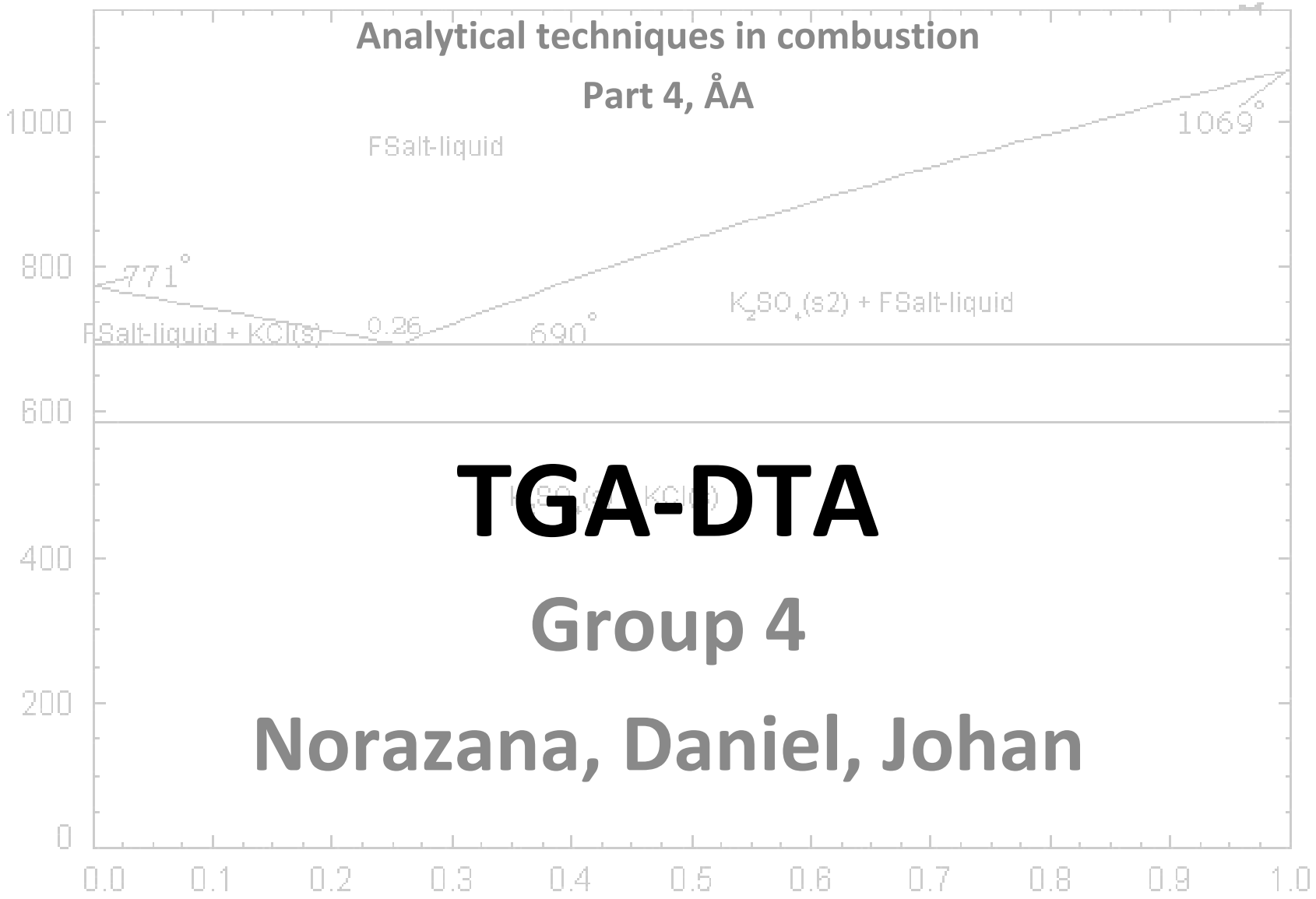
BiofuelsGS-2

Analytical techniques in combustion

Part 4, ÅA



TGA-DTA

Group 4

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Presentation outline

- Comparison of DTA and TGA techniques

- Group works

G1. Indium (DTA)
G3. Oxalate (TGA)
} Method approval

G2. KCl (TGA-DTA)
G5. K2SO4 (TGA-DTA)
G4. KCl-K2SO4 eut mix (TGA-DTA)
} Phase diagram

DTA-TGA

one instrument – two functions

DTA

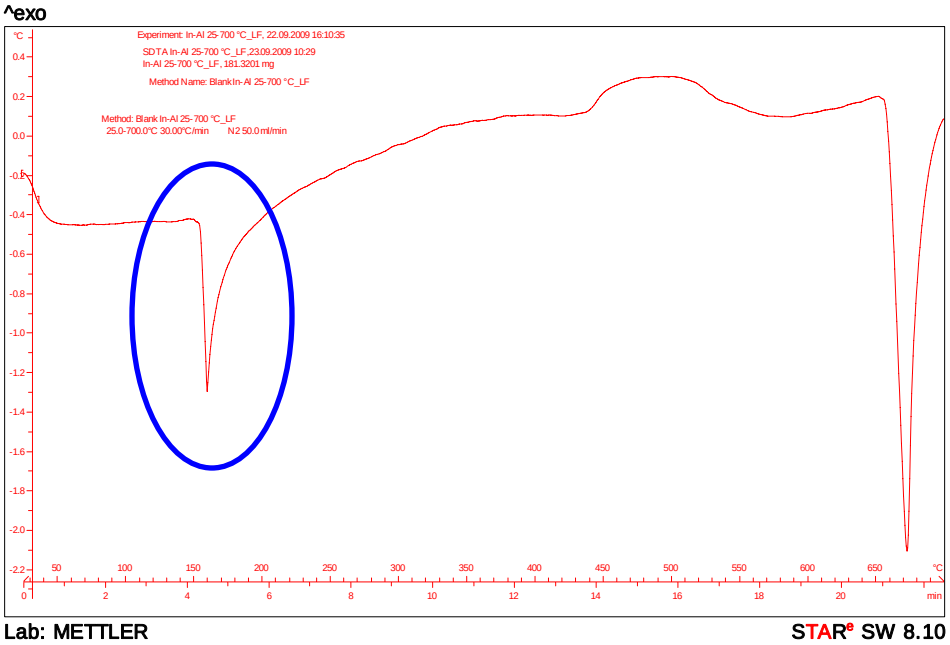
- Temperature difference
- Thermocouple
- Structural changes
  - Chrystallization
  - Melting
- Chemical reactions causing temperature changes

