

Enantiodivergent Aldol Condensation in the Presence of Aziridine/Acid/Water Systems

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² University of Lodz, The Bio-Med-Chem Doctoral School of the University of Lodz Institutes of the Polish Academy of Sciences

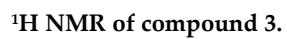
* Correspondence: adam.pieczonka@gmail.com, adam.pieczonka@uni.lodz.pl

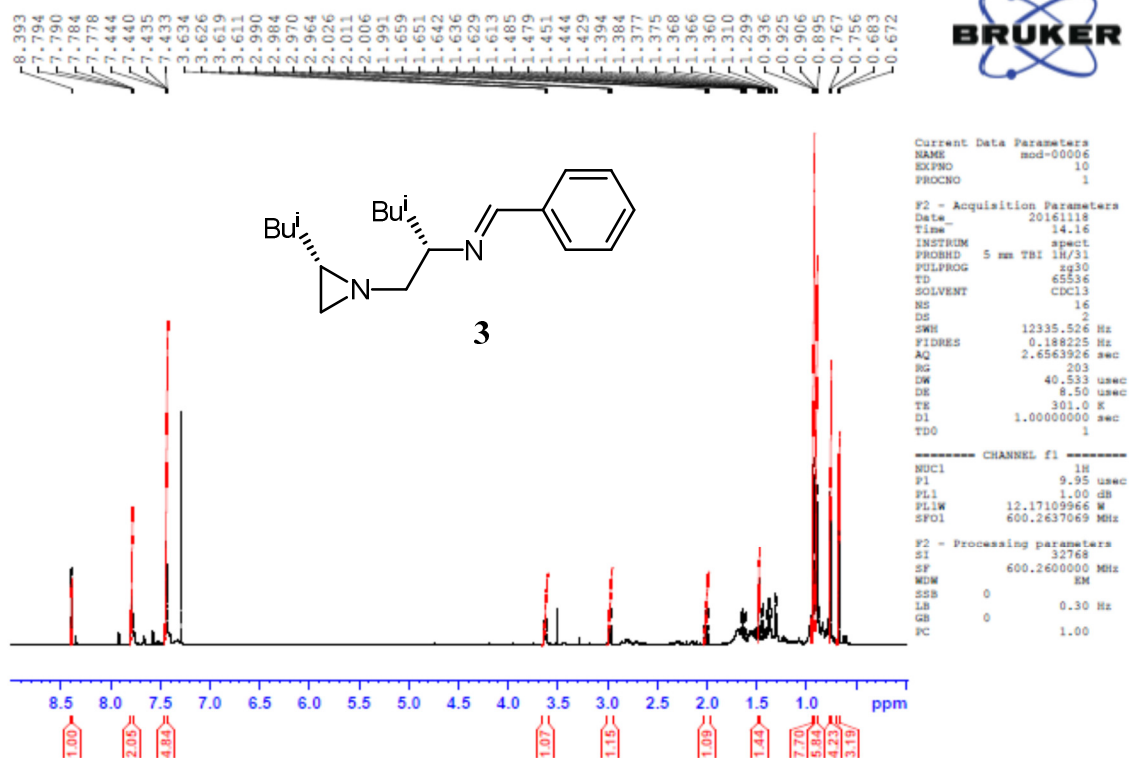
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Copies of NMR Spectra for imines **1-10** S2-S11

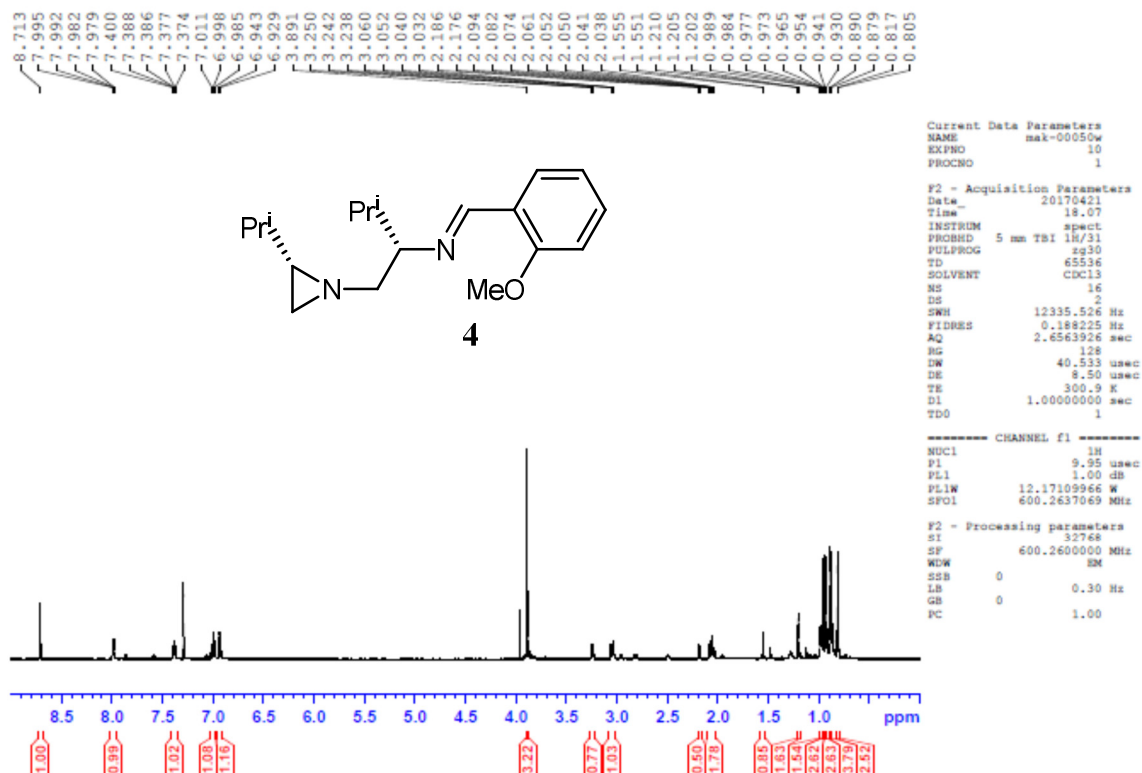
Selected HPLC profiles for the determination of enantiomeric excesses of aldol adducts S12-S26

¹H NMR of compound **2**

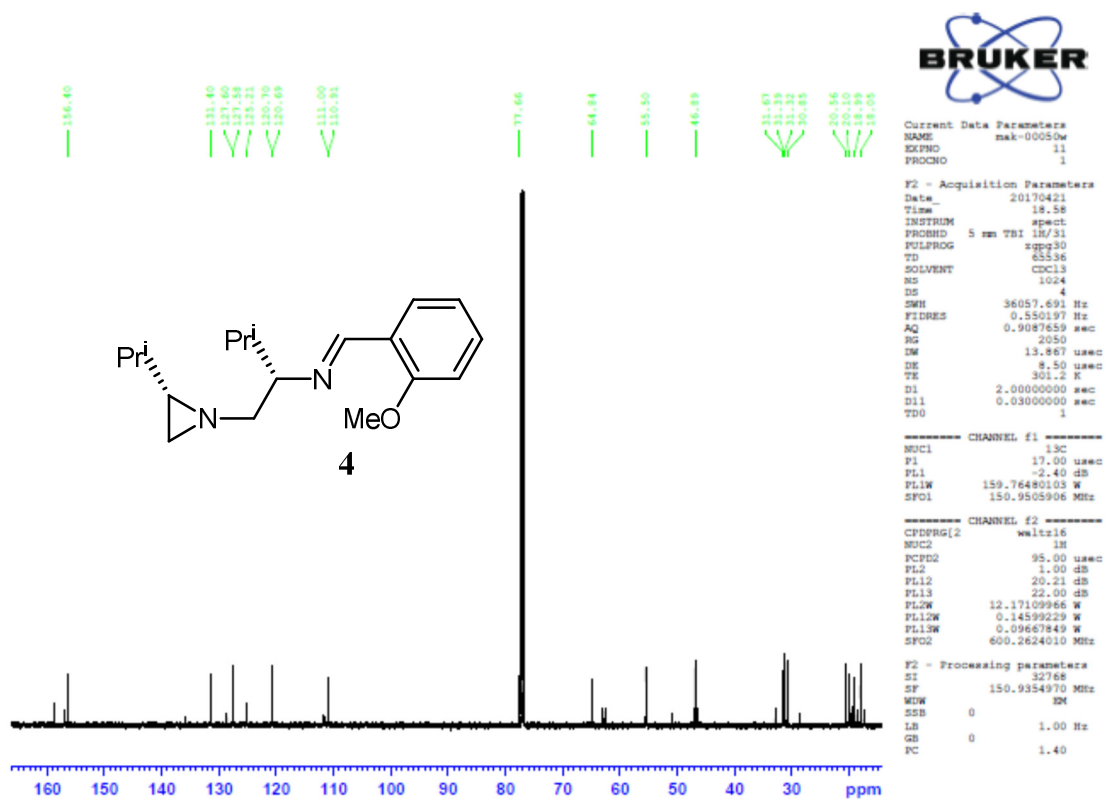




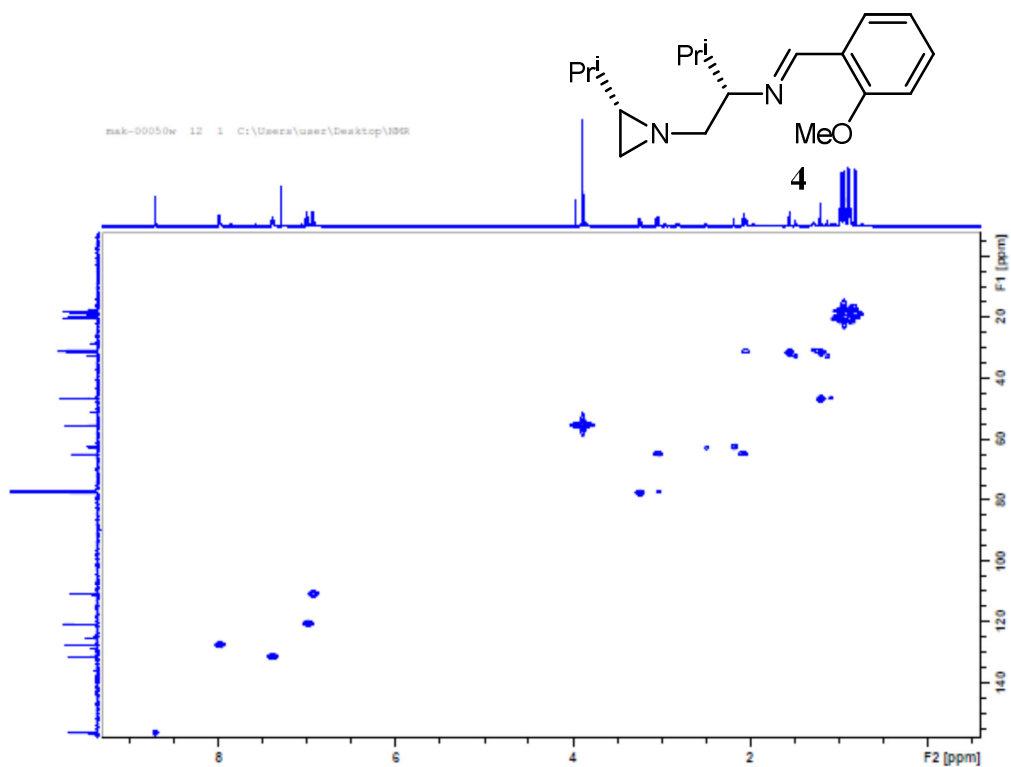
¹H NMR of compound 4.



