Supporting Information

Synthesis of ^{11}C-Epoxides, Aziridines, and Cyclopropanes from Structurally Modified ^{11}C-Sulfur Ylides

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General Experimental

Commercially available reagents/solvents including **4c** (chloro aldehyde), **4d** (methoxy aledhyde), **4f** (epoxypropylbenzene), **4h** (sulfone), **4k** (piperidone), **4n** (Ts aziridine) were obtained from Ambeed, Combi-Blocks, Enamine, Millipore-Sigma, Oakwood Chemical, Thermo-Scientific, TCI America and were used without further purification. Where necessary, reactions were conducted under an Argon atmosphere in flame dried glassware, PTFE coated stir bars were used for all reactions.

Thin layer chromatography was used to monitor reaction progress (Merck Supelco Silica gel 60 F_{254} glass plates) and was visualised by UV_{254} and heat developed $KMnO_4$ or Vanillin. Flash chromatography was conducted on a Biotage Isolera Prime using Biotage Sfär flash purification columns, eluents were Hexanes:EtOAc mixtures (containing 1% Et_3N for epoxide purifications).

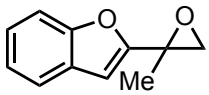
NMR spectra were acquired using a Varian 500 MHz VNMRS (^1H = 500 MHz, ^{13}C = 126 MHz) or a Varian 400 MHz MR (^1H = 400 MHz, ^{13}C = 101 MHz) spectrometer at ambient temperature. TMS free deuterated NMR solvents were obtained from Cambridge Isotope Labs or Thermo-Scientific. Chemical shifts (δ) are reported in ppm relative to TMS and spectra are calibrated using residual solvent peaks. Multiplicity is reported as: s (singlet), d (doublet), t (triplet), q (quadruplet), m (multiplet), br. (broad).

A Shimadzu LC20AD/T LPGEKIT system was used for HPLC analysis, featuring a CBM-20A controller, DGU 20A SR degassing unit, SIL 20AHT injector, CTO-20A column oven, SPD 20A UV/Vis detector and B-FC-1000 gamma radiation detector. Radio-TLC analysis was performed on a Bioscan AR 2000 rTLC scanner (Merck Supelco Silica gel 60 F_{254} glass plates cut to 6.5 × 2.0 cm).

Synthesis of Authentic Standards

Compounds **4a** (naphthalene),[1] **4b** (ester aldehyde),[2, 3] **4e** (pyridine),[4] **4g** (ester ketone),[5] **4i** (benzofuran), **4j** (benzothiophene),[2] **4l** (isatin),[6] **4m** (Ph aziridine),[7, 8] **4o** (quinoline cyclopropane),[9] were prepared according to previously-reported procedures and spectra agreed with values from the prior literature.

(4i) - 2-(2-Methyl-2-oxiranyl)benzofuran (CAS # 64481-23-8)



In a flame dried Schlenk tube held under an $Ar_{(g)}$ atmosphere, dry DMSO (5.0 mL) and dry THF (2.5 mL) were combined with Me_3SI (1.46 g, 7.13 mmol, 1.6 equiv.) and cooled to 0 °C (ice/water). To this solution was carefully added NaH (60% in mineral oil, 258 mg, 6.45 mmol, 1.5 equiv.), following complete addition the reaction mixture was allowed to warm to rt and stirred for 1 h. The reaction mixture was again cooled to 0 °C (ice/water) before 1-(benzofuran-2-yl)ethan-1-one (711 mg, 4.44 mmol, 1.0 equiv.) was introduced as a solution in dry THF (1.5 mL) dropwise over *ca*. 5 mins. The reaction mixture as allowed to warm to rt and stirred for 20 h before quenching with H_2O, diluting with Et_2O (25 mL) and H_2O (50 mL) and transferred to a separatory funnel. The organic fraction was separated and the aqueous further extracted with Et_2O (2 × 25 mL). The pooled extracts were then washed with H_2O (3 × 50 mL) and saturated $NaCl_{(aq.)}$, dried over $MgSO_4$, filtered and concentrated by rotary evaporation under reduced pressure. Residue purified by automated SiO_2 flash column chromatography (Hexanes:Et_3N, 99:1). Compound **4i** obtained as a white solid (627 mg, 81%).

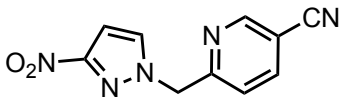
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 (d, *J* = 7.6, 1H), 7.45 (d, *J* = 8.2, 1H), 7.29 (app. t, *J* = 8.0 Hz, 1H), 7.23 (app. t, *J* = 7.6 Hz, 1H), 6.77 (s, 1H), 3.47 (d, *J* = 5.3 Hz, 1H), 3.06 (d, *J* = 5.3 Hz, 1H), 1.81 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 156.0, 154.9, 128.3, 124.7, 123.0, 121.0, 111.4, 105.2, 55.3, 52.5, 19.5.

HRMS (APCI+Q-TOF): *m/z* calcd for $C_{11}H_{11}O_2$: 175.0754; Found 175.0749 $[M+H]^+$.

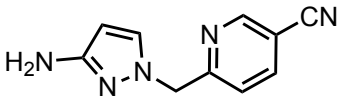
Synthesis of 3p (Apinocaltamide Precursor)

6-((3-nitro-1H-pyrazol-1-yl)methyl)nicotinonitrile (3p-Int-1) (CAS # 1838653-23-8)



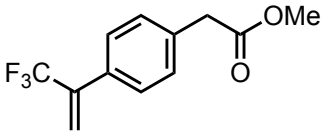
Synthesised as previously reported by Fournier et al. (White solid, 76%).[10]

6-((3-amino-1H-pyrazol-1-yl)methyl)nicotinonitrile (3p-Int-2) (CAS # 1838633-02-5)



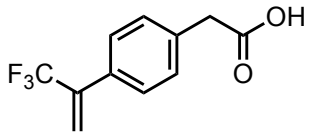
Synthesised as previously reported by Fournier et al. (White solid, 86%).[10]

methyl 2-(4-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)acetate (3p-Int-3)



Synthesised as previously reported by Xiao et al. and Zhang et al. (Pale yellow liquid, 46%).[11, 12]

2-(4-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)acetic acid (3p-Int-4) (CAS # 1994969-25-3)



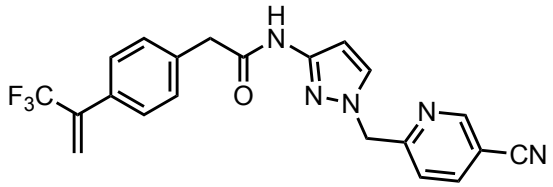
A solution of the ester **3p-Int-3** (498 mg, 2.04 mmol, 1.0 equiv.) in THF (5.5 mL) was cooled to 0 °C (ice/water) and 1.0M $LiOH_{(aq.)}$ (3.0 mL, 3.0 mmol, 1.5 equiv.) added followed by $^{i}PrOH$ (1.1 mL). The reaction mixture was stirred at 0 °C for 10 mins before removal of the cooling bath. After warming to rt the reaction mixture was stirred for 4 h before concentrating by rotary evaporation under reduced pressure. The residue obtained was dissolved in H_2O (7 mL), placed in a separatory funnel and washed with Et_2O (2 × 4 mL). The aqueous layer was then acidified with 1M $HCl_{(aq.)}$ (8 mL) and extracted with EtOAc (4 × 6 mL), the pooled extracts were dried over $MgSO_4$ filtered and the solvent removed by rotary evaporation under reduced pressure. Compound **3p-Int-4** was obtained as a pale orange solid (406 mg, 87%)

^{1}H NMR (500 MHz, $CDCl_3$) δ 10.16 (br. s, 1H), 7.43 (d, *J* = 7.9 Hz, 2H), 7.31 (d, *J* = 7.9 Hz, 2H), 5.96 (s, 1H), 5.77 (s, 1H), 3.68 (s, 2H)

^{13}C NMR (126 MHz, $CDCl_3$) δ 177.53, 138.64 (q, *J* = 30.5 Hz), 134.22, 132.89, 129.77, 127.79, 123.42 (q, *J* = 274.2 Hz), 120.64 (q, *J* = 5.7 Hz), 40.77.

HRMS (EI+): *m/z* calcd for $C_{11}H_9F_3O_2$: 230.0555; Found 230.0562 $[M]^+$.

***N*-(1-((5-cyanopyridin-2-yl)methyl)-1H-pyrazol-3-yl)-2-(4-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)acetamide (3p)**



In a round bottomed flask 2-(4-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)acetic acid **(3p-Int-4)** (360 mg, 1.56 mmol, 1.0 equiv.) was dissolved in dry MeCN (12.0 mL), HATU (629 mg, 1.66 mmol, 1.05 equiv.) was then added followed by DIPEA (0.59 mL, 3.4 mmol, 2.2 equiv.). The reaction mixture was stirred for 5 mins at rt before the addition of 6-((3-amino-1H-pyrazol-1-yl)methyl)nicotinonitrile **(3p-Int-2)** (311 mg, 1.56 mmol, 1.0 equiv.). After 19 h the solvent was removed by rotary evaporation under reduced pressure and the residue dissolved in EtOAc (30 mL) and transferred to a separatory funnel. The organic fraction was washed with 0.1 M $HCl_{(aq.)}$ (20 mL) followed by saturated $NaHCO_{3\ (aq.)}$ (20 mL) and finally H_2O (20 mL). The organic fraction was then dried over $MgSO_4$, filtered and concentrated by rotary evaporation under reduced pressure. The residue was then purified by automated SiO_2 flash column chromatography (Hexanes:EtOAc, 100:0 → 30:70). Compound **3p** was isolated as a white solid (289 mg, 52%).

^{1}H NMR (500 MHz, DMSO) δ 10.74 (s, 1H), 8.98 (d, *J* = 2.2 Hz, 1H), 8.28 (dd, *J* = 8.2, 2.2 Hz, 1H), 7.79 (d, *J* = 1.8 Hz, 1H), 7.44 (d, *J* = 7.9 Hz, 2H), 7.38 (d, *J* = 7.9 Hz, 2H), 7.18 (d, *J* = 8.2 Hz, 1H), 6.53 (d, *J* = 1.8 Hz, 1H), 6.05 (app. d, *J* = 5.3 Hz, 2H), 5.44 (s, 2H), 3.62 (s, 2H).

^{13}C NMR (126 MHz, DMSO) δ 167.9, 161.3, 152.2, 148.0, 140.9, 137.2, 136.7 (q, *J* = 29.3 Hz), 131.9, 130.9, 129.6, 126.9, 123.4 (q, *J* = 274.2 Hz), 121.9 (q, *J* = 5.7 Hz), 121.7, 116.9, 107.9, 97.1, 56.1, 42.1

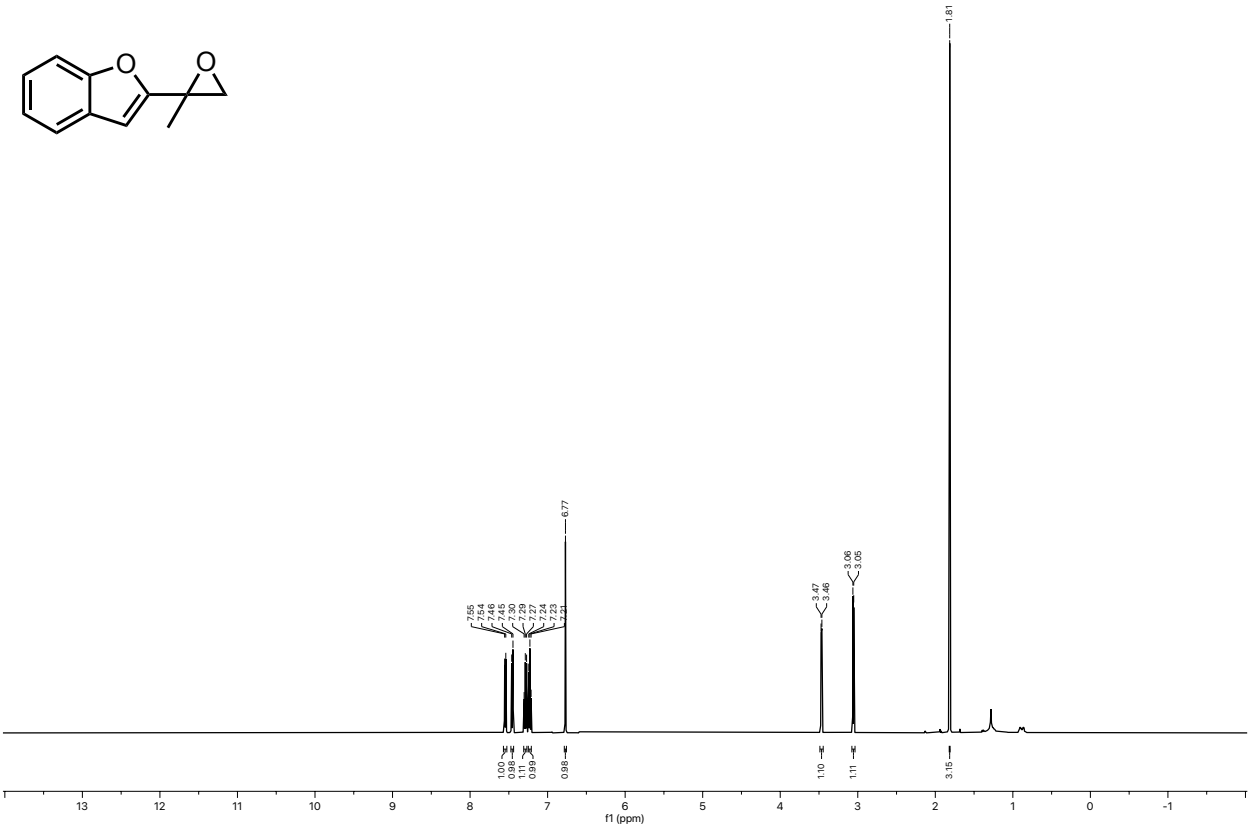
^{19}F NMR (470 MHz, DMSO) δ -63.32.

HRMS (ESI-TOF): *m/z* calcd for $C_{21}H_{17}F_3N_5O$: 412.1380; Found 412.1379 $[M+H]^+$.

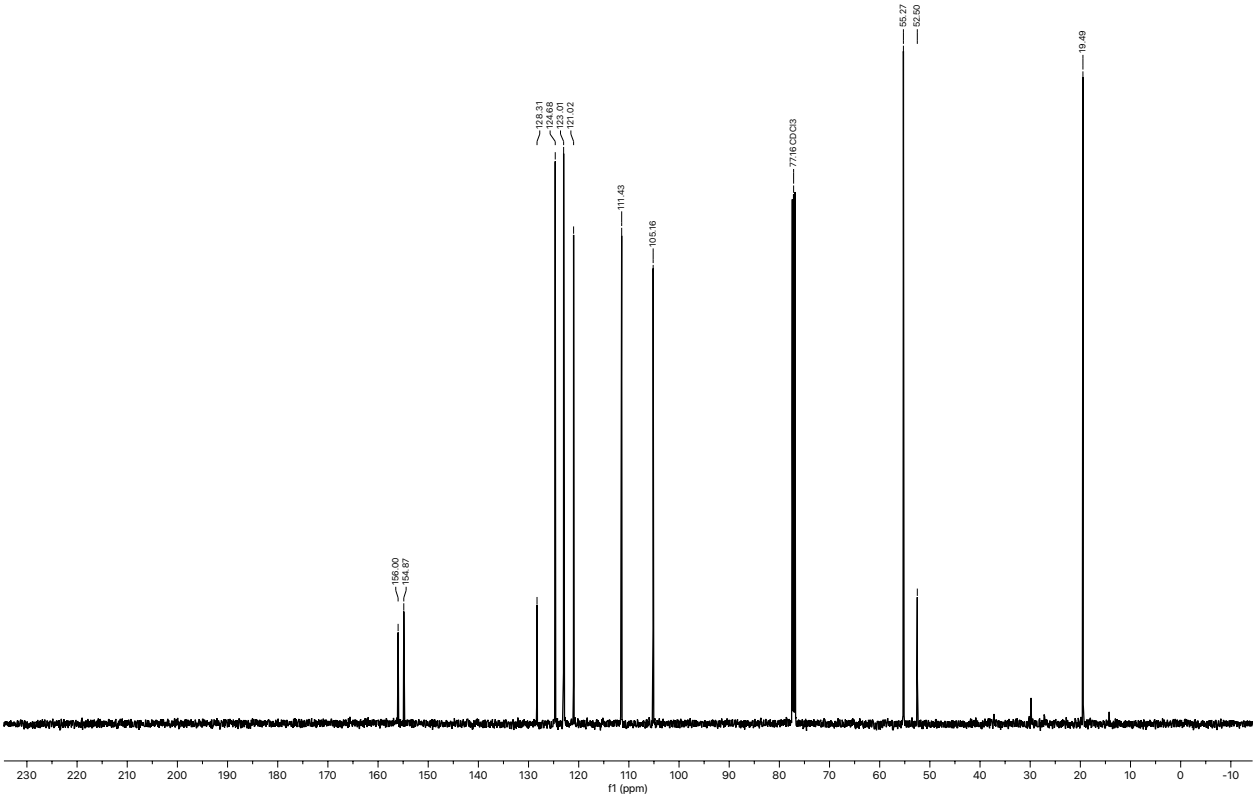
NMR Spectra

(4i) - 2-(2-Methyl-2-oxiranyl)benzofuran (CAS # 64481-23-8)

^{1}H NMR

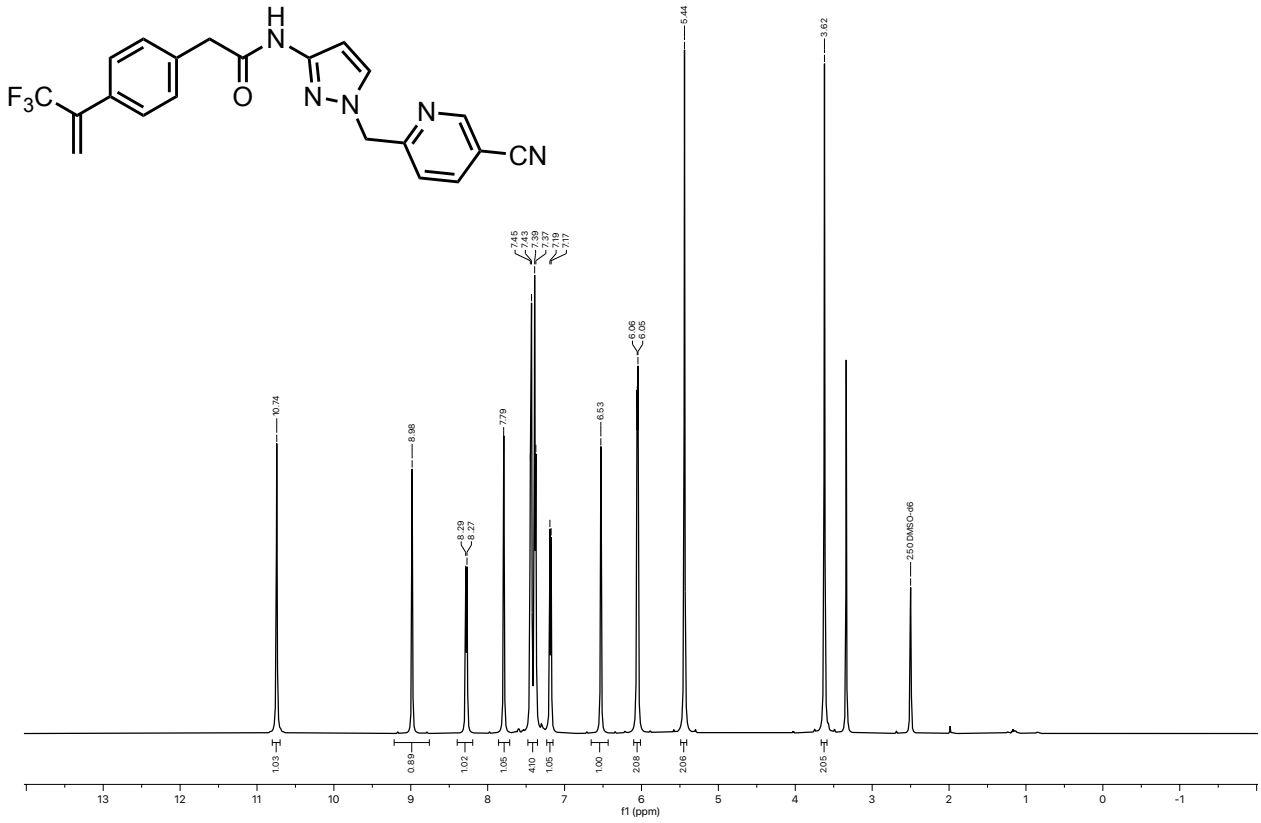


^{13}C NMR

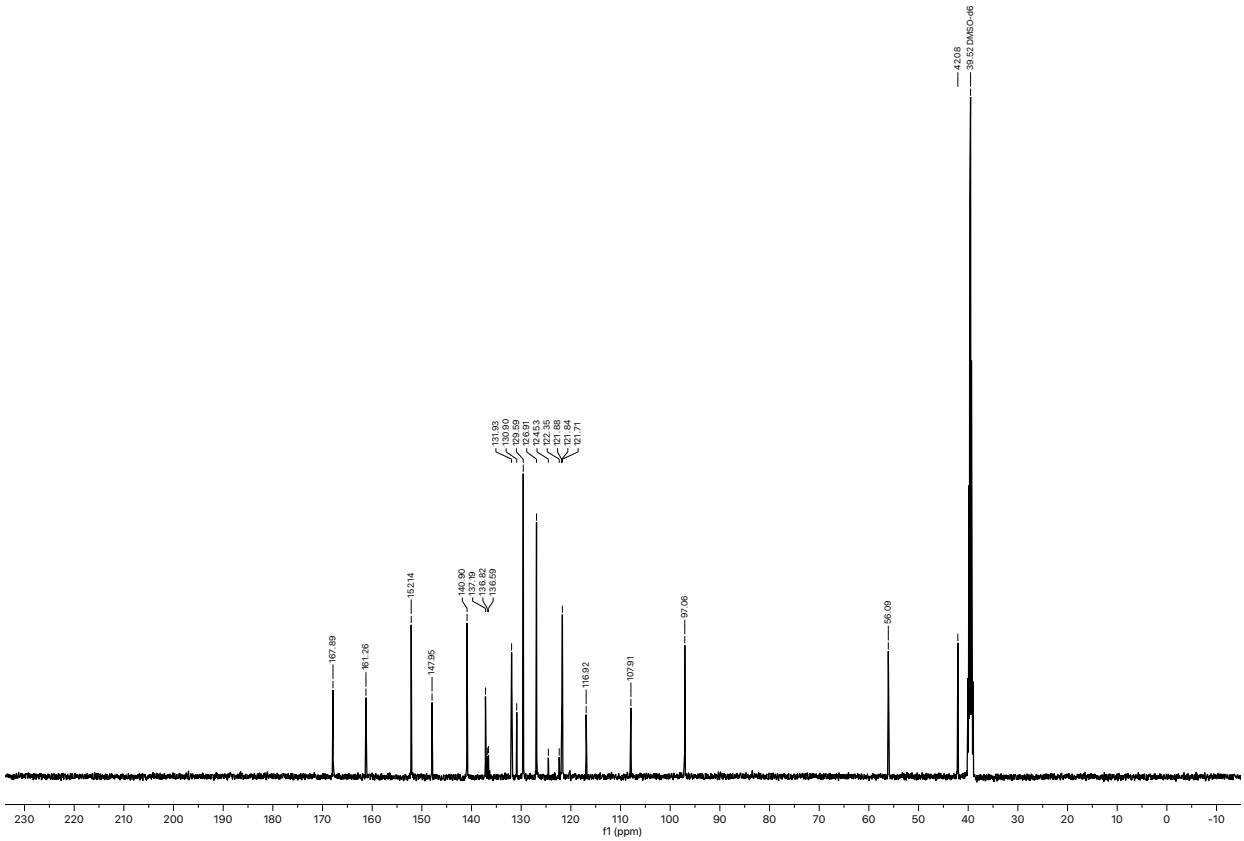


_N_-(1-((5-cyanopyridin-2-yl)methyl)-1H-pyrazol-3-yl)-2-(4-(3,3,3-trifluoroprop-1-en-2-yl)phenyl)acetamide (3p)

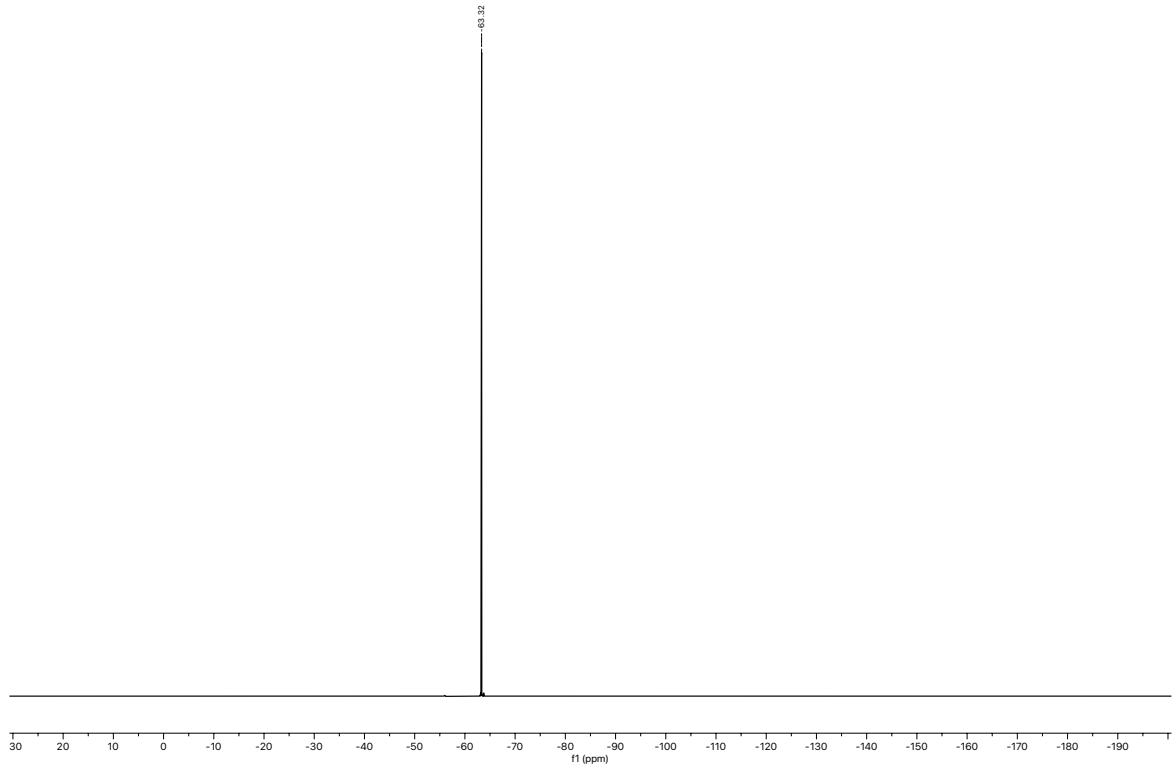
^{1}H NMR



^{13}C NMR



^{19}F NMR


-63.32
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)

Radiochemistry

Preparation of [^{11}C]MeI and [^{11}C]MeOTf

Using a GE TRACERlab $FX_{C\text{-}Pro}$ synthesis module. [^{11}C]CO_2 was produced *via* the $^{14}N(p,\alpha)^{11}C$ nuclear reaction using a GE PETtrace cyclotron in a $^{14}N_2$ + 0.5% O_2 gas target (16.4 MeV proton irradiation at 60 μA for 2 mins or 15 mins). [^{11}C]CO_2 from the target was mixed with H_2 over a preheated nickel oven at 400 °C for conversion to [^{11}C]CH_4. [^{11}C]CH_3I was formed by standard procedures with iodine in a circulation loop and converted to [^{11}C]CH_3OTf by passage through a AgOTf column where applicable. The ^{11}C-methylating reagent was then delivered to a PTFE/silicone septa-equipped vial containing the relevant solvent (1.8 mL) or sulfide solution (10-20 μmol., 1.8 mL) in the case of [^{11}C]CH_3OTf, and this solution was then used in the manual protocol. In the case of the automated protocol, [^{11}C]CH_3I was transferred directly to a GE TRACERlab $FX2_M$ synthesis module through the transfer line.

Manual Radiochemistry General Procedure

The relevant sulfide (*20 μmol*) and AgOTf (*20 μmol*) were loaded into a 4 mL glass vial with PTFE/silicone septa cap and magnetic stir bar. To this was then added a solution of [^{11}C]CH_3I in PhCl (*100-300 μL, depending on amount of activity required*) produced as described above, and additional PhCl if needed (to achieve 300 μL final volume). The reaction vial was then sealed and placed in a preheated heating block at 60 °C with stirring for 5 mins, following this a 200 μL aliquot was transferred to a second 4 mL PTFE/silicone septa capped vial containing the relevant base (*20-40 μmol, 1-2 equiv*.) and a magnetic stir bar. The relevant starting material (20 μmol, 1 equiv.) was then introduced as a solution in MeCN (*200 μL*) and the vial placed in a preheated heating block with stirring (time and temperature as described), for all epoxides, aziridines and cyclopropanes the reaction mixtures were analysed as detailed below. For the formation of diol compound **5**, the reaction vial was briefly cooled (1 min) by removal from the heating block before the addition of H_2O (*8 μL, 400 μmol*) and trifluoroacetic acid (*12 μL, 160 μmol*) and then returned to a preheated heating block at 120 °C with stirring for 5 mins. Analysis of **5** was then performed in an identical fashion to all other compounds (see below).

rHPLC and rTLC Analysis

Following reaction completion, the vial was removed from the heating block and cooled briefly (1 min) before filtration through a HPLC syringe filter (PTFE, 0.2 μm, 13 mm). The solution obtained was then analysed directly by rTLC (SiO_2 glass backed plates 6.5 × 2.0 cm, eluent Hexanes:EtOAc [1:1]) and an aliquot (200 μL) placed in a HPLC vial, if required internal standard was added as a solution in MeCN (10-50 μL), and the sample analysed by rHPLC.

HPLC conditions

The value for **X** is specified for each substrate.

