Isolation of corosin \([1]\) as its acetate \([2]\) has been described earlier on. During present isolation sequence as indicated in the flow sheet (Fig. I) the more soluble acetates as obtained in methanol solution have been investigated. The total extractives from fresh jute roots were separated into two broad fractions - the less methanol soluble solid on acetylation gave corosin acetate, m.p. 311-313 °C dec. (gas bubbling), and the more methanol soluble fraction on acetylation and separation by silica-gel column chromatography gave crystalline acetates, i) m.p. 268–269 °C (feathery needles), \(\delta_{[D]}^\mathrm{C} + 70^\circ\) \((c = 0.02\% \text{ chloroform})\) and ii) m.p. 220–222 °C (Rods, changing to fine needles above 180 °C), \(\delta_{[D]}^\mathrm{C} + 24^\circ\) \((c = 0.101\% \text{ methanol})\). The polar fractions from the column gave after esterification with diazomethane followed by re-chromatography on a silica-gel column, a micro-crystalline product (from \(n\)-hexane) now designated as \(\text{o xo-corosin acetate methyl ester, C}_{36}\text{H}_{54}O_{10}\), m.p. 160 °C, \(\delta_{[D]}^\mathrm{C} - 15^\circ\) \((c = 0.435\% \text{ chloroform})\). The 24-carboxyl in corosin-anhydro-lactone-acetate has been reduced to the methyl group and the resultant lactone on acetylation indicated to be identical with tormentic acid lactone acetate.

Isolation of corosolic acid, ursolic acid as its acetate, corosolic acid as its acetate and indicated to be urs-2\(\alpha,2\beta\)-diol-12-en-28-oic-acid and o xo-corosin isolated as its acetate-methyl-ester, \(\text{C}_{36}\text{H}_{54}O_{10}\), m.p. 160 °C, \(\delta_{[D]}^\mathrm{C} - 15^\circ\) \((c = 0.435\% \text{ methanol})\). The 24-carboxyl in corosin-anhydro-lactone-acetate has been reduced to the methyl group and the resultant lactone on acetylation indicated to be identical with tormentic acid lactone acetate.

In the infrared spectrum an acid carboxyl and acetate carbonyl showed up at 1692 and 1730 cm\(^{-1}\) respectively. A strong end absorption in the ultraviolet spectrum also indicated the presence of a double bond in the molecule. From the mass spectral fragmentation pattern the retro-Diels-Alder sequence \([3, 4]\) in ring e of a \(\Delta^{12}\)-ursene skeleton was easily detected \((I; R_{1} = \text{H}, R_{2} = \text{OAc})\).

The fragment a as expected showed up as a strong peak at \(m/e\) 248 and after decarboxylation of 28-carboxyl b showed up at \(m/e\) 203. These facts indicate the acid acetate to be ursoic acid acetate, variously reported \([5]\) as having m.p. 264° \([6]\), 279–280° \([7]\), 289–290° \([8]\) and m.p. 286°, \(\delta_{[D]}^\mathrm{C} + 73^\circ\) \([9]\). This was confirmed by its NMR spectra which showed bands at 7.85 \((\text{O-CO-CH}_{3})\), 5.83–5.78 \((1\text{H, 3-H})\), 4.77, 4.74 and 4.70 \(\tau\) \((\text{C-12-H})\).

The acetate m.p. 220–222 °C now designated as corosilic acid acetate gave a mass molecular ion at 556 and analysed for \(\text{C}_{36}\text{H}_{56}O_{4}\). In the infrared spectrum it showed an ester acetate at 1745 and the ester carbonyls showed up at 1728 and 1740 cm\(^{-1}\).

The acetate m.p. 268–269 °C gave a mass molecular ion at 498 and analysed for \(\text{C}_{34}\text{H}_{52}O_{6}\). In the

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** Present address: BCSIR Laboratories, P. O. Bayezid Bostami, Chittagong, Bangladesh.