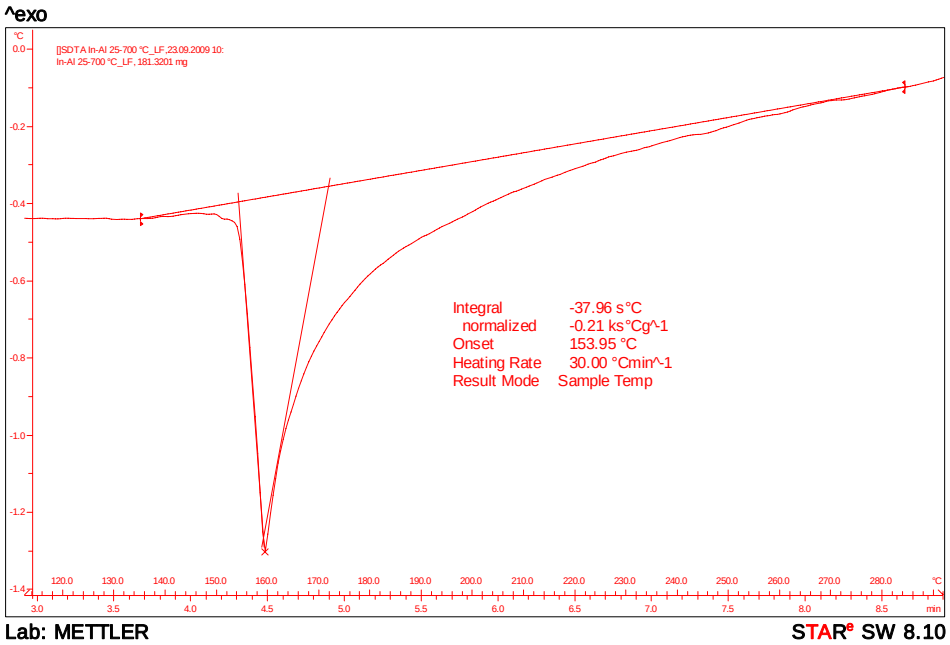
TGA

- Mass change
- Microbalance
- Mass change by phase transition
  - Vaporization
  - Sublimation
- Mass change by chemical reactions

Indium

- Standard for DTA



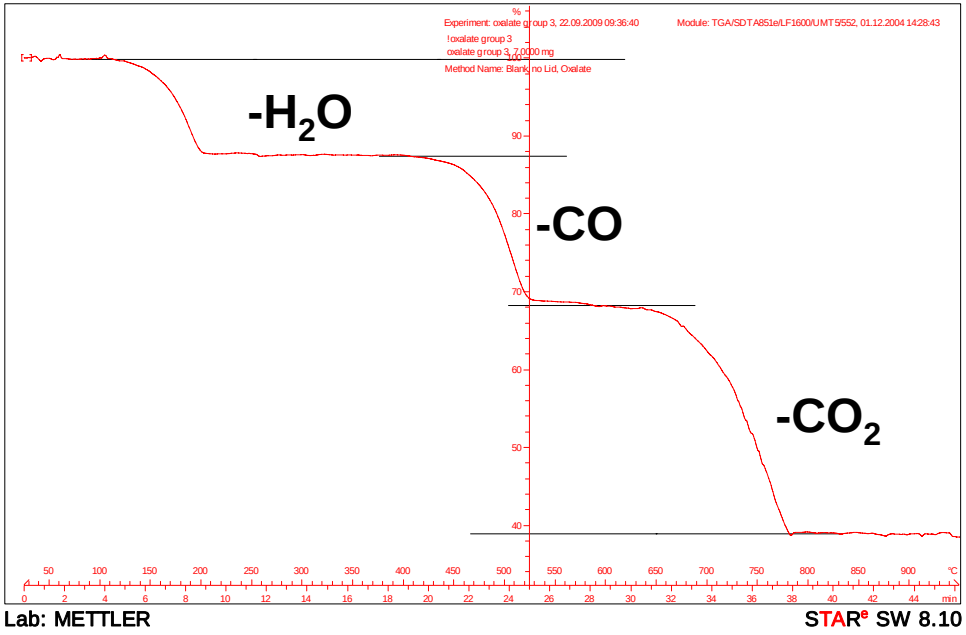


- Melting point:
  - Litterature: 156,6°C
  - Experimental: 153,95 °C

Calcium oxalate, $CaC_2O_4 \bullet H2O$

- Common material to show the TGA performance

- Step 1: release of water
- Step 2: release of CO
- Step 3: release of CO_2



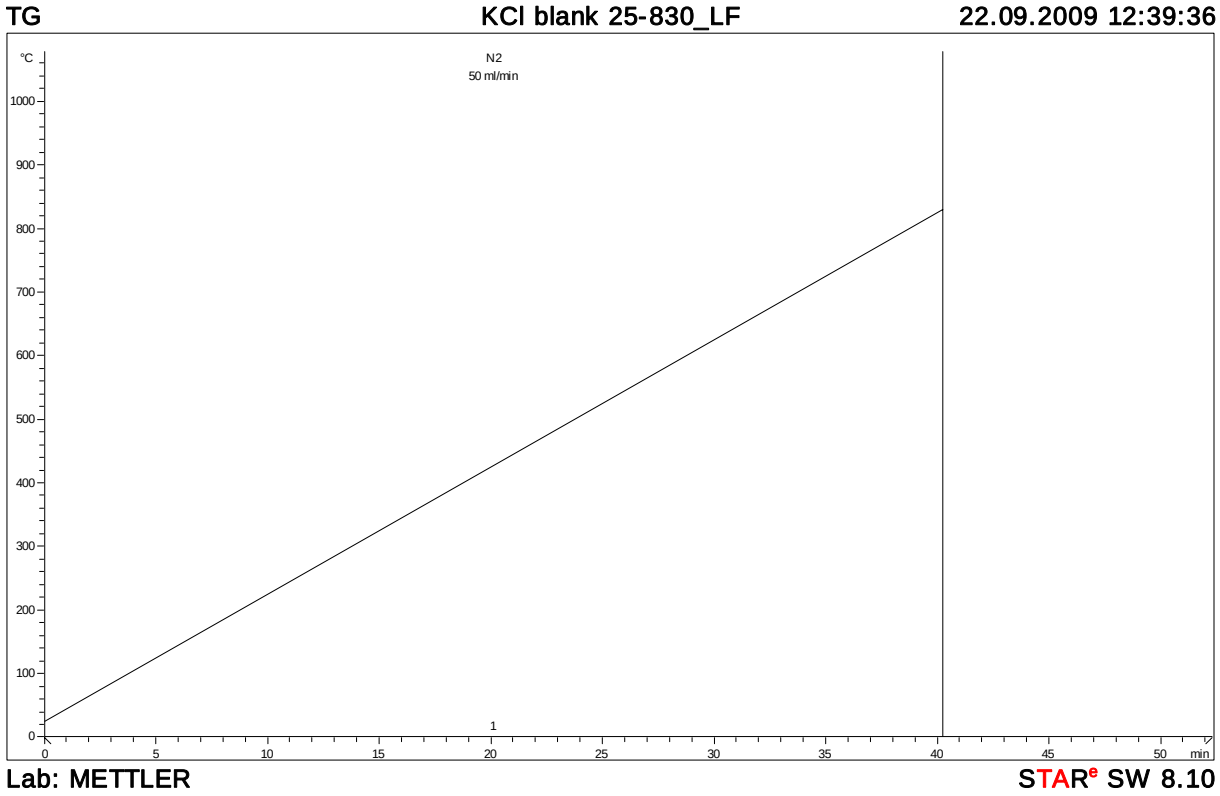
$KCl\text{-}K_2SO_4$ experiments

- In these experiments the 2 pure salts and an eutectic mixture were analyzed by DTA-TGA
- The results were compared with theoretical values
- In order to create a *complete* phase diagram of a salt mixture the melting behavior of *the pure salts and several mixtures* are needed.

