Morinins H−K, Four Novel Phenylpropanol Ester Lipid Metabolites from *Morina chinensis*

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Received March 31, 1999

The medicinal plant, *Morina chinensis*, afforded four novel phenylpropanol ester lipid metabolites, named morinins H−K (**1**−**4**). Their structures were identified on the basis of spectral data interpretation.

In a previous paper, we reported seven new phenylpropanol derivatives, morinins A−G, as well as five other known phenolic compounds from *Morina chinensis* (Dipsacaceae).[1] In a continued study on the chemical constituents of this traditional Chinese medicinal herb, we now wish to report the isolation and structure elucidation of four other novel phenylpropanol ester lipid metabolites, morinins H−K (**1**−**4**) (Chart 1). A $CHCl_3$-soluble fraction of a MeOH extract of the roots of *M. chinensis* (Dipsacaceae) was purified using repeated silica gel column chromatography, gel permeation chromatography and HPLC to afford morinins H−K (**1**−**4**).

Compound **1** was obtained as a colorless oil. The HREIMS of **1** gave an intense parent ion at *m*/*z* 522.3324, corresponding to a molecular formula of $C_{33}H_{46}O_5$, which required 11 degrees of unsaturation. Similar to morinins D and F,[1] the ^{1}H NMR spectral data of **1** showed a cinnamyl alcohol, a methoxyl group at δ_H 3.82 (3H, s), and an oxygenated angeloyl group at δ_H 6.44 (1H, br q, J = 7.2 Hz, H-3′), 2.12 (3H, d, J = 7.2 Hz, H-4′), and 4.77 (2H, br s, H-5′). Similarly to morinins D and F, in compound **1** the C-5′ position of the angeloyl group was esterified, but in this case the moiety seemed to be an unsaturated fatty ester group, after careful analysis of the other signals of ^{1}H NMR spectrum. The ^{13}C NMR and DEPT spectral data of **1** further confirmed the above-mentioned conclusions.

In the HMBC spectrum of **1**, the correlations of δ_H 6.62 (H-7) with δ_C 129.02 (C-1), 127.88 (C-2, C-6), and 65.30 (C-9) and δ_H 4.77 (H-5′) with δ_C 165.85 (C-1′) 127.88 (C-2′), 143.76 (C-3′), and 173.51 (C-1″), indicated clearly that the angeloyl group was connected with C-9, with the unsaturated fatty ester chain attached to C-5′ of the angeloyl group.

On the basis of the above analysis, combined with the number of unsaturations and the molecular formula determined for compound **1**, it was deduced that the unsaturated fatty ester group was a C_{18} unit and bore three bouble bonds. The ^{1}H NMR spectrum showed signals for six olefinic protons at δ_H 5.28−5.43 (6H, m, H-9″, 10″, 12″, 13″, 15″, and 16″), four allylic protons at δ_H 2.01−2.08 (4H, m, H-8″, and 17″), and four double allylic methylene protons at δ_H 2.81 (4H, br s, H-11″ and 14″).[2,3] The ^{13}C NMR and DEPT spectral data also displayed six olefinic methine carbons at δ_C 130.34 (C-9″), 127.79 (C-10″), 128.36 (C-12″), 128.32 (C-13″), 127.18 (C-15″), and 132.03 (C-16″), two allylic methylenes carbons at δ_C 27.28 (C-8″) and 20.62 (C-17″), and two double allylic methylenes carbons at δ_C 25.68 (C-11″) and 25.60 (C-14″). All of these assignments were verified by the correlations in the ^{1}H−^{1}H COSY, HSQC, and HMBC spectra.

According to published reports[4−6] on the ^{13}C NMR chemical shifts of allylic methylene carbons (*Z* alkenes, δ_C < 27 ppm; *E* alkenes, δ_C > 30 ppm) of alkenes, all of the three double bonds of the unsaturated fatty ester group of **1** should be in the *Z* configuration, as deduced from the ^{13}C NMR spectral data of the allylic methylenes (δ_C 27.28, C-8″, 25.68, C-11″, 25.60, C-14″, 20.62, C-17″). In the ^{1}H−^{1}H COSY spectrum, the correlations of δ_H 0.98 (H-18″) with δ_H 2.01−2.08 (H-17″) and of δ_H 2.01−2.08 (H-17″) with δ_H 5.28−5.43 (olefinic protons) suggested that there was a double bond between C-15″ and C-16″. This was supported by the correlations of δ_H 0.98 (H-18″) with δ_C 20.62 (C-17″) and 132.03 (C-16″) in the HMBC spectrum. Accordingly, the other two double bonds must be between C-9″, C-10″, and C-12″, C-13″, respectively. The ^{13}C NMR spectral data of this unsaturated fatty ester group were in good agreement with those of linolenic acid.[6] Thus, the structure of compound **1** has been determined as shown, and named morinin H.

