

Test Report

Molecular weight measurement of Polymer by GPC (VISCOPLEX® 12/10301)

manufactured by
Evonik Resource Efficiency GmbH
Oil Additives

Date: 2018-04-26

Tested by
Evonik Technology & Infrastructure GmbH
Product Line Analytik

1. Testing Laboratory

Adress:

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Analyzer: Jana Ernst

Signature: 

Date: 2018-04-26

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2. Test substance:

VISCOPLEX®12/10301 supplied by Evonik Resource Efficiency GmbH; Oil Additives



CAS No: 2181814-52-6

3. Analytical conditions:

(1) Eluent: Tetrahydrofuran (eluent is continuously distilled and circulated by pump)

(2) GPC column:

5 SDV Columns 8 x 300 mm resp. 8 x 50 mm (company PSS at Mainz)

1 Solvent-Peak Separation Column 8 x 100 mm (company Shodex)

No	Type		Dimension		Serial no.
Precolumn	SDV	10 μ	8 x 50	mm	7040721
1	SDV LinL	10 μ	8 x 300	mm	2062903
2	SDV LinL	10 μ	8 x 300	mm	6082302
3	SDV 100Å	10 μ	8 x 300	mm	0071401
4	SDV 100Å	10 μ	8 x 300	mm	0070508
5	KF-800D		8 x 100	mm	2007012

(3) Detector: RI: temperature 35 °C
(UV: Wavelength 239 nm, not used)

(4) Column temperature: 35 °C

(5) Flow rate: 1 ml/min

(6) Injection volume: 100 μ l

(7) Sample concentration: 2 g/l

(8) Data system: PSS WinGPC-Software

4. GPC calibration

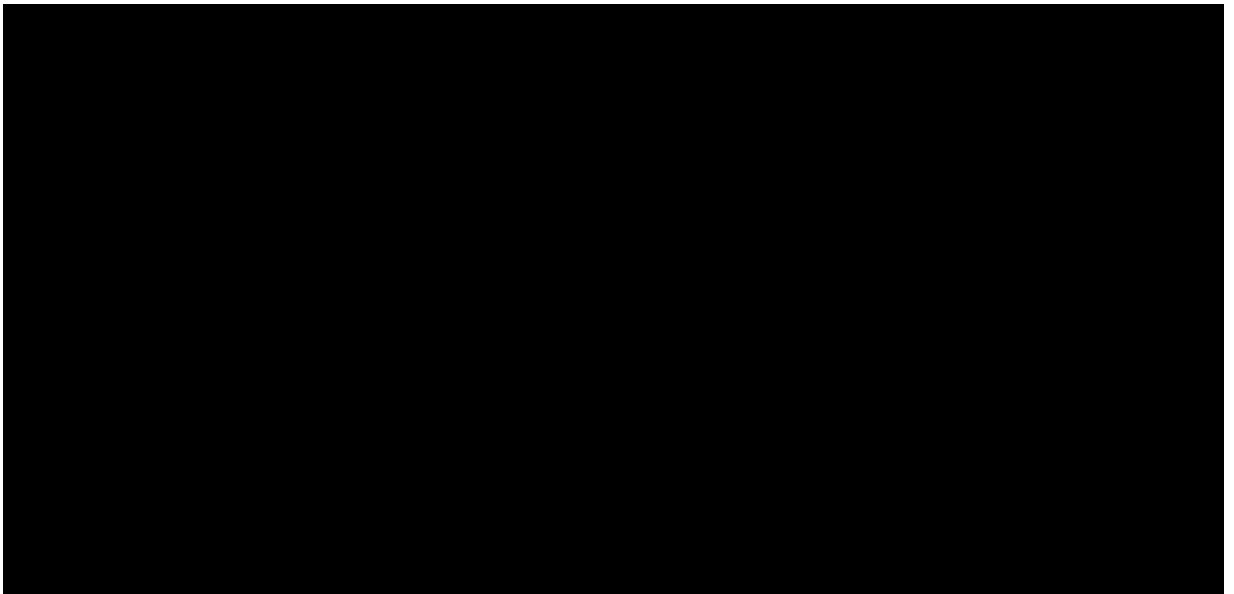
(1) Standard sample: PMMA, PSS (Mainz) or Polymer Laboratories

