Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Enantioselective cyclopropanation of (*Z*)-3-substituted-2-(4-pyridyl)-acrylonitriles catalyzed by *Chincona* ammonium salts.

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Supporting Information

General Methods

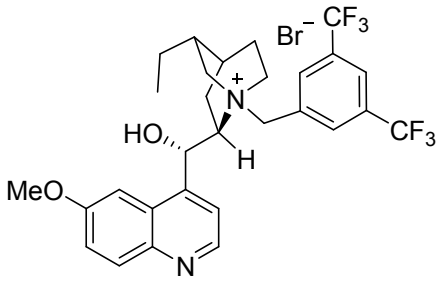
^{1}H, ^{13}C NMR spectra were recorded on a Bruker 400 instrument. Chemical shifts (δ) are reported in ppm relative to residual solvent signals for ^{1}H and ^{13}C NMR (^{1}H NMR: 7.26 ppm for $CDCl_3$, 3.34 ppm for CD_3OD; ^{13}C NMR: 77.0 ppm for $CDCl_3$, 49.00 ppm for CD_3OD). ^{13}C NMR spectra were acquired with ^{1}H broad band decoupled mode. Coupling constants (*J*) are in Hz. Multiplicities are reported as follows: s, singlet, d, doublet, dd, doublets of doublets, t, triplet, q, quartet, m, multiplet, c, complex, and br, broad. Mass spectra were recorded on a Micro mass LCT spectrometer using electrospray (ES) ionisation techniques. Optical rotations were measured on a Perkin-Elmer 241 polarimeter. The enantiomeric excess (*ee*) of the products was determined by chiral stationary phase CSP-HPLC (Daicel Chiralpak AD, Chiralcel OD and Chiralcel AS columns), using a UV detector operating at 254 nm and 210 nm. Infrared (IR) spectra were recorded as thin films between NaCl plates using a Bruker Tensor27 FT-IR instrument. Absorption maximum (νmax) was reported in wavenumbers (cm^{-1}) and only selected peaks are reported. The following abbreviations are used: w, weak, m, medium, s, strong and br, broad.

Materials

Analytical grade solvents and commercially available reagents were used as received, unless otherwise stated. Reactions were checked for completion by TLC (EM Science, silica gel 60 F254). Flash chromatography was performed using silica gel 60 (0.040-0.063 mm, 230-400 mesh). Racemic samples were prepared using tetrabutylammonium bromide as a catalyst at room temperature.

Experimental procedure for the preparation of catalyst *N*-(3,5-bis (trifluoromethyl)) benzyl hydroquinidinium bromide (11).

To a stirred suspension of hydroquinidine (2.00 g, 6.13 mmol) in acetone (35.0 mL), 3,5-bis(trifluoromethyl) benzyl bromide (1.18 ml, 6.44 mmol) was added. The resulting mixture was heated at 60 °C and stirred for 4 h at the same temperature. After cooling to r.t., the solvent was removed *in vacuo*. The residue was washed with EtO_2 affording the title compound as a yellow solid (3.76 g, 97% yield), m.p. 195^{o} (dec).



$[\alpha]_D^{20}$ = +350 (c = 0.50 in CH_3Cl); ^{1}H-NMR (400 MHz, CD_3OD): 8.79 (d, *J* = 4.8 Hz, 1H), 8.42 (s, 2H), 8.25 (s, 1H), 8.05 (d, *J* = 9.2 Hz, 1H), 7.93 (d, *J* = 4.8 Hz, 1H), 7.57 (dd, *J* = 9.4 Hz, 2.6 Hz, 1H), 7.49 (d, *J* = 2.4 Hz, 1H), 6.58 (d, *J* = 1.6 Hz, 1H), 5.25 (d, *J* = 12.4 Hz, 1H), 5.05 (d, *J* = 12.8 Hz, 1H), 4.25 (dt, J_d = 8.8 Hz, J_t = 2.8 Hz, 1H), 4.10 (s, 3H), 3.94 (t, *J* = 10.4 Hz, 2H), 3.54 (t, *J* = 9.6 Hz, 1H), 3.16-3.08 (m, 1H), 2.52 (t, *J* = 11.8 Hz, 1H), 1.98 (m, 1H), 1.90-1.79 (m, 3H), 1.71-1.59 (m, 2H), 1.14-1.10 (m, 1H), 0.95 (t, *J* = 7.6 Hz, 3H); ^{13}C-NMR (100.6 MHz): 160.2, 148.3, 145.5, 144.7, 135.4, 133.8, 133.5, 132.1, 131.9, 127.5, 125.9, 125.6, 123.1, 122.6, 121.7, 103.4, 69.9, 67.0, 63.4, 58.3, 58.2, 56.6, 36.8, 30.6, 25.9, 25.3, 25.2, 22.2, 11.6; HRMS: *m/z* found $[M\text{-}Br]^+$ 553.2275, $C_{29}H_{31}F_6N_2O_2$ requires 553.2284.

Catalysts **4a-j** have been prepared according to the procedure reported in the literature.[1]
Catalyst **4k** has been prepared according to the procedure reported in the literature.[2]

Experimental procedure for the preparation of diethyl-2-bromomalonate (2b)

A solution of bromide (1.3 mL, 25.2 mmol, 1.10 eq) in CCl_4 (16mL) was added dropwise to a stirred mixture of diethyl bromomalonate (3.8 mL, 25.0 mmol, 1.0 eq) and CCl_4 (10 mL) in a two-neck flask equipped with a reflux condenser. After six drops of the bromine solution had been added, the reaction was initiated by irradiating the flask with a 100W incandescent bulb. The remaining bromide solution was added over 15 min to maintain an orange-red color in the reaction mixture. Then the reaction mixture was refluxed for 6h at 100 °C. The reaction mixture was diluted with an equal volume of CCl_4, washed with 5% $NaHCO_3$ (2 x 50 mL), dried over $MgSO_4$, filtered and evaporated *in vacuo*. The residue was purified by flash chromatography on silica gel eluting with Petroleum Ether/AcOEt 95:5 affording the product as a colourless liquid (5.04 g, 84% yield).

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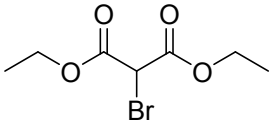
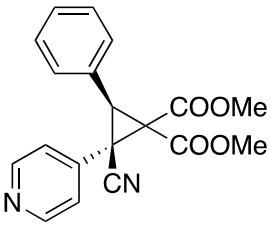
[1] C. Del Fiandra, M. Moccia, V. Cerulli, M. F. A. Adamo, *Chem. Commun.* **2016**, *52*(8), 1697 doi: 10.1039/C5CC08105J.
[2] T. Furukawa, N. Shibata, S. Mizuta, S. Nakamura, T. Toru, M. Shiro, *Angew. Chem. Int. Ed.* **2008**, *47*, 8051.



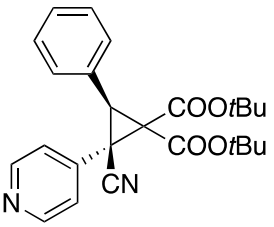
^{1}H-NMR (400 MHz, $CDCl_3$): 4.81 (s, 1H), 4.28 (q, *J* = 7.2 Hz, 4H), 1.30 (t, *J* = 7.2 Hz, 6H); ^{13}C-NMR (100.6 MHz): 164.7, 63.3, 42.5, 14.0; HRMS: *m/z* found $[M+H]^+$ 238.9935, $C_7H_{11}BrO_4$ requires 237.9841.

(2*R*,3*R*)-dimethyl 2-cyano-3-phenyl-2-(pyridin-4-yl)cyclopropane-1,1-dicarboxylate (3)



Reaction time: 120 h. Column chromatography (Solvents DCM:Et_2O 7:3) afforded **3** as a yellow crystals (30.3 mg, 90% yield) m.p. 102-105 ^{o}C. The *ee* of the product was determined by CSP-HPLC using a Chiralpak AS column (*n*-hexane/*i*-PrOH 90:10, flow rate 0.75 mL/min, t_{maj} = 26.28 min, t_{min} = 31.42 min, 78% *ee*); $[\alpha]_D^{20}$ = +35 (c = 0.29 in $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): 8.69 (s, 2H), 7.48-7.38 (m, 7H), 4.06 (s, 1H), 3.75 (s, 3H), 3.51 (s, 3H); ^{13}C-NMR (100.6 MHz): 164.1, 163.4, 150.5, 141.5, 130.2, 129.0, 128.9, 123.7, 115.8, 63.0, 47.5, 35.8, 31.0, 13.9; IR (NaCl) / cm^{-1}: 2241 s, 1734 s; HRMS: *m/z* found $[M]^+$ 336.1121, $C_{19}H_{16}N_2O_4$ requires 336.1110.

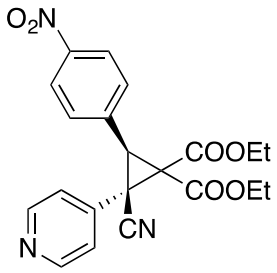
(2*R*,3*R*)-di-*tert*-butyl 2-cyano-3-phenyl-2-(pyridin-4-yl)cyclopropane-1,1-dicarboxylate (13)



Reaction time: 120 h. Column chromatography (Solvents DCM:Et_2O 7:3) afforded **13** as a yellow oil (37.7 mg, 90% yield). The *ee* of the product was determined by CSP-HPLC using a Chiralpak AD column (*n*-hexane/*i*-PrOH 90:10, flow rate 0.50 mL/min, t_{maj} = 13.65 min, t_{min} = 19.05 min, 64% *ee*); $[\alpha]_D^{20}$ = +37 (c = 0.40 in $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): 8.69 (s, 2H), 7.48-7.38 (m, 7H), 4.06 (s, 1H), 1.49 (s, 9H), 1.11 (s, 9H); ^{13}C-NMR (100.6 MHz): 169.4, 166.0, 150.5, 141.5, 130.2, 129.0, 128.9, 123.7, 115.8, 81.6, 80.8, 47.5, 35.8, 31.0, 13.9; IR (NaCl) / cm^{-1}: 2236 s, 1728 s; HRMS: *m/z* found $[M]^+$ 420.2156, $C_{25}H_{28}N_2O_4$ requires 420.2049.

Prepared following general procedure using (*Z*)-3-(2,3-dihydrobenzo[b][1,4]dioxin-5-yl)-2-(4-pyridyl)acrylonitrile **1i** (26.4 mg, 0.10 mmol) and catalyst **11**. Reaction time: 144 h. Column chromatography (Solvents DCM:Et_2O 7:3) afforded **12i** as a yellow oil (32.3 mg, 76% yield). The *ee* of the product was determined by CSP-HPLC using a Chiralcel OD column (*n*-hexane/*i*-PrOH 80:20, flow rate 1.00 mL/min, t_{maj} = 24.98 min, t_{min} = 40.40 min, 60% *ee*); $[\alpha]_D^{20}$ = +27 (c = 0.25 in $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): 8.66 (d, *J* = 6.0 Hz, 2H), 7.40 (d, *J* = 6.2 Hz, 2H), 6.98-6.86 (m, 3H), 4.26 (s, 6H), 4.00-3.89 (m, 3H), 1.24 (t, *J* = 7.2 Hz, 3H), 1.00 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (100.6 MHz): 164.1, 163.4, 150.6, 144.2, 143.9, 141.3, 123.6, 123.0, 122.1, 118.1, 117.9, 115.8, 64.5, 64.4, 63.1, 63.0, 47.4, 35.3, 31.2, 13.9, 13.8; IR (NaCl) / cm^{-1}: 2237 s, 1742 s; HRMS: *m/z* found $[M+H]^+$ 423.1561, $C_{23}H_{23}N_2O_6$ requires 423.1556.

(2*R*,3*R*)-Diethyl 2-cyano-3-(4-nitrophenyl)-2-(pyridin-4-yl)cyclopropane-1,1-dicarboxylate (12k)

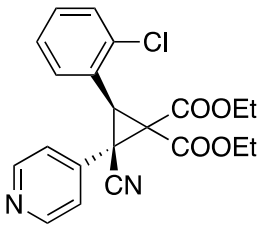


Prepared following general procedure using (*Z*)-3-(4-nitrophenyl)-2-(pyridin-4-yl)acrylonitrile **1k** (25.1 mg, 0.10 mmol) and catalyst **11**. Reaction time: 192 h. Column

chromatography (Solvents DCM:Et_2O 7:3) afforded **12k** as a wine red oil (29.4 mg, 72% yield).

The *ee* of the product was determined by CSP-HPLC using a Chiralcel OD column (*n*-hexane/*i*-PrOH 90:10, flow rate 1.00 mL/min, t_{maj} = 57.53 min, t_{min} = 49.23 min, 16% *ee*); $[\alpha]_D^{20}$ = +17 (c = 0.28 in $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): 8.72 (d, *J* = 5.6 Hz, 2H), 8.29 (d, *J* = 8.7 Hz, 2H), 7.69 (d, *J* = 8.9 Hz, 2H), 7.47 (d, *J* = 6.1 Hz, 2H), 4.29-4.21 (m, 2H), 4.09 (s, 1H), 4.05-3.93 (m, 2H), 1.23 (t, *J* = 7.2 Hz, 3H), 1.04 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (100.6 MHz): 163.5, 162.8, 150.2, 148.2, 141.1, 137.4, 130.3, 124.2, 123.6, 115.1, 63.6, 63.5, 47.6, 35.2, 31.2, 13.9, 13.8; IR (NaCl) / cm^{-1}: 2245 s, 1740 s; HRMS: *m/z* found $[M+Na]^+$ 432.1172, $C_{21}H_{19}N_3O_6$ requires 409.1274.

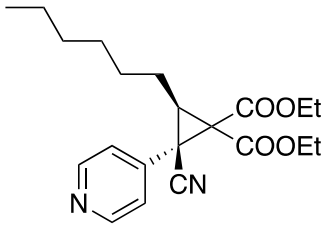
(2*R*,3*R*)-Diethyl 3-(2-chlorophenyl)-2-cyano-2-(pyridin-4-yl)cyclopropane-1,1-dicarboxylate (12l)



Prepared following general procedure using (*Z*)-3-(2-chlorophenyl)-2-(pyridin-4-yl)acrylonitrile **1l** (24.1 mg, 0.10 mmol) and catalyst **11**. Reaction time: 144 h. Column chromatography (Solvents DCM:Et_2O 7:3) afforded **12l** as a yellow oil (27.5 mg, 69% yield).

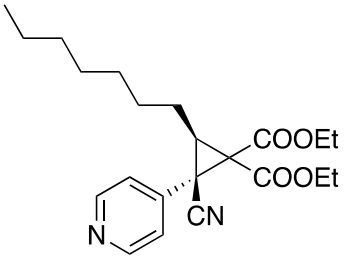
The *ee* of the product was determined by CSP-HPLC using a Chiralpak AS column (*n*-hexane/*i*-PrOH 95:5, flow rate 0.75 mL/min, t_{maj} = 21.76 min, t_{min} = 29.11 min, 52% *ee*); $[\alpha]_D^{20}$ = +5 (c = 0.35 in $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): 8.69 (d, *J* = 5.9 Hz, 2H), 7.55-7.47 (m, 4H), 7.38-7.29 (m, 2H), 4.26 (qd, *J* = 7.1, 1.7 Hz, 2H), 4.09 (s, 1H), 4.01-3.84 (m, 2H), 1.21 (t, *J* = 7.1 Hz, 3H), 0.98 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (100.6 MHz): 163.8, 163.4, 149.9, 141.6, 135.7, 130.3, 130.2, 129.9, 128.7, 127.2, 123.8, 115.6, 63.2, 63.1, 47.6, 34.8, 31.7, 13.8, 13.7; IR (NaCl) / cm^{-1}: 2235 s, 1742 s; HRMS: *m/z* found $[M+Na]^+$ 421.0918, $C_{21}H_{19}ClN_2O_4$ requires 398.1033.

(2*R*,3*R*)-Diethyl 2-cyano-3-hexyl-2-(pyridin-4-yl)cyclopropane-1,1-dicarboxylate (12m)



Prepared following general procedure using (*Z*)-2-(4-pyridyl)non-2-enenitrile **1m** (21.4 mg, 0.10 mmol) and catalyst **11**. Reaction time: 168 h. Column chromatography (Solvents DCM:Et_2O 7:3) afforded **12m** as a yellow oil (34.3 mg, 92% yield). The *ee* of the product was determined by CSP-HPLC using a Chiralpak AD column (*n*-hexane/*i*-PrOH 90:10, flow rate 0.75 mL/min, t_{maj} = 9.86 min, t_{min} = 8.79 min, 82% *ee*); $[\alpha]_D^{20}$ = +32 (c = 0.45 in $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): 8.62 (d, *J* = 6.0 Hz, 2H), 7.29 (d, *J* = 4.4 Hz, 2H), 4.36-4.26 (m, 2H), 3.95-3.87 (m, 2H), 2.70 (t, *J* = 7.2 Hz, 1H), 1.64-1.59 (m, 1H), 1.45-1.41 (m, 2H), 1.37-1.29 (m, 10H), 0.97 (t, *J* = 7.2 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C-NMR (100.6 MHz): 164.3, 163.8, 150.4, 129.1, 123.7, 116.1, 63.3, 62.8, 46.3, 42.5, 32.7, 31.6, 31.0, 29.0, 28.3, 26.2, 22.6, 14.1, 14.0; IR (NaCl) / cm^{-1}: 2243 s, 1744 s; HRMS: *m/z* found $[M+H]^+$ 373.2134, $C_{21}H_{29}N_2O_4$ requires 373.2127.

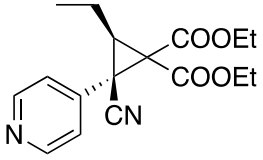
(2*R*,3*R*)-Diethyl 2-cyano-3-heptyl-2-(pyridin-4-yl)cyclopropane-1,1-dicarboxylate (12n)



Prepared following general procedure using (*Z*)-2-(4-pyridyl)dec-2-enenitrile **1n** (22.8 mg, 0.10 mmol) and catalyst **11**. Reaction time: 192 h. Column chromatography (Solvents DCM:Et_2O 7:3) afforded **12n** as a yellow oil (27.0 mg, 70% yield). The *ee* of the product was determined by CSP-HPLC using a Chiralpak AD column (*n*-hexane/*i*-PrOH 90:10, flow rate 0.75 mL/min, t_{maj} = 9.64 min, t_{min} = 8.35 min, 83% *ee*); $[\alpha]_D^{20}$ = +16 (c = 0.22 in $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): 8.62 (d, *J* = 4.4 Hz, 2H), 7.29 (d, *J* = 4.4 Hz, 2H), 4.40-4.31 (m, 2H), 3.94-3.84 (m, 2H), 2.70 (t, *J* = 7.4 Hz, 1H), 1.90-1.86 (m, 1H), 1.73-1.59 (m, 4H), 1.47-1.42 (m, 2H), 1.35 (t, *J* = 7.0 Hz, 3H), 1.31-1.29 (m, 5H), 0.97 (t, *J* = 7.2 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C-NMR (100.6 MHz): 164.3, 163.8, 150.4, 140.9, 123.7, 116.1, 62.9, 62.8,

46.3, 32.7, 31.8, 31.0, 29.3, 29.2, 28.4, 26.3, 22.7, 14.2, 14.1, 13.7; IR (NaCl) / cm^{-1}: 2245 s, 1746 s; HRMS: *m/z* found $[M+H]^+$ 387.2288, $C_{22}H_{31}N_2O_4$ requires 387.2284.

(2*R*,3*R*)-Diethyl 2-cyano-3-ethyl-2-(pyridin-4-yl)cyclopropane-1,1-dicarboxylate (12o)

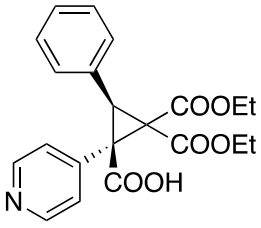


Prepared following general procedure using (*Z*)-2-(pyridin-4-yl)pent-2-enenitrile **1o** (15.8 mg, 0.10 mmol) and catalyst **11**. Reaction time: 192 h. Column chromatography (Solvents DCM:Et_2O 7:3) afforded **12o** as a yellow oil (19.8 mg, 68% yield).

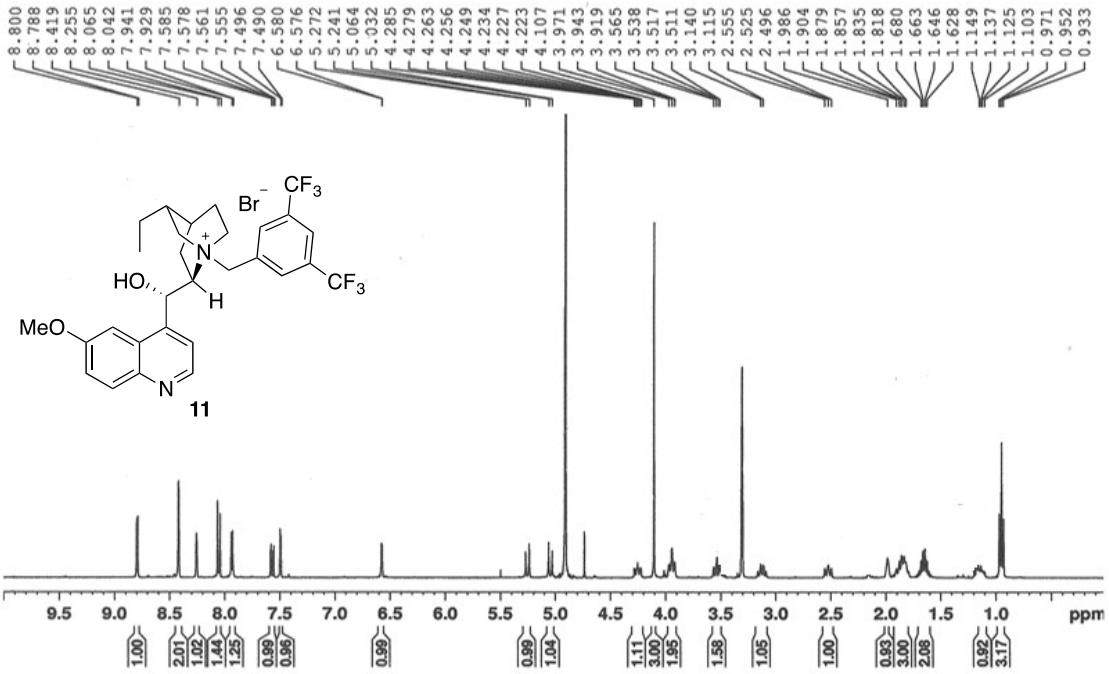
The *ee* of the product was determined by CSP-HPLC using a Chiralpak AD column (*n*-hexane/*i*-PrOH 95:5, flow rate 0.50 mL/min, t_{maj} = 16.02 min, t_{min} = 15.03 min, 21% *ee*); $[\alpha]_D^{20}$ = +10 (c = 0.20 in $CHCl_3$); ^{1}H-NMR (400 MHz, $CDCl_3$): 8.62 (d, *J* = 6.1 Hz, 2H), 7.30 (d, *J* = 6.2 Hz, 2H), 4.41-4.31 (m, 2H), 3.95-3.85 (m, 2H), 2.67 (dd, *J* = 8.7, 6.2 Hz, 1H), 1.98-1.89 (m, 1H), 1.80-1.69 (m, 1H), 1.35 (t, *J* = 7.1 Hz, 3H), 1.25 (t, *J* = 7.4 Hz, 3H), 0.98 (t, *J* = 7.1 Hz, 3H); ^{13}C-NMR (100.6 MHz): 164.3, 163.8, 150.5, 141.0, 123.7, 116.0, 62.9, 62.8, 46.5, 34.2, 30.9, 19.9, 14.1, 13.8, 12.7; IR (NaCl) / cm^{-1}: 2240 s, 1736 s; HRMS: *m/z* found $[M+H]^+$ 317.1516, $C_{17}H_{20}N_2O_4$ requires 316.1423.

Procedure for the preparation of 3-phenyl-2-pyridin-4-yl-cyclopropane-1,1,2-tricarboxylic acid diethyl ester 16

To a solution of (2*R*,3*R*)-diethyl 2-cyano-3-phenyl-2-(4-pyridyl)cyclopropane-1,1-dicarboxylate **12a** (30.0 mg, 0.09 mmol, 1.0 eq) in 500 µL of water, NaOH was added (16.0 mg, 0.4 mmol, 4.5 eq) and the mixture was refluxed for 24 h. After cooling to 0 °C, concentrated hydrochloric acid was added until the solution was neutral and the solvent was removed *in vacuo* affording the title compound **16** as a white solid in 98% yield (28.5 mg). m.p. 127-129^{o}C.



$[\alpha]_D^{20}$ = +26 (c = 0.50 in $CHCl_3$); ^{1}H-NMR (400 MHz, CD_3OD): 8.51 (d, *J* = 4.8 Hz, 2H), 7.71 (d, *J* = 4.8 Hz, 1H), 7.66-7.61 (m, 2H), 7.55-7.49 (m, 1H), 7.40-7.37 (m, 1H), 7.35 (t, *J* = 7.2 Hz, 1H), 7.28 (t, *J* = 7.4 Hz, 1H), 4.21 (dq, *J* = 7.2 Hz, *J* = 1.6 Hz, 2H), 4.01-3.90 (m, 2H), 3.37 (s, 1H), 1.16 (t, *J* = 7.2 Hz, 3H), 1.01 (t, *J* = 7.2 Hz, 3H); ^{13}C-NMR (100.6 MHz): 185.1, 174.9, 174.5, 150.1, 144.2, 134.5, 130.2, 129.8, 129.1, 126.7, 63.1, 63.0, 54.8, 51.8, 45.4, 13.9, 13.8; IR (NaCl) / cm^{-1}: 2239, 1724 s, 1705 s; HRMS: *m/z* found $[M+H]^+$ 384.1441, $C_{21}H_{22}NO_6$ requires 384.1447.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5.064
5.032
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4.256
4.249
4.234
4.227
4.223
4.107
3.971
3.943
3.919
3.565
3.538
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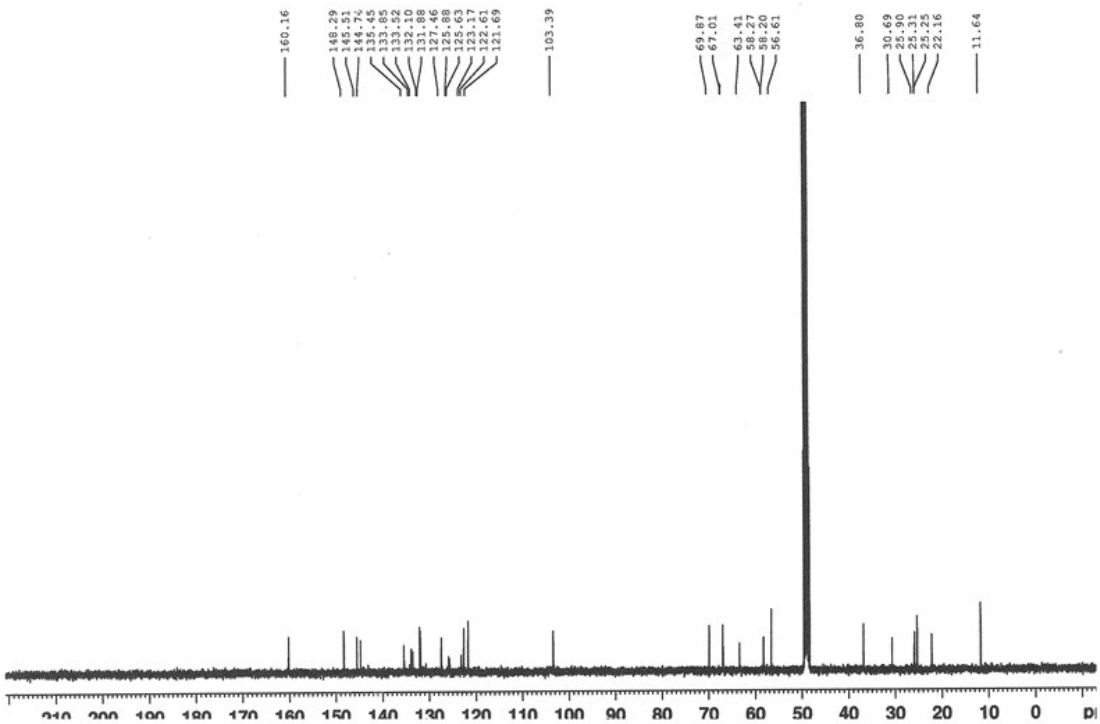
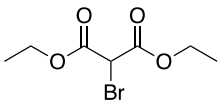
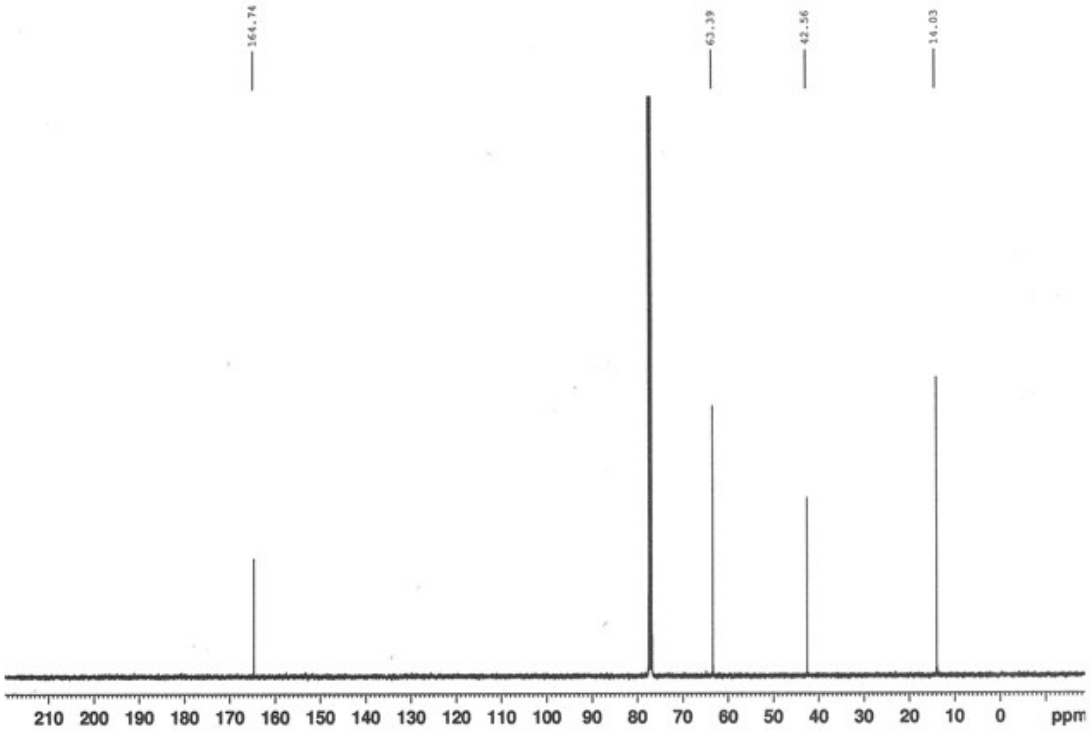
3.140
3.115
2.555
2.525
2.496
1.986
1.904
1.879
1.857
1.835
1.818
1.680
1.663
1.646
1.628
1.149
1.137
1.125
1.103
0.971
0.952
0.933
Br
CF3
CF3
HO
H
MeO
N
N
11
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
2.01
1.02
1.44
1.25
0.99
0.96
0.99
0.99
1.04
1.11
3.00
1.95
1.58
1.05
1.00
0.83
3.00
2.08
0.92
3.17