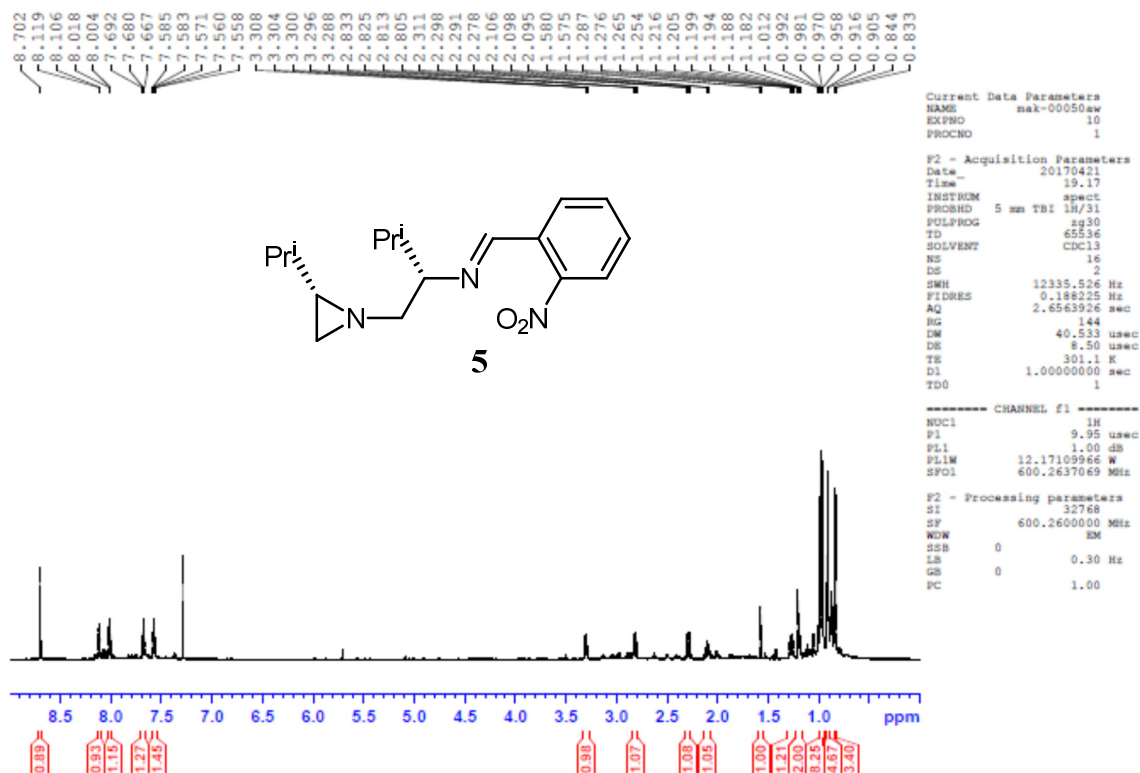
¹³C NMR of compound 4.



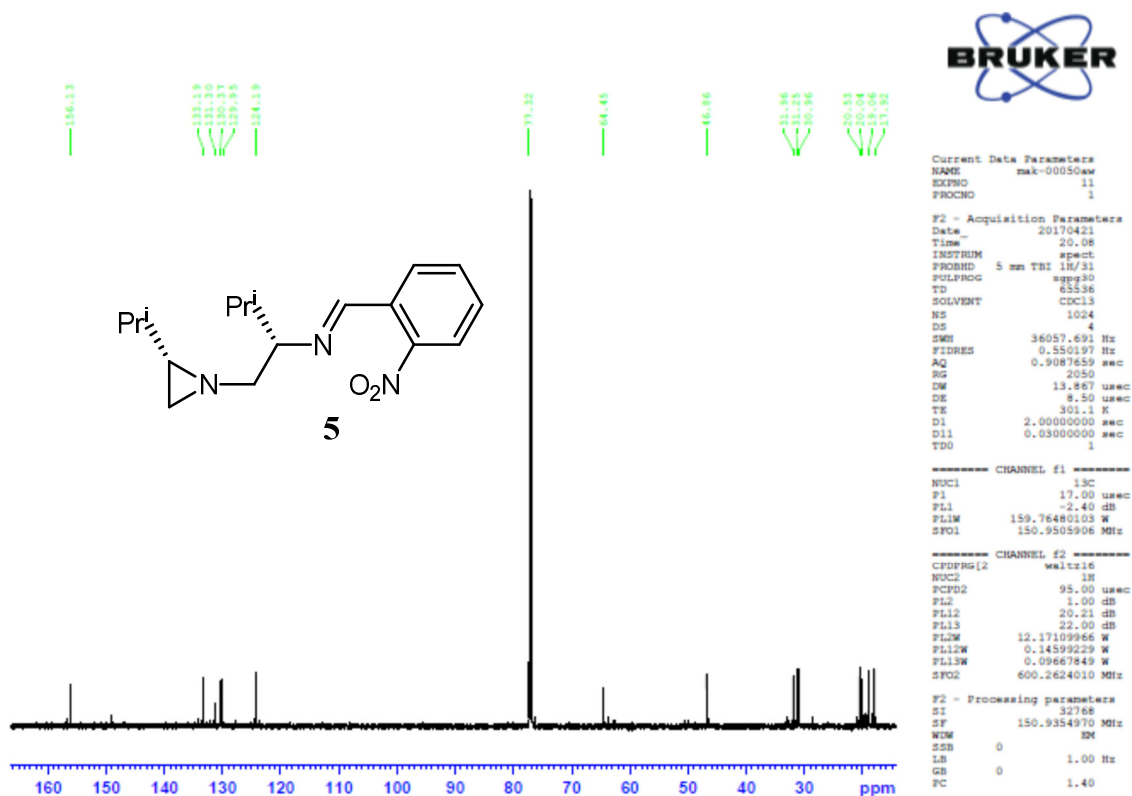
HMQC of compound 4



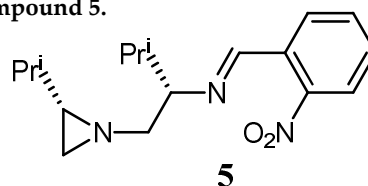
¹H NMR of compound 5.

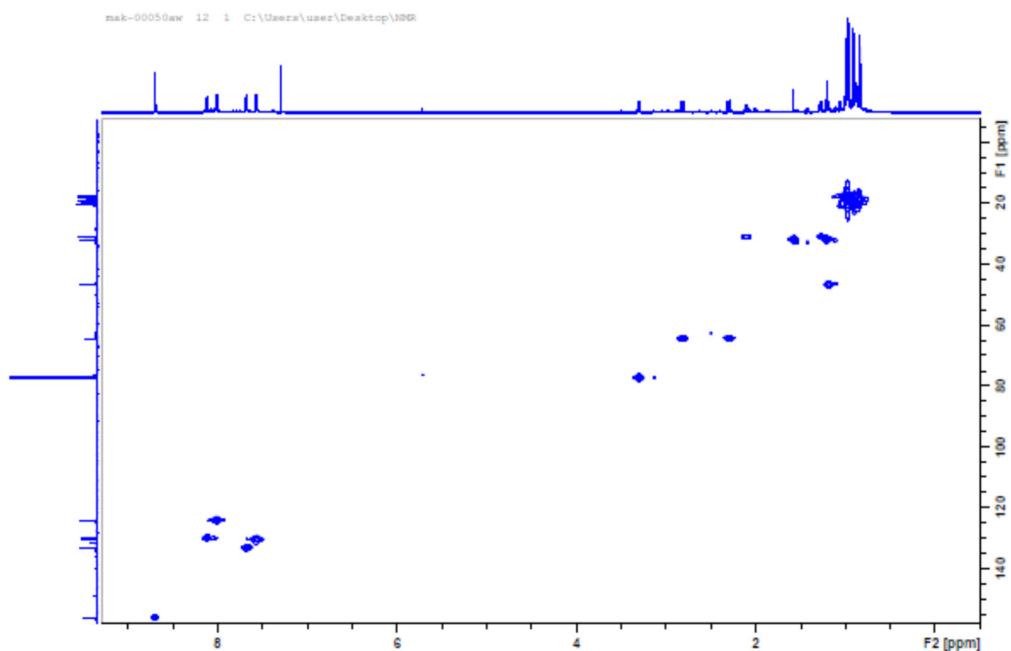


¹³C NMR of compound 5.

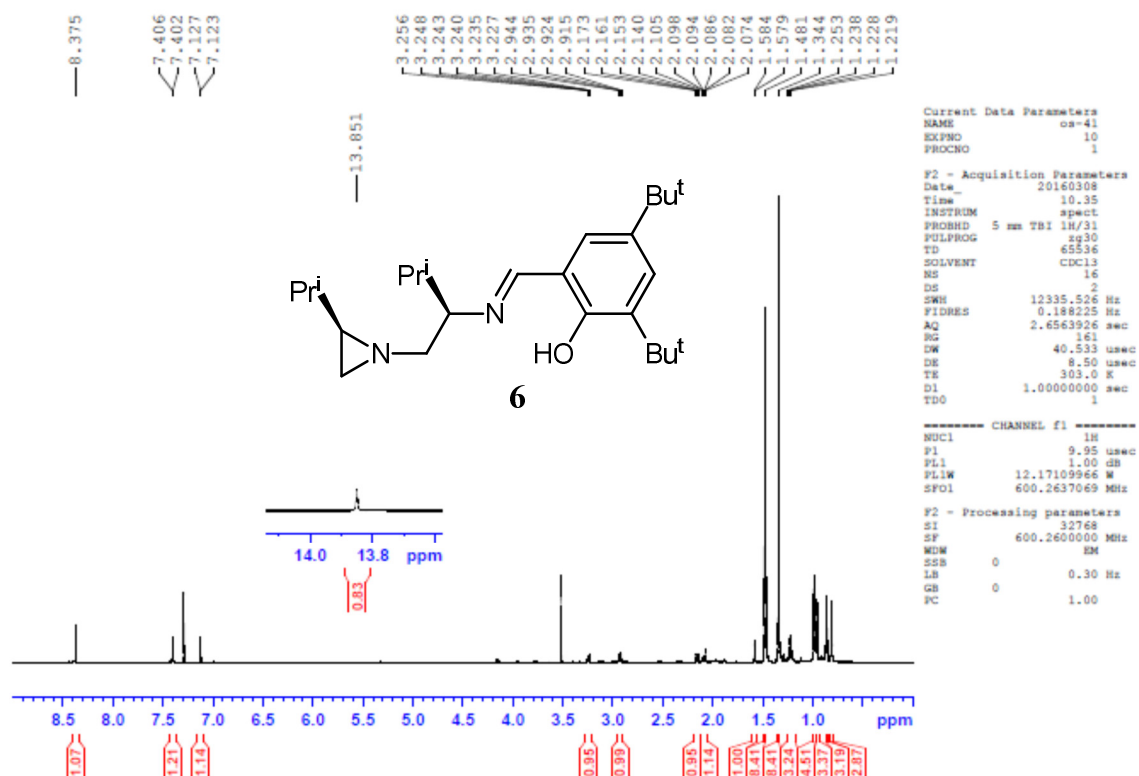


HMQC of compound 5.

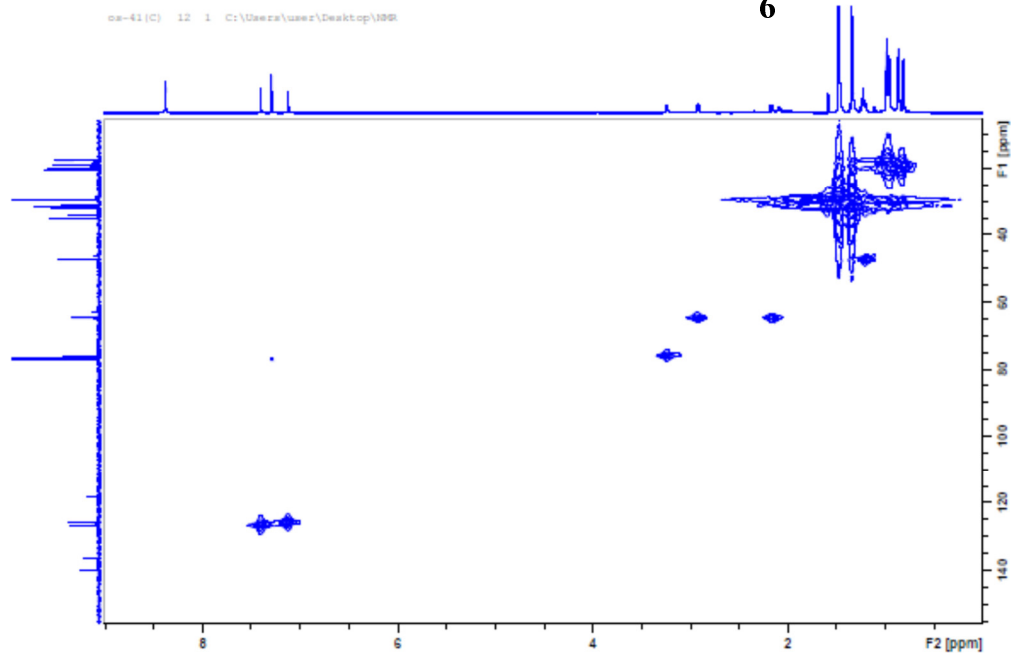
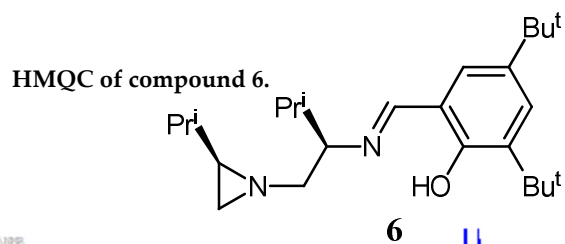