Condition A

UV/Vis Detector:	220 nm
Column:	HydroRP 250 × 4.6 mm
Solvent A:	10 mM $NH_4OAc_{(aq.)}$ at pH 6.5
Solvent B:	MeCN
0 – 8 min	X% B
8 – 8.5 min	X% to 75% B
8.5 – 13 min	75% B
13 – 13.5 min	75% B to X% B
13.5 - 15 min	X% B

Condition B

UV/Vis Detector:	220 nm
Column:	HydroRP 250 × 4.6 mm
Solvent A:	10 mM $NH_4OAc_{(aq.)}$ at pH 6.5
Solvent B:	MeCN
0 – 15 min	X% B
15 – 15.5 min	X% to 75% B
15.5 – 19 min	75% B
19 – 19.5 min	75% B to X% B
19.5 - 23 min	X% B

Condition C

UV/Vis Detector:	220 nm
Column:	HydroRP 250 × 4.6 mm
Solvent A:	10 mM $NH_4OAc_{(aq.)}$ at pH 6.5
Solvent B:	MeCN
0 – 12 min	X% B
12 – 12.5 min	X% to 75% B
12.5 – 16 min	75% B
16 – 16.5 min	75% B to X% B
16.5 - 20 min	X% B

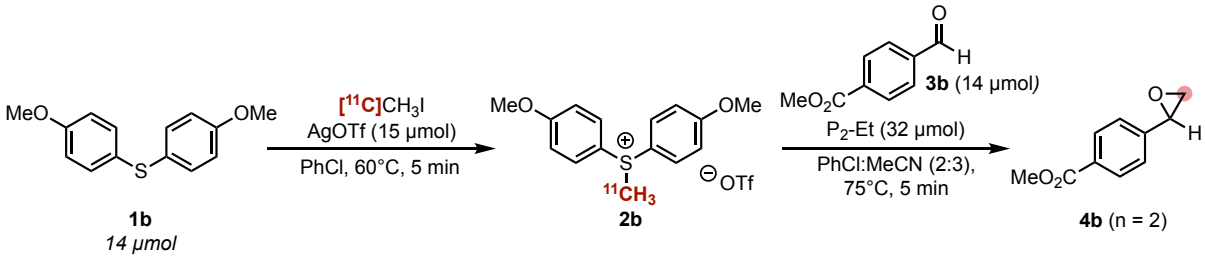
Condition D

UV/Vis Detector:	220 nm
Column:	HydroRP 250 × 4.6 mm
Solvent A:	10 mM $NH_4OAc_{(aq.)}$ at pH 6.5
Solvent B:	MeCN
0 – 8 min	X% B
8 – 8.5 min	X% to 75% B
8.5 – 18 min	75% B
18 – 18.5 min	75% B to X% B
18.5 - 20 min	X% B

Condition E

UV/Vis Detector:	220 nm
Column:	HydroRP 250 × 4.6 mm
Solvent A:	10 mM $NH_4OAc_{(aq.)}$ at pH 6.5
Solvent B:	MeCN
0 – 20 minutes:	X% B
20 – 20.5 minutes:	X% to 75% B
20.5 – 24 minutes:	75% B
24 – 24.5 minutes:	75% B to X% B
24.5 - 28 minutes:	X% B

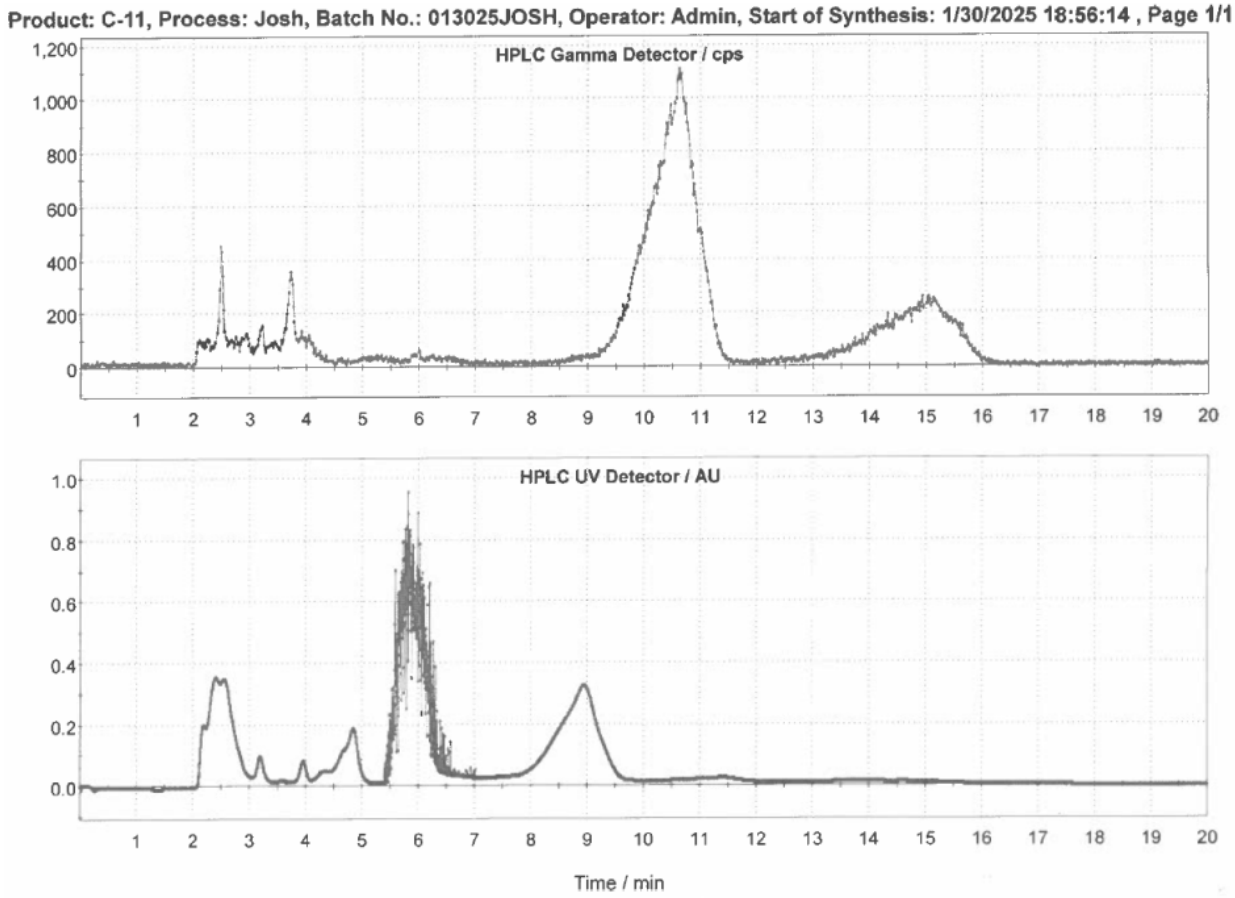
Automated Radiochemistry Procedure



Automation was performed on a GE TRACERlab FX2 M module (see above for [^{11}C]MeI synthesis, see below for module configuration). Into an oven dried glass reactor AgOTf (3.9 mg, 15 µmol, 1 equiv.) and a magnetic stir bar was loaded followed by the addition of bis(4-methoxyphenyl)sulfane **1b** (3.5 mg, 14 µmol, 1 equiv.) as a solution in dry PhCl (200 µL). The reactor was then attached to the module, [^{11}C]CH_3I was then delivered *via* a transfer line and the open valves closed and heated to 60 °C with stirring for 5 mins. The reaction mixture was then cooled to 40 °C with a stream of N_2 before the addition of phosphazene base P_2-Et (11 mg, 32 µmol, 2 equiv.) as a solution in dry MeCN (150 µL) from a 1.5 mL total recovery glass snap top microvial which had been attached in place of valve 1. The reaction mixture was stirred for 15 s before the addition of methyl 4-formylbenzoate **3b** (2.3 mg, 14 µmol, 1 equiv.) as a solution in dry MeCN (150 µL) *via* vial 2 before again closing the open valves and heating to 75 °C for 5 mins. The reaction mixture was cooled to 0 °C in a stream of N_2 before the addition of 0.7 mL of HPLC eluent (10 mM $NH_4OAc_{(aq)}$ pH 6.5, 40% MeCN), stirred for *ca.* 1 min and the majority transferred to an HPLC injection loop for purification, a small fraction was allowed to run to waste and collected for RCC analysis by HPLC (condition D). Synthesis time of 19 mins. Purification was carried out with a Prodigy 10µ ODS-Prep (250 × 10 mm, 10 µ) semi-preparative HPLC column using an isocratic eluent (10 mM $NH_4OAc_{(aq)}$ pH 6.5, 40% MeCN) at a flow rate of 6 mL min^{-1}. The peak of interest (retention time *ca.* 10.5 mins) was isolated, measured for activity and analysed by HPLC (condition A).

Molar activity was calculated based upon the UV peak corresponding to the radioactivity peak of interest. As no significant absorption was observed, discovering the limit of detection of compound **4b** by UV was completed using a range of standard solutions (0.1 mg mL^{-1} to 0.0001 mg mL^{-1}). Molar activity for **4b** was found to be >710 mCi µmol^{-1}.

Automation Crude Semi-Preparative Purification

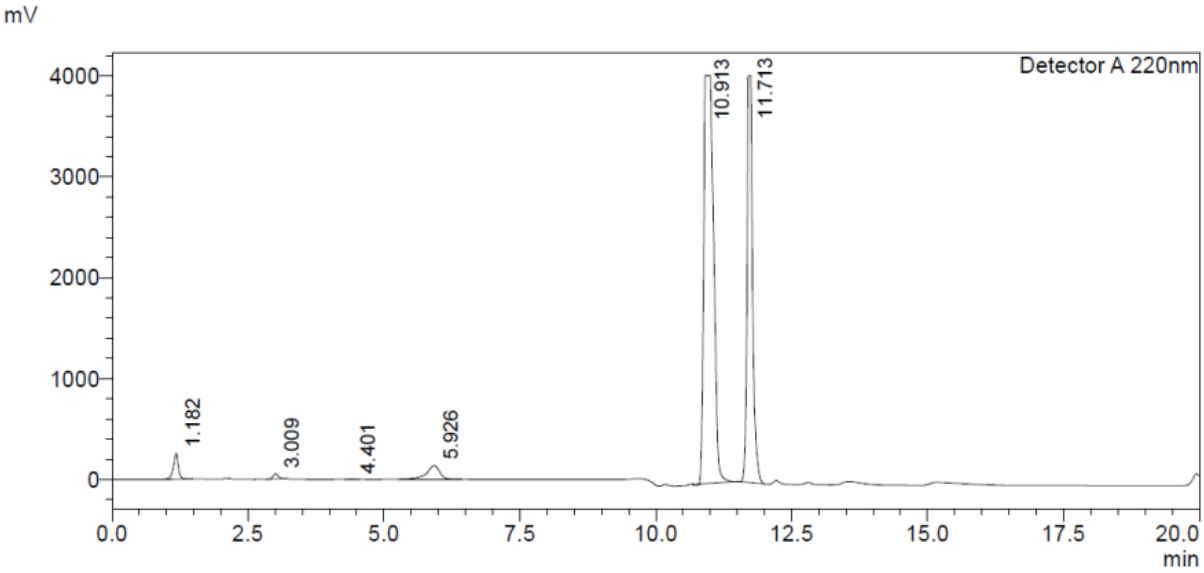


Details:

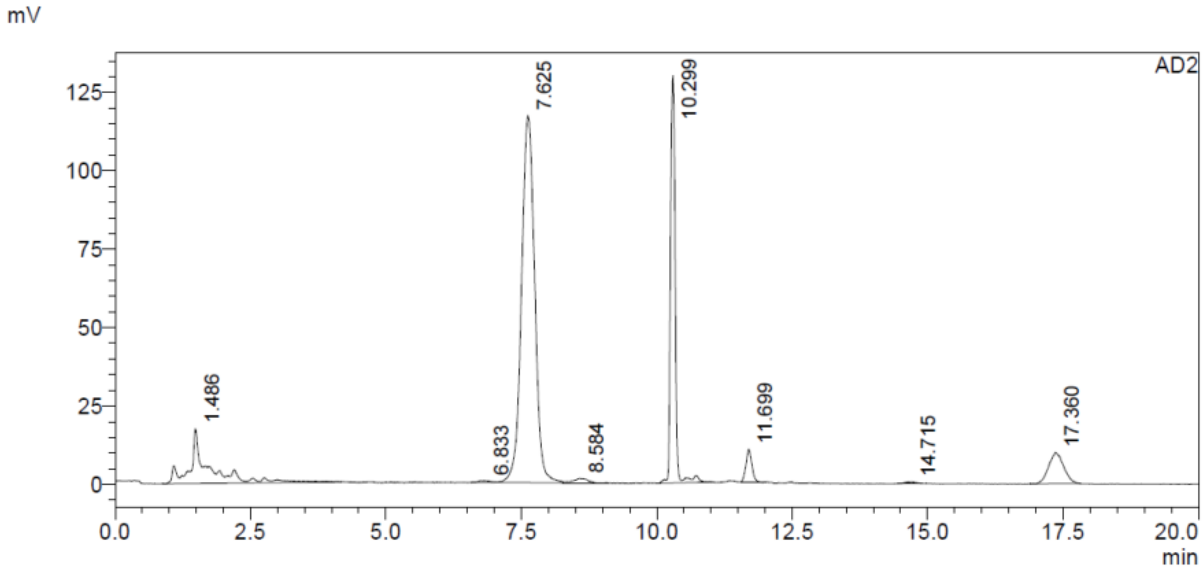
Prodigy 10µ ODS-Prep (250 × 10 mm, 10 µ) HPLC column using an isocratic eluent (10 mM $NH_4OAc_{(aq)}$ pH 6.5, 40% MeCN) at a flow rate of 6 mL min^{-1}, peak at ~10.5 min (gamma) isolated.

Automation - Crude Analytical Data

UV Trace



Gamma Trace

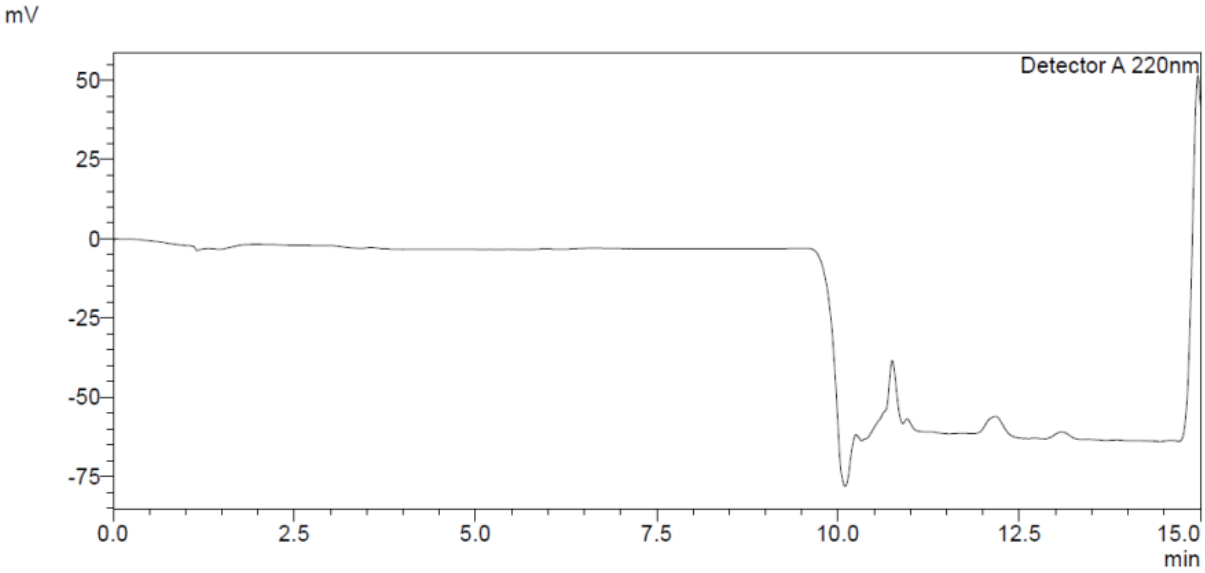


HPLC condition **D** (X = 30), injection volume 5 µL, product peak at 7.625 min (gamma),

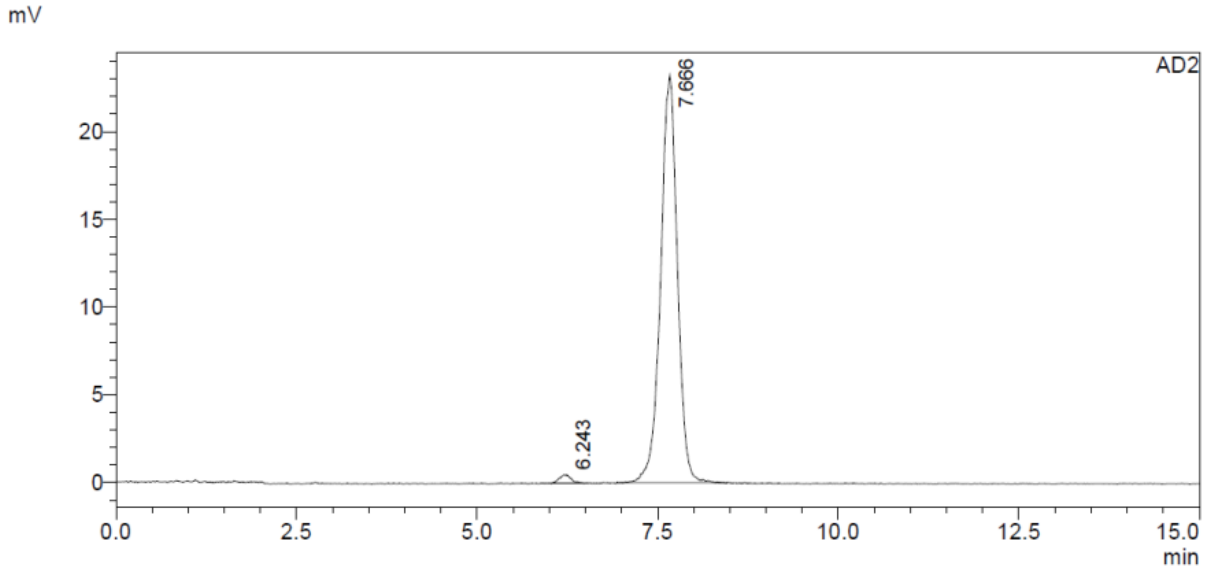
Radiochemical Conversion (non-decay corrected): **57%.**

Automation Isolated Sample (*without internal standard*)

UV Trace



Gamma Trace

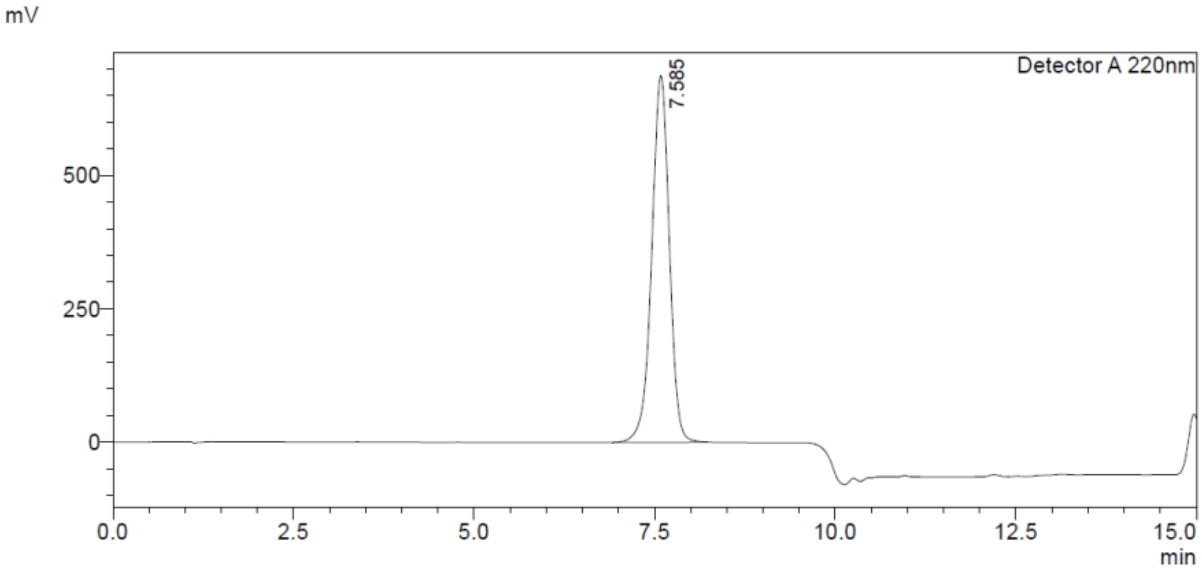


HPLC condition **A** (X = 30), injection volume 10 µL, peak at 7.666 min (gamma)

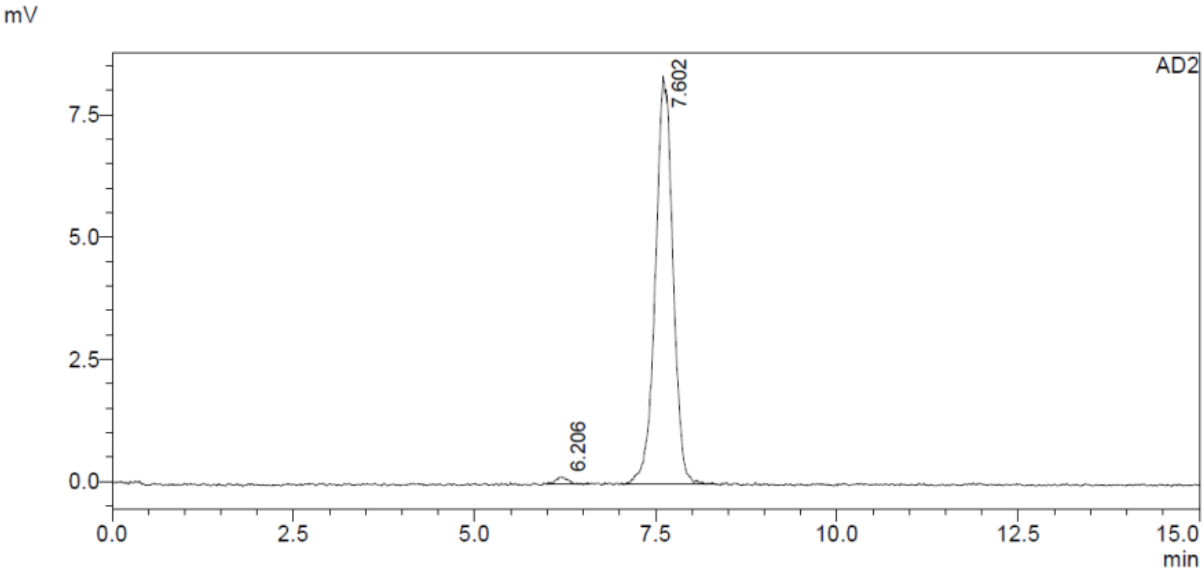
Radiochemical Purity: **99%.**

Automation Isolated Sample (with internal standard)

UV detector

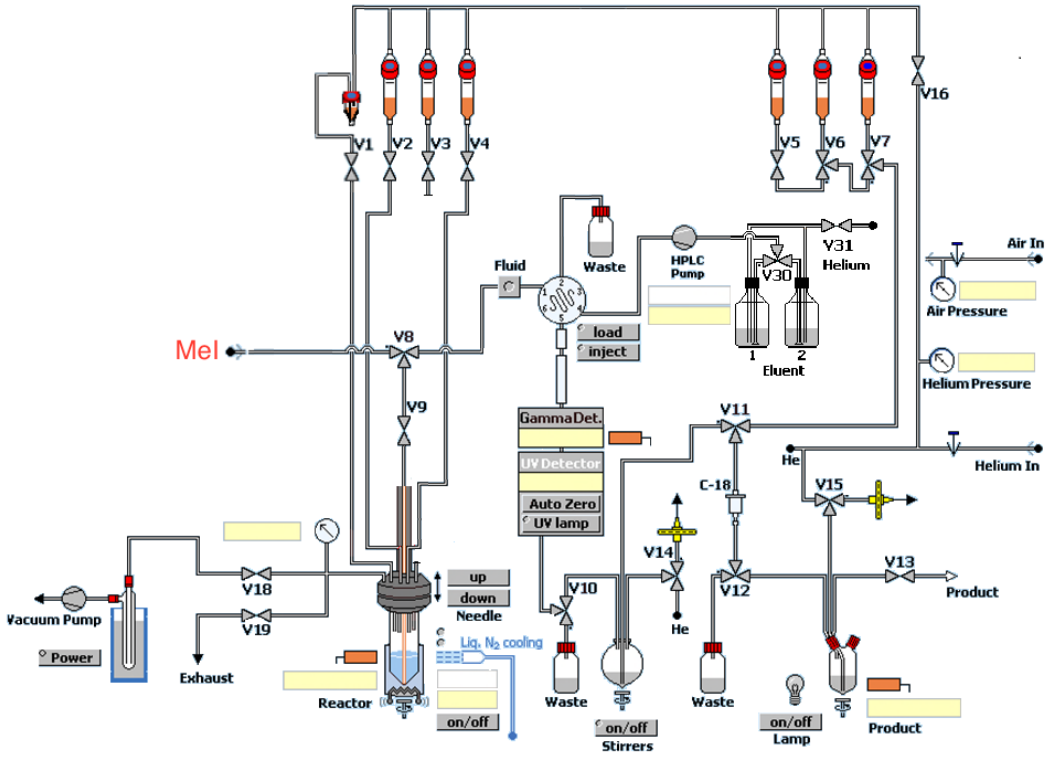


Gamma Trace



HPLC condition **A** (X = 30), injection volume 10 µL, peak at 7.585 min (UV) and 7.602 min (gamma), Radiochemical Purity: **99%.**

Automation GE TRACERlab FX2$_M$ Timelist and Configuration



Time List: Methylation start up J 2/11/2025

Time		Device		Value	Dur.	Comme
0	Set	Power	=	On		
0.1	Set	HPLC Pump Flow Set Point	=	0		
0.3	Set	V19	=	Open		
0.4	Set	Process Control	=	Show message and wait		Waiting for precursor
t1+0	Set	Needle Reactor 1	=	Down		
t1+0.1	Set	V19	=	Close		

Time List: Methylation heat 2/11/2025

Time		Device		Value	Dur.	Comme
0	Set	Power	=	On		
0.2	Set	Stirrer	=	On		
0.4	Set	Process Control	=	Show message and wait		waiting activity
t1+0	Set	V09	=	Open		
t1+0.1	Set	V19	=	Open		
t1+3'40	Set	Needle Reactor 1	=	Up		
t1+3'40	Set	V09	=	Close		
t1+3'41	Set	V19	=	Close		
t1+3'42	Set	V08	=	b (right)		
t1+3'42	Set	Temp. Setpoint Reactor	=	60		
t1+3'42	Wait	Temp. Regulator Status Reactor	=	Temp. OK		
t2+5'2	Set	Temp. Setpoint Reactor	=	40		
t2+5'2	Wait	Temp. Regulator Status Reactor	=	Temp. OK		
t3+0	Set	V19	=	Open		
t3+0.1	Set	V01 Vial1	=	Open		
t3+0.2	Set	V16	=	Open		
t3+20.3	Set	V01 Vial1	=	Close		
t3+20.4	Set	V16	=	Close		
t3+35	Set	V02 Vial2	=	Open		
t3+35.1	Set	V16	=	Open		
t3+55	Set	V02 Vial2	=	Close		
t3+55.1	Set	V16	=	Close		
t3+55.2	Set	V19	=	Close		
t3+55.4	Set	Temp. Setpoint Reactor	=	75		
t3+55.5	Wait	Temp. Regulator Status Reactor	=	Temp. OK		
t4+5'0	Set	Temp. Setpoint Reactor	=	30		
t4+5'1	Wait	Temp. Regulator Status Reactor	=	Temp. OK		
t5+0	Set	V19	=	Open		

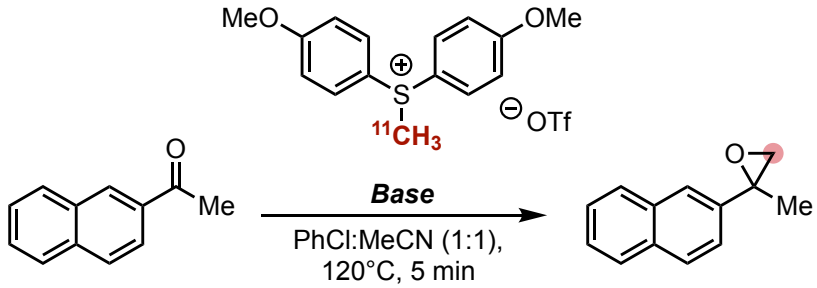
Time List: HPLC reactor 2/11/2025

Time		Device		Value	Dur.	Comme
0.1	Set	HPLC Pump Flow Set Point	=	6		
0.2	Set	V04 Vial4	=	Open		Dilution with HPLC eluent
0.3	Set	V16	=	Open		
30	Set	V04 Vial4	=	Close		
30.1	Set	V16	=	Close		
1'31	Set	V04 Vial4	=	Open		
1'31	Set	V16	=	Open		
1'32	Set	V09	=	Open		
1'32	Set	V19	=	Close		
1'33	Set	Needle Reactor 1	=	Down		Transfer into HPLC loop
1'33	Set	HPLC UV Detector Auto Zero	=	On		
1'33	Set	HPLC UV Detector Auto Zero	=	Off		
1'34	Wait	Fluid Detector	=	ON		
t1+2	Wait	Fluid Detector	=	Off		
t2+0.1	Set	Load/Inject Valve	=	Inject		
t2+20.3	Set	V04 Vial4	=	Close		
t2+20.4	Set	V16	=	Close		
t2+20.5	Set	Needle Reactor 1	=	Up		
t2+30	Wait	Chromatography Peak Detector	=	Start of Peak		Peak Cut
t3+20	Wait	Chromatography Peak Detector	=	End of Peak		
t4+20.1	Set	Load/Inject Valve	=	Load		

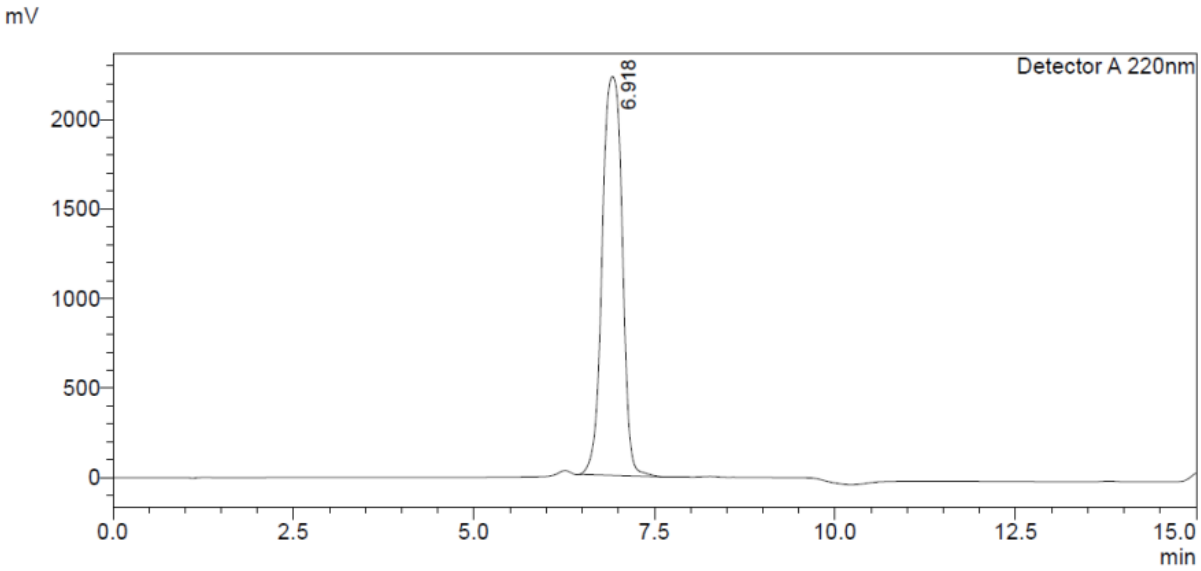
Analytical Data for Manual Substrates

4a

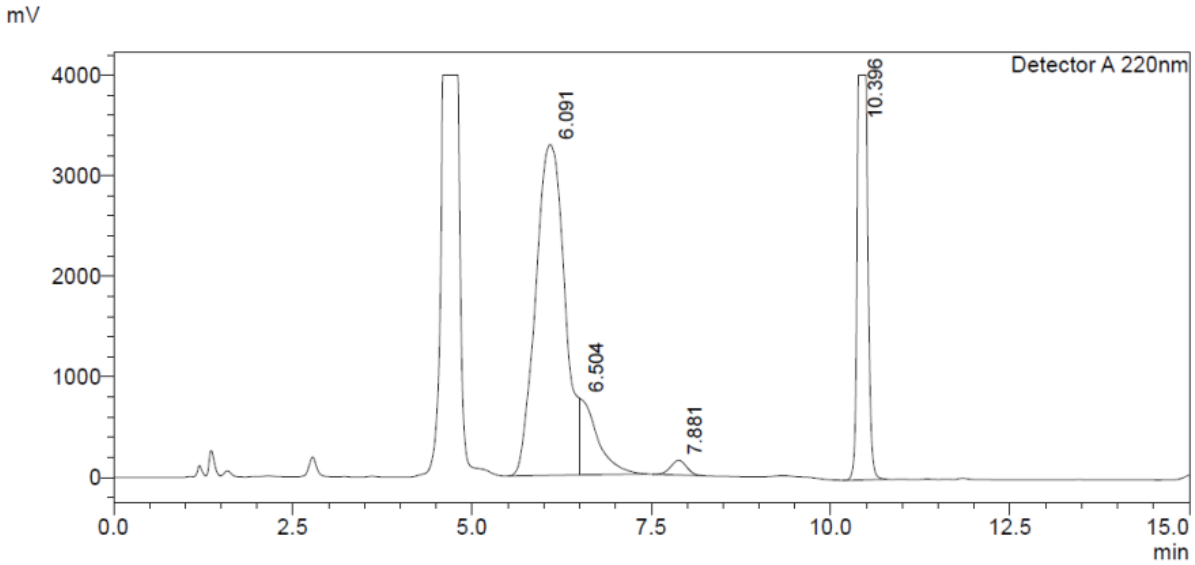
(Base = 2 eq. KOtBu)



