11a and **7** as a clear oil: ^{31}P NMR (benzene-d_6) δ -64.4 ($^1J_{PH}$ = 224.0) and -3.9. To a solution of the mixture in 15 mL of benzene was added 5 mL of 12.5% H_2O_2 and the reaction mixture stirred at ambient temperature for 18 h. The benzene layer was separated and the aqueous layer diluted with 5 mL of water and extracted with $CHCl_3$ (3 × 15 mL). The combined organic extracts were concentrated, yielding a pale oil to which 15 mL of 1% NaOH was added, and the solution was then extracted with $CHCl_3$ (3 × 20 mL). The combined $CHCl_3$ extracts were dried ($MgSO_4$), filtered, and concentrated, yielding 0.2 g of **12** as a clear oil: ^{31}P NMR ($CDCl_3$) δ +54.1; ^{13}C and ^1H NMR were identical with those of **12** prepared by gas-phase reaction of **5**.

The basic aqueous layer was acidified to pH 2 with 2 N H_2SO_4 and reextracted with $CHCl_3$ (3 × 20 mL). The combined extracts were dried ($MgSO_4$) and concentrated to yield 0.25 g of **13** as an oil, which crystallized to an off-white solid upon drying under high vacuum: mp 105-110 °C; ^{31}P NMR ($CDCl_3$) δ +30.4; ^{13}C NMR ($CDCl_3$) δ 116.3-131.3 (complex, unassigned sp^2 carbons), δ 147.5 (C-3); ^1H NMR ($CDCl_3$) δ 5.9-6.3 (t, $^2J_{PH}$ 13.4, $^3J_{HH}$ = 13.2 Hz, PCH=), 7.0-7.4 (m, aromatic). Anal. Calcd for $C_{14}H_{13}O_2P \cdot {}^1/_2H_2O$: C, 66.39; H, 5.38; P, 12.33. Found: 66.39; H, 5.51; P, 12.31.

Phenyl-*cis*-2-styrylphosphinic Acid (13). A solution of 0.6 g (2.6 mmol) of phosphine oxide **5** and 0.5 g (25 mmol) of NaOH in 50 mL of 85% Me_2SO-H_2O was heated at 100 °C for 6 h. Me_2SO and H_2O were removed by distillation at reduced pressure. The residue, dissolved in 40 mL of water, was extracted with $CHCl_3$ (3 × 50 mL) to remove nonacidic organics in the mixture. The aqueous layer was then acidified to pH 3 by addition of 2 N H_2SO_4 and reextracted with $CHCl_3$ (3 × 50 mL). This $CHCl_3$ extract was dried ($MgSO_4$) and concentrated to give 0.35 g (55%) of **13** as a clear oil, which crystallized slowly to a hygroscopic white solid: mp 106-110 °C; ^{31}P NMR ($CDCl_3$) δ +29.5; ^1H and ^{13}C NMR were identical with those of **13** prepared by the reduction method above.

Methylphenyl-*cis*-2-styrylphosphine (16). To a suspension of 1.1 g (3.1 mmol) of phosphonium salt **6** in 25 mL of benzene was added 10 mL of triethylamine. The mixture was refluxed under nitrogen for 2 days. The benzene solution was then decanted and the reaction flask rinsed with several portions of benzene. The solution was concentrated to give 0.30 g (43%) of **16** as a clear oil: ^{31}P NMR (benzene-d_6) δ -45.4; ^1H NMR (benzene-d_6) δ 1.3 (d, J = 6.1 Hz, PCH_3), 6.1-6.4 (d of d, $^2J_{PH}$ = 14.9, $^3J_{HH}$ = 4.1 Hz, PCH=), 7.0-7.9 (m, aromatic H); ^{13}C NMR (benzene-d_6) δ 14.6 (d, J = 12.2 Hz, PCH_3), 143.1 (d, J = 17.1 Hz, C-3), 141.9 (d, J = 13.4 Hz, C-4), 127.2-137.9 (complex, sp^2 C).