Method KCl

Pure KCl

KCl – K_2SO_4 eutectic mixture

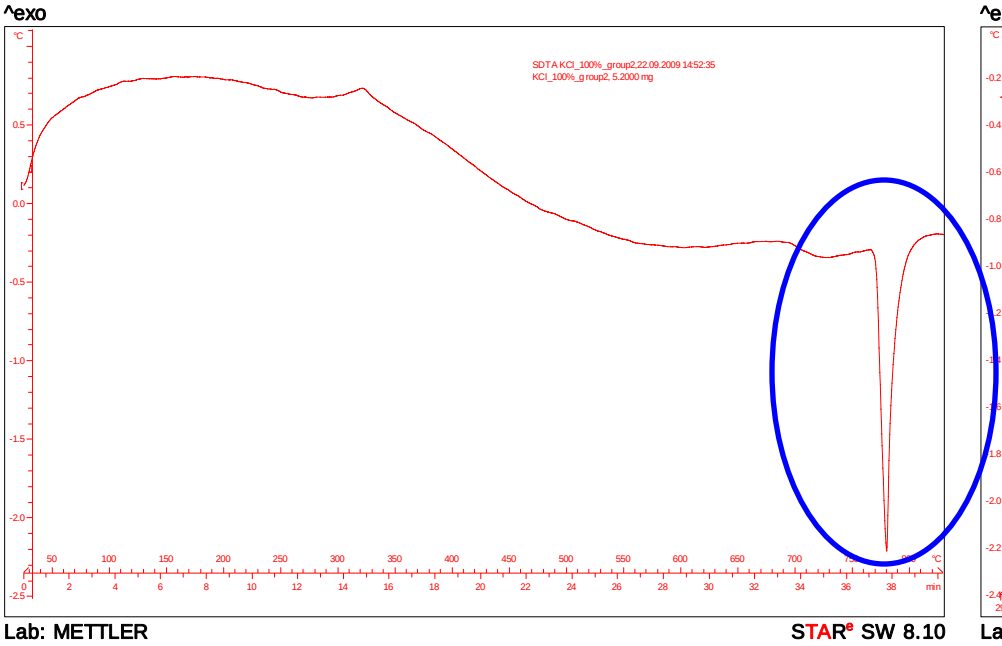


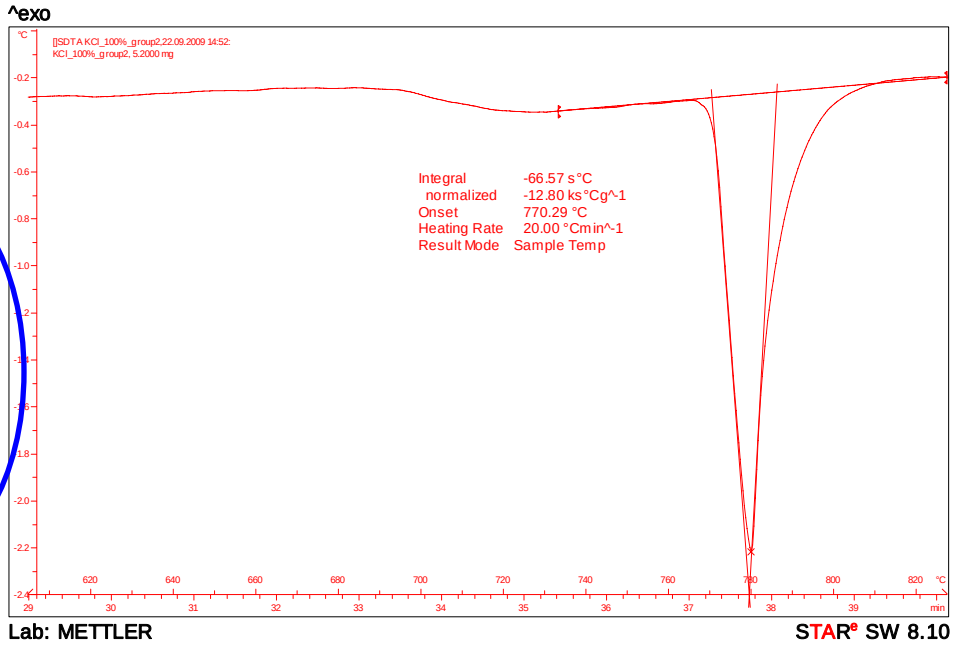
Expected
Mp(KCl): 771 °C
Mp(Eut.): 690 °C

Temp prog.
Start T: 25 °C
End T: 830 °C
Heating rate: 20 °C/min
Gas: N_2
Flow rate: 50 ml / min

