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Brøndby,17.09.2013

Report

Case no.	Report no.	Prep. by	Page	No. of pages incl. 3 enclosures
113-28655	2	MLMA/hld	1	21

This report replaces the report: 113-28655 report 1 on the 12.09.2013

Analysis of bullets for softguns

Background

FORCE Technology received 5 bullets for softguns on the 22.08.2013 for analysis by FTIR-ATR, TGA and SEM-EDX. The bullets were designated:

- 16171 (reference bullet)
- 17290 (0.20 g bullet – Containing PLA)
- 17291 (0.25 g bullet – Containing PLA)
- 16178 (0.36 g bullet – Brown)
- 16290 (0.43 g bullet – Black)

I order to avoid analysing on the grease from the mould during manufacturing of the bullets, the outer shell was removed before analysis.

Results

FTIR-ATR

The 5 bullets were analysed with FTIR-ATR (Fourier Transform InfraRed - Attenuated Total Reflectance) spectroscopy to identify the polymer type. The spectra are represented in enclosure 1.

Bullet no. 16171, 16178 and 16290 consists of polystyrene (PS) – Additionally bullet no. 16178 shows an indication of a content of polyurethane. Bullet no. 17290 and 17291 both consist of polylactic acid (PLA) and calcium carbonate. Furthermore bullet no. 17291 has an indication of magnesium silicate.

TGA

TGA (Thermogravimetric Analysis) was conducted on each bullet in order to perform compositional analysis to determine the amount of polymer and fillers according to ASTM E1131-08. Duplicate determinations were performed on each bullet. The TGA curves can be seen in enclosure 2. Table 1 shows the composition of the bullets.

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Table 1 - Compositional analysis

Bullet	Component	% mass loss
16171	Polymer	41.8±0.1
	Fillers	0.6±0.0
	Ash	57.6±0.1
17290	Polymer	36.5±0.0
	Fillers	27.4±0.0
	Ash	36.1±0.0
17291	Polymer	27.6±0.0
	Fillers	10.7±0.4
	Ash	61.7±0.4
16178	Polyurethane	3.5±0.2
	Polystyrene	11.6±0.5
	Ash	84.9±0.7

The reference bullet (16171) contained 41.8 wt% polymer. The PLA content in bullet no. 17290 and 17291 is 36.5 wt% and 27.6 wt%, respectively.

For bullet no. 16178 it is possible to determine the polyurethane and polystyrene part, 3.5 wt% and 11.6 wt%, respectively. Bullet no. 16290 has an indication of a small bump around 320 °C, but no weight loss is observed. Normally, a weight loss in the first part of the curve is representing the highly volatile matter. The weight loss at 450 °C is probably polystyrene (10.8 wt%). It is not possible to determine the fillers for bullet no. 161780 and 16290 or ash for bullet no. 16290, which is caused by the high iron content. For further investigation of the two bullets the ash content was determined; see Table 2.

Table 2 - Ash content

Bullet	Ash content wt%
16178	93.4±1.0
16290	100.5±0.2

The ash determination of bullet no. 16178 and 16290 gives a content of 93.4 and 100.5 %, which means the bullets mainly consist of inorganic material. The ash content of bullet no. 16290 should not have exceeded 100 %, which would lead to the conclusion that the bullet only consist of inorganic material. Comparing the ash content of the two different methods of analysis, there is a difference in the amount, which is due to the high iron content.

SEM-EDX

The chemical composition of the bullets has been determined by SEM-EDX (Scanning Electron Microscope combine with Energy-Dispersive X-ray Spectroscopy) analysis, giving a semiquantitative elemental analysis. All the bullets displayed inhomogeneities -The content of these inhomogeneities were also determined by SEM-EDX. Table 3 shows the chemical composition of the inhomogeneities in the bullets. The full SEM-EDX report for each bullet can be seen in enclosure 3.

Table 3 - Chemical composition of inhomogeneities

Bullet	Component	Wt%
16171	C	21.9
	O	18.0
	Sr	02.4
	S	10.6
	Ba	47.0
17290	C	23.7
	O	35.8
	Ca	40.5
17291	C	16.6
	O	17.5
	Sr	00.9
	S	11.1
	Ca	00.5
	Ba	53.4
16178	C	17.7
	Si	00.8
	Mn	00.9
	Fe	80.6
16290	C	19.5
	Si	00.6
	Mn	00.8
	Fe	79.1

Based on the SEM-EDX analysis and within the detection limit of the method the bullets do not contain any heavy metals. Bullet no. 17290 and 17291 is comparable, even though bullet no. 17291 contains compounds of strontium, sulphur and barium besides carbon, oxygen and calcium. Bullet no. 16178 and 16290 contains 80.6 and 79.1 weight % iron, respectively, which is responsible for the weight of the bullets. Besides the iron content the bullets contain compounds of carbon, silicium and barium. Furthermore, these bullets were highly magnetic, both before and after the ash determination, caused by the high iron content.

Conclusion

Bullet no. 17290 and 17291 consist of polylactic acid, with a content of 36.5 wt% and 27.6 wt%, respectively. Bullet no. 16171, 16178 and 16290 consist of polystyrene. Furthermore bullet no. 16178 has an indication of polyurethane. Bullet no. 16178 and 16290 do not contain any heavy metals, within the detection limit. The weight of the bullets is caused by the high iron content. It is possible that the high content of iron has affected the different analysis.

FORCE Technology

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Polymer Specialist
Chemistry and Plastics

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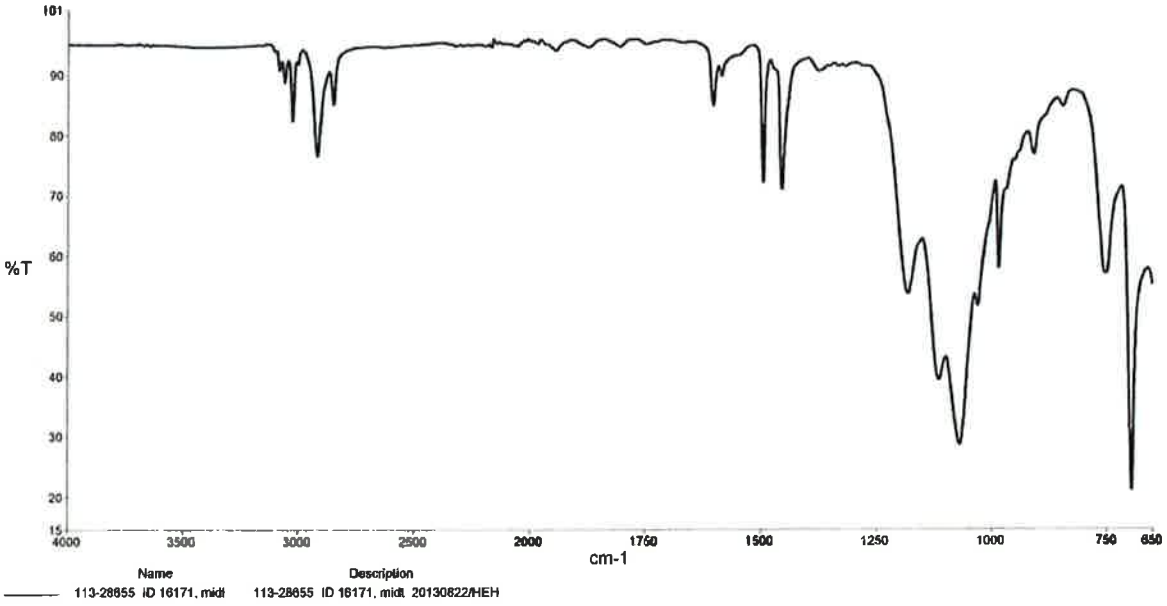
16171



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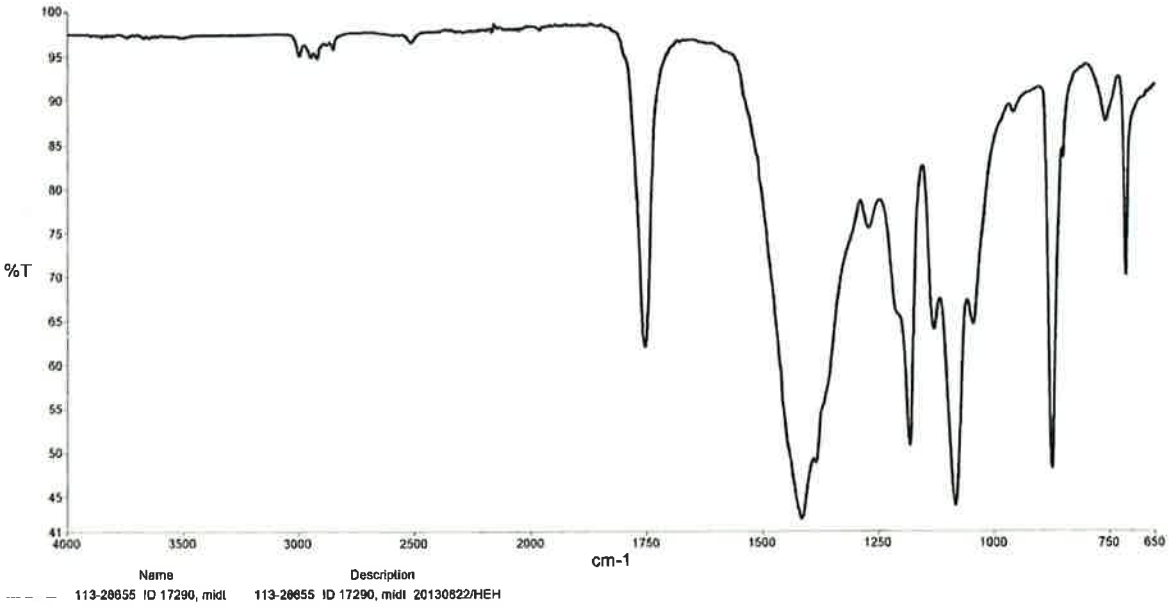

