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# ALKANE DIOLS FROM FLOWER PETALS OF *CARTHAMUS TINCTORIUS*

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**Key Word Index**—*Carthamus tinctorius*; Carthami Flos; Compositae; petals; secondary *syn*-alkane-1,3-diols.

**Abstract**—Eleven novel secondary alkane-1,3-diols were isolated from a methanol extract of dried flower petals of *Carthamus tinctorius*. Their structures were determined to be *syn* (*R,S* and/or *S,R*)-$C_{36}$-alkane-6,8-diol, *syn*-$C_{28}$-, $C_{30}$-, $C_{32}$, $C_{34}$- and $C_{36}$-alkane-7,9-diols, and *syn*-$C_{27}$, $C_{29}$-, $C_{31}$-, $C_{33}$- and $C_{35}$-alkane-8,10-diols by spectral methods. 

## INTRODUCTION

Carthami Flos, the dried flower petals of *Carthamus tinctorius*, is a Chinese crude drug used for the treatment of disease in women [1]. A methanol extract of this species was found to inhibit the 12-*O*-tetradecanoylphorbol-13-acetate (TPA)-induced inflammation and tumour promotion in two-stage carcinogenesis in mice [2], in which sterols [2] and alkanediols [3, 4] were the most active compounds. The alkanediol fraction contained 12 *syn*-alkane-6,8-diols with carbon numbers of $C_{21}$, $C_{23}$, $C_{25}$ and $C_{27}$-$C_{35}$ (**1–12**) [5]. The present paper describes further investigations on this alkanediol fraction, which led us to isolate and characterize a further 11 novel compounds (**13–23**).

## RESULTS AND DISCUSSION

The methanol extract of Carthami Flos was partitioned between *n*-hexane–methanol–water (19:19:2). The *n*-hexane fraction was subjected to silica gel column chromatography, which yielded a mixture of sterols and alkanediols. Further chromatography of the mixture enabled the separation of the alkanediol fraction [5]. Preparative HPLC of the fraction yielded 12 *syn*-alkane-6,8-diols **1–12** [5] and 11 further alkanediols **13–23**.

The EI-mass spectrum of **13** displayed the highest-mass ion at *m/z* 520 ($C_{36}H_{72}O$) [M-$H_2O$]$^+$, accompanied by diagnostic fragmentation ions at *m/z* 467 [M − $C_5H_{11}$]$^+$ (fragment **i**), 423 [M − $C_7H_{15}O$]$^+$ (**ii**), 145 [$C_8H_{17}O_2$]$^+$ (**iii**) and 101 [$C_6H_{13}O$]$^+$ (**iv**), formed by α-cleavage of hydroxyl groups [6]. The fragmentation pattern and $^1$H NMR spectral data (see Experimental) of **13** were essentially the same as those of *syn*-alkane-6,8-diols [5] and, hence, **13** was thought to possess a *syn*-alkane-6,8-diol structure, $C_{36}$ i.e. (6*R*, 8*S* and/or 6*S*, 8*R*)-*syn*-hexatriacontane-6,8-diol.