*** Reprint requests to Prof. Dr. G. Habermehl.

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Ethanol extractive from fresh undried jute roots concentrated to a mainly aqueous liquor in vacuo

Precipitate charcoaled hot in methanol and cooled soluble phase mainly sugars, amino-acids and inorganic salts

Dissolved in methanol

First and second crops \((y, z)\) acetylated and taken in benzene

Mother liquor evaporated \((A)\) → chromatographed on silica-gel-column

1. Ethyl hexadecanoate
2. \(\beta\)-Sitosterol
3. Polar fractions

Precipitate Diluted with ether (1:1)

Mother liquor evaporated \((B)\), acetylated and taken in benzene

Mother liquor evaporated \((C, E)\), crystallised from methanol

Mother liquor evaporated \((F)\) and chromatographed on silica-gel column

10\% methanol in ethyl-acetate elute (M)

Esterified with diazomethane and chromatographed on silica-gel oxo-corosin-acetate-methylester

Fig. 1. Flow sheet for isolation from jute roots.

In the mass spectrum at 248 (ion a) and 203 (ion b). The NMR spectrum showed the presence of two acetate methyls at 8.05 and 7.98 \(\tau\) which is expected of a 2\(a\),3\(\beta\)-diacetoxy triterpenoid. In the literature urs-2\(a\),3\(\beta\)-diol-12-ene-28-oic acid has been reported \([3, 10]\) as its methyl ester acetate and the diol acid, m.p. 139–143 \(^\circ\)C, \(a_{\text{D}}\): +27.2\(\%\) (\(c = 1.1\)),

and m.p. 243–244 \(^\circ\)C, \(a_{\text{D}}\): +42.1 \(\%\) (\(c = 1.02\), pyridine) respectively. The acid obtained from corosolic acid acetate by hydrolysis to give the diol acid, m.p. 240–242 \(^\circ\)C, \(a_{\text{D}}\): +42.0\% (\(c = 0.11\), methanol) had a \(r_{\text{max}}\) at 1692 cm\(^{-1}\) (COOH) and in the mass spectra gave a molecular ion at 472, the ion a and b showed up at \(m/e\) 248 and 203 as expected for an urs-12-en acid (I, \(R_1 = R_2 = OH\)).

The diacetate acid on esterification with diazomethane gave a methylester acetate, m.p. 162 to 164 \(^\circ\)C (puffing up above 100 \(^\circ\)C, unsharp), \(a_{\text{D}}\): +2\% (\(c = 0.18\%), methanol) and had a \(r_{\text{max}}\) at 1740 cm\(^{-1}\). This comparison indicates that corosolic acid is the same as urs-2\(a\),3\(\beta\)-diol-12-en-28-oic acid.

So far a number of C-24, 28 lactones in the ursane series have been reported in the literature:

2a: Corosin anhydro lactone
\((R_1 = R_2 = OH; R_3 = CO_2H)\).

2b: Tormentic acid lactone
\((R_1 = R_2 = OH; R_3 = CH_3)\).

2c: Vanguerolactone
\((R_1 = H; R_2 = OH; R_3 = CH_3)\).

2d: Urs-13(18)-en-2\(a\),3\(\beta\)-diacetoxy-28,20-lactone
\((R_1 = R_2 = OAc; R_3 = CH_3)\).

The C-24 carboxyl group has been reported to be reduced in corosine lactone (2a) to the methyl group with a subsequent opening of the lactone to give urs-13:19(20)-dien-2\(a\),3\(\beta\)-dihydroxy-28-oic acid [2]. The reduction of the C-24 carboxyl without opening of the lactone ring is expected to give tormentic acid lactone (2b). It has now been possible to isolate...
Table I. NMR spectra of ursane derivatives.