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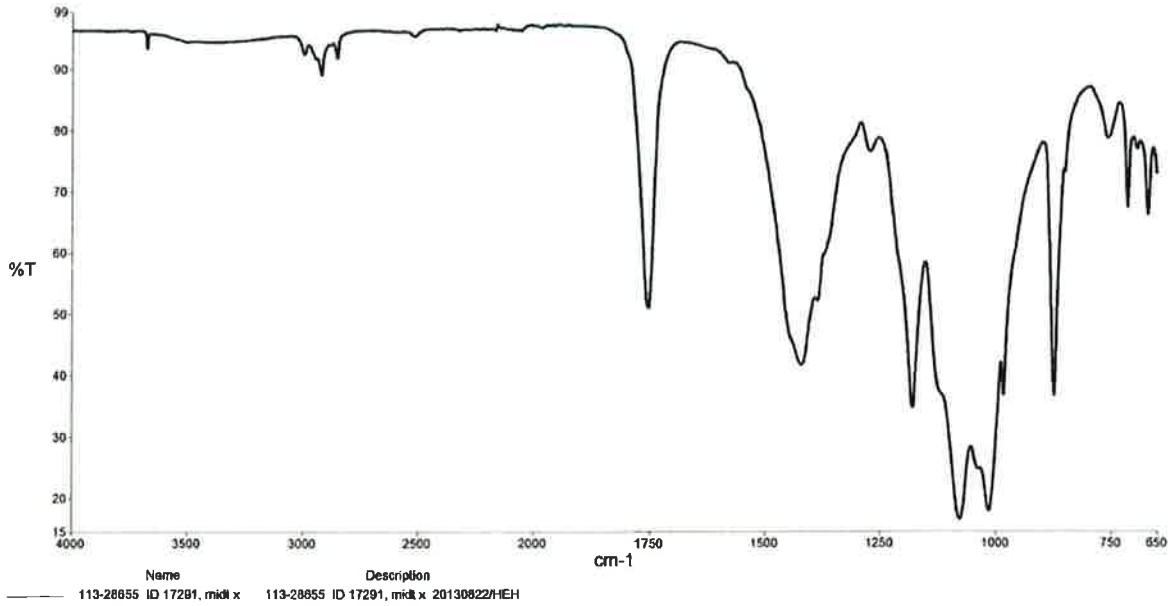

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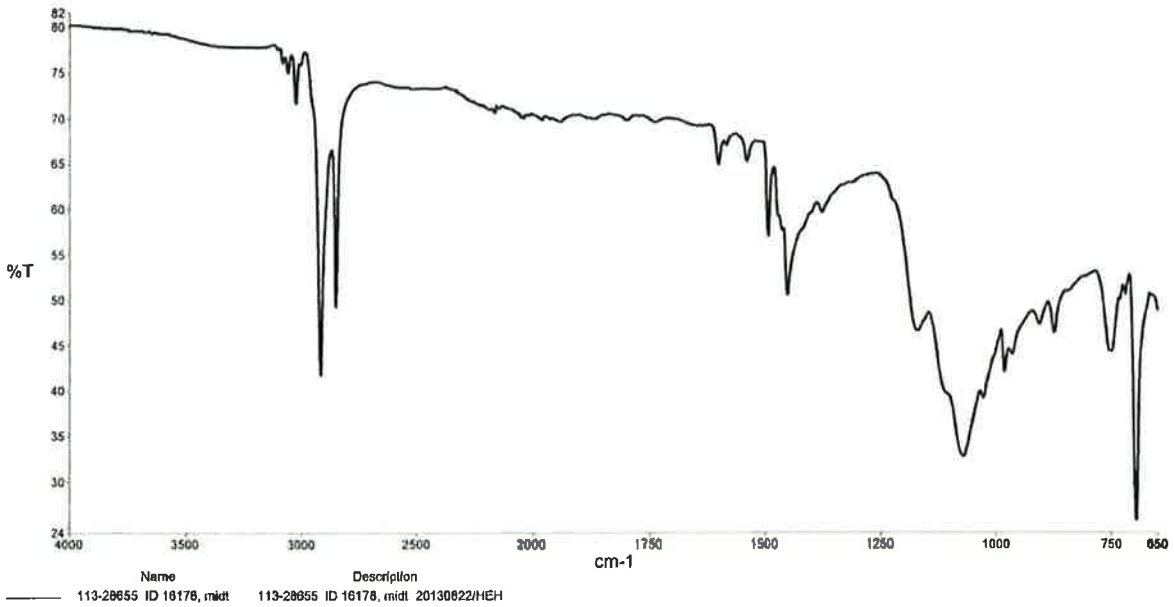

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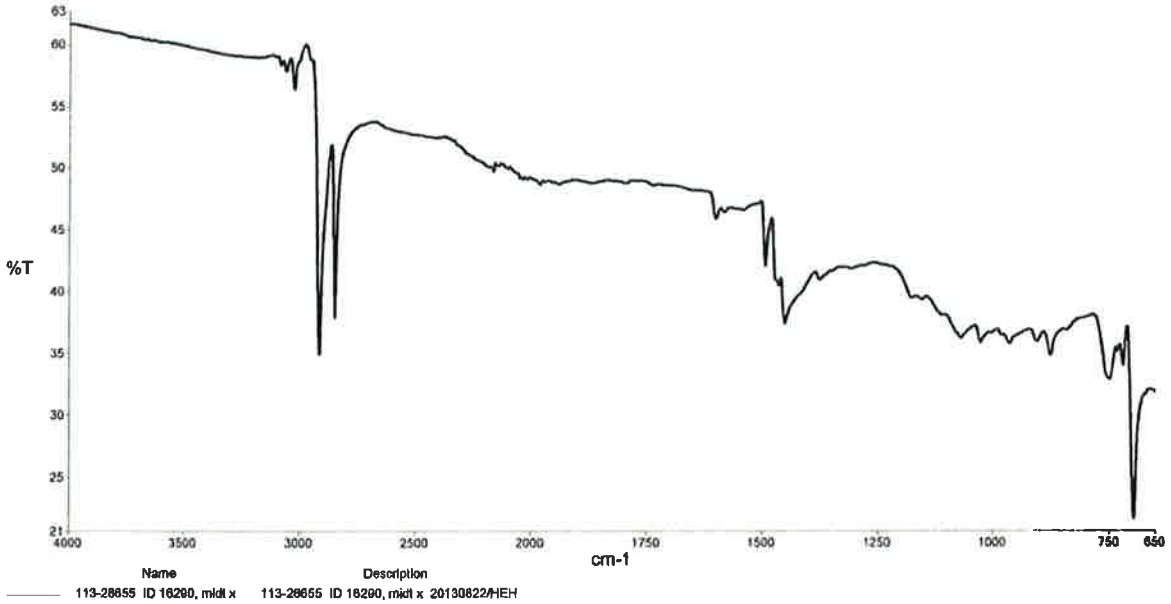
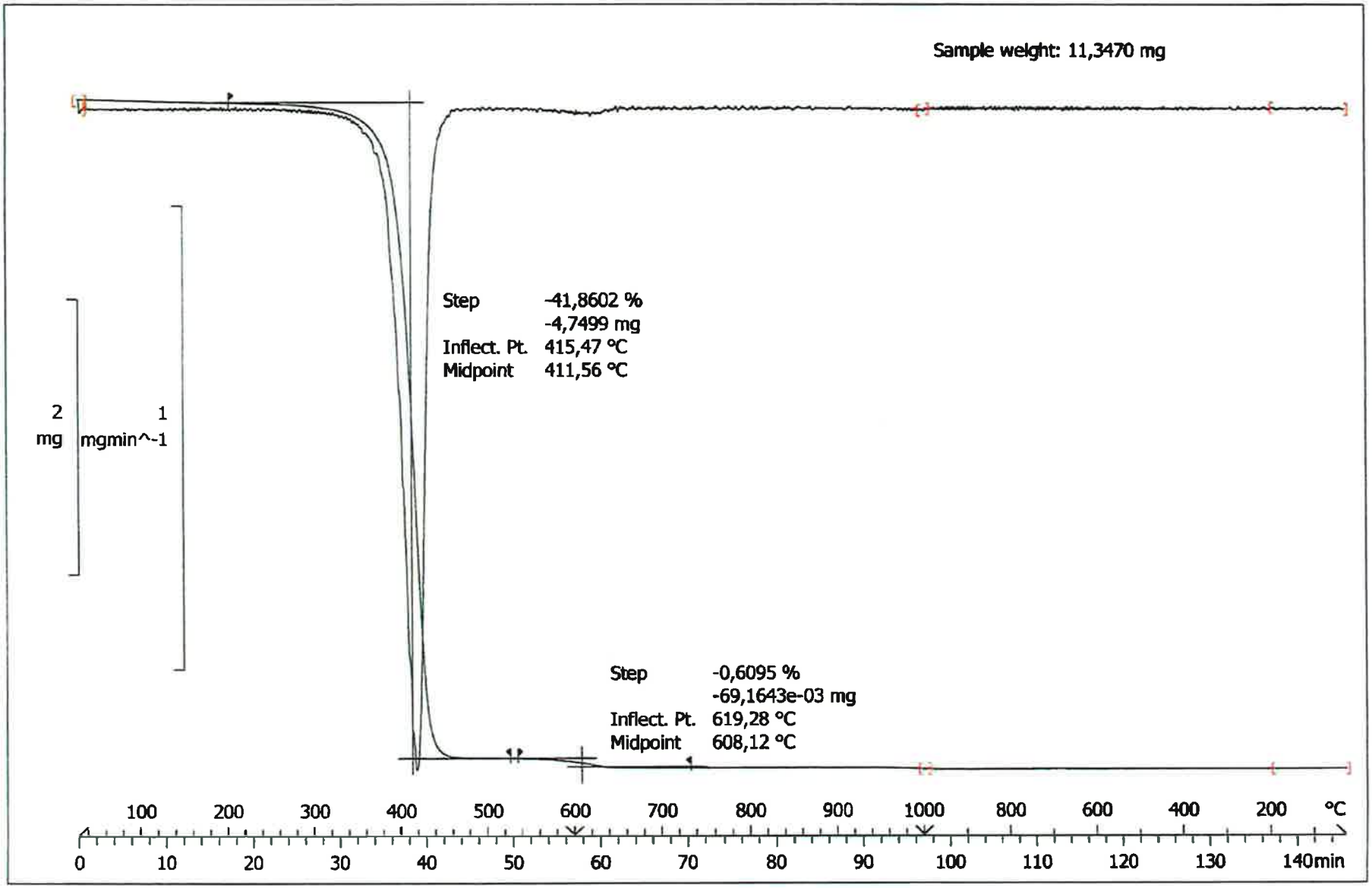