(2) Injection volume: 100 μ l

(3) Standard concentration: 1 g/l (for $M_w > 10^6$: 0.5 g/l, $M_w > 2 \cdot 10^6$: 0.25 g/l)

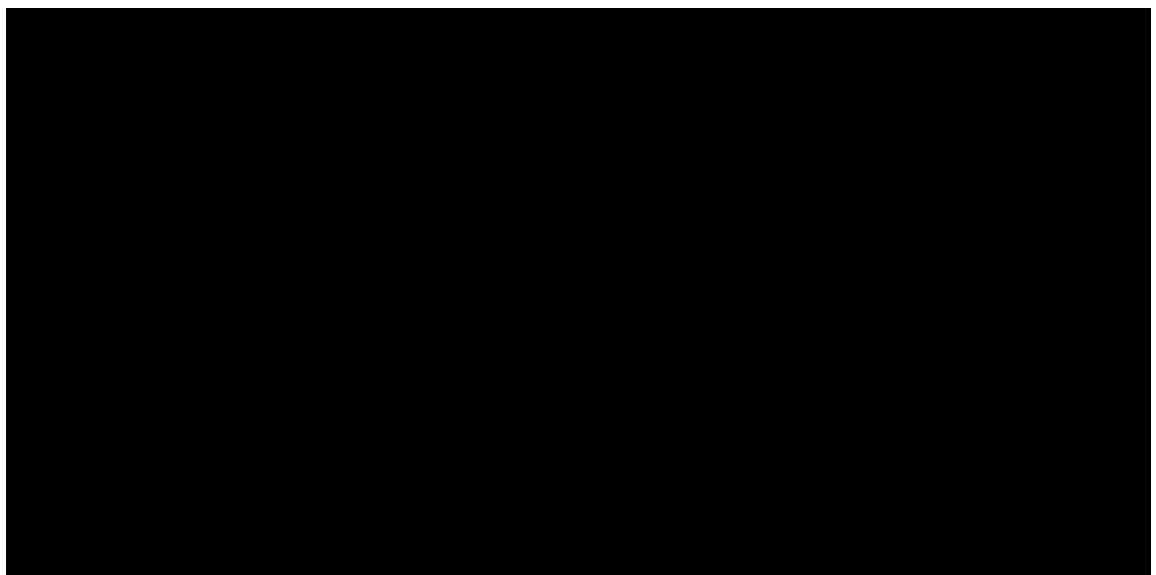
(4) Test date for calibration: 2017-12-01

(5) GPC Chromatogram of Standard Sample:



	No	Sample	Comment
—	1	RI 1100er HP, PMMA Mischung I (2 8 12a 15)	
—	2	RI 1100er HP, PMMA Mischung II (4 10 13 16)	
—	3	RI 1100er HP, PMMA Mischung III (6 12 14 17)	
—	4	RI 1100er HP, PMMA Mischung 1-7	
—	5	RI 1100er HP, PMMA Mischung 3-9	
—	6	RI 1100er HP, PMMA Mischung 5-11	
—	7	RI 1100er HP, PMMA Mischung 21-23	
—	8	RI 1100er HP, PMMA Mischung 22	
—	9	RI 1100er HP, PMMA18 mmg20113 660	
—	10	RI 1100er HP, PMMA1 PS F-850 TS-31 11.200.000 0,25 g/l	
—	11	RI 1100er HP, PMMA2 5911/7/1 4.960.000	
—	12	RI 1100er HP, PMMA3 mm7086 3.250.000	

Figure 1a. GPC chromatograms of PAMA standard samples



Elutions Volumen [-]
Sample: PMMA18 mmg20113 660
Material: PMMA/THF/30°C at 633nm

Figure 1b. Raw chromatogram of the lowest standard sample including system peaks and Internal Standard (The blue-marked line is the start marker of the next inject.)

