Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Carbene-Catalyzed Selective Addition of Isothioureas to Enals for Access to Sulphur-Containing 5,6-Dihydropyrimidin-4-ones

Rakesh Maiti,[a] Jun Xu,[a,b] Jia-Lei Yan,[a] Bivas Mondal,[a] Xing Yang,[a] Huifang Chai,*[b] Lin Hao,[a] Zhichao Jin,[c] and Yonggui Robin Chi*[a, c]

[a]Division of Chemistry & Biological Chemistry, School of Physical & Mathematical Science, Nanyang Technological University, Singapore 637371, Singapore

[b]Guizhou University of Traditional Chinese Medicine, Guiyang 550025, China

[c]Key Laboratory of Green Pesticide and Agriculture Bioengineering, Ministry of Education, Guizhou University, Huaxi District, Guiyang 550025, China

Table of Contents

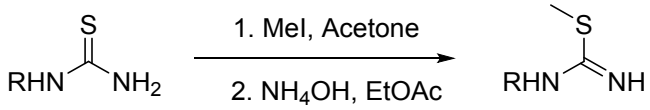
Part 1. General Information

Commercially available materials purchased form TCI or Sigma Aldrich were used as received. All reactions were carried out under nitrogen atmosphere under anhydrous conditions unless otherwise noted. THF was distilled from sodium-benzophenone. Flash chromatography was performed using silica gel (200-300 mesh). Reactions were monitored by thin layer chromatography (TLC). Visualization was achieved under a UV lamp (254nm and 365 nm). 1H and 13C NMR were recorded on Bruker BBFO 400 MHz NMR, Bruker AV400 MHz NMR spectrometer with TMS as the internal standard, and were calibrated using residual undeuterated solvent as an internal reference (CDCl3: ^{1}H NMR = 7.26, ^{13}C NMR = 77.16). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad. Coupling constants (J) are reported in Hertz (Hz). High-resolution Mass spectra (HRMS) were recorded by using Finnigan MAT 95 XP mass spectrometer (Thermo Electron Corporation). The determination of e.r. was performed via chiral HPLC analysis using Shimadzu LC-20AD HPLC workstation. Optical rotations were measured using a 1 mL cell with a 1 dm path length on a Jasco P-1030 polarimeter and are reported as follows: $[\alpha]^{21}_{D}$ (c in g per 100 mL solvent).

Part 2. Experimental Section

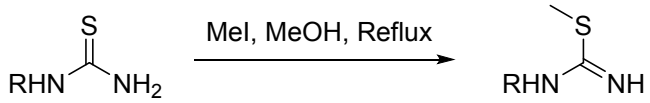
I. General Procedure to Synthesis of Starting Materials:

S-Methyl Isothiourea:[1]



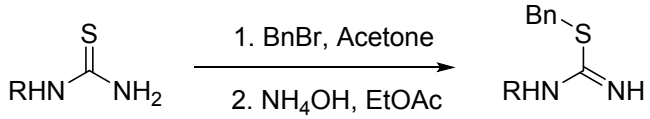
Iodomethane (5 mmol) was added to a solution of thiourea derivative (5 mmol) in acetone and the mixture was continued to stir overnight at room temperature. After that, the mixture was concentrated under vacuum and ethyl acetate (25 mL) was added. Later, H_2O (50 mL) was added and cooled to 0 ^oC. Next, conc. NH_4OH was mixed dropwise until all the solid disappeared and then, stirred for another 30 mins. Organic layer was separated, washed with brine, and concentrated. The product was used directly without further purification.

Synthesis of 2h:[2]



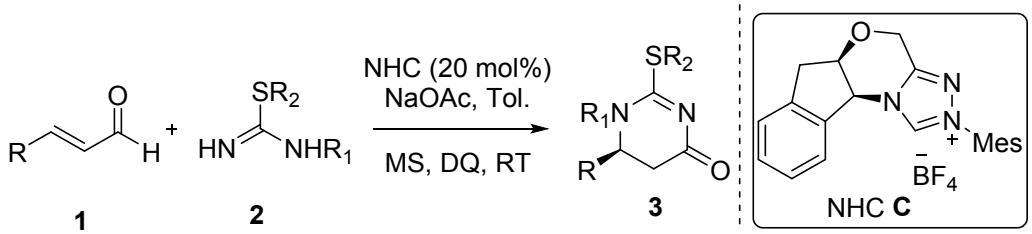
In 100 mL round bottom flask equipped with a magnetic stirring bar and a reflux condenser, thiourea (4.00 g, 51.3 mmol) was dissolved in methanol (40.0 mL). Then, Iodomethane (3.20 mL, 51.3 mmol) was added in one portion and the mixture was continued to reflux for 2 hrs. After the mentioned time, the reaction mixture was cooled to room temperature and concentrated under reduced pressure, giving a yellowish solid. Then, the yellowish solid was mixed with EtOAc (50.0 mL), and the precipitate that did not dissolve was filtered. The precipitate was sequentially washed several times with EtOAc and diethyl ether until it becomes completely colorless. The product was dried under vacuum to provide 8.35 g (38.0 mmol, 74% yield) of white powder.

Synthesis of 2i and 2j:



Benzyl Bromide (5 mmol) was added dropwise to a cooled solution of thiourea derivative (5 mmol) in acetone and the mixture was continued to stir overnight at room temperature. After that, the mixture was concentrated under vacuum and ethyl acetate (25 mL) was added. Later, H_2O (50 mL) was added and cooled to 0 ^oC. Next, conc. NH_4OH was mixed dropwise until all the solid disappeared and then, stirred for another 30 mins. Organic layer was separated, washed with brine, and concentrated. The product was used directly without further purification.

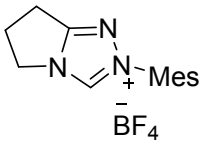
II. General Procedure for the Catalytic Reactions of Enal (1) with S-alkylated isothioura (2) to Synthesize Product 3



1 2 3

A dry 10 mL Schlenk tube with a stir bar was charged with enal **1** (0.18 mmol, 1.8 equiv.), **2** (0.1 mmol, 1.0 equiv.), NHC (8.4 mg, 20 mol%), NaOAc (13 mg, 0.15 mmol, 1.5 equiv.), AcOH (1.8 μL, 30 mol%), DQ (51 mg, 0.125 mmol, 1.25 equiv.) and molecular sieves (100 mg). The tube was evacuated and refilled with nitrogen. Then the mixture was dissolved with the newly distilled solvent toluene (2.0 mL). Then the mixture was stirred at room temperature for 36-48h. When the substrate was consumed completely (monitored by TLC), the mixture was concentrated under vacuum and purified by column chromatography on silica gel (hexane/ethyl acetate = 2:1) to afford the desired product 3, which was confirmed by ^{1}H NMR, ^{13}C NMR spectra, and the enantiomeric ratio was determined by chiral HPLC.

Note: Racemic samples were prepared using NHC below for chiral phase HPLC analysis.

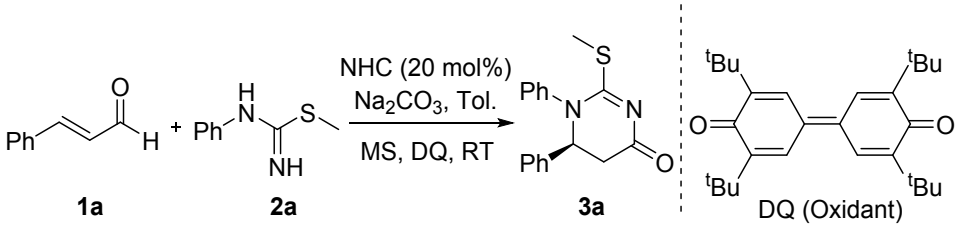


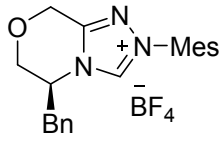
Achiral NHC

III. Additional Results of Condition Optimization

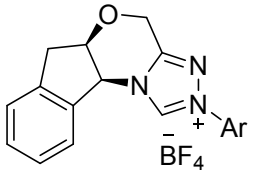
Condition Optimization (additional results) of the Model Reaction (reaction of 1a and 2a) (Tables S1-S2).

Table S1. NHC Catalysts Screening [a, b]

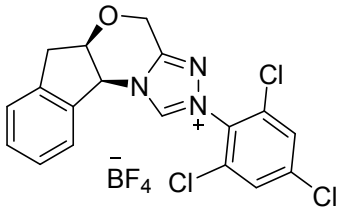




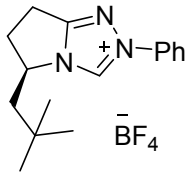
A; 63%, 81:19 e.r.



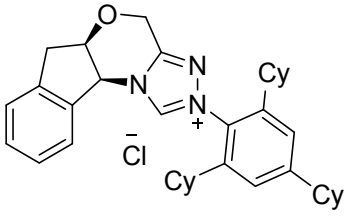
Ar = Ph, **B;** 52%, 84:16 e.r.
= Mes, **C;** 58%, 93:7 e.r.



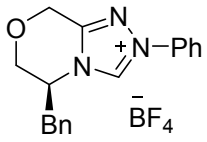
D; 42%, 84:16 e.r.



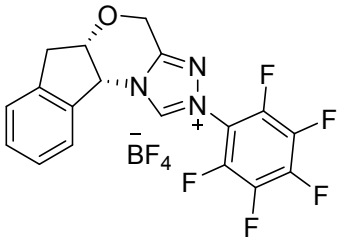
E; 65%, 71:29 e.r.



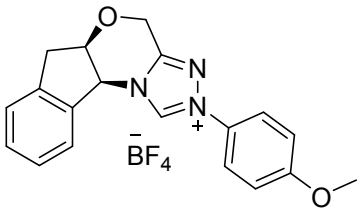
F; NA



G; 52%, 74:26 e.r.



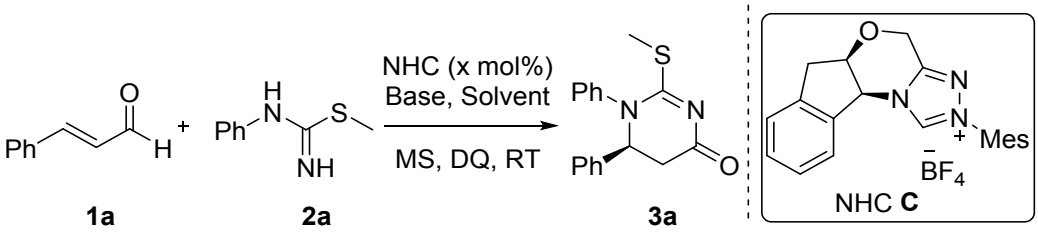
H; 21%, 36:64 e.r.



I; 43%, 84:16 e.r.

[a]Reaction condition: **1a** (0.09 mmol.), **2a** (0.05 mmol), NHC pre-cat. (20 mol%), Na_2CO_3 (1.5 equiv.), DQ (1.25 equiv.), AcOH (30 mol%, additive), Tol. (1 mL), MS (50 mg) at RT for 36-48 hrs. [b]Yield determined by ^{1}HNMR, based on **2a**, by using 1,3,5-trimethoxybenzene as internal standard. The e.r. was determined via chiral-phase HPLC analysis.

Table S2. Bases, Solvents, and amount of cat. Loading Screening [a, b]



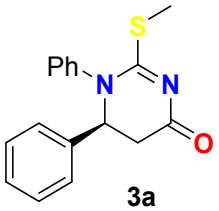
1a 2a 3a

Entry	NHC (mol%)	Base	Solvent	Yield (%)	e.r.
1	**C (20)**	Na_2CO_3	THF	50	83:17
2	**C (20)**	Na_2CO_3	ACN	63	68:32
3	**C (20)**	Na_2CO_3	DCE	73	70:30
4	**C (20)**	Na_2CO_3	EtOAc	72	83:17
5	**C (20)**	Na_2CO_3	$CHCl_3$	31	87:13
6	**C (20)**	Na_2CO_3	Et_2O	77	86:14
7	**C (20)**	K_2CO_3	Tol.	49	92:8
8	**C (20)**	Cs_2CO_3	Tol.	55	91:9
9	**C (20)**	K_3PO_4	Tol.	61	91:9
10	**C (20)**	DABCO	Tol.	25	87:13
11	**C (20)**	DMAP	Tol.	45	92:8
12	**C (20)**	DIPEA	Tol.	36	92:8
13	**C (20)**	NaOAc	Tol.	82	95:5
14	**C (10)**	NaOAc	Tol.	77	93:7
15	**C (5)**	NaOAc	Tol.	75	91:9

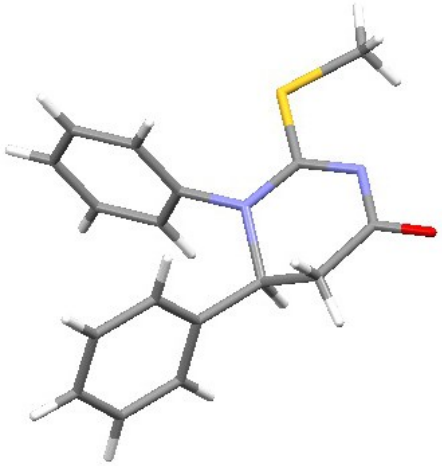
[a]Reaction condition: **1a** (0.09 mmol.), **2a** (0.05 mmol), NHC pre-cat. (mol%), Base (1.5 equiv.), DQ (1.25 equiv.), AcOH (30 mol%, additive), Solvent (1 mL), MS (50 mg) at RT for 36-48 hrs. [b]Yield determined by ^{1}HNMR, based on **2a**, by using 1,3,5-trimethoxybenzene as internal standard. The e.r. was determined via chiral-phase HPLC analysis.

IV. Stereochemistry Determination of 3a via X-ray Crystallographic Analysis

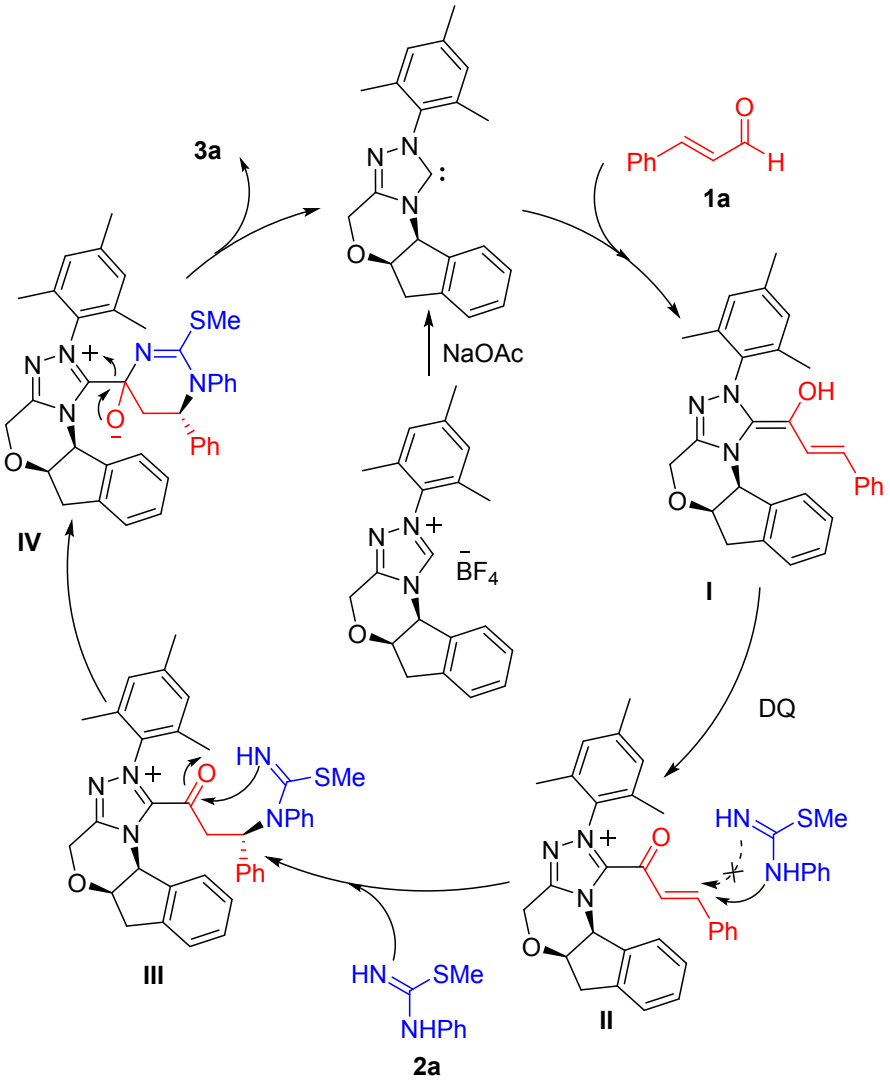
Product 3a was crystallized as a colourless crystal via vaporization of a hexane/CH_2Cl_2 solution, and its absolute configuration was determined by x-ray structure analysis. ***CCDC 2025922*** contains the supplementary crystallographic data that can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data request/cif.



3a

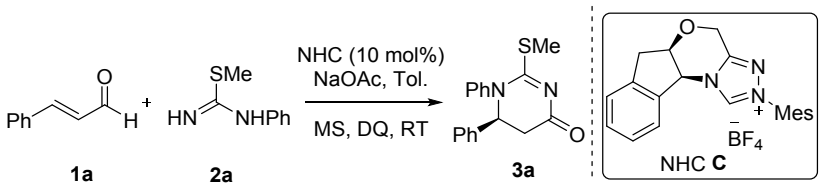


V. Postulated Reaction Mechanism



VI. Procedures for Scale-up Reaction and Synthetic Transformations

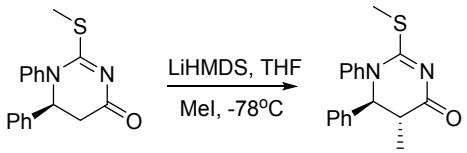
Gram-scale Preparation of 3a



1a **2a** **3a**

A dry 100 mL Schlenk tube with stir bar was charged with cinnamaldehyde **1a** (12 mmol, 1.2 equiv.), **2a** (1.66 g, 10 mmol, 1.0 equiv.), NHC **C** (420 mg, 10 mol%), NaOAc (1.23 gm, 15 mmol, 1.5 equiv.), DQ (5.1 gm, 12.5 mmol, 1.25 equiv.) and molecular sieves (1 gm). The tube was evacuated and refilled with nitrogen. Then the mixture was dissolved with newly distilled solvent Toluene (50 mL). Then the mixture was stirred at room temperature for 36 h. After complete consumption of the substrate **2a** (monitored by TLC), the mixture was concentrated under vacuum and purified by column chromatography on silica gel (hexane/ethyl acetate = 2:1) to afford desired product **3a** (2.13g) with 72% yield and 93:7 e.r.

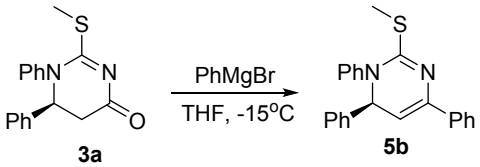
Preparation of 5a[3]



3a **5a**

To a stirred solution of **3a** (30 mg, 0.1 mmol) in anhydrous THF (1 mL) at −78 °C was added LiHMDS (1 M in THF, 0.15 mL, 0.15 mmol), and the solution was stirred for 2 hrs. The iodomethane (28.4 mg, 0.2 mmol) was added dropwise. After that, the temperature of the reaction mixture was slowly raised to room temperature and continued to stir for 20 h at the same temperature. After completion of the reaction, H_2O (1 mL) was added. The organic layer was collected, and the aqueous layer was extracted with EtOAc (twice). The combined organic extracts were washed with brine, dried over MgSO4, filtered, and concentrated under reduced pressure. The residue was purified via silica gel flash chromatography (hexane/ethyl acetate = 5:1) to afford product **5a (**27 mg, 87%, 91:9 e.r., >99:1 d.r.).

Preparation of 5b[4]

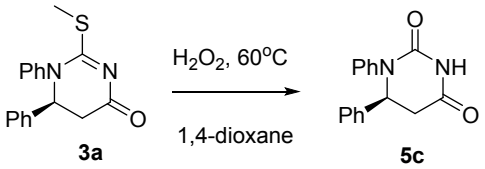


3a **5b**

The product **3a** (30 mg, 0.1 mmol) was suspended in dry THF and then cooled −15 °C. Grignard reagent (PhMgBr, 1.2 equiv.) was then added dropwise, and the reaction mixture was stirred at −15 °C for 5 h. Trifluoroacetic acid (3.0 equiv.) was then carefully added, and the reaction was stirred at −15 °C for another 30 minutes. The mixture was concentrated under reduced pressure, diluted with water, and extracted with EtOAc. The combined organic layers were dried over MgSO4, filtered, and concentrated

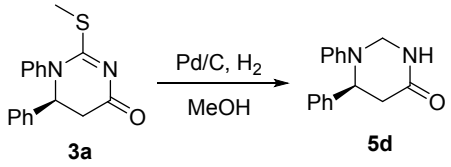
under reduced pressure. The residue was purified by column chromatography (hexane/ethyl acetate = 1:50) to obtain **5b** (35 mg, 98%, 90:10 e.r.).

Preparation of 5c



The product **3a** (30 mg, 0.1 mmol) was dissolved in 1,4-dioxane and then H_2O_2 (35% v/v, 1 mL) was added. Then the solution was heated at 60 °C for 6 hrs. Solvent was evaporated and the reaction mixture was extracted with EtOAc. The combined organic layers were dried over MgSO4, filtered, and concentrated under reduced pressure. Pure **5c** (21.6 mg, 81%, 91:9 e.r.) was obtained by silica gel column chromatography (hexane/ethyl acetate = 5:1).

Preparation of 5d



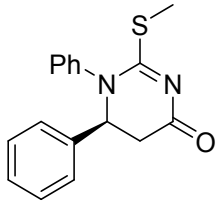
The product **3a** (30 mg, 0.1 mmol) and Pd/C (10 mol%) was added to MeOH (10 ml). Then solution was degassed using pump and bubbled with H_2 gas (5 times). The reaction mixture was stirred until the starting materials consumed completely. When the reaction was finished, MeOH was evaporated and EtOAc was added to the reaction mixture. Excess Pd/C was filtered through celite, solvent was evaporated and concentrated under reduced pressure. Silica gel column chromatography (hexane/ethyl acetate = 3:1) of the residue provided the pure **5d** (18.4 mg, 73%, 93:7 e.r.).

VII. Reference

1. I. Cohen, *Synthesis* 1980, **1980**, 60.
2. K. Mailyan, J. L. Chen, W. Li, A. A. Keller, S. M. Sternisha, B. G. Miller, A. Zakarian, *J. Am. Chem. Soc.* 2018, **140**, 6027.
3. C.-L. Zhang, D.-L. Wang, K.-Q. Chen, S. Ye *Org. Biomol. Chem.,* 2015, **13**, 11255.
4. B. Ranieri, O.Robles, D. Romo, *J. Org. Chem.,* 2013, **78**, 6291.

Part 3. Characterization of products

I. Characterizations of Products



***(S)*-2-(methylthio)-1,6-diphenyl-5,6-dihydropyrimidin-4(1H)-one (3a)**

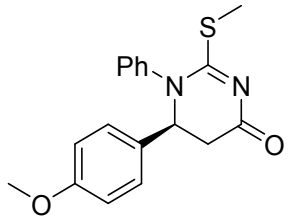
Yield: 23 mg (78%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.24 (m, 6H), 7.24 – 7.16 (m, 2H), 7.12 (s, 2H), 4.91 (dd, *J* = 7.7, 3.6 Hz, 1H), 3.25 (dd, *J* = 15.4, 7.8 Hz, 1H), 2.88 (dd, *J* = 15.4, 3.6 Hz, 1H), 2.46 (s, 3H).**^{13}C NMR (100 MHz, $CDCl_3$)** δ 173.8, 173.1, 140.7, 138.5, 129.7, 129.5, 129.2, 128.8, 128.7, 126.8, 64.9, 38.6, 15.2 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{16}N_2OSH^+$: 297.1062 $(M+H)^+$, found: 297.1063.

$[\alpha]^{21}_D$ = +3.6 (c = 1.0 in $CHCl_3$).

HPLC analysis: 95:5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 22.1 min, R_t (major) = 28.1 min.



***(S)*-6-(4-methoxyphenyl)-2-(methylthio)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3b)**

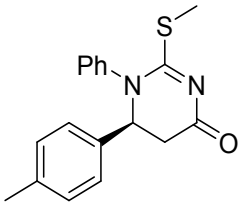
Yield: 25 mg (77%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.34 (dd, *J* = 4.5, 2.1 Hz, 3H), 7.09 (dd, *J* = 6.7, 2.0 Hz, 4H), 6.87 – 6.75 (m, 2H), 4.84 (dd, *J* = 7.6, 3.8 Hz, 1H), 3.77 (s, 3H), 3.21 (dd, *J* = 15.4, 7.6 Hz, 1H), 2.87 (dd, *J* = 15.4, 3.9 Hz, 1H), 2.45 (s, 3H). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 173.6, 173.4, 159.9, 140.8, 130.7, 129.6, 129.5, 128.8, 128.3, 114.6, 64.5, 55.4, 38.8, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{18}H_{18}N_2O_2SH^+$: 327.1167 $(M+H)^+$, found: 327.1169.

$[\alpha]^{21}_D$ = -5.3 (c = 1.0 in $CHCl_3$).

HPLC analysis: 94.5:5.5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 35.7 min, R_t (major) = 38.9 min.



***(S)*-2-(methylthio)-1-phenyl-6-(p-tolyl)-5,6-dihydropyrimidin-4(1H)-one (3c)**

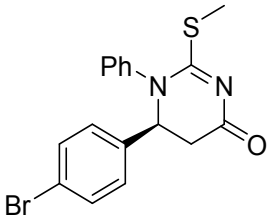
Yield: 24 mg (78%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.42 – 7.28 (m, 3H), 7.18 – 6.97 (m, 6H), 4.86 (dd, *J* = 7.7, 3.7 Hz, 1H), 3.22 (dd, *J* = 15.4, 7.7 Hz, 1H), 2.86 (dd, *J* = 15.4, 3.7 Hz, 1H), 2.45 (s, 3H), 2.30 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.6, 173.2, 140.8, 138.6, 135.6, 129.9, 129.6, 129.4, 128.7, 126.8, 64.8, 38.8, 21.2, 15.2 ppm.

HRMS (ESI, m/z): calculated for $C_{18}H_{18}N_2OSH^+$: 311.1218 $(M+H)^+$, found: 311.1218.

$[\alpha]^{21}_D$ = -1.4 (c = 1.7 in $CHCl_3$).

HPLC analysis: 94.5:5.5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 22.0 min, R_t (major) = 28.4 min.



***(S)*-6-(4-bromophenyl)-2-(methylthio)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3d)**

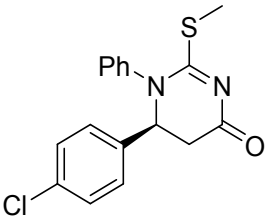
Yield: 30 mg (81%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.48 – 7.39 (m, 2H), 7.39 – 7.30 (m, 3H), 7.16 – 6.99 (m, 4H), 4.87 (dd, *J* = 7.7, 3.4 Hz, 1H), 3.24 (dd, *J* = 15.4, 7.8 Hz, 1H), 2.83 (dd, *J* = 15.4, 3.5 Hz, 1H), 2.45 (s, 3H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.9, 172.6, 140.6, 137.7, 132.5, 129.8, 129.7, 128.6, 128.6, 122.9, 64.4, 38.5, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}BrN_2OSH^+$: 375.0167 $(M+H)^+$, found: 375.0167.

$[\alpha]^{21}_D$ = +3.7 (c = 1.7 in $CHCl_3$).

HPLC analysis: 95.5:4.5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 26.3 min, R_t (major) = 37.4 min.



***(S)*-6-(4-chlorophenyl)-2-(methylthio)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3e)**

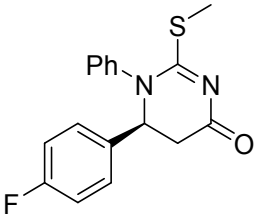
Yield: 27 mg (82%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.41 – 7.31 (m, 3H), 7.31 – 7.27 (m, 2H), 7.18 – 6.99 (m, 4H), 4.88 (dd, *J* = 7.7, 3.5 Hz, 1H), 3.24 (dd, *J* = 15.4, 7.7 Hz, 1H), 2.84 (dd, *J* = 15.4, 3.5 Hz, 1H), 2.45 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.9, 172.7, 140.6, 137.2, 134.8, 129.8, 129.7, 129.5, 128.7, 128.3, 64.4, 38.5, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}ClN_2OSH^+$: 331.0672 $(M+H)^+$, found: 331.0672.

$[\alpha]^{21}_D$ = +12.0 (c = 0.8 in $CHCl_3$).

HPLC analysis: 95:5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 24.2 min, R_t (major) = 30.7 min.



***(S)*-6-(4-fluorophenyl)-2-(methylthio)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3f)**

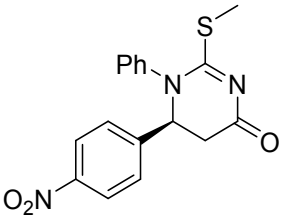
Yield: 27 mg (86%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.43 – 7.29 (m, 3H), 7.22 – 7.13 (m, 2H), 7.10 (s, 2H), 7.03 – 6.92 (m, 2H), 4.90 (dd, *J* = 7.7, 3.6 Hz, 1H), 3.24 (dd, *J* = 15.4, 7.7 Hz, 1H), 2.85 (dd, *J* = 15.4, 3.6 Hz, 1H), 2.45 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.8, 172.9, 164.1, 161.7, 140.6, 134.5 (d, *J* = 3.3 Hz), 129.7 (d, *J* = 14.8 Hz), 128.8 (d, *J* = 8.6 Hz), 116.2, 116.0, 64.1, 38.5, 15.1 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}FN_2OSH^+$: 315.0967 $(M+H)^+$, found: 315.0967.

$[\alpha]^{21}_D$ = -6.4 (c = 2.2 in $CHCl_3$).

HPLC analysis: 94:6 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 23.5 min, R_t (major) = 29.1 min.



***(S)*-2-(methylthio)-6-(4-nitrophenyl)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3g)**

Yield: 30 mg (88%)

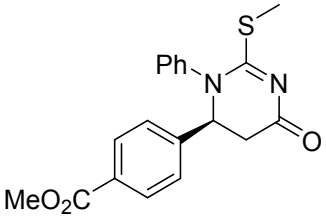
^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.27 – 8.09 (m, 2H), 7.51 – 7.31 (m, 5H), 7.13 (d, *J* = 5.6 Hz, 2H), 5.06 (dd, *J* = 7.8, 3.2 Hz, 1H), 3.31 (dd, *J* = 15.4, 7.9 Hz, 1H), 2.85 (dd, *J* = 15.4, 3.3 Hz, 1H), 2.47 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 171.7, 148.0, 145.7, 140.4, 130.0, 129.9, 128.4, 127.9, 124.6, 64.2, 38.2, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}N_3O_3SH^+$: 342.0912 $(M+H)^+$, found: 342.0912.

$[\alpha]^{21}_D$ = +51.8 (c = 0.3 in $CHCl_3$).

HPLC analysis: 91.5:8.5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 36.3 min, R_t (major) = 71.4 min.



methyl *(S)*-4-(2-(methylthio)-6-oxo-3-phenyl-3,4,5,6-tetrahydropyrimidin-4-yl)benzoate (3h)

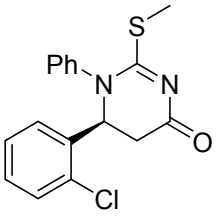
Yield: 28 mg (79%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 8.02 – 7.90 (m, 2H), 7.34 (dd, *J* = 7.6, 3.9 Hz, 3H), 7.28 (d, *J* = 7.0 Hz, 2H), 7.11 (d, *J* = 5.7 Hz, 2H), 4.98 (dd, *J* = 7.8, 3.5 Hz, 1H), 3.90 (s, 3H), 3.27 (dd, *J* = 15.4, 7.8 Hz, 1H), 2.86 (dd, *J* = 15.4, 3.5 Hz, 1H), 2.46 (s, 3H). **^{13}C NMR** (101 MHz, $CDCl_3$) δ 174.0, 172.5, 166.5, 143.5, 140.6, 130.7, 130.5, 129.8, 129.7, 128.6, 126.9, 64.6, 52.3, 38.4, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{19}H_{18}N_2O_3SH^+$: 355.1116 $(M+H)^+$, found: 375.0167.

$[\alpha]^{21}_D$ = +15.7 (c = 1.7 in $CHCl_3$).

HPLC analysis: 95:5 *e.r.* (ORJH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 34.1 min, R_t (major) = 40.2 min.



***(S)*-6-(2-chlorophenyl)-2-(methylthio)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3i)**

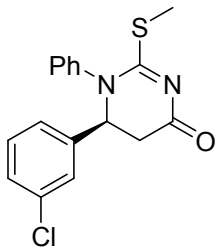
Yield: 24 mg (73%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.54 – 7.44 (m, 1H), 7.43 – 7.33 (m, 3H), 7.30 (dd, *J* = 7.5, 1.0 Hz, 2H), 7.26 – 7.09 (m, 3H), 5.47 (dd, *J* = 7.9, 2.7 Hz, 1H), 3.23 (dd, *J* = 15.4, 7.9 Hz, 1H), 2.90 (dd, *J* = 15.4, 2.8 Hz, 1H), 2.48 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.2, 172.6, 140.9, 135.4, 132.3, 130.4, 129.9, 129.8, 129.6, 128.3, 127.8, 127.7, 61.2, 37.1, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}ClN_2OSH^+$: 331.0672 $(M+H)^+$, found: 331.0672.

$[\alpha]^{21}_D$ = +51.9 (c = 1.9 in $CHCl_3$).

HPLC analysis: 96:4 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 21.9 min, R_t (major) = 53.3 min.



***(S)*-6-(3-chlorophenyl)-2-(methylthio)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3j)**

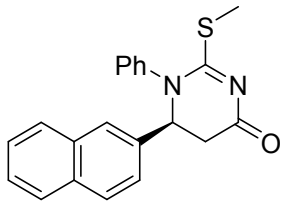
Yield: 18 mg (54%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 7.2, 4.1 Hz, 3H), 7.30 – 7.23 (m, 2H), 7.13 (dd, *J* = 5.4, 2.5 Hz, 4H), 4.89 (dd, *J* = 7.7, 3.4 Hz, 1H), 3.24 (dd, *J* = 15.4, 7.8 Hz, 1H), 2.84 (dd, *J* = 15.4, 3.4 Hz, 1H), 2.46 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.0, 172.5, 140.6, 140.6, 135.1, 130.6, 129.8, 129.7, 129.1, 128.6, 127.1, 124.9, 64.4, 38.5, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}ClN_2OSH^+$: 331.0672 $(M+H)^+$, found: 331.0672.

$[\alpha]^{21}_D$ = +11.9 (c = 1.9 in $CHCl_3$).

HPLC analysis: 92:8 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 21.0 min, R_t (major) = 24.0 min.



***(S)*-2-(methylthio)-6-(naphthalen-2-yl)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3k)**

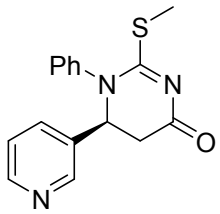
Yield: 27 mg (78%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.86 – 7.69 (m, 3H), 7.59 (s, 1H), 7.52 – 7.40 (m, 2H), 7.38 – 7.27 (m, 4H), 7.14 (s, 2H), 5.07 (dd, *J* = 7.8, 3.6 Hz, 1H), 3.31 (dd, *J* = 15.5, 7.8 Hz, 1H), 2.96 (dd, *J* = 15.5, 3.7 Hz, 1H), 2.49 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.9, 173.0, 140.8, 136.0, 133.4, 133.3, 129.7, 129.5, 129.4, 128.7, 128.2, 127.8, 126.7, 126.7, 126.2, 124.2, 65.2, 38.7, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{21}H_{18}N_2OSH^+$: 347.1218 $(M+H)^+$, found: 347.1218.

$[\alpha]^{21}_D$ = +5.2 (c = 1.1 in $CHCl_3$).

HPLC analysis: 95.5:4.5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 34.9 min, R_t (major) = 48.2 min.



***(S)*-2-(methylthio)-1-phenyl-6-(pyridin-3-yl)-5,6-dihydropyrimidin-4(1H)-one (3l)**

Yield: 20 mg (67%)

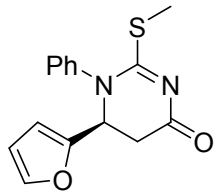
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.56 (d, *J* = 3.7 Hz, 1H), 8.35 (s, 1H), 7.67 (d, *J* = 7.8 Hz, 1H), 7.37 (s, 3H), 7.29 (dd, *J* = 8.8, 6.3 Hz, 1H), 7.10 (s, 2H), 4.97 (dd, *J* = 7.4, 3.6 Hz, 1H), 3.29 (dd, *J* = 15.4, 7.6

Hz, 1H), 2.88 (dd, *J* = 15.4, 3.6 Hz, 1H), 2.46 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.1, 172.3, 150.4, 148.7, 140.3, 134.3, 130.0, 129.8, 128.6, 124.2, 62.6, 38.3, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{16}H_{15}N_3OSH^+$: 298.1014 $(M+H)^+$, found: 298.1017.

$[\alpha]^{21}_D$ = +2.3 (c = 0.9 in $CHCl_3$).

HPLC analysis: 94.5:5.5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 45.6 min, R_t (major) = 50.2 min.



***(S)*-6-(furan-2-yl)-2-(methylthio)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3m)**

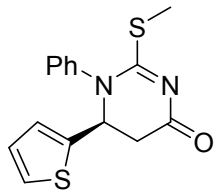
Yield: 19 mg (63%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (dd, *J* = 7.1, 4.3 Hz, 3H), 7.35 (dd, *J* = 1.8, 0.7 Hz, 1H), 7.13 (s, 2H), 6.26 (dd, *J* = 3.3, 1.9 Hz, 1H), 6.19 (d, *J* = 3.2 Hz, 1H), 4.95 (dd, *J* = 7.0, 4.8 Hz, 1H), 3.15 (dd, *J* = 15.6, 7.1 Hz, 1H), 3.04 (dd, *J* = 15.6, 4.8 Hz, 1H), 2.41 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.5, 173.1, 150.4, 143.2, 140.4, 129.7, 129.7, 128.8, 110.6, 109.3, 58.0, 35.9, 15.2 ppm.

HRMS (ESI, m/z): calculated for $C_{15}H_{14}N_2O_2SH^+$: 287.0854 $(M+H)^+$, found: 287.0854.

$[\alpha]^{21}_D$ = -19.1 (c = 1.8 in $CHCl_3$).

HPLC analysis: 90:10 *e.r.* (OD, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 29.2 min, R_t (major) = 31.6 min.



***(S)*-2-(methylthio)-1-phenyl-6-(thiophen-2-yl)-5,6-dihydropyrimidin-4(1H)-one (3n)**

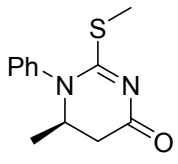
Yield: 18 mg (60%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, *J* = 5.8 Hz, 3H), 7.23 (dd, *J* = 4.3, 2.0 Hz, 1H), 7.13 (s, 2H), 6.89 (dd, *J* = 5.2, 2.7 Hz, 2H), 5.16 (dd, *J* = 7.1, 4.1 Hz, 1H), 3.26 (dd, *J* = 15.5, 7.1 Hz, 1H), 3.00 (dd, *J* = 15.5, 4.1 Hz, 1H), 2.43 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.4, 172.8, 140.6, 140.3, 129.7, 128.9, 127.0, 126.8, 126.2, 60.2, 39.05, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{15}H_{14}N_2OS_2H^+$: 303.0626 $(M+H)^+$, found: 303.0626.

$[\alpha]^{21}_D$ = -79.8 (c = 0.9 in $CHCl_3$).

HPLC analysis: 90:10 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 29.2 min, R_t (major) = 31.6 min.



***(R)*-6-methyl-2-(methylthio)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3o)**

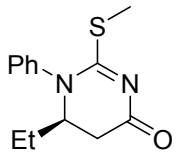
Yield: 20 mg (85%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.42 (m, 3H), 7.32 – 7.21 (m, 2H), 4.10 – 3.86 (m, 1H), 2.95 (dd, *J* = 15.2, 6.4 Hz, 1H), 2.59 (dd, *J* = 15.2, 4.9 Hz, 1H), 2.39 (s, 3H), 1.23 (d, *J* = 6.6 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.9, 172.6, 140.1, 129.9, 129.6, 128.9, 56.4, 38.3, 18.3, 15.1 ppm.

HRMS (ESI, m/z): calculated for $C_{12}H_{14}N_2OSH^+$: 235.0905 $(M+H)^+$, found: 235.0907.

$[\alpha]^{21}_D$ = +18.1 (c = 1.1 in $CHCl_3$).

HPLC analysis: 80.5:19.5 *e.r.* (ASH, 30:70 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 43.6 min, R_t (major) = 80.4 min.



***(R)*-6-ethyl-2-(methylthio)-1-phenyl-5,6-dihydropyrimidin-4(1H)-one (3p)**

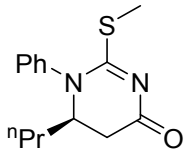
Yield: 20 mg (81%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.42 (m, 2H), 7.33 – 7.22 (m, 2H), 3.86 – 3.63 (m, 1H), 2.92 (dd, *J* = 15.4, 6.9 Hz, 1H), 2.72 (dd, *J* = 15.4, 3.8 Hz, 1H), 2.38 (s, 2H), 1.74 – 1.49 (m, 2H), 0.88 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.0, 172.8, 140.4, 129.9, 129.6, 128.9, 61.9, 34.6, 24.6, 15.1, 9.4 ppm.

HRMS (ESI, m/z): calculated for $C_{13}H_{16}N_2OSH^+$: 249.1062 $(M+H)^+$, found: 249.1062.

$[\alpha]^{21}_D$ = +45.6 (c = 1.8 in $CHCl_3$).

HPLC analysis: 89:11 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 17.7 min, R_t (major) = 19.5 min.



***(R)*-2-(methylthio)-1-phenyl-6-propyl-5,6-dihydropyrimidin-4(1H)-one (3q)**

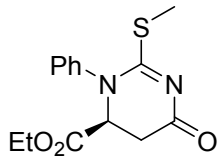
Yield: 23 mg (90%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.55 – 7.39 (m, 3H), 7.34 – 7.21 (m, 2H), 3.96 – 3.63 (m, 1H), 2.92 (dd, *J* = 15.3, 6.8 Hz, 1H), 2.70 (dd, *J* = 15.3, 3.6 Hz, 1H), 2.38 (s, 3H), 1.75 – 1.46 (m, 2H), 1.46 – 1.30 (m, 1H), 1.19 (dddd, *J* = 13.3, 10.5, 9.7, 4.7 Hz, 1H), 0.83 (t, *J* = 7.3 Hz, 3H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.0, 172.7, 140.5, 129.8, 129.5, 128.9, 60.6, 35.1, 33.6, 18.3, 15.1, 13.8 ppm.

HRMS (ESI, m/z): calculated for $C_{14}H_{18}N_2OSH^+$: 263.1218 $(M+H)^+$, found: 263.1218.

$[\alpha]^{21}_D$ = +48.0 (c = 2.1 in $CHCl_3$).

HPLC analysis: 90:10 *e.r.* (ADH, 20:80 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 35.9 min, R_t (major) = 44.0 min.



Ethyl *(S)*-2-(methylthio)-6-oxo-3-phenyl-3,4,5,6-tetrahydropyrimidine-4-carboxylate (3r)

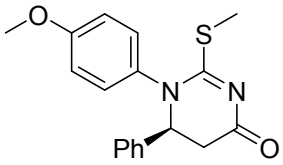
Yield: 19 mg (65%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 – 7.32 (m, 5H), 4.48 (dd, *J* = 7.9, 2.8 Hz, 1H), 4.20 (qq, *J* = 10.8, 7.1 Hz, 2H), 3.11 (dd, *J* = 15.9, 7.9 Hz, 1H), 3.03 (dd, *J* = 15.9, 2.8 Hz, 1H), 2.42 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.2, 171.6, 169.0, 140.9, 130.0, 129.9, 128.7, 62.7, 62.6, 34.0, 15.2, 14.1 ppm.

HRMS (ESI, m/z): calculated for $C14H16N2O_3SH^+$: 293.0960 $(M+H)^+$, found: 293.0960.

$[\alpha]^{21}_D$ = +43.3 (c = 1.4 in $CHCl_3$).

HPLC analysis: 85:15 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 21.9 min, R_t (major) = 26.6 min.



***(S)*-1-(4-methoxyphenyl)-2-(methylthio)-6-phenyl-5,6-dihydropyrimidin-4(1H)-one (4a)**

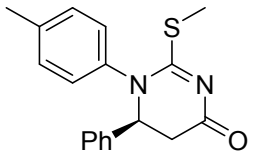
Yield: 27 mg (83%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.22 (m, 3H), 7.22 – 7.12 (m, 2H), 7.00 (s, 2H), 6.81 (d, *J* = 8.2 Hz, 2H), 4.85 (dd, *J* = 7.8, 3.5 Hz, 1H), 3.77 (s, 3H), 3.23 (dd, *J* = 15.4, 7.8 Hz, 1H), 2.86 (dd, *J* = 15.4, 3.5 Hz, 1H), 2.44 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 174.3, 173.1, 160.1, 138.7, 133.3, 129.8, 129.2, 128.7, 126.9, 114.7, 65.06, 55.5, 38.6, 15.2 ppm.

HRMS (ESI, m/z): calculated for $C_{18}H_{18}N_2O_2SH^+$: 327.1167 $(M+H)^+$, found: 327.1167.

$[\alpha]^{21}_D$ = -1.3 (c = 1.9 in $CHCl_3$).

HPLC analysis: 93:7 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 30.6 min, R_t (major) = 35.0 min.



***(S)*-2-(methylthio)-6-phenyl-1-(p-tolyl)-5,6-dihydropyrimidin-4(1H)-one (4b)**

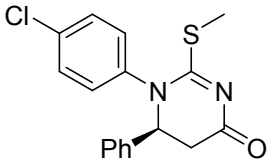
Yield: 24 mg (77%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.30 (ddd, *J* = 10.7, 4.2, 2.7 Hz, 3H), 7.23 – 7.15 (m, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 6.99 (d, *J* = 7.1 Hz, 2H), 4.87 (dd, *J* = 7.8, 3.4 Hz, 1H), 3.23 (dd, *J* = 15.4, 7.8 Hz, 1H), 2.86 (dd, *J* = 15.4, 3.4 Hz, 1H), 2.45 (s, 3H), 2.33 (s, 3H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.9, 173.1, 139.7, 138.7, 138.2, 130.3, 129.2, 128.7, 128.3, 126.8, 65.0, 38.6, 21.3, 15.2 ppm.

HRMS (ESI, m/z): calculated for $C_{18}H_{18}N_2OSH^+$: 311.1218 $(M+H)^+$, found: 311.1218.

$[\alpha]^{21}_D$ = +0.1 (c = 1.9 in $CHCl_3$).

HPLC analysis: 95:5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 20.7 min, R_t (major) = 24.2 min.



