Electronic Supplementary Material (ESI) for Green Chemistry
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SUPPORTING INFORMATION

E-factor minimized protocols for the Polystyryl-BEMP catalyzed conjugate additions of various nucleophiles to α,β-unsaturated carbonyl compounds

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CONTENTS

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Experimental section

All chemicals were purchased and used without any further purification.

GC analyses were performed by using Hewett-Packard HP 5890 series II equipped with a capillary column SPB-5 (30 m, 0.25 mm), a FID detector and hydrogen as gas carrier. GC-EIMS analyses were carried out by using a Hewett-Packard HP 6890 Series GC system/5973 Mass Selective Detector equipped with a electron impact ionizer at 70 eV. All ^{1}H NMR and ^{13}C NMR spectra were recorded at 200 MHz or 400 MHz, and at 50.3 or 100.6 MHz respectively, using a Bruker DRX-ADVANCE 200 MHz and a Bruker DRX-ADVANCE 400 MHz spectromers. Deuterated solvents were used with the residual peak as internal standard, or TMS in the case of $CDCl_3$. Chemical shift was reported in ppm and coupling costants in Hertz. All melting points were measured with Buchi Melting Point 510 apparatus and are uncorrected. Microanalyses were realized by using a Carlo Erba Elemental analyzer mod. 1106. Thin Layer Cromatography analyses were performed on silica gel on aluminum plates and UV and/or $KMnO_4$ were used as revealing systems. Column chromatographies were performed by using silica gel 230-400 mesh and eluting as reported below. PS-BEMP was purchased from Aldrich.

Compounds **4**,[1] **5**,[2] **7**,[1] **8**,[2] **11**,[3] **12**,[4] **14**,[5] **16**,[6] **17**,[7] **18**,[8] **19**,[9] **21a-b**,[10] **22**,[11] **24a-b**,[12] **26**[13] are known compounds, compounds **10**, **23**, **27a-b**, **28a**, **29** have been already prepared but spectroscopic data have not been reported, while compounds **6, 9, 15, 25, 28b** are new compounds. Characterization data (^{1}H NMR, ^{13}C NMR, GC-EIMS, mp, and elemental analyses) for compounds **6**, **9**, **10**, **15**, **23**, **25**, **27a**, **27b**, **28a**, **28b**, **29** are reported below.

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Representative experimental procedure.

In a screw capped vial equipped with a magnetic stirrer PS-BEMP (**1**) (0.048 g, 0.1 mmol, 2.1 mmol/g), *trans*-4-phenyl-3-buten-2-one (**2a**) (0.292 g, 2.0 mmol) and dimethylmalonate (**3a**) (0.229 ml, 2.0 mmol) were consecutively added and the resulting mixture was left under vigorous stirring at 60 °C. After 24 h, methanol was added, the catalyst was recovered by filtration and the organic solvent was evaporated under vacuum to give pure dimethyl-2-(1'-phenyl-3'-oxo-butyl)malonate (**4**) as a colourless oil (0.541 g, 97 % yield).

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E-factor calculation (Waste produced (g)/ Product (g))

Batch conditions:

Table 2, entry 1: reaction of **2a** with **3a** (calculated for 1.0 mmol):
E-factor = [0.146 g (**2a**) + 0.132 g (**3a**) + 1.58 g (MeOH) – 0.270 g (isolated **4)**] / 0.270 g= **5.97**

Table 2, entry 2: reaction of **2a** with **3b** (calculated for 1.0 mmol):
E-factor = [0.146 g (**2a**) + 0.133 g (**3b**) + 2.37 g (MeOH) – 0.265 g (isolated **5)**] / 0.265 g= **9.0**

Table 2, entry 3: reaction of **2a** with **3c** (calculated for 1.0 mmol):
E-factor = [0.146 g (**2a**) + 0.128 g (**3c**) + 1.58 g (MeOH) – 0.247 g (isolated **6)**] / 0.247 g= **6.5**

Table 2, entry 4: reaction of **2b** with **3a** (calculated for 1.0 mmol):
E-factor = [0.096 g (**2b**) + 0.132 g (**3a**) + 2.37 g (MeOH) – 0.217 g (isolated **7)**] / 0.217 g= **11.0**

Table 2, entry 5: reaction of **2b** with **3b** (calculated for 1.0 mmol):
E-factor = [0.096 g (**2b**) + 0.133 g (**3b**) + 1.58 g (MeOH) – 0.213 g (isolated **8)**] / 0.213 g= **7.5**

Table 2, entry 6: reaction of **2b** with **3c** (calculated for 1.0 mmol):
E-factor = [0.096 g (**2b**) + 0.128 g (**3c**) + 2.37 g (MeOH) – 0.211 g (isolated **9)**] / 0.211 g= **11.3**

Table 2, entry 7: reaction of **2c** with **3a** (calculated for 1.0 mmol):
E-factor = [0.086 g (**2c**) + 0.396 g (**3a**) + 1.58 g (MeOH) – 0.186 g (isolated **10)**] / 0.186 g= **10.1**

Table 2, entry 8: reaction of **2c** with **3b** (calculated for 1.0 mmol):
E-factor = [0.086 g (**2c**) + 0.399 g (**3b**) + 1.58 g (MeOH) – 0.197 g (isolated **11)**] / 0.197 g= **9.5**

Table 2, entry 9: reaction of **2c** with **3c** (calculated for 1.0 mmol):
E-factor = [0.086 g (**2c**) + 0.128 g (**3c**) + 2.37 g (MeOH) – 0.197 g (isolated **12)**] / 0.197 g= **12.1**

Table 3, entry 1: reaction of **2a** with **13a** (calculated for 1.0 mmol):
E-factor = [0.146 g (**2a**) + 0.110 g (**13a**) + 1.42 g (MeOH) – 0.249 g (isolated **14)**] / 0.249 g= **5.7**

Table 3, entry 2: reaction of **2a** with **13b** (calculated for 1.0 mmol):
E-factor = [0.146 g (**2a**) + 0.090 g (**13b**) + 1.42 g (MeOH) – 0.225 g (isolated **15)**] / 0.225 g= **6.4**

Table 3, entry 3: reaction of **2b** with **13a** (calculated for 1.0 mmol):
E-factor = [0.096 g (**2b**) + 0.110 g (**13a**) + 1.42 g (MeOH) – 0.190 g (isolated **16)**] / 0.190 g= **7.6**

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Table 3, entry 4: reaction of **2b** with **13b** (calculated for 1.0 mmol):
E-factor = [0.096 g (**2b**) + 0.090 g (**13b**) + 1.42 g (MeOH) – 0.179 g (isolated **17)**] / 0.179 g= **8.0**

Table 3, entry 5: reaction of **2c** with **13a** (calculated for 1.0 mmol):
E-factor = [0.086 g (**2c**) + 0.110 g (**13a**) + 1.42 g (MeOH) – 0.188 g (isolated **18)**] / 0.188 g= **7.6**

Table 3, entry 6: reaction of **2c** with **13b** (calculated for 1.0 mmol):
E-factor = [0.086 g (**2c**) + 0.090 g (**13b**) + 1.42 g (MeOH) – 0.164 g (isolated **19)**] / 0.164 g= **8.8**

Table 4, entry 1: reaction of **2a** with **20a** (calculated for 1.0 mmol):

E-factor = [0.146 g (**2a**) + 0.119 g (**20a**) + 2.37 g (MeOH) – 0.252 g (isolated **21)**] / 0.252 g= **9.5**

Table 4, entry 2: reaction of **2a** with **20b** (calculated for 1.0 mmol):
E-factor = [0.146 g (**2a**) + 0.146 g (**20b**) + 2.37 g (MeOH) – 0.219 g (isolated **22)**] / 0.219 g= **11.2**

Table 4, entry 3: reaction of **2a** with **20c** (calculated for 1.0 mmol):
E-factor = [0.146 g (**2a**) + 0.087 g (**20c**) + 2.37 g (MeOH) – 0.198 g (isolated **23)**] / 0.198 g= **12.2**

Table 4, entry 4: reaction of **2b** with **20a** (calculated for 1.0 mmol):
E-factor = [0.096 g (**2b**) + 0.119 g (**20a**) + 2.37 g (MeOH) – 0.198 g (isolated **24)**] / 0.198 g= **12.1**

Table 4, entry 5: reaction of **2b** with **20b** (calculated for 1.0 mmol):
E-factor = [0.096 g (**2b**) + 0.146 g (**20b**) + 2.37 g (MeOH – 0.220 g (isolated **25)**] / 0.220 g= **10.9**

Table 4, entry 6: reaction of **2b** with **20c** (calculated for 1.0 mmol):
E-factor = [0.096 g (**2b**) + 0.087 g (**20c**) + 2.37 g (MeOH) – 0.186 g (isolated **26)**] / 0.186 g= **12.7**