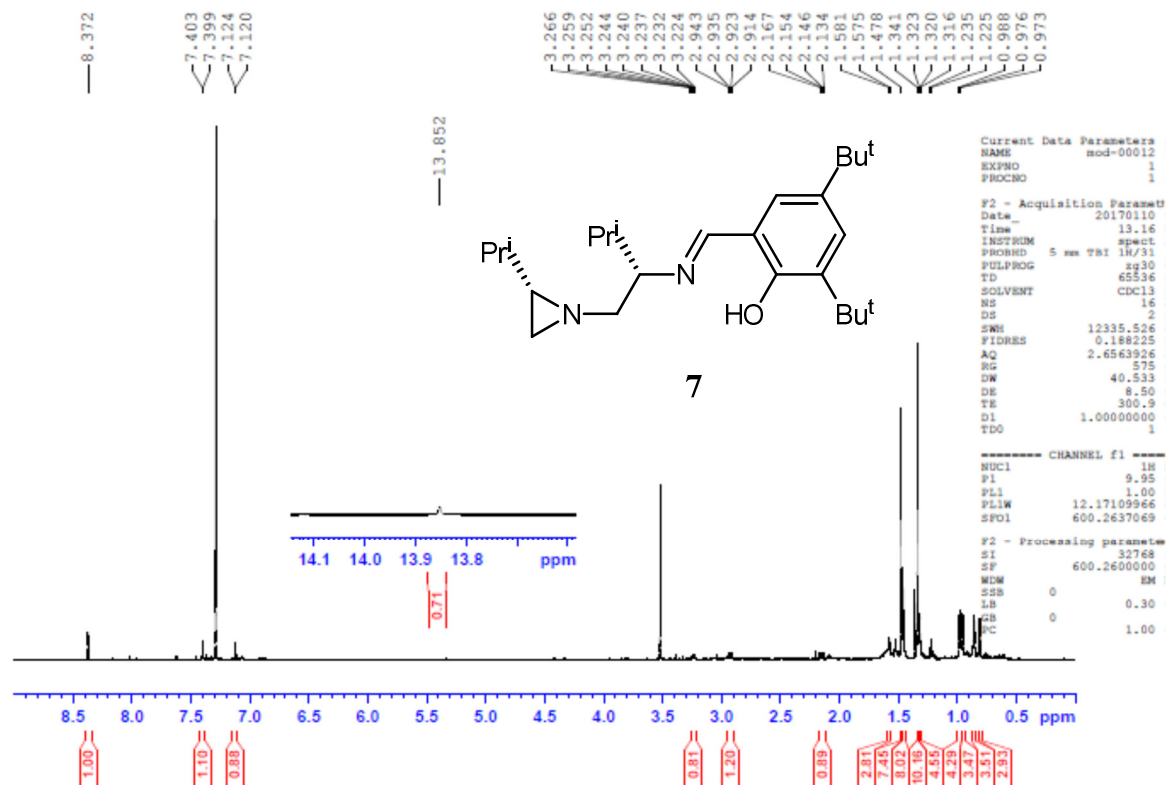
^1H NMR of compound 6.



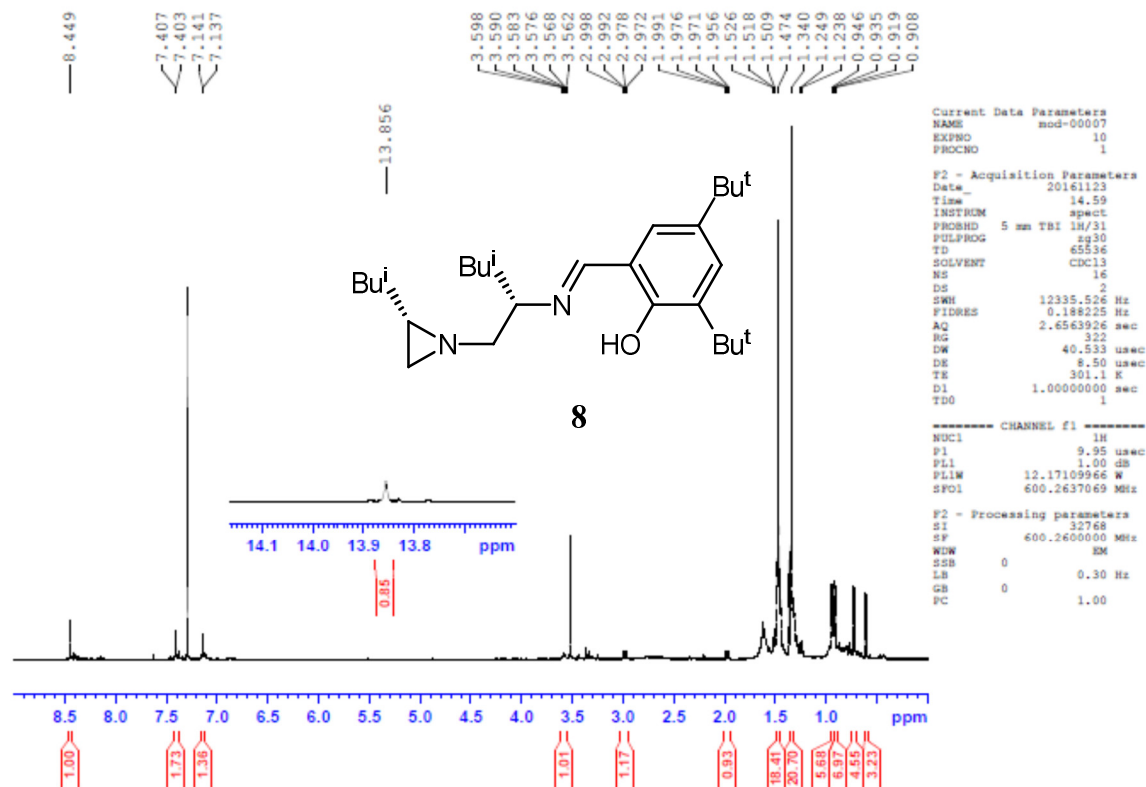
^{13}C NMR of compound 6.



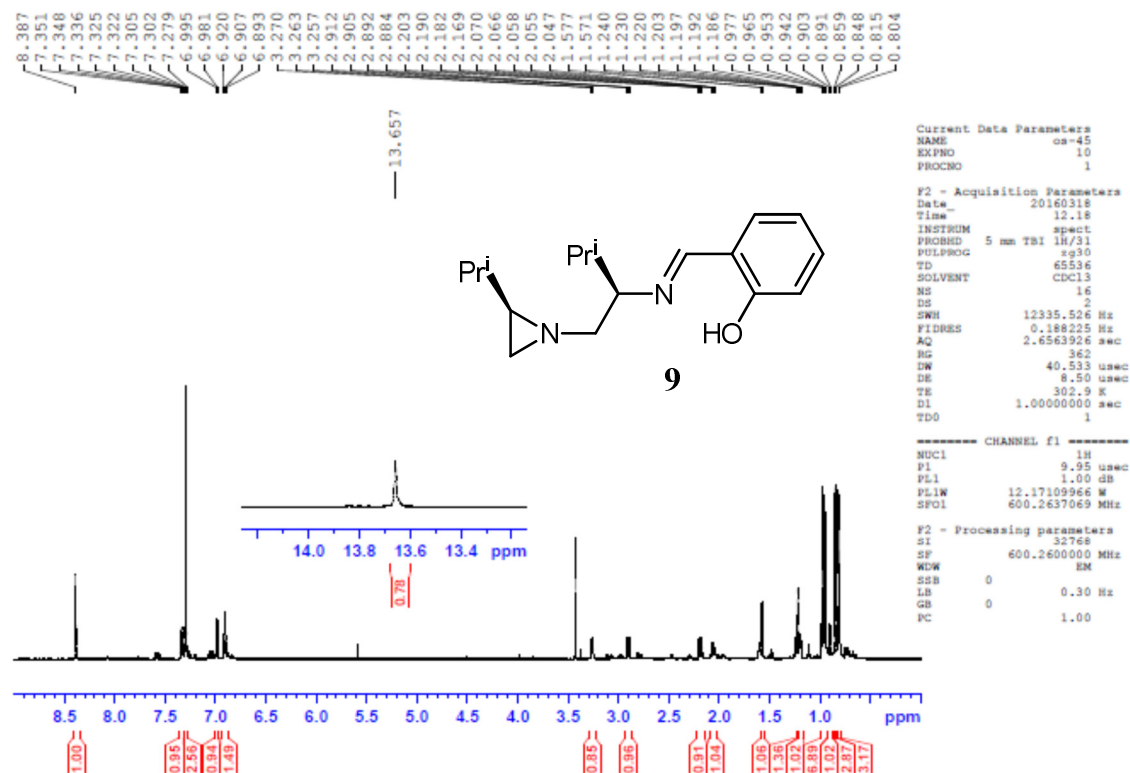
¹H NMR of compound 7.



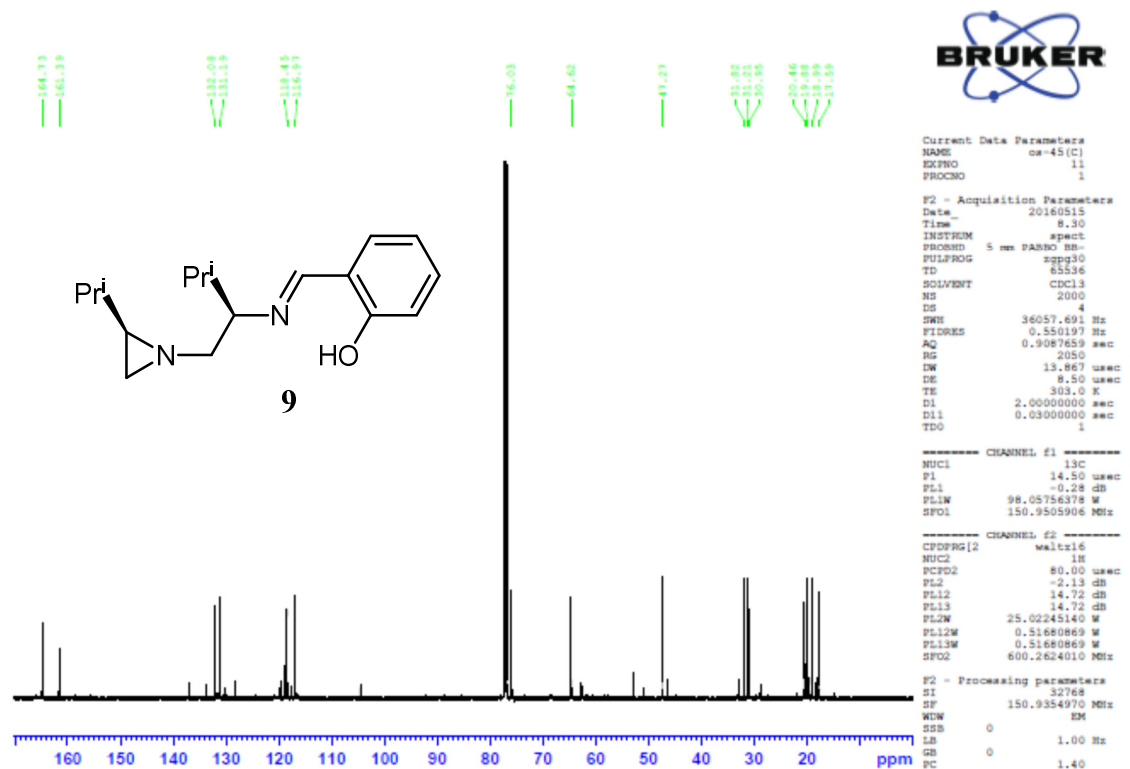
¹H NMR of compound 8.



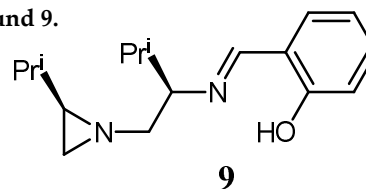
¹H NMR of compound 9.