Compound **2** was obtained as a colorless oil. The molecular ion (HREIMS) of compound **2**, at *m*/*z* 524.3511 ($C_{33}H_{48}O_5$), was 2 amu greater than that of compound **1**, while both compounds exhibited the same intense fragments at *m*/*z* 147, 163, 216, and 244 in the EIMS. The ^{1}H NMR spectral data of **2** were nearly identical with the data of **1**, except for the olefinic protons (δ_H 5.30−5.42, m, H-9″, 10″, 12″ and 13″), of which there were four, and the double allylic methylene protons (δ_H 2.77, t, J = 6.4 Hz, H-11″), of which there were only two. The ^{13}C NMR spectral data also showed four olefinic methines at δ_C 130.10 (C-9″), 128.09 (C-10″), 127.98 (C-12″), and 130.27 (C-13″) and only one double allylic methylene at δ_C 25.68 (C-11″). In the HMBC spectrum, the observed correlations of δ_H 4.82 (H-9) with δ_C 165.83 (C-1′), δ_H 4.77 (H-5′) with δ_C 165.83 (C-1′) and 173.49 (C-1″) were similar to those seen for **1**. In the EIMS of compound **2**, two strong fragments appeared at *m*/*z* 277 and 377, corresponding to *m*/*z* 275 and 375 in compound **1**. Thus, compound **2** was a dihydro derivative of compound **1**.

In the ^{1}H−^{1}H COSY spectrum of **2**, the terminal methyl signal at δ_H 0.89 (H-18″) did not show any correlation with an allylic methylene proton signal, but it showed correlated with overlapped methylene signals at δ_H 1.27−1.37, suggesting that the double bond between C-15″ and C-16″ present in compound **1** was hydrogenated in compound **2**. In the HMBC spectrum of **2**, the correlations of δ_H 0.89 (H-18″) with δ_C 22.64 (C-17″) and 31.58 (C-16″); δ_H 5.30−5.42 (olefinic protons) with δ_C 25.68 (C-11″) and 27.26 (C-8″, C-14″); δ_H 2.03−2.08 (H-8″, 14″) with the undisturbed

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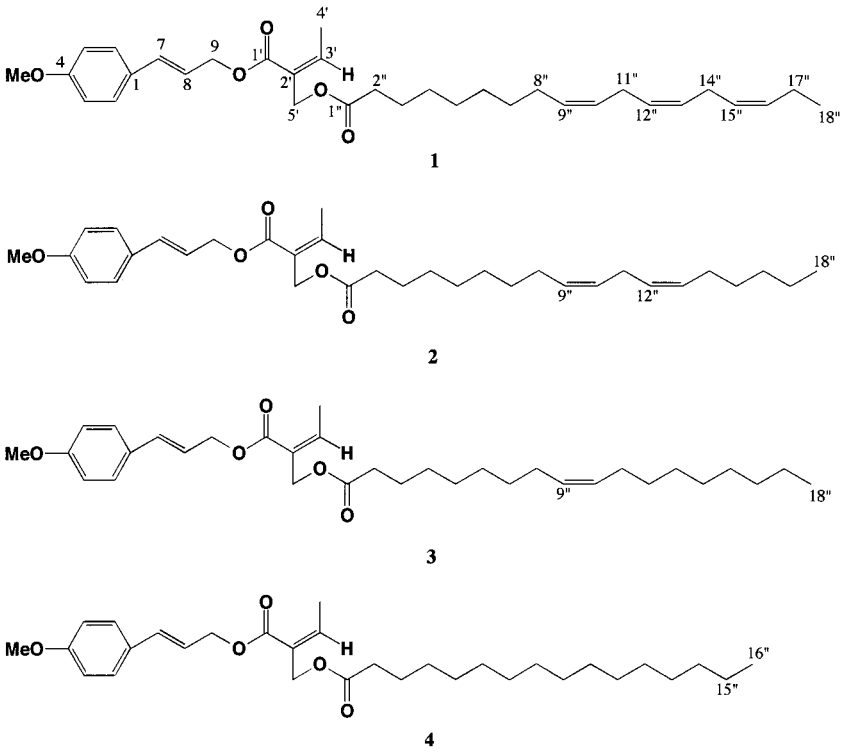
10.1021/np990145n CCC: $18.00
Published on Web 08/05/1999