The alkanediol **15** showed a [M+H]$^+$ at *m/z* 455 ($C_{30}H_{63}O_2$) in its CI-mass spectrum, accompanied by diagnostic ions at *m/z* 437 [M+H−$H_2O$]$^+$ and 419 [M+H−2$H_2O$]$^+$. An absorption band due to a hydroxyl group at 3308 cm$^{-1}$ in the IR spectrum suggested that **15** was dihydroxy $C_{30}$ alkane. The EI-mass spectrum of **15**, which displayed the highest-mass ion at *m/z* 436 ($C_{30}H_{60}O$) [M−$H_2O$]$^+$, gave prominent fragmentation ions at *m/z* 369 [M − $C_6H_{13}$]$^+$ (fragment **i**), 325 [M − $C_8H_{17}O$]$^+$ (**ii**), 159 [$C_9H_{19}O_2$]$^+$ (**iii**) and 115 [$C_7H_{15}O$]$^+$ (**iv**), formed by α-cleavage of hydroxyl groups. This suggested that the two hydroxyl groups are located at C-7 and C-9, and **15** was, thus, considered to have the structure, triacontane-7,9-diol. The $^1$H NMR spectrum of **15** exhibited signals due to two terminal methyls [δ 0.88 (3H, *t*, *J* = 7.1 Hz) and 0.90 (3H, *t*, *J* = 6.9 Hz)], two hydroxyl-bearing methines [δ 2.77 (2H, *m*)], one methine adjacent to two hydroxyl-bearing methines [-CH(OH)-C$H_2$-CH(OH)-] [δ 1.45 (1H, *ddd*, *J* = 10.0, 11.1, 14.6 Hz) and 1.61 (1H, *ddd*, *J* = 2.2, 2.2, 14.6 Hz)] and methylenes (δ 1.25, *br s*), which were consistent with the proposed structure of **15**. The coupling patterns of the methylene proton signals at δ 1.45 and 1.61 of **15** were in good agreement with those observed for *syn*-pentane-

2,4-diol [$\delta$ 1.48 (1H, *ddd*, $J$ = 9.8, 9.8, 14.2 Hz); $\delta$ 1.51 (1H, *ddd*, $J$ = 2.7, 2.7, 14.2 Hz)] but different from those for the *anti*-isomer [$\delta$ 1.54 (2H), *ddd*, $J$ = 3.0, 8.5, 14.2 Hz] [7], which suggested that **15** has a (7*R*, 9*S* and/or 7*S*, 9*R*)-*syn*-configuration with respect to the diol system. The *syn*-configuration of **15** was supported from the $^1$H NMR data of its cyclic acetal derivative (**24**), prepared by treatment with acetaldehyde in the presence of *p*-toluenesulphonic acid [5, 8]. The acetal **24**, which occurred in the most stable chair form with an equatorially oriented methyl group at C-1′ [8], displayed the C-8 methylene ($H_A$ and $H_B$) signals as an ABXY-system in the $^1$H NMR spectrum at $\delta$ 1.20 [$H_A$, *ddd*, $J_{AB}$ = 13.3 Hz, $J_{AX}$ (ax.-ax.) = $J_{AY}$ (ax.-ax.) = 11.3 Hz] and 1.50 [$H_B$, *ddd*, $J_{AB}$ = 13.3 Hz, $J_{BX}$ (eq.-ax.) = $J_{BY}$ (eq.-ax.) = 2.3 Hz]. The coupling constants of the ABXY-spin system were in agreement with those observed for *syn*-hentriacontane-6,8-diol acetal [5] and the *syn*-form of 5,7-dialkyl-1,3-dioxane derivatives [9] but different from those observed for the *anti*-isomer of the dioxane derivatives [9].

The other four alkanediols were characterized as $C_{28}$ (**14**), $C_{32}$ (**16**), $C_{34}$ (**17**) and $C_{36}$ (**18**) *syn*-alkane-7,9-diols based on their EI-mass spectral data ($[M-H_2O]^+$ and diagnostic fragmentation ions **i–iv**) and $^1$H NMR spectral similarities with those of **15**.