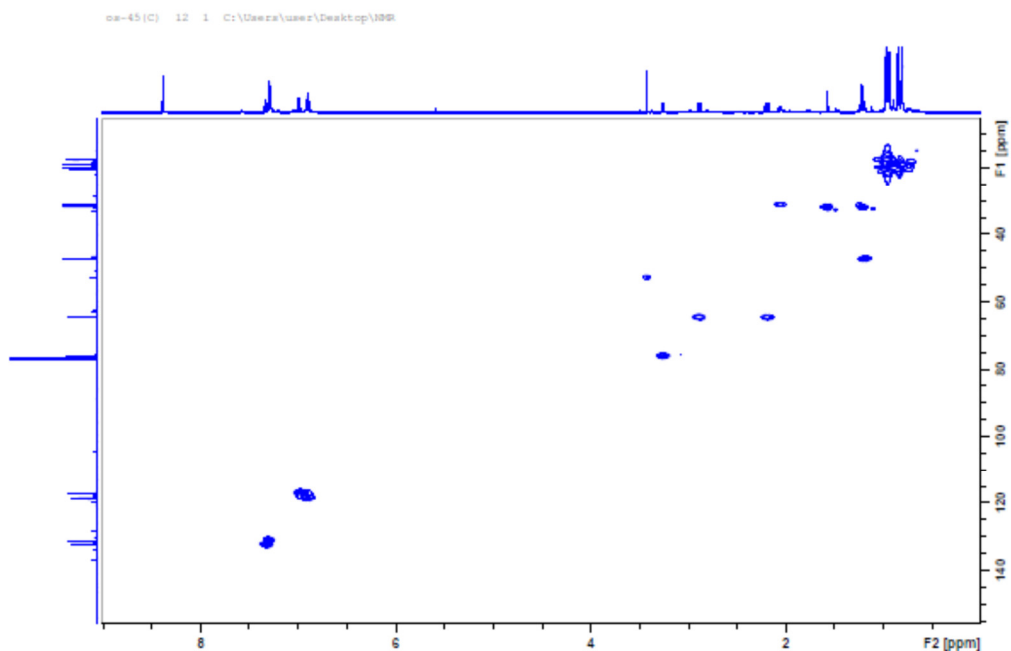


¹³C NMR of compound 9.

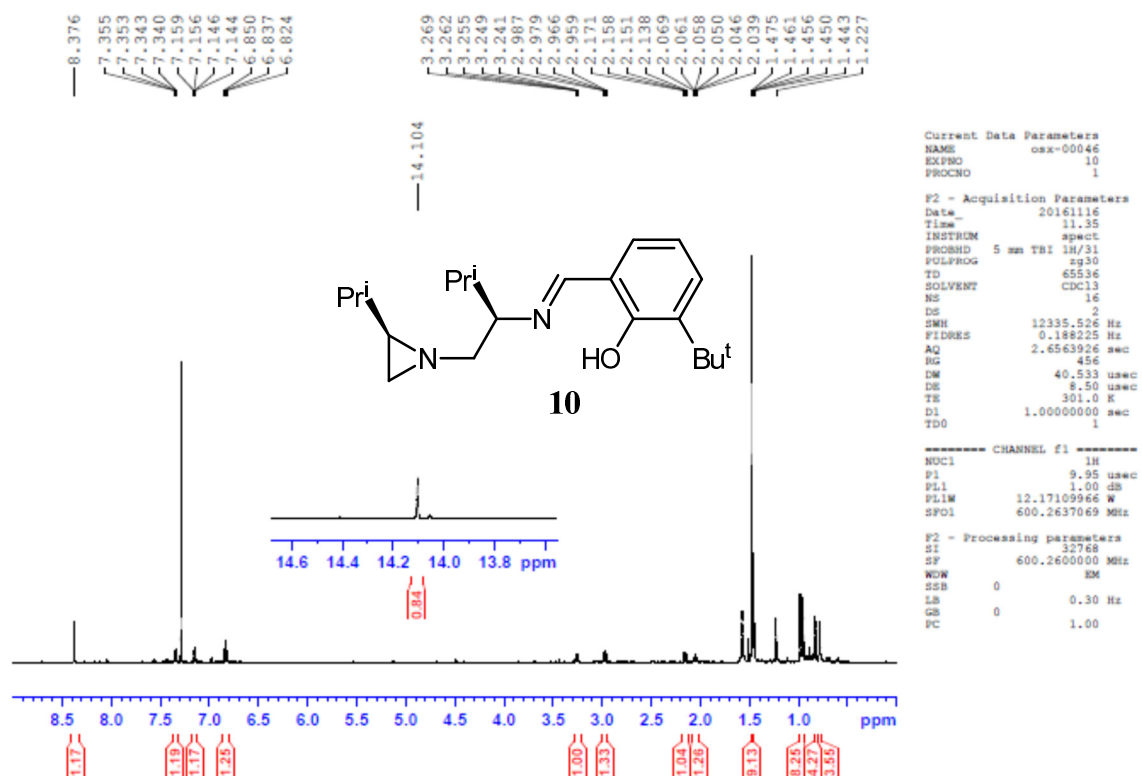


HMQC of compound 9.

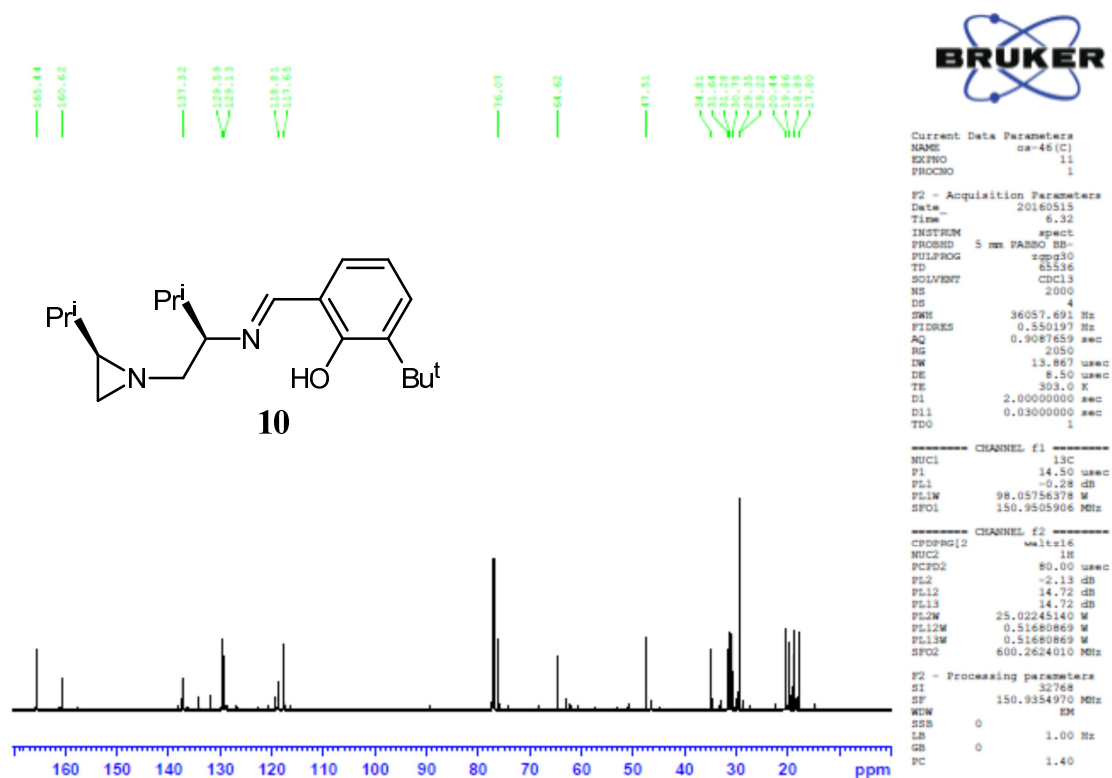




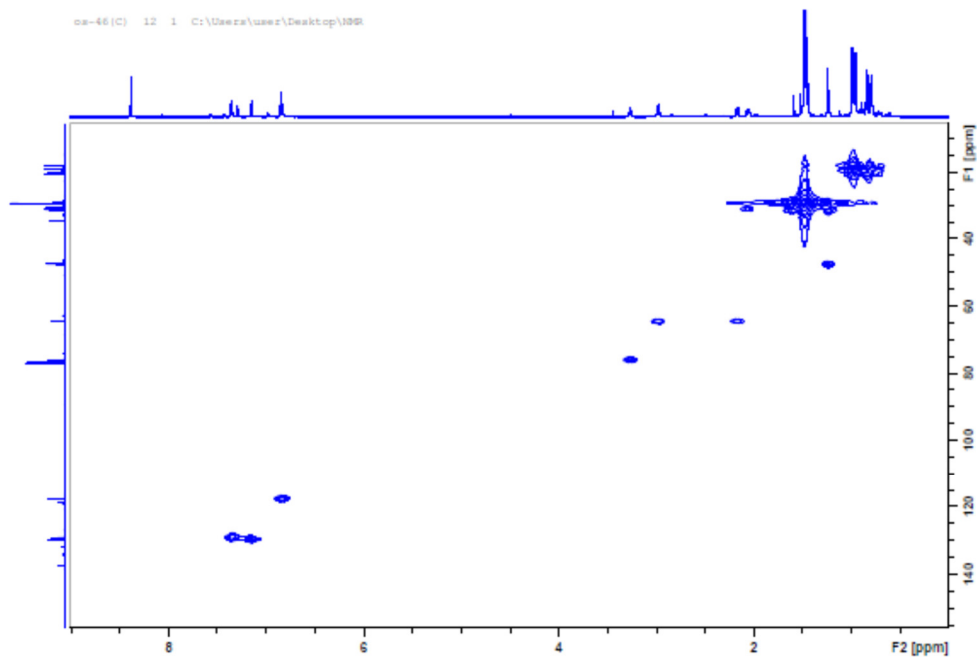
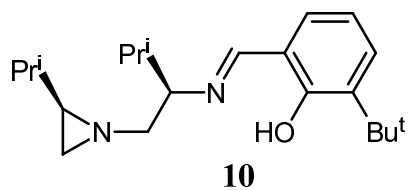
^1H NMR of compound 10.



^{13}C NMR of compound 10.



HMQC of compound 10.

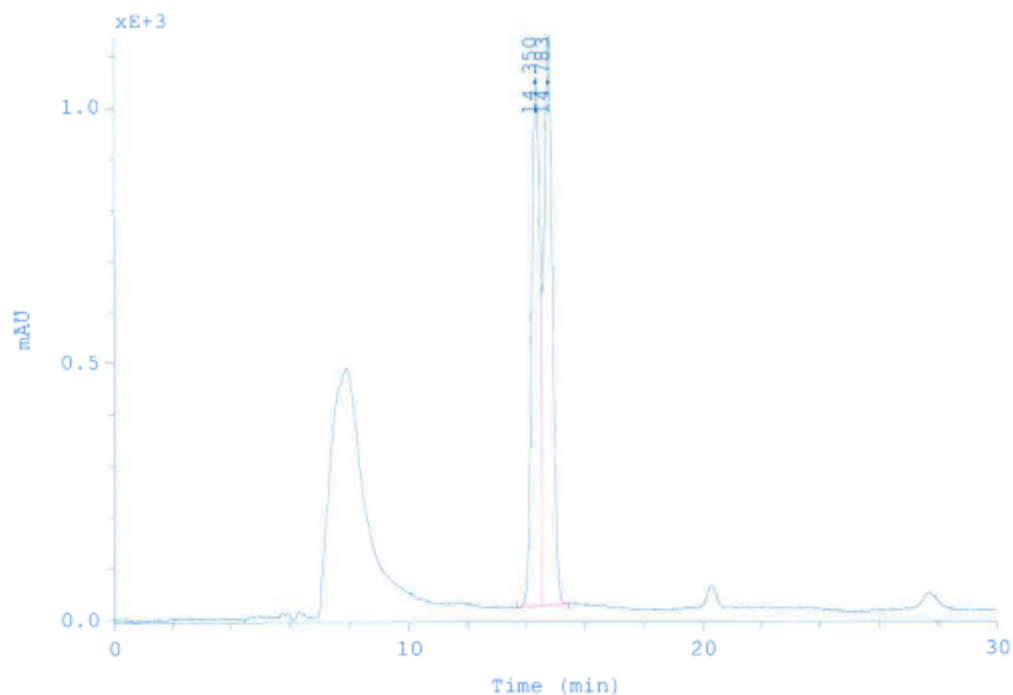


Selected HPLC profiles

HPLC data for racemic aldol adduct (obtained in the use of racemic proline as a catalyst)

Chiral AD-H, iPrOH/*n*-hexane 10/90, flow: 1 ml/min., $\lambda = 254$ nm

Operator: Rachwalski
Object name: Kondensacja aldolowa MIX11 25.IV.2017_Det1-A - Created: 25-04-2017, 11:47
Path: >Testy>Chromatogramy



Operator: Rachwalski
Object name: Kondensacja aldolowa MIX11 25.IV.2017_Det1-A - Created: 25-04-2017, 12:18
Path: >Testy>Chromatogramy

