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Synthesis of enantiomerically pure 2,9-dicycloalkyl substituted perhydro hexaazadibenzotetracenes

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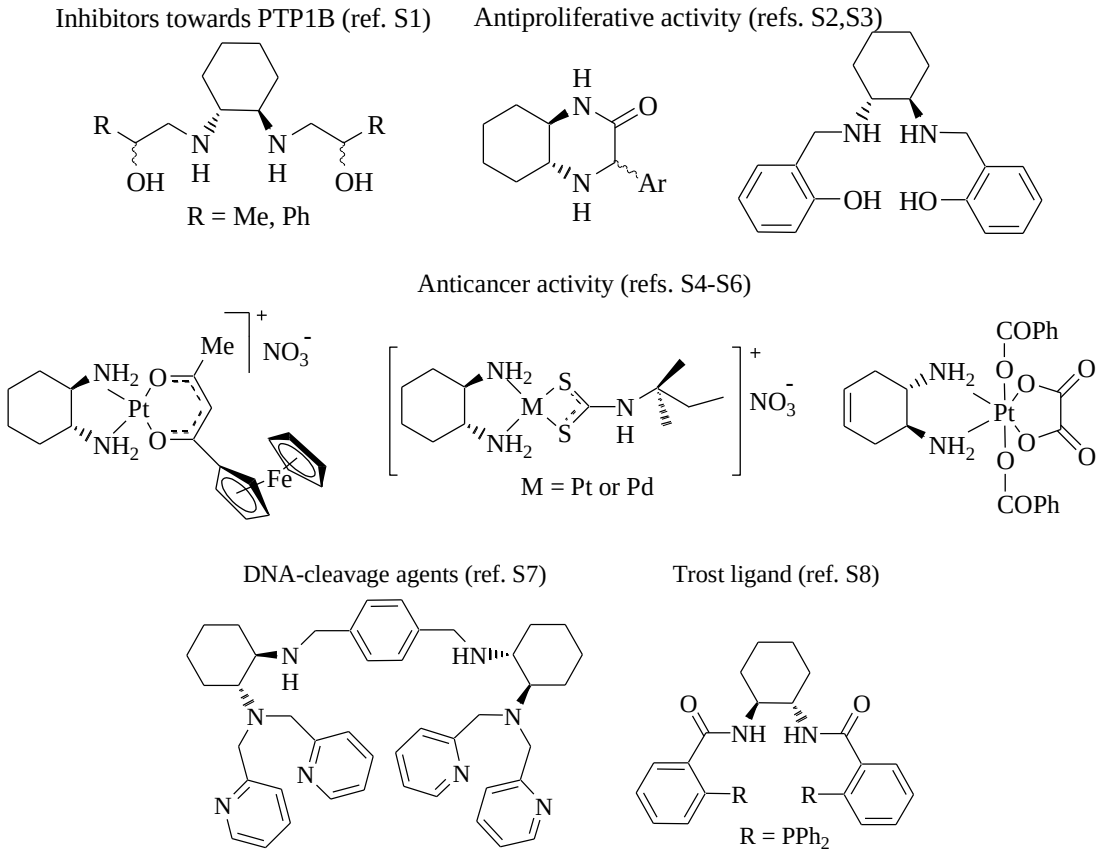
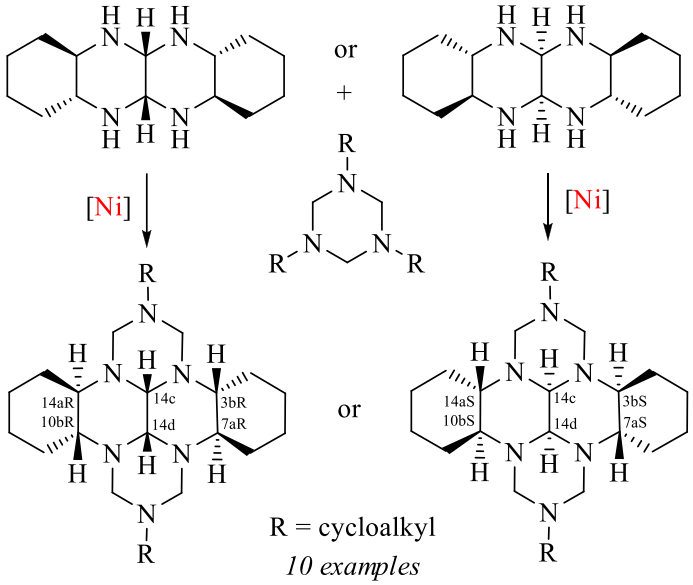


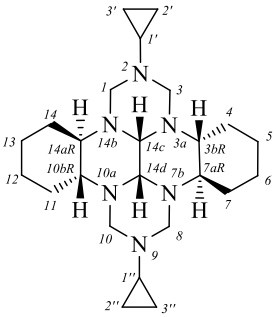
Figure S1 Application of *trans*-1,2-diaminocyclohexane derivatives.

General information. The ^{1}H and ^{13}C NMR spectra, including two dimensional homo- (COSY) and heteronuclear (HSQC, HMBC) spectra, were recorded on a Bruker Avance 500 spectrometer (500.17 MHz for ^{1}H and 125.78 MHz for ^{13}C) or a Bruker Avance 400 spectrometer (400.13 MHz for ^{1}H and 100.61 MHz for ^{13}C) according to standard Bruker protocols using $CDCl_3$ as solvent and TMS as internal standard. The high-resolution mass spectra were recorded on a Bruker MaXis Impact instrument (TOF mass analyzer, electrospray ionization). The melting points were measured on a PHMK 80/2617 melting point apparatus. Specific rotations were measured on Perkin-Elmer-341 polarimeter. Commercially available reagents used in this work were purchased from Sigma–Aldrich and Acros Organics.

Recyclization of 1,3,5-tricycloalkyl-1,3,5-triazinanes with (*R*,*R*,*R*,*R*)-/(*S*,*S*,*S*,*S*)-perhydro-5,6,11,12-tetraazatetracenes (2, 2′) (general method). A round-bottomed flask equipped with a magnetic stirrer bar was charged with MeOH (5 mL), $NiCl_2 \cdot 6H_2O$ (0.012 g, 0.05 mmol), and the appropriate 1,3,5-tricycloalkyl-1,3,5-triazinane (2.00 mmol), prepared *in situ* by the reported procedure [S9,S10], and the mixture was stirred at r.t. for 30 min. (*R*,*R*,*R*,*R*)-/(*S*,*S*,*S*,*S*)-Perhydro-5,6,11,12-tetraazatetracenes (**2** or **2′**; 1.00 mmol) in MeOH (5 mL), prepared by the reaction of (*R*,*R*)-(–)/(*S*,*S*)-(+)-1,2-diminocyclohexanes (0.23 g, 2.00 mmol) with 40 wt% aq glyoxal (0.14 g, 1.00 mmol) was added, and the mixture was stirred at 20 °C for 3 h, then concentrated. The white precipitate was filtered and washed twice with MeOH (5 mL). Compounds **3a–e** and **3′a–e** were obtained as white powders. The final products **3a–e** and **3′a–e** were identified by spectroscopic methods.

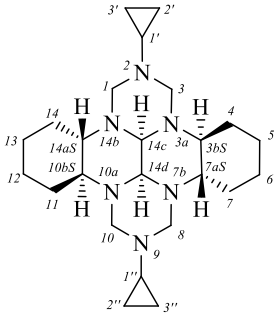


Analytical data for compounds 3a–e, 3′a–e

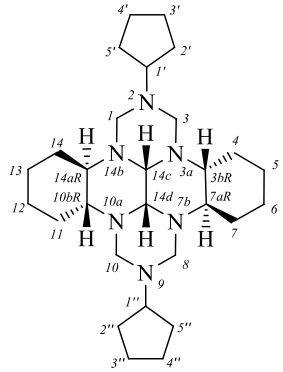


(3b*R*,7a*R*,10b*R*,14a*R*,*cis*-14c,14d)-2,9-Dicyclopropyl-octadecahydro-1*H*,8*H*-2,3a,7b,9,10a,14b-hexaazadibenzo[*fg*,*op*]tetracene (3a). White powder; yield 0.19 g (46%); mp 246–248 °C; R_f 0.75 (MeOH), $[\alpha]_D$ –28.9 (22 °C, *c* 0.057, $CHCl_3$). ^{1}H NMR (500.17 MHz, $CDCl_3$): δ = 0.29–0.34 and 0.40–0.49 (m, 8H, CH_2, H-2′,2″,3′,3″), 0.97 (qd, $^2J_{ab}$ = 12.5 Hz, ^3J = 3.5 Hz, 2H, CH_2, H_a-7,14), 1.17–1.32 (m, 6H, CH_2, H_a-4,5,6,11,12,13), 1.60–1.64 (m, 2H, CH, H-1′,1″), 1.69–1.79 (m, 4H, CH_2, H_b-4,5,11,12; 2H, CH, H-7a,14a), 1.98–2.07 (m, 4H, CH_2, H_b-6,7,13,14), 2.56 (d, $^2J_{ab}$ = 8.5 Hz, 2H, CH_2, H_a-1,8), 3.21 (d, $^2J_{ab}$ = 12 Hz, 2H, CH_2, H_a-3,10), 3.22–3.25 (m, 2H, CH, H-3b,10b), 3.32 (br. s, 2H, CH, H-14c,14d), 4.19 (d, $^2J_{ba}$ = 12 Hz, 2H, CH_2, H_b-3,10), 4.25 (d, $^2J_{ba}$ = 8 Hz, 2H, CH_2, H_b-1,8). ^{13}C NMR (125.78 MHz, $CDCl_3$): δ = 5.3 and 5.9 (C-2′,2″,3′,3″), 24.4 (C-4,11), 24.6 (C-5,12), 27.1 (C-6,13), 28.2 (C-7,14), 33.1 (C-1′,1″), 55.3

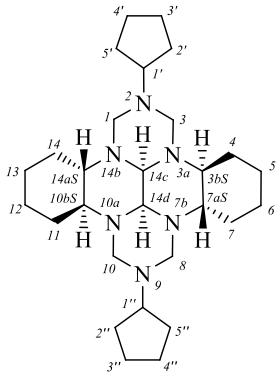
(C-3b,10b), 63.9 (C-7a,14a), 68.7 (C-3,10), 74.4 (C-1,8), 77.7 (C-14c,14d). Anal. Calcd for $C_{24}H_{40}N_6$: C, 69.86; H, 9.77; N, 20.37. Found: C, 69.72; H, 9.70; N, 20.23.



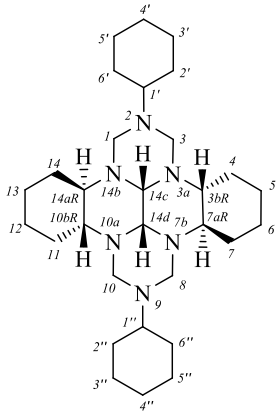
(3b*S*,7a*S*,10b*S*,14a*S*,*cis*-14c,14d)-2,9-Dicyclopropyl-octadecahydro-1*H*,8*H*-2,3a,7b,9,10a,14b-hexaazadibenzo[*fg*,*op*]tetracene (3′a). White powder; yield 0.18 g (44%); mp 240–242 °C; R_f 0.75 (MeOH), $[\alpha]_D$ +26.2 (22 °C, *c* 0.141, $CHCl_3$). ^{1}H NMR (500.17 MHz, $CDCl_3$): δ = 0.29–0.34 and 0.39–0.49 (m, 8H, CH_2, H-2′,2″,3′,3″), 0.97 (qd, $^2J_{ab}$ = 12 Hz, ^3J = 3 Hz, 2H, CH_2, H_a-7,14), 1.16–1.33 (m, 6H, CH_2, H_a-4,5,6,11,12,13), 1.59–1.64 (m, 2H, CH, H-1′,1″), 1.69–1.79 (m, 4H, CH_2, H_b-4,5,11,12; 2H, CH, H-7a,14a), 1.98–2.07 (m, 4H, CH_2, H_b-6,7,13,14), 2.57 (d, $^2J_{ab}$ = 8.5 Hz, 2H, CH_2, H_a-1,8), 3.21 (d, $^2J_{ab}$ = 12 Hz, 2H, CH_2, H_a-3,10), 3.22–3.25 (m, 2H, CH, H-3b,10b), 3.32 (br. s, 2H, CH, H-14c,14d), 4.19 (d, $^2J_{ba}$ = 12 Hz, 2H, CH_2, H_b-3,10), 4.26 (d, $^2J_{ba}$ = 8.5 Hz, 2H, CH_2, H_b-1,8). ^{13}C NMR (125.78 MHz, $CDCl_3$): δ = 5.3 and 5.9 (C-2′,2″,3′,3″), 24.3 (C-4,11), 24.6 (C-5,12), 27.1 (C-6,13), 28.2 (C-7,14), 33.1 (C-1′,1″), 55.3 (C-3b,10b), 63.9 (C-7a,14a), 68.7 (C-3,10), 74.3 (C-1,8), 77.7 (C-14c,14d). Anal. Calcd for $C_{24}H_{40}N_6$: C, 69.86; H, 9.77; N, 20.37. Found: C, 69.75; H, 9.71; N, 20.23.