The alkanediol **20** showed a $[M+H]^+$ at $m/z$ 441 ($C_{29}H_{61}O_2$), accompanied by fragment ions at $m/z$ 423 $[M+H-H_2O]^+$ and 405 $[M+H-2H_2O]^+$ in the CI-mass spectrum, and hydroxyl absorption in the IR spectrum (3310 cm$^{-1}$), suggesting that it was a dihydroxy $C_{29}$ alkane. The EI-mass spectrum of **20** displayed the highest-mass ion at $m/z$ 422 ($C_{29}H_{58}O$) $[M-H_2O]^+$ accompanied by diagnostic fragment ions at 341 $[M-C_7H_{15}]^+$ (fragment **i**), 297 $[M-C_9H_{19}O]^+$ (**ii**), 173 $[C_{10}H_{21}O_2]^+$ (**iii**) and 129 $[C_8H_{17}O]^+$ (**iv**) due to $\alpha$-cleavage of hydroxyl groups. This indicated that the two hydroxyl groups are located at C-8 and C-10 and, hence, **20** was considered to be nonacosane-8,10-diol. The $^1$H NMR spectra of **20** and its cyclic acetal **25** (see Experimental) were essentially the same as those of **15** and **24**, respectively, which enabled the assignment of the *syn*-configuration with respect to the diol system of **20**. Thus, **20** was (8*R*, 10*S* and/or 8*S*, 10*R*)-*syn*-nonacosane-8,10-diol. The other four alkanediols were characterised as $C_{27}$ (**19**), $C_{31}$ (**21**), $C_{33}$ (**22**) and $C_{35}$ (**23**) *syn*-alkane-8,10-diols based on their EI-mass spectral data ($[M-H_2O]^+$ and diagnostic fragmentation ions **i–iv**) and $^1$H NMR spectral similarities with those of **20**.

Our recent [5] and present study has thus revealed that Carthami Flos contains 23 *syn*-alkane-1,3-diols: 13 *syn*-alkane-6,8-diols [**1** (2.5%) of alkanediol fraction), **2** (1.3%), **3** (0.9%), **4** (8.3%), **5** (5.4%), **6** (7.3%), **7** (2.8%), **8** (28.6%), **9** (4.3%), **10** (14.5%), **11** (2.0%), **12** (2.4%) and **13** (0.5%)], five *syn*-alkane-7,9-diols [**14** (0.1%), **15** (0.1%), **16** (0.8%), **17** (0.6%) and **19** (0.1%)] and five *syn*-alkane-8,10-diols [**19** (0.1%), **20** (0.2%), **21** (0.6%), **22** (1.1%) and **23** (0.3%)], amongst which, eleven (**13–23**) were isolated and characterized for the first time from a natural source. Major components were alkane-6,8-diols with an odd carbon number should be noted that only even carbon numbered alkane-7,9-diols and odd carbon numbered alkane-8,10-diols were detected.

## EXPERIMENTAL

Recrystallizations: $Me_2CO$–MeOH. Mp: uncorr. HPLC: $C_{18}$ silica column (Superiorex ODS S-5 $\mu$m column, 25 cm × 10 mm i.d.; Shiseido), MeOH as mobile phase (flow rate 4 ml min$^{-1}$). GC: DB-17 fused-silica capillary column (30 m × 0.3 mm i.d.), column temp. 275°. $RR_t$ on HPLC and GC expressed relative to cholesterol (cholest-5-en-3$\beta$-ol). IR spectra were recorded in KBr discs. EI-MS (70 eV) and CI-MS (100 eV, *iso*butane): probe. $^1$H NMR spectra (400 MHz) were determined in $CDCl_3$ with TMS as int. standard. $^1$H NMR signal assignments were performed by comparison of the spectral data with those of related compounds [5]. Acetalization of **15** and **20** was carried out as described previously [5, 8]. The source and extraction of Carthami Flos (366 g) and separation of the alkanediol fr. (312 mg; 0.45% of MeOH extract) were as described in our previous paper [5]. Isolation of individual alkanediols from this fr. was achieved by HPLC. Composition of the alkanediol fr. was determined from HPLC and GC data.