Enclosure 2

^exo 113-28655 ID: 16171 A 03.09.2013 14:22:37



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Enclosure 2

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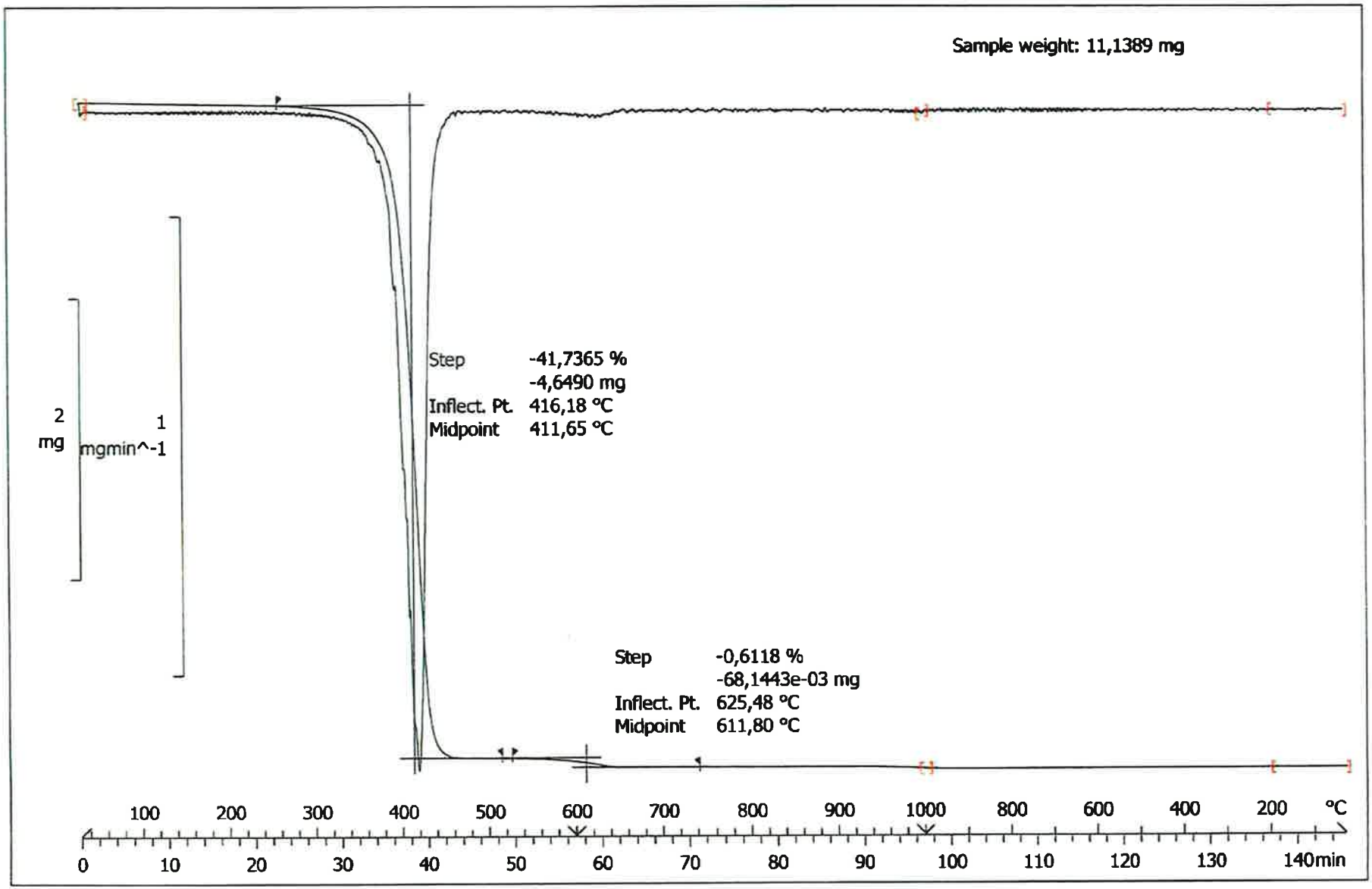