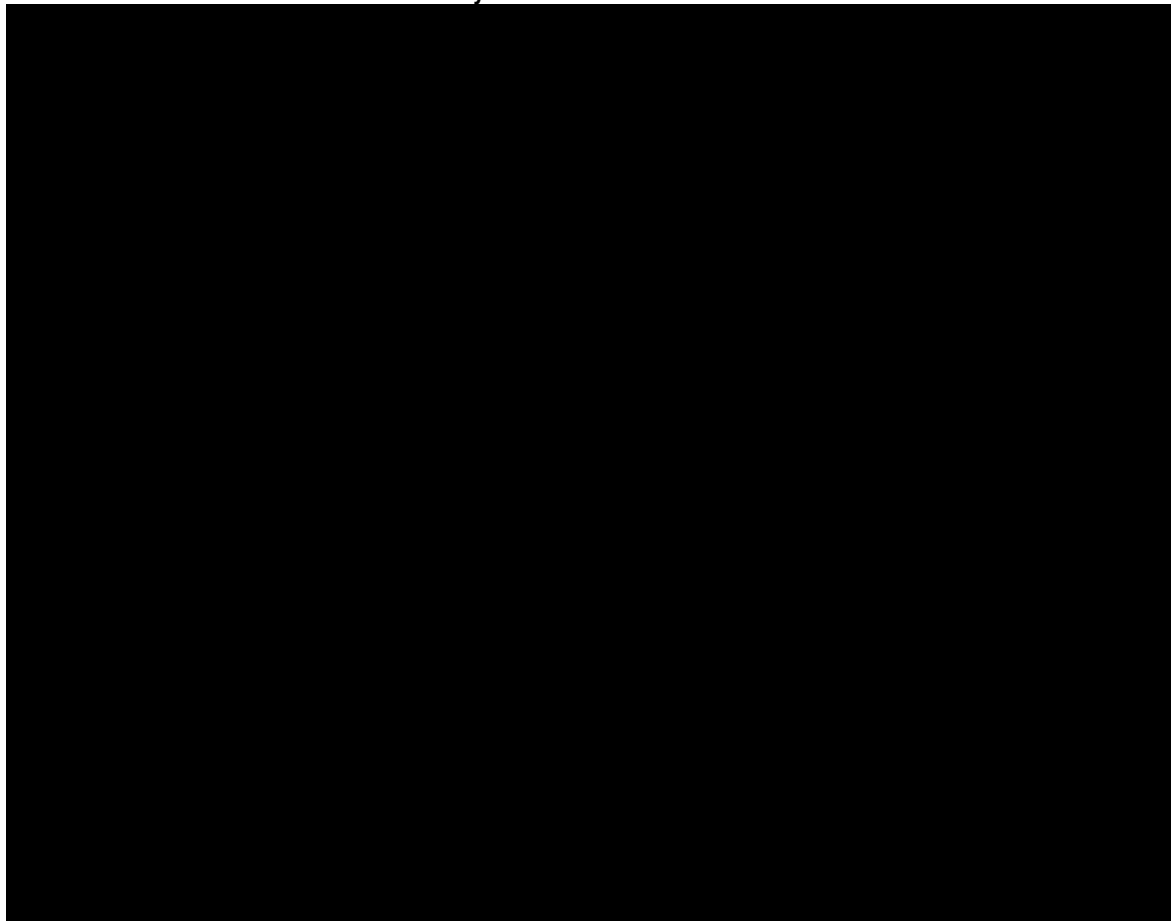
(6) Calibration plot:



WINGPC Report Calibration

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File: T:\GPC\Calib\Sys HP\173HPPMMA.CAL



Calibration Parameters:

Operator: Se/ErJ (valid from 2017-12-01)
Columns: Satz HP: VS/LinL/LinL/100/100/SSC

Mark-Houwink Coefficients:

$a = 0,688$ $K = 0,01298$

Fit Characteristics:

Fit Type: PSS Poly 5

Internal Standard:

Name: o-Dichlorbenzol 0,2 μ l / 100 μ l (IP)

Date of Printing: 2018-04-26



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Figure 2. Calibration plot

Table 1. Calibration Table



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File: D:\AP\GPC\Calib\Sys HP\173HPPMMA.CAL
Polymer Standards: Poly(methyl methacrylate)
Calibration Table:

The calibration table content is completely redacted with a large black rectangle.