***(S)*-1-(4-chlorophenyl)-2-(methylthio)-6-phenyl-5,6-dihydropyrimidin-4(1H)-one (4c)**

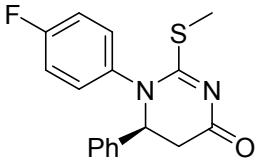
Yield: 23 mg (70%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.37 – 7.27 (m, 1H), 7.17 (dd, *J* = 7.0, 2.3 Hz, 1H), 7.05 (d, *J* = 7.9 Hz, 1H), 4.86 (dd, *J* = 7.6, 3.9 Hz, 1H), 3.23 (dd, *J* = 15.5, 7.7 Hz, 1H), 2.88 (dd, *J* = 15.5, 3.9 Hz, 1H), 2.46 (s, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.5, 172.9, 139.1, 138.3, 135.5, 130.1, 129.9, 129.3, 129.0, 126.8, 65.05, 38.6, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}ClN_2OSH^+$: 331.0672 $(M+H)^+$, found: 331.0672.

$[\alpha]^{21}_D$ = -3.3 (c = 1.8 in $CHCl_3$).

HPLC analysis: 96:4 *e.r.* (OD, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 29.4 min, R_t (major) = 32.0 min.



***(S)*-1-(4-fluorophenyl)-2-(methylthio)-6-phenyl-5,6-dihydropyrimidin-4(1H)-one (4d)**

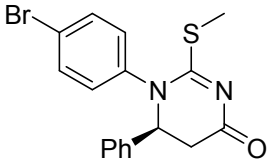
Yield: 26 mg (83%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.28 (m, 3H), 7.17 (dd, *J* = 7.2, 2.3 Hz, 2H), 7.08 (d, *J* = 3.0 Hz, 2H), 7.00 (t, *J* = 8.1 Hz, 2H), 4.86 (dd, *J* = 7.7, 3.9 Hz, 1H), 3.23 (dd, *J* = 15.5, 7.7 Hz, 1H), 2.88 (dd, *J* = 15.5, 3.9 Hz, 1H), 2.46 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.9, 172.9, 164.0, 161.5, 138.4, 136.6, 130.7 (d, *J* = 8.9 Hz), 129.1 (d, *J* = 38.8 Hz), 126.9, 116.7 (d, *J* = 22.9 Hz), 65.1, 38.6, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}FN_2OSH^+$: 315.0967 $(M+H)^+$, found: 315.0967.

$[\alpha]^{21}_D$ = -14.5 (c = 2.0 in $CHCl_3$).

HPLC analysis: 95:5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 22.6 min, R_t (major) = 25.6 min.



***(S)*-1-(4-bromophenyl)-2-(methylthio)-6-phenyl-5,6-dihydropyrimidin-4(1H)-one (4e)**

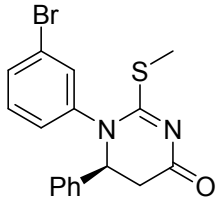
Yield: 26 mg (83%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.45 (d, *J* = 8.7 Hz, 2H), 7.36 – 7.28 (m, 3H), 7.22 – 7.11 (m, 2H), 6.99 (d, *J* = 8.0 Hz, 2H), 4.86 (dd, *J* = 7.7, 3.8 Hz, 1H), 3.23 (dd, *J* = 15.5, 7.7 Hz, 1H), 2.88 (dd, *J* = 15.5, 3.8 Hz, 1H), 2.46 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.4, 172.8, 139.7, 138.3, 132.9, 130.4, 129.3, 129.0, 126.8, 123.6, 65.0, 38.6, 15.2 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}BrN_2OSH^+$: 375.0167 $(M+H)^+$, found: 375.0167.

$[\alpha]^{21}_D$ = -0.6 (c = 2.3 in $CHCl_3$).

HPLC analysis: 95.5:4.5 *e.r.* (OD, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 30.5 min, R_t (major) = 32.9 min.



***(S)*-1-(3-bromophenyl)-2-(methylthio)-6-phenyl-5,6-dihydropyrimidin-4(1H)-one (4f)**

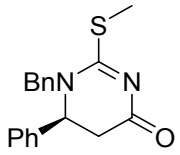
Yield: 23 mg (61%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.53 – 7.42 (m, 1H), 7.38 – 7.27 (m, 4H), 7.24 – 7.10 (m, 3H), 7.03 (d, *J* = 7.5 Hz, 1H), 4.89 (dd, *J* = 7.6, 3.8 Hz, 1H), 3.23 (dd, *J* = 15.4, 7.6 Hz, 1H), 2.88 (dd, *J* = 15.4, 3.8 Hz, 1H), 2.47 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.4, 172.8, 141.9, 138.2, 132.7, 131.9, 130.8, 129.3, 129.0, 127.5, 126.8, 122.8, 65.0, 38.7, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{17}H_{15}BrN_2OSH^+$: 375.0167 $(M+H)^+$, found: 375.0166.

$[\alpha]^{21}_{D}$ = +8.9 (c = 1.8 in $CHCl_3$).

HPLC analysis: 95:5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 23.6 min, R_t (major) = 28.5 min.



***(S)*-1-benzyl-2-(methylthio)-6-phenyl-5,6-dihydropyrimidin-4(1H)-one (4g)**

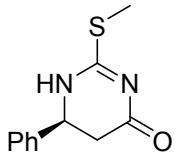
Yield: 18 mg (58%)

^{1}H NMR (400 MHz, $CDCl_3$) *δ* 7.44 – 7.29 (m, 6H), 7.26 – 7.19 (m, 2H), 7.16 – 7.05 (m, 2H), 5.12 (d, *J* = 16.3 Hz, 1H), 4.56 (dd, *J* = 7.9, 2.7 Hz, 1H), 4.08 (d, *J* = 16.3 Hz, 1H), 2.96 (dd, *J* = 15.4, 8.0 Hz, 1H), 2.70 (dd, *J* = 15.4, 2.8 Hz, 1H), 2.62 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.8, 172.6, 138.0, 135.1, 129.5, 129.3, 128.8, 128.5, 127.5, 126.4, 59.6, 53.0, 38.5, 15.0 ppm.

HRMS (ESI, m/z): calculated for $C_{18}H_{18}N_2OSH^+$: 311.1218 $(M+H)^+$, found: 311.1218.

$[\alpha]^{21}_{D}$ = +26.4 (c = 0.8 in $CHCl_3$).

HPLC analysis: 89.5:10.5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 25.2 min, R_t (major) = 28.3 min.



***(S)*-2-(methylthio)-6-phenyl-5,6-dihydropyrimidin-4(1H)-one (4h)**

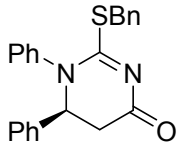
Yield: 14.5 mg (66%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (s, 1H), 7.48 – 7.34 (m, 4H), 7.34 – 7.26 (m, 1H), 4.84 (dd, *J* = 12.2, 5.2 Hz, 1H), 2.82 (dd, *J* = 16.7, 5.2 Hz, 1H), 2.61 – 2.43 (m, 4H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 169.8, 152.5, 142.3, 128.8, 127.5, 126.4, 58.8, 38.4, 13.4.

HRMS (ESI, m/z): calculated for $C_{11}H_{12}N_2OSH^+$: 221.0749 $(M+H)^+$, found: 221.0746.

$[\alpha]^{21}_D$ = +67.0 (c = 0.5 in $CHCl_3$).

HPLC analysis: 87:13 *e.r.* (ID, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (major) = 17.9 min, R_t (minor) = 21.7 min.



***(S)*-2-(benzylthio)-1,6-diphenyl-5,6-dihydropyrimidin-4(1H)-one (4i)**

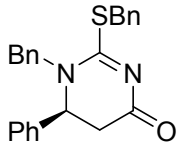
Yield: 19 mg (51%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.14 (m, 12H), 7.10 (s, 2H), 4.90 (dd, *J* = 7.7, 3.6 Hz, 1H), 4.39 (s, 2H), 3.25 (dd, *J* = 15.4, 7.7 Hz, 1H), 2.89 (dd, *J* = 15.4, 3.6 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 173.0, 172.9, 140.6, 138.5, 136.3, 129.7, 129.6, 129.5, 129.3, 128.8, 128.7, 128.7, 127.6, 126.9, 65.0, 38.7, 37.1 ppm.

HRMS (ESI, m/z): calculated for $C_{23}H_{20}N_2OSH^+$: 373.1375 $(M+H)^+$, found: 373.1372.

$[\alpha]^{21}_D$ = -15.3 (c = 0.3 in $CHCl_3$).

HPLC analysis: 95.5:4.5 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 28.0 min, R_t (major) = 34.2 min.



***(S)*-1-benzyl-2-(benzylthio)-6-phenyl-5,6-dihydropyrimidin-4(1H)-one (4j)**

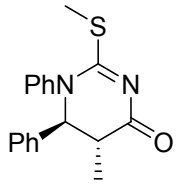
Yield: 20 mg (52%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 – 7.40 (m, 2H), 7.40 – 7.26 (m, 9H), 7.24 – 7.16 (m, 2H), 7.13 (dd, *J* = 7.5, 1.8 Hz, 2H), 5.10 (d, *J* = 16.2 Hz, 1H), 4.69 – 4.42 (m, 3H), 4.06 (t, *J* = 16.6 Hz, 1H), 2.96 (dd, *J* = 15.4, 8.0 Hz, 1H), 2.70 (dd, *J* = 15.4, 2.8 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 172.6, 172.0, 137.9, 136.2, 135.0, 129.6, 129.5, 129.3, 128.9, 128.5, 127.8, 127.5, 126.4, 59.5, 53.1, 38.5, 36.9 ppm.

HRMS (ESI, m/z): calculated for $C_{24}H_{22}N_2OSH^+$: 387.1531 $(M+H)^+$, found: 387.1529.

$[\alpha]^{21}_D$ = -1.6 (c = 0.7 in $CHCl_3$).

HPLC analysis: 90:10 *e.r.* (ADH, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 21.2 min, R_t (major) = 24.9 min.



(5*R*,6*S*)-5-methyl-2-(methylthio)-1,6-diphenyl-5,6-dihydropyrimidin-4(1H)-one (5a)

Yield: 27 mg (87%)

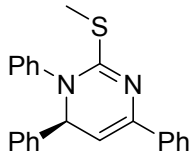
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.23 (m, 6H), 7.20 – 7.14 (m, 2H), 7.11 (d, *J* = 3.1 Hz, 2H), 4.50 (d, *J* = 4.0 Hz, 1H), 2.88 (qd, *J* = 7.1, 4.1 Hz, 1H), 2.47 (s, 3H), 1.44 (d, *J* = 7.1 Hz, 3H). **^{13}C NMR** (100

MHz, $CDCl_3$) δ 177.4, 172.7, 140.9, 138.2, 129.7, 129.4, 129.2, 128.7, 126.9, 71.8, 42.9, 17.0, 15.3 ppm.

HRMS (ESI, m/z): calculated for $C_{18}H_{18}N_2OSH^+$: 311.1218 $(M+H)^+$, found: 311.1212.

$[\alpha]^{21}_D$ = -26.4 (c = 1.2 in $CHCl_3$).

HPLC analysis: 91:9 *e.r.* (IA, 15:85 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (major) = 20.7 min, R_t (minor) = 25.4 min.



***(S)*-2-(methylthio)-1,4,6-triphenyl-1,6-dihydropyrimidine (5b)**

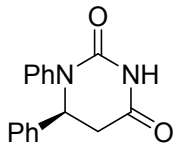
Yield: 35 mg (98%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 – 7.84 (m, 2H), 7.36 (t, *J* = 7.4 Hz, 2H), 7.33 – 7.26 (m, 9H), 7.11 – 7.00 (m, 2H), 5.71 (d, *J* = 5.0 Hz, 1H), 5.30 (d, *J* = 4.9 Hz, 1H), 2.50 (s, 3H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 158.7, 143.6, 142.3, 140.6, 138.5, 129.3, 129.2, 128.8, 128.3, 128.3, 128.2, 127.9, 127.7, 125.6, 103.1, 65.8, 14.8 ppm.

HRMS (ESI, m/z): calculated for $C_{23}H_{20}N_2SH^+$: 357.1425 $(M+H)^+$, found: 357.1425.

$[\alpha]^{21}_D$ = +12.7 (c = 1.4 in $CHCl_3$).

HPLC analysis: 90:10 *e.r.* (ADH, 3:97 ^{i}PrOH/Hexane, 0.3 mL/min), R_t (minor) = 21.1 min, R_t (major) = 25.2 min.



***(S)*-1,6-diphenyldihydropyrimidine-2,4(1H,3H)-dione (5c)**

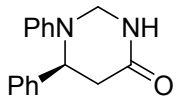
Yield: 21.6 mg (81%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.43 – 7.29 (m, 5H), 7.28 – 7.14 (m, 5H), 5.08 (dd, *J* = 6.7, 2.6 Hz, 1H), 3.34 (dd, *J* = 16.6, 6.8 Hz, 1H), 2.95 (dd, *J* = 16.6, 2.6 Hz, 1H). **^{13}C NMR** (100 MHz, $CDCl_3$) δ 168.2, 151.8, 140.7, 138.6, 129.4, 129.3, 128.7, 127.2, 126.0, 125.9, 60.0, 39.4 ppm.

HRMS (ESI, m/z): calculated for $C_{16}H_{14}N_2O_2H^+$: 267.1134 $(M+H)^+$, found: 267.1126.

$[\alpha]^{21}_D$ = +139.8 (c = 0.4 in $CHCl_3$).

HPLC analysis: 91:9 *e.r.* (ADH, 30:70 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (minor) = 30.7 min, R_t (major) = 43.9 min.



***(S)*-1,6-diphenyltetrahydropyrimidin-4(1H)-one (5d)**

Yield: 18.4 mg (73%)

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.34 (m, 4H), 7.33 – 7.19 (m, 3H), 6.97 – 6.84 (m, 3H), 6.71 (s, 1H), 4.97 (t, *J* = 5.7 Hz, 1H), 4.81 (dd, *J* = 12.2, 2.9 Hz, 1H), 4.63 (dd, *J* = 12.1, 2.8 Hz, 1H), 2.93 (dd,

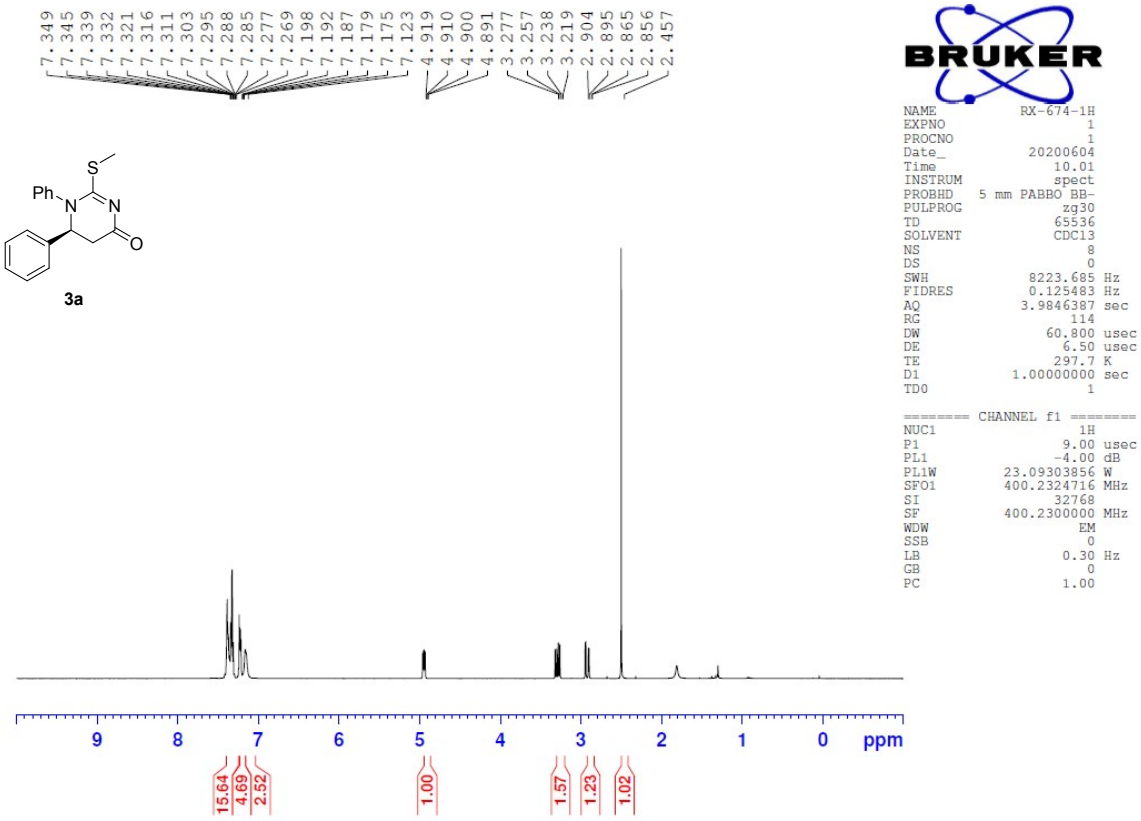
J = 16.4, 6.4 Hz, 1H), 2.76 (dd, *J* = 16.4, 5.0 Hz, 1H).**^{13}C NMR** (100 MHz, $CDCl_3$) δ 171.2, 148.3, 141.0, 129.6, 129.1, 127.9, 126.6, 120.8, 116.6, 59.6, 56.0, 36.9 ppm.

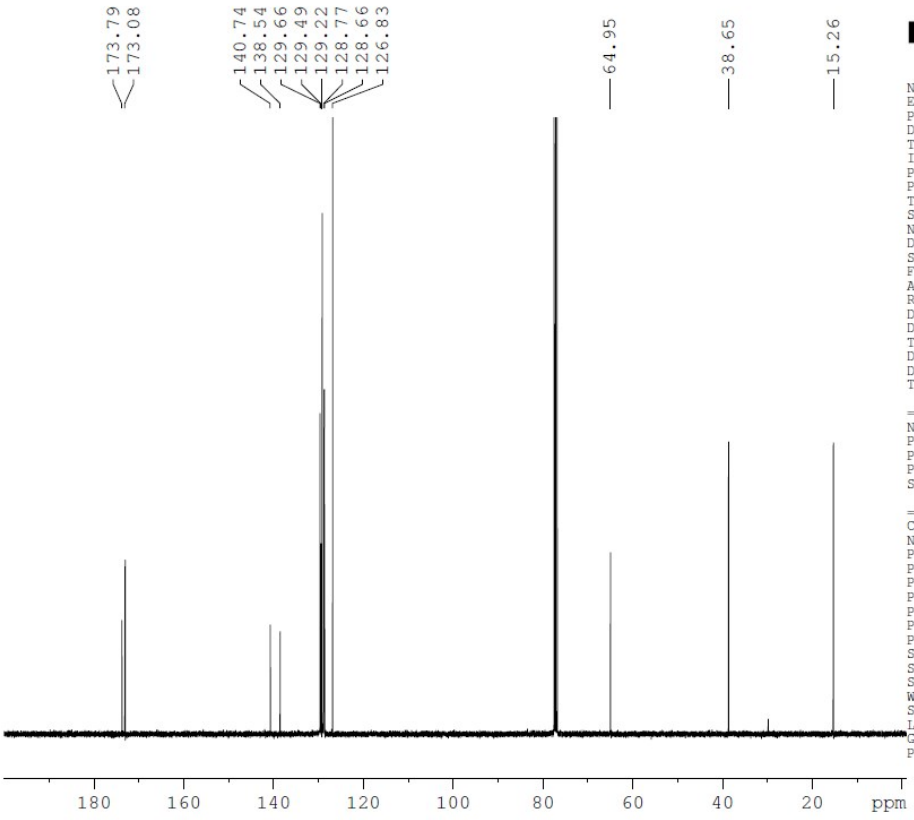
HRMS (ESI, m/z): calculated for $C_{16}H_{16}N_2OH^+$: 253.1341 $(M+H)^+$, found: 253.1341.

$[\alpha]^{21}_{D}$ = +94.8 (c = 0.8 in $CHCl_3$).

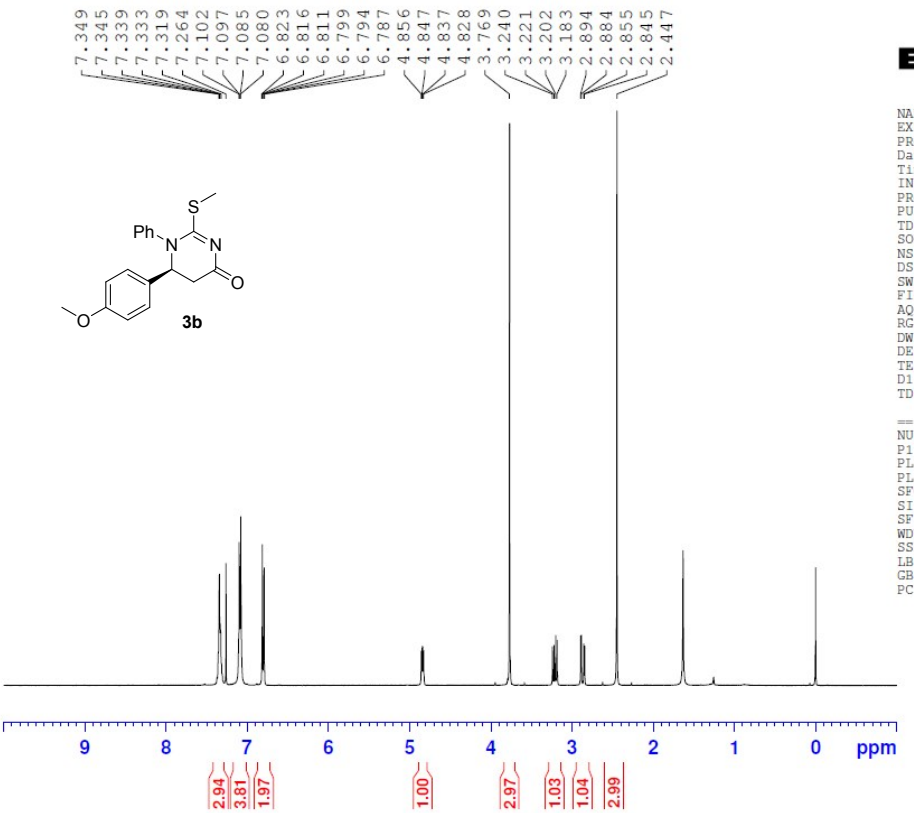
HPLC analysis: 93:7 *e.r.* (ADH, 20:80 ^{i}PrOH/Hexane, 0.5 mL/min), R_t (major) = 16.3 min, R_t (minor) = 17.9 min.

II. ^{1}H, ^{13}C NMR and HPLC Spectra

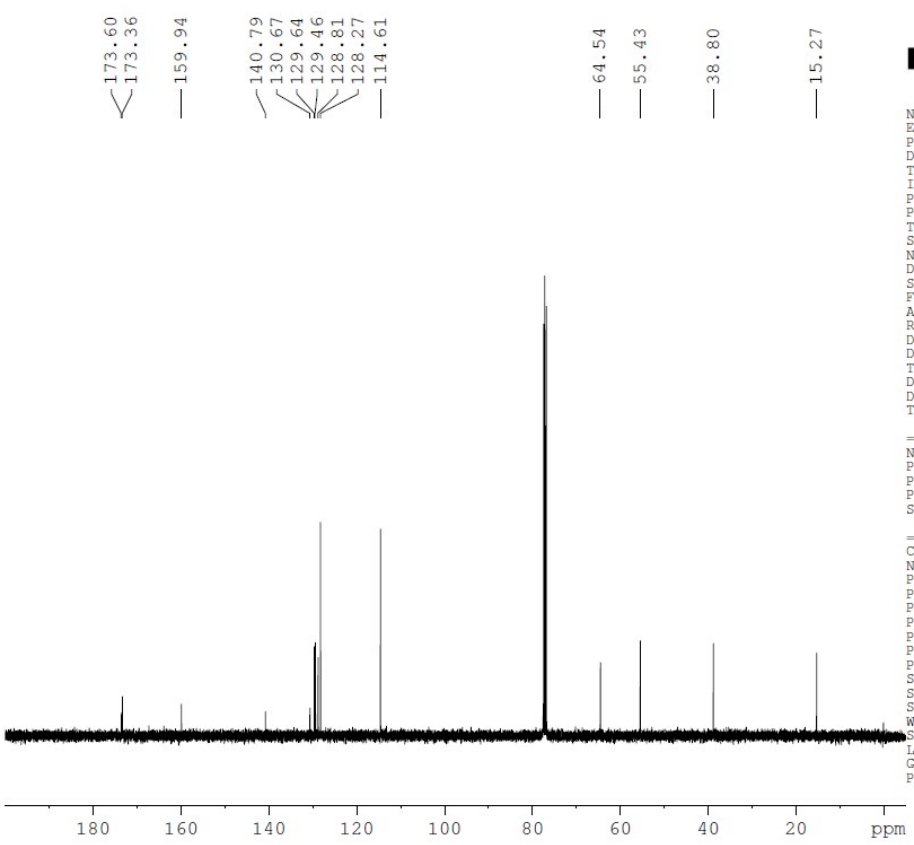





```
NAME            RX-674-13C
EXPNO                    1
PROCNO                   1
Date_             20200604
Time                 10.06
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   3621
DS                      4
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ             1.2976629 sec
RG                   22.6
DW                19.800 usec
DE                 6.50 usec
TE                298.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   100

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PL1                -2.00 dB
PL1W         59.71607590 W
SFO1        100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               14.98 dB
PL13               13.89 dB
PL2W         23.09303856 W
PL12W         0.29206610 W
PL13W         0.37538856 W
SFO2        400.2316009 MHz
SI                 32768
SF          100.6379047 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
```

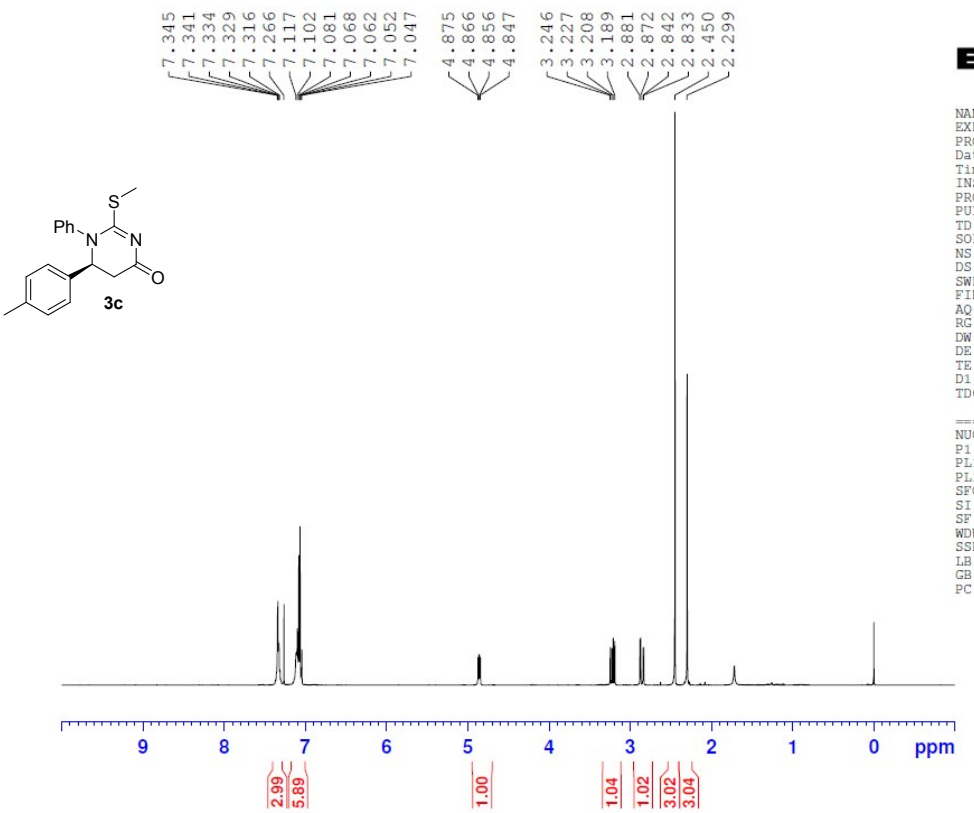



```
NAME       RX-663-1-1-1H
EXPNO                  1
PROCNO                 1
Date_           20191119
Time               18.59
INSTRUM            spect
PROBHD  5 mm PABBO BB-
PULPROG             zg30
TD                 65536
SOLVENT           CDCl3
NS                     8
DS                     0
SWH          8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                   181
DW                60.800 usec
DE                 6.50 usec
TE                299.8 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.00 usec
PL1               -4.00 dB
PL1W        23.09303856 W
SFO1       400.2324716 MHz
SI                 32768
SF         400.2300189 MHz
WDW                  EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



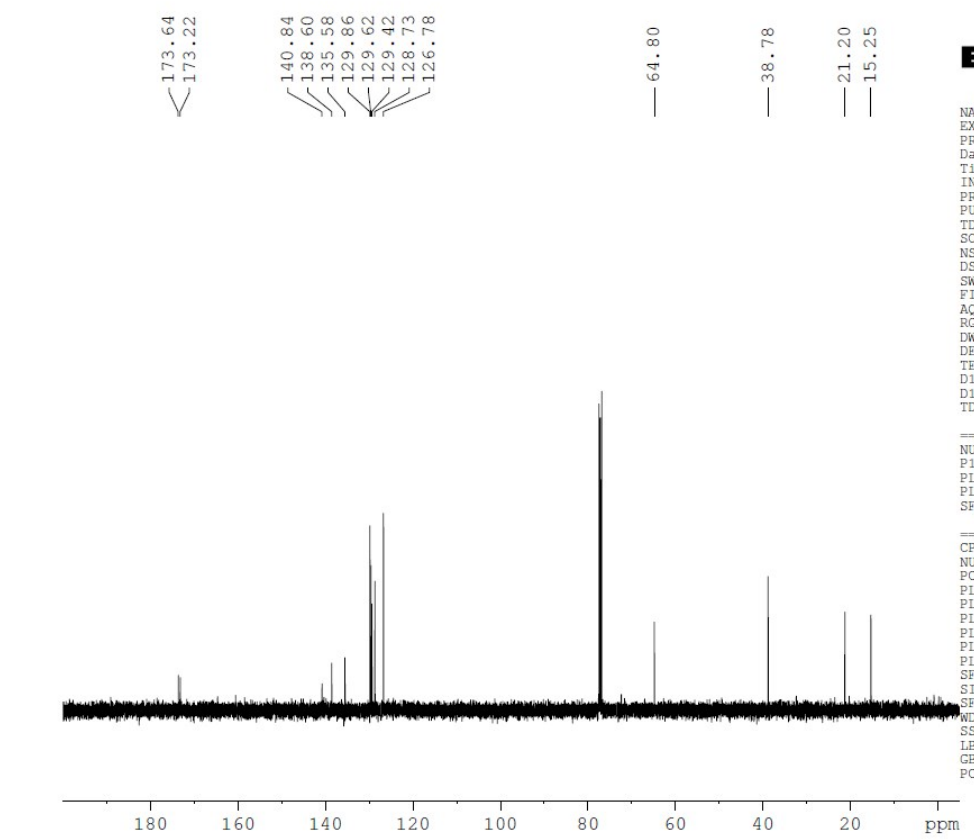
BRUKER

NAME	RX-663-1-1-13C
EXPNO	1
PROCNO	1
Date_	20191119
Time	19.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDC13
NS	712
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	25.4
DW	19.800 usec
DE	6.50 usec
TE	300.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	100
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	-2.00 dB
PL1W	59.71607590 W
SFO1	100.6499905 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-4.00 dB
PL12	14.98 dB
PL13	13.89 dB
PL2W	23.09303856 W
PL12W	0.29206610 W
PL13W	0.37538856 W
SFO2	400.2316009 MHz
SI	32768
SF	100.6378992 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40



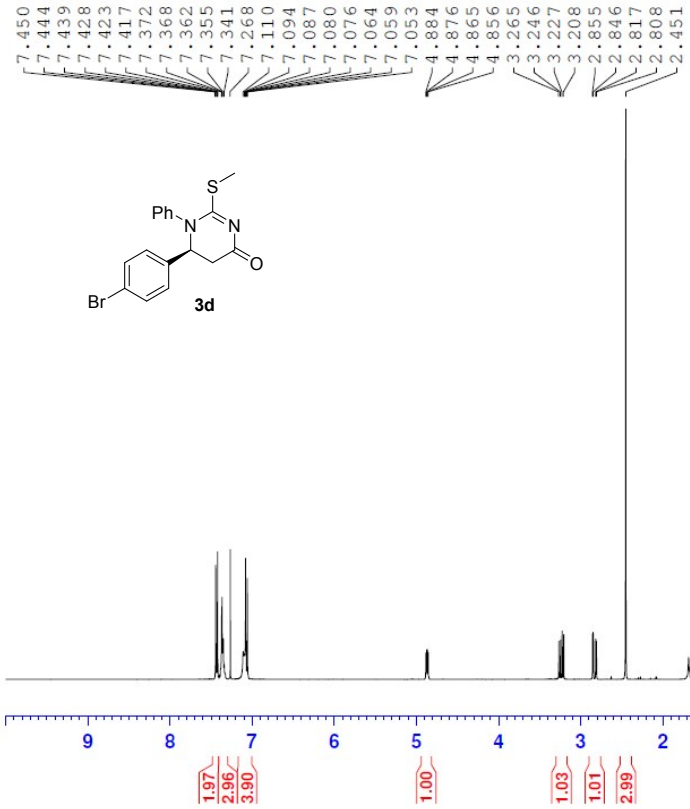
BRUKER

```
NAME          RX-663-2-1-1H
EXPNO                    1
PROCNO                   1
Date_             20191119
Time                 19.47
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                       8
DS                       0
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     128
DW                60.800 usec
DE                 6.50 usec
TE                 300.2 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.00 usec
PL1                -4.00 dB
PL1W         23.09303856 W
SFO1         400.2324716 MHz
SI                   32768
SF           400.2300180 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   1.00
```



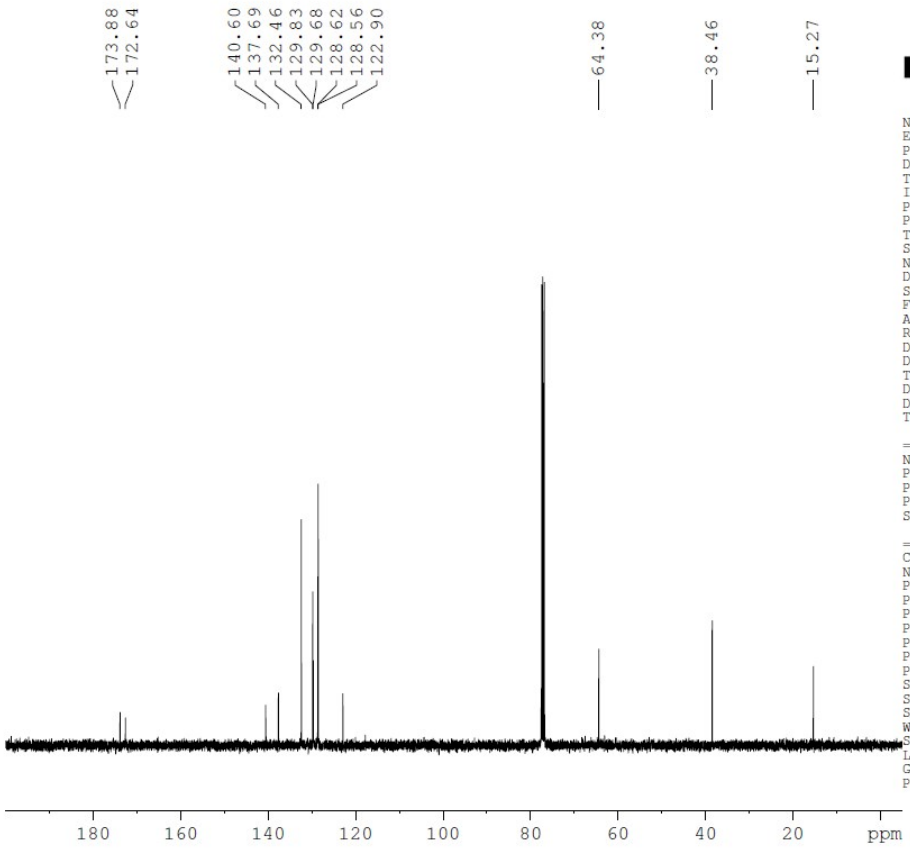
BRUKER

```
NAME          RX-663-2-1-13C
EXPNO                    1
PROCNO                   1
Date_             20191119
Time                 19.57
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     198
DS                       4
SWH             25252.525 Hz
FIDRES           0.385323 Hz
AQ               1.2976629 sec
RG                    25.4
DW                19.800 usec
DE                 6.50 usec
TE                 300.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    100

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -2.00 dB
PL1W         59.71607590 W
SFO1         100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               14.98 dB
PL13               13.89 dB
PL2W         23.09303856 W
PL12W         0.29206610 W
PL13W         0.37538856 W
SFO2         400.2316009 MHz
SI                   32768
SF           100.6379019 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   1.40
```



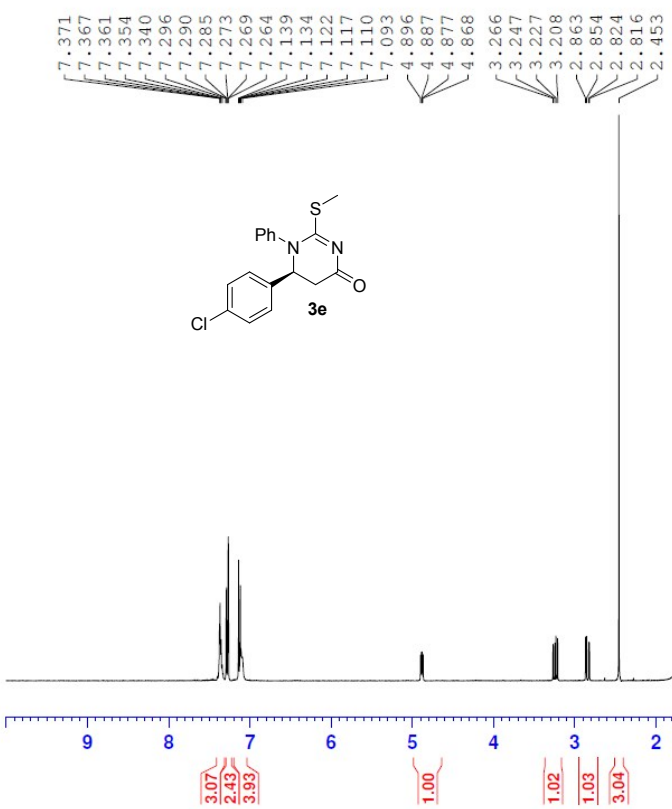
BRUKER

```
NAME          RX-663-3-1-1H
EXPNO                    1
PROCNO                   1
Date_             20191119
Time                 20.07
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDC13
NS                       8
DS                       0
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     144
DW                60.800 usec
DE                 6.50 usec
TE                 300.1 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.00 usec
PL1                -4.00 dB
PL1W         23.09303856 W
SFO1         400.2324716 MHz
SI                   32768
SF           400.2300174 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   1.00
```



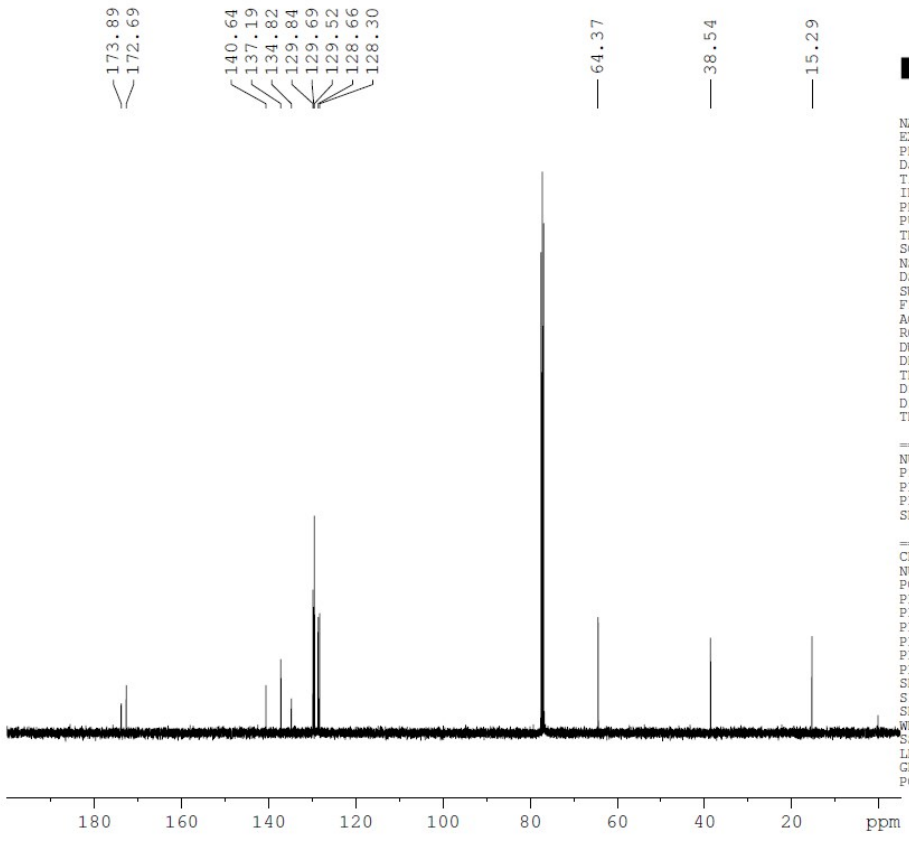
BRUKER

```
NAME          RX-663-3-1-13C
EXPNO                    1
PROCNO                   1
Date_             20191119
Time                 20.09
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                   65536
SOLVENT              CDC13
NS                     196
DS                       4
SWH              25252.525 Hz
FIDRES           0.385323 Hz
AQ               1.2976629 sec
RG                    25.4
DW                19.800 usec
DE                 6.50 usec
TE                 300.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    100

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -2.00 dB
PL1W         59.71607590 W
SFO1         100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               14.98 dB
PL13               13.89 dB
PL2W         23.09303856 W
PL12W         0.29206610 W
PL13W         0.37538856 W
SFO2         400.2316009 MHz
SI                   32768
SF           100.6379016 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```



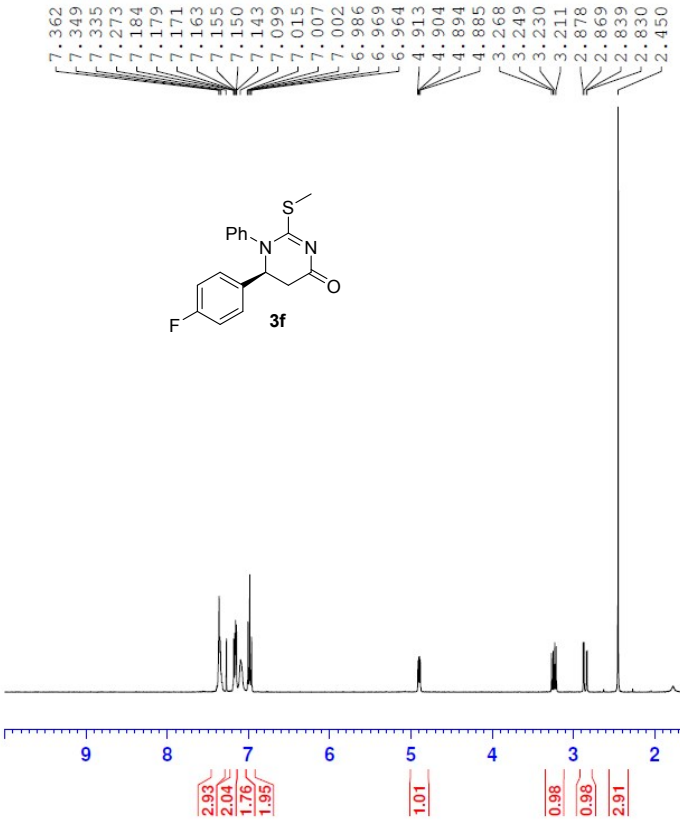
BRUKER

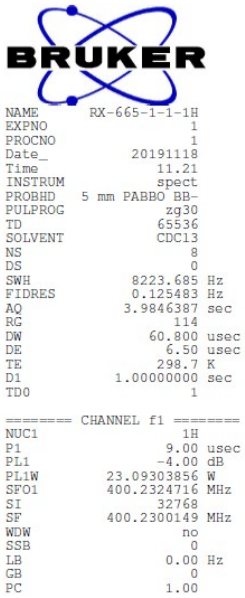
```
NAME          RX-663-4-1-1H
EXPNO                    1
PROCNO                   1
Date_             20191119
Time                 20.24
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               8223.685 Hz
FIDRES           0.125483 Hz
AQ                3.9846387 sec
RG                     161
DW                  60.800 usec
DE                   6.50 usec
TE                  300.0 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.00 usec
PL1                 -4.00 dB
PL1W          23.09303856 W
SFO1          400.2324716 MHz
SI                   32768
SF            400.2300190 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

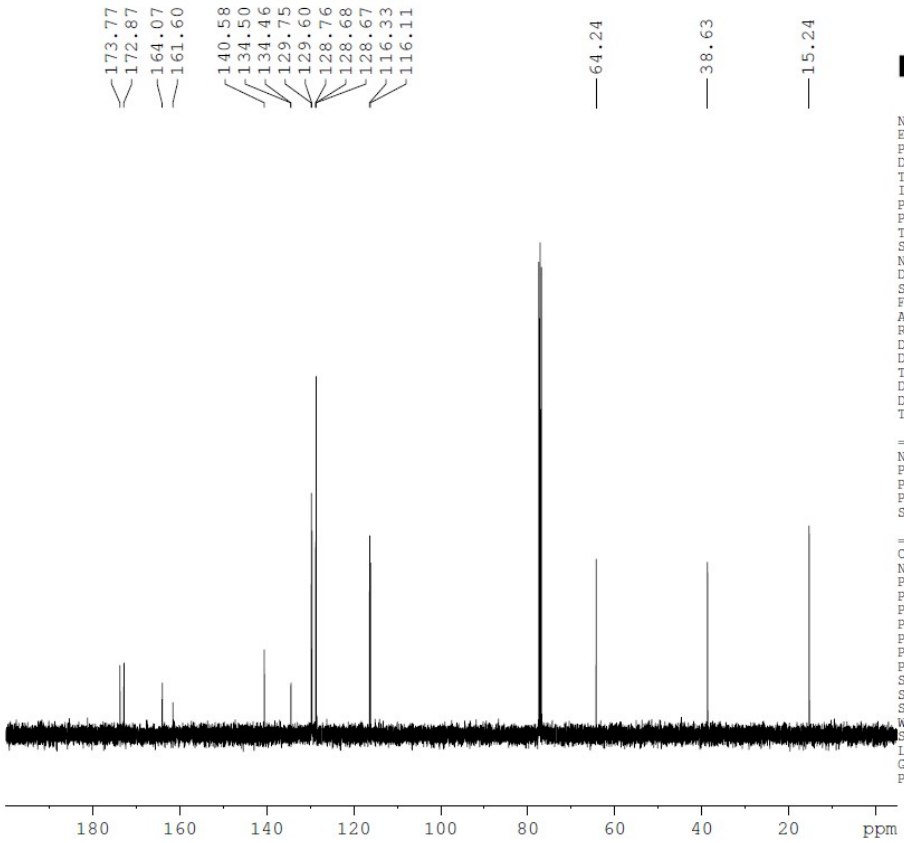


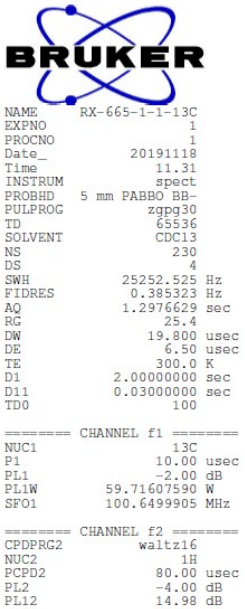
BRUKER

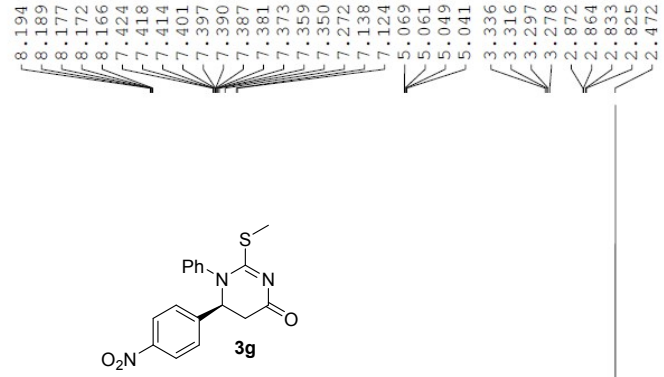
```
NAME          RX-663-4-1-13C
EXPNO                    1
PROCNO                   1
Date_             20191119
Time                 20.31
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     986
DS                       4
SWH              25252.525 Hz
FIDRES           0.385323 Hz
AQ                1.2976629 sec
RG                    25.4
DW                  19.800 usec
DE                   6.50 usec
TE                  300.9 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                    100