Pure KCl

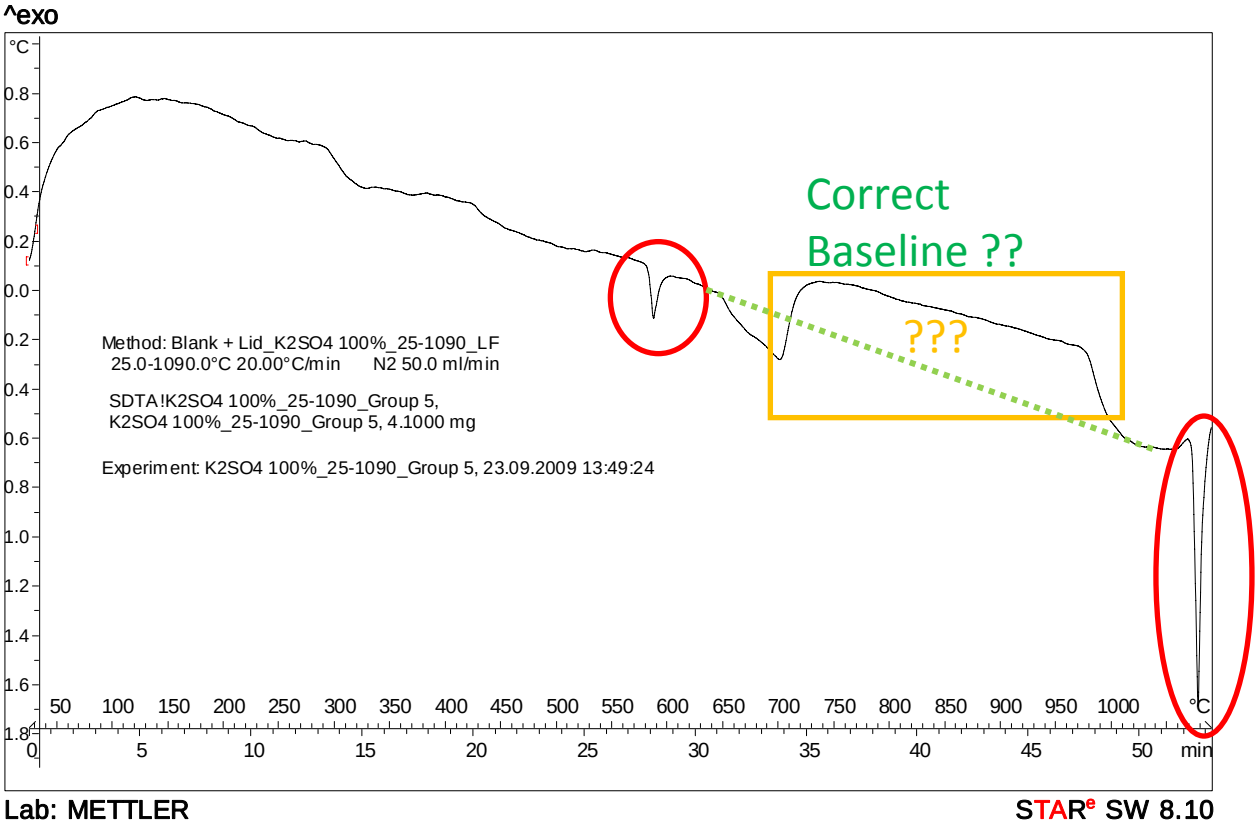
- Melting point:
  - Litterature: 771 °C
  - Experimental: 770,29 °C





Pure K_2SO_4

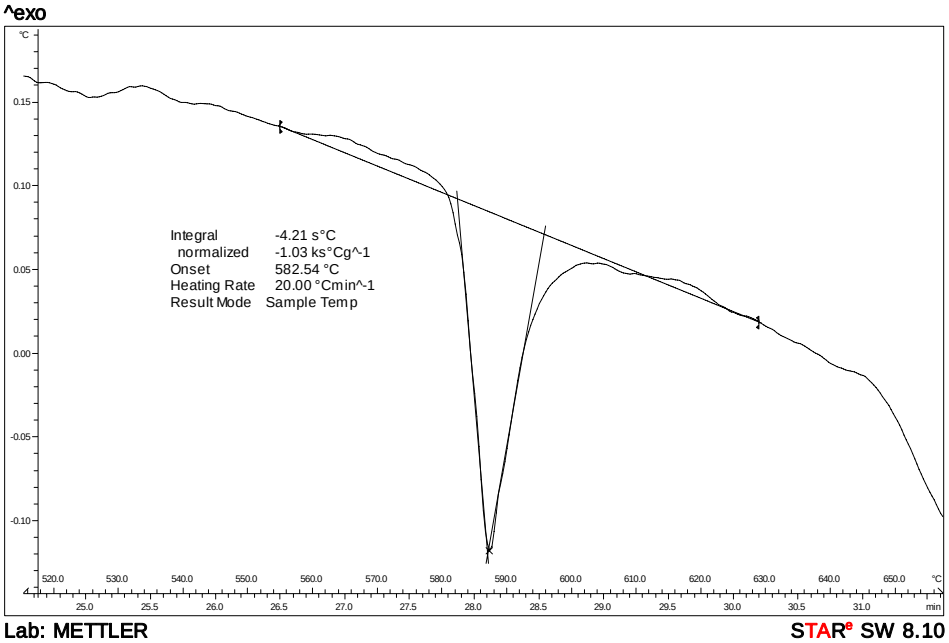
- Phase transition:
  - Litterature: 587 °C
- Melting point:
  - Litterature: 1069 °C