Phosphine **16** was analyzed as its methyl iodide salt, prepared by adding excess methyl iodide to a benzene solution of **16**. The resulting precipitate was filtered and washed with benzene, and a small amount was then recrystallized from methanol: mp 155-157 °C; ^{31}P NMR ($CDCl_3$) δ +12.5; ^{13}C NMR ($CDCl_3$) δ 11.7 (d, 57.4, PCH_3), 111.7 (d, 80.5, =CP), 127.6, 128.3, and 129.9 (styryl ring carbons), 129.7 (d, J = 12.2 Hz, phenyl ortho C), 131.2 (d, J = 11.0 Hz, phenyl meta C), 133.5 (d, J = 8.5 Hz, phenyl ipso C), 133.9 (d, J = 2.5 Hz, para C), 155.9 (C=CP). Anal. Calcd for $C_{16}H_{18}IP$: C, 52.19; H, 4.94; P, 8.41. Found: C, 52.06; H, 5.02; P, 7.95.

Epimerization of 1a to 8. A solution of 250 mg of dihydrophosphindole **1a** in 10 mL of xylene was heated at 150 °C for 16.5 h under nitrogen. The ^{31}P spectrum of an aliquot showed a 70:30 mixture of **1a** (δ -26.2) and **8** (δ -24.0); the gas chromatogram was identical with that for the mixture formed from **1a** at 345-370 °C.

Isomerization of 1a to 7. A solution of 0.5 g of **1a** in 15 mL of 15% NaOH was stirred at 25 °C for 3 h. The basic solution was neutralized with 2 N H_2SO_4 and extracted with $CHCl_3$ (3 × 25 mL). The organic extract was dried ($MgSO_4$) and concentrated to give 0.45 g of **7** as a clear oil: ^{31}P NMR (benzene-d_6) δ -3.9; ^{13}C and ^1H NMR were identical with those of **7** prepared by alternate methods.

Registry No. **1a**, 86901-20-4; **3** (R = C_6H_5), 76549-54-7; **4** (R = C_6H_5), 86941-21-1; **5**, 86940-55-8; **6**, 86901-21-5; **7**, 86901-22-6; **7** methyl iodide salt, 86901-28-2; **8**, 86941-22-2; **11a**, 86901-23-7; **11b**, 86901-24-8; **12**, 31236-96-1; **13**, 86901-25-9; **16**, 86901-26-0; **17**, 86901-27-1; $HSiCl_3$, 10025-78-2; 3,4-dibromo-1-phenylphospholane 1-oxide, 72620-95-2.

Crystal Structure of Orthosphenic Acid

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Received December 15, 1982

A new triterpenic compound, orthosphenic acid, has been isolated from *Orthosphenia mexicana* and its structure determined by X-ray analysis.

The study of Celastraceae has attracted considerable attention as this family contains physiologically active quinones.[1] From a member of this group of plants, *Orthosphenia mexicana* Standley,[2] we have isolated two triterpenic compounds, celastrol (**8**) and, to the best of our knowledge, a new compound, which we named orthosphenic acid (**1**).

The structure (**1**) given to this compound was based on the following data. The acid **1** ($C_{30}H_{48}O_5$) reacted with diazomethane to yield a methyl ester (**2**). Examination of the ^1H NMR spectrum of the latter showed absence of unsaturation and the presence of a secondary alcohol and of six methyl groups. Acetylation of **1**, with acetic anhydride in pyridine, gave the monoacetate **3** and the diacetate **5** in a 1:1 ratio. The proton geminal to the secondary alcohol group was shifted from δ 4.35 in **1** to δ 5.04 in the monoacetate **3** and to δ 5.85 in the diacetate **5**. When the

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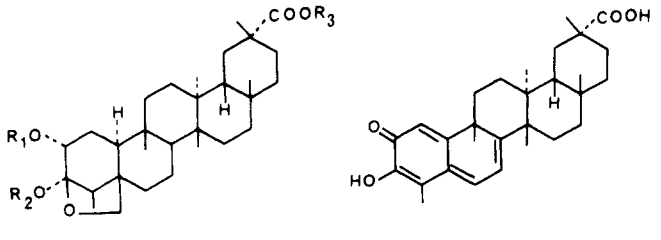