<table>
<thead>
<tr>
<th>Substance</th>
<th>H</th>
<th>H-2*</th>
<th>H-3</th>
<th>C-2</th>
<th>C-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Corosin acetate</td>
<td>4.70*</td>
<td>4.30</td>
<td>5.19*</td>
<td>8.05</td>
<td>7.94</td>
</tr>
<tr>
<td>2. Corosin ester acetate</td>
<td>4.68*</td>
<td>4.34</td>
<td>5.17*</td>
<td>8.05</td>
<td>7.96</td>
</tr>
<tr>
<td>3. Tormentosolic acetate ester</td>
<td>4.54b</td>
<td></td>
<td>5.80a</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4. Corosolic acid acetate</td>
<td>4.74*</td>
<td>4.96</td>
<td>5.29b</td>
<td>8.05</td>
<td>7.98</td>
</tr>
<tr>
<td>5. Ursolic acid acetate</td>
<td>4.74b</td>
<td></td>
<td>5.80a</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6. Oxo-corosin ester acetate</td>
<td>4.58*</td>
<td>4.30</td>
<td>5.17a</td>
<td>8.04</td>
<td>7.94</td>
</tr>
<tr>
<td>7. Corosin anhydro lactone acetate</td>
<td></td>
<td>4.12</td>
<td>5.17a</td>
<td>8.03</td>
<td>7.93</td>
</tr>
<tr>
<td>8. Urs-13(18)-en-2α,3β-diacetoxy-20,28-lactone</td>
<td></td>
<td>4.85</td>
<td>5.29a</td>
<td>8.05</td>
<td>7.98</td>
</tr>
</tbody>
</table>

Spectra were taken on 100 Mcs Varian XL-100 instrument in CDC13 solution using TMS as internal standard.

The peak positions were read from the chart and are expressed in r. The multiplet and unresolved humps, doublets and triplets as seen in the generally small peaks have been indicated.

* Unresolved humps or multiplet; a doublet; b triplet.

Table II. Optical rotations*.

<table>
<thead>
<tr>
<th>Substances</th>
<th>a256</th>
<th>a436</th>
<th>a546</th>
<th>a578</th>
<th>a589</th>
<th>Conc. in g/100 ml</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Ursolic acid acetate (I, R1 = H; R2 = OAc)</td>
<td>+216</td>
<td>+142</td>
<td>+83</td>
<td>+71</td>
<td>+70</td>
<td>0.024</td>
</tr>
<tr>
<td>2. Corosolic acid acetate (I, R1 = R2 = OAc)</td>
<td>+63</td>
<td>+40</td>
<td>+25</td>
<td>+24</td>
<td>+24</td>
<td>0.101</td>
</tr>
<tr>
<td>3. Corosolic acid (I, R1 = R2 = OH)</td>
<td>+135</td>
<td>+70</td>
<td>+46</td>
<td>+42</td>
<td>+42</td>
<td>0.11</td>
</tr>
<tr>
<td>4. Corosalic acid methyl ester (I, R1 = R2 = OAc; 28-OCH3)</td>
<td>+98</td>
<td>+41</td>
<td>+23</td>
<td>+21</td>
<td>+21</td>
<td>0.18</td>
</tr>
<tr>
<td>5. Oxo-corosin acetate methyl ester</td>
<td>—58</td>
<td>—34</td>
<td>—19</td>
<td>—16</td>
<td>—15</td>
<td>0.435</td>
</tr>
<tr>
<td>6. corosin anhydro lactone acetate (II, R1 = R2 = OAc; R3 = COOH)</td>
<td>—334</td>
<td>—185</td>
<td>—99</td>
<td>—84</td>
<td>—77</td>
<td>0.06</td>
</tr>
<tr>
<td>7. Urs-13(18)-en-2α,3β-diol-28-oic-20,28-lactone (II, R1 = R2 = OH; R3 = CH3)</td>
<td>—464</td>
<td>—253</td>
<td>—136</td>
<td>—116</td>
<td>—108</td>
<td>0.073</td>
</tr>
<tr>
<td>8. Urs-13(18)-en-2α,3β-diacetoxy-28-oic-20,28-lactone (II d)</td>
<td>—395</td>
<td>—224</td>
<td>—121</td>
<td>—105</td>
<td>—95</td>
<td>0.089</td>
</tr>
</tbody>
</table>

