WACKER

Final Report

Determination of Molecular Weight Distribution of SILRES® BS 1703

CAS number:

Study number: N-0002175739

WACKER – CONFIDENTIAL

Table of Contents

Page

Cover ……1

Table of Contents 2

1 General Part 3

2 ISO Certificate 5

3 Summary of Results 6

4 Molecular Weight Distribution 7

1 General Part

1.1 Identification of Test Substance

Trade name: SILRES® BS 1703

Product number:

Synonym: SILRES® BS 68507, SLM 68507

CAS number:

1.2 Administrative Information

Company name: Wacker Chemie AG

Sponsor:
Dr. Daniel Schildbach
Wacker Chemie AG
S-C-C-T4/BGH
Johannes-Hess-Strasse 24
D-84489 Burghausen

Product safety unit (monitor):
Dr. Wolfgang Nüdling
Wacker Chemie AG
Corporate Product Safety
WL-CS-P/BGH
Johannes-Hess-Strasse 24
D-84489 Burghausen

Test facility:
Wacker Chemie AG
Corporate Analytics
WL-CS-A/BGH
Johannes-Hess-Strasse 24
D-84489 Burghausen

Test facility manager: Dr. Johannes Wiedemann

Study director: Dr. Ing. Thomas Ehmann

Date: 15 December 2016

Signature:

(Study director)

2 ISO Certificate

ZERTIFIKAT ■ CERTIFICATE ■ 認證證書 ■ СЕРТИФИКАТ ■ CERTIFICADO ■ CERTIFICAT

TÜV SÜD Management Service

CERTIFICATE

The Certification Body
of TÜV SÜD Management Service GmbH
certifies that

Wacker Chemie AG
Hanns-Seidel-Platz 4
81737 München
Germany

including the sites and scope of application
see enclosure

has established and applies
a Quality Management System.

An audit was performed, Report No. **707022378**.

Proof has been furnished that the requirements
according to

ISO 9001:2008

are fulfilled. The certificate is from **2015-03-11** until **2018-03-10**.

Certificate Registration No. **12 100 49144 TMS**

Product Compliance Management
Munich, 2015-03-16

Page 1 of 8

TÜV SÜD Management Service GmbH • Zertifizierungsstelle • Ridlerstraße 65 • 80339 München • Germany
www.tuev-sued.de/certificate-validity-check

3 Summary of Results

Physico-chemical property	Result	Method
Molecular Weight Distribution		EC test guideline A.18 and A.19 Size Exclusion Chromato-graphy (SEC) according to DIN 55672-1

4 Molecular Weight Distribution

4.1 General

Test guideline:	EC test guideline A.18 and A.19
Method:	Size Exclusion Chromatography (SEC) according to DIN 55672-1

4.2 Materials/Parameters/Conditions

Balance:	Toledo AX 304; (Mettler Toledo)
Pump:	Isocratic pump Agilent 1200 (Agilent Technologies)
Flow rate:	0.3 mL/min
Injection system:	Autosampler Agilent 1200 (Agilent Technologies)
Injection volume:	20 µL
Eluent:	THF > 99.9%, p.A., (Merck)
Eluent reservoir:	Brown glass bottle, 5 L; pre-cleaned (Duran)
Degasser:	4 Channel degasser (PSS)

Column:

Stationary phase: Polystyrene-divinylbenzene

Manufacturer: Agilent Technologies

Supplier: Agilent Technologies

Arrangement	Description / Particle size [µm] / Pore size [Å]	Molecular weight range [g/mol]	Length [mm]	Diameter [mm]
1	Guard column / 5 / MiniMIX-C	–	50	4.6
2	PLgel / 5 / 10,000	1,000 – 600,000	250	4.6
3	PLgel / 5 / 500	100 – 30,000	250	4.6
4	PLgel / 5 / 100	100 – 4,000	250	4.6

Column temperature: Oven temperature 45°C (Phenomenex)

Detector: Refractive index
Measuring principle: Deflection
Type: Agilent 1200 (Agilent Technologies)
Cell volume: 8 µL
Temperature: 40 °C

The differential refractometer trace is taken as concentration signal.

Operating system: Windows 7 Enterprise SP1

Software: PSS WinGPC UniChrom, Build 5377,
(Polymer Standard Service, Mainz, Germany)

4.3 System Calibration

Blank run 1: Injection of the eluent THF (Appendix 1)

Blank run 2: Injection of the eluent THF + ISTD toluene (Appendix 2)

Remark: Please note the different scaling of the signal axis.

Calibration substances: EasyCal PS-2 A and B (Agilent) and single standard (Appendices 3a – 3c)
M_p values in [g/mol] used for calibration as provided by the manufacturer.