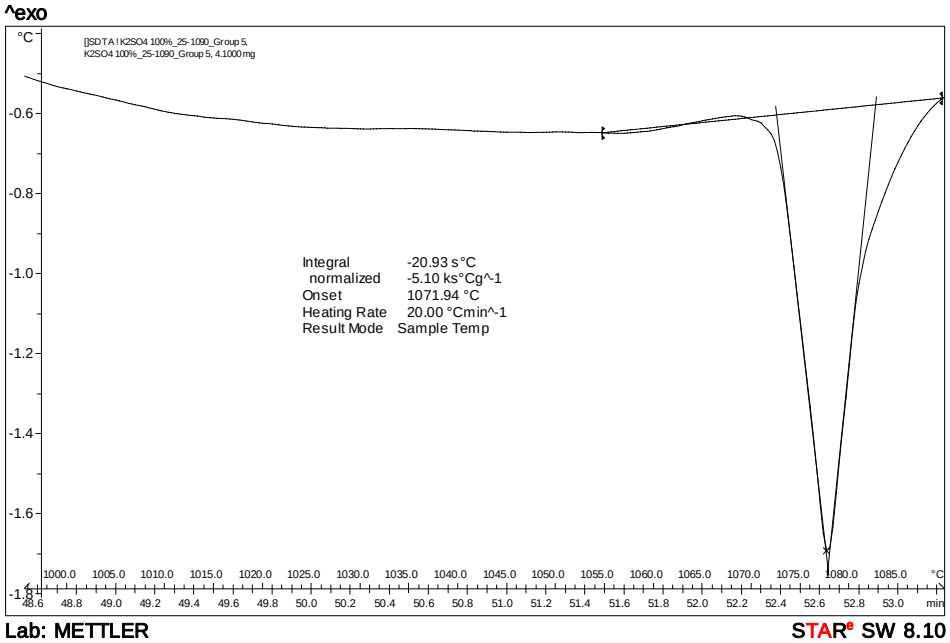
Phase transition and melting

Experimental

- 582,54 °C



- 1071,94 °C

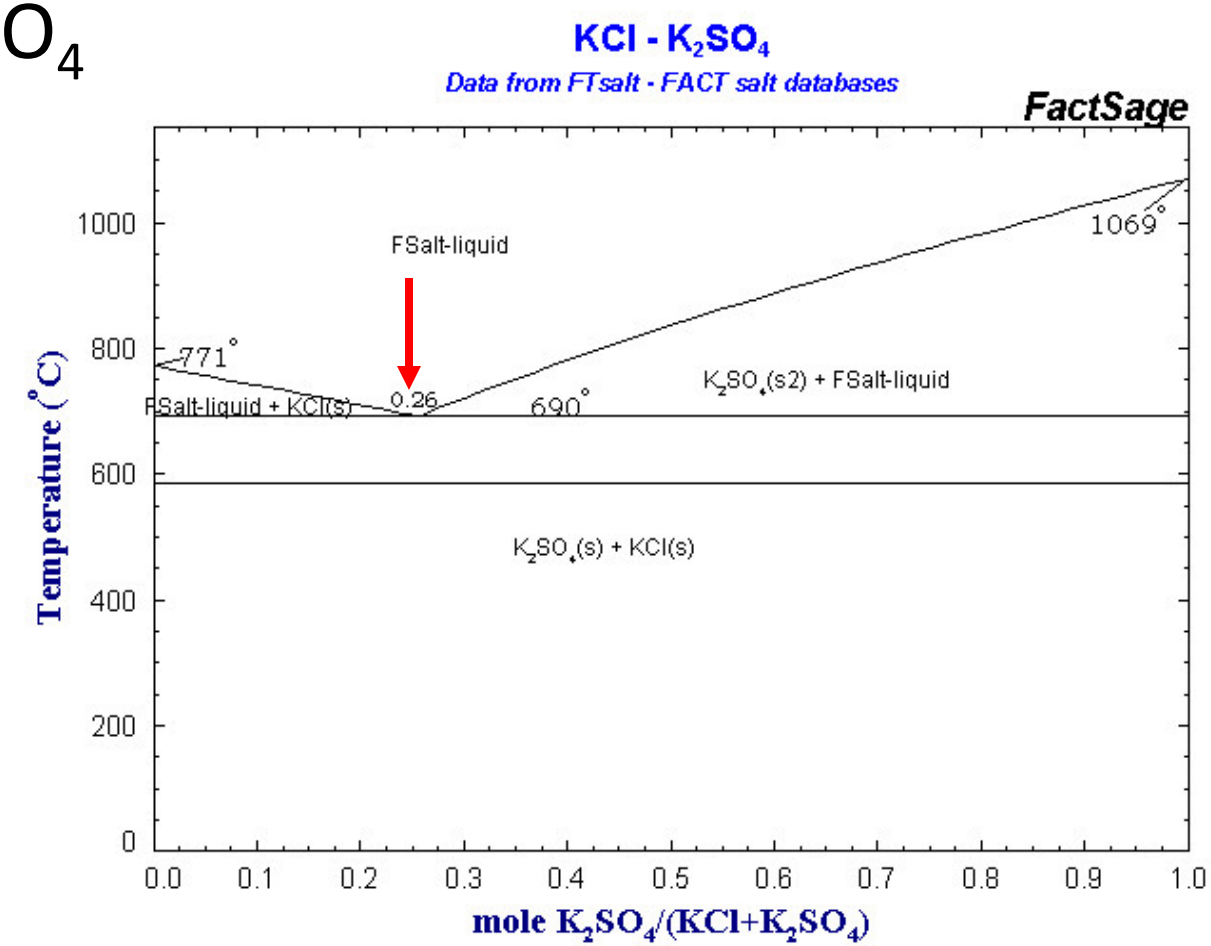


Sample preparation

$KCl – K_2SO_4$ eutectic mixture

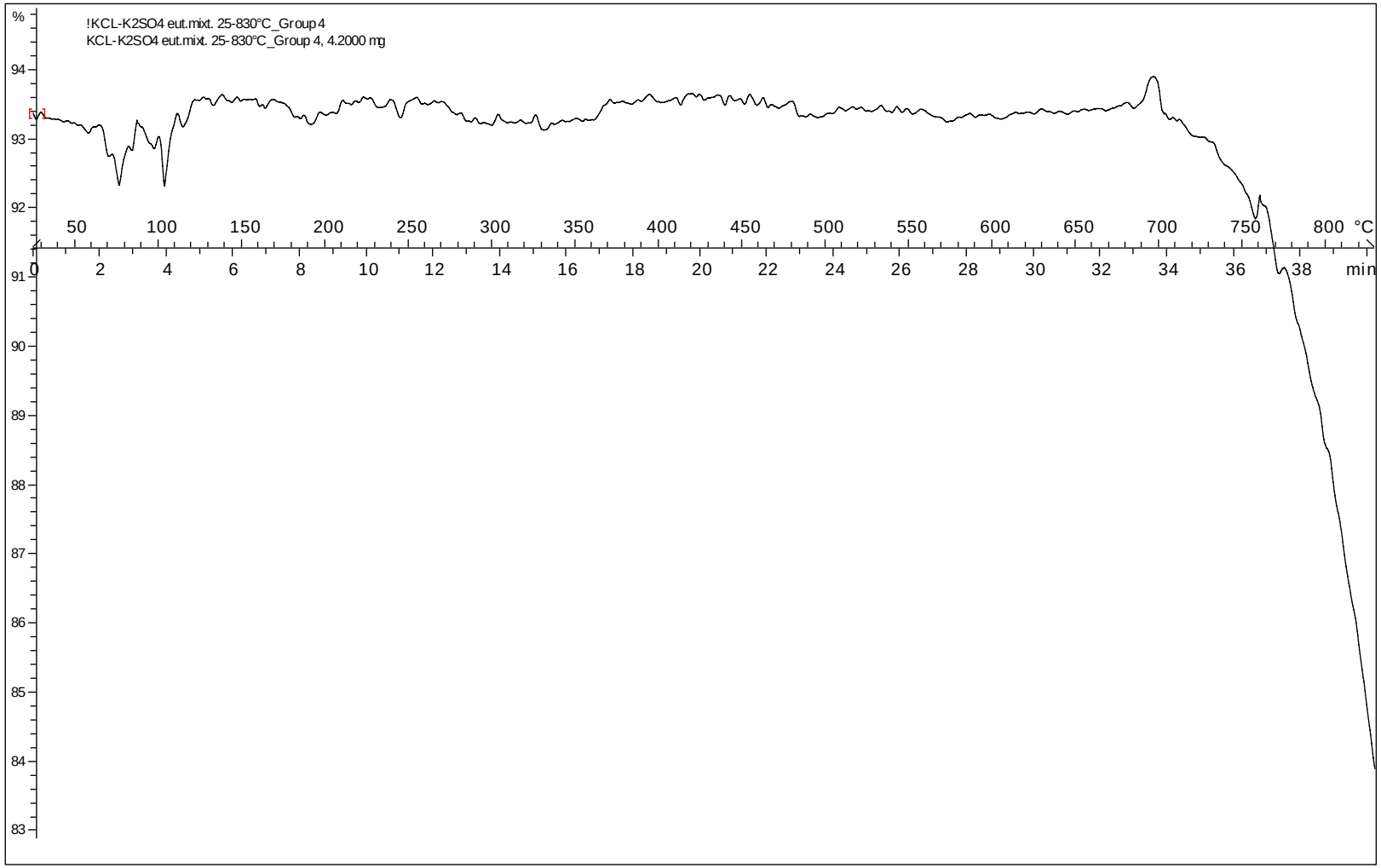
- 26 mole-% K_2SO_4
- 74 mole-% KCl

  →
- 45 mg K_2SO_4
- 55 mg KCl