* Determined at room temperature on Perkin Elmer 141 Polarimeter.
tion with a drop of conc. hydrochloric acid in methanol solution gave a product, m. p. 300–302 °C decomp., having the lactone structure, \( \alpha \beta \delta \delta \) = 108° (\( e=0.073\% \) methanol), \( \lambda_{\text{max}} \) 205, 230 nm (\( e=8,400; 6,000 \)).

In the earlier paper a tentative assignment for the NMR bands were reported. It has now been possible to have a direct comparison with tormentosolic acetate methylester [12]. The NMR bands assigned to the \( 2\beta,3\alpha \)-protons and acetate methyls appear to be better represented as tabulated in Table I. The \( 2\alpha \)-acetate methyl and the \( 3\alpha \)-H are now indicated to have the higher \( r \) value as compared to the \( 3\beta \)-acetate methyl and the \( 2\beta \)-H in the corosin compounds.

**Experimental**

Unless otherwise stated all infrared spectra were determined in KBr, ultraviolet spectra were determined in methanol solution, optical rotations were determined in methanol solution, NMR was determined in CDC\(_6\) solution using Varian XL-100 spectrophotometer at 100 MHz. Melting points were determined on electric blocks and are uncorrected, the higher melting solids were somewhat heat sensitive and were determined at a moderate heating rate (\( \geq 5 \) °C/min). In the text symbols s, m, w, v and i denote strong, medium, weak, very and inflexion respectively. Analytical samples were dried at 90 °C (unless otherwise stated) in evacuated drying pistols over P2O5, KOH pellets and wax for 24 hrs. Rf values are reported for TLC on silicagel sheets (Kieselgel 60 F\(_{254}\)) using ethyl acetate: chloroform (40:60 for development).

1. **Extraction of Jute roots**

The extraction was carried out as described earlier on [1, 2]. The concentrated mainly aqueous extract gave a precipitate which was redissolved in methanol and charcoaled. The precipitate was collected with further solids obtained by ether dilution of the methanol mother liquor (1:1) (mother liquor, A). The combined solids were dissolved in methanol and allowed to crystallise. After collection of the first (7 g, Y) and second (6 g, Z) crops, the mother liquor was evaporated (B). The mother liquor A on chromatography on silicagel gave with benzene elution firstly a colourless oil, \( n^\circ = 1.4501 \), which distilled at 140 °C/0.15 mm to give ethyl hexadecanoate reported earlier on, followed by a fraction which on crystallisation from methanol gave \( \beta \)-sitosterol, m. p. 135–138 °C.

The solids Y and Z (13 g) were acetylated and the product taken in benzene. The crystalline first crop from benzene (5.5 g, m. p. 269–271 °C, colourless rods, mother liquor C) on recrystallisation from ethanol gave corosin acetate, m. p. 311–313 °C decomp. (earlier reported m. p. at slow heating).

\( C_{39}H_{60}O_9 \) (602.7) dried at 140 °C in vacuo

Calcd C 67.75 H 8.36

Found C 68.14 H 8.48

The benzene mother liquor C was evaporated and crystallised from methanol (mother liquor F), to give plates which on recrystallisations from the same solvent gave \( \beta \)-sitosteryl acetate, m. p. 135–137 °C.

\( C_{36}H_{32}O_2 \cdot 0.5 \text{CH}_3\text{OH} \) (454.7)

Calcd C 80.01 H 11.53

Found C 79.75 H 11.44

Evaporated mother liquor B (15.7 g) was acetylated and taken in benzene. A small amount of precipitate was collected on cooling (1.1 g, mother liquor E).