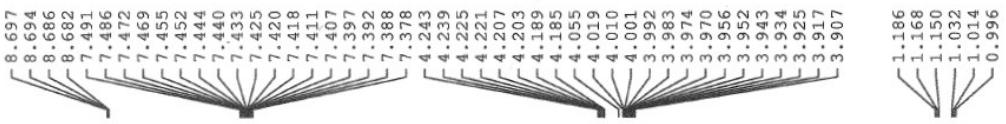

160.16
148.29
145.51
144.74
135.45
133.85
133.52
132.10
131.88
127.46
125.88
125.63
123.17
122.61
121.69
103.39
69.87
67.01
63.41
58.27
58.20
56.61
36.80
30.69
25.90
25.31
25.25
22.16
11.64
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0



2b

4.814
4.314
4.296
4.278
4.260
1.324
1.306
1.288
9.5 9.0 8.5 8.0 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
1.00
4.00
6.16


164.74
63.39
42.56
14.03
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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8.686
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7.486
7.472
7.469
7.455
7.452
7.444
7.440
7.433
7.425
7.420
7.418
7.411
7.407
7.397
7.392
7.388
7.378
4.243
4.239
4.225
4.221
4.207
4.203
4.189
4.185
4.055
4.019
4.010
4.001
3.992
3.983
3.974
3.970
3.956
3.952
3.943
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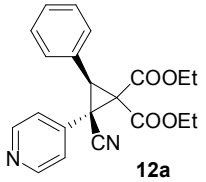
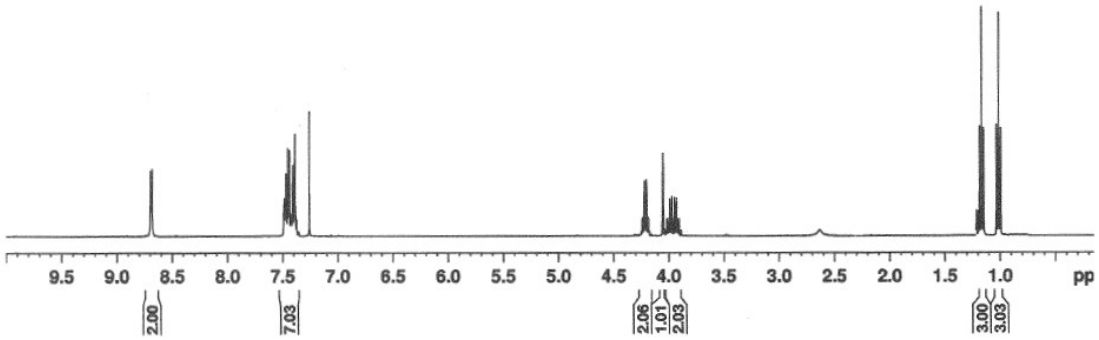
3.925
3.917
3.907
1.186
1.168
1.150
1.032
1.014
0.996


COOEt
COOEt
CN
N

12a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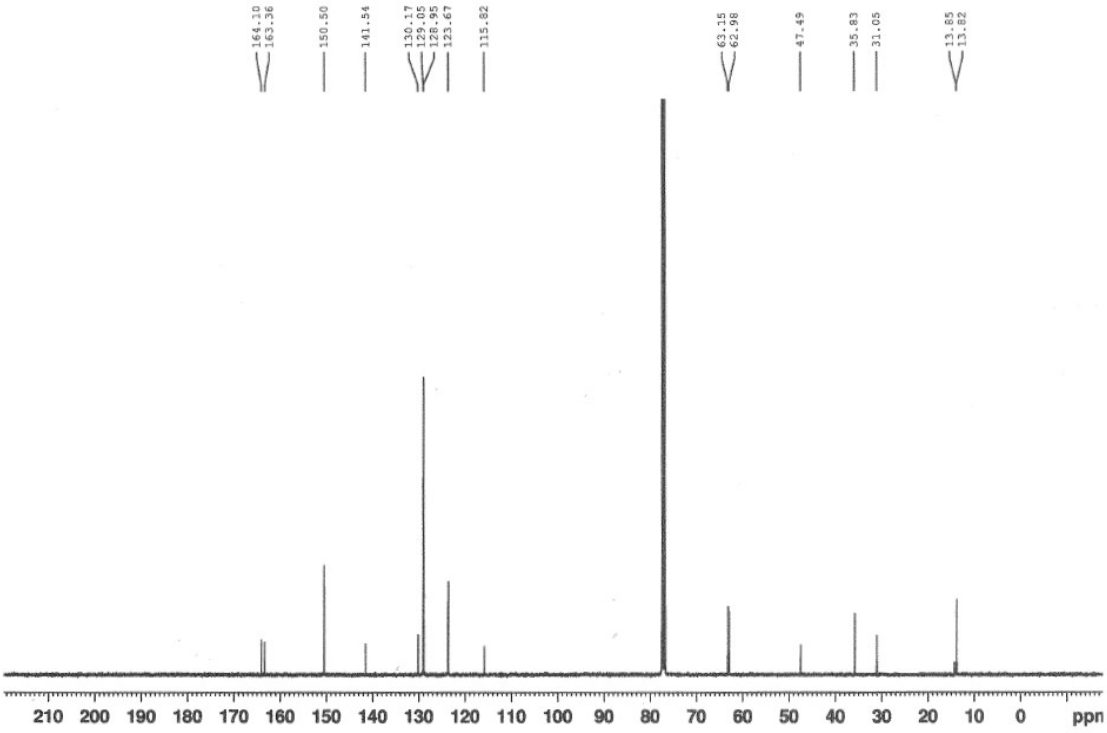
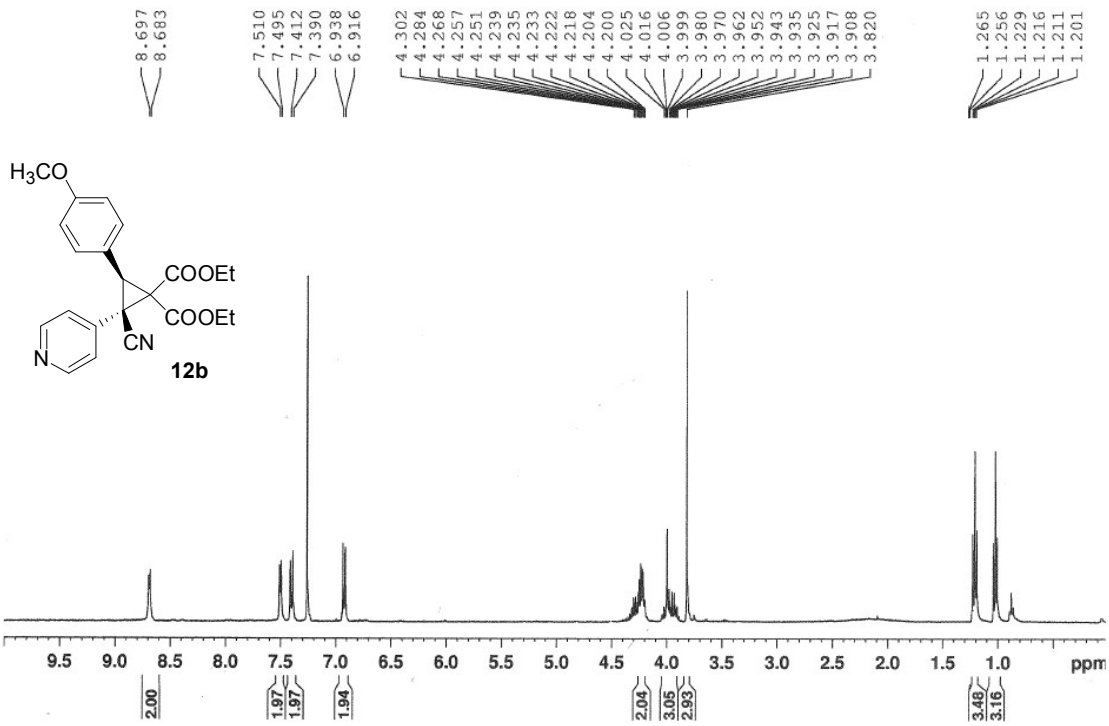
7.03
2.06
1.01
2.03
3.00
3.03


164.10
163.36
150.50
141.54
130.17
129.05
128.95
123.67
115.82
63.15
62.98
47.49
35.83
31.05
13.85
13.82
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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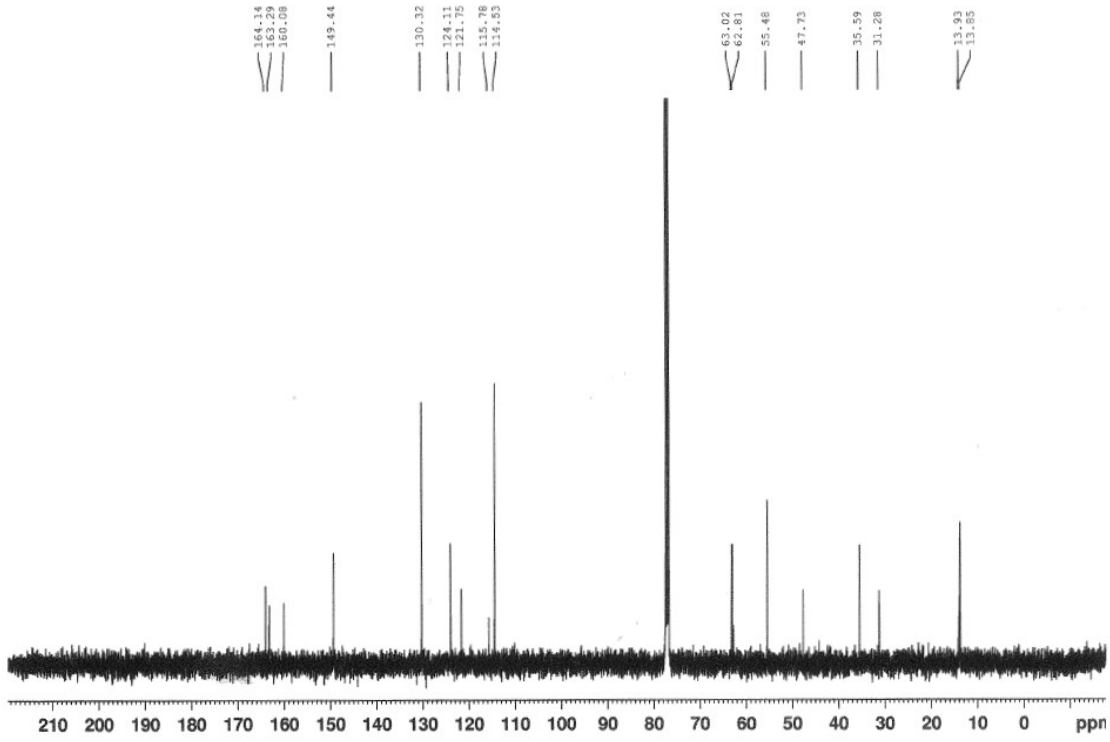
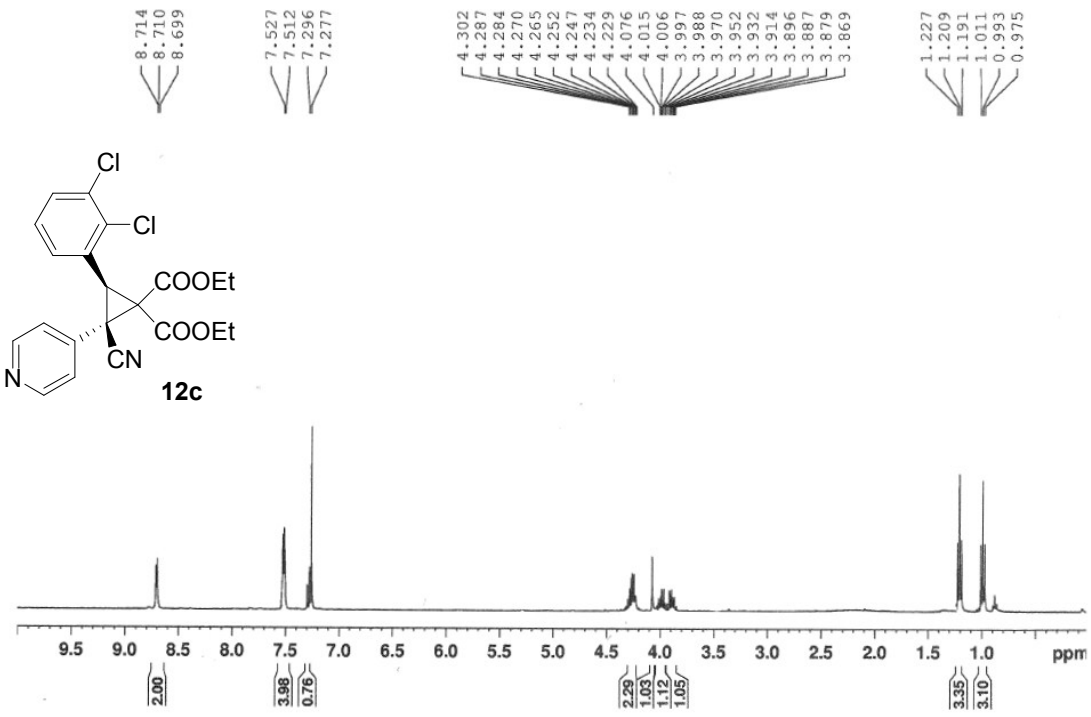
8.683
7.510
7.495
7.412
7.390
6.938
6.916
4.302
4.284
4.268
4.257
4.251
4.239
4.235
4.233
4.222
4.218
4.204
4.200
4.025
4.016
4.006
3.999
3.980
3.970
3.962
3.952
3.943
3.935
3.925
3.917
3.908
3.820
1.265
1.256
1.229
1.216
1.211
1.201
H3CO
COOEt
COOEt
CN
N
12b
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.00
1.97
1.97
1.94
2.04
3.05
2.93
3.48
3.16


164.14
163.29
160.08
149.44
130.32
124.11
121.75
115.78
114.53
63.02
62.81
55.48
47.73
35.59
31.28
13.93
13.85
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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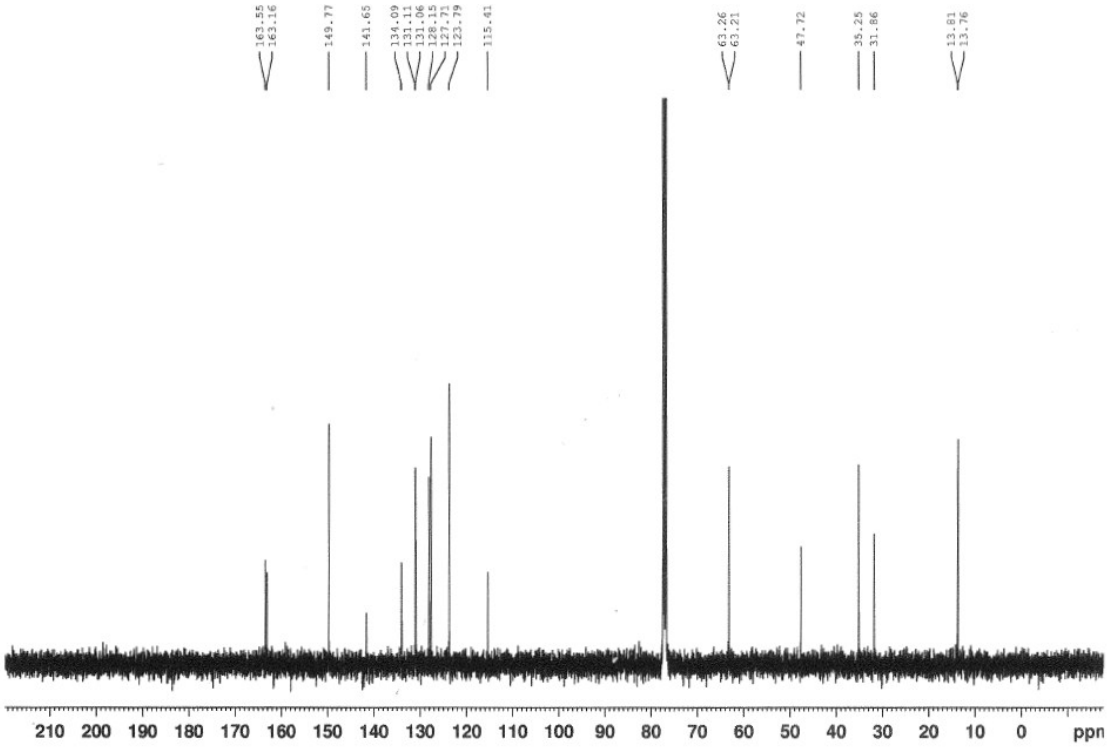
8.710
8.699
7.527
7.512
7.296
7.277
4.302
4.287
4.284
4.270
4.265
4.252
4.247
4.234
4.229
4.076
4.015
4.006
3.997
3.988
3.970
3.952
3.932
3.914
3.896
3.887
3.879
3.869
1.227
1.209
1.191
1.011
0.993
0.975
Cl
Cl
COOEt
COOEt
CN
N
12c
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm
2.00
3.98
0.76
2.29
1.03
1.12
1.05
3.35
3.10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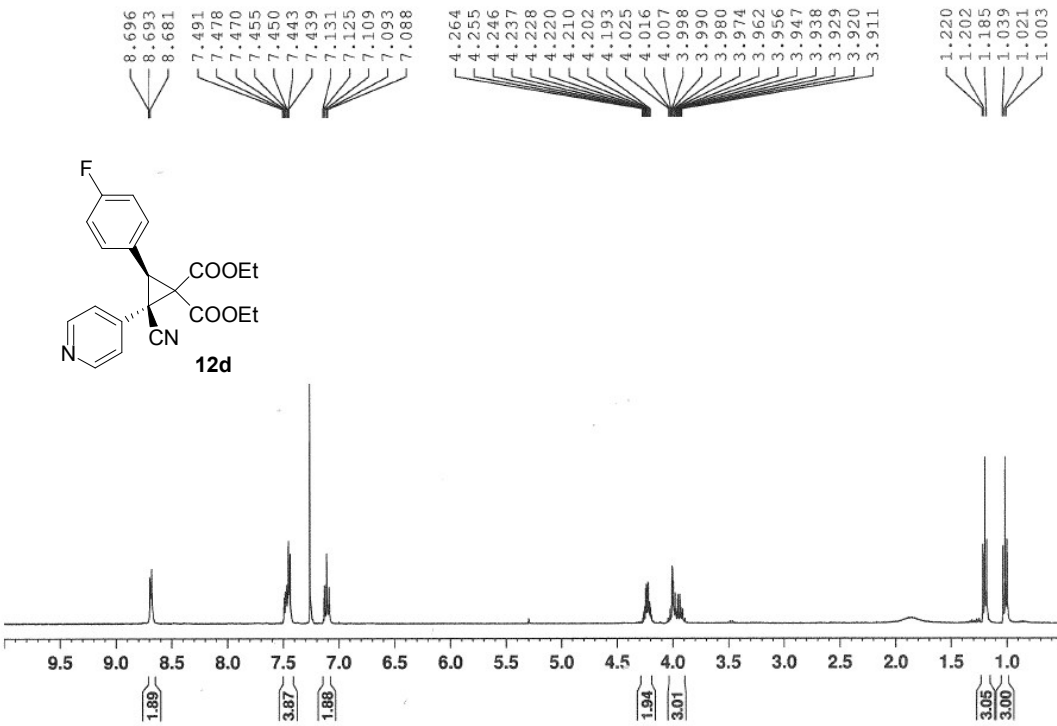
163.16
149.77
141.65
134.09
131.11
130.96
128.15
127.71
123.79
115.41
63.26
63.21
47.72
35.25
31.86
13.81
13.76
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm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8.681
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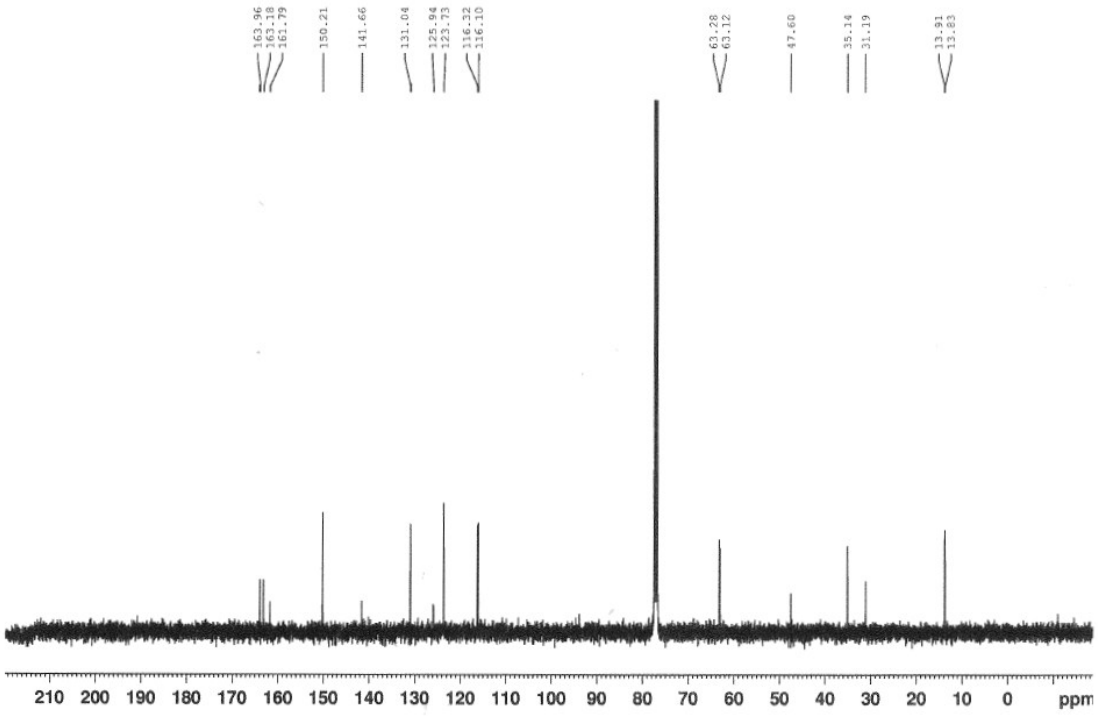
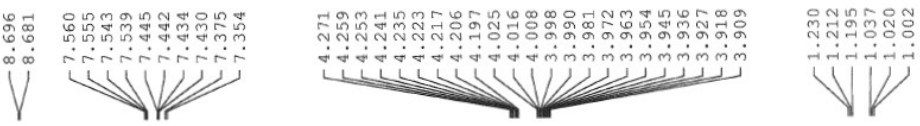
7.470
7.455
7.450
7.443
7.439
7.131
7.125
7.109
7.093
7.088
4.264
4.255
4.246
4.237
4.228
4.220
4.210
4.202
4.193
4.025
4.016
4.007
3.998
3.990
3.980
3.974
3.962
3.956
3.947
3.938
3.929
3.920
3.911
1.220
1.202
1.185
1.039
1.021
1.003
F
COOEt
COOEt
CN
N
12d
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
1.89
3.87
1.88
1.94
3.01
3.05
3.00


163.96
163.18
161.79
150.21
141.66
131.04
125.94
123.73
116.32
116.10
63.28
63.12
47.60
35.14
31.19
13.91
13.83
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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8.681
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7.555
7.543
7.539
7.445
7.442
7.434
7.430
7.375
7.354
4.271
4.259
4.253
4.241
4.235
4.223
4.217
4.206
4.197
4.025
4.016
4.008
3.998
3.990
3.981
3.972
3.963
3.954
3.945
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3.927
3.918
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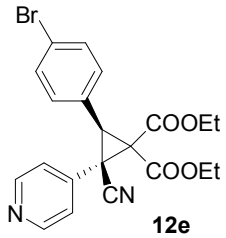
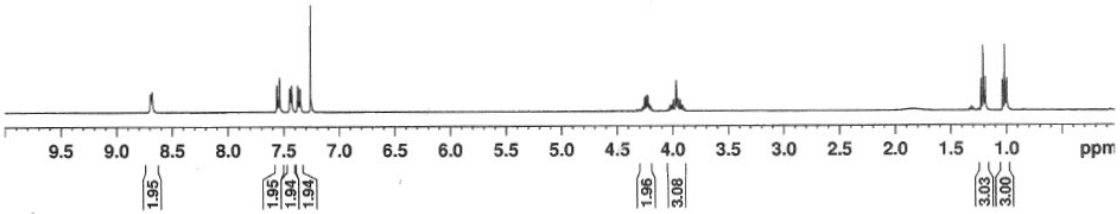
1.230
1.212
1.195
1.037
1.020
1.002