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                 -2.00 dB
PL1W          59.71607590 W
SFO1          100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                14.98 dB
PL13                13.89 dB
PL2W          23.09303856 W
PL12W          0.29206610 W
PL13W          0.37538856 W
SFO2          400.2316009 MHz
SI                   32768
SF            100.6378992 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40
```


7.362
7.349
7.335
7.273
7.184
7.179
7.171
7.163
7.155
7.150
7.143
7.099
7.015
7.007
7.002
6.986
6.969
6.964
4.913
4.904
4.894
4.885
3.268
3.249
3.230
3.211
2.878
2.869
2.839
2.830
2.450
S
Ph
N
N
O
F
3f
9
8
7
6
5
4
3
2
1
0
ppm
2.93
2.04
1.76
1.95
1.01
0.98
0.98
2.91


BRUKER
NAME RX-665-1-1-1H
EXPNO 1
PROCNO 1
Date_ 20191118
Time 11.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 -4.00 dB
PL1W 23.09303856 W
SFO1 400.2324716 MHz
SI 32768
SF 400.2300149 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

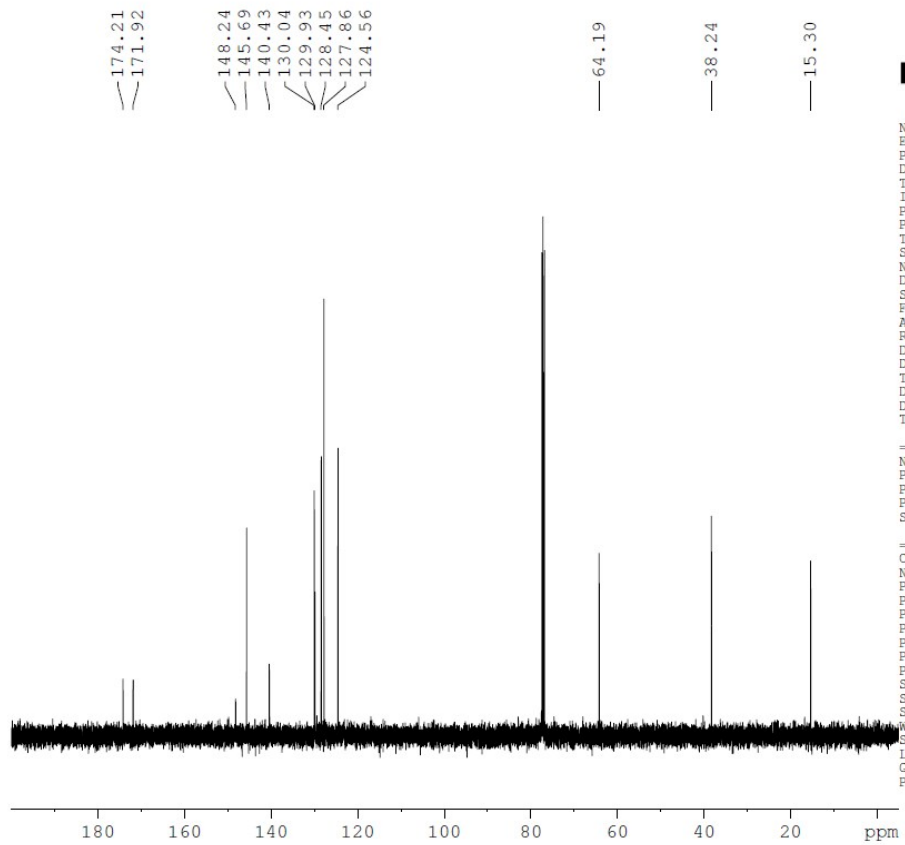

173.77
172.87
164.07
161.60
140.58
134.50
134.46
129.75
129.60
128.76
128.68
128.67
116.33
116.11
64.24
38.63
15.24
180
160
140
120
100
80
60
40
20
ppm


BRUKER
NAME RX-665-1-1-13C
EXPNO 1
PROCNO 1
Date_ 20191118
Time 11.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 230
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 25.4
DW 19.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 100
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -2.00 dB
PL1W 59.71607590 W
SFO1 100.6499905 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 14.98 dB
PL13 13.89 dB
PL2W 23.09303856 W
PL12W 0.29206610 W
PL13W 0.37538856 W
SFO2 400.2316009 MHz
SI 32768
SF 100.6379031 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40

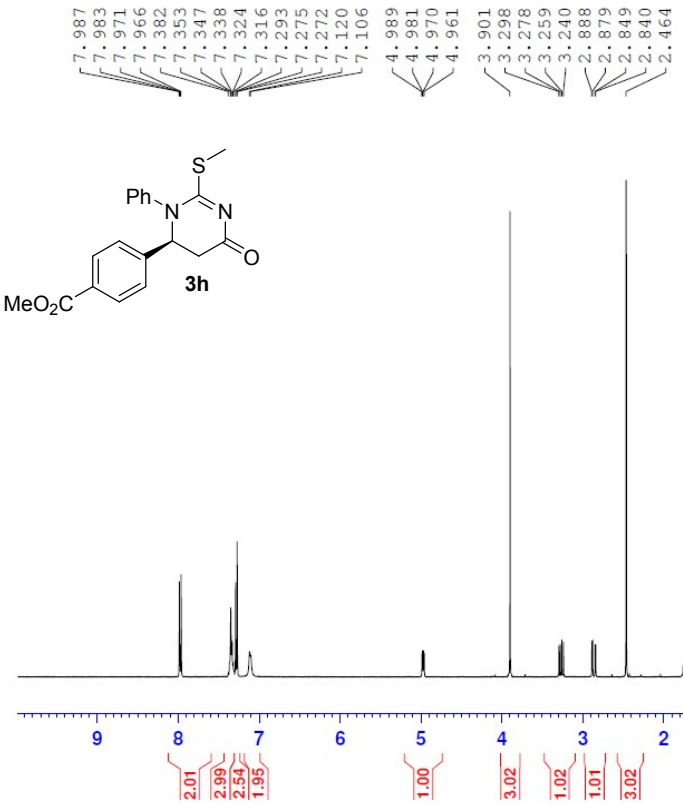


3g


```
NAME          RX-665-3-1-1H
EXPNO                    1
PROCNO                   1
Date_             20191118
Time                 12.06
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               8223.685 Hz
FIDRES           0.125483 Hz
AQ                3.9846387 sec
RG                     114
DW                  60.800 usec
DE                   6.50 usec
TE                  299.4 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   1H
P1                   9.00 usec
PL1                 -4.00 dB
PL1W           23.09303856 W
SFO1          400.2324716 MHz
SI                   32768
SF            400.2300153 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

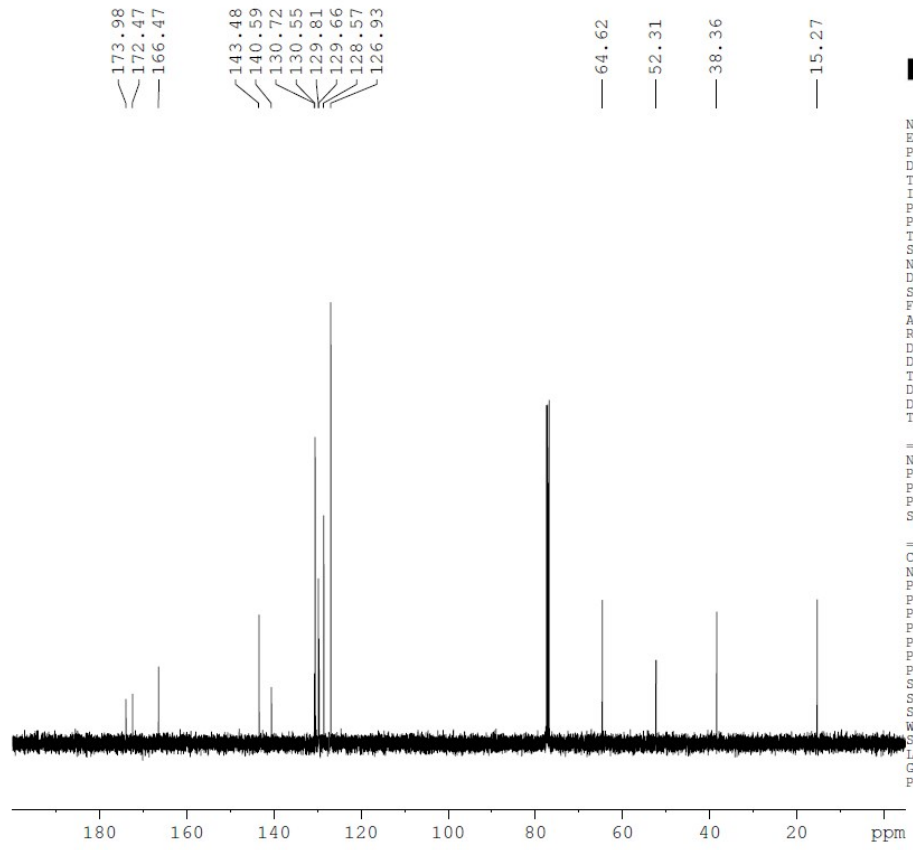



```
NAME          RX-665-3-1-13C
EXPNO                    1
PROCNO                   1
Date_             20191118
Time                 12.10
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     200
DS                       4
SWH              25252.525 Hz
FIDRES           0.385323 Hz
AQ                1.2976629 sec
RG                    25.4
DW                  19.800 usec
DE                   6.50 usec
TE                  299.5 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                    100

======== CHANNEL f1 ========
NUC1                  13C
P1                  10.00 usec
PL1                 -2.00 dB
PL1W           59.71607590 W
SFO1          100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                14.98 dB
PL13                13.89 dB
PL2W           23.09303856 W
PL12W           0.29206610 W
PL13W           0.37538856 W
SFO2          400.2316009 MHz
SI                   32768
SF            100.6379023 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.40
```



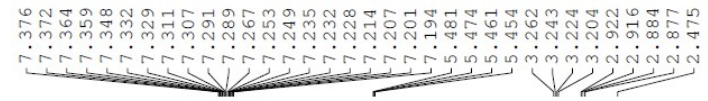
BRUKER

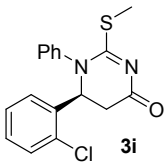
```
NAME          RX-665-5-1-1H
EXPNO                    1
PROCNO                   1
Date_             20191118
Time                 12.26
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    101
DW                 60.800 usec
DE                  6.50 usec
TE                 299.5 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.00 usec
PL1                -4.00 dB
PL1W          23.09303856 W
SFO1          400.2324716 MHz
SI                  32768
SF            400.2300140 MHz
WDW                    no
SSB                     0
LB                  0.00 Hz
GB                      0
PC                   1.00
```



BRUKER

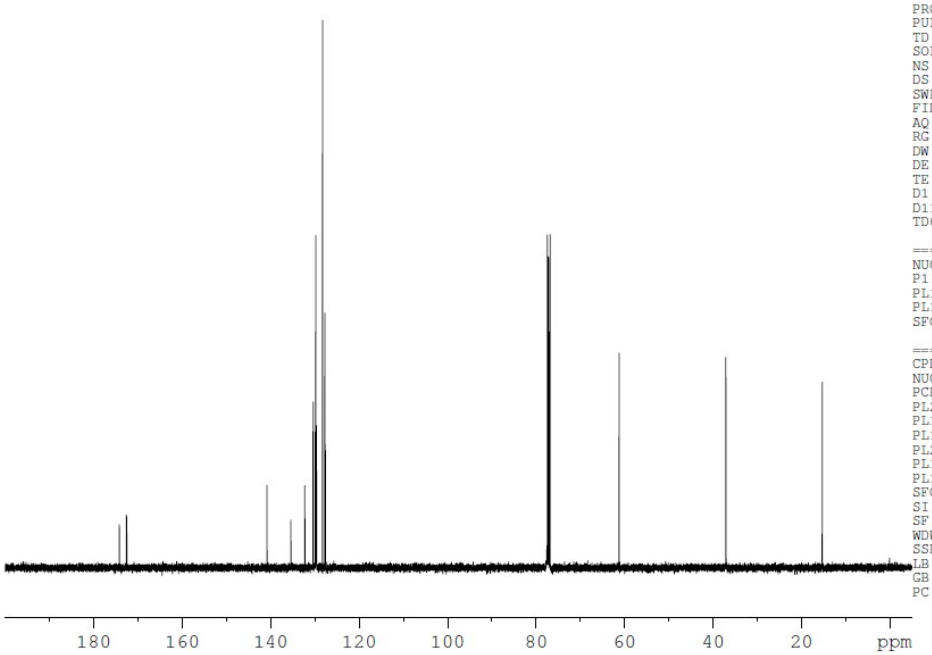
```
NAME          RX-665-5-1-13C
EXPNO                    1
PROCNO                   1
Date_             20191118
Time                 12.36
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    177
DS                      4
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ             1.2976629 sec
RG                   22.6
DW                 19.800 usec
DE                  6.50 usec
TE                 300.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                   100

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -2.00 dB
PL1W          59.71607590 W
SFO1          100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               14.98 dB
PL13               13.89 dB
PL2W          23.09303856 W
PL12W          0.29206610 W
PL13W          0.37538856 W
SFO2          400.2316009 MHz
SI                  32768
SF            100.6379030 MHz
WDW                    no
SSB                     0
LB                  0.00 Hz
GB                      0
PC                   1.40
```



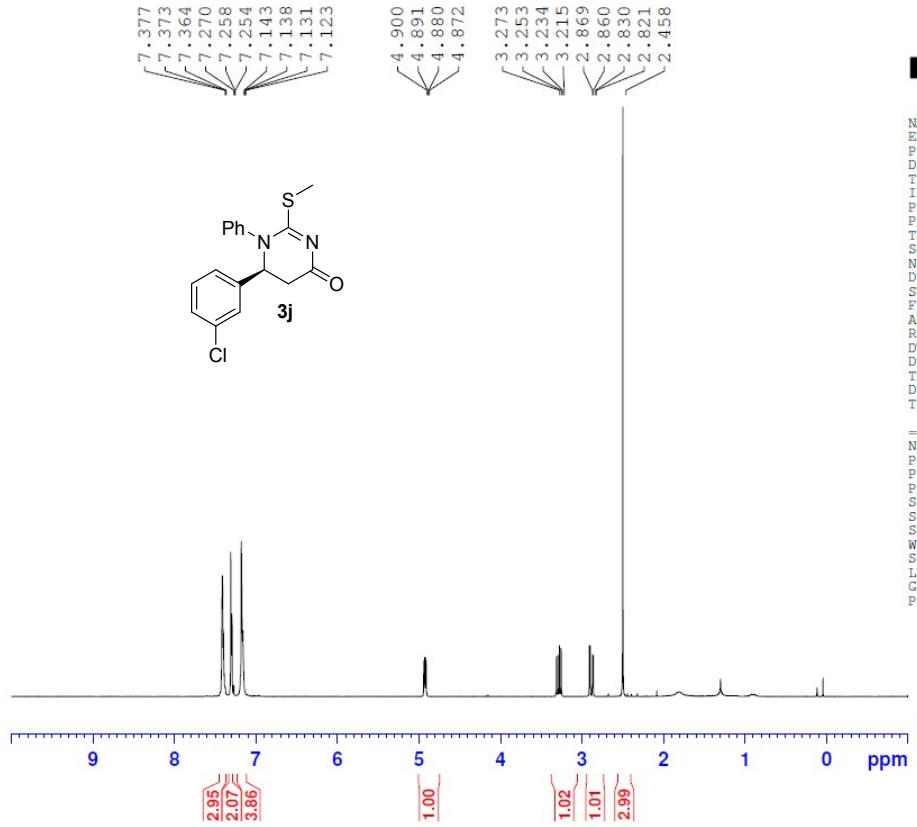


3i

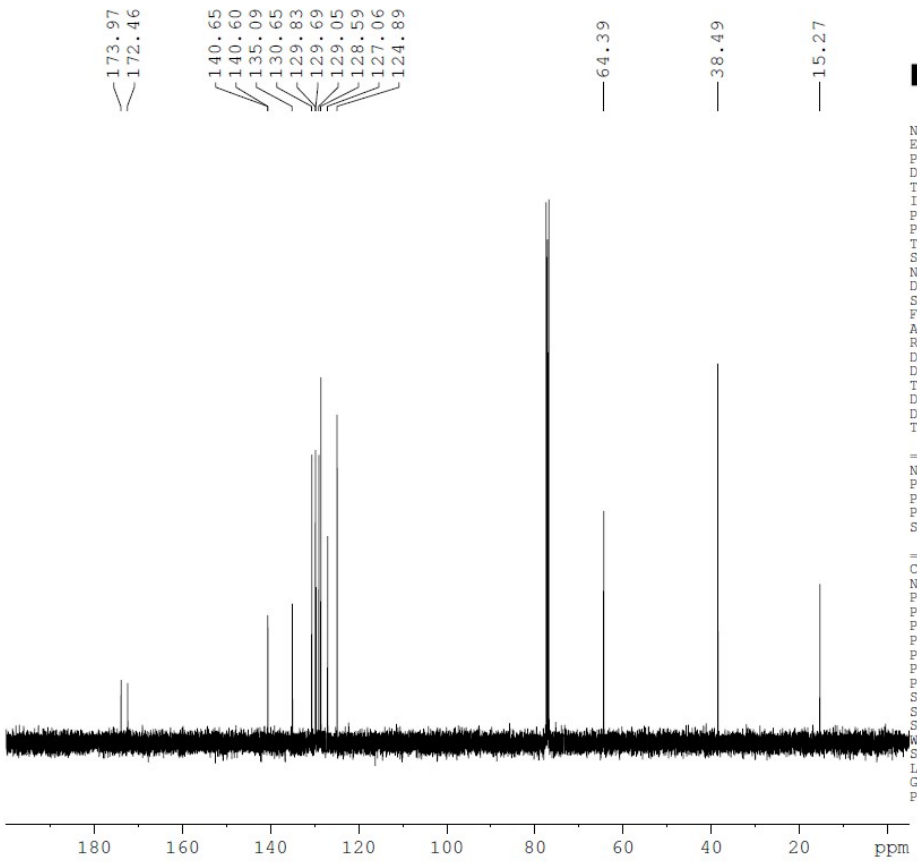

```
NAME          RX-667-4-1-1H
EXPNO                    1
PROCNO                   1
Date_             20191118
Time                 12.46
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                      8
DS                      0
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                    114
DW                 60.800 usec
DE                  6.50 usec
TE                 299.5 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  9.00 usec
PL1                -4.00 dB
PL1W          23.09303856 W
SFO1          400.2324716 MHz
SI                  32768
SF            400.2300172 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   1.00
```



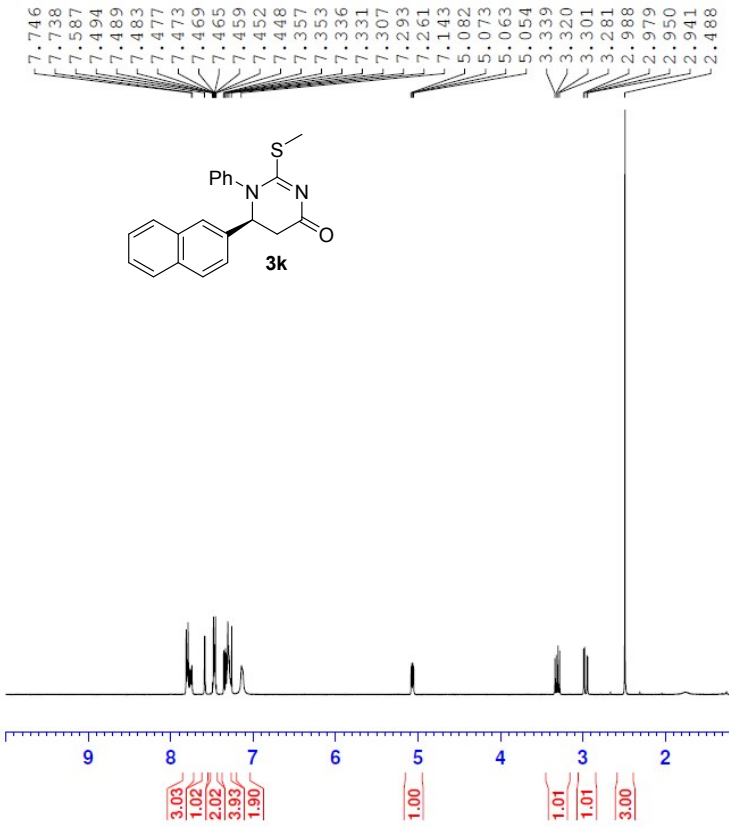

```
NAME         RX-667-4-1-13C
EXPNO                    1
PROCNO                   1
Date_             20191118
Time                 12.56
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG            zgpg30
TD                  65536
SOLVENT             CDC13
NS                   1231
DS                      4
SWH             25252.525 Hz
FIDRES           0.385323 Hz
AQ               1.2976629 sec
RG                   22.6
DW                 19.800 usec
DE                  6.50 usec
TE                 300.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                   100

======== CHANNEL f1 ========
NUC1                  13C
P1                 10.00 usec
PL1                -2.00 dB
PL1W          59.71607590 W
SFO1          100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -4.00 dB
PL12               14.98 dB
PL13               13.89 dB
PL2W          23.09303856 W
PL12W          0.29206610 W
PL13W          0.37538856 W
SFO2          400.2316009 MHz
SI                  32768
SF            100.6379021 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   1.40
```



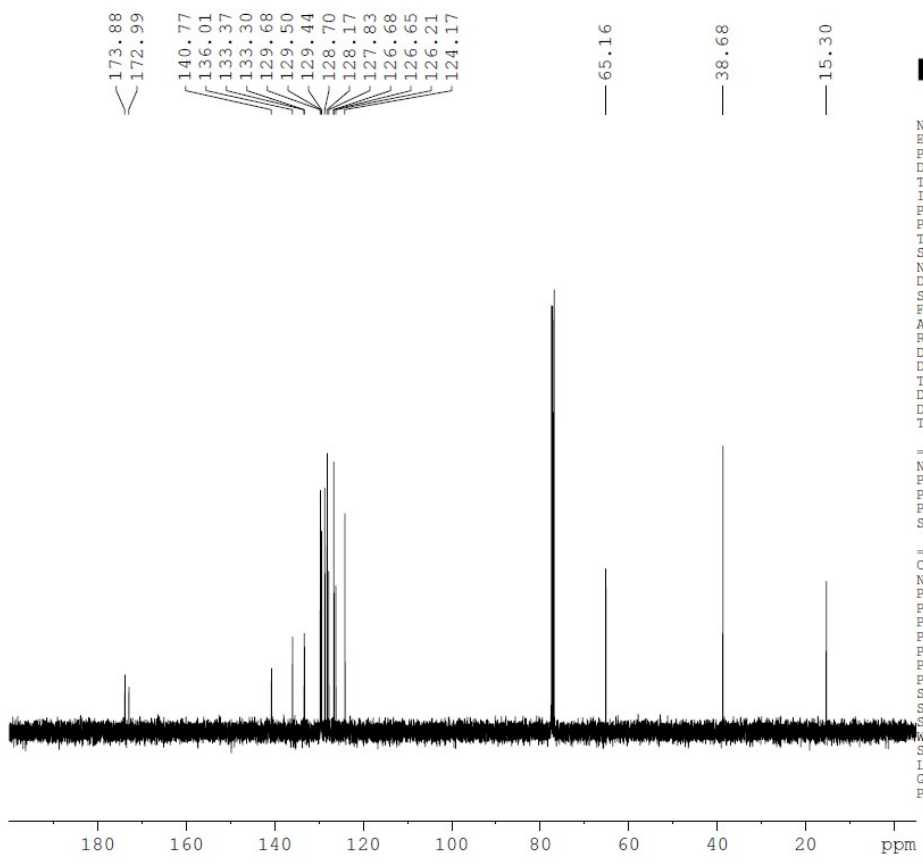

```
NAME          RX-713-3-1HNMR
EXPNO                      1
PROCNO                     1
Date_               20200114
Time                   20.12
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                        8
DS                        0
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      114
DW                   60.800 usec
DE                    6.50 usec
TE                   299.4 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.00 usec
PL1                   -4.00 dB
PL1W           23.09303856 W
SFO1           400.2324716 MHz
SI                    32768
SF             400.2300000 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00
```



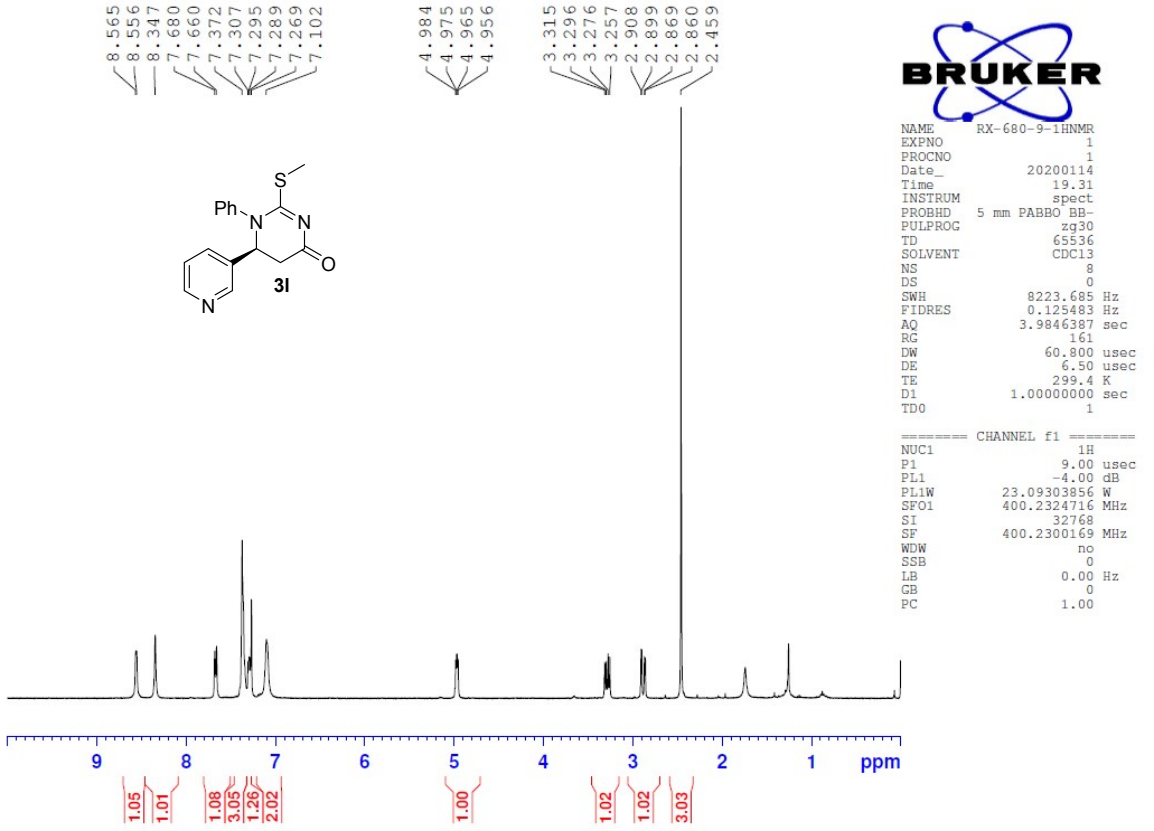

```
NAME          RX-713-3-13CNMR
EXPNO                      1
PROCNO                     1
Date_               20200114
Time                   20.21
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDC13
NS                      256
DS                        4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                       32
DW                   19.800 usec
DE                    6.50 usec
TE                   300.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                     100

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.00 usec
PL1                   -2.00 dB
PL1W           59.71607590 W
SFO1           100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  14.98 dB
PL13                  13.89 dB
PL2W           23.09303856 W
PL12W           0.29206610 W
PL13W           0.37538856 W
SFO2           400.2316009 MHz
SI                    32768
SF             100.6379025 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.40
```



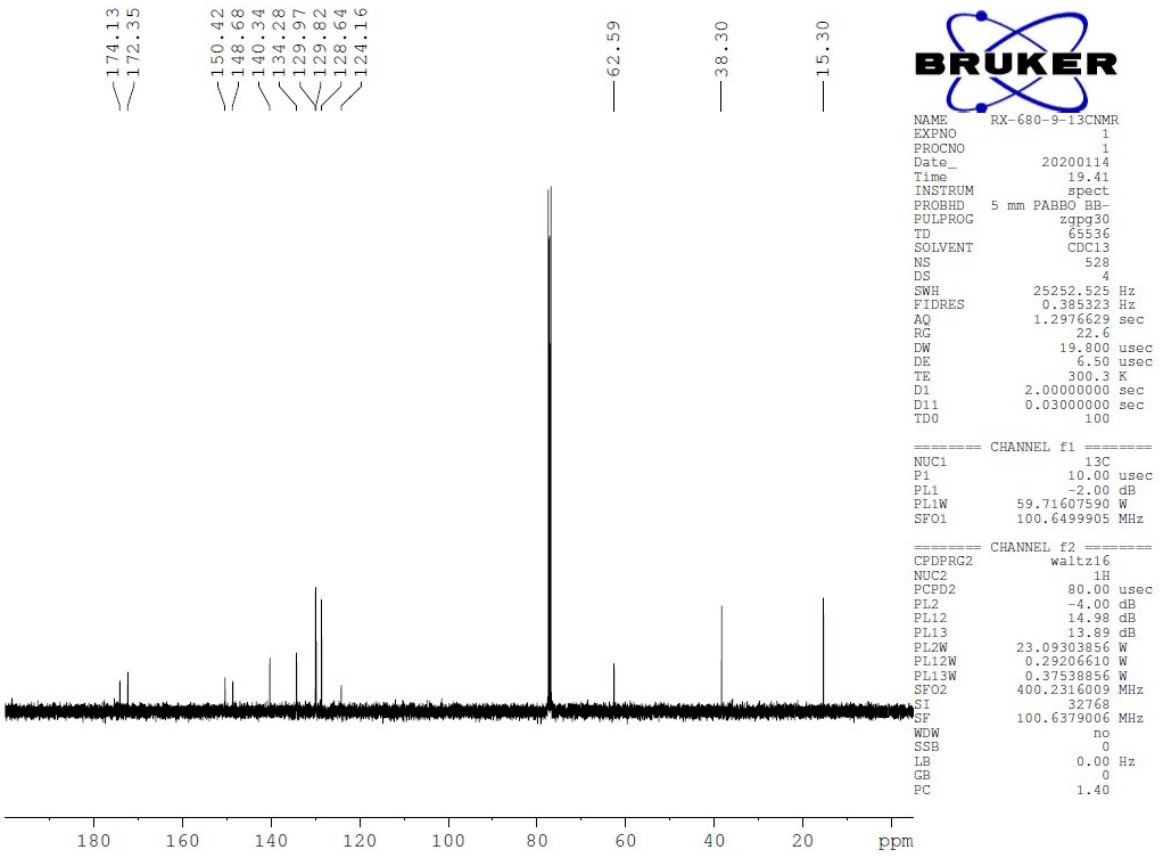

```
NAME          RX-665-2-1-1H
EXPNO                    1
PROCNO                   1
Date_             20191118
Time                 11.47
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     101
DW                  60.800 usec
DE                   6.50 usec
TE                  299.2 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.00 usec
PL1                 -4.00 dB
PL1W           23.09303856 W
SFO1           400.2324716 MHz
SI                   32768
SF             400.2300198 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```



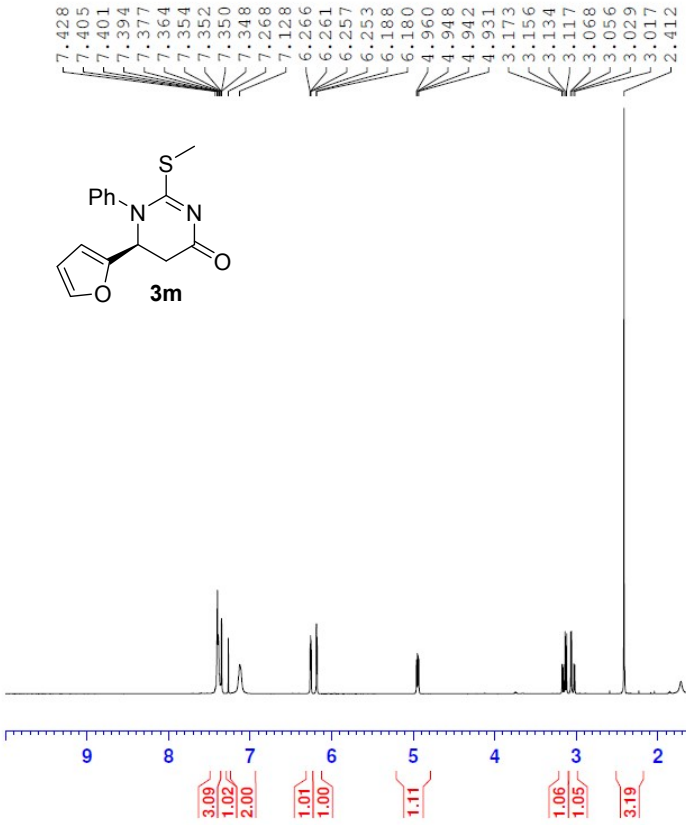

```
NAME          RX-665-2-1-13C
EXPNO                    1
PROCNO                   1
Date_             20191118
Time                 11.54
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     203
DS                       4
SWH              25252.525 Hz
FIDRES            0.385323 Hz
AQ                1.2976629 sec
RG                    22.6
DW                  19.800 usec
DE                   6.50 usec
TE                  300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                    100

======== CHANNEL f1 ========
NUC1                   13C
P1                  10.00 usec
PL1                 -2.00 dB
PL1W           59.71607590 W
SFO1           100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                14.98 dB
PL13                13.89 dB
PL2W           23.09303856 W
PL12W           0.29206610 W
PL13W           0.37538856 W
SFO2           400.2316009 MHz
SI                   32768
SF             100.6379037 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.40
```




```
NAME          RX-680-9-1HNMR
EXPNO                      1
PROCNO                     1
Date_               20200114
Time                   19.31
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDC13
NS                         8
DS                         0
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                       161
DW                    60.800 usec
DE                     6.50 usec
TE                    299.4 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.00 usec
PL1                   -4.00 dB
PL1W              23.09303856 W
SFO1              400.2324716 MHz
SI                       32768
SF                400.2300169 MHz
WDW                       no
SSB                        0
LB                     0.00 Hz
GB                         0
PC                     1.00
```

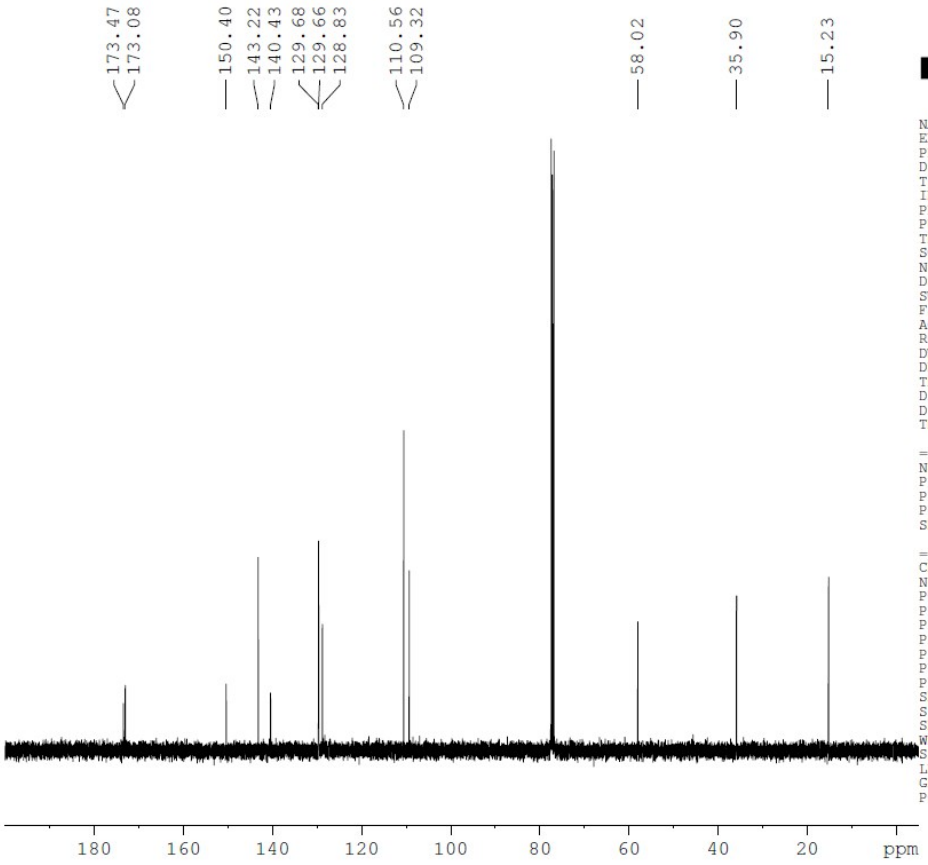



```
NAME         RX-680-9-13CNMR
EXPNO                      1
PROCNO                     1
Date_               20200114
Time                   19.41
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                     65536
SOLVENT                CDC13
NS                       528
DS                         4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976629 sec
RG                      22.6
DW                    19.800 usec
DE                     6.50 usec
TE                    300.3 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                      100

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.00 usec
PL1                   -2.00 dB
PL1W              59.71607590 W
SFO1              100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  14.98 dB
PL13                  13.89 dB
PL2W              23.09303856 W
PL12W              0.29206610 W
PL13W              0.37538856 W
SFO2              400.2316009 MHz
SI                       32768
SF                100.6379006 MHz
WDW                       no
SSB                        0
LB                     0.00 Hz
GB                         0
PC                     1.40
```



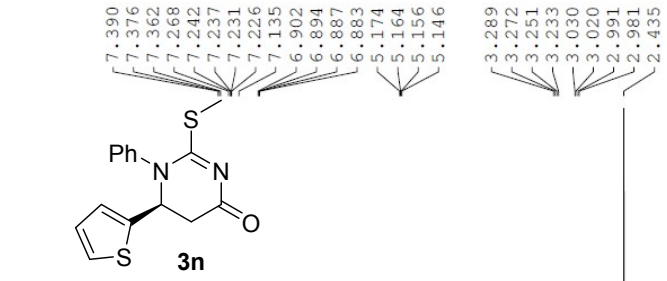
BRUKER

```
NAME          RX-719-1-1-HNMR
EXPNO                       1
PROCNO                      1
Date_              20200219
Time                   22.36
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDC13
NS                         8
DS                         0
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       144
DW                    60.800 usec
DE                     6.50 usec
TE                    298.7 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.00 usec
PL1                   -4.00 dB
PL1W              23.09303856 W
SFO1              400.2324716 MHz
SI                     32768
SF                400.2300174 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.00
```

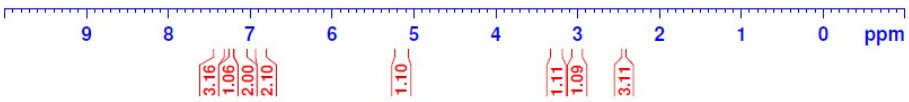


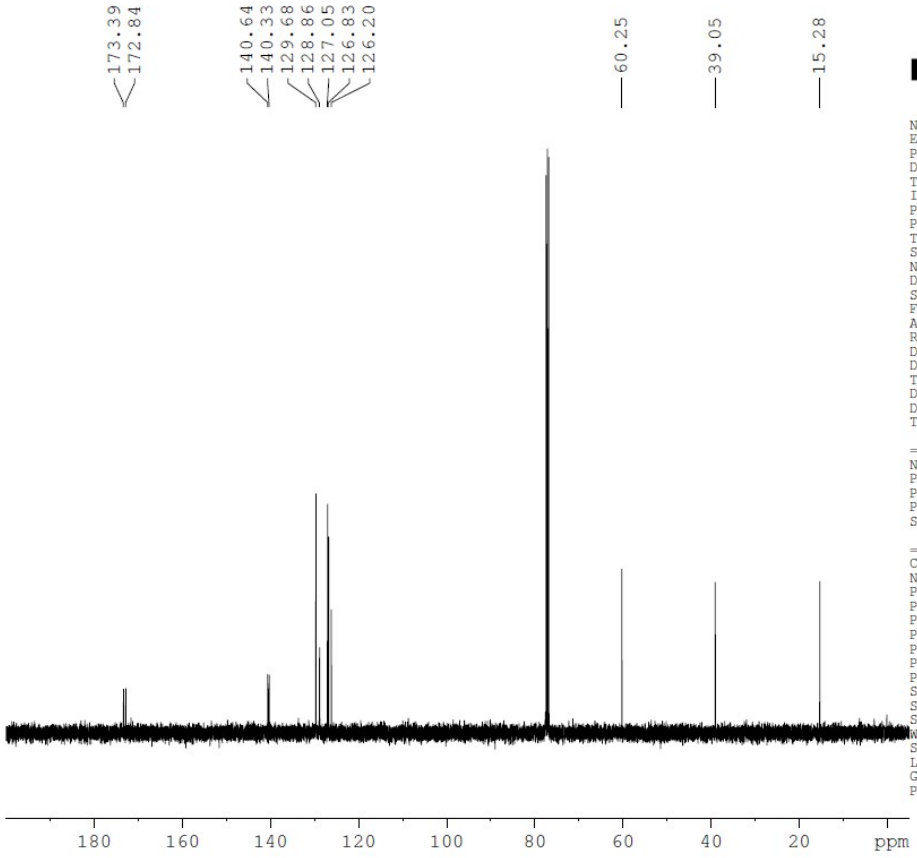
BRUKER

```
NAME          RX-719-1-1-CNMR
EXPNO                       1
PROCNO                      1
Date_              20200219
Time                   22.46
INSTRUM                spect
PROBHD     5 mm PABBO BB-
PULPROG              zgpg30
TD                     65536
SOLVENT                CDC13
NS                       480
DS                         4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                      22.6
DW                    19.800 usec
DE                     6.50 usec
TE                    300.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                      100

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.00 usec
PL1                   -2.00 dB
PL1W              59.71607590 W
SFO1              100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  14.98 dB
PL13                  13.89 dB
PL2W              23.09303856 W
PL12W              0.29206610 W
PL13W              0.37538856 W
SFO2              400.2316009 MHz
SI                     32768
SF                100.6379014 MHz
WDW                       no
SSB                        0
LB                     0.00 Hz
GB                         0
PC                      1.40
```



3n



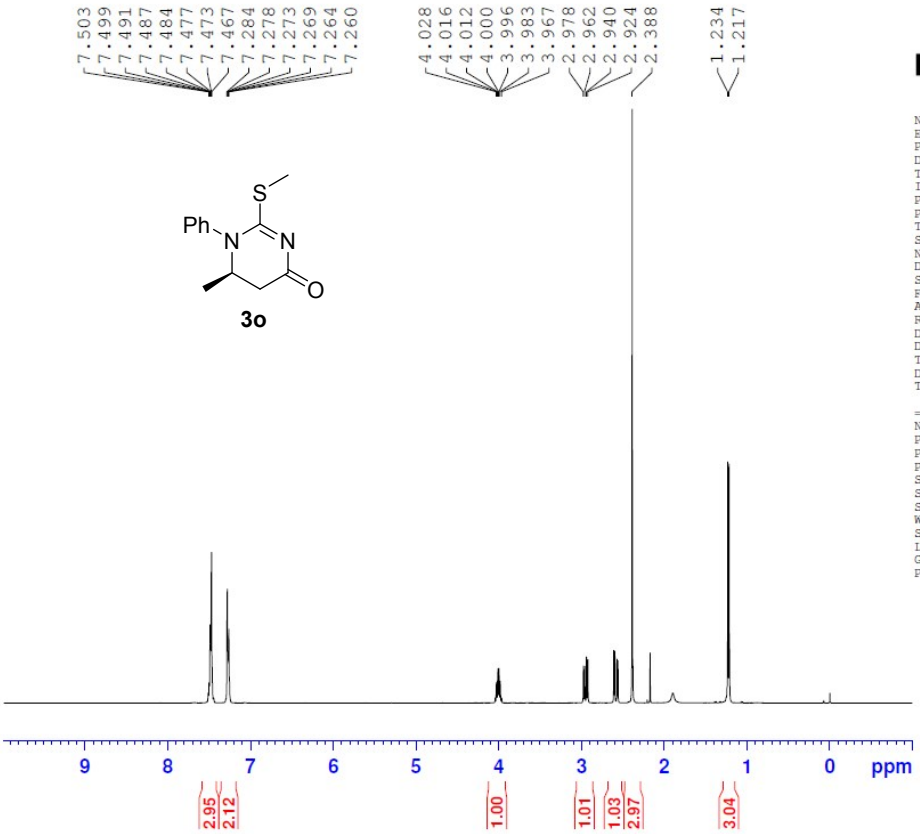


BRUKER

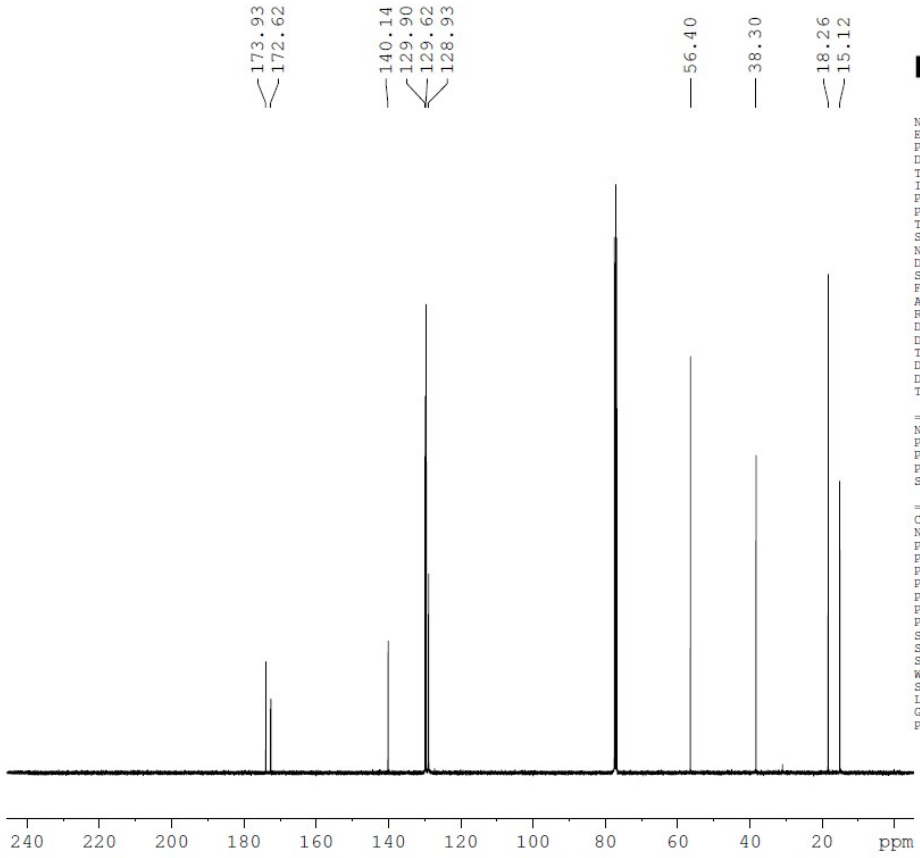
```
NAME          RX-719-2-1-HNMR
EXPNO                       1
PROCNO                      1
Date_                20200219
Time                    10.08
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDC13
NS                         8
DS                         0
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                3.9846387 sec
RG                       144
DW                    60.800 usec
DE                     6.50 usec
TE                    298.4 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.00 usec
PL1                   -4.00 dB
PL1W             23.09303856 W
SFO1            400.2324716 MHz
SI                      32768
SF               400.2300176 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