Key code	M_p [g/mol]	Batch number	Elution volume [ml]
PS-2A	483,400	0006208548	5.09
	113,300		5.53
	19,540		6.19
	4,830		6.83
	1,390		7.52
PS-2B	224,900	0006208548	5.32
	51,150		5.82
	9,970		6.48
	2,780		7.11
	580		7.96
Single Standard*	523,000	20139-22	5.06

Concentration: 0.4 g/L (EasiCal, pre-prepared Polystyrene Calibrants,
provided by the manufacturer)
*0.3 g/L from the same manufacturer

Injection Volume: 20 µL

Remarks: Data processing (calibration and sample run) based on retention times.
Toluene was used as internal flow marker maintaining a reproducible flow rate (retention time fixed at 9.55 mL elution volume).

Calibration curve fitting: Polynomial fit PSS 3rd order
Regression coefficient: R = 0.999963
(Appendix 4)

4.4 Evaluation of the SEC System

PSS system test in accordance with ISO 13885-1 (Appendix 5b):

Test substance:	ethyl benzene
Concentration:	2.0 mg/mL in THF
Injection volume:	20 µL
Number of theoretical plates n:	51,163 $^{1}/_{m}$
Asymmetry factor:	0.871
Resolution R:	5.466
Separation efficiency:	6.098 cm

4.5 Sample Preparation

Three independent samples were prepared.

About 17-22 mg of the test substance were dissolved in the solvent THF (c = about 5 mg/mL).

The samples were completely soluble in the eluent.

4.6 Results

All calculations were done on the basis of the elution volume.

Item	[redacted]
Weight average Mw [Dalton]:	
Number average Mn [g/mol]:	
Area % > 1000:	
Area % < 1000:	
Area % < 500:	

* The results were referred to the molar masses of polystyrene standards (polystyrene mass-equivalents).

Appendices 7a – 7c: The ranges of molar masses used for quantification were marked in the corresponding chromatograms.

Experimental start date: **14 December 2016**

Experimental termination date: **15 December 2017**

Appendices:

1	Injection of the eluent THF
2	Injection of the eluent THF and toluene as ISTD
3a, 3b, 3c	Elution profile of polystyrene standards (PS-2 A, PS-2 B and single standard)
4	Calibration curve
5a, 5b	Elution profile of ethyl benzene and system test
6a, 6b	Chromatogram of polystyrene standard Mw 88,000 and corresponding report of System Qualification Test
7a, 7b, 7c	Elution profile of test samples (three independent runs)
8a, 8b, 8c	Processed test samples (three independent runs) (integration ranges with overlaid calibration curve explaining the integration range settings / molecular mass distribution / results)
9a, 9b, 9c	Slice tables of molar mass distribution of the diagram from appendices 8a - 8c