2. **Ursolic acid acetate and corosolic acid acetate**

(\( I, R_1 = H; R_2 = O\text{Ac} \) and \( I, R_1 = R_2 = O\text{Ac} \))

The methanol mother liquor F obtained after separation of the bulk of sitosteryl acetate was evaporated (5.25 g) and chromatographed on a column of silicagel 60 (150 g). 100 ml fractions were collected. The benzene (10 fractions), chloroform in benzene (20%, 40%, 60%) fractions (17), chloroform (4 fractions), and 40% ethyl acetate in chloroform (3 fractions) gave a small amount of elutes (\( \approx 0.3 \) g). The 35th, 36th and 37th gave an impure solid (2.2 g). The 60% ethyl acetate in chloroform, and ethyl acetate fractions (18 total) were not investigated. Finally, the 10% methanol in ethyl acetate gave amongst other materials a polar acetate in the 50th and 60th fractions (0.5 g; M) which with vanillin-phosphoric acid spray development (140 °C) on the TLC showed turquoise blue spots as compared to blue-violet spots of the other fractions.

The 35th to 37th fractions were combined and rechromatographed on a silicagel column (50 g) and eluted with chloroform. The first chloroform elutes (400 ml) and 10% ethyl acetate in chloroform elutes (100 ml) gave no eluents. The next 20 ml gave a product having a \( RF \) value = 0.59 and on crystallisation from methanol gave \( \text{ursolic acid acetate} \), m. p. 268–269 °C, \( \alpha(D) = +70° \) (\( c=0.02\% \), chloroform).

\( C_{36}H_{50}O_4 \) (498.7)

Calcd C 77.06 H 10.29

Found C 76.65 H 9.84

It gave a mass molecular ion at \( m/e = 498 \), and in the NMR signals at 7.85 (OCOC\(_2\)H\(_5\)), 5.83, 5.78 (3\( \alpha \)-H), 4.77, 4.74, 4.70 \( \tau \) (C-12 proton). It had \( \nu_{\text{max}} \) 1730 (acetate) and 1697 (COOH) cm\(^{-1}\), and showed end absorption \( \lambda_{\text{max}} \) 205 nm in the ultraviolet spectrum.

The next 20 ml elute gave a mixture of two compounds, and the following 60 ml elute gave a product having a \( RF \) value = 0.56 which crystallised from benzene/ligroin (80–100 °C) to give colourless
rods, m.p. 220 °C (changing to fine needles 185 to 190 °C). This on recrystallisation from the same solvent gave corosolic acid acetate, m.p. 220–222 °C (needles above 180 °C), \([\alpha]_D^2 + 24^\circ (c = 0.1\%)\).

\[
C_{24}H_{22}O_8 \text{(556.8)}
\]
Caled \(C 73.34\) H 9.42, Found \(C 73.71\) H 9.31.

It gave a mass molecular ion at \(m/e = 556\) and had a \(\lambda_{max} = 1735 (OCOCH_3), 1697 (COOH) \text{ cm}^{-1}\) and a \(\lambda_{max} = 204 \text{ nm} (e 6,600)\). In NMR it showed signals at \(\tau = 9.28, 9.12, 8.98, 8.91, 8.79, 8.05 (2\alpha-OAc), 798 (3\beta-OAc), 5.34, 5.24 (2\beta-H), 4.96\) (multiplet, D-12 proton), 476 (multiplet, 3\alpha-H).

