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:
   Evonik Technology & Infrastructure GmbH
   Kirschenallee
   64293 Darmstadt
   Germany

   Person in Charge: Dr. Anja Niklaus

   [redacted]

   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

   [redacted]

   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 x100 mm (company Shodex)

No	Type		Dimension		Serial no.
Precolumn	SDV	10µ	8 x 50	mm	7040721
1	SDV LinL	10µ	8 x 300	mm	2062903
2	SDV LinL	10µ	8 x 300	mm	6082302
3	SDV 100Å	10µ	8 x 300	mm	0071401
4	SDV 100Å	10µ	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 µl

(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 µl

(3) Standard concentration: 1 g/l (for Mw > 10^6: 0.5 g/l, Mw > $2*10^6$: 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

Page: 1

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

[redacted]

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 0,2µl / 100µl (IP)

Date of Printing: 2018-04-26

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Figure 2. Calibration plot

Table 1. Calibration Table

WINGPC Report Calibration

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

Calibration Table:

[redacted]

Date of Printing: 2018-04-09

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WINGPC Report Calibration

Page: 3

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

[redacted]

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

Material: **Viscoplex 12/10301 100%-ig**

Weighted Sample / 20ml THF: [redacted] RI HP

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

Operator: Ernst_J

Date: 16.01.2018 13:17:25

Baseline from: [redacted]

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: [redacted]

Detector 2: [redacted]

[redacted] Druck HP Delay Volume: 0,000 ml

Calibration 173HPPMMA.CAL

Polymer Standards: Poly(methyl methacrylate)

Internal Standard: 49,100 ml

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

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

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

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

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[redacted]				
[redacted]				

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

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[redacted]				

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

* The used GPC software produce two slice tables, one with elution volume, molar mass, detector signal (height) and integral distribution, the other with molar mass, differential distribution (area), and integral distribution. The scaling of both tables is different. The molar mass scaling is logarithmical and the elution table is normal. Even though the number of data points in both tables is

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 [%].