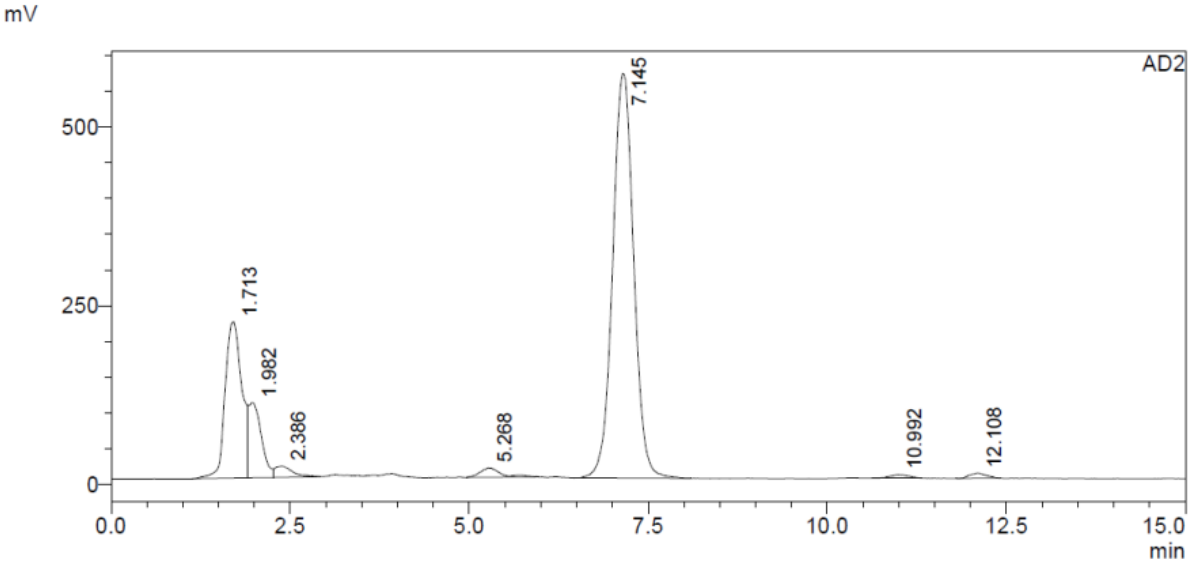
Standard - UV Detector



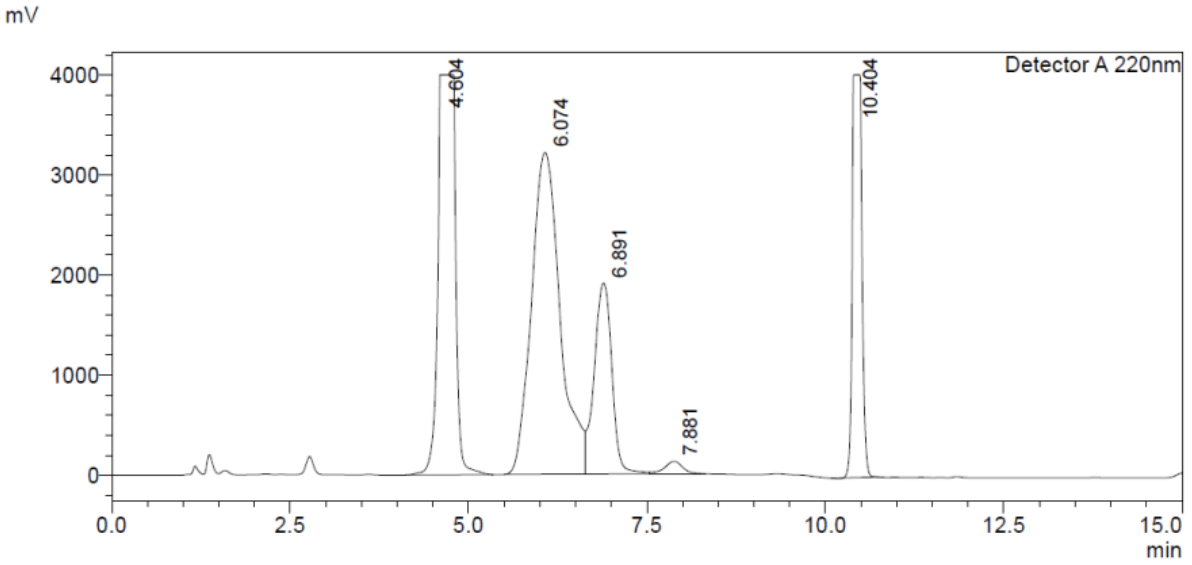
Crude Reaction Mixture (without internal standard) – UV Detector



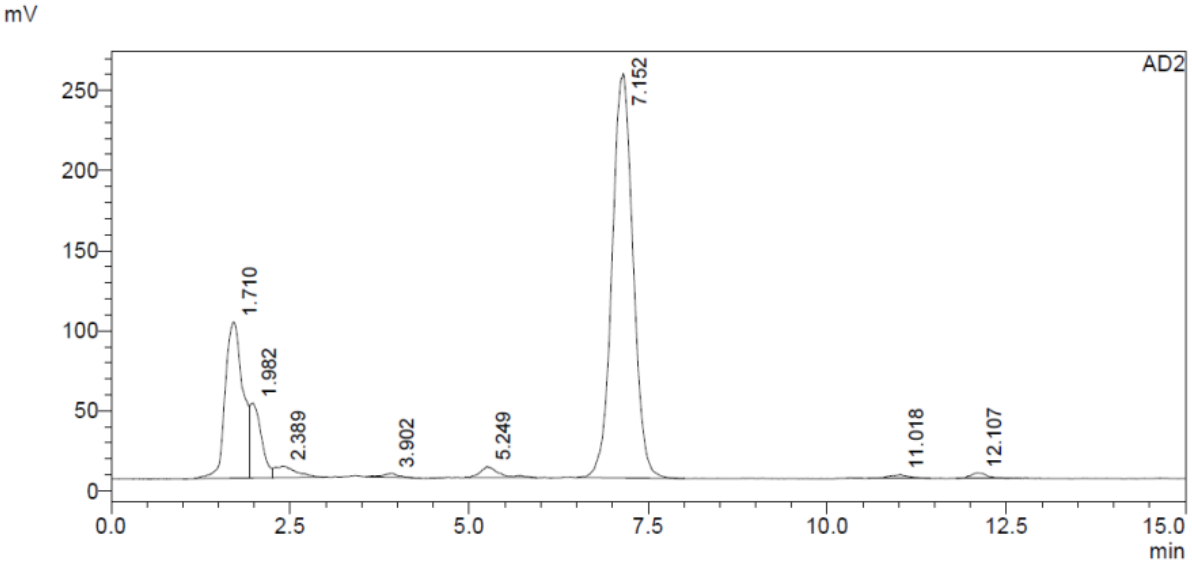
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



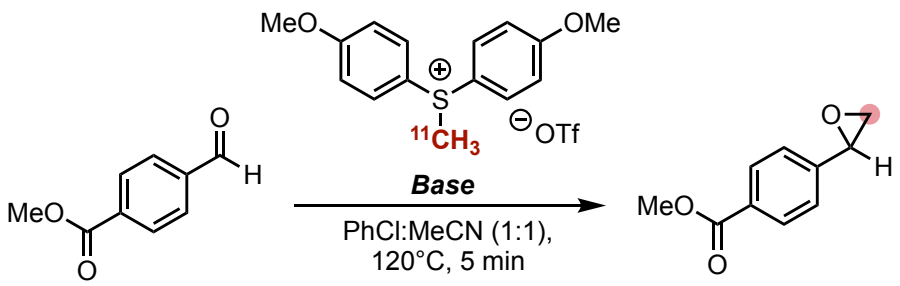
Crude Reaction Mixture – Gamma Detector



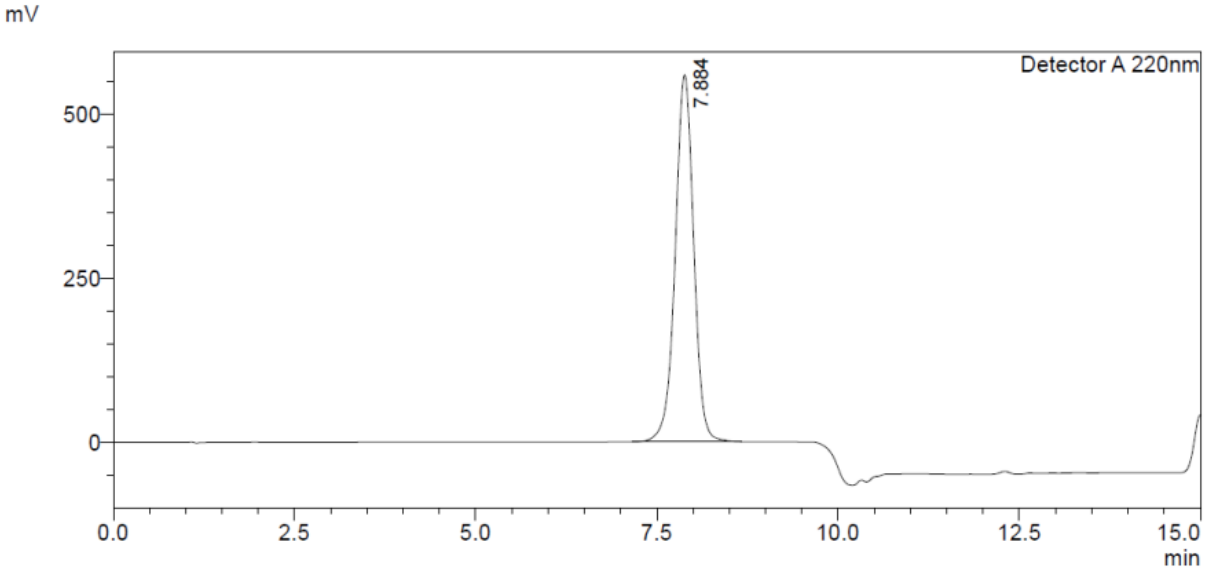
HPLC condition **A (X = 50),** injection volume 5 µL, peak at 6.891 (UV) and 7.152 (Rad), RCY **67%**.

4b

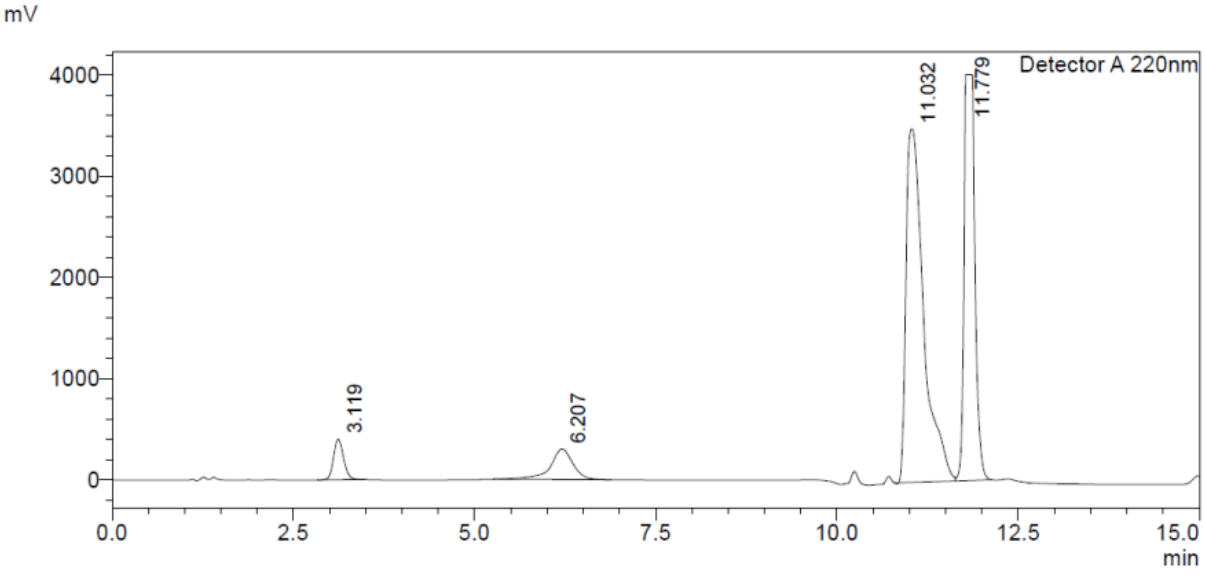
(Base = 1eq. ^{t}BuOK)



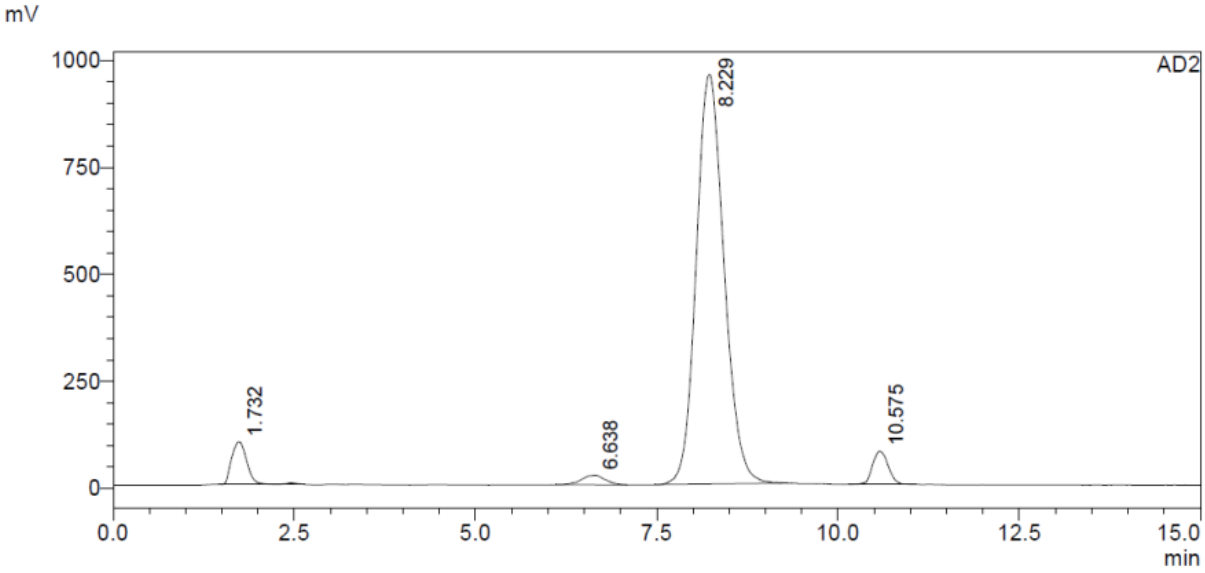
Standard - UV Detector



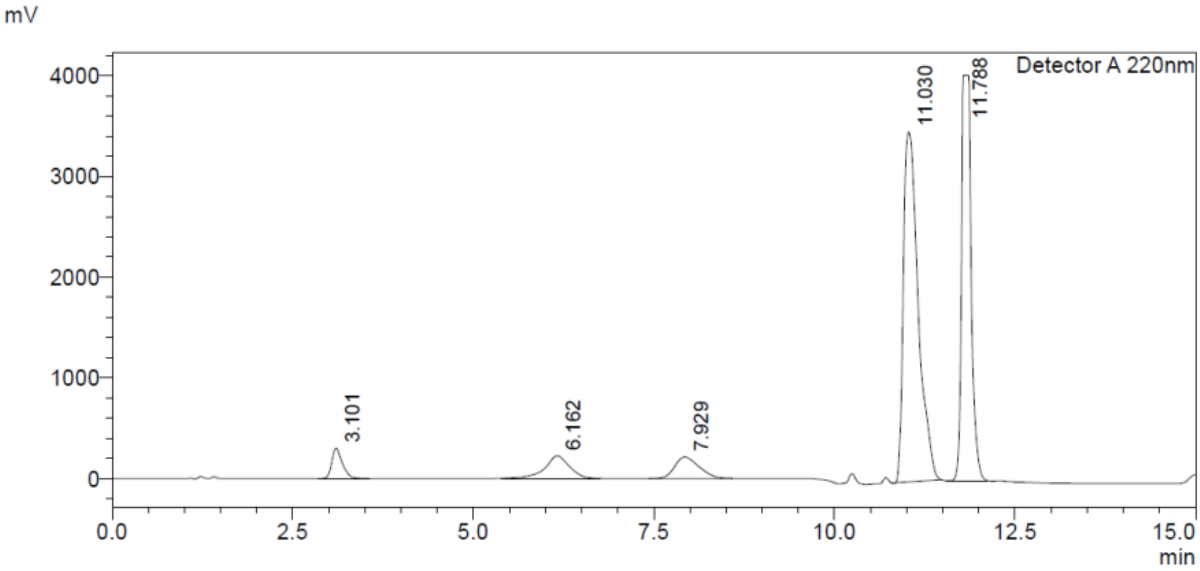
Crude Reaction Mixture (without internal standard) – UV Detector



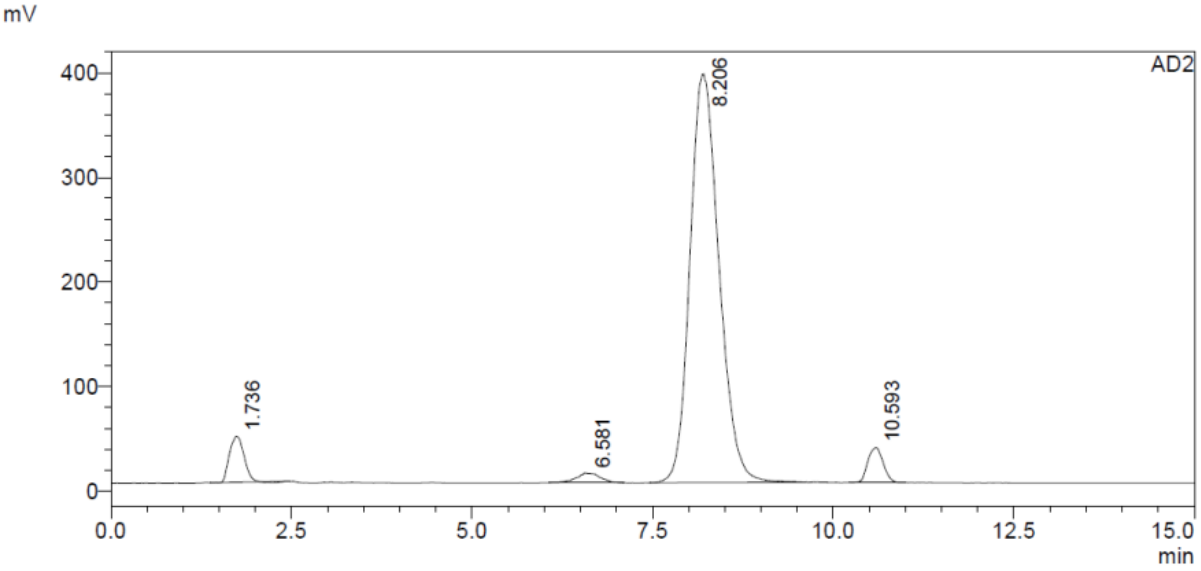
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



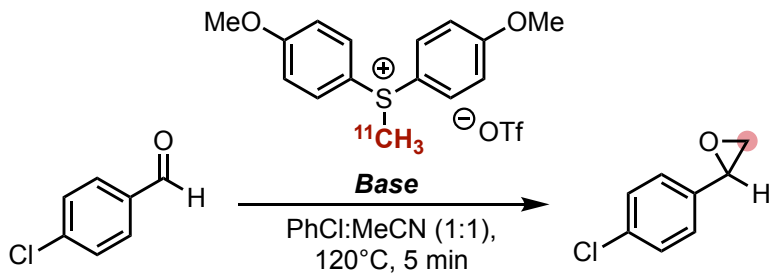
Crude Reaction Mixture – Gamma Detector



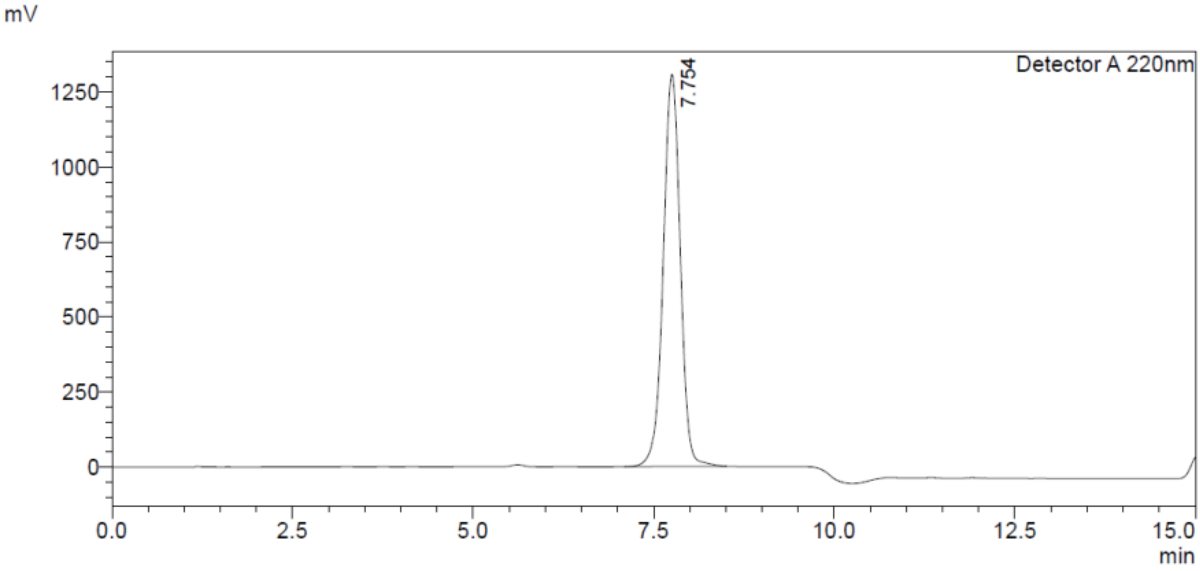
HPLC condition **A (X = 30),** injection volume 5 µL, peak at 7.929 (UV) and 8.206 (Rad), RCY 89%.

4c

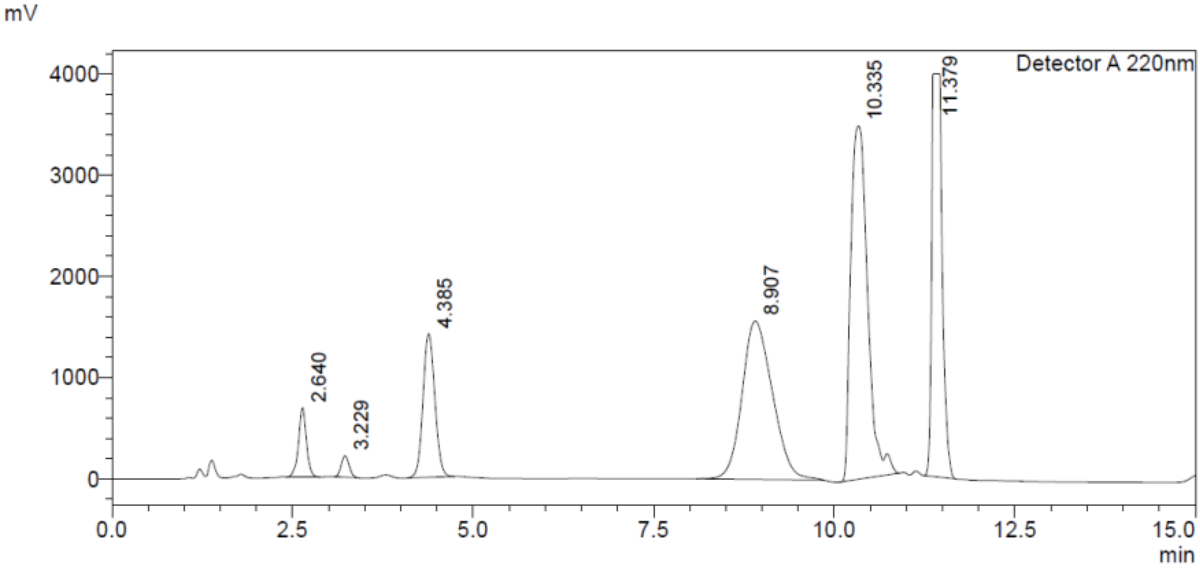
(Base = 1 eq. KOtBu)



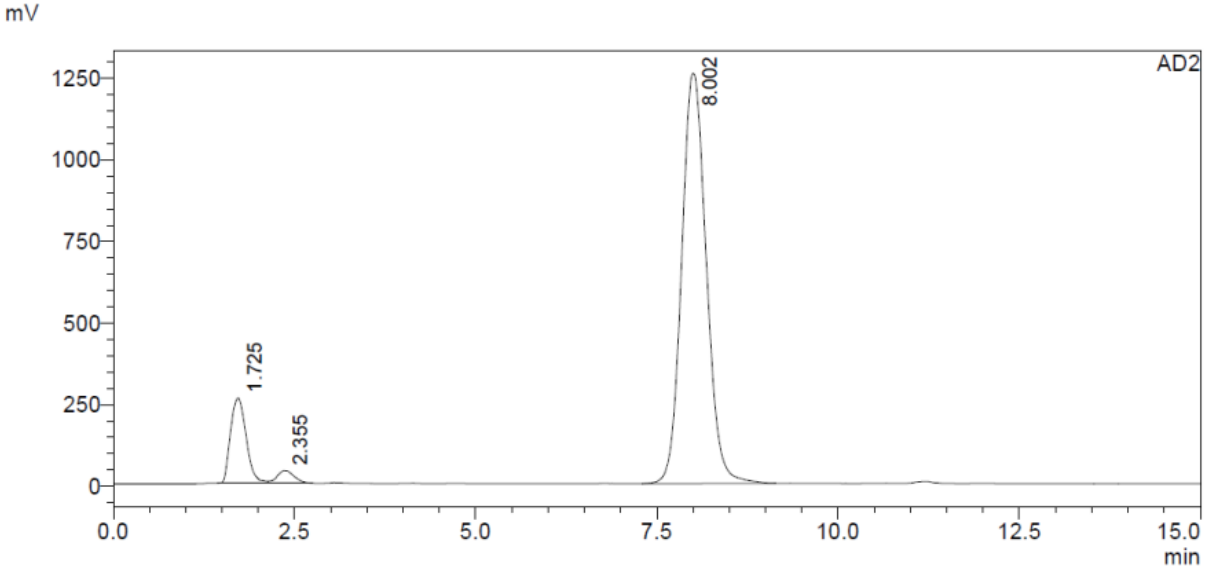
Standard - UV Detector



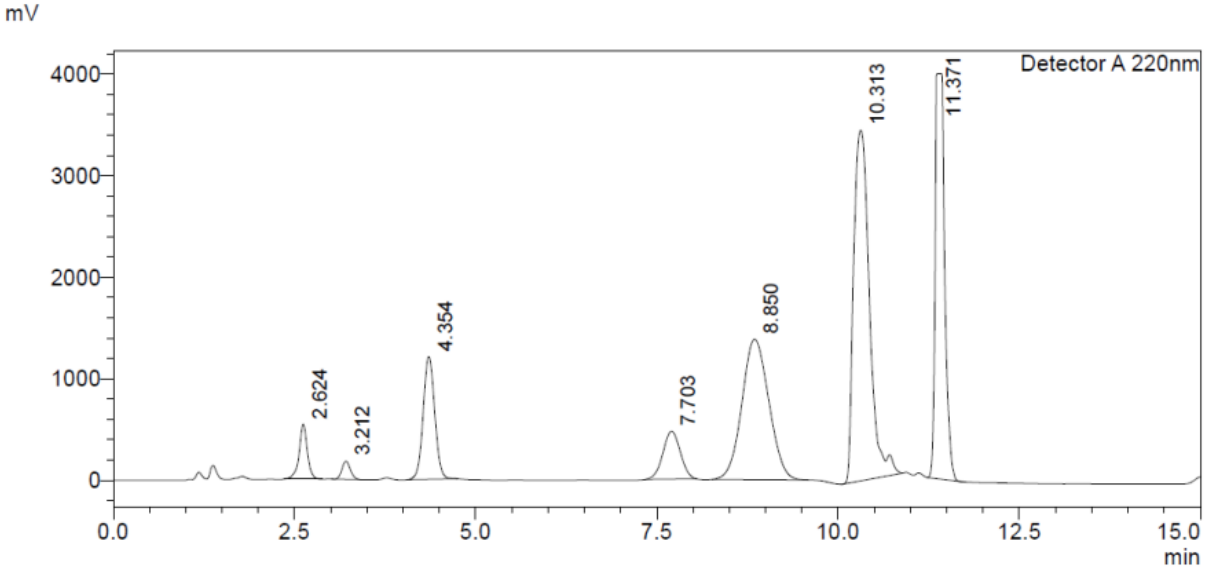
Crude Reaction Mixture (without internal standard) – UV Detector



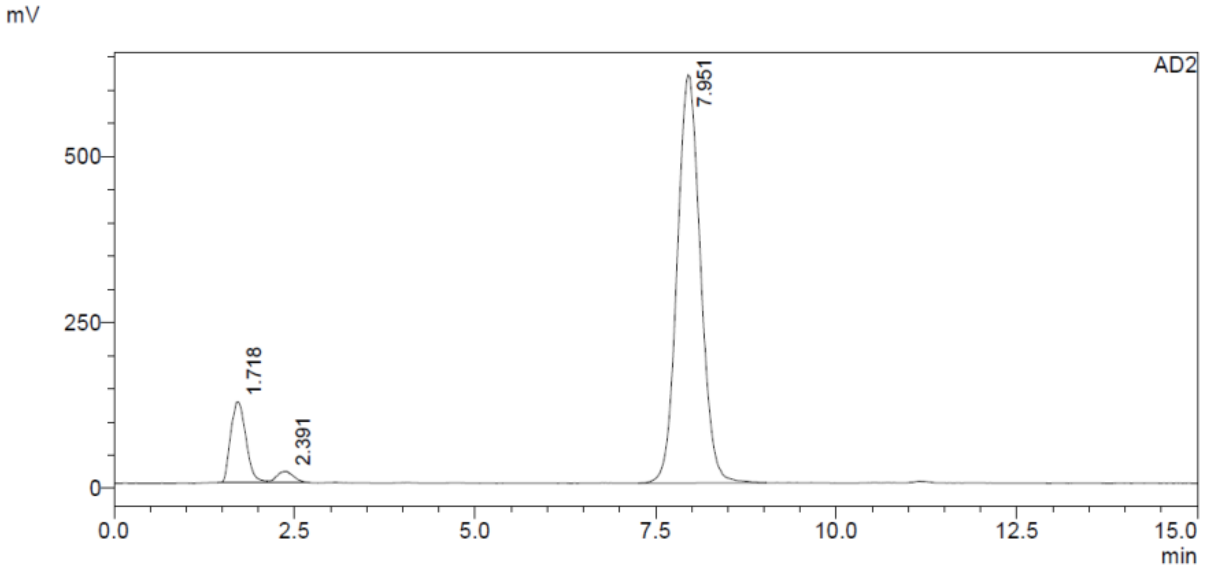
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