Chart 1



signal δ_C 31.58 (C-16″), indicated that the two double bonds should be between C-9″, C-10″ and C-12″, C-13″, respectively. Both of the double bonds were in the *Z* configuration according to the chemical shifts of the allylic methylenes (δ_C 27.26, C-8″, 14″; δ_C 25.68, C-11″).[4−6] All of the ^{13}C NMR spectral data of the determined fatty ester group were also in good agreement with those of the linolelaidic acid.[6] Thus, the structure of morinin I has been identified as **2**.

Compound **3** was obtained as a colorless oil. The molecular ion (HREIMS) of compound **3** at *m*/*z* 526.3670 ($C_{33}H_{50}O_5$) was 2 amu greater than that of compound **2** and 4 amu greater than that of compound **1**, and it also showed intense fragments at *m*/*z* 147, 163, 216, and 244 in the EIMS. The ^{1}H and ^{13}C NMR spectral data of **3** were very similar to the data of compounds **1** and **2**. In compound **3**, it was shown that only one double bond was present in the fatty ester group both with signals at δ_H 5.30−5.39 (2H, m, H-9″, 10″) and δ_C 129.83 (C-9″) and 130.07 (C-10″), and there were no signals of double allylic methylenes near δ_H 2.80 and δ_C 26.00, and just two allylic methylenes at δ_H 2.00−2.05 (4H, m, H-8″, 11″) and δ_C 27.30 (C-8″) and 27.29 (C-11″). In the HMBC spectrum, the correlations of δ_H 4.82 (H-9) with δ_C 165.87 (C-1′) and of δ_H 4.77 (H-5′) with δ_C 165.87 (C-1′) and 173.54 (C-1″) were observed as for compounds **1** and **2**. The double bond was in the *Z* configuration according to the chemical shifts of the allylic methylenes (δ_C 27.30, C-8″; δ_C 27.29, C-11″).[4−6] The ^{13}C NMR spectral data of this unsaturated fatty ester group were in good agreement with those of elaidic acid.[6,7] Hence, the structure of compound **3** was identified as shown, and named morinin J.

Compound **4** was also obtained as a colorless oil. The ^{1}H and ^{13}C NMR spectral data of **4** were similar to those of compounds **1**−**3**. However, the NMR spectra showed that the fatty ester group was saturated. The HREIMS of compound **4** gave the parent molecular ion at *m*/*z* 500.3537, characteristic of the molecular formula, $C_{31}H_{48}O_5$. The unsaturation value was 8, suggesting not only that the fatty ester group had no double bond, but also that the chain length was C_{16}, with a palmitoyl group connected with the C-5′ of the angeloyl group in compound **4**. The structure was confirmed by the correlations of ^{1}H−^{1}H COSY, HSQC and HMBC. We named this compound morinin K.

Polyacetylenes, lipids, and related compounds have attracted much interest over the years for their broad range of biological activities, and many of these compounds have been isolated from natural sources, especially from marine sponges.[2,3,8,9] Morinins H−K (**1**−**4**), in which the lipid ester groups are connected with the angeloyl groups of phenylpropanol derivatives represent a rare compound class in higher plants.

Experimental Section

General Experimental Procedures and Plant Material. These were as described in a previous contribution.[1]

Extraction and Isolation. Powdered air-dried roots of *Morina chinensis* were extracted with MeOH (3 × 15 L) at 60 °C. After concentration of the combined extracts under reduced pressure, the residue (200 g) was suspended with water and then extracted with $CHCl_3$ and *n*-butanol, respectively.