Br
COOEt
COOEt
CN
N

12e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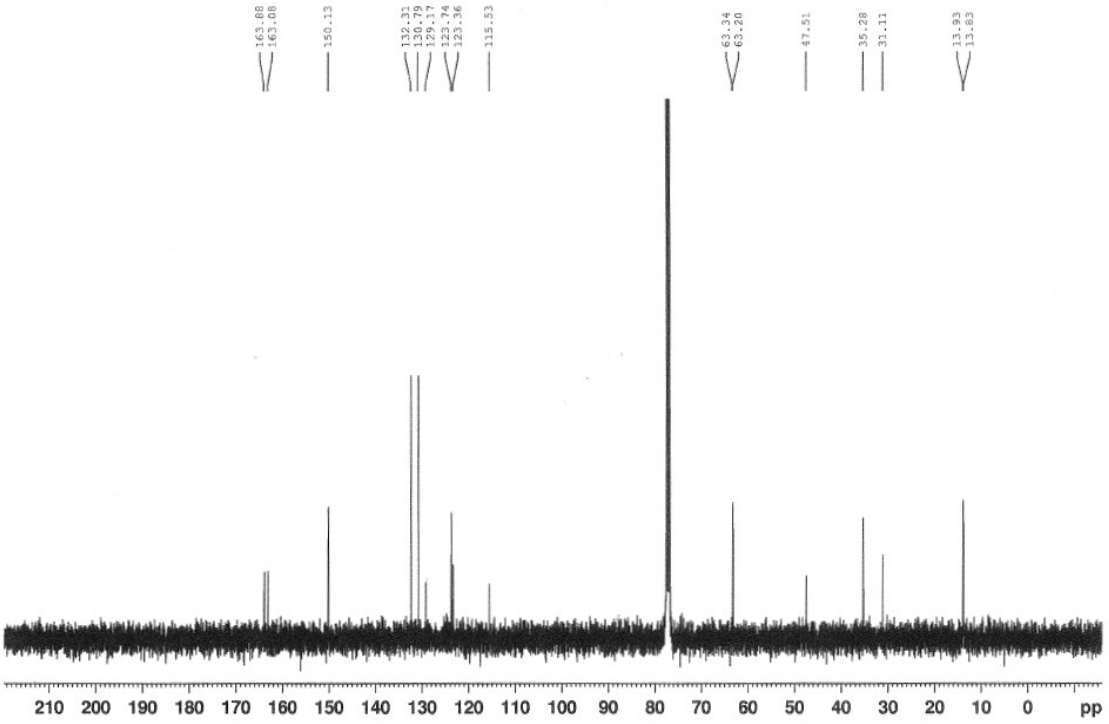
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.95
1.95
1.94
1.94
1.96
3.08
3.03
3.00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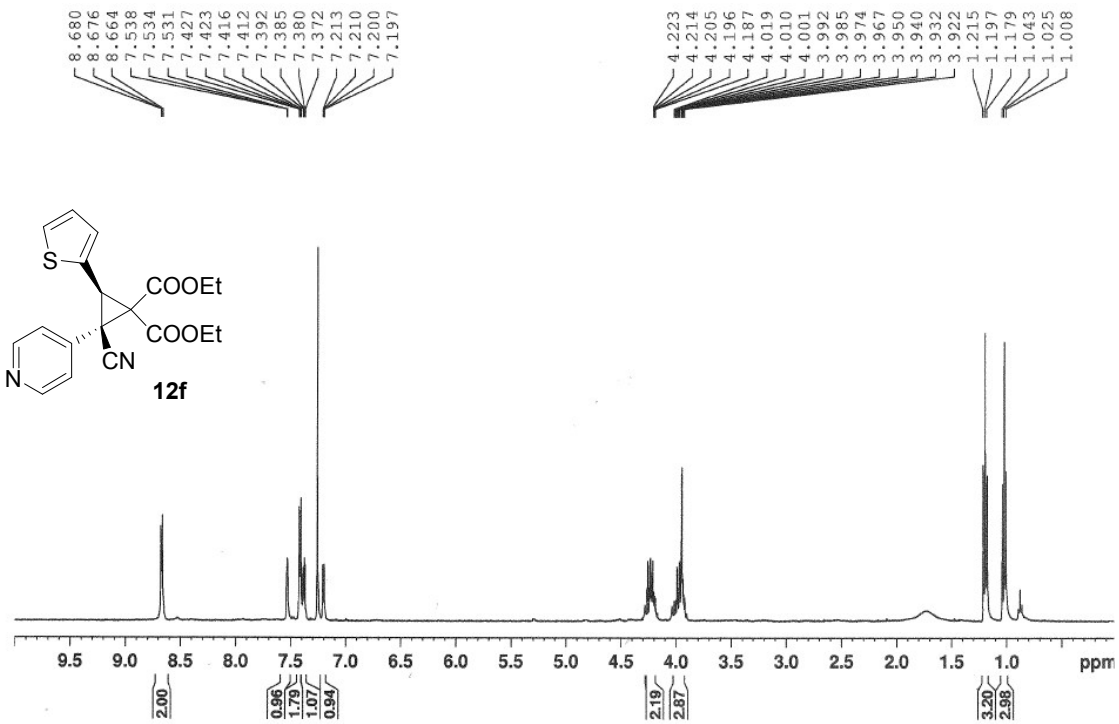
163.08
150.13
132.31
130.79
129.77
123.74
123.36
115.53
63.34
63.20
47.51
35.28
31.11
13.93
13.63
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
pp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7.534
7.531
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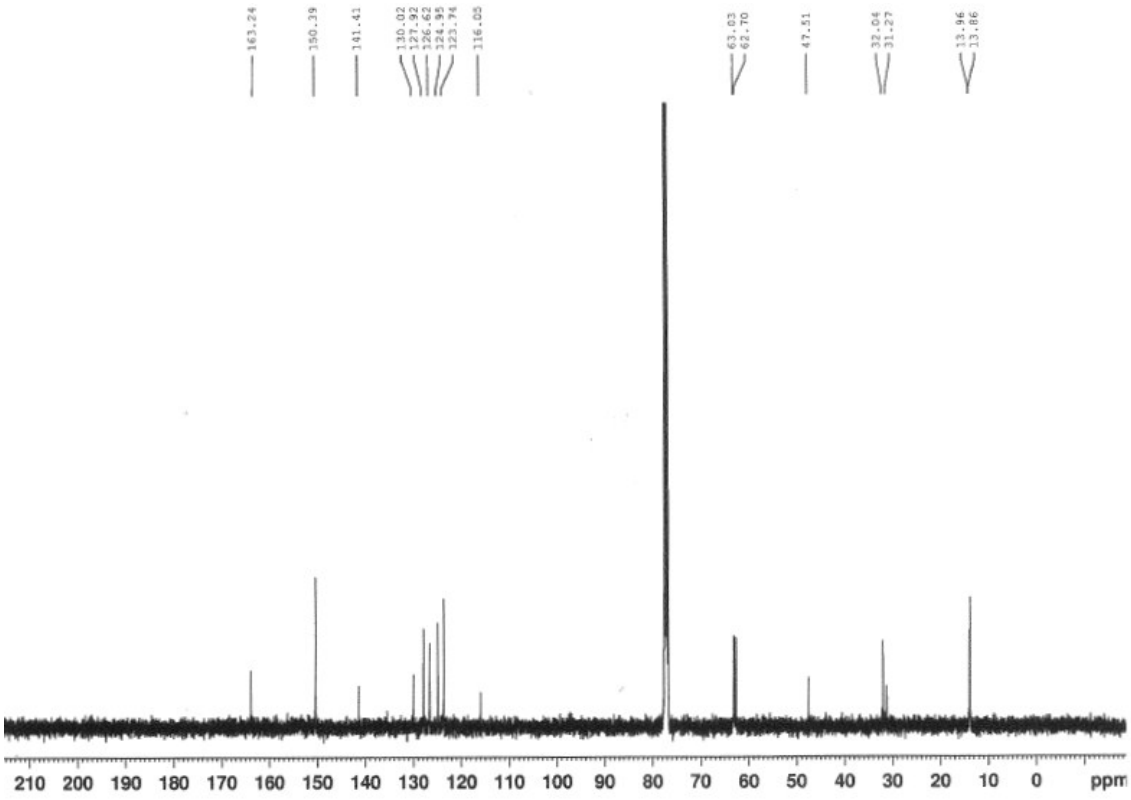
7.423
7.416
7.412
7.392
7.385
7.380
7.372
7.213
7.210
7.200
7.197
4.223
4.214
4.205
4.196
4.187
4.019
4.010
4.001
3.992
3.985
3.974
3.967
3.950
3.940
3.932
3.922
1.215
1.197
1.179
1.043
1.025
1.008
S
COOEt
COOEt
CN
N
12f
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
2.00
0.96
1.79
1.07
0.94
2.19
2.87
3.20
2.98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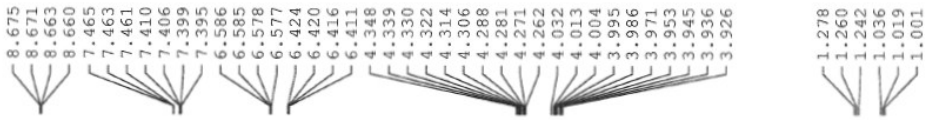
150.39
141.41
130.02
127.92
126.62
124.95
123.74
116.05
63.03
62.70
47.51
32.04
31.27
13.96
13.86
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm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7.461
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7.406
7.399
7.395
6.586
6.585
6.578
6.577
6.424
6.420
6.416
6.411
4.348
4.339
4.330
4.322
4.314
4.306
4.288
4.281
4.271
4.262
4.032
4.013
4.004
3.995
3.986
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1.260
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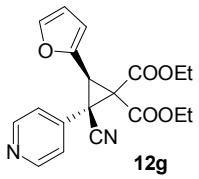
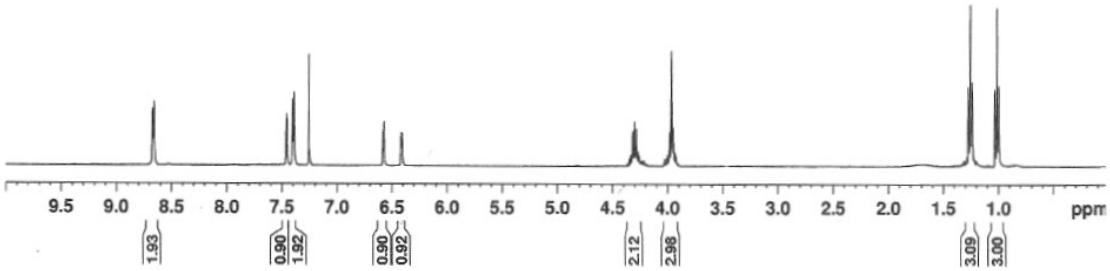
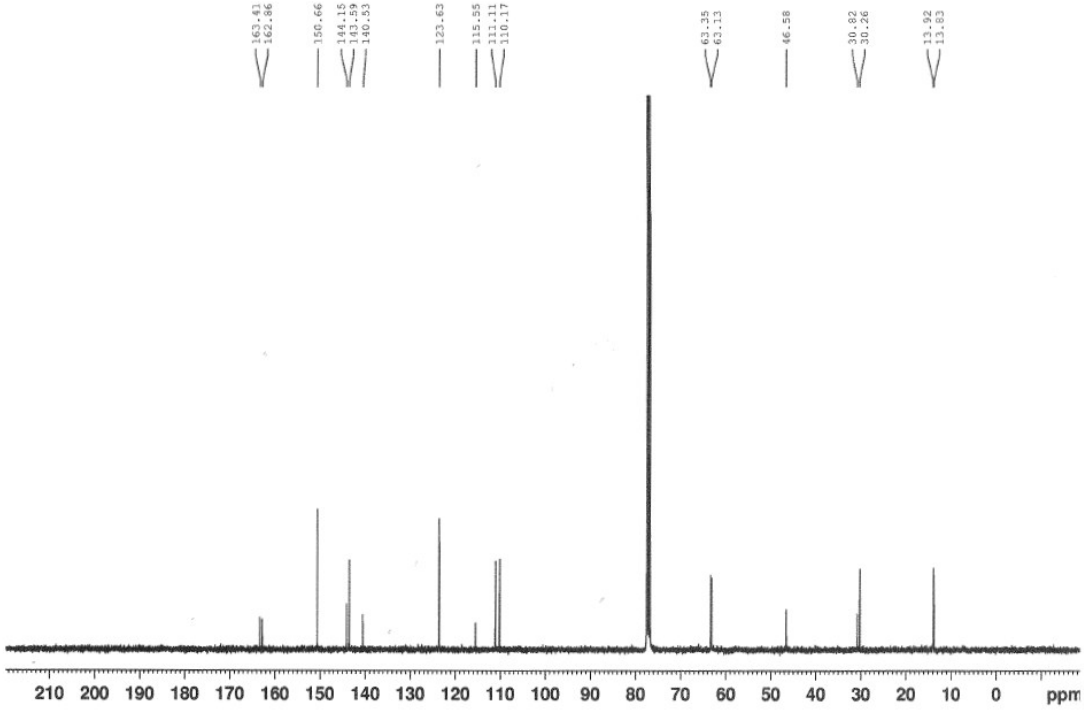
1.036
1.019
1.001


O
COOEt
COOEt
CN
N

12g


9.5 9.0 8.5 8.0 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
1.93
0.90
1.92
0.90
0.92
2.12
2.98
3.09
3.00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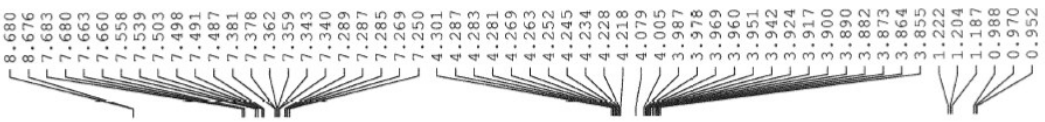
162.86
150.66
144.15
143.59
140.53
123.63
115.55
111.11
110.17
63.35
63.13
46.58
30.82
30.26
13.92
13.83
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
ppm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7.660
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7.539
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7.487
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7.378
7.362
7.359
7.343
7.340
7.289
7.287
7.285
7.269
7.250
4.301
4.287
4.283
4.281
4.269
4.263
4.252
4.245
4.234
4.228
4.218
4.079
4.005
3.987
3.978
3.969
3.960
3.951
3.942
3.924
3.917
3.900
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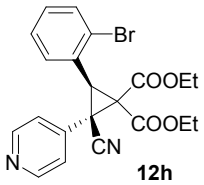
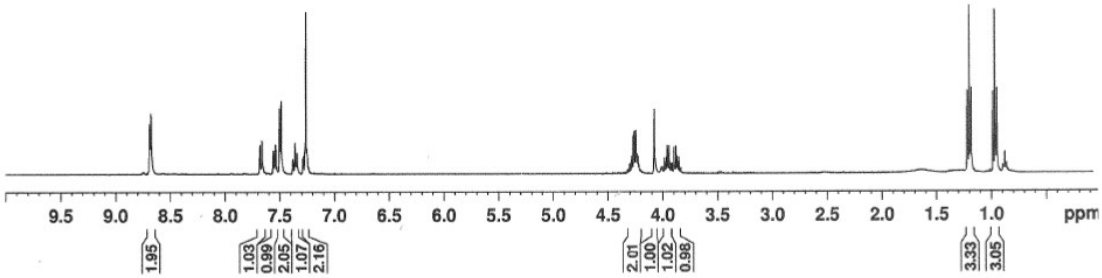
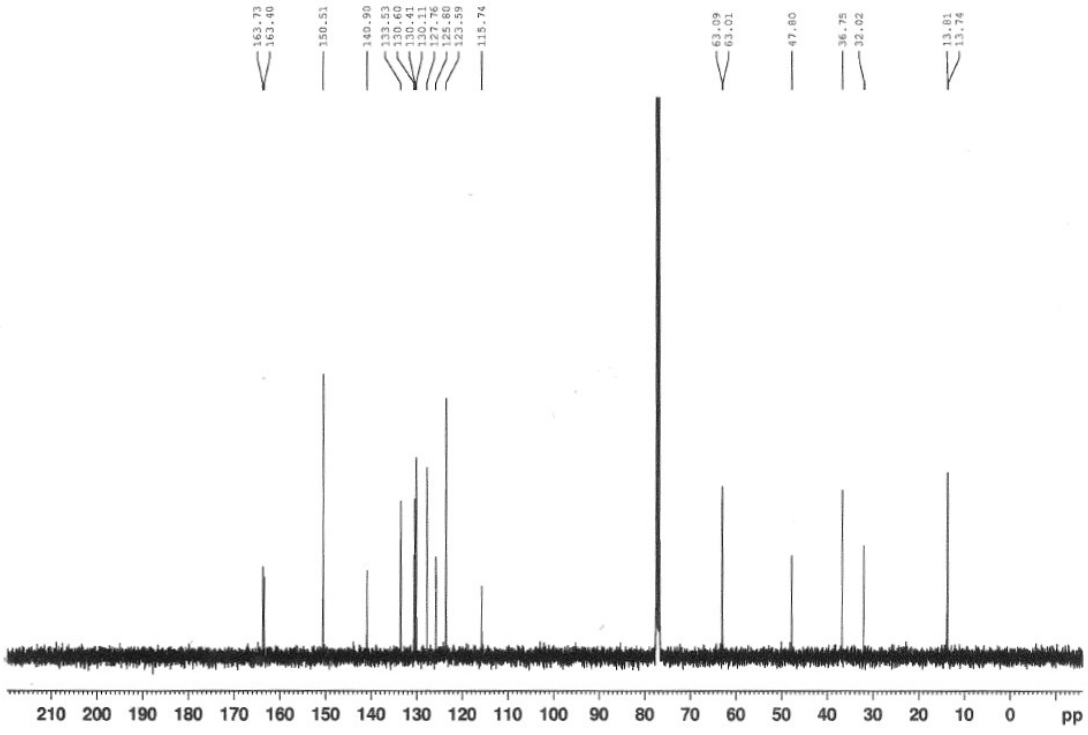
0.970
0.952


Br
COOEt
COOEt
CN
N

12h


9.5 9.0 8.5 8.0 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
1.95
1.03
0.99
2.05
1.07
2.16
2.01
1.00
1.02
0.98
3.33
3.05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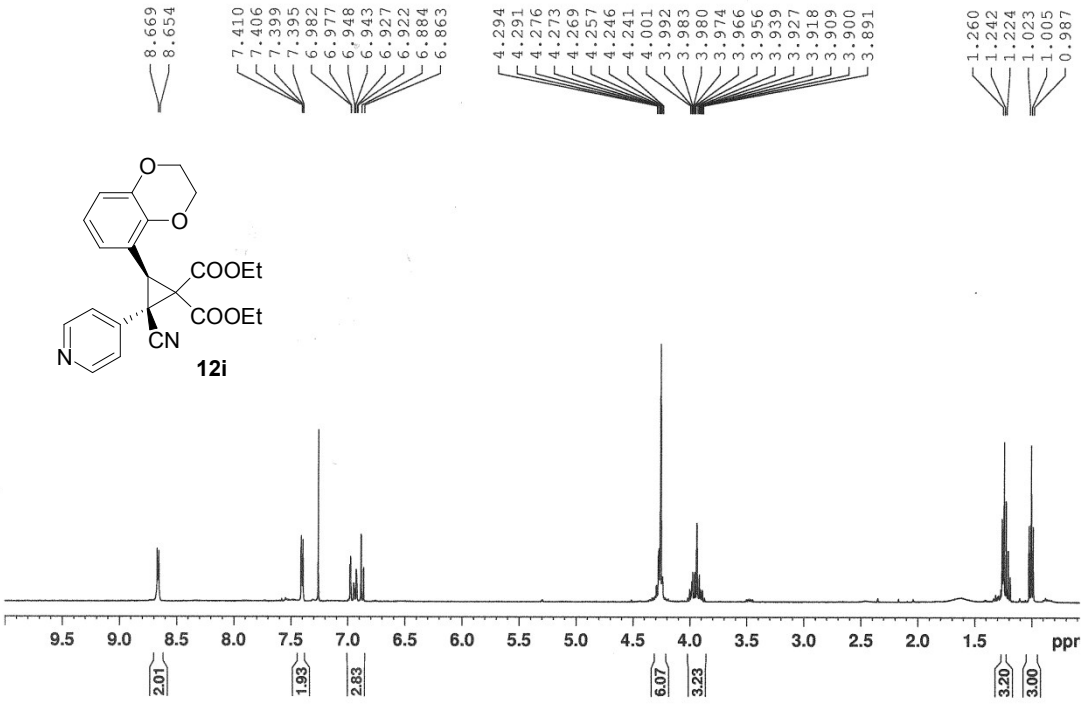
133.53
130.60
130.41
130.11
127.76
125.80
123.59
115.74
63.09
63.01
47.80
36.75
32.02
13.81
13.74
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 pp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6.977
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6.927
6.922
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6.863
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3.927
3.918
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3.900
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1.242
1.224
1.023
1.005
0.987
O
O
COOEt
COOEt
CN
N
12i
9.5 9.0 8.5 8.0 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 ppm
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1.93
2.83
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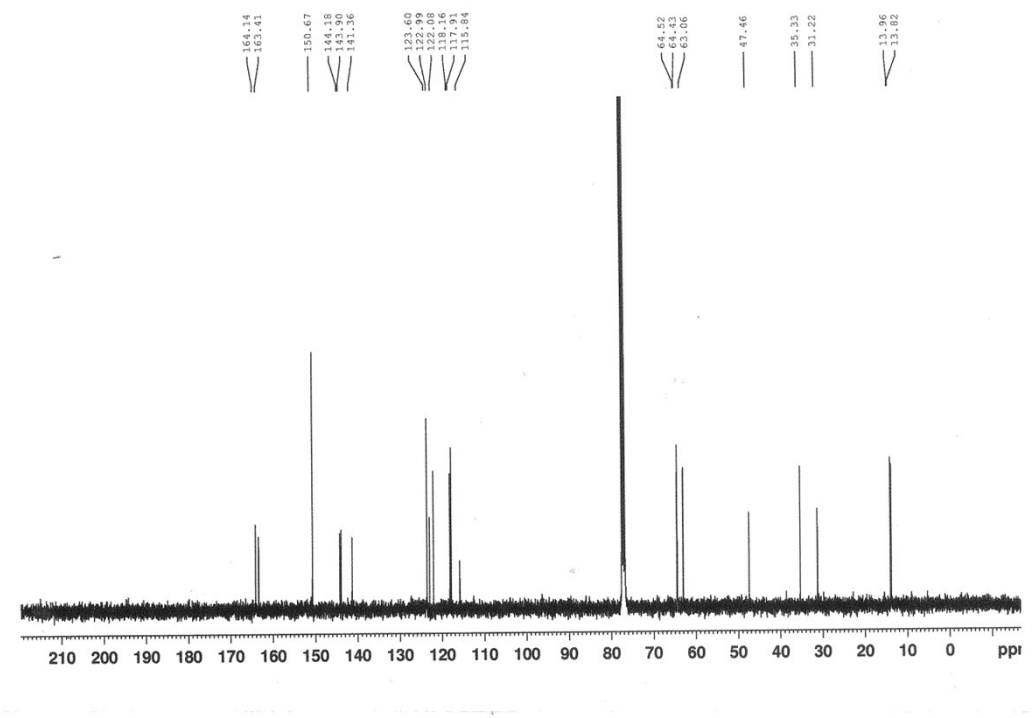
3.00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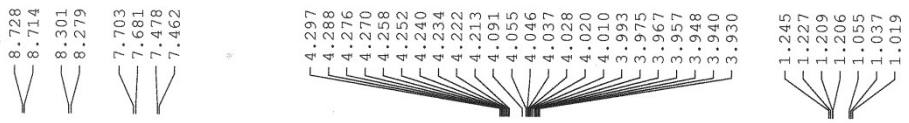
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4.046
4.037
4.028
4.020
4.010
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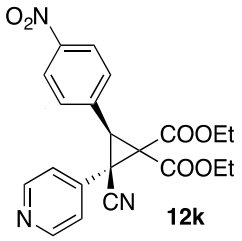
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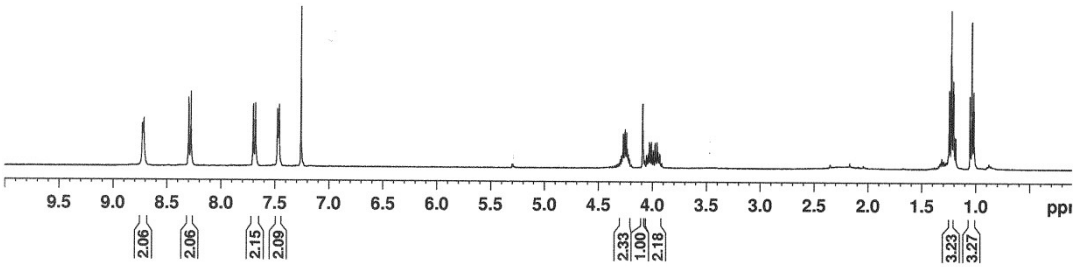
COOEt
COOEt
CN
N

12k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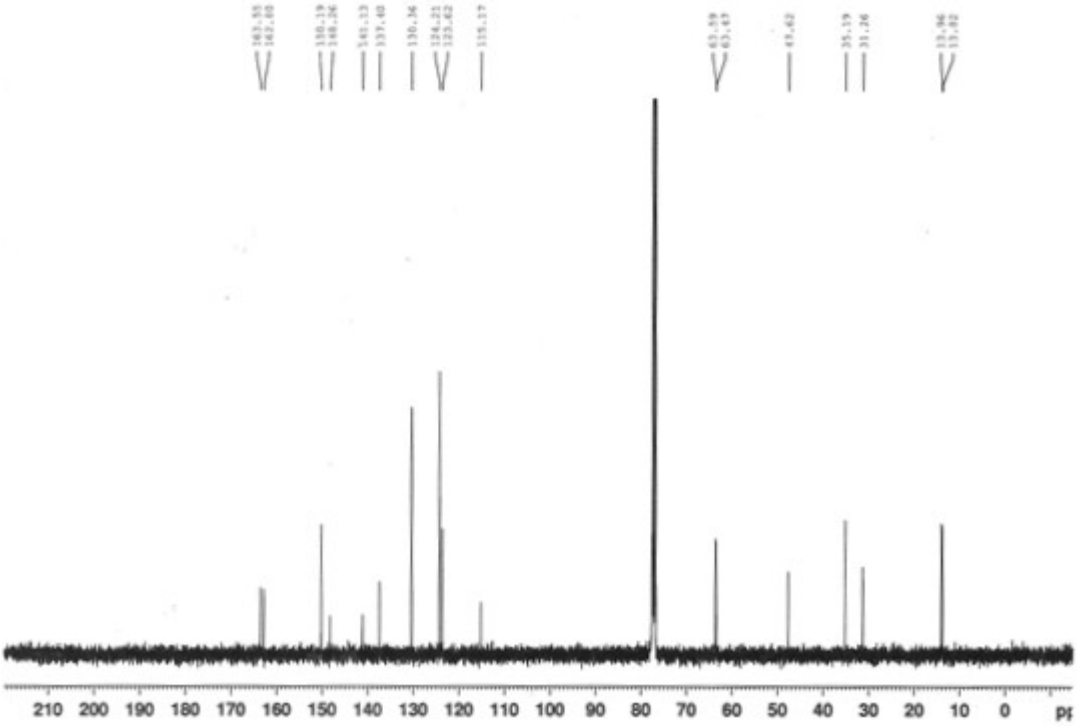
2.33
1.00
2.18
3.23
3.27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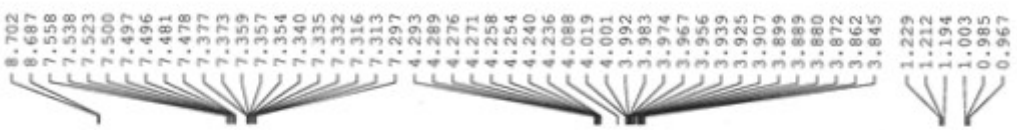
pp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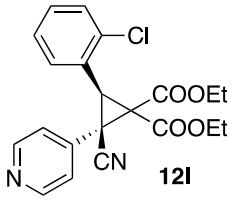
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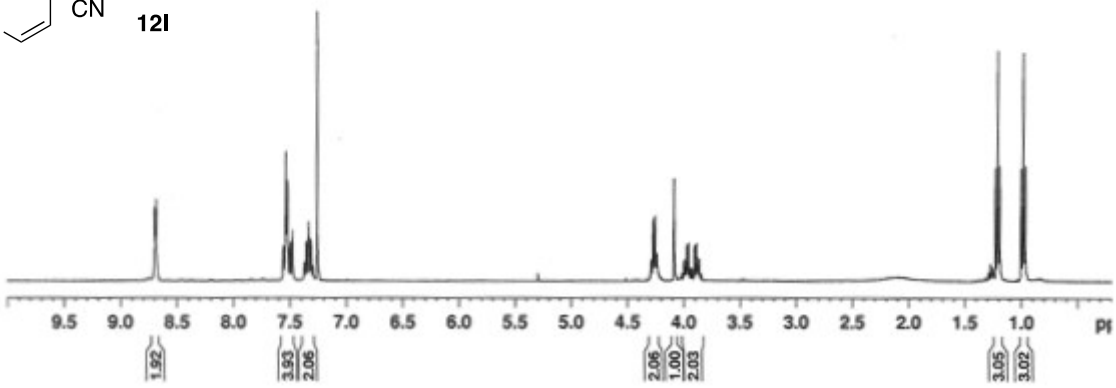
COOEt
CN
N

12l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2.06
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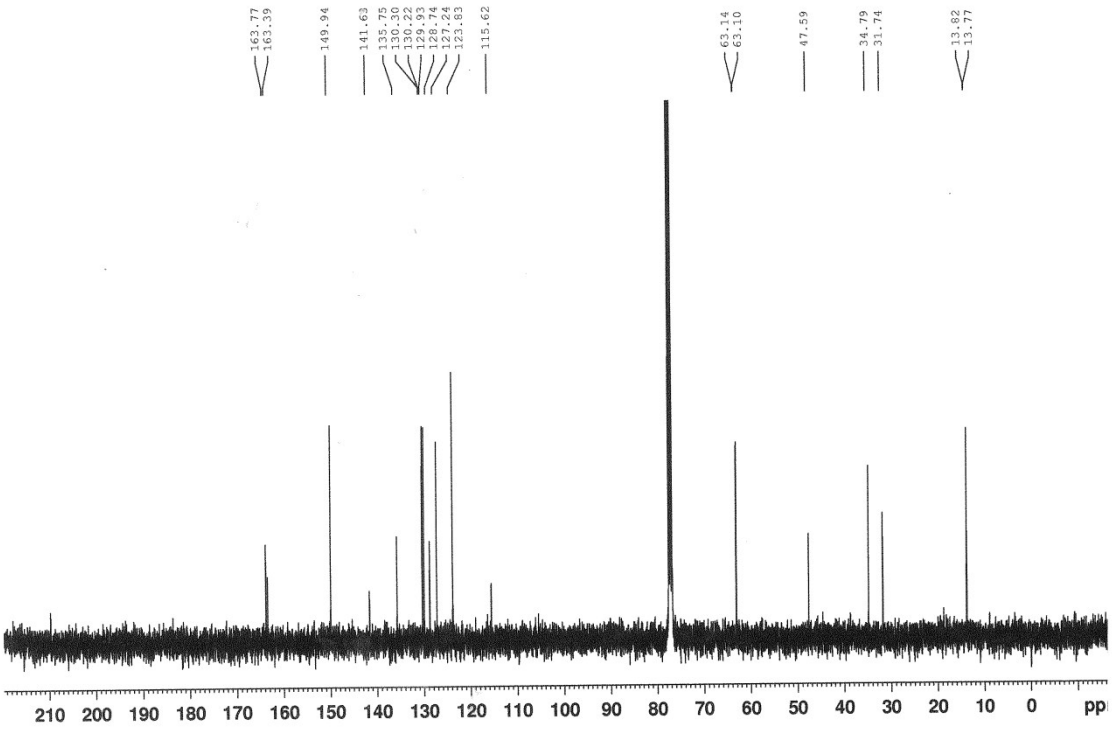
2.03
3.05
3.02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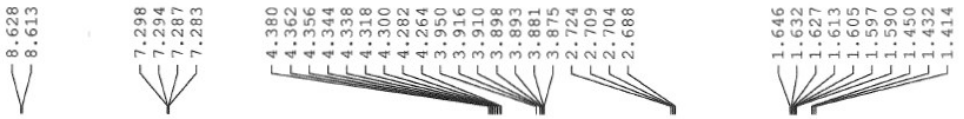
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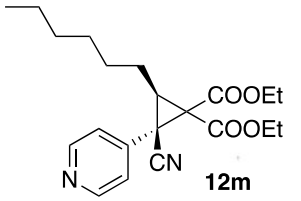
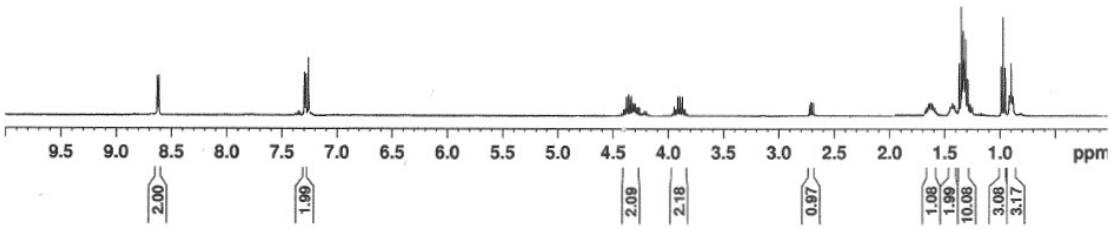
1.414