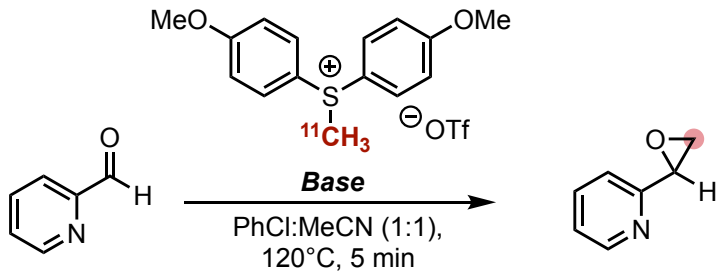
Crude Reaction Mixture – Gamma Detector



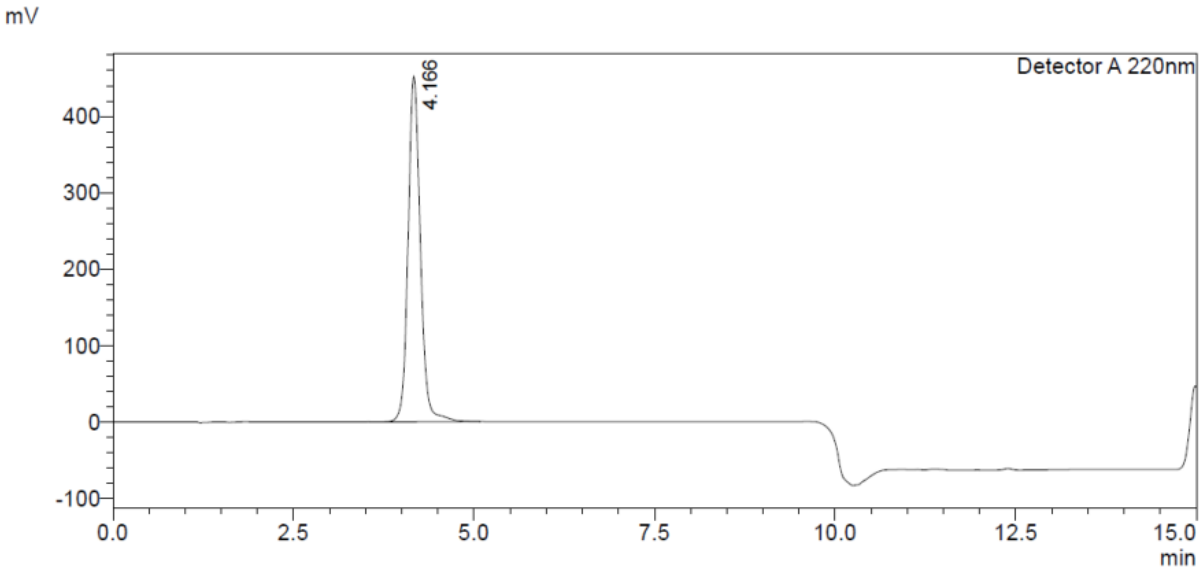
HPLC condition **A (X = 40),** injection volume 5 µL, peak at 7.703 (UV) and 7.951 (Rad), RCY **87%.**

4e

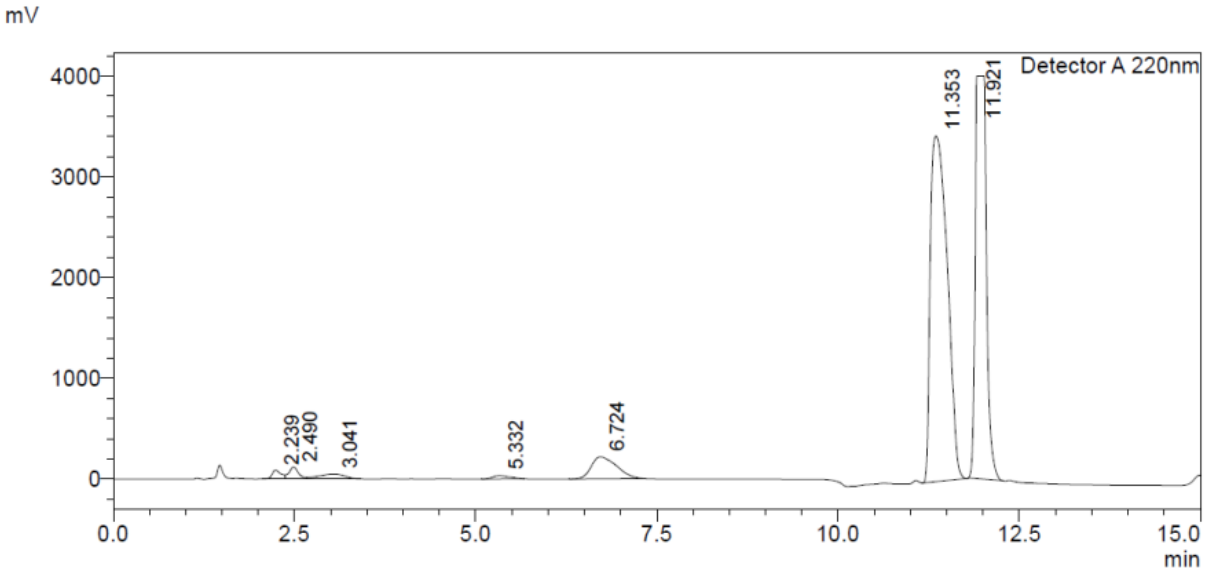
(Base = 1 eq. KOtBu)



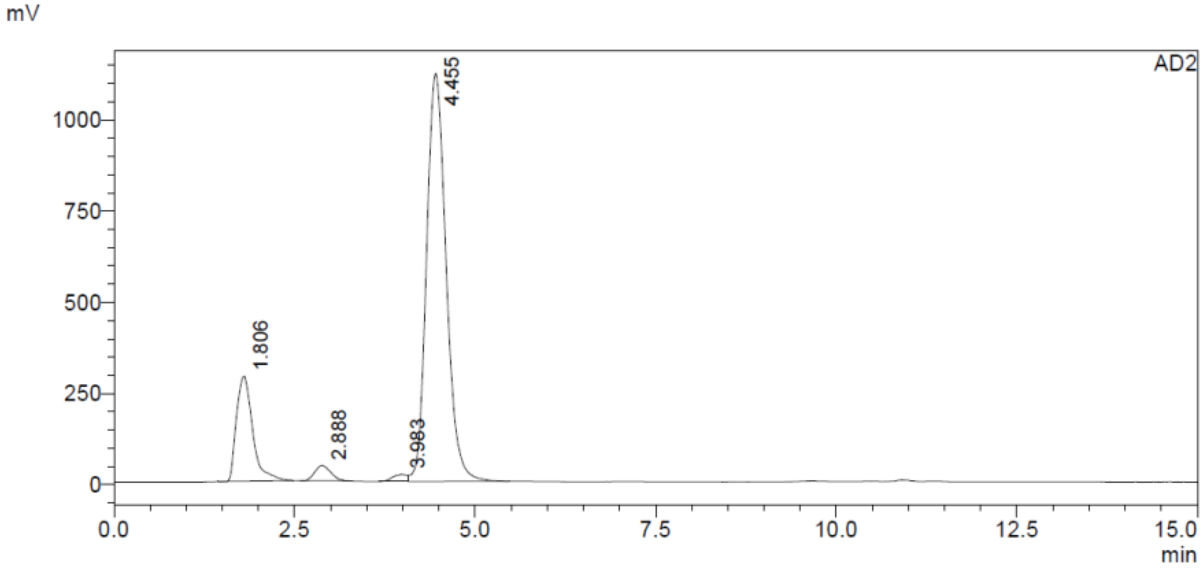
Standard - UV Detector



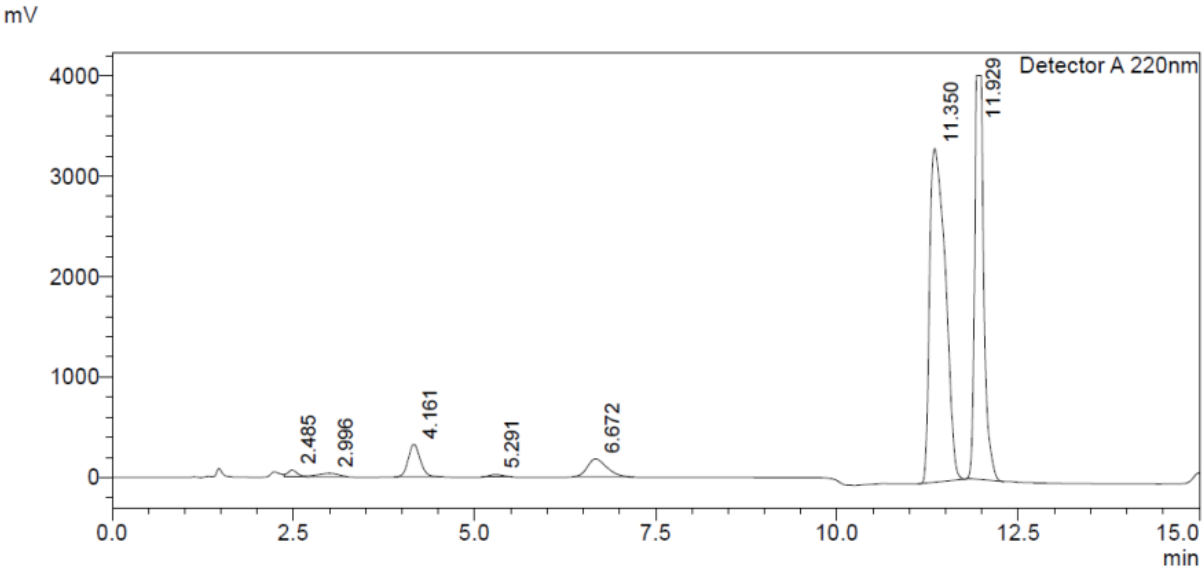
Crude Reaction Mixture (without internal standard) – UV Detector



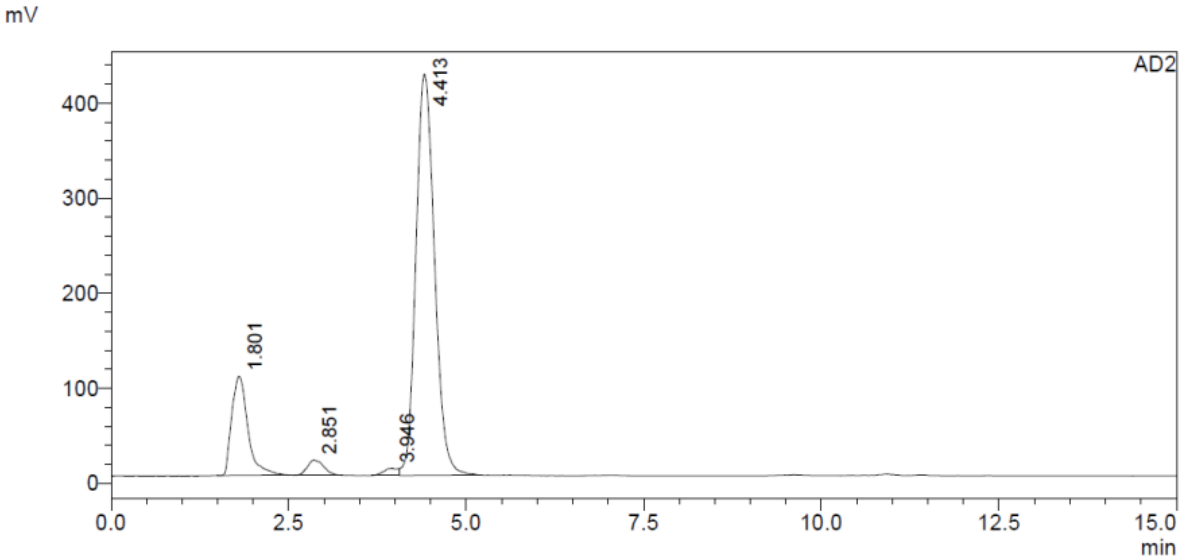
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



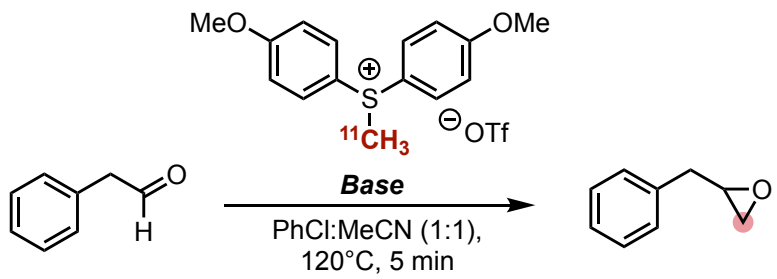
Crude Reaction Mixture – Gamma Detector



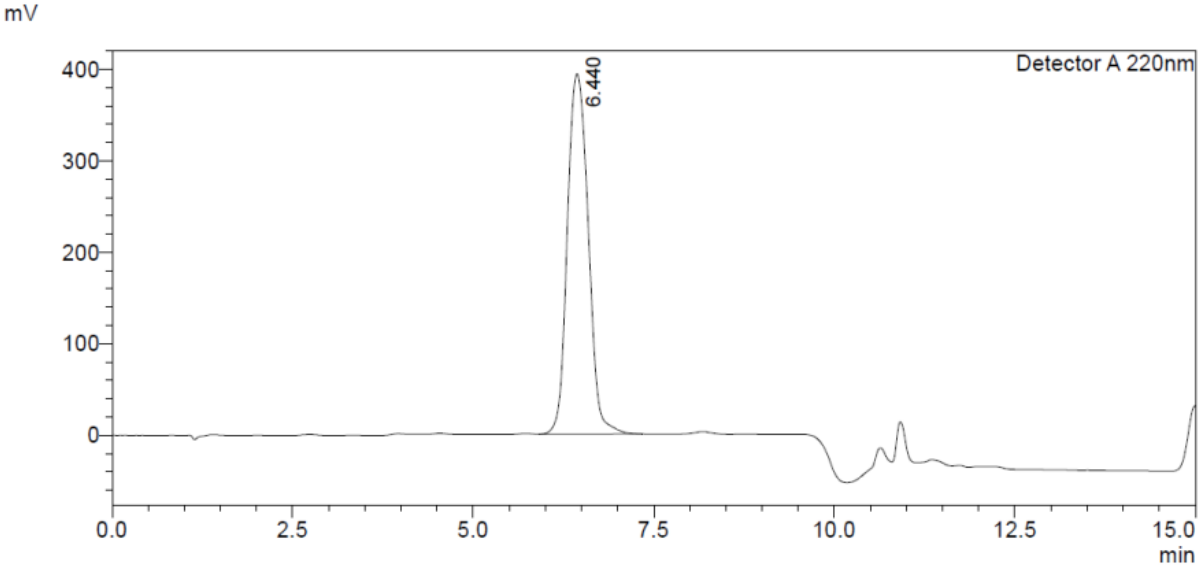
HPLC condition **A (X = 20),** injection volume 5 µL, peak at 4.161 (UV) and 4.413 (Rad), RCY **80%**.

4f

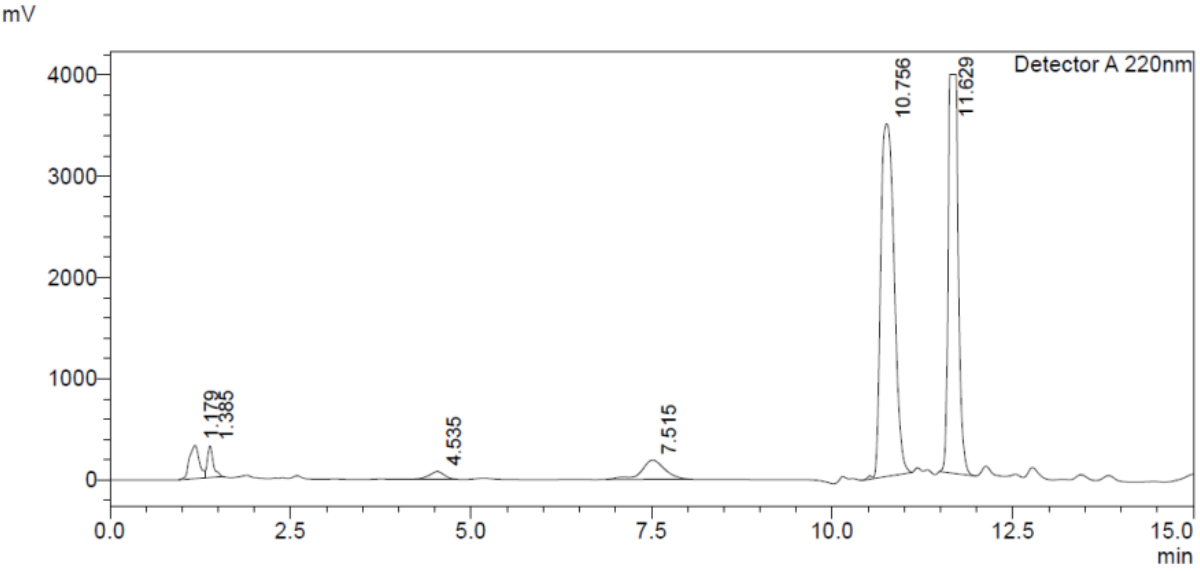
(Base = 2 eq. KOtBu)



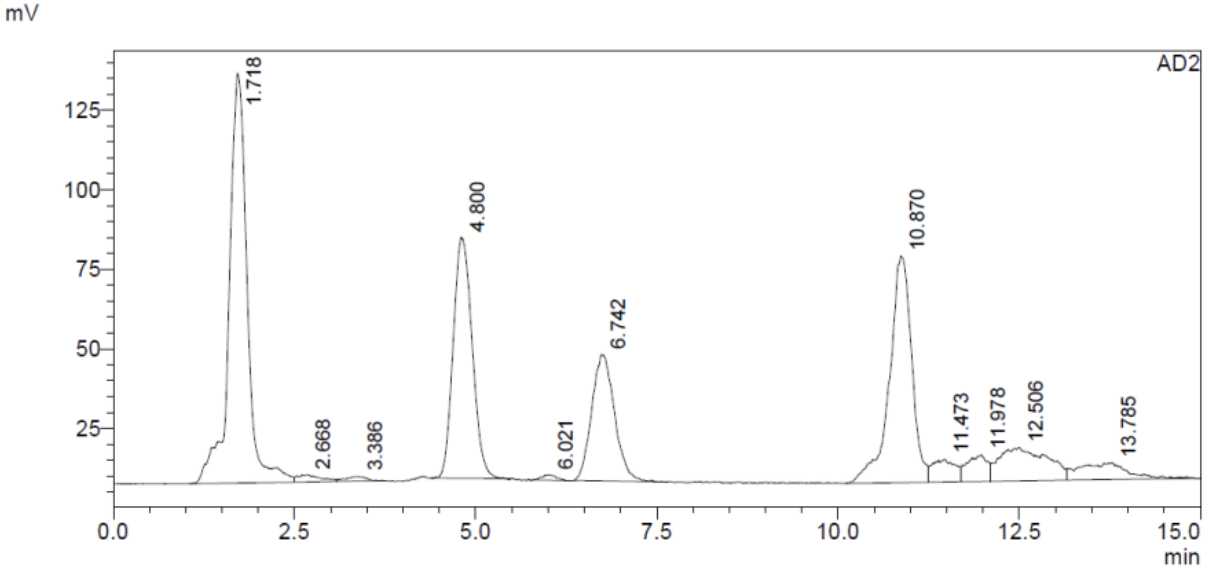
Standard - UV Detector



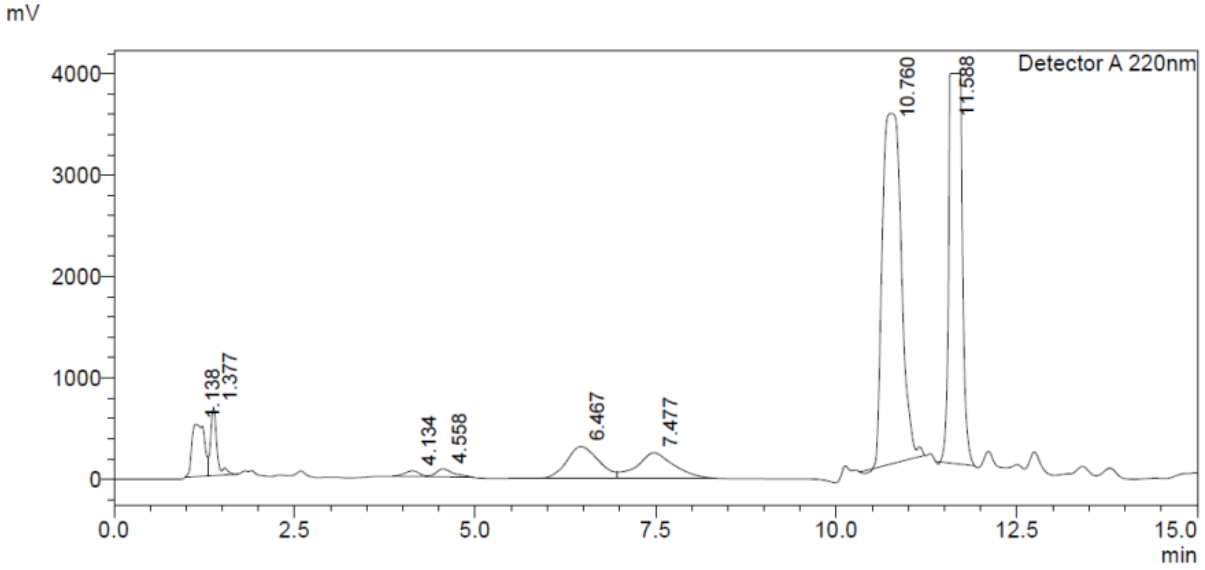
Crude Reaction Mixture (without internal standard) – UV Detector



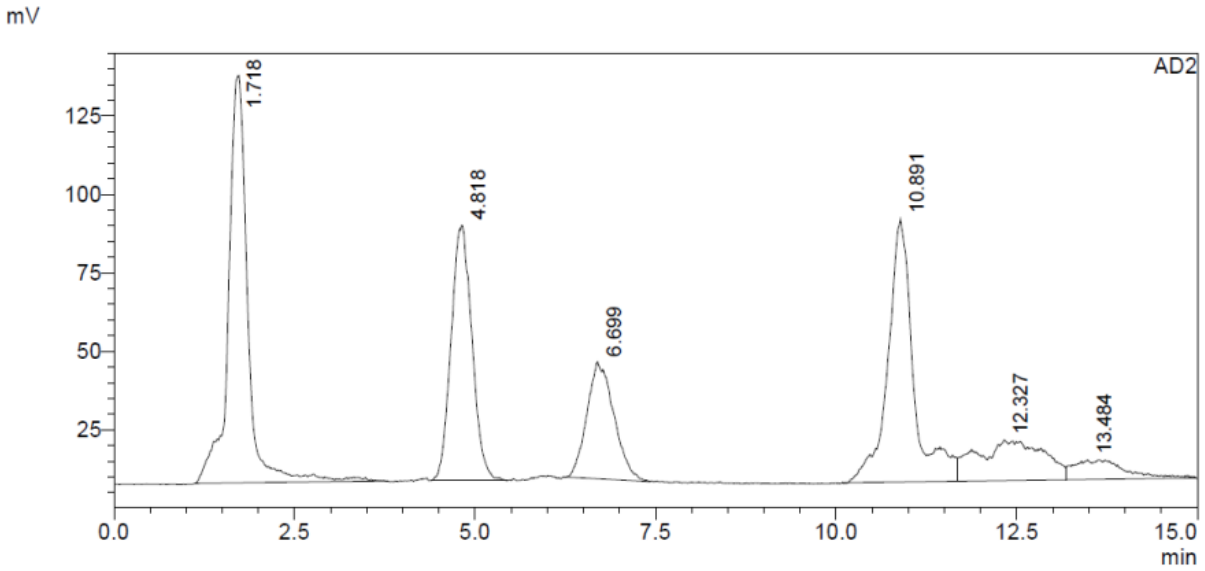
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



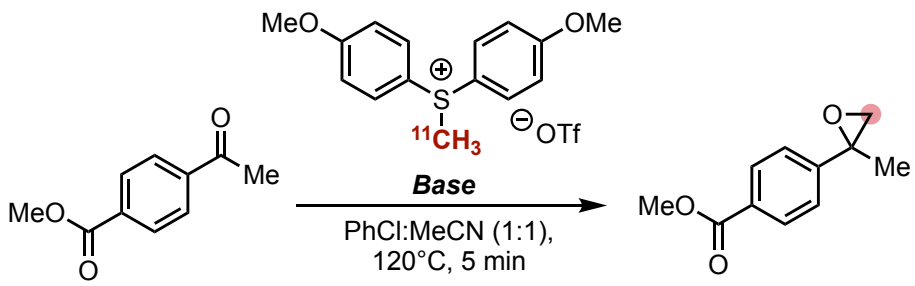
Crude Reaction Mixture – Gamma Detector



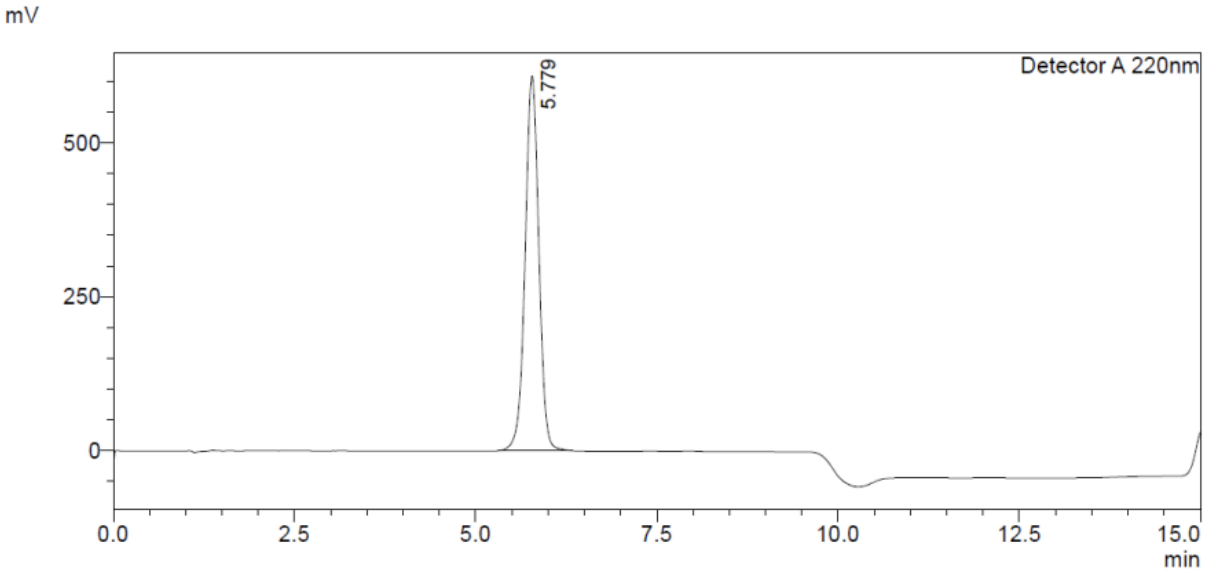
HPLC condition **A (X = 35),** injection volume 5-10 µL, peak at 6.467 (UV) and 6.699 (Rad), RCY **12%**.