BRUKER

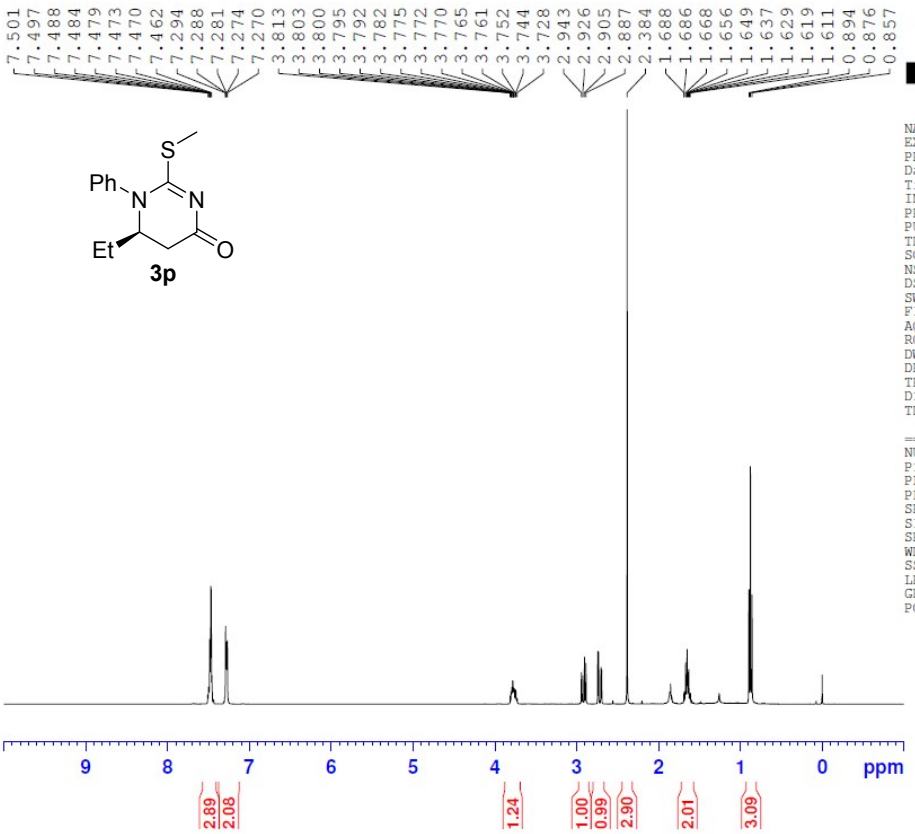
```
NAME          RX-719-2-1-CNMR
EXPNO                       1
PROCNO                      1
Date_                20200219
Time                    10.34
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                     65536
SOLVENT                CDC13
NS                       447
DS                         4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                1.2976629 sec
RG                      25.4
DW                    19.800 usec
DE                     6.50 usec
TE                    299.9 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      100

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.00 usec
PL1                   -2.00 dB
PL1W             59.71607590 W
SFO1            100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  14.98 dB
PL13                  13.89 dB
PL2W             23.09303856 W
PL12W             0.29206610 W
PL13W             0.37538856 W
SFO2            400.2316009 MHz
SI                      32768
SF               100.6379010 MHz
WDW                        no
SSB                        0
LB                      0.00 Hz
GB                         0
PC                      1.40
```



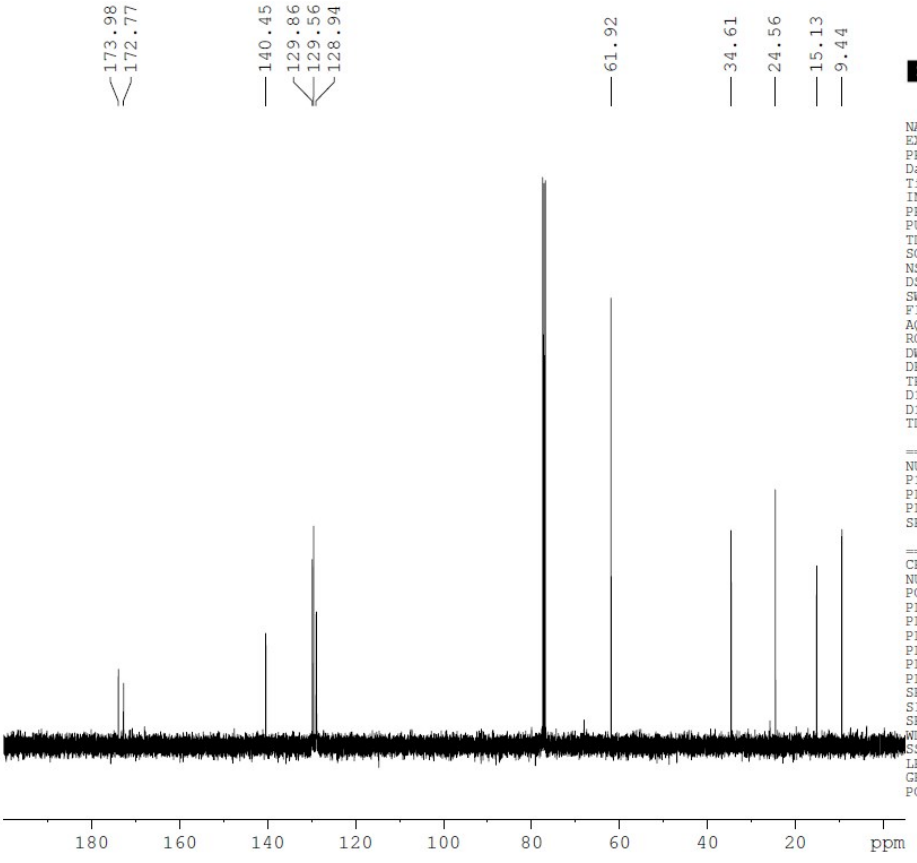

```
NAME        RX-713-5-1-1HNMR(2nd)
EXPNO                          1
PROCNO                         1
Date_                   20200117
Time                       23.50
INSTRUM                    spect
PROBHD     5 mm PABBO BB-
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                             8
DS                             0
SWH                     8223.685 Hz
FIDRES                  0.125483 Hz
AQ                      3.9846387 sec
RG                           114
DW                        60.800 usec
DE                         6.50 usec
TE                        298.3 K
D1                      1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
NUC1                          1H
P1                         9.00 usec
PL1                       -4.00 dB
PL1W                23.09303856 W
SFO1                400.2324716 MHz
SI                         32768
SF                  400.2300111 MHz
WDW                           EM
SSB                            0
LB                         0.30 Hz
GB                             0
PC                          1.00
```



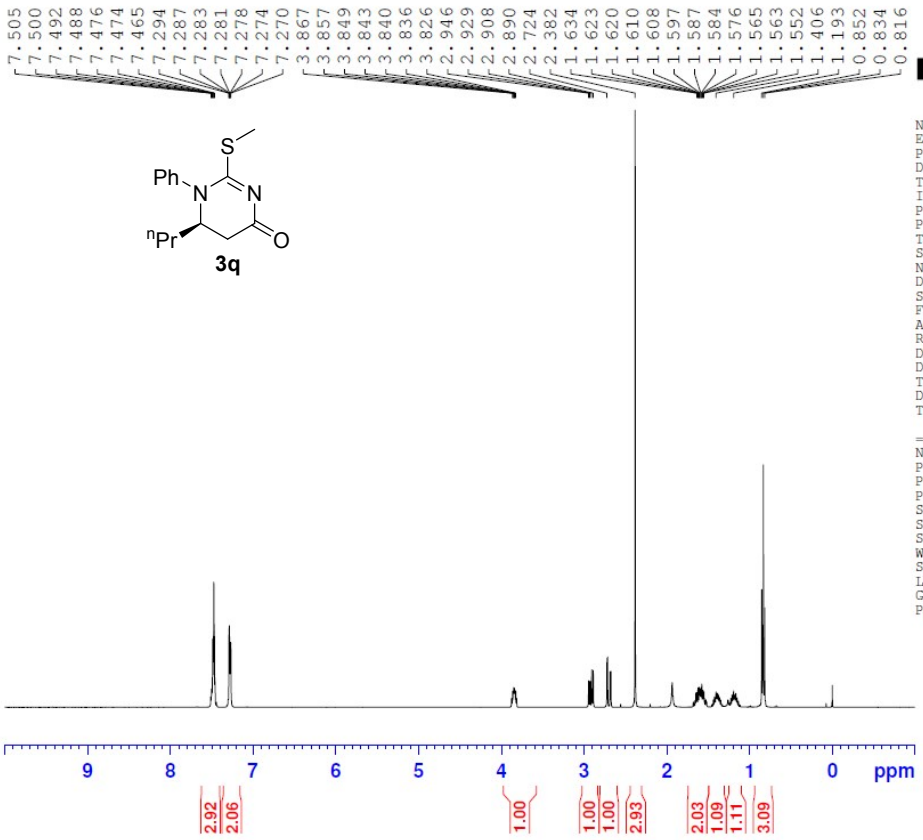

```
NAME        RX-713-5-1-13CNMR(2nd)
EXPNO                          1
PROCNO                         1
Date_                   20200118
Time                        0.02
INSTRUM                    spect
PROBHD     5 mm PABBO BB-
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                         10000
DS                             4
SWH                    25252.525 Hz
FIDRES                  0.385323 Hz
AQ                      1.2976629 sec
RG                          25.4
DW                        19.800 usec
DE                         6.50 usec
TE                        299.6 K
D1                      2.00000000 sec
D11                     0.03000000 sec
TD0                          100

======== CHANNEL f1 ========
NUC1                         13C
P1                        10.00 usec
PL1                       -2.00 dB
PL1W                59.71607590 W
SFO1                100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2                  waltz16
NUC2                          1H
PCPD2                     80.00 usec
PL2                       -4.00 dB
PL12                      14.98 dB
PL13                      13.89 dB
PL2W                23.09303856 W
PL12W                0.29206610 W
PL13W                0.37538856 W
SFO2                400.2316009 MHz
SI                         32768
SF                  100.6379040 MHz
WDW                           no
SSB                            0
LB                         0.00 Hz
GB                             0
PC                          1.40
```



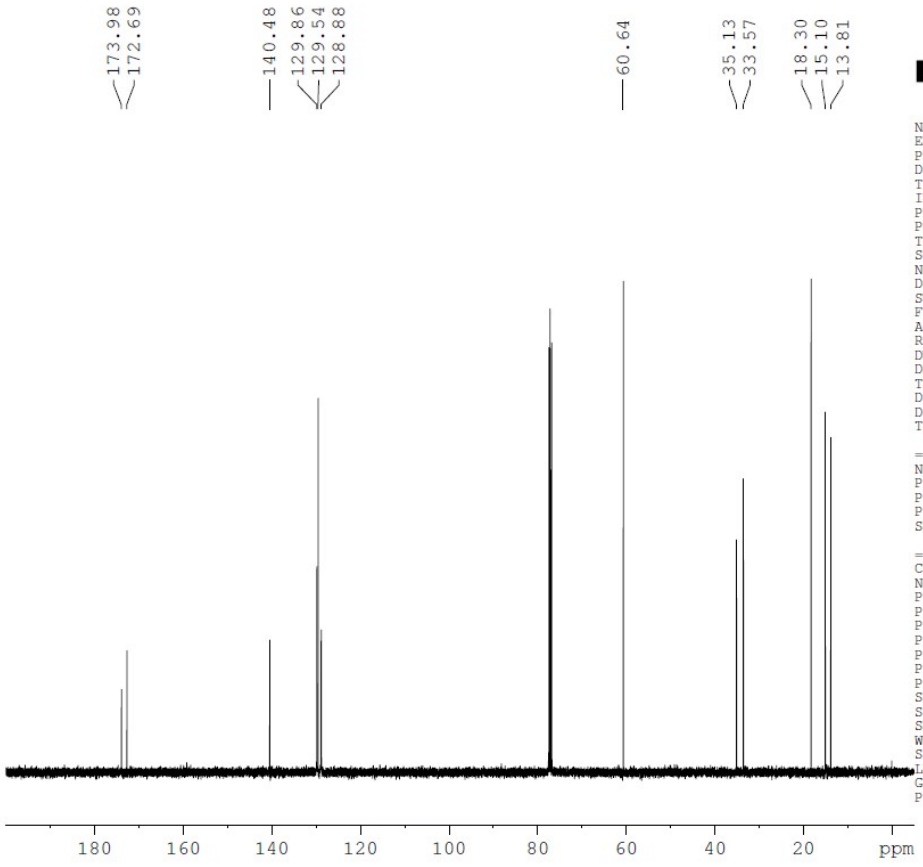

```
NAME          RX-719-3-1-HNMR
EXPNO                       1
PROCNO                      1
Date_              20200224
Time                   20.12
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                        8
DS                        0
SWH               8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                      101
DW                  60.800 usec
DE                   6.50 usec
TE                  299.3 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.00 usec
PL1                 -4.00 dB
PL1W          23.09303856 W
SFO1         400.2324716 MHz
SI                    32768
SF           400.2300123 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```



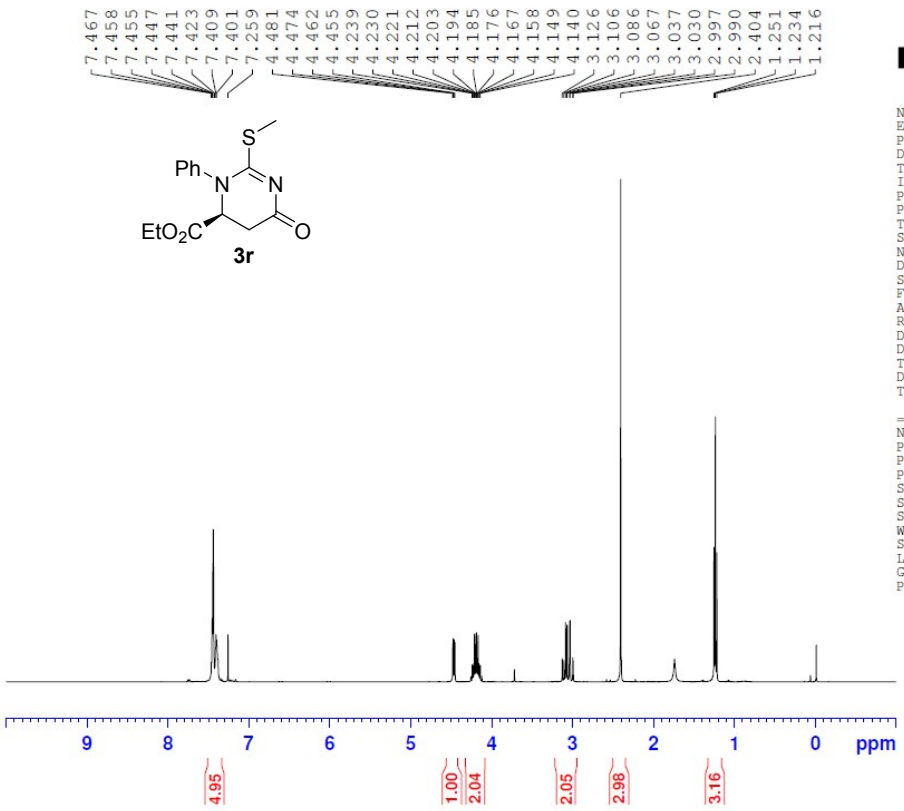

```
NAME          RX-719-3-1-CNMR
EXPNO                       1
PROCNO                      1
Date_              20200224
Time                   20.24
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDC13
NS                      261
DS                        4
SWH              25252.525 Hz
FIDRES           0.385323 Hz
AQ               1.2976629 sec
RG                     25.4
DW                  19.800 usec
DE                   6.50 usec
TE                  300.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     100

======== CHANNEL f1 ========
NUC1                   13C
P1                  10.00 usec
PL1                 -2.00 dB
PL1W          59.71607590 W
SFO1         100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                14.98 dB
PL13                13.89 dB
PL2W          23.09303856 W
PL12W          0.29206610 W
PL13W          0.37538856 W
SFO2         400.2316009 MHz
SI                    32768
SF           100.6379041 MHz
WDW                     no
SSB                      0
LB                  0.00 Hz
GB                       0
PC                    1.40
```




```
NAME        RX-719-4-1-HNMR
EXPNO                     1
PROCNO                    1
Date_              20200224
Time                 20.38
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                    80.6
DW                60.800 usec
DE                 6.50 usec
TE                299.6 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.00 usec
PL1                 -4.00 dB
PL1W           23.09303856 W
SFO1           400.2324716 MHz
SI                   32768
SF             400.2300096 MHz
WDW                     EM
SSB                      0
LB                0.30 Hz
GB                       0
PC                    1.00
```

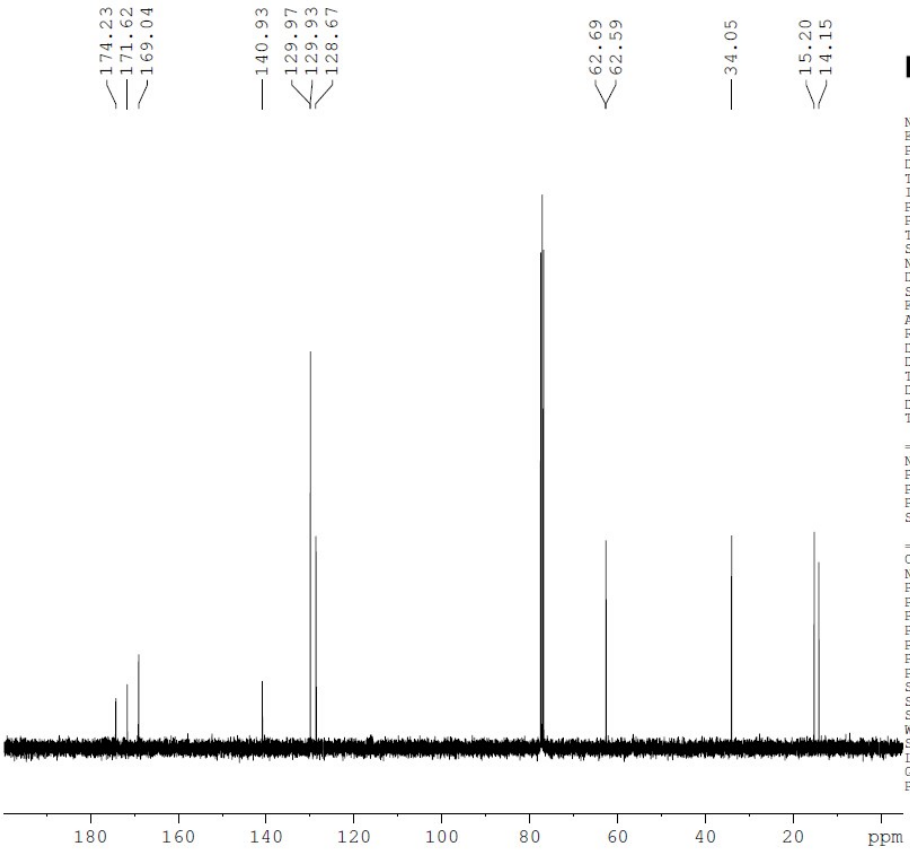



```
NAME        RX-719-4-1-CNMR
EXPNO                     1
PROCNO                    1
Date_              20200224
Time                 20.48
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     758
DS                       4
SWH            25252.525 Hz
FIDRES          0.385323 Hz
AQ              1.2976629 sec
RG                    25.4
DW                19.800 usec
DE                 6.50 usec
TE                300.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    100

======== CHANNEL f1 ========
NUC1                   13C
P1                   9.50 usec
PL1                 -2.00 dB
PL1W           59.71607590 W
SFO1           100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                 -4.00 dB
PL12                13.89 dB
PL13                13.89 dB
PL2W           23.09303856 W
PL12W           0.37538856 W
PL13W           0.37538856 W
SFO2           400.2316009 MHz
SI                   32768
SF             100.6379045 MHz
WDW                     no
SSB                      0
LB                0.00 Hz
GB                       0
PC                    1.40
```



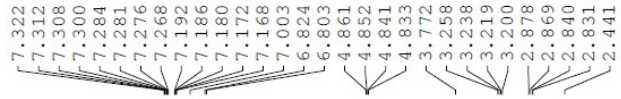
BRUKER

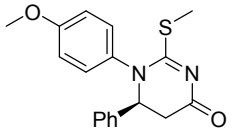
```
NAME          RX-713-4-1-HNMR
EXPNO                      1
PROCNO                     1
Date_               20200224
Time                   19.32
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDC13
NS                        8
DS                        0
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      114
DW                   60.800 usec
DE                    6.50 usec
TE                   299.2 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.00 usec
PL1                   -4.00 dB
PL1W             23.09303856 W
SFO1            400.2324716 MHz
SI                    32768
SF               400.2300208 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



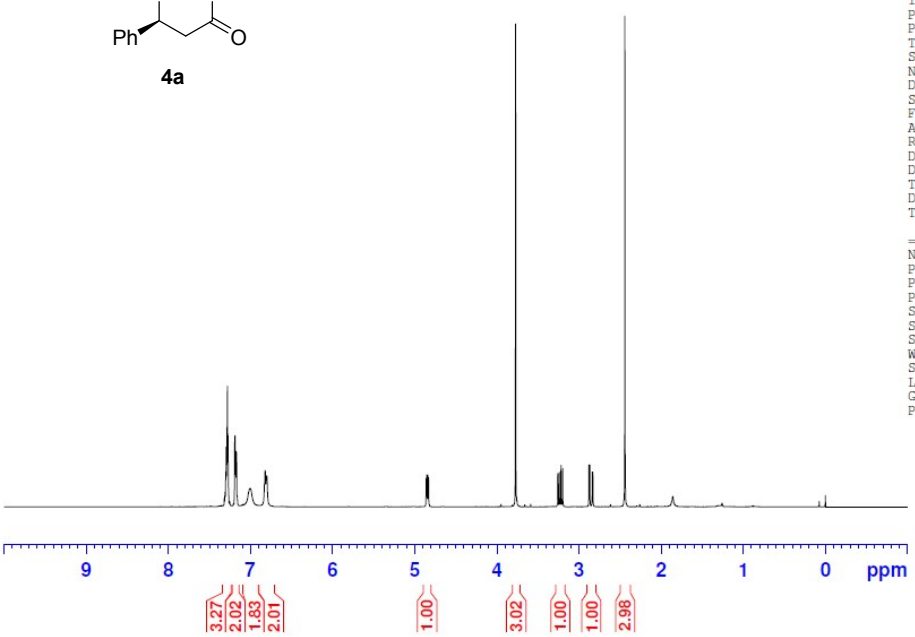
BRUKER

```
NAME          RX-713-4-1-CNMR
EXPNO                      1
PROCNO                     1
Date_               20200224
Time                   19.44
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDC13
NS                      389
DS                        4
SWH               25252.525 Hz
FIDRES            0.385323 Hz
AQ                1.2976629 sec
RG                     25.4
DW                   19.800 usec
DE                    6.50 usec
TE                   300.3 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                     100

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.00 usec
PL1                   -2.00 dB
PL1W             59.71607590 W
SFO1            100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  14.98 dB
PL13                  13.89 dB
PL2W             23.09303856 W
PL12W             0.29206610 W
PL13W             0.37538856 W
SFO2            400.2316009 MHz
SI                    32768
SF               100.6379015 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.40
```

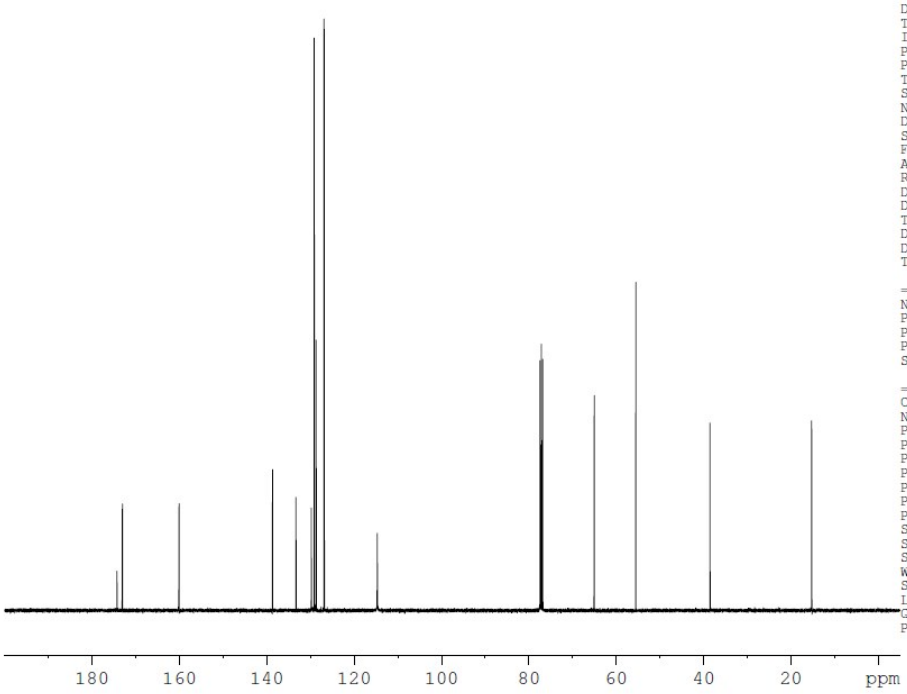




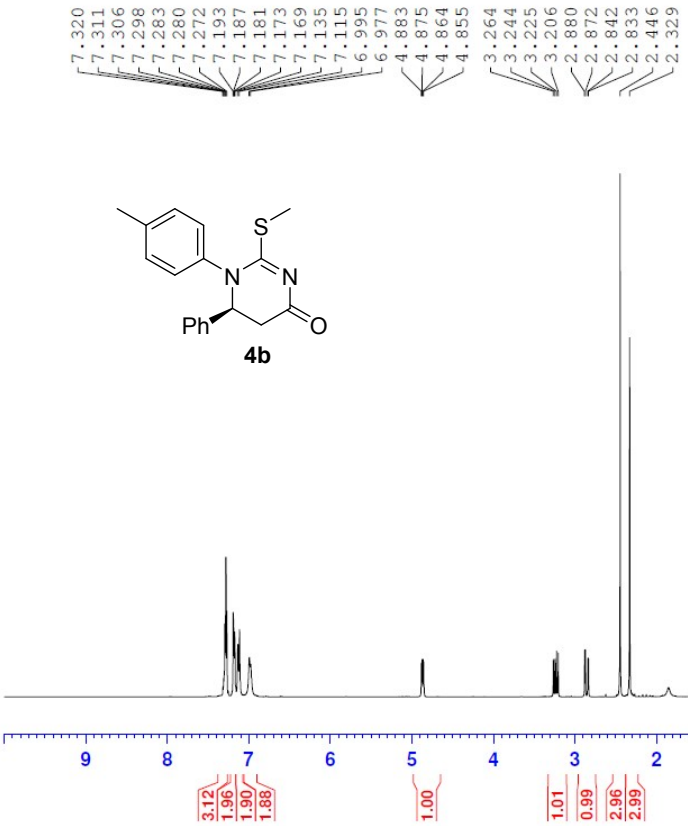
4a




```
NAME        RX-703-4-1-1HNMR
EXPNO                      1
PROCNO                     1
Date_               20191231
Time                   17.48
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      90.5
DW                   60.800 usec
DE                    6.50 usec
TE                   300.2 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.00 usec
PL1                   -4.00 dB
PL1W              23.09303856 W
SFO1              400.2324716 MHz
SI                       32768
SF                400.2300142 MHz
WDW                        EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                     1.00
```

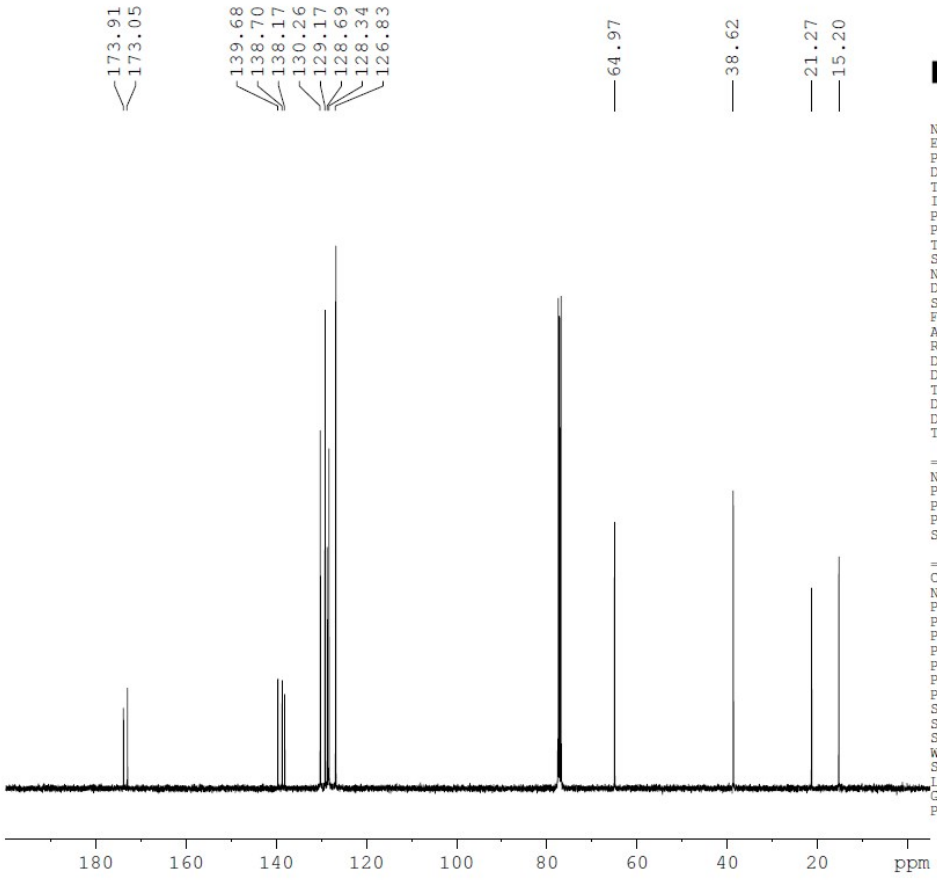



```
NAME        RX-703-4-1-13CNMR
EXPNO                      1
PROCNO                     1
Date_               20191231
Time                   17.57
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     2127
DS                        4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                       32
DW                   19.800 usec
DE                    6.50 usec
TE                   301.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                      100

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.00 usec
PL1                   -2.00 dB
PL1W              59.71607590 W
SFO1              100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  14.98 dB
PL13                  13.89 dB
PL2W              23.09303856 W
PL12W              0.29206610 W
PL13W              0.37538856 W
SFO2              400.2316009 MHz
SI                       32768
SF                100.6379054 MHz
WDW                       no
SSB                        0
LB                     0.00 Hz
GB                         0
PC                     1.40
```



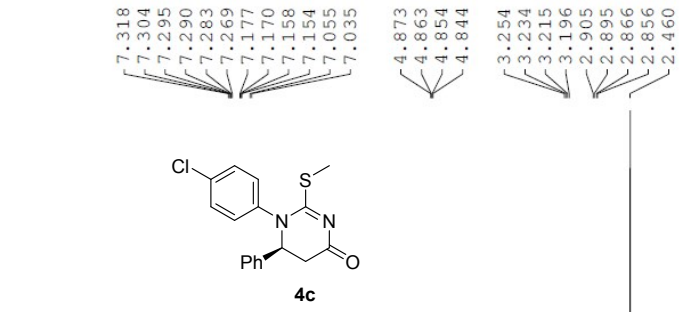
BRUKER

```
NAME          RX-699-3-1-1HNMR
EXPNO                        1
PROCNO                       1
Date_                 20191220
Time                     15.32
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9846387 sec
RG                         101
DW                      60.800 usec
DE                       6.50 usec
TE                      299.4 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       9.00 usec
PL1                     -4.00 dB
PL1W                23.09303856 W
SFO1                400.2324716 MHz
SI                       32768
SF                  400.2300159 MHz
WDW                         EM
SSB                          0
LB                       0.30 Hz
GB                           0
PC                       1.00
```

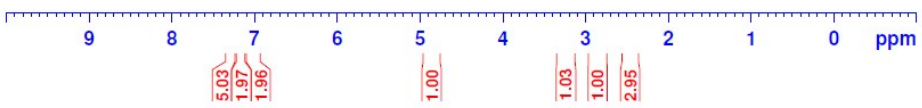


BRUKER

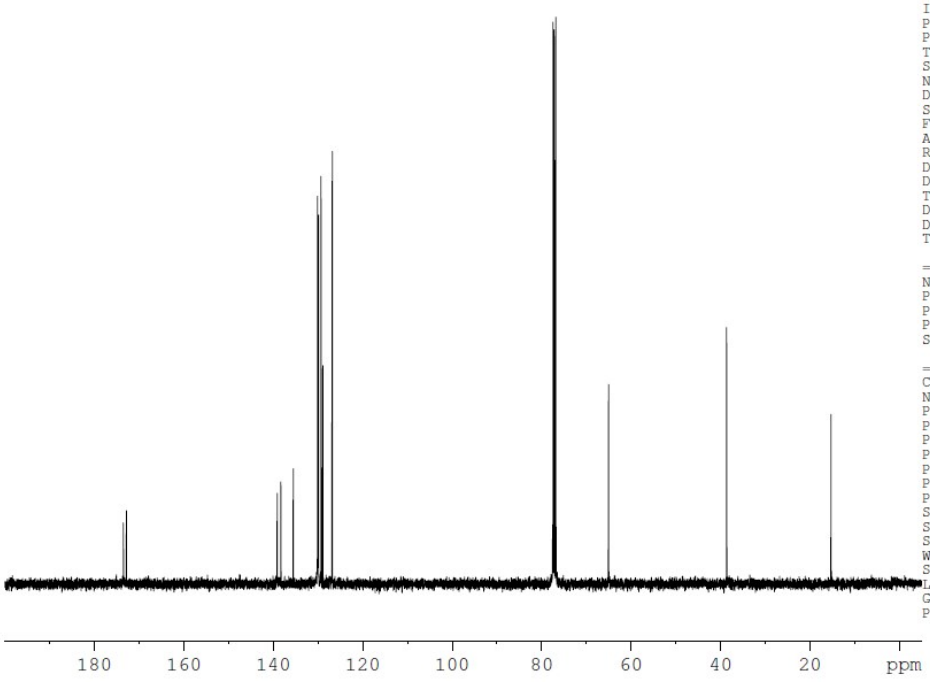
```
NAME          RX-699-3-1-13CNMR
EXPNO                        1
PROCNO                       1
Date_                 20191220
Time                     15.42
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         818
DS                           4
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976629 sec
RG                          32
DW                      19.800 usec
DE                       6.50 usec
TE                      300.5 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                        100

======== CHANNEL f1 ========
NUC1                        13C
P1                      10.00 usec
PL1                     -2.00 dB
PL1W                59.71607590 W
SFO1                100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                     -4.00 dB
PL12                    14.98 dB
PL13                    13.89 dB
PL2W                23.09303856 W
PL12W                0.29206610 W
PL13W                0.37538856 W
SFO2                400.2316009 MHz
SI                       32768
SF                  100.6379046 MHz
WDW                         EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                       1.40
```



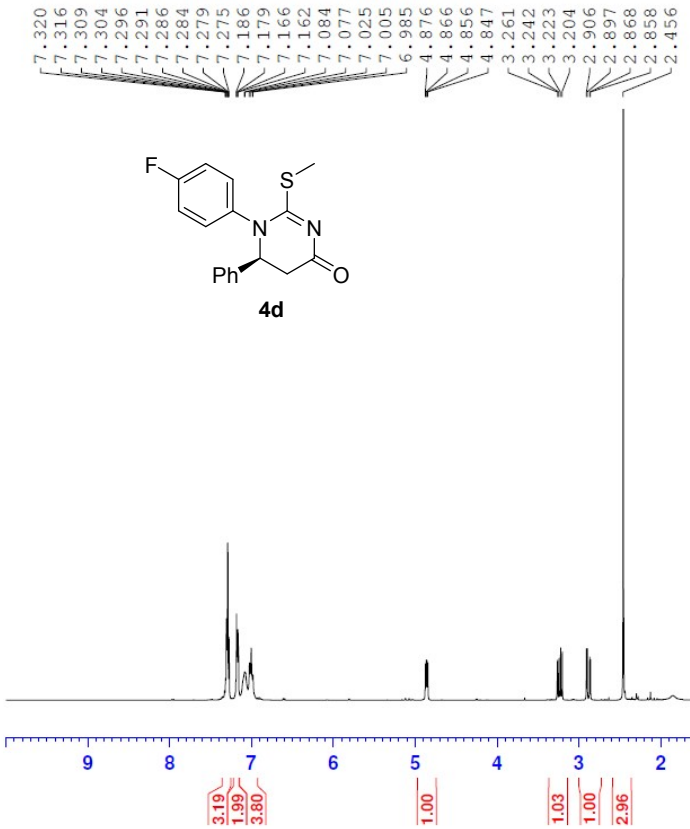
4c




```
NAME          RX-682-4-1-1HNMR
EXPNO                        1
PROCNO                       1
Date_             20191220
Time                     14.31
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                         144
DW                    60.800 usec
DE                     6.50 usec
TE                     299.6 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       1H
P1                       9.00 usec
PL1                     -4.00 dB
PL1W              23.09303856 W
SFO1              400.2324716 MHz
SI                       32768
SF                400.2300169 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                        1.00
```

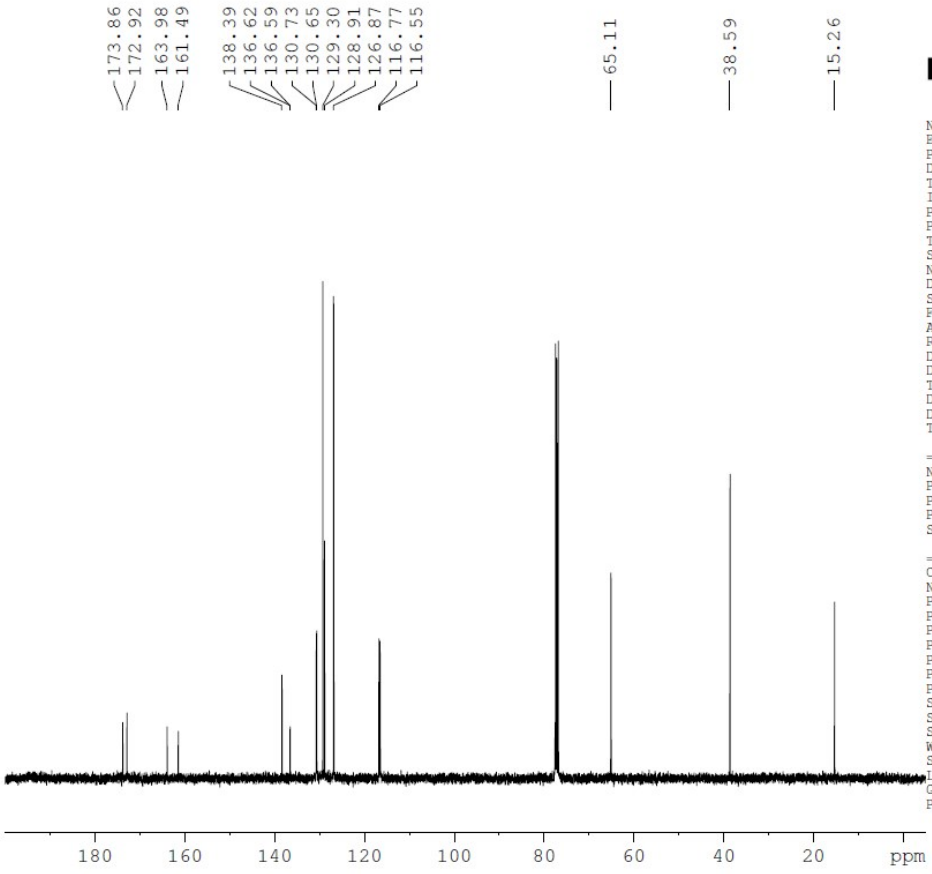



```
NAME         RX-682-4-1-13CNMR
EXPNO                        1
PROCNO                       1
Date_             20191220
Time                     14.41
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         379
DS                           4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976629 sec
RG                          32
DW                    19.800 usec
DE                     6.50 usec
TE                     300.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        100

======== CHANNEL f1 ========
NUC1                       13C
P1                      10.00 usec
PL1                     -2.00 dB
PL1W              59.71607590 W
SFO1              100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   80.00 usec
PL2                     -4.00 dB
PL12                    14.98 dB
PL13                    13.89 dB
PL2W              23.09303856 W
PL12W              0.29206610 W
PL13W              0.37538856 W
SFO2              400.2316009 MHz
SI                       32768
SF                100.6379031 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        1.40
```



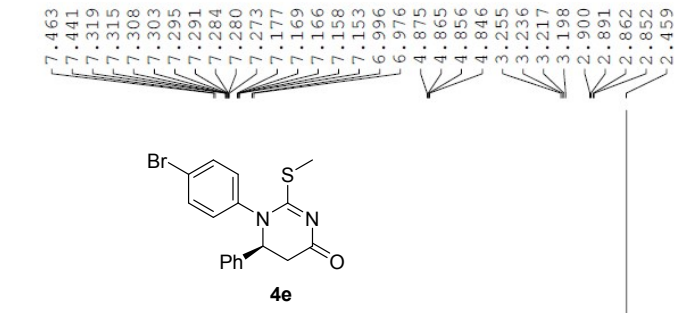
BRUKER

```
NAME          RX-703-3-1-1HNMR
EXPNO                        1
PROCNO                       1
Date_                 20191231
Time                     17.09
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDC13
NS                          8
DS                          0
SWH                  8223.685 Hz
FIDRES               0.125483 Hz
AQ                   3.9846387 sec
RG                        101
DW                     60.800 usec
DE                      6.50 usec
TE                     299.4 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       9.00 usec
PL1                     -4.00 dB
PL1W               23.09303856 W
SFO1               400.2324716 MHz
SI                       32768
SF                 400.2300144 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                        1.00
```



BRUKER

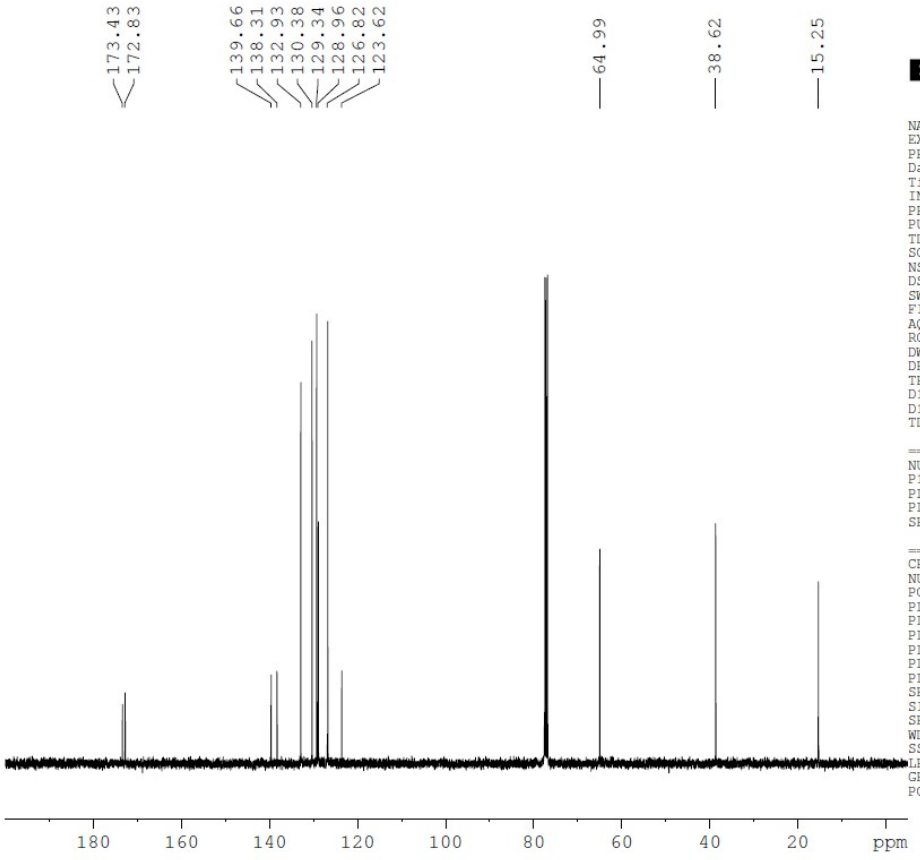
```
NAME         RX-703-3-1-13CNMR
EXPNO                        1
PROCNO                       1
Date_                 20191231
Time                     17.27
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         300
DS                           4
SWH                25252.525 Hz
FIDRES               0.385323 Hz
AQ                   1.2976629 sec
RG                        22.6
DW                     19.800 usec
DE                      6.50 usec
TE                     299.8 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                        100

======== CHANNEL f1 ========
NUC1                        13C
P1                      10.00 usec
PL1                     -2.00 dB
PL1W               59.71607590 W
SFO1               100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                     -4.00 dB
PL12                    14.98 dB
PL13                    13.89 dB
PL2W               23.09303856 W
PL12W               0.29206610 W
PL13W               0.37538856 W
SFO2               400.2316009 MHz
SI                       32768
SF                 100.6379044 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        1.40
```