TGA curve

$KCl – K_2SO_4$ eutectic mixture

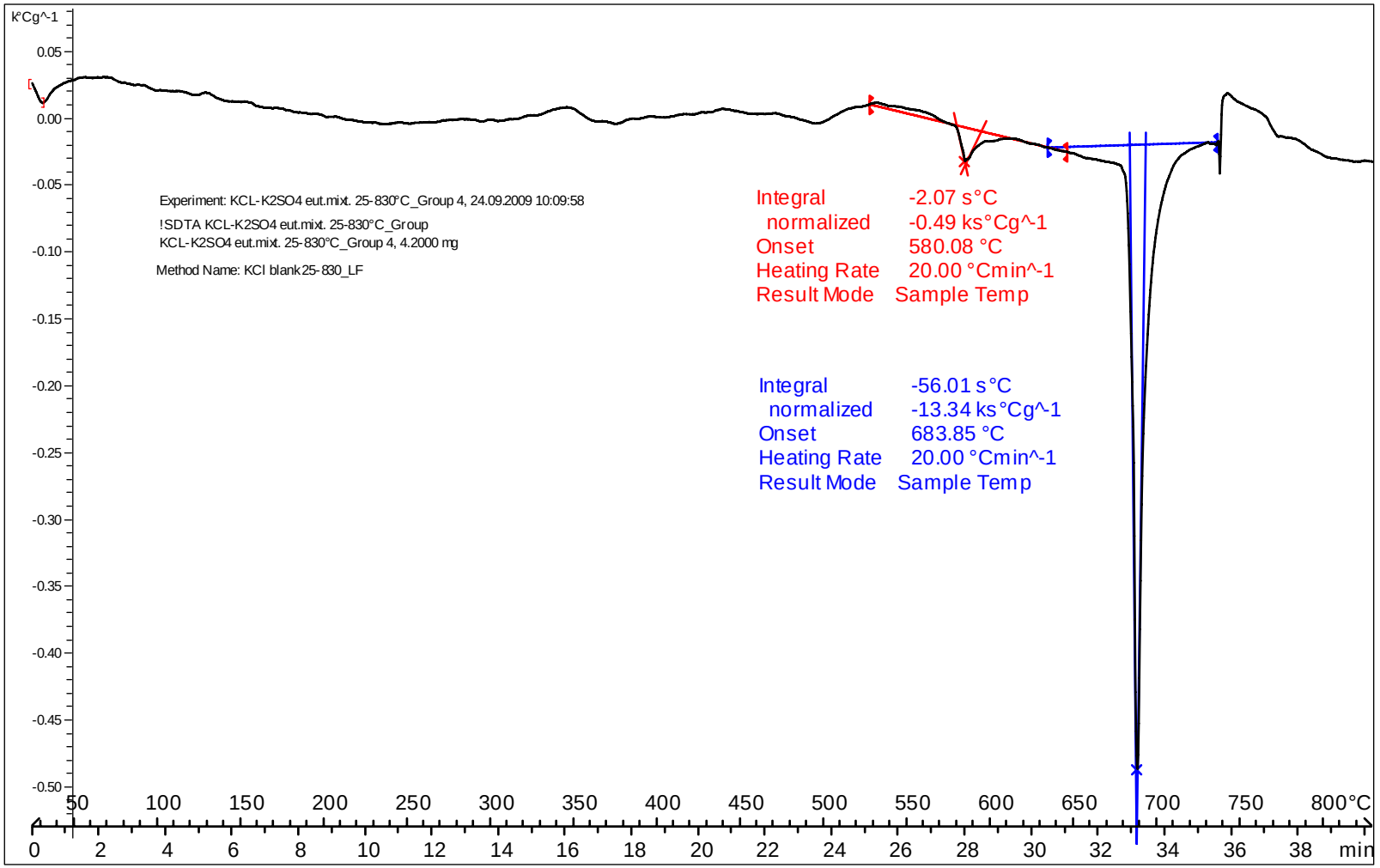


Lab: METTLER

STAR^e SW 8.10

SDTA curve

$KCl – K_2SO_4$ eutectic mixture

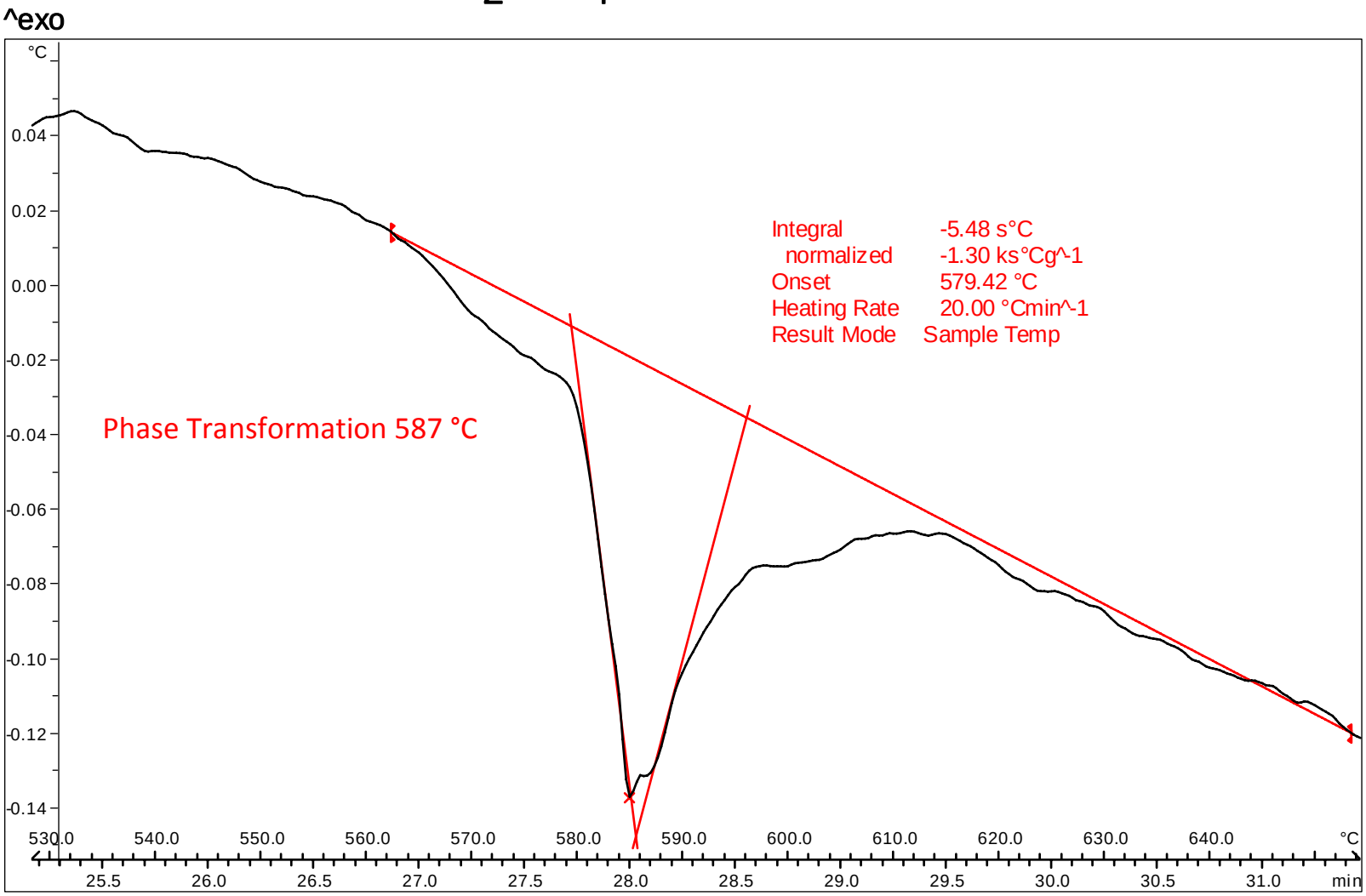


Lab: METTLER

STAR^e SW 8.10

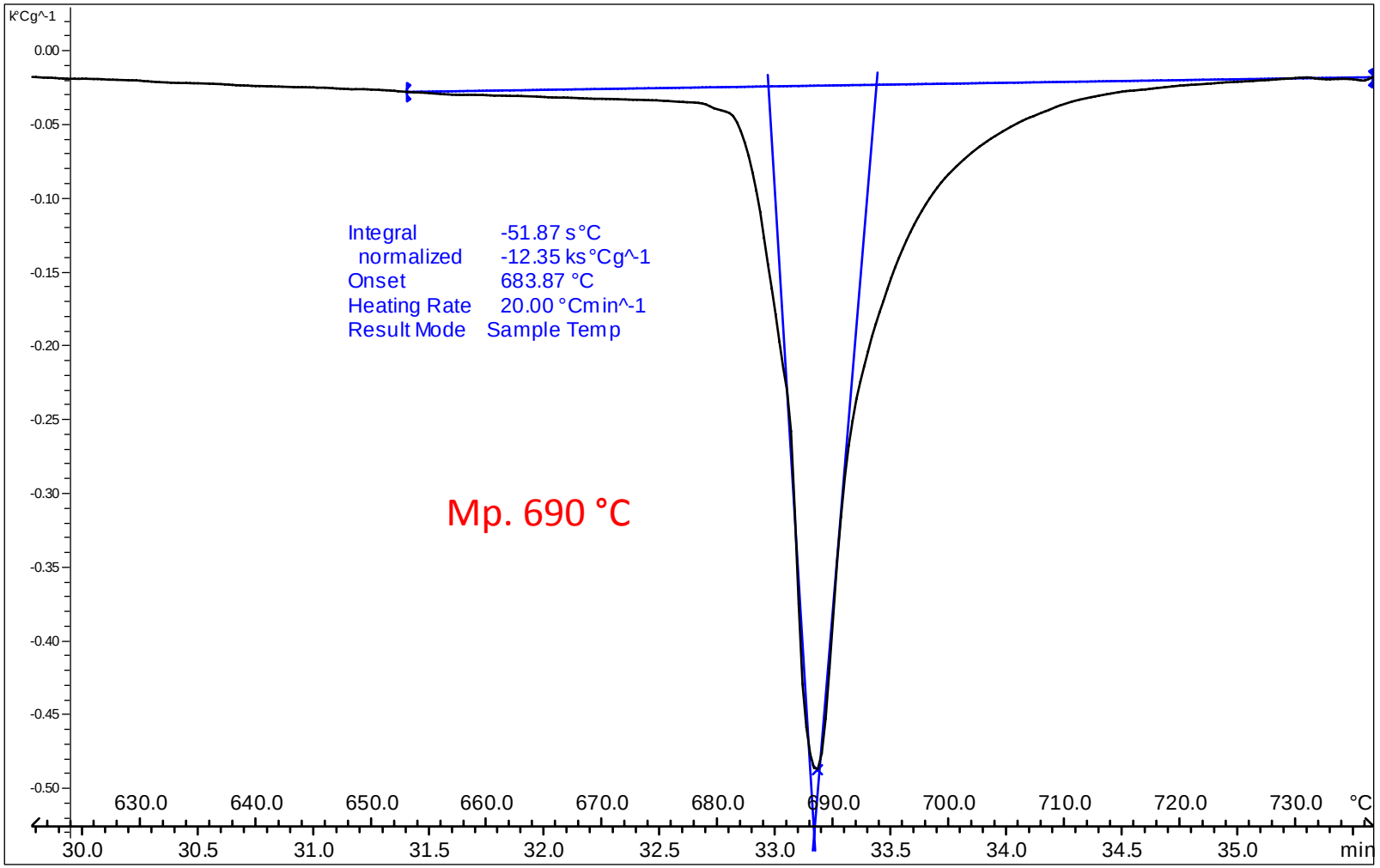
Phase transformation peak