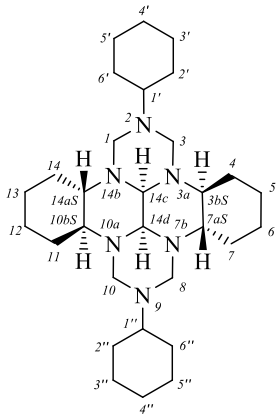
(3b*R*,7a*R*,10b*R*,14a*R*,*cis*-14c,14d)-2,9-Dicyclopenthyl-octadecahydro-1*H*,8*H*-2,3a,7b,9,10a,14b-hexaazadibenzo[*fg*,*op*]tetracene (3b). White powder; yield 0.26 g (55%); mp 228–230 °C; R_f 0.74 (MeOH), $[\alpha]_D$ –41.7 (21°C, *c* 0.145, $CHCl_3$). ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 0.91–1.01 (m, 2H, CH_2, H_a-7,14), 1.16–1.27 (m, 4H, CH_2, H_a-5,6,12,13), 1.33–1.42 (m, 4H, CH_2, H_a-4,11; H_a-2′,2″), 1.46–1.58 (m, 6H, CH_2, H_a-3′,3″,4′,4″5′,5″), 1.64–1.84 (m, 12H, CH_2, H_b-4,5,11,12; H_b-2′,2″,3′,3″,4′,4″5′,5″; 2H, CH, H-7a,14a), 1.96 (d, $^2J_{ba}$ = 10 Hz, 2H, CH_2, H_b-6,13), 2.06 (d, $^2J_{ba}$ = 10.8 Hz, 2H, CH_2, H_b-7,14), 2.28 (d, $^2J_{ab}$ = 7.6 Hz, 2H, CH_2, H_a-1,8), 2.34–2.42 (m, 2H, CH, H-1′,1″), 2.91 (d, $^2J_{ab}$ = 11.6 Hz, 2H, CH_2, H_a-3,10), 3.27 (br. s, 2H, CH, H-14c,14d), 3.31–3.35 (m, 2H, CH, H-3b,10b), 4.21 (d, $^2J_{ba}$ = 11.6 Hz, 2H, CH_2, H_b-3,10), 4.30 (d, $^2J_{ba}$ = 7.6 Hz, 2H, CH_2, H_b-1,8). ^{13}C NMR (100.61 MHz, $CDCl_3$): δ = 23.9 (C-3′,3″,4′,4″), 24.3 (C-4,11), 24.7 (C-5,12), 27.2 (C-6,13), 28.2 (C-7,14), 30.0 and 30.1 (C-2′,2″,5′,5″), 55.1 (C-3b,10b), 62.4 (C-1′,1″), 63.8 (C-7a,14a), 68.5 (C-3,10), 74.2 (C-1,8), 77.5 (C-14c,14d). Anal. Calcd for $C_{28}H_{48}N_6$: C, 71.75; H, 10.32; N, 17.93. Found: C, 71.65; H, 10.24; N, 17.81.



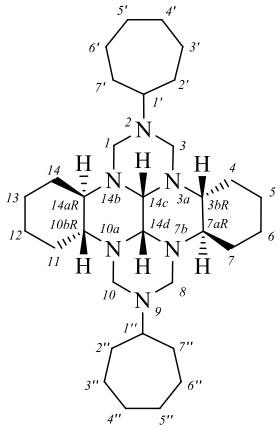
(3b*S*,7a*S*,10b*S*,14a*S*,*cis*-14c,14d)-2,9-Dicyclopenthyl-octadecahydro-1*H*,8*H*-2,3a,7b,9,10a,14b-hexaazadibenzo[*fg*,*op*]tetracene (3′b). White powder; yield 0.27 g (58%); mp 230–232 °C; R_f 0.74 (MeOH), $[\alpha]_D$ +44.1 (21 °C, *c* 0.111, $CHCl_3$). ^{1}H NMR (400.13 MHz, $CDCl_3$): δ = 0.92–1.02 (m, 2H, CH_2, H_a-7,14), 1.16–1.28 (m, 4H, CH_2, H_a-5,6,12,13), 1.32–1.43 (m, 4H, CH_2, H_a-4,11; H_a-2′,2″), 1.44–1.58 (m, 6H, CH_2, H_a-3′,3″,4′,4″5′,5″), 1.63–1.87 (m, 12H, CH_2, H_b-4,5,11,12; H_b-2′,2″,3′,3″,4′,4″5′,5″; 2H, CH, H-7a,14a), 1.96 (d, $^2J_{ba}$ = 10 Hz, 2H, CH_2, H_b-6,13), 2.06 (d, $^2J_{ba}$ = 11.2 Hz, 2H, CH_2, H_b-7,14), 2.28 (d, $^2J_{ab}$ = 8 Hz, 2H, CH_2, H_a-1,8), 2.34–2.42 (m, 2H, CH, H-1′,1″), 2.91 (d, $^2J_{ab}$ = 11.6 Hz, 2H, CH_2, H_a-3,10), 3.27 (br. s, 2H, CH, H-14c,14d), 3.30–3.36 (m, 2H, CH, H-3b,10b), 4.21 (d, $^2J_{ba}$ = 11.2 Hz, 2H, CH_2, H_b-3,10), 4.30 (d, $^2J_{ba}$ = 7.6 Hz, 2H, CH_2, H_b-1,8). ^{13}C NMR (100.61 MHz, $CDCl_3$): δ = 23.9 (C-3′,3″,4′,4″), 24.3 (C-4,11), 24.7 (C-5,12), 27.2 (C-6,13), 28.2 (C-7,14), 30.0 and 30.1 (C-2′,2″,5′,5″), 55.1 (C-3b,10b), 62.4 (C-1′,1″), 63.8 (C-7a,14a), 68.5 (C-3,10), 74.2 (C-1,8), 77.5 (C-14c,14d). Anal. Calcd for $C_{28}H_{48}N_6$: C, 71.75; H, 10.32; N, 17.93. Found: C, 71.68; H, 10.25; N, 17.82.



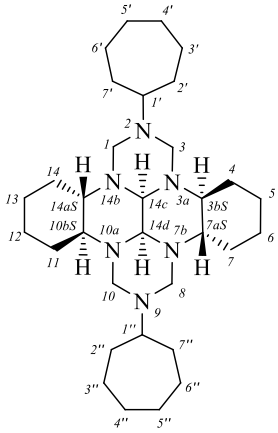
(3b*R*,7a*R*,10b*R*,14a*R*,*cis*-14c,14d)-2,9-Dicyclohexyl-octadecahydro-1*H*,8*H*-2,3a,7b,9,10a,14b-hexaazadibenzo[*fg*,*op*]tetracene (3c). White powder; yield 0.25 g (50%); mp 232–234 °C; R_f 0.72 (MeOH), $[\alpha]_D$ –42.9 (22 °C, *c* 0.49, $CHCl_3$). ^{1}H NMR (500.17 MHz, $CDCl_3$): δ = 0.88–0.98 (m, 2H, CH_2, H_a-7,14), 1.08–1.35 (m, 18H, CH_2, H_a-4,5,6,11,12,13; H_a-2′,2″,3′,3″,5′,5″,6′,6″, H-4′,4″), 1.58–1.63 (m, 2H, CH_2, H_b-5′,5″), 1.69–1.80 (m, 8H, CH_2, H_b-4,5,11,12; H_b-3′,3″,6′,6″; 2H, CH, H-7a,14a), 1.84–1.88 (m, 2H, CH_2, H_b-2′,2″), 1.94–2.03 (m, 4H, CH_2, H_b-6,7,13,14), 2.23–2.27 (m, 2H, CH, H-1′,1″), 2.66 (d, $^2J_{ab}$= 7.5 Hz, 2H, CH_2, H_a-1,8), 3.18 (d, $^2J_{ab}$ = 11.5 Hz, 2H, CH_2, H_a-3,10), 3.24 (br. s, 2H, CH, H-14c,14d), 3.28–3.32 (m, 2H, CH, H-3b,10b), 4.20 (d, $^2J_{ba}$ = 9.5 Hz, 4H, CH_2, H_b-1,3,8,10). ^{13}C NMR (125.78 MHz, $CDCl_3$): δ = 24.4 (C-4,11), 24.7 (C-5,12), 25.7 (C-3′,3″), 26.0 (C-4′,4″), 26.2 (C-5′,5″), 27.2 (C-6,13), 28.0 (C-2′,2″), 28.2 (C-7,14), 30.3 (C-6′,6″), 54.8 (C-3b,10b), 60.4 (C-1′,1″), 63.8 (C-7a,14a), 65.1 (C-3,10), 72.3 (C-1,8), 78.0 (C-14c,14d). Mass-spectrum: Found *m/z* 497.4323 $[M + H]^+$, Calculated for $C_{30}H_{53}N_6^+$ 497.4332.



(3b*S*,7a*S*,10b*S*,14a*S*,*cis*-14c,14d)-2,9-Dicyclohexyl-octadecahydro-1*H*,8*H*-2,3a,7b,9,10a,14b-hexaazadibenzo[*fg*,*op*]tetracene (3′c). White powder; yield 0.24 g (48%); mp 230–232 °C; R_f 0.72 (MeOH), $[\alpha]_D$ +46.9 (22 °C, *c* 0.37, $CHCl_3$). ^{1}H NMR (500.17 MHz, $CDCl_3$): δ = 0.90–0.98 (m, 2H, CH_2, H_a-7,14), 1.10–1.35 (m, 18H, CH_2, H_a-4,5,6,11,12,13; H_a-2′,2″,3′,3″,5′,5″,6′,6″, H-4′,4″), 1.58–1.62 (m, 2H, CH_2, H_b-5′,5″), 1.71–1.80 (m, 8H, CH_2, H_b-4,5,11,12; H_b-3′,3″,6′,6″; 2H, CH, H-7a,14a), 1.83–1.87 (m, 2H, CH_2, H_b-2′,2″), 1.92–2.03 (m, 4H, CH_2, H_b-6,7,13,14), 2.24–2.28 (m, 2H, CH, H-1′,1″), 2.66 (d, $^2J_{ab}$= 6 Hz, 2H, CH_2, H_a-1,8), 3.16 (d, $^2J_{ab}$ = 11 Hz, 2H, CH_2, H_a-3,10), 3.24 (br. s, 2H, CH, H-14c,14d), 3.28–3.31 (m, 2H, CH, H-3b,10b), 4.20 (d, $^2J_{ba}$ = 8 Hz, 4H, CH_2, H_b-1,3,8,10). ^{13}C NMR (125.78 MHz, $CDCl_3$): δ = 24.4 (C-4,11), 24.7 (C-5,12), 25.7 (C-3′,3″), 26.0 (C-4′,4″), 26.2 (C-5′,5″), 27.2 (C-6,13), 28.0 (C-2′,2″), 28.2 (C-7,14), 30.3 (C-6′,6″), 54.8 (C-3b,10b), 60.4 (C-1′,1″), 63.8 (C-7a,14a), 65.1 (C-3,10), 72.3 (C-1,8), 78.0 (C-14c,14d). Mass-spectrum: Found *m/z* 497.4321 $[M + H]^+$, Calculated for $C_{30}H_{53}N_6^+$ 497.4332.



(3b*R*,7a*R*,10b*R*,14a*R*,*cis*-14c,14d)-2,9-Dicycloheptyl-octadecahydro-1*H*,8*H*-2,3a,7b,9,10a,14b-hexaazadibenzo[*fg*,*op*]tetracene (3d). White powder; yield 0.28 g (53%); mp 216–218 °C; R_f 0.73 (MeOH), $[\alpha]_D$ –51.8 (23 °C, *c* 0.54, $CHCl_3$). ^{1}H NMR (500.17 MHz, $CDCl_3$): δ = 0.93 (qd, $^2J_{ab}$ = 12.5 Hz, ^3J = 3 Hz, 2H, CH_2, H_a-7,14), 1.15–1.30 (m, 4H, CH_2, H_a-5,6,12,13), 1.32–1.44 (m, 8H, CH_2, H_a-4,11; H_a-2′,2″,3′,3″,6′,6″), 1.46–1.58 (m, 10H, CH_2, H-4′,4″,5′,5″; H_a-7′,7″) 1.63–1.68 (m, 4H, CH_2, H_b-3′,3″,6′,6″), 1.70–1.78 (m, 4H, CH_2, H_b-4,5,11,12; 2H, CH, H-7a,14a), 1.80–1.86 (m, 4H, CH_2, H_b-2′,2″,7′,7″), 1.96 (d, $^2J_{ba}$ = 11.5 Hz, 2H, CH_2, H_b-6,13), 2.03 (d, $^2J_{ba}$ = 11.5 Hz, 2H, CH_2, H_b-7,14), 2.48–2.51 (m, 2H, CH, H-1′,1″), 2.66 (d, $^2J_{ab}$ = 7.5 Hz, 2H, CH_2, H_a-1,8), 3.20 (br. s, 2H, CH_2, H_a-3,10), 3.22 (br. s, 2H, CH, H-14c,14d), 3.27–3.32 (m, 2H, CH, H-3b,10b), 4.11 (d, $^2J_{ba}$ = 12 Hz, 2H, CH_2, H_b-3,10), 4.12 (br. s, 2H, CH_2, H_b-1,8). ^{13}C NMR (125.78 MHz, $CDCl_3$): δ = 24.4 (C-4,11), 24.8 (C-5,12), 25.4 and 25.5 (C-3′,3″,6′,6″), 27.2 (C-6,13), 27.8 and 27.9 (C-4′,4″,5′,5″), 28.3 (C-7,14), 28.4 (C-2′,2″), 32.4 (C-7′,7″), 54.7 (C-3b,10b), 62.4 (C-1′,1″), 64.0 (C-7a,14a), 64.4 (C-3,10), 72.5 (C-1,8), 78.0 (C-14c,14d). Anal. Calcd for $C_{32}H_{56}N_6$: C, 73.23; H, 10.76; N, 16.01. Found: C, 73.12; H, 10.67; N, 15.90.