Table 4, entry 7: reaction of **2c** with **20a** (calculated for 1.0 mmol):
E-factor = [0.086 g (**2c**) + 0.119 g (**20a**) + 2.37 g (MeOH) – 0.193 g (isolated **27)**] / 0.193 g= **12.4**

Table 4, entry 8: reaction of **2c** with **20b** (calculated for 1.0 mmol):
E-factor = [0.258 g (**2c**) + 0.146 g (**20b**) + 2.37 g (MeOH) – 0.223 g (isolated **28)**] / 0.223 g= **11.5**

Table 4, entry 9: reaction of **2c** with **20c** (calculated for 1.0 mmol):
E-factor = [0.172 g (**2c**) + 0.087 g (**20c**) + 1.58 g (MeOH) – 0.171 g (isolated **29)**] / 0.171 g= **9.8**

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Cyclic continuous-flow conditions for the reaction of **2c** with **3c** (calculated for 50.0 mmol):

E-factor = [4.3 g (**2c**) + 6.4 g (**3c**) + 4.75 g (MeOH) – 10.17 g (isolated **12)**] / 10.17 g**= 0.52**

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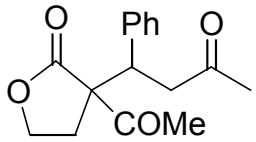
Characterization data

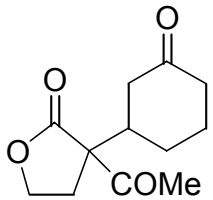
3-Acetyl-3-(3'-oxo-1'-phenyl-butyl)-dihydro-furan-2-one (6)



6

Diastereisomeric mixture isolated in 90% overall yield. Diastereoisomer A: ^{1}H-NMR ($CDCl_3$, 400 MHz) δ = 1.92-1.98 (m, 1H), 2.08 (s, 3H), 2.38-2.44 (m, 1H), 3.07-3.10 (m, 1H), 3.12-3.21 (m, 1H), 3.30-3.34 (m, 1H), 3.47-3.52 (m, 1H), 4.12-4.16 (m, 1H), 7-27-7.35 (m, 5H). ^{13}C-NMR ($CDCl_3$, 100.6 MHz) δ = 193.6, 174.0, 164.5, 138.9, 129.0, 128.3, 124.2, 66.5, 57.4, 48.8, 35.6 (2 carbons), 29.5, 28.4, 24.3. Diastereoisomer B: ^{1}H-NMR ($CDCl_3$, 400 MHz) δ = 2.07 (s, 3H), 2-23-2.38 (m, 2H), 2.74-2.76 (m, 2H), 3.83-3.90 (m, 2H), 4.17-4.23 (m, 1H), 7-22-7.33 (m, 5H). ^{13}C-NMR ($CDCl_3$, 100.6 MHz) δ = 195.3, 175.6, 163.3, 138.4, 128.7, 127.9, 124.3, 65.7, 59.1, 45.9, 44.5, 33.9, 29.3, 28.0, 24.6. GC-EIMS (m/z, %) diastereoisomer A: 258 (13), 126 (100), 115 (77), 91 (40); diastereoisomer B: 256 (94), 210 (100), 128 (96), 115 (100), 91 (83).

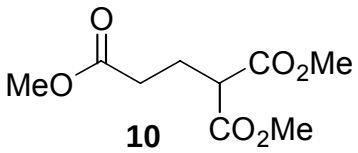
3-Acetyl-3-(3'-oxo-cyclohexyl)-dihydro-furan-2-one (9)



9

Isolated in 94% overall yield as a colourless oil. ^{1}H-NMR ($CDCl_3$, 400 MHz) δ = 1.60-1.80 (m, 2H), 2.00-2.80 (m, 1H), 2.00-2.32 (m, 4H), 2.28 (s, 3H), 2.40-2.50 (m, 2H), 2.68-2.85 (m, 1H), 2.85-2.98 (m, 1H), 4.05-4.20 (m, 1H), 4.20-4.40 (m, 1H). ^{13}C-NMR ($CDCl_3$, 100.6 MHz) δ = 208.3, 201.3, 201.0, 173.7, 66.2, 66.2, 65.9, 65.5, 43.2, 42.5, 41.5, 41.1, 40.8, 40.7, 26.5, 26.3, 25.4, 25.0, 24.5, 24.3, 24.0. Anal. Calc. for $C_{12}H_{16}O_4$ (FW 224): C, 64.27; H, 7.19; O, 28.54. Found: C, 64.01; H, 7.16. GC-EIMS (m/z, %) diastereoisomer A: 181 (M^+, 22), 112 (47), 97 (38), 43 (100); diastereoisomer B: 181 (M^+, 63), 112 (46), 97 (36), 43 (100).

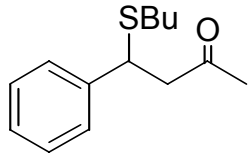
2-Methoxycarbonyl-pentandioic acid dimethylester (10)



10

Isolated by column chromatography (petroleum ether/ethyl acetate = 95/5) in 85% yield. Colourless oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 2.15-2.27 (m, 2H), 2.36-2.43 (m, 2H), 3.48 (t, *J* = 7.3 Hz, 1H), 3.67 (s, 3H), 3.73 (s, 6H). ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ = 172.7, 169.3, 52.6, 51.7, 50.4, 31.1, 23.7. Anal. calc. for $C_9H_{14}O_6$ (FW 218): C, 49.54; H, 6.47. Found: C, 48.62; H 6.44. GC-EIMS (m/z, %) 187 (28), 158 (54), 155 (100), 145 (24), 132 (43), 126 (29), 113 (83), 100 (30), 59 (100), 55 (55), 43 (13).

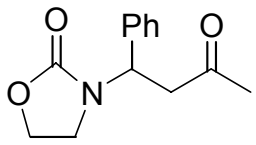
4-(Butylthio)-4-phenylbutan-2-one (15)



15

Isolated in 95% yield. Yellowish oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 0.83 (t, *J* = 7.3 Hz, 3H), 1.20-1.40 (m, 2H), 1.40-1.53 (m, 2H), 2.09 (s, 3H), 2.19-2.40 (m, 2H), 2.96 (d, *J* = 7.3 Hz, 2H), 4.31 (t, *J* = 7.3 Hz, 1H), 7.15-7.40 (m, 5H). ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ = 205.6, 142.0, 128.5, 127.7, 127.2, 50.1, 44.1, 31.2, 31.0, 30.7, 21.9, 13.6. Anal. calc. for $C_{14}H_{20}OS$ (FW 236): C, 71.14; H, 8.53; S, 13.57. Found: C, 70.8; H 8.57; S 13.52. GC-EIMS (m/z, %) 236 (M+, 28), 179 (26), 148 (41), 104 (31), 43 (100).

3-(3'-Oxo-1'-phenyl-butyl)-oxazolidin-2-one (23)



23

Isolated in 72% yield. Yellowish oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 2.23 (s, 3H), 3.01 (dd, *J* = 5.7, 16.1 Hz, 1H), 3.35 (dd, *J* = 8.2, 16.4 Hz, 1H), 3.40-3.58 (m, 2H), 4.24 (t, *J* = 8.0, 2H), 5.23 (dd, *J* = 5.7, 9.7 Hz, 1H), 7.26-7.45 (m, 5H). ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ = 205.6, 157.6, 137.9, 128.8, 128.1, 127.0, 61.8, 53.7, 45.2, 42.3, 29.6. Anal. calc. for $C_{13}H_{15}NO_3$ (FW 233): C, 66.94; H, 6.48; N, 6.00. Found: C, 66.62; H, 6.45; N, 6.03.

3-(5'-Phenyl-tetrazol-2'-yl)-cyclohexanone (25)

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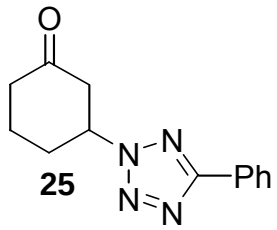
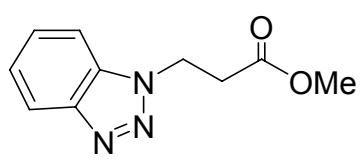


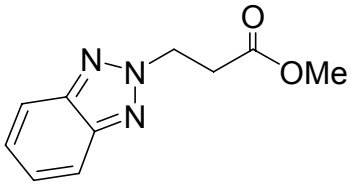
25

Isolated in 91% overall yield as a yellow solid (m.p. = 63-65 °C). ^{1}H-NMR ($CDCl_3$, 400 MHz) δ = 1.78-2.00 (m, 1H), 2.10-2.25 (m, 1H), 2.35-2.63 (m, 4H), 3.02 (dd, *J* = 5.0, 14.8 Hz, 1H), 3.16 (dd, *J* = 9.9, 14.8 Hz, 1H), 5.15-5.35 (m, 1H), 7.45-7.60 (m, 3H), 8.10-8.25 (m, 2H). ^{13}C-NMR ($CDCl_3$, 100.6 MHz) δ = 205.8, 165.0, 130.4, 128.9, 127.2, 126.8, 61.5, 46.2, 40.4, 30.8, 21.3. Anal. Calc. for $C_{13}H_{14}N_4O$ (FW 242): C, 64.45; H, 5.82; N, 23.23; O, 6.60. Found: C, 64.76; H, 5.84; N, 23.04. GC-EIMS (m/z, %) 103 (100), 76 (38).