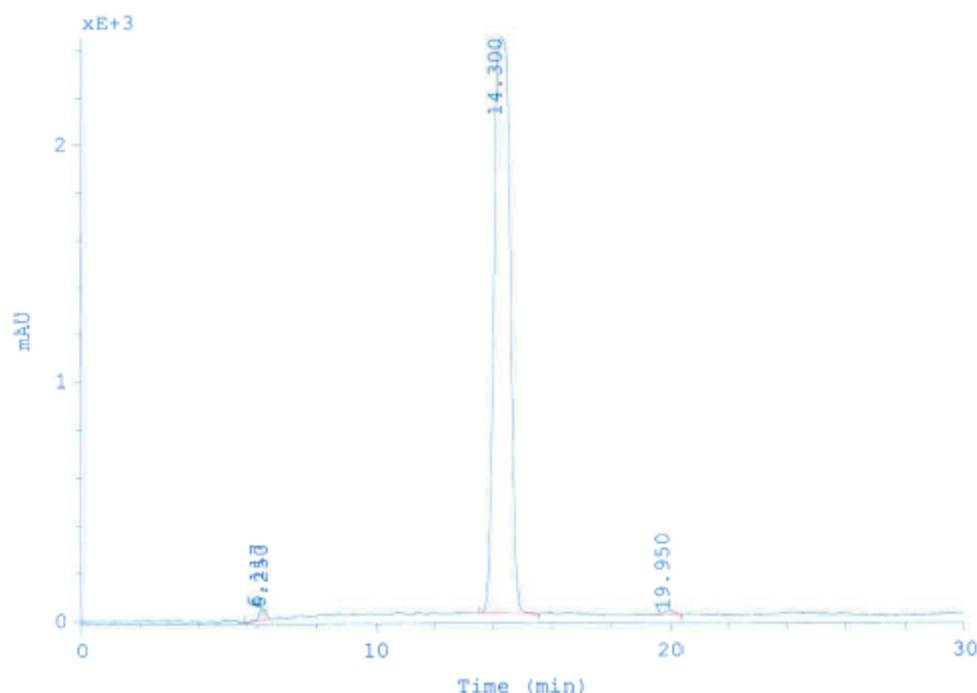
	Ret.time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	14.350	13.72	14.55		0		335.856	1032.51	47.3213	0.260	BP
2	14.783	14.55	15.45		0		373.879	1112.01	52.6787	0.260	PB

HPLC data for aldol adduct (Table 2, entry 2).

Chiral AD-H, iPrOH/*n*-hexane 10/90, flow: 1 ml/min., $\lambda = 254$ nm

$[\alpha]_D^{23} -60.7$ (c 1.00; chloroform)¹

Operator: Rachwalski
Object name: Kondensacja aldolowa MIX7 12.IV.2017_Det1-A - Created: 12-04-2017, 13:01
Path: >Testy>Chromatogramy



Operator: Rachwalski
Object name: Kondensacja aldolowa MIX7 12.IV.2017_Det1-A - Created: 12-04-2017, 13:31
Path: >Testy>Chromatogramy

	Ret.time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	6.117	5.53	6.20		0		8.76111	52.2315	0.5704	0.121	BP
2	6.250	6.20	6.48		0		5.70132	45.3216	0.3712	0.092	PB
3	14.300	13.52	15.55		0		1517.13	2421.18	98.7679	0.599	BB
4	19.950	19.62	20.40		0		4.46274	13.6007	0.2905	0.319	BB

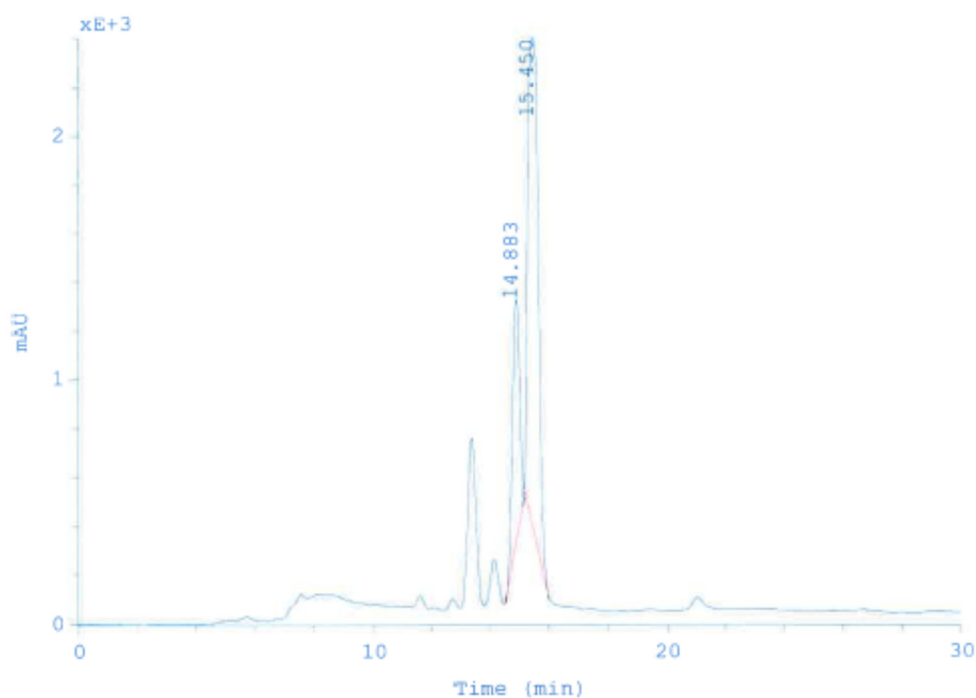
HPLC data for aldol adduct (Table 2, entry 16).

Chiral AD-H, iPrOH/*n*-hexane 10/90, flow: 1 ml/min., $\lambda = 254$ nm

$[\alpha]_D^{23} -30.6$ (c 1.00; chloroform)

¹ M. Rachwalski, S. Lesniak, P. Kiełbasinski *Tetrahedron: Asymmetry* **2011**, 22, 1325; J. Paradowska, M. Pasternak, B. Gut, B. Gryzłó, J. Młynarski *J. Org. Chem.* **2012**, 77, 173.

Operator: Rachwałski
 Object name: Kondensacja aldolowa MIX4 BIS EtOH 2.III_Det1-A - Created: 02-03-2017, 12:42
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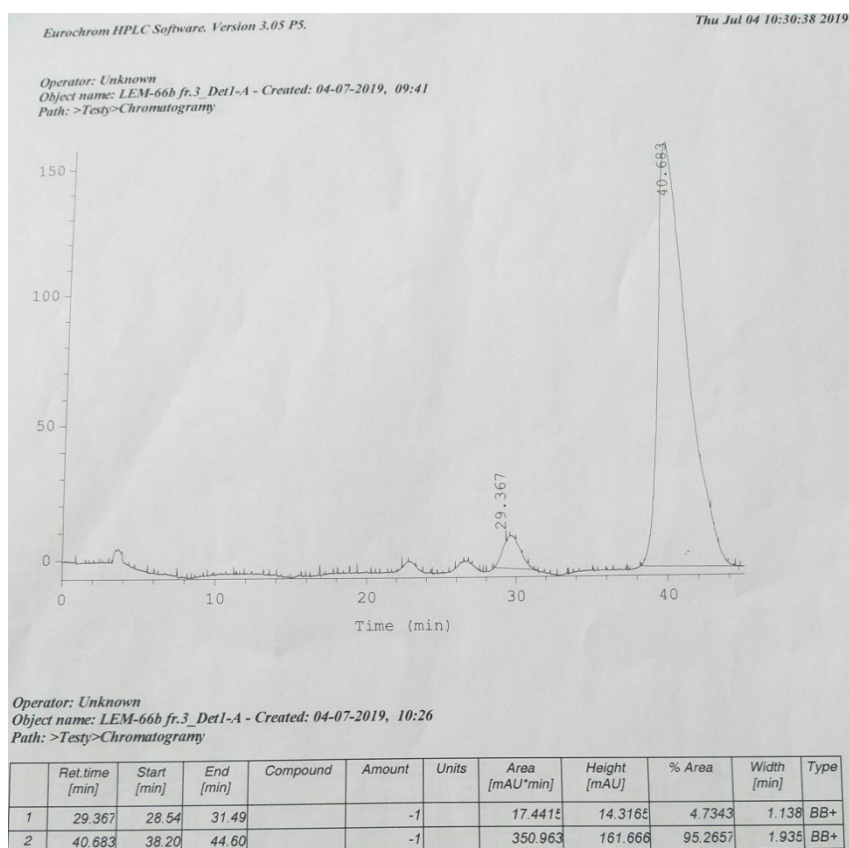
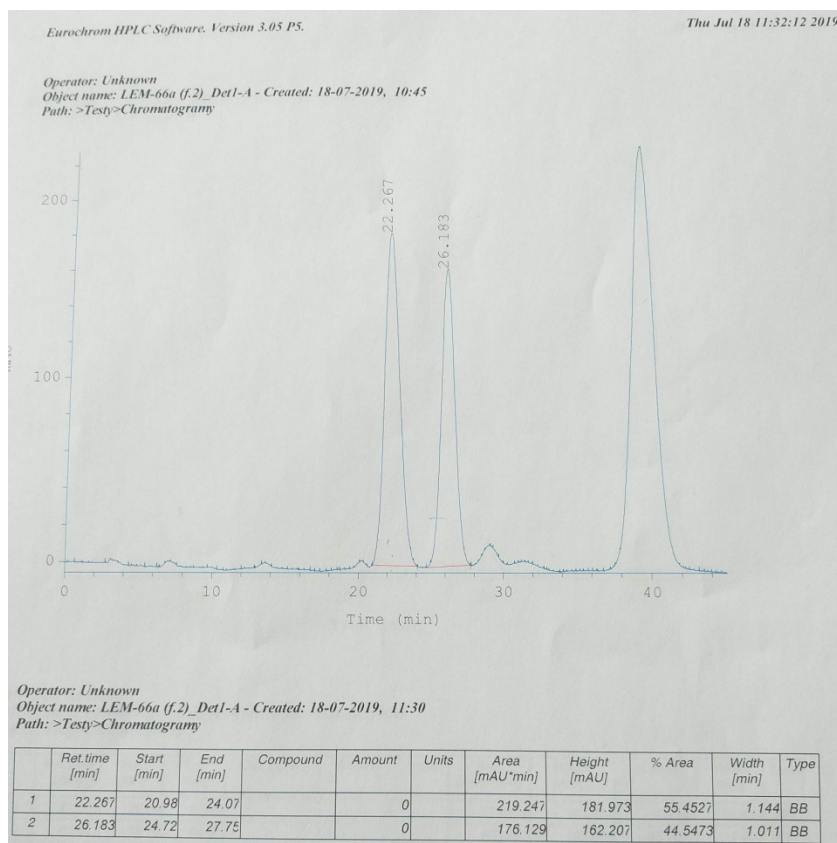


Operator: Rachwałski
 Object name: Kondensacja aldolowa MIX4 BIS EtOH 2.III_Det1-A - Created: 02-03-2017, 13:13
 Path: >Testy>Chromatogramy