8

1 $R_1=R_2=R_3=H$
2 $R_1=R_2=H$ $R_3=CH_3$
3 $R_1=COCH_3$ $R_2=R_3=H$
4 $R_1=COCH_3$ $R_2=H$ $R_3=CH_3$
5 $R_1=R_2=COCH_3$ $R_3=H$
6 $R_1=R_2=COCH_3$ $R_3=CH_3$
7 $R_1=R_2=C(CH_3)_2$ $R_3=CH_3$

methyl ester **2** was refluxed with dry acetone and copper sulfate, the acetonide **7** was obtained. These results and the ^1H NMR data of **3** and **6** show that our compound (**1**) has a friedo-oleane skeleton.[3] The mass spectral fragmentation pattern of the orthosphenic acid derivatives are in accordance with the proposed structure.[3a,c,4]

The stereochemistry of the alcohol group at C-2 was given as α on the basis of the NMR of **1**. On the other hand, the presence of celastrol (**8**) in this plant suggests the C-29 position for the acid group.

Structure for orthosphenic acid (**1**) was confirmed by X-ray analysis (see Figure 1).

$C_{30}H_{48}O_5 \cdot H_2O$ crystallizes in the monoclinic system $P2_1$, with two molecules in the cell: $a = 7.4413$ (4) Å, $b = 30.275$ (1) Å, $c = 6.3812$ (3) Å, and $\beta = 113.54$ (2)° $\rho_x = 1.278$ g cm^{-3}. The intensities of the 2290 independent Friedel pairs for $2 < \theta < 65°$ were collected on a four-circle diffractometer with graphite-monochromated Cu Kα radiation. Each reflection was scanned during 0.5 min in the $\omega/2\theta$ mode. The structure was solved by direct methods and refined by full-matrix least-squares methods using the 2254 observed Friedel pairs with $I > 2\sigma(I)$. All hydrogen atoms were found in a difference map, and then a weighted anisotropic refinement (fixed isotropic for H atoms) converged to $R_{obsd} = 0.041$ and $R_{w(obsd)} = 0.045$.[5] The 88 more relevant Bijvoet pairs with $\Delta F_c > 0.08$[6] indicate the actual absolute configuration of the molecule, shown in Figure 1, giving an averaged Bijvoet difference of 0.194 vs. 0.276 for the wrong enantiomer. Tables I–V listing final atomic and anisotropic thermal parameters, bond lengths, bond angles, and torsion angles are available as supplementary material.

The atoms of the triterpenoid backbone do not deviate more than 1 Å from its best plane, although it is slightly convex (~15°) toward the β face of the molecule. This overall stretched conformation is the usual when rings A–C are chairs and D and E are boats. The Cremer conformational parameters[7] have been calculated for the six-membered rings and for the five-membered one, beginning at atoms 2, 10, 11, 13, 19, and 4, respectively, in a counterclockwise manner. θ_2 values are 22°, 23°, 5°, 79°, and 92°, Φ_2 values are 124°, 238°, 264°, 18°, and −4° for rings A–E, respectively, and $\Phi_2 = 189°$ for the five-membered ring. These values mean that A and B are more distorted chairs than C; ring D is between twist and boat, and E is a boat. The five-membered ring is almost exactly between a half-chair and an envelope. The torsion angle 23–20–24–2 is −2.3°.