3-Benzotriazol-1-yl-propionic acid methyl ester (27a)/3-Benzotriazol-2-yl-propionic acid methyl ester (27b)



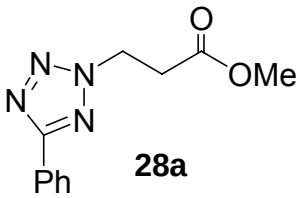
27a (80%)



27b (20%)

Mixture of two regioisomeric products (27a:27b=4:1 from ^{1}H-NMR). Isolated in 94% overall yield. Yellow oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 3.13 (t, *J* = 6.7 Hz, 2H, A), 3.20 (t, *J* = 7.0 Hz, 2H, B), 3.67 (s, 3H, A), 3.73 (s, 3H, B), 4.93 (t, *J* = 6.8 Hz, 2H, A), 5.05 (t, *J* = 7.0 Hz, 2H, B), 7.35-7.42 (m, 1H A + 1H B), 7.52 (t, *J* = 7.6 Hz, 1H, A), 7.62 (d, *J* = 8.3 Hz, 1H, A), 7.82-7.95 (m, 2H, B), 8.06 (d, *J* = 8.3 Hz, 1H, A). ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ = 171.0 (A+B), 145.8 (A), 144.3 (B), 133.1 (A), 127.5 (A); 126.4 (B), 124.0 (A), 119.9 (A), 118.0 (B), 109.5, (A), 52.1 (A+B), 51.6 (B), 43.3 (A), 34.1 (A), 33.9 (B). Anal. calc. for $C_{10}H_{11}N_3O_2$ (FW 205): C, 58.53; H, 5.40; N, 20.48. Found: C, 58.27; H 5.41; N 20.56. GC-EIMS (m/z, %) A: 205 (M+, 44), 104 (99), 91 (57), 87 (32), 77 (90), 64 (41), 59 (100), 51 (35); B: 205 (M+, 57), 146 (100), 91 (47), 77 (31), 64 (31), 59 (40), 55 (36).

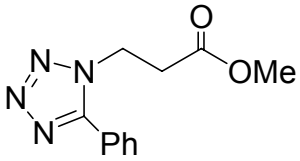
3-(5'-Phenyl-tetrazol-2'-yl)-propionic acid methyl ester (28a)



28a

Mixture of two regioisomeric products (**28a**:**28b**=89:11 from ^{1}H-NMR). Isolated by column chromatography (petroleum ether/ethyl acetate = 3/1) in 96% overall yield. **28a**: white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 3.14 (t, *J* = 7.0 Hz, 2H), 3.74 (s, 3H), 4.96 (t, *J* = 7.0 Hz, 2H), 7.45-7.60 (m, 3H), 8.10-8.25 (m, 2H). ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ = 170.1, 165.2, 130.4, 128.9, 127.2, 126.8, 52.3, 48.4, 33.3. Anal. Calc. for $C_{11}H_{12}N_4O_2$ (FW 232): C, 56.89; H, 5.21; N, 24.12; O, 13.78. Found: C, 57.13; H, 5.19; N, 24.23. GC-EIMS (m/z, %) 232 (M+, 11), 159 (100), 131 (20).

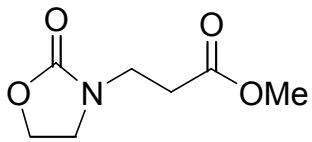
3-(5'-Phenyl-tetrazol-1'-yl)-propionic acid methyl ester (28b)



28b

Mixture of two regioisomeric products (**28a**:**28b**=89:11 from ^{1}H-NMR). Isolated by column chromatography (petroleum ether/ethyl acetate = 3/1) in 96% overall yield. **28b**: colourless oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 3.10 (t, *J* = 6.8 Hz, 2H), 3.66 (s, 3H), 4.67 (t, *J* = 6.8 Hz, 2H), 7.50-7.65 (m, 3H), 7.65-7.80 (m, 2H). ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ = 170.2, 154.6, 131.3, 129.3, 128.8, 123.7, 52.3, 43.4, 33.3. Anal. Calc. for $C_{11}H_{12}N_4O_2$ (FW 232): C, 56.89; H, 5.21; N, 24.12; O, 13.78. Found: C, 57.13; H, 5.19; N, 24.23. GC-EIMS (m/z, %) 232 (M+, 43), 131 (27), 118 (31), 104 (69), 89 (47), 77 (78), 63 (37), 59 (100), 55 (43), 51 (31).

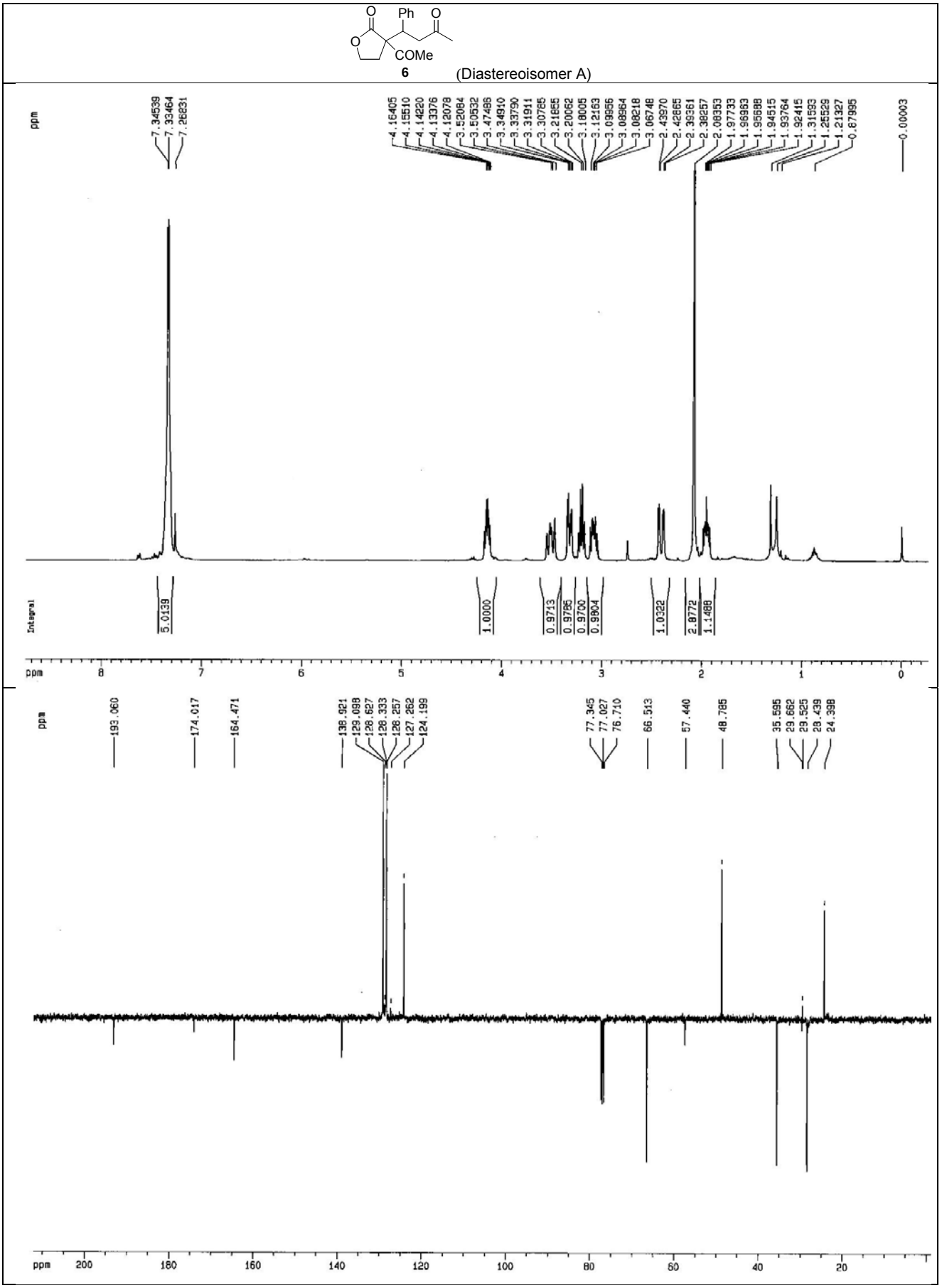
3-(2'-Oxo-oxazolidin-3'-yl)-propionic acid methyl ester (29)