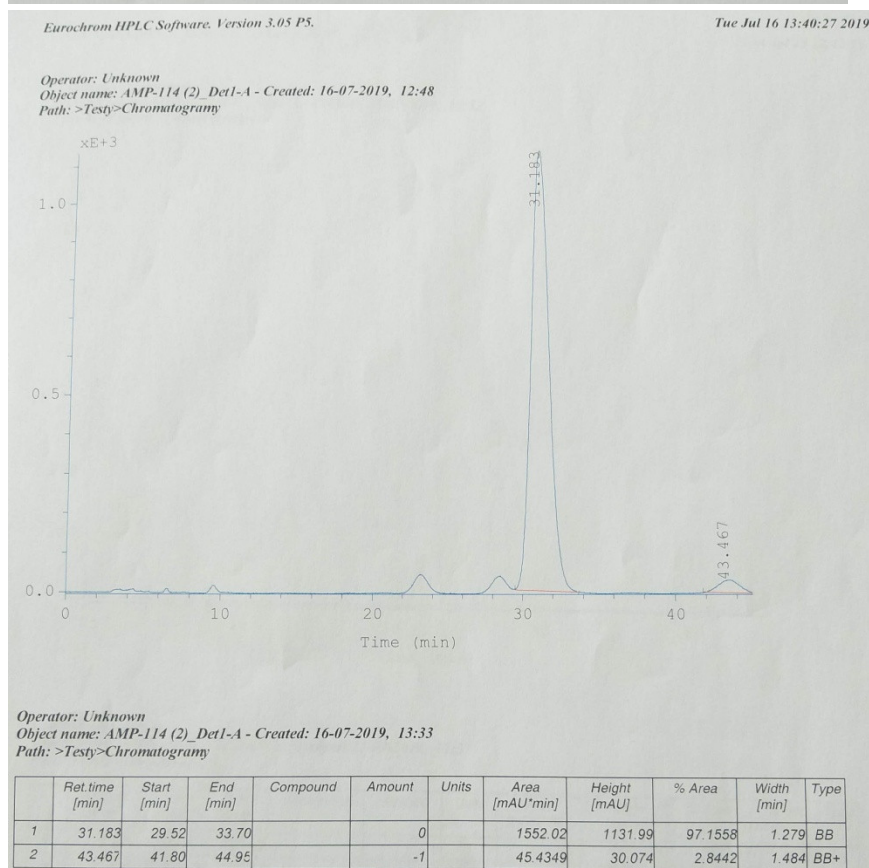
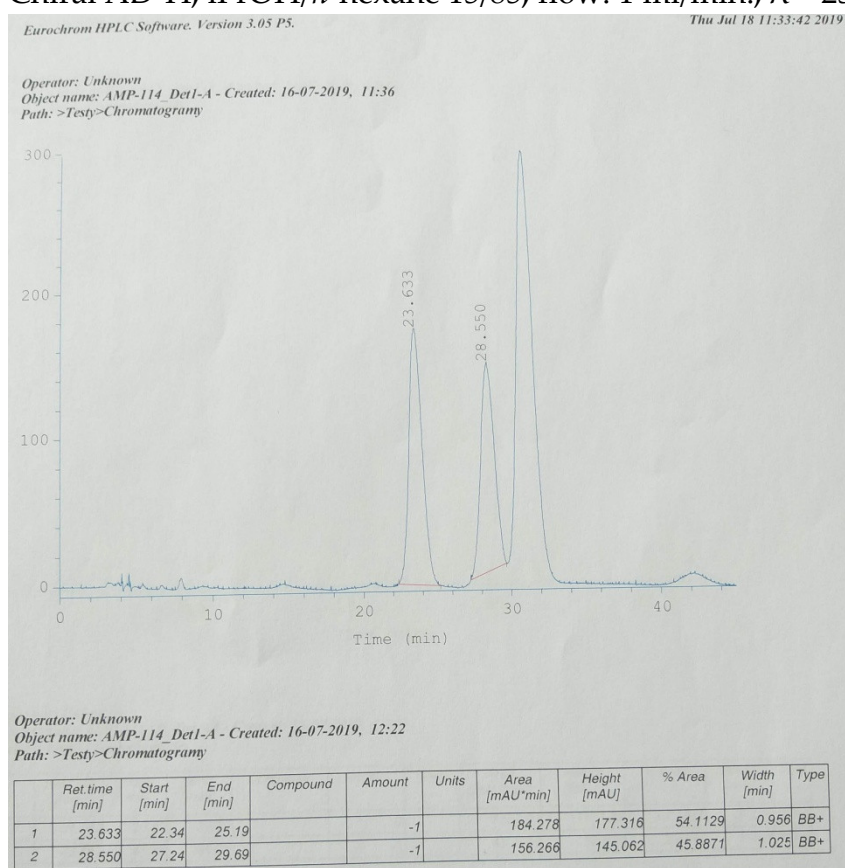
	Ret.time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	14.883	14.51	15.13		-1		273.025	991.04	24.8155	0.276	BB+
2	15.450	15.16	15.97		-1		827.195	1997.13	75.1845	0.414	BB+

HPLC data for aldol adduct (Table 4, entry 3).

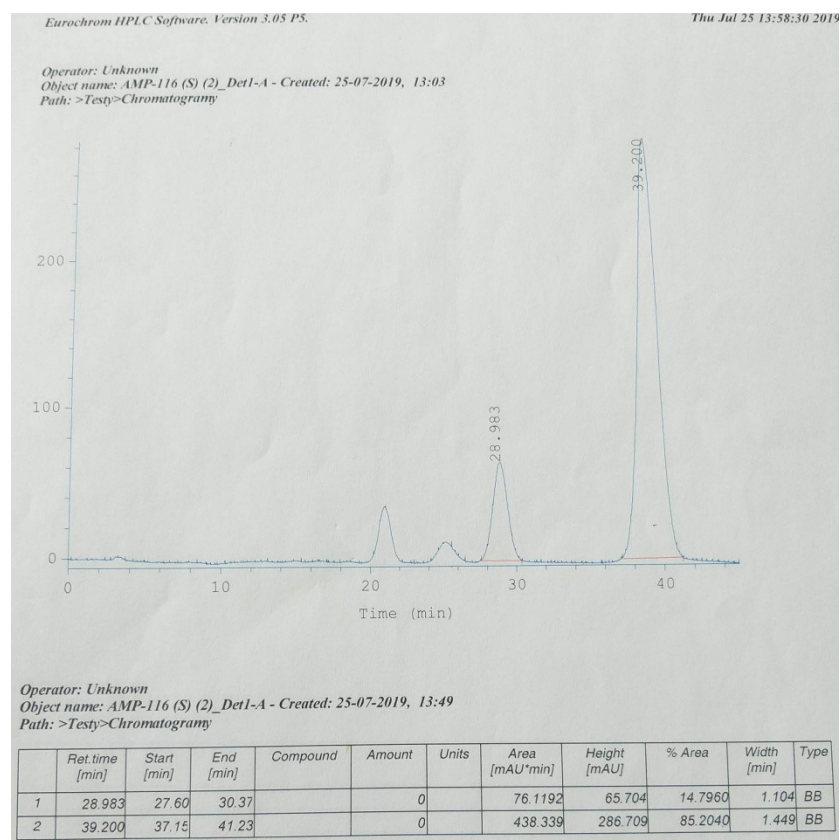
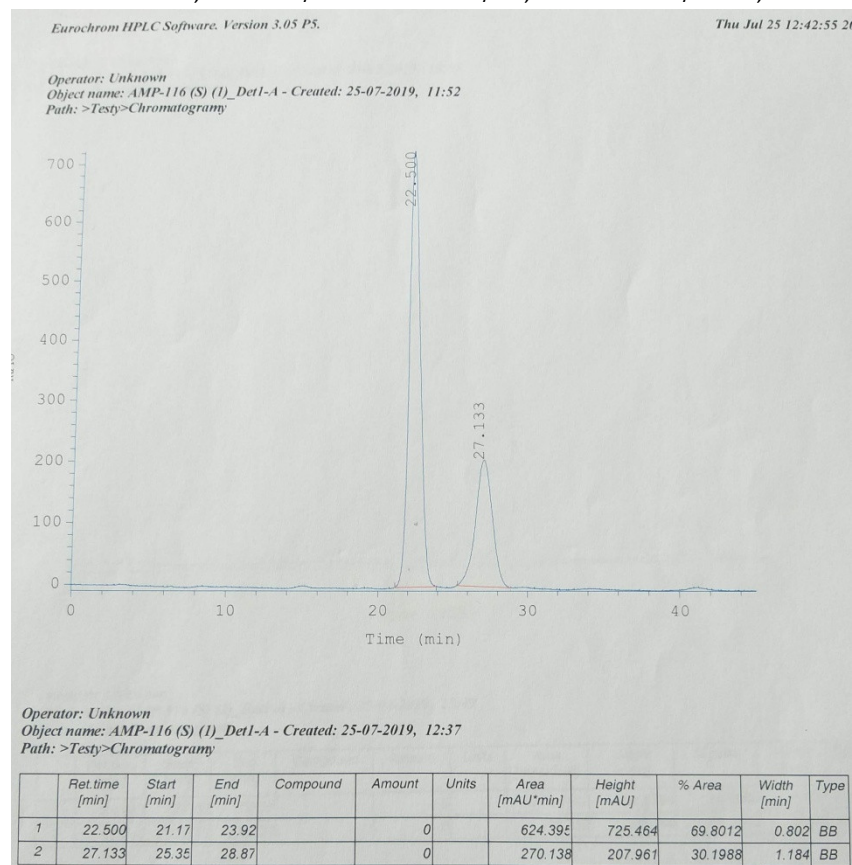
Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., λ = 254 nm



HPLC data for aldol adduct (Table 4, entry 4).

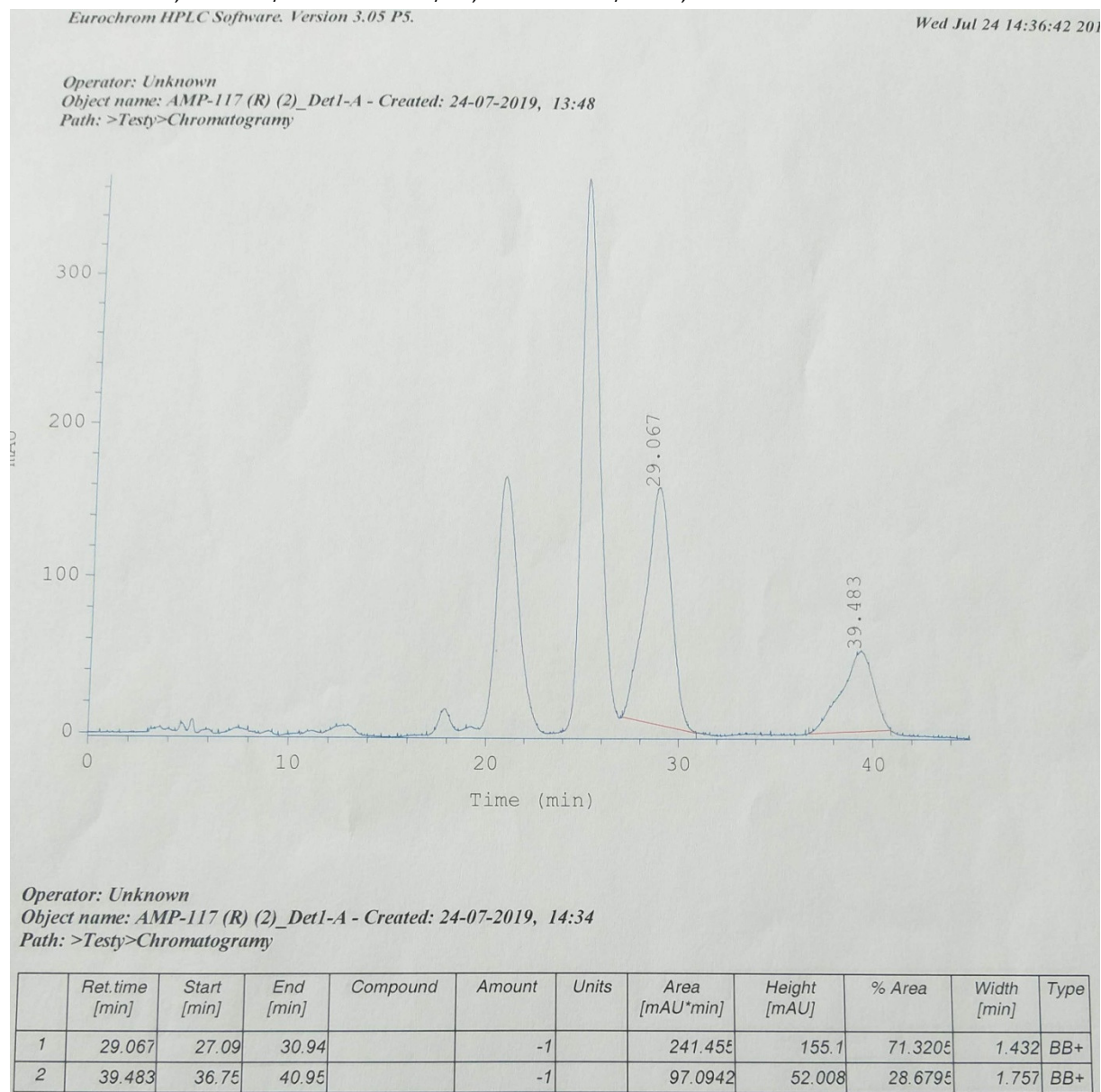
Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., $\lambda = 254$ nm

HPLC data for aldol adduct (Table 4, entry 5).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., $\lambda = 254$ nm

HPLC data for aldol adduct (Table 4, entry 6).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., $\lambda = 254$ nm



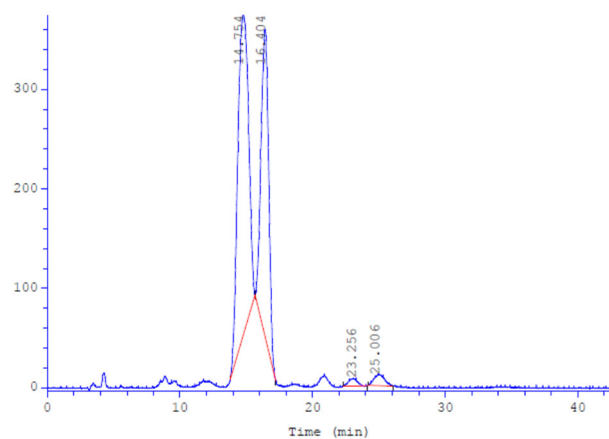
HPLC data for aldol adduct (Table 7, entry 1).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., $\lambda = 254$ nm

Eurochrom HPLC Software, Version 3.05 P5.