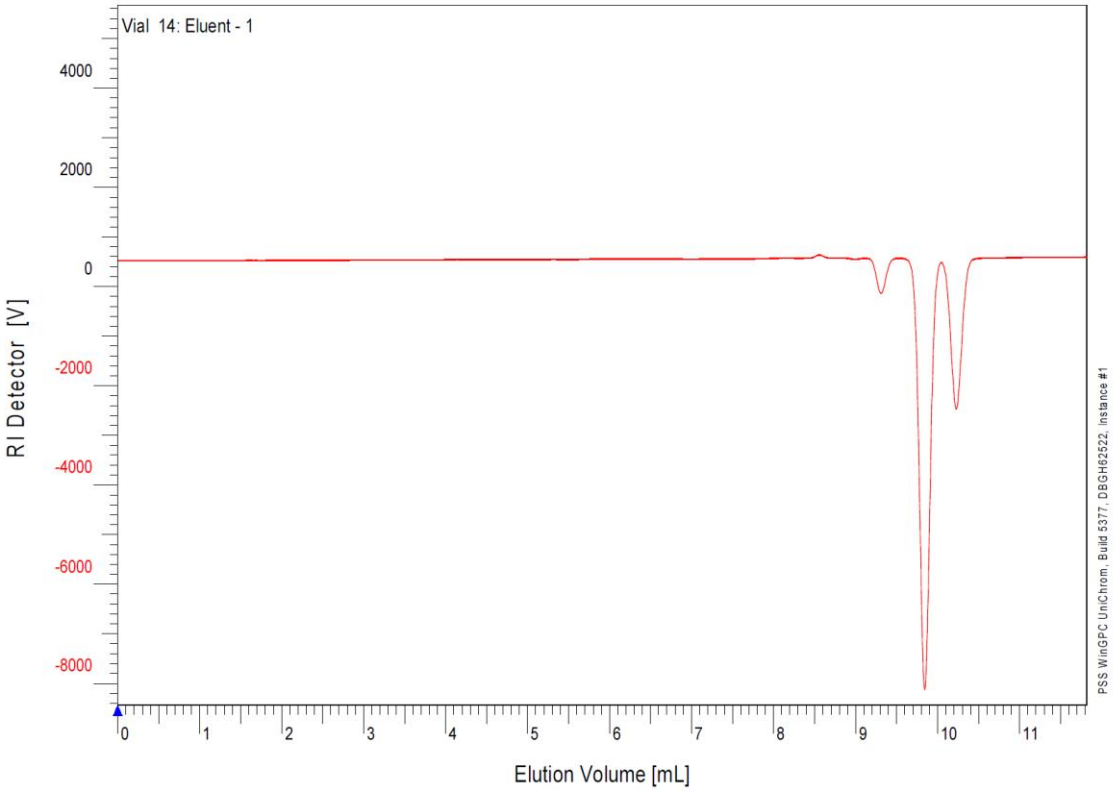
Appendix 1

WACKER
Liquid Chromatography
Corporate Analytics

Sample	**Eluent**
Eluent	THF
Column	Mini_10E4+500+100
Column temperature	45°C
Flow rate	0.30 mL/min
Pressure	40.1 bar
Detector 1	RI-Detector
Calibration	conventional Polynomial 3 04_Mini_10e4+500+100_151216_PS corrected by internal standard

Customer	**Fr. Genzinger**
Operator	Gohla A.
Date of injection	14-Dez-2016 20:35:24
Injection volume	20 µL
Concentration	1.00 mg/mL
Sample filtrated	**no**

Elugram (raw)



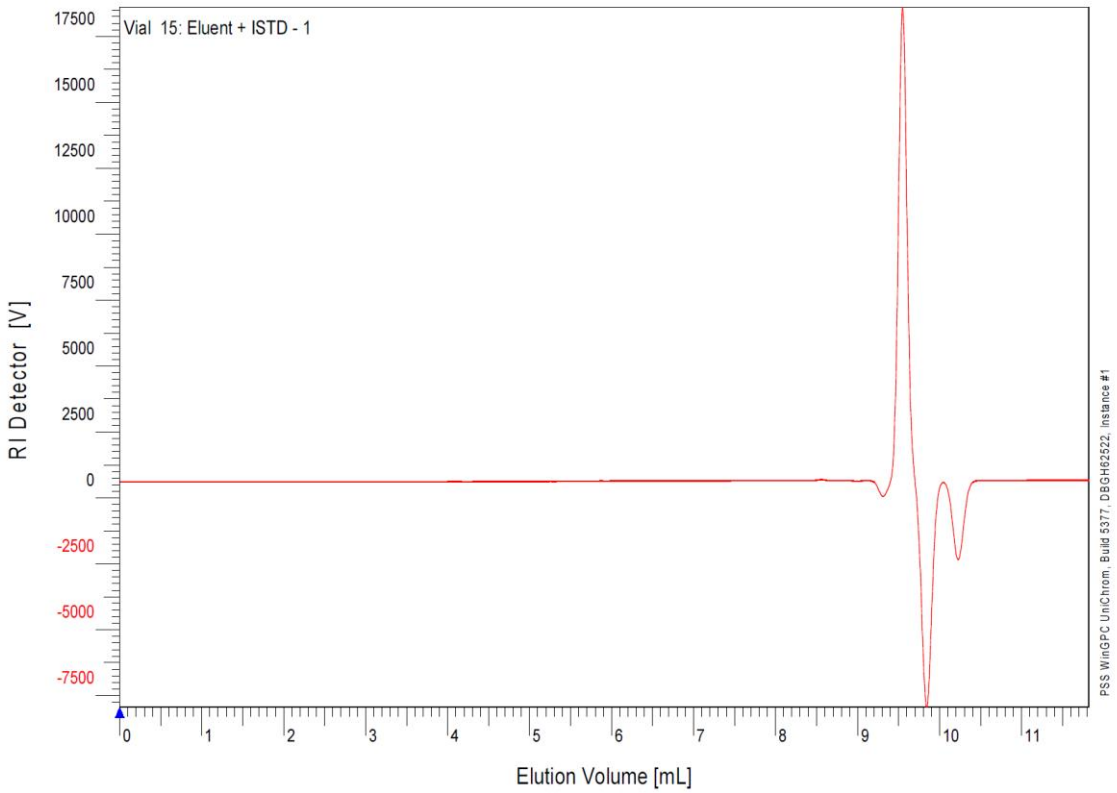
Appendix 2

WACKER
Liquid Chromatography
Corporate Analytics

Sample	**Eluent + ISTD**
Eluent	THF
Column	Mini_10E4+500+100
Column temperature	45°C
Flow rate	0.30 mL/min
Pressure	40.2 bar
Detector 1	RI-Detector
Calibration	conventional Polynomial 3 04_Mini_10e4+500+100_151216_PS corrected by internal standard

