

WITHANOLIDES OF *DATURA* SPP. AND HYBRIDS

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(Received 3 January 1984)

Key Word Index—*Datura* spp. and hybrids; Solanaceae; withanolides; steroidal lactones; withaferoxolide.

Abstract—A new steroidal lactone of the withanolide group has been isolated from two *Datura* species and three hybrids and characterized as (20*R*,22*R*)-5 α ,12 α -dihydroxy-1-oxo-6 α ,7 α -epoxywitha-2,24-dienolide. The distribution of six withanolides throughout section Stramonium of the genus has been investigated.

INTRODUCTION

Many highly oxygenated C₂₈ steroidal lactones (withanolides) have been isolated from various genera of the Solanaceae including *Acnistus* (*Dunalia*), *Datura*, *Jaborosa*, *Lycium*, *Nicandra* and *Physalis*. These compounds have considerable phytochemical, chemotaxonomic and medicinal interest.

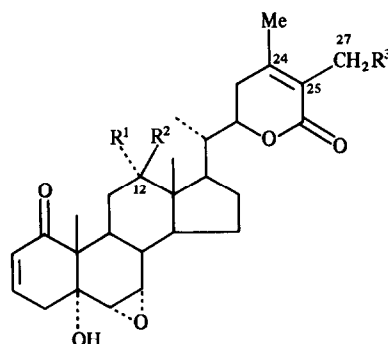
In the genus *Datura* withanolides appear to have been reported from two species only. From *D. quercifolia* leaves, daturalactones-1(1), -2(2), -3(3) and -4(4) have been characterized [1–5]. *D. stramonium*, collected in Tashkent in 1975, also contained daturalactone-1 [6] and the variety *violacea*, reported on in 1977 from the same area [7], gave this withanolide and the novel withastramonolide (5). However this second paper reported that the white-flowered form of the plant was withanolide-free.

We record here our investigations on the withanolides of some 15 species and varieties of *Datura* together with three hybrids. The structure determination of a new 12 α -hydroxywithanolide (6) is reported together with comparative ¹³C NMR and ¹H NMR spectral data for ring C oxygenated compounds of this group.

RESULTS AND DISCUSSION

Preparative TLC of the alkaloid-free, ethanol-chloroform extracts of the aerial parts of a number of *Datura* spp. and hybrids resulted in the overall separation of seven steroidal lactones (withanolides) of which six were fully characterized. The distribution of these compounds is given in Table 1. In typical isolations the yield of total withanolides (% dry wt) was: *D. ferox* leaves 0.6, stems 0.02; *D. quercifolia* stems and leaves 0.06; *D. ferox* × *D. quercifolia* F₁ hybrid leaves, 0.4; and *D. stramonium* var. *godronii* × *D. quercifolia* F₁ hybrid stems and leaves, 0.02. Sitosterol (0.001%) was characterized from the ether-soluble portion of the original plant extract of *D. ferox*. No withanolides were detected in the roots of the above plants nor in the aerial parts of the following species: *D. stramonium* (see Table 1), *D. leichhardtii*, *D. discolor*, *D. metel*, *D. meteloides*, *D. innoxia*, *D. ceratocaula*, *D. candida* cultivar, *D. aurea*, *D. suaveolens* and *D. sanguinea*.

Of the six compounds which were fully characterized one was a new 12 α -hydroxy derivative; its structure was determined as (20*R*,22*R*)-5 α ,12 α -dihydroxy-1-oxo-6 α ,7 α -



- 1 R¹ = OH; R² = R³ = H; 24,25 – epoxy
- 2 R¹ R² = O; R³ = H; 24,25 – epoxy
- 3 R¹ = R³ = H; R² = OH
- 4 R¹ = R² = R³ = H; 24,25 – epoxy
- 5 R¹ = R³ = OH; R² = H
- 6 R¹ = OH; R² = R³ = H
- 7 R¹ = R² = R³ = H
- 8 R¹ R² = O; R³ = H
- 9 R¹ = OH; R² = R³ = H; 2,3 – dihydro

epoxywitha-2,24-dienolide (6) which we have designated withaferoxolide. In *D. ferox* it constituted about 80% and 60% of the total withanolide content of the leaves and stems respectively. The other five compounds are known as constituents of various solanaceous species. Identification was achieved by comparative spectroscopy [¹H NMR (Table 2), ¹³C NMR (Table 3), MS, UV and IR].