The $CHCl_3$ extract (120 g) was chromatographed over a Si gel column (11 × 100 cm, Merck silica gel 60, 1.6 kg) and eluted with *n*-hexane−acetone (15:1 to 1:1), acetone, and MeOH. Thirteen fractions were collected. Fraction 1 (0.8 g) was chromatographed over a Si gel column (2.0 × 70 cm) and eluted with hexane−EtOAc (10:1), to give eight fractions (Fr1.1−Fr1.8). Fr1.3 was purified by using gel filtration chromatography ($CHCl_3$) to give seven fractions (Fr1.3.1−Fr1.3.7). Fr1.3.3 was then purified by HPLC (Si gel 60, Hibar RT 250-25, hexane−EtOAc, 10:1), to afford compound **1** (22 mg) and a mixture (48 mg), and then the mixture was further purified using the same HPLC column, using hexane−EtOAc (18:1), to produce the pure compounds **2** (12 mg), **3** (19 mg), and **4** (11 mg).

Morinin H (1): $[\alpha]_D^{24}$ +2.3° (*c* 0.78, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ϵ) 266.2 (3.89) nm; UV (MeOH) λ_{max} (log ϵ) 202.8 (3.96), 269.0 (3.41) nm; IR (KBr) ν_{max} 2952, 2368, 1712, 1655, 1608, 1578, 1514, 1388, 1351, 1253, 1231, 1155, 1038, 968 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.33 (2H, d, J = 8.6 Hz, H-2 and

H-6), 6.86 (2H, d, J = 8.6 Hz, H-3 and H-5), 6.62 (1H, d, J = 15.9 Hz, H-7), 6.44 (1H, br q, J = 7.2 Hz, H-3′), 6.18 (1H, dt, J = 15.9, 6.5 Hz, H-8), 5.28−5.43 (6H, m, H-9″, 10″, 12″, 13″, 15″, and 16″), 4.82 (2H, br d, J = 6.5 Hz, H-9), 4.77 (2H, br s, H-5′), 3.82 (3H, s, OMe), 2.81 (4H, br s, H-11″ and 14″), 2.28 (2H, t, J = 7.5 Hz, H-2″), 2.12 (3H, d, J = 7.2 Hz, H-4′), 2.01−2.08 (4H, m, H-8″ and 17″), 1.59 (2H, m, H-3″), 1.26−1.39 (8H, m, H-4″ to H-7″), 0.98 (3H, t, J = 7.6 Hz, H-18″); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 173.51 (s, C-1″), 165.85 (s, C-1′), 159.67 (s, C-4), 143.76 (d, C-3′), 134.00 (d, C-7), 132.03 (d, C-16″), 130.34 (d, C-9″), 129.02 (s, C-1), 128.36 (d, C-12″), 128.32 (d, C-13″), 127.88 (d, C-2 and C-6; s, C-2′), 127.79 (d, C-10″), 127.18 (d, C-15″), 120.82 (d, C-8), 114.08 (d, C-3 and C-5), 65.38 (t, C-5′), 65.30 (t, C-9), 55.35 (q, OMe), 34.36 (t, C-2″), 29.64 (t, C-7″), 29.22 (t, C-6″), 29.18 (t, C-5″), 29.15 (t, C-4″), 27.28 (t, C-8″), 25.68 (t, C-11″), 25.60 (t, C-14″), 24.98 (t, C-3″), 20.62 (t, C-17″), 15.93 (q, C-4′), 14.36 (q, C-18″); EIMS m/z 522 $[M]^+$ (56), 523 (22), 375 (15), 277 (11), 275 (78), 261 (28), 244 (42), 216 (16), 185 (17), 163 (100), 147 (175), 135 (31) 131 (34), 121 (32), 117 (23), 115 (54), 103 (40), 91 (90), 82 (99), 79 (90), 67 (80), 55 (59), 41 (58); HREIMS m/z 522.3324 (calcd for $C_{33}H_{46}O_5$, 522.3345).