Customer	**Fr. Genzinger**
Operator	Gohla A.
Date of injection	14-Dez-2016 21:21:47
Injection volume	20 µL
Concentration	1.00 mg/mL
Sample filtrated	**no**

Elugram (raw)

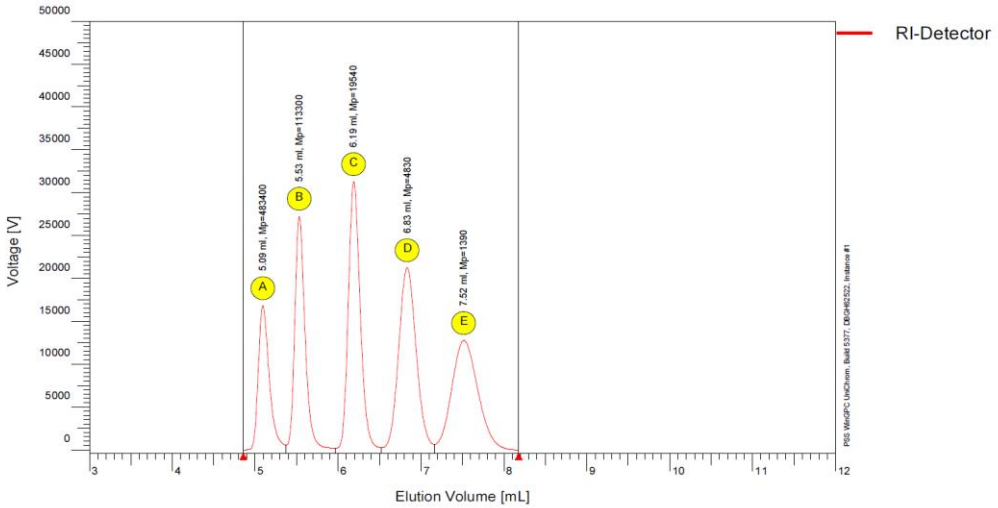


Appendix 3a

WACKER
Liquid Chromatography
Corporate Analytics

Sample	**PS 2 A 141216**	**Customer**	**Fr. Genzinger**
Eluent	THF		
Column	Mini_10E4+500+100		
Column temperature	45°C	Operator	Gohla A.
Flow rate	0.30 mL/min	Date of injection	14-Dez-2016 16:43:38
Pressure	40.2 bar	Injection volume	20 µL
Detector 1	RI-Detector	Concentration	0.40 mg/mL
Calibration	conventional	**Sample filtrated**	**no**
	none or not available		
	04_Mini_10e4+500+100_151216_PS	Baseline from	4.86 mL to 8.18 mL
	corrected by internal standard	Integration from	4.86 mL to 8.18 mL

Elugram (processed)



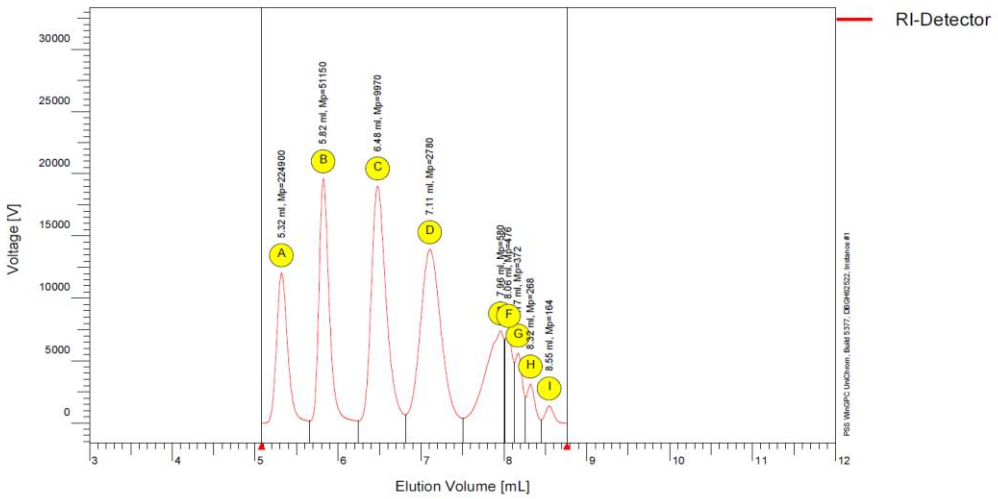
Peak	Component	VP [mL]	Mp [g/mol]	F [V·mL]	F [%]
A	Mp=483400	5.09	10	2 595	12.00
B	Mp=113300	5.53	10	3 935	18.19
C	Mp=19540	6.19	10	5 191	24.00
D	Mp=4830	6.83	10	5 230	24.18
E	Mp=1390	7.52	10	4 682	21.64