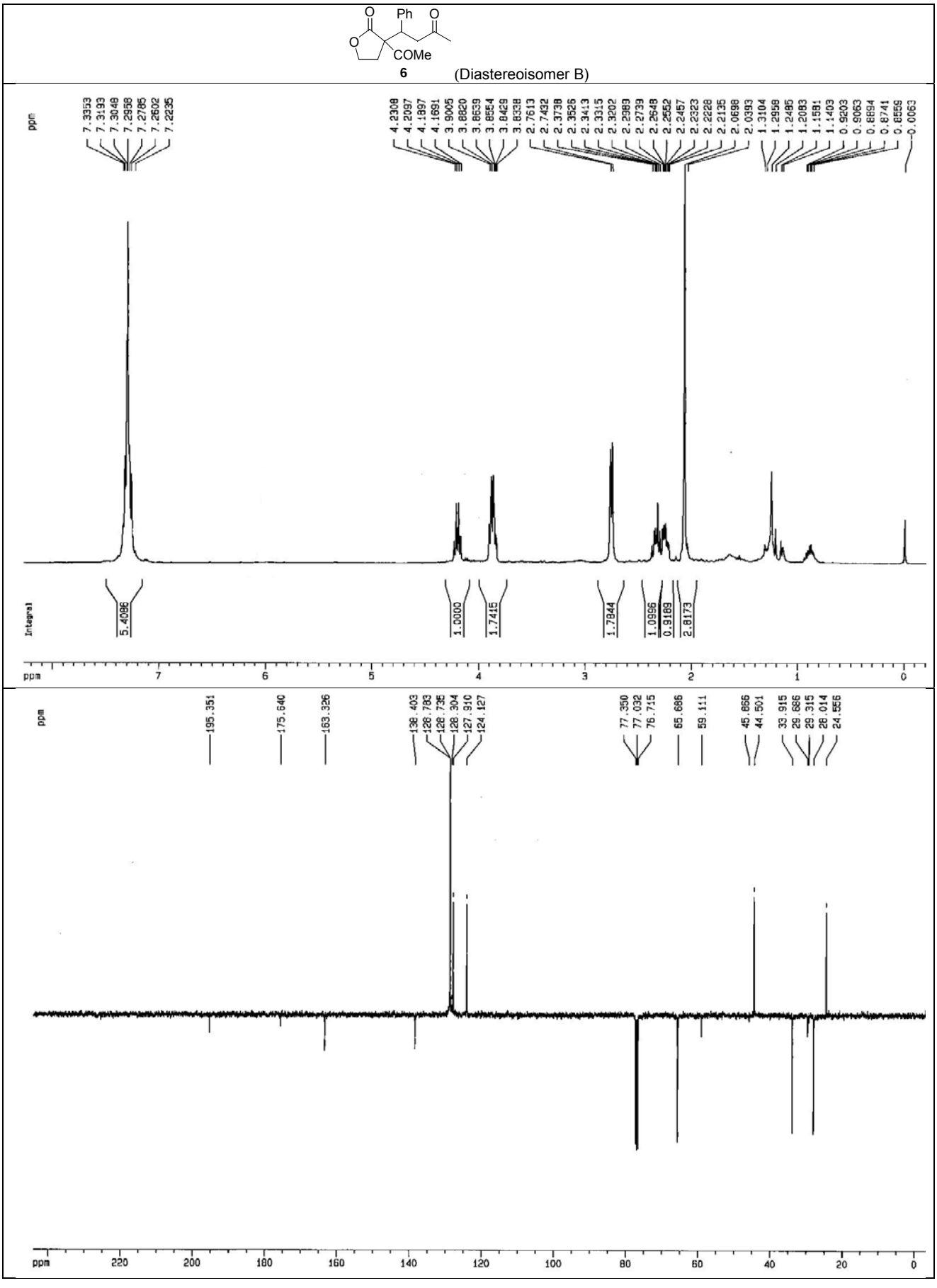


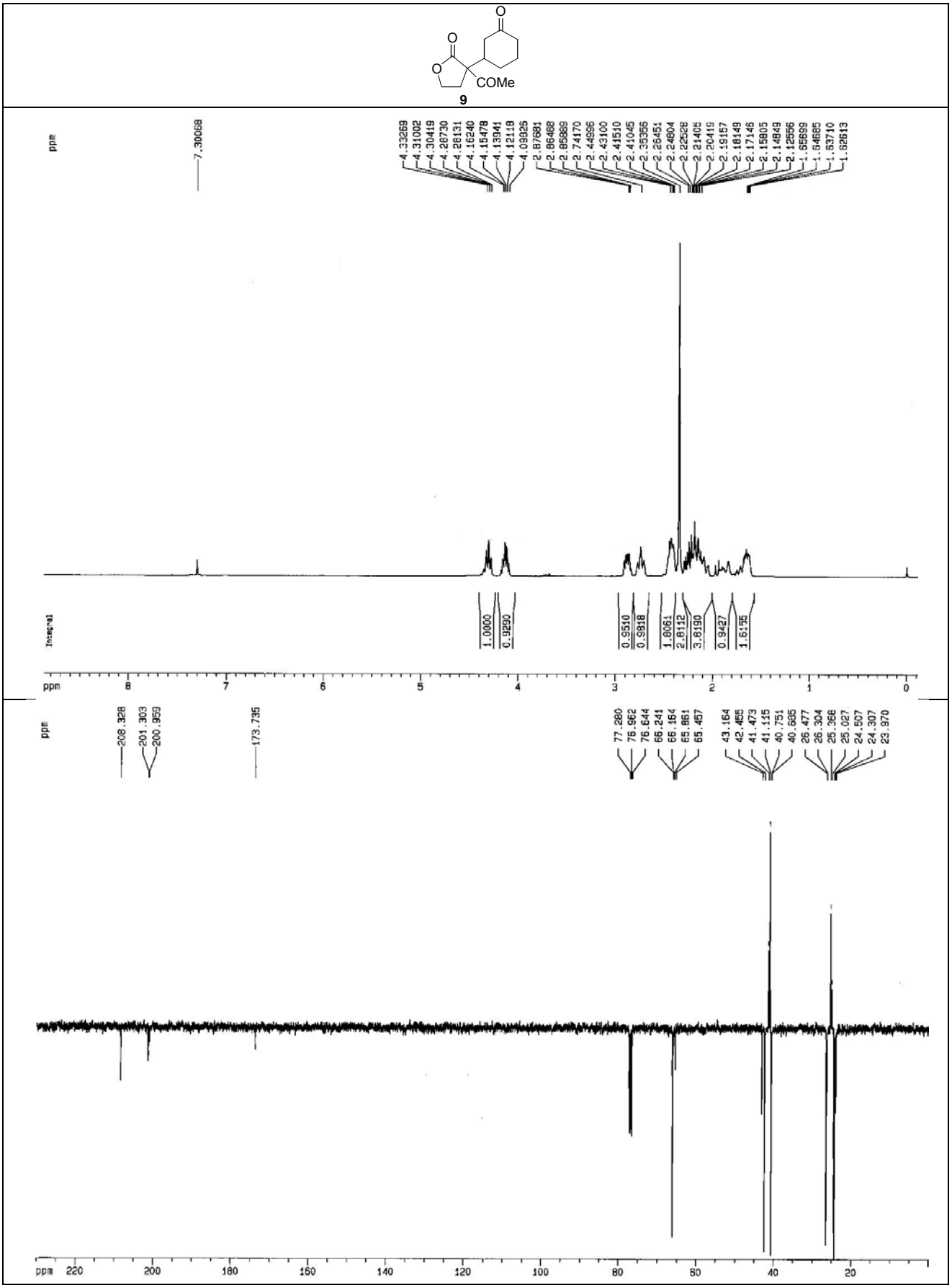
29

Isolated in 99% yield. Colourless oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 2.61 (t, *J* = 6.6 Hz, 2H), 3.54 (t, *J* = 6.6 Hz, 2H,), 3.61 (t, *J* = 8.5 Hz, 2H), 3.68 (s, 3H), 4.29 (t, *J* = 8.0 Hz, 2H). ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ = 171.9, 158.3, 61.8, 51.8, 45.2, 40.1, 32.5. Anal. calc. for $C_7H_{11}NO_4$ (FW 173): C, 48.55; H, 6.40; N, 8.09. Found: C, 48.33; H 6.37; N 8.12. GC-EIMS (m/z, %) 142 (15), 129 (20), 113 (20), 100 (39), 70 (27), 56 (100), 43 (13).