3. Oxo-corosin acetate methyl ester

The polar acetate fraction (M; 0.5 g) was esterified with diazomethane in ether and rechromatographed on a silicagel (18 g SiO\(_2\)) column. The benzene elutes (4 \(\times\) 30 ml), 20% chloroform in benzene (3 \(\times\) 30 ml), 50% chloroform in benzene (3 \(\times\) 30 ml), 80% chloroform in benzene (3 \(\times\) 30 ml), chloroform (3 \(\times\) 30 ml) fractions did not give any eluents. The 30% ethyl acetate in chloroform in the second fraction eluted out a light yellow band (12 ml) containing the major portion of the product (0.29 g). After crystallisation from \(n\)-hexane a micro-crystalline product having \(Rf\) value 0.59, oxo-corosin acetate methyl ester, m.p. 160 °C (puffs up above 100 °C, unsharp), \([\alpha]_D^160° = -173 (c = 0.06\%)\), \(\lambda_{max} = 260 (5.5), 255 (6.5), 208 \text{ nm} (c = 400; 525; 5,000 and 9,100).\) It was analysed with one mole of ethanolic of crystallisation which very clearly showed up in the NMR at 6.3 \(\tau\) (quartet, 2H; \(-CH_2O-\)) and 8.72 \(\tau\) (CH\(_3\)). This solvent molecule could be removed by heating at 150 °C only in vacuo over 24 h, whence the colourless crystals became translucent, to give corosin anhydro lactone acetate (anhyd.). It gave a mass molecular ion at \(m/e 548\).

\[
C_{36}H_{44}O_{10} \text{(646.8)}
\]
Caled \(C 69.84\) H 8.27, Found \(C 69.67\) H 8.36.

4. Corosolic acid (I, \(R_1 = R_2 = OH\))

Corosolic acid acetate (0.1 g) was taken in methanol (25 ml) and potassium hydroxide pellets (2.5 g) added. After refluxing for 90 min the solvent was removed in vacuo, the residue taken in water, acidified with dil. hydrochloric acid and extracted with ether. The etherial extract was washed with water, dried (Na\(_2\)SO\(_4\)) and evaporated to give a solid (0.1 g) which on crystallisation from chloroform/methanol, followed by recrystallisation from methanol gave corosolic acid, m.p. 240–242 °C, \([\alpha]_D^2 + 42^\circ (c = 0.11\%)\). It gave a mass molecular ion at \(m/e 472\) and had a \(\lambda_{max} = 1692 \text{ cm}^{-1}\) (COOH).

\[
C_{36}H_{44}O_4 \text{(472.7)}
\]
Caled \(C 76.22\) H 10.23, Found \(C 76.10\) H 10.10.

5. Corosolic acid methyl ester (I, \(R_1 = R_2 = OAc\); 28-OCH\(_3\))

Corosolic acid acetate (40 mg) was taken in dry ether and treated with excess ethereal diazomethane. Solvent was removed at pump and the residue chromatographed on silicagel column (1.5 g). The benzene and chloroform elutes did not give any eluents, the 20% ethyl acetate in chloroform gave a product which was crystallised from aqueous methanol to give corosolic acid methyl ester, m.p. 162–164 °C (puffs up above 100 °C, unsharp), \([\alpha]_D^162° = +2^\circ (c = 0.18\%)\). It gave a mass molecular ion at \(m/e 570\) and had a \(\lambda_{max} = 1740 \text{ cm}^{-1}\).

\[
C_{38}H_{40}O_8 \text{(580.8)}
\]
Caled \(C 71.39\) H 9.58, Found \(C 71.39\) H 9.20.

6. Ure-13(18)-en-2a,3\beta-diacetoxy-28-oic-20,28-lactone (11d)

The corosin anhydro lactone acetate prepared from corosolic acid gave colourless rods, m.p. 317–321 °C (decomp., \([\alpha]_D = -77^\circ (c = 0.06\%)\), \(\lambda_{max2} = 268, 250, 227, 208 \text{ nm} (c = 400; 525; 5,000 and 9,100).\) It was analysed with one mole of ethanol of crystallisation which very clearly showed up in the NMR at 6.3 \(\tau\) (quartet, 2H; \(-CH_2O-\)) and 8.72 \(\tau\) (CH\(_3\)). This solvent molecule could be removed by heating at 150 °C only in vacuo over 24 h, whence the colourless crystals became translucent, to give corosin anhydro lactone acetate (anhyd.). It gave a mass molecular ion at \(m/e 548\).

\[
C_{38}H_{44}O_8 \text{(584.7)}
\]
Caled \(C 69.84\) H 8.27, Found \(C 69.67\) H 8.36.