4g

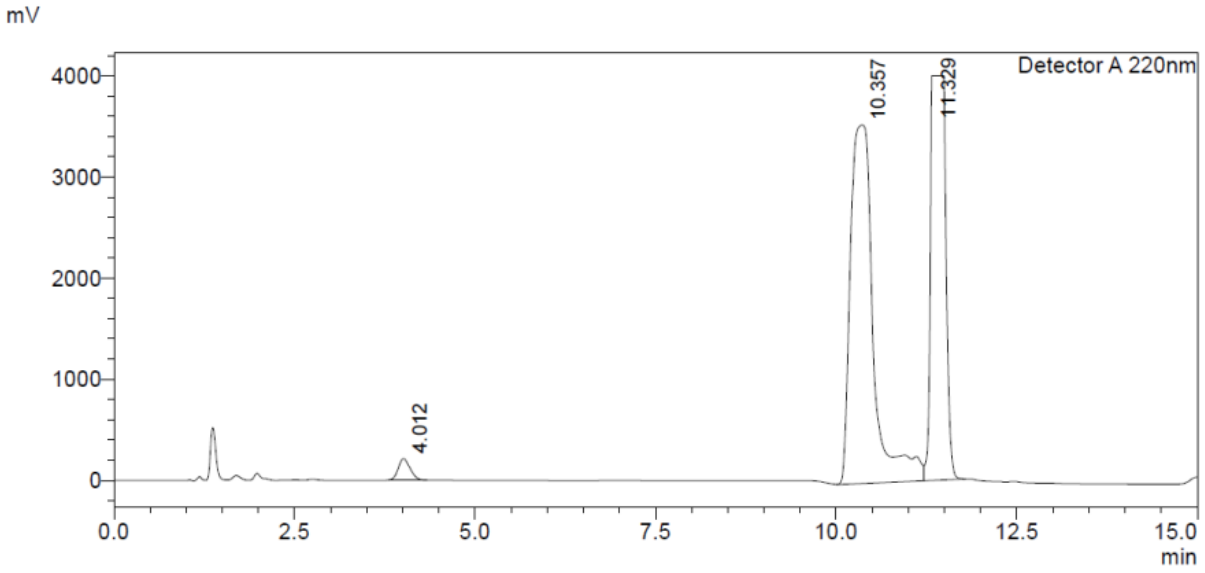
(Base = 2 eq. KOtBu)



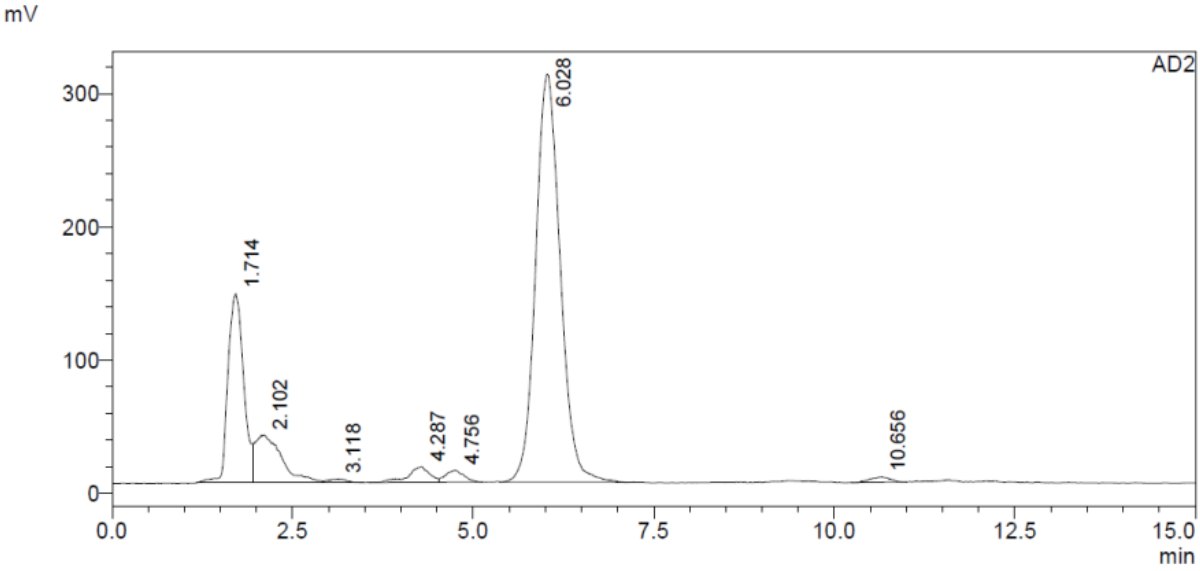
Standard - UV Detector



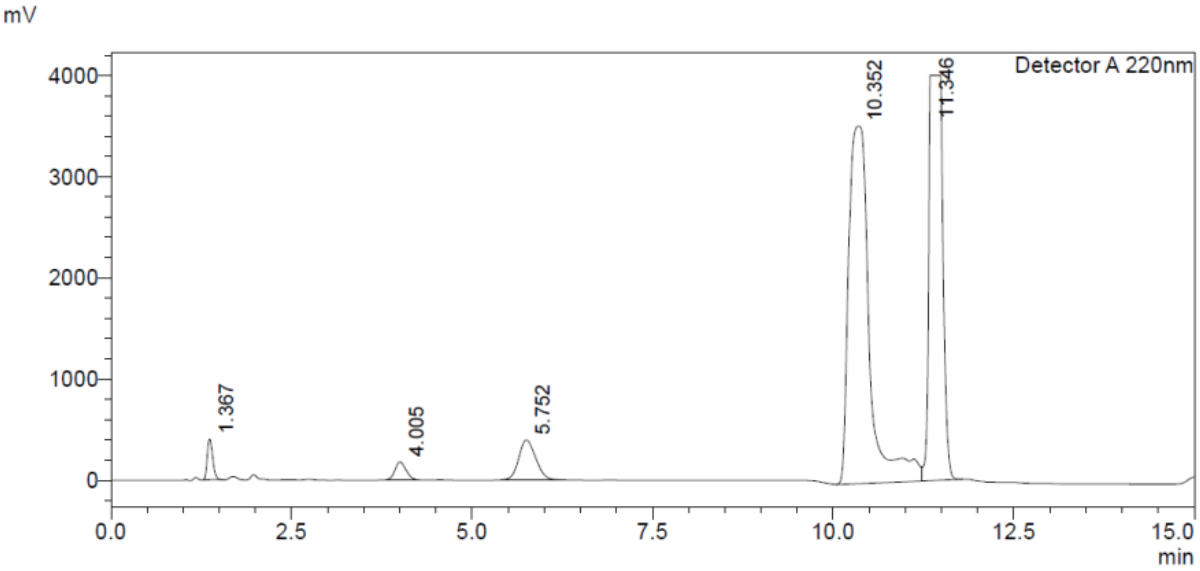
Crude Reaction Mixture (without internal standard) – UV Detector



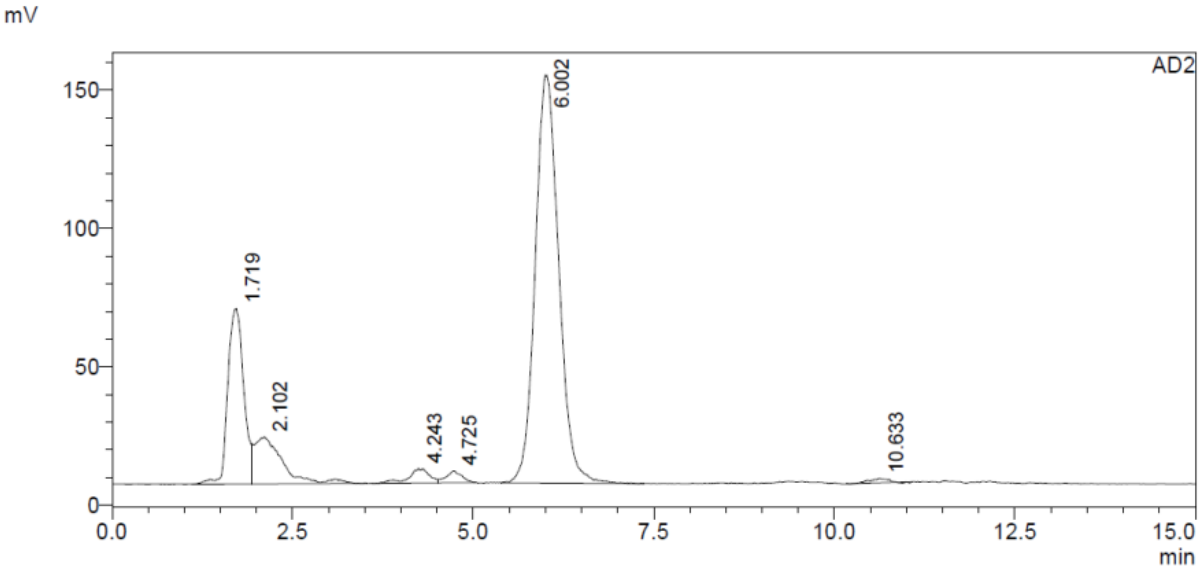
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



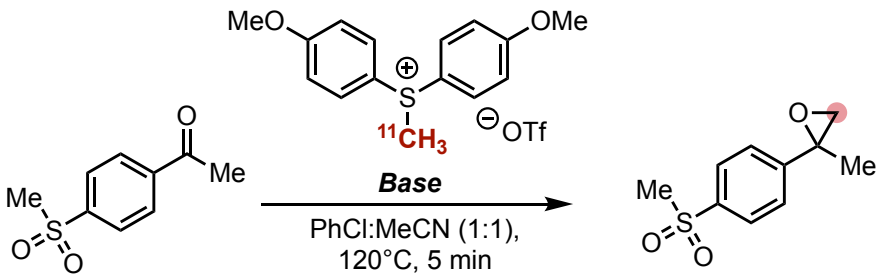
Crude Reaction Mixture – Gamma Detector



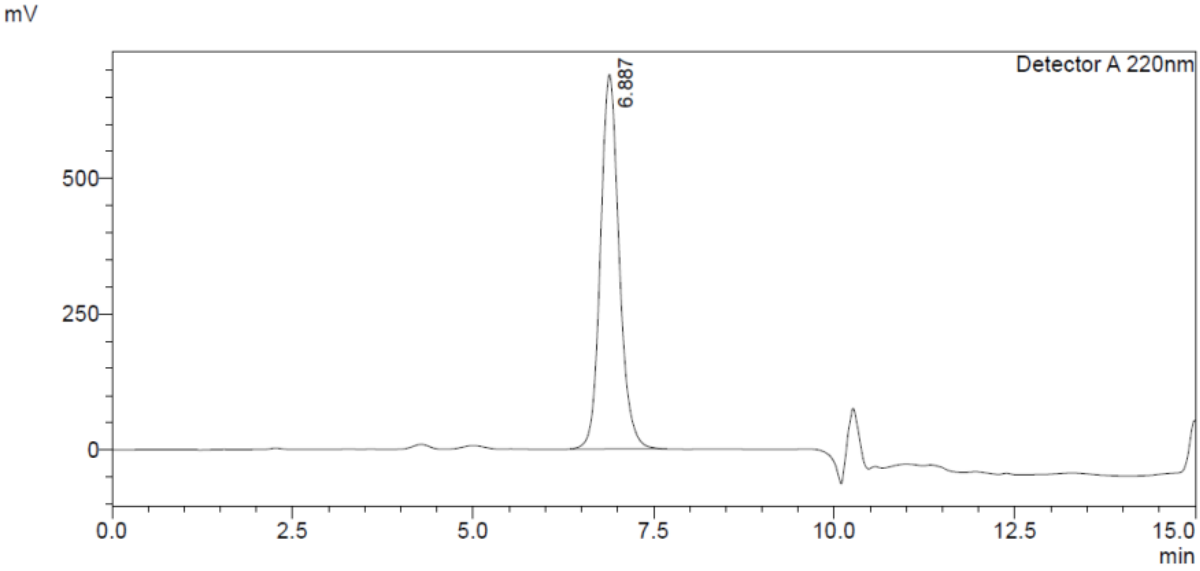
HPLC condition A (X = 40), injection volume 10 µL, peak at 5.752 (UV) and 6.002 (Rad), RCY 67%.

4h

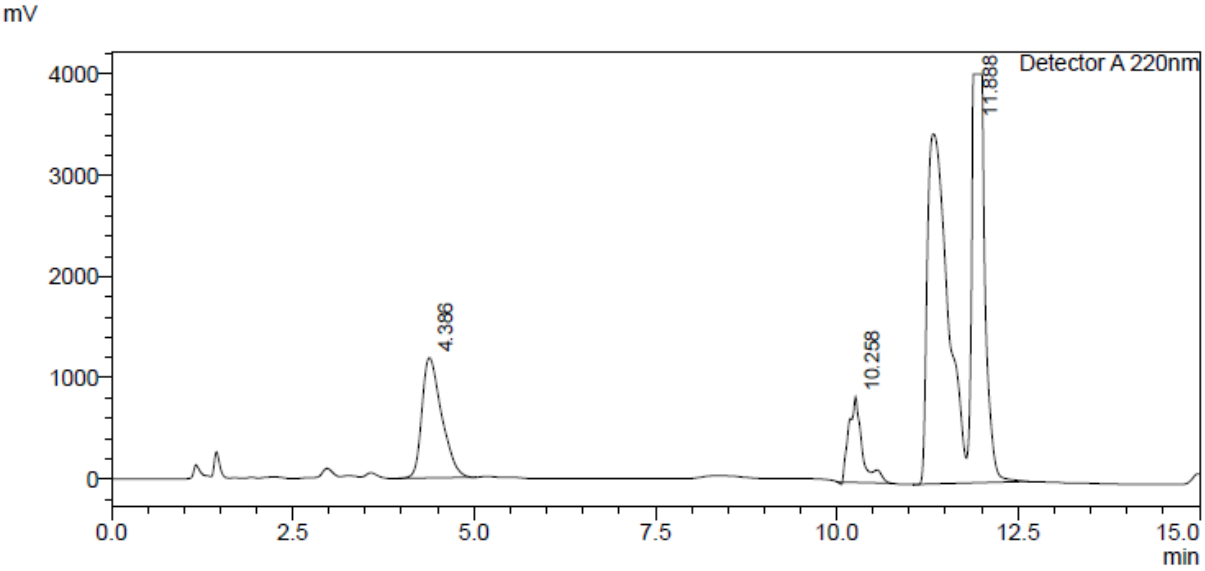
(Base = 2 eq. KOtBu)



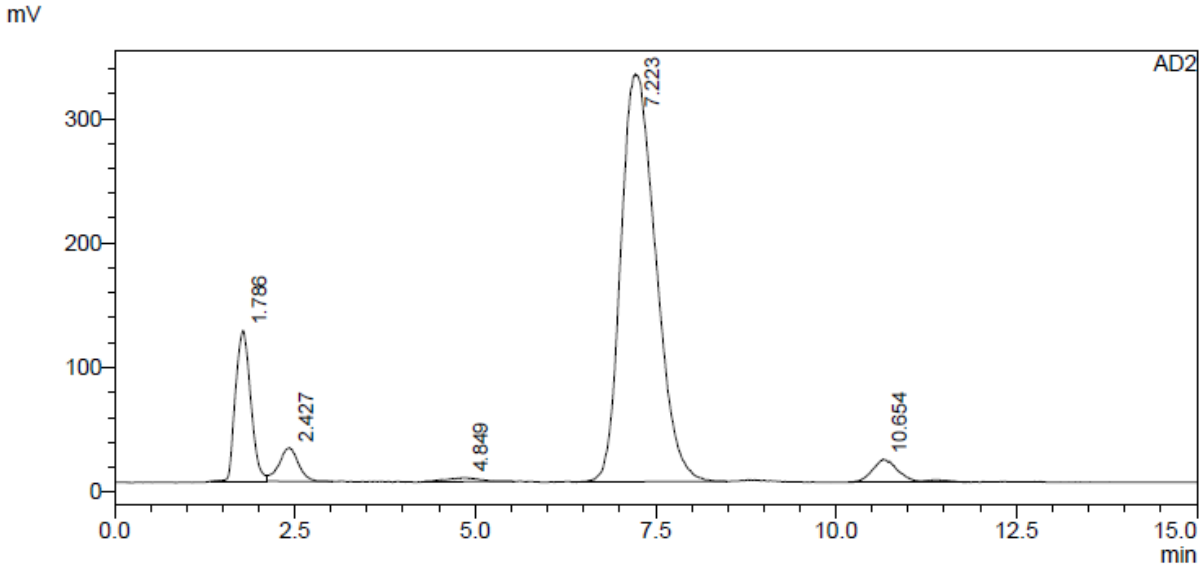
Standard - UV Detector



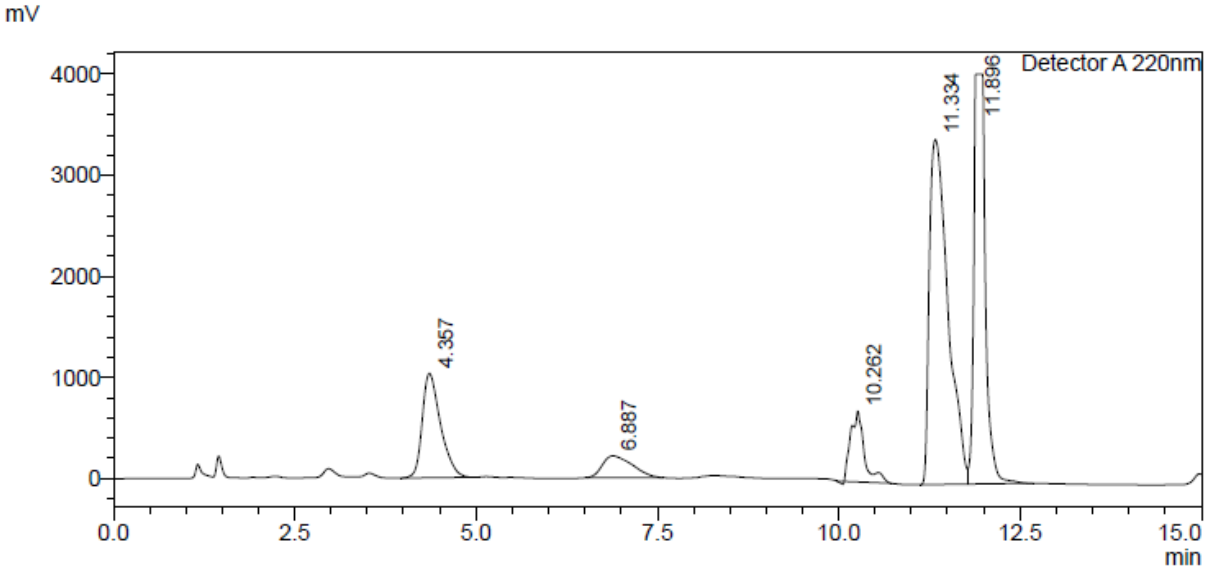
Crude Reaction Mixture (without internal standard) – UV Detector



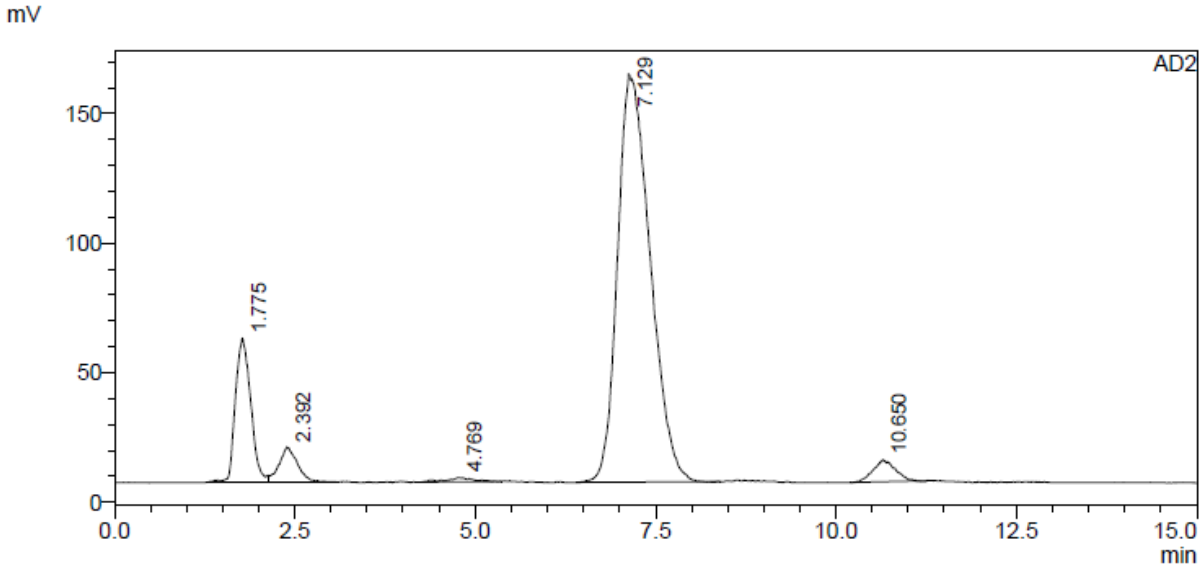
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



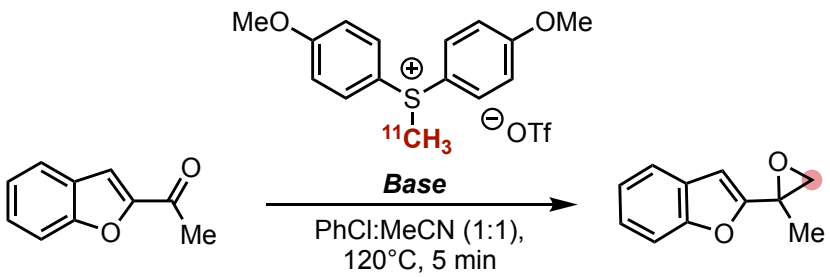
Crude Reaction Mixture – Gamma Detector



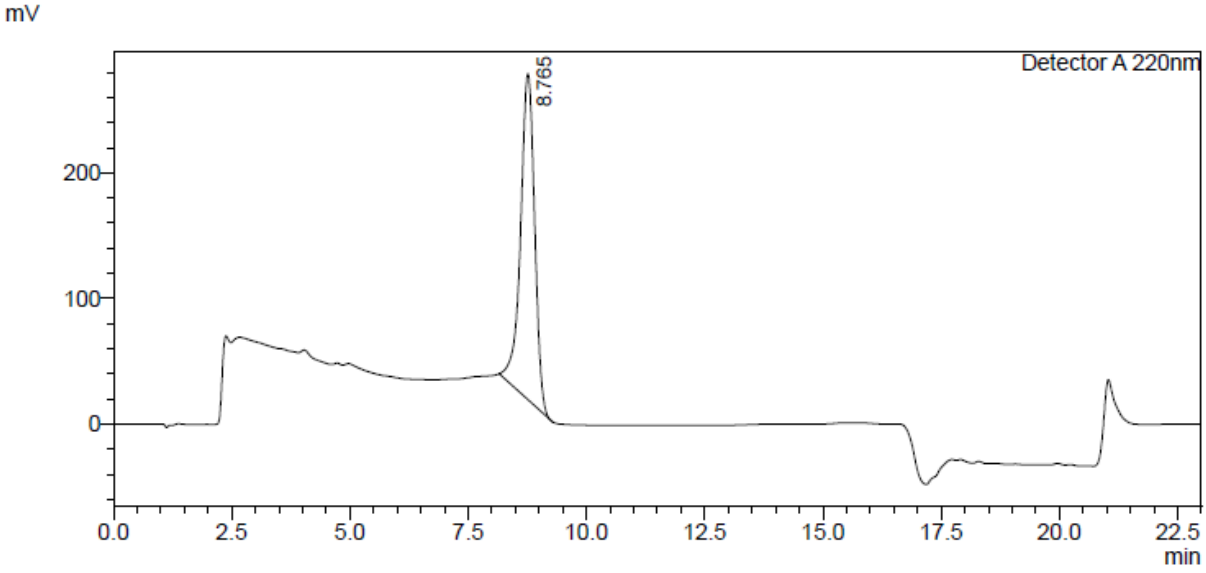
HPLC condition **A (X = 20),** injection volume 5 µL, peak at 6.887 (UV) and 7.129 (Rad), RCY **79%**.

4i

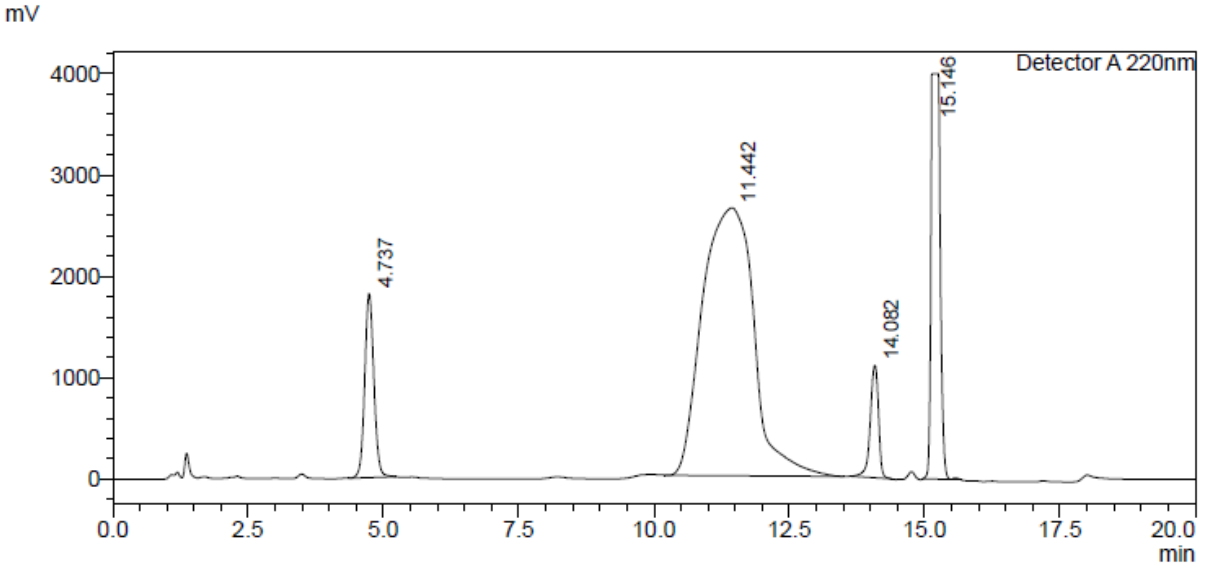
(Base = 2eq. ^{t}BuOK)



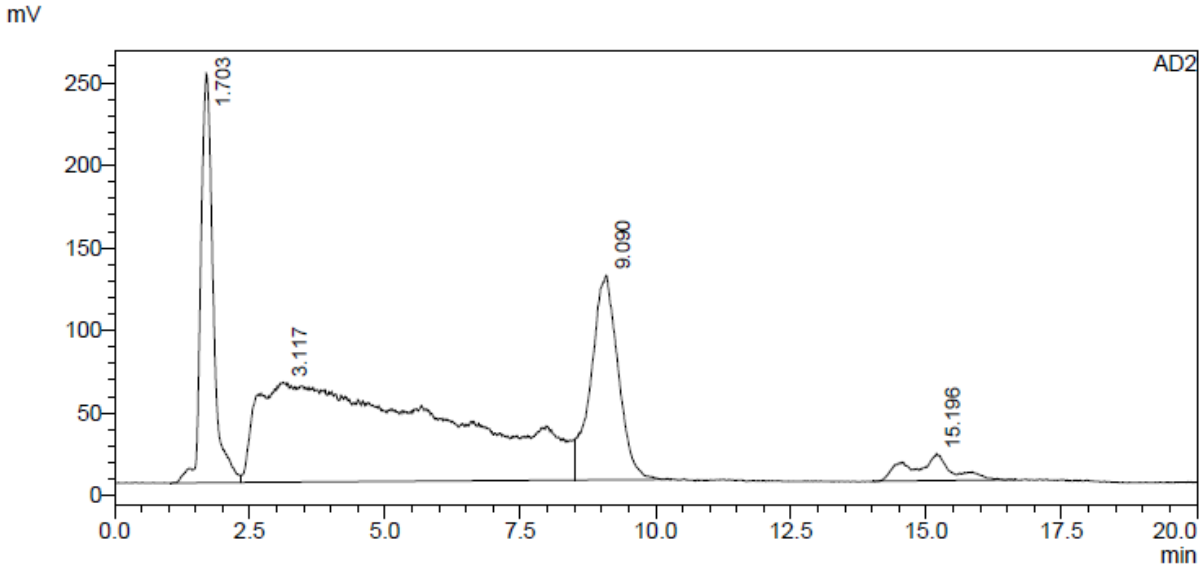
Standard - UV Detector



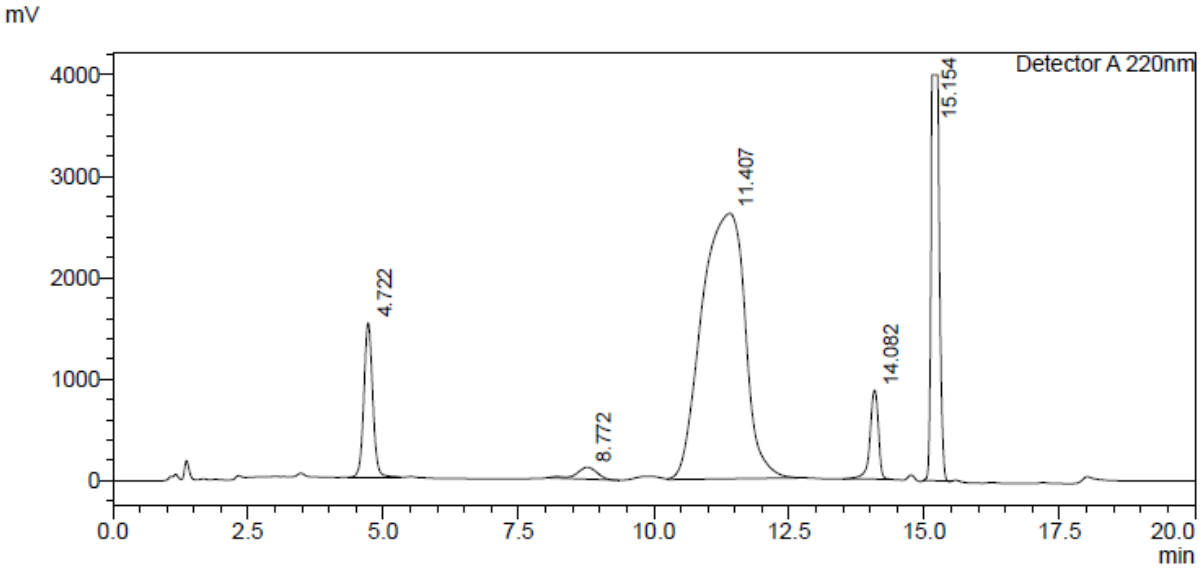
Crude Reaction Mixture (without internal standard) – UV Detector



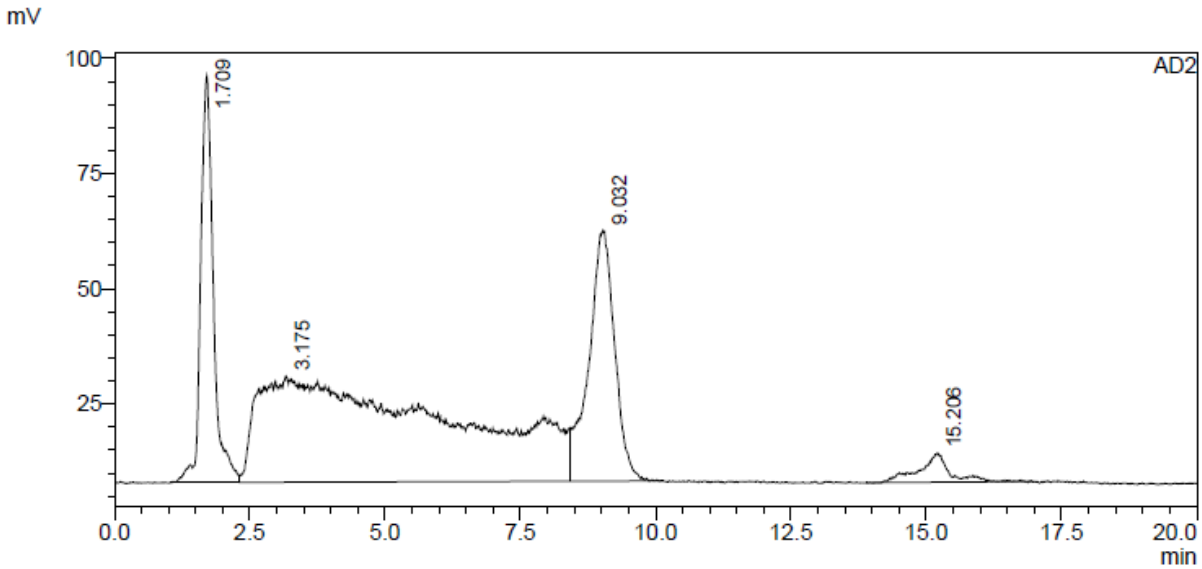
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



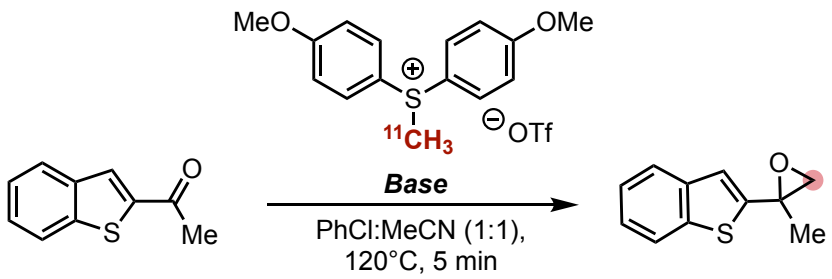
Crude Reaction Mixture – Gamma Detector



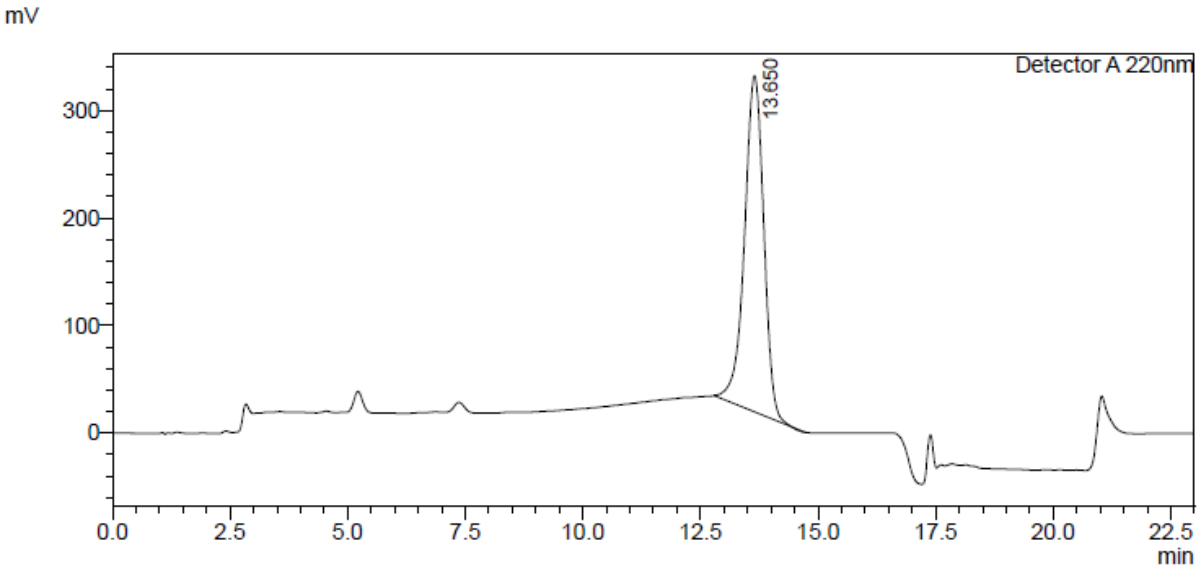
HPLC condition **C (X = 40),** injection volume 5 µL, peak at 8.772 (UV) and 9.032 (Rad), RCY **20%**.