Morinin I (2): $[\alpha]_D^{24}$ +0.3° (c 1.25, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ϵ) 266.3 (3.78) nm; UV (MeOH) λ_{max} (log ϵ) 203.5 (3.85), 266.0 (3.40) nm; IR (KBr) ν_{max} 2938, 1713, 1652, 1606, 1582, 1516, 1461, 1386, 1351, 1260, 1232, 1157, 1033, 967 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.33 (2H, d, J = 8.6 Hz, H-2 and H-6), 6.86 (2H, d, J = 8.6 Hz, H-3 and H-5), 6.62 (1H, d, J = 15.9 Hz, H-7), 6.44 (1H, br q, J = 7.3 Hz, H-3′), 6.18 (1H, dt, J = 15.9, 6.4 Hz, H-8), 5.30−5.42 (4H, m, H-9″, 10″, 12″, and 13″), 4.82 (2H, br d, J = 6.4 Hz, H-9), 4.77 (2H, br s, H-5′), 3.82 (3H, s, OMe), 2.77 (2H, t, J = 6.4 Hz, H-11″), 2.28 (2H, t, J = 7.5 Hz, H-2″), 2.12 (3H, d, J = 7.3 Hz, H-4′), 2.03−2.08 (4H, m, H-8″ and 14″), 1.59 (2H, m, H-3″), 1.27−1.37 (14H, m, H-4″ to H-7″, and H-15″ to 17″), 0.89 (3H, t, J = 6.9 Hz, H-18″); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 173.49 (s, C-1″), 165.83 (s, C-1′), 159.66 (s, C-4), 143.74 (d, C-3′), 133.98 (d, C-7), 130.27 (d, C-13″), 130.10 (d, C-9″), 129.00 (s, C-1), 128.09 (d, C-10″), 127.98 (d, C-12″), 127.91 (d, C-2 and C-6), 127.90 (s, C-2′), 120.80 (d, C-8), 114.06 (d, C-3 and C-5), 65.37 (t, C-5′), 65.28 (t, C-9), 55.33 (q, OMe), 34.35 (t, C-2″), 31.58 (t, C-16″), 29.67 (t, C-7″), 29.40 (t, C-15″), 29.21 (t, 6″), 29.17 (t, C-4″), 29.14 (t, C-5″), 27.26 (t, C-8″ and 14″), 25.68 (t, C-11″), 24.97 (t, C-3″), 22.64 (t, C-17″), 15.91 (q, C-4′), 14.14 (q, C-18″); EIMS m/z 524 $[M]^+$ (21), 497 (8), 377 (10), 361 (12), 343 (8), 277 (47), 263 (26), 244 (55), 185 (11), 163 (100), 148 (51), 147 (222), 135 (14), 131 (13), 121 (20), 115 (16), 99 (20), 82 (99), 69 (24), 55 (45), 54 (43), 41 (28); HREIMS m/z 524.3511 (calcd for $C_{33}H_{48}O_5$, 524.3502).

Morinin J (3): $[\alpha]_D^{24}$ +1.0° (c 1.15, $CHCl_3$); UV ($CHCl_3$) λ_{max} (log ϵ) 267.4 (3.75) nm; UV (MeOH) λ_{max} (log ϵ) 206.3 (3.92), 263.5 (3.78) nm; IR (KBr) ν_{max} 2932, 2848, 1717, 1703, 1678, 1656, 1639, 1604, 1517, 1460, 1160, 1149, 1026, 970 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.33 (2H, d, J = 8.7 Hz, H-2 and H-6), 6.86 (2H, d, J = 8.7 Hz, H-3 and H-5), 6.62 (1H, d, J = 15.9 Hz, H-7), 6.44 (1H, br q, J = 7.3 Hz, H-3′), 6.18 (1H, dt, J = 15.9, 6.5 Hz, H-8), 5.30−5.39 (2H, m, H-9″ and 10″), 4.82 (2H, br d, J = 6.5 Hz, H-9), 4.77 (2H, br s, H-5′), 3.82 (3H, s, OMe), 2.28 (2H, t, J = 7.5 Hz, H-2″), 2.12 (3H, d, J = 7.3 Hz, H-4′), 2.00−2.05 (4H, m, H-8″ and 11″), 1.59 (2H, m, H-3″), 1.26−1.38 (18H, m, H-4″ to H-7″, and H-12″ to 17″), 0.89 (3H, t, J = 6.9 Hz, H-18″); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 173.54 (s, C-1″), 165.87 (s, C-1′), 159.68 (s, C-4), 143.77 (d, C-3′), 134.01 (d, C-7), 130.07 (d, C-10″), 129.83 (d, C-9″), 129.04 (s, C-1), 127.94 (d, C-2 and C-6), 127.93 (s, C-2′), 120.84 (d, C-8), 114.09 (d, C-3 and C-5), 65.39 (t, C-5′), 65.31 (t, C-9), 55.36 (q, OMe), 34.39 (t, C-2″), 31.99 (t, C-16″), 29.85 (t, C-12″), 29.78 (t, C-7″ and 13″), 29.71 (t, C-6″), 29.51 (t, C-5″ and 14″), 29.32 (t, C-15″), 29.30 (t, C-4″), 27.30 (t, C-8″), 27.29 (t, C-11″), 25.00 (t, C-3″), 22.77 (t, C-17″), 15.94 (q, C-4′), 14.20 (q, C-18″); EIMS m/z 526 $[M]^+$ (26), 527 (10), 363 (27), 345 (13), 279 (14), 265 (36), 245 (15), 244 (75), 216 (15), 215 (9), 200 (7), 199 (5), 185 (14), 177 (9), 164 (14), 163 (100), 161 (31), 147 (244), 135 (21), 131 (21), 121 (30), 115 (27), 103 (21), 83 (38), 82 (100), 69 (55), 55 (87), 43 (58), 41 (59); HREIMS m/z 526.3670 (calcd for $C_{33}H_{50}O_5$, 526.3658).