COOEt
COOEt
CN
N

12m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2.18
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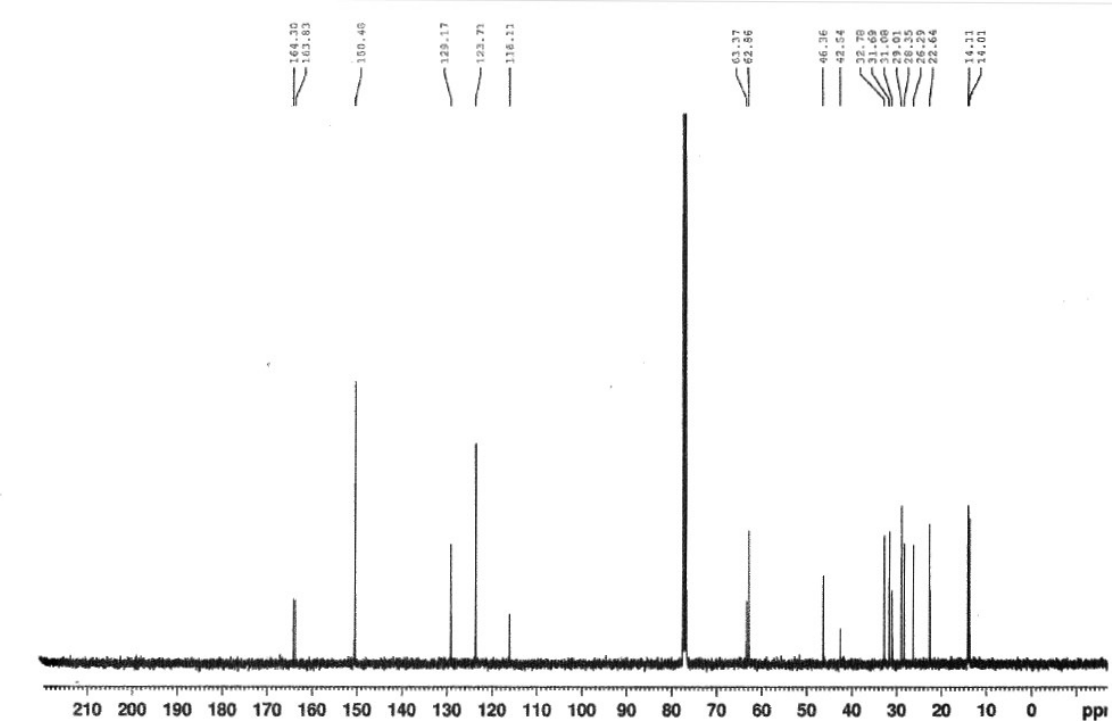
1.08
1.99
10.08
3.08
3.17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46.36
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29.01
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25.29
22.64
14.11
14.01
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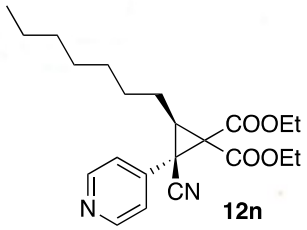
ppm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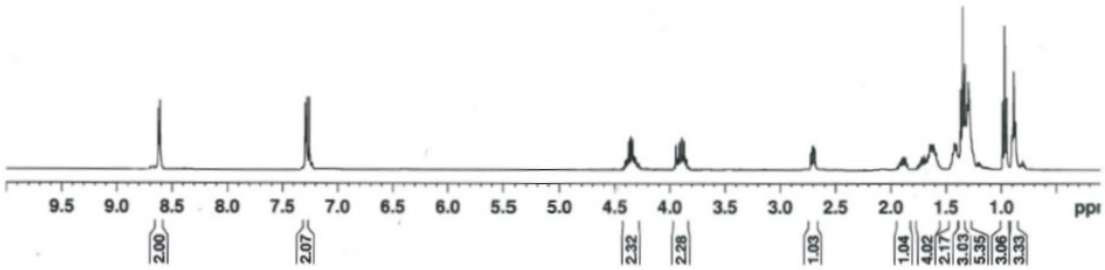
COOEt
CN
N

12n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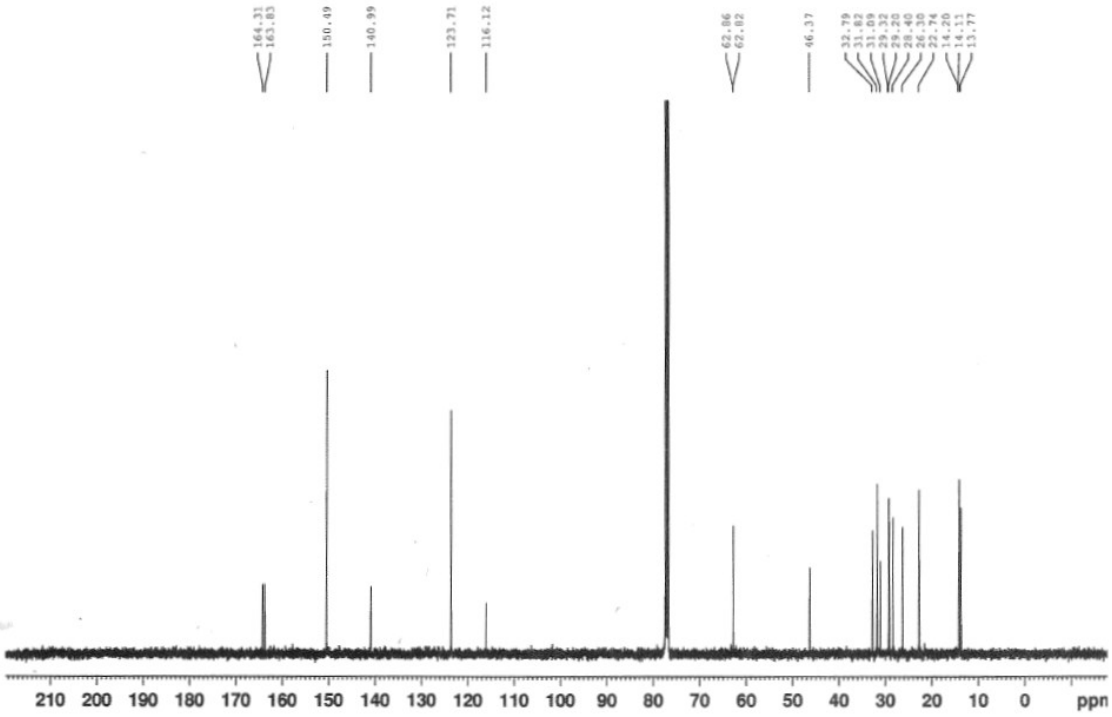
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29.32
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26.30
22.74
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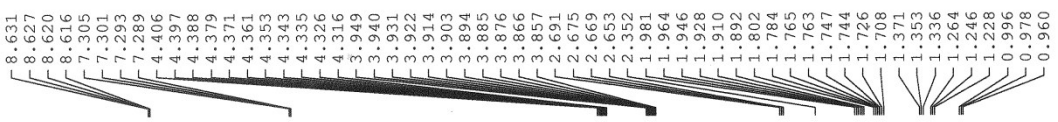
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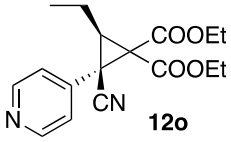
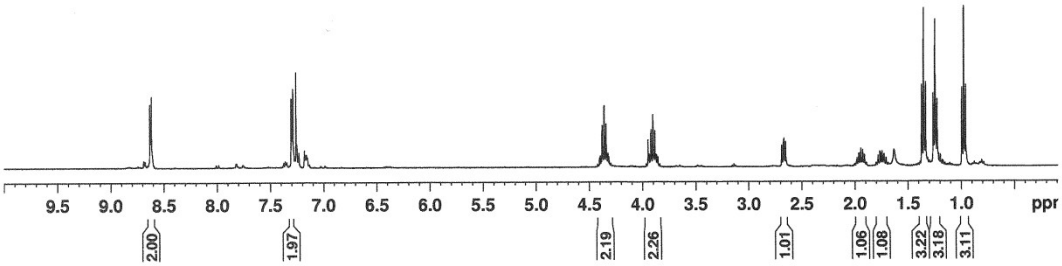
0.960


COOEt
COOEt
CN
N

12o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1.06
1.08
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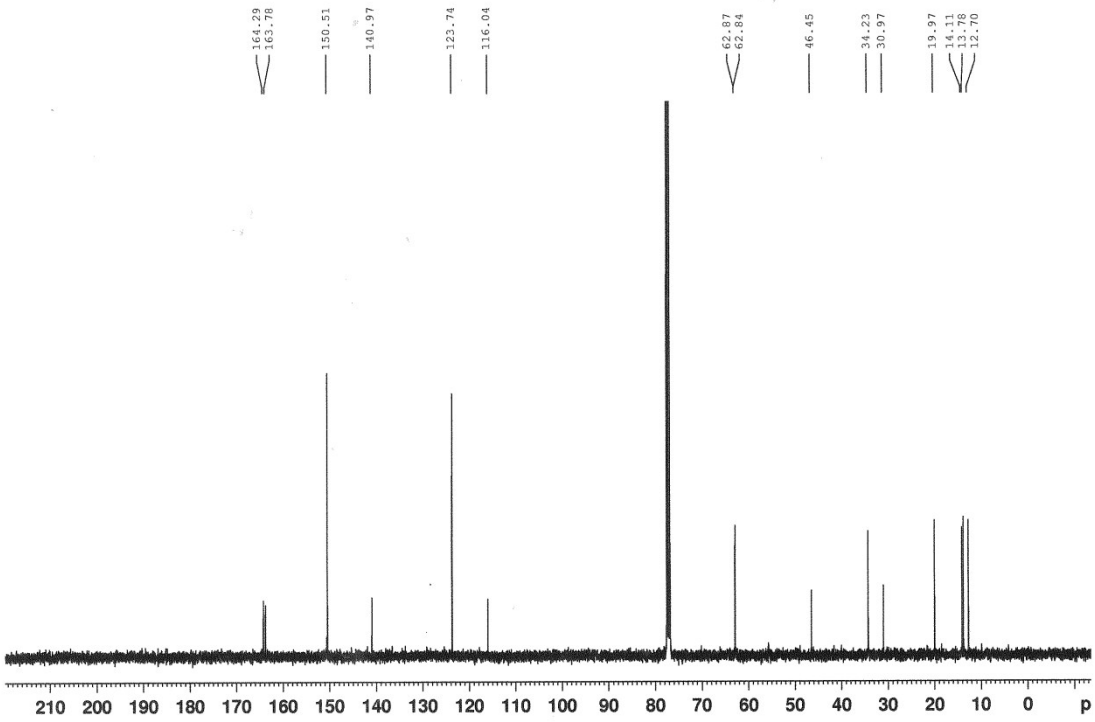
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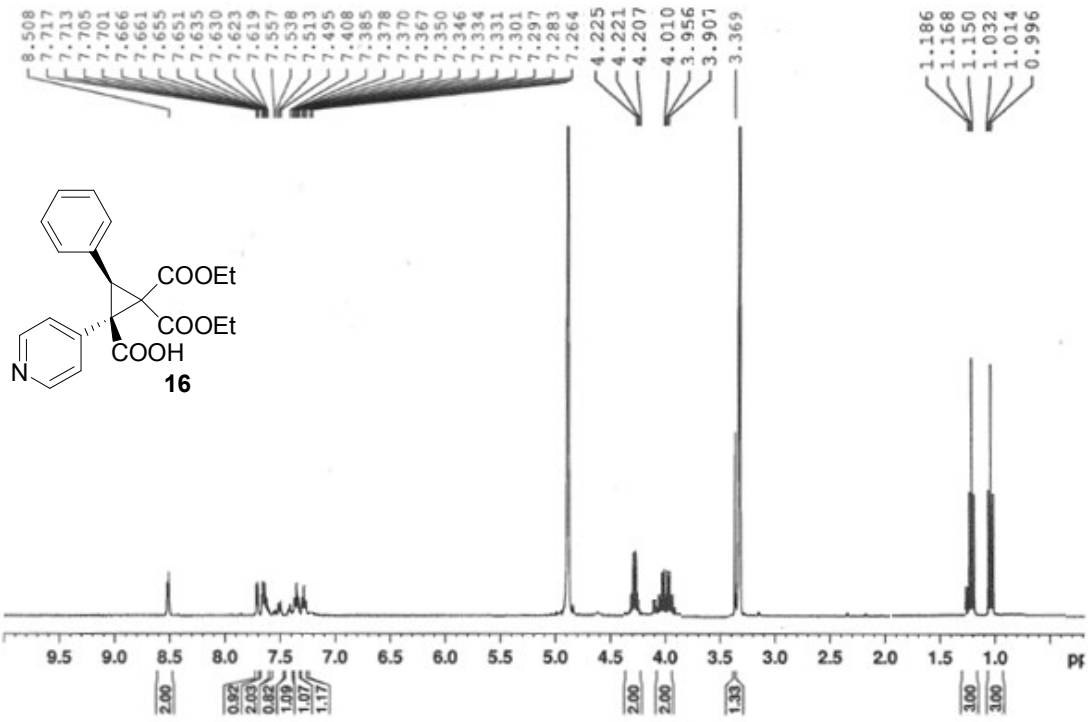
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COOEt
COOH
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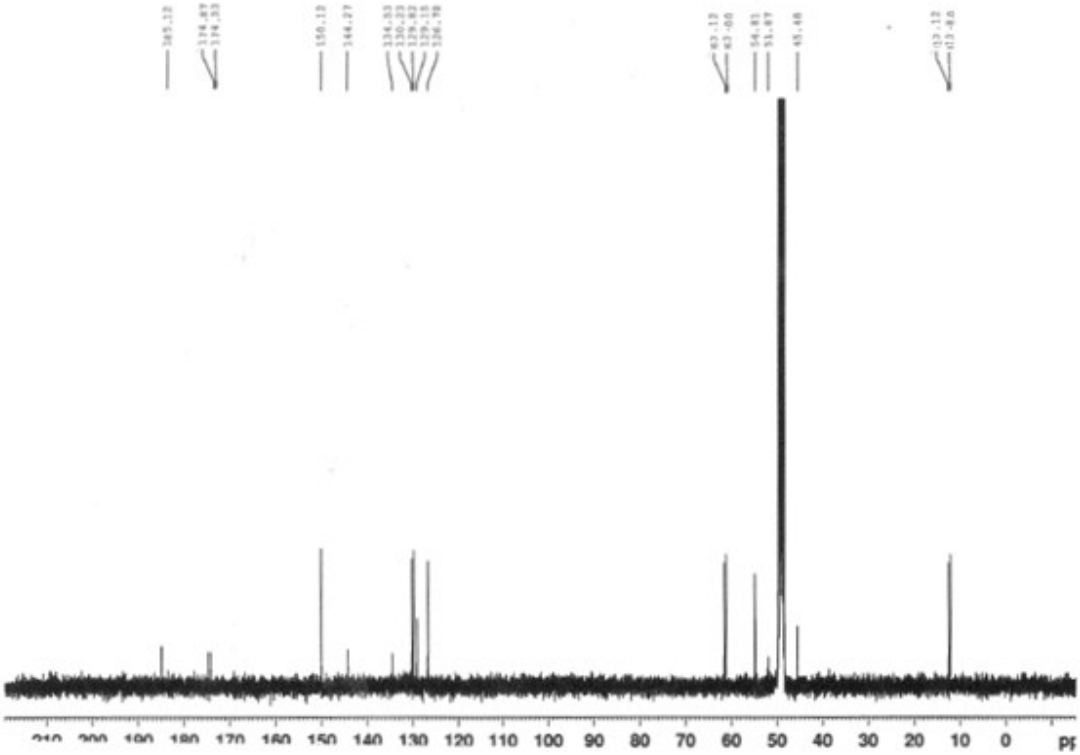
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126.78
63.13
63.00
54.81
51.67
45.48
13.13
13.46
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

Compound **12a**

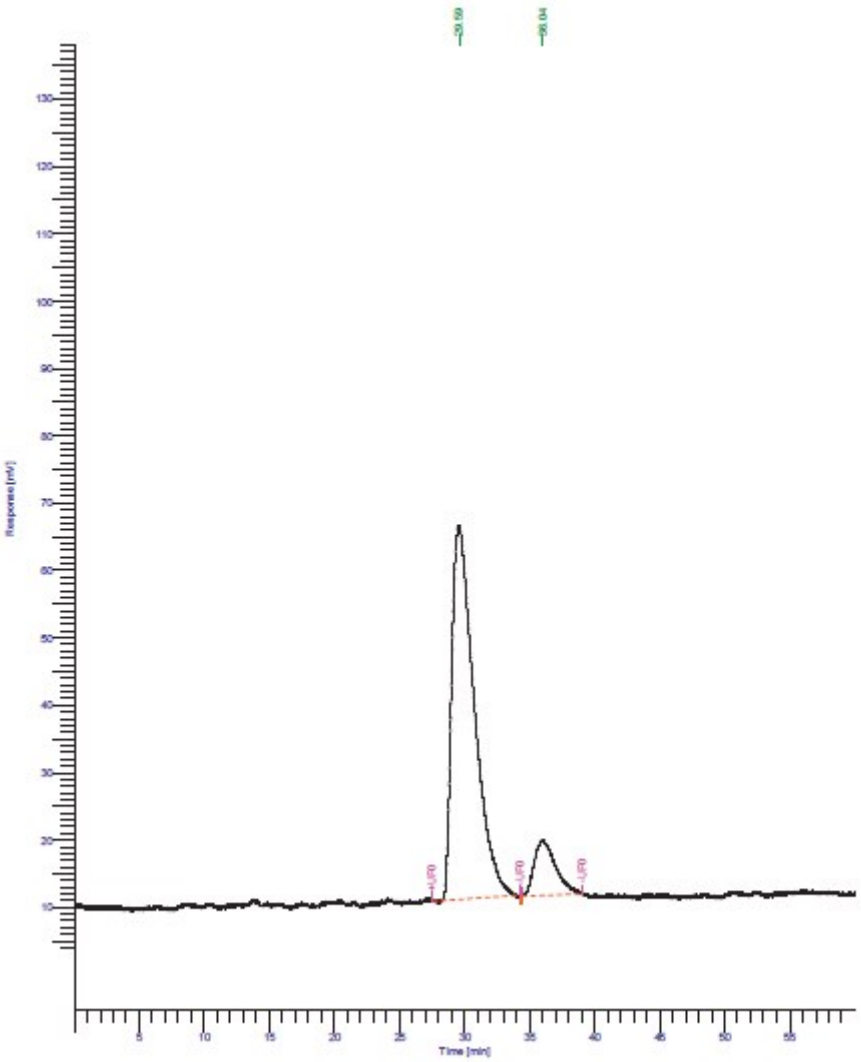
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		29.590	6658711.60	55616.78	90.89	90.89			*MM	6.6587	6.6587
2		36.036	667627.28	7277.34	9.11	9.11			*MM	0.6676	0.6676
			7326338.88	62894.12	100.00	100.00				7.3263	7.3263

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



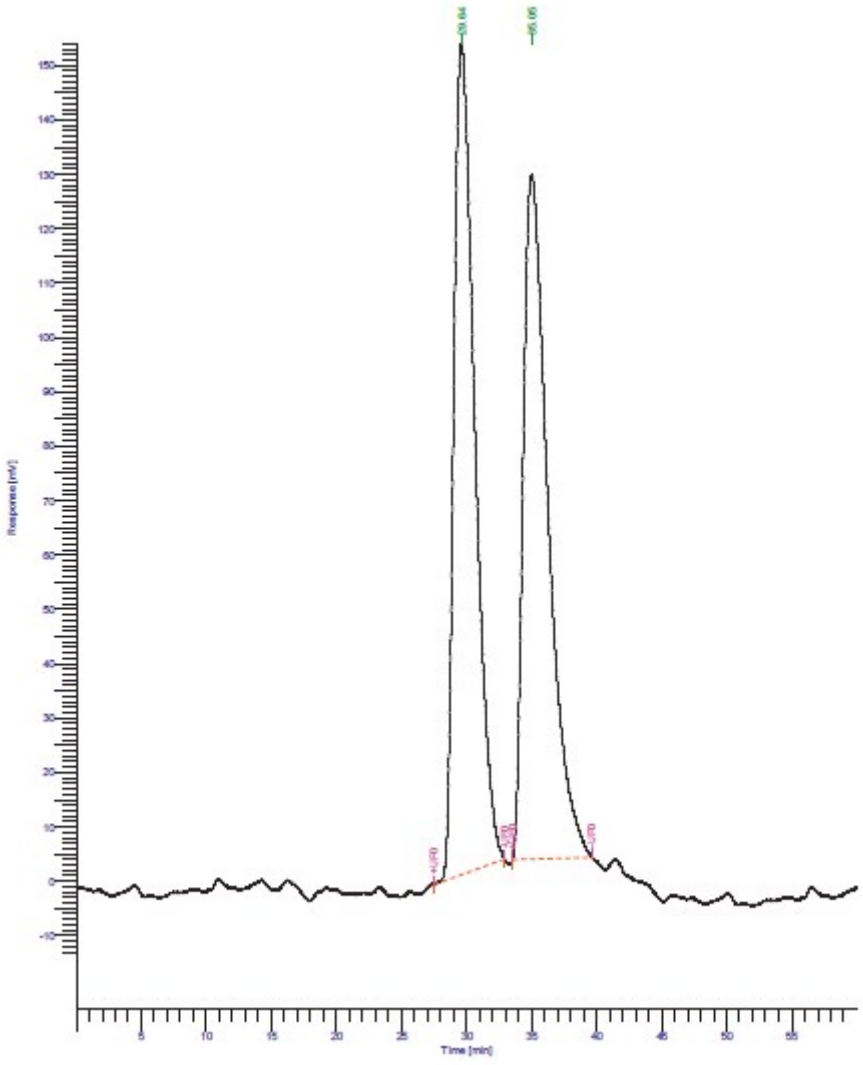
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		29.641	17103345.39	152855.60	50.62	50.62			*MM	17.1033	17.1033
2		35.046	16686611.01	126044.75	49.38	49.38			*MM	16.6866	16.6866
			33789956.40	278900.35	100.00	100.00				33.7900	33.7900

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12b**

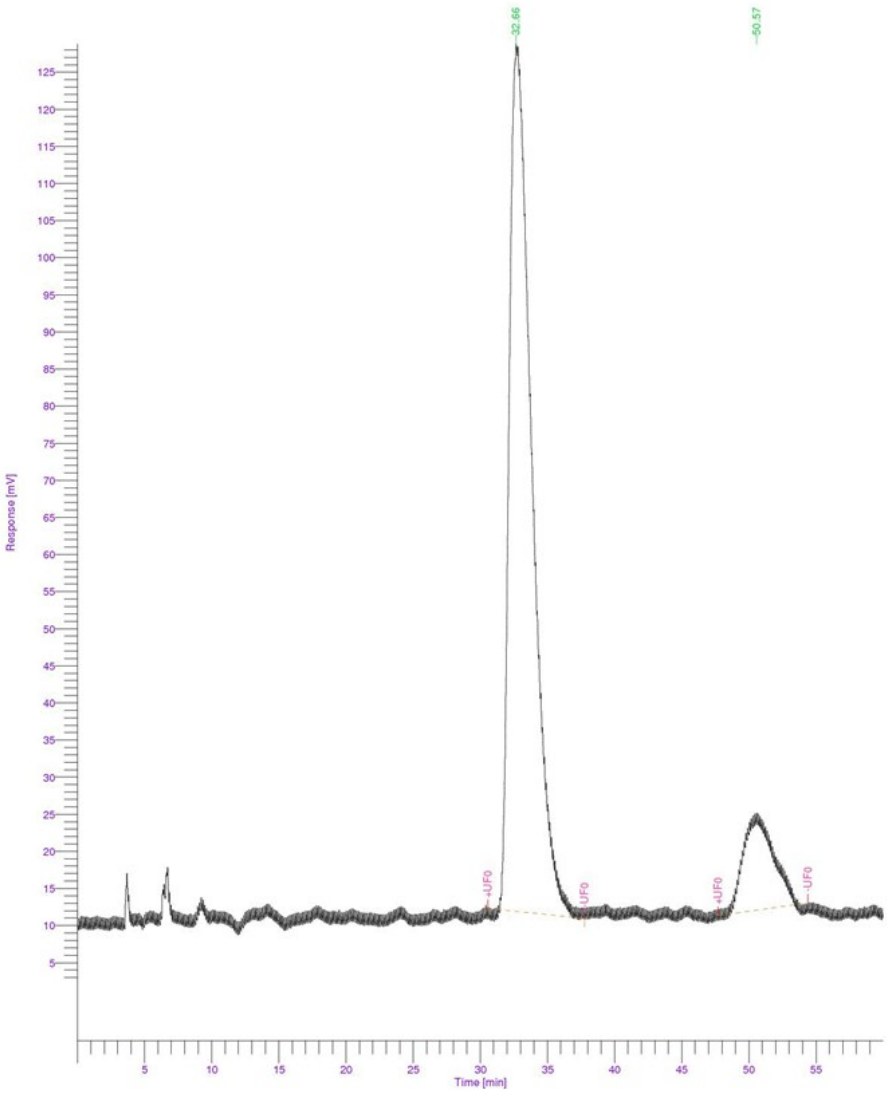
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		32.663	13956764.61	117952.32	87.39	87.39			*MM	13.9568	13.9568
2		50.571	2013462.91	13006.68	12.61	12.61			*MM	2.0135	2.0135
			15970227.52	130959.00	100.00	100.00				15.9702	15.9702

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



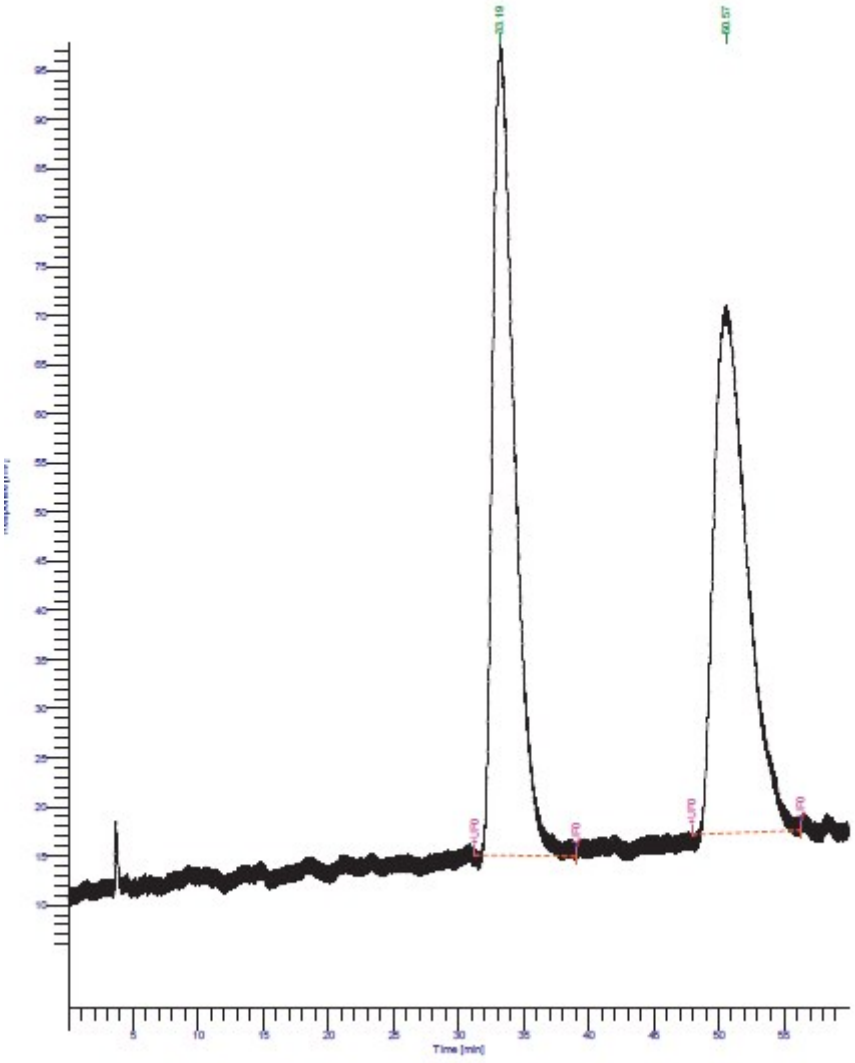
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		33.191	9632264.83	82780.62	50.63	50.63			*MM	9.6323	9.6323
2		50.571	9391953.78	53658.68	49.37	49.37			*MM	9.3920	9.3920
			19024218.61	136439.30	100.00	100.00				19.0242	19.0242

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12c**

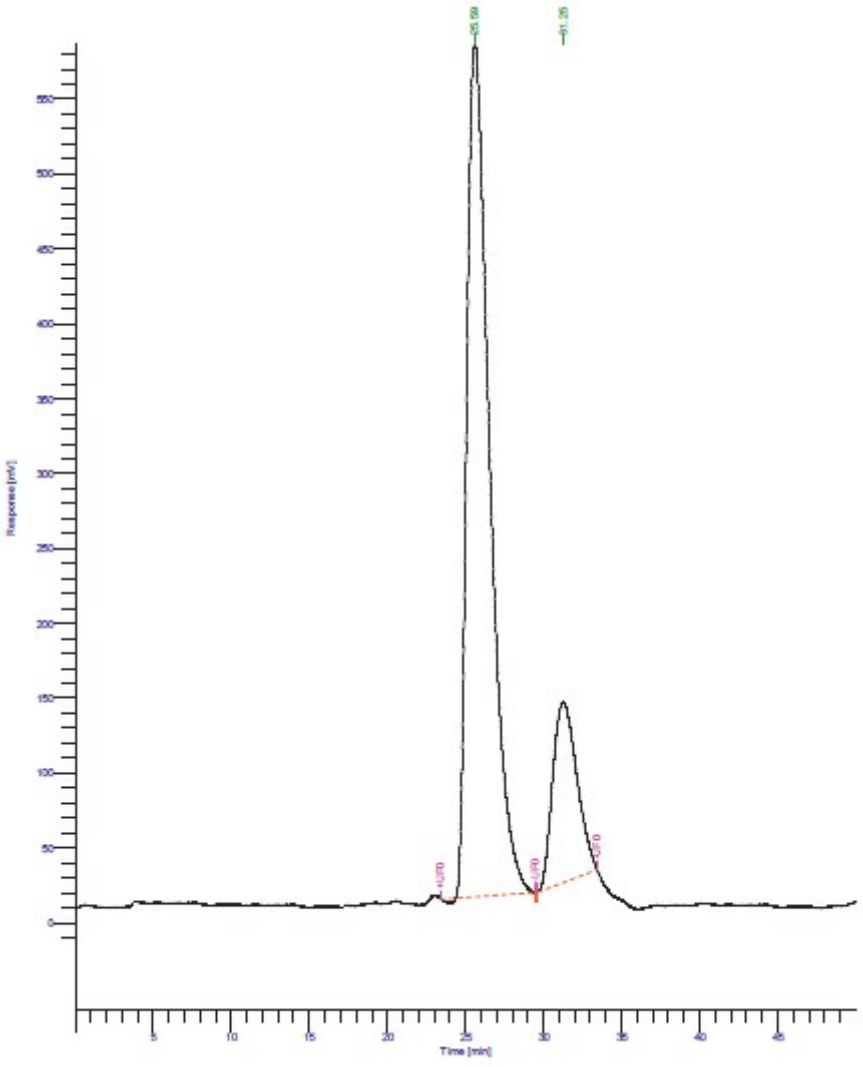
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		25.593	57478230.29	569861.78	81.59	81.59			*MM	57.4782	57.4782
2		31.255	12965404.05	120474.21	18.41	18.41			*MM	12.9654	12.9654
			70443634.34	690335.99	100.00	100.00				70.4436	70.4436

