Recycling Compatibility of Polyamide Copolymer PA6/IPDI as layer in PE based packaging films

Test Report 2776-2024-004286 | 17/07/2024 (rev. 07/08/2024) | Project No. 2776-001-2024

Customer:

Envalior Deutschland GmbH
CHEMPARK Dormagen
41538 Dormagen, Germany

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Institut cyclos-HTP GmbH
Institute for Recyclability
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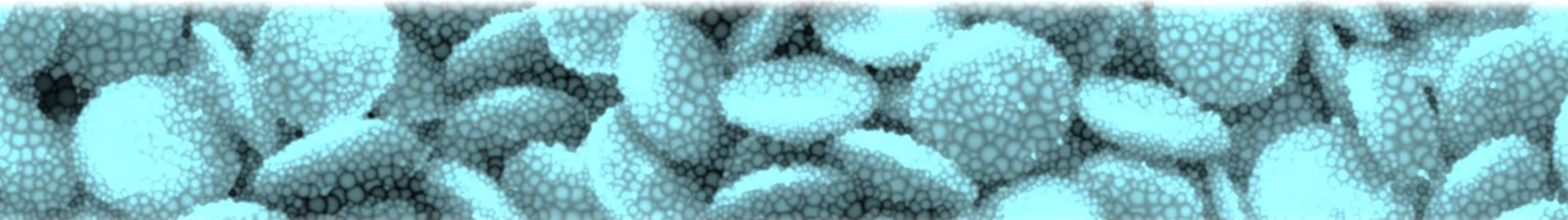


Content

1.1 Task and samples

Project task:

In this project, the recycling compatibility of **the PA6/IPDI copolymer family "Durethan C38"** in a PE-based packaging film is investigated. For this purpose, compounds are produced with PCR-LDPE and tested in the most important recycling applications of blown film and injection molding. The final evaluation of compatibility is based on a comparison with a PE-based reference film without PA.

This test was made according to the CHI standard method **CHI C8 PEF 1/4.1**. This CHI test method was developed by cyclos-HTP for LDPE-based flexible packaging samples with at least one material component or layer in the packaging structure with unknown or theoretically critical properties for the mechanical recycling to produce post-consumer recyclates based on LDPE films.

According to the CHI standard, a test is considered to be "passed" if no significant, negative deviations in the measurement results are found between the tested samples compared to the reference. If there is at least one significant negative deviation compared to the reference, a test is considered "failed" for the respective sample. Positive deviations to the advantage of the tested sample compared to the reference are permitted but are not evaluated as compensation for negative deviations.

Sample films provided by the customer:

- Sample **PCK-R0 |** PE-reference film without PA
- Sample **PCK-S1 |** PE-sample film with PA (Durethan C38FA)

Reference PCR recyclate:

- Sample **REF**: Commercial LDPE-based recyclate

PCK-R0 (PE reference film)

Single components	Thickness*	Density	Grammage	Percentage total
LDPE	32.5 µm	0.924 g/cm³	30.0 g/m²	41.1%
Yparex (LLDPE MAH-modified)	7.0 µm	0.928 g/cm³	6.5 g/m²	8.9%
Yparex (LLDPE MAH-modified)	7.0 µm	0.928 g/cm³	6.5 g/m²	8.9%
LDPE	32.5 µm	0.924 g/cm³	30.0 g/m²	41.1%
Sum	79.0 µm		73.1 g/m²	100.0%

**Evaluated by microscopy cross-section analysis*

PCK-S1 (PE sample film with PA)

Single components	Thickness*	Density	Grammage	Percentage total
LDPE	27.5 µm	0.924 g/cm³	25.4 g/m²	24.4%
Yparex (LLDPE MAH-modified)	3.0 µm	0.928 g/cm³	2.8 g/m²	2.7%
Durethan C38FA (PA6-IPDI copolymer)	34.0 µm	1.130 g/cm³	38.4 g/m²	**36.9%**
Yparex (LLDPE MAH-modified)	8.0 µm	0.928 g/cm³	7.4 g/m²	7.1%
LDPE	32.5 µm	0.924 g/cm³	30.0 g/m²	28.9%
Sum	105.0 µm		104.1 g/m²	100.0%

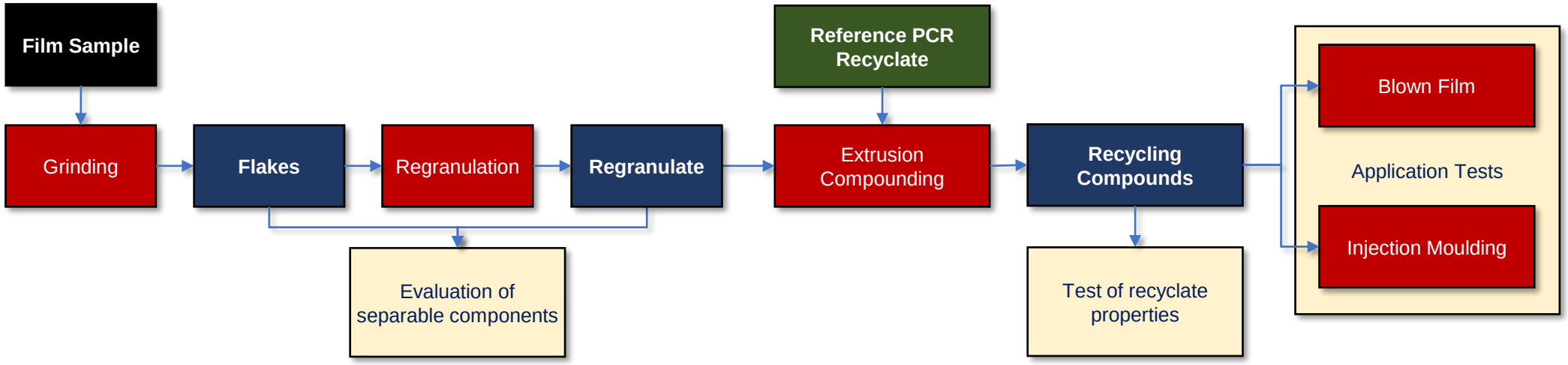
**Evaluated by microscopy cross-section analysis*

1.2 Test program and material preparation

1. **Grinding –** The samples are ground into flakes on a granulator with a 5-8 mm sieve to achieve a suitable particle size

2. **Homogenization of grinded flakes –** Each of the grinded samples must be homogenized separately by manual mixing of the flakes required for the entire test program. This is to achieve a uniform quality of the sample flakes.

3. **Homogenization of the reference recyclate –** Manual mixing of the PCR granules from all containers and batches required for the entire test program. This is to achieve a uniform quality of the recyclate so that all samples produced can be compared with each other. The reason is that the composition and quality of the PCR granulate can vary also within a production lot.



1.2 Test program and material preparation

4. **Regranulation –** A twin screw extruder ZSE35 MAXX was used to produce the regranulated samples (RG-R0 and RG-S1) from the grinded flakes. The effective melt temperature was **226 °C** (**PCK-R0** and **PCK-S1**). To reach a melt temperature in that range, suitable melt pressure settings and the correct temperature settings of the extruder zones were selected. The regranulation parameters can be found in the appendix (see p. 21).

The granule samples after extrusion showed a slight yellow-brown discoloration which was stronger for the reference R0 than for the sample S1.

*Figure: Flakes (left) and regranulate (right) of **PCK-R0 (PE-reference without PA)**.*

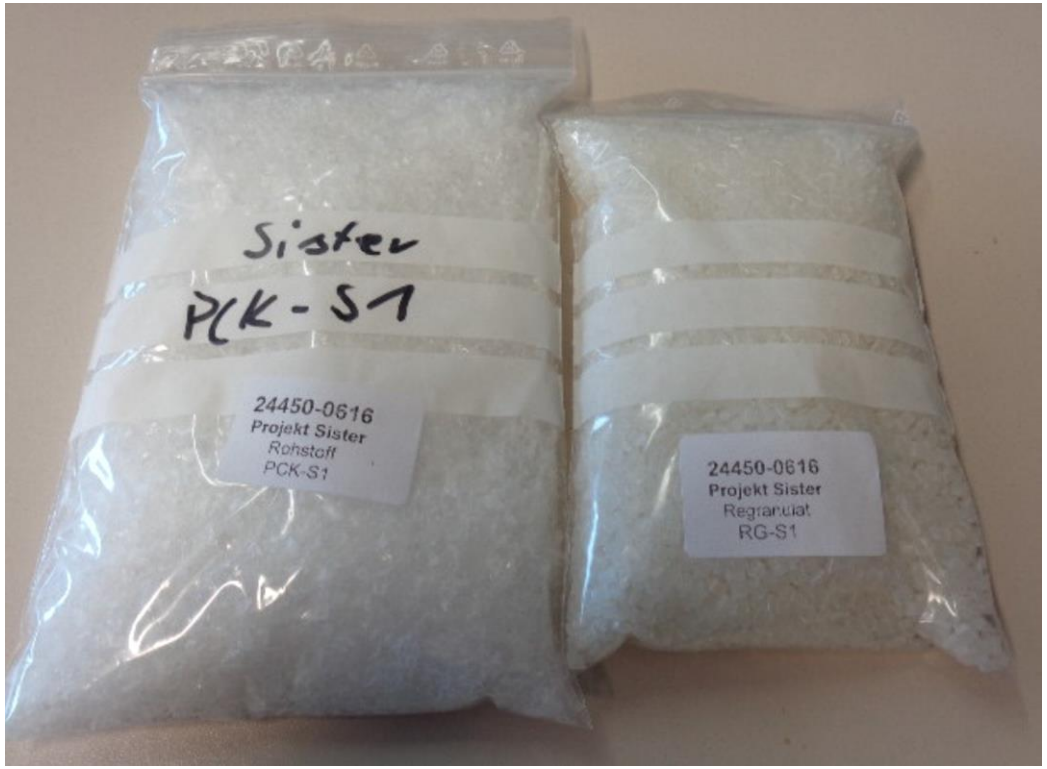


*Figure: Flakes (left) and regranulate (right) of **PCK-S1 (PE-sample film with PA)**.*

1.2 Test program and material preparation

5. **Compounding** – Each regranulate is blended with the reference PCR material (**PCR-REF**) in two different ratios (5% and 30%) to simulate different concentrations of the samples in the LDPE waste stream. The extrusion compounding was carried-out with the same twin-screw extruder. The effective melt temperature was in the range of **212-215 °C**. See the appendix for the compounding parameters (see p. 22)

Compound	RG-R0	RG-S1	PCR-REF
REF05	5 %		95 %
CHI05-1		5 %	95 %
REF30	30 %		70 %
CHI30-1		30 %	70 %

Figure: Comparison of the 5 wt.% compounds ***REF05*** *(reference) and* ***CHI05-1*** *(sample)*

Figure: Comparison of the 30 wt.% compounds ***REF30*** *(reference) and* ***CHI30-1*** *(sample)*

Content

2.1 Characterization of recyclates | DSC

The thermal properties of the sample films are tested with Dynamic scanning calorimetry (DSC). Determination of the melting and crystallisation behaviour by DSC was carried out with a heat flow calorimeter according to DIN EN ISO 11357-1. The DSC diagrams can be found in the appendix (see p. 25 - 36).