4j

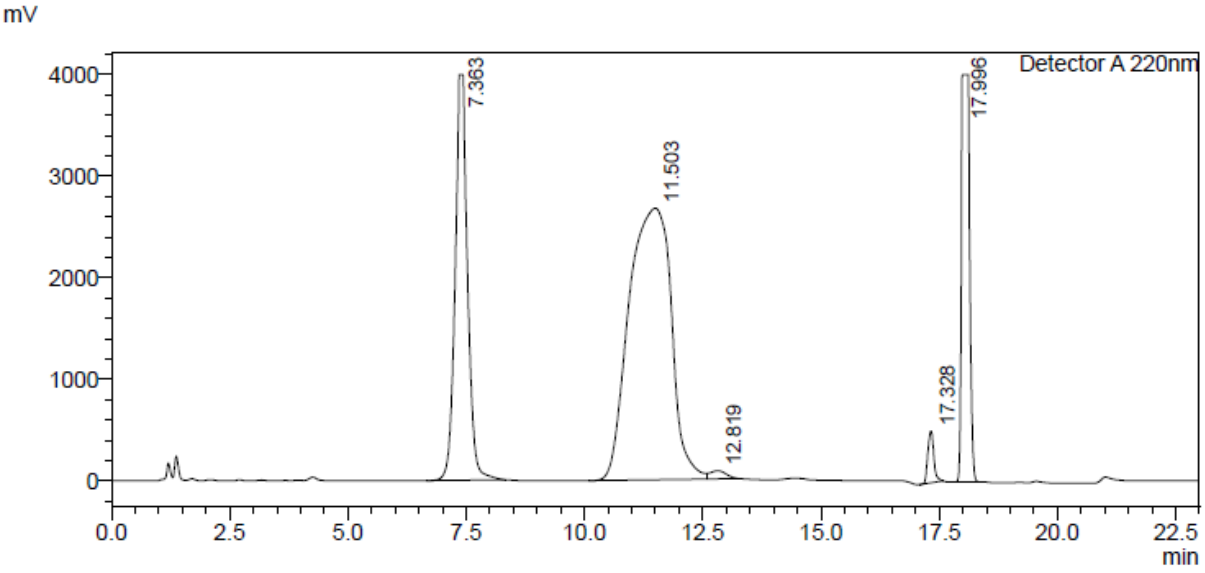
(Base = 2 eq. ^{t}BuOK)



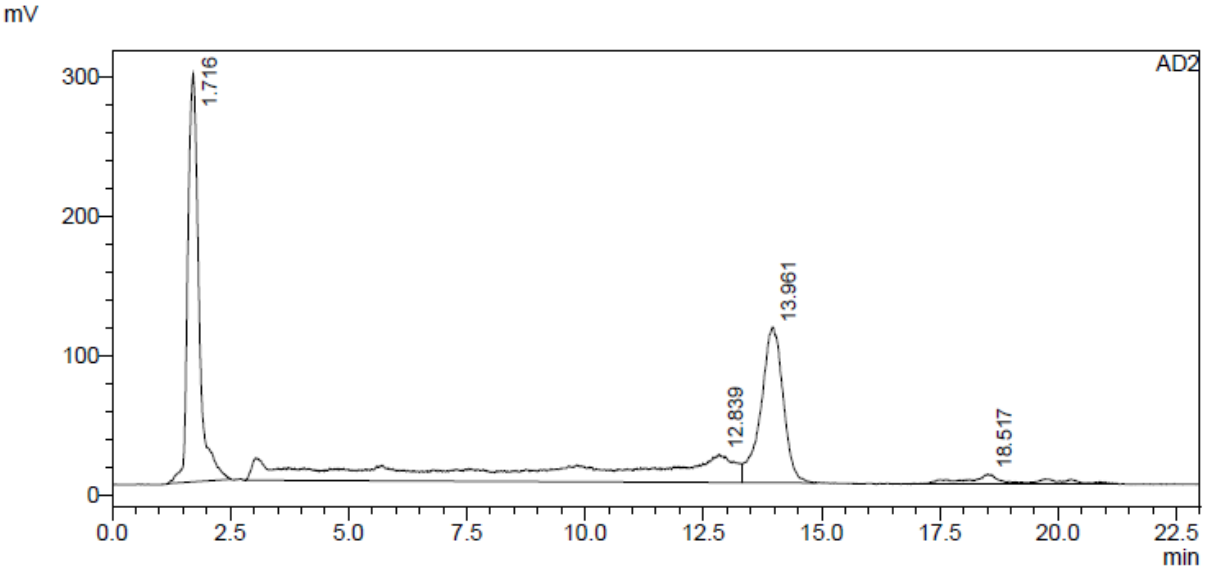
Standard - UV Detector



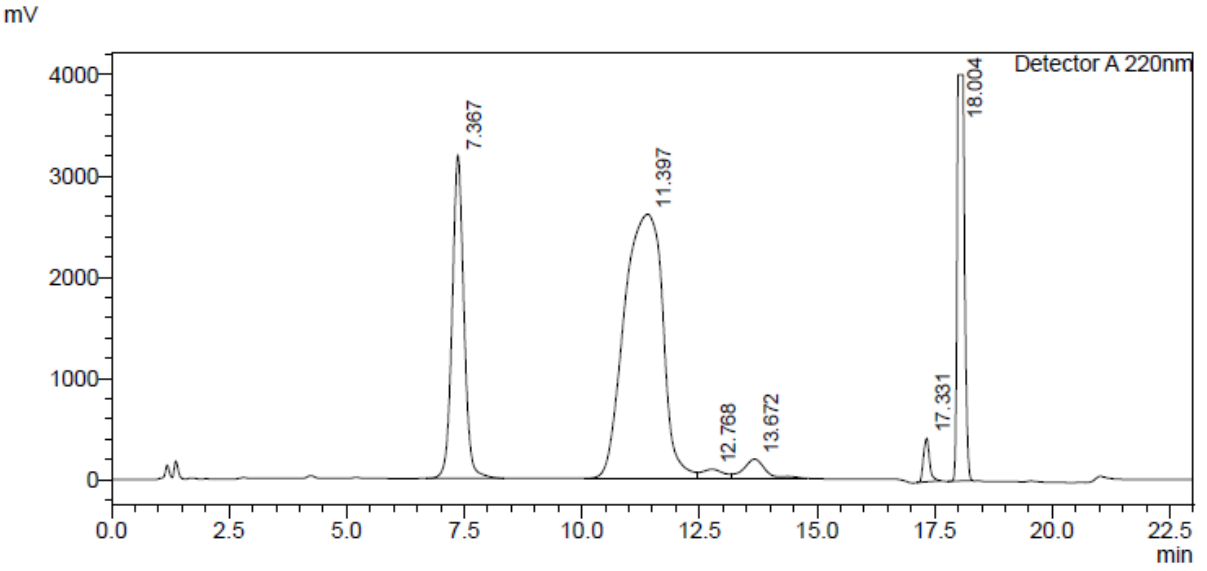
Crude Reaction Mixture (without internal standard) – UV Detector



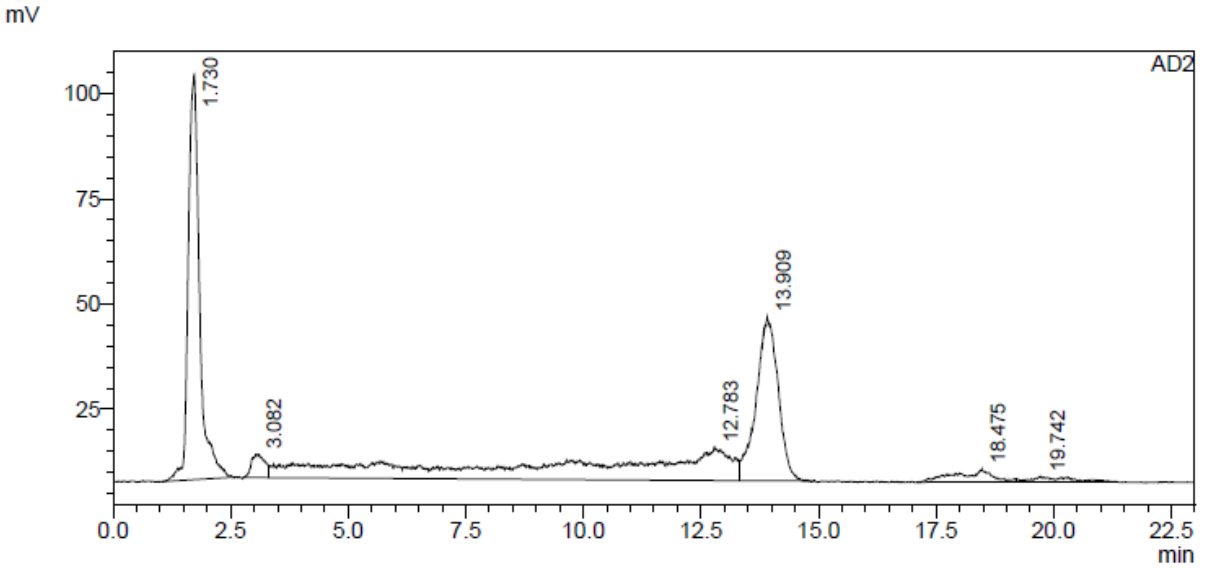
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



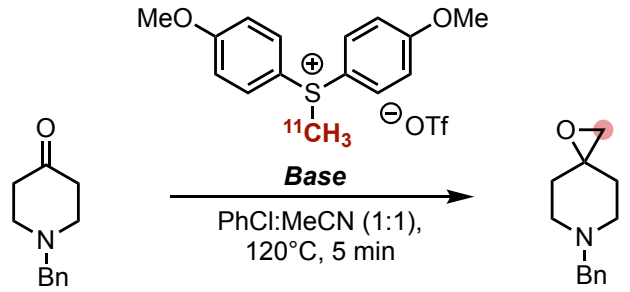
Crude Reaction Mixture – Gamma Detector



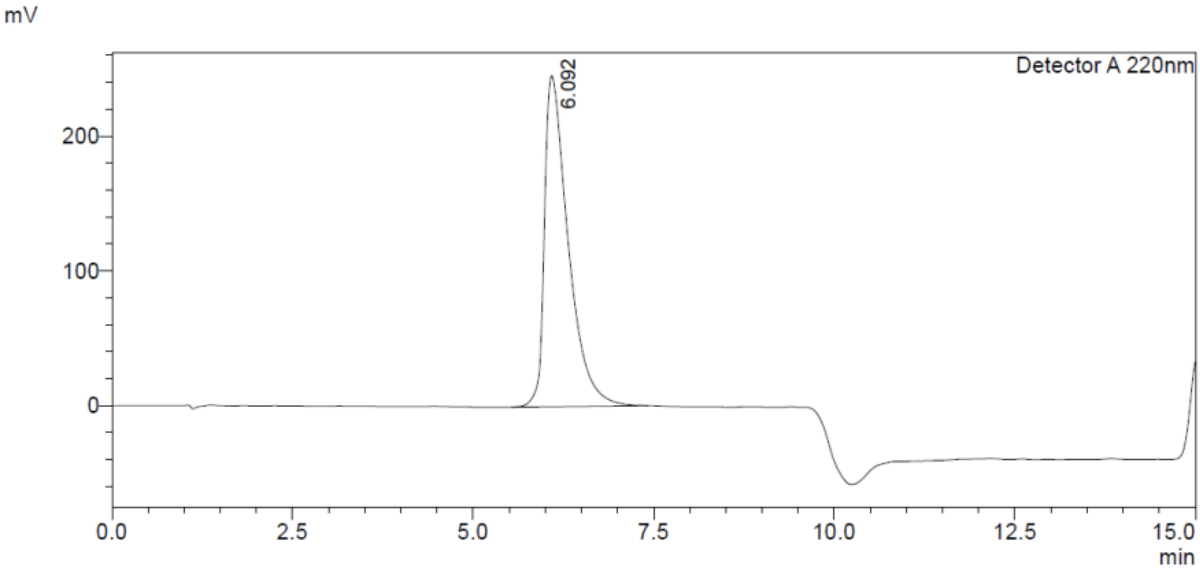
HPLC condition **B (X = 40),** injection volume 5 µL, peak at 13.672 (UV) and 13.909 (Rad), RCY **25%**.

4k

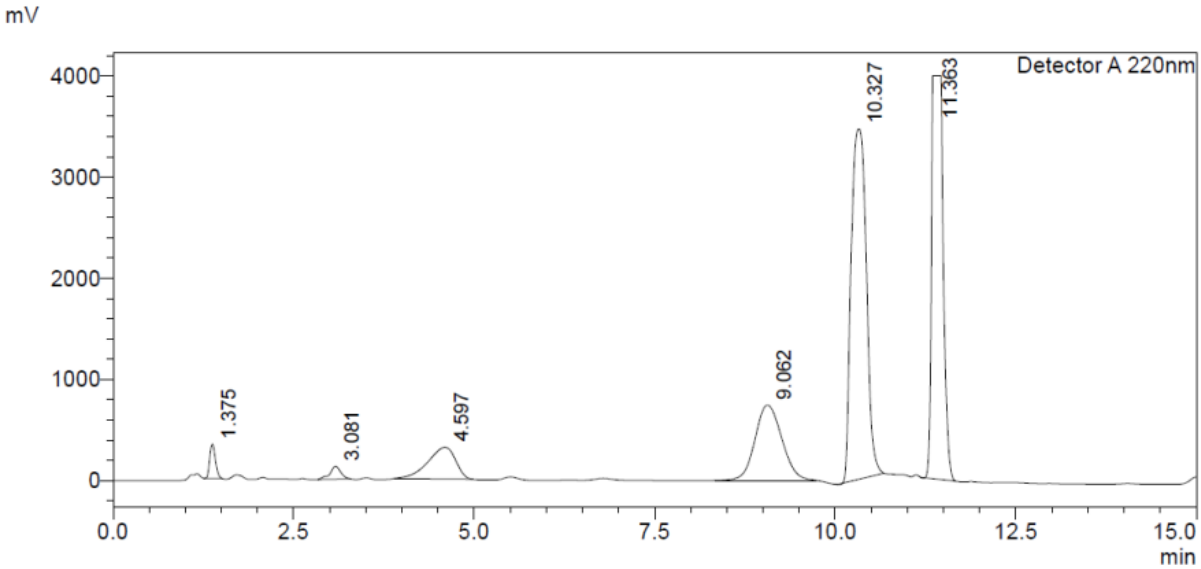
(Base = 2 eq. KOtBu)



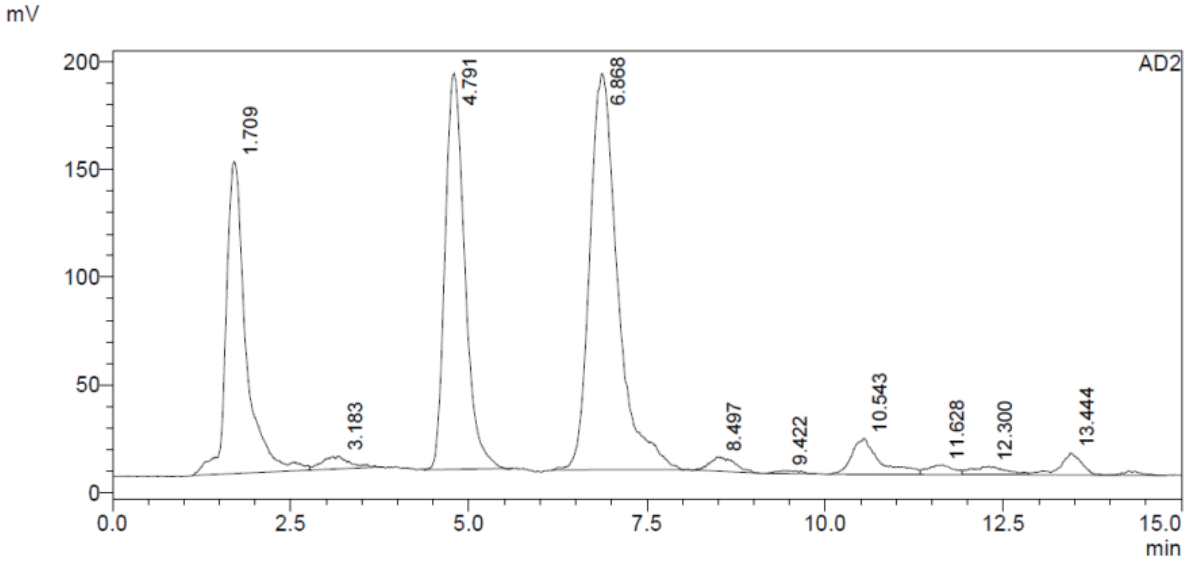
Standard - UV Detector



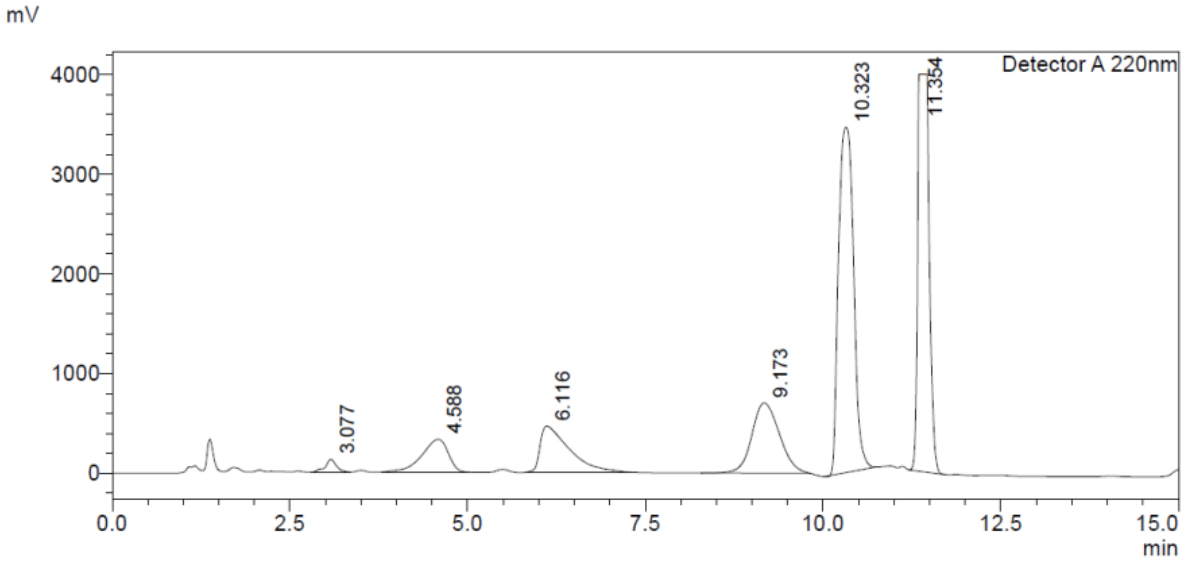
Crude Reaction Mixture (without internal standard) – UV Detector



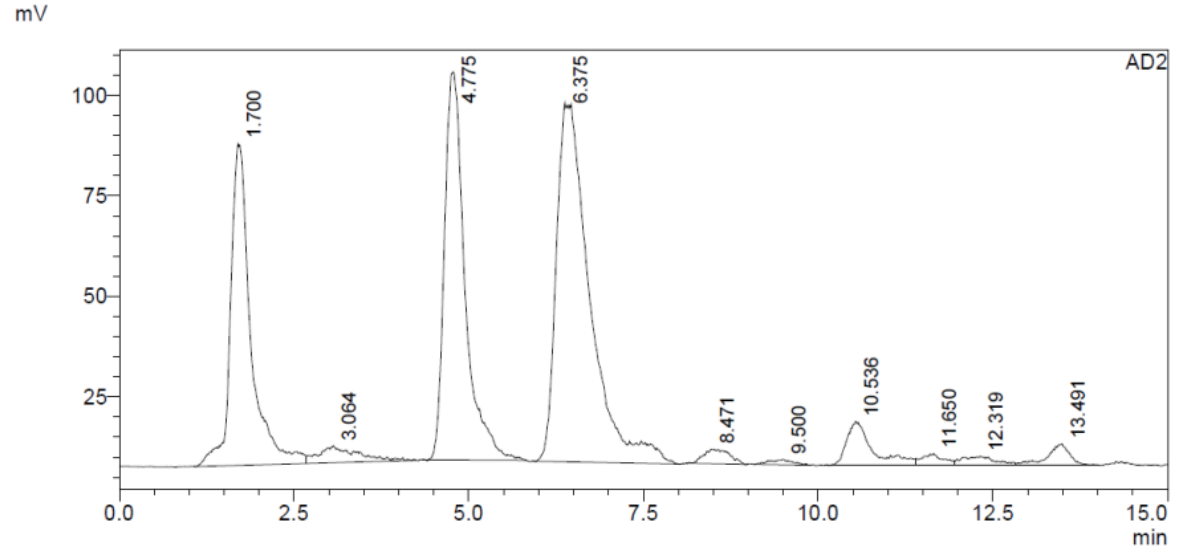
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



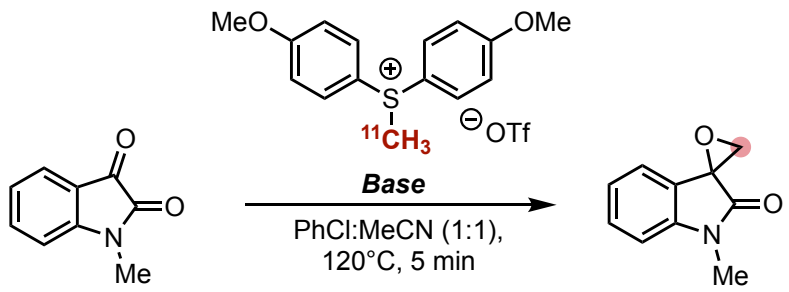
Crude Reaction Mixture – Gamma Detector



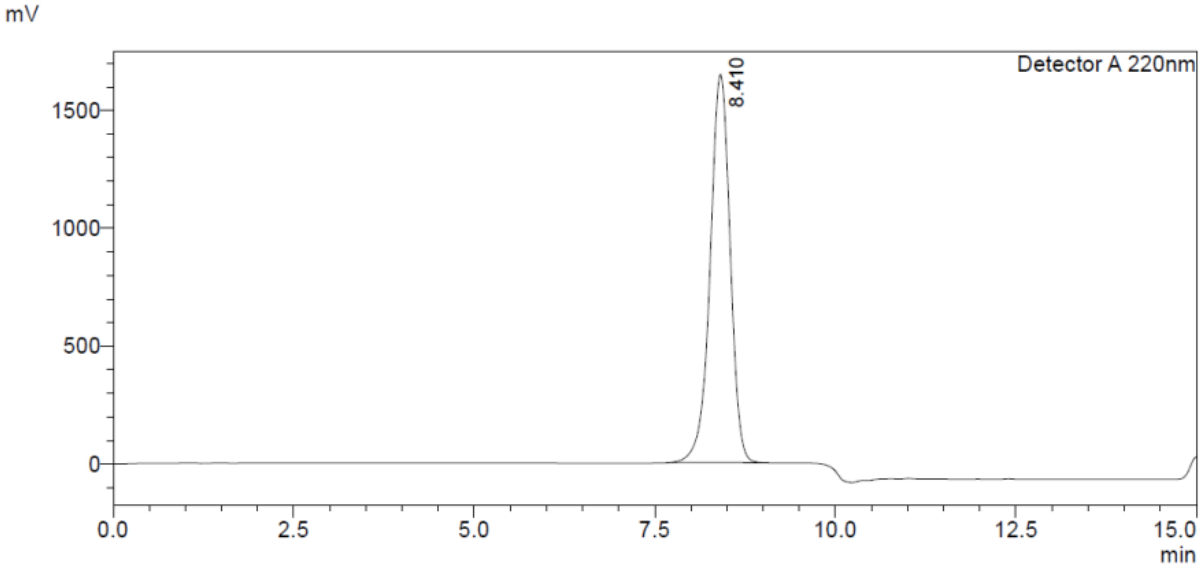
HPLC condition **A (X = 40),** injection volume 5 µL, in crude reaction mixture compound of interest peak at 6.116 (UV) and 6.375 (Rad); RCY **40%.**

4l

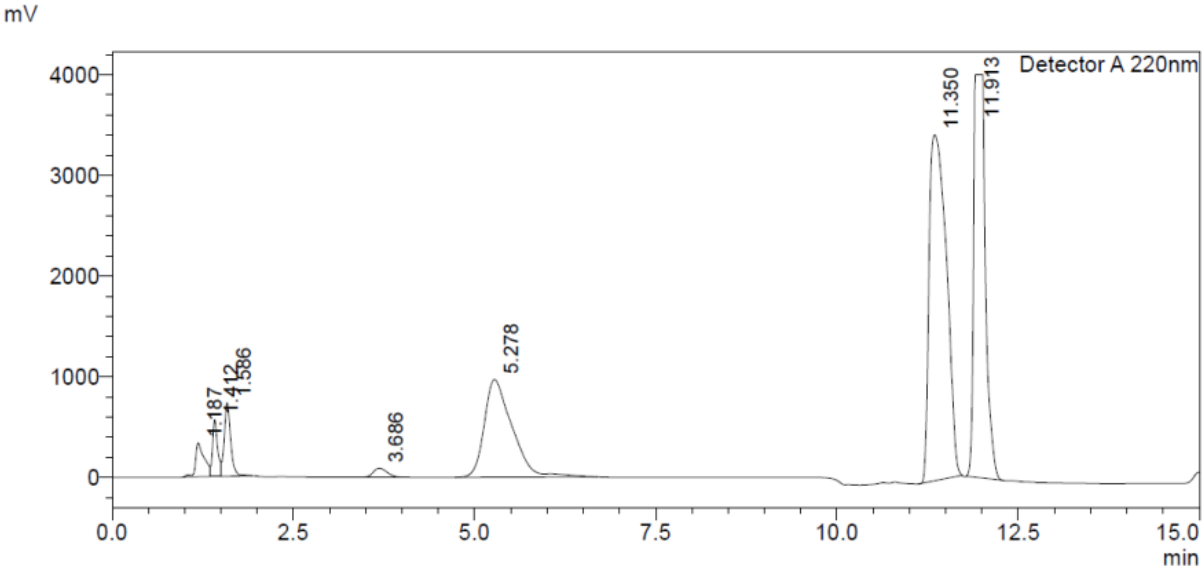
(Base = 1 eq. KOtBu)



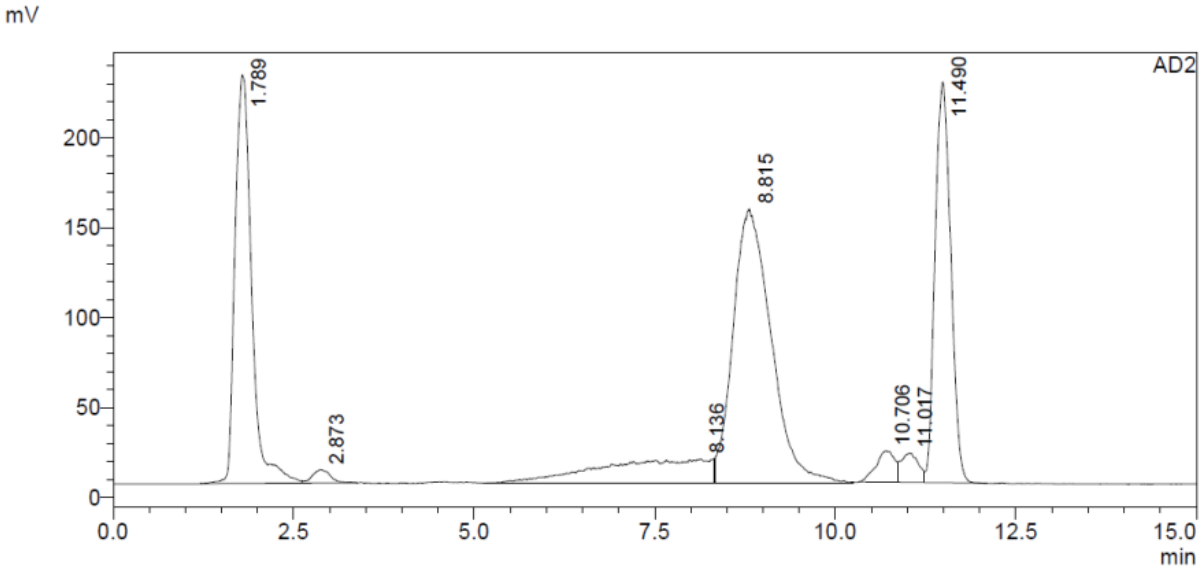
Standard - UV Detector



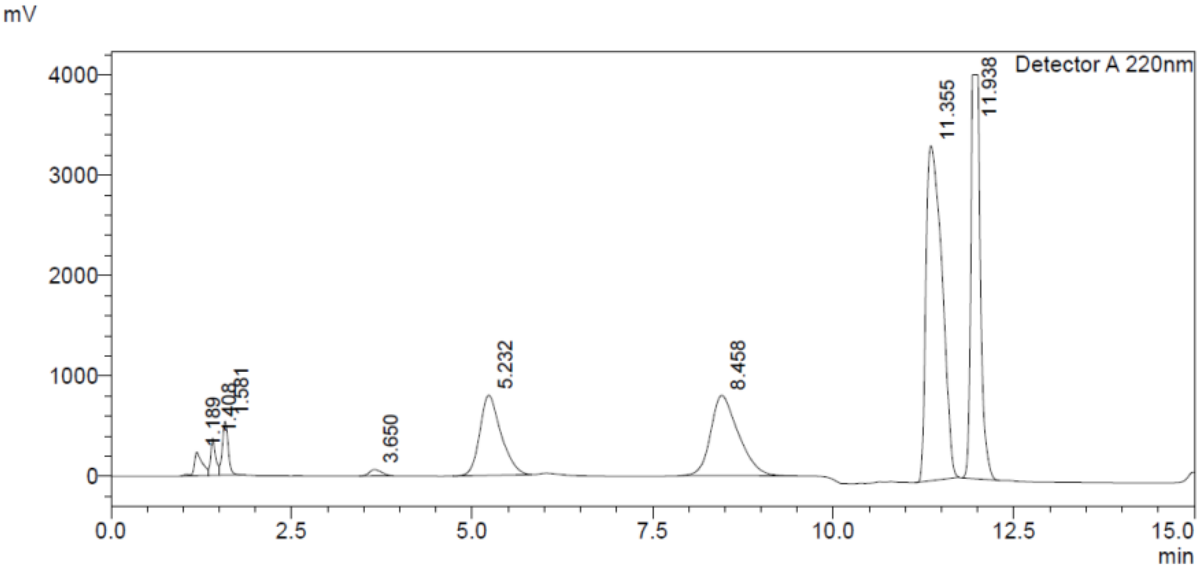
Crude Reaction Mixture (without internal standard) – UV Detector