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



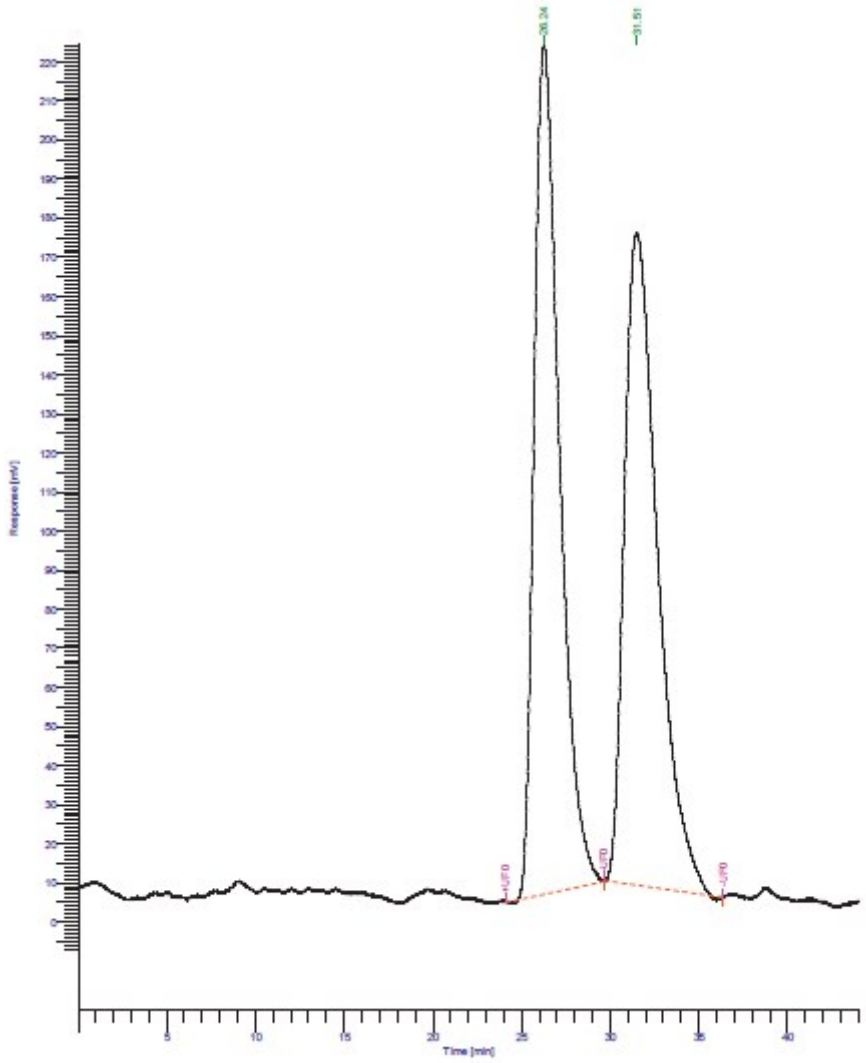
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		26.239	21714383.49	217732.43	49.95	49.95			*MM	21.7144	21.7144
2		31.511	21759818.66	167039.66	50.05	50.05			*MM	21.7598	21.7598
			43474202.15	384772.09	100.00	100.00				43.4742	43.4742

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12d**

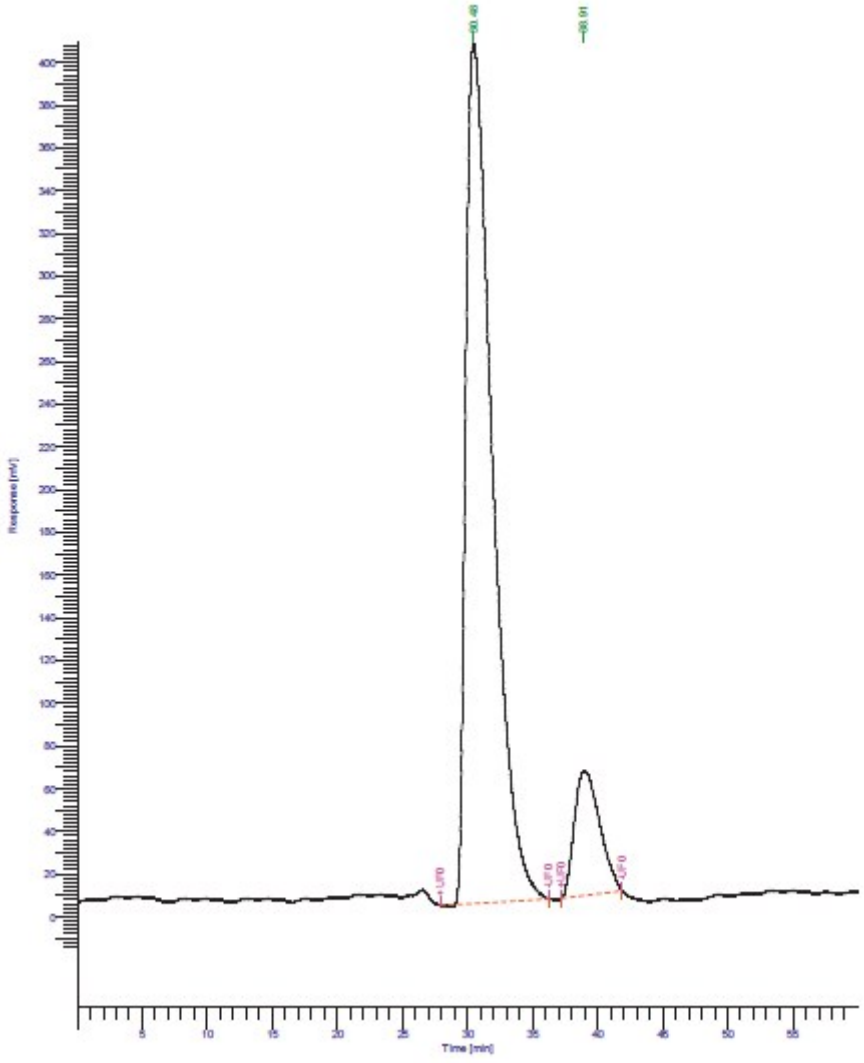
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		30.485	57043275.80	403527.46	87.84	87.84			*MM	57.0433	57.0433
2		38.911	7898797.70	58523.16	12.16	12.16			*MM	7.8988	7.8988
			64942073.50	462050.62	100.00	100.00				64.9421	64.9421

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



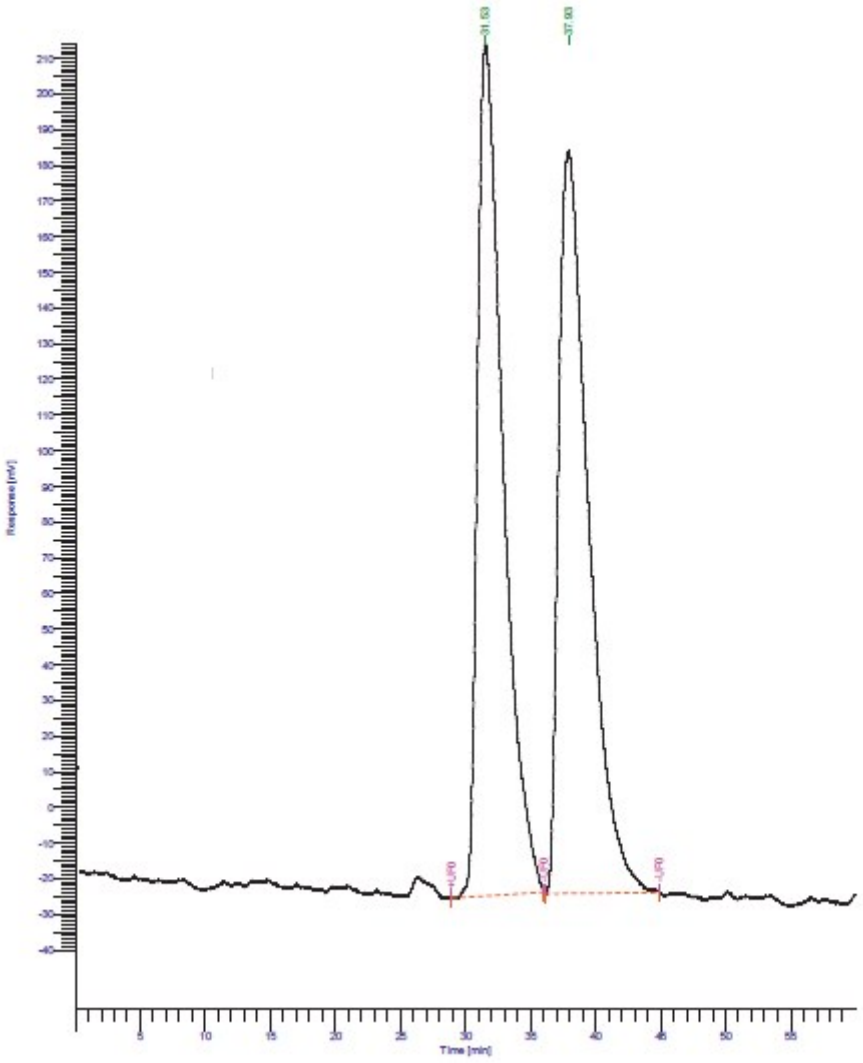
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		31.533	32802594.52	239042.29	49.59	49.59			*MM	32.8026	32.8026
2		37.928	33346888.49	208196.13	50.41	50.41			*MM	33.3469	33.3469
			66149483.02	447238.42	100.00	100.00				66.1495	66.1495

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12e**

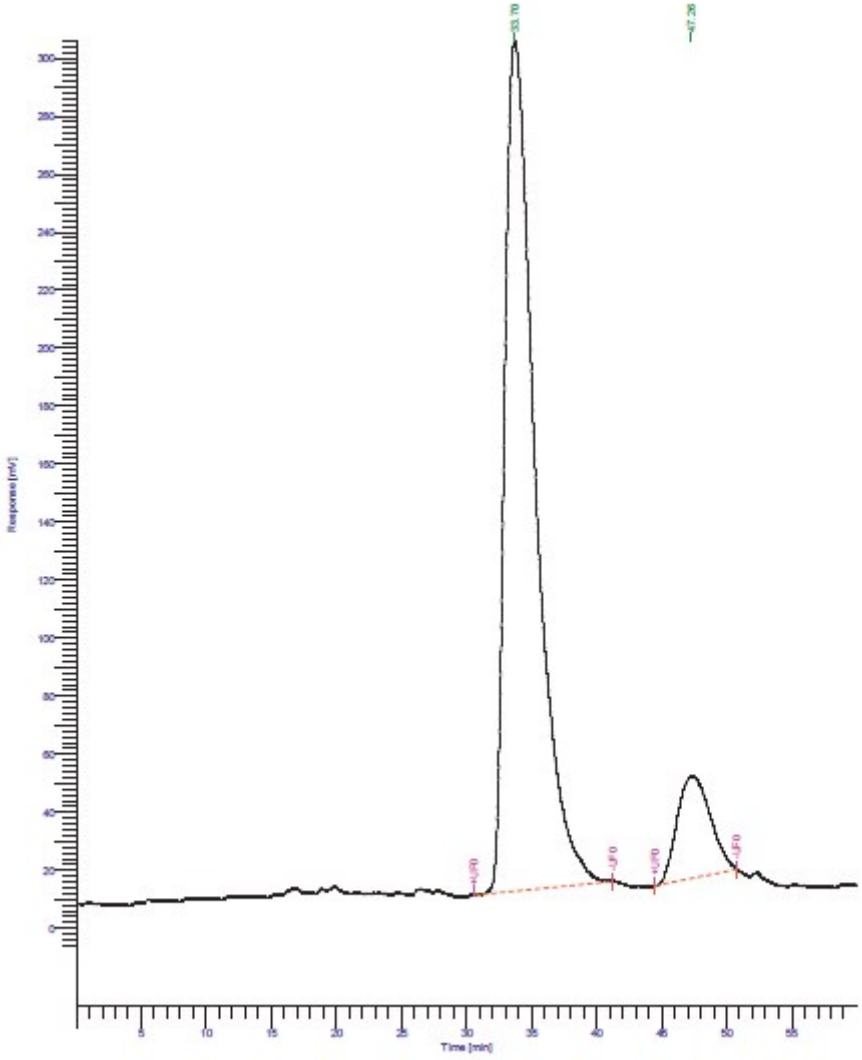
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		33.704	47825175.84	293198.75	88.47	88.47			*MM	47.8252	47.8252
2		47.263	6231561.04	35576.34	11.53	11.53			*MM	6.2316	6.2316
			54056736.88	328775.09	100.00	100.00				54.0567	54.0567

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



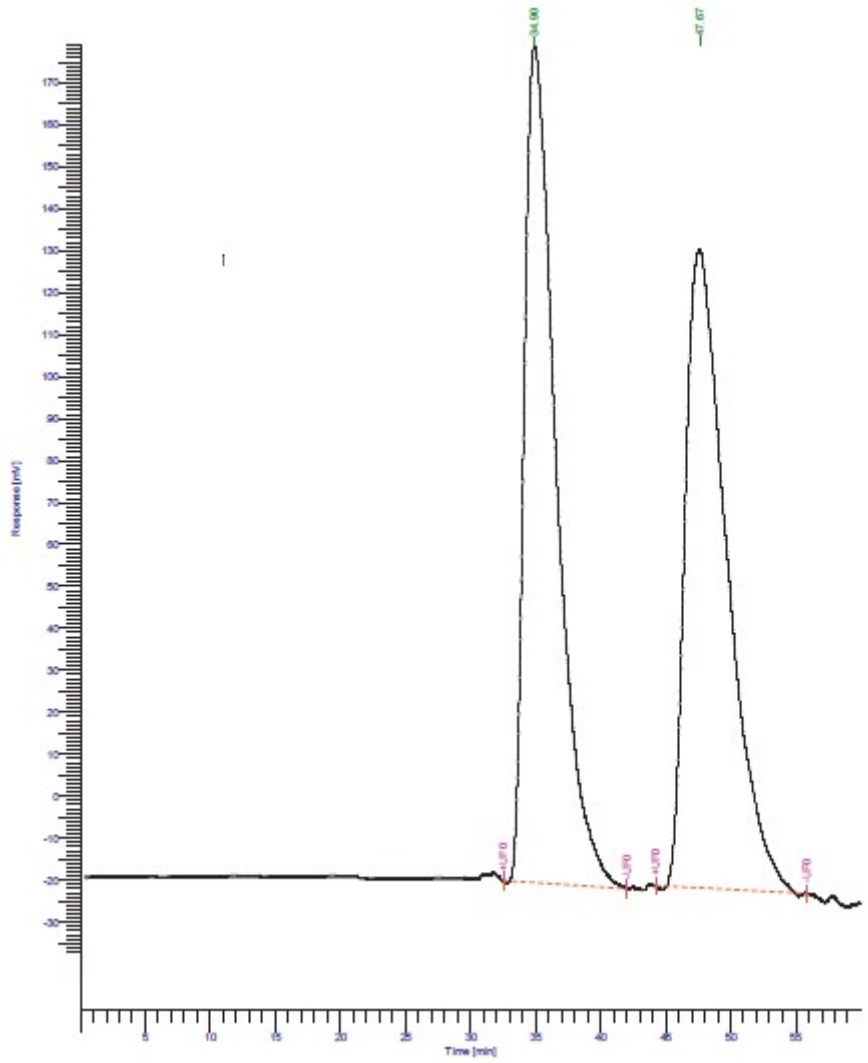
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		34.899	33864006.91	199778.99	50.08	50.08			*MM	33.8640	33.8640
2		47.667	33755004.42	152240.85	49.92	49.92			*MM	33.7550	33.7550
			67619011.33	352019.84	100.00	100.00				67.6190	67.6190

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12f**

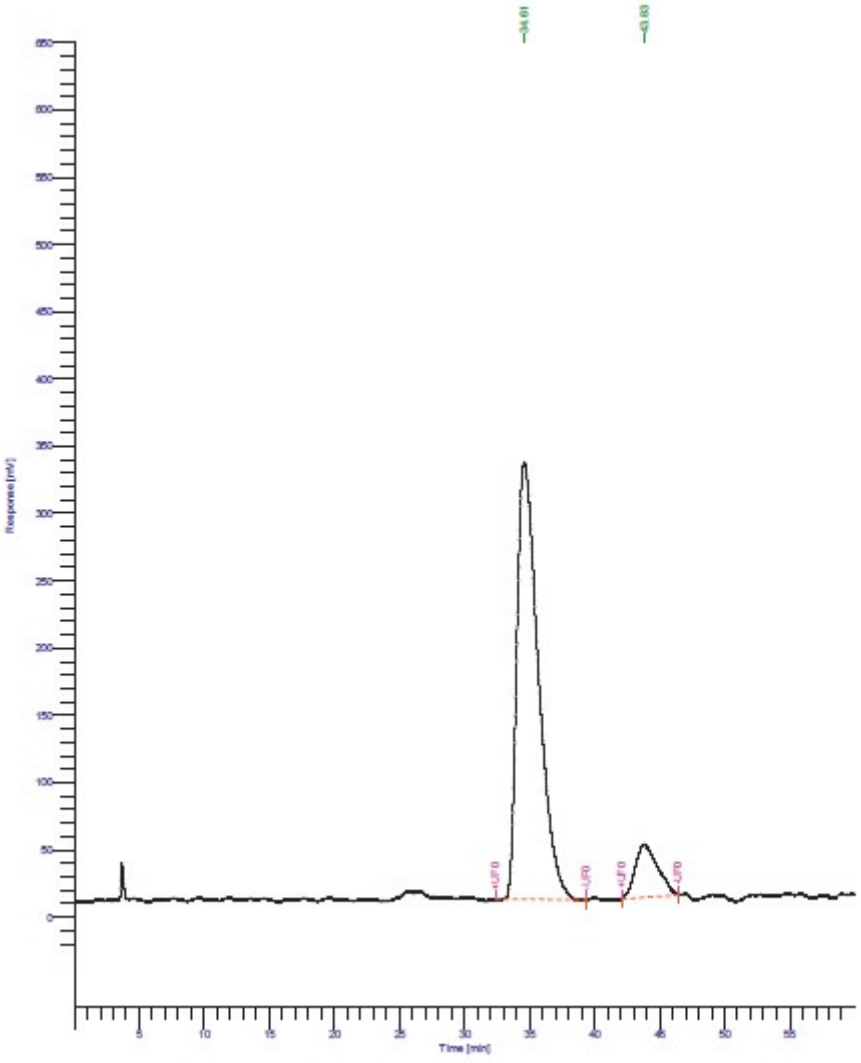
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		34.613	37103064.69	325522.29	88.80	88.80			*MM	37.1031	37.1031
2		43.831	4677467.07	39760.95	11.20	11.20			*MM	4.6775	4.6775
			41780531.77	365283.24	100.00	100.00				41.7805	41.7805

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



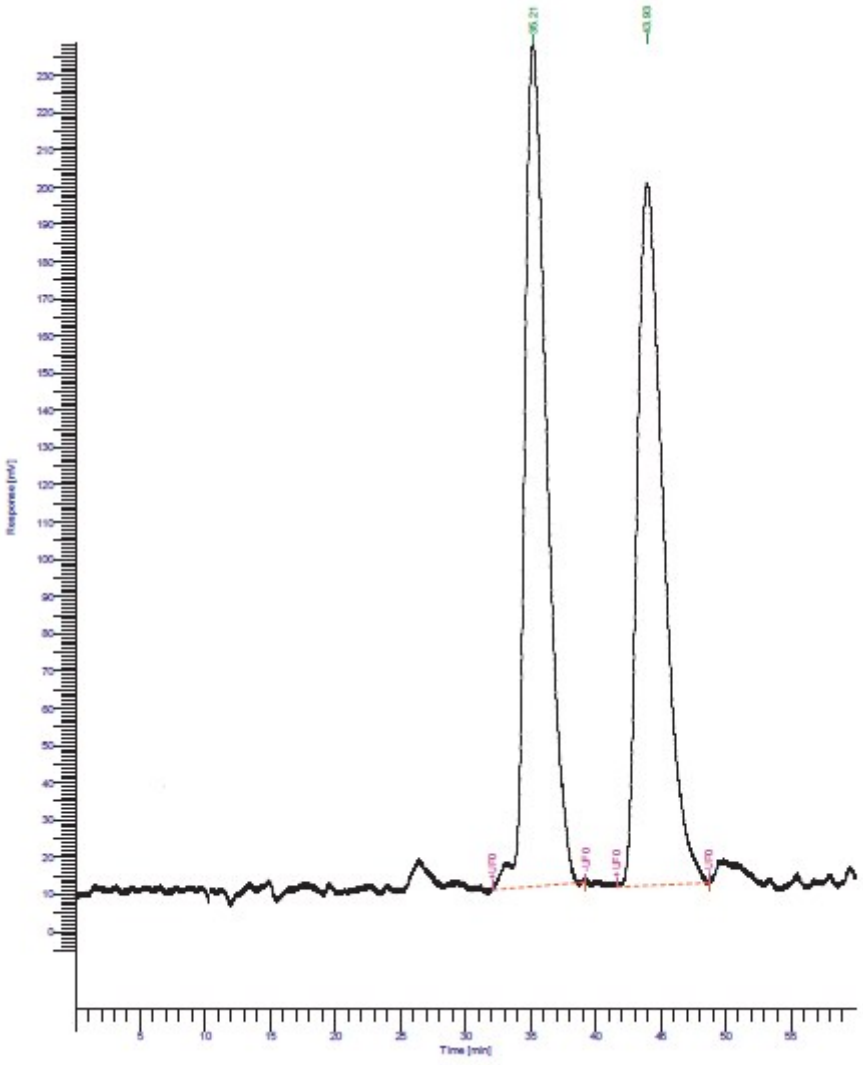
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		35.207	26563135.61	226695.04	50.36	50.36			*MM	26.5631	26.5631
2		43.927	26179152.07	188835.90	49.64	49.64			*MM	26.1792	26.1792
			52742287.68	415530.95	100.00	100.00				52.7423	52.7423

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12g**

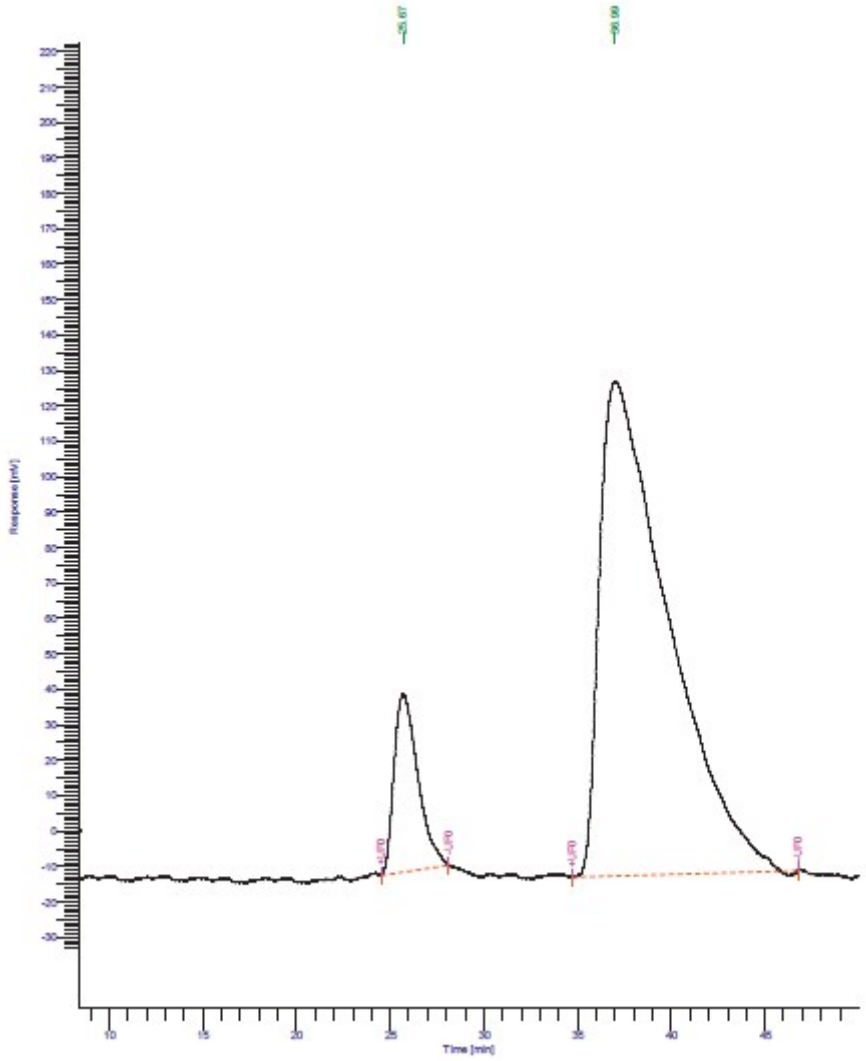
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		25.674	4448985.24	50324.09	11.12	11.12			*MM	4.4490	4.4490
2		36.989	35562382.71	139722.87	88.88	88.88			*MM	35.5624	35.5624
			40011367.95	190046.96	100.00	100.00				40.0114	40.0114

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



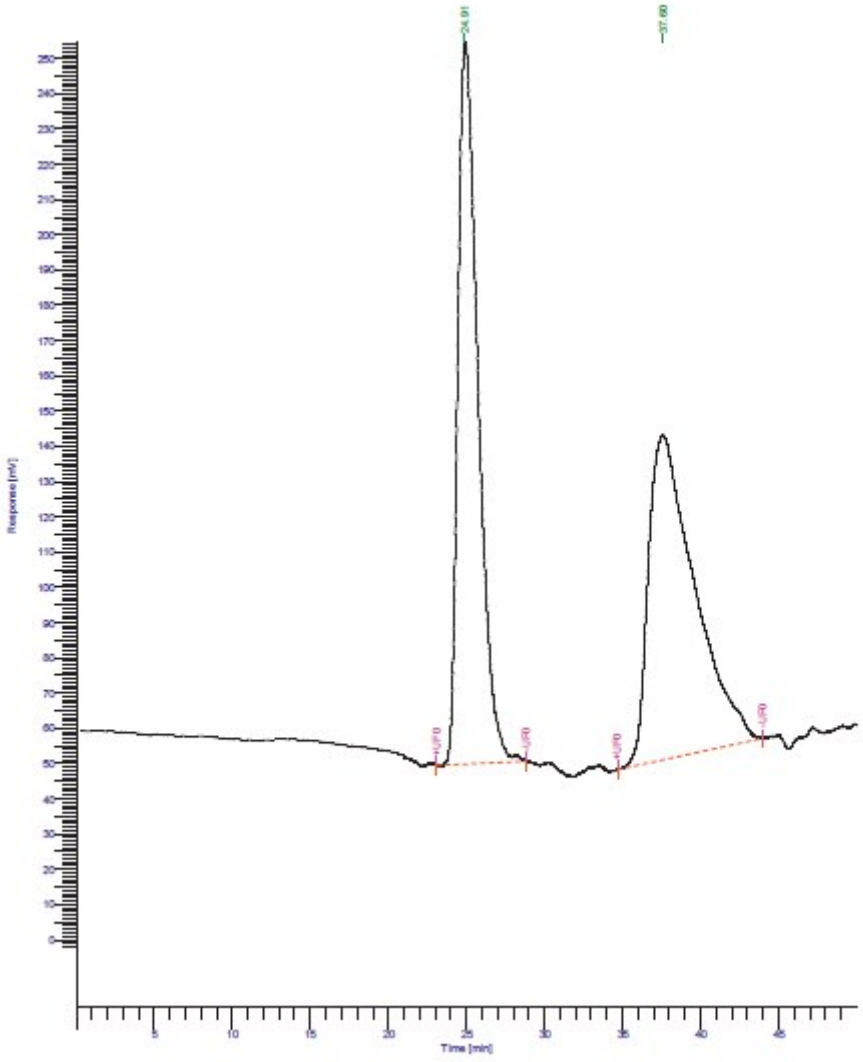
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		24.911	18370503.68	204976.06	49.32	49.32			*MM	18.3705	18.3705
2		37.598	18877094.71	92203.47	50.68	50.68			*MM	18.8771	18.8771
			37247598.39	297179.53	100.00	100.00				37.2476	37.2476

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12h**

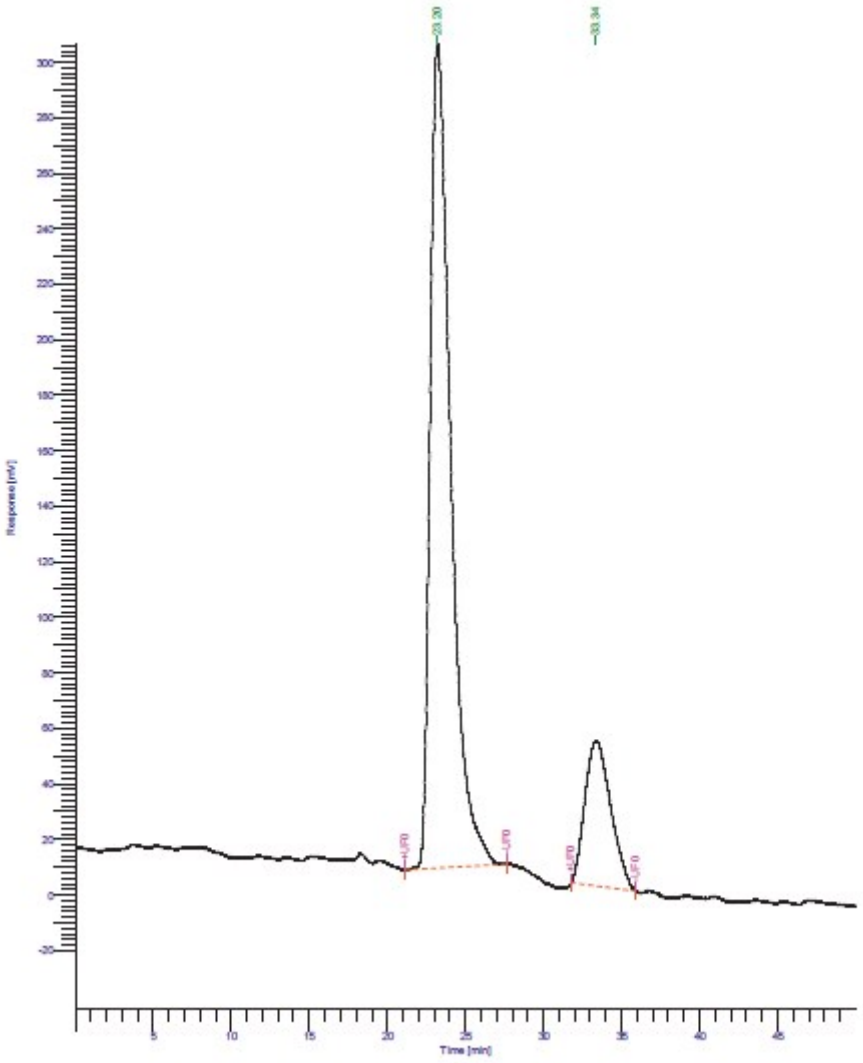
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		23.203	27189471.95	297123.05	81.83	81.83			*MM	27.1895	27.1895
2		33.337	6037084.29	52537.60	18.17	18.17			*MM	6.0371	6.0371
			33226556.24	349660.65	100.00	100.00				33.2266	33.2266