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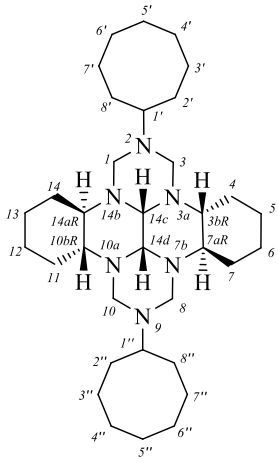

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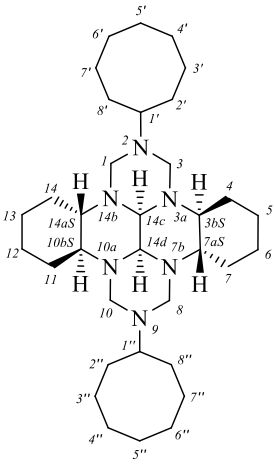

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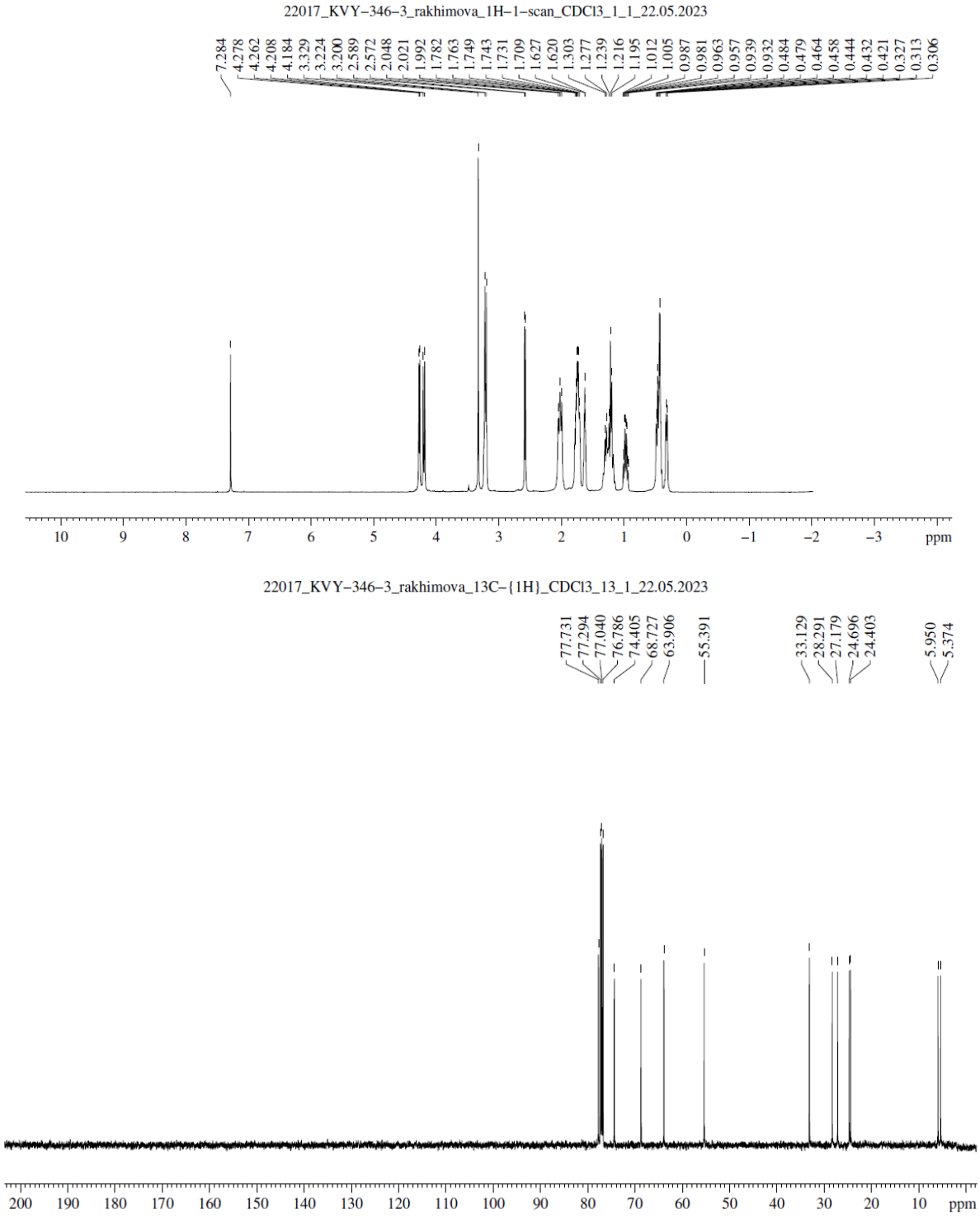
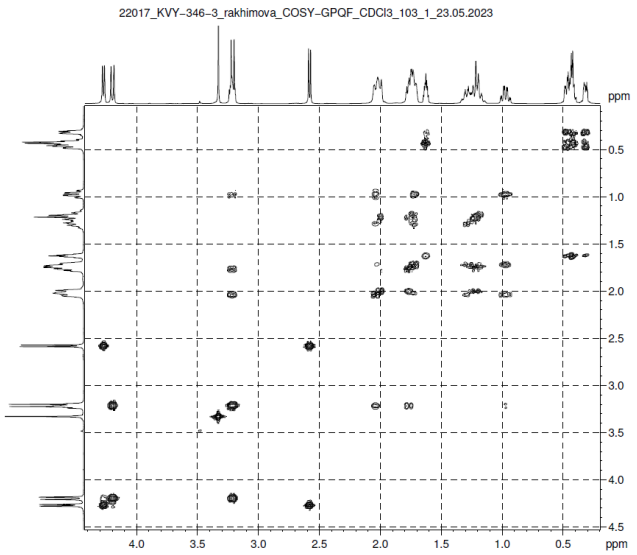
NMR spectra of compounds 3a-e, 3′a–e

Figure S2. NMR spectra of compound **3a**

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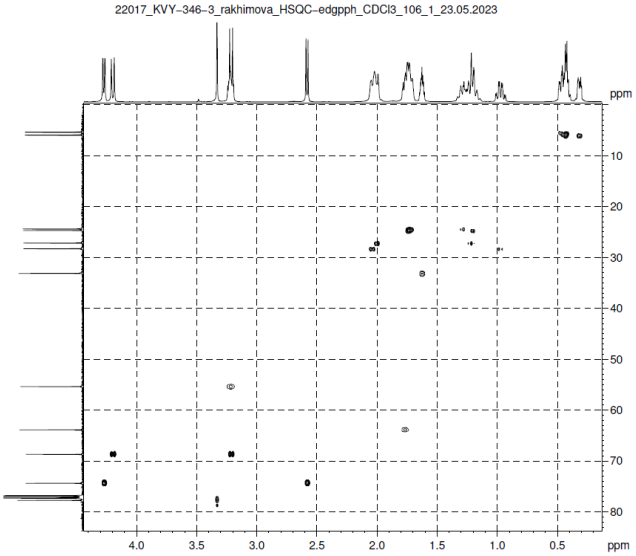
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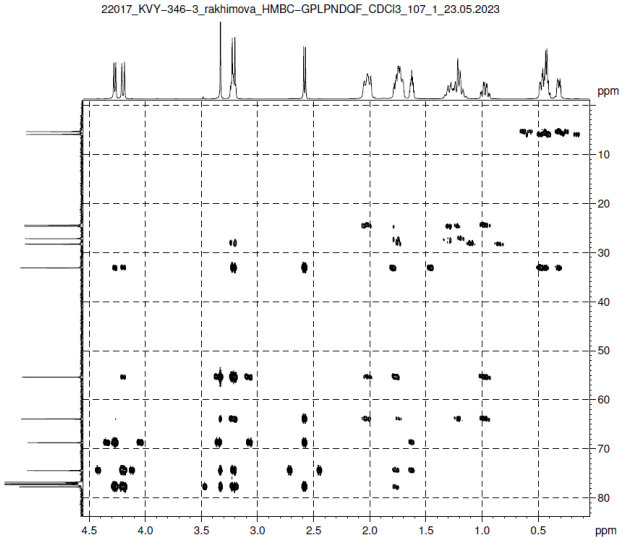
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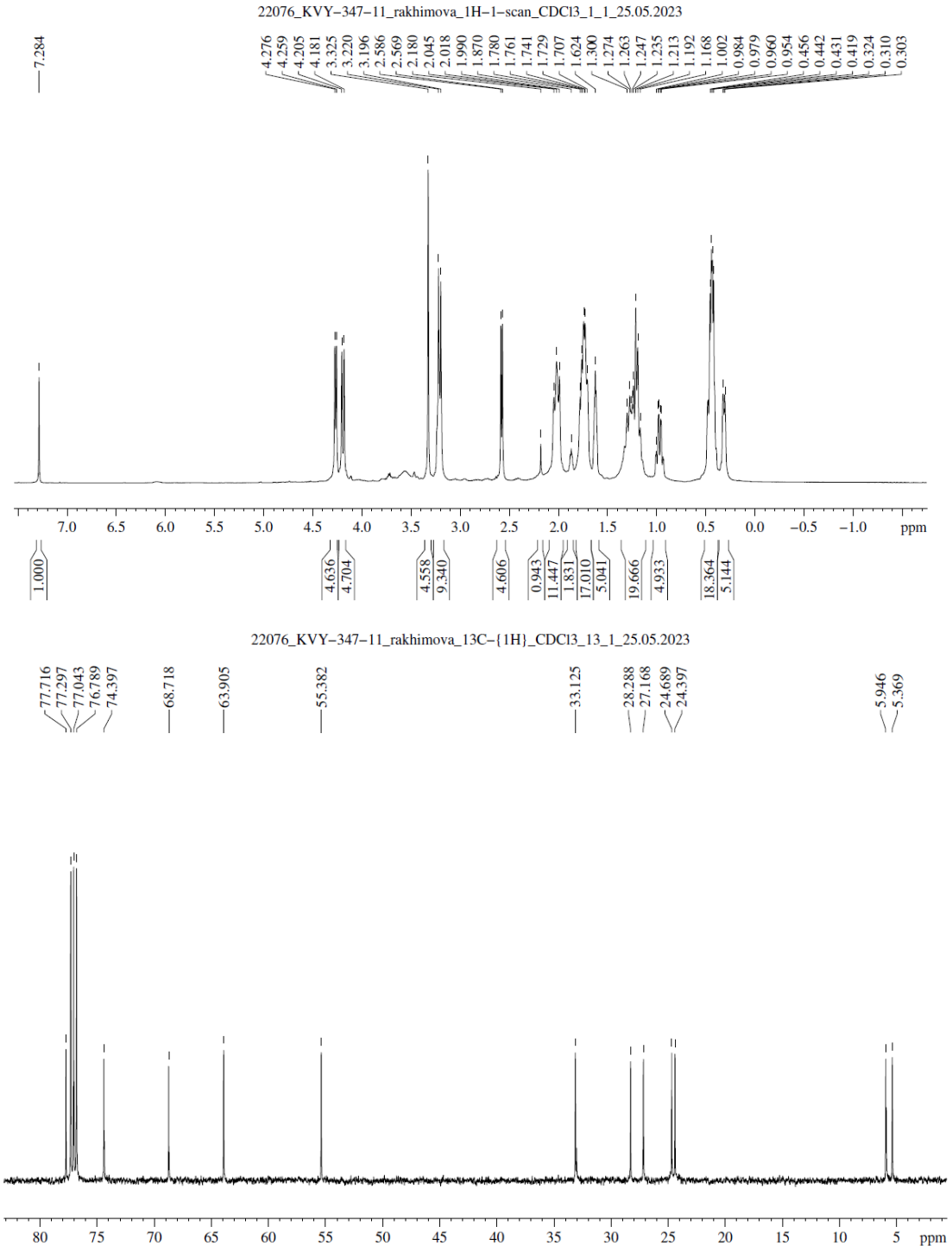
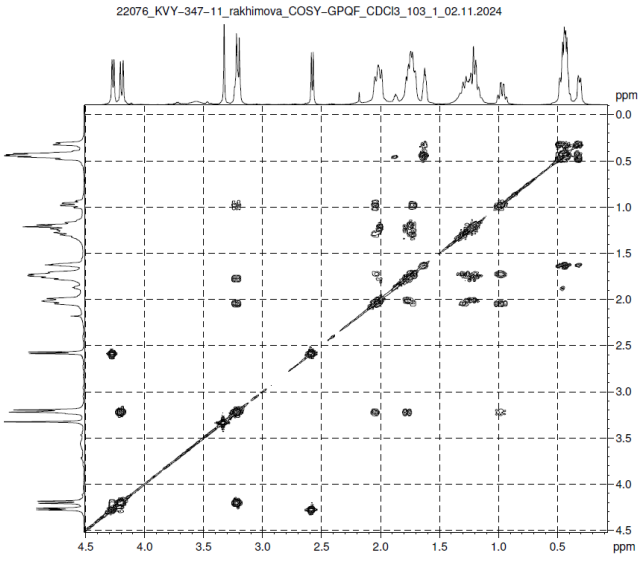
Figure S3. NMR spectra of compound **3′a**

