (lit. [5] mp 291°). Acetate (Ac_2O $\text{C}_{30}\text{H}_{48}\text{O}_5$), mp $288-289^\circ$ (lit. [6] mp $289-290^\circ$). IR, MS and ^1H NMR identical to those reported in the literature [6, 7].

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Table 1. ^{13}C NMR spectral data of compounds 1-3 (75 MHz, CDCl_3)

C	1	2	3
1	71.27	23.50	39.94
2	42.84	34.23	34.12
3	169.55	174.78	217.28
4	89.00	85.90	47.41
5	42.84	49.10	55.47
6	21.63	23.50	19.72
7	30.94	31.05	34.71
8	41.75	39.28	40.47
9	51.16	51.16	50.04
10	40.05	39.28	36.97
11	21.04	22.69	22.02
12	24.83	23.50	26.94
13	44.07	43.49	43.42
14	50.46	50.46	50.26
15	31.46	31.46	31.27
16	24.83	25.00	26.84
17	49.23	53.33	49.43
18	15.90	14.92	15.95
19	15.48	16.01	15.29
20	89.55	89.82	89.79
21	24.88	25.40	25.55
22	28.95	29.16	25.10
23	30.94	32.41	29.20
24	176.42	176.53	176.34
28	23.65	26.85	26.84
29	24.88	31.05	21.05
30	15.40	18.28	16.27
OCOME	170.48		
OCOCH ₃	20.58		

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