4e

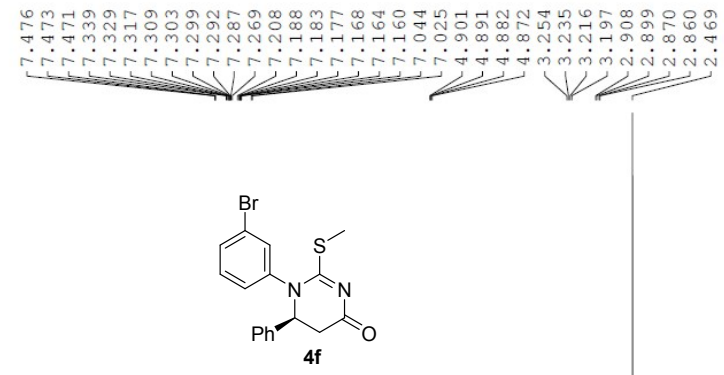
BRUKER

```
NAME          RX-701-2-1-1HNMR
EXPNO                        1
PROCNO                       1
Date_             20191220
Time                     20.03
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDC13
NS                           8
DS                           0
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                         128
DW                    60.800 usec
DE                     6.50 usec
TE                     299.3 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       9.00 usec
PL1                     -4.00 dB
PL1W             23.09303856 W
SFO1             400.2324716 MHz
SI                       32768
SF                400.2300153 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                        1.00
```



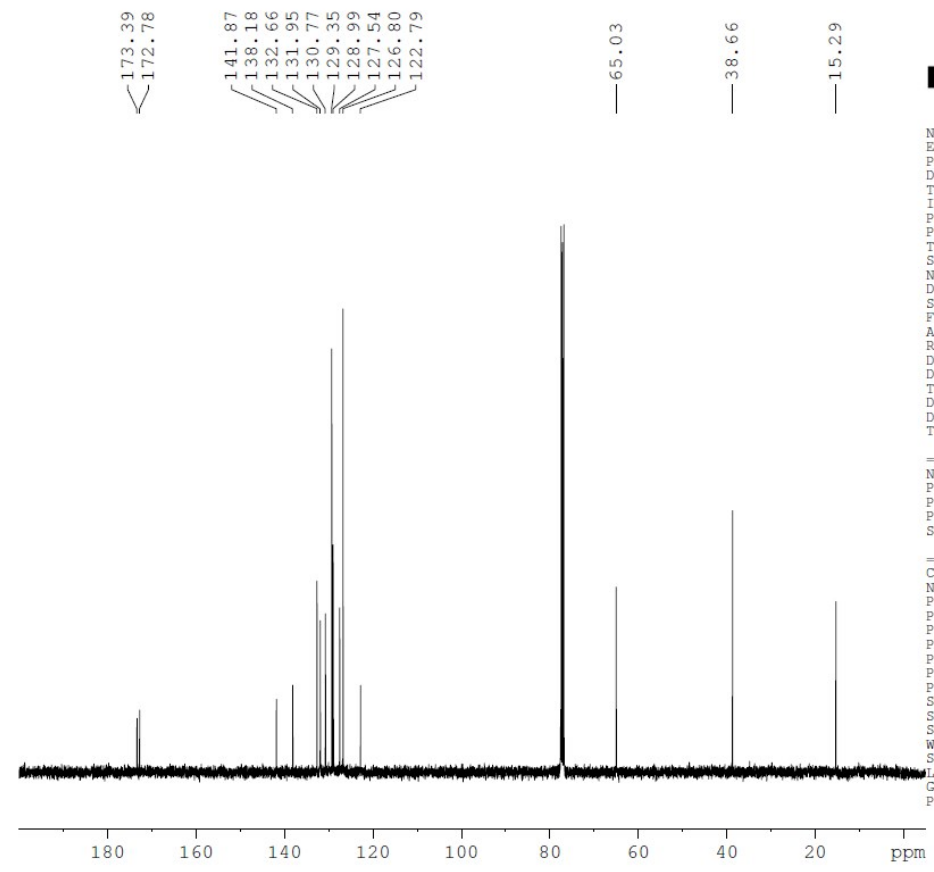
BRUKER

```
NAME          RX-701-2-1-13CNMR
EXPNO                        1
PROCNO                       1
Date_             20191220
Time                     20.11
INSTRUM                  spect
PROBHD     5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDC13
NS                         326
DS                           4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976629 sec
RG                          32
DW                    19.800 usec
DE                     6.50 usec
TE                     300.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        100

======== CHANNEL f1 ========
NUC1                       13C
P1                      10.00 usec
PL1                     -2.00 dB
PL1W             59.71607590 W
SFO1             100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                   80.00 usec
PL2                     -4.00 dB
PL12                    14.98 dB
PL13                    13.89 dB
PL2W             23.09303856 W
PL12W             0.29206610 W
PL13W             0.37538856 W
SFO2             400.2316009 MHz
SI                       32768
SF                100.6379043 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                        1.40
```

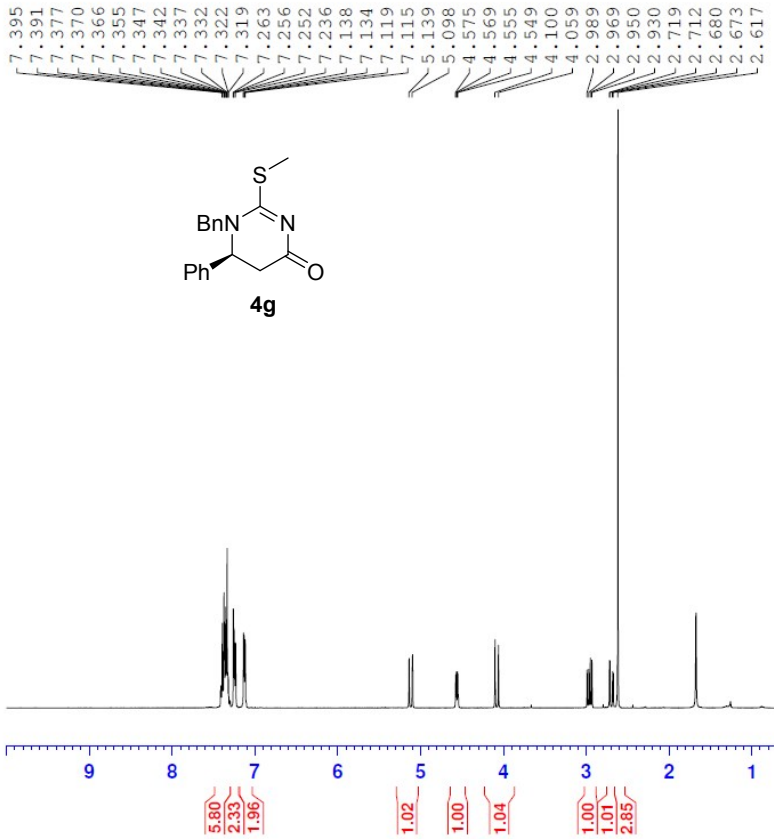


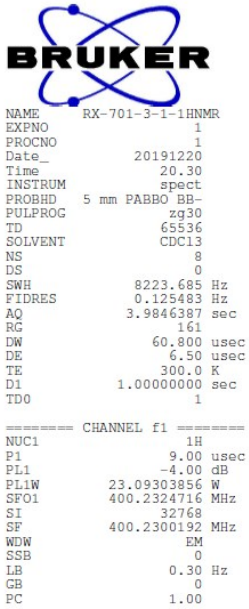
4f


```
NAME        RX-699-1-1-1HNMR
EXPNO                      1
PROCNO                     1
Date_               20191220
Time                   15.01
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      144
DW                   60.800 usec
DE                    6.50 usec
TE                   299.9 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.00 usec
PL1                   -4.00 dB
PL1W           23.09303856 W
SFO1           400.2324716 MHz
SI                    32768
SF             400.2300168 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

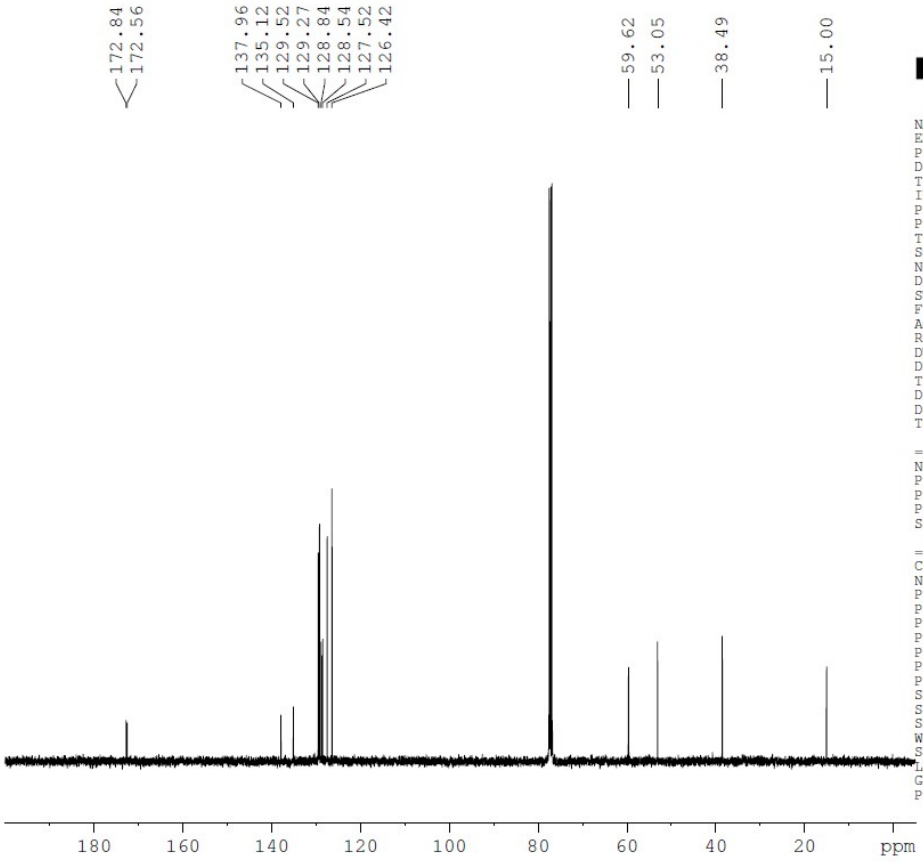



```
NAME        RX-699-1-1-13CNMR
EXPNO                      1
PROCNO                     1
Date_               20191220
Time                   15.11
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      300
DS                        4
SWH            25252.525 Hz
FIDRES           0.385323 Hz
AQ               1.2976629 sec
RG                       32
DW                   19.800 usec
DE                    6.50 usec
TE                   300.6 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                     100

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.00 usec
PL1                   -2.00 dB
PL1W           59.71607590 W
SFO1           100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  14.98 dB
PL13                  13.89 dB
PL2W           23.09303856 W
PL12W           0.29206610 W
PL13W           0.37538856 W
SFO2           400.2316009 MHz
SI                    32768
SF             100.6379025 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

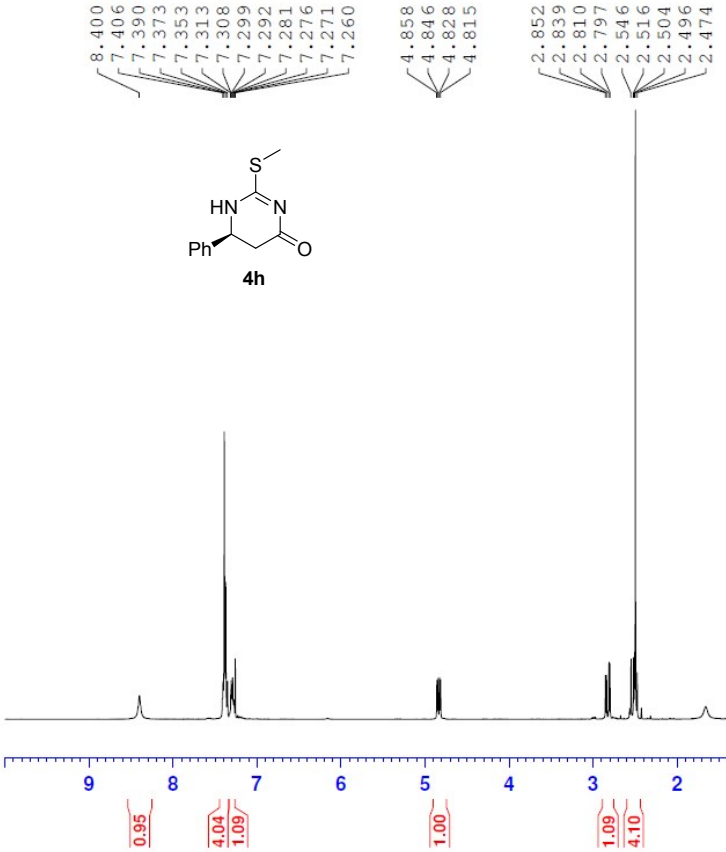




```
NAME          RX-701-3-1-1HNMR
EXPNO                        1
PROCNO                       1
Date_             20191220
Time                     20.30
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                  zg30
TD                      65536
SOLVENT                 CDC13
NS                           8
DS                           0
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                         161
DW                    60.800 usec
DE                     6.50 usec
TE                     300.0 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                        9.00 usec
PL1                      -4.00 dB
PL1W              23.09303856 W
SFO1              400.2324716 MHz
SI                       32768
SF                400.2300192 MHz
WDW                         EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```

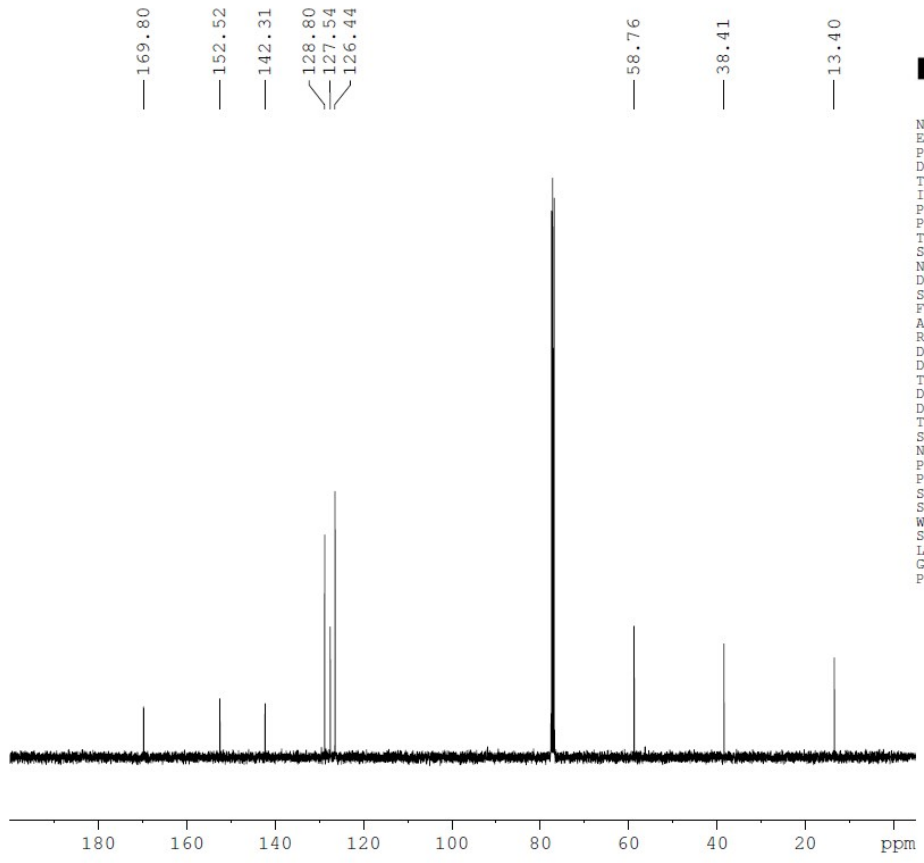


```
NAME         RX-701-3-1-13CNMR
EXPNO                        1
PROCNO                       1
Date_             20191220
Time                     20.39
INSTRUM                  spect
PROBHD    5 mm PABBO BB-
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDC13
NS                         366
DS                           4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976629 sec
RG                          36
DW                    19.800 usec
DE                     6.50 usec
TE                     300.8 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        100

======== CHANNEL f1 ========
NUC1                       13C
P1                       10.00 usec
PL1                      -2.00 dB
PL1W              59.71607590 W
SFO1              100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    80.00 usec
PL2                      -4.00 dB
PL12                     14.98 dB
PL13                     13.89 dB
PL2W              23.09303856 W
PL12W              0.29206610 W
PL13W              0.37538856 W
SFO2              400.2316009 MHz
SI                       32768
SF                100.6379012 MHz
WDW                         EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```



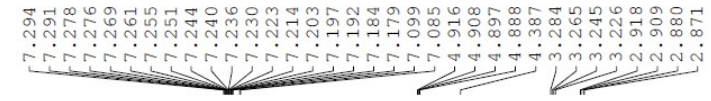
BRUKER

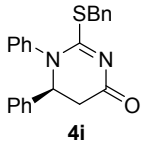
```
NAME          RX-777-10-3-1H
EXPNO                      3
PROCNO                     1
Date_               20200901
Time                  21.05 h
INSTRUM                spect
PROBHD    Z108618_0239 (
PULPROG                 zg30
TD                     65536
SOLVENT                CDC13
NS                         8
DS                         0
SWH                 8223.685 Hz
FIDRES              0.250967 Hz
AQ                  3.9846387 sec
RG                       144
DW                    60.800 usec
DE                     6.50 usec
TE                    298.1 K
D1                 1.00000000 sec
TD0                        1
SFO1              400.1324710 MHz
NUC1                       1H
P0                     5.00 usec
P1                    15.00 usec
SI                     32768
SF                400.1300101 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.00
```



BRUKER

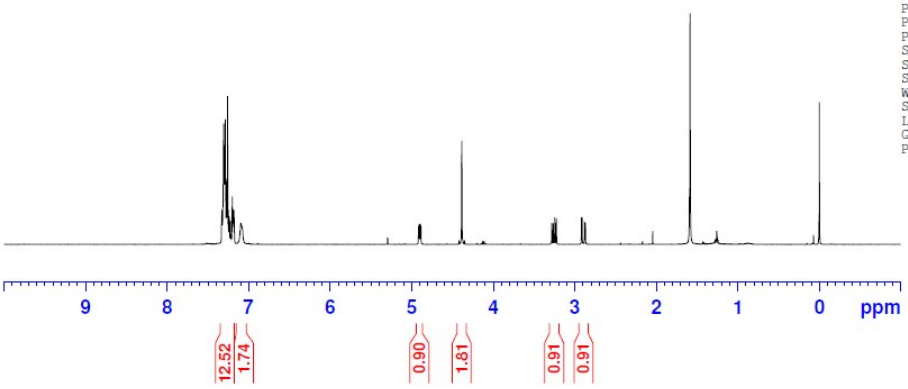
```
NAME          RX-777-10-3-13C
EXPNO                      4
PROCNO                     1
Date_               20200901
Time                  21.28 h
INSTRUM                spect
PROBHD    Z108618_0239 (
PULPROG              zgpg30
TD                     65536
SOLVENT                CDC13
NS                       312
DS                         0
SWH                25252.525 Hz
FIDRES              0.770646 Hz
AQ                  1.2976629 sec
RG                       144
DW                    19.800 usec
DE                     6.50 usec
TE                    298.7 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                      100
SFO1              100.6248425 MHz
NUC1                       13C
P0                     3.33 usec
P1                    10.00 usec
SI                     32768
SF                100.6127555 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                      1.40
```

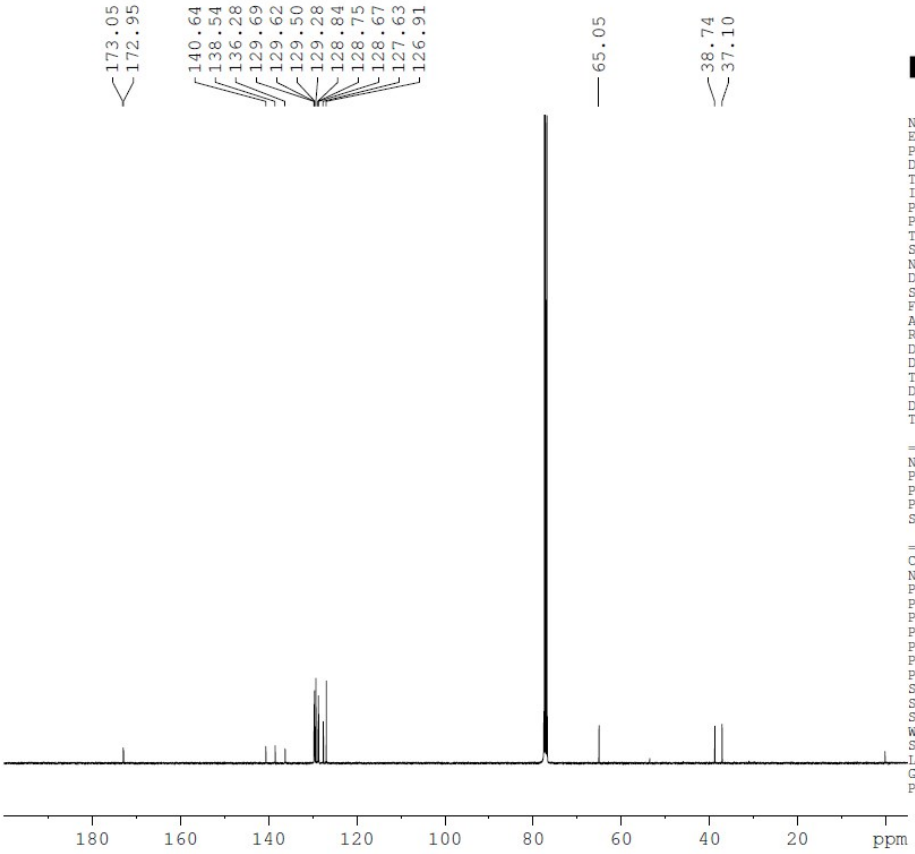




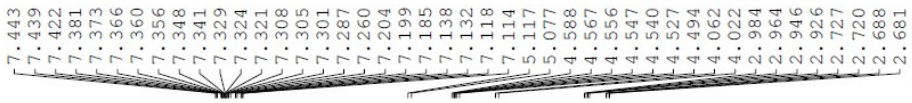
4i

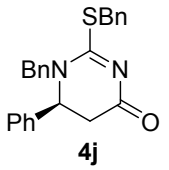
```
NAME          RX-717-1-1-HNMR
EXPNO                       1
PROCNO                      1
Date_                20200219
Time                    23.20
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDC13
NS                         8
DS                         0
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                3.9846387 sec
RG                       203
DW                    60.800 usec
DE                     6.50 usec
TE                    299.1 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.00 usec
PL1                   -4.00 dB
PL1W             23.09303856 W
SFO1            400.2324716 MHz
SI                     32768
SF              400.2300203 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



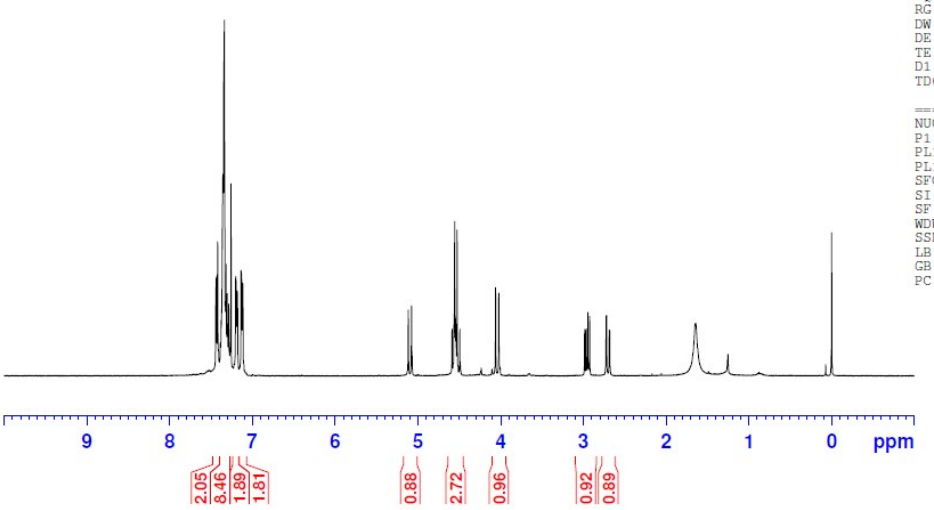


```
NAME          RX-717-1-1-CNMR
EXPNO                       1
PROCNO                      1
Date_                20200219
Time                    23.30
INSTRUM                 spect
PROBHD   5 mm PABBO BB-
PULPROG              zgpg30
TD                     65536
SOLVENT                CDC13
NS                     10000
DS                         4
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                1.2976629 sec
RG                      22.6
DW                    19.800 usec
DE                     6.50 usec
TE                    300.1 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      100

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.00 usec
PL1                   -2.00 dB
PL1W             59.71607590 W
SFO1            100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -4.00 dB
PL12                  14.98 dB
PL13                  13.89 dB
PL2W             23.09303856 W
PL12W             0.29206610 W
PL13W             0.37538856 W
SFO2            400.2316009 MHz
SI                     32768
SF              100.6378991 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

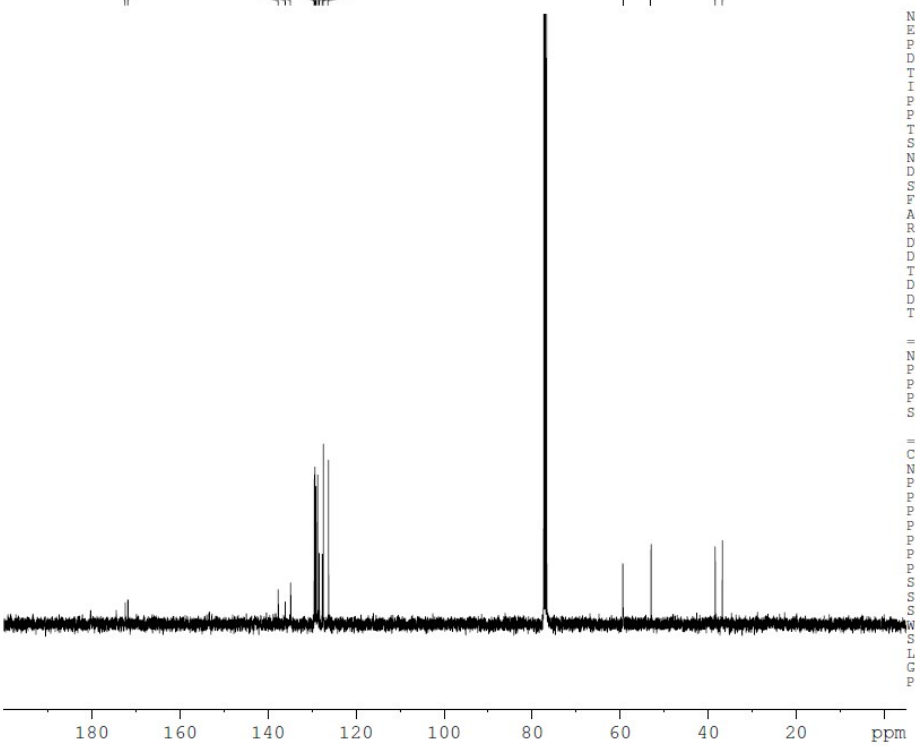




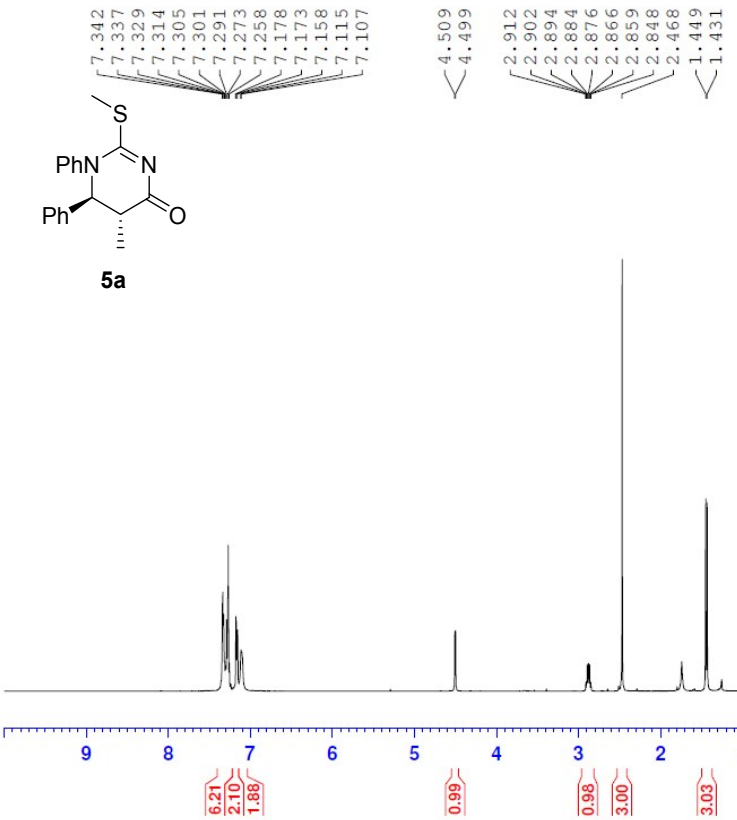
4j




```
NAME        RX-728-2-1HNMR
EXPNO                    1
PROCNO                   1
Date_             20200316
Time                 10.54
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH             8223.685 Hz
FIDRES          0.125483 Hz
AQ              3.9846387 sec
RG                     161
DW                60.800 usec
DE                 6.50 usec
TE                297.2 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   9.00 usec
PL1                 -4.00 dB
PL1W          23.09303856 W
SFO1          400.2324716 MHz
SI                   32768
SF            400.2300206 MHz
WDW                     EM
SSB                      0
LB                0.30 Hz
GB                       0
PC                1.00
```



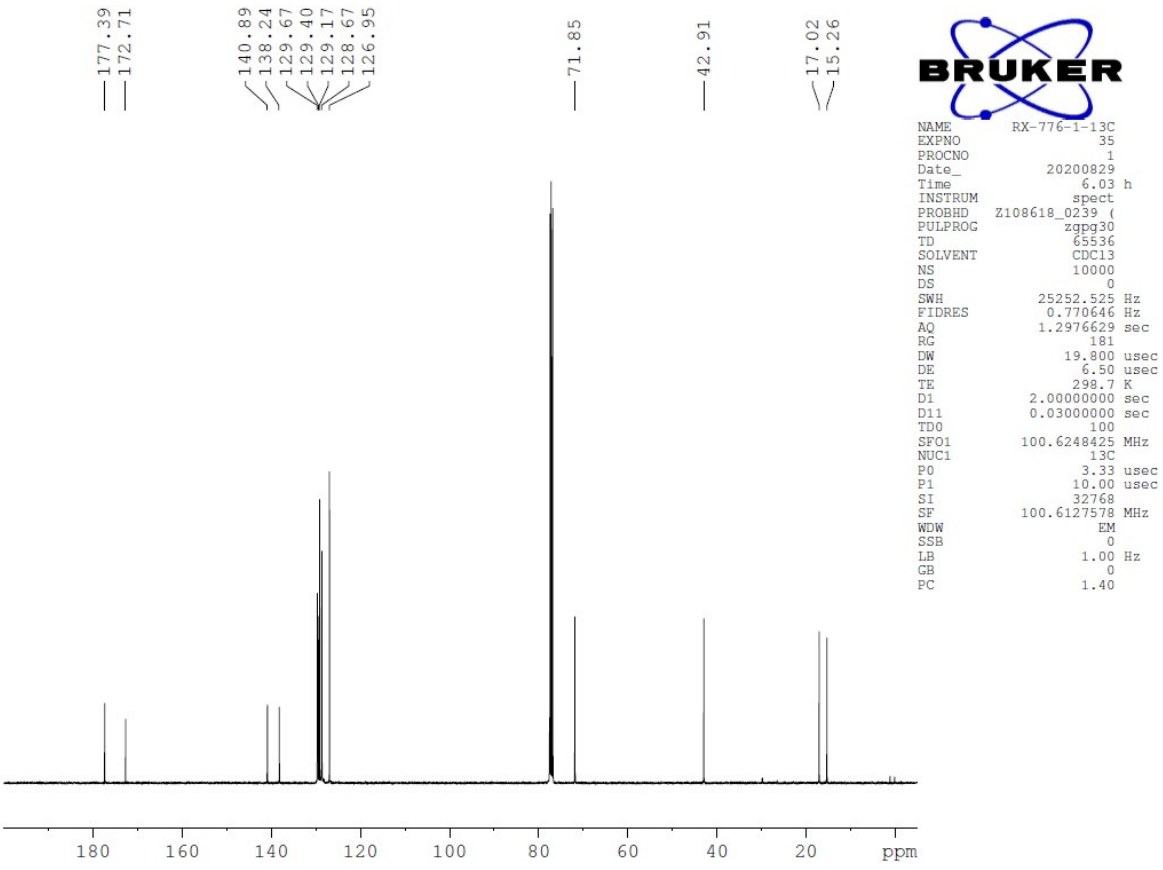

```
NAME       RX-728-2-13CNMR
EXPNO                    1
PROCNO                   1
Date_             20200316
Time                 11.07
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     442
DS                       4
SWH            25252.525 Hz
FIDRES          0.385323 Hz
AQ              1.2976629 sec
RG                    25.4
DW                19.800 usec
DE                 6.50 usec
TE                298.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    100

======== CHANNEL f1 ========
NUC1                   13C
P1                  10.00 usec
PL1                 -2.00 dB
PL1W          59.71607590 W
SFO1          100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                 -4.00 dB
PL12                14.98 dB
PL13                13.89 dB
PL2W          23.09303856 W
PL12W          0.29206610 W
PL13W          0.37538856 W
SFO2          400.2316009 MHz
SI                   32768
SF            100.6379140 MHz
WDW                     EM
SSB                      0
LB                1.00 Hz
GB                       0
PC                1.40
```



5a

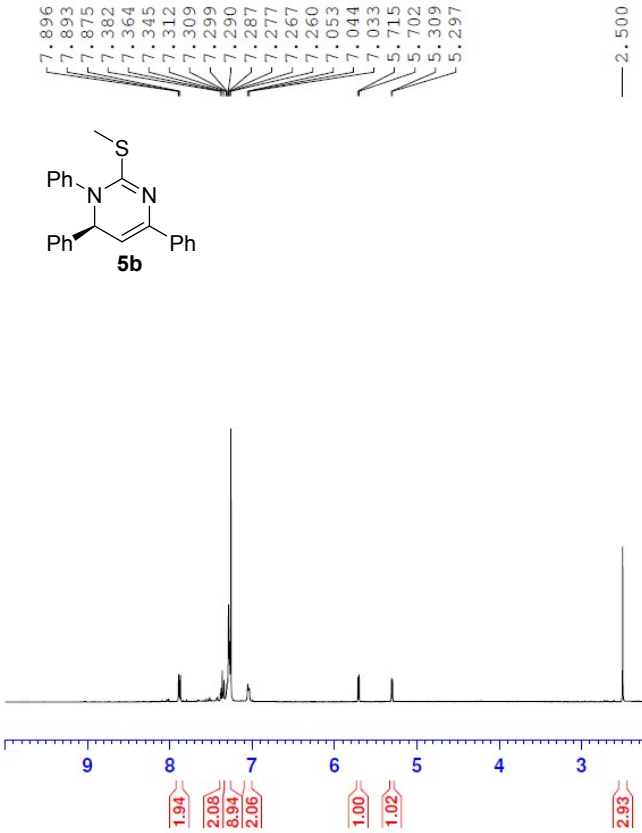
BRUKER

```
NAME            RX-776-1-1H
EXPNO                    34
PROCNO                    1
Date_              20200828
Time                 20.35 h
INSTRUM                spect
PROBHD    Z108618_0239 (
PULPROG                zg30
TD                    65536
SOLVENT             CDC13
NS                        8
DS                        0
SWH               8223.685 Hz
FIDRES            0.250967 Hz
AQ                3.9846387 sec
RG                      128
DW                 60.800 usec
DE                  6.50 usec
TE                  298.4 K
D1              1.00000000 sec
TD0                       1
SFO1           400.1324710 MHz
NUC1                     1H
P0                  5.00 usec
P1                 15.00 usec
SI                    32768
SF             400.1300066 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                     1.00
```

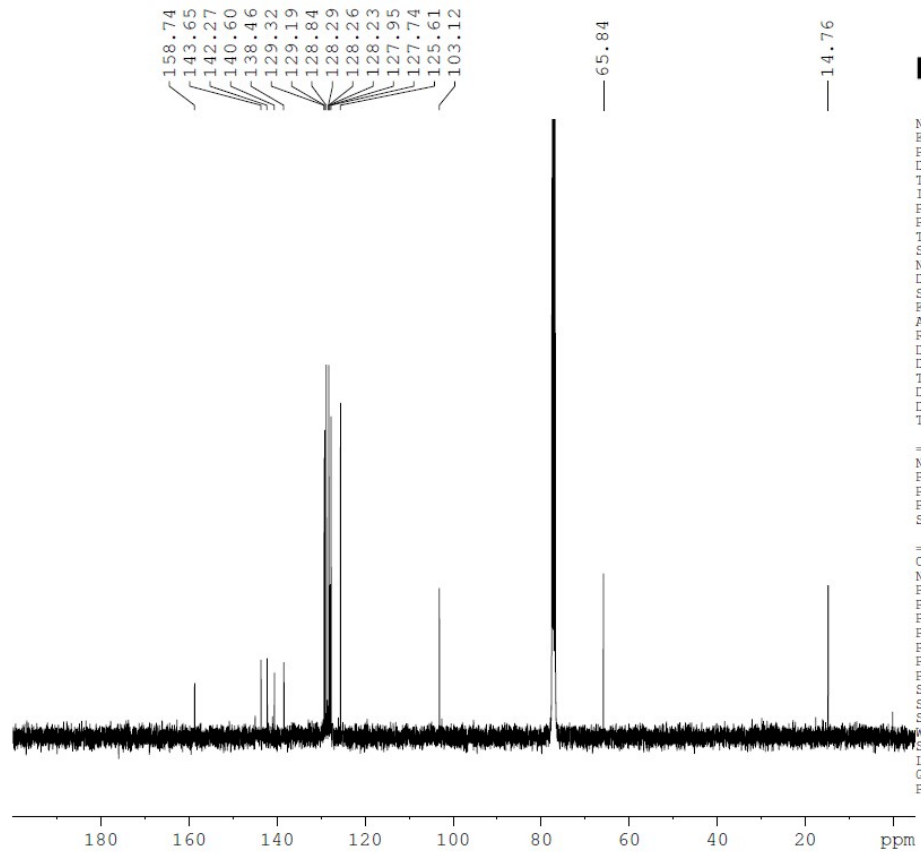


BRUKER

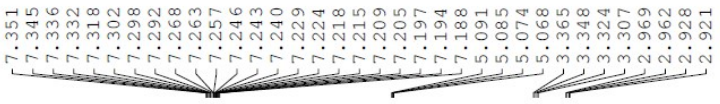
```
NAME            RX-776-1-13C
EXPNO                     35
PROCNO                     1
Date_               20200829
Time                  6.03 h
INSTRUM                spect
PROBHD    Z108618_0239 (
PULPROG             zgpg30
TD                    65536
SOLVENT             CDC13
NS                    10000
DS                        0
SWH              25252.525 Hz
FIDRES            0.770646 Hz
AQ                1.2976629 sec
RG                      181
DW                 19.800 usec
DE                  6.50 usec
TE                  298.7 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     100
SFO1           100.6248425 MHz
NUC1                    13C
P0                  3.33 usec
P1                 10.00 usec
SI                    32768
SF             100.6127578 MHz
WDW                      EM
SSB                       0
LB                   1.00 Hz
GB                        0
PC                     1.40
```

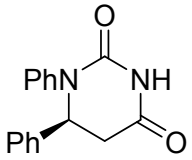


```
NAME          RX-776-2-1H(2nd)
EXPNO                       1
PROCNO                      1
Date_              20200922
Time                   20.59
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        64
DS                         0
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                       144
DW                  60.800 usec
DE                   6.50 usec
TE                   298.3 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     7.50 usec
PL1                   -3.38 dB
PL1W           20.00124741 W
SFO1           400.2324716 MHz
SI                     32768
SF             400.2300202 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

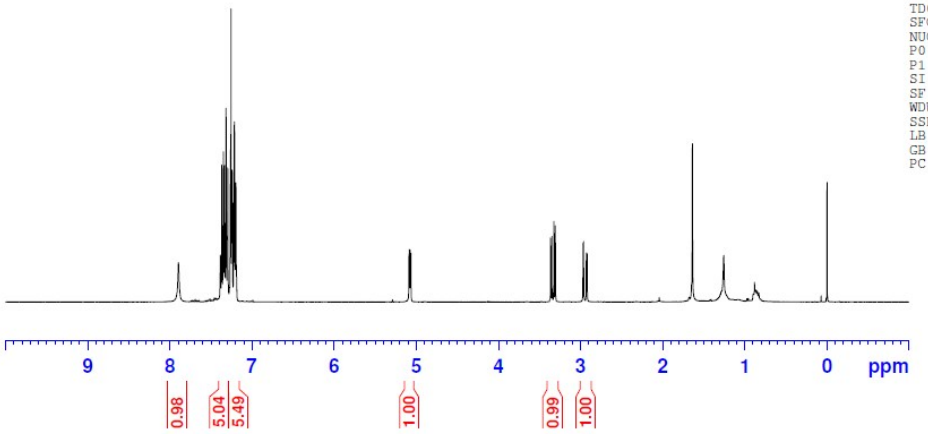


```
NAME          RX-776-2-13C(2nd)
EXPNO                       1
PROCNO                      1
Date_              20200922
Time                   21.10
INSTRUM                spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                     10000
DS                         4
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                       114
DW                  19.800 usec
DE                   6.50 usec
TE                   299.3 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      100

======== CHANNEL f1 ========
NUC1                     13C
P1                    10.00 usec
PL1                   -2.69 dB
PL1W           69.99890900 W
SFO1           100.6499905 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                 80.00 usec
PL2                   -3.38 dB
PL12                  17.18 dB
PL13                  13.89 dB
PL2W           20.02078438 W
PL12W           0.17598718 W
PL13W           0.37538856 W
SFO2           400.2316009 MHz
SI                     32768
SF             100.6379010 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                      1.40
```

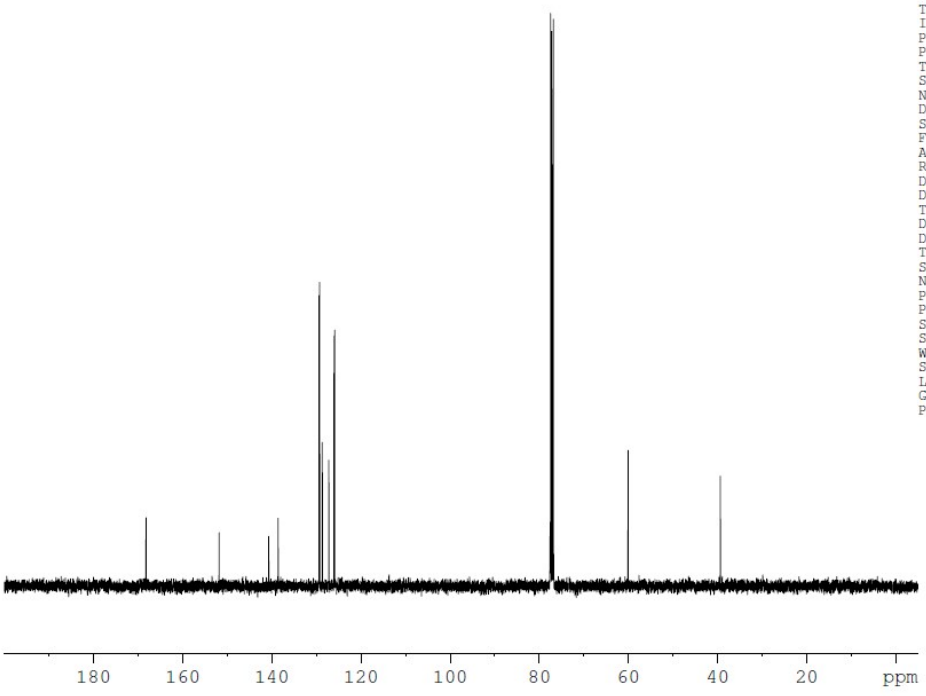




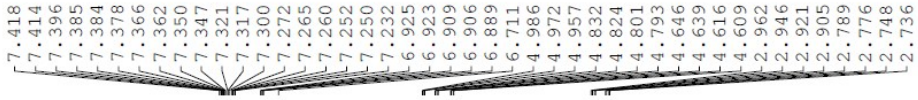
5c

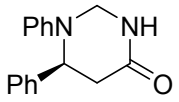



```
NAME        RX-789-2-1H(2nd)
EXPNO                     6
PROCNO                    1
Date_              20201009
Time                 21.11 h
INSTRUM              spect
PROBHD     Z108618_0238 (
PULPROG              zg30
TD                   65536
SOLVENT              CDC13
NS                       8
DS                       0
SWH             8223.685 Hz
FIDRES          0.250967 Hz
AQ              3.9846387 sec
RG                     161
DW                60.800 usec
DE                 6.50 usec
TE                298.4 K
D1             1.00000000 sec
TD0                      1
SFO1          400.1324710 MHz
NUC1                   1H
P0                 5.00 usec
P1                15.00 usec
SI                   32768
SF            400.1300108 MHz
WDW                    EM
SSB                      0
LB                 0.30 Hz
GB                       0
PC                 1.00
```



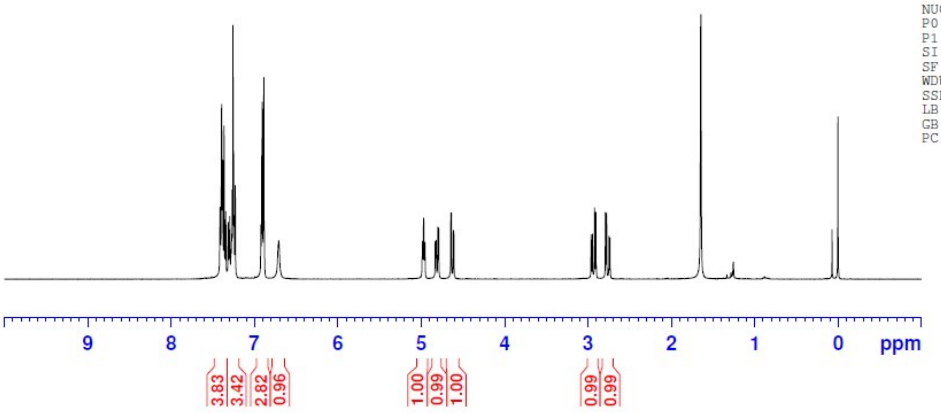