		PCK-R0	RG-R0	PCK-S1	RG-S1
1. Heating	Peak-temp. [°C]	111.6 122.4	116.0	112.5 123.6 212.8	115.1 212.1
2. Heating	Peak-temp. [°C]	111.6 122.4	111.0 120.1	110.1 119.9 210.1	110.9 120.2 211.1
	Areas [J/g]	135.8 10.3	124.6 16.1	92.7 7.9 15.7	93.1 5.3 11.8
Cooling	Peak-temp. [°C]	63.6 99.9	100.0	99.8 164.2	99.6 168.0

Observations:

- As a result of the regranulation, only one instead of two peaks occur in ***Sample R0*** during the first heating and cooling. Furthermore, there is a slight shift in the peak temperature.
- For ***Sample S1***, there are also changes observed due to the regranulation. These changes are mainly limited to the 1st heating, where the central peak disappears.
- Comparing the samples with each other, it is noticeable that ***S1*** has additional peaks compared to ***R0***. These are found in the 1st and 2nd heating between 210.1 °C and 212.8 °C. The reason for this is the PA.

Summary:

⇨ **Apart from the additional peaks at S1, which are caused by the PA, no other influences on the melting and crystallisation behaviour of the material after recycling can be observed.**

⇨ **There are no restrictions due to the DSC results.**

2.1 Characterization of recyclates | TGA

Thermogravimetric analysis (TGA) is used to determine the ash and volatile content of the sample flakes (PCK-R0, PCK-S1) and the corresponding regranulates (RG-R0, RG-R1). The test is carried out at the temperatures used in the recycling process according to EN ISO 11358-1.

Compound	Volatile Components [%]	Polymer share [%]	Oxidizable Components [%]	Residual mass [%]
PCK-R0	0	97.4	0.7	1.9
RG-R0	0	99.8	0.1	0.2
PCK-S1	0	99.6	0.2	0.2
RG-S1	0	99.8	0.2	0.1

Observations:

- The TGA results of the samples ***RG-R0***, ***PCK-S1*** and ***RG-S1*** are characterized by a very high degree of coincidence.
- Only the Polymer Share of ***PCK-R0*** is slightly lower than that of the other samples. As a result, the levels of Oxidizable Components and Remaining Mass are slightly higher.
- Overall, however, no significant deviations were found between the samples or due to regranulation.

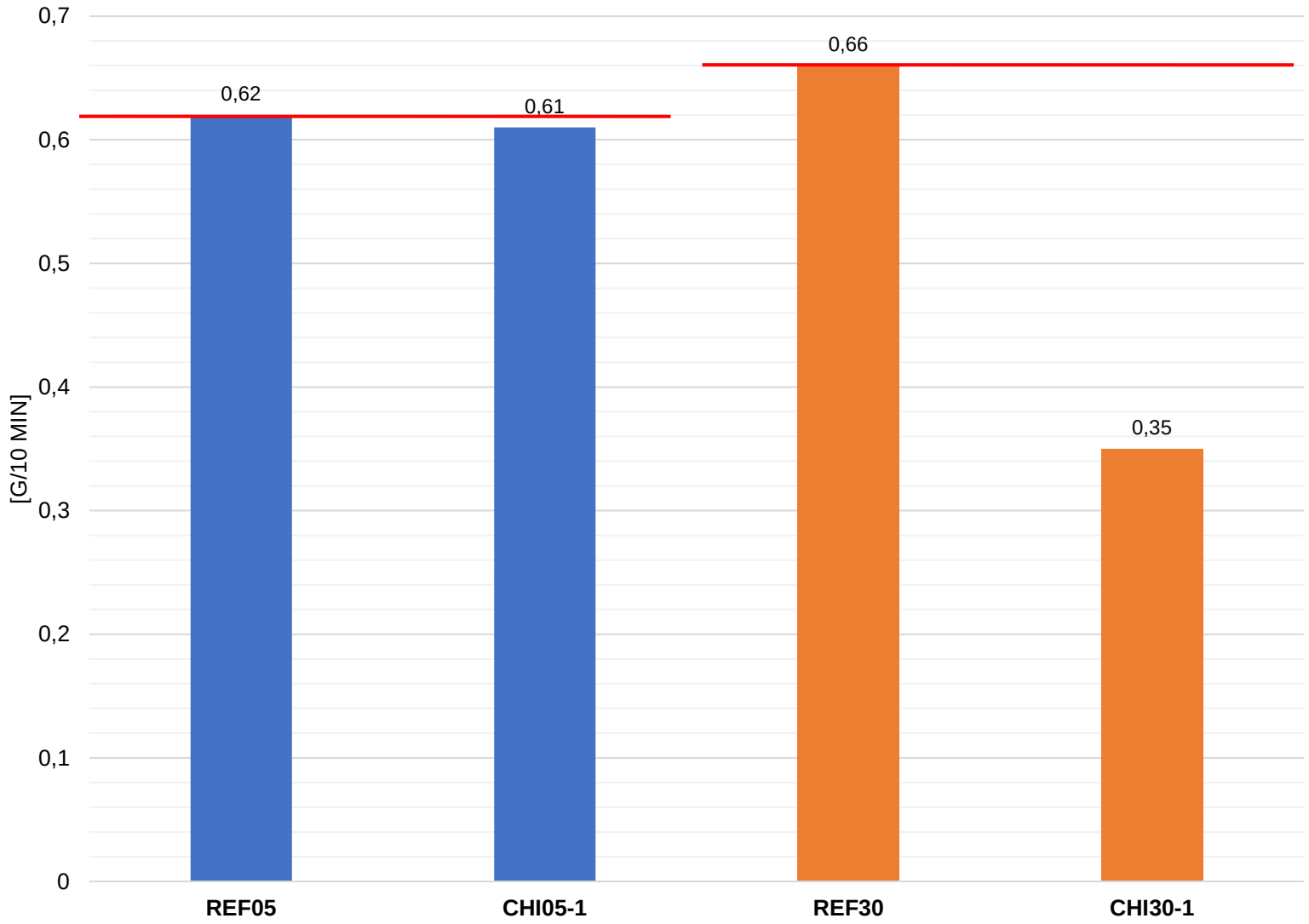
Summary:

⇨ **The PA has only minor effects on the thermal degradation behavior of the material after recycling.**

⇨ **There are no restrictions due to TGA results.**

2.1 Characterization of recyclates | MFR

The Melt Flow Rate (MFR) is used to test whether the recycling compounds produced are within the viscosity range of the respective PCR reference. The determination is carried out according to ISO 1133-1 at 190 °C with 2.16kg load.



Observations:

- ***CHI05-1*** shows nearly no changes in the MFR compared to ***REF05***. The MFR for ***REF30*** is also at a comparable level.
- ***CHI30-1*** shows a sharp increase in viscosity. The MFR is almost half of the value of ***REF30***.

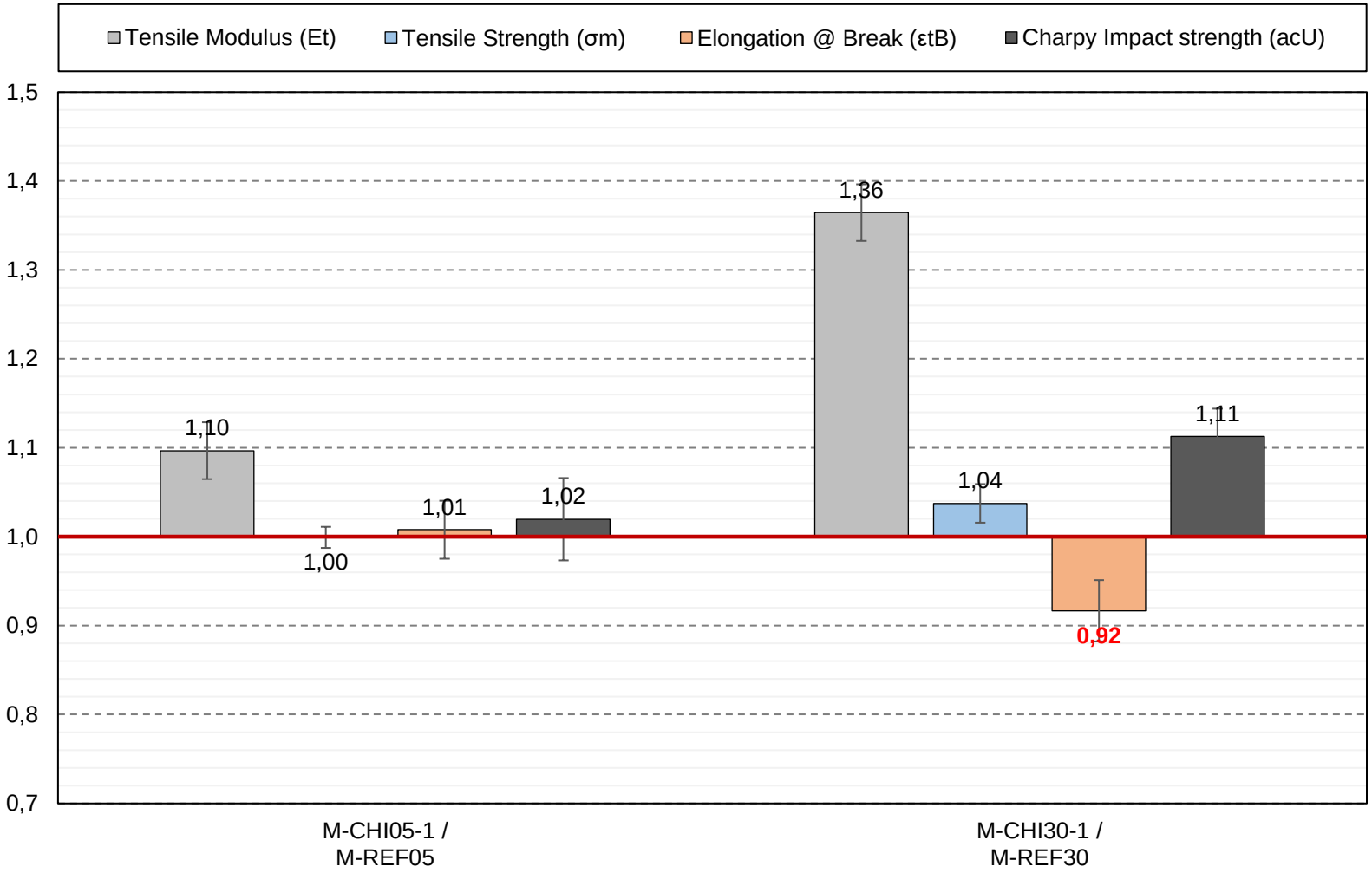
Summary:

- ⇨ **The increase in viscosity for the CHI30-1 sample must be viewed critically. This could theoretically lead to problems in injection moulding applications.**
- ⇨ **Further analyses of the MFR for CHI30-1 are recommended.**
- ⇨ **Nevertheless, the MFR results are still acceptable.**

2.2 Injection moulding application tests | Mechanical properties

The injection moulding samples (M-REF05, M-CHI05,…) are produced from the recycling compounds according to DIN EN ISO 3167. Subsequently, the characterization of the injection moulding specimens is carried out by tensile testing (DIN EN ISO 527-1) and Charpy notched impact strength (DIN EN ISO 179-2 1eA). The parameters for injection molding specimen production can be found in the appendix (see p. 23).