*Hexatriacontane-6,8-diol* (**13**). Mp 88–90°. $RR_t$: 3.70 (HPLC), 4.73 (GC). MS $m/z$ (rel. int.): 520 (1), 502 (2), 467 (2), 449 (2), 431 (2), 423 (2), 405 (1), 145 (11), 127 (10), 109 (17), 101 (7), 83 (53), 55 (100); HR-EI-MS $m/z$: 520.5545 ($C_{36}H_{72}O$ $[M-H_2O]^+$, requires 520.5579), 502.5481 ($C_{36}H_{70}$ $[M-2H_2O]^+$), 467.4771 ($C_{31}H_{63}O_2$, fragment **i**), 423.4522 ($C_{29}H_{59}O$, **ii**), 145.1195 ($C_8H_{17}O_2$, **iii**), 101.0962 ($C_6H_{13}O$, **iv**). $^1$H NMR: $\delta$ 0.88 (3H, *t*, $J$ = 6.9 Hz, $H_3$-36), 0.90 (3H, *t*, $J$ = 6.9 Hz, $H_3$-1), 1.25 (*br s*), 1.45 (1H, *ddd*, $J$ = 11.1, 11.1, 13.6 Hz, H-7), 1.61 (1H, *ddd*, $J$ = 2.2, 2.2, 13.6 Hz, H-7), 2.77 (2H, *m*, 6-OH, 8-OH), 3.85 (2H, *m*, $W_{1/2}$ = 20 Hz, H-6, H-8).

*Triacontane-7,9-diol* (**15**). Mp 59–61°. $RR_t$: 1.22 (HPLC), 1.24 (GC). IR $\nu_{max}$ cm$^{-1}$. 3308 (OH), 2916, 2848, 1471, 1135, 1087, 840, 718. MS $m/z$ (rel. int.): 436 (2), 418 (3), 369 (3), 351 (13), 333 (3), 325 (8), 159 (40), 141 (27), 123 (37), 115 (20), 112 (41), 97 (56), 43 (100); HR-EI-MS $m/z$: 436.4639 ($C_{30}H_{60}O$ $[M-H_2O]^+$, requires 436.3840), 418.4506 ($C_{30}H_{58}$ $[M-2H_2O]^+$), 369.3680 ($C_{24}H_{49}O_2$, fragment **i**), 325.3472 ($C_{22}H_{45}O$, **ii**), 159.1375 ($C_9H_{19}O_2$, **iii**), 115.1123 ($C_7H_{15}O$, **iv**). HR-CI-MS $m/z$: 455.4824 ($C_{30}H_{63}O_2$ $[M+H]^+$ requires 455.4824), 437.4656 ($C_{30}H_{61}O$ $[M+H-H_2O]^+$), 419.4540 ($C_{30}H_{59}$ $[M+H-2H_2O]^+$). $^1$H NMR: $\delta$ 0.88 (3H, *t*, $J$ = 7.1 Hz, $H_3$-30), 0.90 (3H, *t*, $J$ = 6.9 Hz, $H_3$-1), 1.25 (*br s*), 1.45 (1H, *ddd*, $J$ = 10.0, 11.1, 14.6 Hz, H-8), 1.61 (1H, *ddd*, $J$ = 2.2, 2.2, 14.6 Hz, H-8), 2.77 (2H, *m*, 7-OH, 9-OH), 3.85 (2H, *m*, $W_{1/2}$ = 20 Hz, H-7, H-9). $^1$H NMR spectra of other alkane-7,9-diols described

below (**14**, **16–18**) were essentially the same as that of **15**.