```
NAME        RX-789-2-13C(2nd)
EXPNO                     7
PROCNO                    1
Date_              20201009
Time                 21.28 h
INSTRUM              spect
PROBHD     Z108618_0238 (
PULPROG            zgpg30
TD                   65536
SOLVENT              CDC13
NS                     239
DS                       0
SWH            25252.525 Hz
FIDRES          0.770646 Hz
AQ              1.2976629 sec
RG                     144
DW                19.800 usec
DE                 6.50 usec
TE                298.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    100
SFO1          100.6248425 MHz
NUC1                   13C
P0                 3.33 usec
P1                10.00 usec
SI                   32768
SF            100.6127555 MHz
WDW                    EM
SSB                      0
LB                 1.00 Hz
GB                       0
PC                 1.40
```



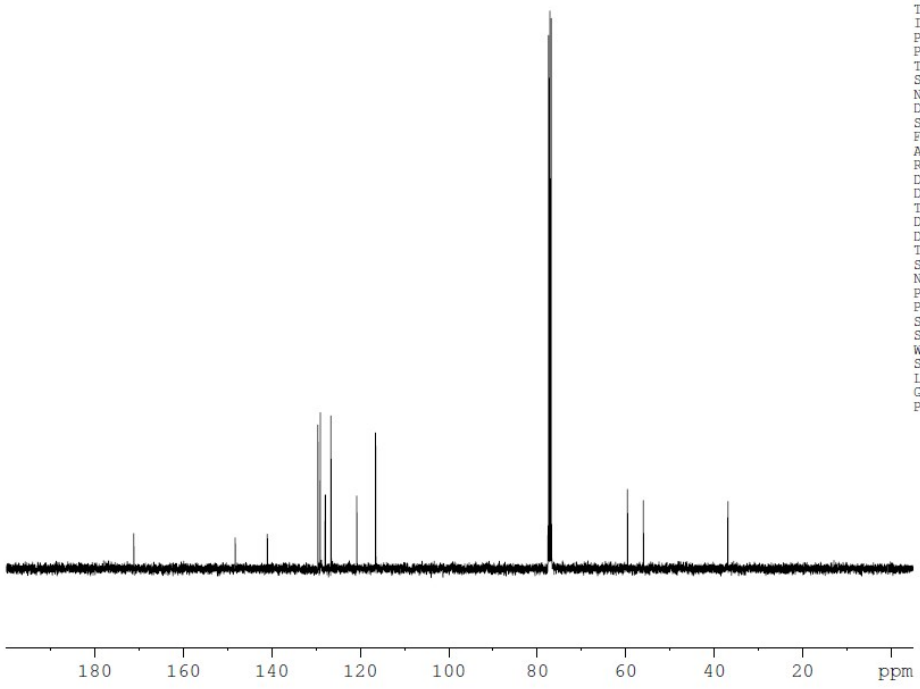


5d

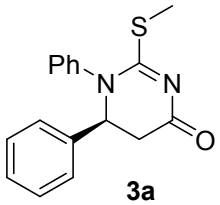
```
NAME          RX-789-1-1H
EXPNO                   4
PROCNO                  1
Date_            20201016
Time                11.00 h
INSTRUM              spect
PROBHD    Z116098_0542 (
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                      8
DS                      0
SWH              8223.685 Hz
FIDRES           0.250967 Hz
AQ               3.9846387 sec
RG                    128
DW                 60.800 usec
DE                  6.50 usec
TE                 298.3 K
D1              1.00000000 sec
TD0                     1
SFO1           400.2324716 MHz
NUC1                   1H
P0                  3.33 usec
P1                 10.00 usec
SI                  32768
SF             400.2300099 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