The following applies: **1.0 = same level as REF reference.**



Observations:

- ***M-CHI05-1*** is nearly at a comparable level to the corresponding reference ***M-REF05***. No negative deviations can be observed.
- For ***M-CHI30-1***, the tensile modulus and Charpy impact strength are higher compared to ***M-REF30***. In addition, a significant reduction of Elongation @ Break can be observed.

Summary:

⇨ **M-CHI05-1 passed the injection molding application test.**

⇨ **M-CHI30-1 failed the mechanical test of the injection molding application due to the significant reduction of Elongation @ Break.**

2.3 Blown film application tests | Processing

The blown film samples are produced according to the CHI test method CHI-C8-BFPE,. The films contain 60% of the corresponding recycled compounds and 40% new PE-LD/LLD. The corresponding parameters can be found in the appendix (see p. 24).

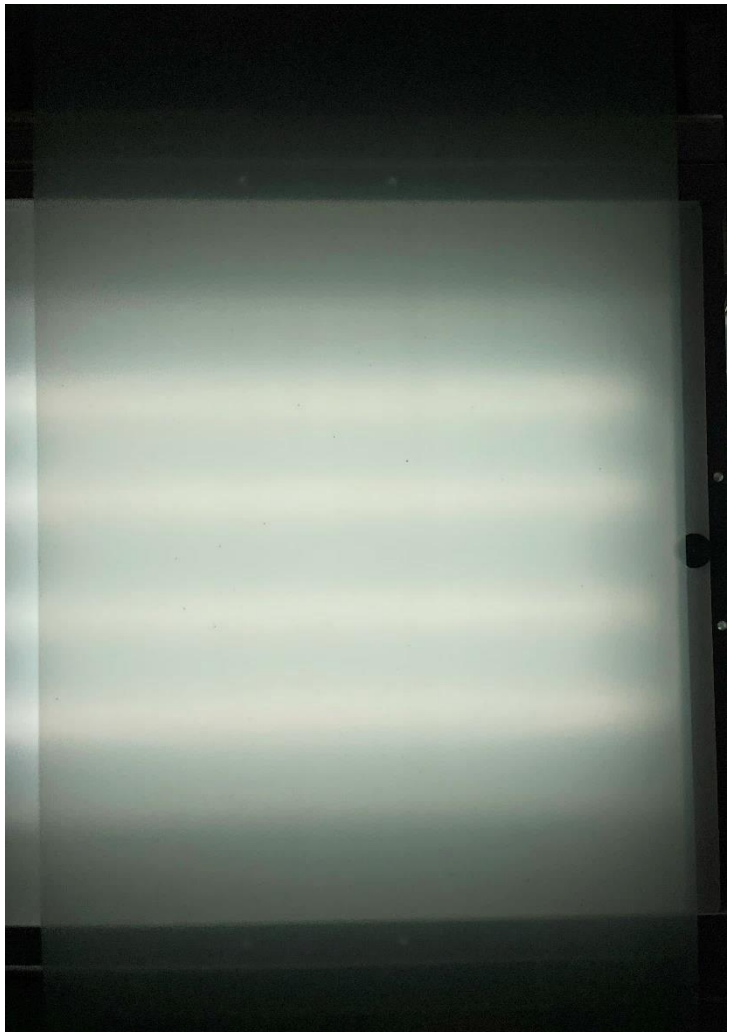


*Figure: Blown film production of **F-CHI05-1***

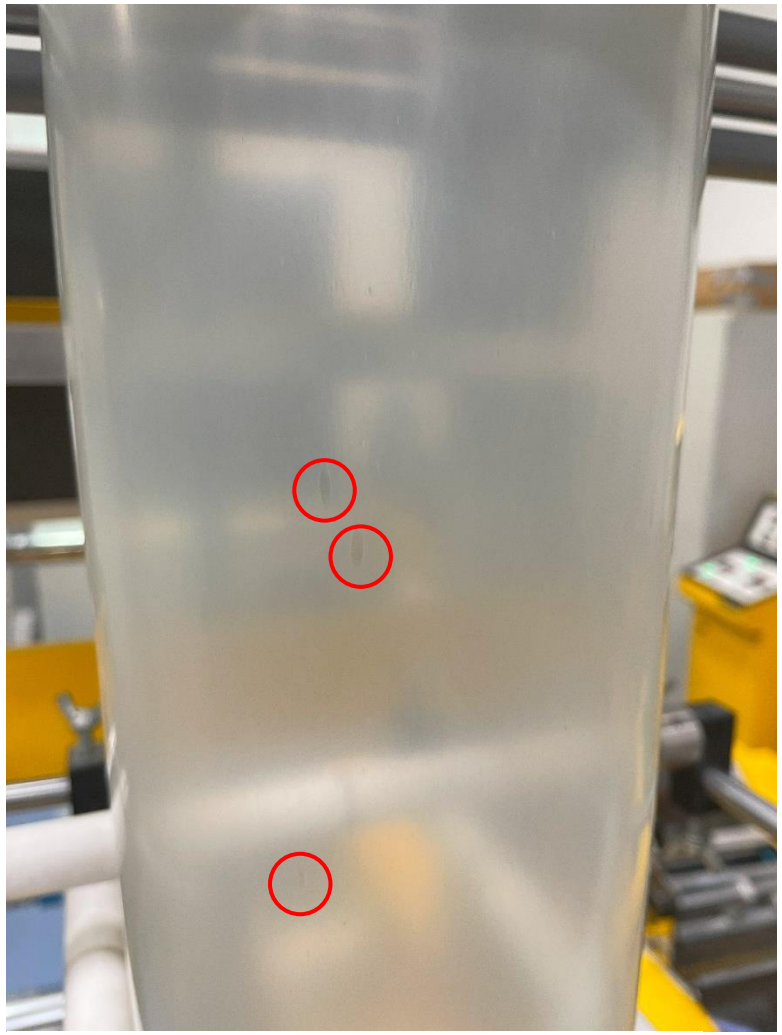


*Figure: Production of blown film **F-CHI30-1**, with holes marked in the film that occurred during production.*

Observations:

- The films F-REF05, F-REF30 and F-CHI05-1 could be produced without problems.
- During the production of F-CHI30-1, holes appeared in the blown film from time to time which was caused by a too high moisture content of the compound.
- After additional drying of the compound CHI30 for 2h at 80°C, the blown film production was possible without holes.

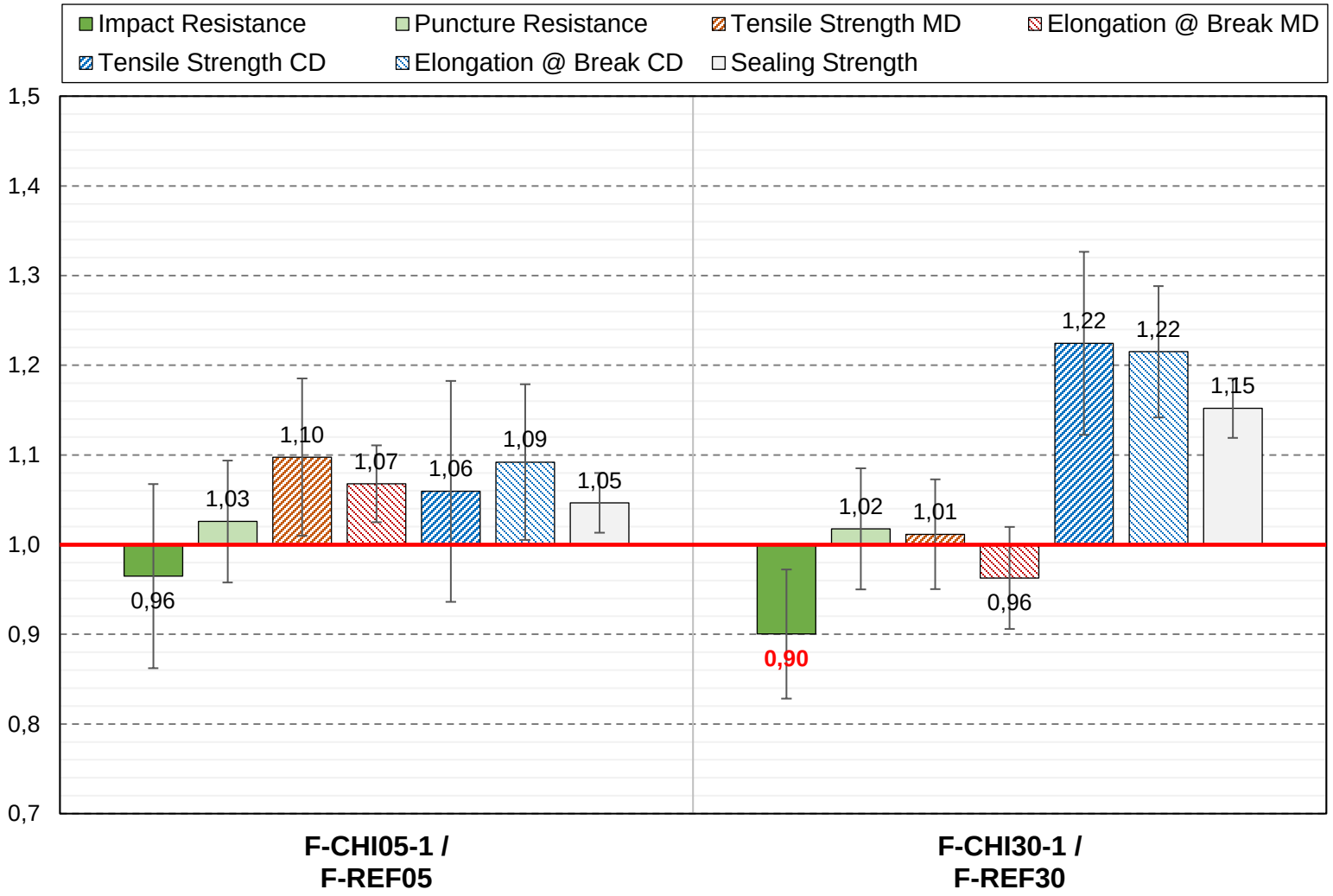
Summary:

⇨ **All samples passed the blown film processing test.**

2.3 Blown film application tests | Mechanical properties

The blown film samples are characterized by tensile test (DIN EN ISO 527-3). The test was carried out at 500 mm/min. In addition, the impact strength of the blown films was tested by Dart Drop (DIN EN ISO 7765-1) as well as the sealing properties (DIN 55529).

The following applies: **1.0 = same level as PE reference.**



Observations:

- ***F-CHI05-1*** shows slight but significant improvements in Tensile Test and Sealing Strength in comparison to ***F-REF05***.
- Compared to ***F-REF30***, ***F-CHI30-1*** shows a significant decrease of 10% in impact resistance. In contrast, the parameters from the Sealing Test and the Tensile Test in CD are noticeably and significantly higher.