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



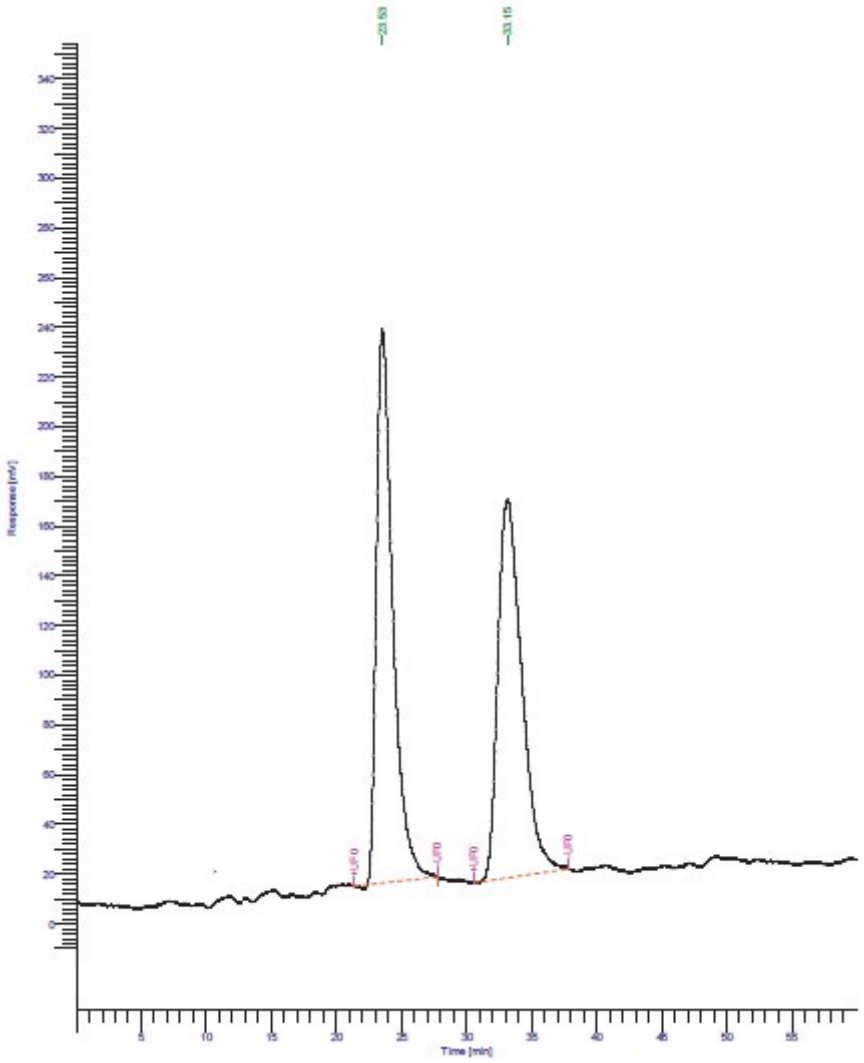
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		23.533	19635723.09	223053.22	50.23	50.23			*MM	19.6357	19.6357
2		33.147	19456369.47	152430.82	49.77	49.77			*MM	19.4564	19.4564
			39092092.56	375484.04	100.00	100.00				39.0921	39.0921

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12i**

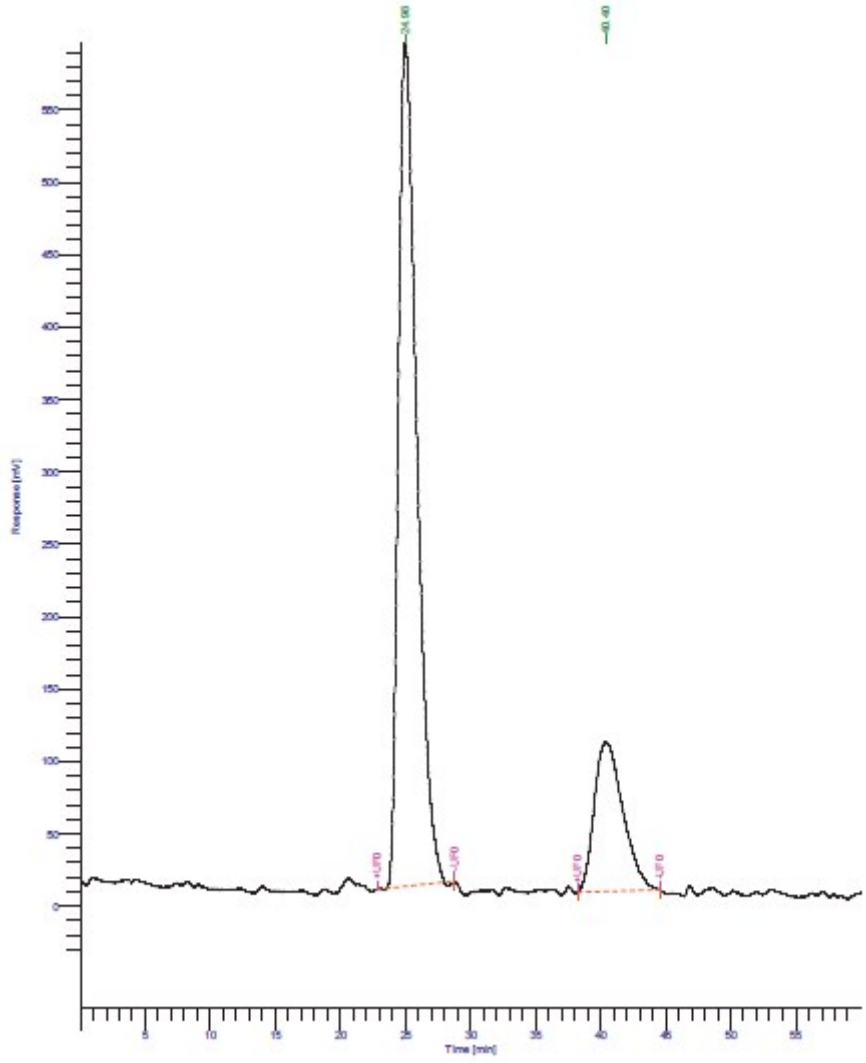
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		24.985	57735652.32	582720.26	78.47	78.47			*MM	57.7357	57.7357
2		40.399	15844162.34	103273.21	21.53	21.53			*MM	15.8442	15.8442
			73579814.66	685993.47	100.00	100.00				73.5798	73.5798

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



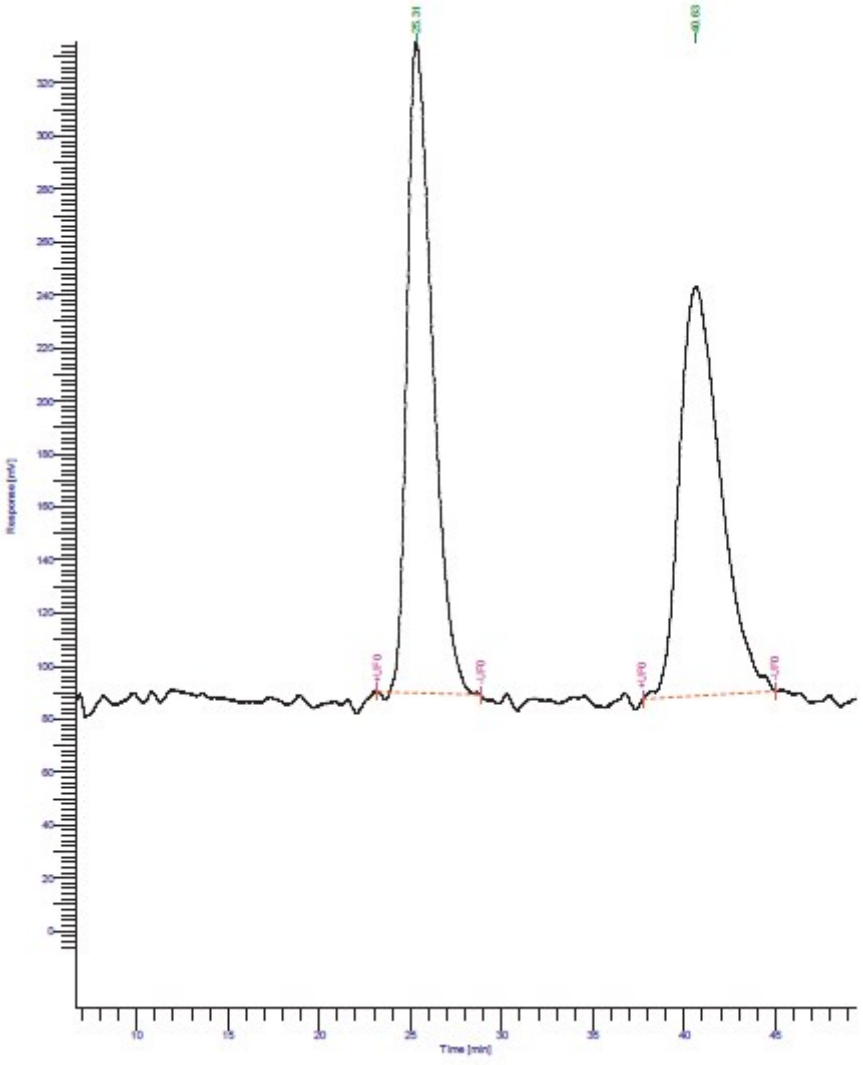
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		25.315	23737183.13	245816.76	49.75	49.75			*MM	23.7372	23.7372
2		40.627	23973885.17	154552.27	50.25	50.25			*MM	23.9739	23.9739
			47711068.30	400369.03	100.00	100.00				47.7111	47.7111

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12k**

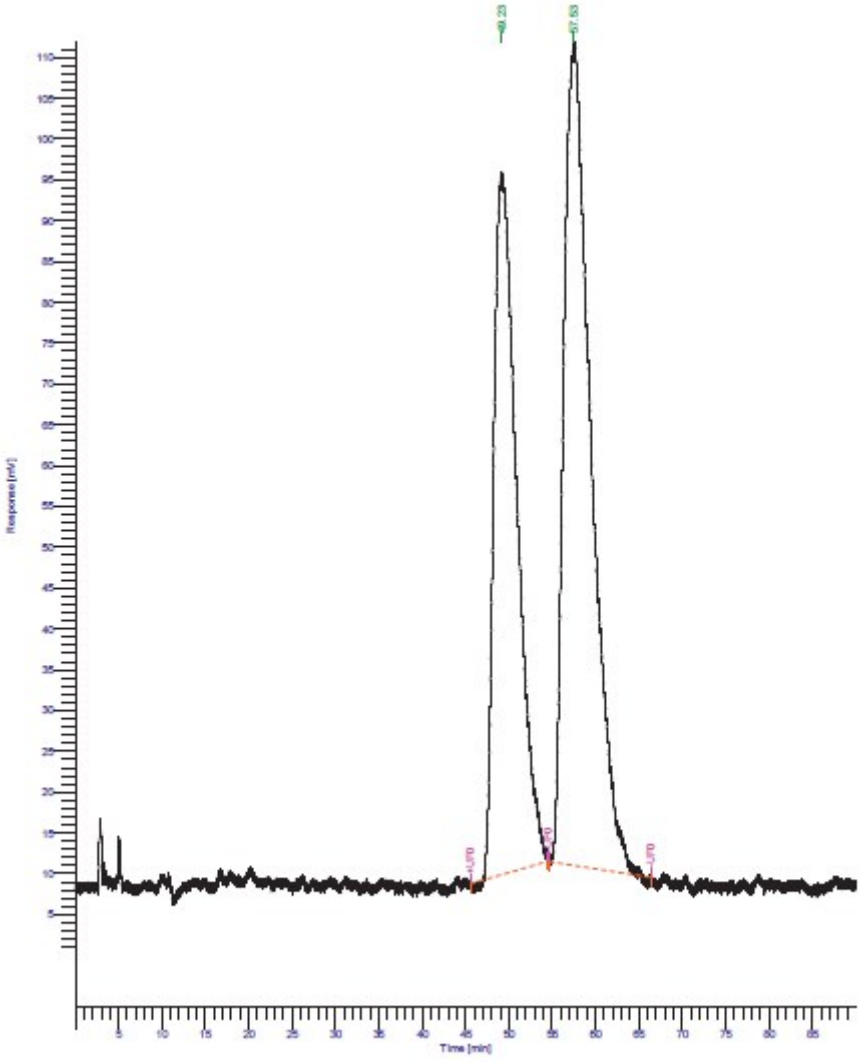
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		49.229	15949776.94	86099.11	42.05	42.05			*MM	15.9498	15.9498
2		57.530	21983489.96	100903.09	57.95	57.95			*MM	21.9835	21.9835
			37933266.89	187002.21	100.00	100.00				37.9333	37.9333

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



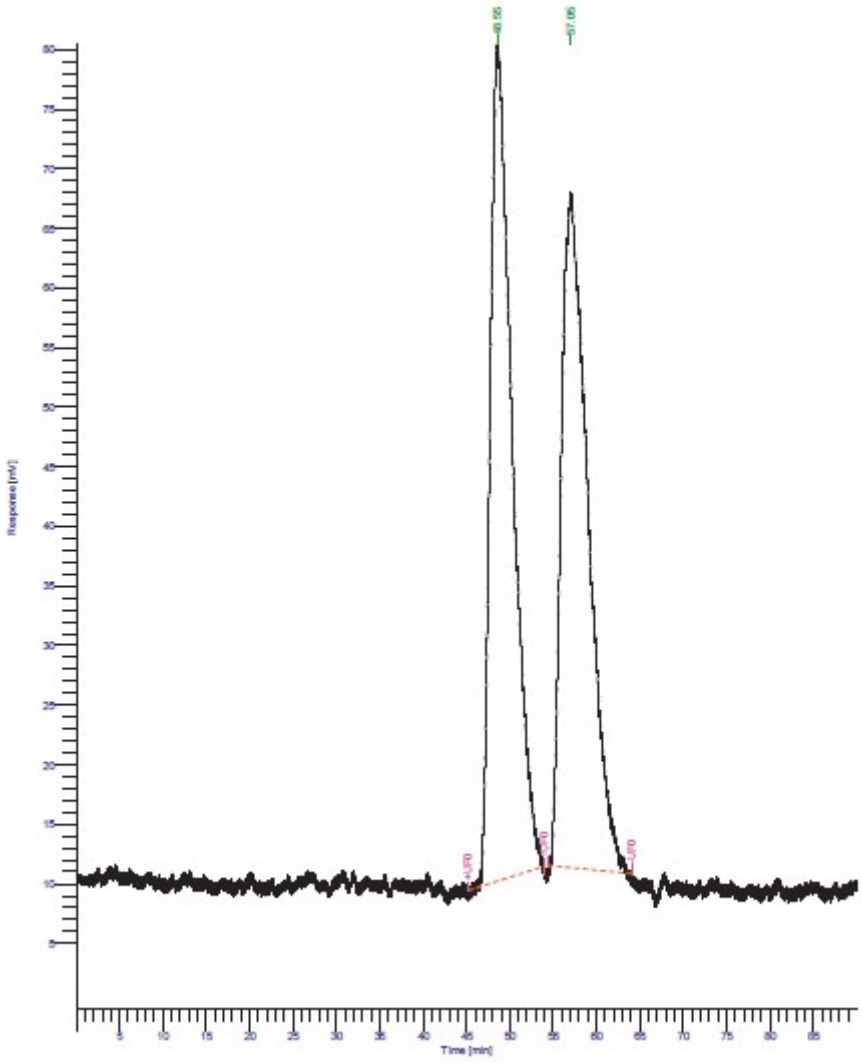
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		48.547	12429462.41	70311.34	50.53	50.53			*MM	12.4295	12.4295
2		57.046	12168215.36	56666.62	49.47	49.47			*MM	12.1682	12.1682
			24597677.77	126977.96	100.00	100.00				24.5977	24.5977

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12l**

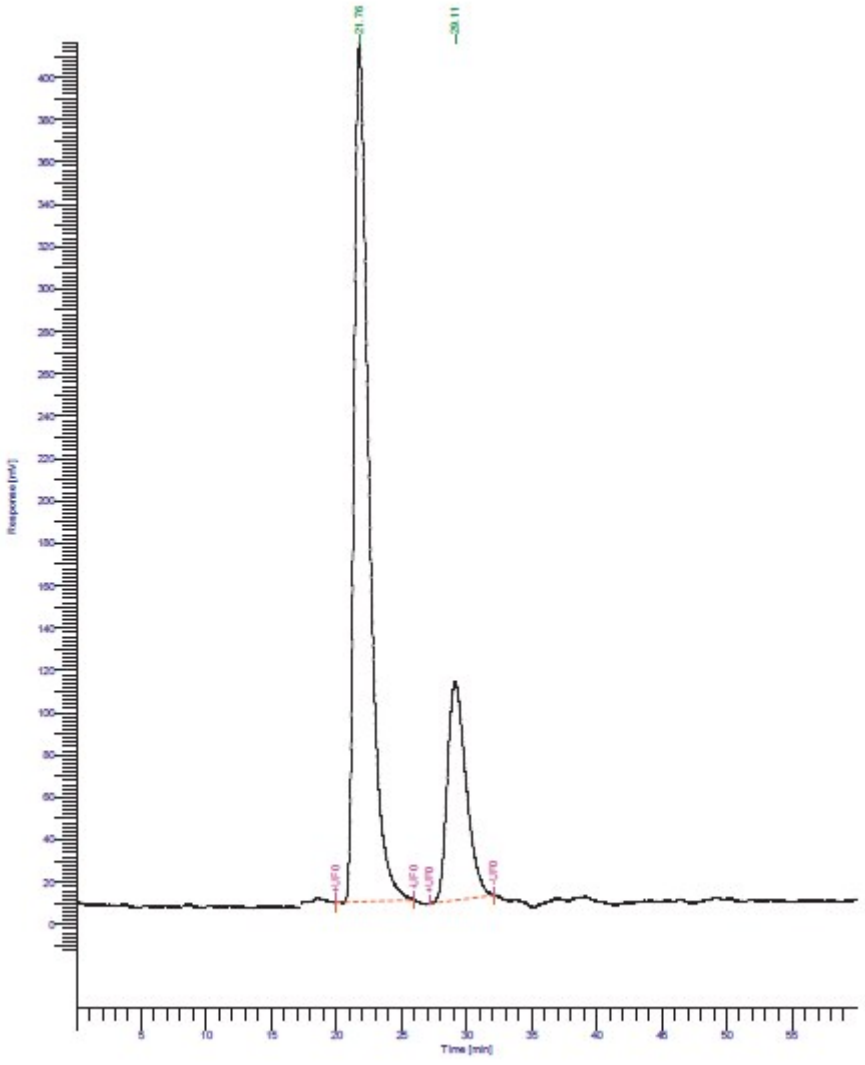
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		21.758	32223943.20	405645.44	75.70	75.70			*MM	32.2239	32.2239
2		29.113	10346614.71	102926.26	24.30	24.30			*MM	10.3466	10.3466
			42570557.90	508571.70	100.00	100.00				42.5706	42.5706

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



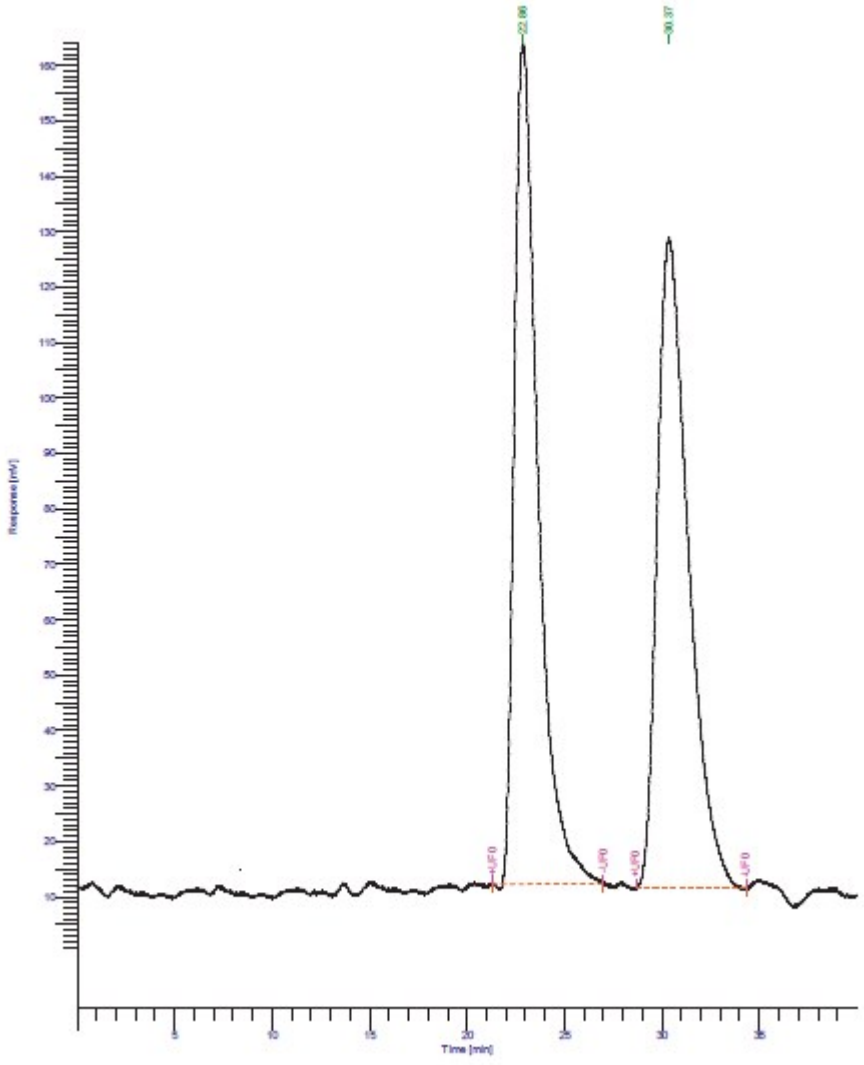
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		22.858	12787494.65	151790.77	49.48	49.48			*MM	12.7875	12.7875
2		30.367	13057762.31	117275.23	50.52	50.52			*MM	13.0578	13.0578
			25845256.97	269066.00	100.00	100.00				25.8453	25.8453

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12m**

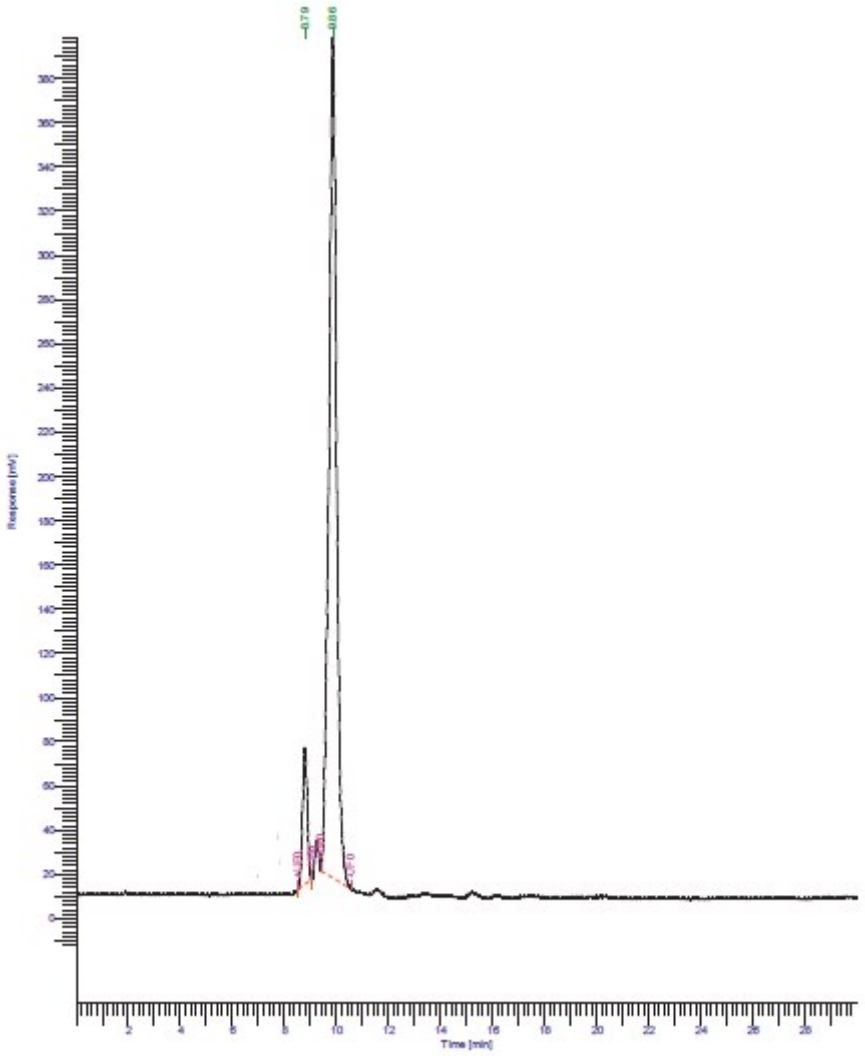
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		8.793	779367.04	61732.12	9.03	9.03			*MM	0.7794	0.7794
2		9.863	7852353.06	380237.86	90.97	90.97			*MM	7.8524	7.8524
			8631720.11	441969.98	100.00	100.00				8.6317	8.6317

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



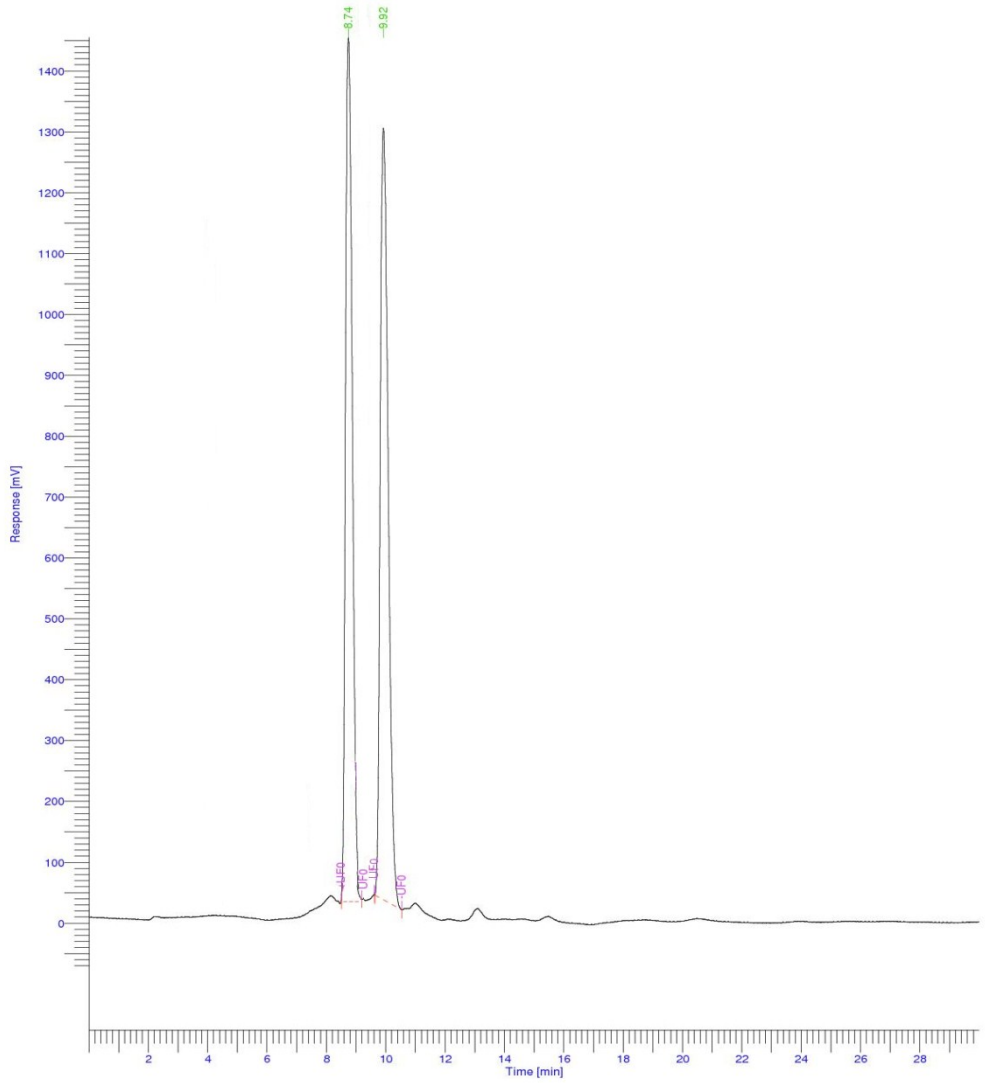
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		8.741	20650676.04	1.22e+06	49.48	49.48			*MM	20.6507	20.6507
2		9.922	21086575.66	1.17e+06	50.52	50.52			*MM	21.0866	21.0866
			41737251.70	2.40e+06	100.00	100.00				41.7373	41.7373