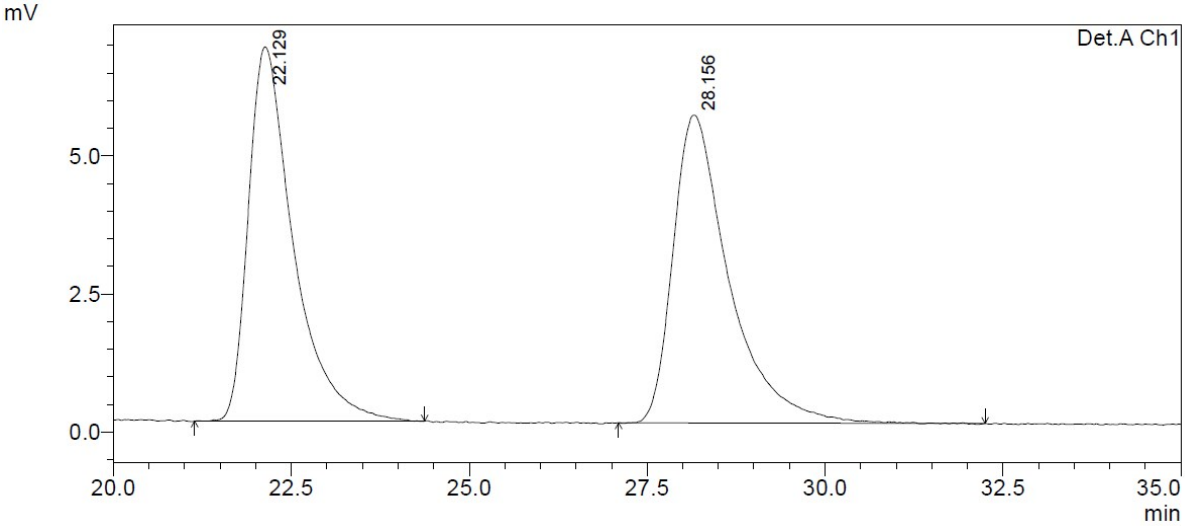
```
NAME          RX-789-1-13C
EXPNO                   5
PROCNO                  1
Date_            20201016
Time                11.22 h
INSTRUM              spect
PROBHD    Z116098_0542 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDC13
NS                    333
DS                      4
SWH             25252.525 Hz
FIDRES           0.770646 Hz
AQ               1.2976629 sec
RG                    114
DW                 19.800 usec
DE                  6.50 usec
TE                 298.8 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                   100
SFO1           100.6499905 MHz
NUC1                   13C
P0                  3.33 usec
P1                 10.00 usec
SI                  32768
SF             100.6379001 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



HPLC Data:

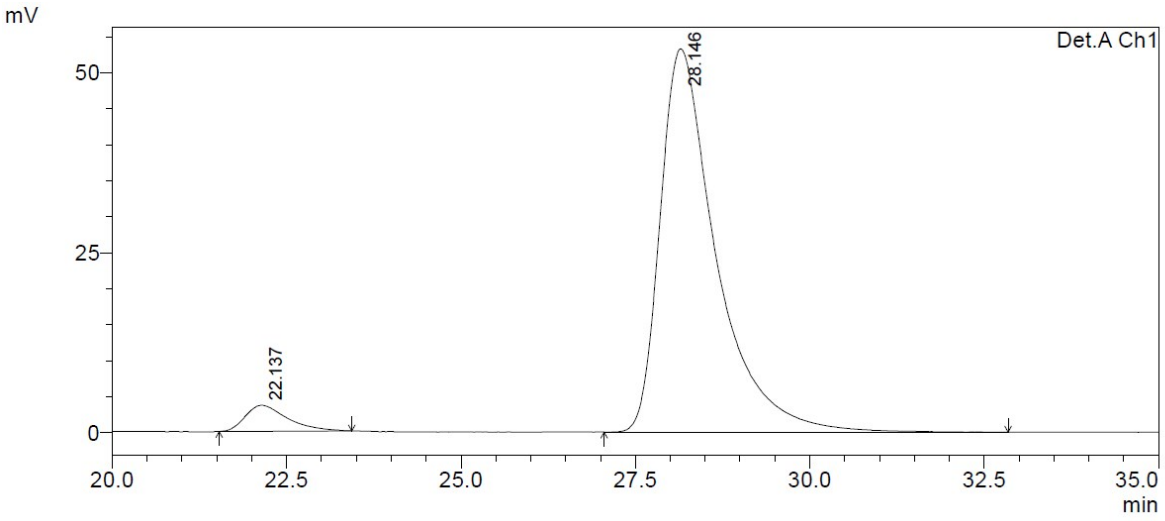


3a



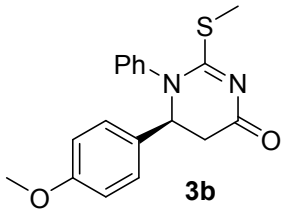
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.129	297217	6771	49.424	54.869
2	28.156	304144	5569	50.576	45.131
Total		601361	12340	100.000	100.000

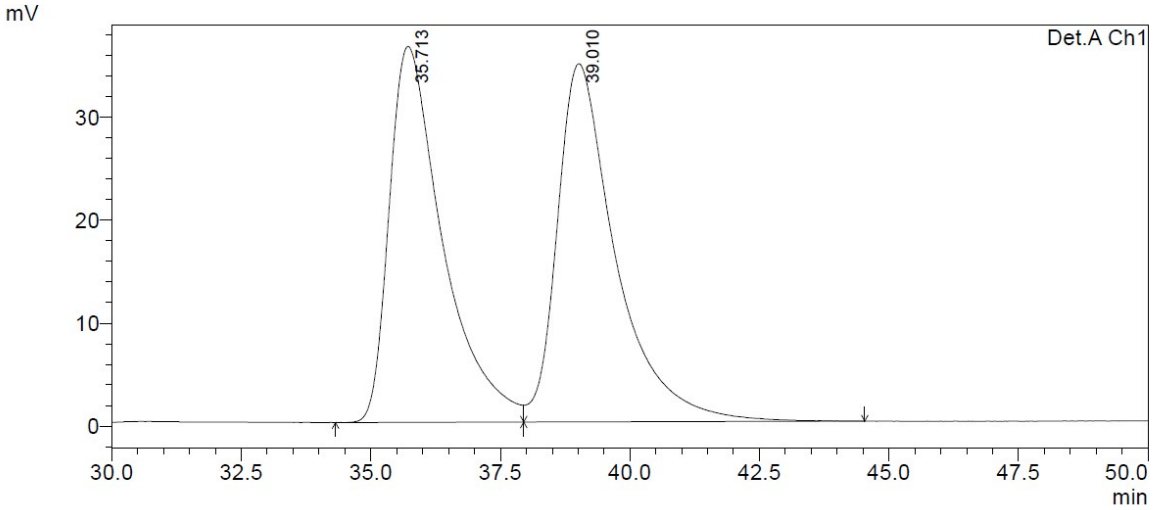


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.137	151189	3606	4.858	6.345
2	28.146	2961267	53222	95.142	93.655
Total		3112456	56828	100.000	100.000

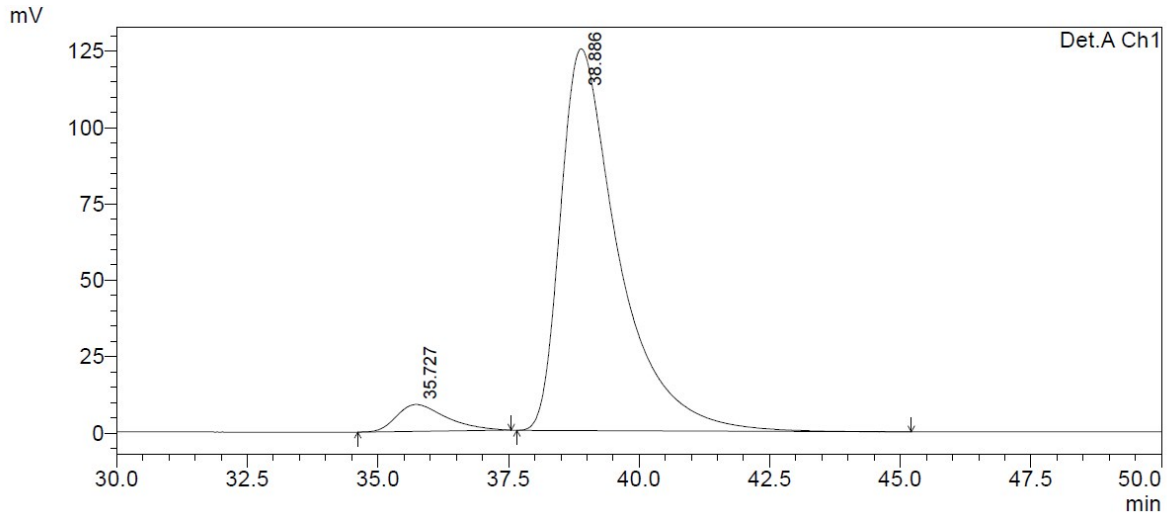


3b



Detector A Ch1 254nm

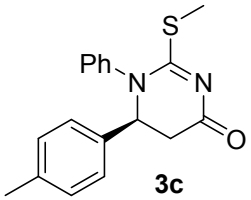
Peak#	Ret. Time	Area	Height	Area %	Height %
1	35.713	2618551	36514	48.960	51.210
2	39.010	2729757	34789	51.040	48.790
Total		5348309	71303	100.000	100.000



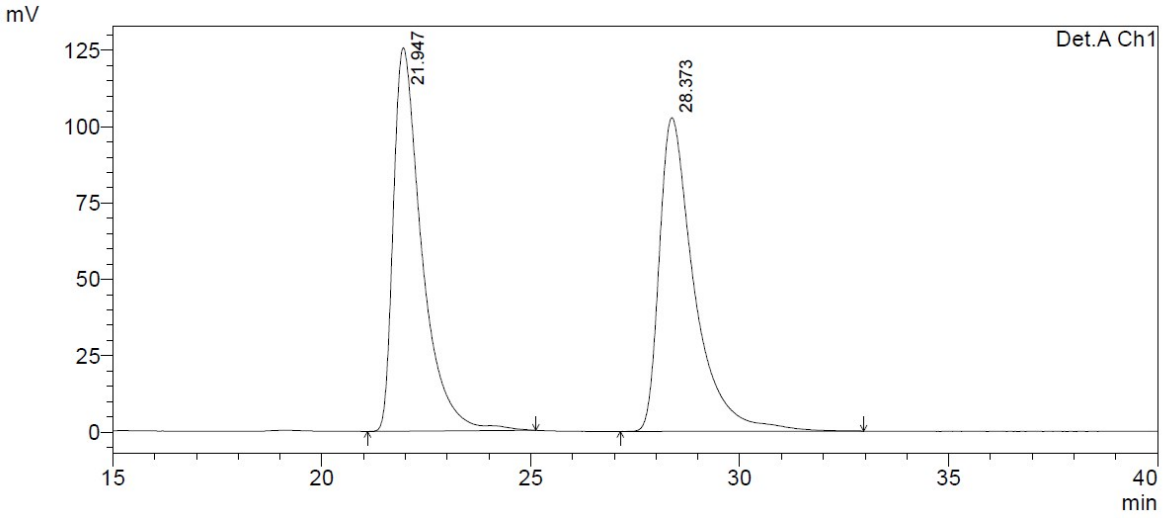
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	35.727	580571	8778	5.667	6.565
2	38.886	9664035	124928	94.333	93.435
Total		10244607	133706	100.000	100.000

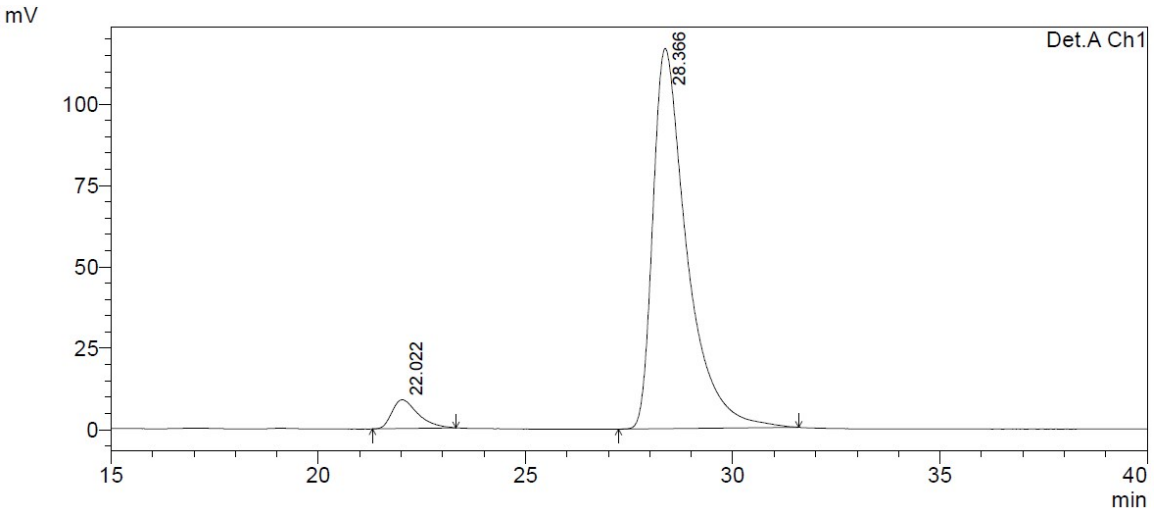


3c



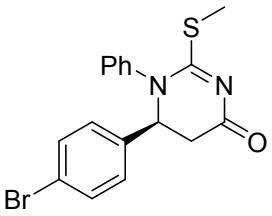
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.947	5928782	125502	49.845	55.009
2	28.373	5965756	102645	50.155	44.991
Total		11894539	228147	100.000	100.000

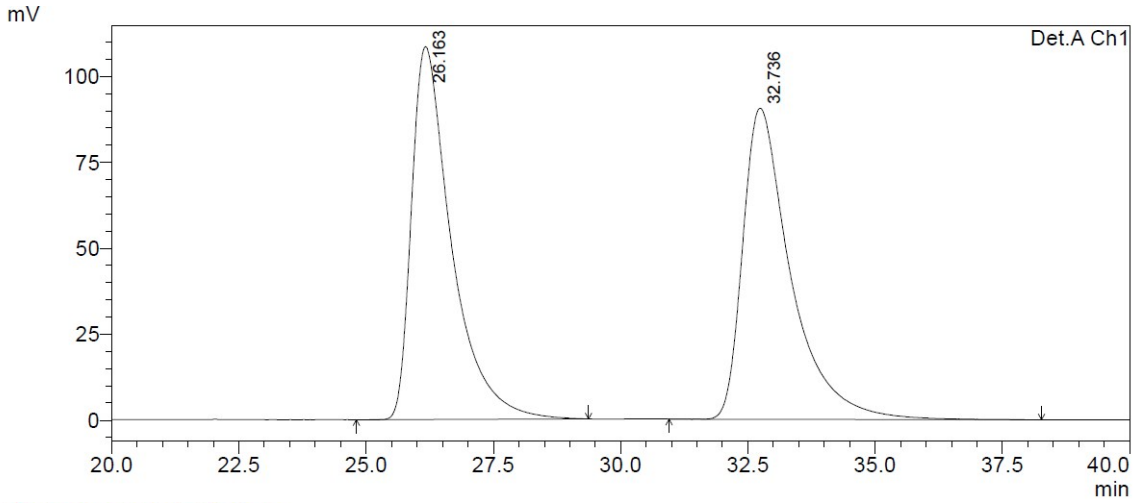


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.022	385661	8827	5.477	7.025
2	28.366	6655879	116829	94.523	92.975
Total		7041540	125656	100.000	100.000

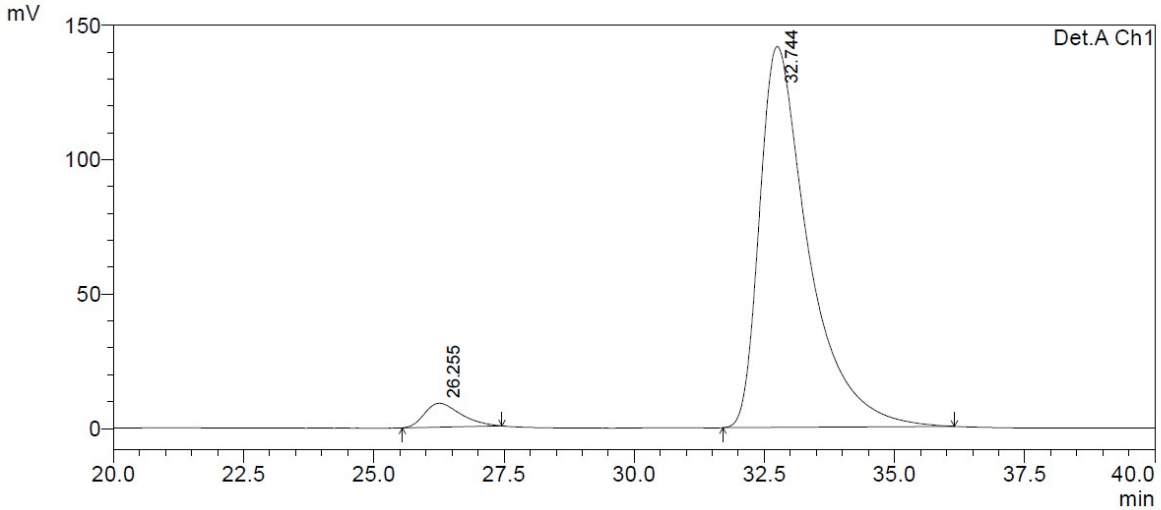


3d



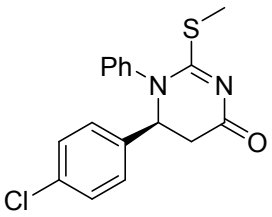
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.163	5920708	108396	49.959	54.511
2	32.736	5930393	90456	50.041	45.489
Total		11851101	198853	100.000	100.000

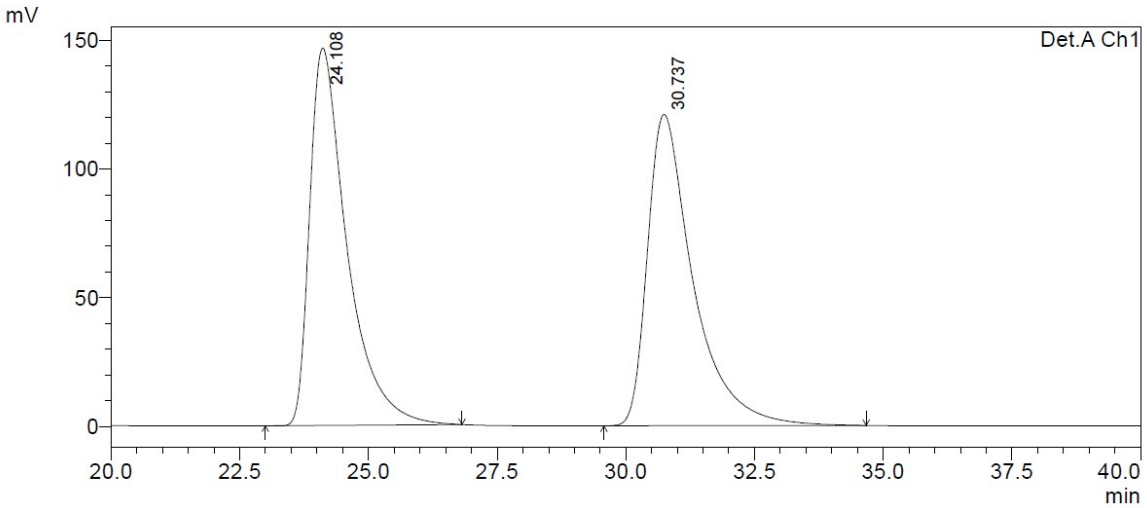


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.255	429988	8904	4.438	5.910
2	32.744	9258346	141752	95.562	94.090
Total		9688334	150657	100.000	100.000

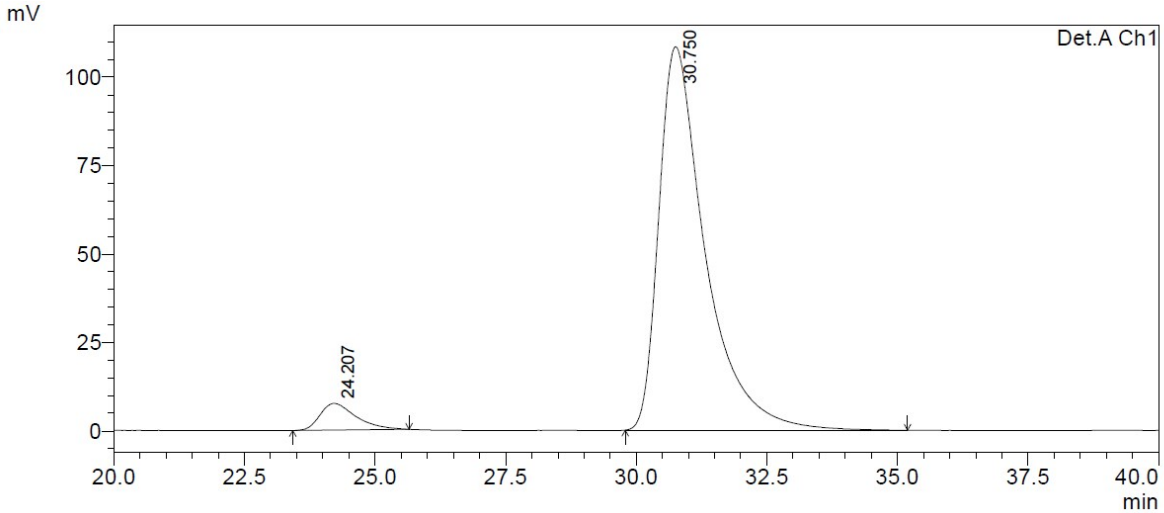


3e



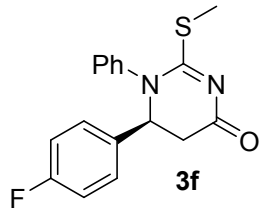
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.108	7377864	146590	49.774	54.801
2	30.737	7444993	120903	50.226	45.199
Total		14822858	267493	100.000	100.000

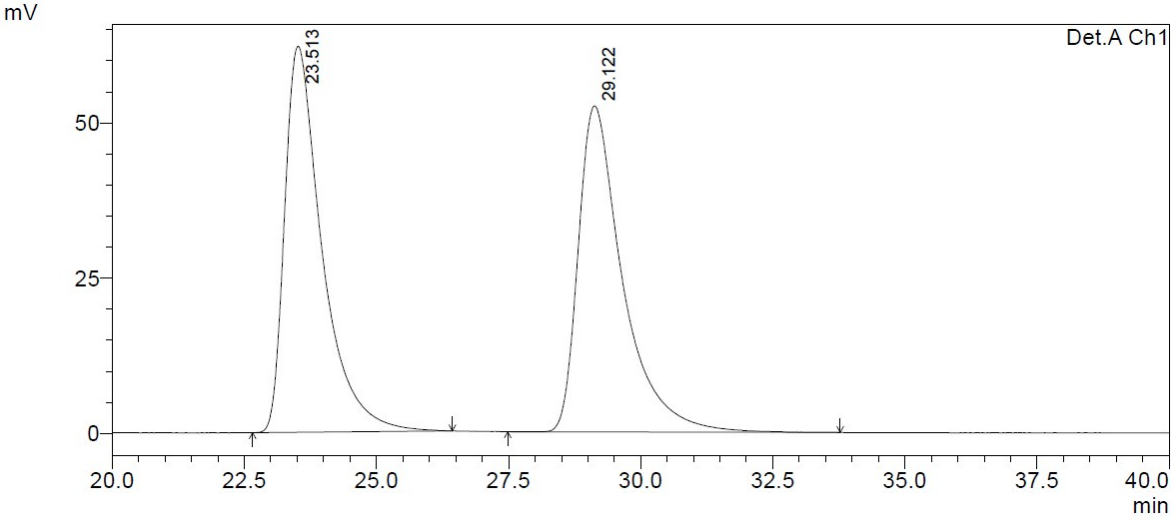


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.207	358984	7510	5.094	6.481
2	30.750	6688085	108363	94.906	93.519
Total		7047068	115873	100.000	100.000

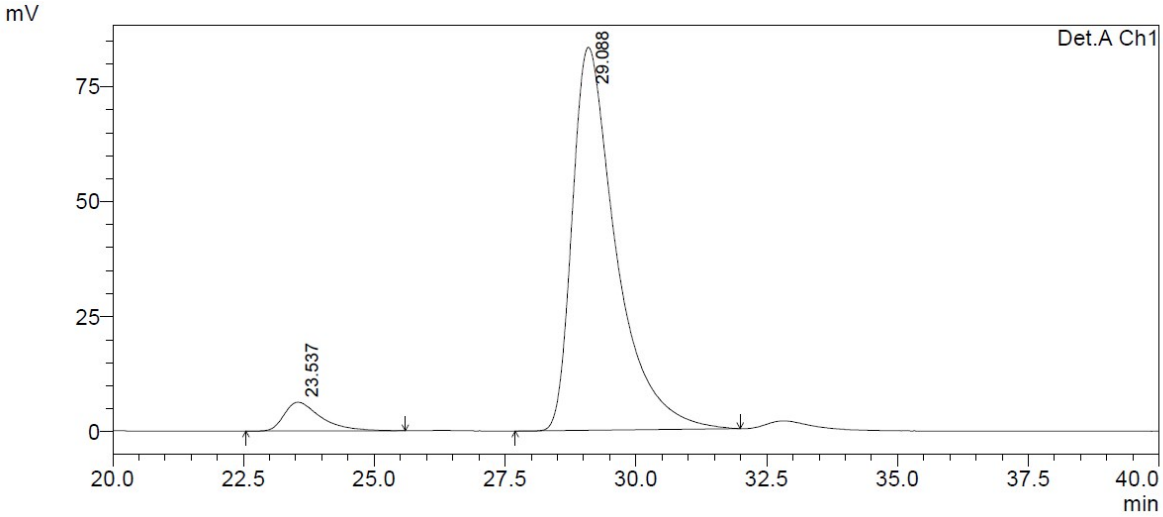


3f



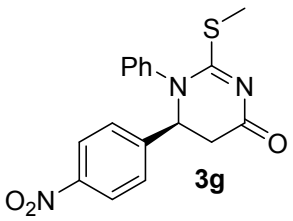
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.513	3057126	62214	49.897	54.224
2	29.122	3069786	52520	50.103	45.776
Total		6126911	114734	100.000	100.000

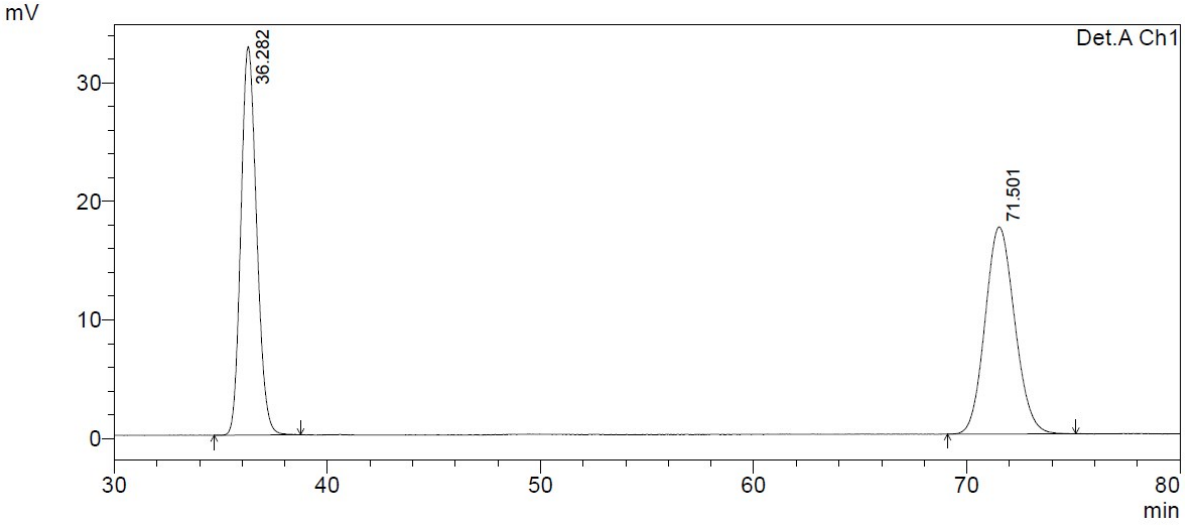


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.537	304563	6244	5.909	6.970
2	29.088	4850051	83343	94.091	93.030
Total		5154615	89587	100.000	100.000

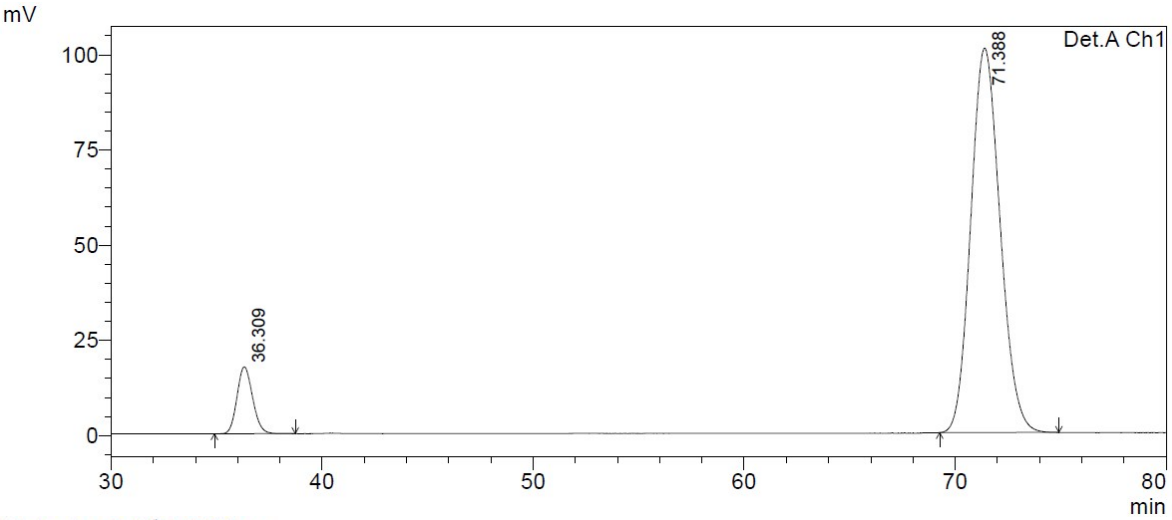


3g



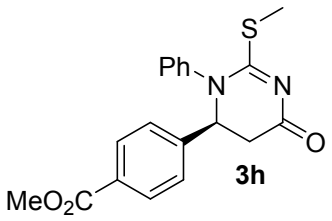
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	36.282	1680655	32746	49.891	65.221
2	71.501	1687983	17461	50.109	34.779
Total		3368638	50207	100.000	100.000

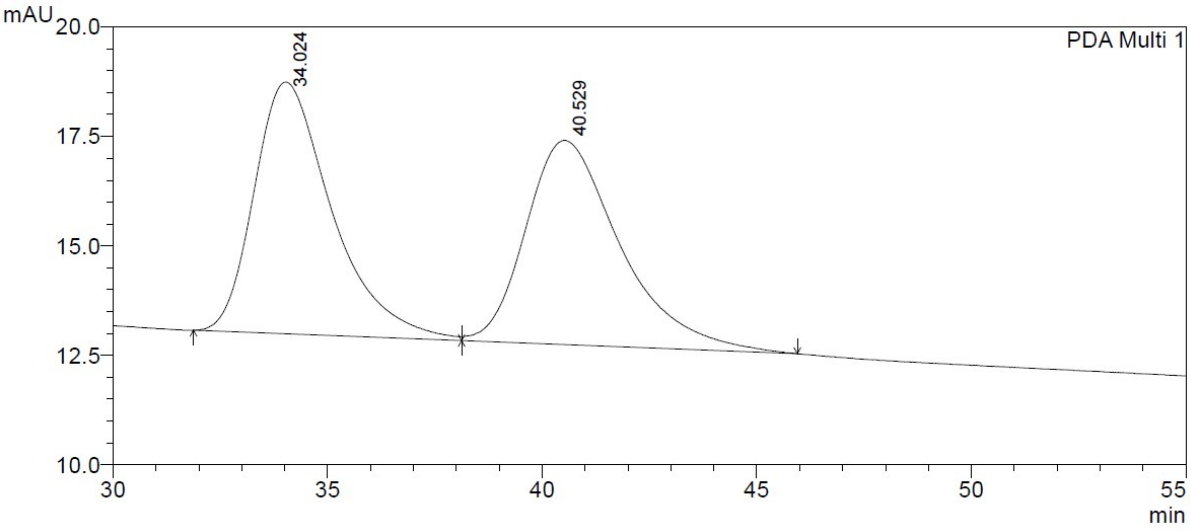


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	36.309	900971	17569	8.458	14.819
2	71.388	9751244	100995	91.542	85.181
Total		10652214	118564	100.000	100.000

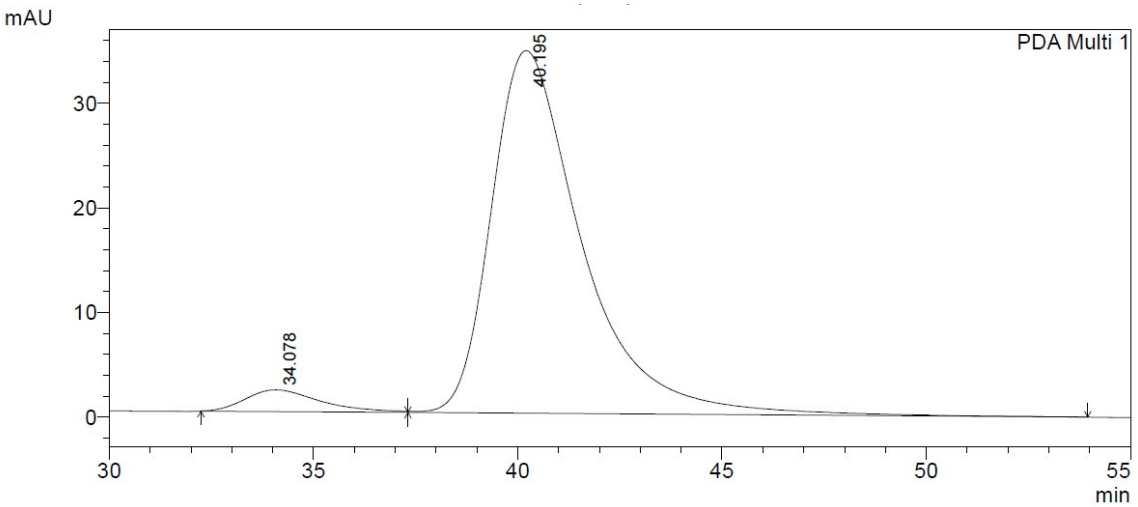


3h



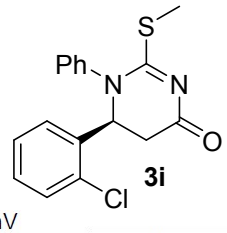
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.024	723368	5750	50.302	55.201
2	40.529	714684	4667	49.698	44.799
Total		1438052	10417	100.000	100.000

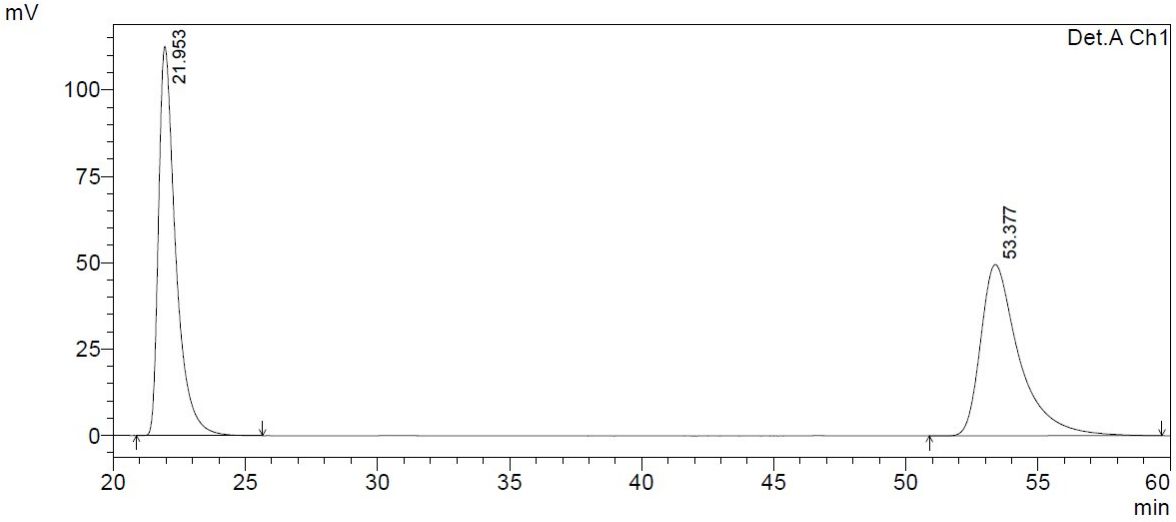


PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.078	264525	2082	4.700	5.669
2	40.195	5363811	34636	95.300	94.331
Total		5628336	36718	100.000	100.000

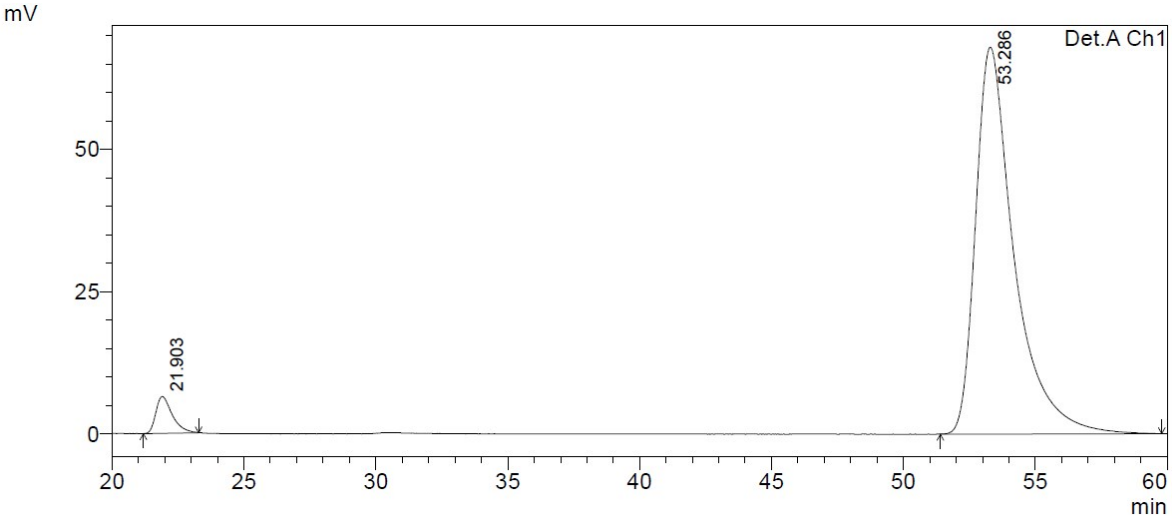


3i



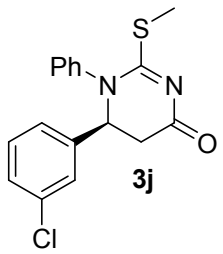
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.953	5061775	112574	50.168	69.435
2	53.377	5027787	49555	49.832	30.565
Total		10089561	162129	100.000	100.000

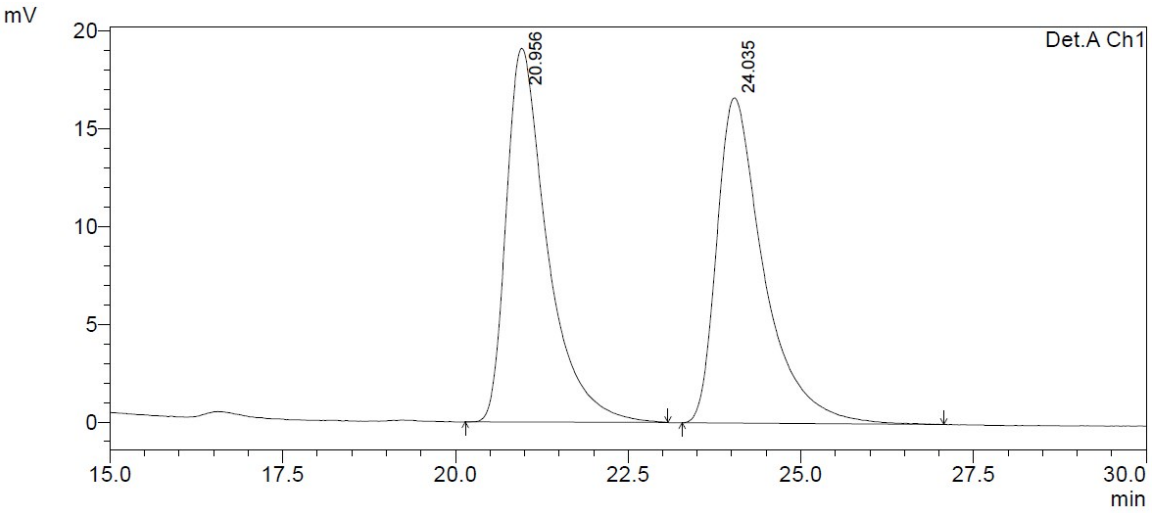


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.903	278250	6464	3.874	8.682
2	53.286	6903536	67984	96.126	91.318
Total		7181786	74448	100.000	100.000

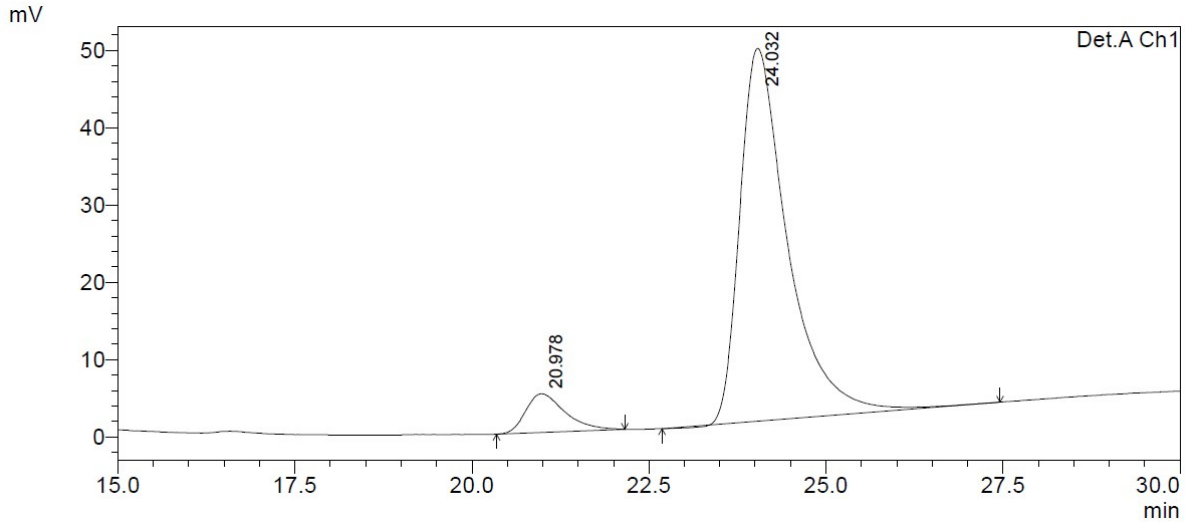


3j



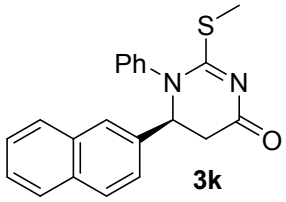
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.956	788033	19113	49.999	53.472
2	24.035	788055	16631	50.001	46.528
Total		1576088	35744	100.000	100.000

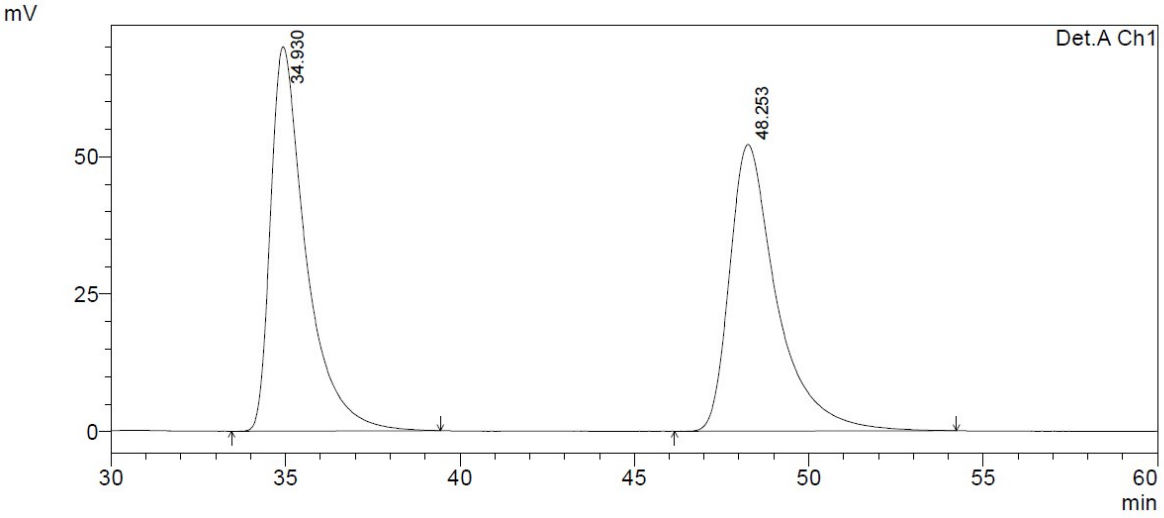


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.978	193573	5009	7.870	9.405
2	24.032	2266081	48253	92.130	90.595
Total		2459654	53262	100.000	100.000

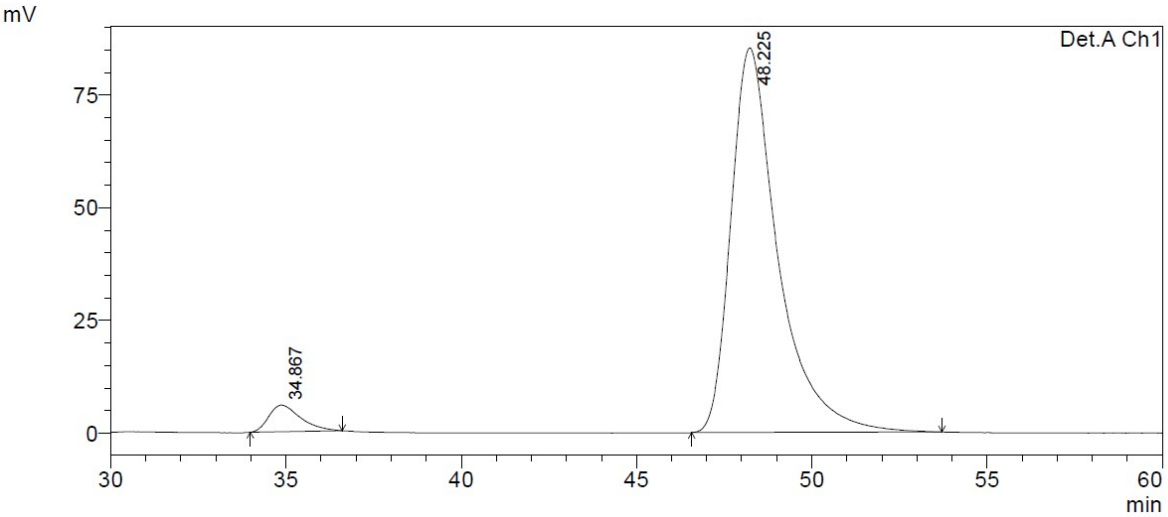


3k



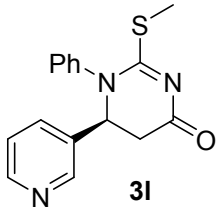
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.930	4928341	69950	50.133	57.278
2	48.253	4902233	52173	49.867	42.722
Total		9830574	122123	100.000	100.000

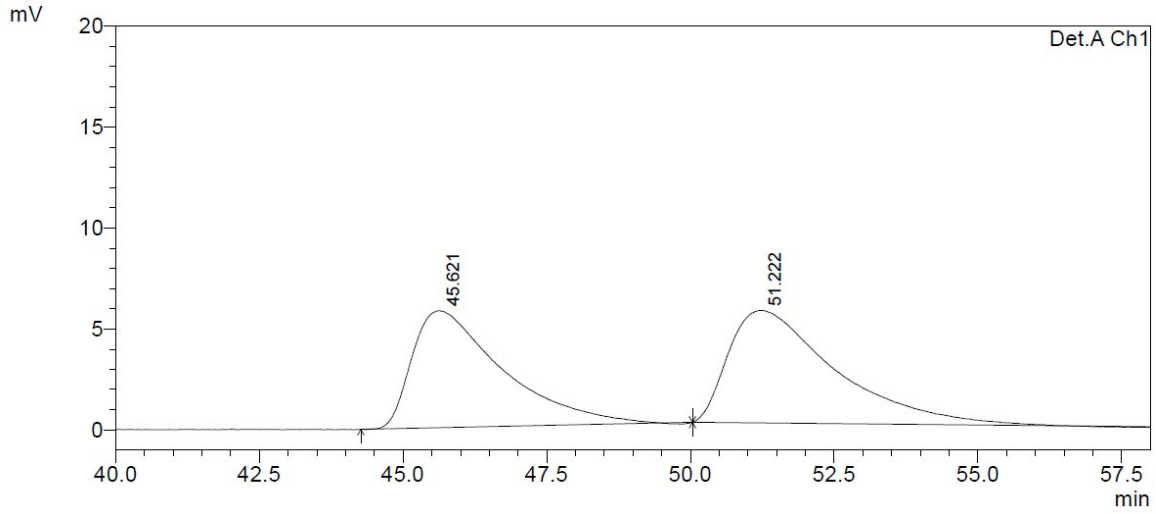


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.867	372373	5897	4.469	6.469
2	48.225	7960744	85259	95.531	93.531
Total		8333117	91157	100.000	100.000

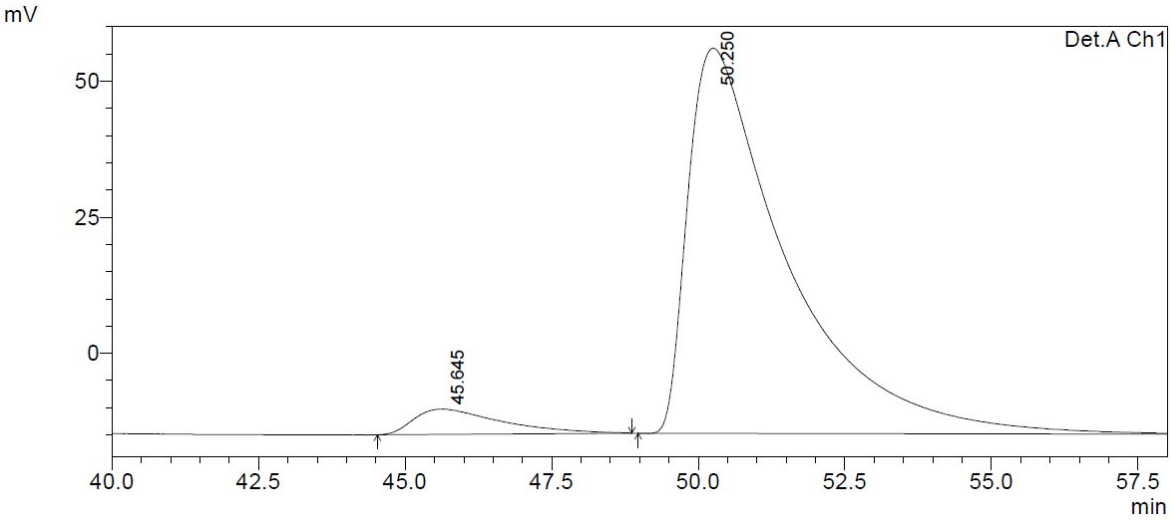


3l



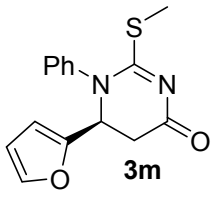
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	45.621	628758	5792	46.739	50.983
2	51.222	716495	5569	53.261	49.017
Total		1345253	11360	100.000	100.000

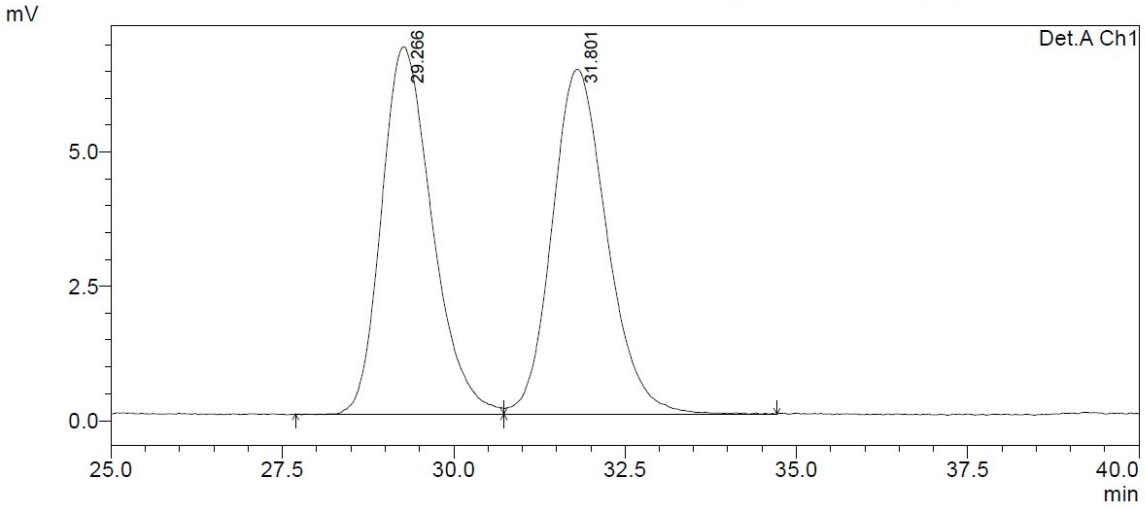


Detector A Ch1 254nm

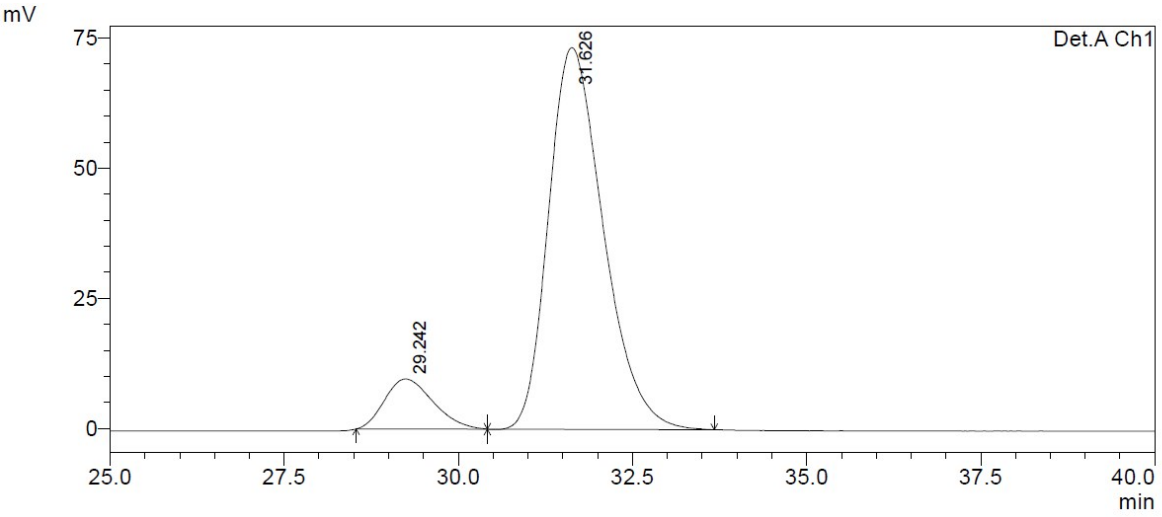
Peak#	Ret. Time	Area	Height	Area %	Height %
1	45.645	478821	4635	5.423	6.143
2	50.250	8350995	70824	94.577	93.857
Total		8829816	75459	100.000	100.000



3m

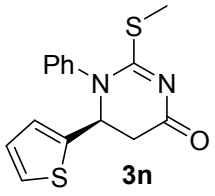


Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.266	350964	6837	49.585	51.587
2	31.801	356842	6417	50.415	48.413
Total		707806	13254	100.000	100.000

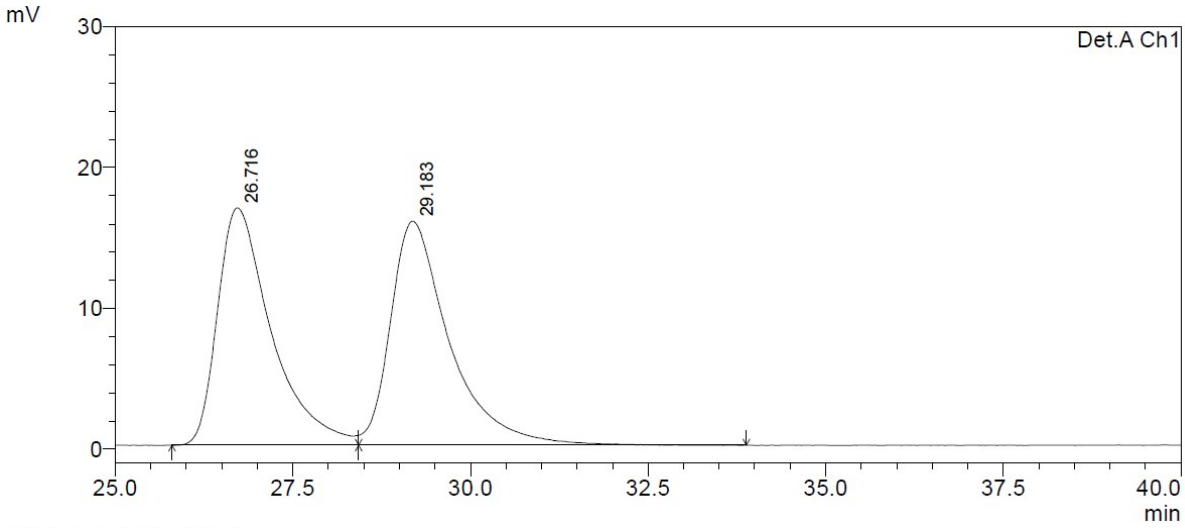


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.242	465870	9619	10.391	11.591
2	31.626	4017380	73366	89.609	88.409
Total		4483250	82984	100.000	100.000

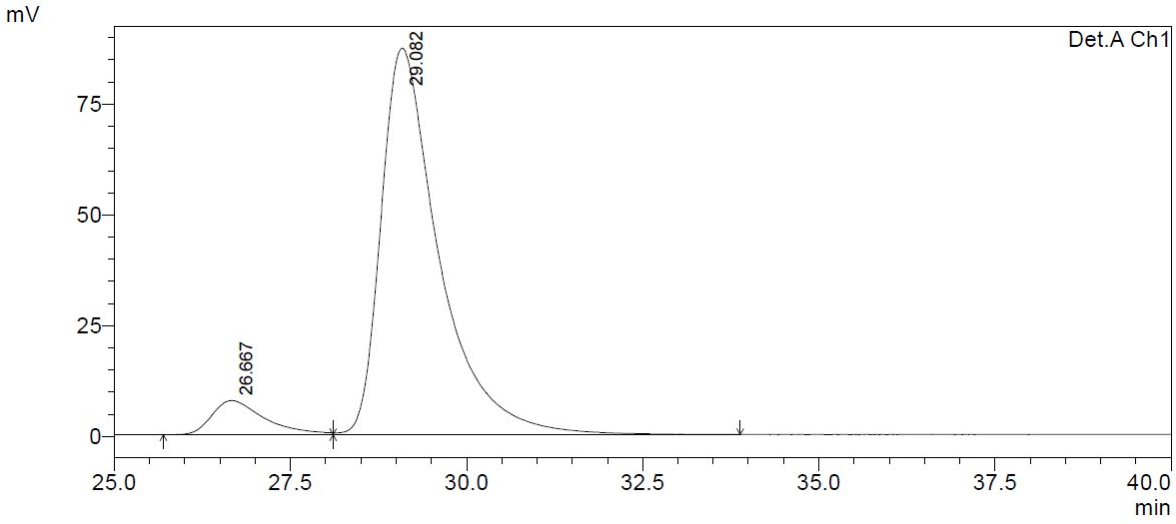


3n



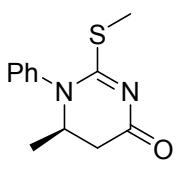
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.716	888331	16866	49.138	51.451
2	29.183	919480	15914	50.862	48.549
Total		1807811	32781	100.000	100.000

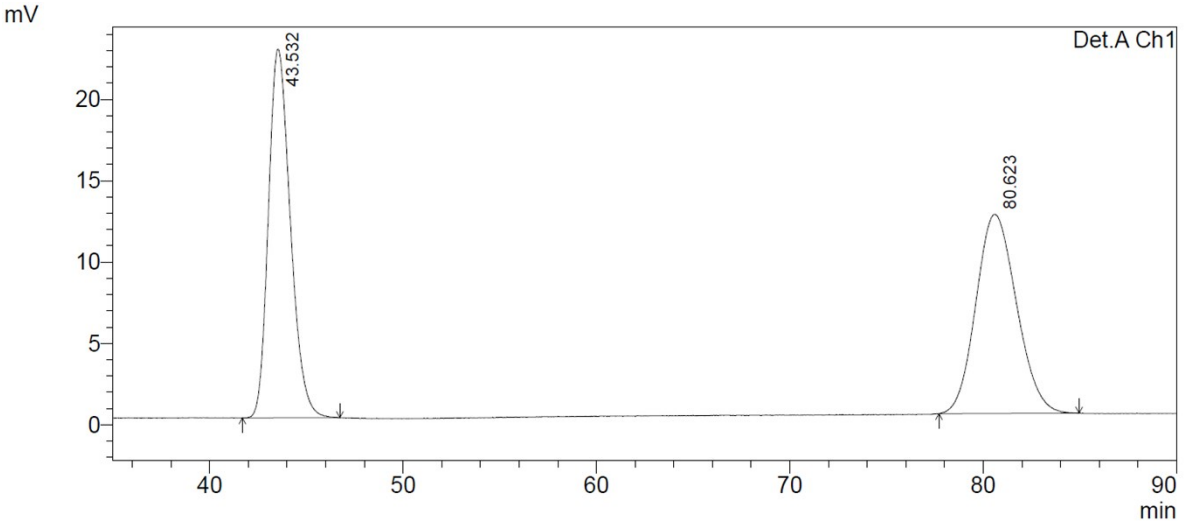


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.667	398314	7692	7.374	8.102
2	29.082	5003019	87246	92.626	91.898
Total		5401333	94938	100.000	100.000

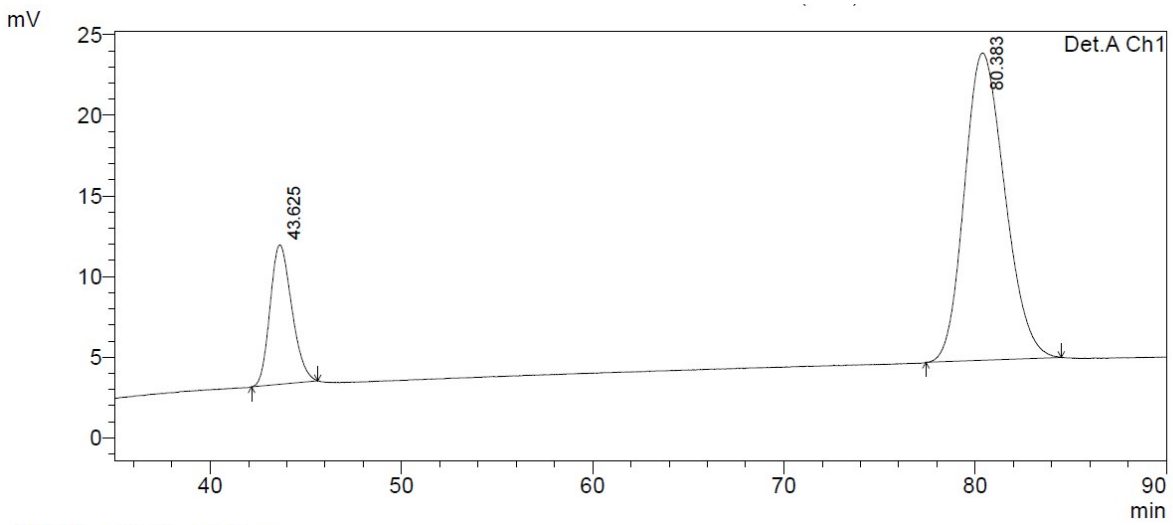


3o



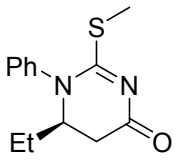
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	43.532	1789689	22668	50.056	64.936
2	80.623	1785709	12240	49.944	35.064
Total		3575398	34908	100.000	100.000

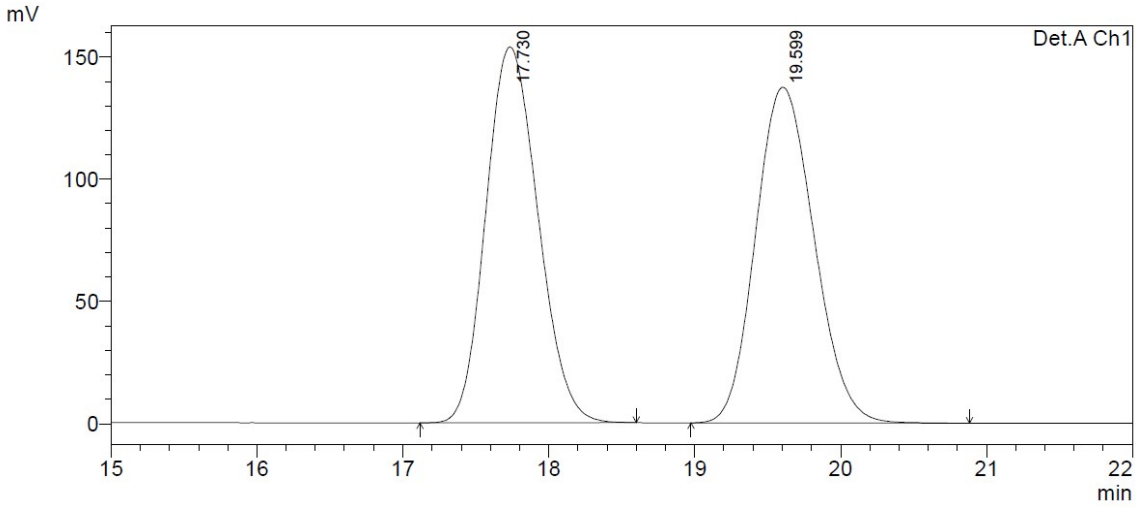


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	43.625	682469	8634	19.599	31.188
2	80.383	2799772	19050	80.401	68.812
Total		3482240	27684	100.000	100.000