Date of Printing: 2018-04-09



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WINGPC Report Calibration

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File: D:\AP\GPC\Calib\Sys HP\173HPPMMA.CAL

Polymer Standards: [REDACTED]

Calibration Table:

[REDACTED]					

5. Test method

The molecular weight results (number-average and weight-average molecular weight and distribution) were measured by GPC (Gel permeation chromatography) in dependence on DIN 55672-1

6. Test result

Table 2. Molecular weights, polydispersity, % content below 1000 g/mol and % content below 500 g/mol of VISCOPLEX[®] 12/10301

Mn [g/mol]	Mw [g/mol]	Mp [g/mol]	Mw/Mn	content below 500 g/mol [%]	content below 1000 g/mol [%]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]

Mn: number-average molecular weight

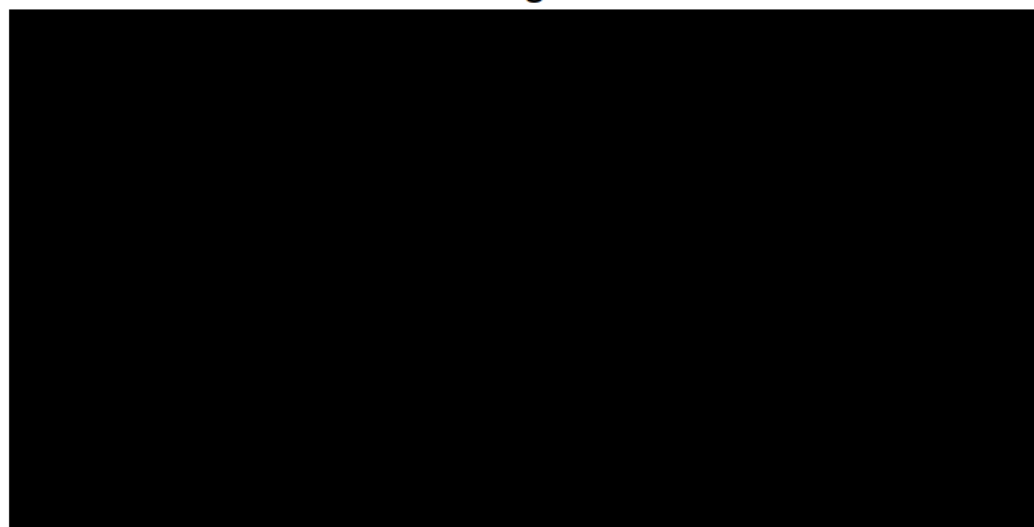
Mw: weight-average molecular weight

Mp: Peak average molecular weight

Mw/Mn: polydispersity

Sample test date: 16. January 2018 (see Date line on Fig. 3)

Molecular Weight Distribution



Sample: 2018_45 [CA-IM-OA-PRD] 011181851850

Material: Viscoplex 12/10301 100%-ig

Weighted Sample / 20ml THF:

Project: D:\AP\GPC\Daten WinGPC8\HP01-2018.LDX

Operator: Ernst_J

Date: 16.01.2018 13:17:25

Baseline from:

Internal Standard: o-Dichlorbenzol 0,2µl / 100µl (IP) at 49,193 ml

Method

System: System HP

Flow Rate: 1,00 ml / min

Eluent: THF

Inject Volume: 100 µl

Column 1: HP VS 01.12.2017 SN7040722

Column 2: HP S1+S2 LinL 2062903 + 6082302

Column 3: HP S3+S4 100A 71401 + 70508

Column 4: HP S5 SSC 01.01.2000 2007012

Detector 1:

Detector 2:

Druck HP Delay Volume: 0,000 ml

Calibration

Polymer Standards: 173HPPMMA.CAL Poly(methyl methacrylate)

Internal Standard: 49,100 ml

Detector	Mn g/mol	Mw g/mol	D	Mp g/mol	Area ml*V	Mass Fraction below	
						500 g/mol	1000 g/mol

Date of Printing: 2018-04-10



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Figure 3. GPC Chromatogram of VISCOPLEX® 12/10301



Sample: 2018_45 [CA-IM-OA-PRD] 011181851850
Material: Viscoplex 12/10301 100%-ig

Figure 4. Raw chromatogram of VISCOPLEX® 12/10301 including system peaks and Internal Standard (The blue-marked line is the start marker of the next inject.)

Table 3. GPC slice table

[illegible]

Elution Volume* [ml]	Molar Mass [g/mol]	RI-Detector signal (height) [V]	Integral [%]	100 - Integral [%]

[illegible]

Elution Volume* [ml]	Molar Mass [g/mol]	RI-Detector signal (height) [V]	Integral [%]	100 - Integral [%]

[illegible]

similar, the pairs of elution volume / molar mass correlate not exactly. Therefore we used the first table (see above) and we added the column “100-Integral [%]”, which is manually calculated on base of Integral [%].