^{1}H and ^{13}C-NMR Spectra




Ph
O
O
O
COMe
6
(Diastereoisomer B)
ppm
Integral
5.4096
1.0000
1.7415
1.7844
1.0986
0.9189
2.8173
195.351
175.640
163.326
138.403
128.783
128.735
128.304
127.910
124.127
77.350
77.032
76.715
65.686
59.111
45.866
44.501
33.915
29.686
29.315
26.014
24.556


O
O
O
COMe
9
ppm
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4.31002
4.30419
4.28730
4.28131
4.16240
4.15478
4.13941
4.12119
4.09925
2.87681
2.86468
2.85889
2.74170
2.44996
2.43100
2.41510
2.41045
2.35356
2.26451
2.24804
2.22528
2.21405
2.20419
2.19157
2.18149
2.17146
2.15805
2.14849
2.12556
1.55699
1.54685
1.53710
1.52613
Integral
1.0000
0.9290
0.9510
0.9818
1.8061
2.8112
3.8190
0.9427
1.6155
ppm
8
7
6
5
4
3
2
1
0
ppm
208.328
201.303
200.959
173.735
77.280
76.962
76.644
66.241
66.164
65.861
65.457
43.164
42.455
41.473
41.115
40.751
40.685
26.477
26.304
25.368
25.027
24.507
24.307
23.970
ppm
220
200
180
160
140
120
100
80
60
40
20

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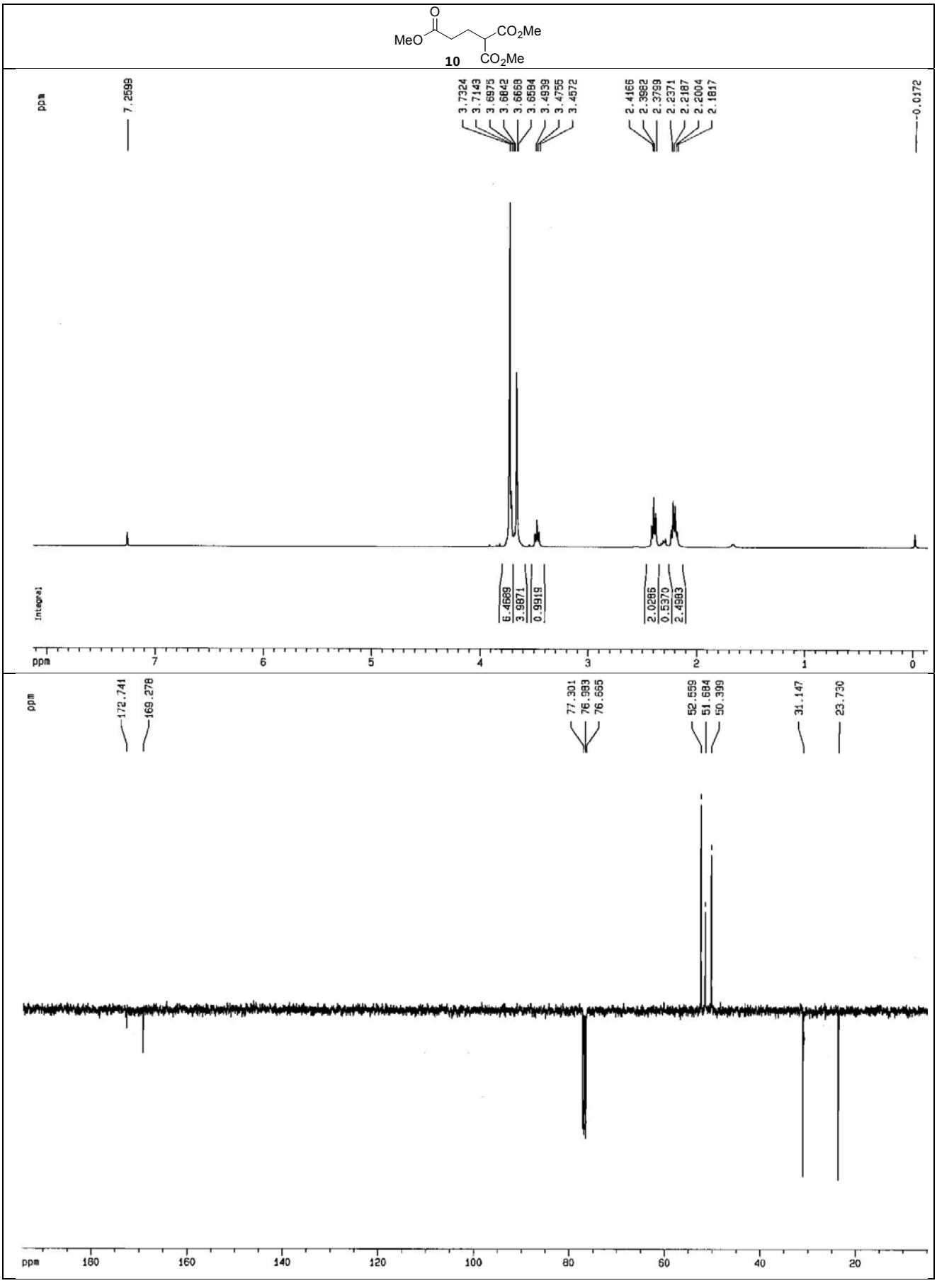