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12n**

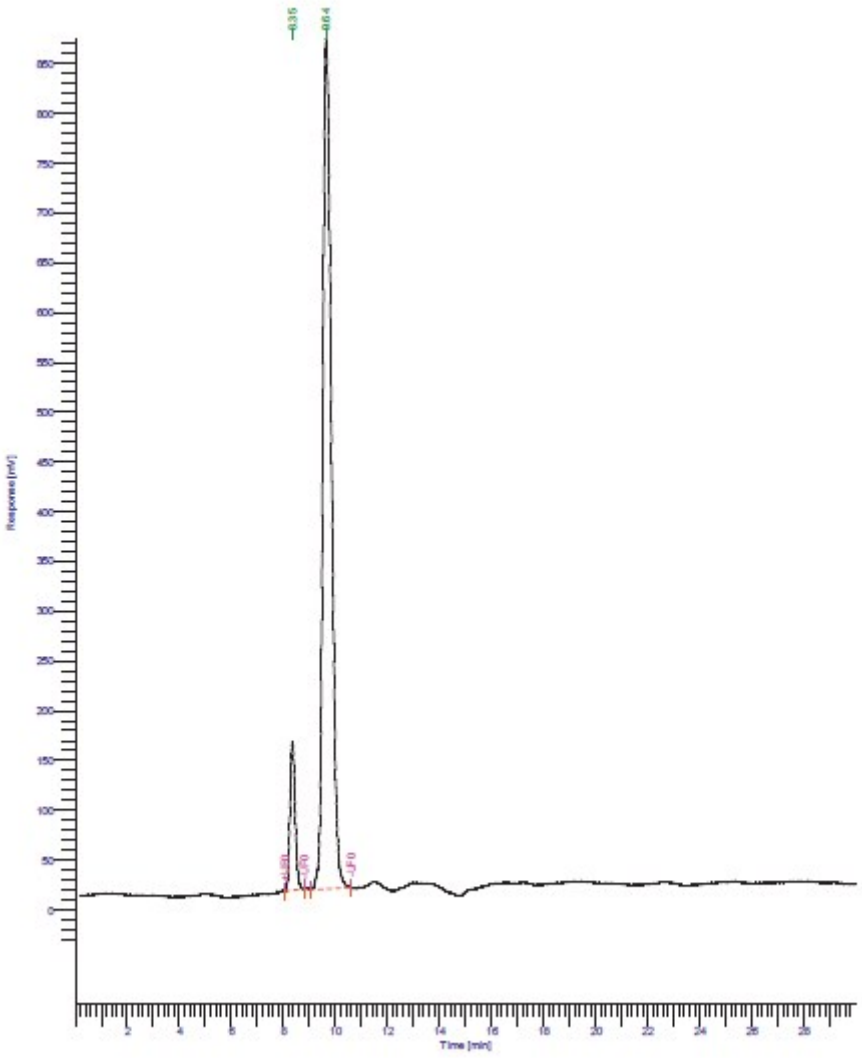
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		8.353	1815200.50	141022.64	8.55	8.55			*MM	1.8152	1.8152
2		9.643	19421651.98	854393.49	91.45	91.45			*MM	19.4217	19.4217
			21236852.48	995416.13	100.00	100.00				21.2369	21.2369

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



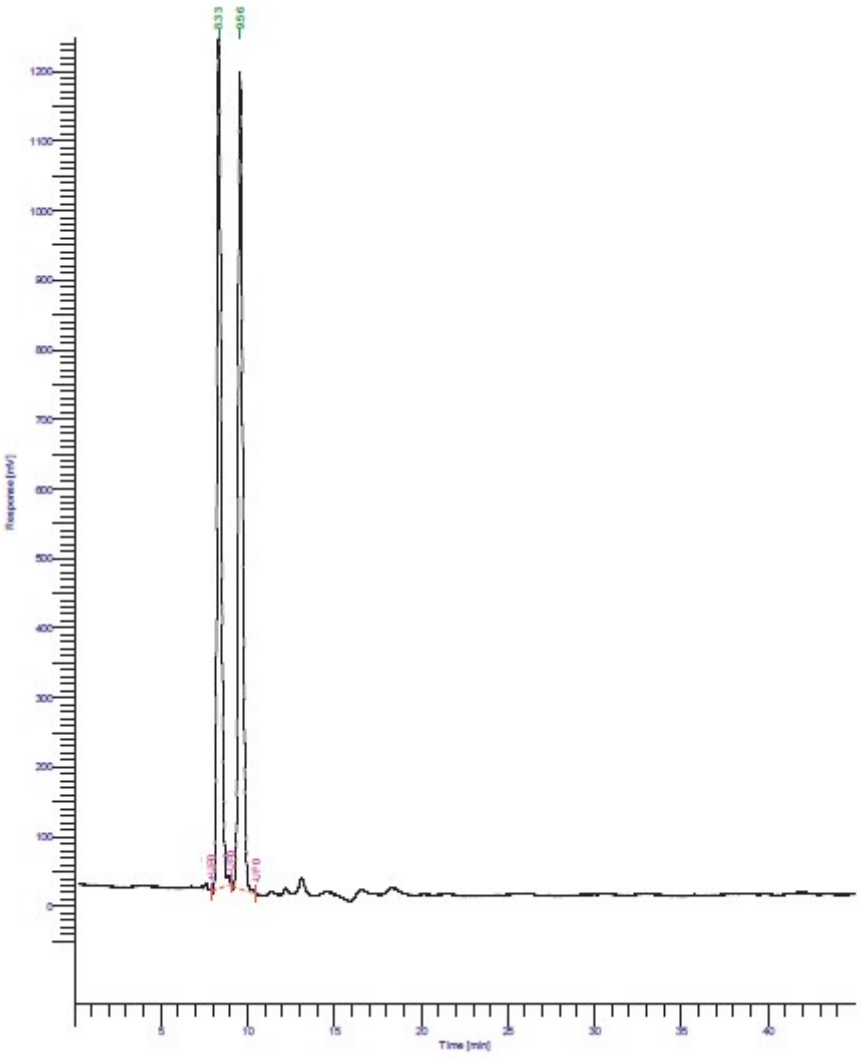
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		8.331	20650676.04	1.22e+06	49.48	49.48			*MM	20.6507	20.6507
2		9.563	21086575.66	1.17e+06	50.52	50.52			*MM	21.0866	21.0866
			41737251.70	2.40e+06	100.00	100.00				41.7373	41.7373

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **12o**

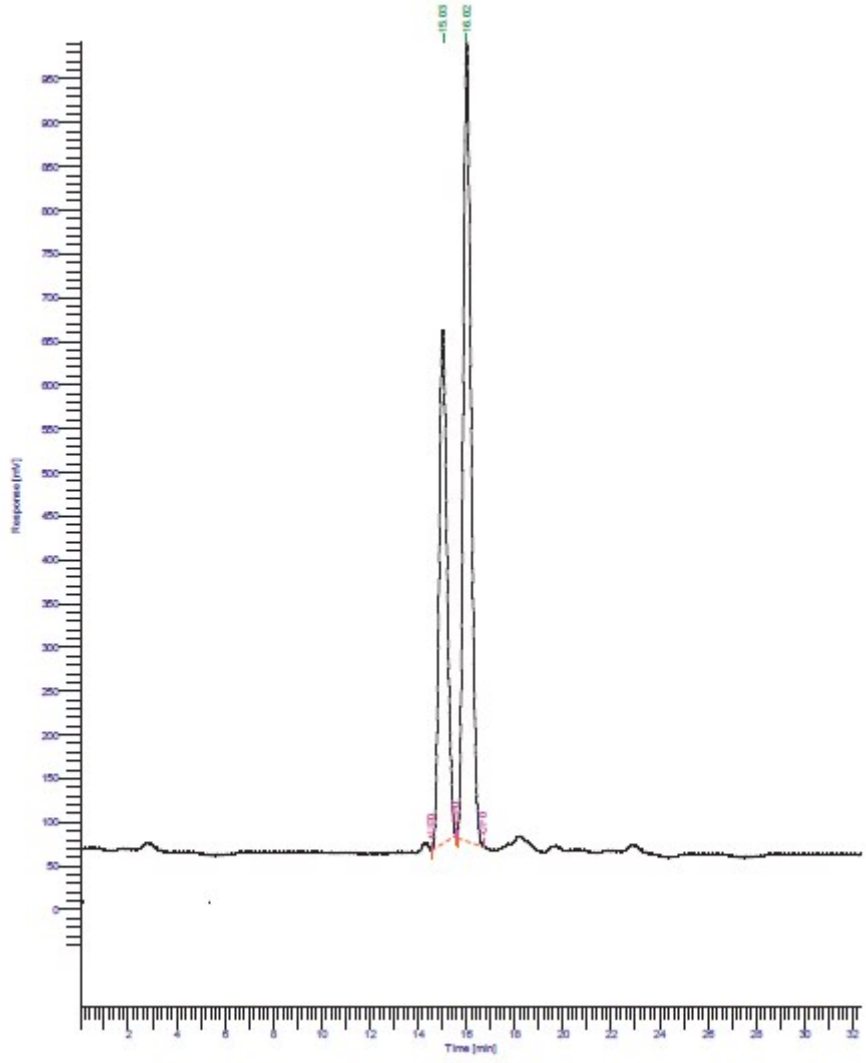
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		15.026	13394492.95	586781.42	39.51	39.51			*MM	13.3945	13.3945
2		16.016	20505366.36	914804.91	60.49	60.49			*MM	20.5054	20.5054
			33899859.31	1.50e+06	100.00	100.00				33.8999	33.8999

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



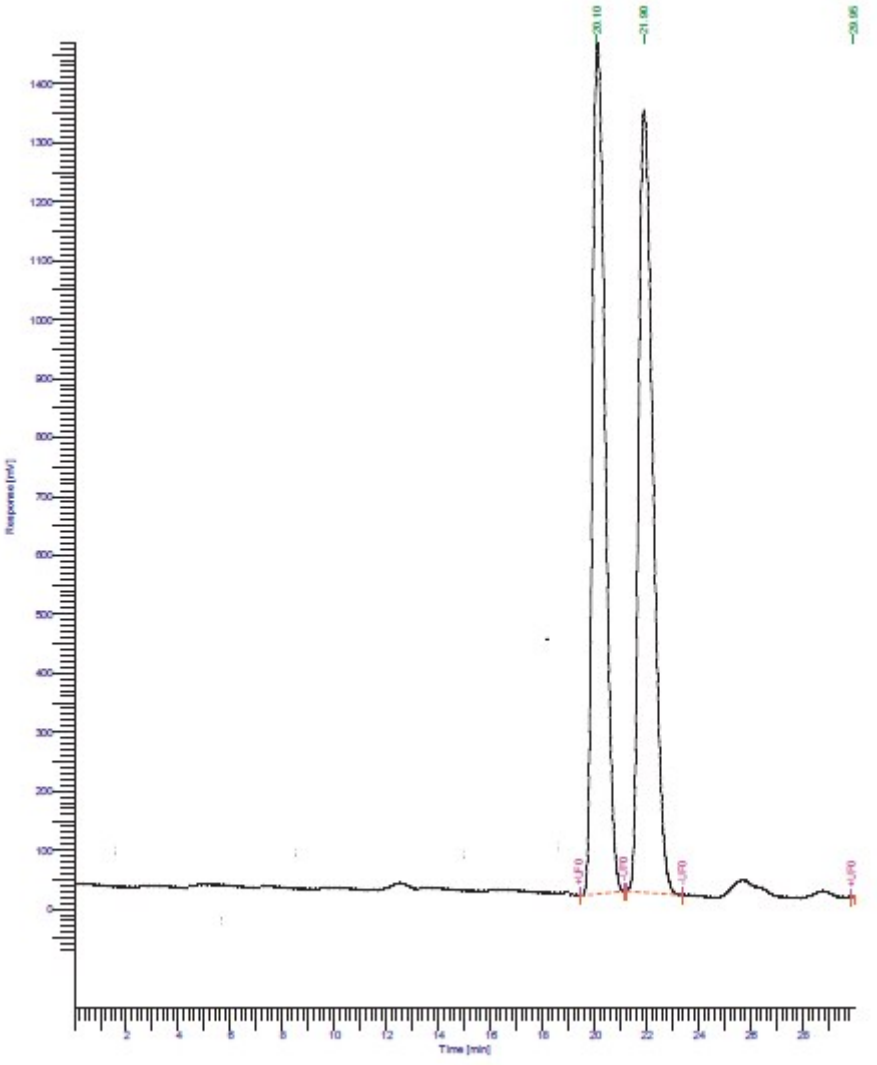
DEFAULT REPORT

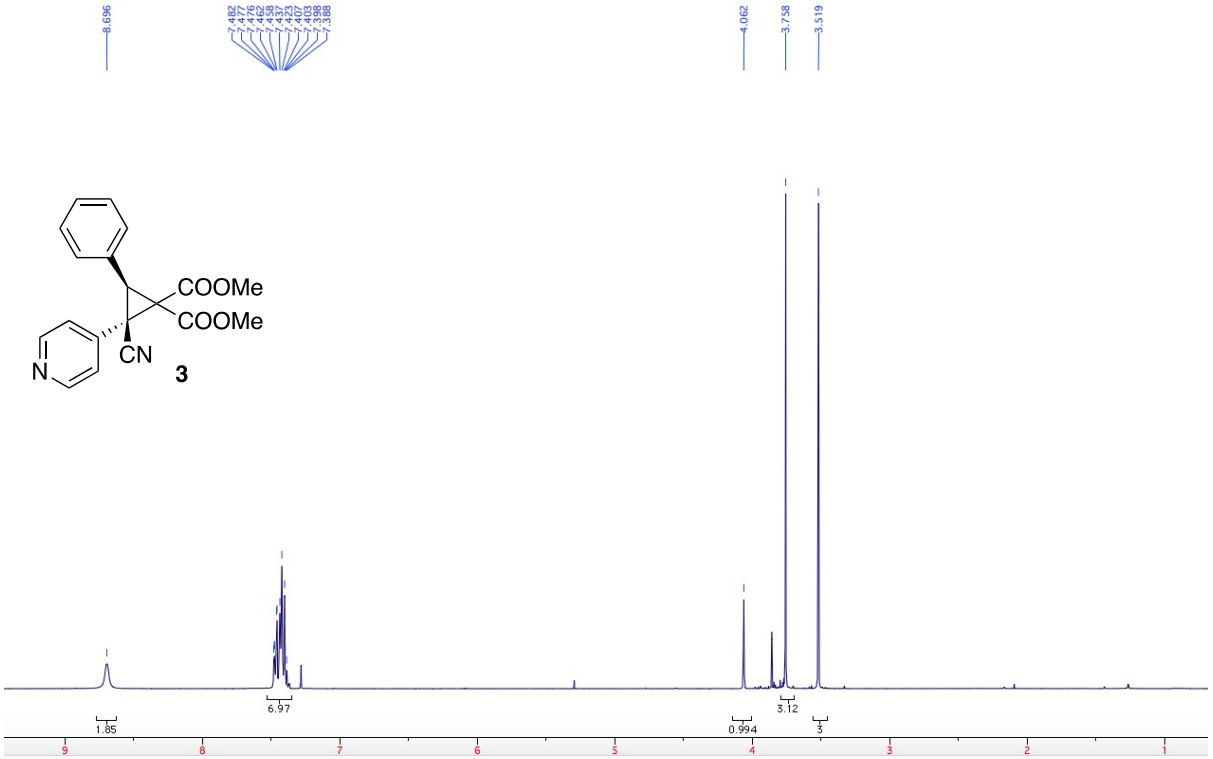
Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		20.101	48829867.44	1.44e+06	49.09	49.09			*MM	48.8299	48.8299
2		21.897	50629283.15	1.33e+06	50.90	50.90			*MM	50.6293	50.6293
3		29.949	2691.12	1235.16	0.00	0.00			*MM	0.0027	0.0027
			99461841.71	2.77e+06	100.00	100.00				99.4618	99.4618

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found

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7.476
7.472
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7.403
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COOMe
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3
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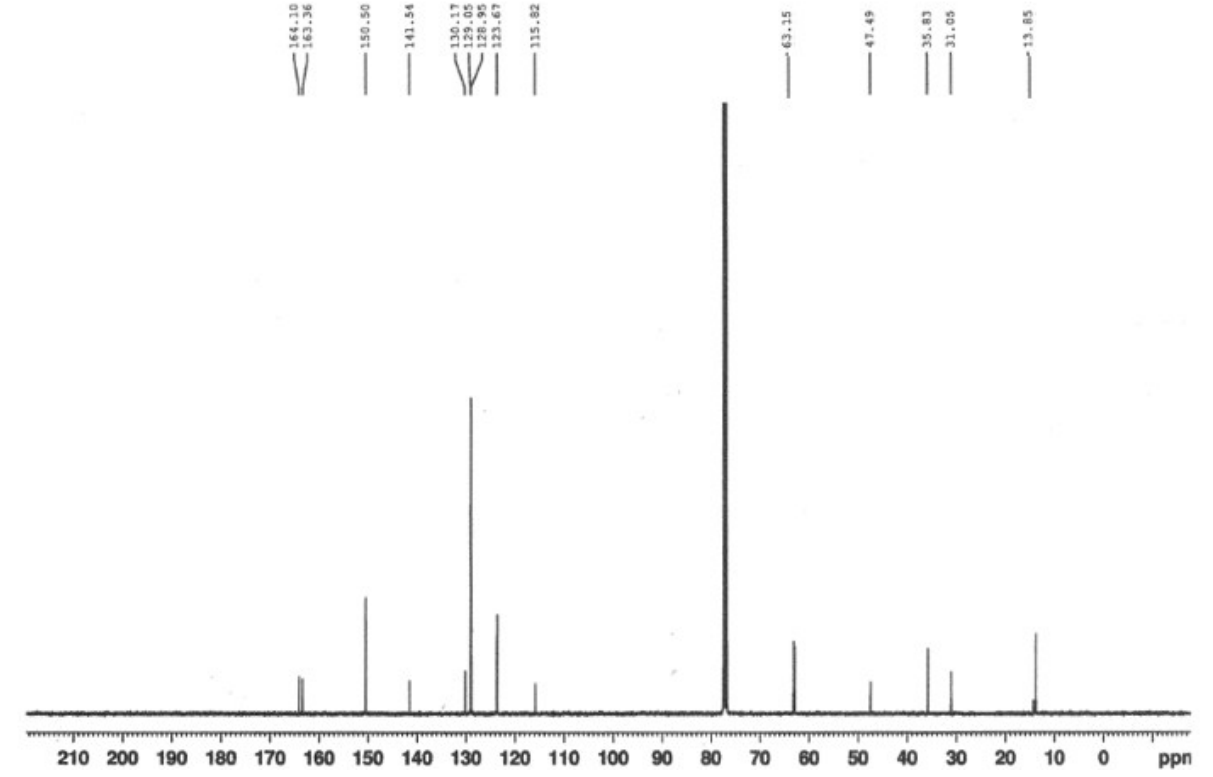
0.994
3.12
3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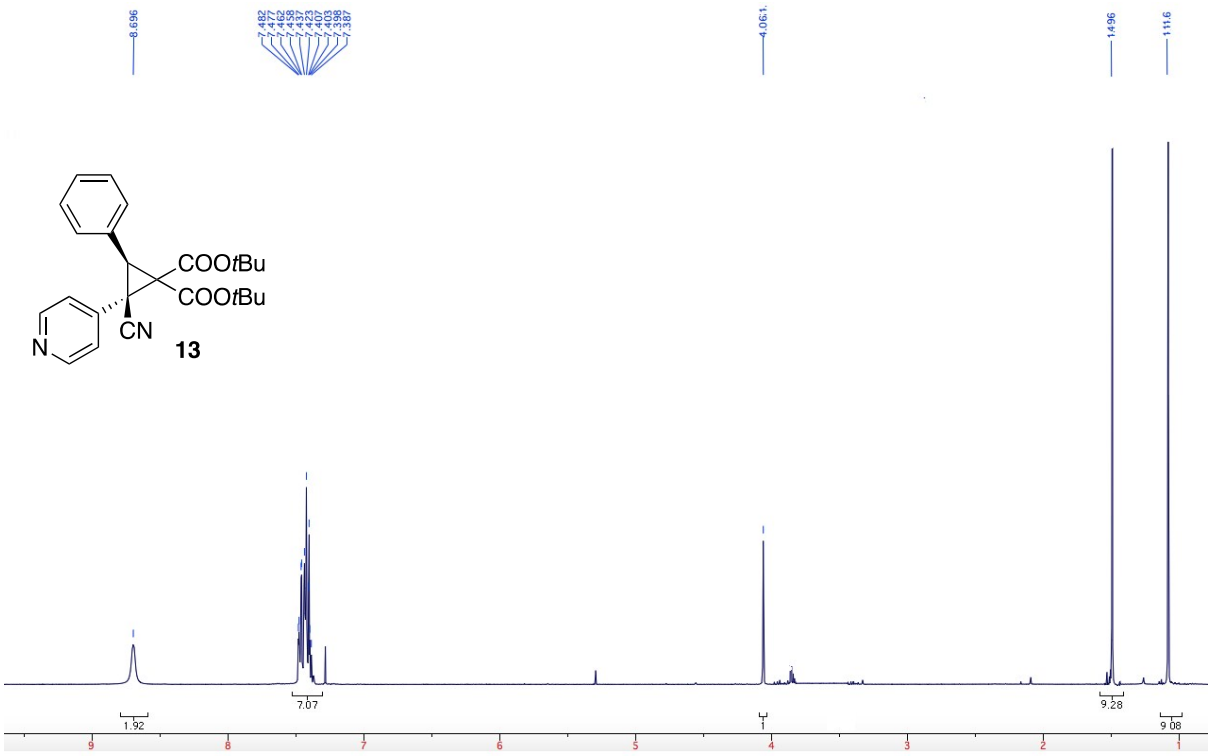
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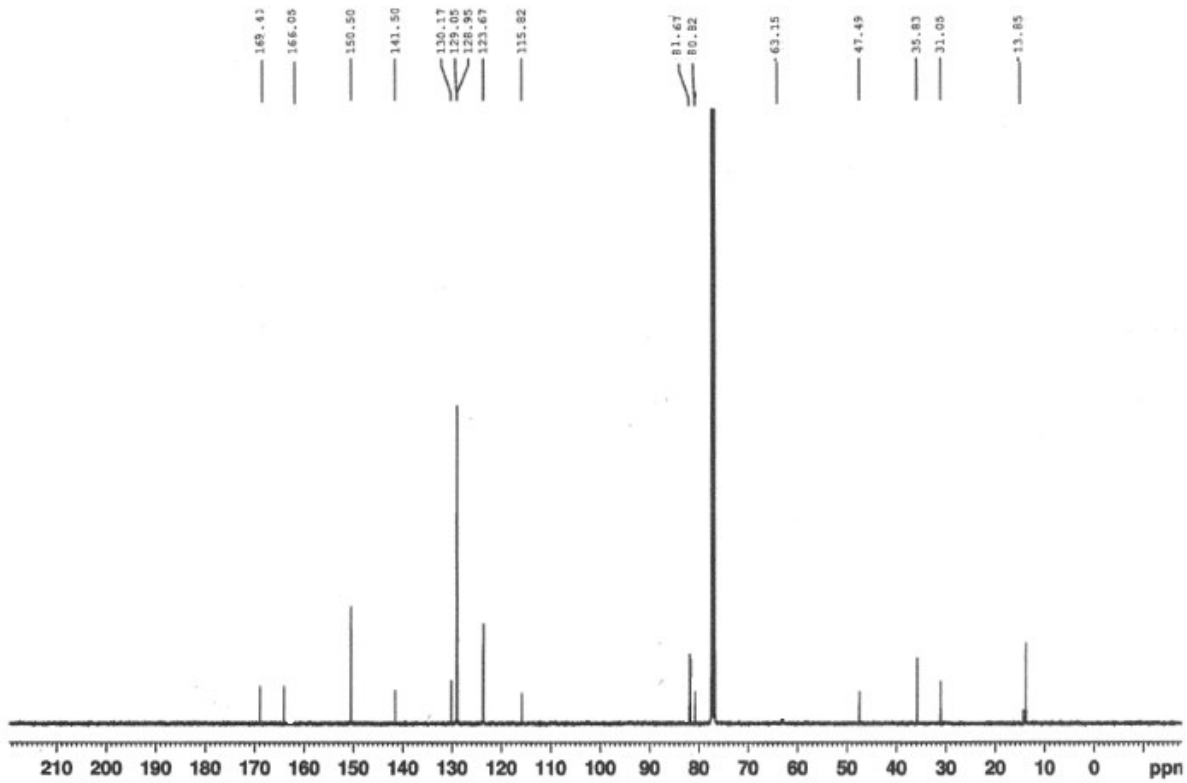
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150.50
141.50
130.17
129.05
128.95
123.67
115.82
81.67
80.82
63.15
47.49
35.83
31.05
13.85
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Compound 3

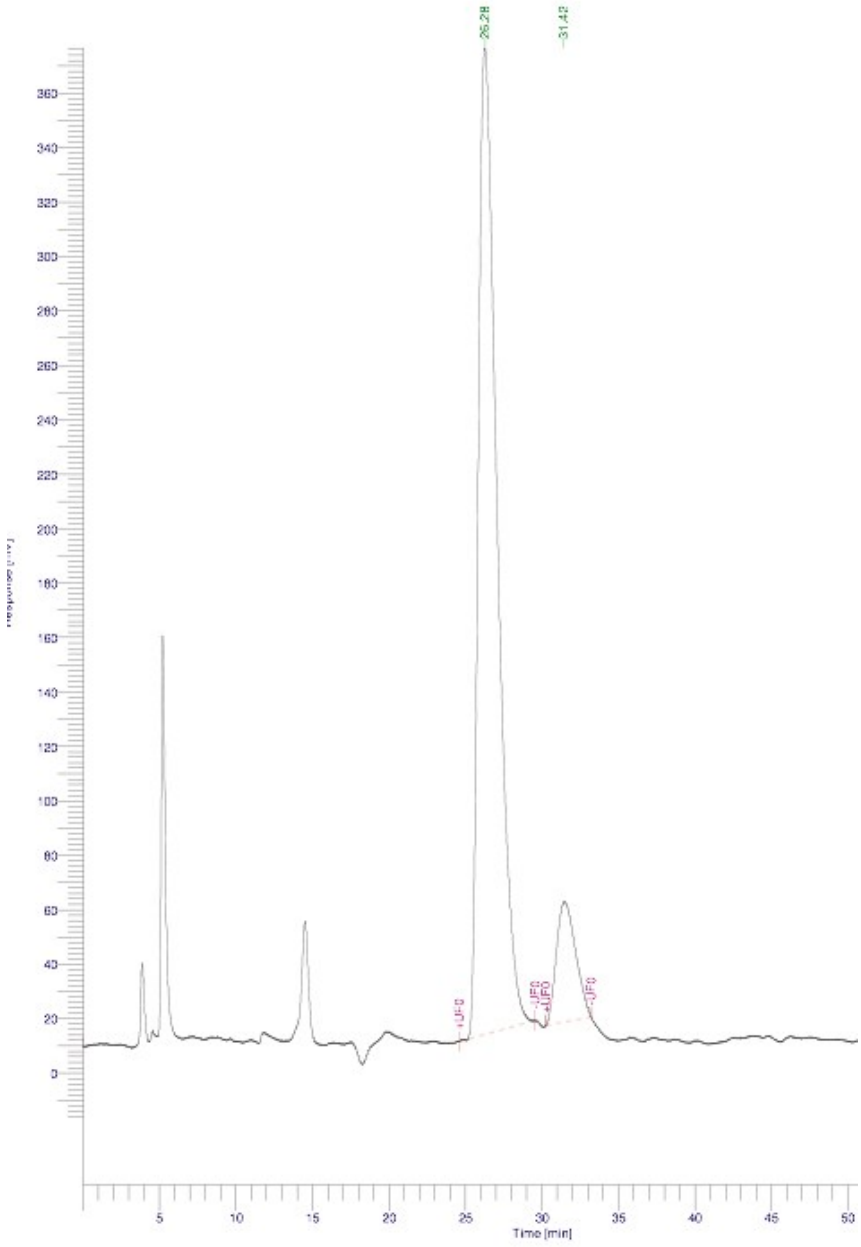
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		26.275	30974526.90	362318.32	88.92	88.92			*MM	30.9745	30.9745
2		31.416	3859334.41	44074.25	11.08	11.08			*MM	3.8593	3.8593
			34833861.31	406392.57	100.00	100.00				34.8339	34.8339

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



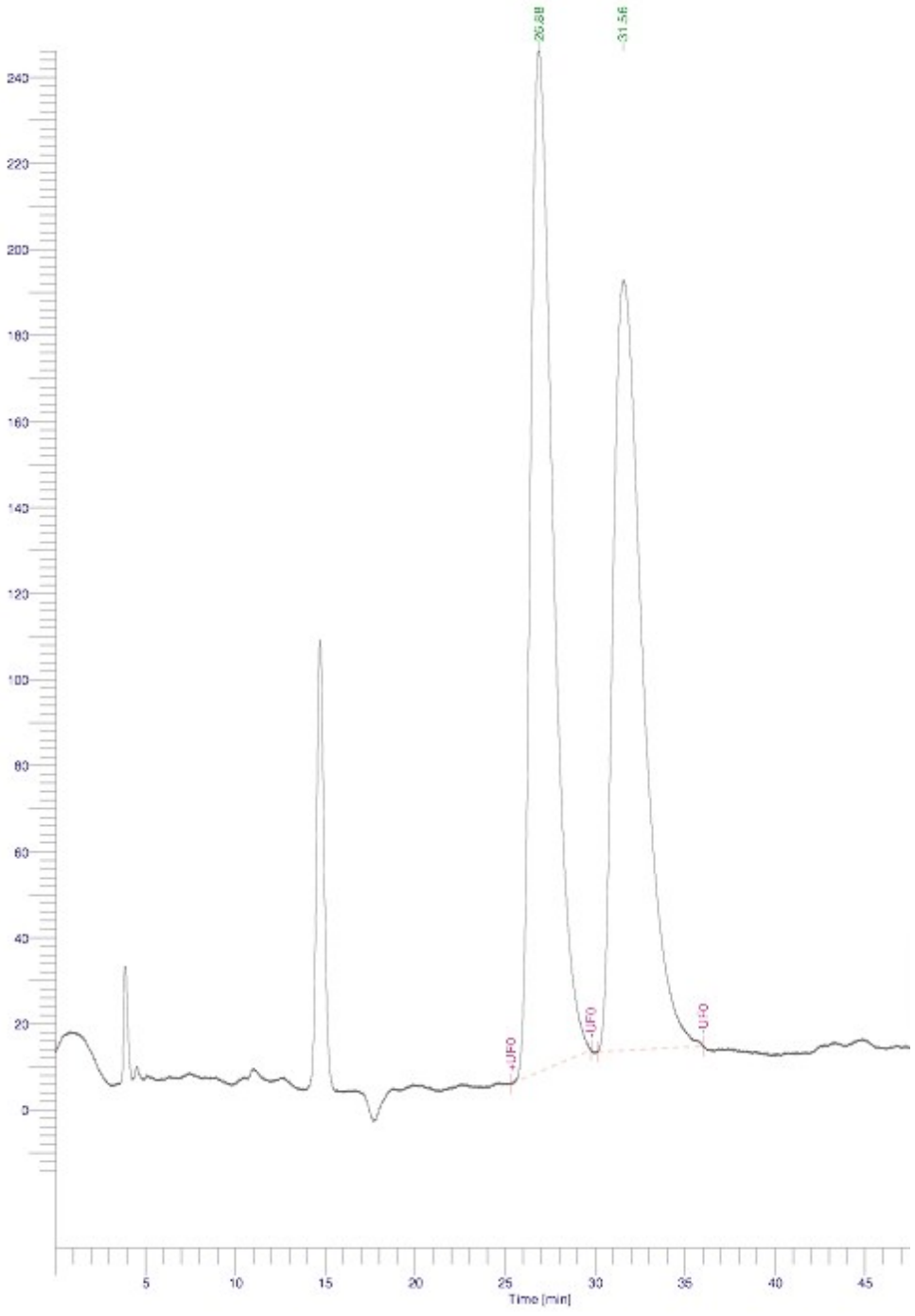
DEFAULT REPORT

Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		26.884	20866612.39	237380.66	50.46	50.46			*MM	20.8666	20.8666
2		31.555	20485605.08	179036.05	49.54	49.54			*MM	20.4856	20.4856
			41352217.48	416416.71	100.00	100.00				41.3522	41.3522