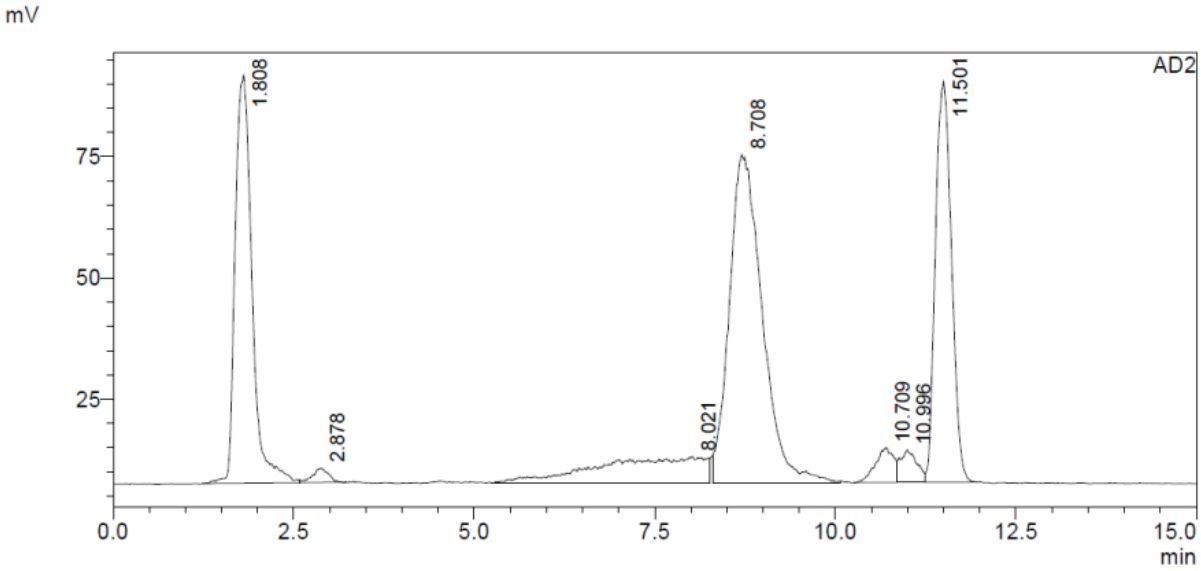
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



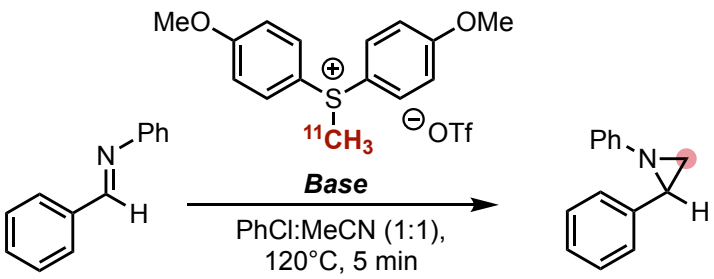
Crude Reaction Mixture – Gamma Detector



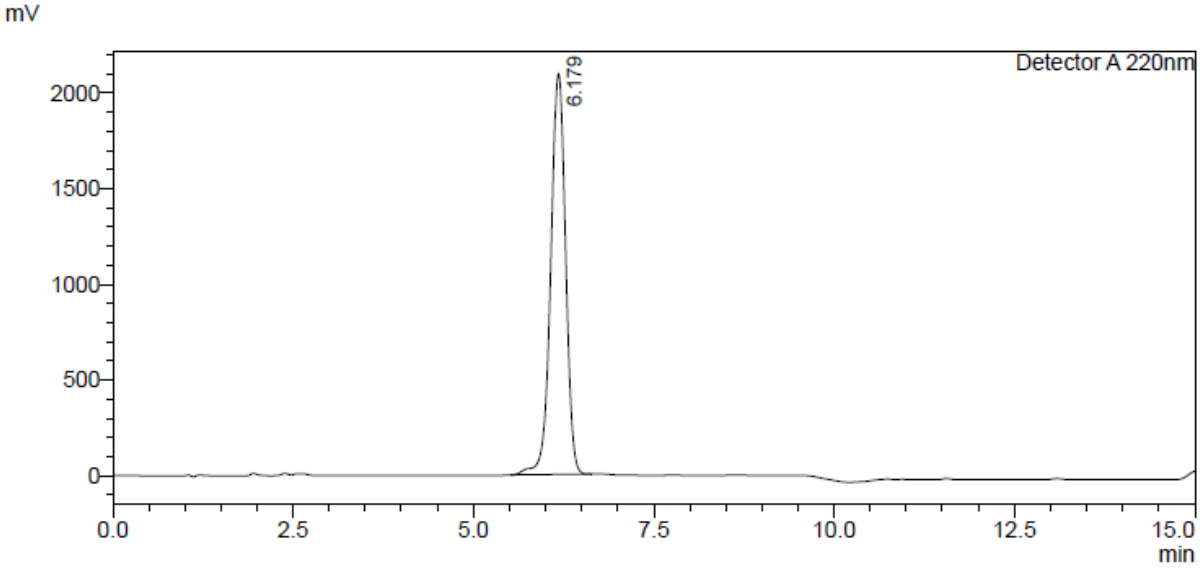
HPLC condition A (X = 20), injection volume 5 µL, peak at 8.458 (UV) and 8.708 (Rad), RCY 38%.

4m

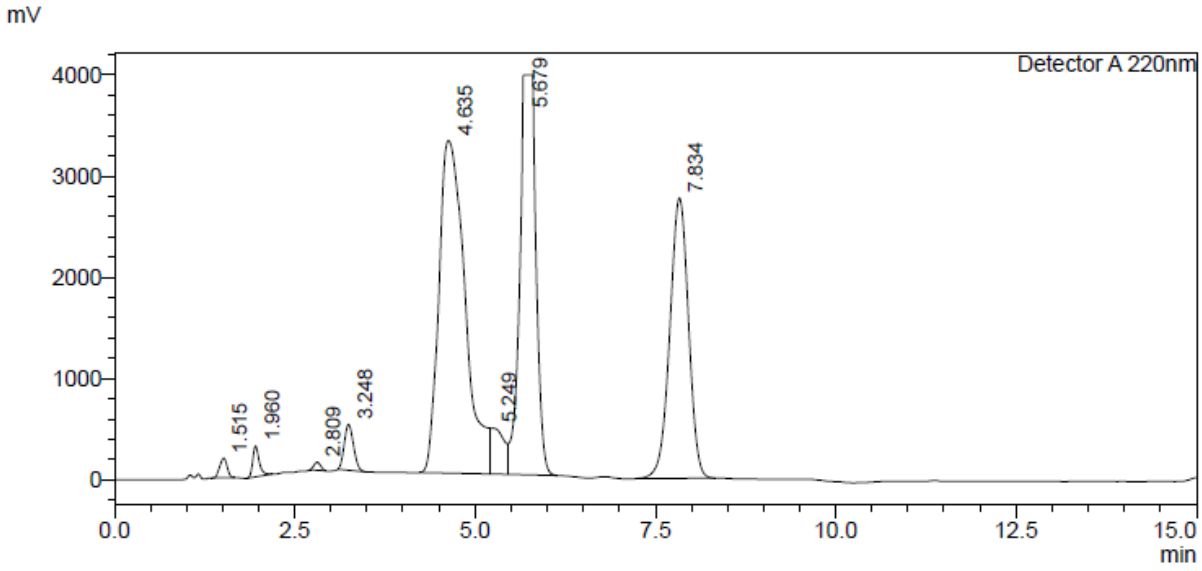
(Base = 1eq. ^{t}BuOK)



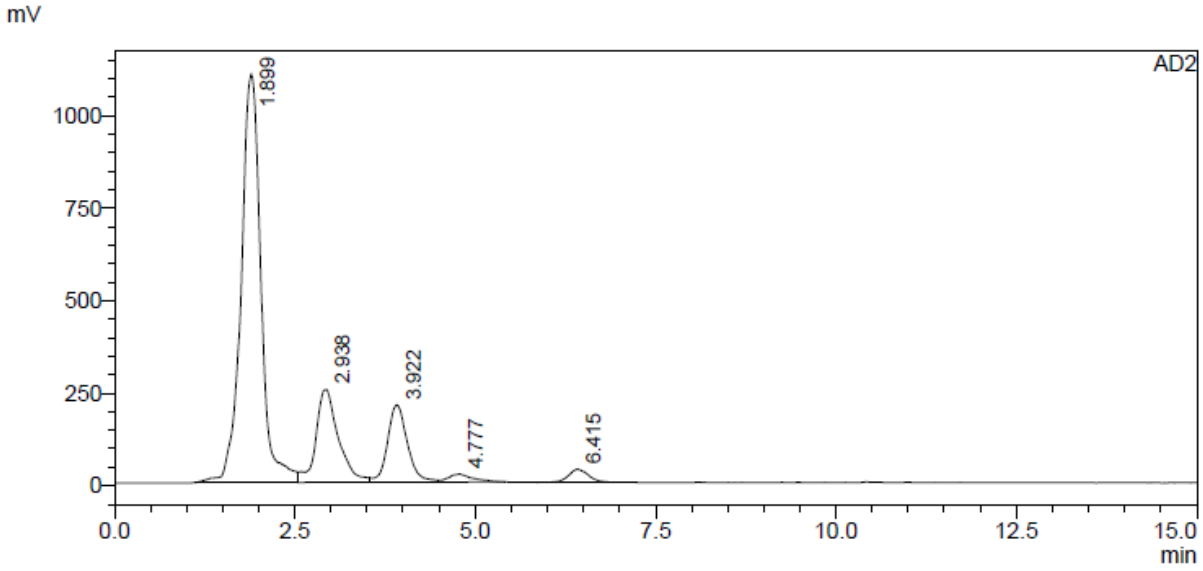
Standard - UV Detector



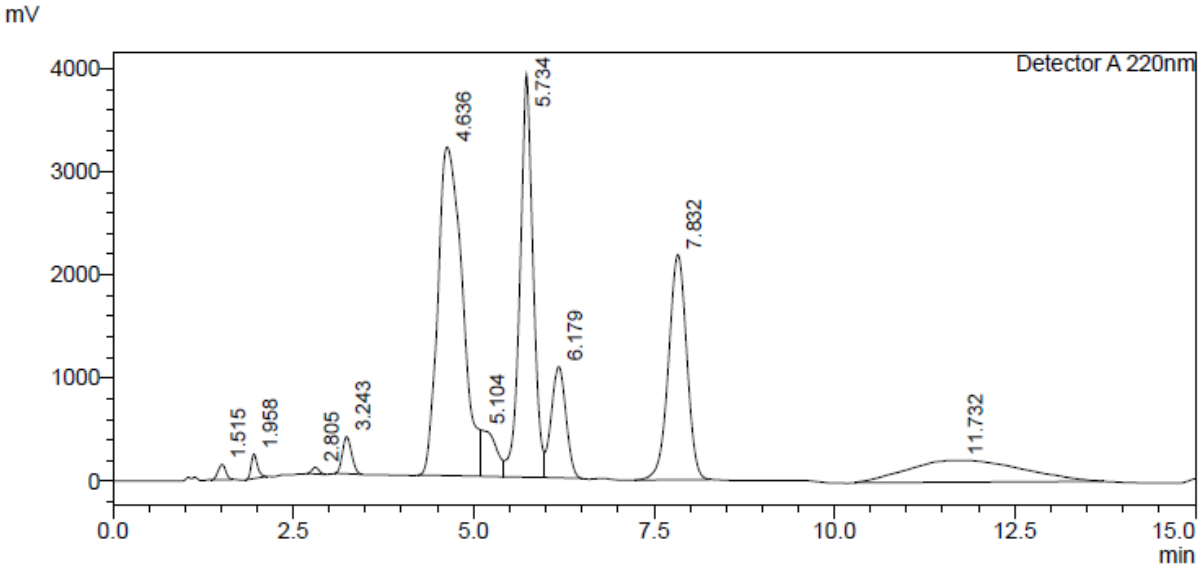
Crude Reaction Mixture (without internal standard) – UV Detector



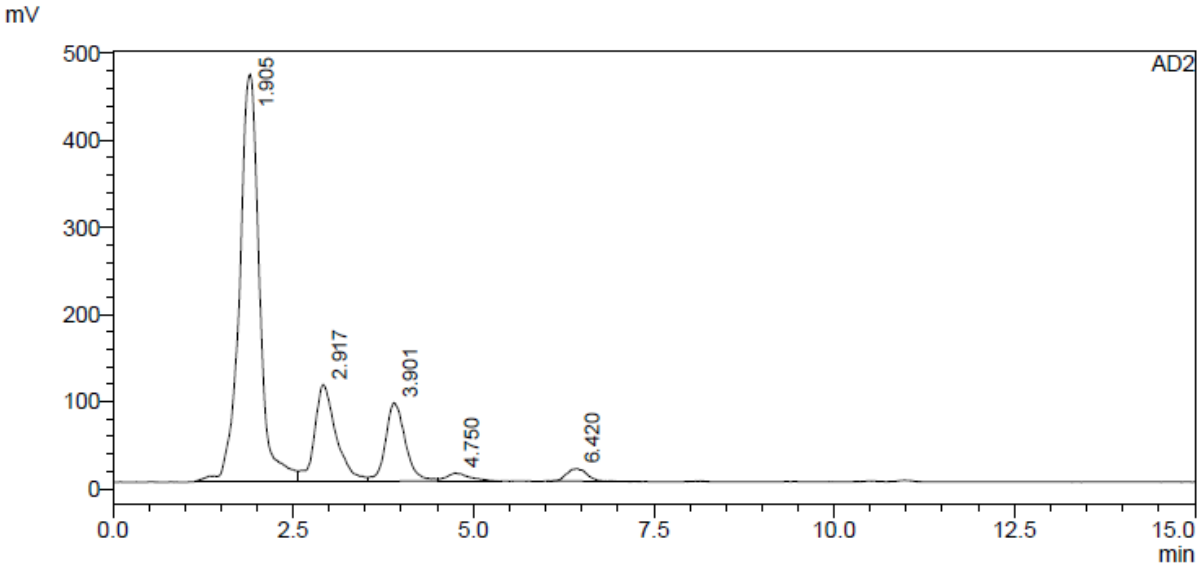
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



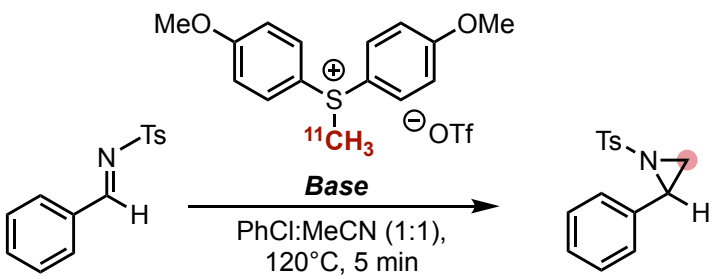
Crude Reaction Mixture – Gamma Detector



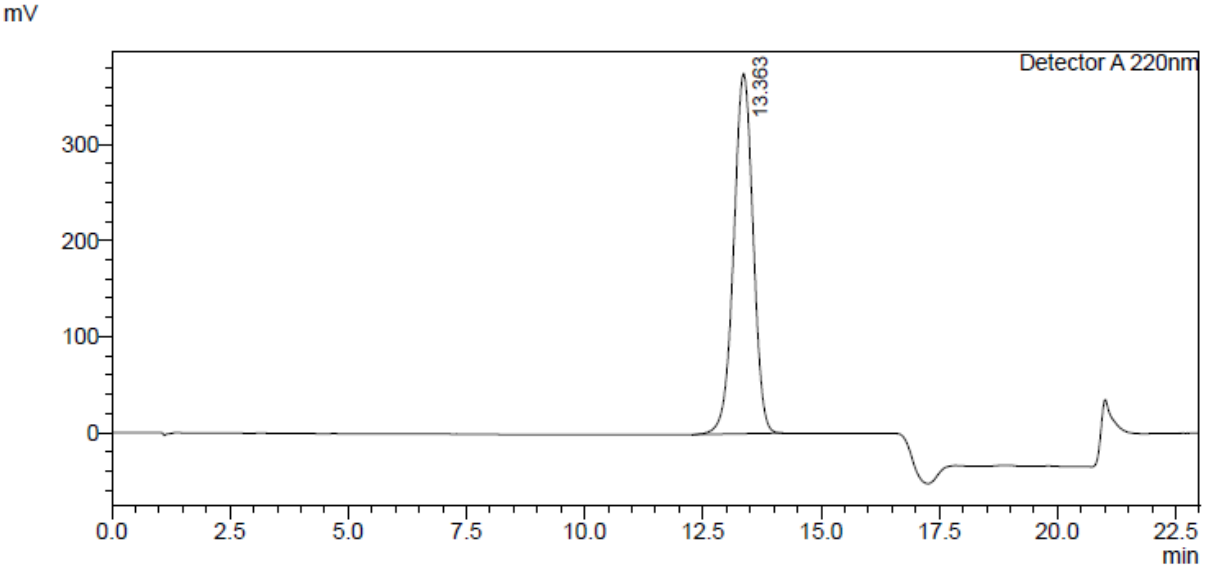
HPLC condition **A (X = 55),** injection volume 5 µL, peak at 6.179 (UV) and 6.420 (Rad), RCY **2%**.

4n

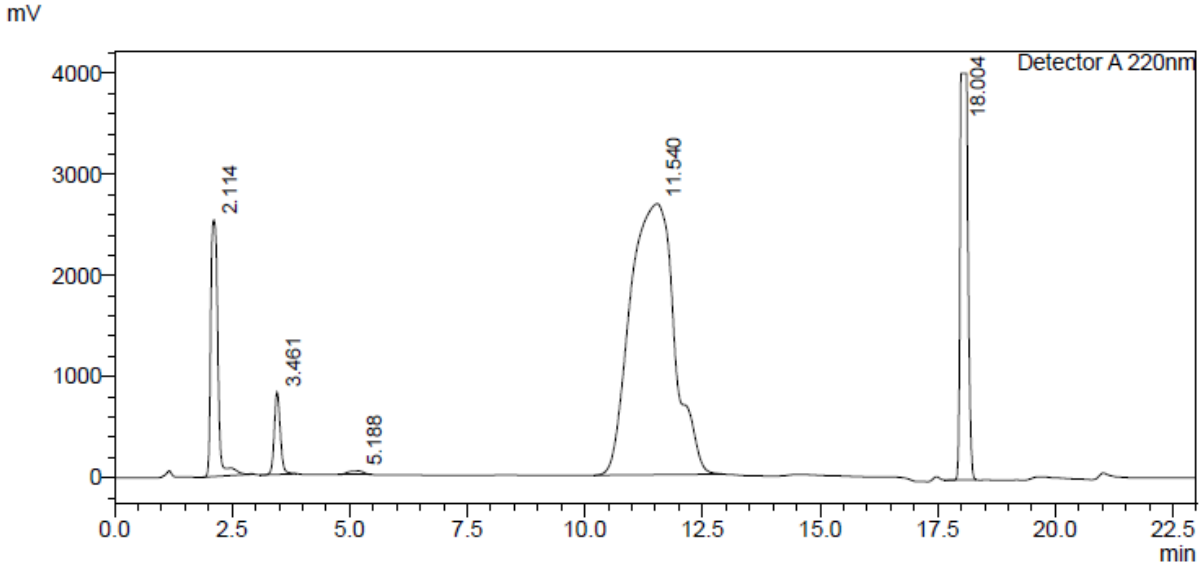
(Base = 1eq. P_2-et)



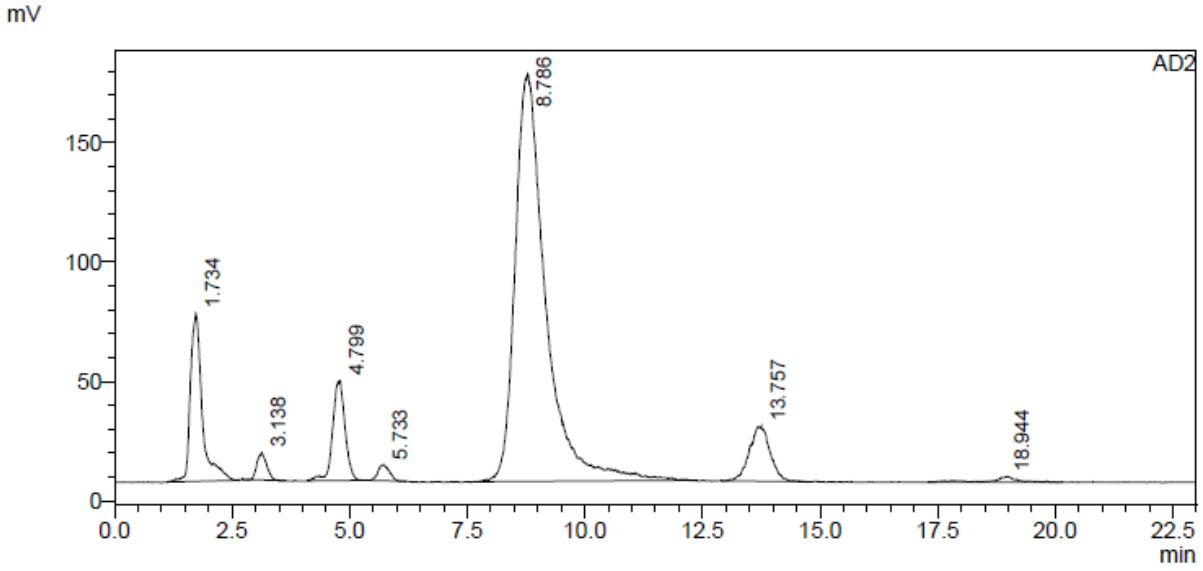
Standard - UV Detector



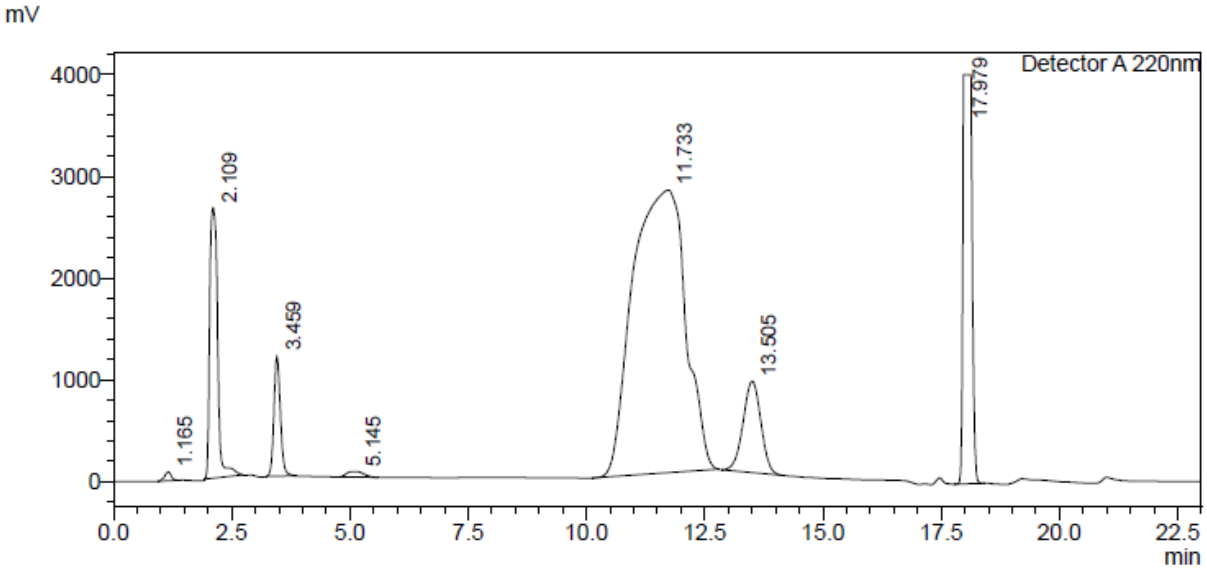
Crude Reaction Mixture (without internal standard) – UV Detector



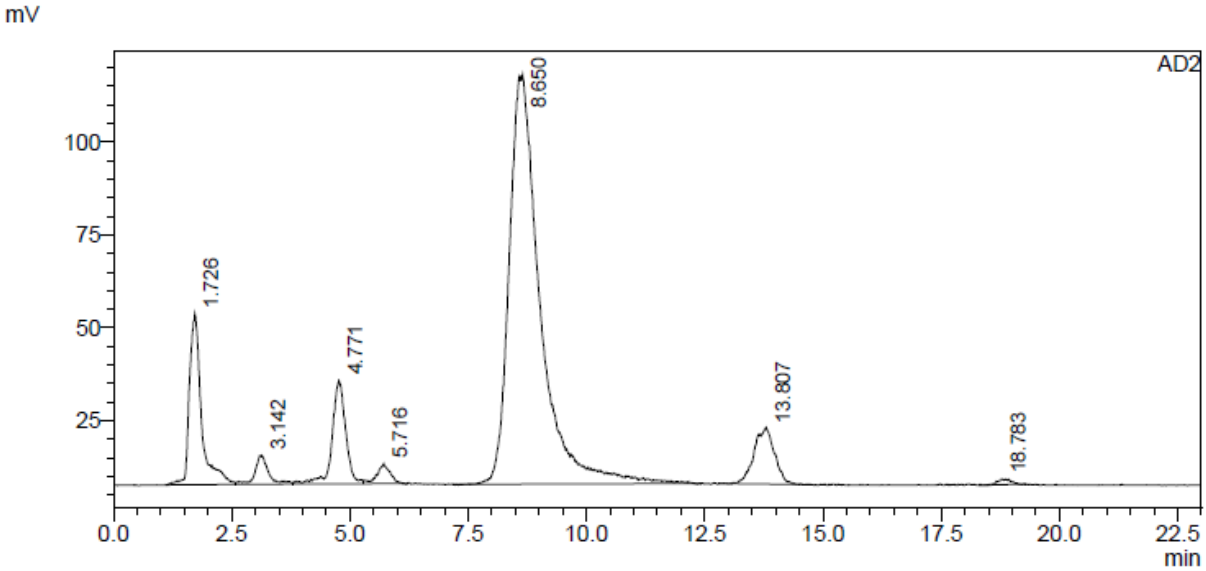
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



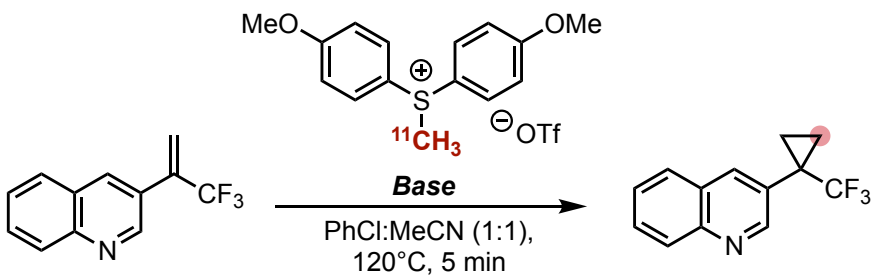
Crude Reaction Mixture – Gamma Detector



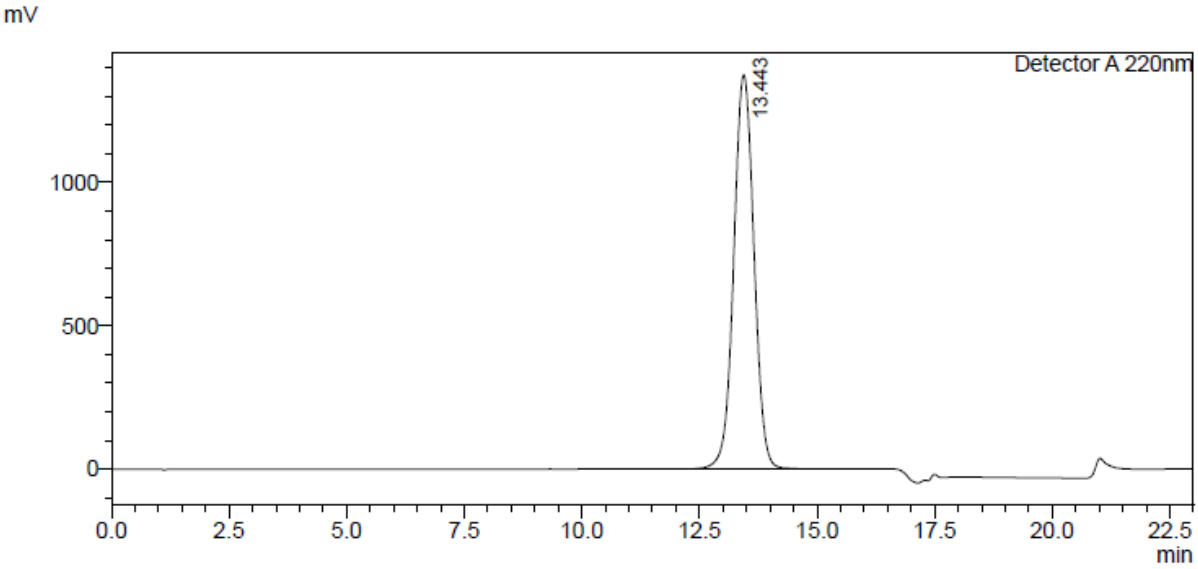
HPLC condition **B (X = 40),** injection volume 5-10 µL, peak at 13.505 (UV) and 13.807 (Rad), RCY **7%**.