The lactone (0.7 g) after conversion into the C-24 acid chloride by thionyl chloride (7.5 ml) under reflux (30 min), could be reduced in refluxing dry toluene (35 ml) in presence of 10% Pd charcoal (fresh catalyst, 0.4 g) with dry hydrogen gas. A catalyst replacement (0.3 g) after 24 h was found essential for satisfactory reduction. On crystallisation from chloroform/methanol urs-13(18)-en-24-oic-2a,3\beta-diacetoxy-28-oic and 28-lactone, m.p. 320 to 330 °C (decomp. (sinters at 270 °C and resolidifies to rods) was obtained (0.425 g) [2]. It gave a mass molecular ion at \(m/e 568\).

The C-24 aldehyde lactone diacetate (0.14 g) was taken in diethylene glycol (8 ml) and hydrazine hydrate (1.4 ml) added. It was heated under dry nitrogen until a clear solution was obtained (\(\approx 150^\circ \text{ C bath temp.}\)) and then allowed to reflux for 30 min (180 °C bath temp.). During 40 min, 2.5 ml of distillate was allowed to distill over (bath temp. to 210 °C) and cooled down to 80 °C. Potassium
Hydroxide pellets (0.4 g) were added, the solution heated to 190 °C (bath) and allowed to cool down slowly to room temperature (90 min). It was poured in dilute hydrochloric acid and extracted with ether/ethyl acetate mixture. The organic extractive was washed with water, dried (Na₂SO₄) and evaporated. The residue was crystallized from chloroform/methanol to give conglomerates of flat needles (48 mg), m.p. 307-308 °C decomp., \([\alpha]_D\): —155° (c = 0.078%), \(A_{\text{max}}\): 207, 239, 250 nm (e 10,800; 12,700; 10,000). Mass molecular ion at m/e = 470.

\[\text{C}_{30}\text{H}_{46}\text{O}_4 \cdot 5\text{H}_2\text{O} (470.7)\]
Calcd C 75.55 H 9.92,
Found C 75.80 H 9.78.

This product (8 mg) was dissolved in methanol containing a drop of conc. hydrochloric acid, evaporated in vacuo (steam bath) and crystallized from methanol/water to give rods, m.p. 300-302 °C, decomp., \([\alpha]_D\): —108° (c = 0.073%), \(A_{\text{max}}\): 206, 230 nm (e = 8,400; 6,000).

The mother liquor from the reduction product gave a second crop, m.p. 293-298 °C, decomp., and a third crop m.p. 230-270 °C decomp. (35 mg), which showed in the ultraviolet spectrum maxima at 206 and 230 nm only. In the mass spectrum it showed a mass molecular ion at m/e 470. The third crop (35 mg) was dissolved in acetic anhydride (3 ml) and dry pyridine (1 ml) and left at room temperature overnight. The solvents were removed at the pump and the last traces removed by chloroform/methanol evaporation. The residue was crystallized from methanol/chloroform to give \(\text{urs-13(18)-en-2\alpha,3\beta-diacetoxy-28-oic-20,28-lactone}\) (19 mg), m.p. 310-320 °C decomp. (changing to rods above 280 °C), \([\alpha]_D\): —95° (c = 0.089%), \(A_{\text{max}}\): 207, 227 nm (e: 10,000; 6,000).

\[\text{C}_{34}\text{H}_{50}\text{O}_6 \cdot 0.5\text{CH}_3\text{OH} (554.7)\]
Calcd C 72.59 H 9.18,
Found C 72.42 H 9.01.

The mass molecular ion peak was observed at m/e = 554 and it gave fragments at m/e = 234, 201, 189. In the NMR spectrum it showed signals at \(\tau = 9.21, 9.54, 9.02, 8.98, 8.93, 8.80, 8.66, 8.54, 8.35, 8.05, 7.98, 5.34, 5.23\) and 4.92 (multiplet).

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