Appendix 3b

WACKER
Liquid Chromatography
Corporate Analytics

Sample	**PS 2 B 141216**	**Customer**	**Fr. Genzinger**
Eluent	THF		
Column	Mini_10E4+500+100		
Column temperature	45°C	Operator	Gohla A.
Flow rate	0.30 mL/min	Date of injection	14-Dez-2016 17:30:02
Pressure	40.2 bar	Injection volume	20 µL
Detector 1	RI-Detector	Concentration	0.40 mg/mL
Calibration	conventional	**Sample filtrated**	**no**
	none or not available		
	04_Mini_10e4+500+100_151216_PS	Baseline from	5.08 mL to 8.76 mL
	corrected by internal standard	Integration from	5.08 mL to 8.76 mL

Elugram (processed)



Peak	Component	VP [mL]	Mp [g/mol]	F [V·mL]	F [%]
A	Mp=224900	5.32	10	1 865	11.33
B	Mp=51150	5.82	10	2 794	16.97
C	Mp=9970	6.48	10	3 989	24.23
D	Mp=2780	7.11	10	4 046	24.57
E	Mp=580	7.96	10	1 906	11.58
F	Mp=476	8.06	10	768	4.67
G	Mp=372	8.17	10	592	3.60
H	Mp=268	8.32	10	345	2.10
I	Mp=164	8.55	10	159	0.97

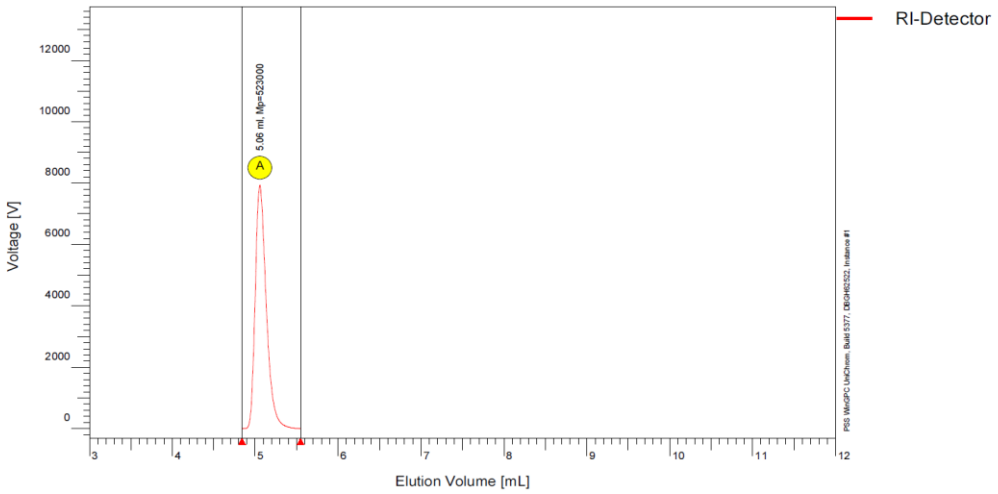
Appendix 3c

WACKER
Liquid Chromatography
Corporate Analytics

Sample	**Mw:523000**
Eluent	THF
Column	Mini_10E4+500+100
Column temperature	45°C
Flow rate	0.30 mL/min
Pressure	40.3 bar
Detector 1	RI-Detector
Calibration	conventional none or not available 04_Mini_10e4+500+100_151216_PS corrected by internal standard

Customer	**Fr. Genzinger**
Operator	Gohla A.
Date of injection	14-Dez-2016 18:16:24
Injection volume	20 µL
Concentration	0.30 mg/mL
Sample filtrated	**no**
Baseline from	4.84 mL to 5.55 mL
Integration from	4.84 mL to 5.55 mL

Elugram (processed)