*Triacontane*-7,9-*diol acetal* (**24**). Mp 67–69°. $RR_t$: 4.36 (HPLC), 0.73 (GC). Ms *m/z* (rel. int.): 480 (2), 465 (29), 418 (3), 395 (3), 351 (12), 333 (3), 325 (6), 322 (10), 185 (30), 141 (31), 123 (26), 43 (100); HR-EI-MS *m/z*: 480.4885 ($C_{32}H_{64}O_2$ [M]$^+$, requires 480.4902), 465.4662 ($C_{31}H_{61}O_2$ [M−Me]$^+$), 418.4495 ($C_{30}H_{58}$ [M−$C_2H_6O_2$ {62, acetal}]$^+$), 395.3876 ($C_{26}H_{51}O_2$, fragment **i**), 333.3518 ($C_{24}H_{45}$, *i*−62), 185.1555 ($C_{11}H_{21}O_2$, **iii**), 123.1183 ($C_9H_{15}$, **iii**−62). $^1$H NMR: $\delta$ 0.88 (6H, *t*, $J$ = 7.1 Hz, $H_3$-1, $H_3$-30), 1.20 (1H, *ddd*, $J$ = 11.3, 11.3, 13.3 Hz, ax. $H_A$-8), 1.25 (*br s*), 1.33 (3H, *d*, $J$ = 5.2 Hz, $H_3$-2′), 1.50 (1H, *ddd*, $J$ = 2.3, 2.3, 13.3 Hz, eq. $H_B$-8), 3.55 (2H, *m*, $W_{1/2}$ = 22 Hz, H-7, H-9), 4.67 (1H, *q*, $J$ = 5.2 Hz, H-1′).

*Octacosane*-7,9-*diol* (**14**). Mp 56–58°. $RR_t$: 0.84 (HPLC), 0.78 (GC). MS *m/z* (rel. int.): 408 (1), 390 (2), 341 (2), 323 (10), 305 (2), 297 (6), 279 (2), 159 (30), 141 (24), 123 (35), 115 (18), 112 (33), 97 (48), 43 (100).; HR-EI-MS *m/z*: 408.4310 ($C_{28}H_{56}O$ [M−$H_2O$]$^+$, requires 408.4327), 390.4071 ($C_{28}H_{54}$ [M−2$H_2O$]$^+$), 341.3557 ($C_{22}H_{45}O_2$, fragment **i**), 297.3140 ($C_{20}H_{41}O$, **ii**), 159.1362 ($C_9H_{19}O_2$, **iii**), 115.1118 ($C_7H_{15}O$, **iv**).

*Dotriacontane*-7,9-*diol* (**16**). Mp 77–80°. $RR_t$: 1.76 (HPLC), 1.95 (GC). MS *m/z* (rel. int.): 464 (1), 446 (2), 397 (2), 379 (10), 361 (2), 353 (5), 159 (34), 141 (23), 123 (29), 115 (19), 112 (33), 97 (51), 43 (100),; HR-EI-MS *m/z*: 464.4942 ($C_{32}H_{64}O$ [M−$H_2O$]$^+$, requires 464.4953), 446.4836 ($C_{32}H_{628}$ [M−2$H_2O$]$^+$), 397.4079 ($C_{26}H_{53}O_2$, fragment **i**), 353.3807 ($C_{24}H_{49}O$, **ii**), 159.1386 ($C_9H_{19}O_2$, **iii**), 115.1102 ($C_7H_{15}O$, **iv**).

*Tetratriacontane*-7,9-*diol* (**17**). Mp 74–78°. $RR_t$: 2.52 (HPLC), 3.06 (GC). MS *m/z* (rel. int.): 492 (1), 474 (3), 425 (1), 407 (9), 389 (2), 381 (5), 159 (36), 141 (24), 123 (30), 115 (21), 112 (35), 97 (52), 43 (100); HR-EI-MS *m/z*: 492.5239 ($C_{34}H_{68}O$ [M−$H_2O$]$^+$, requires 492.5212), 474.5179 ($C_{34}H_{66}$ [M−2$H_2O$]$^+$), 425.4344 ($C_{28}H_{57}O_2$, fragment **i**), 381.4143 ($C_{26}H_{53}O$, **ii**), 159.1362 ($C_9H_{19}O_2$, **iii**), 115.1106 ($C_7H_{15}O$, **iv**).