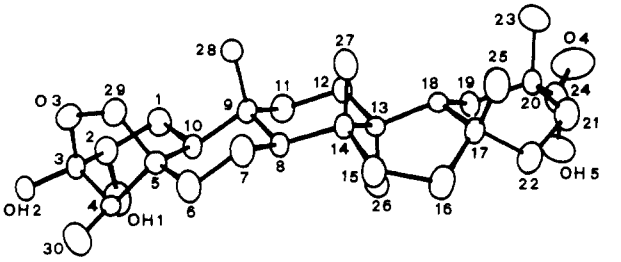
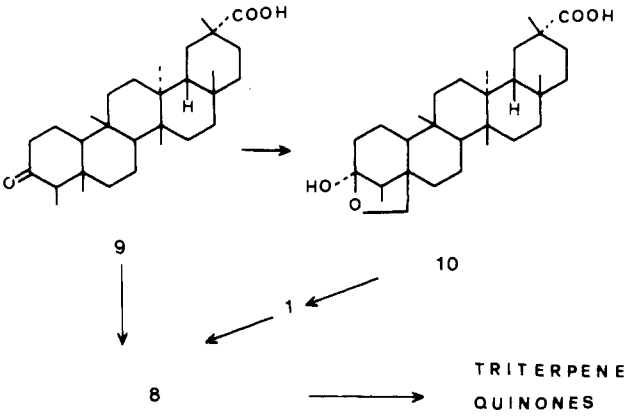


Figure 1. Structure for orthosphenic acid (**1**).

Scheme I



The water molecule is involved in four hydrogen bonds with different molecules, two as acceptors from OH5 (2.69 Å) and OH1 (3.12 Å), and two as donors to O3 (2.79 Å) and OH21 (2.82 Å). There is also another intermolecular H bond (O2H···O4) of 2.66 Å. The crystal structure is built through the H bonds which have been described above.

Salaspermic acid (**10**)[3a] and benulin[9] are two other examples of triterpenes with a hemiketal function occurring in plants.

Orthosphenic acid (**1**), (2*R*,3*R*,4*R*,5*S*,8*S*,9*R*,10*S*,13*S*,-14*R*,17*R*,18*R*,20*R*)-2,24-dihydroxy-3-oxofriedelan-29-oic acid hemiketal, may be an intermediate in the biosynthetic pathway that leads from polpunonic acid (**9**) to the triterpene quinones via salaspermic acid (**10**)[8b] (Scheme I). This makes the route more plausible than the alternative direct way of **9** to **8**.[8a]

Experimental Section

Melting points are uncorrected. ^1H NMR spectra were obtained with a Perkin-Elmer R-12B and R-32 at 60 and 90 MHz, respectively, by using deuterated solvents and tetramethylsilane as an internal standard. Mass spectra were obtained with a VG Micromass ZAB-2F. IR spectra were recorded on a Perkin-Elmer 257.

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From the overstem bark roots of *Orthosphenia mexicana* Standley (Celastraceae) was obtained a methanolic extract (13 g). After chromatography over silica gel, two major components, celastrol (**9**, 0.9 g) and orthosphenic acid (**1**, 300 mg), were isolated.

Orthosphenic Acid (1). This pale yellow compound had the following: mp 298–300 and 330 °C (double); IR (KBr) 3520–2800, 3210, 2980, 2950, 2920, 2900, 2875, 1695, 1450, 1438, 1390, 1240, 1210, 1190, 1145, 1065, 955 cm^{-1}; ^1H NMR (C_5D_5N) δ 0.85 (s, 3 H),0.92 (s, 3 H), 1.15 (br s, 9 H), 1.22 (s, 3 H), 3.74 (d, 1 H, J = 9 Hz), 4.22 (d, 1 H, J = 9 Hz), 4.35 (br s, 1 H, $W_{1/2}$ = 8 Hz) (partially overlapping with doublet at 4.22); mass spectrum, m/z (relative intensity) 488 (M^+, 20), 412 (32.9), 189 (28.6), 163 (15.0), 161 (16.0), 155 (20.8), 149 (21.6), 147 (16.9), 135 (21.7), 133 (17.3), 125 (99.1), 123 (19.5), 122 (18.7), 121 (34.2), 119 (33.3), 109 (100.0); calcd for $C_{30}H_{48}O_5$ mol wt 488.3501, found mol wt 488.3525 (high-resolution mass spectroscopy).