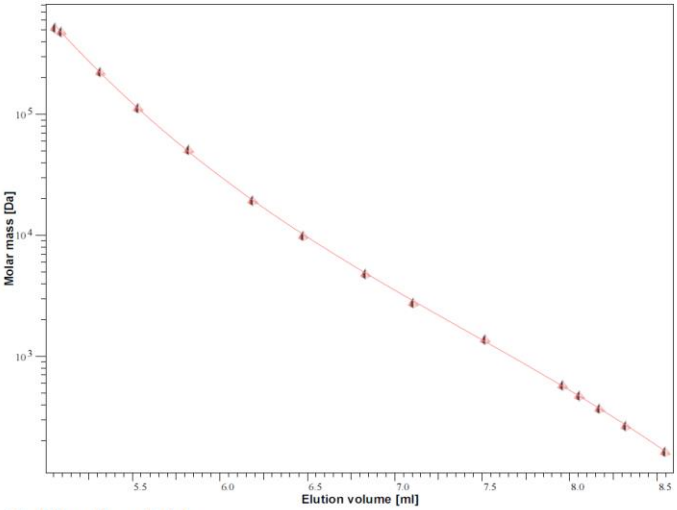
Peak	Component	VP [mL]	Mp [g/mol]	F [V·mL]	F [%]
A	Mp=523000	5.06	10	1 228	100.00

Appendix 4

WACKER
Liquid Chromatography
Corporate Analytics

WINGPC Calibration Report

Datei **N:\CD-WL\CS\Analytics\LC\Calib\04_THF\Mini\04_Mini_10e4+500+100_151216_PS**



Calibration parameter

Calibration prepared by	Gohla A.
Date	15-Dez-2016
Column	Mini_10E4+500+100

Quality of fitting

Type of fitting	Polynomial 3
R	0.999963
Highest calibration point	543 034 g/mol at 5.06 mL
Lowest calibration point	165 g/mol at 8.55 mL

internal Standard

used substance	Toluene in THF
elution at	9.6 mL

Calibration table

Elution volume [mL]	Molar mass [g/mol]	Sample name	Slope	Deviation [%]
5.06	523 000	Mw:523000 - 1	-1.59	3.83
5.09	483 400	PS 2 A 141216 - 1	-1.56	-1.15
5.32	224 900	PS 2 B 141216 - 1	-1.43	-1.56
5.53	113 300	PS 2 A 141216 - 1	-1.31	-1.05
5.82	51 150	PS 2 B 141216 - 1	-1.17	-3.59
6.19	19 540	PS 2 A 141216 - 1	-1.02	0.47
6.48	9 970	PS 2 B 141216 - 1	-0.94	2.70
6.83	4 830	PS 2 A 141216 - 1	-0.86	1.70
7.11	2 780	PS 2 B 141216 - 1	-0.83	3.79
7.52	1 390	PS 2 A 141216 - 1	-0.82	-4.21
7.96	580	PS 2 B 141216 - 1	-0.86	-1.95
8.06	476	PS 2 B 141216 - 1	-0.87	-1.10
8.17	372	PS 2 B 141216 - 1	-0.89	0.17
8.32	268	PS 2 B 141216 - 1	-0.93	1.50
8.55	164	PS 2 B 141216 - 1	-0.99	0.88
			Average of deviation	0.03
			Variation coefficient of deviation	5.53