Fri Feb 07 12:10:13 2020

Operator: Rachwalski
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 Path: >Testy>Chromatography



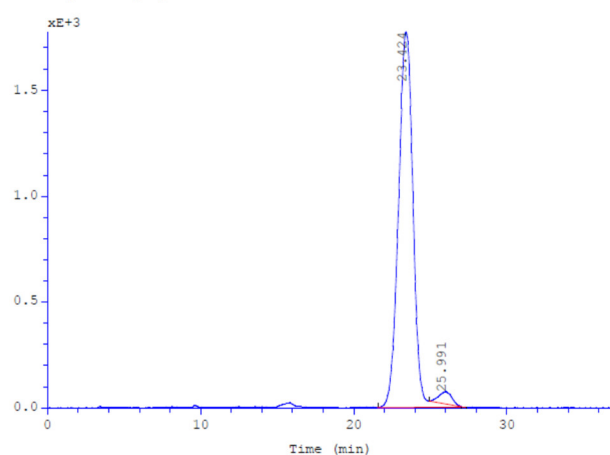
Operator: Rachwalski
 Object name: LEM-100a f. 3_Det1-A - Created: 07-02-2020, 12:07
 Path: >Testy>Chromatography

	Ret time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU·min]	Height [mAU]	% Area	Width [min]	Type
1	14.754	13.79	15.67		0		305.085	322.711	58.0190	0.946	BB
2	16.404	15.67	17.22		0		204.378	307.573	38.8673	0.650	BB
3	23.256	22.31	24.11		-1		5.78996	8.21457	1.1011	0.695	BB+
4	25.006	24.16	26.05		-1		10.584	11.3478	2.0128	0.893	BB+

Eurochrom HPLC Software, Version 3.05 P5.

Thu Feb 06 15:17:58 2020

Operator: Rachwalski
 Object name: LEM-100b f. 4.5_Det1-A - Created: 06-02-2020, 14:39
 Path: >Testy>Chromatography



Operator: Rachwalski
 Object name: LEM-100b f. 4.5_Det1-A - Created: 06-02-2020, 15:17
 Path: >Testy>Chromatography

	Ret time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU·min]	Height [mAU]	% Area	Width [min]	Type
1	23.424	21.57	27.36		0		1967.36	1774.3	97.2320	1.010	BB
2	25.991	24.96	27.06		0		56.0073	57.6413	2.7680	0.926	SS

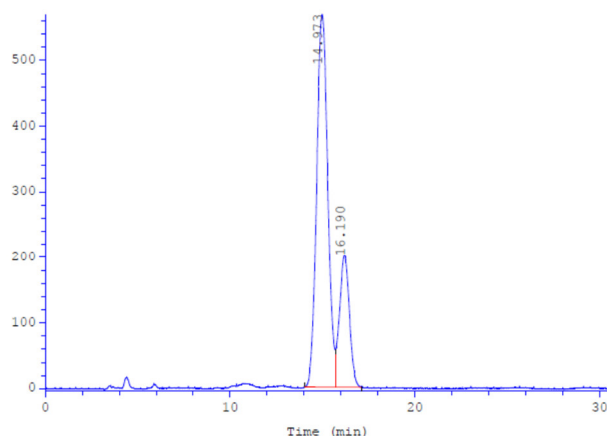
HPLC data for aldol adduct (Table 7, entry 2).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., λ = 254 nm

Eurochrom HPLC Software, Version 3.05 P5.

Thu Feb 06 13:06:10 2020

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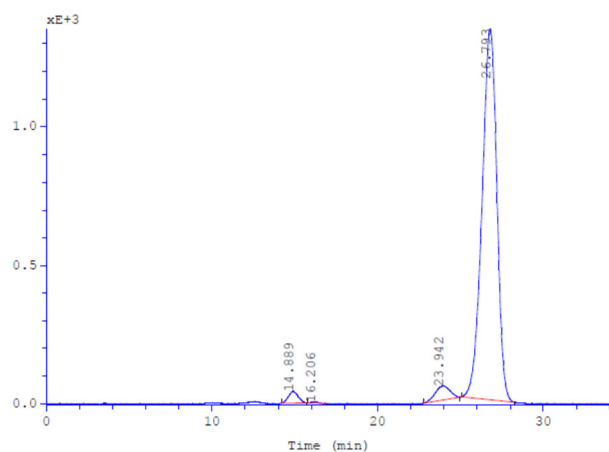
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 Path: >Testy>Chromatogramy

	Rettime [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU·min]	Height [mAU]	% Area	Width [min]	Type
1	14.973	14.06	15.69		0		394.633	567.294	75.8493	0.619	BP
2	16.190	15.69	17.09		0		125.653	200.608	24.1507	0.526	PB

Eurochrom HPLC Software, Version 3.05 P5.

Thu Feb 06 13:51:49 2020

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 Path: >Testy>Chromatogramy



Operator: Rachwalski
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 Path: >Testy>Chromatogramy

	Rettime [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU·min]	Height [mAU]	% Area	Width [min]	Type
1	14.889	14.17	15.74		0		28.7287	42.2144	1.9521	0.648	BB
2	16.206	15.77	16.90		-1		2.15636	4.22829	0.1465	0.559	BB+
3	23.942	22.76	24.99		0		52.6602	49.9239	3.5782	1.023	BB
4	26.793	25.09	28.26		0		1388.16	1338.12	94.3232	0.969	BB

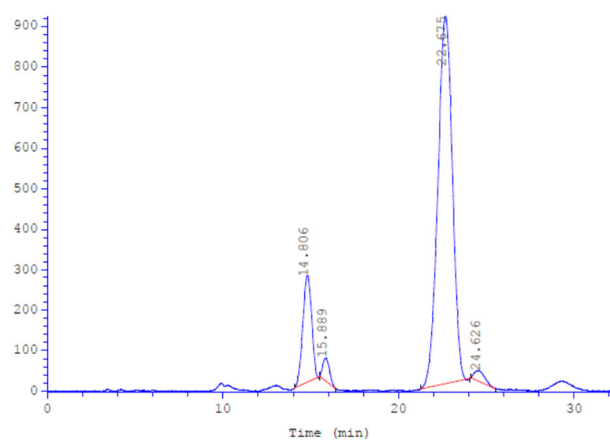
HPLC data for aldol adduct (Table 7, entry 3).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., λ = 254 nm

Eurochrom HPLC Software, Version 3.05 P5.

Wed Feb 19 13:41:02 2020

Operator: Rachwalski
 Object name: LEM-101 II a f. 3-6_Det1-A - Created: 19-02-2020, 13:06
 Path: >Test>Chromatogram



Operator: Rachwalski
 Object name: LEM-101 II a f. 3-6_Det1-A - Created: 19-02-2020, 13:39
 Path: >Test>Chromatogram

	Ret time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	14.806	14.08	15.48		-1		151.876	262.02	14.2690	0.561	BB+
2	15.889	15.52	16.41		-1		24.6004	57.4865	2.3113	0.451	BB+
3	22.675	21.28	24.07		-1		869.97	906.95	81.7354	0.900	BB+
4	24.626	24.11	25.54		-1		17.9275	27.8019	1.6843	0.723	BB+

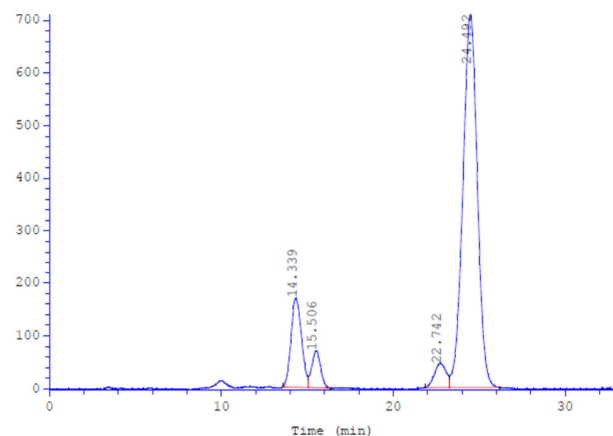
HPLC data for aldol adduct (Table 7, entry 4).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., $\lambda = 254$ nm

Eurochrom HPLC Software, Version 3.05 P5.

Wed Feb 19 11:41:32 2020

Operator: Rachwalski
 Object name: LEM-99 II a f. 4-12_Det1-A - Created: 19-02-2020, 11:04
 Path: >Test>Chromatogram



Operator: Rachwalski
 Object name: LEM-99 II a f. 4-12_Det1-A - Created: 19-02-2020, 11:38
 Path: >Test>Chromatogram

	Ret time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	14.339	13.57	15.04		0		118.509	169.712	13.3352	0.633	BP
2	15.506	15.04	16.32		0		42.0104	69.9121	4.7272	0.505	PB
3	22.742	21.84	23.28		0		36.5868	45.6823	4.1168	0.640	BP
4	24.492	23.28	26.19		0		691.588	709.042	77.8207	0.893	PB

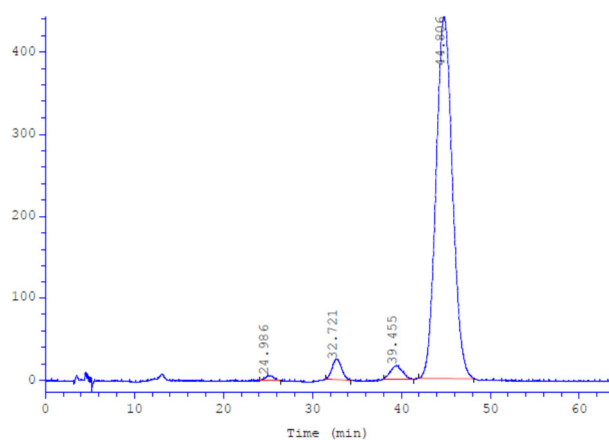
HPLC data for aldol adduct (Table 7, entry 5).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., λ = 254 nm

Eurochrom HPLC Software, Version 3.05 P5.

Tue Feb 04 13:53:04 2020

Operator: Rachwalski
 Object name: LEM-51a.f.3_Det1-A - Created: 04-02-2020, 12:46
 Path: >Test>Chromatogram



Operator: Rachwalski
 Object name: LEM-51a.f.3_Det1-A - Created: 04-02-2020, 13:50
 Path: >Test>Chromatogram

	Ret time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	24.986	24.12	26.45		-1		6.54545	7.21648	0.6493	1.079	BB+
2	32.721	31.53	34.29		-1		30.5184	24.8228	3.0274	1.160	BB+
3	39.455	37.96	41.35		-1		24.9503	17.0129	2.4751	1.509	BB+
4	44.806	41.97	48.16		0		946.05	441.188	93.8482	2.002	BB

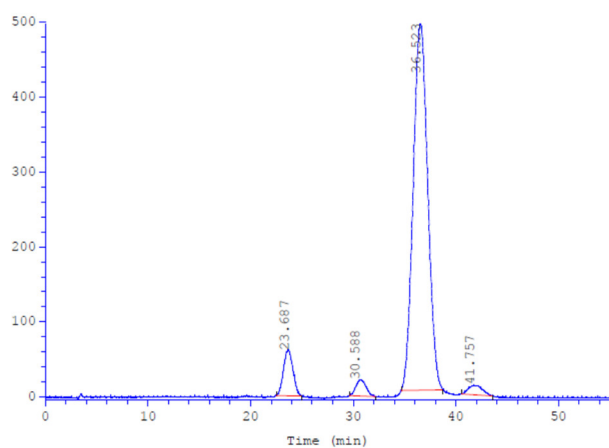
HPLC data for aldol adduct (Table 7, entry 6).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., λ = 254 nm

Eurochrom HPLC Software, Version 3.05 P5.