Methyl Orthosphenate (2). Esterification of **1** (20 mg) with diazomethane yielded **2**: mp 258–260 °C; IR (KBr) 3495, 3300, 2920, 2860, 1760, 1500, 1375, 1255, 1215, 1185, 1150, 1135, 1060, 1050, 980, 910 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.82 (s, 6 H), 0.91 (d, 3 H, J = 9 Hz), 0.93 (s, 3 H), 1.06 (s, 3 H), 1.16 (s, 3 H), 3.57 (d, 1 H, J = 9 Hz) (partially overlapping with singlet at 3.63) 3.63 (s, 3 H), 3.86 (br s, 1 H, $W_{1/2}$ = 8 Hz), 4.08 (d, 1 H, J = 9 Hz); mass spectrum, m/z (relative intensity) 5.02 (M^+, 26.4), 169 (28.3), 163 (12.2), 125 (100), 121 (24.1), 109 (39.3); calcd for $C_{31}H_{50}O_5$ mol wt 502.3658, found mol wt 502.3660 (high-resolution mass spectroscopy).

Monoacetate of Orthosphenic Acid (3). Esterification of **1** (25 mg) with acetic anhydride in pyridine at room temperature for 4 h yielded 12 mg of **3** and 11 mg of **5**. Compound **3** had the following: mp 250–252 C; IR (KBr) 3650–3100, 2920, 1740, 1450, 1370 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.86 (s, 3 H), between 0.91 (s), 0.95 (s), and 0.98 (s) (12 H), 2.10 (s, 3 H), 3.64 (d, 1 H, J = 9 Hz), 4.12 (d, 1 H, J = 9 Hz), 5.04 (br s, 1 H, $W_{1/2}$ = 9 Hz); mass spectrum, m/z (relative intensity) 530 (M^+, 64.0), 412 (32.0), 235 (6.0), 189 (52.0), 163 (89.0), 161 (4.0), 155 (10.0), 149 (62.0), 135 (6.2), 125 (100.0), 121 (113.0), 112 (4.7), 109 (10.0), 105 (64.0), 95 (18.4); calcd for $C_{32}H_{50}O_6$ mol wt 530.3607, found mol wt 530.3619 (high-resolution mass spectroscopy).

Monoacetate of Methyl Orthosphenate (4). Compound **3** (12 mg) was suspended in ethyl ether and esterified with diazomethane, yielding 11 mg of **4**: mp 232–234 °C; IR (KBr) 3420, 2910, 2850, 1740, 1720, 1450, 1380, 1370, 1250, 1220, 1135, 1040 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.83 (s, 6 H), 0.93 (s, 3 H), 0.94 (d, 3 H, J = 8 Hz), 1.06 (s, 3 H), 1.17 (s, 3 H), 2.10 (s, 3 H), 3.62 (d, 1 H, J = 9 Hz), 3.64 (s, 3 H), 4.10 (d, 1 H, J = 9 Hz), 5.02 (br s, 1 H, $W_{1/2}$ = 9 Hz); mass spectrum, m/z (relative intensity) 544 (M^+); calcd for $C_{33}H_{52}O_6$ mol wt 544.3764, found mol wt 544.3787 (high-resolution mass spectroscopy).

Diacetate of Orthosphenic Acid (5). This compound had the following: mp 106–110 °C; IR (KBr) 3600–3000, 2920, 2860, 1730, 1450, 1380, 1250, 1230, 1140 cm^{-1}; ^1H NMR ($CDCl_3$) δ (0.84 (d, 3 H, J = 7 Hz), 0.86 (s, 3 H), 0.96 (s) and 0.98 (s) (9 H), 1.09 (s, 3 H), 1.99 (s, 3 H), 2.07 (s, 3 H), 3.74 (d, 1 H, J = 9 Hz), 4.27 (d, 1 H, J = 9 Hz), 5.85 (br s, 1 H, $W_{1/2}$ = 9 Hz); mass spectrum, m/z (relative intensity) 530 (M^+ – 42) (73.4), 412 (4.6), 383 (5.5), 373 (4.2), 259 (4.8), 235 (9.7), 189 (5.5), 163 (9.1), 155 (7.6), 149 (6.6), 147 (5.1), 135 (7.1), 133 (5.7), 125 (100.0), 121 (12.7), 109 (21.6), 95 (21.4); calcd for $C_{32}H_{50}O_6$ (M^+ – 42) mol wt 530.3607, found mol wt 530.3589 (high-resolution mass spectroscopy).