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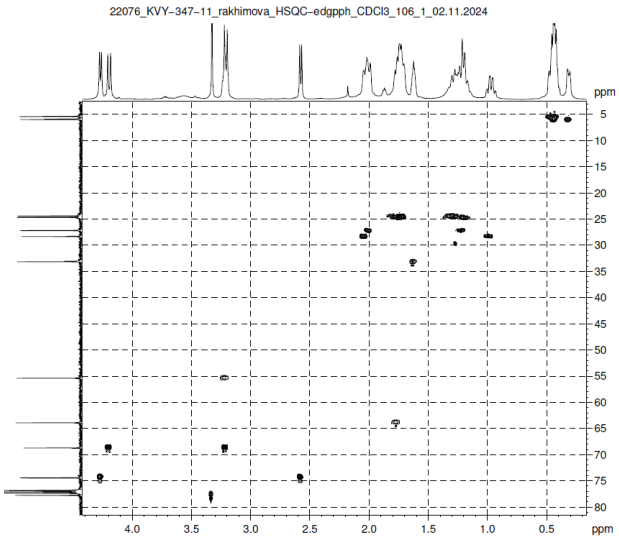
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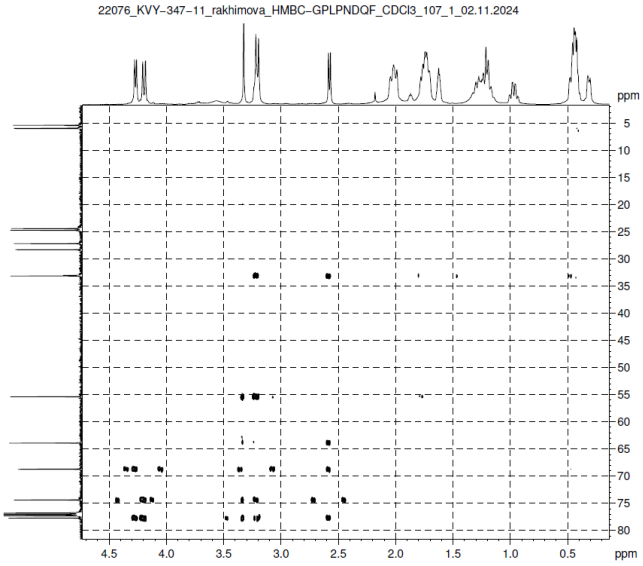
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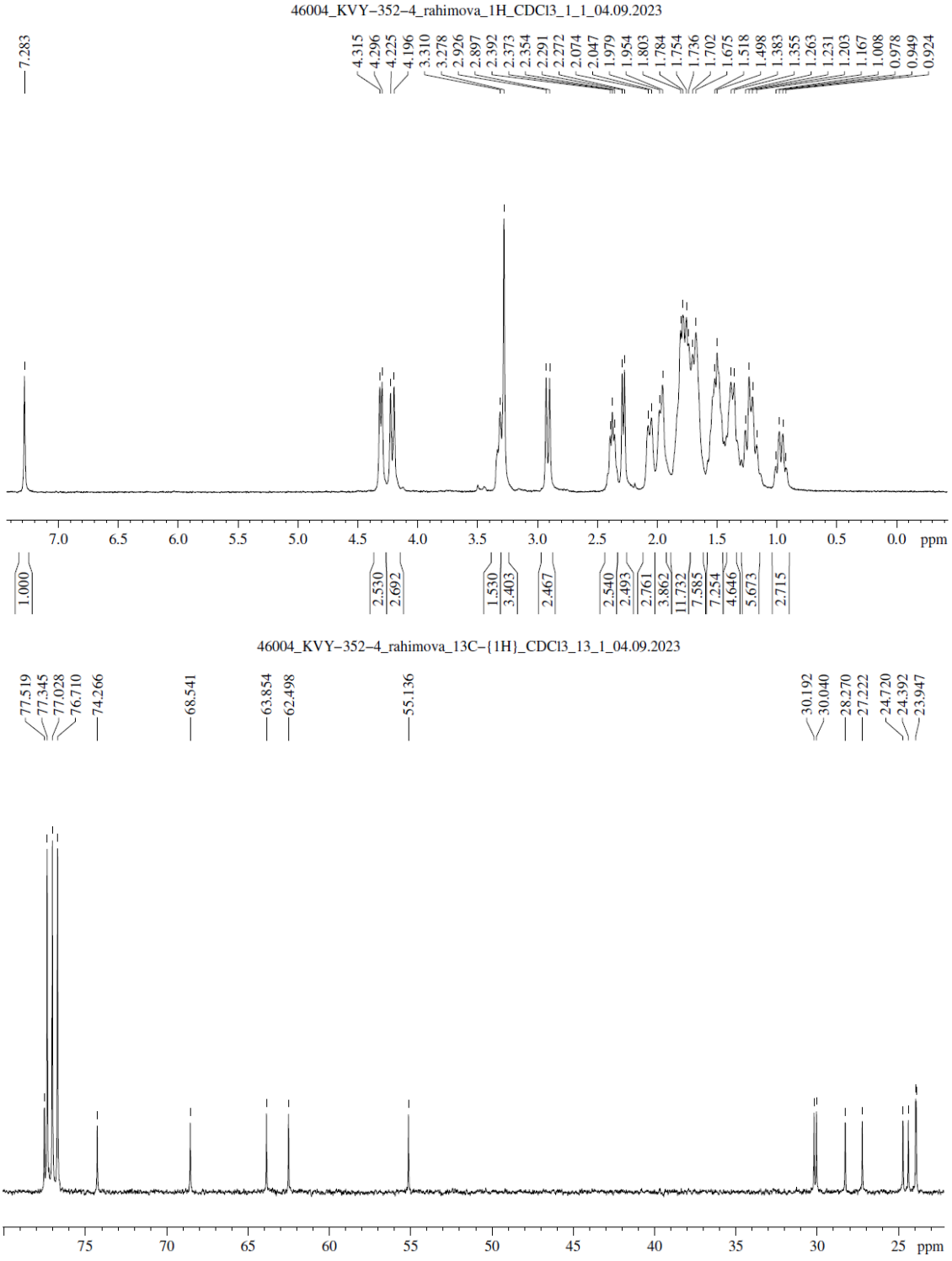
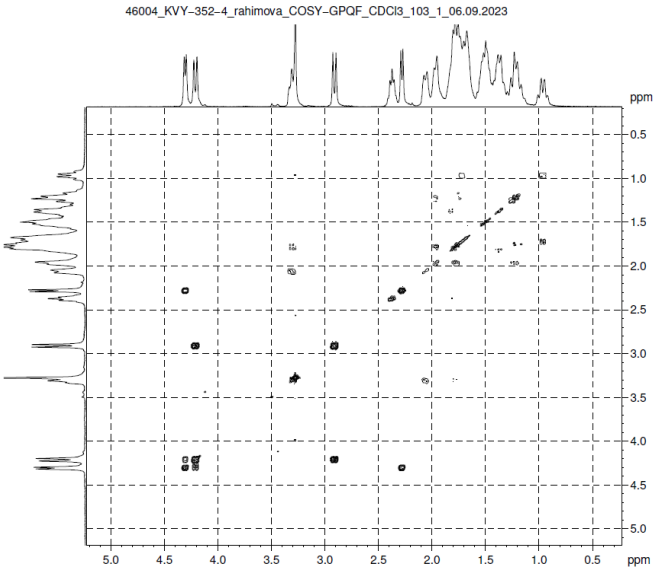
Figure S4. NMR spectra of compound **3b**

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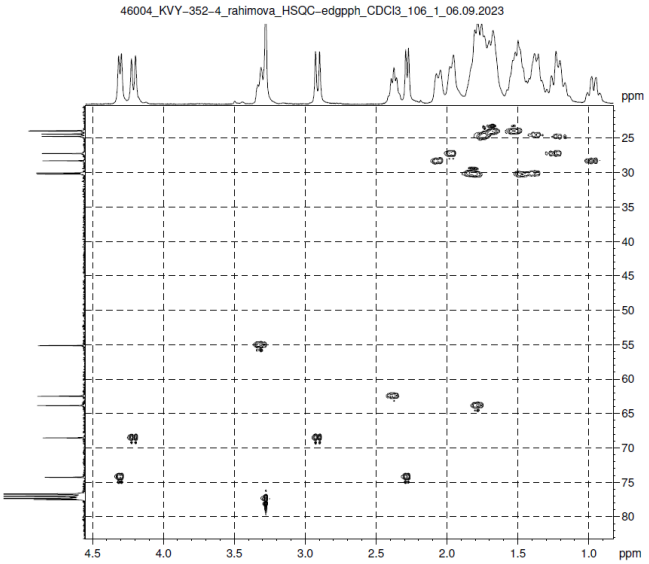
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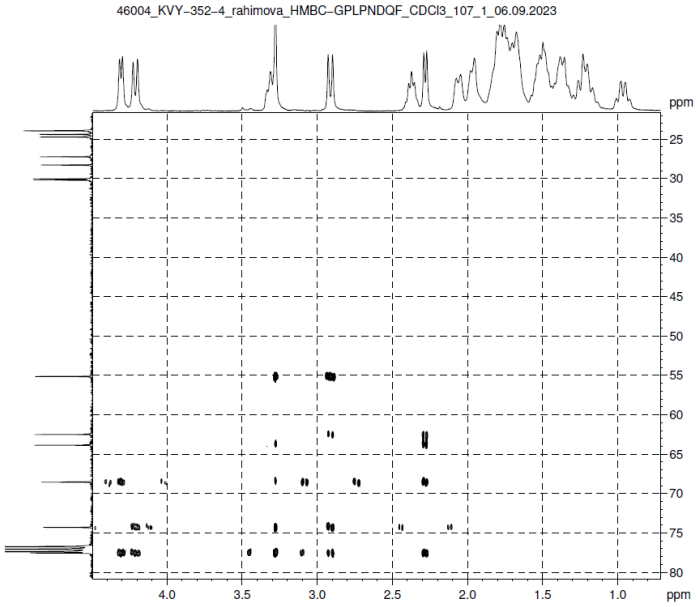
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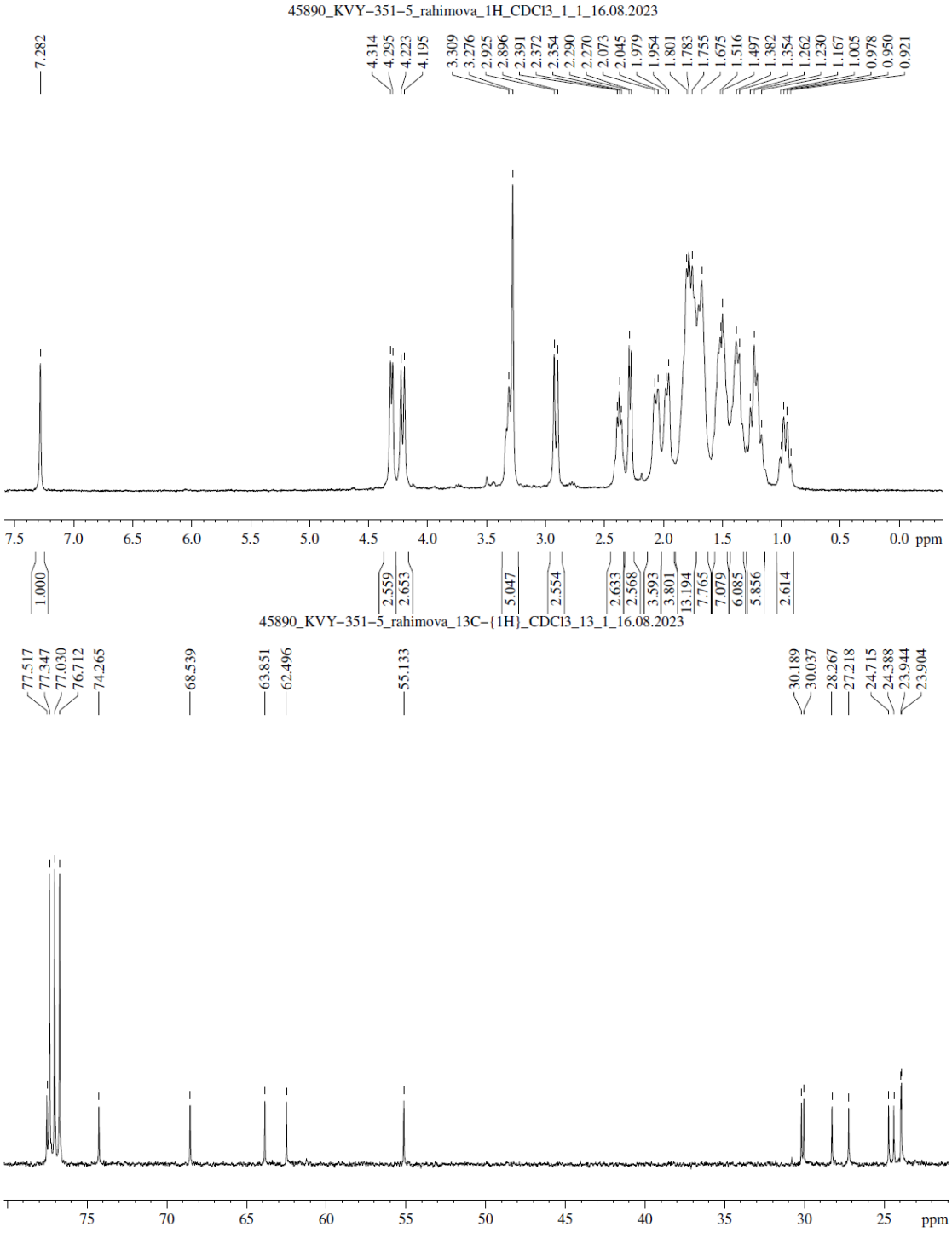
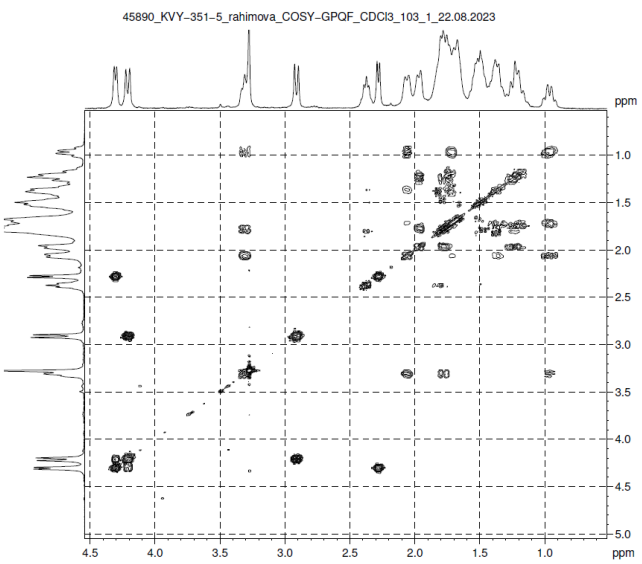
Figure S5. NMR spectra of compound **3′b**