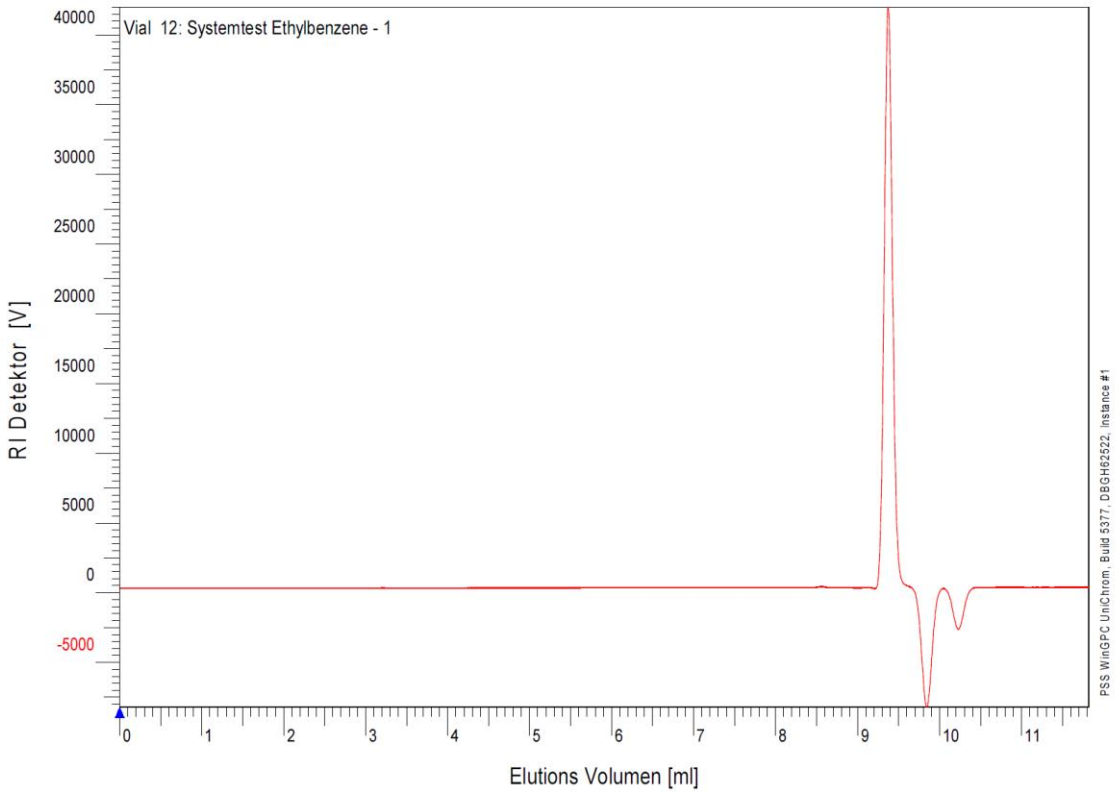
Appendix 5a

WACKER
Liquid Chromatography
Corporate Analytics

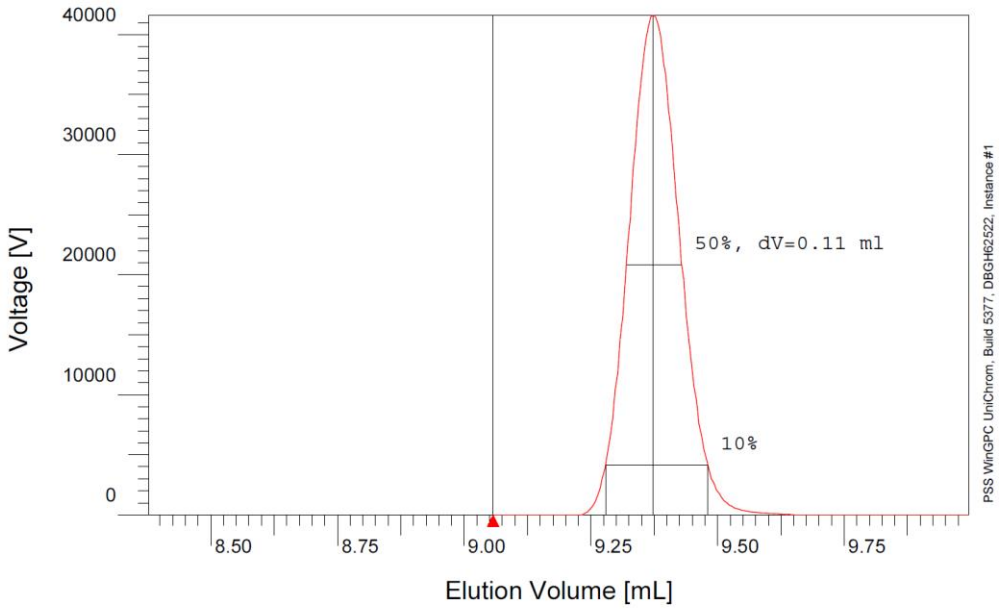
Sample	**Systemtest Ethylbenzene**
Eluent	THF
Column	Mini_10E4+500+100
Column temperature	45°C
Flow rate	0.30 mL/min
Pressure	40.3 bar
Detector 1	RI-Detector
Calibration	conventional Polynomial 3 04_Mini_10e4+500+100_151216_PS corrected by internal standard

Customer	**Fr. Genzinger**
Operator	Gohla A.
Date of injection	14-Dez-2016 19:02:44
Injection volume	20 µL
Concentration	2.00 mg/mL
Sample filtrated	**no**

Elugram (raw)



Appendix 5b



Sample :	Vial 12: Systemtest Ethylbenzene - 1	**Method :**	N:\CD-WL\CS\Analytics\LC\Methoden\04_THF\rechts
Baseline from :	9.057 ml	**to :**	10.446 ml
Integration from :	9.057 ml	**to :**	10.446 ml
Calibration File :	04_Mini_10e4+500+100_151216_PS.CAL	**Eluent :**	THF
MHK - A (Cal.):	0.000E+0	**MHK - K (Cal.):**	1.000E+0 ml/g
Int.stand.-cal.:	9.550 ml	**Int.stand.-sam.:**	------- ml
Pump :	Agilent	**Flowrate :**	0.300 ml/min
Concentration :	2.000 g/l	**Inject volume :**	20.000 ul
Column 1 :	Mini_10E4+500+100	**Temperature :**	45.000 °C
Length :	80.000 cm	**Diameter :**	0.460 cm
Detector 1 :	I4: RID 1, RI Signal	**Delay volume :**	0.124 ml
Detector 2 :	I4: IsoPump 1, Pressure	**Delay volume :**	0.000 ml
Operator :	Gohla A.	**Acquisition interval**	1.000 sec

PSS Systemtest nach ISO 13885-1/DIN 55672-1 :

	Results	Requirements	Unit
Vol. Peakmax.:	9.373		ml
Plate Count :	51163	> 20000	1/m
Asymmetry :	0.871	> 0.85 < 1.15	-
Resolution :	5.466	> 2.5	-
Efficiency :	6.098	> 6	cm
signal drift :	2.028E+2	< 5 % Imax	V/h
signal noise :	9.801E+0	-	V
signal wander :	3.881E+1	-	V
signal/noise :	8496.708	> 100	-
pump pressure fluct. :	5.600E-2	-	bar
column temp fluct. :	0.000E+0	-	°C

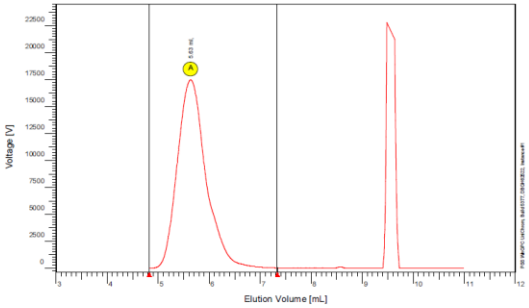
Appendix 6a

WACKER
Liquid Chromatography
Corporate Analytics

Sample	**Mw:88000**
Eluent	THF
Column	Mini_10E4+500+100
Column temperature	45°C
Flow rate	0.30 mL/min
Pressure	40.4 bar
Detector 1	RI-Detector
Calibration	conventional Polynomial 3 04_Mini_10e4+500+100_151216_PS corrected by internal standard

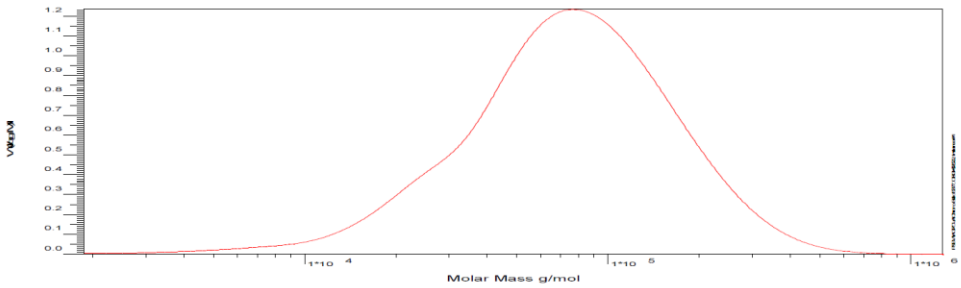
Customer	**Fr. Genzinger**
Operator	Gohla A.
Date of injection	14-Dez-2016 19:49:04
Injection volume	20 µL
Concentration	3.00 mg/mL
Sample filtrated	**no**
Baseline from	4.82 mL to 10.98 mL
Integration from	4.82 mL to 7.34 mL

Chromatogram



Peak	Component	VP [mL]	Mp [g/mol]	Area [µV·mL]	Area [%]
A		5.63	83 458	11 349	100.00

Molar mass distribution



SEC results *(measurement accuracy at full hundred molecular weight)*

Detector	Mw [g/mol]	Mn [g/mol]	Polydispersity
RI-Detector	97 295	50 124	1.94

Appendix 6b

Complete inspection of testing equipment
Anlage 04_THF_RI

Columns: Mini - 10,000Å, 500Å, 100Å

For inspecting the testing equipment a broad distributed polystyrene standard of nominal molar weight of Mw = 88,000 (PSS Mainz, Germany, Lot No.psbr88k) was used.

The difference between Mw specified by the vendor and our target value was caused by the different set of columns used as well as the different calibration standards.

Date	Mw target		Operator
24.08.2009	**102,000**		*A. Pastl*
14.12.2016	97,295	actual	
	-4.6	deviation %	*A. Pastl*

Appendix 7a

WACKER

[redacted]

Appendix 7b

[redacted]

Appendix 7c

WACKER
Liquid Chromatography
Corporate Analytics

Sample **SILRES BS 1703 (III)**

[redacted]

Appendix 8a

[redacted]

Appendix 8b

WACKER
Liquid Chromatography
Corporate Analytics

Sample **SILRES BS 1703 (II)**

[redacted]

Appendix 8c

WACKER
Liquid Chromatography
Corporate Analytics

Sample **SILRES BS 1703 (III)**

[redacted]

Appendix 9a – Slice table

[redacted]

Appendix 9b – Slice table

[redacted]

Appendix 9c – Slice table

[redacted]