*Hexatriacontane*-7,9-*diol* (**18**). Mp 81–84°. $RR_t$: 3.54 (HPLC), 4.73 (GC). MS *m/z* (rel. int.): 520 (1), 502 (1), 453 (2), 435 (3), 417 (1), 409 (2), 391 (2), 159 (11), 141 (8), 123 (17), 115 (11), 112 (20), 97 (36), 43 (100); HR-EI-MS *m/z*: 520.5542 ($C_{36}H_{72}O$ [M−$H_2O$]$^+$, requires 520.5578), 502.5541 ($C_{36}H_{70}$ [M−2$H_2O$]$^+$), 453.3754 ($C_{31}H_{59}O_2$, fragment **i**), 409.4431 ($C_{28}H_{57}O$, **ii**), 159.1356 ($C_9H_{19}O_2$, **iii**), 115.1125 ($C_7H_{15}O$, **iv**).

*Nonacosane*-8,10-*diol* (**20**). Mp 52–55°C. $RR_t$: 0.99 (HPLC), 0.98 (GC). IR $\nu_{max}$ cm$^{-1}$3310 (OH), 2913, 2849, 1471, 1136, 1092, 842, 719. MS *m/z* (rel. int.): 422 (7), 404 (12), 341 (21), 323 (18), 305 (3), 297 (11), 295 (7), 294 (8), 279 (1), 173 (43), 155 (32), 137 (28), 129 (25), 126 (43), 111 (26), 43 (100); HR-EI-MS *m/z*: 422.4503 ($C_{29}H_{58}O$ [M−$H_2O$]$^+$, requires 422.4485), 404.4400 ($C_{29}H_{56}$ [M−2$H_2O$]$^+$), 341.3423 ($C_{22}H_{45}O_2$, fragment **i**), 297.3184 ($C_{20}H_{41}O$, **ii**), 173.1559 ($C_{10}H_{21}O_2$, **iii**), 129.1267 ($C_8H_{17}O$, **iv**). HR-CI-MS *m/z*: 441.4652 ($C_{29}H_{61}O_2$ [M+H]$^+$, requires 441.4668), 423.4542 ($C_{29}H_{59}O$ [M+H−$H_2O$]$^+$), 405.4444 ($C_{29}H_{57}$ [M+H−2$H_2O$]$^+$). $^1$H NMR: $\delta$ 0.88 (6H, *t*, $J$ = 6.0 Hz, $H_3$-1, $H_3$-29), 1.25 (*br s*), 1.45 (1H, *ddd*, $J$ = 10.4, 11.3, 14.6 Hz, H-9), 1.61 (1H, *ddd*, $J$ = 2.2, 2.2, 14.6 Hz, H-9), 2.75 (2H, *m*, 8-OH, 10-OH), 3.84 (2H, *m*, $W_{1/2}$ = 23 Hz, H-8, H-10). $^1$H NMR spectra of other alkane-8,10-diols described below (**19**, **21–23**) were essentially the same as that of **20**.

*Nonacosane*-8,10-*diol acetal* (**25**). Mp 65–67°. $RR_t$: 3.63 (HPLC), 0.57 (GC). MS *m/z* (rel. int.): 466 (1), 451 (24), 404 (2), 367 (3), 323 (10), 305 (1), 294 (7), 199 (19), 155 (23), 137 (12), 129 (8), 126 (37), 43 (100); HR-EI-MS *m/z*: 466.4709 ($C_{31}H_{62}O_2$ [M]$^+$, requires 466.4745), 451.4470 ($C_{30}H_{59}O_2$ [M−Me]$^+$), 404.4374 ($C_{29}H_{56}$ [M−$C_2H_6O_2$ {62, acetal}]$^+$), 367.3588 ($C_{24}H_{47}O_2$, fragment **i**), 305.3189 ($C_{22}H_{41}$, **i**−62), 199.1686 ($C_{12}H_{23}O_2$, **iii**), 137.1301 ($C_{10}H_{17}$, **iii**−62). $^1$H NMR: $\delta$ 0.88 (6H, *t*, $J$ = 7.1 Hz, $H_3$-1, $H_3$-29), 1.19 (1H, *ddd*, $J$ = 11.3, 11.3, 13.2 Hz, ax. $H_A$-9), 1.25 (*br s*), 1.33 (3H, *d*, $J$ = 4.9 Hz, $H_3$-2′), 1.50 (1H, *ddd*, $J$ = 2.2, 2.2, 13.2 Hz, eq. $H_B$-9), 3.54 (2H, *m*, $W_{1/2}$ = 22 Hz, H-8, H-10), 4.67 (1H, *q*, $J$ = 5.2 Hz, H-1′).