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Enclosure 2

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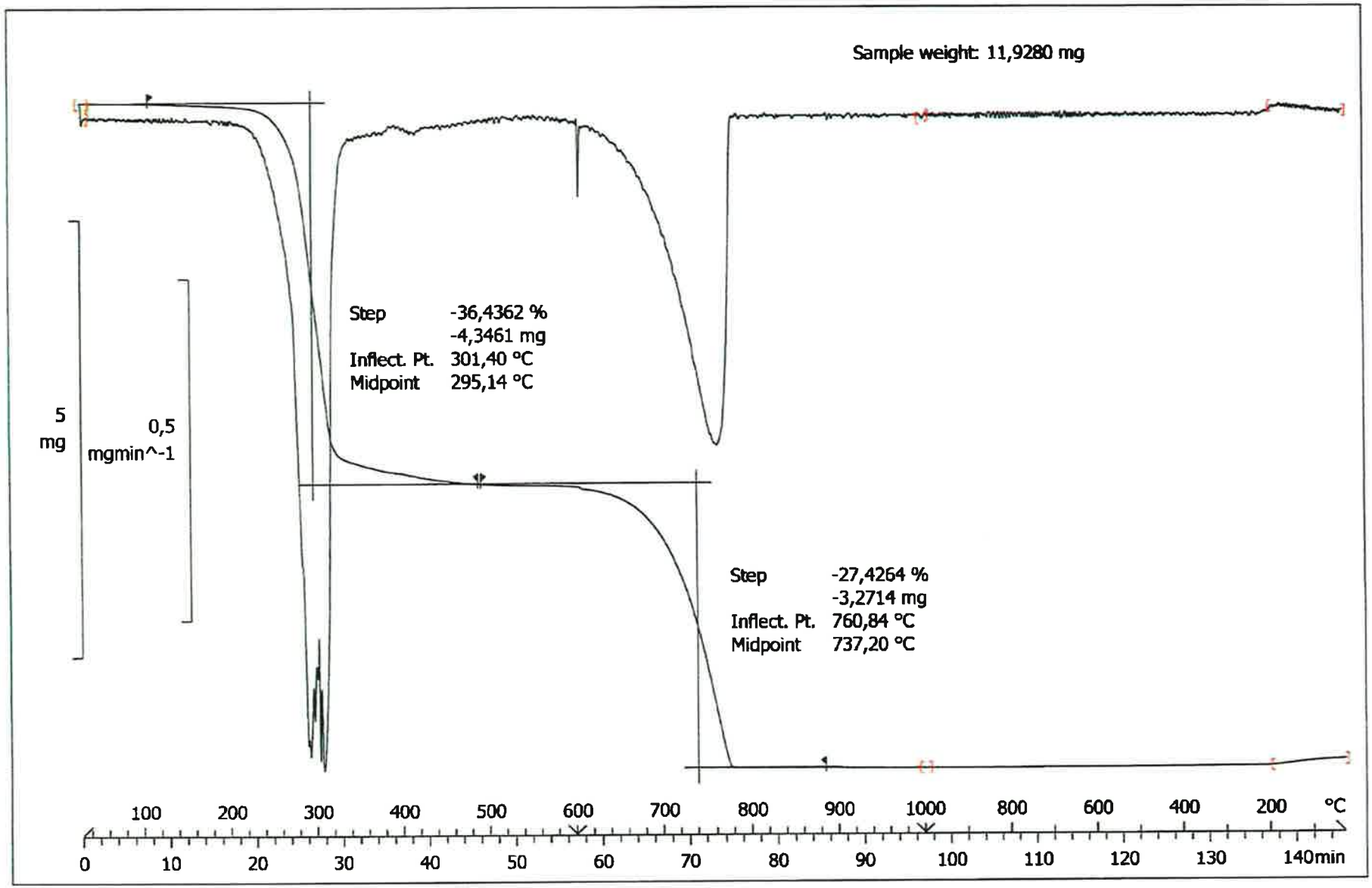

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Enclosure 2

^exo

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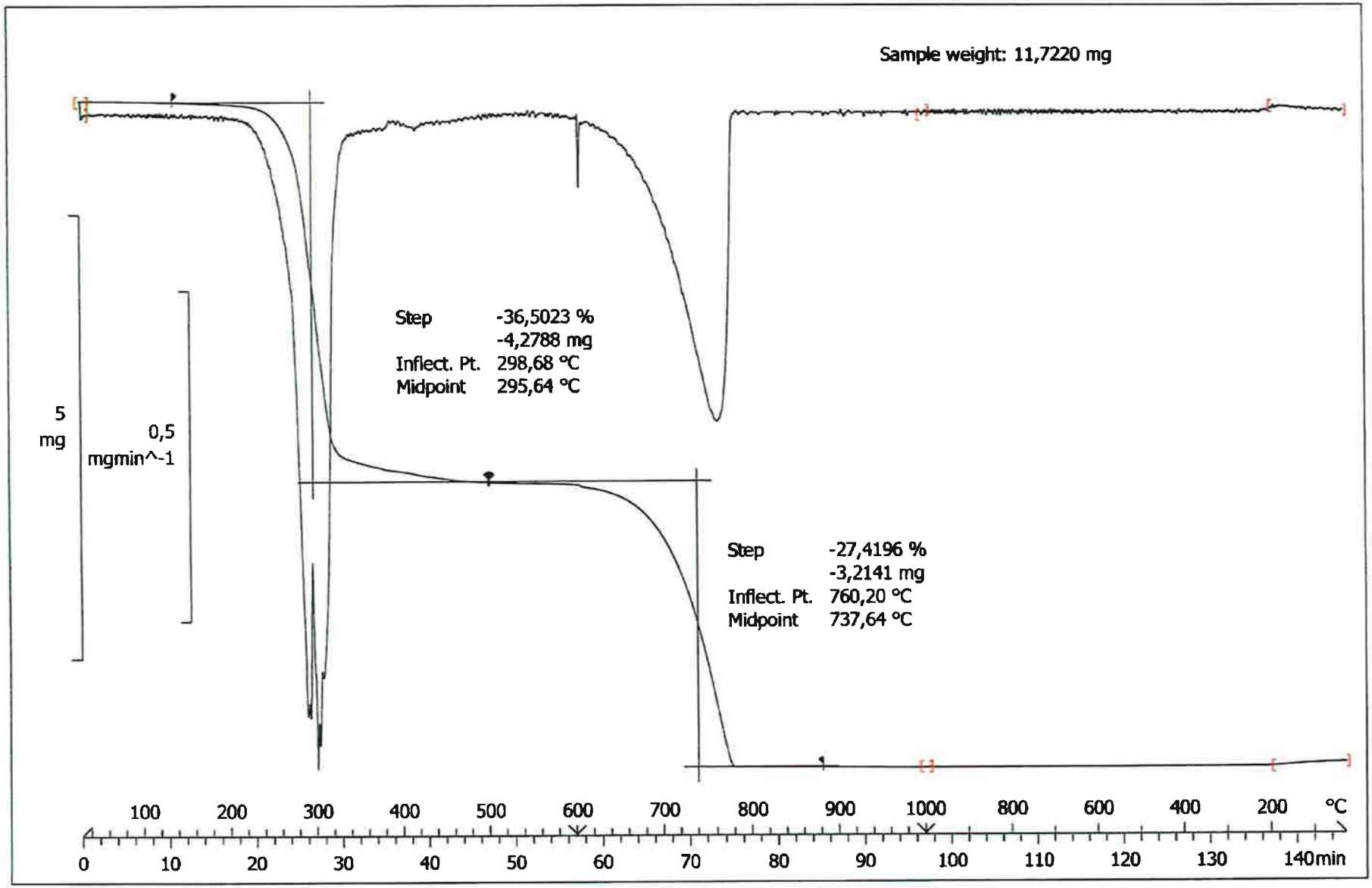

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Enclosure 2

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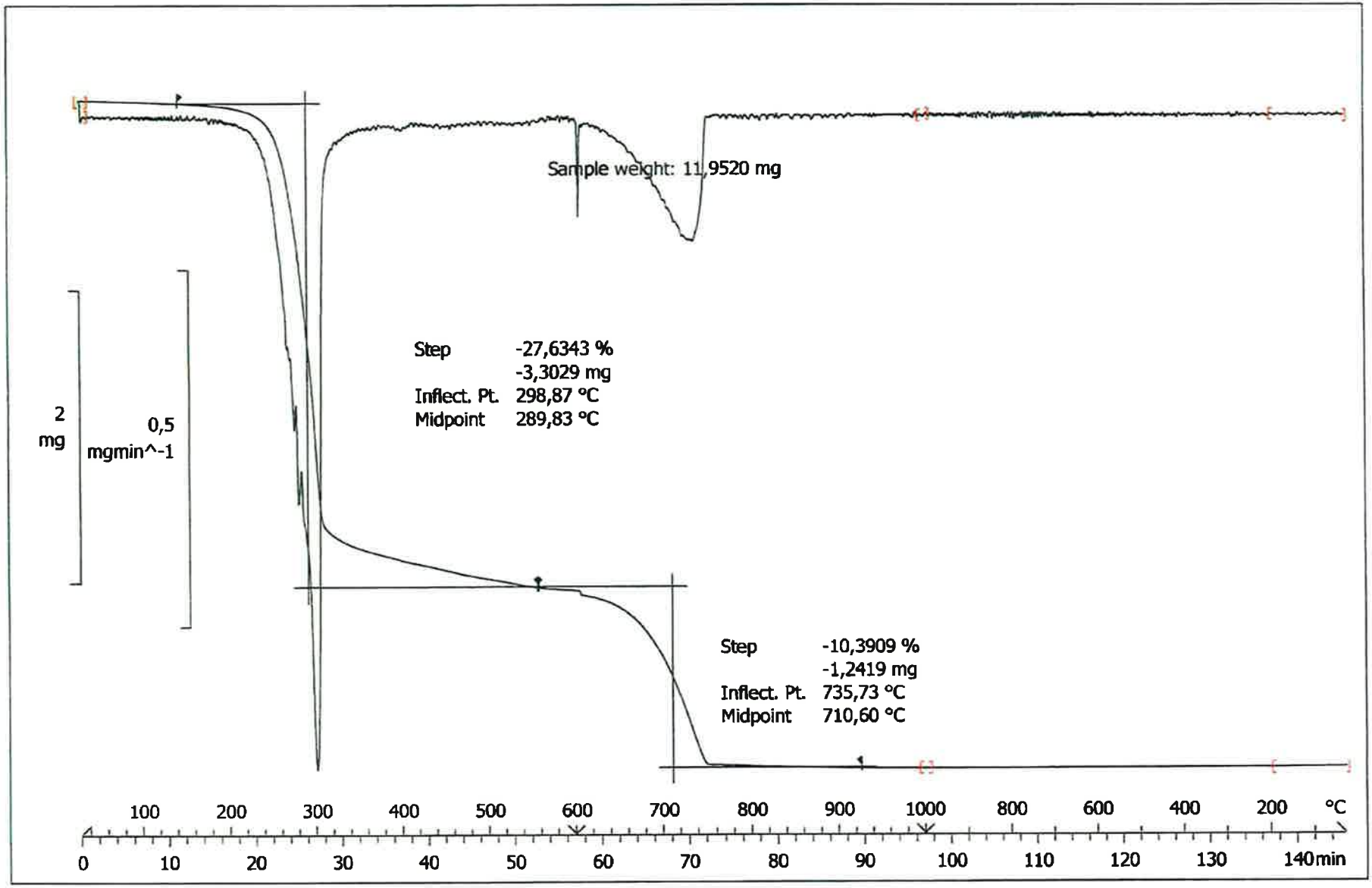