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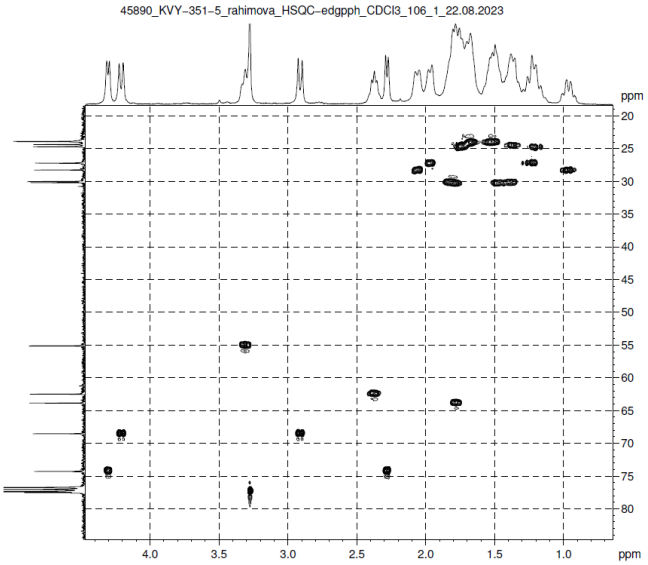
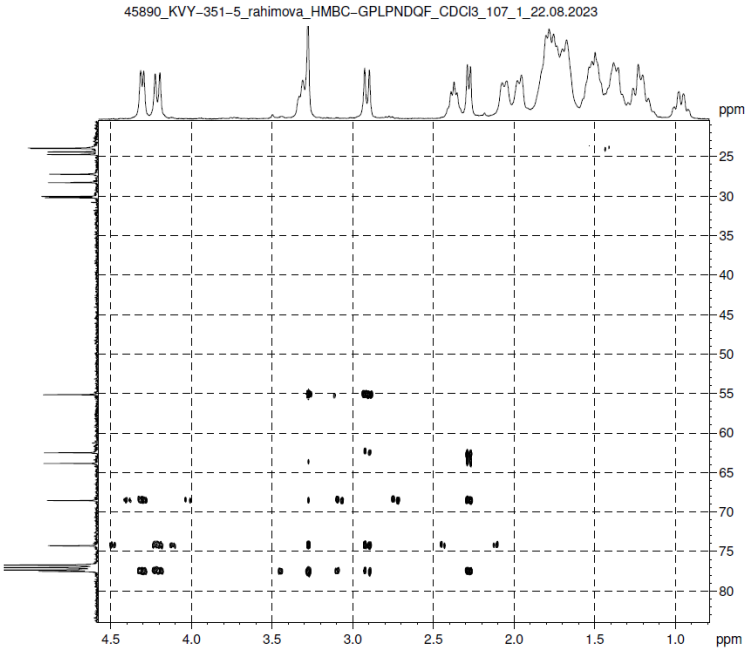

45890_KVY-351-5_rahimova_HSQC-edgpph_CDCl3_106_1_22.08.2023
ppm
20
25
30
35
40
45
50
55
60
65
70
75
80
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


45890_KVY-351-5_rahimova_HMBC-GPLPNDQF_CDCl3_107_1_22.08.2023
ppm
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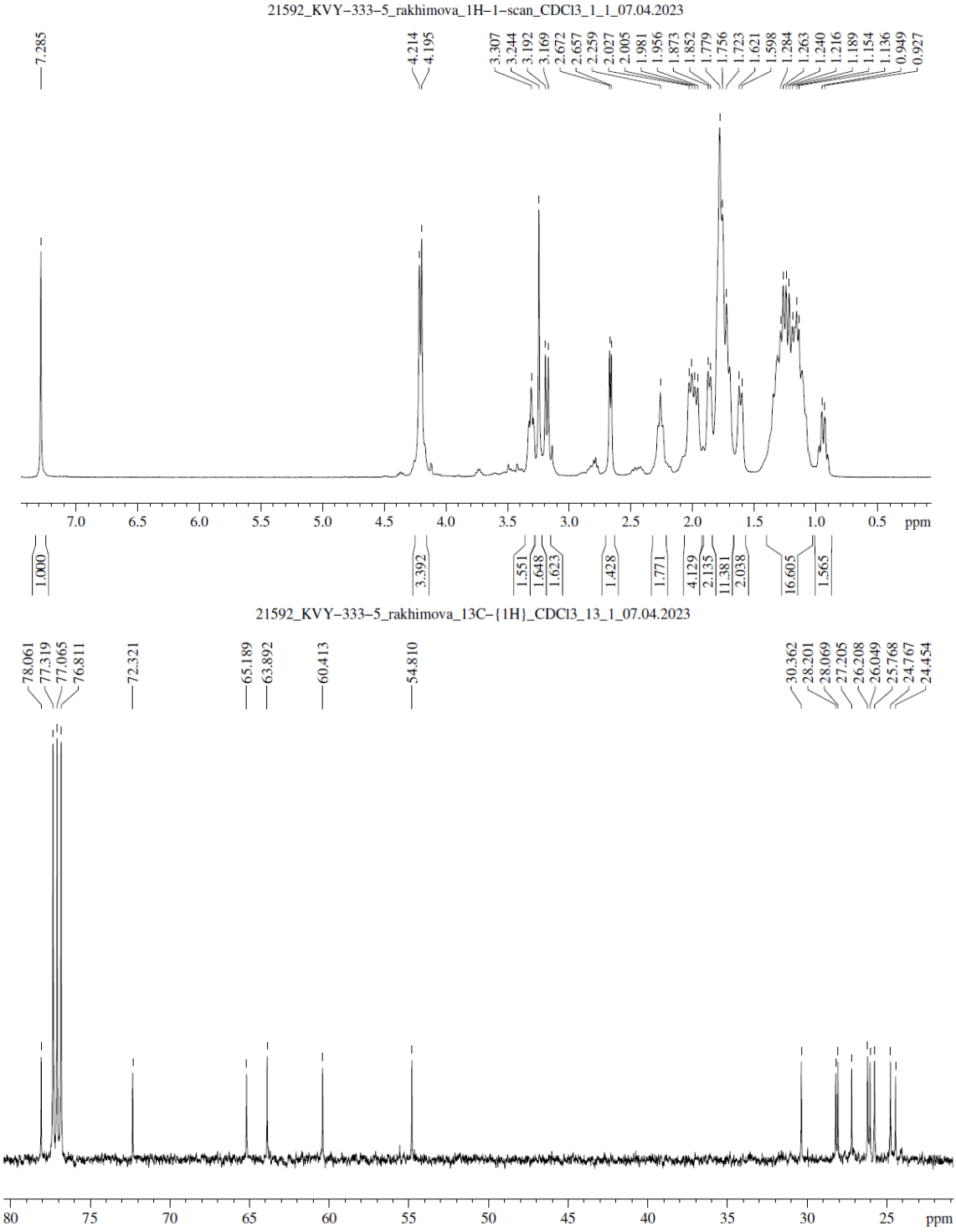
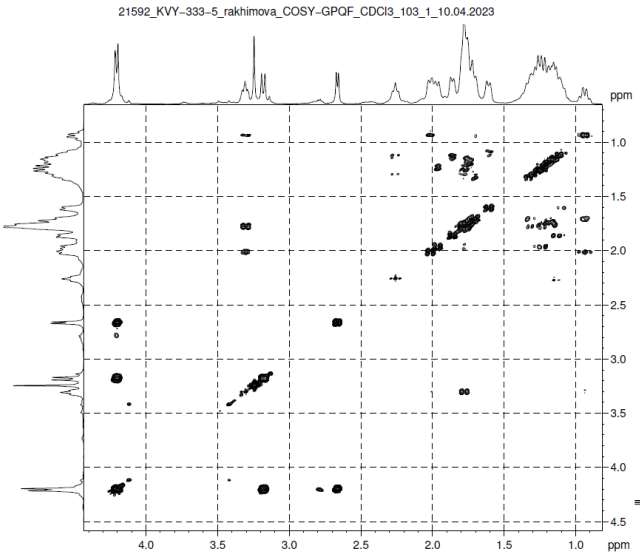
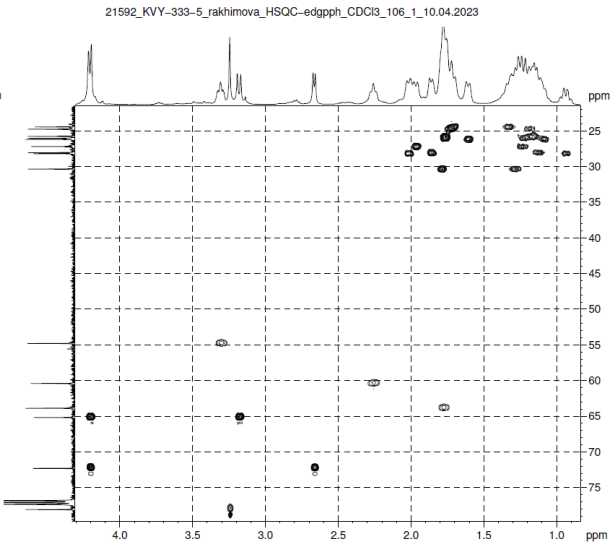
30
35
40
45
50
55
60
65
70
75
80
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

Figure S6. NMR spectra of compound **3c**




21592_KVY-333-5_rakhimova_COSY-GPQF_CDCl3_103_1_10.04.2023
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


21592_KVY-333-5_rakhimova_HSQC-edgpph_CDCl3_106_1_10.04.2023
ppm
25
30
35
40
45
50
55
60
65
70
75
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