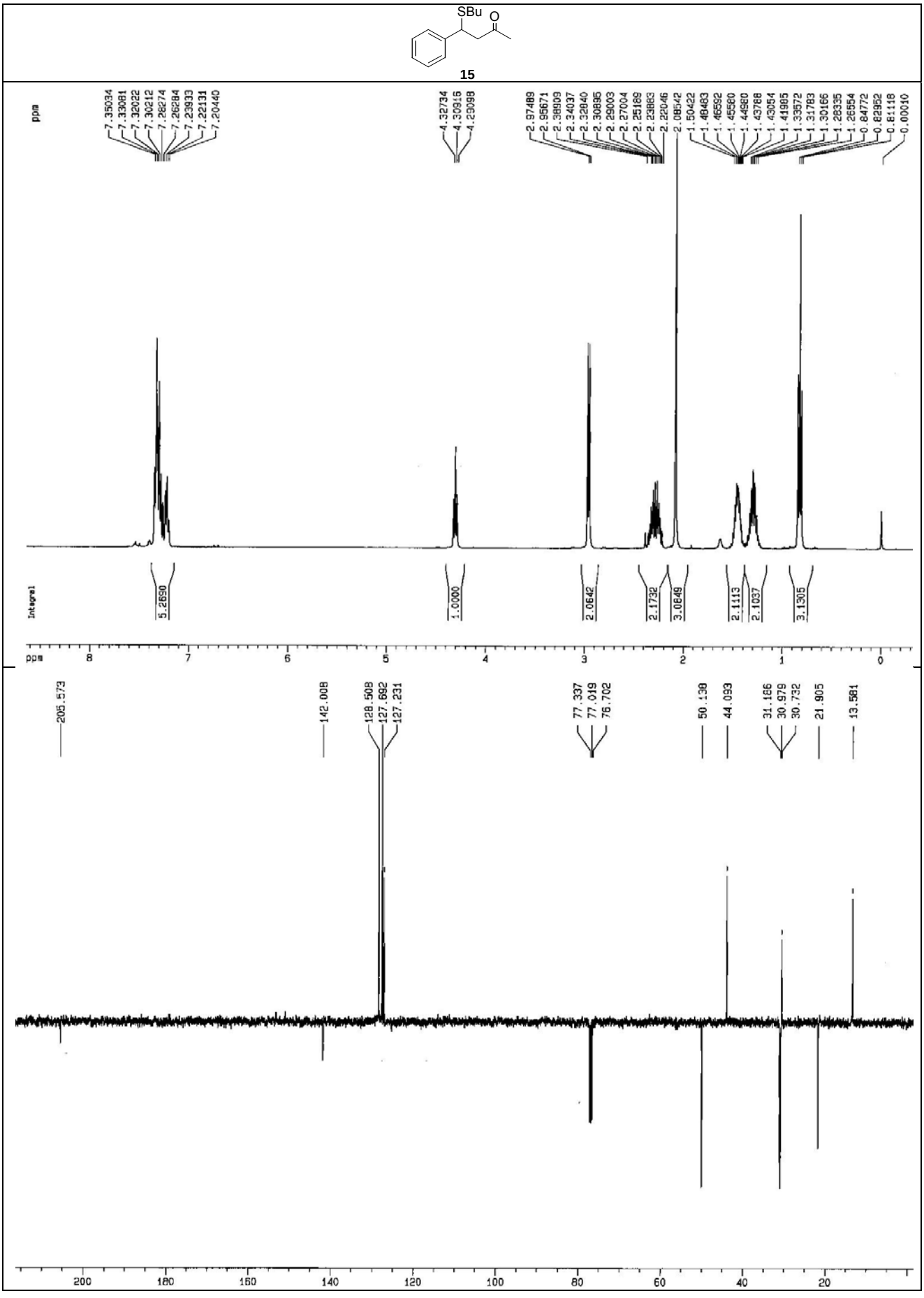
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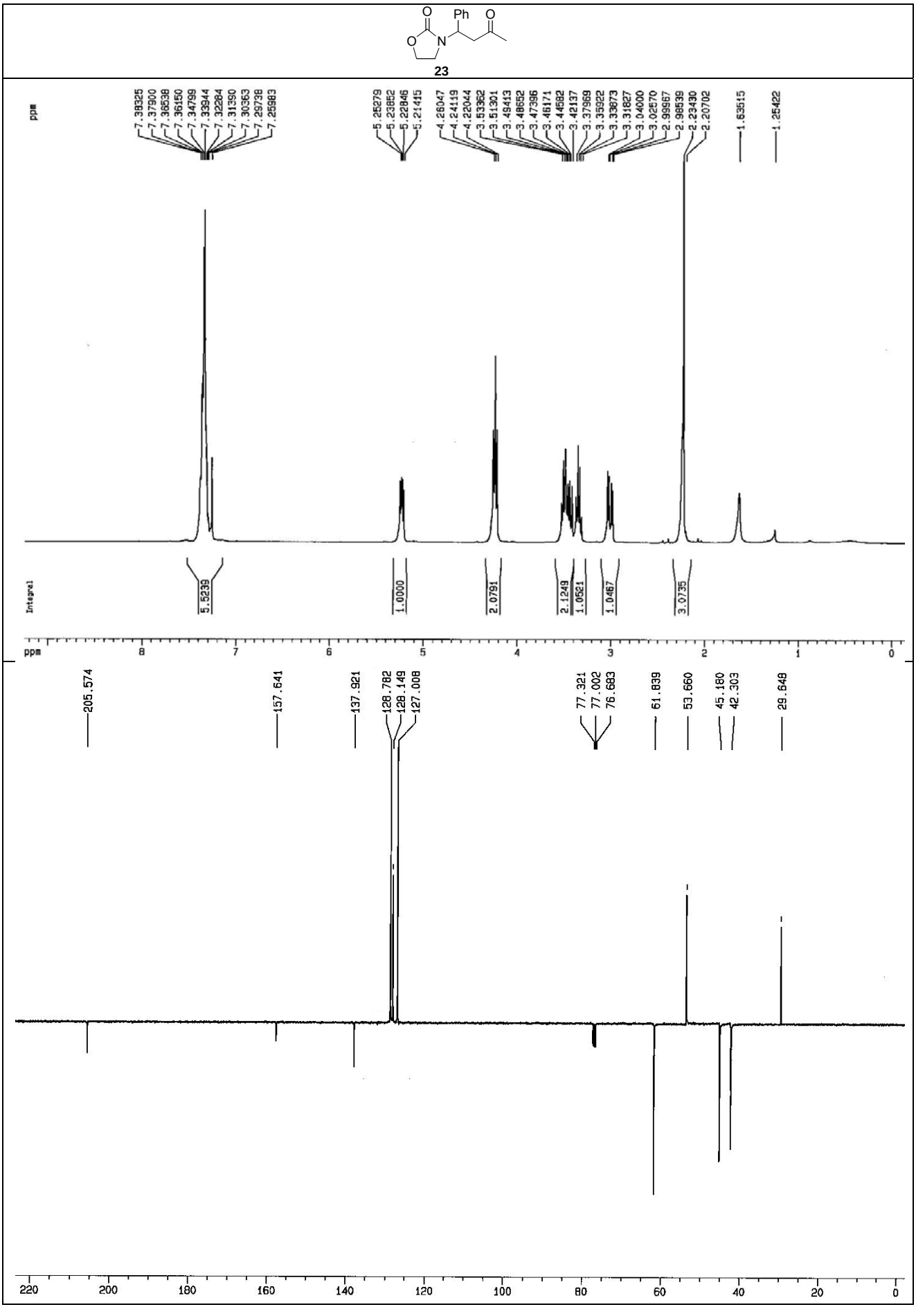
7.22131
7.20440
4.32734
4.30916
4.29098
2.97489
2.95671
2.38909
2.34037
2.32640
2.30895
2.29003
2.27004
2.25189
2.23863
2.22046
2.08542
1.50422
1.48483
1.46592
1.45580
1.44980
1.43788
1.43054
1.41985
1.33572
1.31783
1.30166
1.28335
1.26554
0.84772
0.82952
0.81118
0.00010
Integral
5.2650
1.0000
2.0642
2.1732
3.0649
2.1113
2.1037
3.1305
ppm
8
7
6
5
4
3
2
1
0
205.573
142.008
128.508
127.682
127.231
77.337
77.019
76.702
50.130
44.093
31.186
30.979
30.732
21.905
13.581
200
180
160
140
120
100
80
60
40
20


Ph
O
O
O
N
23
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7.36538
7.36150
7.34799
7.33944
7.32284
7.31350
7.30363
7.29738
7.25983
5.25279
5.23852
5.22846
5.21415
4.26047
4.24119
4.22044
3.53362
3.51301
3.49413
3.48652
3.47396
3.46171
3.44582
3.42137
3.37969
3.35922
3.33873
3.31827
3.04000
3.02570
2.99967
2.98539
2.23430
2.20702
1.63515
1.25422
Integral
5.5239
1.0000
2.0791
2.1249
1.0521
1.0467
3.0735
ppm
205.574
157.641
137.921
128.782
128.149
127.008
77.321
77.002
76.683
61.839
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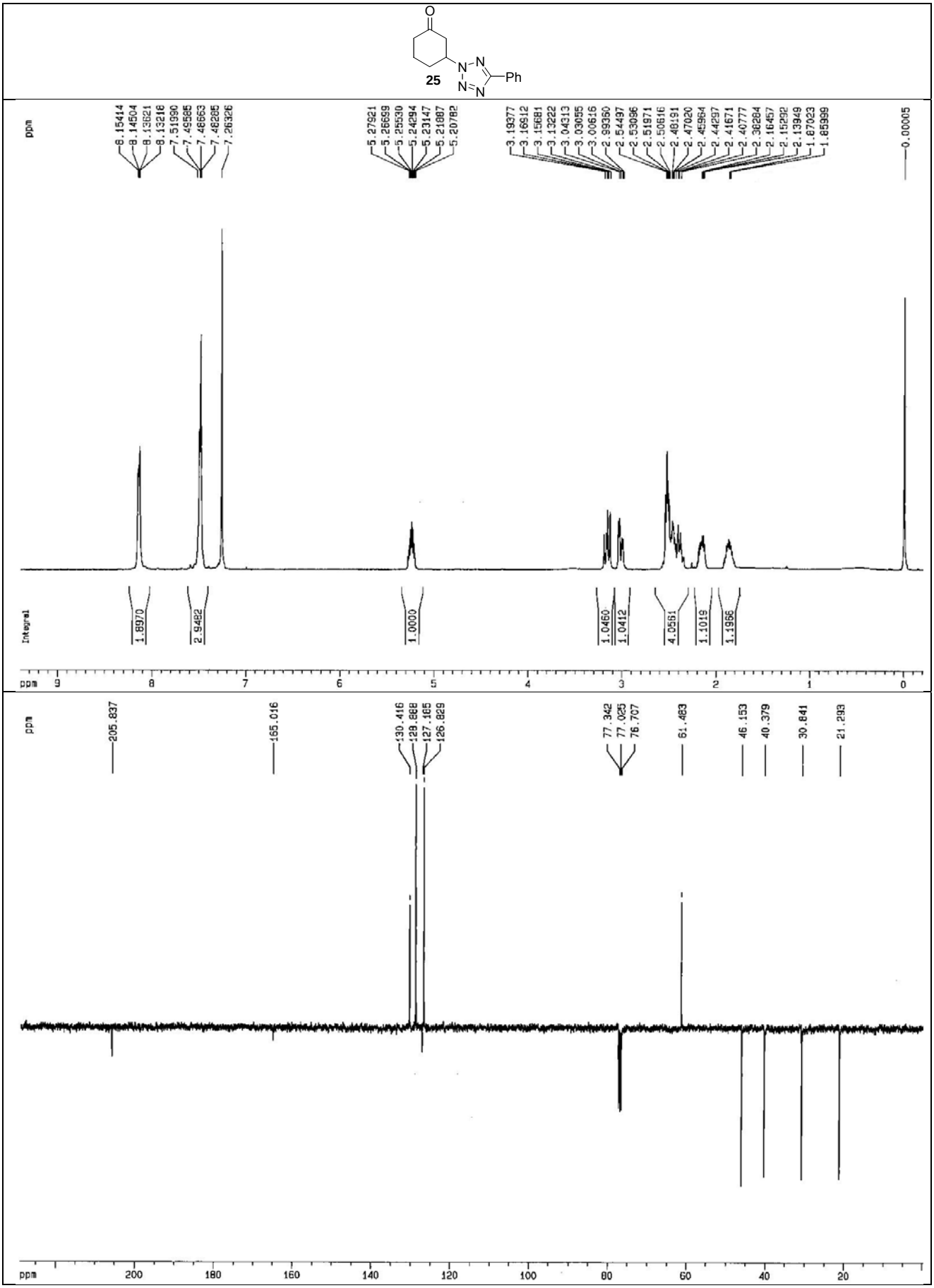
45.180
42.303
29.648