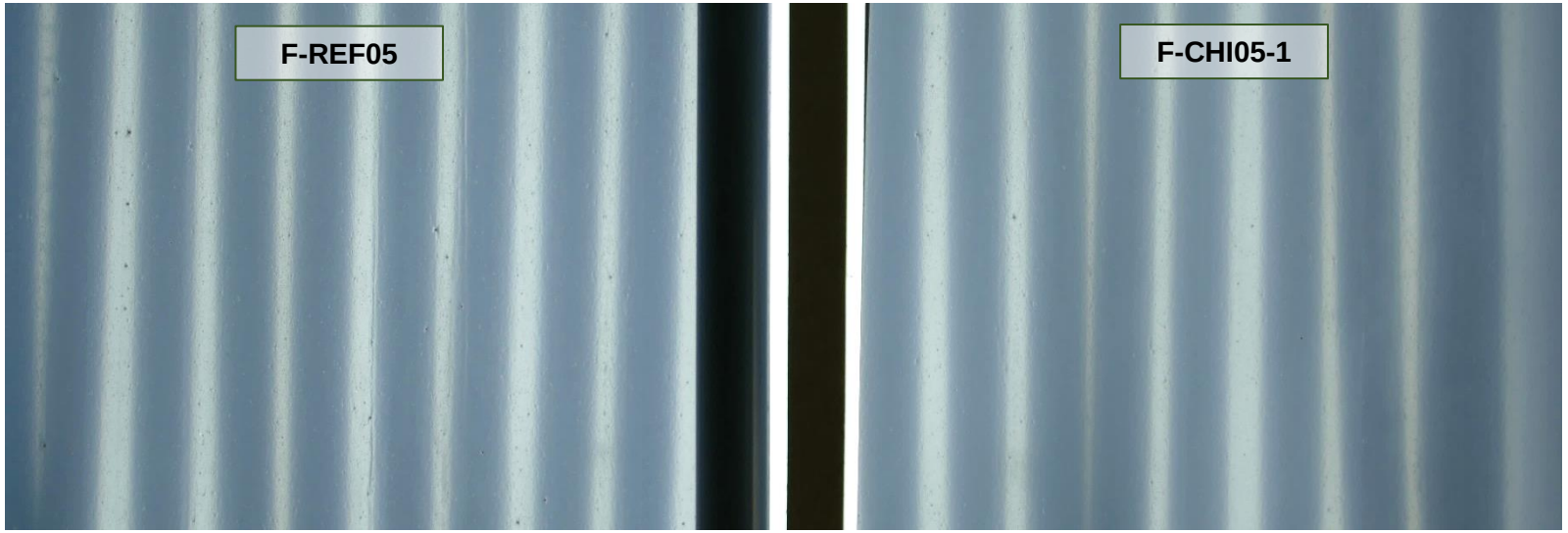
Summary:

⇨ **F-CHI05-1 passed the blown film application test.**

⇨ **Based on the result of the impact resistance test, F-CHI30-1 did not pass the blown film application test.**

2.3 Blown film application tests | Visual inspection

The blown films are then visually inspected using a high-contrast light source. For this purpose, the samples are compared with the corresponding reference sample in terms of transparency, opacity, black spots and gels.



Observations:

- The films ***F-REF05*** and ***F-CHI05-1*** have a comparable and good appearance.
- ***F-REF30*** and ***F-CHI30-1*** have less specks than ***F-CHI05*** and ***F-REF05***, confirming that the specks come from the recyclate present in the sample.
- ***F-CHI30-1*** shows an increased opacity, possibly caused by interfaces between polar PA and non-polar PE. This results is still acceptable.

Summary:

⇨ **F-CHI05-1 passed the visual test**

⇨ **F-CHI30-1 is still acceptable**

2.3 Blown film application tests | Sealing tightness

The sealing tightness is tested according to CHI-C8-BFPE. For this purpose, the blown film samples are prepared with an impulse sealing equipment with 2.4 mm seam width. The sealing time is adjusted with the reference sample to be at the lower end of the window where all reference samples are still tight.

At least 3 samples of each variant were tested. The tightness of the bags produced was evaluated according to the following criteria:

- 3 out of 3 or 4 out of 5 samples must be tight (without leakages) to pass the test
- No water penetrates through the seam within 1t minute after the sealed tube is filled
- The seam of the sample is not breaking within 5 minutes after the sealed tube is filled

Sample	Film 1	Film 2	Film 3	Film 4	Film 5	Assessment
REF05	✓	✓	✓	-	-	*Reference*
F-CHI05-1	✓	✓	✓	-	-	**Passed**
REF30	✓	✓	✓	-	-	*Reference*
F-CHI30-1	✓	✘	✓	✓	✓	**Passed**

Content

3.1 Conclusion | Summary

No.	Test criteria	CHI05-1 *(compared to REF05)*	CHI30-1 *(compared to REF30)*
1	Regranulation, Compounding	Passed ✓	Passed ✓
2	MFR (Viscosity of Compounds)	Passed ✓	Acceptable ✓
3	DSC / TGA thermal analysis	Passed ✓	Passed ✓
4	Injection molding | Processing	Passed ✓	Passed ✓
5	Injection molding | Mechanical properties	Passed ✓	Failed ×
6	Blown films | Processing	Passed ✓	Passed ✓
7	Blown films | Visual inspection	Passed ✓	Acceptable ✓
8	Blown films | Mechanical properties	Passed ✓	Failed ×
9	Blown films | Sealing tightness	Passed ✓	Passed ✓
Conclusion acc. to CHI standard		**Passed ✓**	**Failed ×**

In this project, the recyclability of the **PA6/IPDI copolymer "Durethan C38FA"** in a PE-based packaging film was investigated. For this purpose, the sample film was regranulated. Compounds were produced by mixing with PCR-LDPE at defined ratios (*CHI05-1* and *CHI30-1*) and tested in the most important recycling applications of blown film and injection moulding. In the same way, compounds were produced and tested from a PE-based reference film without PA (*REF05* and *REF30*). The final evaluation of compatibility was carried out by comparing the values of the sample with those of the reference.

For a proportion of 5 wt.% of the PA-containing film (*CHI05-1*), no significant degradation was documented compared to the corresponding reference (*REF05*). In contrast, a proportion of 30 wt.% (*CHI30-1*) showed significant decrease of elongation at break in the injection moulding application test. There was also evidence of a reduction in Impact Resistance for Blown Film applications compared to the corresponding reference *REF30*.

Conclusions:

Based on the results with CHI05-1, the PA6/IPDI copolymer grade **"Durethan C38"** and its neighbor grades can be regarded under tested conditions as *compatible in the recycling of LDPE films*.

Classification as a valuable material is not possible due to the CHI30-1 test series. Its results have shown that the copolymer has a negative impact at higher concentrations of the sample material in the recycling stream.

3.2 Conclusion | Classification according to CHI Standard

- For this assessment, the composition of the packaging and the results of this compatibility test program were considered. The detection and sorting performance was not tested. By testing, it could be proven that the **PA6-IPDI copolymer** has no negative effects at realistic concentrations of the packaging film in the recycling stream.
- The PA copolymer was therefore re-classified as CAT 2 component in the packaging structure as material that cannot be separated using the established methods in the recycling process, but in practice do not or do not significantly impair the recycle properties. The corresponding proportion is not counted as valuable material within the scope of evaluation criterion C 1.

Criteria		**Analysis**	**Original assessment for PCK-S1 “PE film with Durethan C38FA”**		
C 0	Path allocation		**Path 1**: LDPE films		
C 1	Valuable material content	Evaluation of material properties **before test**	∑ 100%: LDPE, LLDPE		63.1 %
			CAT 1	-	-
			CAT 2	-	-
			CAT 3	**PA6/IPDI copolymer**	36.9 %

Before the testing, the structure was classified as “not recyclable”

Criteria		**Analysis**	**New assessment for PCK-S1 “PE film with Durethan C38FA”**		
C 0	Path allocation		**Path 1**: LDPE films		
C 1	Valuable material content	Evaluation of material properties **after test**	∑ 100%: LDPE, LLDPE		63.1 %
			CAT 1	-	-
			CAT 2	**PA6/IPDI copolymer**	36.9 %
			CAT 3	-	-

3.3 Conclusion | Issued Certificate

No. 2776-2024-004286

Polyamide Copolymer PA6/IPDI

Including the Envalior grades Durethan C38F, C38FA, C38FAM, C38FKAM, C38FKS

as layer in coextruded PE-based packaging films; in combination with ≥ 0.3 g per g PA of maleic anhydride-grafted PE as tie layer specified for PA/PE.

The PA was tested with up to 37 % by weight as component in a packaging sample and after recycling with up to 11 % by weight as part of a PCR LDPE recycle.

According to the CHI standard, this PA resin had no significant quality-reducing effects on the recycle in the tested applications for injection moulding and blown films. Based on this assessment, the tested PA material can be rated as:

Recycling Compatible

under tested conditions for the application in PE-based flexible packaging

CERTIFICATE

Recyclability of Packaging Material

Envalior Deutschland GmbH
CHEMPARK Dormagen
41538 Dormagen | Germany

The company receives the certification of recycling compatibility for the following material:

Designation

Polyamide Copolymer PA6/IPDI

Including the Envalior grades Durethan C38F, C38FA, C38FAM, C38FKAM, C38FKS

as layer in coextruded PE-based packaging films; in combination with ≥ 0.3 g per g PA of maleic anhydride-grafted PE as tie layer specified for PA/PE.

Test result

Assessment via path: Path 1: Plastic films / LDPE

Test standard:
☒ **Requirements and assessment catalogue of the cyclos-HTP Institute** for EU-wide certification

Within the certification process, conformity with the following standards was also checked:
☒ **Minimum standard for measuring the recycling capacity** of the ZSVR (state 31/08/2023); also integrated.
☒ **DIN EN 13430** with regard to material recyclability in the post-use phase; also integrated.

The following reference processes, materials and applications are taken into consideration within the certification process:
- Recyclate use for injection moulding and blown film applications.
- Test program based on CHI test method **CHI-C8-PEF-1** with the use of PCR-based LDPE recyclate as reference.

The PA was tested with up to 37 % by weight as component in a packaging sample and after recycling with up to 11 % by weight as part of a PCR LDPE recyclate.

According to the CHI standard, this PA resin had no significant quality-reducing effects on the recycle in the tested applications for injection moulding and blown films. Based on this assessment, the tested PA material can be rated as:

Recycling Compatible

under tested conditions for the application in PE-based flexible packaging

The recyclability of a packaging is dependent on the final packaging design for which this material will be used (including composition, size, colour, and others).

This certificate (No. 2776-2024-004286-R1) is valid until **31/07/2025** (1 year upon issue).

This certificate will lose validity in case of qualitative or quantitative changes of material properties or chemical structures and compositions.

Aachen, dated 17/07/2024 (revised 07/08/2024)

Dr. Roland Bothor
Publicly appointed and sworn expert for packaging disposal
Competent authority:
Chamber of Industry and Commerce, Aachen
The detailed results are documented in the corresponding test report (No. 2776-2024-004286).