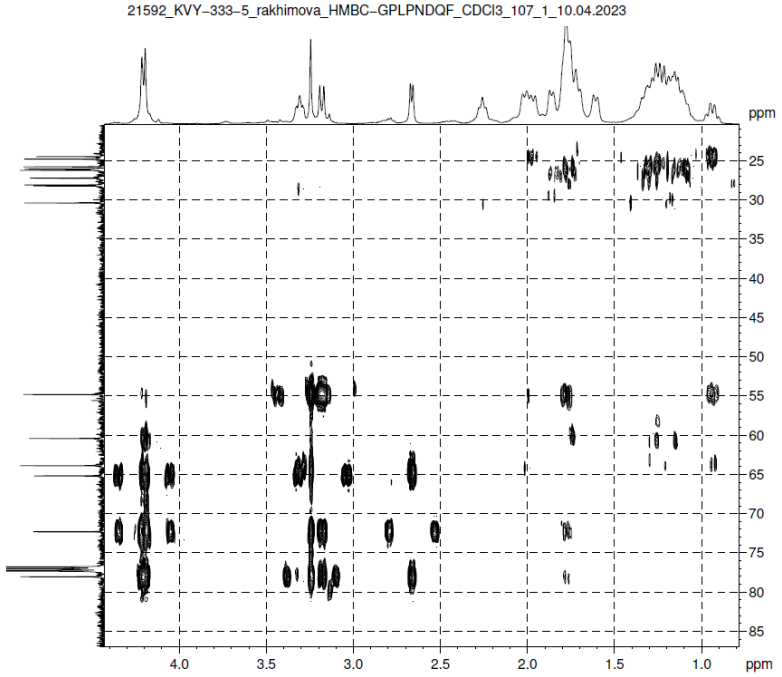
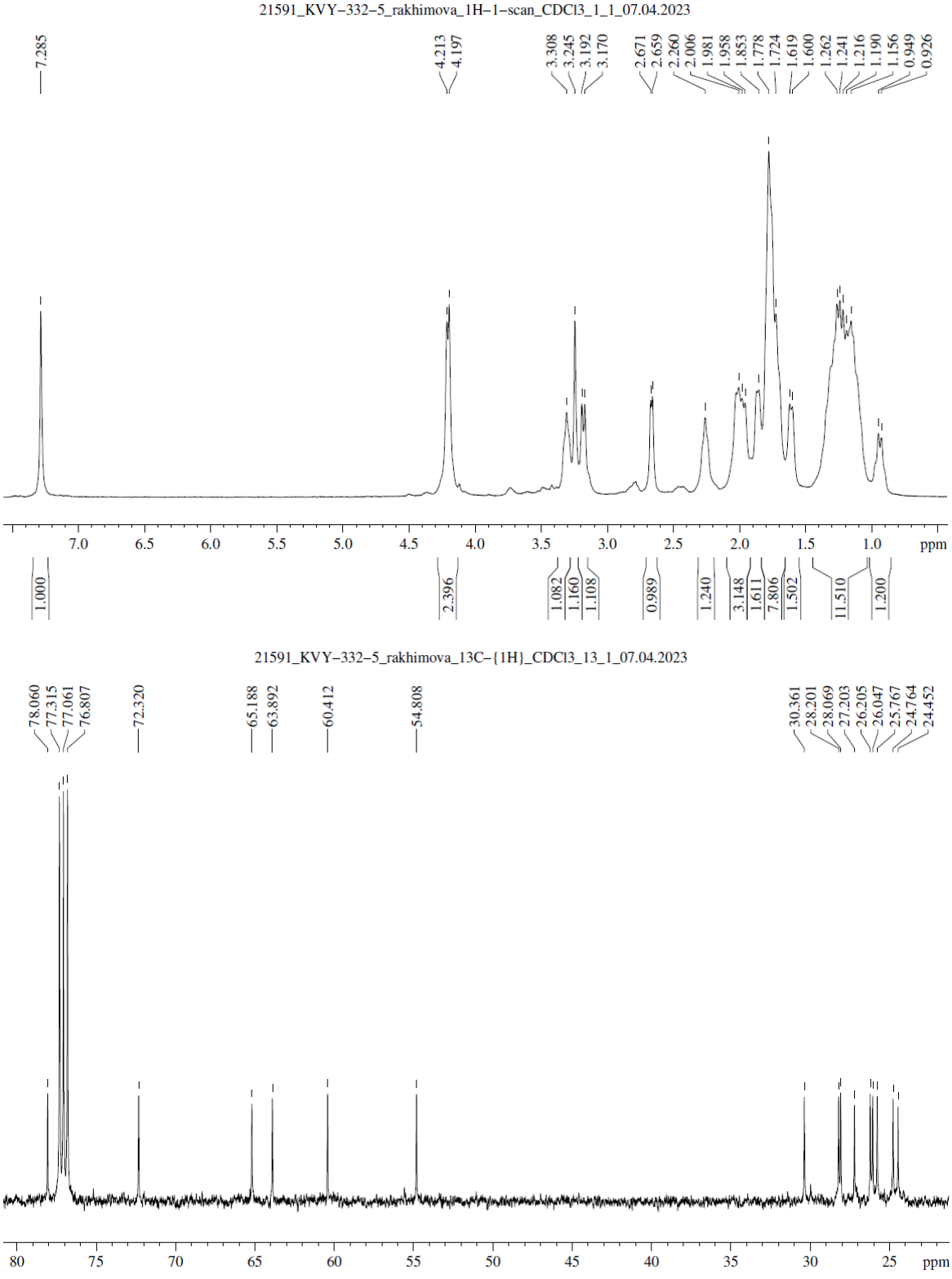
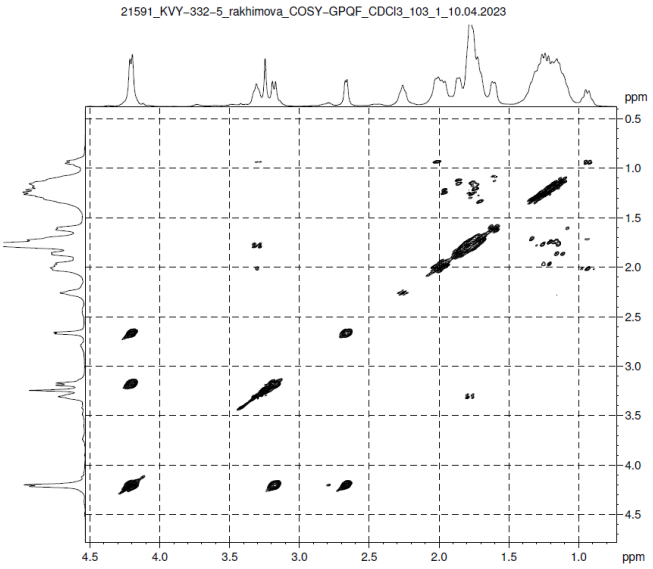
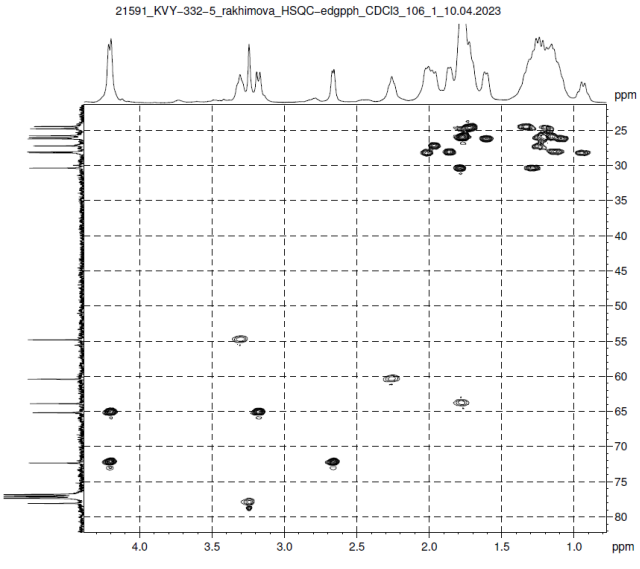

21592_KVY-333-5_rakhimova_HMBC-GPLPNDQF_CDCl3_107_1_10.04.2023
ppm
25
30
35
40
45
50
55
60
65
70
75
80
85
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

Figure S7. NMR spectra of compound **3′c**




21591_KVY-332-5_rakhimova_COSY-GPQF_CDCl3_103_1_10.04.2023
ppm
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


21591_KVY-332-5_rakhimova_HSQC-edgpph_CDCl3_106_1_10.04.2023
ppm
25
30
35
40
45
50
55
60
65
70
75
80
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

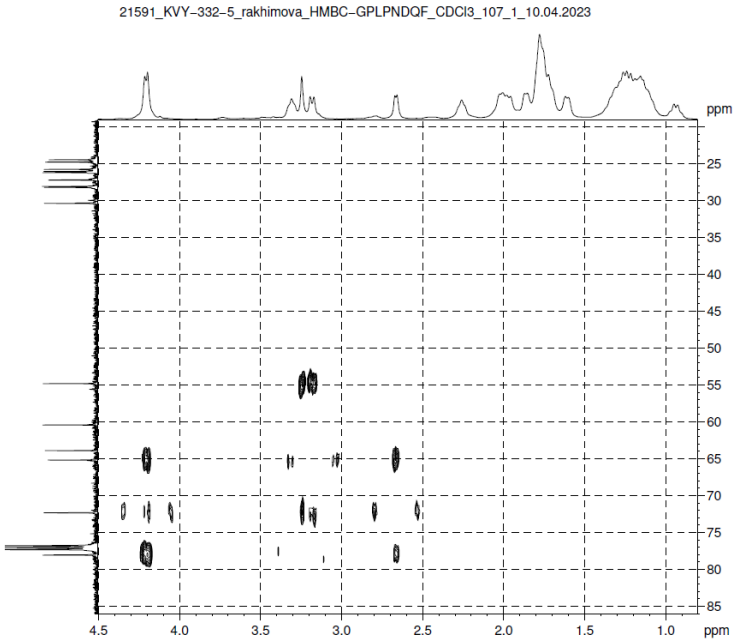
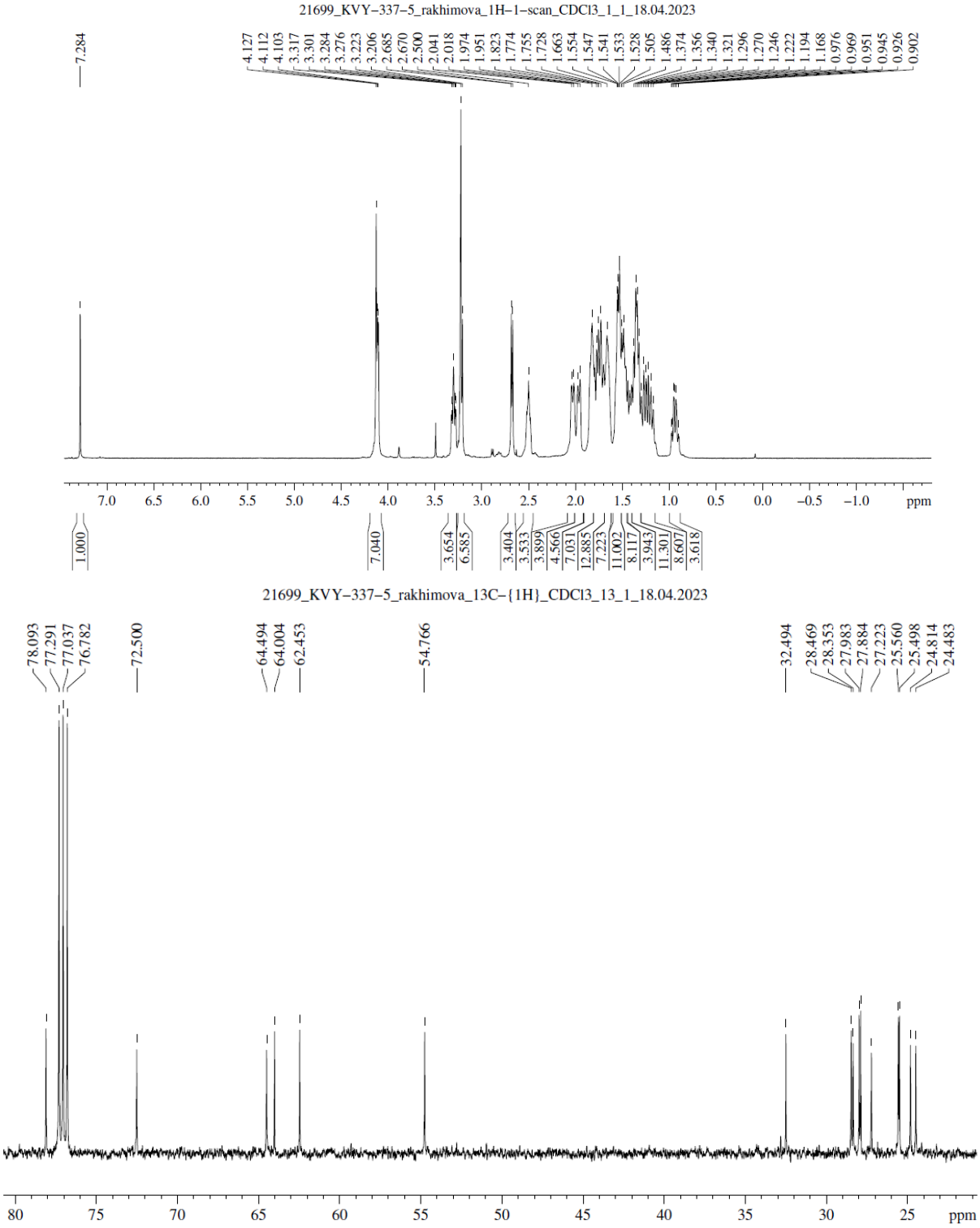
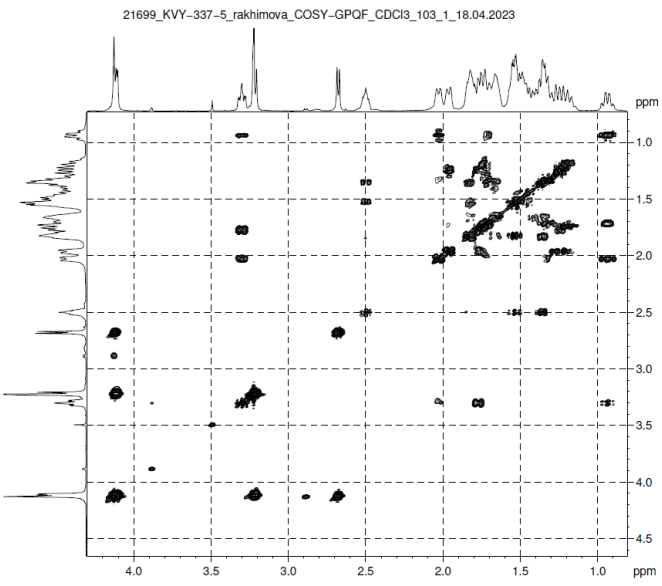

21591_KVY-332-5_rakhimova_HMBC-GPLPNDQF_CDCl3_107_1_10.04.2023
ppm
25
30
35
40
45
50
55
60
65
70
75
80
85
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