O
N
N
N
N
Ph
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ppm
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8.14504
8.13621
8.13216
7.51990
7.49585
7.48663
7.48285
7.26326
5.27921
5.26659
5.25530
5.24294
5.23147
5.21887
5.20782
3.19377
3.16512
3.15681
3.13222
3.04313
3.03055
3.00616
2.99350
2.54497
2.53096
2.51971
2.50616
2.48191
2.47020
2.45964
2.44297
2.41671
2.40777
2.38284
2.16457
2.15292
2.13949
1.87023
1.85999
0.00005
Integral
1.8970
2.9482
1.0000
1.0460
1.0412
4.0561
1.1019
1.1966
ppm 9 8 7 6 5 4 3 2 1 0
ppm
205.837
165.016
130.416
129.888
127.165
126.829
77.342
77.025
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61.483
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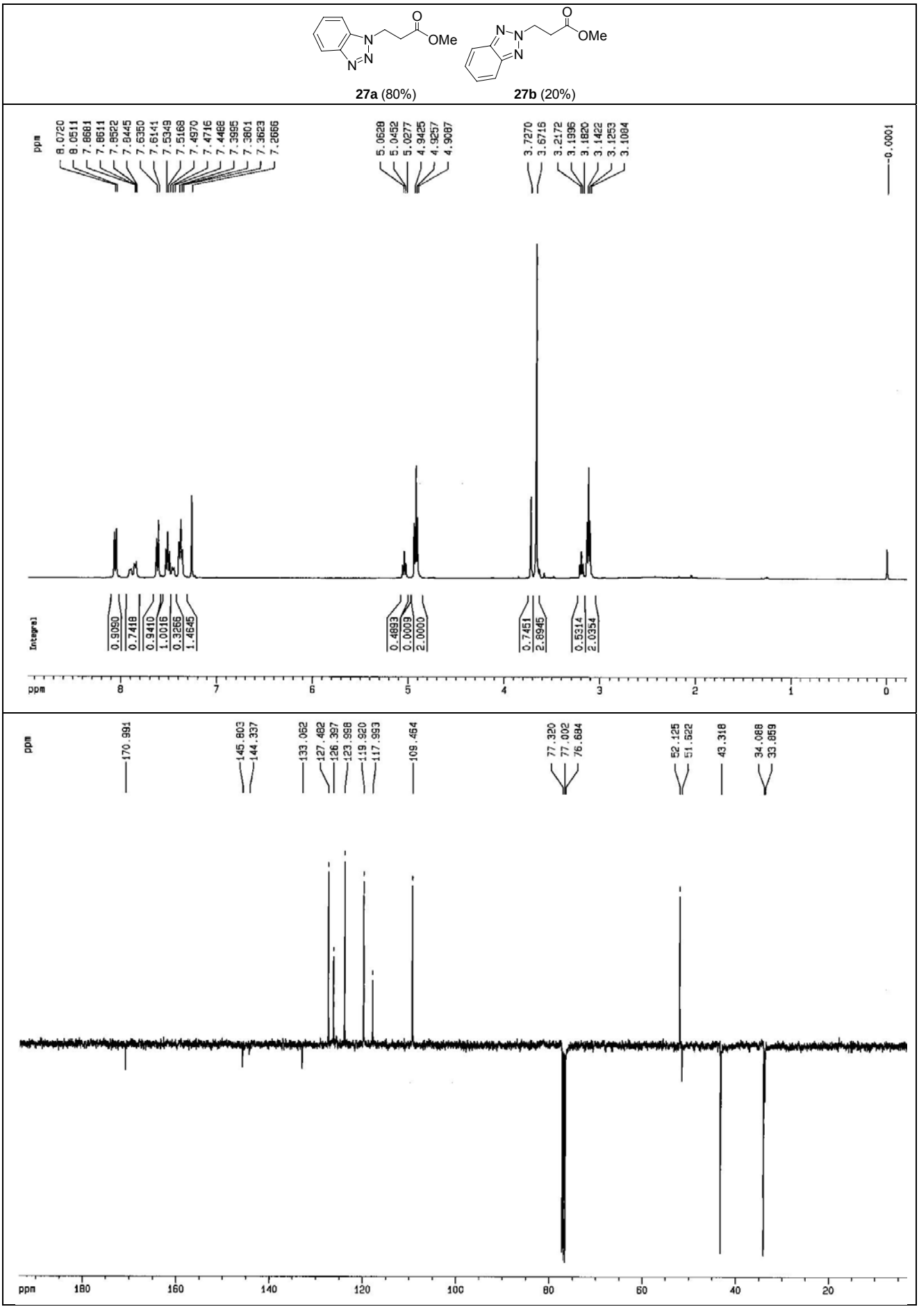
40.379
30.841
21.293
ppm 200 180 160 140 120 100 80 60 40 20


27a (80%)
27b (20%)
8.0720
8.0511
7.8881
7.8611
7.8522
7.8445
7.6350
7.6141
7.5349
7.5168
7.4970
7.4716
7.4488
7.3995
7.3801
7.3623
7.2666
5.0628
5.0452
5.0277
4.9425
4.9257
4.9087
3.7370
3.5715
3.2172
3.1996
3.1820
3.1422
3.1253
3.1084
-0.0001
Integral
0.9090
0.7418
0.9410
1.0016
0.3266
1.4645
0.4893
0.0009
2.0000
0.7451
2.8945
0.5314
2.0354
ppm
170.991
145.803
144.337
133.062
127.482
126.397
123.998
119.920
117.993
109.464
77.320
77.002
76.684
52.125
51.522
43.318
34.088
33.859

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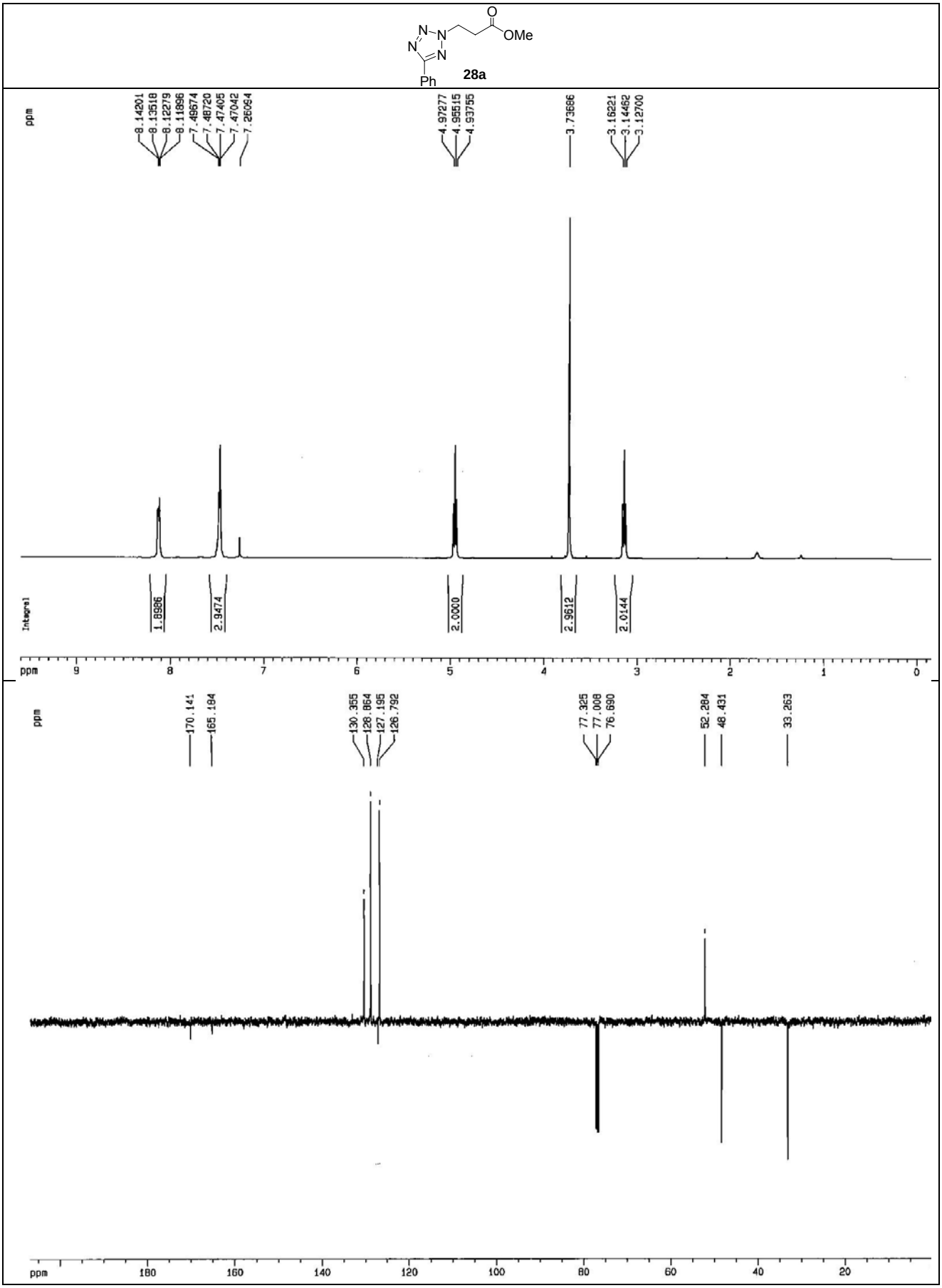