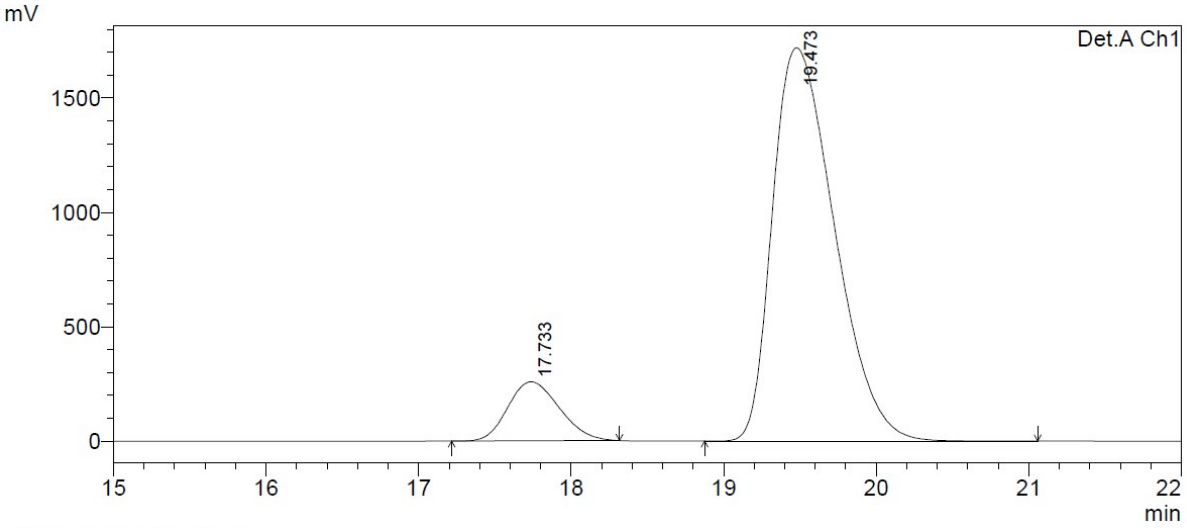


3p



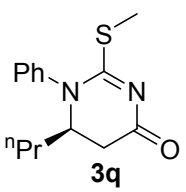
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.730	3815433	153739	49.928	52.823
2	19.599	3826394	137309	50.072	47.177
Total		7641827	291048	100.000	100.000

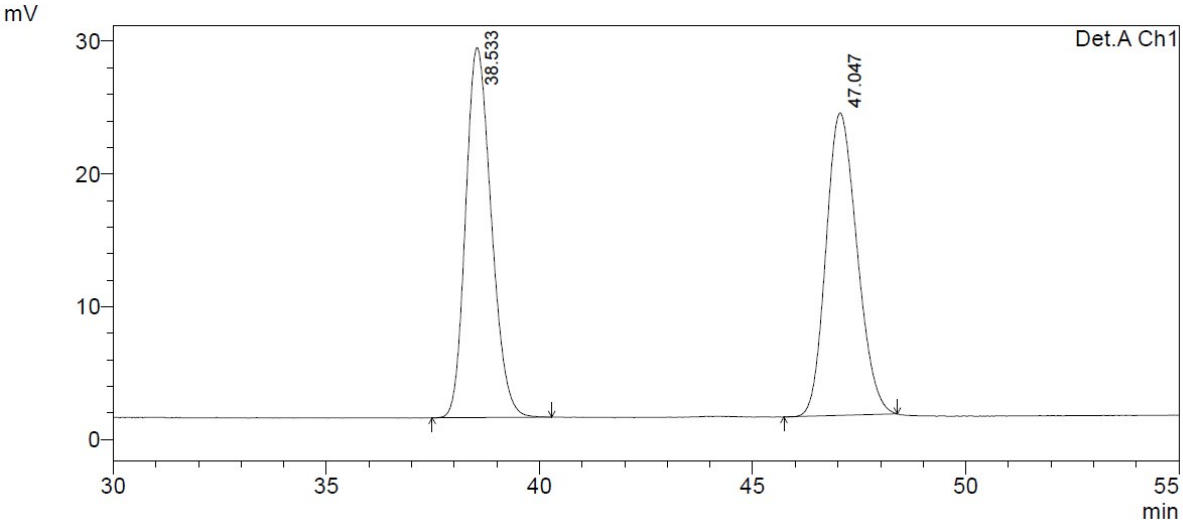


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.733	6072327	258179	11.112	13.060
2	19.473	48572983	1718736	88.888	86.940
Total		54645310	1976915	100.000	100.000

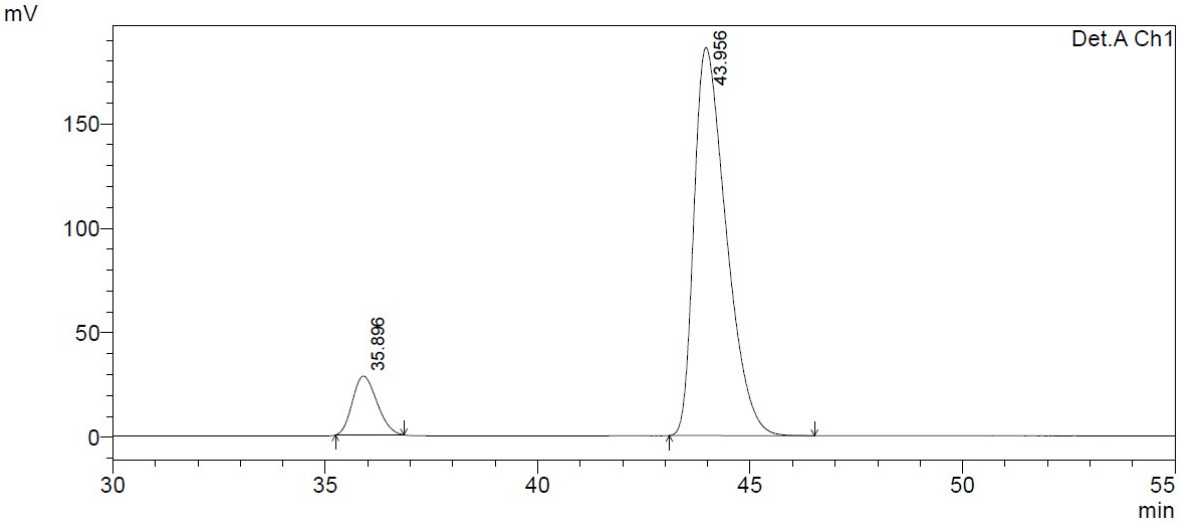


3q



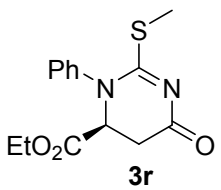
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	38.533	1167540	27874	50.228	55.005
2	47.047	1156918	22801	49.772	44.995
Total		2324457	50675	100.000	100.000

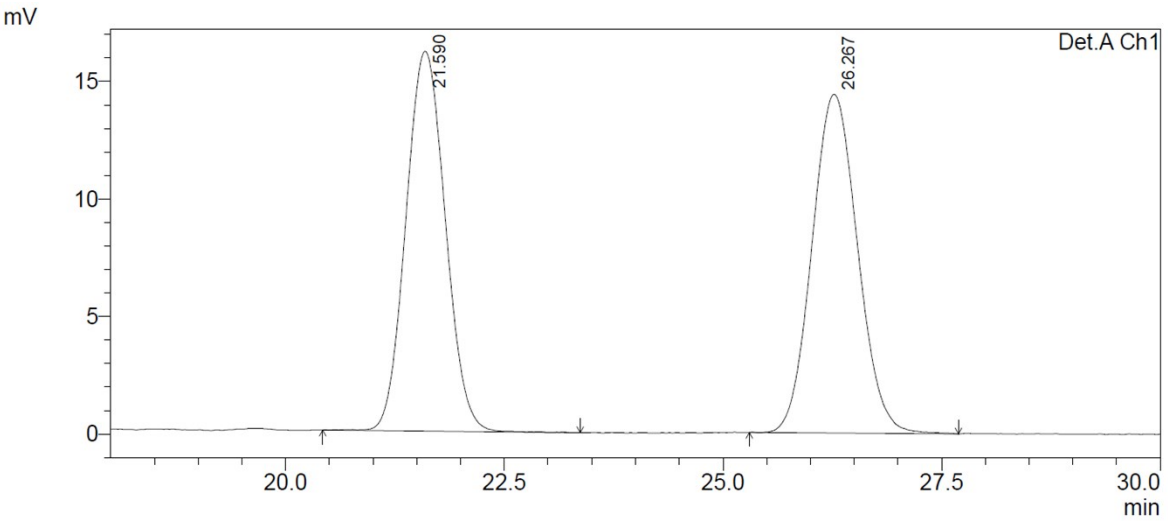


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	35.896	1129258	28174	10.362	13.174
2	43.956	9768326	185682	89.638	86.826
Total		10897584	213856	100.000	100.000

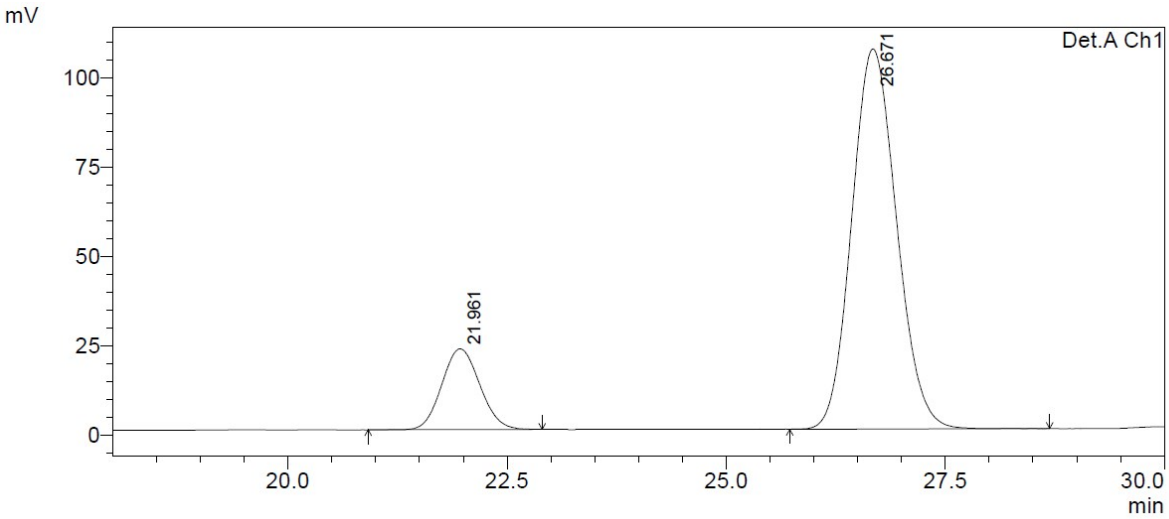


3r



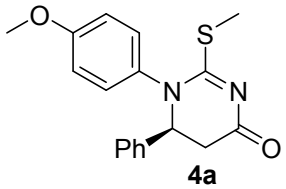
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.590	506359	16166	49.200	52.860
2	26.267	522820	14417	50.800	47.140
Total		1029179	30583	100.000	100.000

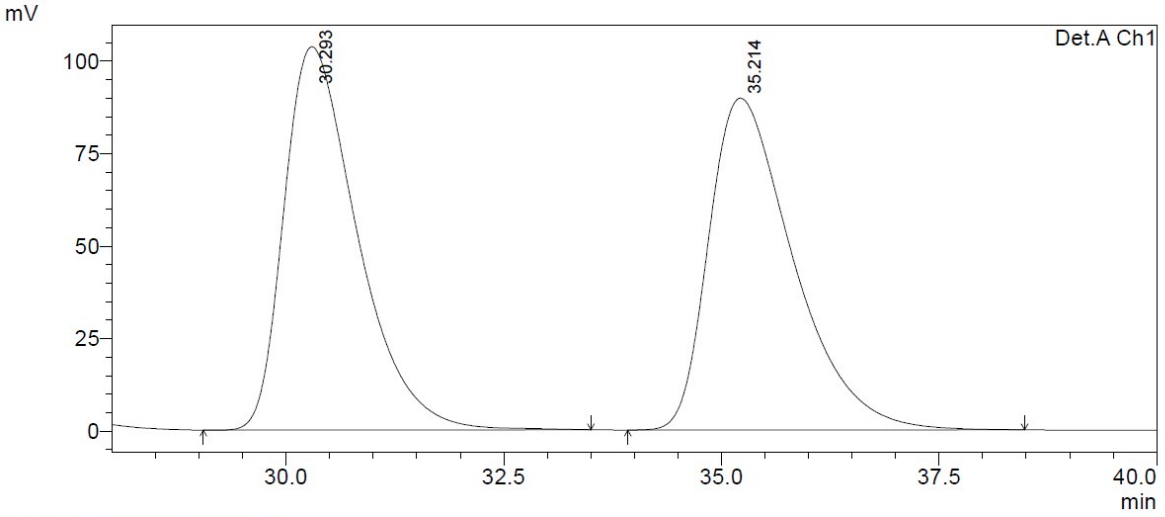


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.961	682609	22608	15.291	17.525
2	26.671	3781512	106391	84.709	82.475
Total		4464121	128999	100.000	100.000

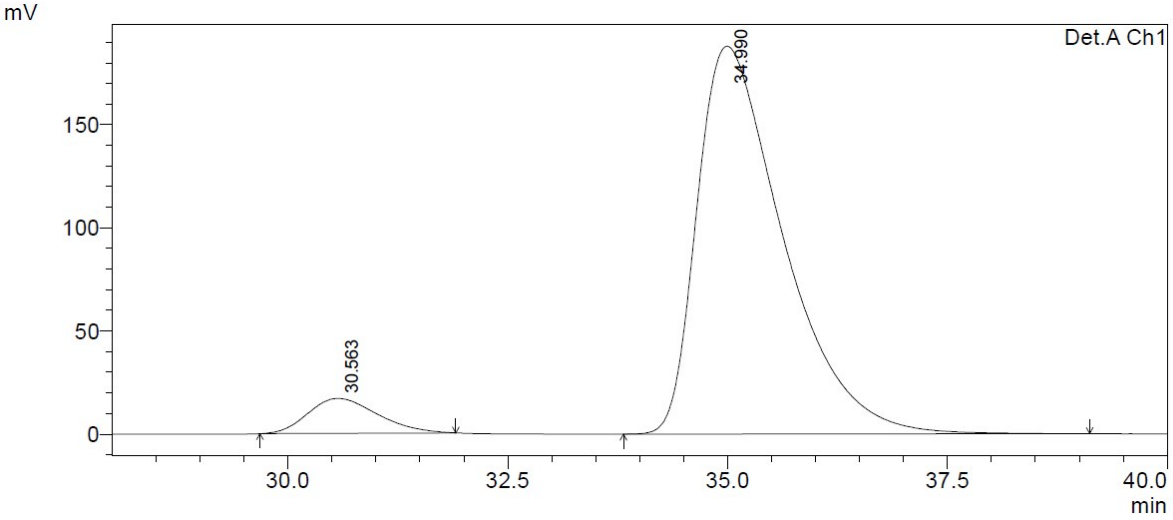


4a



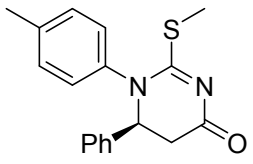
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.293	6076919	103628	50.047	53.600
2	35.214	6065490	89709	49.953	46.400
Total		12142409	193337	100.000	100.000

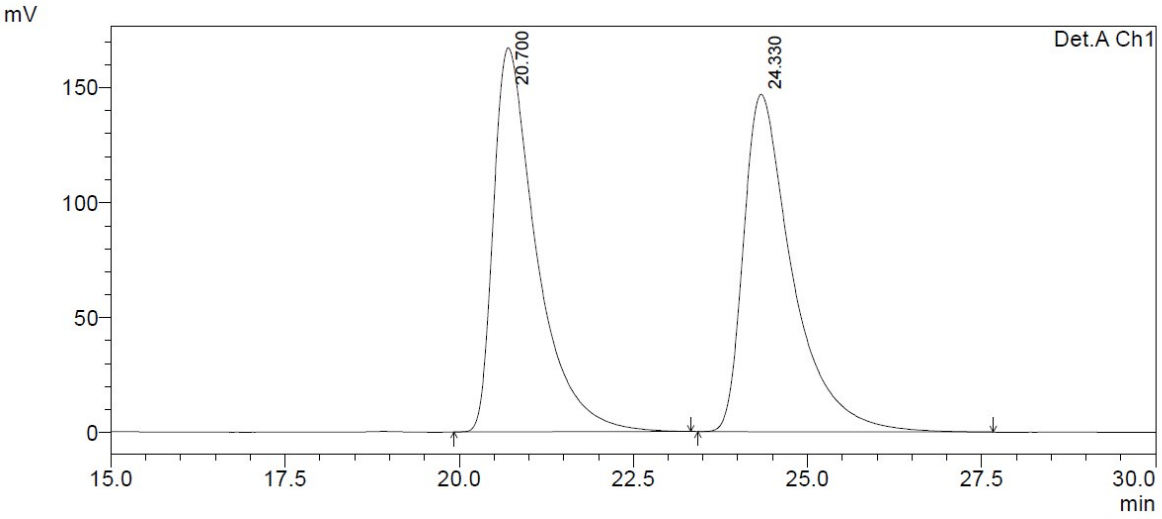


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.563	929990	16933	6.774	8.266
2	34.990	12799086	187920	93.226	91.734
Total		13729076	204853	100.000	100.000

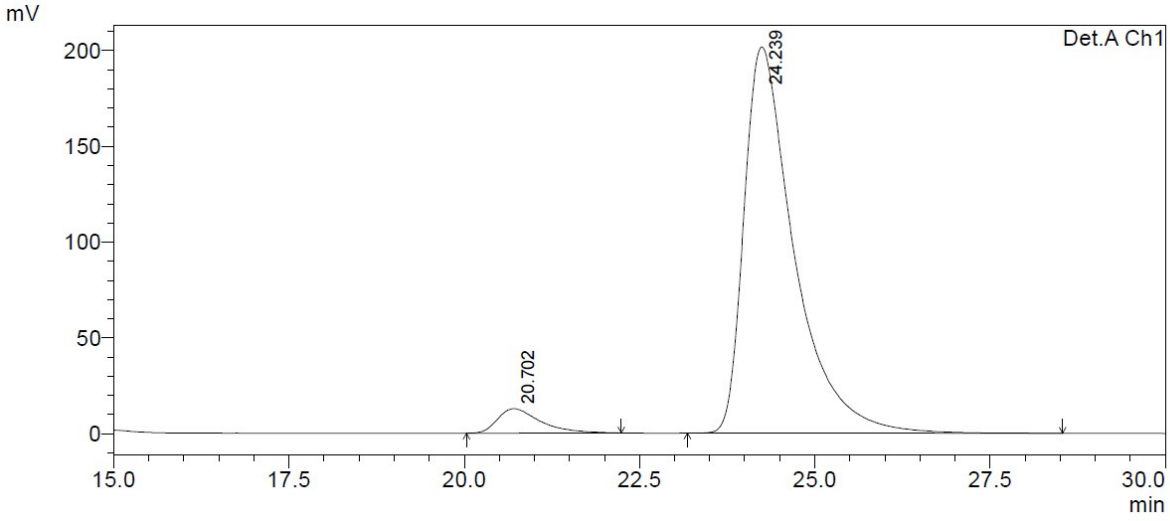


4b



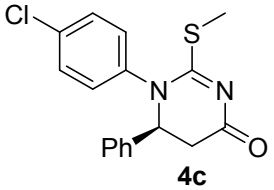
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.700	7145525	166999	49.940	53.237
2	24.330	7162760	146690	50.060	46.763
Total		14308285	313689	100.000	100.000

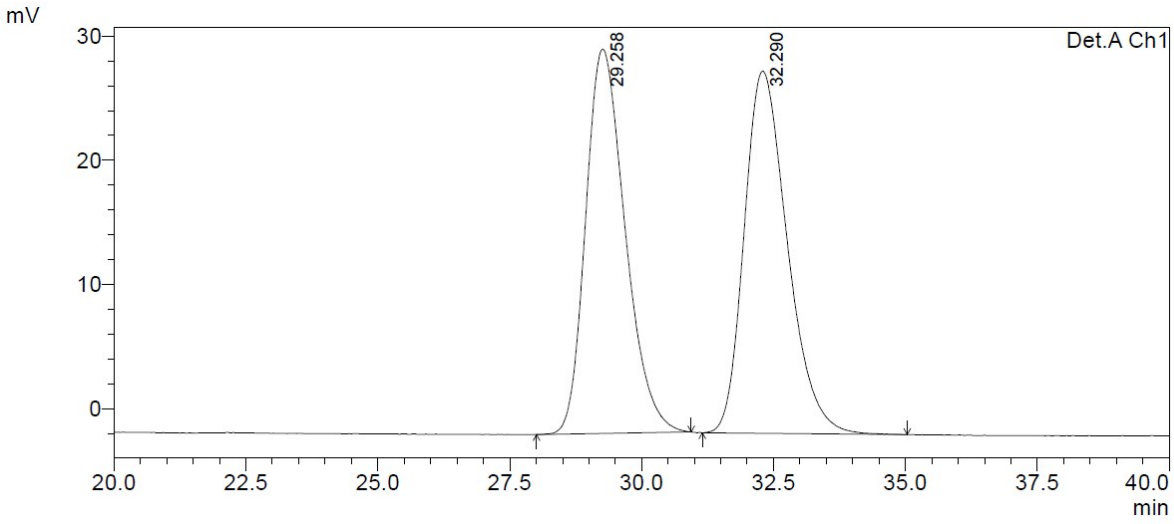


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.702	536472	12730	5.079	5.941
2	24.239	10027068	201523	94.921	94.059
Total		10563541	214253	100.000	100.000

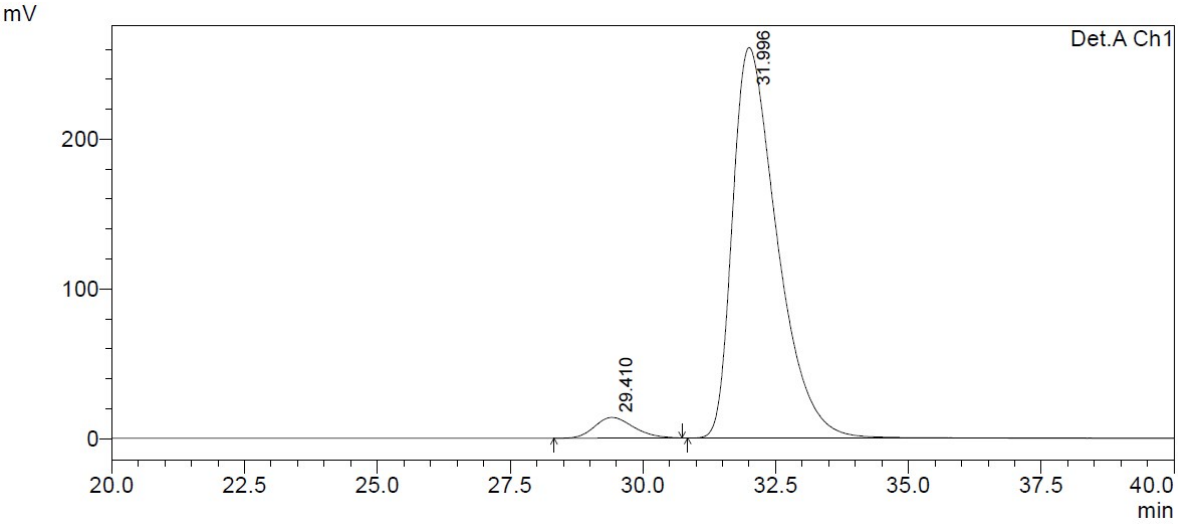


4c



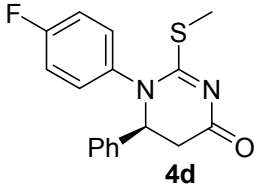
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.258	1609588	30924	49.247	51.474
2	32.290	1658780	29153	50.753	48.526
Total		3268368	60077	100.000	100.000

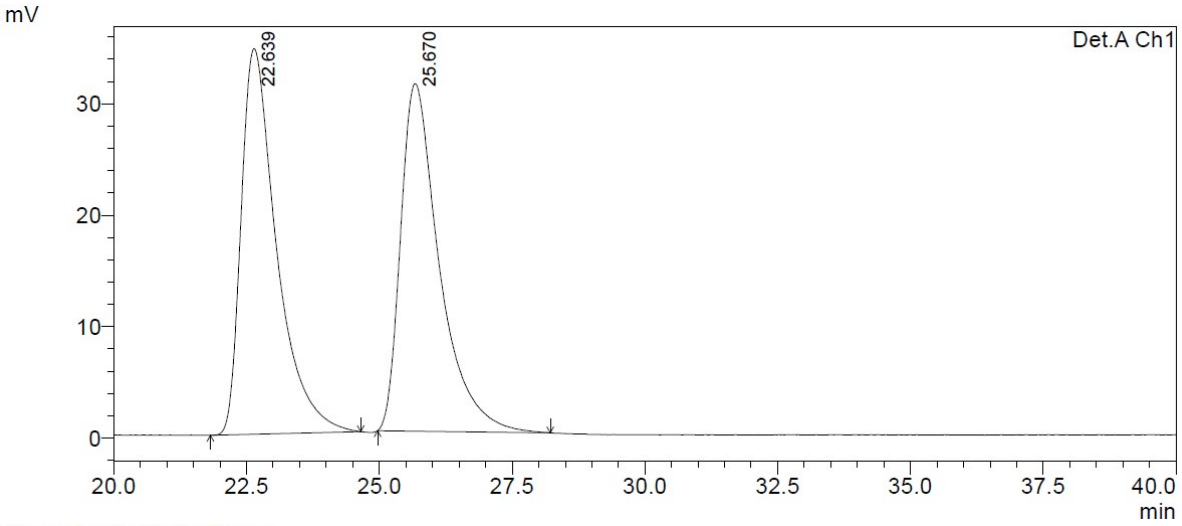


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.410	697836	13766	4.384	5.017
2	31.996	15219025	260634	95.616	94.983
Total		15916861	274401	100.000	100.000

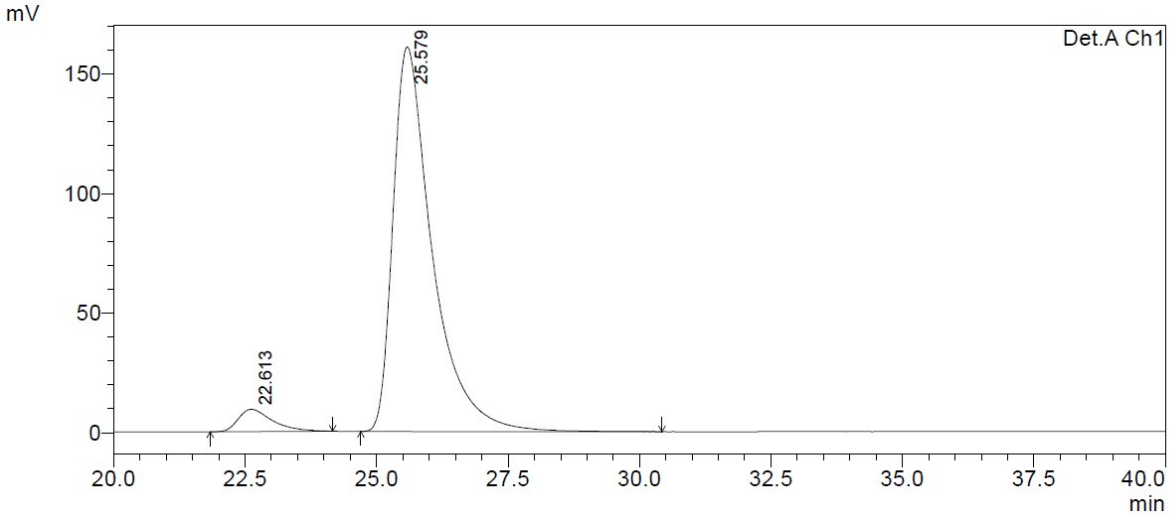


4d



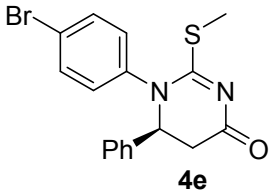
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.639	1604405	34588	50.371	52.598
2	25.670	1580780	31171	49.629	47.402
Total		3185185	65759	100.000	100.000

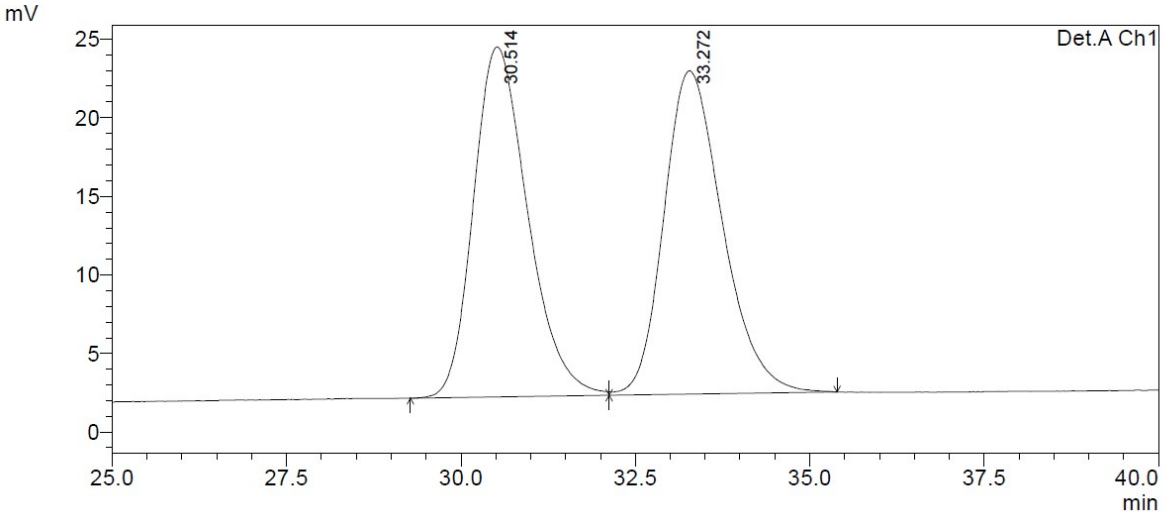


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.613	421049	9332	4.808	5.486
2	25.579	8336471	160770	95.192	94.514
Total		8757520	170102	100.000	100.000

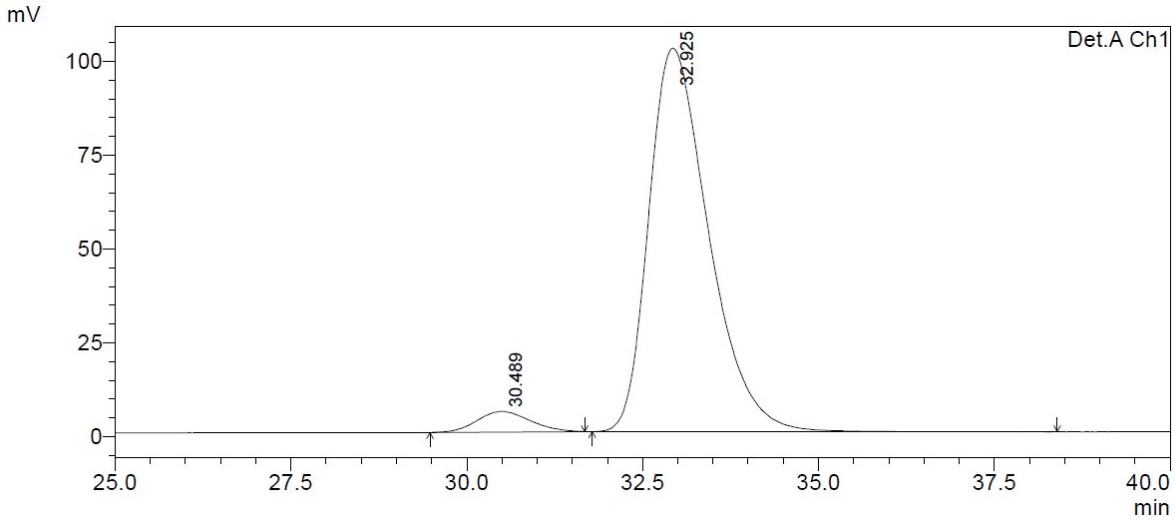


4e



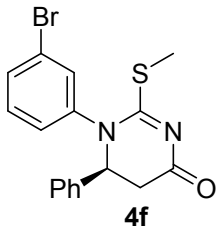
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.514	1203855	22264	50.038	51.984
2	33.272	1202037	20565	49.962	48.016
Total		2405891	42829	100.000	100.000

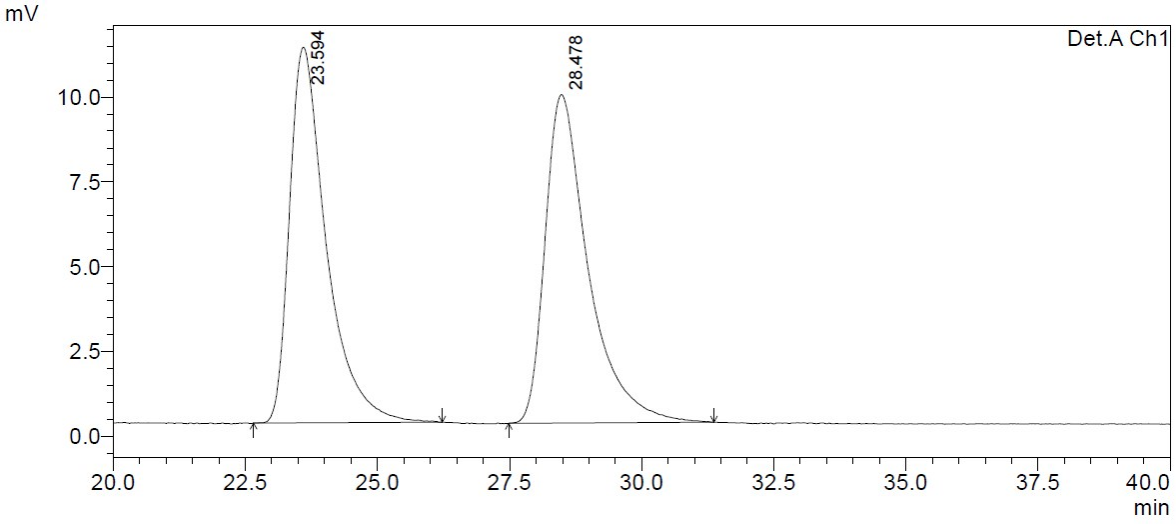


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.489	287168	5488	4.533	5.094
2	32.925	6048395	102260	95.467	94.906
Total		6335562	107749	100.000	100.000

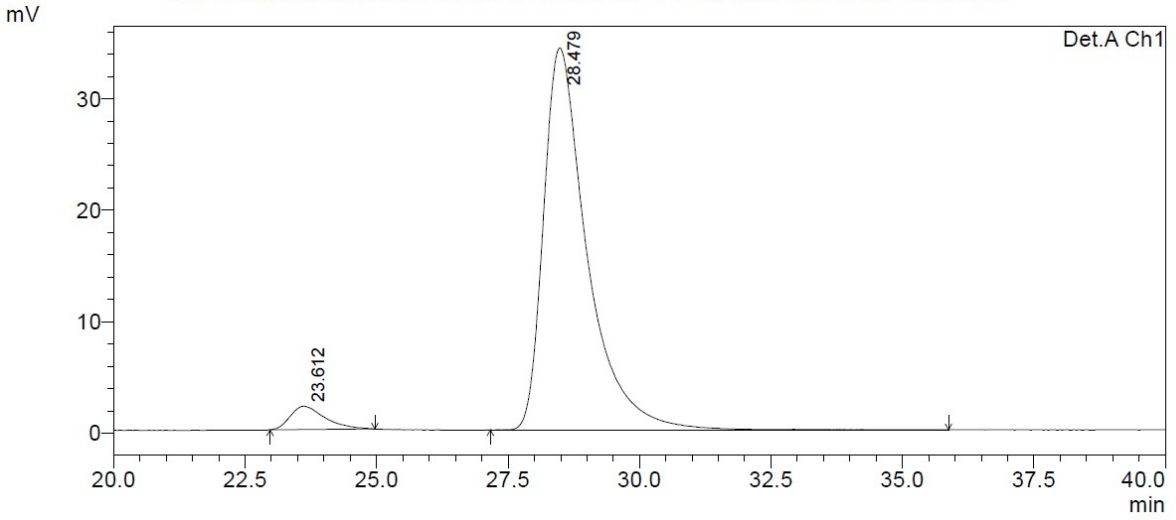


4f



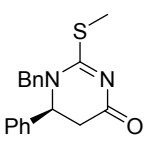
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.594	536143	11074	49.368	53.339
2	28.478	549863	9687	50.632	46.661
Total		1086006	20761	100.000	100.000

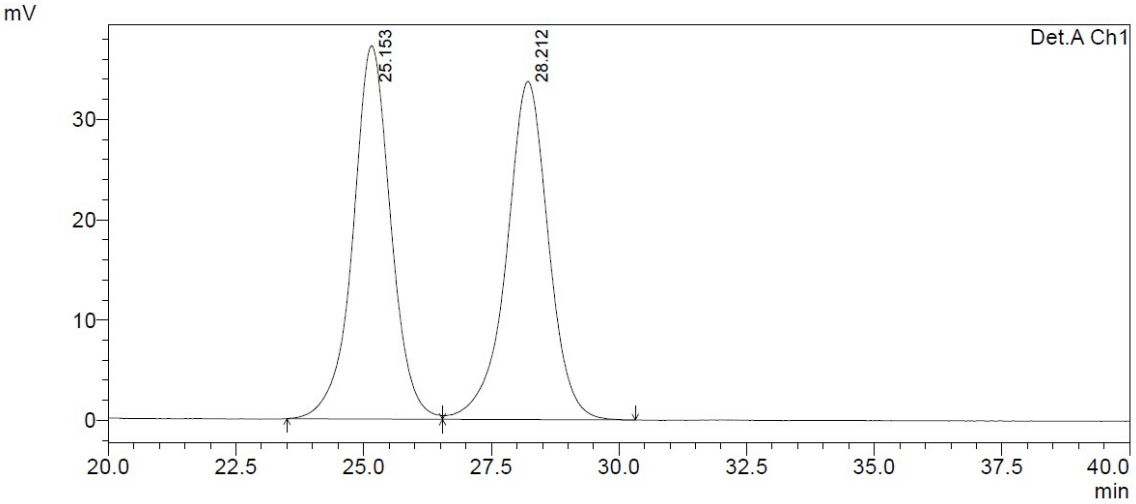


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	23.612	95823	2085	4.630	5.735
2	28.479	1973984	34272	95.370	94.265
Total		2069806	36357	100.000	100.000

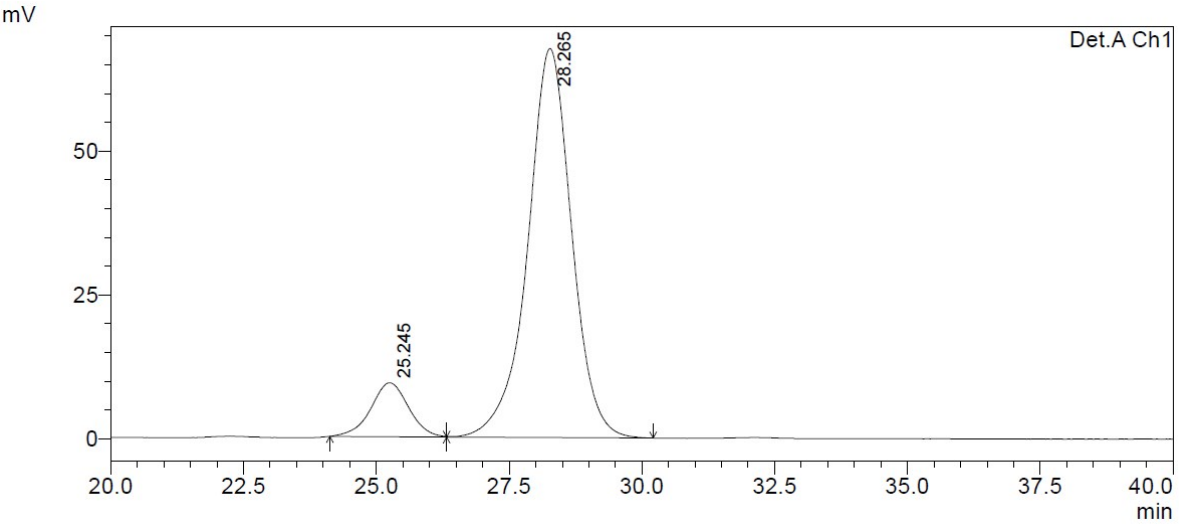


4g



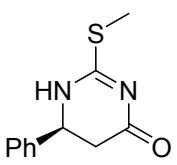
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.153	1916128	37179	49.878	52.459
2	28.212	1925496	33693	50.122	47.541
Total		3841624	70872	100.000	100.000

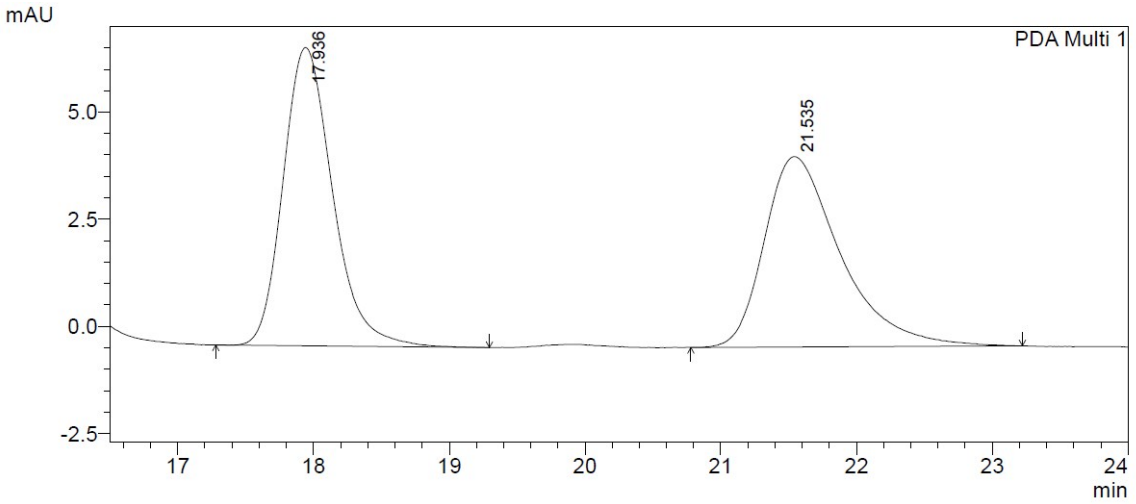


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.245	462687	9368	10.629	12.169
2	28.265	3890385	67611	89.371	87.831
Total		4353072	76979	100.000	100.000

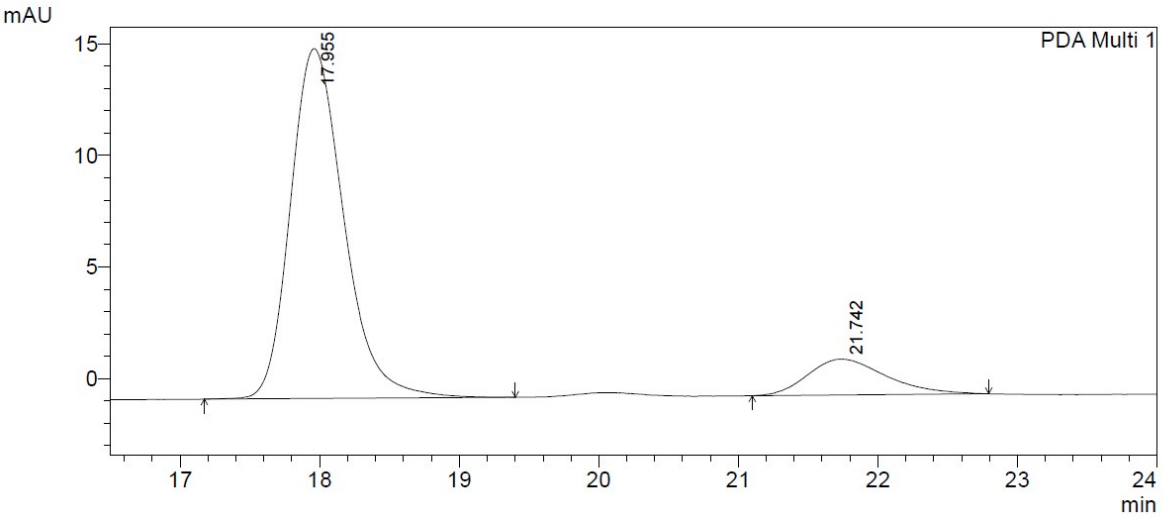


4h



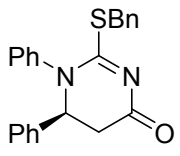
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.936	176492	6964	50.566	61.060
2	21.535	172543	4441	49.434	38.940
Total		349035	11405	100.000	100.000

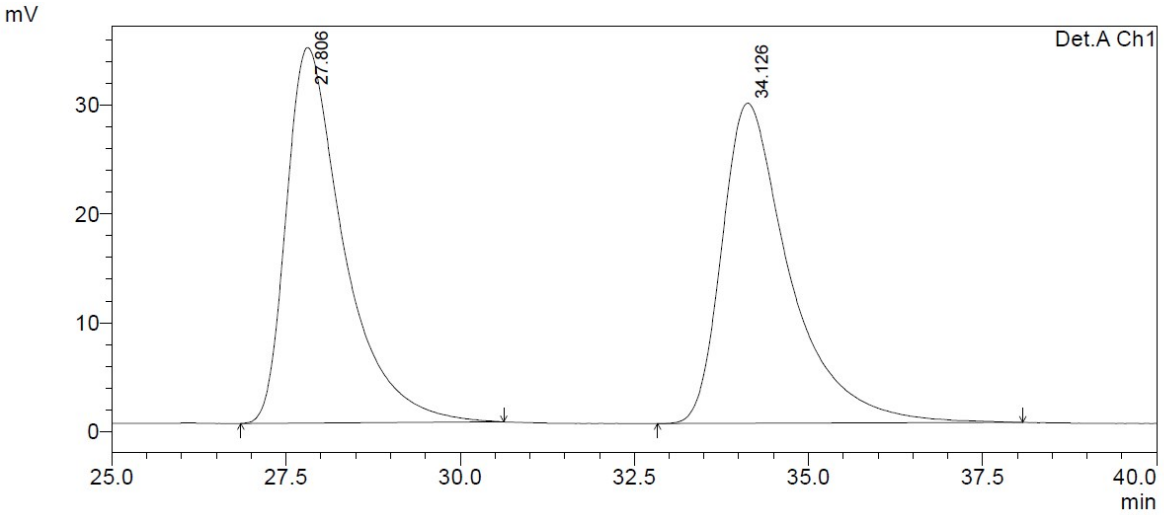


PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.955	417201	15672	86.954	90.713
2	21.742	62595	1604	13.046	9.287
Total		479796	17277	100.000	100.000

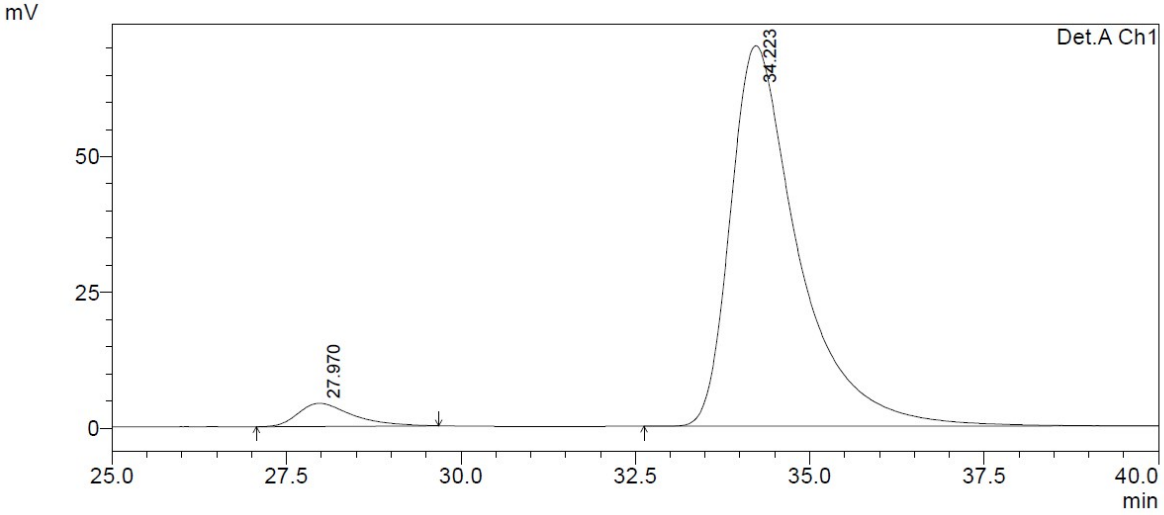


4i



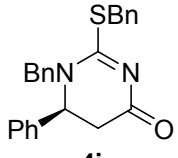
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.806	1978587	34515	49.763	53.974
2	34.126	1997453	29432	50.237	46.026
Total		3976039	63947	100.000	100.000

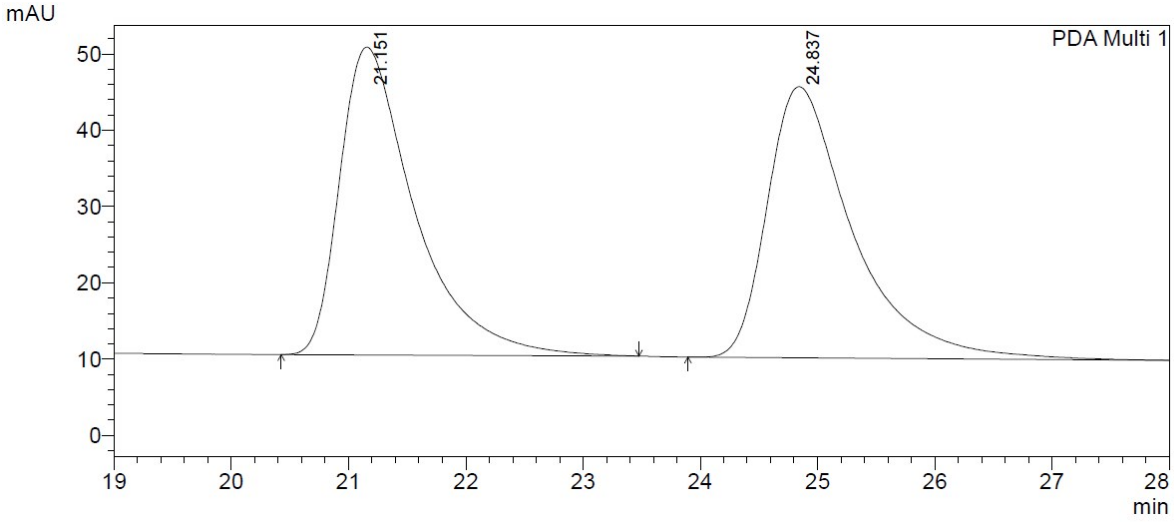


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.970	228949	4257	4.597	5.730
2	34.223	4751820	70049	95.403	94.270
Total		4980769	74306	100.000	100.000

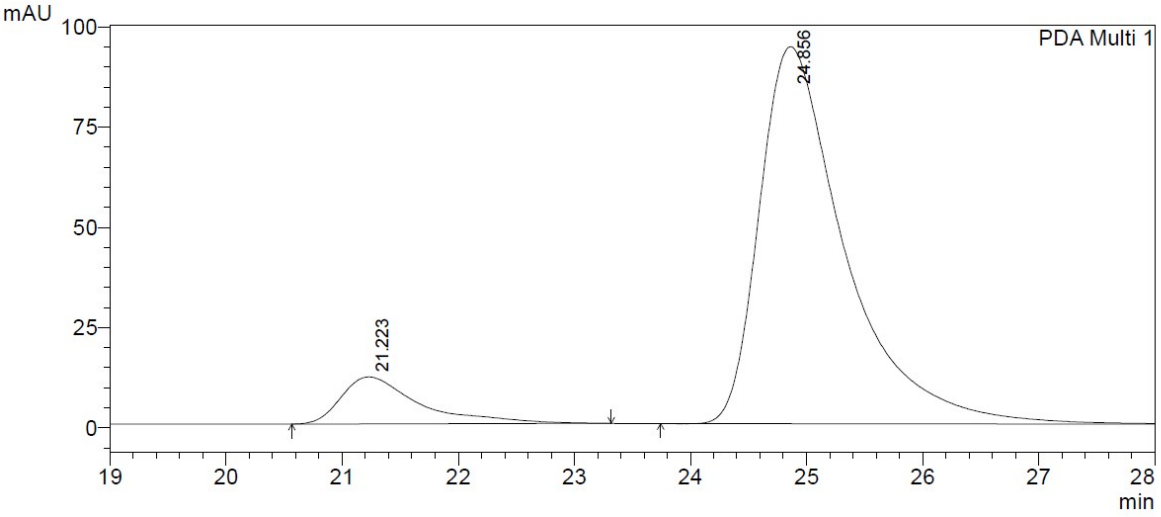


4j



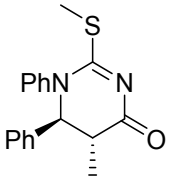
PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.151	1794962	40335	49.857	53.154
2	24.837	1805252	35548	50.143	46.846
Total		3600213	75883	100.000	100.000

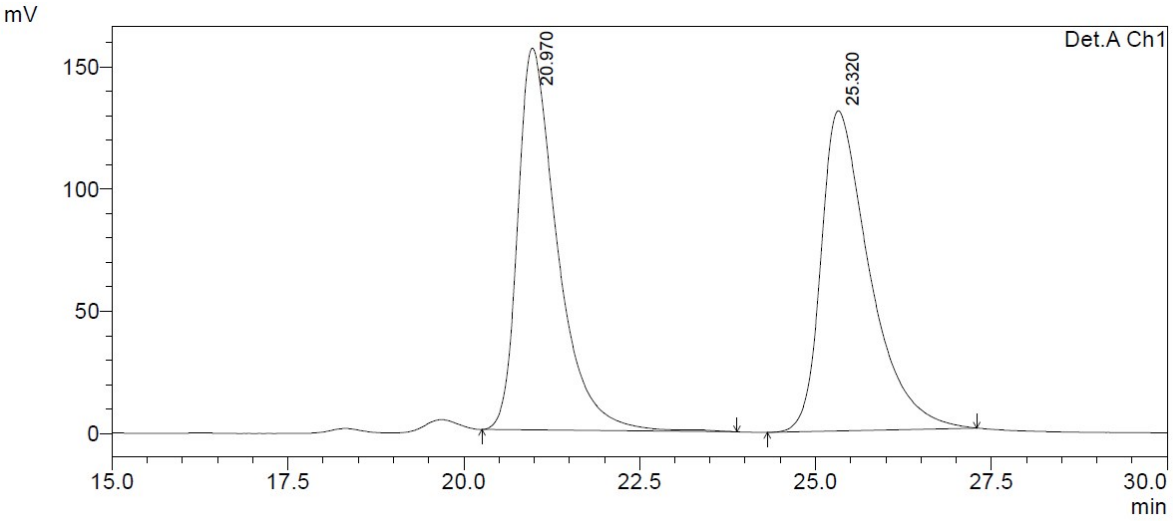


PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.223	548887	11723	10.211	11.086
2	24.856	4826713	94030	89.789	88.914
Total		5375600	105753	100.000	100.000

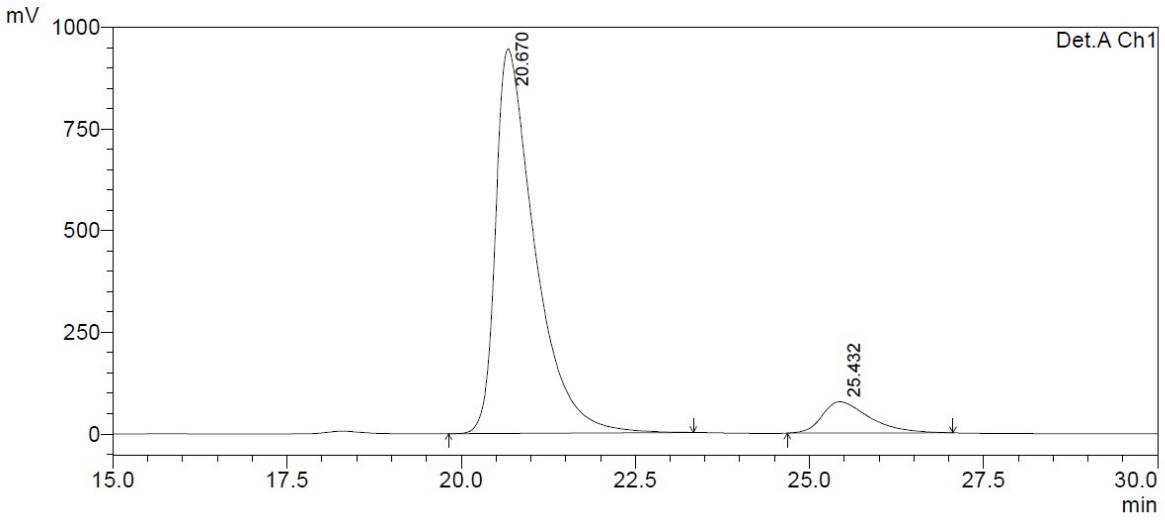


5a



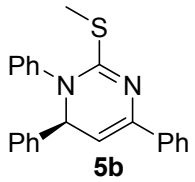
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.970	6123344	156132	49.306	54.393
2	25.320	6295640	130911	50.694	45.607
Total		12418983	287043	100.000	100.000

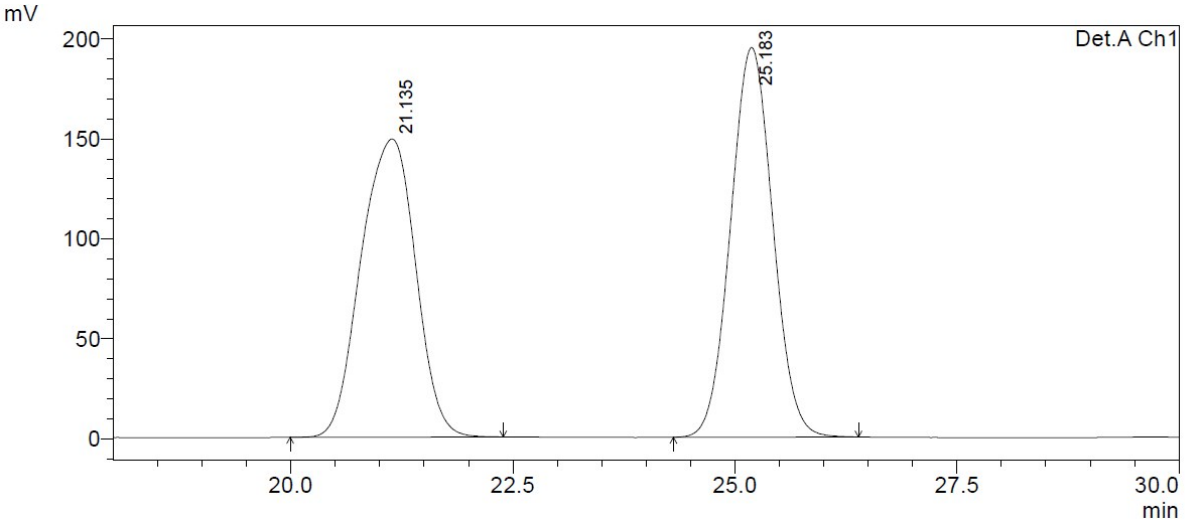


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.670	37014319	945665	91.097	92.495
2	25.432	3617568	76732	8.903	7.505
Total		40631887	1022397	100.000	100.000

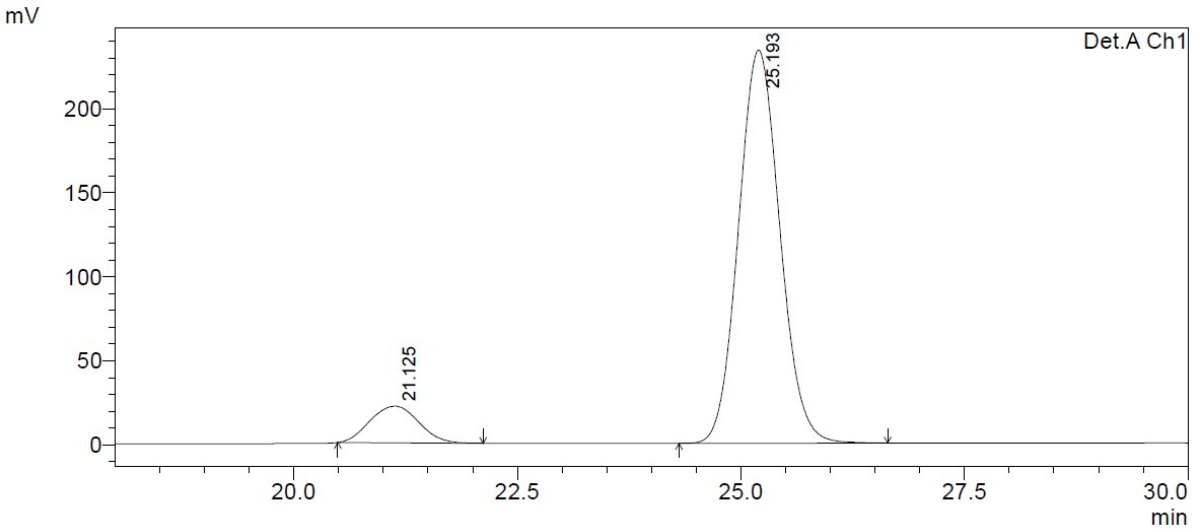


5b



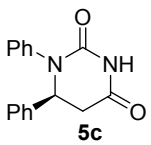
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.135	6388276	149144	49.771	43.353
2	25.183	6447024	194878	50.229	56.647
Total		12835300	344022	100.000	100.000

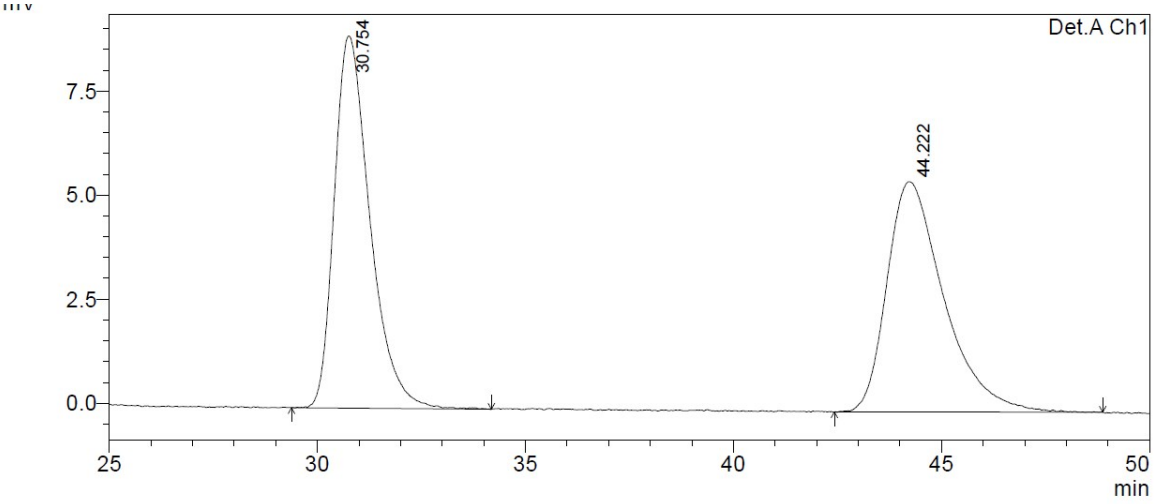


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.125	824440	21794	9.877	8.526
2	25.193	7522766	233841	90.123	91.474
Total		8347207	255636	100.000	100.000

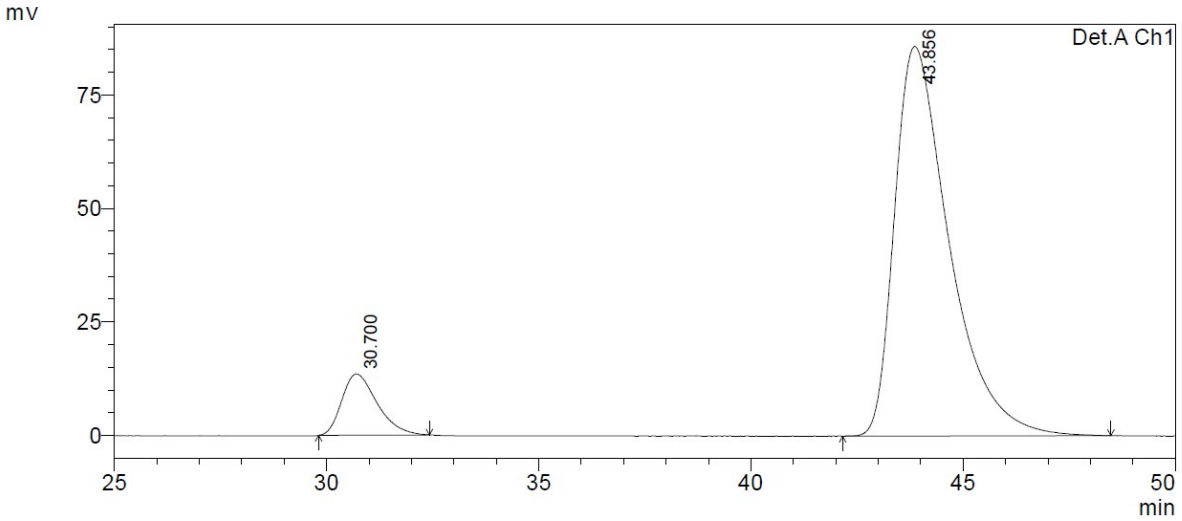


5c



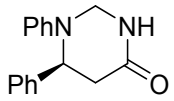
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.754	533775	8937	50.353	61.750
2	44.222	526295	5536	49.647	38.250
Total		1060070	14473	100.000	100.000

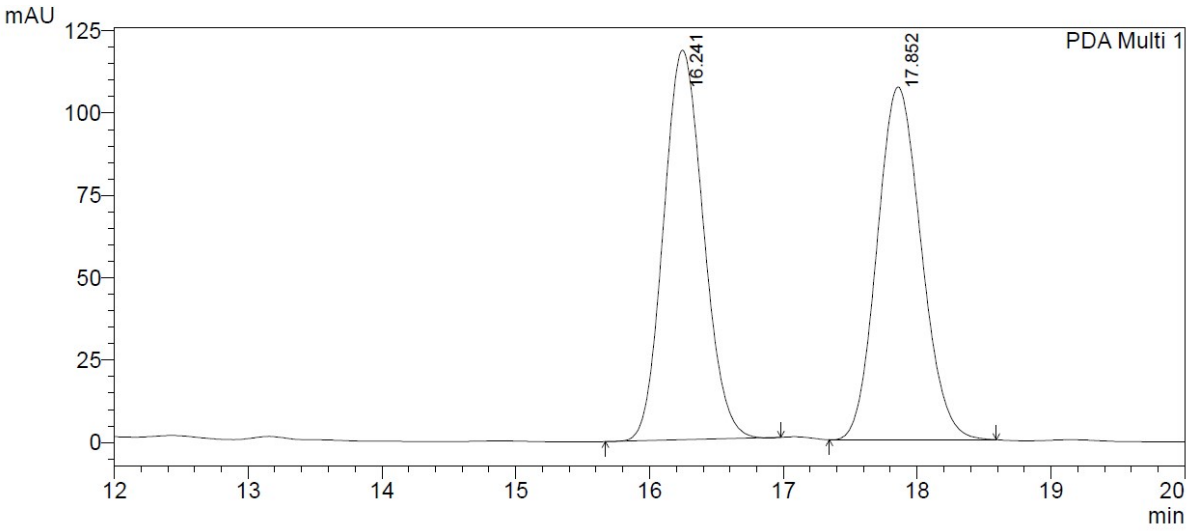


Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	30.700	778357	13482	9.195	13.585
2	43.856	7686495	85761	90.805	86.415
Total		8464852	99243	100.000	100.000

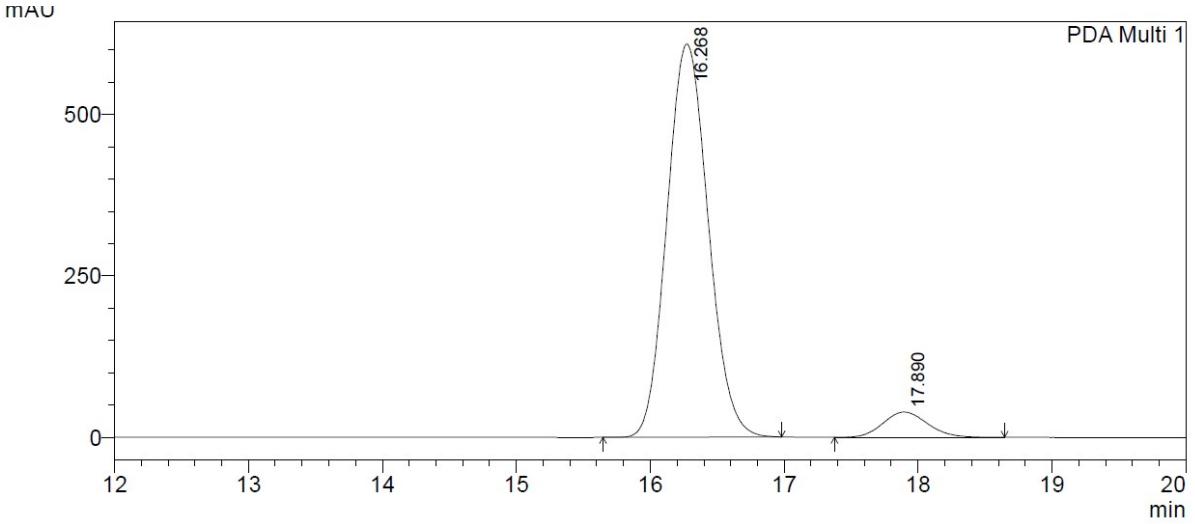


5d



PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.241	2452299	118323	49.967	52.481
2	17.852	2455508	107134	50.033	47.519
Total		4907807	225457	100.000	100.000



PDA Ch1 254nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.268	13048788	608549	93.537	93.965
2	17.890	901634	39082	6.463	6.035
Total		13950421	647631	100.000	100.000