4o

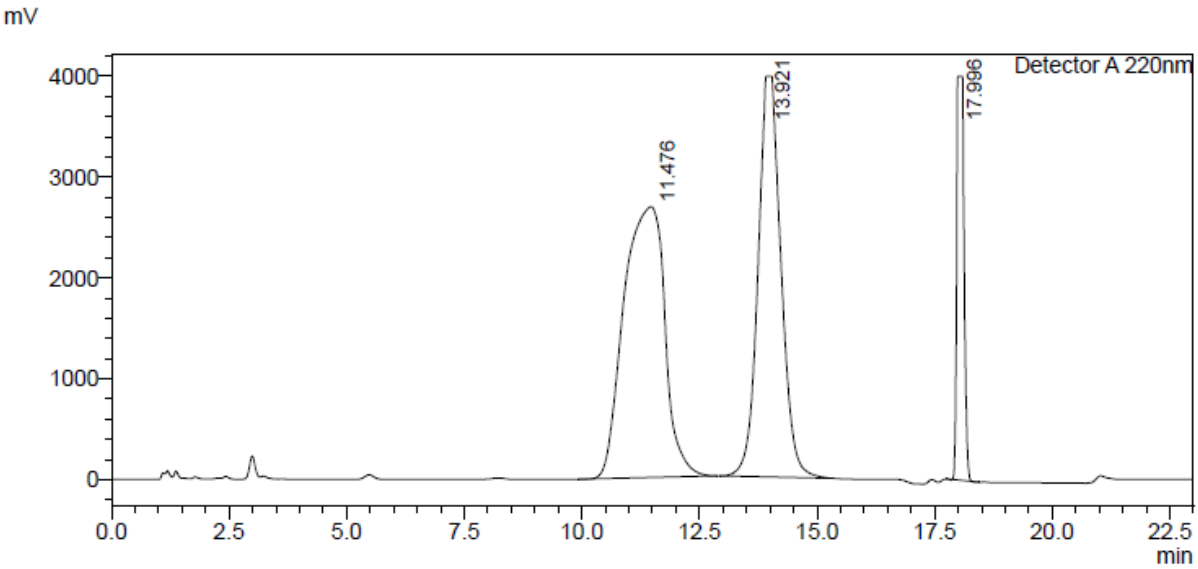
(1eq. ^{t}BuOK)



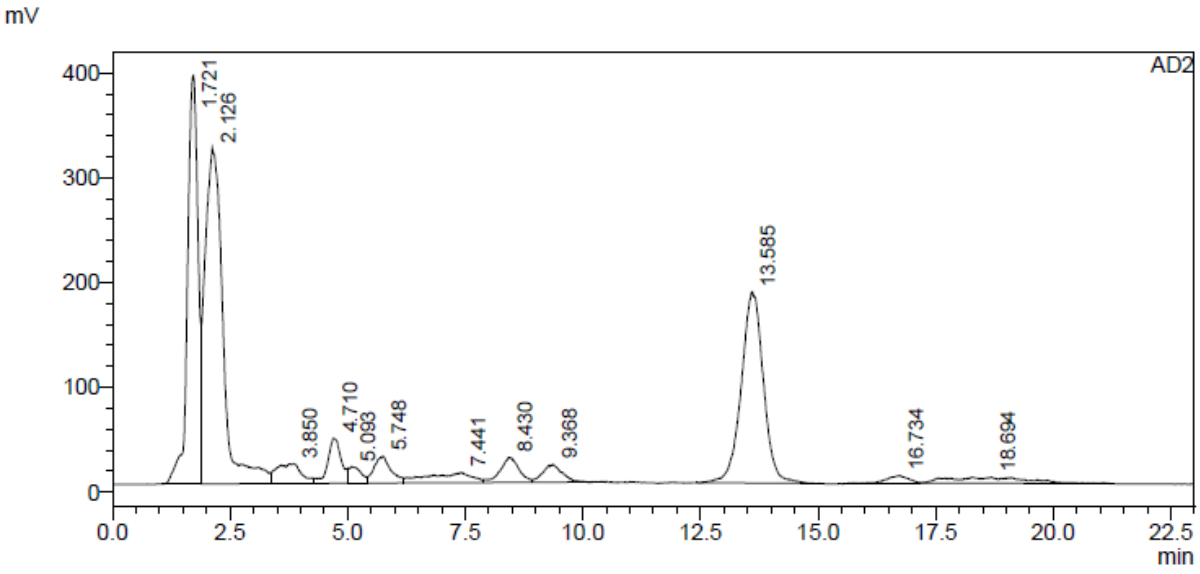
Standard - UV Detector



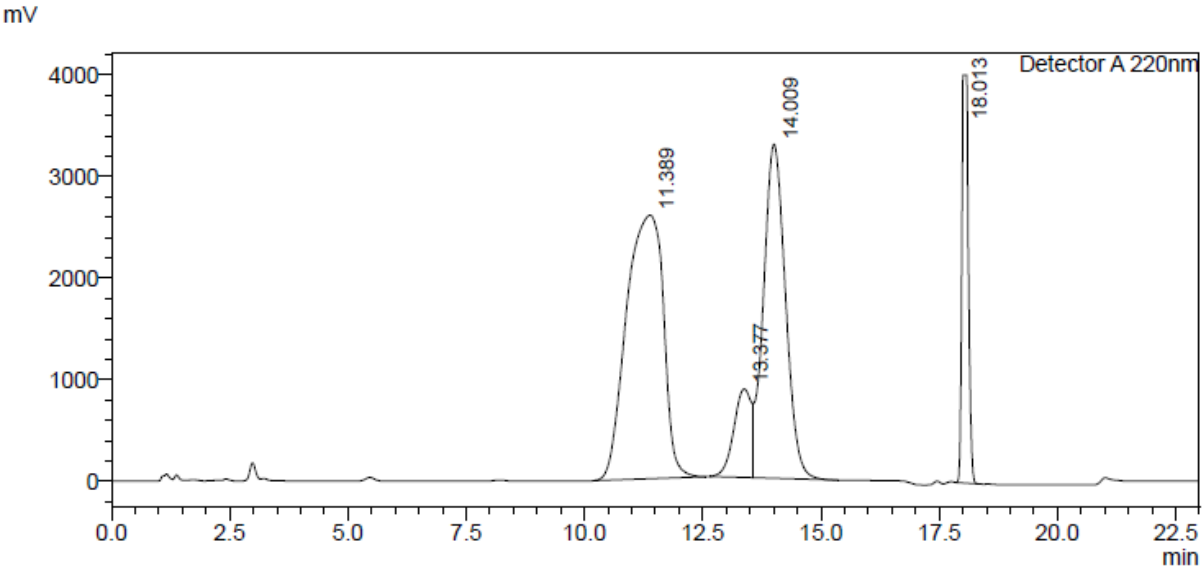
Crude Reaction Mixture (without internal standard) – UV Detector



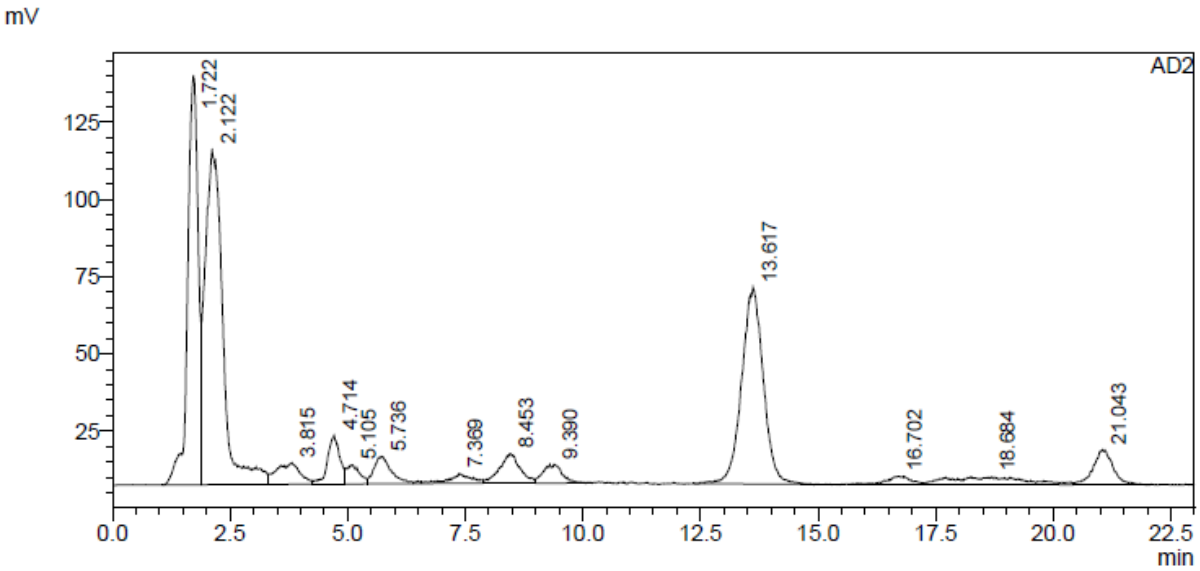
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



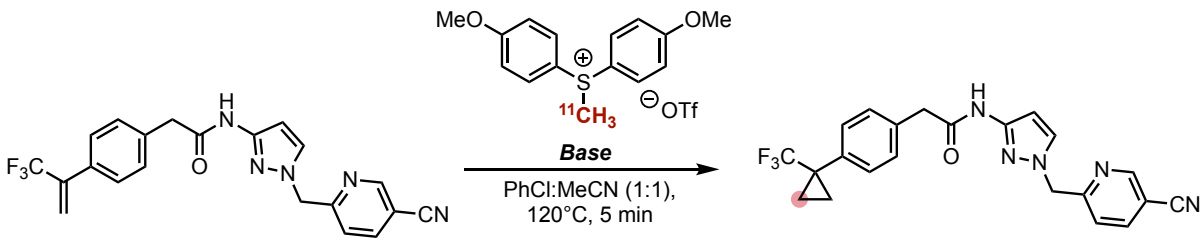
Crude Reaction Mixture – Gamma Detector



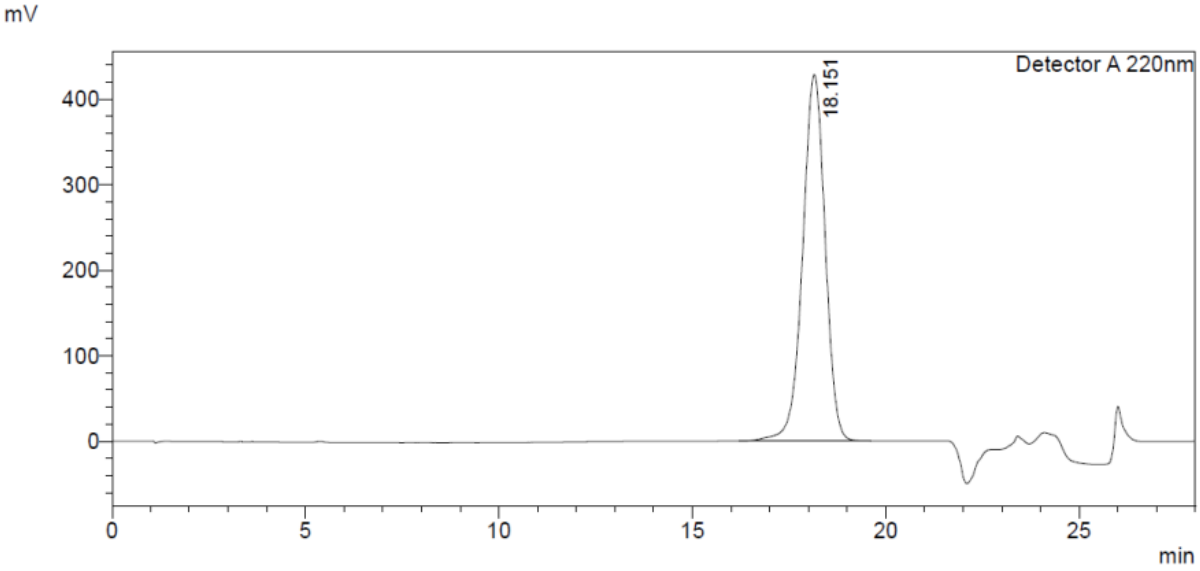
HPLC condition **B (X = 40),** injection volume 5 µL, peak at 13.377 (UV) and 13.617 (Rad), RCY **23%**.

4p

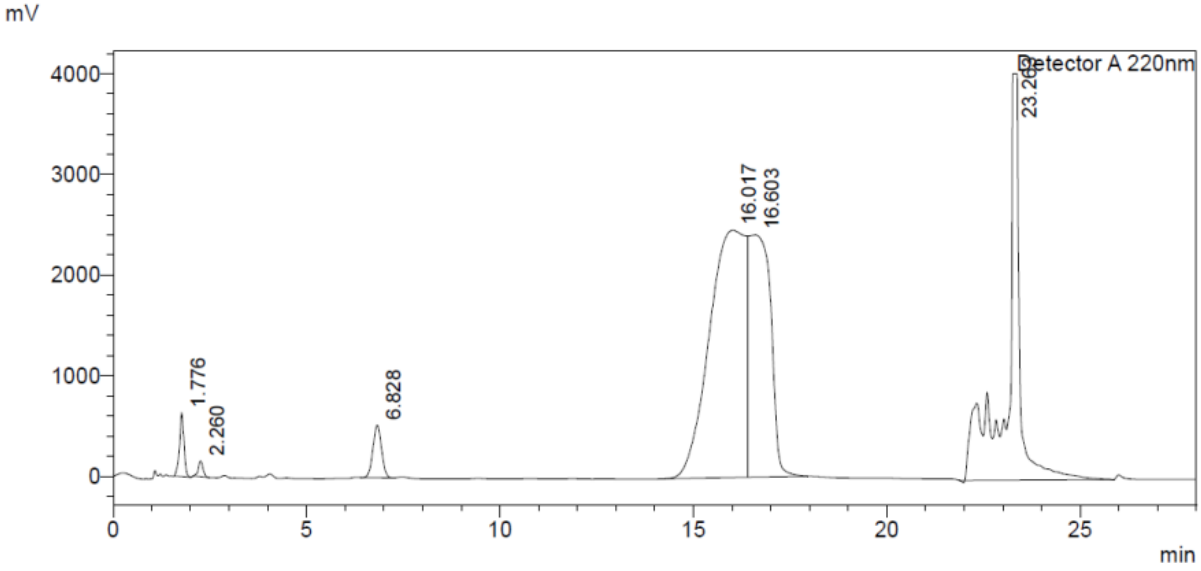
(Base = 2 eq. KOtBu)



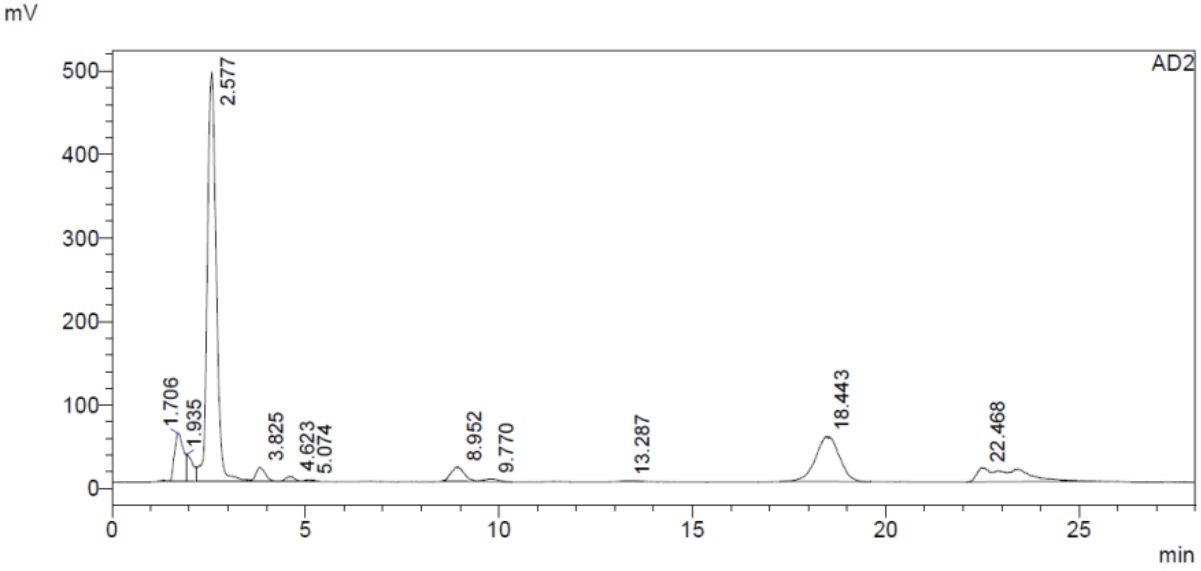
Standard - UV Detector



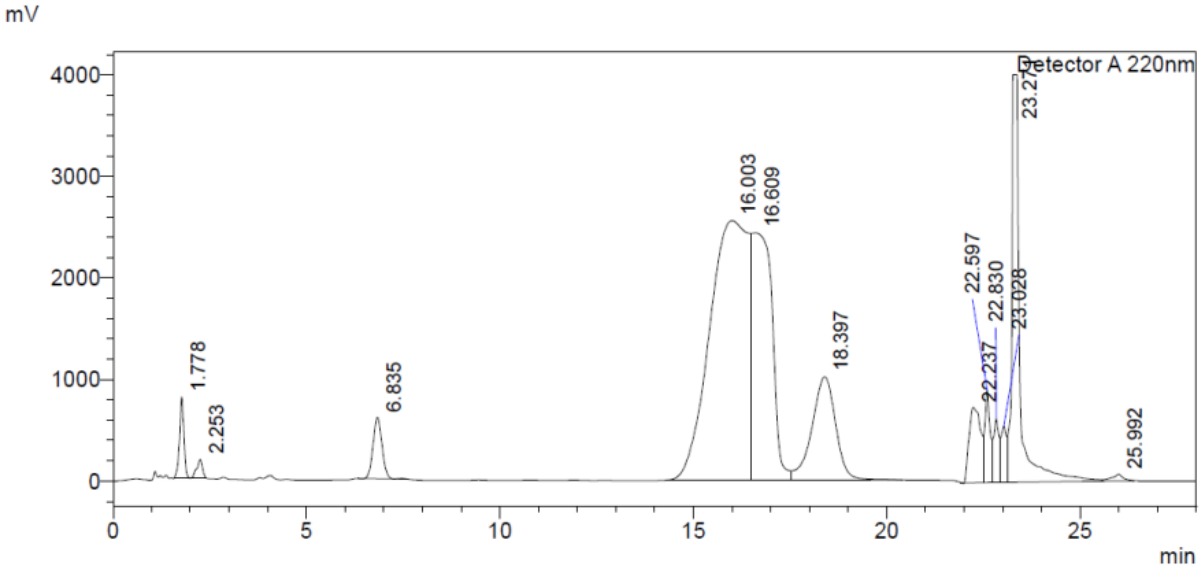
Crude Reaction Mixture (without internal standard) – UV Detector



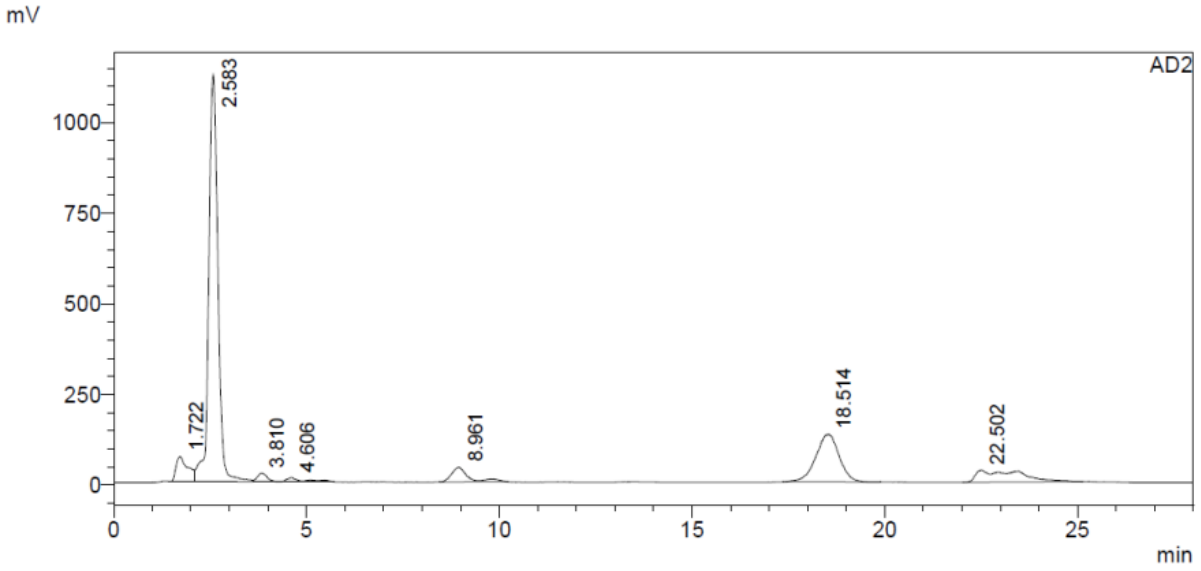
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



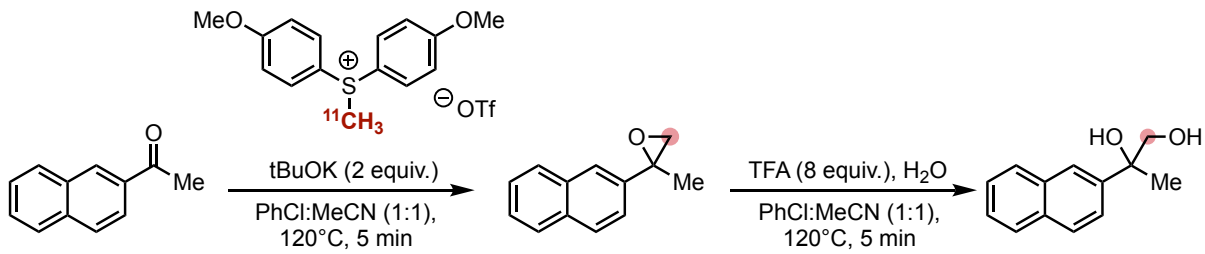
Crude Reaction Mixture – Gamma Detector



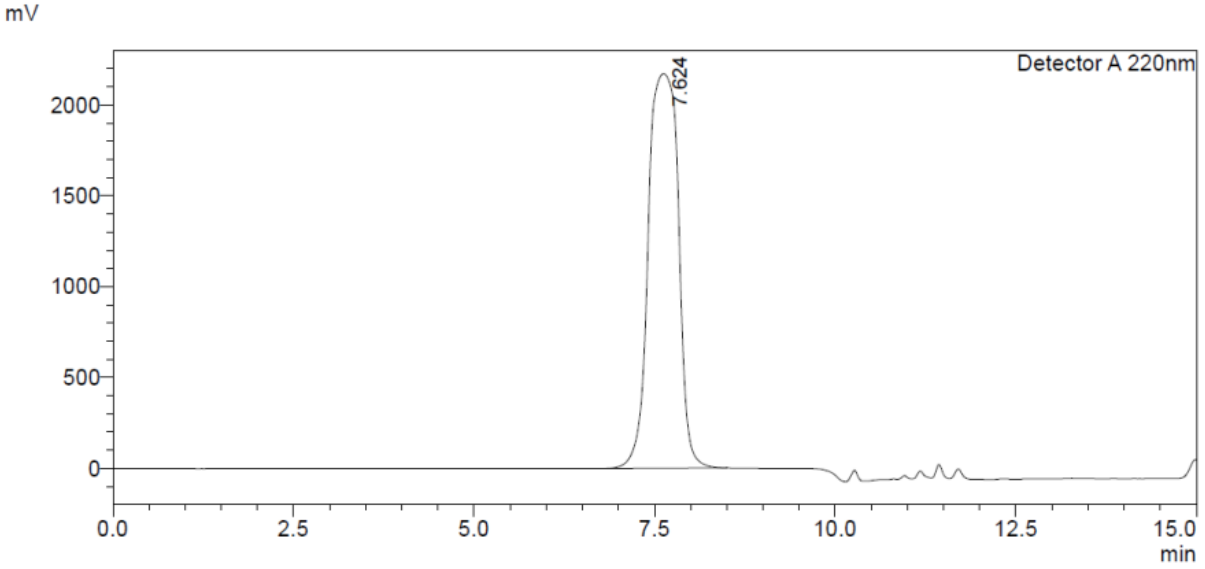
HPLC condition **E (X = 35),** injection volume 5 µL, peak at 18.397 (UV) and 18.514 (Rad), RCY **18%**.

5

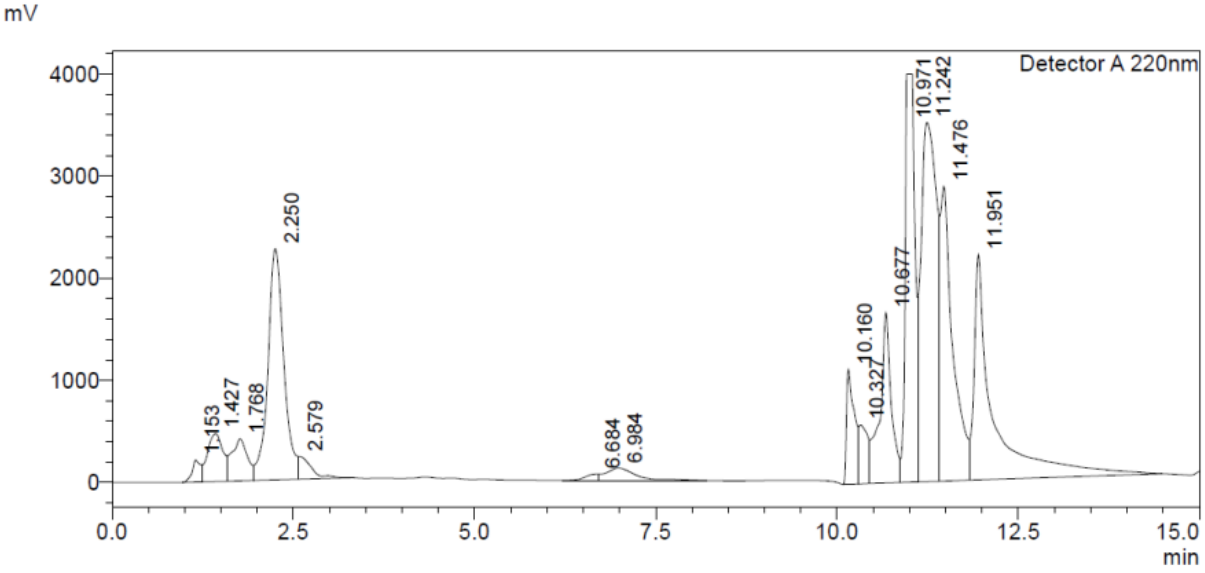
(Base = 2 eq. KOtBu)



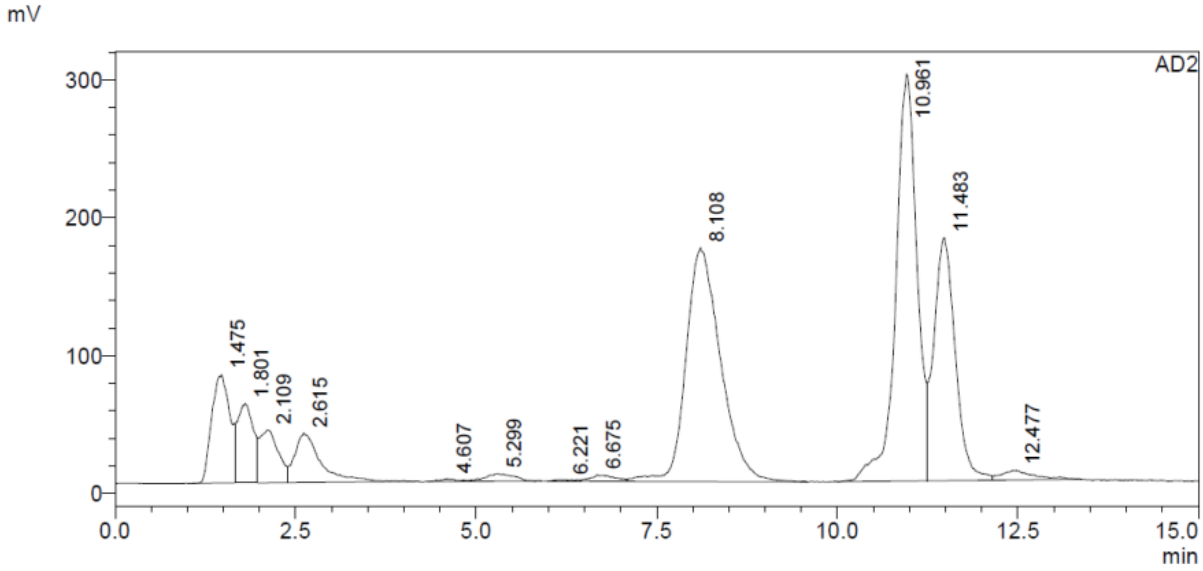
Standard - UV Detector



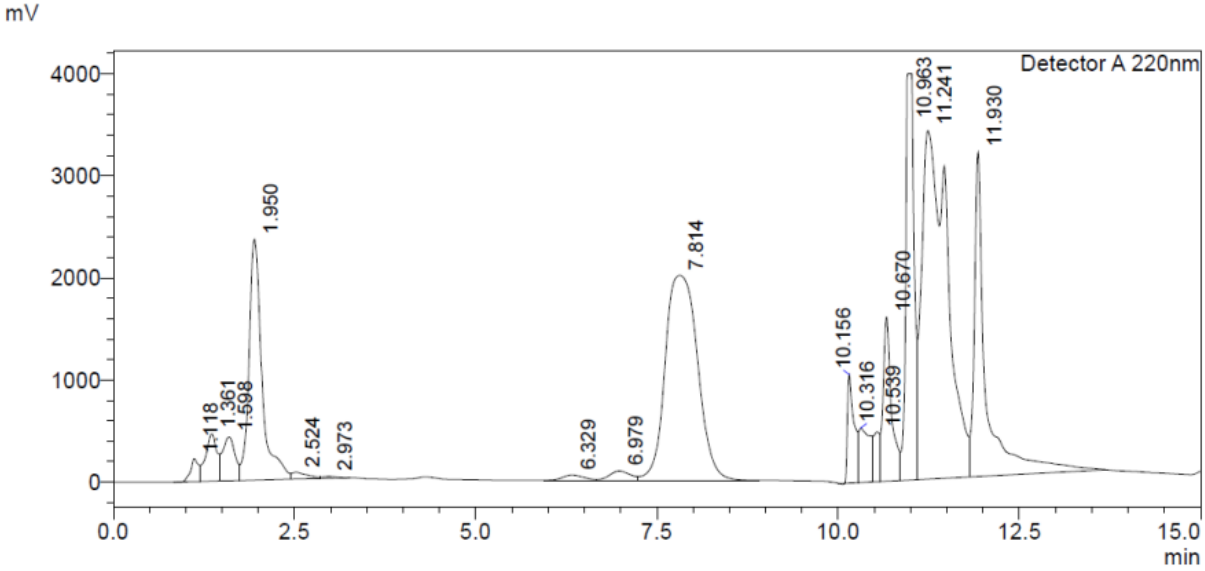
Crude Reaction Mixture (without internal standard) – UV Detector



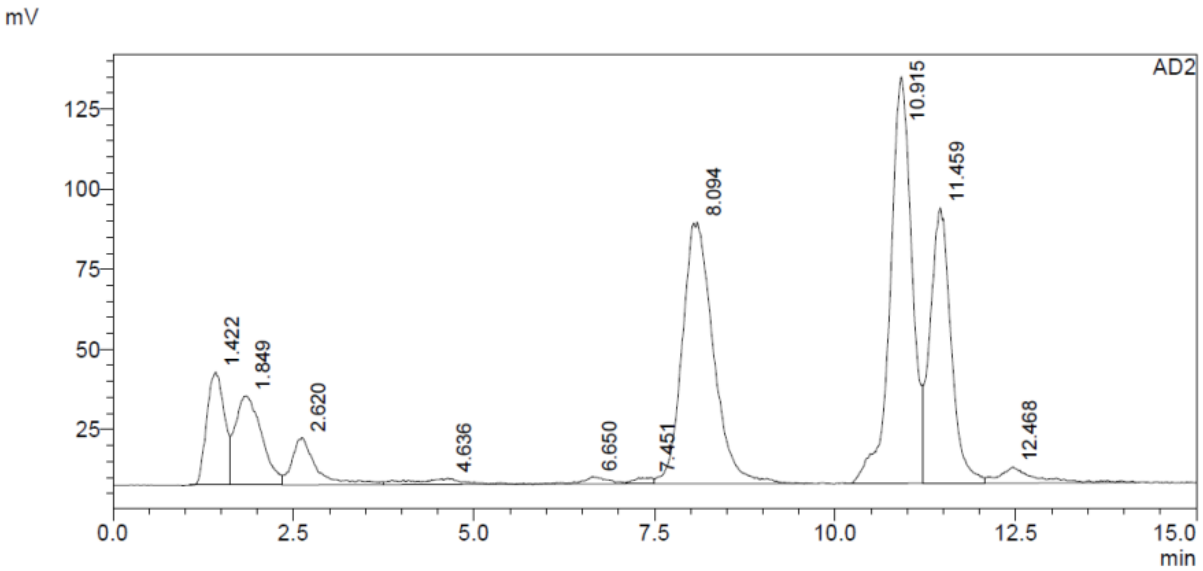
Crude Reaction Mixture – Gamma Detector



Crude Reaction Mixture (with internal standard) – UV Detector



Crude Reaction Mixture – Gamma Detector



HPLC condition **A (X = 25),** injection volume 5 µL, peak at 7.814 (UV) and 8.094 (Rad), RCY **29%.**

References

1. A. Theodorou, G. N. Papadopoulos and C. G. Kokotos, *Tetrahedron*, 2013, **69**, 5438-5443.
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