CHI | cyclos-HTP Institute
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Content

4. Annex | Regranulation parameters

	PCK-R0	PCK-S1
Pre-treatment raw materials	none	
Extruder	ZSE35 MAXX	
Temperatures (Feed -> Nozzle)	190°C / 190°C / 195°C / 195°C / 200°C / 200°C / 200°C / 205°C / 210°C / 210°C / 215°C / 220°C / 220°C	
Dosing points	feed-in zone	
Screw speed	220min^{-1}	
Throughput	8kg/h	8kg/h
Torque	27%	30%
Mass temperature	226°C	226°C
Degassing	vacuum	
Special observation	none	

4. Annex | Compounding parameters

	Sister REF05	Sister CHI05-1	Sister REF30	Sister CHI30-1
Pre-treatment raw materials	none			
Extruder	ZSE35 MAXX			
Temperatures (Feed -> Nozzle)	180°C / 180°C / 180°C / 180°C / 185°C / 185°C / 190°C / 190°C / 190°C / 195°C / 195°C / 195°C / 195°C			
Dosing points	feed-in zone			
Screw speed	250min^{-1}			
Throughput	30kg/h	30kg/h	30kg/h	30kg/h
Torque	59%	58%	58%	59%
Mass temperature	212°C	213°C	215°C	212°C
Degassing	vacuum			
Special observation	typical PE-recycling odour during compounding			

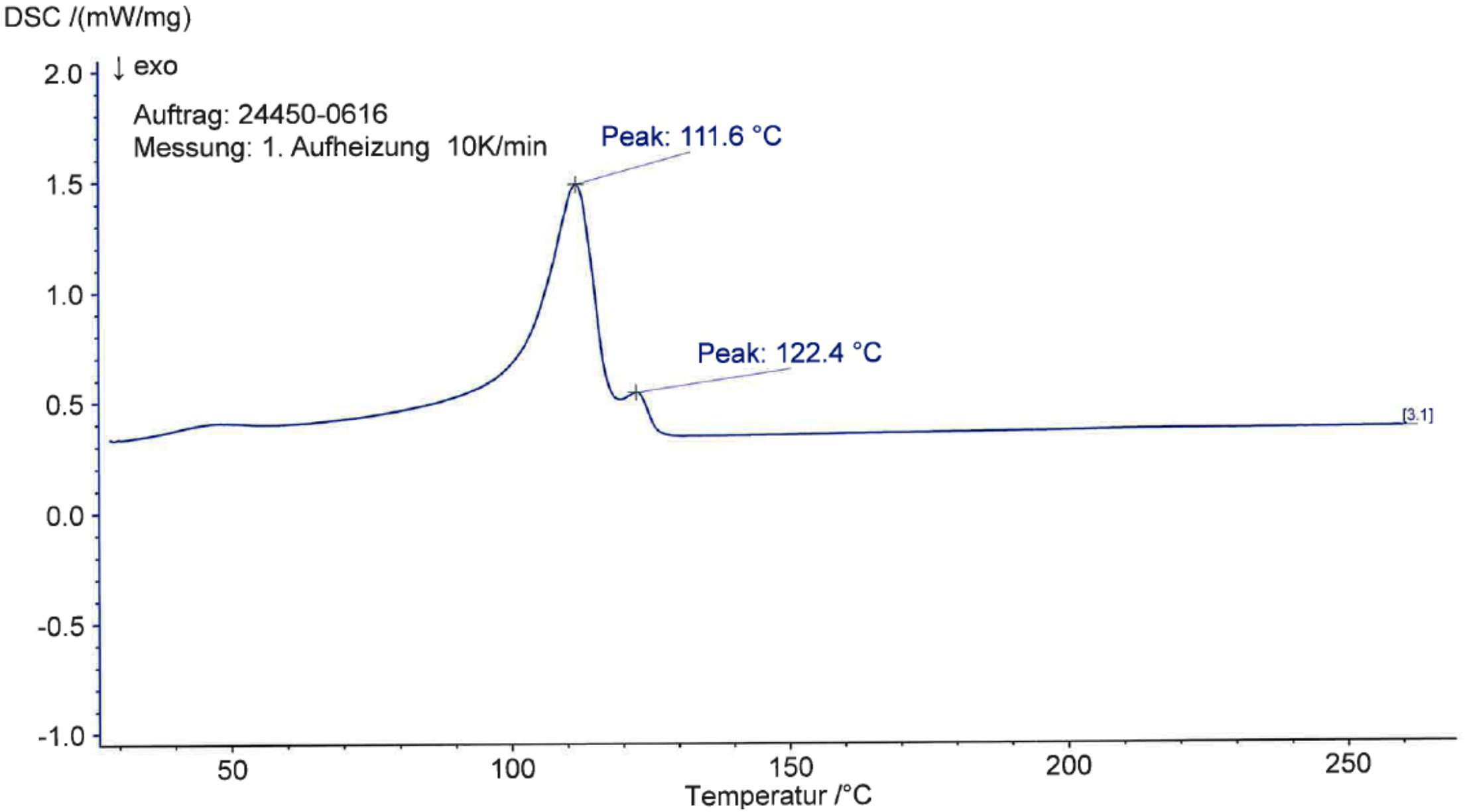
4. Annex | Injection Moulding parameters

	Sister M-REF05	Sister M-CHI05-1	Sister M-REF30	Sister M-CHI30-1
Drying	60°C / 5h			
Machine	Arburg Allrounder 320M with Campus tool			
Screw diameter	30mm			
Temperatures (feed -> nozzle)	220°C / 225°C / 230°C / 230°C / 235°C			
Mold temperature	60°C			
Dosage	18m/min with 30bar holding pressure			
Injection speed	25cm^3/s			
Back pressure	600bar / 25s			
Remaining cooling time	15s			
Total cycle time	53,5s			
Special observation	No special observations.			

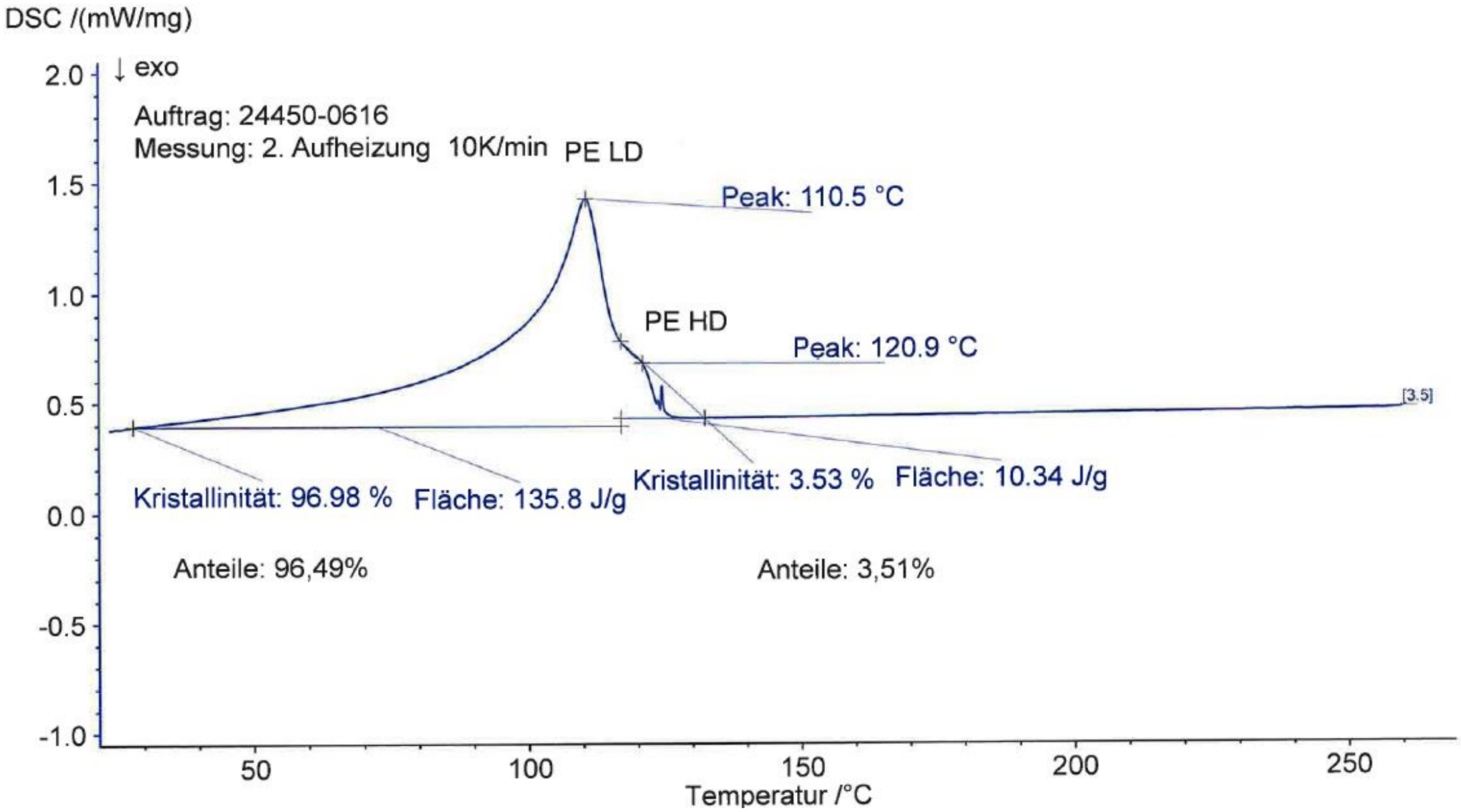
4. Annex | Blown Film production parameters

	Sister F-REF05	Sister F-CHI05	Sister F-REF30	Sister F-CHI30
Pre-treatment raw materials	drying 60°C / 5h / hot air dryer			
Extruder	Labtech single screw extruder with screw diameter 25mm			
Temperatures (Feed -> Nozzle)	195°C / 210°C / 220°C / 225°C / 225°C			
Temperatures blow film die	below: 225°C / middle: 225°C / upper: 225°C			
Temperatures Feeder	225°C			
Screw speed	$60min^{-1}$			
Mass pressure	141bar	147bar	132bar	145bar
Torque	44%	42%	43%	43%
Mass temperature	213°C	214°C	214°C	214°C
Blow ratio	2,7			
Special observation	No special observations.			

