

Supplementary Material

Novel L-threonine-based ionic liquid supported organocatalyst for asymmetric *syn*-aldol reaction: activity and recyclability design

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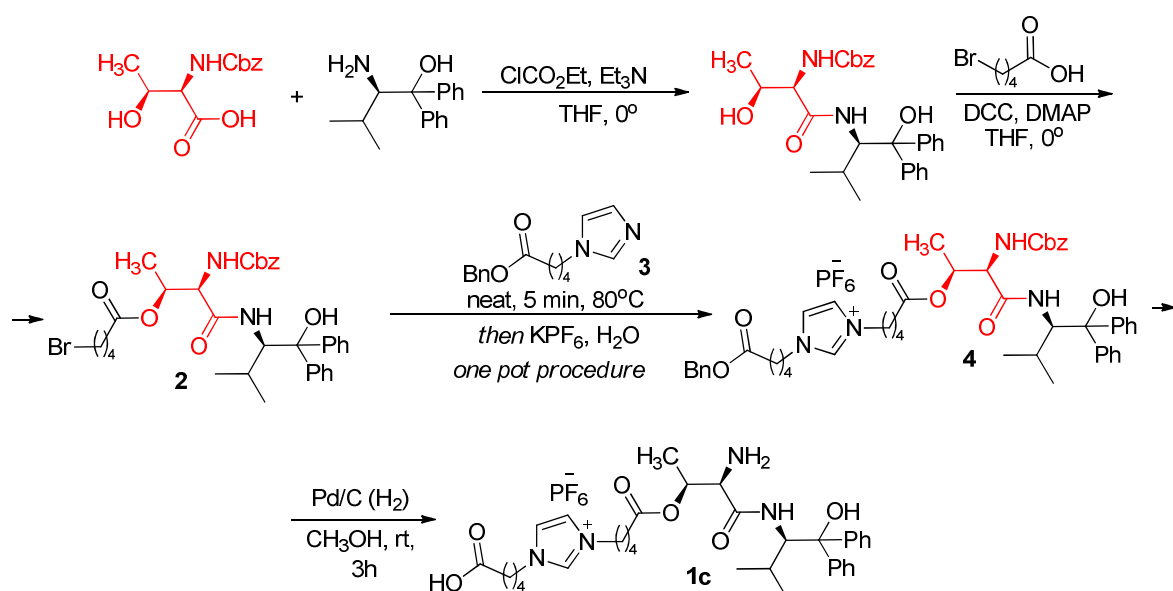
Table of Contents

General information	S2
General scheme of catalyst 1c synthesis	S2
Synthesis and characterization of 4	S3
Synthesis and characterization of 1c	S8
General procedure for <i>syn</i> -aldol reaction	S13
General procedure for recycling experiment	S13
Characterization of (3R,4S)-4-(2,4-dimethoxyphenyl)-3,4-dihydroxybutan-2-one (7i)	S14
HPLC traces for 7	S17
References	S21

General information

^1H and ^{13}C NMR spectra were recorded with a Bruker AM 300 spectrometer in CDCl_3 and $\text{DMSO-}d_6$. The chemical shifts of ^1H and ^{13}C signals were measured relative to Me_4Si or CDCl_3 , respectively. The high-resolution mass spectra (HRMS) were measured with a Bruker microTOF II spectrometer using electrospray ionization (ESI). The measurements were taken either in the positive ion mode (interface capillary voltage 4500 V) or in the negative ion mode (3200 V) in a mass range $m/z = 50\text{--}3000$ Da; external or internal calibration was done with electrospray calibrant solution (Fluka). Syringe injection was used for solution in $\text{MeCN}/\text{H}_2\text{O}$ (1:1, v/v) (flow rate $3\ \mu\text{L}/\text{min}$). Nitrogen was applied as a dry gas, and the interface temperature was set at $180\ ^\circ\text{C}$. Silica gel $0.060\text{--}0.200\ \mu\text{m}$ (Acros) was used for column chromatography. Threonineamide **2** and benzyl 5-(1Himidazol-1-yl)pentanoate **3** were synthesized according to known methods. Compounds **5** and **6** were purchased from Aldrich and used without purification. The solvents were purified by standard procedures. For experimental details and spectral or HPLC data see Supporting Information.

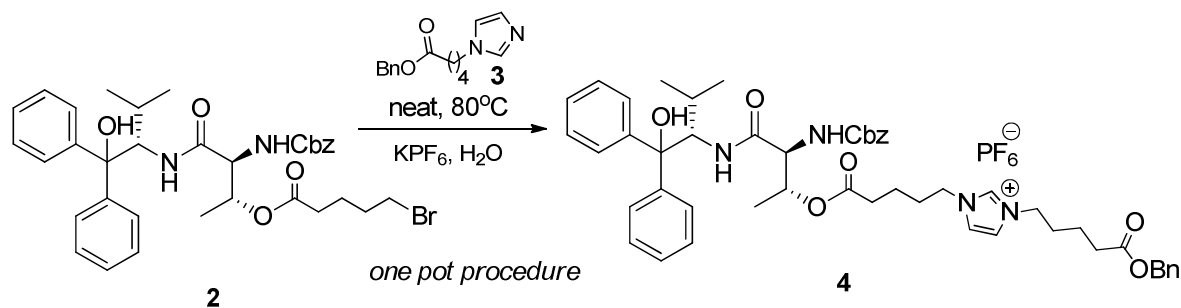
General scheme of catalyst **1c** synthesis



Steps before compound **2** are described in ref [1].

Synthesis and characterization of 4

3-(5-(benzyloxy)-5-oxopentyl)-1-(5-(((2R,3S)-3-(((benzyloxy)carbonyl)amino)-4-(((S)-1-hydroxy-3-methyl-1,1-diphenylbutan-2-yl)amino)-4-oxobutan-2-yl)oxy)-5-oxopentyl)-1H-imidazol-3-ium hexafluorophosphate



Benzyl 5-(1H-imidazol-1-yl)pentanoate **4** (0.22 g, 0.83 mmol) was gradually added to a solution of (2R,3S)-3-(((benzyloxy)carbonyl)amino)-4-(((S)-1-hydroxy-3-methyl-1,1-diphenylbutan-2-yl)amino)-4-oxobutan-2-yl 5-bromopentanoate **3** (0.45 g, 0.69 mmol) in CH₃OH (2 mL). The reaction mixture was kept at ambient temperature for 10 min and evaporated under reduced pressure (20 Torr) at 40 °C. The residue was heated at the same pressure (rotary evaporator, 80 °C) for 5 min, cooled to ambient temperature and diluted with distilled water (3.0 mL). A solution of KPF₆ (128 mg, 0.69 mmol) in distilled water (1.5 mL) was added to the resulting aqueous solution and the reaction mixture was stirred for 1h at ambient temperature. The precipitate was filtered and washed successively with distilled water (3 x 3 mL) and then 2x1 mL of Et₂O, then dried on filter to obtain 0.612 g (90%) of white powder 3-(5-(benzyloxy)-5-oxopentyl)-1-(5-(((2R,3S)-3-(((benzyloxy)carbonyl)amino)-4-(((S)-1-hydroxy-3-methyl-1,1-diphenylbutan-2-yl)amino)-4-oxobutan-2-yl)oxy)-5-oxopentyl)-1H-imidazol-3-ium hexafluorophosphate **4**. White powder, m.p. = 97-100 °C,

¹H NMR (600 MHz, DMSO-*d*₆): 0.65 (d, *J* = 6.5 Hz, 3H, CH₃); 0.70 (d, *J* = 6.5 Hz, 3H, CH₃); 0.87 (d, *J* = 6.5 Hz, 3H, CH₃); 1.38-1.45 (m, 2H, CH₂); 1.48-1.55 (m, 2H, CH₂); 1.69-1.78 (m, 3H, CH₂ + CH(CH₃)₂); 1.78-1.85 (m, 2H, CH₂); 2.13-2.24 (m, 2H, CH₂); 2.40 (t, *J* = 7.3 Hz, 2H, CH₂); 3.99 (t, *J* = 8.2 Hz, 1H, CH); 4.11 (t, *J* = 6.9 Hz, 2H, CH₂); 4.18 (t, *J* = 6.9 Hz, 2H, CH₂); 4.84 (m, 1H, CH); 4.89 (d, *J* = 9.5 Hz, 1H, CH); 5.04 (2H, CH₂ AB system, *J*_{HH}=12.66 Hz); 5.10 (s, 2H, CH₂); 5.64 (s, 1H, OH); 7.08 (t, *J* = 7.2 Hz, 1H, CH); 7.13-7.21 (m, 3H, CH); 7.26-7.41 (m, 12H, CH); 7.46-7.55 (m, 4H, CH); 7.60 (d, *J* = 10.0 Hz, 1H, NH); 7.71 (d, *J* = 8.9 Hz, 1H, NH); 7.79 (d, *J* = 5.0 Hz, 2H, NCHCHN); 9.15-9.24 (m, 1H, NCHN);

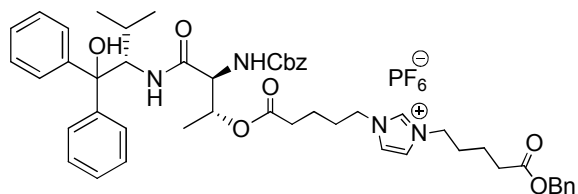
¹³C NMR (125.76 MHz, DMSO-*d*₆): 16.6, 18.2, 21.3, 21.4, 23.2, 29.05, 29.15, 33.1, 33.2, 48.9, 58.2, 59.2, 65.9, 69.8, 81.3, 122.9, 125.7, 125.8, 126.6, 128.0, 128.1, 128.3, 128.4, 128.5, 128.8, 128.9, 136.4, 136.6, 137.5, 146.5, 147.7, 156.5, 169.3, 172.1, 172.9;

Elemental analysis calcd for C₄₉H₅₉F₆N₄O₈P: C, 60.24; H, 6.09; N, 5.73; found: C, 60.06; H, 6.14, N, 5.79.

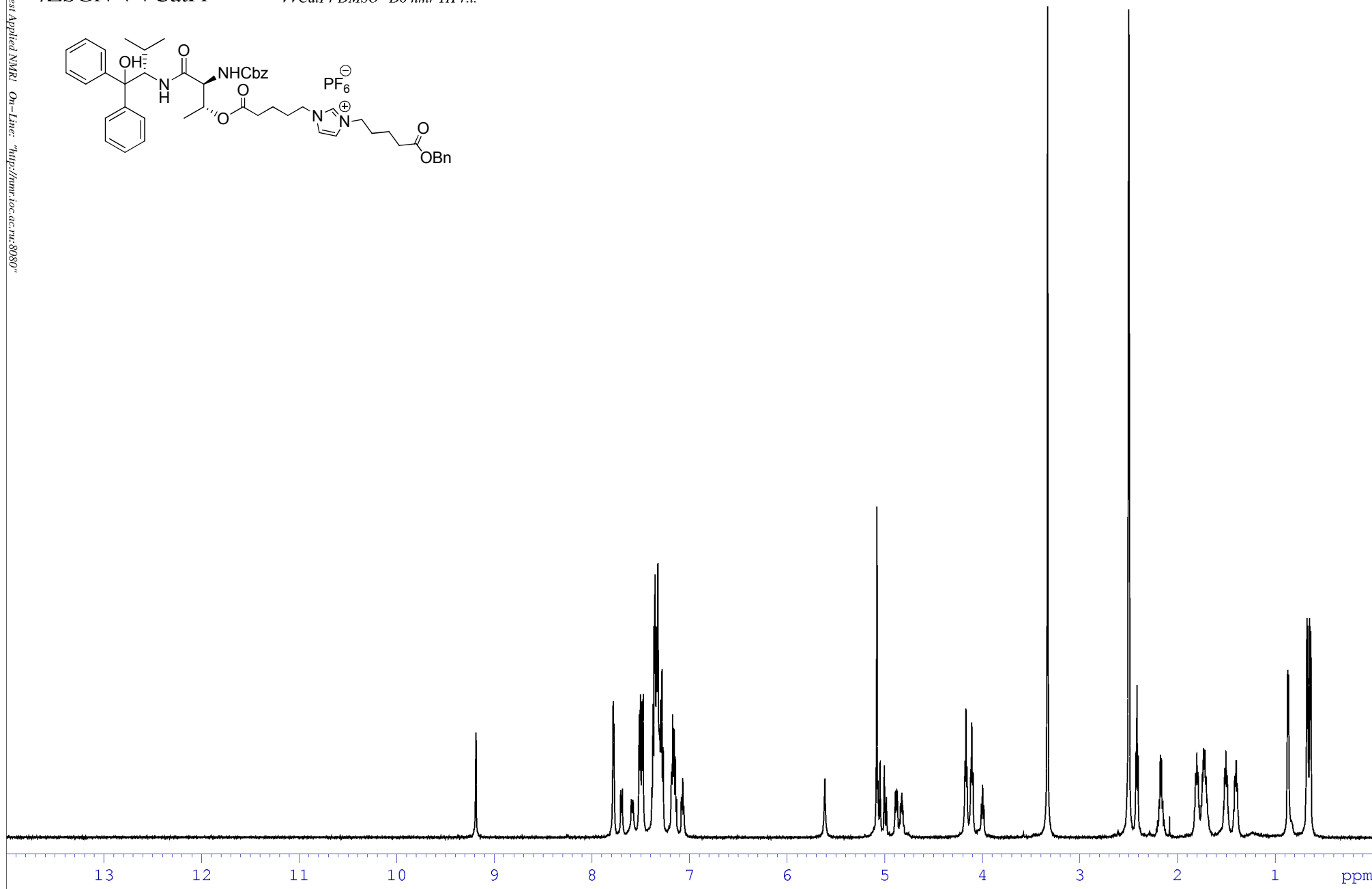
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker AV600 SF=600.13 MHz {1H} SI=32K SW=12019 O1=4800 PW=10.0 AQ=2.721 RD=0.10 NS=1 SR=7.51 TE=296K 10 February 2016 Opr: Fakhrutdinov A.N.; Solv: DMSO-d6;

/ZSGN VVCatPr

VVCatPr DMSO-D6 nmr 1H r.t.



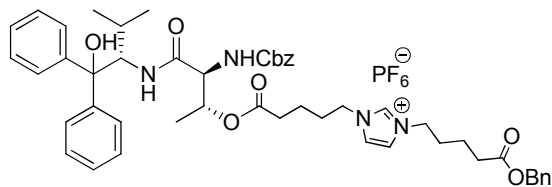
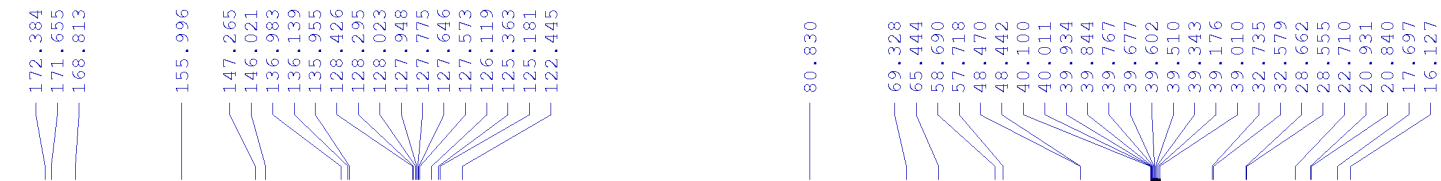
The Best Applied NMR On-Line: <http://nmr.toc.ac.cn:8080/>



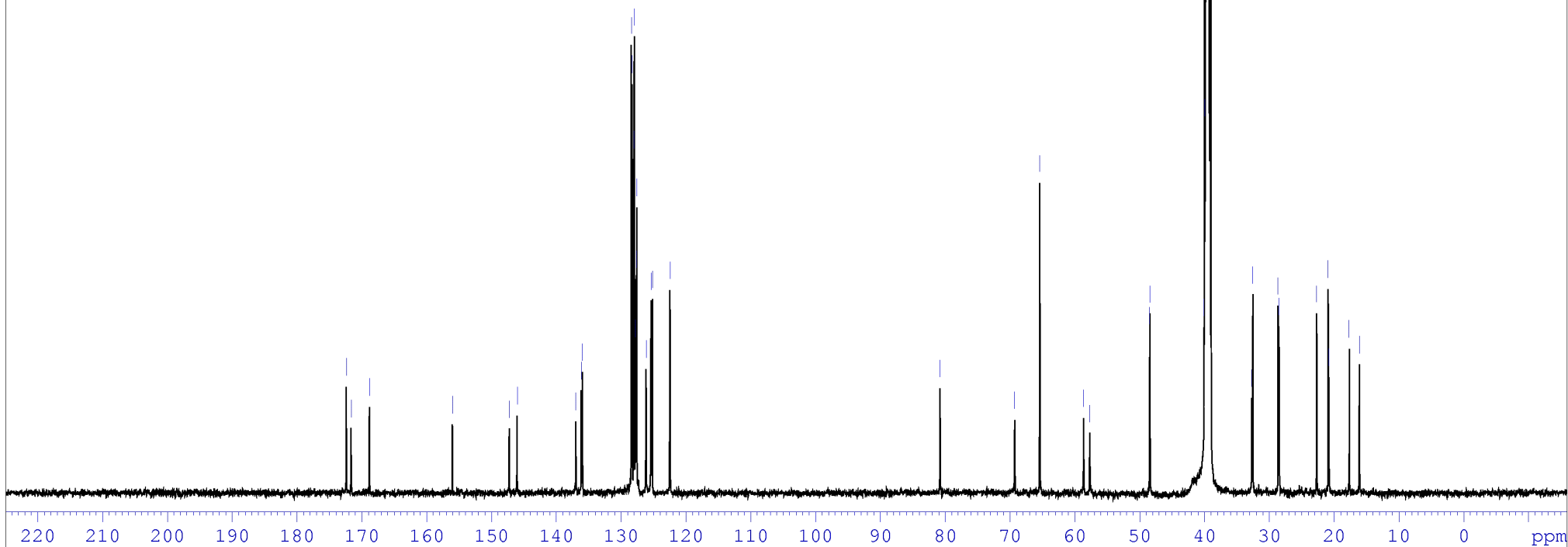
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker DRX500 SF=125.76 MHz {13C} SI=64K SW=30300 O1=13205 PW=12.0 AQ=1.079 RD=0.60 NS=16384 SR=62.39 TE=298K 10 February 2016 Opr: Fakhрутdinov A.N.; Solv: DMSO-d6;

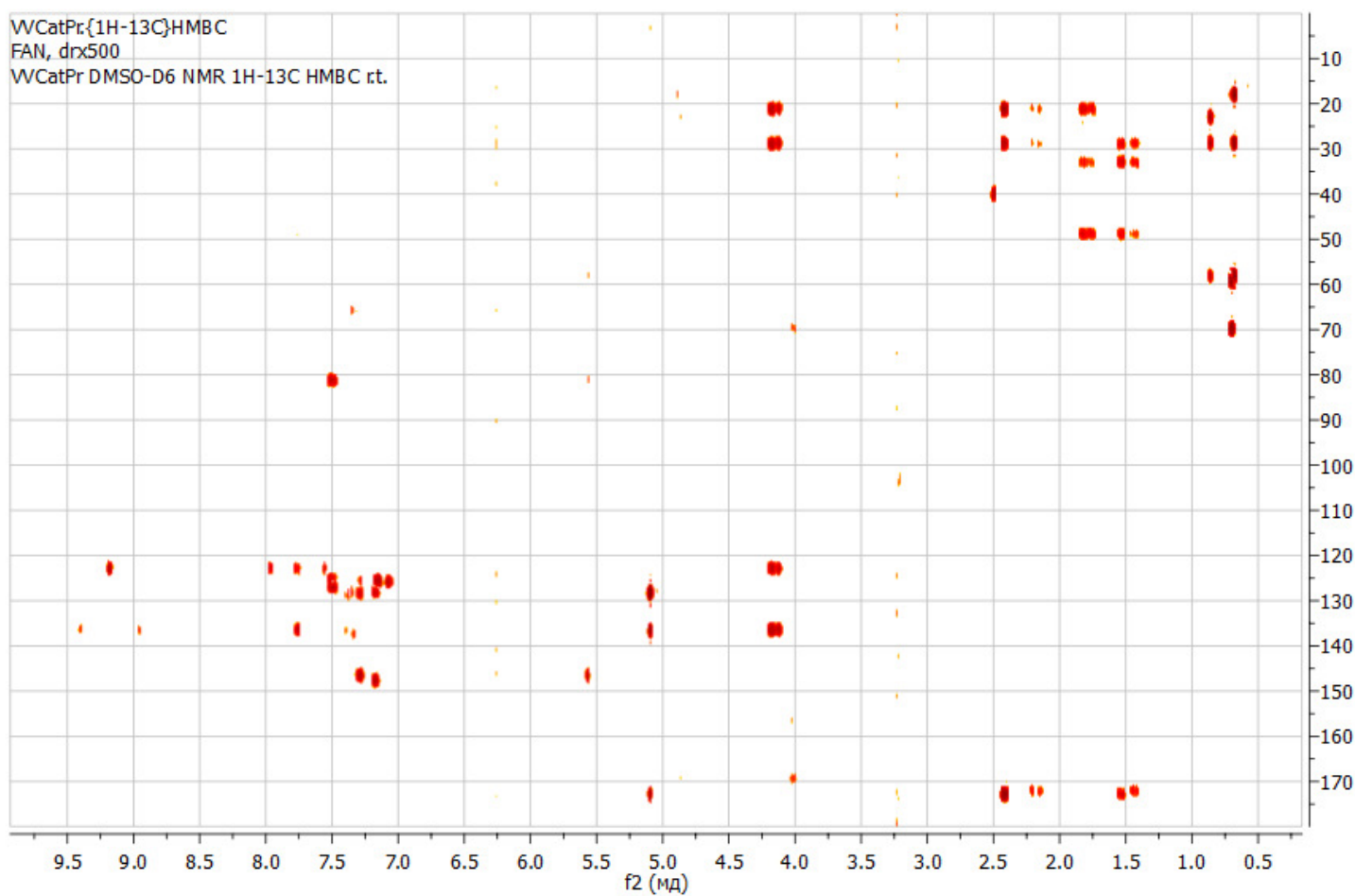
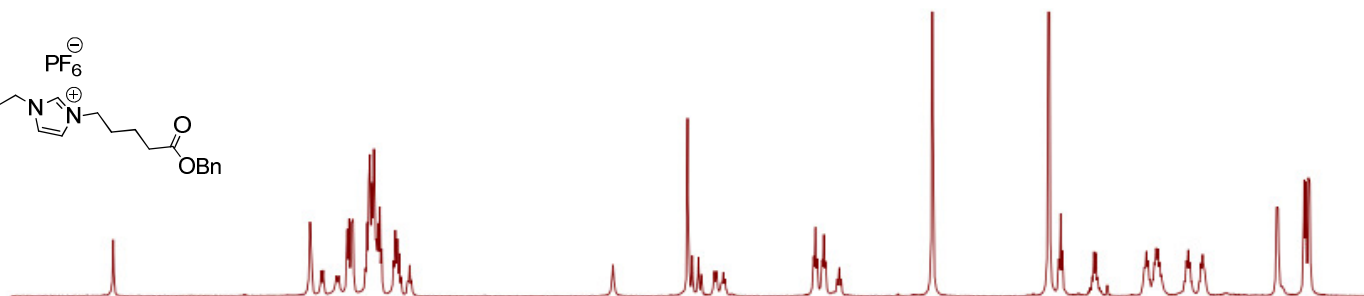
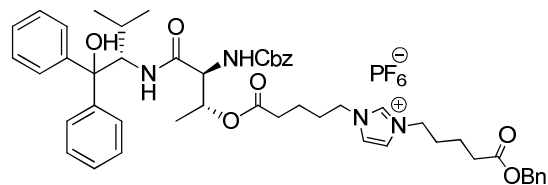
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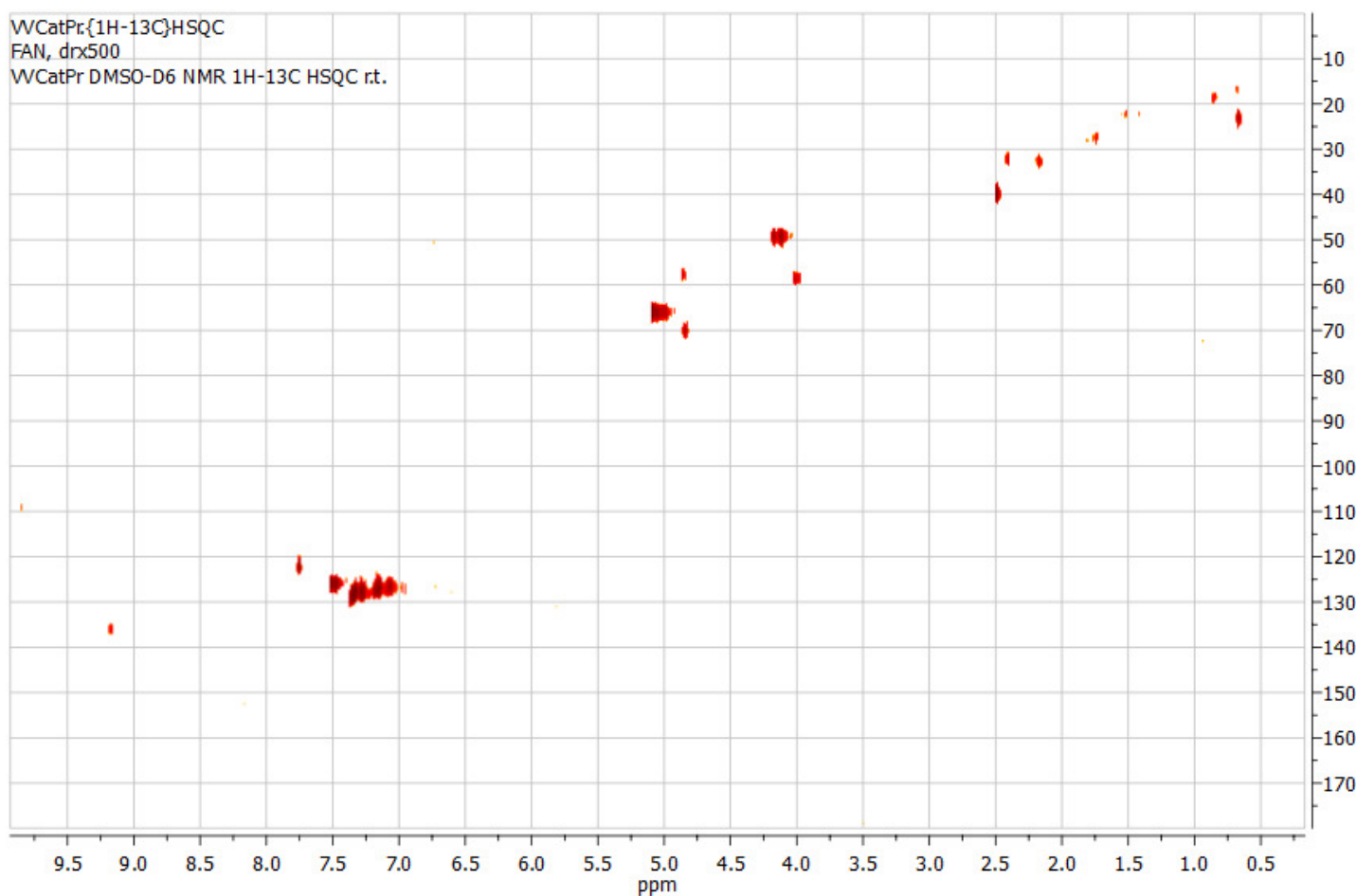
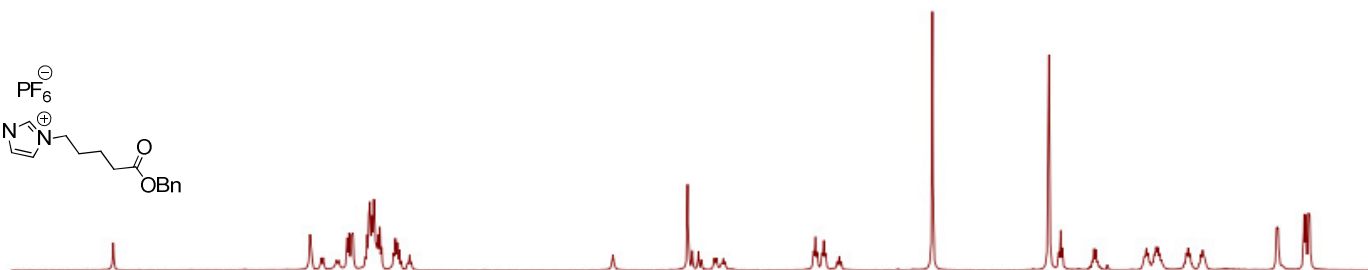
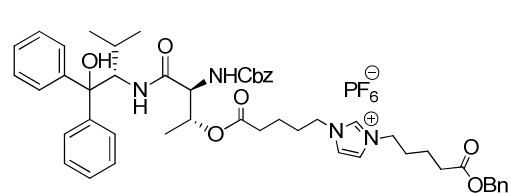
VVCatPr DMSO-D6 NMR 13C r.t.



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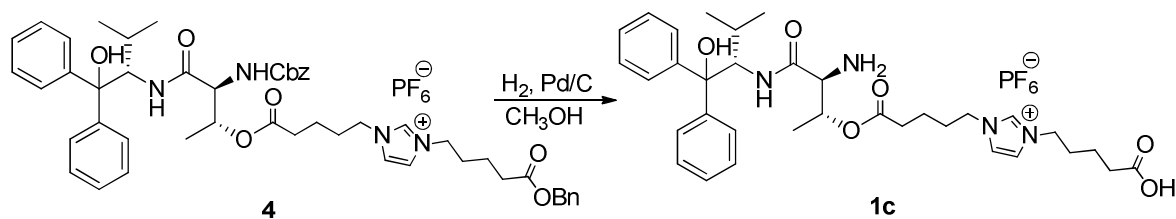




ppm

Synthesis and characterization of 1c

1-(5-(((2R,3S)-3-amino-4-(((S)-1-hydroxy-3-methyl-1,1-diphenylbutan-2-yl)oxy)-4-oxobutan-2-yl)oxy)-5-oxopentyl)-3-(4-carboxybutyl)-1H-imidazol-3-ium hexafluorophosphate



The 5% Pd/C (50 mg) was added to a solution of **4** (120 mg, 0.12 mmol) in freshly distilled methanol (3 mL) and the reaction mixture was vigorously stirred under H₂ atmosphere (~ 1 bar) for 5 h at ambient temperature. The reaction mixture was filtered and evaporated under reduced pressure (20 Torr). The residue was dried in vacuo (2 Torr) at 40 °C for 1 h to afford 89 mg (96%) of **1c**.

Light yellow powder, m.p. = 89-91 °C,

¹H NMR (600 MHz, DMSO-*d*₆): 0.58 (d, *J* = 3.2 Hz, 3H, CH₃); 0.68-0.73 (m, 3H, CH₃); 0.80-0.90 (m, 3H, CH₃); 1.40-1.53 (m, 4H, 2xCH₂); 1.62-1.74 (m, 1H, CH *i*-Pr); 1.71-1.87 (m, 4H, 2xCH₂); 2.18 (t, *J* = 7.1 Hz, 2H, CH₂); 2.28 (t, *J* = 7.2 Hz, 2H, CH₂); 3.64-3.72 (m, 1H, CH₃CHOH); 3.98 (t, *J* = 7.4 Hz, 1H, CH(NH)CONH); 4.13-4.24 (m, 4H, 2xCH₂); 4.50-4.62 (m, 1H, CH(*i*-Pr)NH); 4.87 (d, *J* = 9.5 Hz, 1H, OH); 5.67 (s, 1H, OH); 7.06-7.23 (m, 4H, CH); 7.29 (t, *J* = 7.7 Hz, 2H, CH); 7.42 (d, *J* = 10.1 Hz, 1H, NH); 7.49 (t, *J* = 6.7 Hz, 4H, CH); 7.81 (d, *J* = 11.7 Hz, 2H, NCHCHN); 7.94 (d, *J* = 8.4 Hz, 1H, NH); 9.24 (s, 1H, NCHN); 12.08 (s, 1H, COOH);

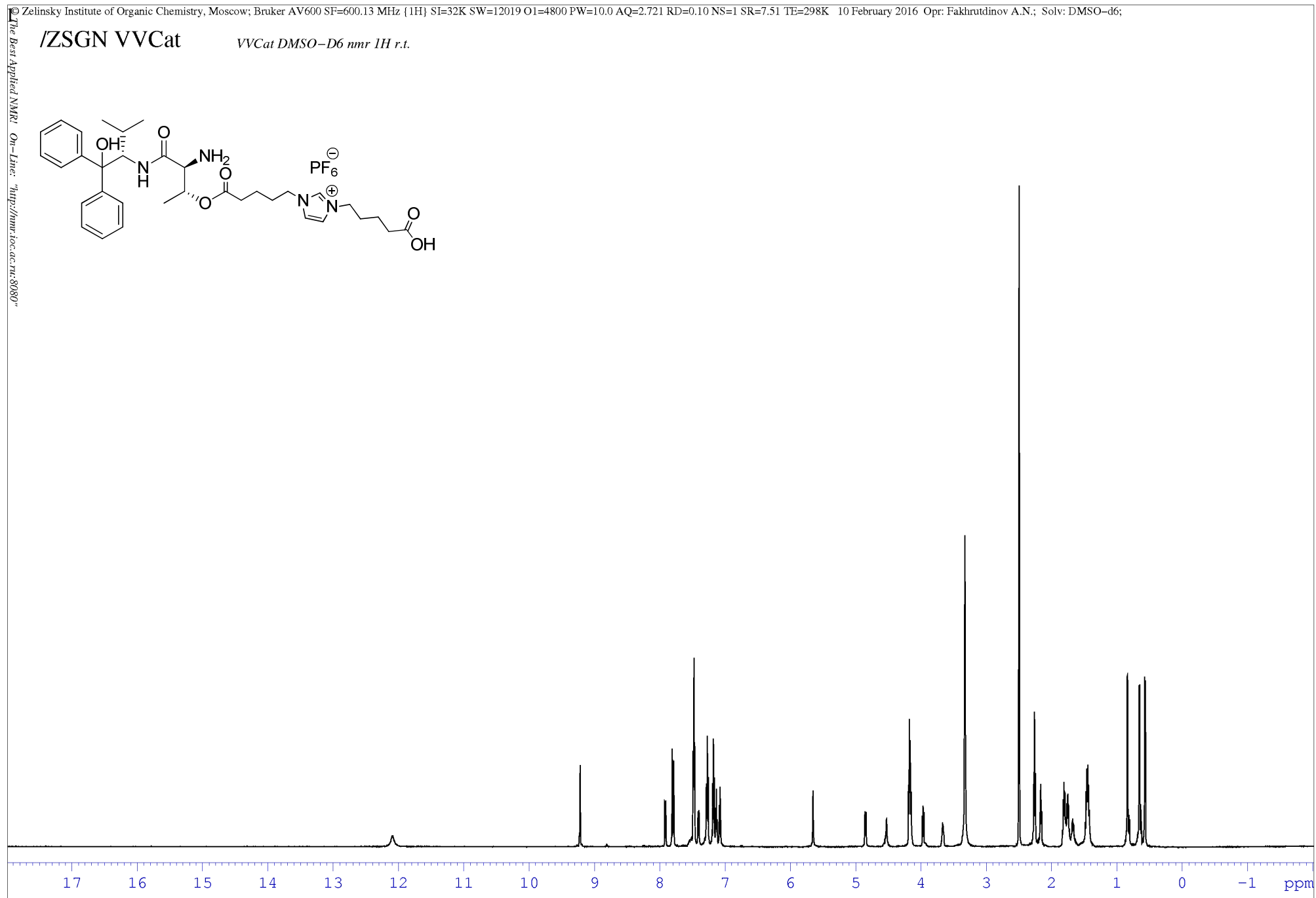
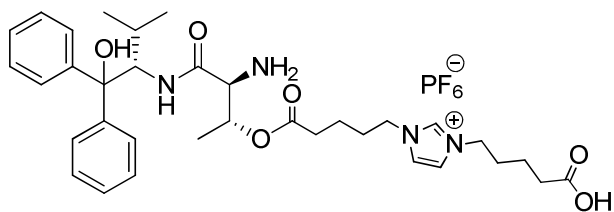
¹³C NMR (δ): 18.2, 19.9, 21.5, 22.2, 23.3, 28.9, 29.1, 29.3, 29.6, 33.3, 34.6, 49.0, 49.1, 57.9, 59.4, 66.1, 81.3, 122.9, 125.6, 126.0, 126.6, 128.1, 128.5, 136.4, 146.7, 170.8, 172.1, 174.5.

HRMS (ESI): *m/z* calcd. for C₃₄H₄₇N₄O₆⁺: 607.3490, found: 607.3493

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/ZSGN VVCat

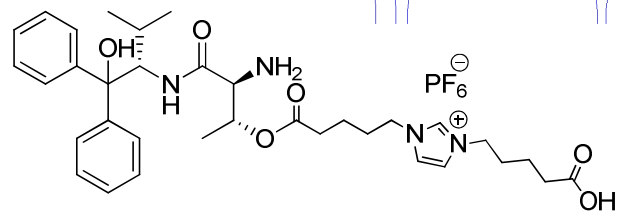
VVCat DMSO-D6 nmr 1H r.t.



© Melinsky Institute of Organic Chemistry, Moscow; Bruker DRX500 SF=125.76 MHz (13C) SI=64K SW=30300 O1=13205 PW=12.0 AQ=1.079 RD=0.60 NS=3142 SR=62.39 TE=298K 10 February 2016 Opr: Fakhrutdinov A.N.; Solv: DMSO-d6;

/ZSGN VVCat

VVCat DMSO-D6 NMR 13C r.t.

174.051
171.659
170.277147.315
146.250

135.934

127.979

127.634

126.074

125.477

125.169

122.459

80.782

65.646

58.907

57.406

48.555

48.506

40.093

40.002

39.925

39.836

39.759

39.669

39.593

39.502

39.335

39.168

39.001

34.062

32.777

28.791

28.558

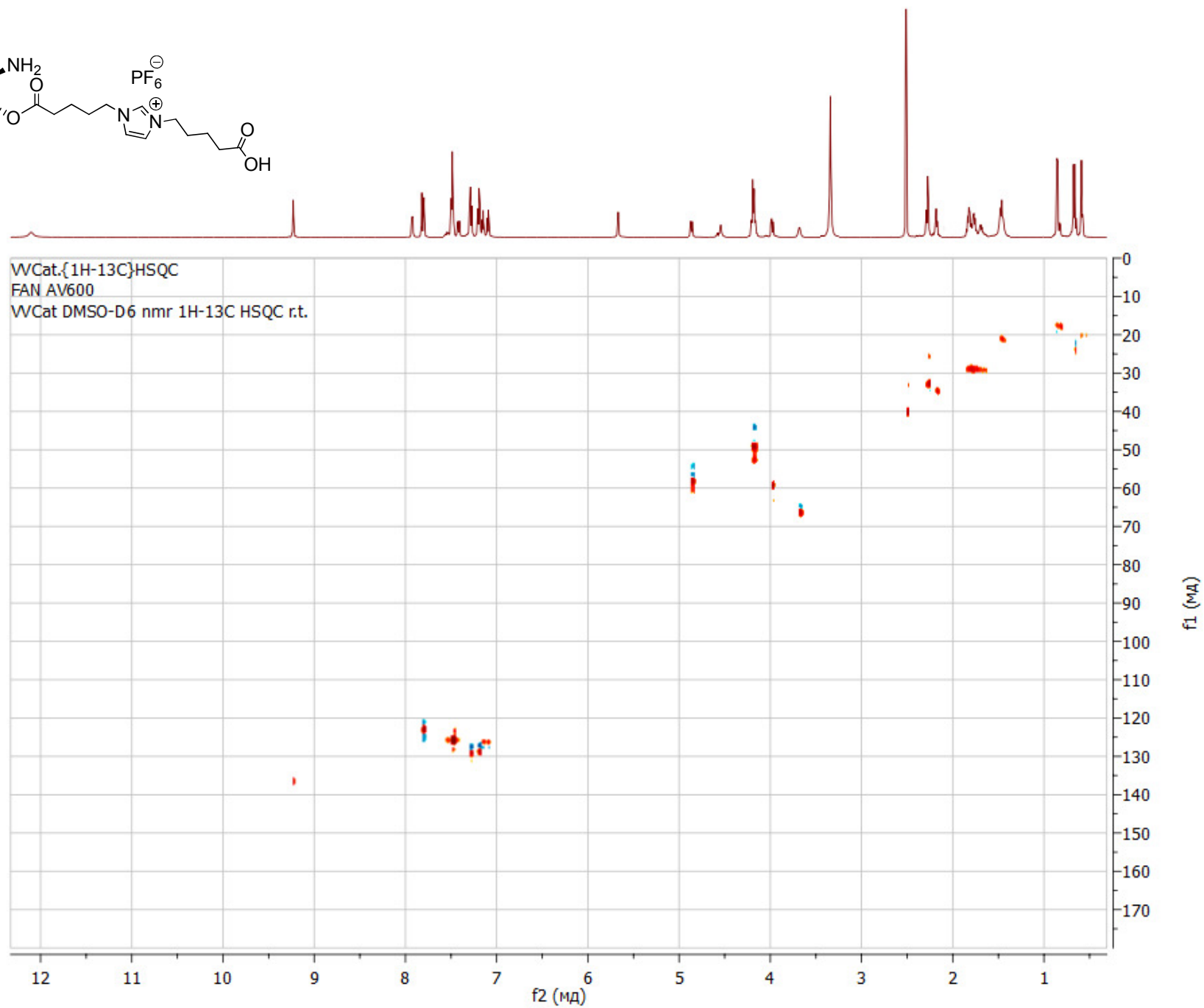
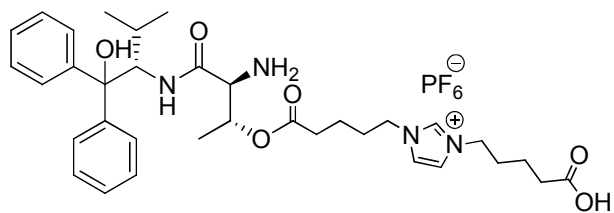
22.784

21.748

20.993

19.399

17.686



Display Report

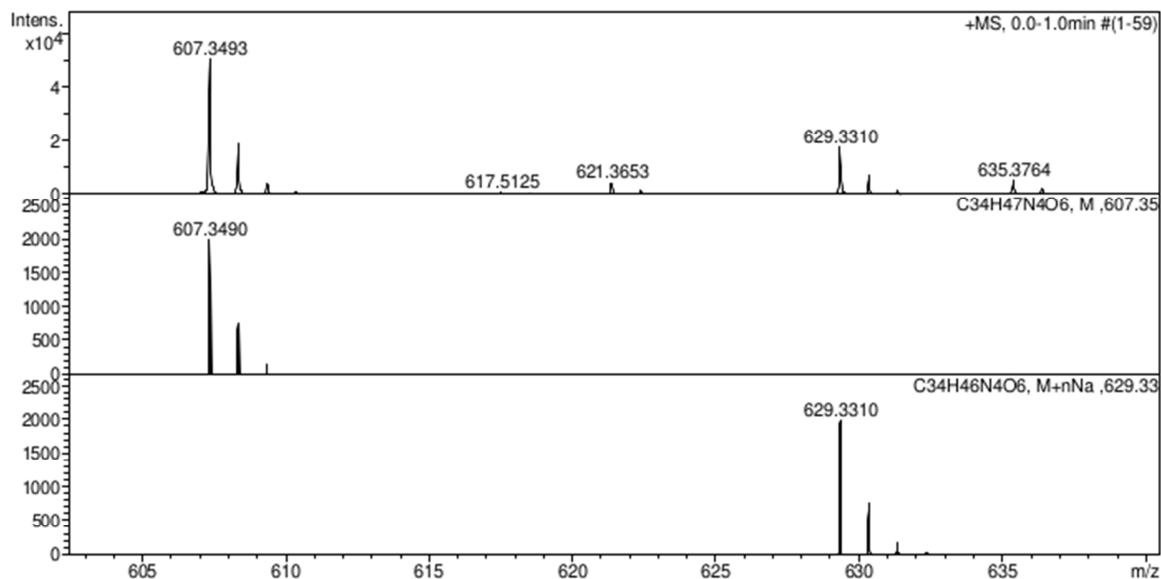
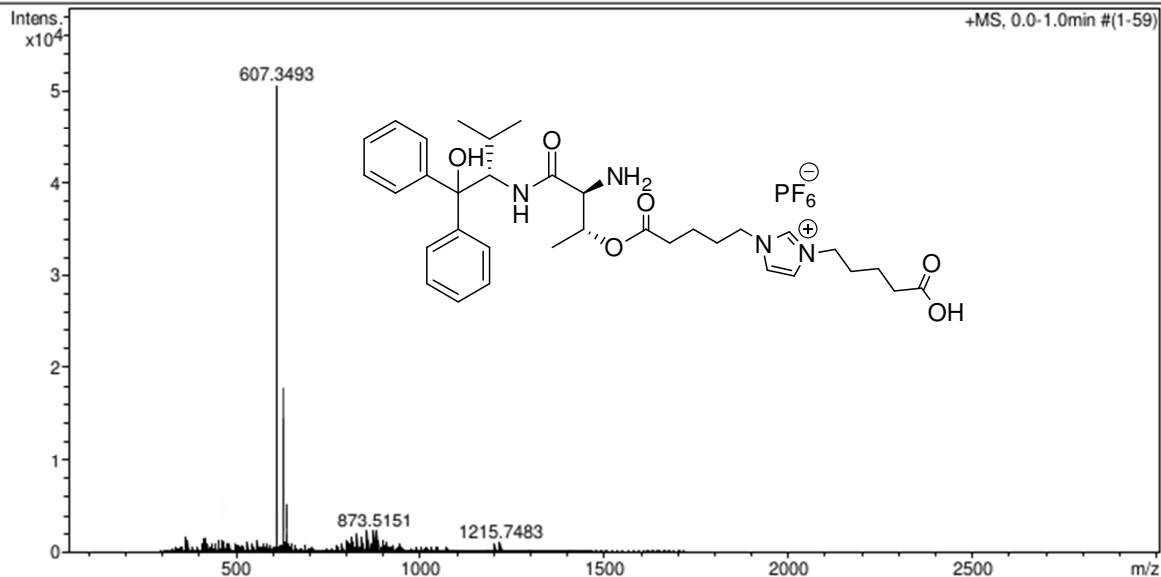
Analysis Info

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 Comment CH3OH 100 %, dil. 200, no calibrant added

Acquisition Date 21.12.2015 18:16:25
 Operator BDAL@DE
 Instrument / Ser# micrOTOF 10248

Acquisition Parameter

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Scan End	3000 m/z	Set End Plate Offset	-500 V	Set Divert Valve	Waste

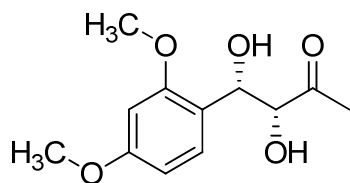


General procedure for *syn*-aldol reaction

Aldehyde **6a-i** (0.066 mmol) and catalyst **1c** (7.5 mg, 0.01 mmol) were dissolved in dry toluene (90 μ L). Then, ketone **5a-c** (0.2 mmol) was added to the resulting solution. The reaction mixture was stirred at ambient temperature for 24-48 h (TLC-monitoring), filtered through a silica gel pad and evaporated (40 $^{\circ}$ C, 8 mbar). Conversions and *dr* values of aldol products **7a-l** were measured by ^1H NMR spectroscopy. The *ee* values of aldol products **7** were determined by chiral HPLC column (Daicel Chiralpak AD-H).

General procedure for recycling experiment

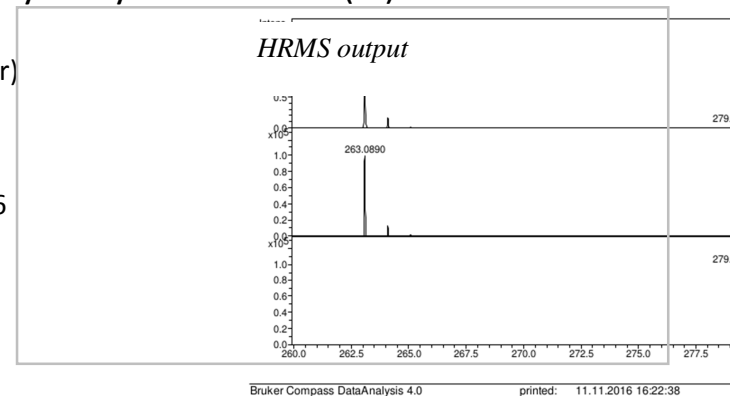
After 24 h, the mixture of hydroxyacetone (**5a**) (74 mg, 70 μ L, 1 mmol), 2-chlorobenzaldehyde (**6d**) (46.8 mg, 0.33 mmol), catalyst **1c** (37.5 mg, 0.05 mmol) and toluene (0.45 mL) was gently evaporated (40 $^{\circ}$ C, 8 mbar). Product **7d** and unchanged starting compounds were carefully extracted from the residue by Et_2O (3 x 0.7 mL). Fresh portions of reagents and toluene were added to the remaining catalyst **1c** and catalytic procedure was re-performed as described above.

Characterization of (3*R*,4*S*)-4-(2,4-dimethoxyphenyl)-3,4-dihydroxybutan-2-one (**7i**)

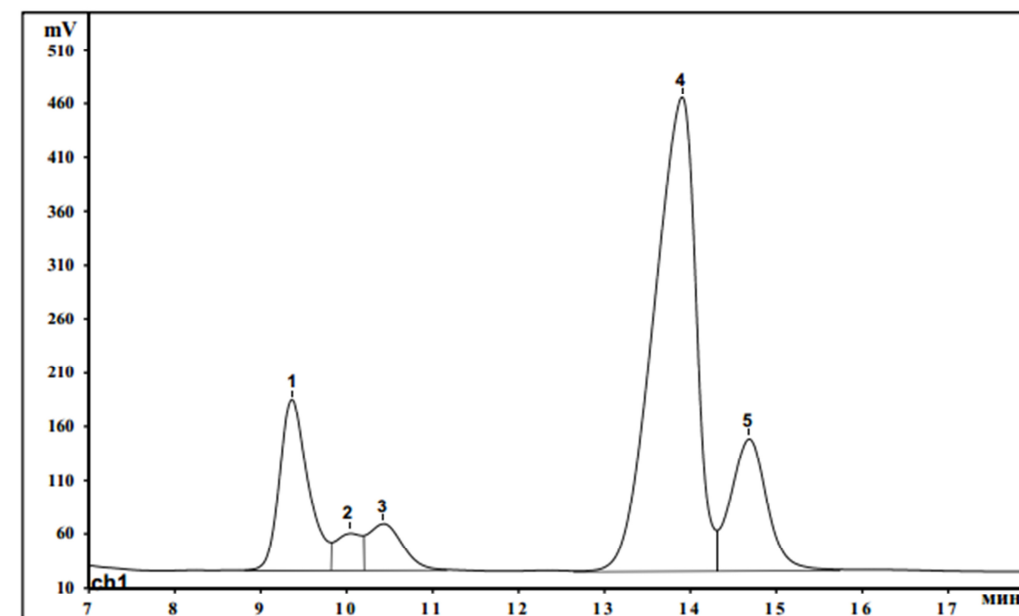
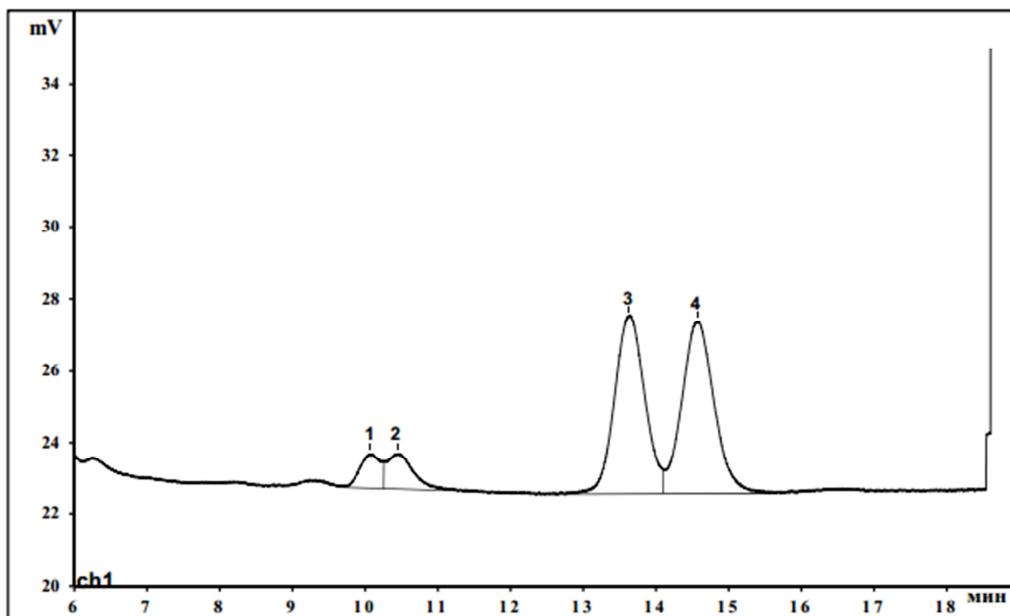
^1H NMR (500 MHz, CDCl_3): 2.27 (s, 3H, CH_3), 3.82 (d, 6H, (OCH_3)₂), 4.41 (s, 1H), 5.31 (s, 1H), 6.42-6.60 (m, 2H, Ar), 7.31 (m, 1H, Ar)

^{13}C NMR (125 MHz, CDCl_3): 26.4, 55.8, 56.0, 69.5, 71.5, 80.2, 80.4, 99.0, 104.8, 104.9, 121.5, 128.1, 129.0, 157.3, 161.13, 208.86

HRMS (ESI) m/z calcd. for $[\text{C}_{12}\text{H}_{16}\text{O}_5+\text{Na}]$: 263.0890; found: 263.0890



HPLC traces (Chiralpak AD-H, 1 ml/min, hexane:*i*-PrOH=80:20, $\lambda=254$ nm):



RESULTS

Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention мин	Area mV*сек	Area Name %
1	10.01	16.11	6.05
2	10.47	14.08	5.28
3	13.63	118.32	44.41
4	14.58	117.92	44.26

1 } *anti*
2 }
3 } *syn*
4 }

RESULTS

Quantitation method: Нормировка отклика
Standard component: Нет

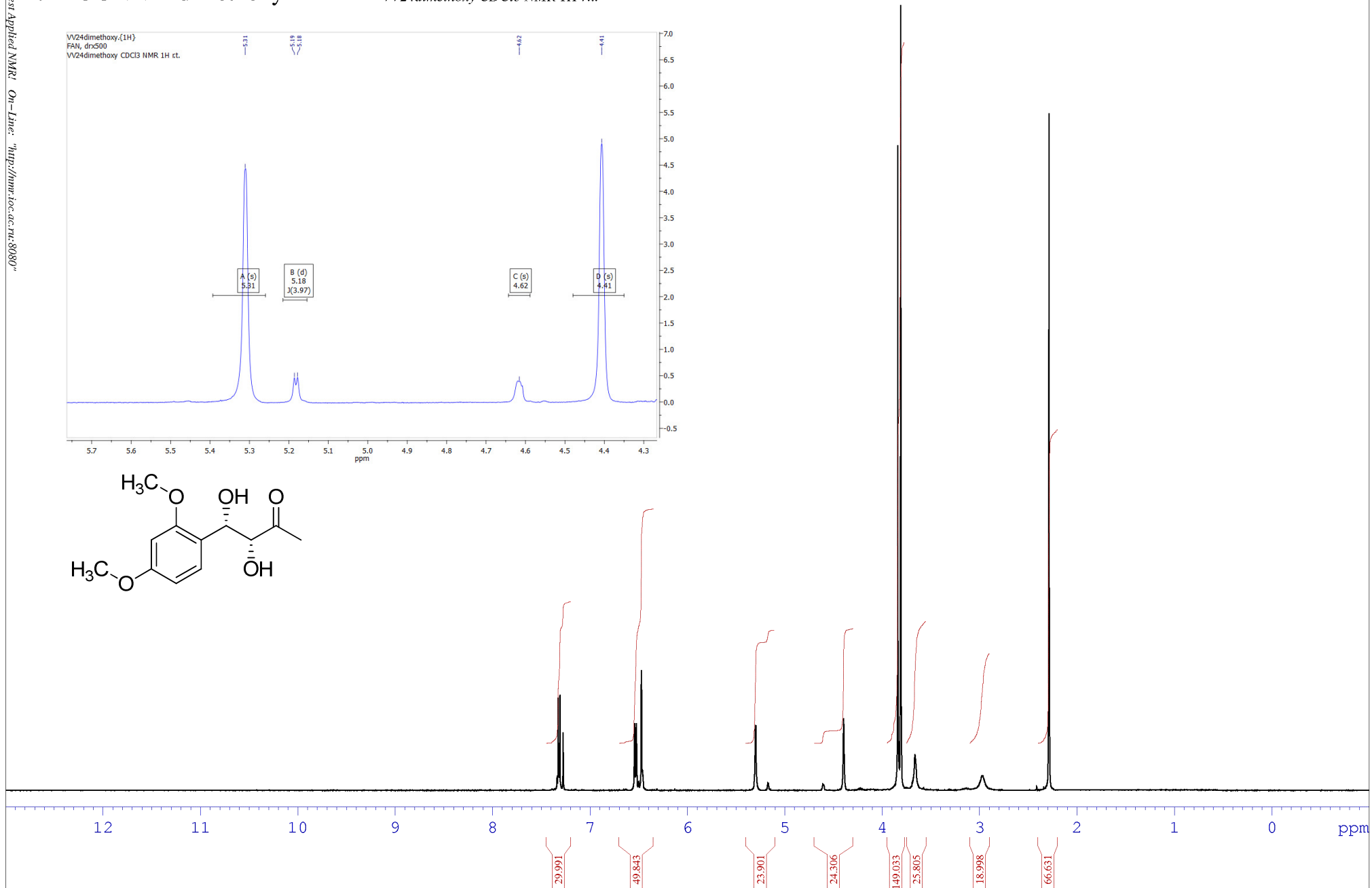
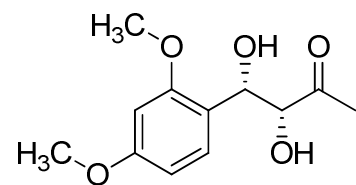
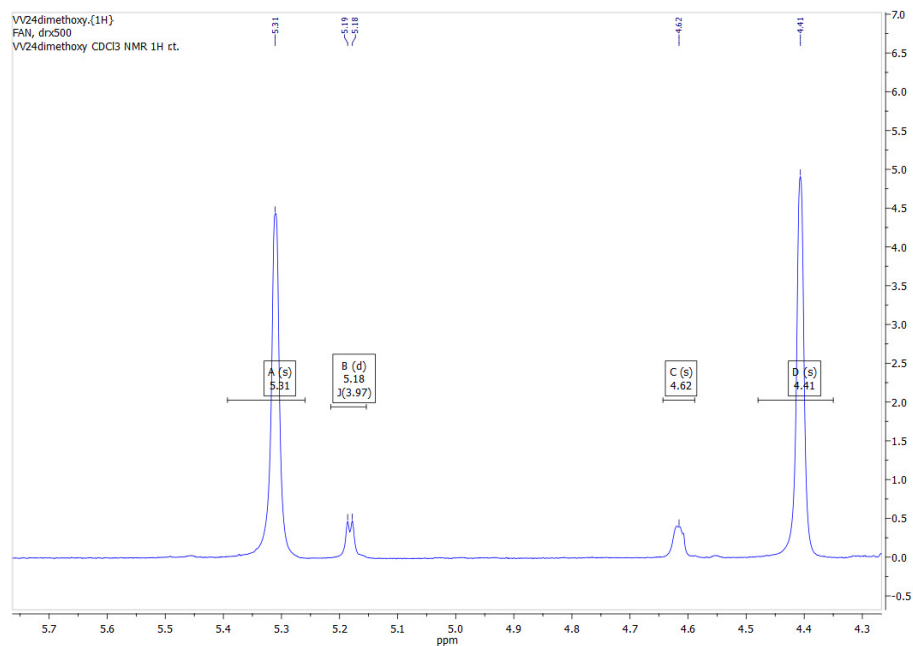
No	Retention мин	Area mV*сек	Area Name %
1	9.36	3719.67	15.40
2	10.05	686.93	2.84
3	10.43	1264.35	5.23
4	13.9	14917.03	61.75

2 } *anti*
3 }
4 } *syn*
5 }

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/FROG VV24dimethoxy

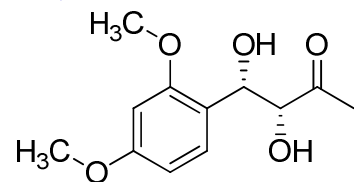
VV24dimethoxy CDCl3 NMR 1H r.t.



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/FROG VV24dimethoxy

VV24dimethoxy CDCl3 NMR 13C r.t.



208.860

161.134

157.338

128.982

128.146

121.486

104.941

104.848

99.041

80.374

80.161

77.919

77.664

77.410

71.494

69.476

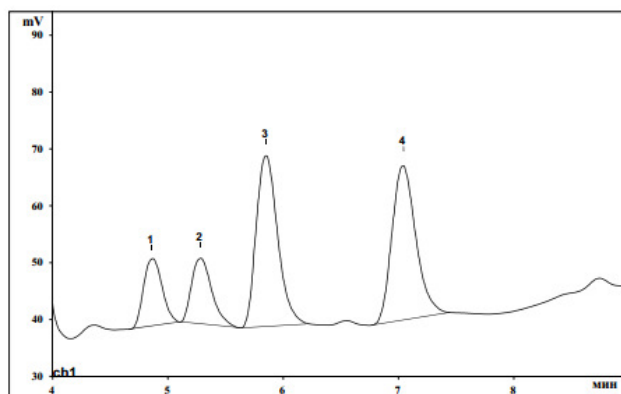
55.978

55.790

26.351

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

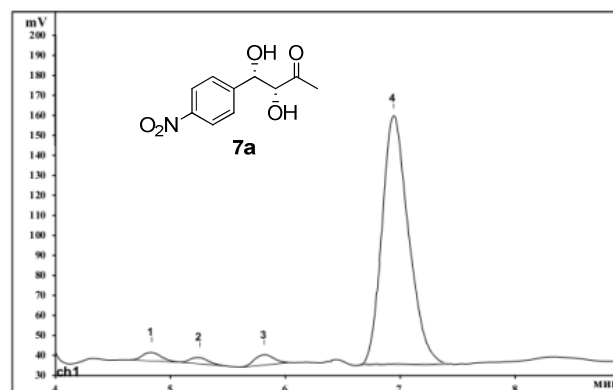
HPLC traces for 7

Chiralpak AD-H, 1.0 ml/min, Hexane:iPrOH = 70:30, λ =254 nm

RESULTS
Quantitation method: Нормировка отклика
Standart component: Нет

No	Retention min	Area mV*сек	Area Name %
1	4.89	98.07	10.11
2	5.30	99.14	10.22
3	5.87	381.66	39.36
4	7.05	390.83	40.30
4	8.95	969.70	100.00

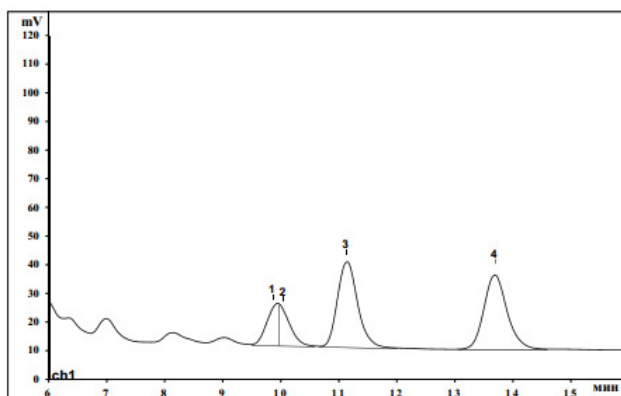
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RESULTS
Quantitation method: Нормировка отклика
Standart component: Нет

No	Retention min	Area mV*сек	Area Name %
1	4.90	44.21	2.52
2	5.31	31.90	1.82
3	5.84	49.13	2.80
4	7.03	1630.79	92.87
4	8.97	1756.03	100.00

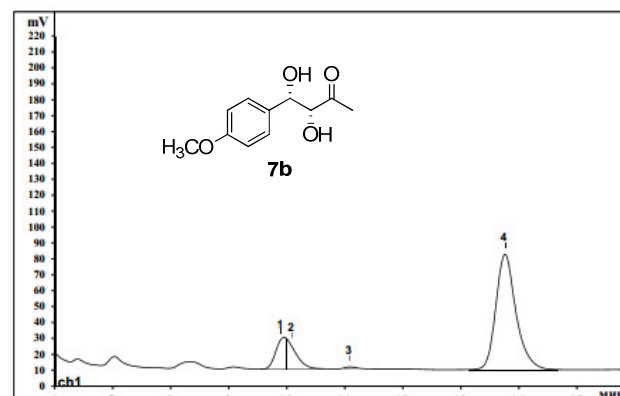
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Chiralpak AD-H, 1 ml/min, Hexane:iPrOH = 85:15, λ =280 nm

RESULTS
Quantitation method: Нормировка отклика
Standart component: Нет

No	Retention min	Area mV*сек	Area Name %
1	9.86	177.41	11.78
2	10.10	139.80	9.28
3	11.17	587.13	38.98
4	13.72	602.01	39.96
4	15.89	1506.35	100.00

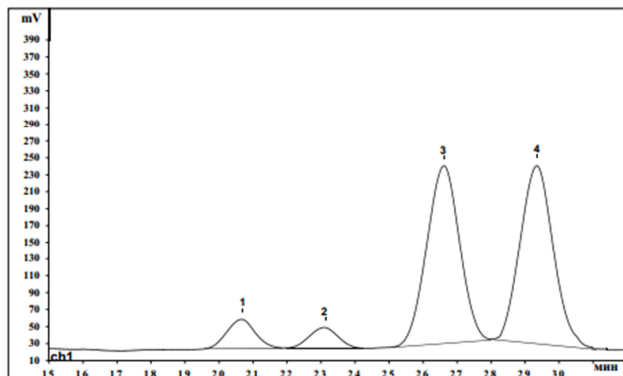
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RESULTS
Quantitation method: Нормировка отклика
Standart component: Нет

No	Retention min	Area mV*сек	Area Name %
1	9.88	149.14	5.54
2	10.12	101.31	3.77
3	11.11	17.27	0.64
4	13.79	2422.18	90.05
4	15.95	2689.90	100.00

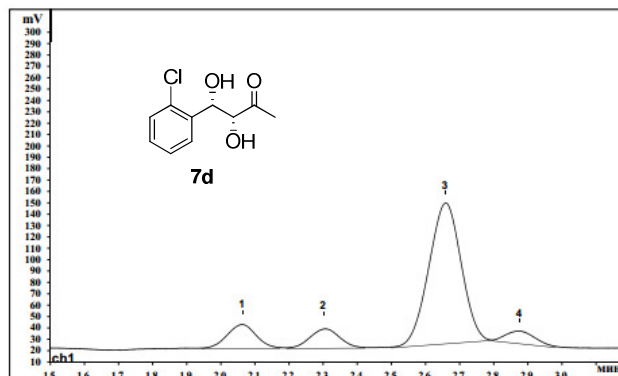
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Chiralpak AD-H, 1ml/min, Hexane:iPrOH = 95:5, λ = 220 nm

RESULTS
Quantitation method: Нормировка отклика
Standard component: Нев

No	Retention мин	Area mV*сек	Area Name %
1	20.61	1440.12	4.75
2	23.04	1322.72	4.37
3	26.60	13827.92	45.65
4	28.86	13698	45.22
4	31.90	30288.76	100.00

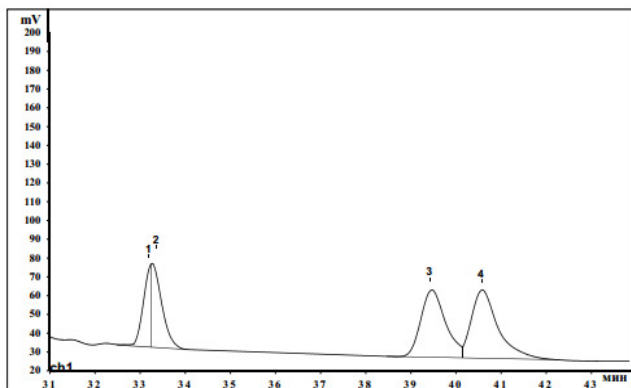
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RESULTS
Quantitation method: Нормировка отклика
Standard component: Нев

No	Retention мин	Area mV*сек	Area Name %
1	20.63	929.08	8.45
2	23.07	761.41	6.92
3	26.59	8896.38	80.87
4	28.84	413.94	3.76
4	31.89	11000.81	100.00

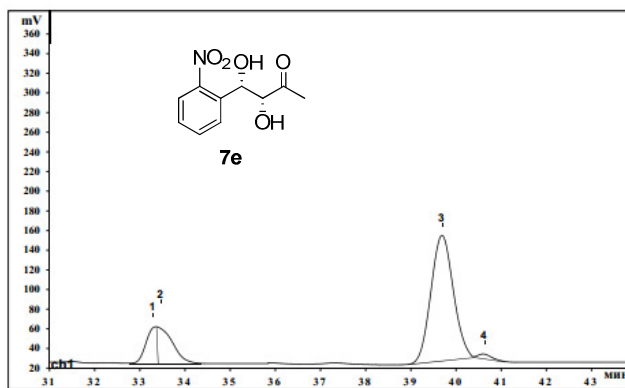
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Chiralpak AD-H, 0.6ml/min, Hexane:iPrOH = 90:10, λ = 254 nm

RESULTS
Quantitation method: Нормировка отклика
Standard component: Нев

No	Retention мин	Area mV*сек	Area Name %
1	33.17	419.90	10.46
2	33.38	584.13	14.55
3	39.46	1488.71	37.09
4	40.57	1521.39	37.90
4	43.87	4014.13	100.00

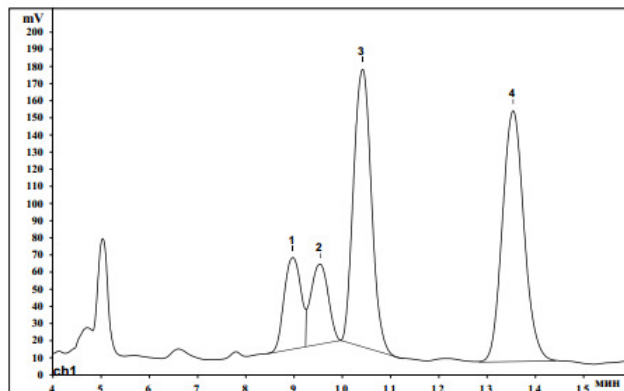
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RESULTS
Quantitation method: Нормировка отклика
Standard component: Нев

No	Retention мин	Area mV*сек	Area Name %
1	33.16	294.41	5.74
2	33.62	310.09	6.04
3	39.71	4439.07	86.48
4	40.59	89.73	1.75
4	43.99	5133.30	100.00

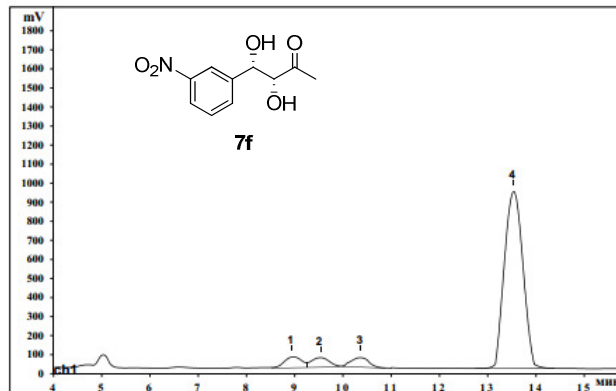
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Chiralpak AD-H, 1.0 ml/min, Hexane:iPrOH = 85:15, λ =254 nm

RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention min	Area mV*сек	Area Name %
1	9.00	1049.10	9.08
2	9.57	991.17	8.58
3	10.43	4733.27	40.96
4	13.55	4782.08	41.38
4	15.87	11555.62	100.00

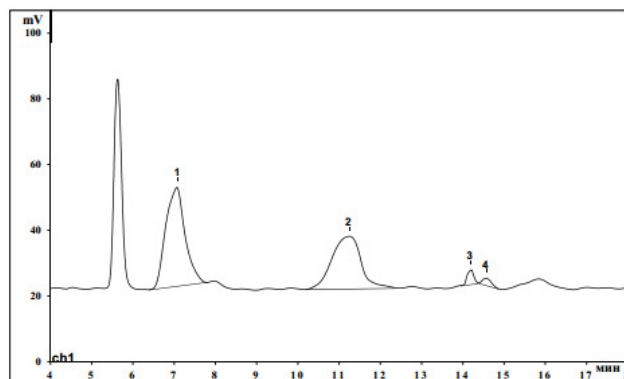
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RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention min	Area mV*сек	Area Name %
1	9.01	449.67	1.71
2	9.55	392.12	1.49
3	10.49	481.90	1.83
4	13.51	24993.08	94.97
4	15.89	26316.77	100.00

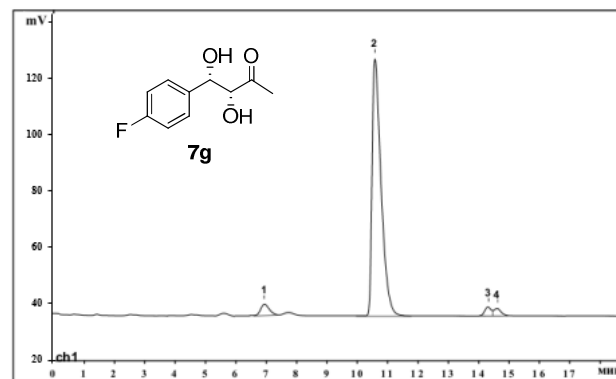
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Chiralpak AD-H, 1 ml/min, Hexane:iPrOH = 96:4, λ =254 nm, 24 °C

RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention min	Area mV*сек	Area Name %
1	7.09	1281.73	50.06
2	11.32	1233.90	48.19
3	14.14	21.58	0.84
4	14.59	23.11	0.90
4	17.92	2560.32	100.00

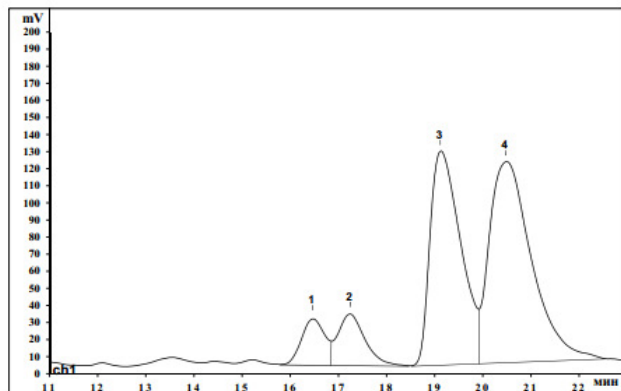
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RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention min	Area mV*сек	Area Name %
1	7.01	112.97	5.12
2	10.60	1939.02	87.86
3	14.12	86.71	3.93
4	14.71	68.14	3.09
4	18.93	2206.84	100.00

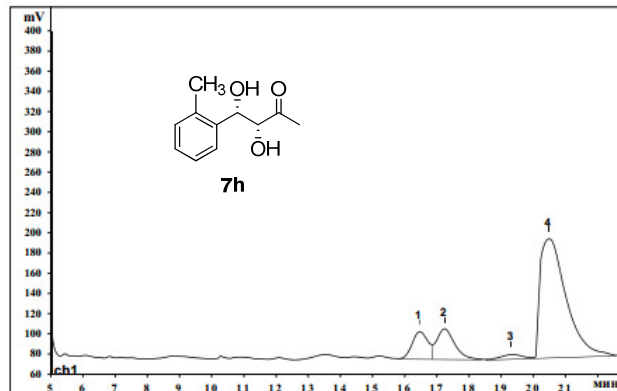
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Chiralpak AS-H, 1 ml/min, Hexane:iPrOH = 92:8, λ = 220 nm

RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention мин	Area mV*сек	Area Name %
1	16.54	739.12	8.65
2	17.27	810.44	9.48
3	19.18	3379.78	39.54
4	20.56	3618.04	42.33
4	22.92	8547.38	100.00

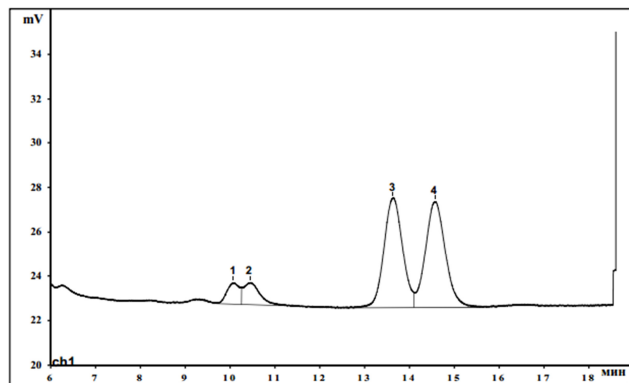
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RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention мин	Area mV*сек	Area Name %
1	16.55	664.21	11.19
2	17.23	751.89	12.66
3	19.16	204.20	3.44
4	20.51	4318.04	72.71
4	22.92	5938.34	100.00

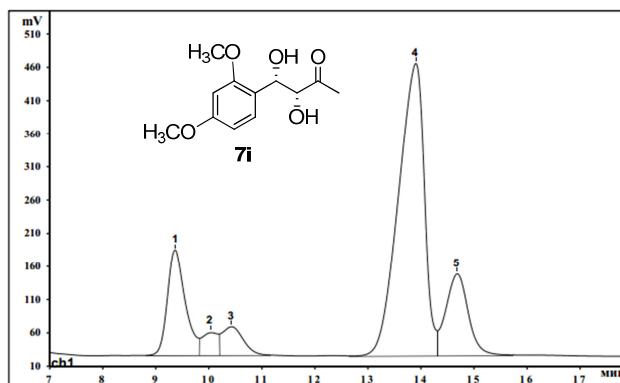
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Chiralpak AD-H, 1 ml/min, Hexane:iPrOH=80:20, λ =254 nm

RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention мин	Area mV*сек	Area Name %
1	10.01	16.11	6.05
2	10.47	14.08	5.28
3	13.63	118.32	44.41
4	14.58	117.92	44.26
4	18.97	266.43	100.00

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RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention мин	Area mV*сек	Area Name %
1	9.36	3719.67	15.40
2	10.05	686.93	2.84
3	10.43	1264.35	5.23
4	13.9	14917.03	61.75
5	14.68	3569.38	14.78
5	17.99	24157.36	100.00

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