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Enclosure 2

^exo

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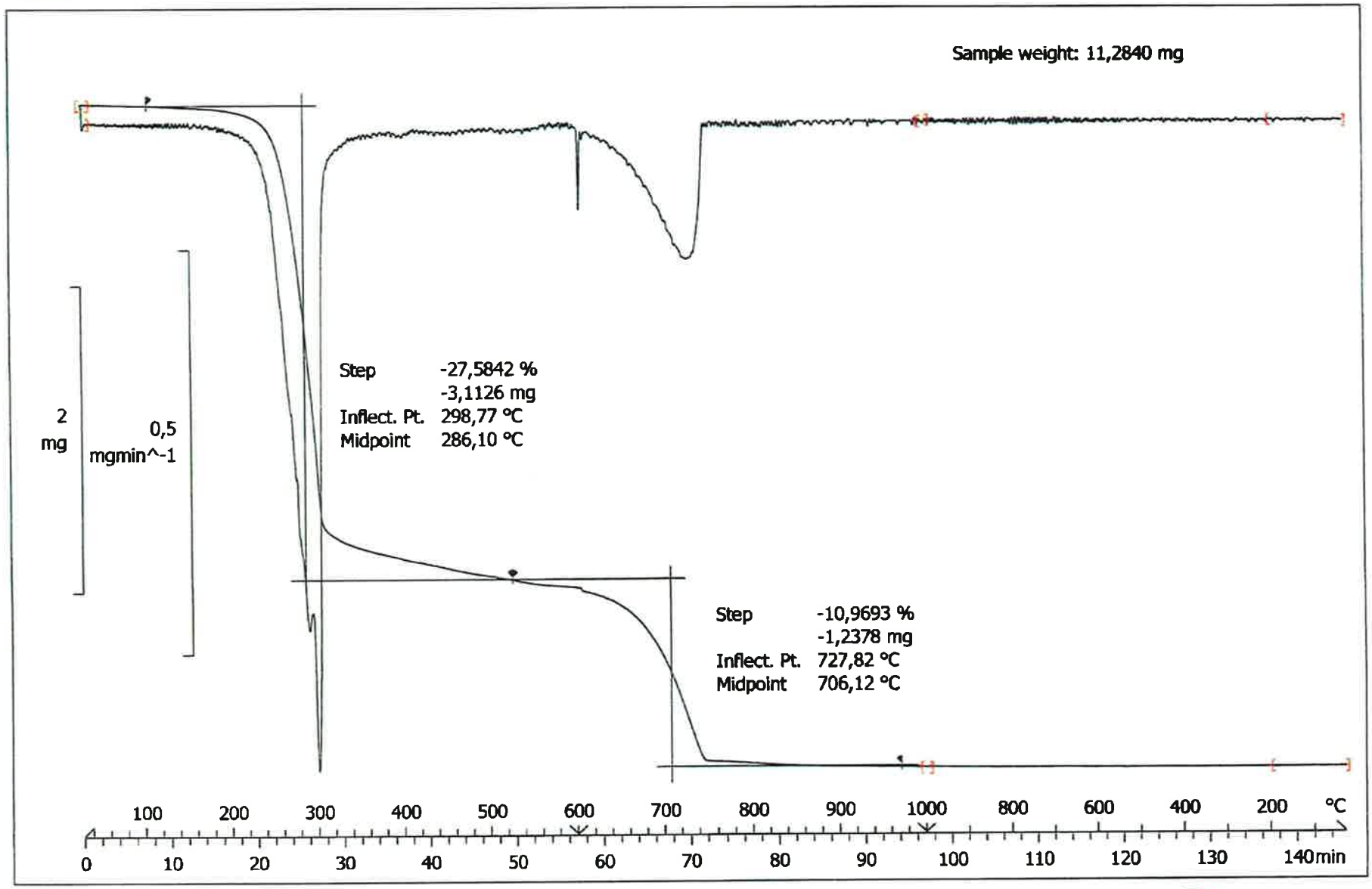

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Enclosure 2

^exo

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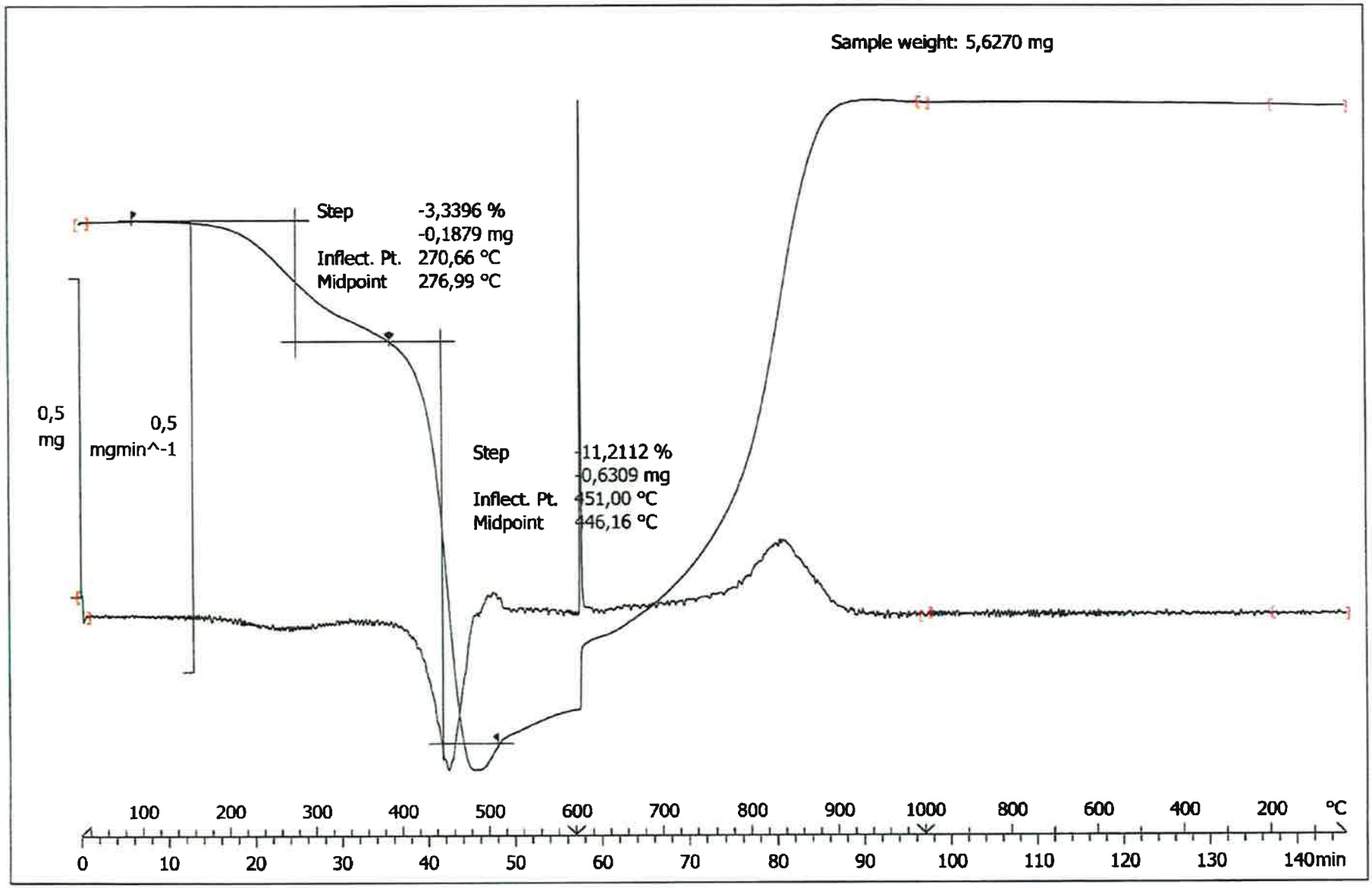

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^exo

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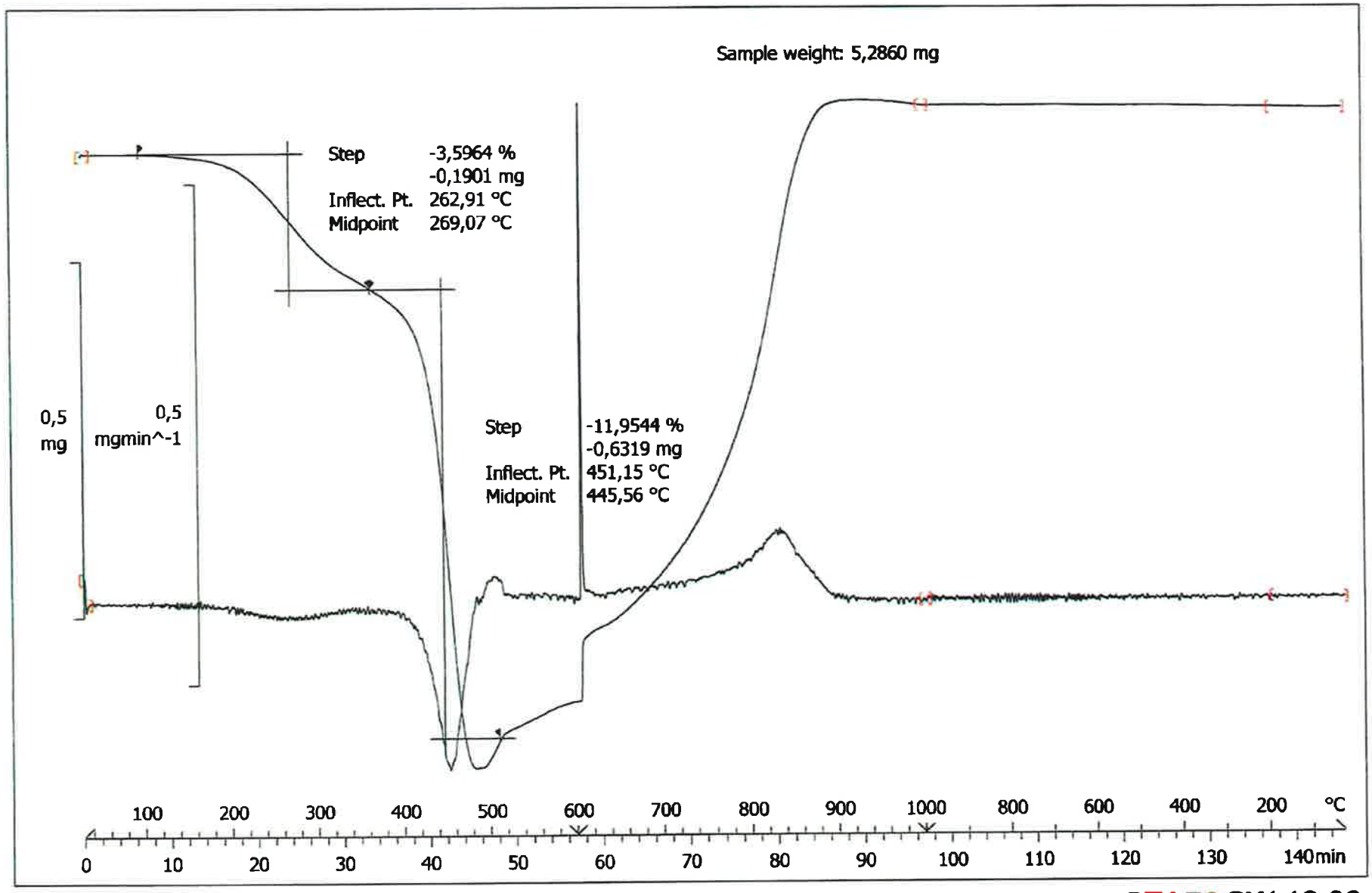

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Enclosure 2

^exo

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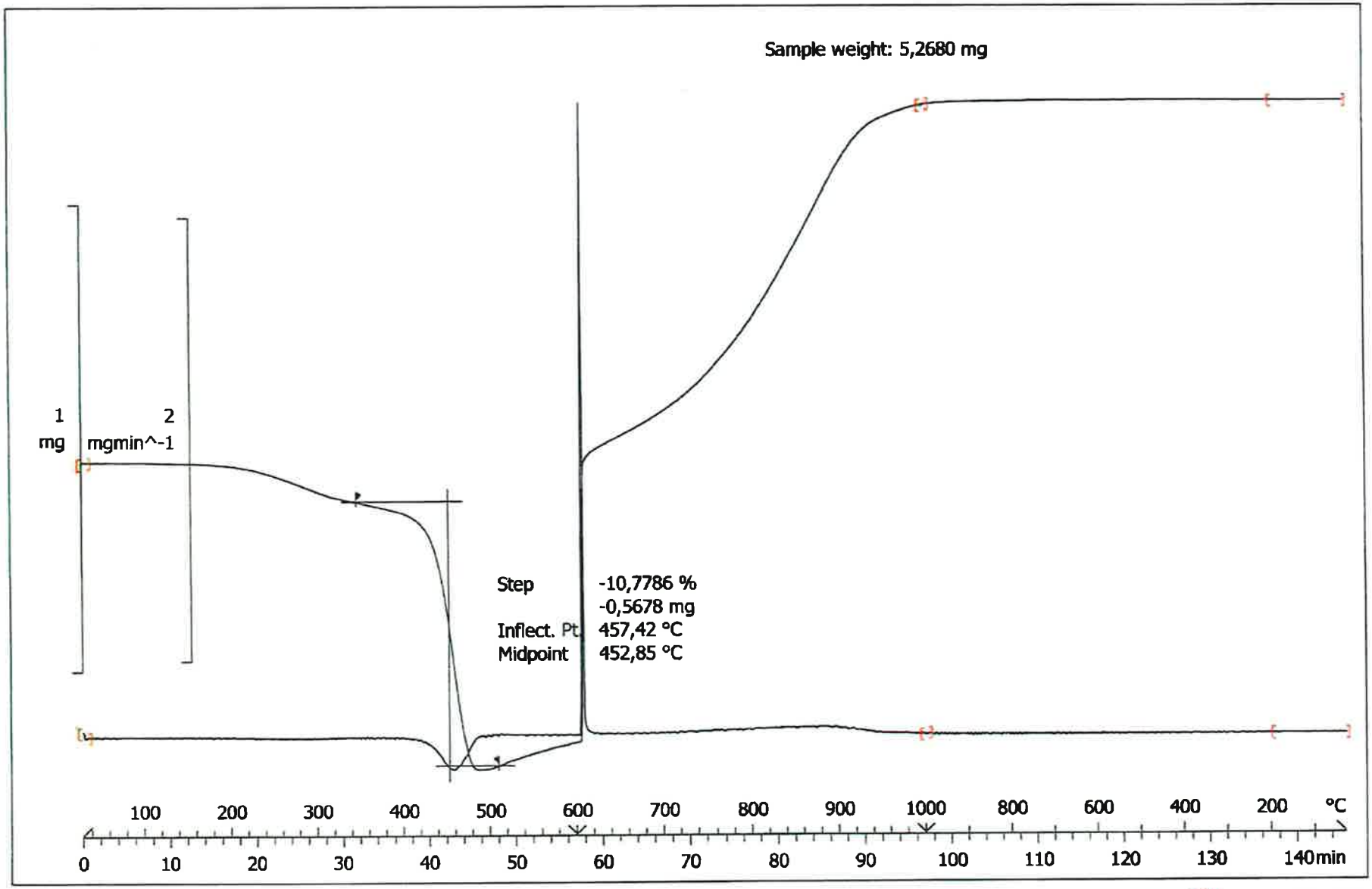