Chemotaxonomically, the available evidence suggests that in *Datura*, withanolides are confined to the three members of Section Stramonium (Gaertner). This is in contrast to the other characteristic secondary metabolites, tropane alkaloids, which are a feature of the whole genus [10]. However in keeping with the occurrence of withanolides in other genera of the Solanaceae [11] there is a great variability in both the chemical constitution, and the proportion of individual withanolides found in a particular species. In the present instance our plant materials gave a different range of compounds to those previously

Table 1. Withanolides of aerial parts of *Datura* species*

	Lycium substance B (7)†	Withani- candrin (8)‡	Datura lactone-3 (3)	Withafer- oxolide (6)	Datura lactone-1 (1)	Withastra- monolide (5)
<i>D. ferox</i>	+	++	+	+++	+	—
<i>D. quercifolia</i>	+	+	trace§	+	?+§	—
<i>D. stramonium</i>	—	—	—	—	§ (but variable)	—
<i>D. stramonium</i> var. <i>tatula</i>	—	—	—	—	§	§
<i>D. stramonium</i> var. <i>godronii</i>	—	—	—	—	—	—
<i>D. ferox</i> × <i>D. quercifolia</i> F ₁	—	+	+	++	+	—
<i>D. stramonium</i> var. <i>godronii</i> × <i>D. ferox</i> F ₁	—	+(TLC)	+(TLC)	+(TLC)	—	+(TLC)
<i>D. stramonium</i> var. <i>godronii</i> × <i>D. quercifolia</i> F ₁	+	+	—	+	?+	+

*The compounds are arranged (left to right) in order of decreasing R_f values for the systems used.

†First characterized from *Lycium chinense* leaves [8].

‡First characterized from *Nicandra physaloides* leaves [9].

§Reported by other workers: see 'Introduction'.

Table 2. ¹H NMR Chemical shift (δ) data for diagnostic protons of ring C oxygenated withanolides*