Wed Feb 05 13:41:34 2020

Operator: Rachwalski
 Object name: LEM-53b.f.5_Det1-A - Created: 05-02-2020, 12:44
 Path: >Test>Chromatogram



Operator: Rachwalski
 Object name: LEM-53b.f.5_Det1-A - Created: 05-02-2020, 13:39
 Path: >Test>Chromatogram

	Ret time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	23.687	22.52	24.97		0		65.2474	60.8566	7.1726	1.028	BB
2	30.588	29.68	32.19		-1		25.3993	22.0007	2.7921	1.113	BB+
3	36.523	34.69	38.76		0		799.089	488.973	87.8436	1.545	BB
4	41.757	40.58	43.58		-1		19.9364	13.4937	2.1916	1.496	BB+

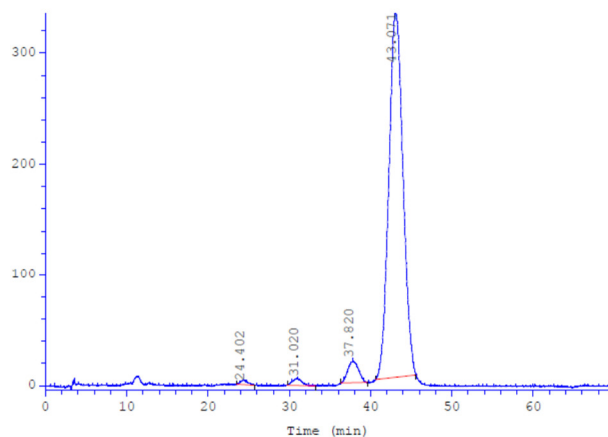
HPLC data for aldol adduct (Table 7, entry 7).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., $\lambda = 254$ nm

Eurochrom HPLC Software, Version 3.05 P5.

Tue Feb 04 15:20:23 2020

Operator: Rachwaliski
Object name: LEM-82b f. 4-7 Det1-A - Created: 04-02-2020, 14:09
Path: >Testy>Chromatography



Operator: Rachwaliski
Object name: LEM-82b f. 4-7 Det1-A - Created: 04-02-2020, 15:19
Path: >Testy>Chromatography

	Ret.time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	24.402	23.54	25.70		-1		4.34548	4.19447	0.6148	1.020	BB+
2	31.020	29.72	33.27		-1		8.35966	7.67085	1.1828	1.168	BB+
3	37.820	36.37	39.61		-1		30.327	22.3582	4.2910	1.338	BB+
4	43.071	40.65	45.59		0		663.732	328.674	93.9114	1.894	BB

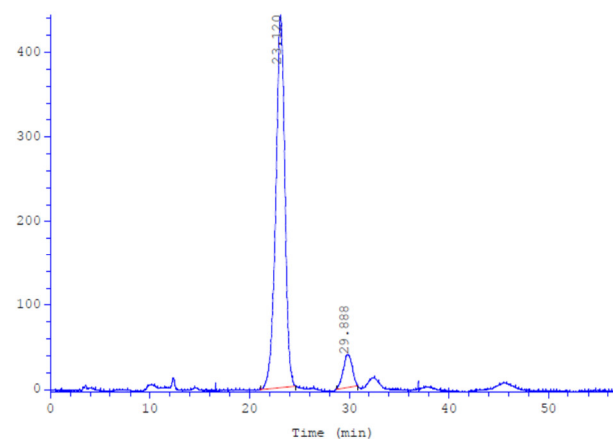
HPLC data for aldol adduct (Table 7, entry 8).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., $\lambda = 254$ nm

Eurochrom HPLC Software, Version 3.05 P5.

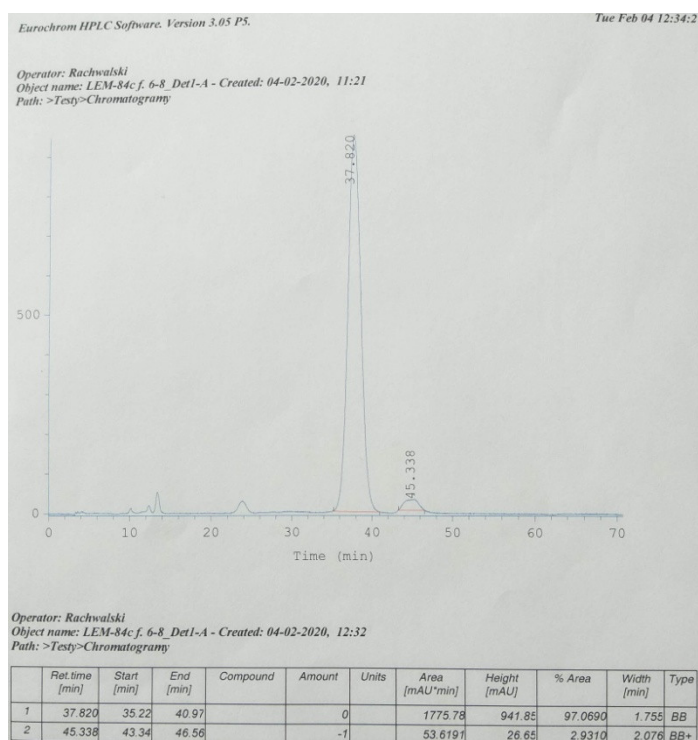
Tue Feb 04 10:14:26 2020

Operator: Rachwaliski
Object name: LEM-84a f. 3,4 Det1-A - Created: 03-02-2020, 12:27
Path: >Testy>Chromatography



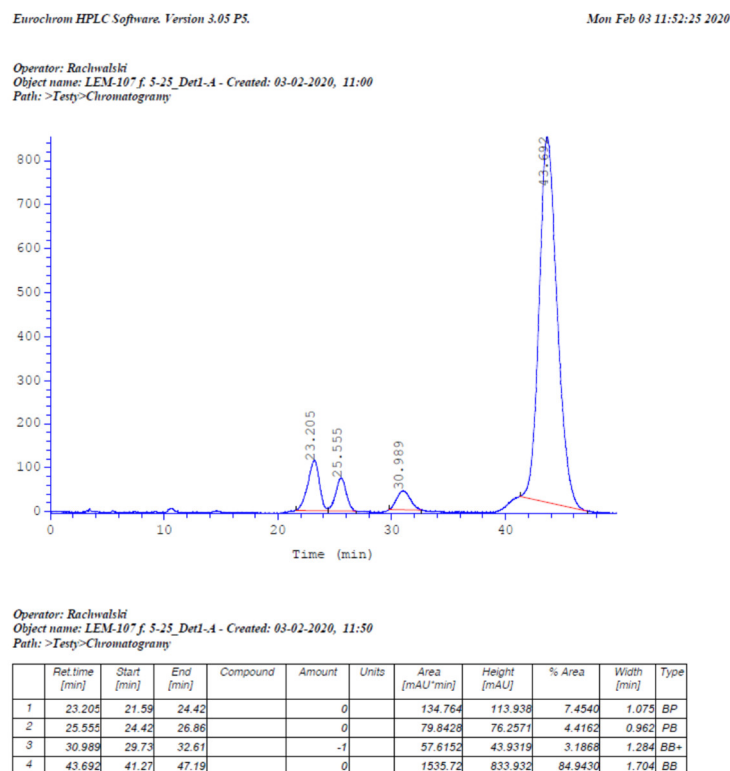
Operator: Rachwaliski
Object name: LEM-84a f. 3,4 Det1-A - Created: 03-02-2020, 13:24
Path: >Testy>Chromatography

	Ret.time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	23.120	21.09	24.64		0		466.744	441.914	92.0056	0.960	BB
2	29.888	28.74	30.84		0		40.5553	38.9532	7.9944	1.029	BB



HPLC data for aldol adduct (Table 7, entry 9).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., λ = 254 nm



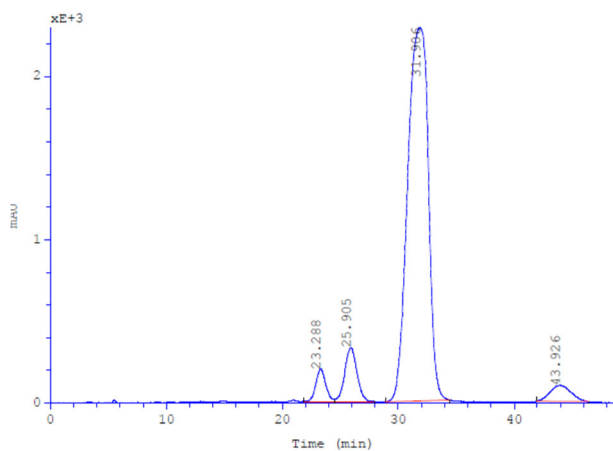
HPLC data for aldol adduct (Table 7, entry 10).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., λ = 254 nm

Eurochrom HPLC Software, Version 3.05 P5.

Fri Jan 31 14:43:18 2020

Operator: Rachwałski
 Object name: LEM-9* f. 4-15 Det1-A - Created: 31-01-2020, 13:53
 Path: >Test>Chromatogram



Operator: Rachwałski
 Object name: LEM-9* f. 4-15 Det1-A - Created: 31-01-2020, 14:42
 Path: >Test>Chromatogram

	Ret.time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	23.288	21.85	24.49		0		196.412	198.845	3.5561	0.891	BP
2	25.905	24.49	27.92		0		400.292	329.539	7.2474	1.095	PB
3	31.905	28.91	34.46		0		4721.36	2289.57	85.4818	1.984	BB
4	43.926	41.94	46.43		-1		205.171	97.8949	3.7147	2.024	BB*

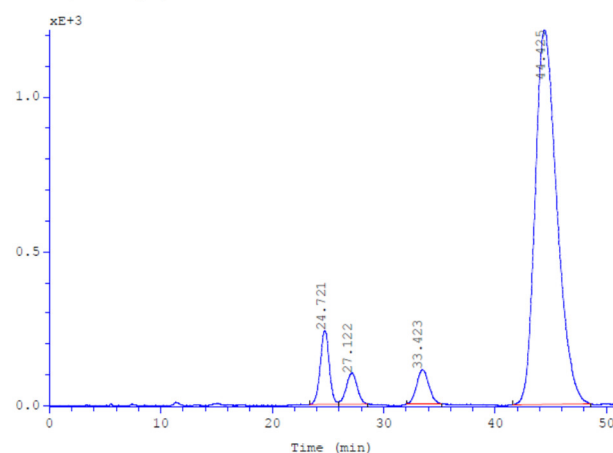
HPLC data for aldol adduct (Table 7, entry 11).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., λ = 254 nm

Eurochrom HPLC Software, Version 3.05 P5.

Fri Jan 31 13:38:01 2020

Operator: Rachwałski
 Object name: LEM-96a f. 3-10 Det1-A - Created: 31-01-2020, 12:45
 Path: >Test>Chromatogram



Operator: Rachwałski
 Object name: LEM-96a f. 3-10 Det1-A - Created: 31-01-2020, 13:36
 Path: >Test>Chromatogram

	Ret.time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	24.721	23.39	25.95		0		222.065	237.958	6.8678	0.848	BP
2	27.122	25.95	28.54		0		114.057	100.393	3.5274	1.022	PB
3	33.423	32.07	35.26		0		139.725	111.701	4.3213	1.162	BB
4	44.425	41.59	48.63		0		2757.57	1212.6	85.2835	2.093	BB

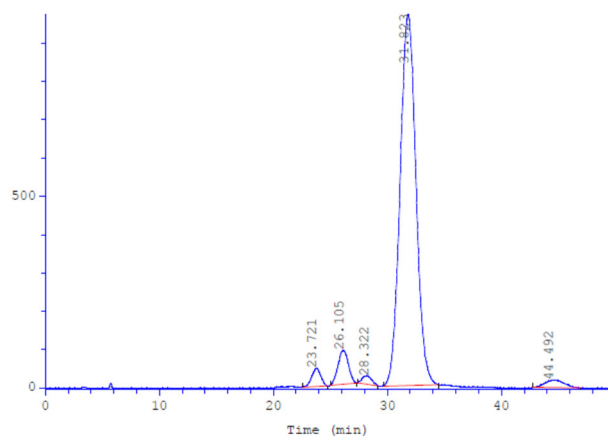
HPLC data for aldol adduct (Table 7, entry 12).

Chiral AD-H, iPrOH/*n*-hexane 15/85, flow: 1 ml/min., $\lambda = 254$ nm

Eurochrom HPLC Software, Version 3.05 P5.

Fri Jan 31 12:33:30 2020

Operator: Rachwalski
 Object name: LEM-85d f. 3,4 Det1-A - Created: 31-01-2020, 11:40
 Path: >Testy>Chromatograny



Operator: Rachwalski
 Object name: LEM-85d f. 3,4 Det1-A - Created: 31-01-2020, 12:30
 Path: >Testy>Chromatograny

	Ret time [min]	Start [min]	End [min]	Compound	Amount	Units	Area [mAU*min]	Height [mAU]	% Area	Width [min]	Type
1	23.721	22.59	24.75		0		43.1578	47.8964	2.4639	0.859	BB
2	26.105	25.00	27.32		0		93.3312	89.2104	5.3283	1.003	BB
3	28.322	27.37	29.19		-1		20.7917	22.2232	1.1870	0.973	BB+
4	31.823	29.66	34.47		0		1555.68	969.051	88.8145	1.480	BB
5	44.492	42.76	47.00		-1		38.6454	20.3298	2.2063	1.860	BB+