*Heptacosane*-8,10-*diol* (**19**). Mp 54–56°. $RR_t$: 0.68 (HPLC), 0.67 (GC). MS *m/z* (rel. int.): 394 (1), 376 (2), 313 (3), 295 (12), 277 (3), 269 (8), 173 (30), 155 (24), 137 (25), 129 (20), 126 (31), 111 (23), 43 (100); HR-EI-MS *m/z*: 394.4104 ($C_{27}H_{54}O$ [M−$H_2O$]$^+$, requires 394.4171), 376.4084 ($C_{27}H_{52}$ [M−2$H_2O$]$^+$), 313.2998 ($C_{20}H_{41}O_2$, fragment **i**), 269.2819 ($C_{18}H_{37}O$, **ii**), 173.1517 ($C_{10}H_{21}O_2$, **iii**), 129.1233 ($C_8H_{17}O$, **iv**).

*Hentriacontane*-8,10-*diol* (**21**). Mp 64–66°. $RR_t$: 1.43 (HPLC), 1.55 (GC). MS *m/z* (rel. int.): 450 (1), 432 (1), 369 (2), 351 (10), 333 (2), 325 (5), 173 (27), 155 (21), 137 (21), 129 (16), 126 (27), 111 (23), 43 (100); HR-EI-MS *m/z*: 450.4761 ($C_{31}H_{62}O$ [M−$H_2O$]$^+$, requires 450.4797), 432.4664 ($C_{31}H_{60}$ [M−2$H_2O$]$^+$), 369.3745 ($C_{24}H_{49}O_2$, fragment **i**), 325.3451 ($C_{22}H_{45}O$, **ii**), 173.1539 ($C_{10}H_{21}O_2$, **iii**), 129.1265 ($C_8H_{17}O$, **iv**).

*Tritriacontane*-8,10-*diol* (**22**). Mp 79–82°. $RR_t$: 2.06 (HPLC), 2.44 (GC). MS *m/z* (rel. int.): 478 (1), 460 (3), 397 (10), 379 (11), 361 (2), 353 (7), 335 (1), 173 (33), 155 (26), 137 (25), 129 (20), 111 (29), 43 (100); HR-EI-MS *m/z*: 478.5076 ($C_{33}H_{66}O$ [M−$H_2O$]$^+$, requires 478.5110), 460. 4953 ($C_{33}H_{64}$ [M−2$H_2O$]$^+$), 397.4049 ($C_{26}H_{66}O_2$, fragment **i**), 353.3800 ($C_{24}H_{49}O$, **ii**), 173.1528 ($C_{10}H_{21}O_2$, **iii**), 129.1261 ($C_8H_{17}O$, **iv**).

*Pentatriacontane*-8,10-*diol* (**23**). Mp 75–78°C. $RR_t$: 2.95 (HPLC), 3.83 (GC). MS *m/z* (rel. int.): 506 (1), 488 (2) 425 (1), 407 (3), 389 (1), 381 (1), 173 (33), 155 (25), 137 (23), 129 (19), 126 (33), 111 (25), 43 (100); HR-EI-MS *m/z*: 506.5490 ($C_{35}H_{70}O$ [M−$H_2O$]$^+$, requires 506.5423), 488.5356 ($C_{35}H_{68}$ [M−2$H_2O$]$^+$), 425.4346 ($C_{28}H_{57}O_2$, fragment **i**), 381.4133 ($C_{26}H_{53}O$, **ii**), 173.1530 ($C_{10}H_{21}O_2$, **iii**), 129.1267 ($C_8H_{17}O$, **iv**).

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