$KCl – K_2SO_4$ eutectic mixture



Melting peak

$KCl - K_2SO_4$ eutectic mixture

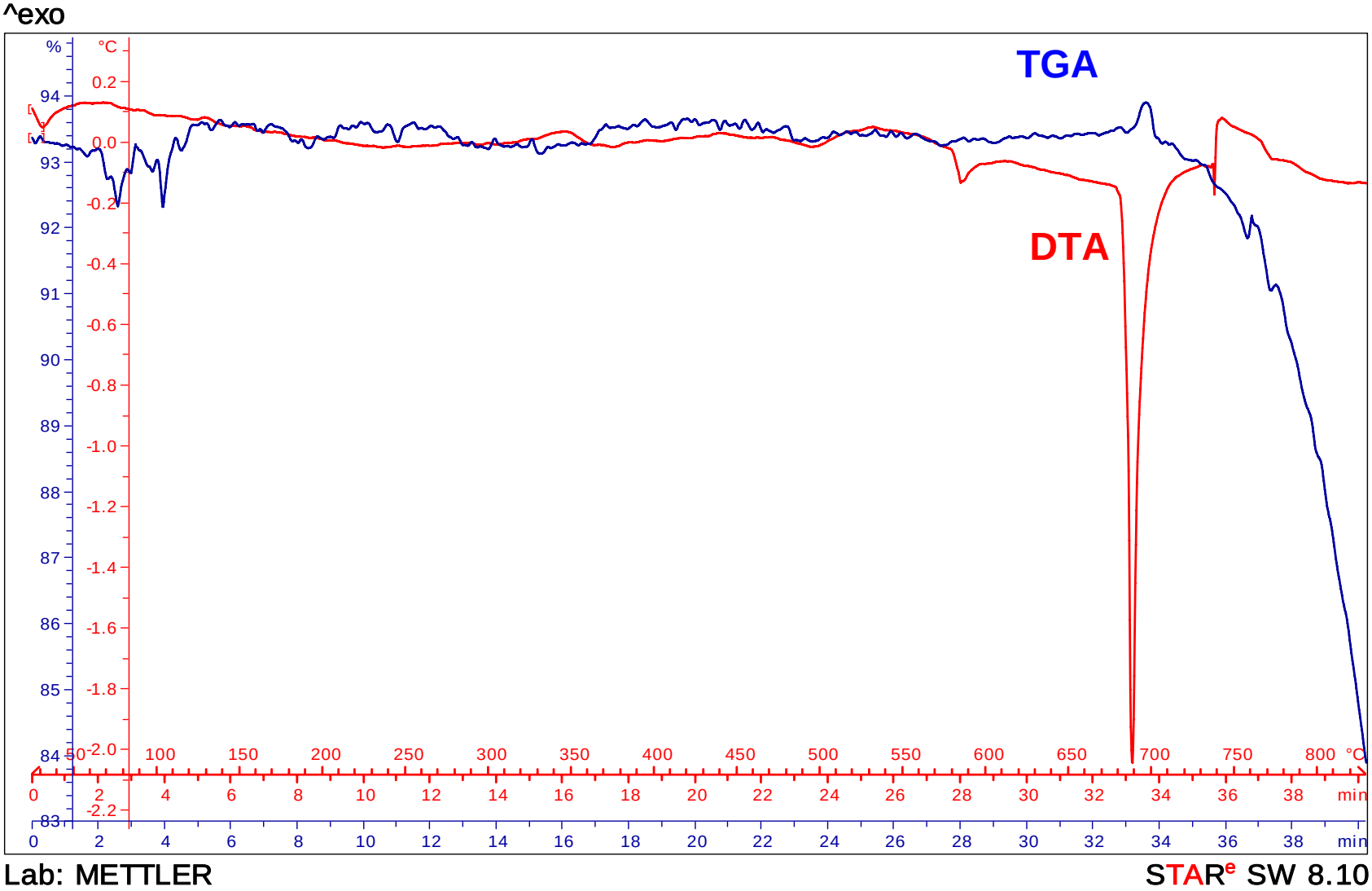


Lab: METTLER

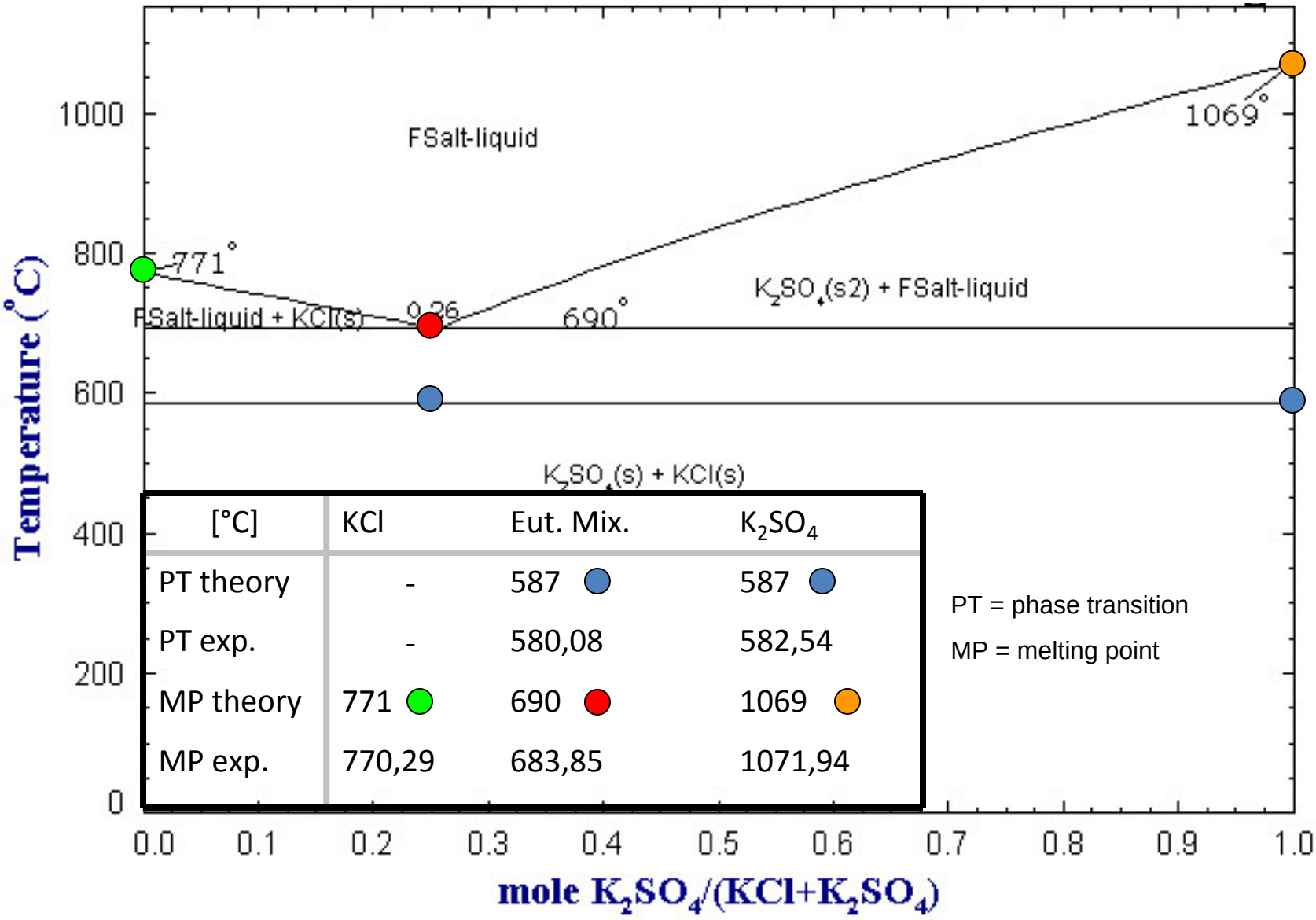
STAR^e SW 8.10

TGA-DTA

$KCl - K_2SO_4$ eutectic mixture



Summary



[°C]	KCl	Eut. Mix.	K_2SO_4
PT theory	-	587	587
PT exp.	-	580,08	582,54
MP theory	771	690	1069
MP exp.	770,29	683,85	1071,94

PT = phase transition

MP = melting point

Conclusions

- KCl and K_2SO_4 system:

  DTA results are in good agreement with the theoretical phase diagram

- Depending on fuel mixtures the melting range of the possibly forming salts have to be taken into careful consideration