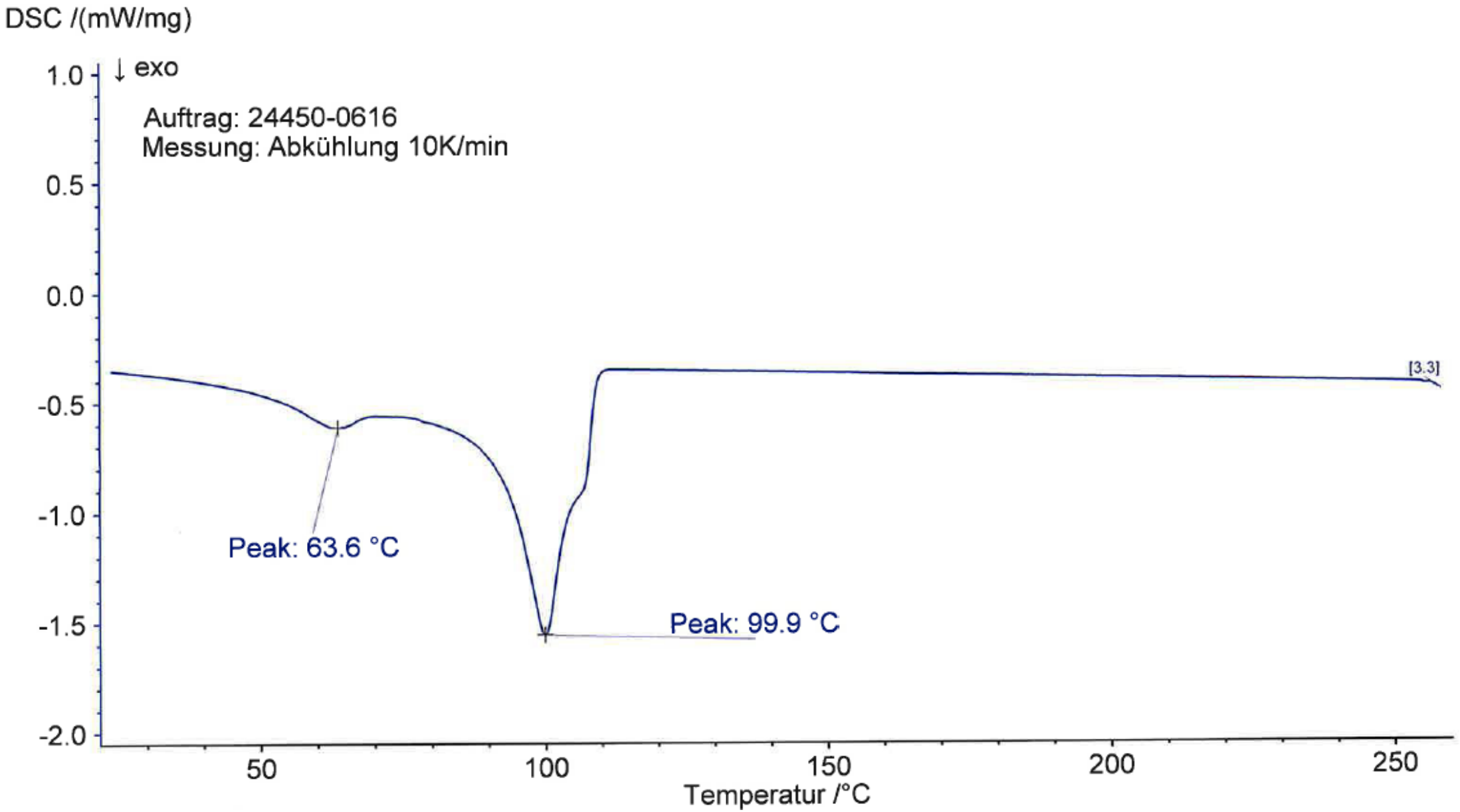
4. Annex | DSC (PCK-R0 | 1. Heating)



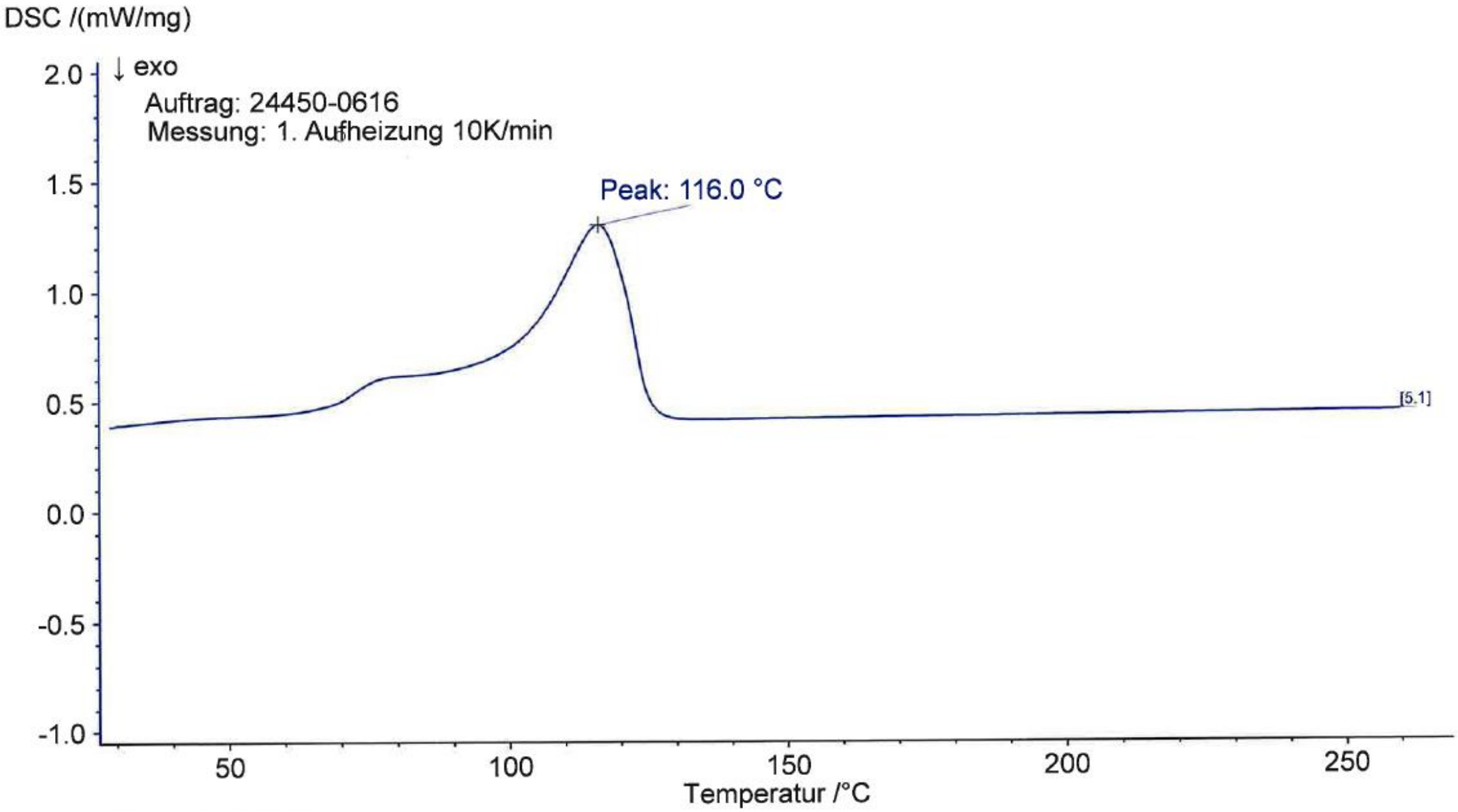
4. Annex | DSC (PCK-R0 | 2. Heating)



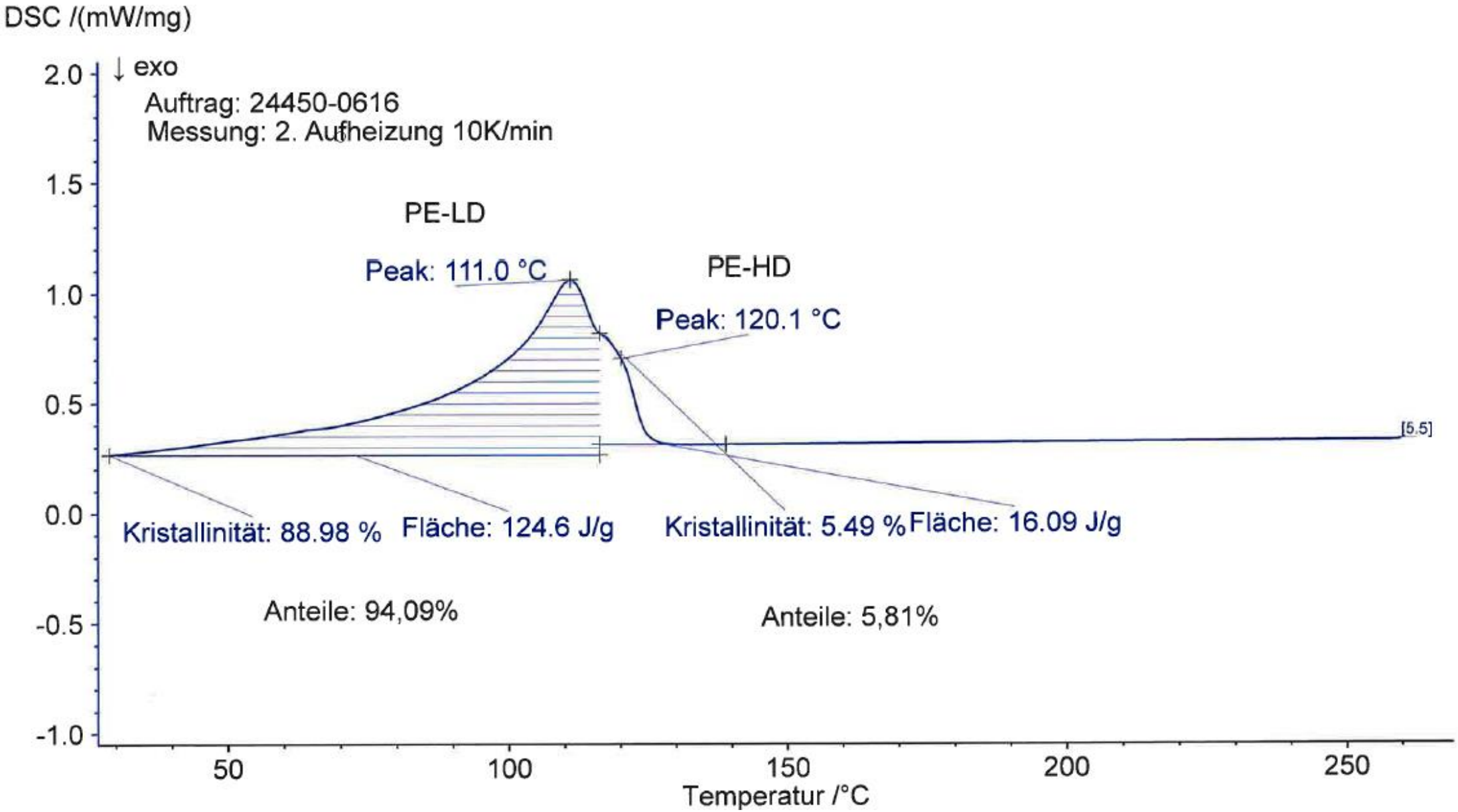
4. Annex | DSC (PCK-R0 | Cooling)