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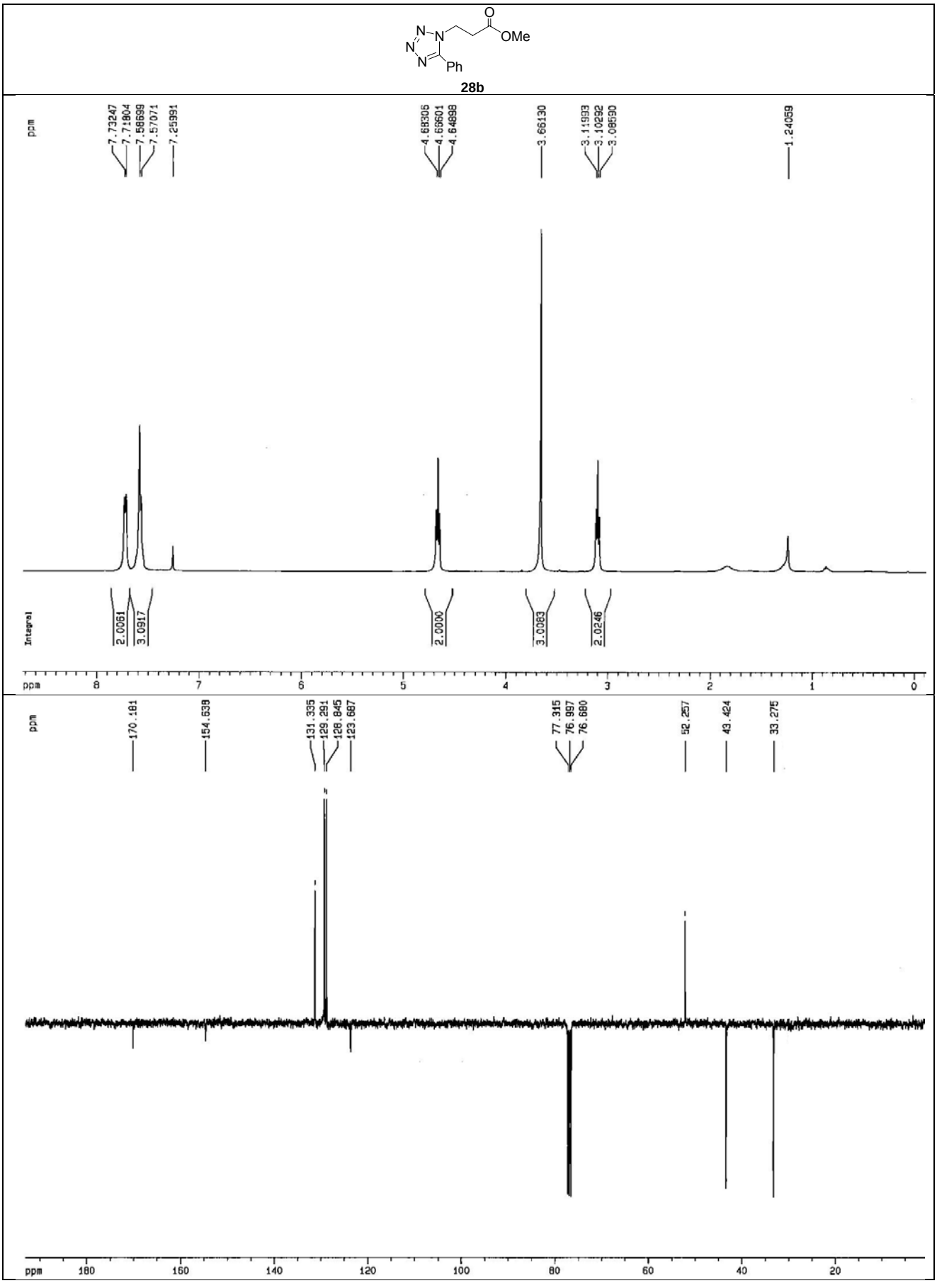

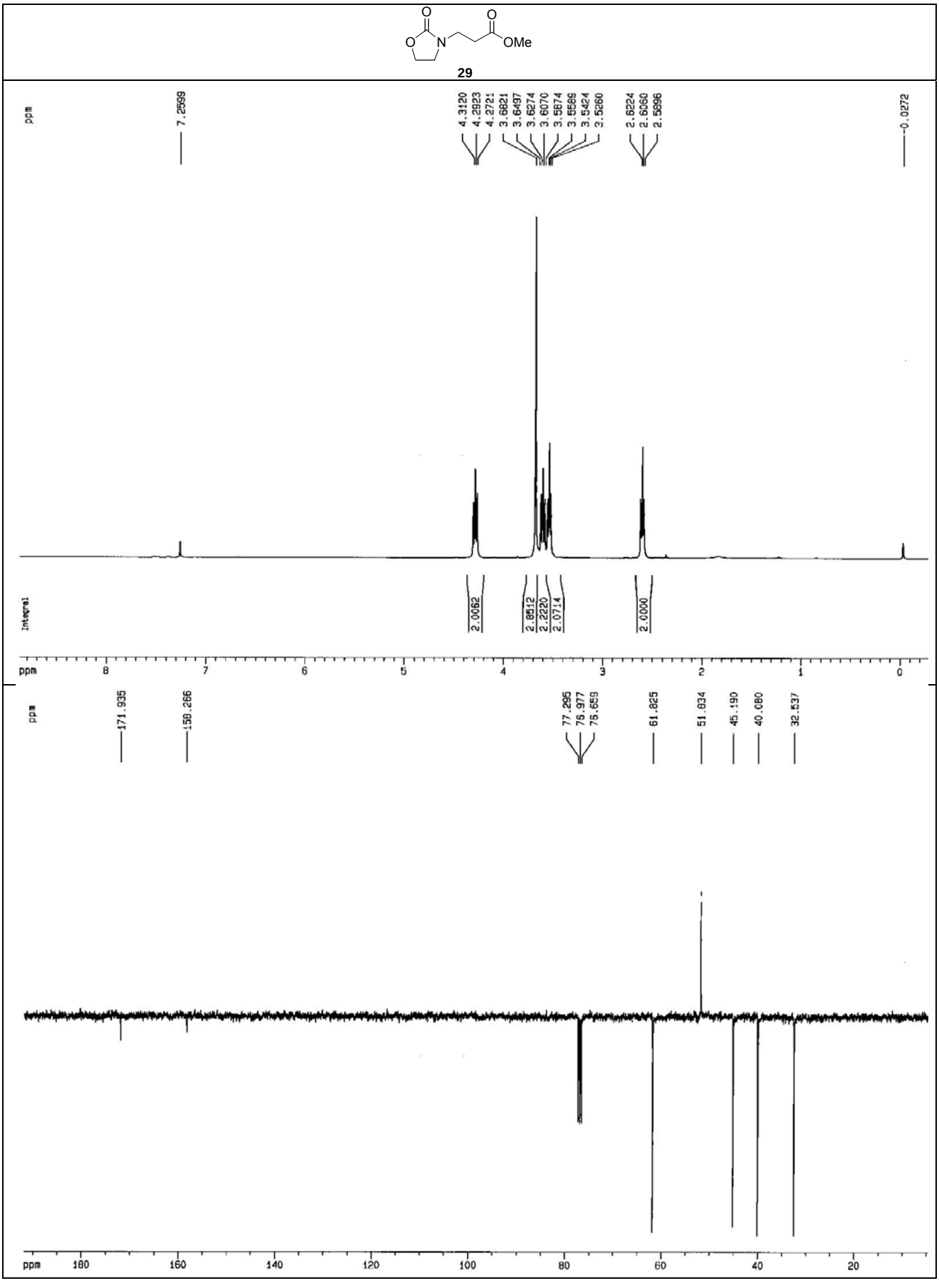
O
O
O
N
OMe
29
ppm
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4.2923
4.2721
3.6821
3.6497
3.6274
3.6070
3.5874
3.5589
3.5424
3.5260
2.6224
2.6060
2.5896
0.0272
Integral
2.0062
2.8512
2.2220
2.0714
2.0000
ppm
8
7
6
5
4
3
2
1
0
ppm
171.935
158.266
77.295
76.977
76.659
61.825
51.834
45.190
40.080
32.537
ppm
180
160
140
120
100
80
60
40
20