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



Compound **13**

DEFAULT REPORT

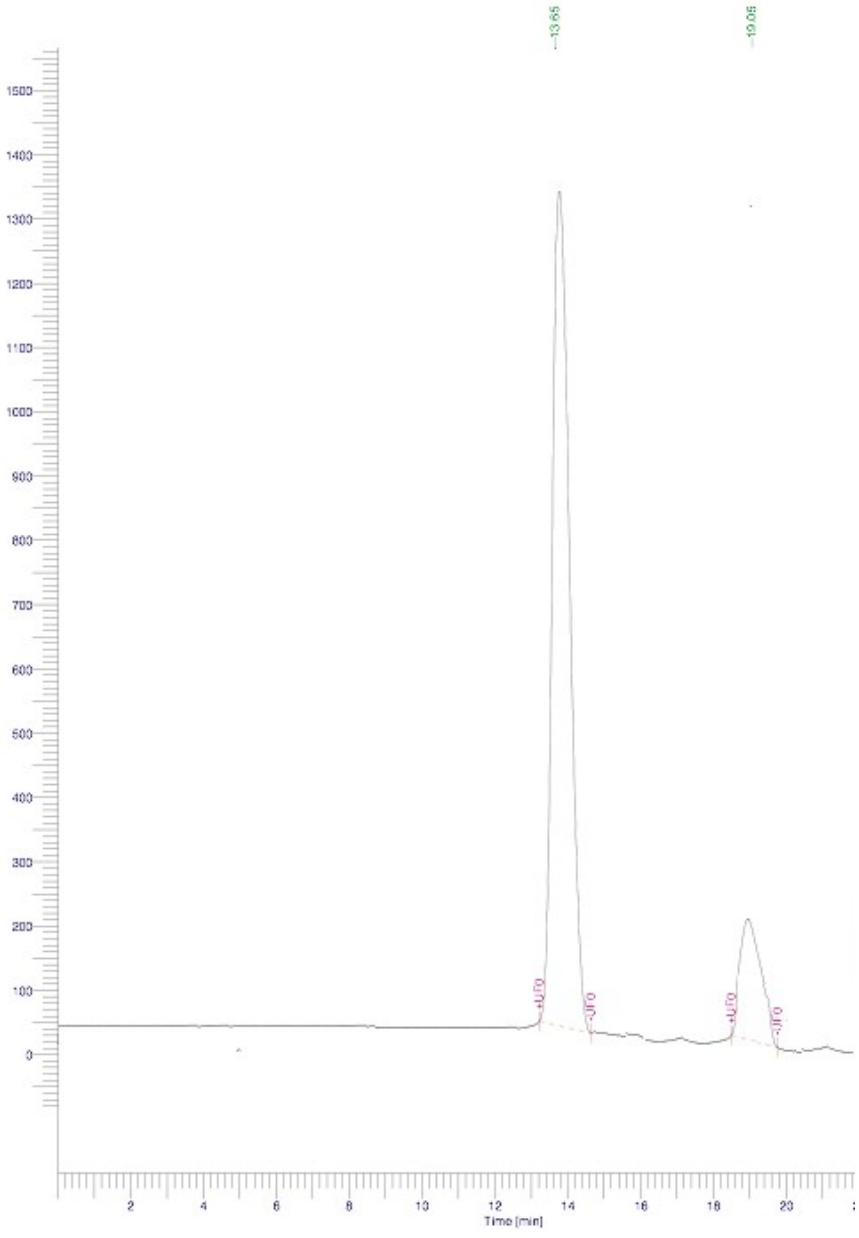
Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		13.655	30974526.90	362318.32	82.24	82.24		+	*MM	30.9745	30.9745
2		19.052	3859334.41	44074.25	17.76	17.76			*MM	3.8593	3.8593
			34833861.31	406392.57	100.00	100.00				34.8339	434.8339

Warning -- Signal level out-of-range in peak

Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



DEFAULT REPORT

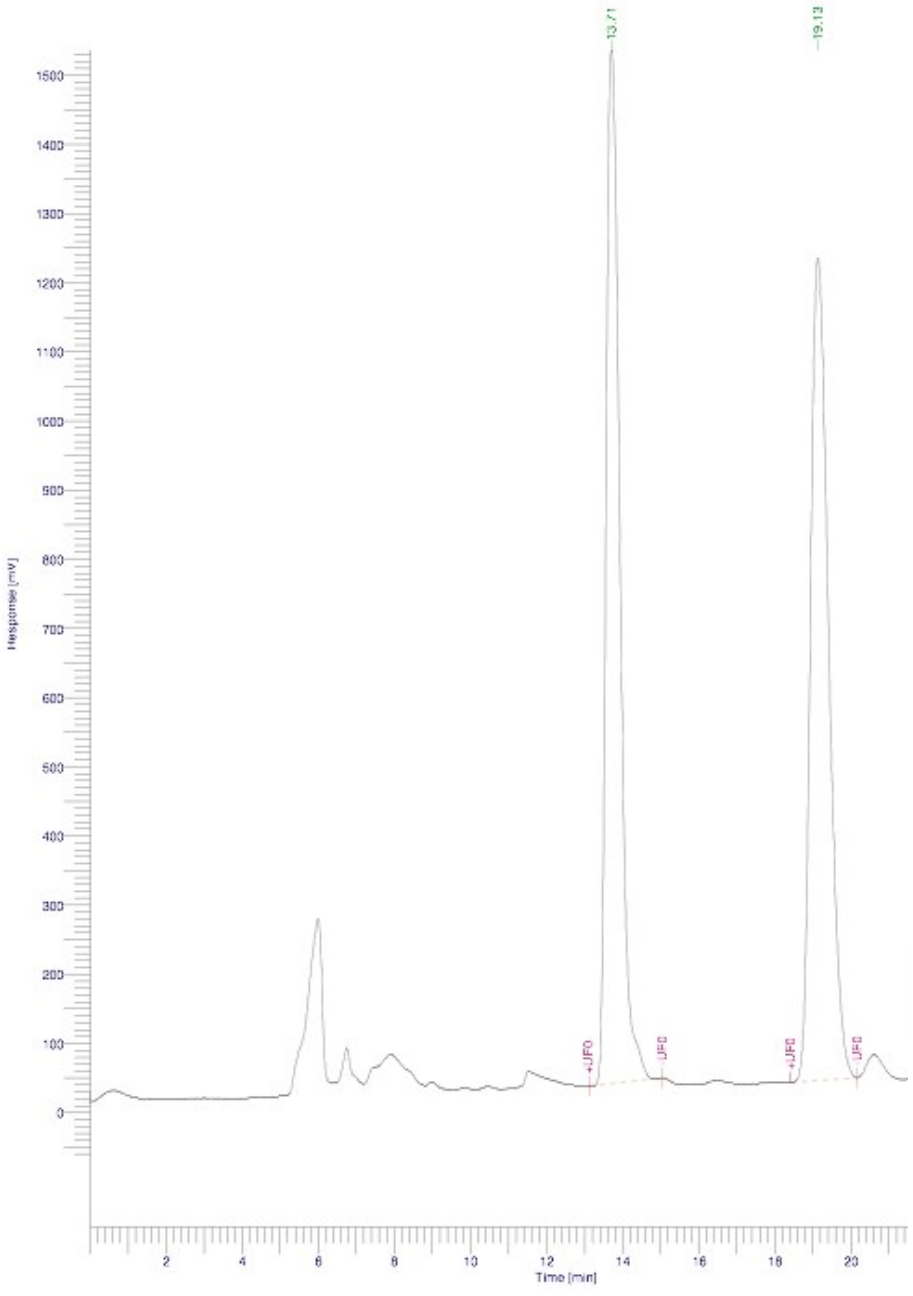
Peak #	Component Name	Time [min]	Area [uV*sec]	Height [uV]	Area [%]	Norm. Area [%]	Cal. Range	Volt Range	BL	Raw Amount	Adjusted Amount
1		13.706	37769910.34	1.49e+06	49.03	49.03		+	*MM	----------	----------
2		19.133	39269801.37	1.19e+06	50.97	50.97			*MM	39.2698	39.2698
			77039711.71	2.68e+06	100.00	100.00				39.2698	39.2698

Warning -- Signal level out-of-range in peak

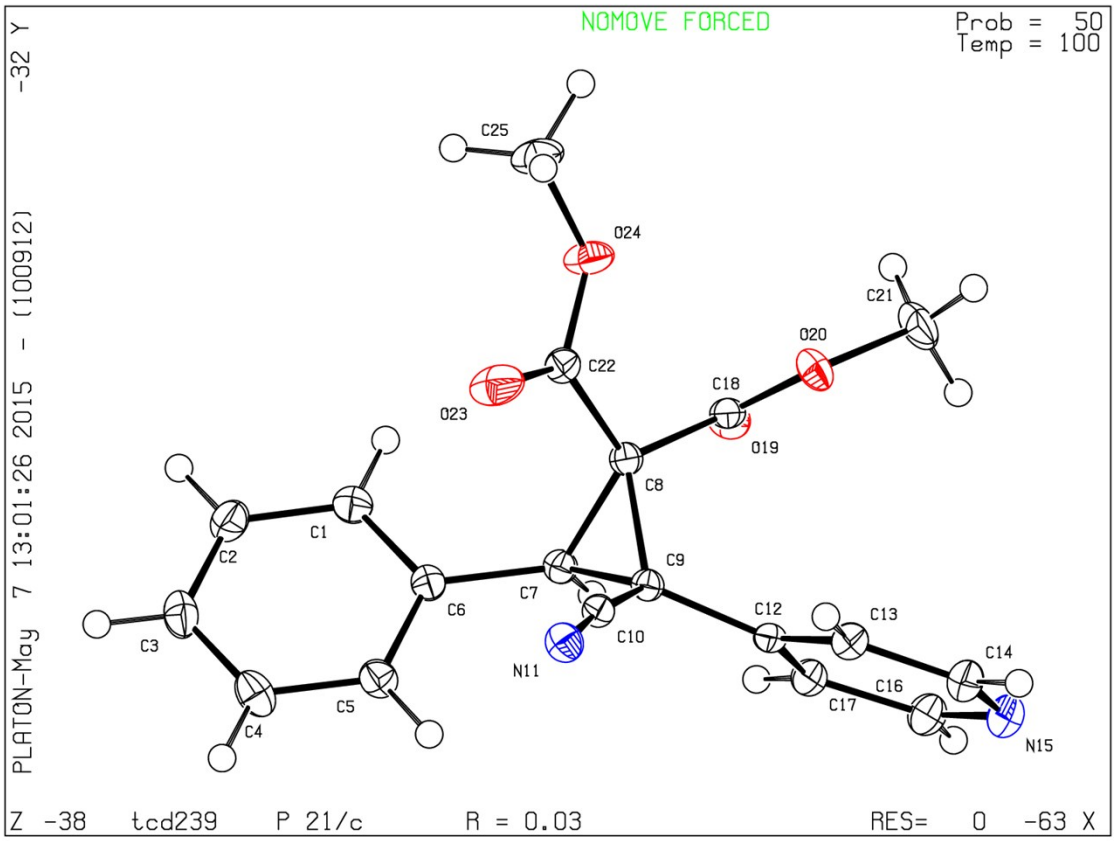
Missing Component Report

Component	Expected Retention (Calibration File)

All components were found



X-ray of compound **3a**



A specimen of $C_{19}H_{16}N_2O_4$, approximate dimensions 0.090 mm x 0.220 mm x 0.320 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 100(2)K using an Oxford Cryosystems Cobra low temperature device using a MiTeGen micromount. See Table 1 for collection parameters and exposure time. Bruker APEX software was used to correct for Lorentz and polarization effects.

A total of 1904 frames were collected. The total exposure time was 9.52 hours. The integration of the data using a monoclinic unit cell yielded a total of 38423 reflections to a maximum θ angle of 30.14° (0.71 Å resolution), of which 4916 were independent (average redundancy 7.816, completeness = 99.8%, R_{int} = 1.92%, R_{sig} = 1.06%) and 4327 (88.02%) were greater than $2\sigma(F^2)$.The final cell constants of a = 9.5875(4) Å, b = 10.4816(5) Å, c = 16.9137(8) Å, β = 100.3989(12)°, volume = 1671.78(13) $Å^3$, are based upon the refinement of the XYZ-centroids of reflections above 20 σ(I).Data were corrected for absorption effects using the multi-scan method (SADABS). The ratio of minimum to maximum apparent transmission was 0.961. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.7166 and 0.7460.

The structure was solved and refined using the Bruker SHELXTL Software Package, using the space group $P2_1/c$, with Z = 4 for the formula unit, $C_{19}H_{16}N_2O_4$.The final anisotropic full-

matrix least-squares refinement on F^2 with 228 variables converged at R1 = 3.50%, for the observed data and wR2 = 10.07% for all data. The goodness-of-fit was 1.039. The largest peak in the final difference electron density synthesis was 0.417 $e^-/Å^3$ and the largest hole was -0.206 $e^-/Å^3$ with an RMS deviation of 0.043 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.336 g/cm^3 and F(000), 704 e^-.

References:

Bruker APEX2 v2014.11-0, Bruker AXS Inc., Madison, Wisconsin, USA.

SADABS (2014/5) Bruker AXS Inc., Madison, Wisconsin, USA; Sheldrick, G. M. University of Göttingen, Germany.

SHELXL-2014, (2014), Bruker AXS Inc., Madison, Wisconsin, USA; Sheldrick, G. M. University of Göttingen, Germany.

Table 1: Data collection details for TCD239.

Axis	dx/mm	2θ/°	ω/°	φ/°	χ/°	Width/°	Frames	Time/s	Wavelength/Å	Voltage/kV	Current/mA	Temperature/K
Omega	45.000	27.36	18.58	255.00	-54.74	0.60	206	18.00	0.71073	50	30.0	100
Omega	45.000	27.36	18.58	102.00	-54.74	0.60	206	18.00	0.71073	50	30.0	100
Omega	45.000	28.47	278.50	355.86	43.94	0.60	203	18.00	0.71073	50	30.0	100
Omega	45.000	27.36	18.58	204.00	-54.74	0.60	206	18.00	0.71073	50	30.0	100
Omega	45.000	28.47	279.83	82.48	41.28	0.60	202	18.00	0.71073	50	30.0	100
Omega	45.000	12.36	2.68	0.00	-54.74	0.60	209	18.00	0.71073	50	30.0	100
Phi	45.000	27.36	17.69	278.20	-57.06	0.60	286	18.00	0.71073	50	30.0	100
Phi	45.000	27.36	287.26	230.00	23.00	0.60	180	18.00	0.71073	50	30.0	100
Omega	45.000	27.36	18.58	51.00	-54.74	0.60	206	18.00	0.71073	50	30.0	100

Table 2. Crystal data and structure refinement for tcd239.

Identification code	tcd239	
Empirical formula	$C_{19}H_{16}N_2O_4$	
Formula weight	336.34	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 9.5857(4) Å	α= 90°.
	b = 10.4800(5) Å	β= 100.4013(12)°.
	c = 16.9107(8) Å	γ = 90°.
Volume	1670.90(13) $Å^3$	
Z	4	
Density (calculated)	1.337 Mg/m^3	
Absorption coefficient	0.095 mm^{-1}	
F(000)	704	
Crystal size	0.320 x 0.220 x 0.090 mm^3	
Theta range for data collection	2.160 to 27.510°.	
Index ranges	$-12\leq h\leq 12$, $-13\leq k\leq 13$, $-19\leq l\leq 21$	
Reflections collected	32841	
Independent reflections	3841 [R(int) = 0.0182]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7460 and 0.7166	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3841 / 0 / 228	
Goodness-of-fit on F^2	1.051	
Final R indices [$I>2\sigma(I)$]	R1 = 0.0327, wR2 = 0.0920	
R indices (all data)	R1 = 0.0365, wR2 = 0.0961	
Largest diff. peak and hole	0.344 and -0.185 $e.Å^{-3}$	

Table 3. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for tcd239. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	-588(1)	1500(1)	537(1)	21(1)
C(2)	-1844(1)	1290(1)	-4(1)	24(1)
C(3)	-3107(1)	1120(1)	277(1)	23(1)
C(4)	-3107(1)	1122(1)	1097(1)	23(1)
C(5)	-1844(1)	1306(1)	1638(1)	19(1)
C(6)	-580(1)	1522(1)	1363(1)	16(1)
C(7)	812(1)	1665(1)	1924(1)	15(1)
C(8)	1703(1)	2863(1)	1973(1)	15(1)
C(9)	1003(1)	2442(1)	2692(1)	14(1)
C(10)	-211(1)	3164(1)	2854(1)	15(1)
N(11)	-1172(1)	3643(1)	3044(1)	19(1)
C(12)	1998(1)	2048(1)	3448(1)	15(1)
C(13)	2231(1)	2869(1)	4106(1)	19(1)
C(14)	3238(1)	2539(1)	4772(1)	23(1)
N(15)	4015(1)	1472(1)	4821(1)	26(1)
C(16)	3761(1)	681(1)	4190(1)	23(1)
C(17)	2765(1)	917(1)	3498(1)	19(1)
C(18)	3293(1)	2657(1)	2101(1)	16(1)
O(19)	3849(1)	1752(1)	1845(1)	21(1)
O(20)	3969(1)	3589(1)	2547(1)	21(1)
C(21)	5502(1)	3476(1)	2726(1)	27(1)
C(22)	1174(1)	4034(1)	1500(1)	17(1)
O(23)	22(1)	4492(1)	1464(1)	31(1)
O(24)	2172(1)	4462(1)	1115(1)	25(1)
C(25)	1854(1)	5613(1)	640(1)	29(1)

Table 4. Bond lengths [Å] and angles [°] for tcd239.

C(1)-C(2)	1.3917(14)	C(21)-H(21C)	0.9800
C(1)-C(6)	1.3949(14)	C(22)-O(23)	1.1956(12)
C(1)-H(1)	0.9500	C(22)-O(24)	1.3290(12)
C(2)-C(3)	1.3892(15)	O(24)-C(25)	1.4500(12)
C(2)-H(2)	0.9500	C(25)-H(25A)	0.9800
C(3)-C(4)	1.3871(15)	C(25)-H(25B)	0.9800
C(3)-H(3)	0.9500	C(25)-H(25C)	0.9800
C(4)-C(5)	1.3936(14)		
C(4)-H(4)	0.9500	C(2)-C(1)-C(6)	120.53(9)
C(5)-C(6)	1.3930(13)	C(2)-C(1)-H(1)	119.7
C(5)-H(5)	0.9500	C(6)-C(1)-H(1)	119.7
C(6)-C(7)	1.4990(12)	C(3)-C(2)-C(1)	119.99(10)
C(7)-C(8)	1.5122(13)	C(3)-C(2)-H(2)	120.0
C(7)-C(9)	1.5148(13)	C(1)-C(2)-H(2)	120.0
C(7)-H(7)	1.0000	C(4)-C(3)-C(2)	119.93(9)
C(8)-C(22)	1.5023(13)	C(4)-C(3)-H(3)	120.0
C(8)-C(18)	1.5159(12)	C(2)-C(3)-H(3)	120.0
C(8)-C(9)	1.5550(13)	C(3)-C(4)-C(5)	120.00(10)
C(9)-C(10)	1.4556(13)	C(3)-C(4)-H(4)	120.0
C(9)-C(12)	1.5083(12)	C(5)-C(4)-H(4)	120.0
C(10)-N(11)	1.1451(13)	C(6)-C(5)-C(4)	120.51(9)
C(12)-C(17)	1.3886(14)	C(6)-C(5)-H(5)	119.7
C(12)-C(13)	1.3925(14)	C(4)-C(5)-H(5)	119.7
C(13)-C(14)	1.3883(14)	C(5)-C(6)-C(1)	118.99(9)
C(13)-H(13)	0.9500	C(5)-C(6)-C(7)	122.17(9)
C(14)-N(15)	1.3383(15)	C(1)-C(6)-C(7)	118.63(8)
C(14)-H(14)	0.9500	C(6)-C(7)-C(8)	123.18(8)
N(15)-C(16)	1.3383(15)	C(6)-C(7)-C(9)	123.58(8)
C(16)-C(17)	1.3928(14)	C(8)-C(7)-C(9)	61.82(6)
C(16)-H(16)	0.9500	C(6)-C(7)-H(7)	113.0
C(17)-H(17)	0.9500	C(8)-C(7)-H(7)	113.0
C(18)-O(19)	1.2047(12)	C(9)-C(7)-H(7)	113.0
C(18)-O(20)	1.3298(12)	C(22)-C(8)-C(7)	121.06(8)
O(20)-C(21)	1.4509(12)	C(22)-C(8)-C(18)	115.28(8)
C(21)-H(21A)	0.9800	C(7)-C(8)-C(18)	115.65(8)
C(21)-H(21B)	0.9800	C(22)-C(8)-C(9)	119.74(8)

C(7)-C(8)-C(9)	59.17(6)	C(12)-C(17)-H(17)	120.7
C(18)-C(8)-C(9)	114.36(7)	C(16)-C(17)-H(17)	120.7
C(10)-C(9)-C(12)	112.76(8)	O(19)-C(18)-O(20)	125.48(9)
C(10)-C(9)-C(7)	117.65(8)	O(19)-C(18)-C(8)	124.00(9)
C(12)-C(9)-C(7)	122.64(8)	O(20)-C(18)-C(8)	110.50(8)
C(10)-C(9)-C(8)	118.23(8)	C(18)-O(20)-C(21)	115.39(8)
C(12)-C(9)-C(8)	116.41(7)	O(20)-C(21)-H(21A)	109.5
C(7)-C(9)-C(8)	59.01(6)	O(20)-C(21)-H(21B)	109.5
N(11)-C(10)-C(9)	172.92(10)	H(21A)-C(21)-H(21B)	109.5
C(17)-C(12)-C(13)	118.13(9)	O(20)-C(21)-H(21C)	109.5
C(17)-C(12)-C(9)	122.19(9)	H(21A)-C(21)-H(21C)	109.5
C(13)-C(12)-C(9)	119.57(9)	H(21B)-C(21)-H(21C)	109.5
C(14)-C(13)-C(12)	118.81(10)	O(23)-C(22)-O(24)	125.43(9)
C(14)-C(13)-H(13)	120.6	O(23)-C(22)-C(8)	125.12(9)
C(12)-C(13)-H(13)	120.6	O(24)-C(22)-C(8)	109.43(8)
N(15)-C(14)-C(13)	123.83(10)	C(22)-O(24)-C(25)	116.98(8)
N(15)-C(14)-H(14)	118.1	O(24)-C(25)-H(25A)	109.5
C(13)-C(14)-H(14)	118.1	O(24)-C(25)-H(25B)	109.5
C(14)-N(15)-C(16)	116.65(9)	H(25A)-C(25)-H(25B)	109.5
N(15)-C(16)-C(17)	123.93(10)	O(24)-C(25)-H(25C)	109.5
N(15)-C(16)-H(16)	118.0	H(25A)-C(25)-H(25C)	109.5
C(17)-C(16)-H(16)	118.0	H(25B)-C(25)-H(25C)	109.5
C(12)-C(17)-C(16)	118.60(10)		

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for tcd239. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	19(1)	24(1)	20(1)	1(1)	5(1)	-2(1)
C(2)	27(1)	26(1)	17(1)	1(1)	1(1)	-2(1)
C(3)	18(1)	23(1)	26(1)	-1(1)	-4(1)	1(1)
C(4)	16(1)	25(1)	29(1)	-6(1)	5(1)	-1(1)
C(5)	18(1)	21(1)	19(1)	-3(1)	5(1)	-2(1)
C(6)	15(1)	13(1)	18(1)	0(1)	2(1)	1(1)
C(7)	14(1)	14(1)	17(1)	1(1)	3(1)	1(1)
C(8)	13(1)	15(1)	16(1)	1(1)	3(1)	1(1)
C(9)	13(1)	14(1)	16(1)	2(1)	2(1)	1(1)
C(10)	16(1)	15(1)	15(1)	1(1)	1(1)	-2(1)
N(11)	17(1)	19(1)	21(1)	-1(1)	4(1)	1(1)
C(12)	13(1)	17(1)	16(1)	4(1)	2(1)	-2(1)
C(13)	19(1)	19(1)	18(1)	2(1)	2(1)	0(1)
C(14)	24(1)	26(1)	18(1)	1(1)	-1(1)	-3(1)
N(15)	23(1)	28(1)	23(1)	6(1)	-2(1)	0(1)
C(16)	20(1)	23(1)	26(1)	7(1)	0(1)	3(1)
C(17)	17(1)	18(1)	20(1)	3(1)	1(1)	0(1)
C(18)	14(1)	17(1)	17(1)	4(1)	4(1)	0(1)
O(19)	17(1)	19(1)	27(1)	1(1)	7(1)	3(1)
O(20)	13(1)	21(1)	27(1)	-2(1)	2(1)	-2(1)
C(21)	12(1)	30(1)	38(1)	0(1)	1(1)	-3(1)
C(22)	17(1)	16(1)	17(1)	1(1)	2(1)	0(1)
O(23)	22(1)	34(1)	40(1)	19(1)	12(1)	12(1)
O(24)	24(1)	23(1)	29(1)	12(1)	11(1)	5(1)
C(25)	32(1)	24(1)	31(1)	14(1)	10(1)	3(1)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for tcd239.

	x	y	z	U(eq)
H(1)	271	1629	342	25
H(2)	-1838	1265	-565	28
H(3)	-3969	1001	-93	28
H(4)	-3969	998	1290	28
H(5)	-1844	1285	2200	23
H(7)	1392	867	1979	18
H(13)	1711	3641	4099	23
H(14)	3384	3104	5218	28
H(16)	4293	-88	4216	28
H(17)	2613	318	3070	23
H(21A)	5767	2728	3068	41
H(21B)	5910	4245	3006	41
H(21C)	5865	3377	2223	41
H(25A)	1221	5406	134	43
H(25B)	2736	5980	525	43
H(25C)	1390	6233	941	43

Table 7. Torsion angles [°] for tcd239.

C(6)-C(1)-C(2)-C(3)	0.92(16)	C(17)-C(12)-C(13)-C(14)	1.51(14)
C(1)-C(2)-C(3)-C(4)	-1.80(17)	C(9)-C(12)-C(13)-C(14)	-174.67(9)
C(2)-C(3)-C(4)-C(5)	0.52(17)	C(12)-C(13)-C(14)-N(15)	0.41(16)
C(3)-C(4)-C(5)-C(6)	1.65(16)	C(13)-C(14)-N(15)-C(16)	-1.56(16)
C(4)-C(5)-C(6)-C(1)	-2.51(15)	C(14)-N(15)-C(16)-C(17)	0.81(16)
C(4)-C(5)-C(6)-C(7)	-177.18(9)	C(13)-C(12)-C(17)-C(16)	-2.19(14)
C(2)-C(1)-C(6)-C(5)	1.23(15)	C(9)-C(12)-C(17)-C(16)	173.89(9)
C(2)-C(1)-C(6)-C(7)	176.08(9)	N(15)-C(16)-C(17)-C(12)	1.06(16)
C(5)-C(6)-C(7)-C(8)	-118.85(10)	C(22)-C(8)-C(18)-O(19)	-117.14(11)
C(1)-C(6)-C(7)-C(8)	66.47(12)	C(7)-C(8)-C(18)-O(19)	32.16(13)
C(5)-C(6)-C(7)-C(9)	-42.92(14)	C(9)-C(8)-C(18)-O(19)	98.17(11)
C(1)-C(6)-C(7)-C(9)	142.39(9)	C(22)-C(8)-C(18)-O(20)	64.51(10)
C(6)-C(7)-C(8)-C(22)	5.18(14)	C(7)-C(8)-C(18)-O(20)	-146.19(8)
C(9)-C(7)-C(8)-C(22)	-108.36(9)	C(9)-C(8)-C(18)-O(20)	-80.18(10)
C(6)-C(7)-C(8)-C(18)	-142.21(9)	O(19)-C(18)-O(20)-C(21)	0.31(14)
C(9)-C(7)-C(8)-C(18)	104.25(9)	C(8)-C(18)-O(20)-C(21)	178.63(8)
C(6)-C(7)-C(8)-C(9)	113.54(10)	C(7)-C(8)-C(22)-O(23)	47.41(15)
C(6)-C(7)-C(9)-C(10)	-4.98(13)	C(18)-C(8)-C(22)-O(23)	-165.08(10)
C(8)-C(7)-C(9)-C(10)	107.95(9)	C(9)-C(8)-C(22)-O(23)	-22.42(15)
C(6)-C(7)-C(9)-C(12)	143.68(9)	C(7)-C(8)-C(22)-O(24)	-131.16(9)
C(8)-C(7)-C(9)-C(12)	-103.39(9)	C(18)-C(8)-C(22)-O(24)	16.34(12)
C(6)-C(7)-C(9)-C(8)	-112.93(10)	C(9)-C(8)-C(22)-O(24)	159.01(8)
C(22)-C(8)-C(9)-C(10)	3.58(13)	O(23)-C(22)-O(24)-C(25)	2.88(16)
C(7)-C(8)-C(9)-C(10)	-106.98(9)	C(8)-C(22)-O(24)-C(25)	-178.55(9)
C(18)-C(8)-C(9)-C(10)	146.57(8)		
C(22)-C(8)-C(9)-C(12)	-135.61(9)		
C(7)-C(8)-C(9)-C(12)	113.83(9)		
C(18)-C(8)-C(9)-C(12)	7.38(12)		
C(22)-C(8)-C(9)-C(7)	110.55(9)		
C(18)-C(8)-C(9)-C(7)	-106.45(9)		
C(10)-C(9)-C(12)-C(17)	147.51(9)		
C(7)-C(9)-C(12)-C(17)	-2.52(14)		
C(8)-C(9)-C(12)-C(17)	-71.14(12)		
C(10)-C(9)-C(12)-C(13)	-36.48(12)		
C(7)-C(9)-C(12)-C(13)	173.50(8)		
C(8)-C(9)-C(12)-C(13)	104.88(10)		

Table 8. Hydrogen bonds for tcd239 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C(16)-H(16)...N(15)#1	0.95	2.54	3.3371(14)	142

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y,-z+1