Figure S8. NMR spectra of compound **3d**




21699_KVY-337-5_rakhimova_COSY-GPQF_CDCl3_103_1_18.04.2023
ppm
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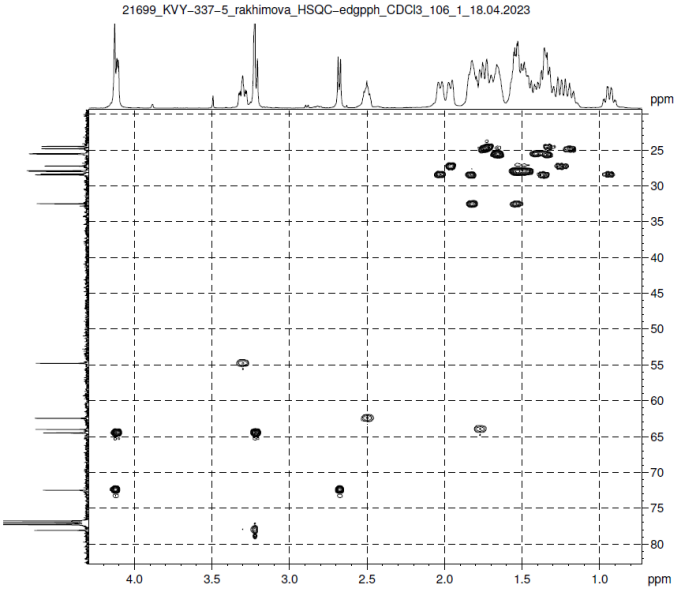
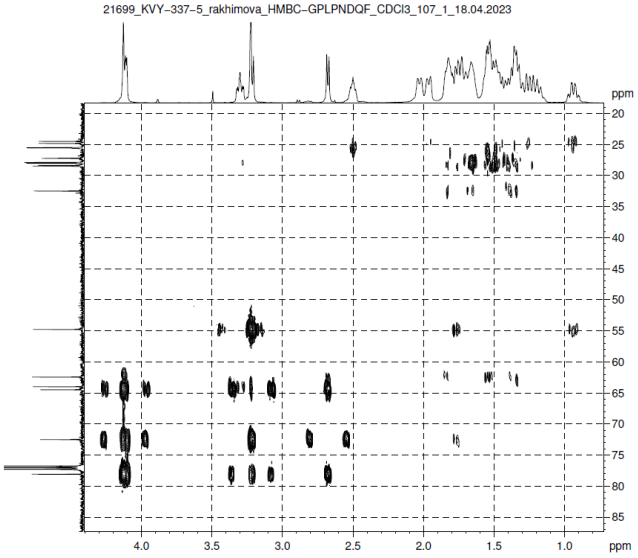
1.5
2.0
2.5
3.0
3.5
4.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


21699_KVY-337-5_rakhimova_HSQC-edgpph_CDCl3_106_1_18.04.2023
ppm
25
30
35
40
45
50
55
60
65
70
75
80
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


21699_KVY-337-5_rakhimova_HMBC-GPLPNDQF_CDCl3_107_1_18.04.2023
ppm
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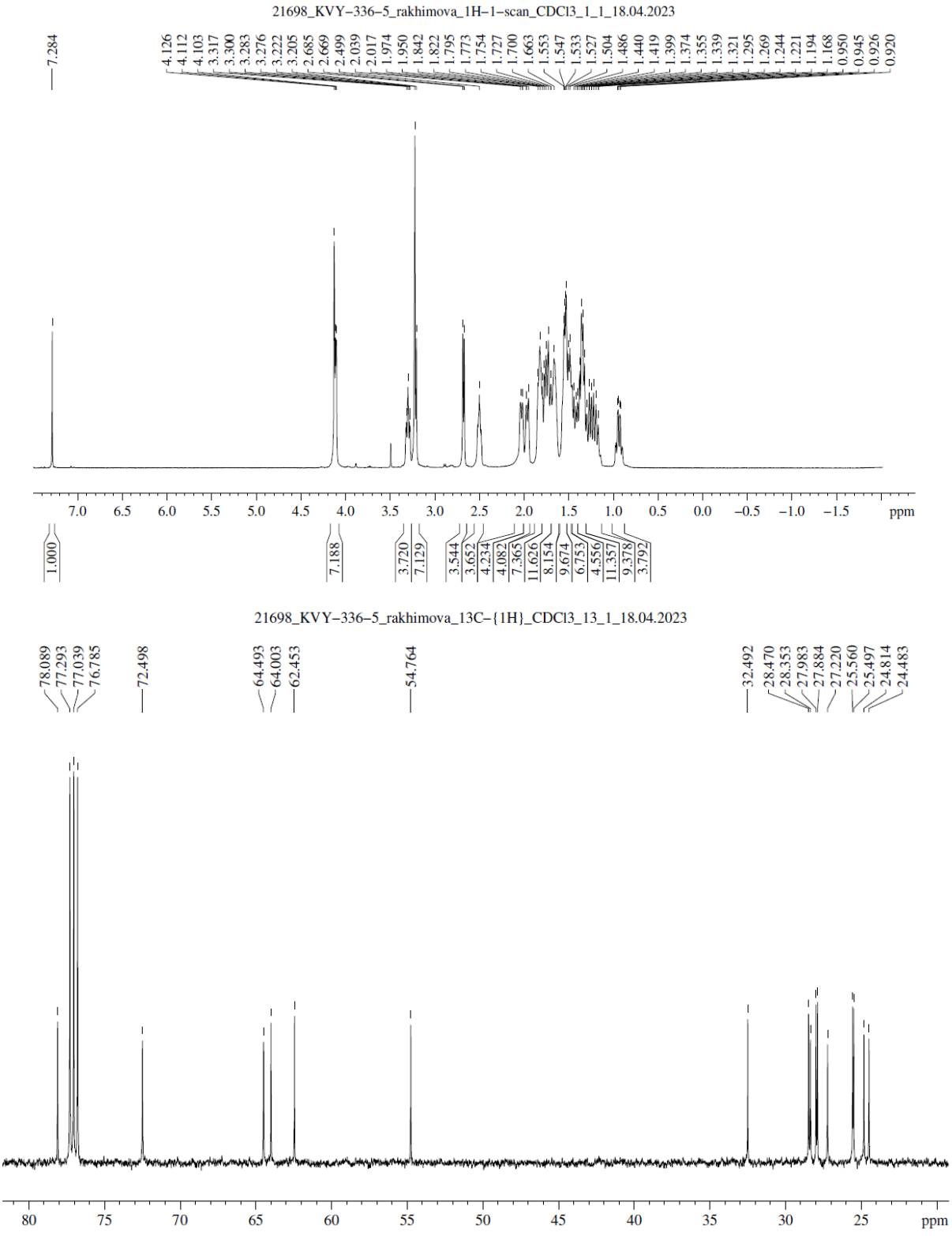
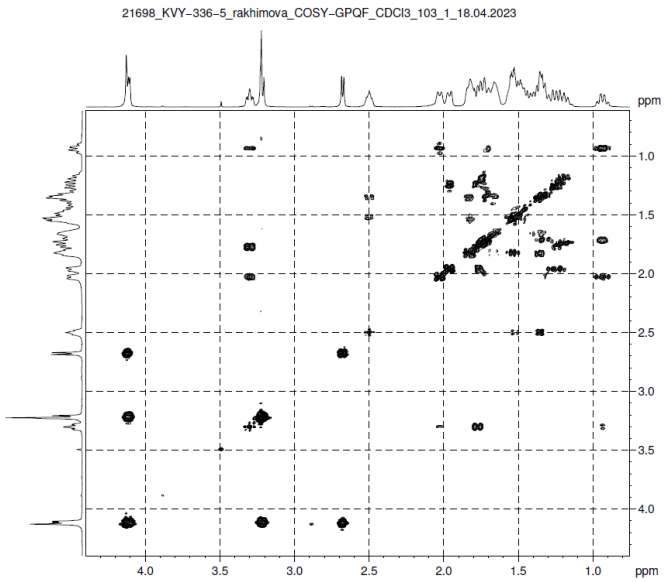
25
30
35
40
45
50
55
60
65
70
75
80
85
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

Figure S9. NMR spectra of compound **3′d**

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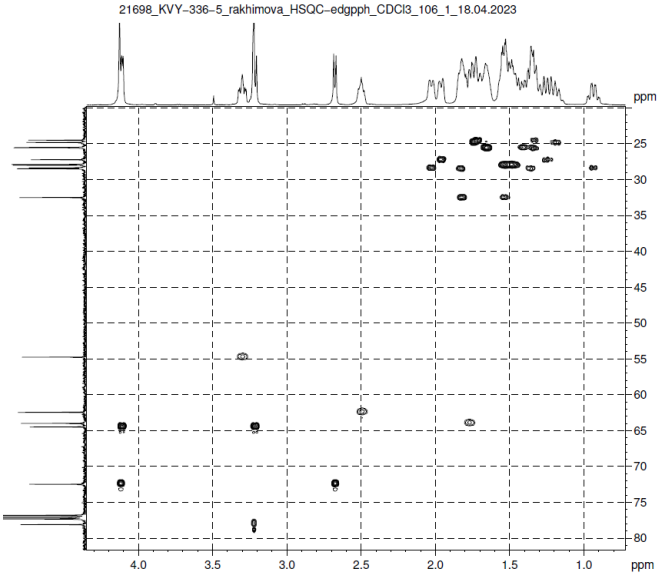
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


21698_KVY-336-5_rakhimova_HSQC-edgpph_CDCl3_106_1_18.04.2023
ppm
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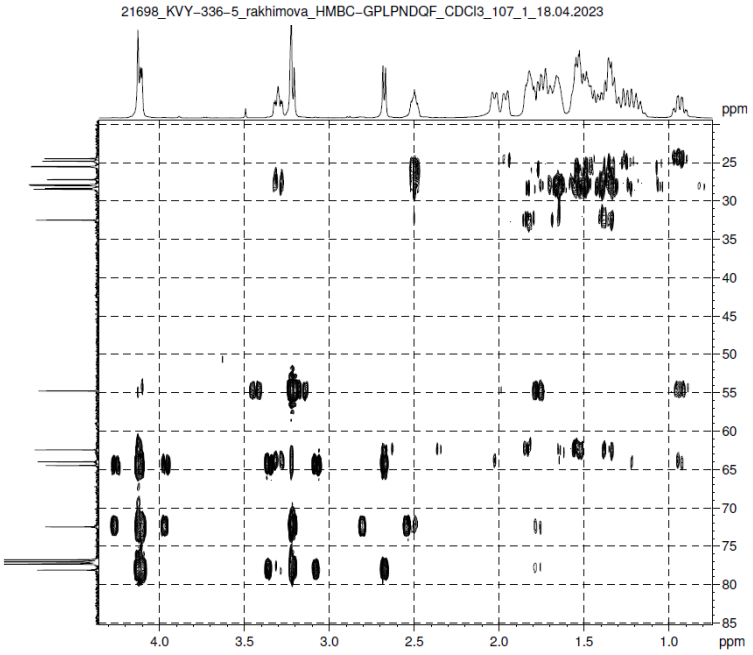
30
35
40
45
50
55
60
65
70
75
80
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