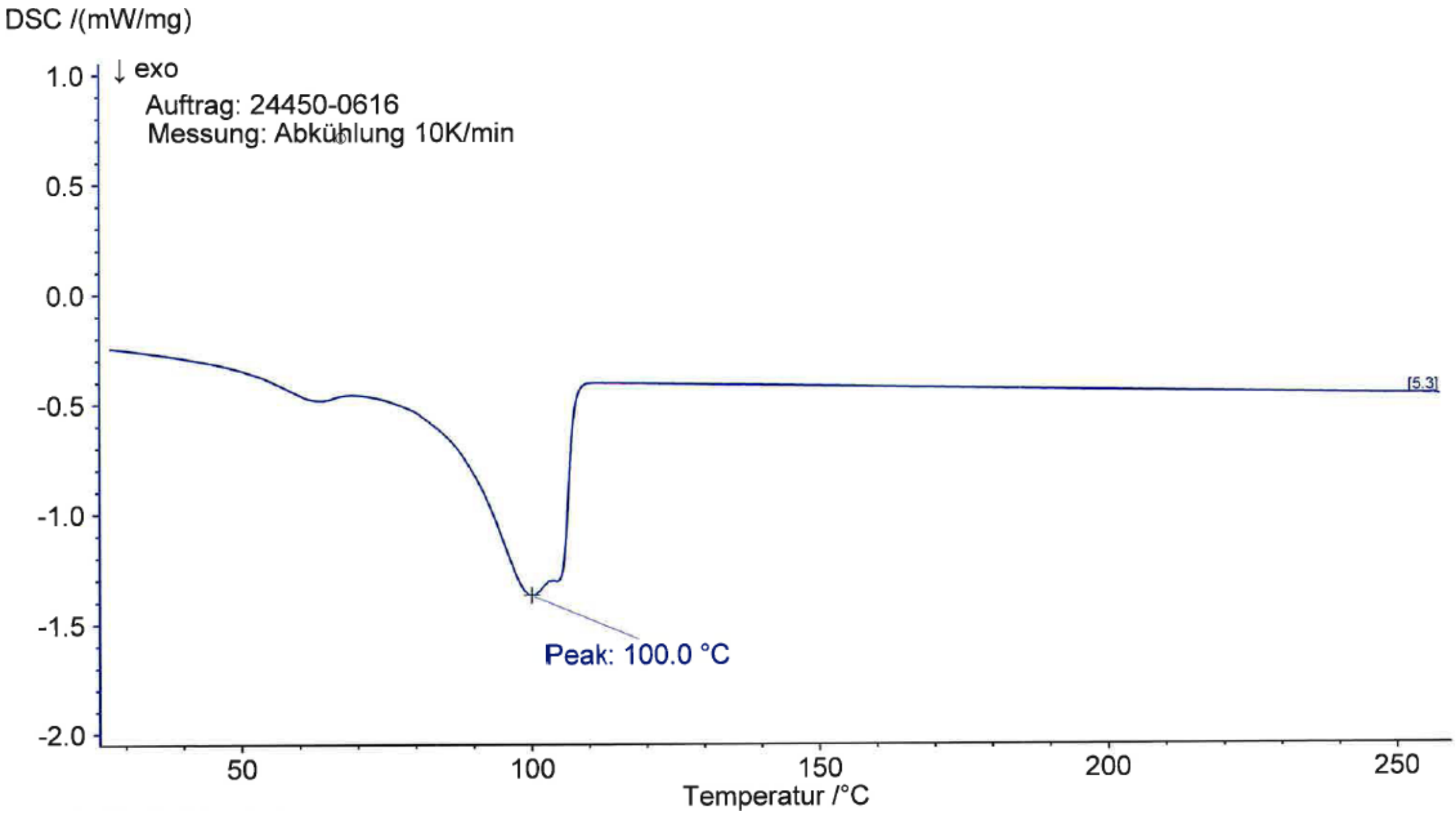
4. Annex | DSC (RG-R0 | 1. Heating)



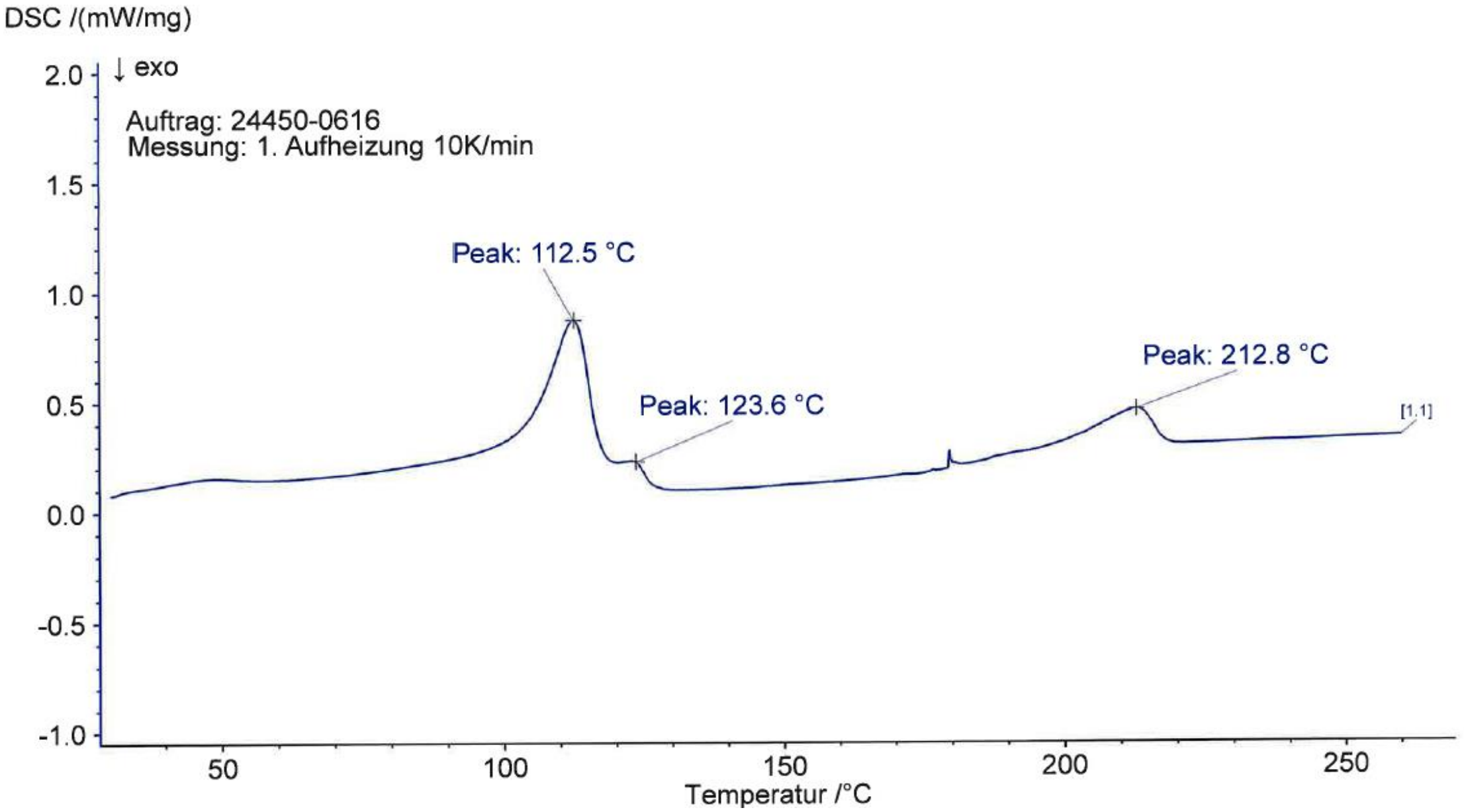
4. Annex | DSC (RG-R0 | 2. Heating)



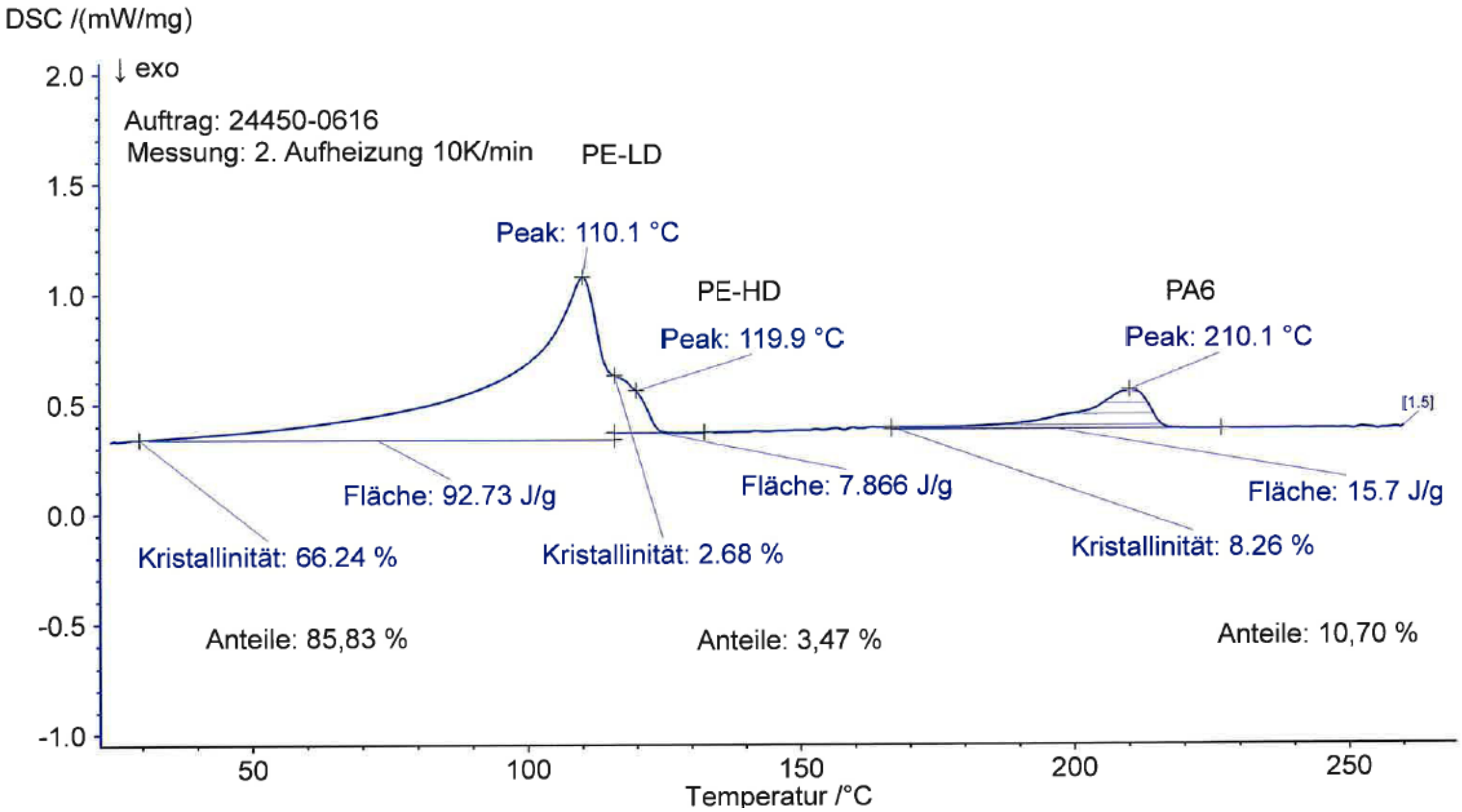
4. Annex | DSC (RG-R0 | Cooling)