Diacetate of Methyl Orthosphenate (6). Esterification of **5** (11 mg) with diazomethane yielded 10 mg of **6**: mp 144–146 °C; IR (KBr) 2910, 1860, 1750, 1730, 1460, 1450, 1380, 1370, 1250, 1220, 1140 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.83 (s, 6 H), 0.92 (s, 3 H), 0.93 (d, 3 H, J = 9 Hz), 1.06 (s, 3 H), 1.16 (s, 3 H), 1.98 (s, 3 H), 2.07 (s, 3 H), 3.64 (s, 3 H), 3.72 (d, 1 H, J = 11 Hz), 4.27 (d, 1 H, J = 11 Hz); mass spectrum, m/z 586 (M^+); calcd for $C_{32}H_{50}O_6$ (M^+ – 42) mol wt 544.3764, found mol wt 544.3761 (high-resolution mass spectroscopy).

Acetonide of Methyl Orthosphenate (7). This compound was obtained by refluxing **2** (20 mg) in dry acetone and copper sulfate, yielding 18 mg of **7**: mp 228–230 °C; IR (KBr) 2910, 2860, 1720, 1380, 1190, 1160, 1110 cm^{-1}; ^1H NMR ($CDCl_3$) δ 0.86 (s, 6 H), 0.94 (d, 3 H, J = 8 Hz), 0.96 (s, 3 H), 1.08 (s, 3 H), 1.18 (s, 3 H), 2.76 (s, 2 H), 3.63 (d, 1 H, J = 9 Hz), 3.66 (s, 3 H), 3.90 (br s, 1 H, $W_{1/2}$ = 9 Hz), 4.13 (d, 1 H, J = 9 Hz); mass spectrum, m/z (relative intensity) 502 (M^+ – 40, 33.7), 319 (6.0), 249 (7.3), 189 (8.0), 169 (28.8), 163 (11.1), 149 (71.0), 135 (9.2), 125 (100.0), 109 (34.1), 95 (24.6); calcd for $C_{31}H_{50}O_5$ (M^+ - 40) mol wt 502.3658, found mol wt 502.3665 (high-resolution mass spectroscopy).

Acknowledgment. Thanks are given to Prof. S. Garcia-Blanco for his support and to the Centro de Proceso de Datos de JEN, Madrid, for computer facilities. E.F. thanks the Instituto de Cooperación Iberoamericana and UNESCO for a fellowship.

Registry No. 1, 86632-20-4; 2, 86632-21-5; 3, 86632-22-6; 4, 86632-24-8; 5, 86632-23-7; 6, 86632-25-9; 7, 86632-26-0; 8, 34157-83-0.

Total Synthesis of (–)-Vertinolide. A General Approach to Chiral Tetronic Acids and Butenolides from Allylic Alcohols†

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Received April 7, 1983

A new method for the synthesis of chiral tetronic acids and butenolides from achiral allylic alcohols is described. Asymmetric epoxidation, followed by a one-step epoxide isomerization and opening under conditions of heating with NaCN, furnishes β-hydroxybutyrolactones upon acidic workup. These alcohols can either be dehydrated to the corresponding optically active α,β-unsaturated butyrolactones or oxidized in Me_2SO–trifluoroacetic anhydride to chiral tetronic acids. To illustrate the method, a chiral synthesis of vertinolide (**3**), a tetronic acid of fungal origin, is described which for the first time establishes this metabolite's absolute configuration.

Chiral, 4-substituted butenolides are natural products of considerable interest to man as ecologically important pheromones,[1] flavoring components in fruit and other foods,[2] and potent mycotoxins.[3] Tetronic acids comprise a subclass of β-hydroxybutenolides with general structure **1**, perhaps the best known of which is ascorbic acid (**2**) (vitamin C). Many tetronic acids and their derivatives

† Dedicated to the memory of the late Professor Daniel Swern.

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