Morinin K (4): $[\alpha]_D^{24}$ +3.9° (c 0.57, $CHCl_3$); UV (CHCl3) λ_{max} (log ϵ) 266.9 (3.73) nm; UV (MeOH) λ_{max} (log ϵ) 207.4 (3.85), 263.6 (3.70) nm; IR (KBr) ν_{max} 2982, 2935, 1721, 1651, 1606, 1516, 1459, 1385, 1358, 1169, 1031, 967 cm^{-1}; ^{1}H NMR (CDCl3, 400 MHz) δ 7.33 (2H, d, J = 8.7 Hz, H-2 and H-6), 6.86 (2H, d, J = 8.7 Hz, H-3 and H-5), 6.63 (1H, d, J = 15.9 Hz, H-7), 6.44 (1H, br q, J = 7.3 Hz, H-3′), 6.19 (1H, dt, J = 15.9, 6.5 Hz, H-8), 4.82 (2H, br d, J = 6.5 Hz, H-9), 4.77 (2H, br s, H-5′), 3.82 (3H, s, OMe), 2.28 (2H, t, J = 7.5 Hz, H-2″), 2.12 (3H, d, J = 7.3 Hz, H-4′),1.59 (2H, m, H-3″), 1.24−1.39 (24H, m, H-4″ to H-15″), 0.89 (3H, t, J = 6.8 Hz, H-16″); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 173.58 (s, C-1″), 165.87 (s, C-1′), 159.68 (s, C-4), 143.78 (d, C-3′), 134.01 (d, C-7), 129.04 (s, C-1), 127.95 (d, C-2 and C-6), 127.91 (s, C-2′), 120.85 (d, C-8), 114.10 (d, C-3 and C-5), 65.40 (t, C-5′), 65.32 (t, C-9), 55.37 (q, OMe), 34.41 (t, C-2″), 32.01 (t, C-14″), 29.78 (t, C-4″, 6″, and 13″), 29.77 (t, C-11″ and 12″), 29.76 (t, C-5″), 29.57 (t, C-10″), 29.45 (t, C-7″), 29.35 (t, C-9″), 29.21 (t, C-8″), 25.02 (t, C-3″), 22.78 (t, C-15″), 15.94 (q, C-4′), 14.22 (q, C-16″); EIMS m/z 500 $[M]^+$ (7), 388 (8), 337 (29), 244 (49), 239 (34), 185 (6), 163 (63), 161 (19), 148 (20), 147 (100), 135 (6), 132 (5), 131 (8), 121 (12), 115 (9), 103 (7), 99 (10), 91 (11), 83 (10), 82 (51), 71 (11), 57 (22), 55 (16), 54 (17), 43 (28), 41 (14); HREIMS m/z 500.3537 (calcd for $C_{31}H_{48}O_5$, 500.3502).

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NP990145N