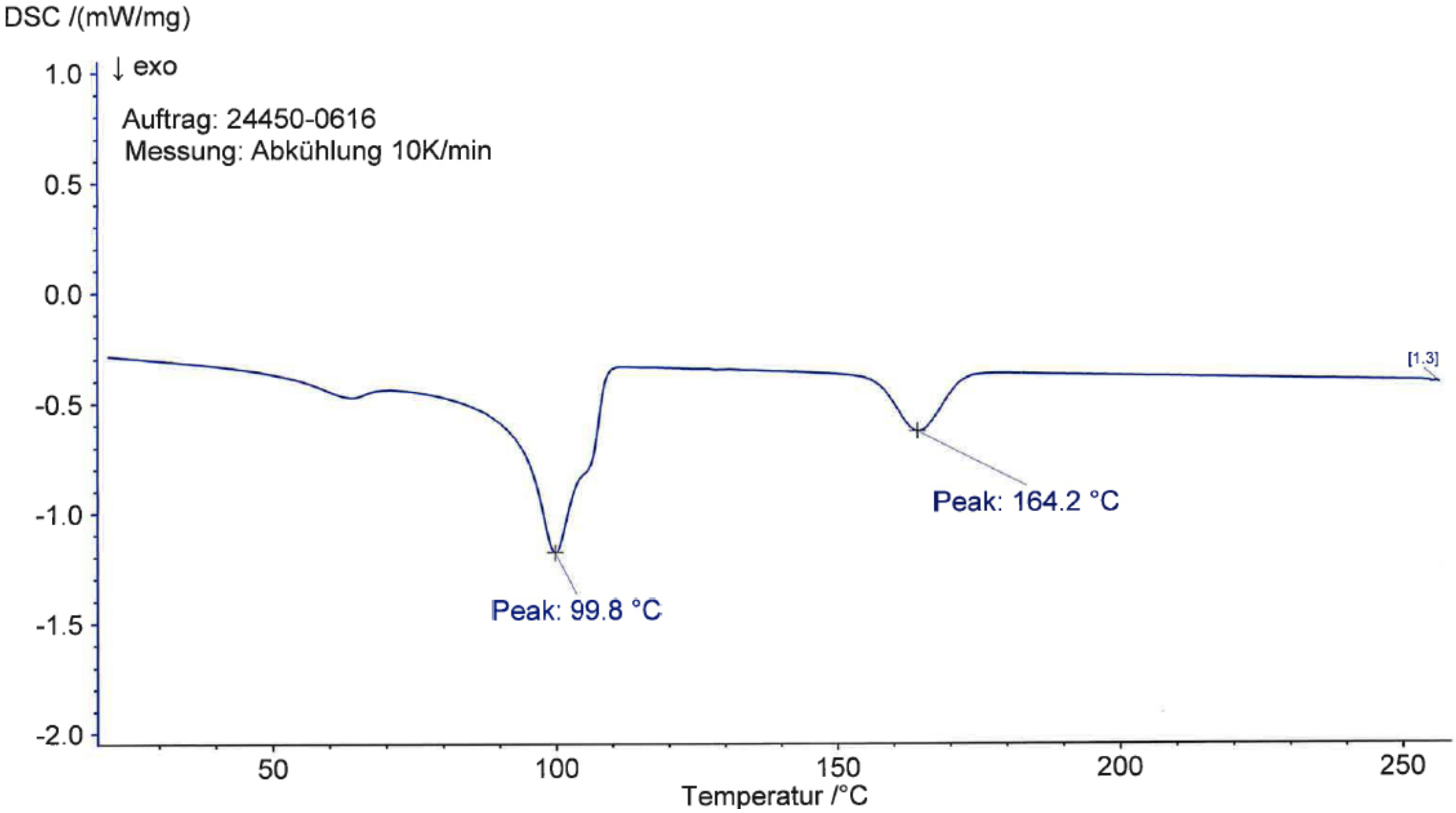
4. Annex | DSC (PCK-S1 | 1. Heating)



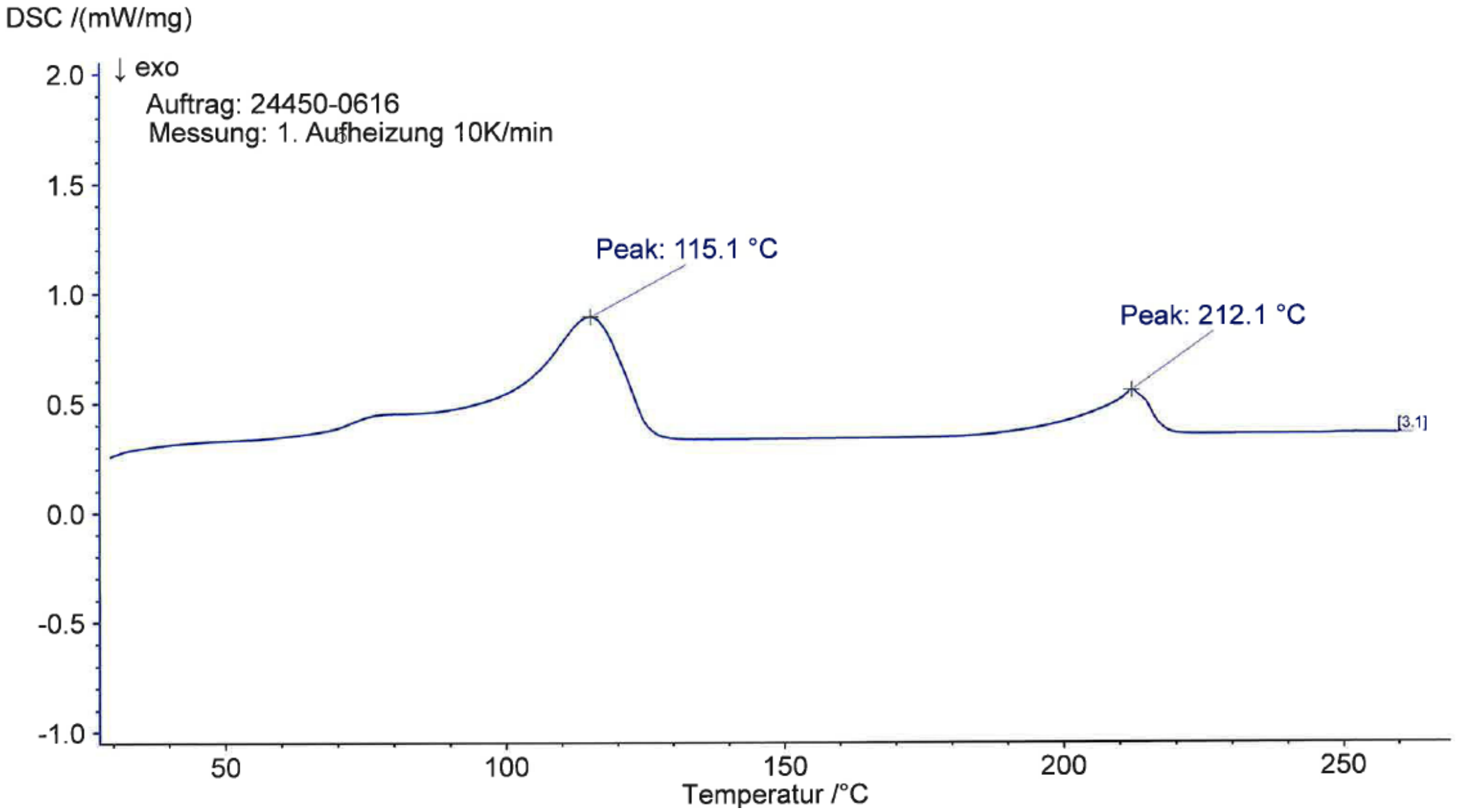
4. Annex | DSC (PCK-S1 | 2. Heating)



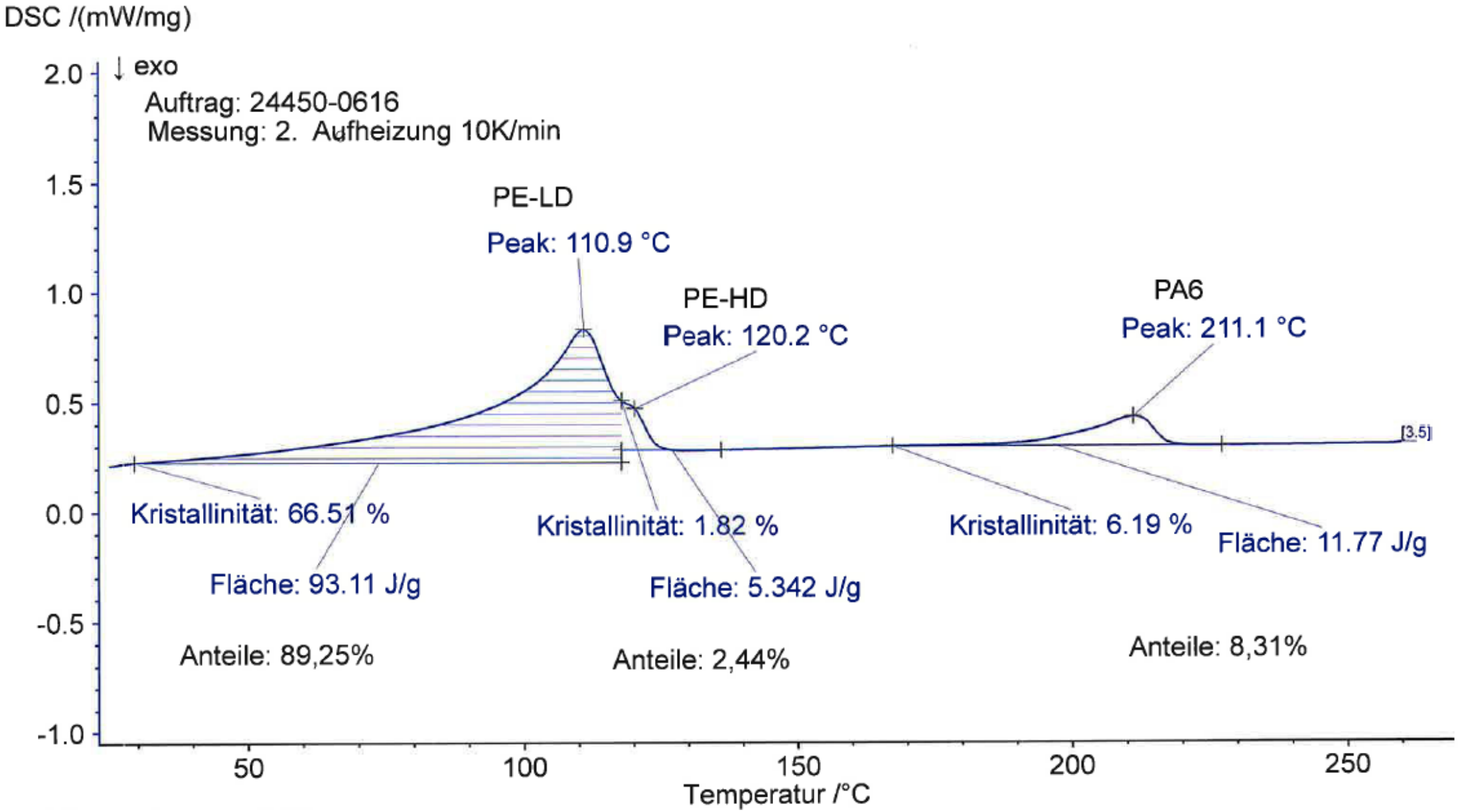
4. Annex | DSC (PCK-S1 | Cooling)



4. Annex | DSC (RG-S1 | 1. Heating)



4. Annex | DSC (RG-S1 | 2. Heating)



4. Annex | DSC (RG-S1 | Cooling)