	7	8	3	6†	1	5	9‡
H-2	5.86 <i>dd</i> (10.1, 2.2)	5.86 <i>dd</i> (10.4, 2.0)	5.86 <i>dd</i> (10.1, 2.0)	5.83 <i>dd</i> (10.1, 1.8)	5.84 <i>dd</i> (10.1, 2.2)	5.84 <i>dd</i> (10.0, 2.1)	
H-3	6.60 <i>ddd</i> (10.1, 5.0, 2.2)	6.63 <i>ddd</i> (10.4, 5.2, 2.4)	6.61 <i>ddd</i> (10.1, 4.8, 2.4)	6.60 <i>ddd</i> (10.1, 4.9, 2.4)	6.61 <i>ddd</i> (10.1, 4.9, 2.4)	6.61 <i>ddd</i> (10.0, 4.9, 2.1)	
H-6	3.05 <i>d</i> (3.7)	3.10 <i>d</i> (3.7)	3.06 <i>d</i> (3.7)	3.05 <i>d</i> (4.0)	3.04 <i>d</i> (3.7)	3.04 <i>d</i> (4.0)	3.04 <i>d</i> (3.5)
H-7	3.32 <i>br s</i>	3.41 <i>dd</i> (3.7, 1.8)	3.33 <i>d</i> (3.7)	3.31 <i>dd</i> (3.6, 1.8)	3.31 <i>dd</i> (3.7, 2.2)	3.32 <i>dd</i> (3.7, 1.8)	3.30 <i>br s</i>
H-12			3.55 <i>dd</i> (10.4, 4.6)	4.03 <i>s</i> § ($W_{1/2} = 6$)	4.00 <i>br s</i> ($W_{1/2} = 7$)	4.04 <i>br s</i> ($W_{1/2} = 6$)	4.0 <i>s</i> ($W_{1/2} = 7$)
H-22	4.39 <i>dt</i> (13.2, 3.4)	4.39 <i>dt</i> (13.1, 3.4)	4.49 <i>ddd</i> (13.1, 3.4, 4.6)	4.37 <i>dt</i> (13.4, 3.1)	4.55 <i>ddd</i> (10.0, 5.0, 2.5)	4.44 <i>dt</i> (13.4, 3.3)	4.36 <i>dt</i>
18-H ₃	0.77 <i>s</i>	1.12 <i>s</i>	0.77 <i>s</i>	0.77 <i>s</i> [0.74 <i>s</i>]¶	0.75 <i>s</i>	0.78 <i>s</i>	0.86 <i>s</i>
19-H ₃	1.19 <i>s</i>	1.25 <i>s</i>	1.18 <i>s</i>	1.175 <i>s</i> [1.22 <i>s</i>]¶	1.17 <i>s</i>	1.18 <i>s</i>	1.20 <i>s</i>
21-H ₃	1.03 <i>d</i> (6.7)	0.985 <i>d</i> (6.7)	1.18 <i>d</i> (6.7)	1.09 <i>d</i> (5.8) [1.31]¶	1.05 <i>d</i> (5.5)	1.10 <i>d</i> (6.1)	1.07 <i>d</i> (7)
27-H ₃	1.95 <i>s</i>	1.97 <i>s</i>	1.95 <i>s</i>	1.94 <i>s</i>	1.50 <i>s</i>	4.38 <i>d</i> ** (5.6)	1.95 <i>s</i>
28-H ₃	1.89 <i>s</i>	1.89 <i>s</i>	1.88 <i>s</i>	1.88 <i>s</i>	1.57 <i>s</i>	2.05 <i>s</i>	1.95 <i>s</i>

Coupling constants in parentheses.

*250 MHz.

†Spectrum in DMSO-*d*₆ showed 0.5 mol solvate (CHCl₃).

‡60 MHz.

§Spectrum in CDCl₃-CCl₃CONCO showed shift of H-12 signal from 4.03 → 5.12 and appearance of new signals at δ 8.88 and 8.50 (2 × NH).

||2- α -Acetate, H-12, δ 5.15; Ac, δ 2.13.

¶[] spectrum in pyridine-*d*₅.

**27-H₂.

recorded [1–7] for *D. quercifolia* and *D. stramonium*. Also Eastwood *et al.* [12] have shown that hybrids between chemical races of *Withania somnifera* produce withanolides not found in either parent. Analogously, as reported above, F₁ hybrids of *D. stramonium* var. *godronii* with *D. ferox* and *D. quercifolia* respectively produce withastra-

monolide, not isolated from either of our parents but which had been characterized from *D. stramonium* by other workers [7].

The empirical formula of withaferoxolide, C₂₈H₃₈O₆, was established by elemental analysis (of the solvate) and by mass spectrometry, although its molecular ion [M]⁺

Table 3. ^{13}C NMR chemical shift data for ring C oxygenated withanolides*

Compound C-12 subst.	7 H	8 =O	3 β -OH	6 α -OH	5 α -OH
C-1	203.0	202.1	202.8	203.1	203.1
C-2	129.1	128.8	129.0	128.9	128.9
C-3	139.5	139.8	139.9	139.8	139.9
C-4	36.8	36.7	36.7	36.8	36.8
C-5	73.3	73.2	73.2	73.3	73.2
C-6	56.2	56.2	56.3	56.2	56.1
C-7	57.3	56.8	57.0	57.0	57.0
C-8	35.8	35.7	38.6	36.1	36.1
C-9	35.6	37.7	35.1	28.7	28.7
C-10	51.5	51.5	50.9	50.6	50.5
C-11	21.9	38.4	31.3	30.1	30.2
C-12	39.9	212.0	77.6	72.5	72.4
C-13	43.6	57.7	48.5	47.1	47.0
C-14	52.0	52.9	53.4	43.7	43.7
C-15	23.6	23.6	22.8	23.1	23.1
C-16	27.3	27.7	26.9	26.6	26.6
C-17	51.0	42.8	49.6	42.9	42.8
C-18	12.1†	11.5†	7.6	12.0†	11.9†
C-19	14.7	14.7	14.6	14.7	14.6
C-20	39.2	39.5	37.7	39.1	39.0
C-21	13.3†	13.1†	14.9	12.4†	12.4†
C-22	78.3	76.3	79.0	79.0	78.7
C-23	29.7	29.1	32.7	29.7	29.8
C-24	149.0	149.3	149.1	149.2	153.0
C-25	122.1	121.8	122.0	122.0	125.6
C-26	167.0	166.8	166.9	167.0	167.0
C-27	12.5	12.5	12.5	12.5	57.0
C-28	20.4	20.5	20.5	20.5	19.9

* Recorded at 62.9 MHz for solutions in CDCl_3 with TMS as internal standard. Multiplicities were verified by the off-resonance decoupled spectra.

† Values in any vertical column may be interchanged.

could not be readily observed without overloading the spectrum of the remaining ions. The UV spectrum (λ_{max} 222 nm, $\log \epsilon$ 4.21) indicated an $\alpha\beta$ -unsaturated ketone and an $\alpha\beta$ -unsaturated lactone. The former was readily confirmed as a steroidal 2-en-1-one system by ^1H NMR signals at δ 5.83 and 6.60 for the alkene protons and by an IR absorption at ν_{max} 1695 cm^{-1} . These alkene signals were removed by reduction (1 mol. equiv. of hydrogen taken up) and the carbonyl ν_{max} was shifted to 1715 cm^{-1} .

The mass spectrum of **6** showed m/z 125 as the base peak and is attributable to fission of ring A and to cleavage at the C-20–C-22 bond; in 2,3-dihydro-**6** the intensity of this ion was much reduced. The fragment ion at m/z 299 arises by fission of the C-17–C-20 bond and concomitant loss of the elements of water and appears to be a series characteristic; all the 12α - and 12β -hydroxywithanolides examined exhibited similar fission. The presence of two hydroxyl functions was revealed by the ^1H NMR spectrum of the bis-trichloroacetyl carbamate (formed *in situ* from the corresponding isocyanate), two characteristic N–H signals appeared at δ 8.88 and 8.50. The presence of one tertiary and one secondary hydroxyl function was indicated since only one proton signal (δ 4.0, $W_{1/2} = 6$ Hz) was deshielded (to δ 5.12). This was confirmed by the

^{13}C NMR spectrum which showed signals at 73.3 and 72.5, a singlet and a doublet respectively, in the off resonance decoupled spectrum. The ^1H NMR spectrum of **6** showed a pyridine induced shift ($\delta\text{CDCl}_3 - \delta$ pyridine) of -0.03 for the 19-Me thus confirming a 5α -hydroxy group [13]. Acetate formation occurred only at elevated temperature and oxidation by Jones' reagent at 0° afforded withanolide **8**.

The ^1H NMR spectrum of compound **8** showed deshielding of the 18-Me and 21-Me signals which is consistent only with a 12-oxo substituent. The oxidation product **8**, which co-occurs abundantly with **6**, appeared to be identical with withanicandrin [9]. The remaining structural features were assigned by comparison with spectroscopic data of related compounds.

The isolation of four ring-C oxygenated withanolides together with the unsubstituted compound allowed a study of the substituent effects in ^{13}C NMR. Assignments were made from off-resonance decoupled spectra, consideration of β and γ effects and by comparison of spectra. Ring A and lactone chemical shifts were in excellent agreement with published data [14]. The 12-oxo compound **2** showed the expected deshielding at C-11 and C-13. The axial 12α -hydroxy group in compounds **1**, **5** and **6** is gauche to the axial hydrogens at C-7, C-14 and C-17, the γ -effect produced is shielding by 6.9–8.3 ppm. The equatorial 12β -hydroxy group in **3** produced smaller shielding effects at C-9 and C-17 and a characteristic high field signal for C-18 (δ 7.6) [15].

EXPERIMENTAL

Mps were measured in a Buchi 510 apparatus and are uncorr. Optical rotations were determined in CHCl_3 on a Bendix automatic polarimeter 143C. EIMS were run at PCMU (Harwell) and CIMS at the School of Pharmacy, London. Prep TLC was carried out on silica gel (Merck) GF₂₅₄ and Al_2O_3 (Merck) GF₂₅₄.

Plant material. Produced from seeds of authenticated stock, grown in open garden or greenhouse in Nottingham and oven-dried at 60° .

Isolation of withanolides. A mixture of the relevant dried *Datura* sp. (1.5 kg), $\text{Ca}(\text{OH})_2$ (300 g) and H_2O (600 ml) was exhaustively extracted with Et_2O . The plant residues were exhaustively percolated with cold CHCl_3 – EtOH (9:1) and the extract was concd to small vol.; alkaloids were removed with 0.25 M H_2SO_4 . The residue (22 g) was chromatographed on Al_2O_3 (Merck, 70–230 mesh, 160 g), eluted with petrol (bp 60–80°) and petrol– Et_2O (1:1) to remove plant pigments; Et_2O first removed sitosterol (17 mg, identical with an authentic sample) and then Et_2O , CHCl_3 – Et_2O (up to 3:2) and CHCl_3 – Me_2CO eluted a withanolide mixture (9 g). Prep. TLC with silica gel (CHCl_3 – Me_2CO , 4:1) gave in order of development compounds **7** (fast running), **8**, **3** and **6**. The lower sixth of the slowest running band was resolved by multiple development on silica gel (CHCl_3 – Me_2CO – EtOH , 40:5:1) and gave further **6** and **1**. Compounds **7**, **8**, **3** and **6** were visualised at 365 nm and compound **1** was revealed (purple-brown colour) with Dragendorff's reagent (according to Munier)-iodine.

Compound 7 [(20R,22R)-5 α -Hydroxy-1-oxo-6 α ,7 α -epoxy-witha-2,24-dienolide (*Lycium* substance B [8])]. Needles, mp 283° (EtOAc) (lit. [8] 260 – 265°); UV λ_{max} nm: 224 ($\log \epsilon$ 4.19) (lit. [8] 225 nm, $\log \epsilon$ 4.18); IR ν_{max} cm^{-1} : 3450 (OH), 1705 (unsatd δ -lactone), 1690 (α,β -unsatd 6-ring C=O), 1655 (C=C); EIMS (probe) 70 eV, m/z (rel. int.): 436 [$\text{M} - \text{H}_2\text{O}$] $^+$ (10), 153 [C-17 side-chain] $^+$ (7), 125 [lactone, ring A] (100).

(Found: C, 74.0; H, 8.1. Calc. for $C_{28}H_{38}O_5$: C, 74.0; H, 8.4%.)

Compound 8 [(20R,22R)-5 α -hydroxy-1,12-dioxo-6 α ,7 α -epoxywitha-2,24-dienolide (withanicandrin [9])]. Needles, mp 303° from $CHCl_3$, or prisms, mp 267° (lit. [9] mp 267–269) from EtOAc; $[\alpha]_D^{25}$ 113.1° (c 0.24, $CHCl_3$) (lit. [9] 105°, c 0.25); IR ν_{max} cm^{-1} : 1708, 1690, 1655. UV λ_{max} nm: 222 (log ϵ 4.26) ($2 \times C=C-C=O$) (lit. [9] 222 nm, log ϵ 4.26); EIMS (probe) 70 eV, m/z (rel.int.): 468.2490 $[M]^+$ (2), $C_{28}H_{36}O_6$ requires m/z 468.2512, 315 $[M - \text{side chain}]^+$ (3), 275 $[C_{16}H_{19}O_4]^+$ (11); 125 (100). (Found: C, 71.8; H, 7.9; $C_{28}H_{36}O_6$ requires C, 71.8; H, 7.7%.)

Compound 3 [(20R,22R)-5 α ,12 β -dihydroxy-1-oxo-6 α ,7 α -epoxywitha-2,24-dienolide(daturalactone-3 [3])]. Prisms, mp 278° ($CHCl_3$ -EtOH) (lit. [3] 285–286°); UV λ_{max} nm: 222 (log ϵ 4.22) (lit. [3] 223 nm, log ϵ 4.23); IR ν_{max} cm^{-1} : 3620, 3460, 1708, 1690, 1655; CIMS (NH_3 , probe) m/z 453 $[(M+1) - H_2O]^+$; EIMS (probe) 70 eV m/z : 452.2597 $[M - H_2O]^+$ (7), $C_{28}H_{36}O_5$ requires 452.2563, 317 $[M - \text{side chain}]^+$ (5), 299 $[M - \text{side chain} - H_2O]^+$ (8), 125 $[\text{lactone} + \text{ring A}]^+$ (100). (Found: C, 71.3; H, 8.2. Calc. for $C_{28}H_{38}O_6$: C, 71.5; H, 8.1%.)

Compound 6 [(20R,22R)-5 α ,12 α -dihydroxy-1-oxo-6 α ,7 α -epoxywitha-2,24-dienolide]. Large prisms, mp 278°, from $CHCl_3$; $[\alpha]_D^{25}$ 116.4° (c 0.4, $CHCl_3$); IR ν_{max} cm^{-1} : 3560, 3450 (OH), 1710 (α,β -unsatd- δ -lactone), 1690 (α,β -unsatd 6 ring $C=O$), 1685; UV λ_{max} nm: 222 (log ϵ , 4.21) ($2 \times C=C-C=O$); EIMS (probe) 70 eV, m/z (rel. int.): 470.2678 $[M]^+$, $C_{28}H_{38}O_6$ requires m/z 470.2668 (obtained only by overloading all other peaks), 452 $[M - H_2O]^+$ (29), 434 $[M - 2H_2O]^+$ (17), 299 $[M - \text{side chain} - H_2O]^+$ (38), 281 $[M - \text{side chain} - 2H_2O]^+$ (33), 153 $[C-17 \text{ side chain}]^+$ (31), 125 $[\text{lactone and ring A}]^+$ (100). (Found: C, 64.8; H, 7.5; $C_{28}H_{38}O_6$, 0.5 $CHCl_3$ requires C, 64.55; H, 7.3%.)

Acetylation of 6. Compound 6 (67 mg) was treated with Ac_2O (1 ml) and pyridine (0.1 ml) at 80° for 2 hr and from the neutralized mixture the acetate was recovered ($CHCl_3$) and was obtained after prep. TLC (Kieselgel GF₂₅₄; $CHCl_3$ - Me_2CO , 4:1) as a gum, which, after several months, turned into an amorphous solid, mp 260°; IR ν_{max} cm^{-1} : 3560 (OH), 1740 (OCOMe), 1720 (unsatd δ -lactone), 1695 (unsatd 6-membered ring $C=O$); UV λ_{max} 225 nm. EIMS (probe) 70 eV m/z (rel.int.): 452 $[M - HOAc]^+$ (100). (Found C, 69.8; H, 8.2. $C_{30}H_{40}O_7$ requires C, 70.3; H, 7.9%.)

Oxidation of 6. Compound 6, in Me_2CO at 0°, was treated with Jones' reagent for 1 hr. The product was identical in all respects with compound 8.

Hydrogenation of 6. Compound 6 (33 mg) was hydrogenated (uptake: 1 mol equiv) over 10% Pd-C in absolute EtOH. Prep. TLC (alumina with $CHCl_3$ - Me_2CO -EtOH, 40:10:1) and recovery of the faster running band gave (20R,22R)-5 α -12 α -dihydroxy-1-oxo-6 α ,7 α -epoxywitha-24-enolide (9) as needles mp 258–260° ($CHCl_3$ -EtOH). UV λ_{max} nm: 213 (log ϵ 4.09) ($C=C-C=O$); IR ν_{max} cm^{-1} : 3710, 3530 (OH), 1715 (α,β -unsatd- δ -lactone and 6-membered ring $C=O$), 1660 ($C=C$); EIMS (probe) 70 eV, m/z (rel. int.): 454 $[M - H_2O]^+$ (3), 301 $[M - \text{side chain} - H_2O]^+$ (6), 283 $[M - \text{side chain} - 2H_2O]^+$ (5). (Found: C, 70.9; H, 8.8. $C_{28}H_{40}O_6$ requires C, 71.2; H, 8.5%.)

Compound 1 [(20R,22R)-5 α ,12 α -dihydroxy-1-oxo-6 α ,7 α -24,25-diepoxywitha-2-enolide (daturalactone-1 [4])]. Prisms, mp

264–267° from EtOH- $CHCl_3$ (lit. [4] 260–261°); UV λ_{max} nm: 224.5 (log ϵ 4.0) ($C=C-C=O$); IR ν_{max} cm^{-1} : 3590–3500 (OH), 1730 (satd δ -lactone), 1690 (unsatd 6-ring $C=O$); CIMS (CH_4 , probe) m/z (rel. int.): 486 $[M]^+$ (4), 468 $[M - H_2O]^+$ (30), 299 $[M - \text{side chain} - H_2O]^+$ (96), 141 $[\text{epoxylactone}]^+$ (38), 125 $[\text{ring A}]^+$ (52). (Found: C, 71.3; H, 8.1; $C_{28}H_{38}O_6$ requires C, 71.5; H, 8.1%.)

Compound 5 [(20R,22R)-5 α ,12 α ,27-trihydroxy-1-oxo-6 α -7 α -epoxywitha-2,24-dienolide (withastramonolide [7])]. Crystals, mp 269–270° (MeOH) (lit. [7] mp 264–266°) or mp 288–290° ($CHCl_3$). UV λ_{max} nm 219 (log ϵ 4.28) ($2 \times C=C-C=O$), lit. [7] λ_{max} nm 225 (log ϵ 4.17) IR: ν_{max} cm^{-1} 3520, 3500 (O-H), 1710 (α,β -unsatd- δ -lactone), 1695 (α,β -unsatd 6-ring ketone), 1685; EIMS (probe) 70 eV, m/z (rel. int.): 468.2496 $[M - H_2O]^+$ (25), $C_{28}H_{36}O_6$ requires 468.2512, 450 $[M - 2H_2O]^+$ (19), 345 $[M - \text{lactone}]^+$ (31), 327 $[M - \text{lactone} - H_2O]^+$ (55), 309 $[M - \text{lactone} - 2H_2O]^+$ (20), 299 $[M - \text{side chain} - H_2O]^+$ (30), 141 $[\text{lactone}]^+$ (55), 125 $[\text{ring A}]^+$ (100), 123 $[\text{lactone} - H_2O]^+$ (70). (Found: C, 69.0; H, 8.1. Calc. for $C_{28}H_{38}O_7$: C, 69.1; H, 7.9%.)

Acknowledgement—One of us (MKM) acknowledges the award of a scholarship by the Government of Ghana.

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