21698_KVY-336-5_rakhimova_HMBC-GPLPNDQF_CDCl3_107_1_18.04.2023
ppm
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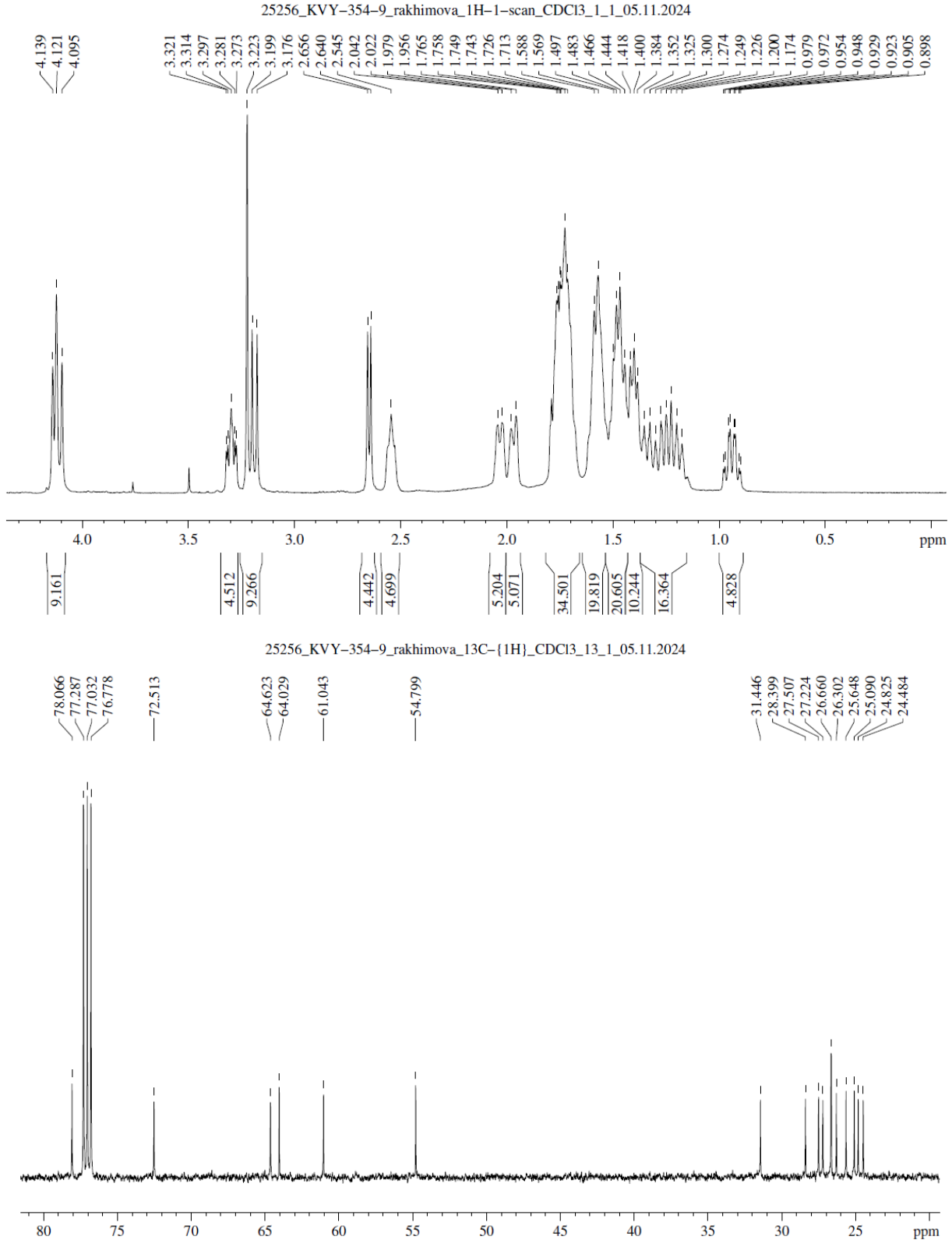
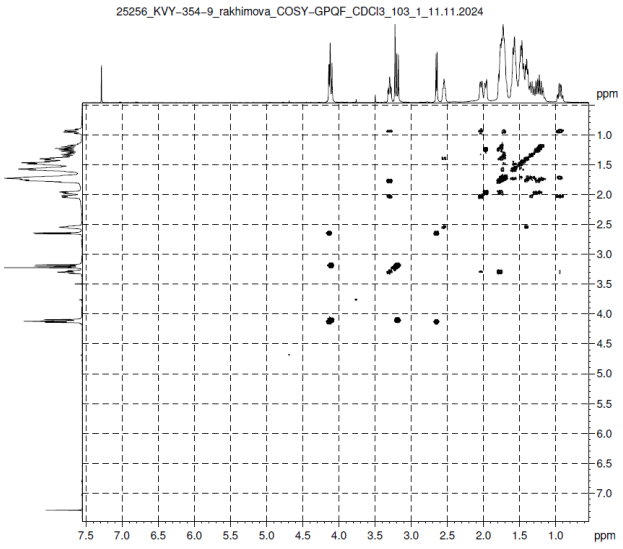
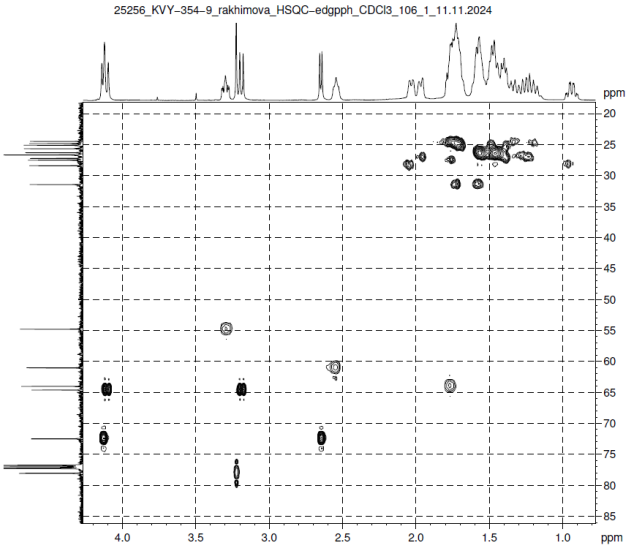
30
35
40
45
50
55
60
65
70
75
80
85
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

Figure S10. NMR spectra of compound **3e**




25256_KVY-354-9_rakhimova_COSY-GPQF_CDCl3_103_1_11.11.2024
ppm
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm


25256_KVY-354-9_rakhimova_HSQC-edgpph_CDCl3_106_1_11.11.2024
ppm
4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

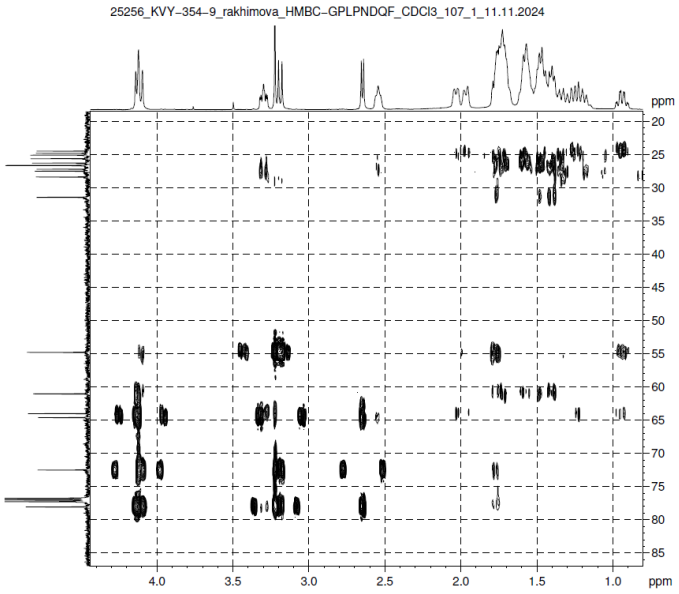
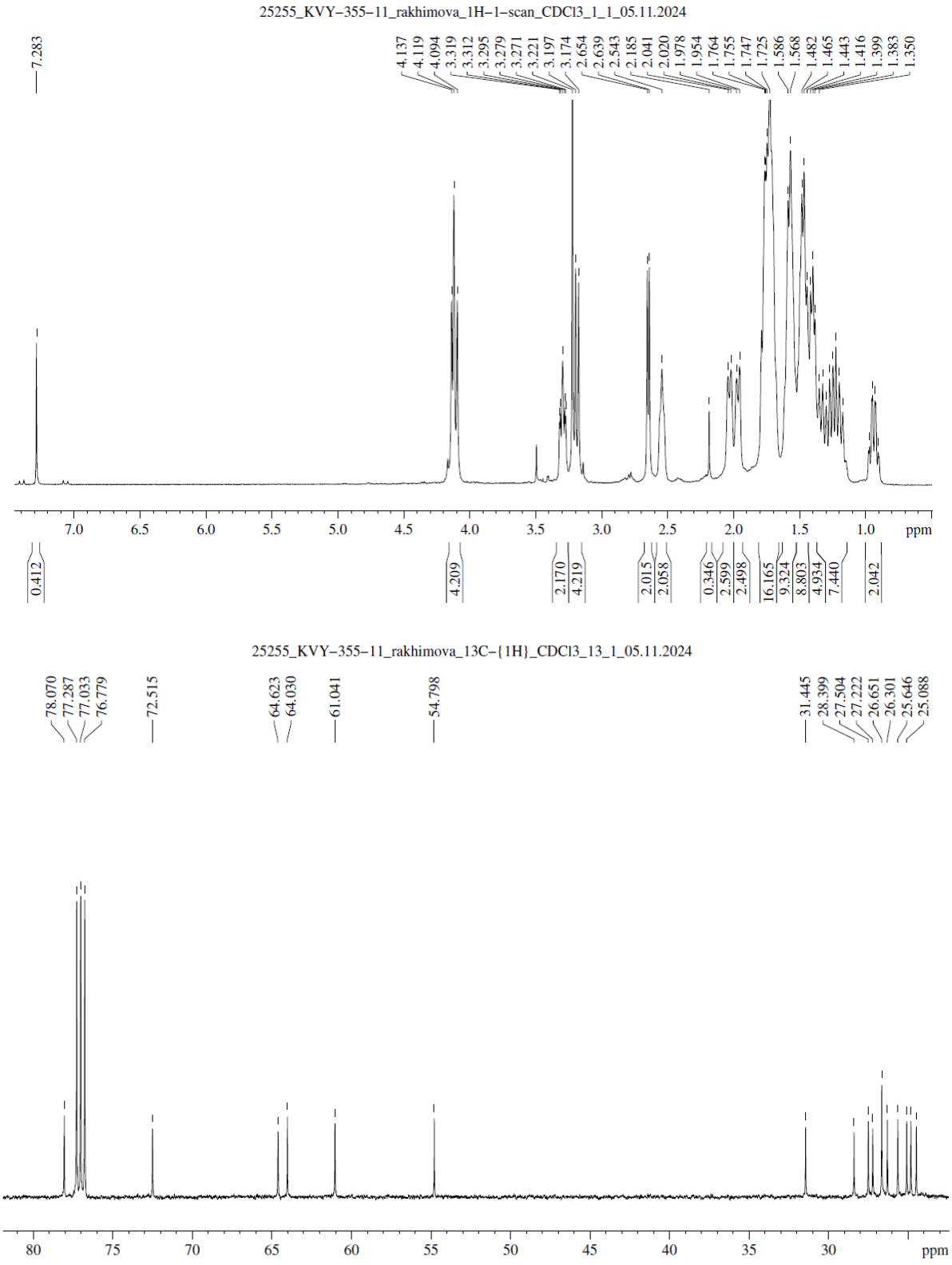
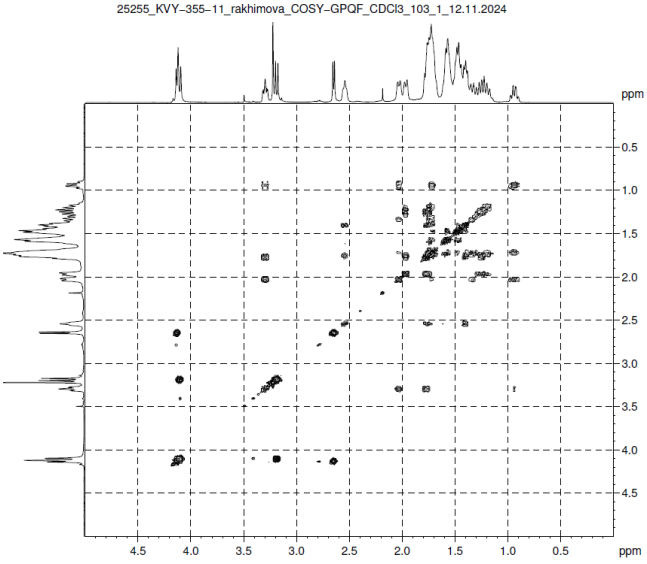

25256_KVY-354-9_rakhimova_HMBC-GPLPNDQF_CDCl3_107_1_11.11.2024
ppm
4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

Figure S11. NMR spectra of compound **3′e**

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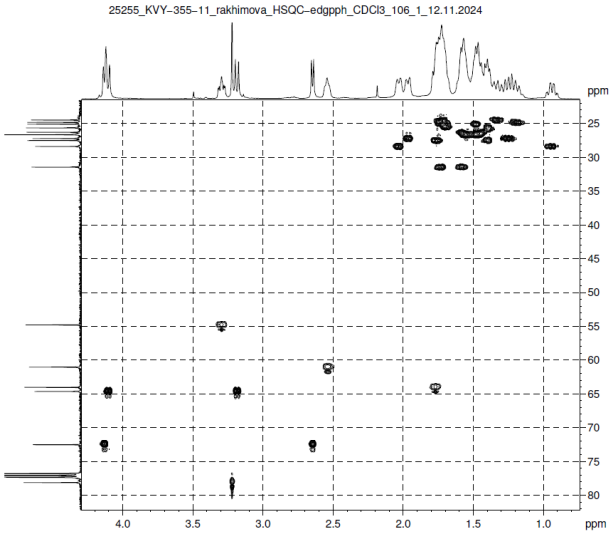
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
ppm
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm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ppm
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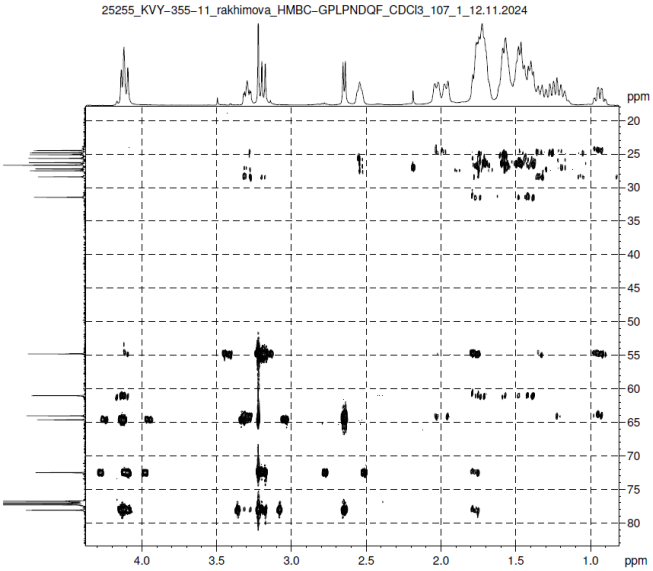
30
35
40
45
50
55
60
65
70
75
80
ppm
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm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ppm
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25
30
35
40
45
50
55
60
65
70
75
80
ppm
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm