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

Vasiliy V. Gerasimchuk,[a] Roman R. Romanov,[b] Woo Hoi Teng Gladys,[c] Igor A. Dmitriev,[a] Alexander S. Kucherenko,[a] and Sergei G. Zlotin*[a]

[a]Zelinsky Institute of Organic Chemistry, Leninsky prospect 47, 119991, Moscow, Russia
[b]Moscow Chemical Lyceum, Tamogeny proezd 4, 111033, Moscow, Russia
[c]National Junior College, 37 Hillcrest Road, 288913, Singapore
Email: zlotin@ioc.ac.ru

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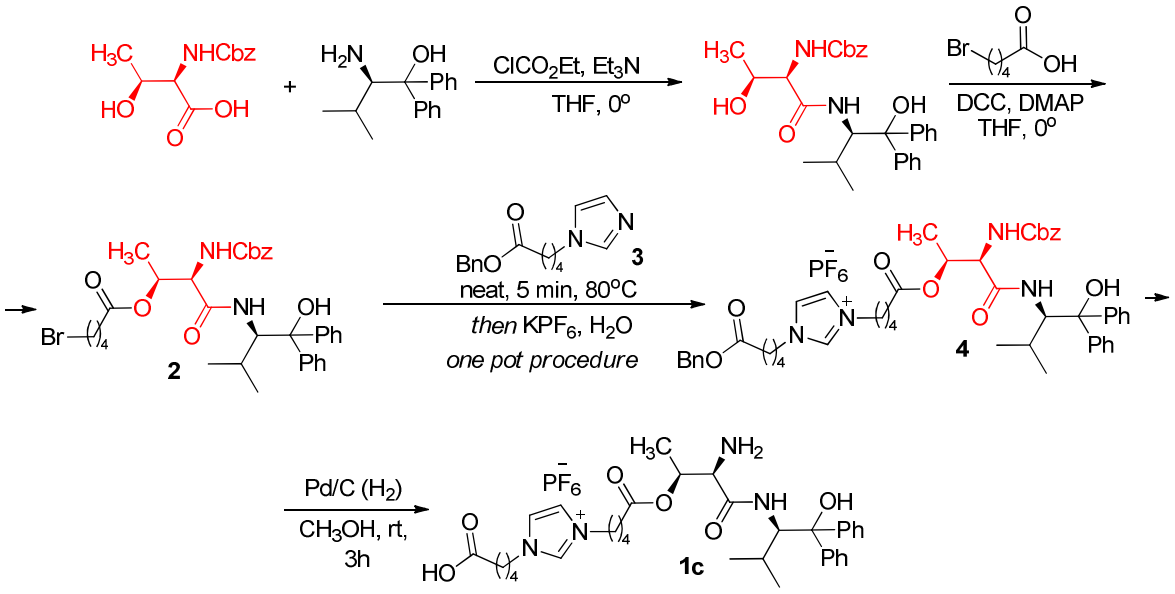
General information

^{1}H and ^{13}C NMR spectra were recorded with a Bruker AM 300 spectrometer in $CDCl_3$ and DMSO-d_6. The chemical shifts of ^{1}H 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 = 50–3000 Da; external or internal calibration was done with electrospray calibrant solution (Fluka). Syringe injection was used for solution in MeCN/H_2O (1:1, v/v) (flow rate 3 µL/min). Nitrogen was applied as a dry gas, and the interface temperature was set at 180 °C. Silica gel 0.060–0.200 µm (Acros) was used for column chromatography. Threonineamide **2** and benzyl 5-(1*H*imidazol-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.

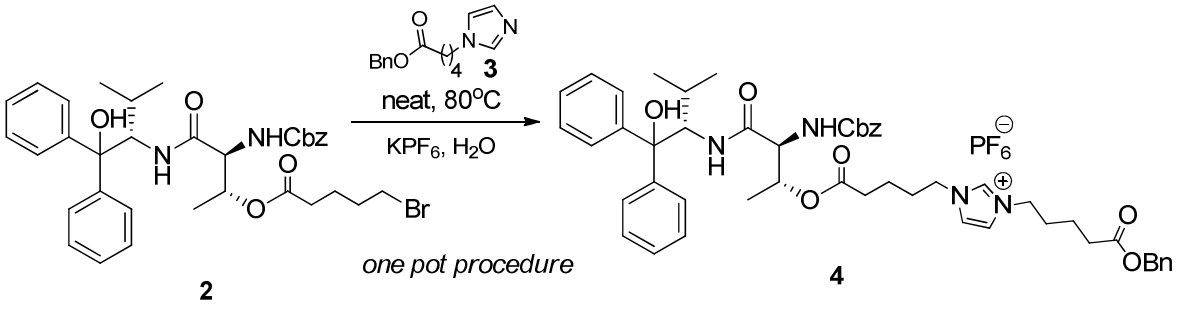
General scheme of catalyst 1c synthesis



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 CH_3OH (2 mL). The reaction mixture was kept at ambient temperature for 10 min and evaporated under reduced pressure (20 Torr) at 40 ^oC. The residue was heated at the same pressure (rotary evaporator, 80 ^oC) for 5 min, cooled to ambient temperature and diluted with distilled water (3.0 mL). A solution of KPF_6 (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 Et_2O, 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,

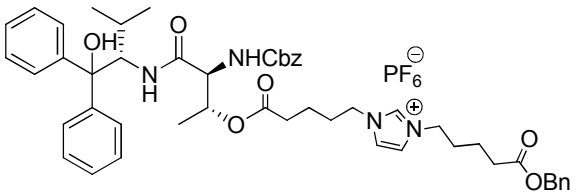
^{1}H NMR (600 MHz, DMSO-d_6): 0.65 (d, *J* = 6.5 Hz, 3H, CH_3); 0.70 (d, *J* = 6.5 Hz, 3H, CH_3); 0.87 (d, *J* = 6.5 Hz, 3H, CH_3); 1.38-1.45 (m, 2H, CH_2); 1.48-1.55 (m, 2H, CH_2); 1.69-1.78 (m, 3H, CH_2 + C*H*$(CH_3)_2$); 1.78-1.85 (m, 2H, CH_2); 2.13-2.24 (m, 2H, CH_2); 2.40 (t, *J* = 7.3 Hz, 2H, CH_2); 3.99 (t, *J* = 8.2 Hz, 1H, CH); 4.11 (t, *J* = 6.9 Hz, 2H, CH_2); 4.18 (t, *J* = 6.9 Hz, 2H, CH_2); 4.84 (m, 1H, CH); 4.89 (d, *J* = 9.5 Hz, 1H, CH); 5.04 (2H, CH_2 AB system, J_{HH}=12.66 Hz); 5.10 (s, 2H, CH_2); 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);

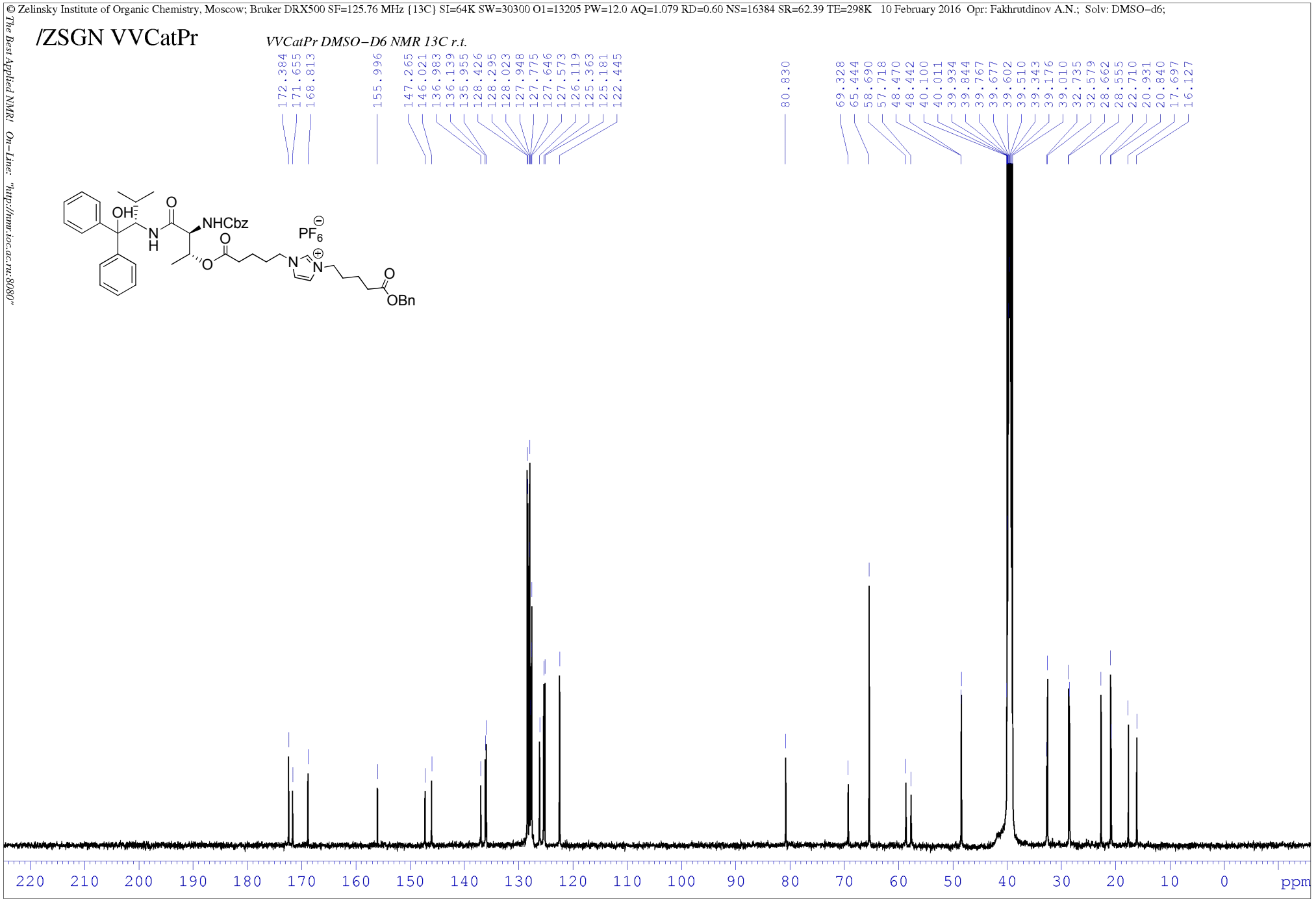
^{13}C NMR (125.76 MHz, DMSO-d6): 16.6, 18.2, 21.3, 21.4, 23.2, 29.05, 29.15, 33.1, 33.2, 48.9, 58.2, 59.2, 65.9, 69.8, 81.3, 122.9, 125.7, 125.8, 126.6, 128.0, 128.1, 128.3, 128.4, 128.5, 128.8, 128.9, 136.4, 136.6, 137.5, 146.5, 147.7, 156.5, 169.3, 172.1, 172.9;

Elemental analysis calcd for $C_{49}H_{59}F_6N_4O_8P$: C, 60.24; H, 6.09; N, 5.73; found: C, 60.06; H, 6.14, N, 5.79.

/ZSGN VVCatPr

VVCatPr DMSO–D6 nmr 1H r.t.




© 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: Fakhrutdinov A.N.; Solv: DMSO-d6;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
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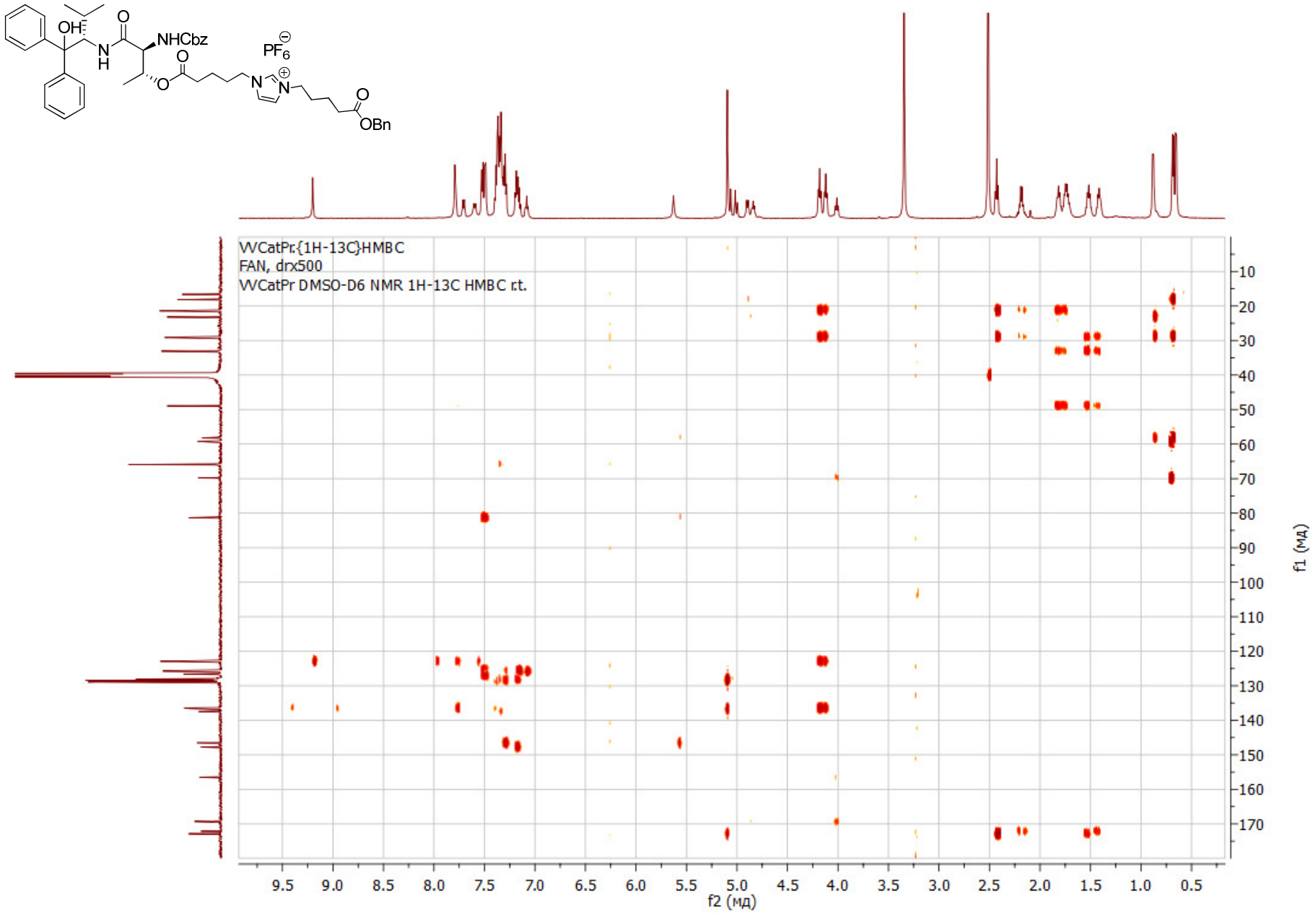
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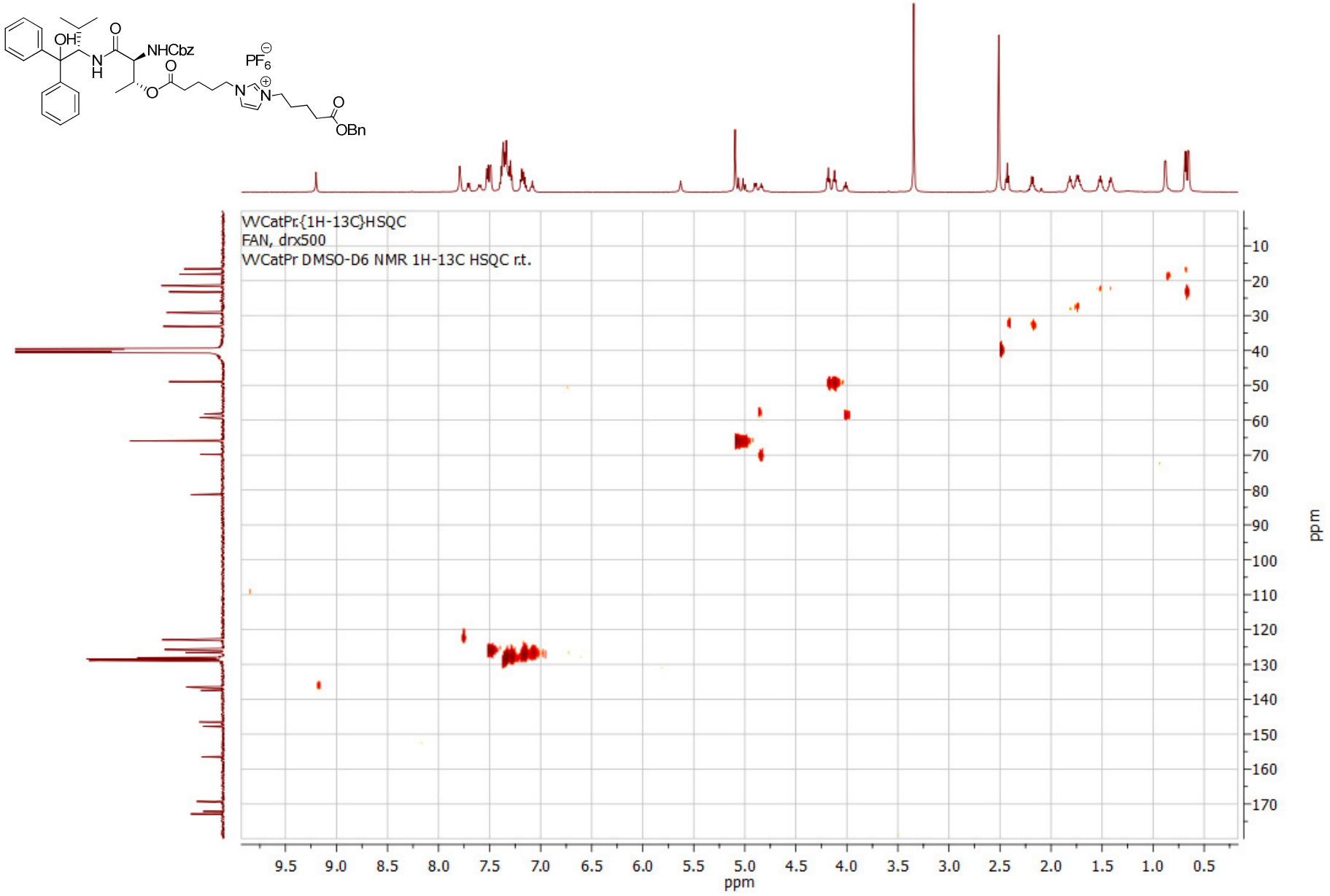
f2 (мд)
f1 (мд)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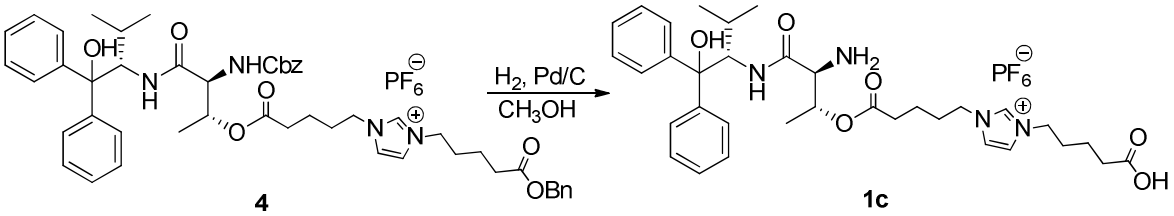
ppm
10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170
ppm

Synthesis and characterization of 1c

1-(5-(((2R,3S)-3-amino-4-(((S)-1-hydroxy-3-methyl-1,1-diphenylbutan-2-yl)amino)-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 H_2 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,

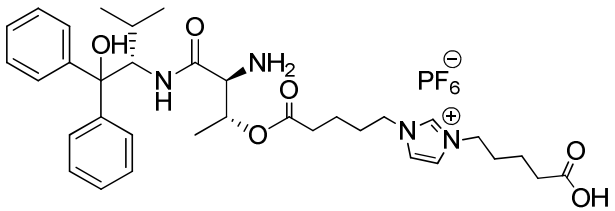
^{1}H NMR (600 MHz, DMSO-d_6): 0.58 (d, *J* = 3.2 Hz, 3H, CH_3); 0.68-0.73 (m, 3H, CH_3); 0.80-0.90 (m, 3H, CH_3); 1.40-1.53 (m, 4H, 2xCH_2); 1.62-1.74 (m, 1H, CH *i*-Pr); 1.71-1.87 (m, 4H, 2xCH_2); 2.18 (t, *J* = 7.1 Hz, 2H, CH_2); 2.28 (t, *J* = 7.2 Hz, 2H, CH_2); 3.64-3.72 (m, 1H, CH_3C*H*OH); 3.98 (t, *J* = 7.4 Hz, 1H, C*H*(NH)CONH); 4.13-4.24 (m, 4H, 2xCH_2); 4.50-4.62 (m, 1H, C*H*(*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);

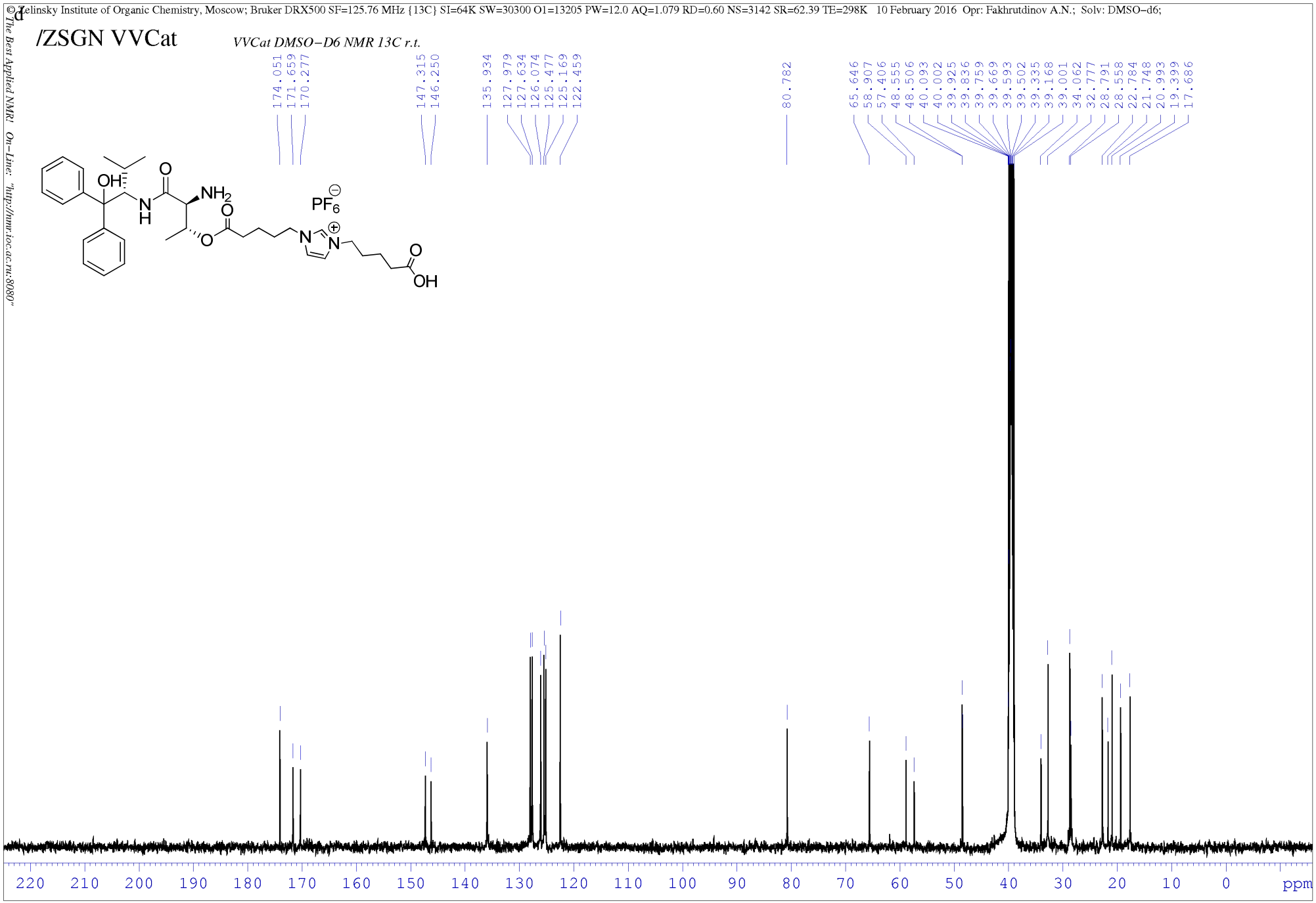
^{13}C NMR (): 18.2, 19.9, 21.5, 22.2, 23.3, 28.9, 29.1, 29.3, 29.6, 33.3, 34.6, 49.0, 49.1, 57.9, 59.4, 66.1, 81.3, 122.9, 125.6, 126.0, 126.6, 128.1, 128.5, 136.4, 146.7, 170.8, 172.1, 174.5.

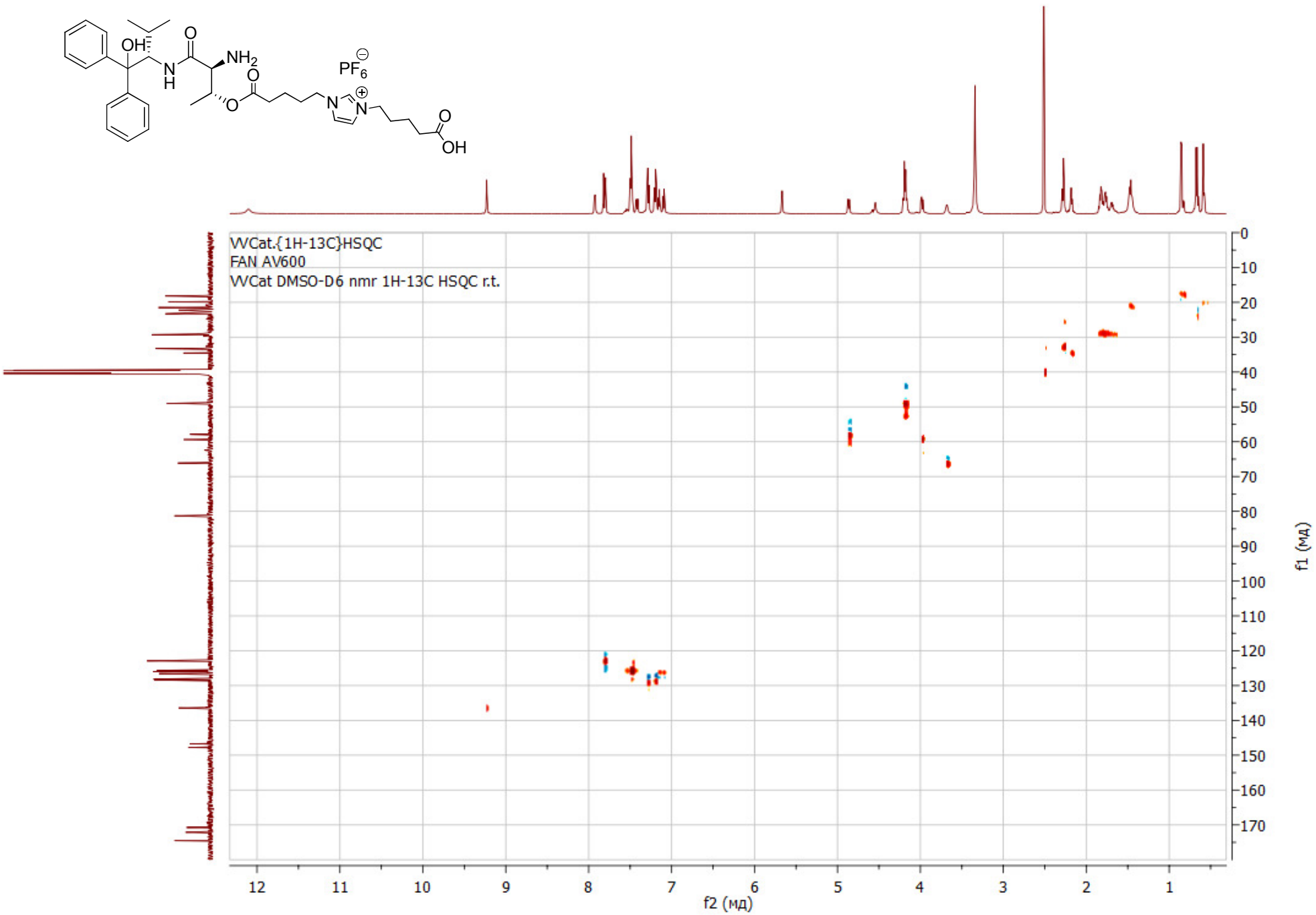
HRMS (ESI): *m/z* calcd. for $C_{34}H_{47}N_4O_6^+$: 607.3490, found: 607.3493

/ZSGN VVCat

VVCat DMSO–D6 nmr 1H r.t.



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FAN AV600
VVCat DMSO-D6 nmr 1H-13C HSQC r.t.
f2 (мд)
f1 (мд)

Display Report

Analysis Info

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Sample Name	/ZSGN VVCat-2	Instrument / Ser#	micrOTOF 10248
Comment	CH3OH 100 %, dil. 200, no calibrant added		

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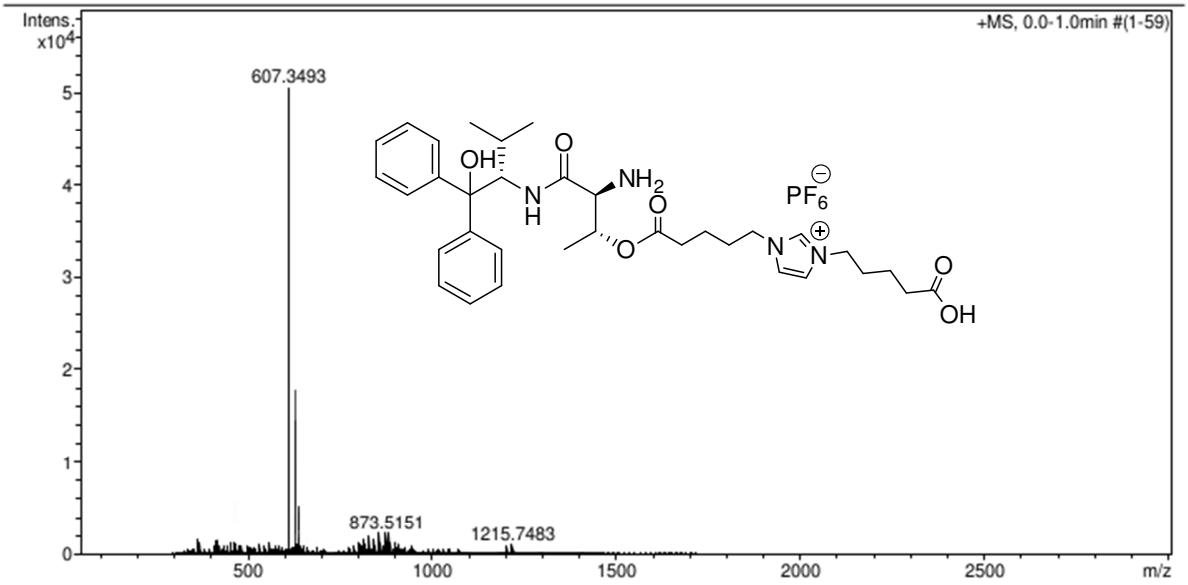
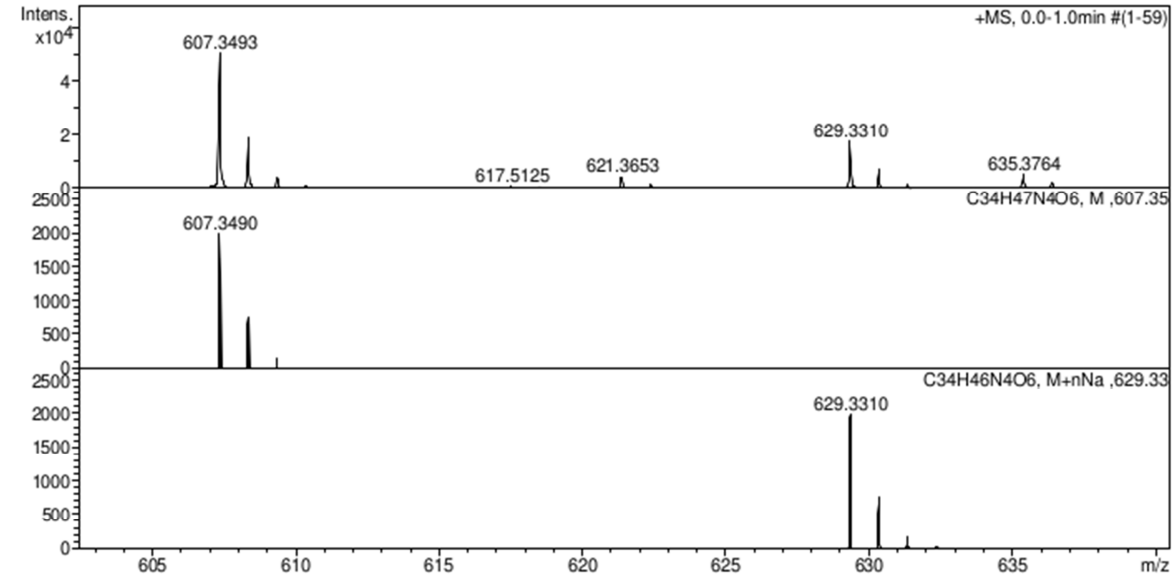




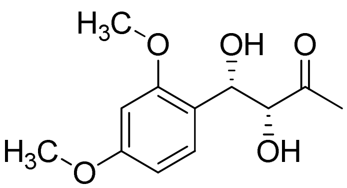
General procedure for *syn*-aldol reaction

Aldehyde **6a-i** (0.066 mmol) and catalyst **1c** (7.5 mg, 0.01 mmol) were dissolved in dry toluene (90 µL). 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 °C, 8 mbar). Conversions and *dr* values of aldol products **7a-l** were measured by ^{1}H 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 µl, 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 °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.

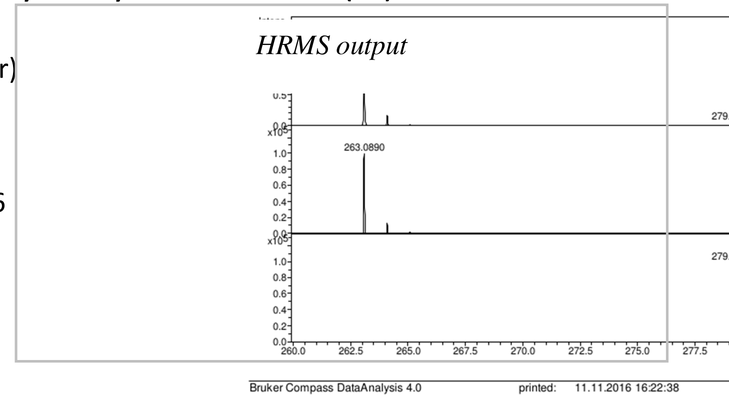
Characterization of (3*R*,4*S*)-4-(2,4-dimethoxyphenyl)-3,4-dihydroxybutan-2-one (**7i**)



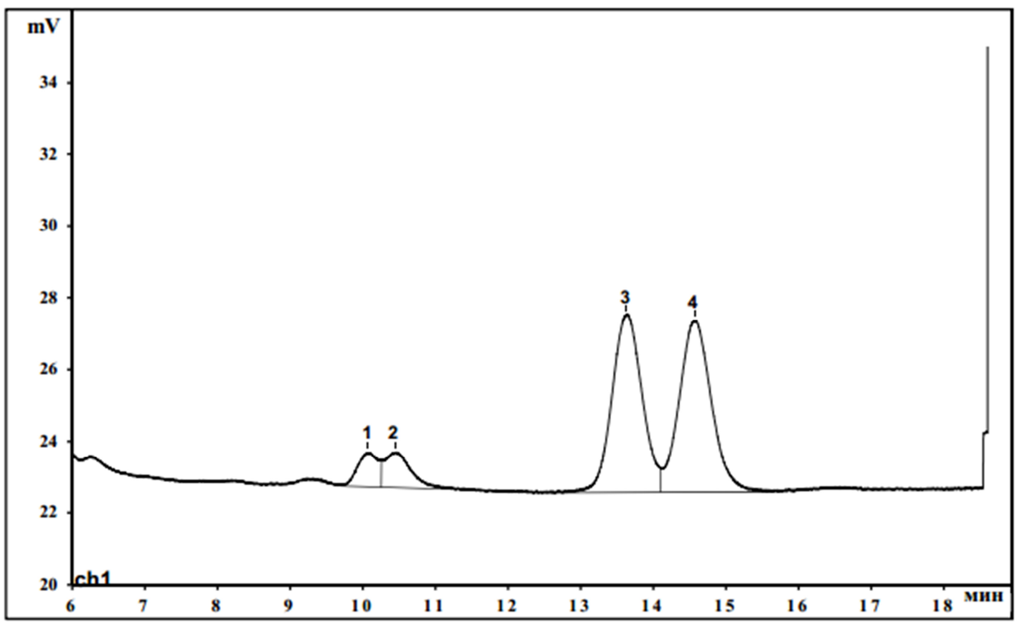
^{1}H NMR (500 MHz, $CDCl_3$): 2.27 (s, 3H, CH_3), 3.82 (d, 6H, $(OCH_3)_2$), 4.41 (s, 1H), 5.31 (s, 1H), 6.42-6.60 (m, 2H, Ar), 7.31 (m, 1H, Ar)

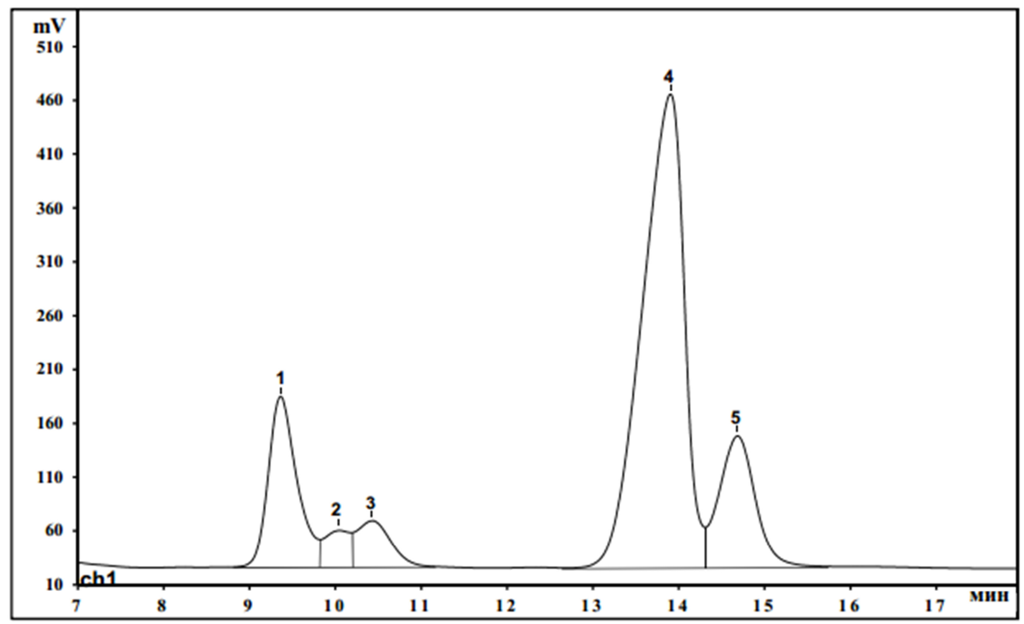
^{13}C NMR (125 MHz, $CDCl_3$): 26.4, 55.8, 56.0, 69.5, 71.5, 80.2, 80.4, 99.0, 104.8, 104.9, 121.5, 128.1, 129.0, 157.3, 161.13, 208.86

HRMS (ESI) m/z calcd. for $[C_{12}H_{16}O_5+Na]$: 263.0890; found: 263.0890



HPLC traces (Chiralpak AD-H, 1 ml/min, hexane:*i*-PrOH=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	

1, 2 – *anti*
3, 4 – *syn*

```
RESULTS
Quantitation method: Нормировка отклика
Standard component:  Нет
```

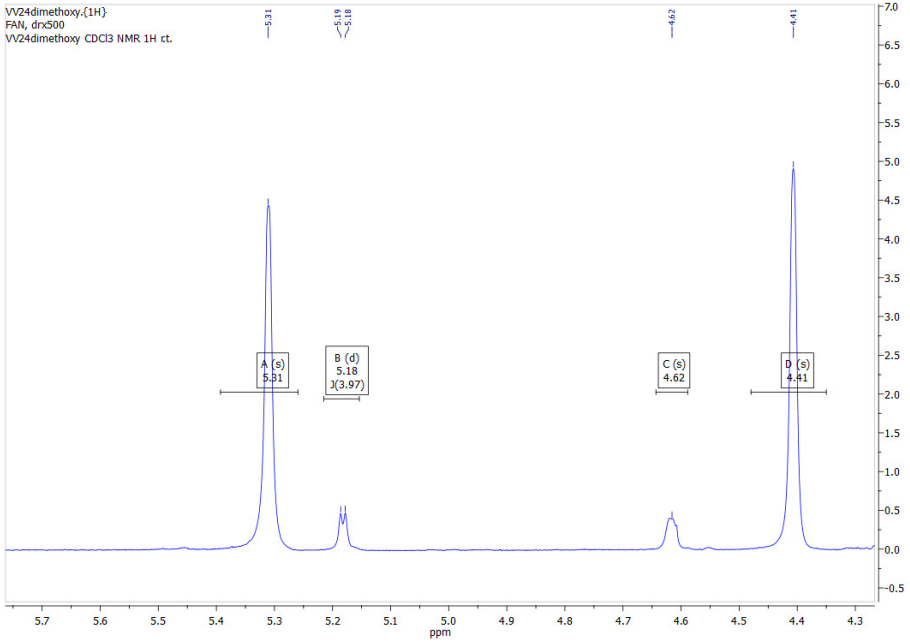
No	Retention мин	Area mV*сек	Area %	Name
1	9.36	3719.67	15.40	
2	10.05	686.93	2.84	
3	10.43	1264.35	5.23	
4	13.9	14917.03	61.75	

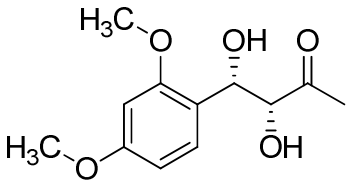
2, 3 – *anti*
4, 5 – *syn*

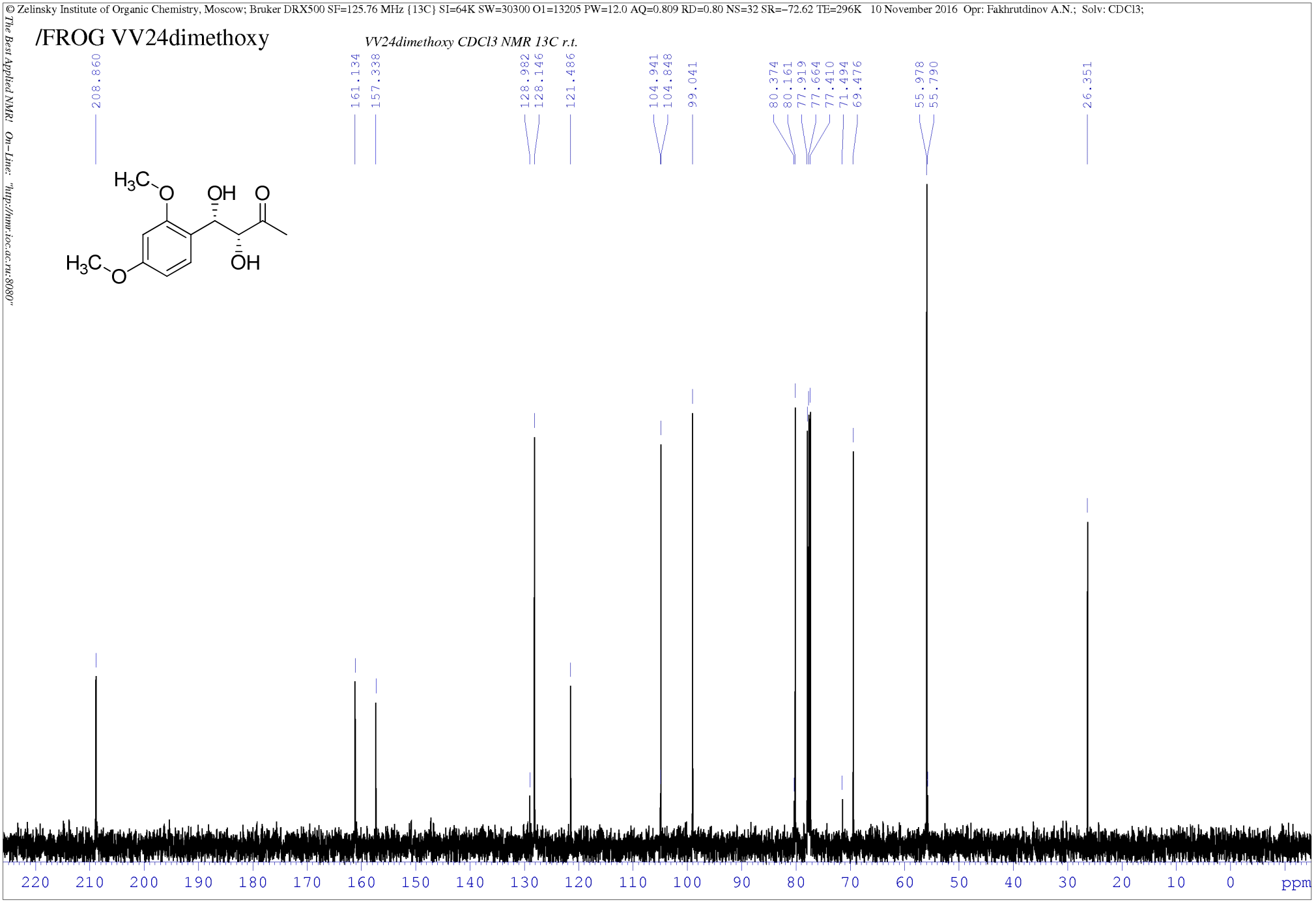
© Zelinsky Institute of Organic Chemistry, Moscow; Bruker DRX500 SF=500.13 MHz {1H} SI=64K SW=8993 O1=3751 PW=16.0 AQ=3.636 RD=0.10 NS=1 SR=4.34 TE=296K 10 November 2016 Opr: Fakhrutdinov A.N.; Solv: CDCl3;

/FROG VV24dimethoxy

VV24dimethoxy CDCl3 NMR 1H r.t.






© Zelinsky Institute of Organic Chemistry, Moscow; Bruker DRX500 SF=125.76 MHz {13C} SI=64K SW=30300 O1=13205 PW=12.0 AQ=0.809 RD=0.80 NS=32 SR=-72.62 TE=296K 10 November 2016 Opr: Fakhrutdinov A.N.; Solv: CDCl3;
The Best Applied NMR! On-Line: "http://nmr.ioc.ac.ru:8080"
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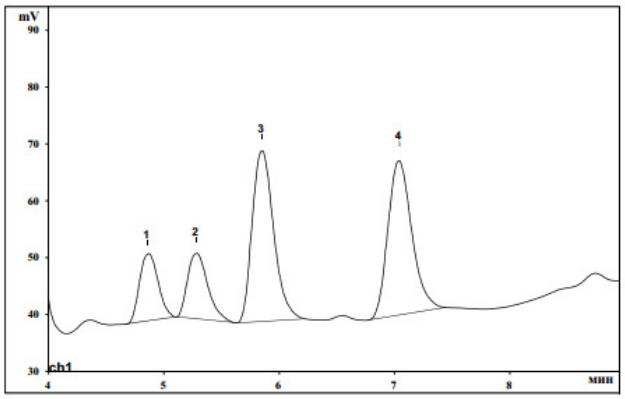
OH
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

HPLC traces for **7**

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



RESULTS
Quantititation method: Нормировка отклика
Standart component: Нет

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2	5.30	99.14	10.22
3	5.87	381.66	39.36
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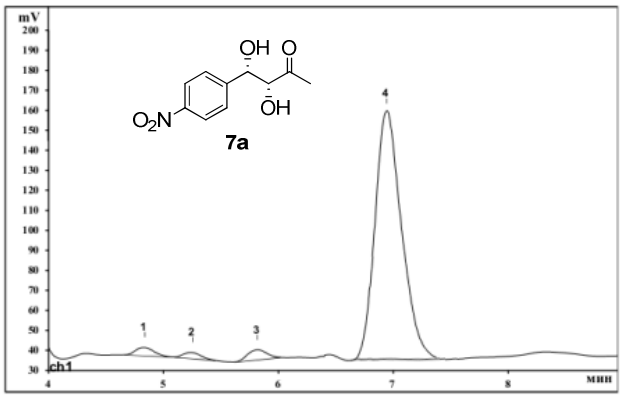

7a

RESULTS
Quantititation method: Нормировка отклика
Standart component: Нет

No	Retention мин	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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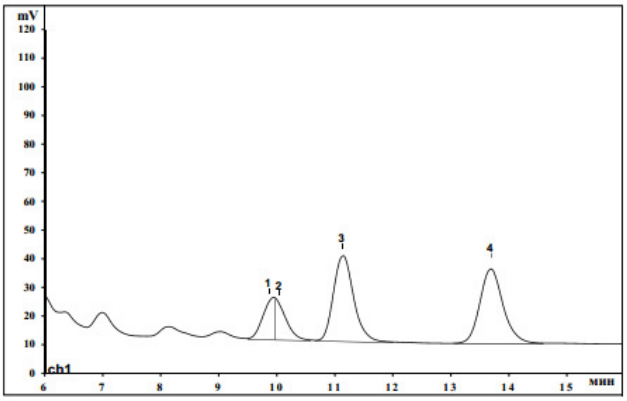
Chiralpak AD-H, 1 ml/min, Hexane:iPrOH = 85:15, λ =280 nm



RESULTS
Quantititation method: Нормировка отклика
Standart component: Нет

No	Retention мин	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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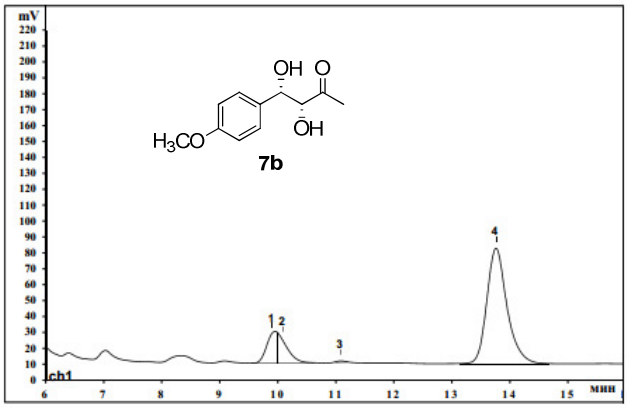

7b

RESULTS
Quantititation method: Нормировка отклика
Standart component: Нет

No	Retention мин	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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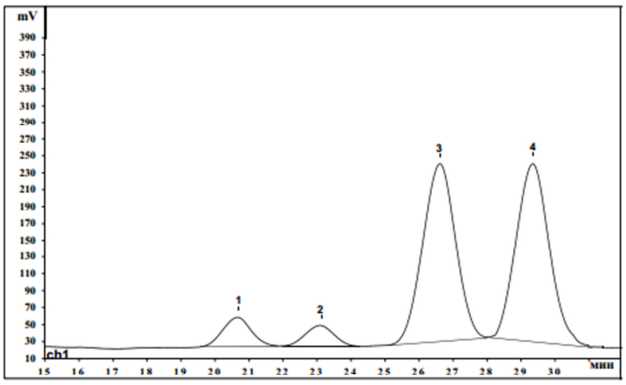
Chiralpak AD-H, 1ml/min, Hexane:iPrOH = 95:5, λ =220 nm



RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

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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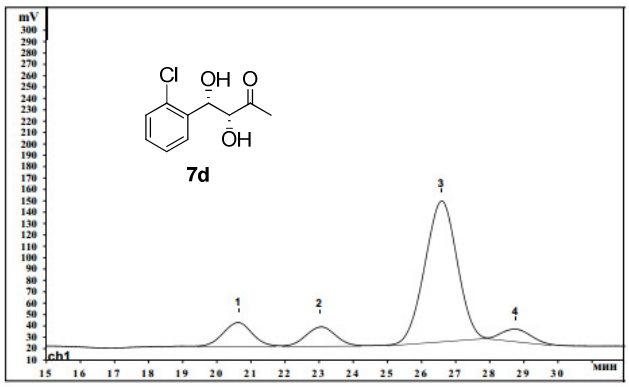

RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

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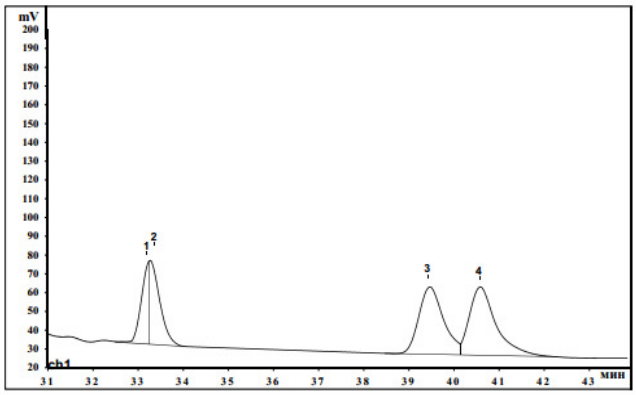
Chiralpak AD-H, 0.6ml/min, Hexane:iPrOH = 90:10, λ =254 nm



RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

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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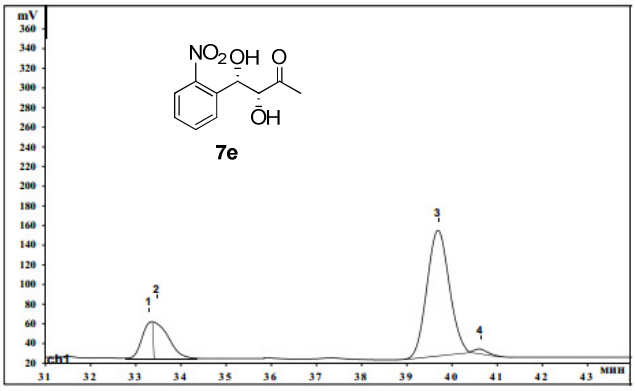

RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

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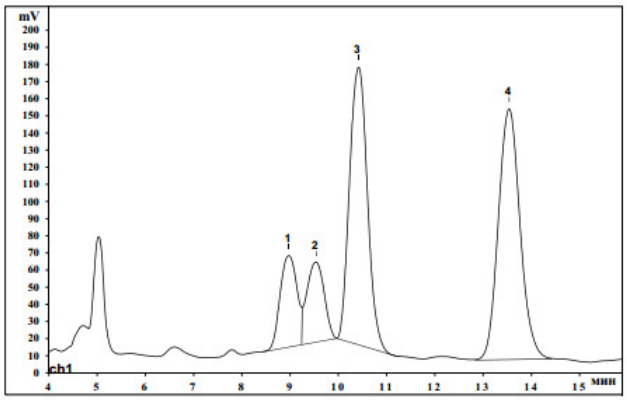
Chiralpak AD-H, 1.0 ml/min, Hexane:iPrOH = 85:15, λ =254 nm



RESULTS
Quantititation method: Нормировка отклика
Standart component: Нет

No	Retention мин	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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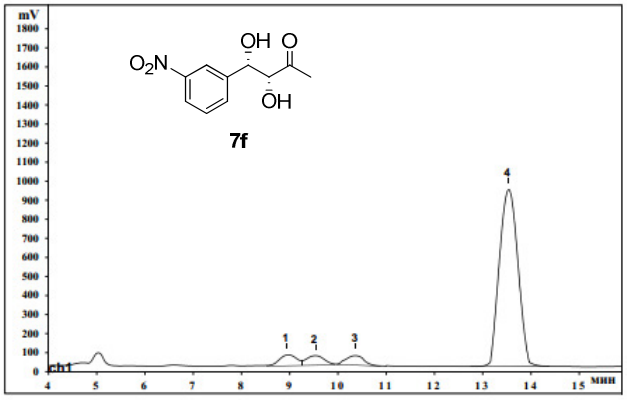

7f

RESULTS
Quantititation method: Нормировка отклика
Standart component: Нет

No	Retention мин	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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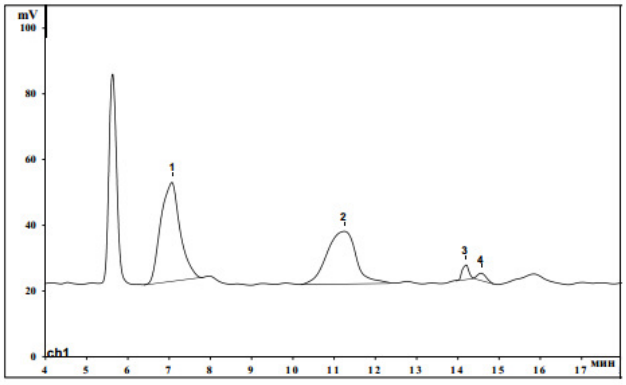
Chiralpak AD-H, 1 ml/min, Hexane:iPrOH = 96:4, λ =254 nm, 24 ^{o}C



RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention мин	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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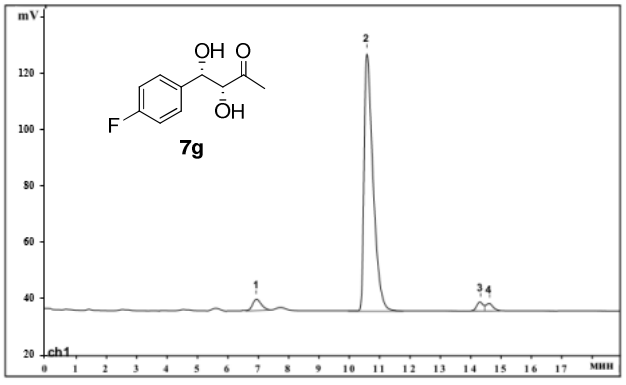

7g

RESULTS
Quantitation method: Нормировка отклика
Standard component: Нет

No	Retention мин	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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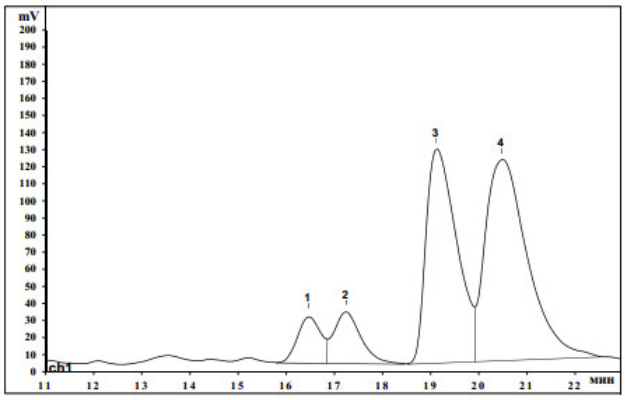
Chiralpak AS-H, 1 ml/min, Hexane:iPrOH = 92:8, λ =220 nm



RESULTS
Quantititation method: Нормировка отклика
Standart 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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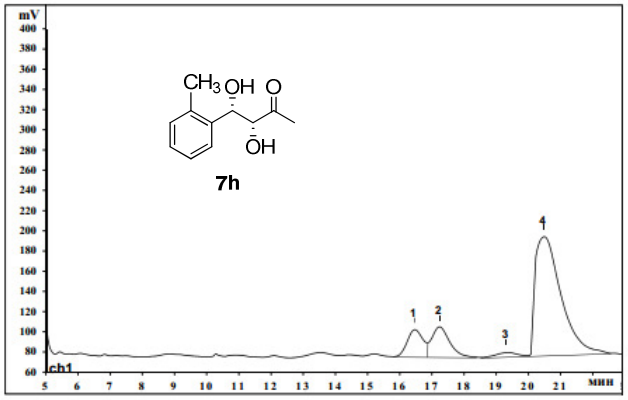

7h

RESULTS
Quantititation method: Нормировка отклика
Standart 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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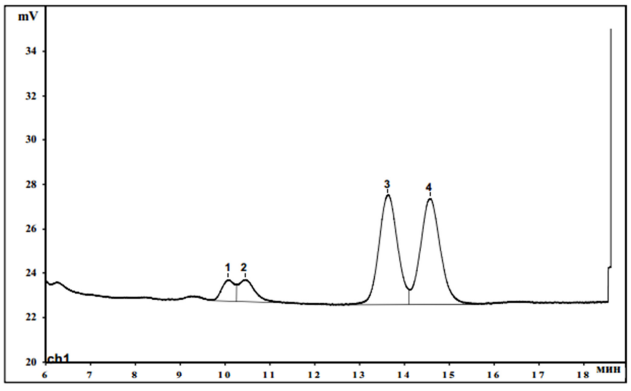
Chiralpak AD-H, 1 ml/min, Hexane:*i*PrOH=80:20, λ=254 nm



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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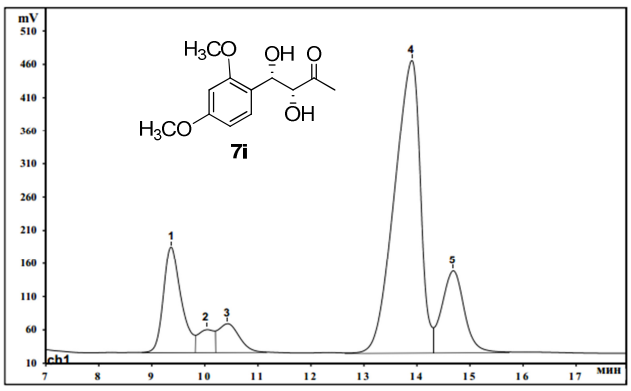

7i

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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