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Enclosure 2

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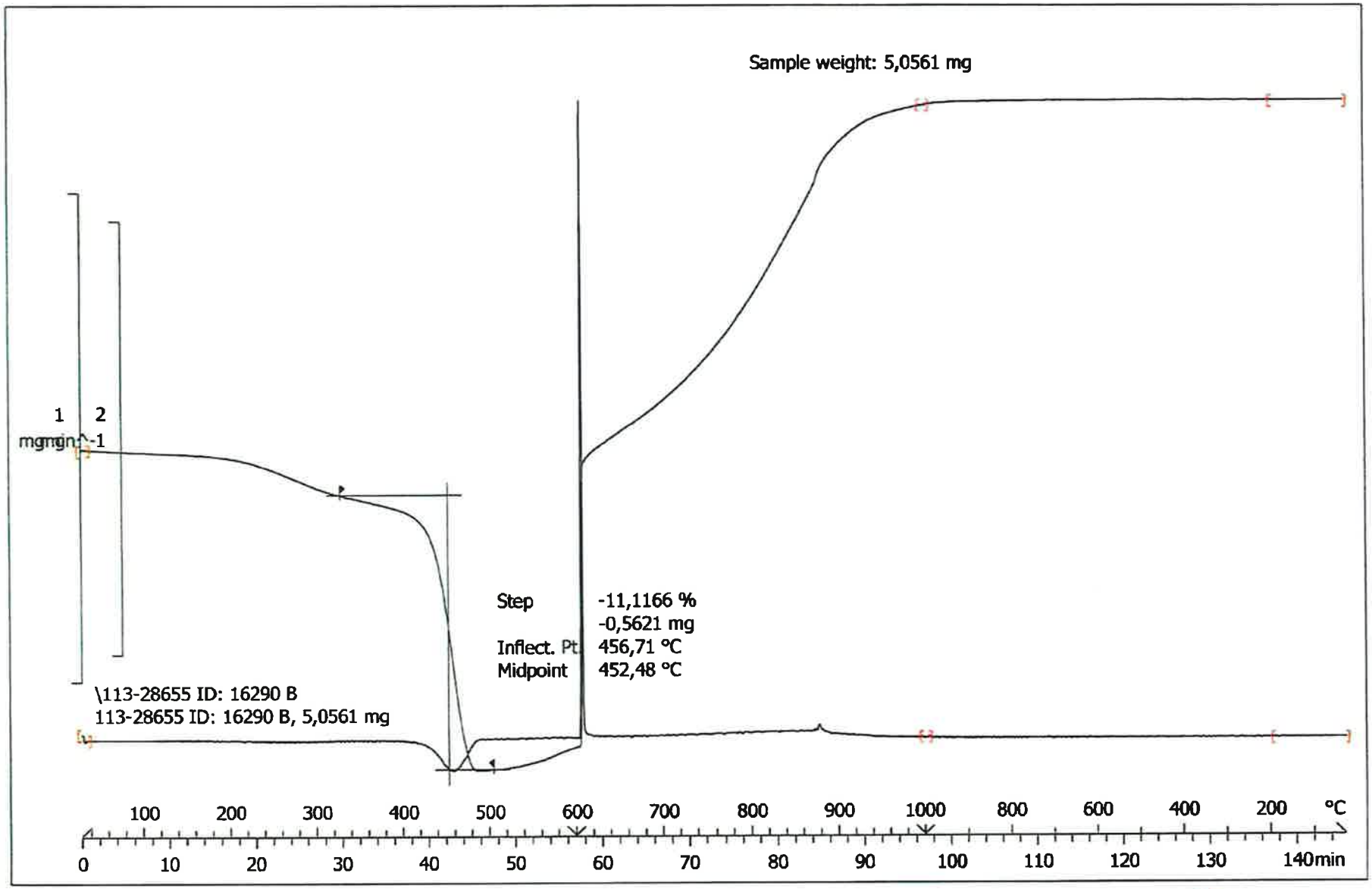

FORCE Technology : Kemi & Plast

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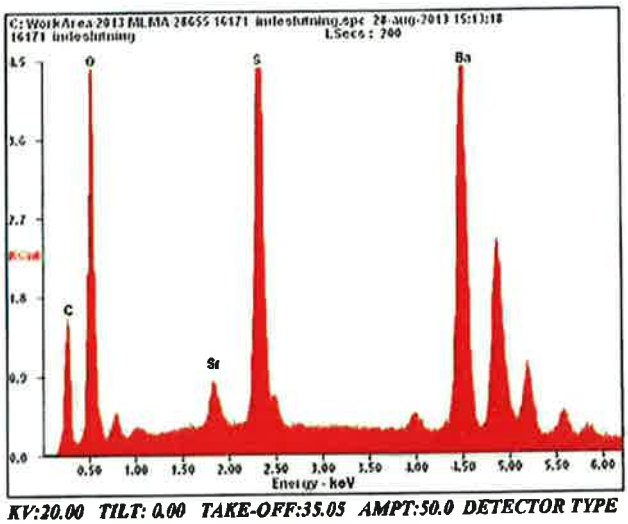
Sags nr: 13-28655 **MLMA/PJ**

16171 indeslutning

28-Aug-2013



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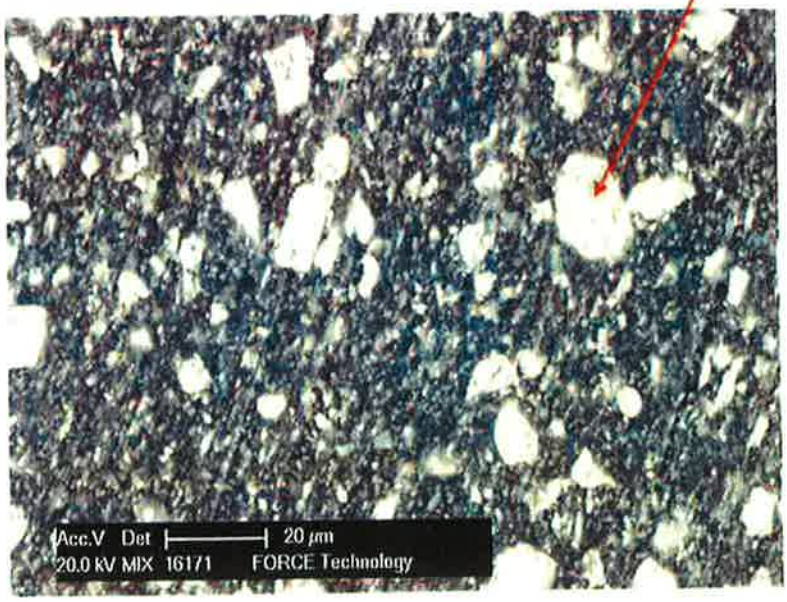
Element	Wt %	At %
C K	21.97	50.04
O K	18.01	30.80
SrL	02.39	00.75
S K	10.60	09.05
BaL	47.02	09.37

EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : DEFAULT



Den energidispersive røntgen analyse er en semi-kvantitativ grundstofbestemmelse.
Resultaterne skal følgelig tages med dette forbehold.

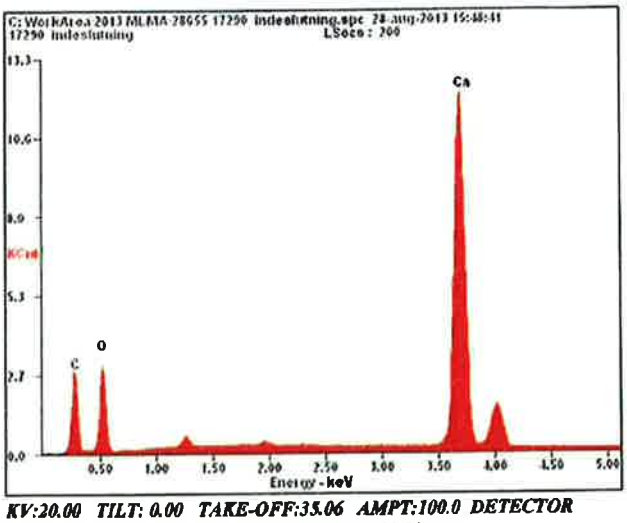
Kemisk Analyse

Karina Brotoft / Pia Hansen /
Jacob B. Markussen / Thomas P. Pedersen

Sags nr: 113-28655 **MLMA/PJ**

17290 indeslutning:

28-Aug-2013



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Element	*Wt %*	*At %*
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O K	35.78	42.82
CaK	40.48	19.34

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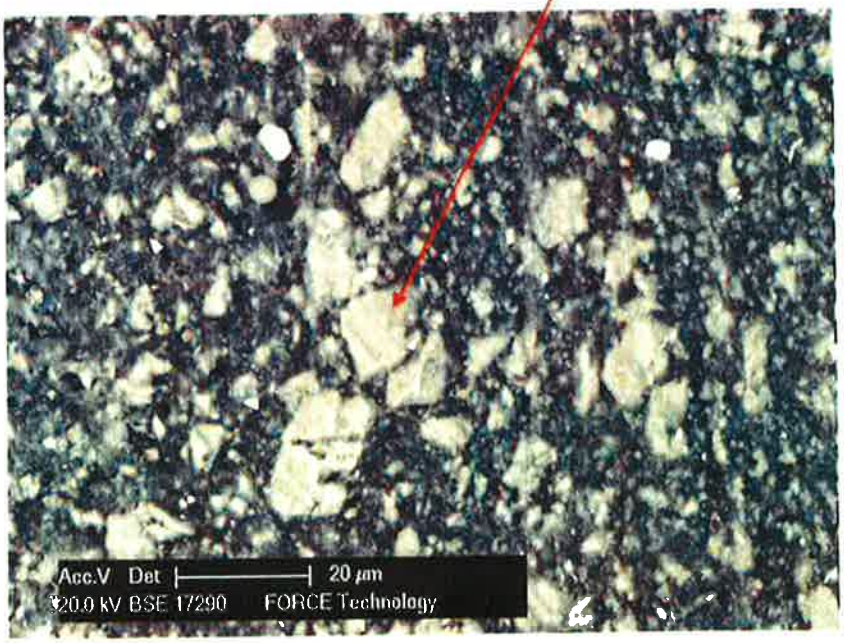

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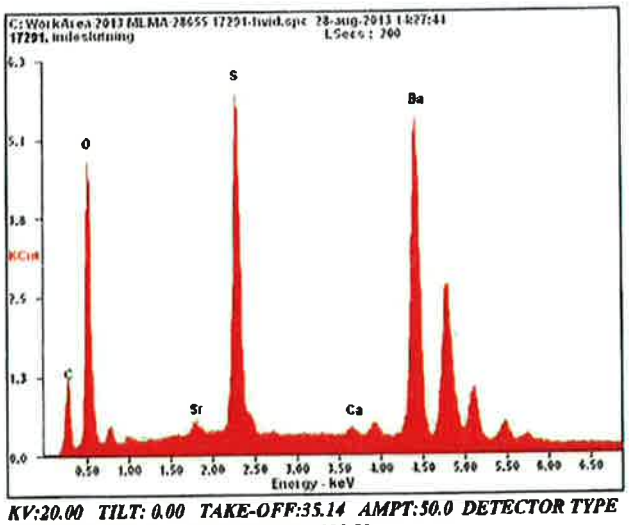
Kemisk Analyse

Karina Brotoft / Pia Hansen /
Jacob B. Markussen / Thomas F. Pedersen

Sags nr: 113-28655 **MLMA/PJ**

17291 indeslutning:

28-Aug-2013



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C K	16.56	42.68
O K	17.52	33.90
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CaK	00.47	00.36
BaL	53.41	12.04

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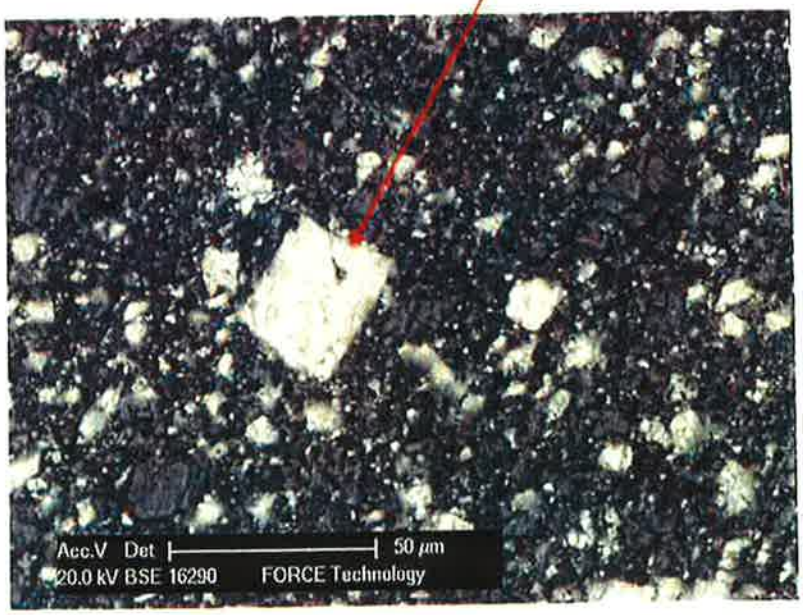


Den energidispersive røntgen analyse er en semi-kvantitativ grundstofbestemmelse.
Resultaterne skal følgelig tages med dette forbehold.

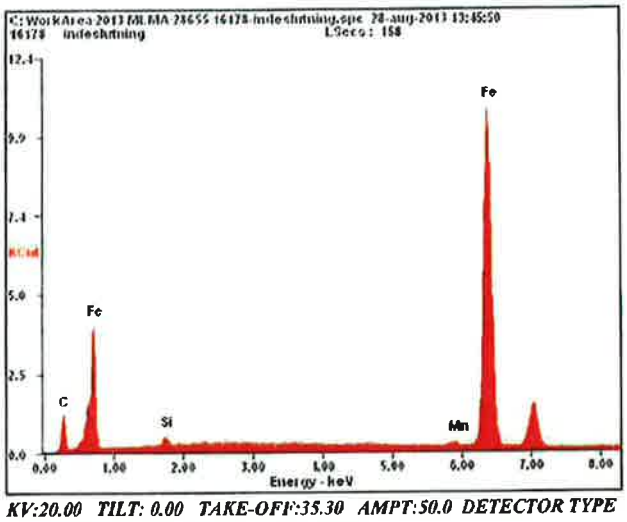
Kemisk Analyse

Karina Brotoft / Pia Hansen /
Jacob B. Markussen / Thomas F. Pedersen

Sags nr: 113-28655 **MLMA/PJ**

16178 indeslutning:

28-Aug-2013



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Element	*Wt %*	*At %*
C K	17.68	49.73
SiK	00.78	00.94
MnK	00.97	00.60
FeK	80.57	48.73

EDAX ZAF QUANTIFICATION STANDARDLESS SEC TABLE : DEFAULT

Den energidispersive røntgen analyse er en semi-kvantitativ grundstofbestemmelse.
Resultaterne skal følgelig tages med dette forbehold.

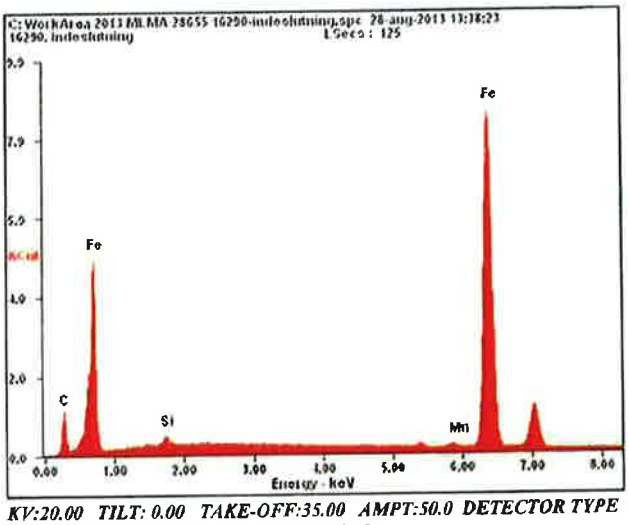
Kemisk Analyse

Karina Brotoft / Pia Hansen /
Jacob B. Markussen / Thomas F. Pedersen

Sags nr: 113-28655 **MLMA/PJ**

16290: indeslutning

28-Aug-2013



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Element	Wt %	At %
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MnK	00.79	00.47
FeK	79.12	46.07

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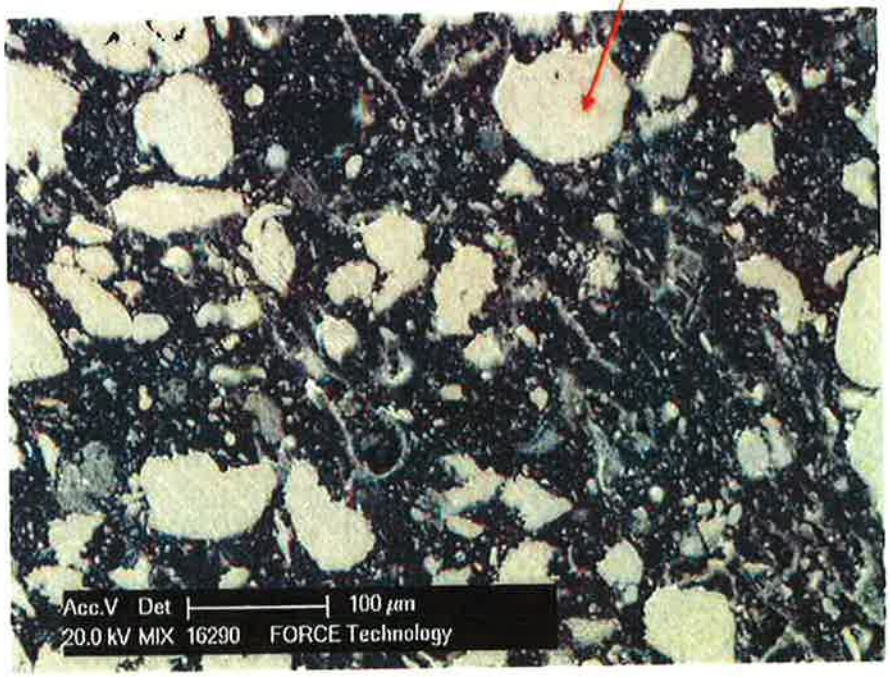

Den energidispersive røntgen analyse er en semi-kvantitativ grundstofbestemmelse.
Resultaterne skal følgelig tages med dette forbehold.

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Karina Brotoft / Pia Hansen /
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