Final Reaction Mass Decomposition

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Synthesis reaction - Nitration

The desired reaction is a nitration

An organic substrate is added to sulphonitric mixture at 0 °C

At the end of addition there's the product isolation and purification

The problem

The reaction was in scaling up phase from lab scale to plant scale

At the end of a batch in pilot reactor, a sample of reaction mass was taken and analyzed to determine the conversion

The synthesis was complete

After analysis the sample remained in quality control lab at room temperature

48 hours after nitrous fumes were observed in the sample container

The container was pressurized

Custom synthesis (no communication)

The reaction was a custom synthesis

After some questions the customer said:

“Sometimes it happens to us”

“Sometimes the cap jumps”

“Two or three times the container exploded ”

!!

The customer didn't say anything before

!!

Synthesis reaction

Reaction Enthalpy = -120 kJ/kg (normalized to reaction mass)

Adiabatic temperature rise = 80 °C

MTSR* = 0 + 80 = 80 °C

All these data were known in Company

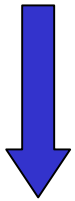
but

they didn't know anything about the thermal stability of the final reaction mass

*Maximum Temperature attainable by the Synthesis Reaction

Thermal stability test

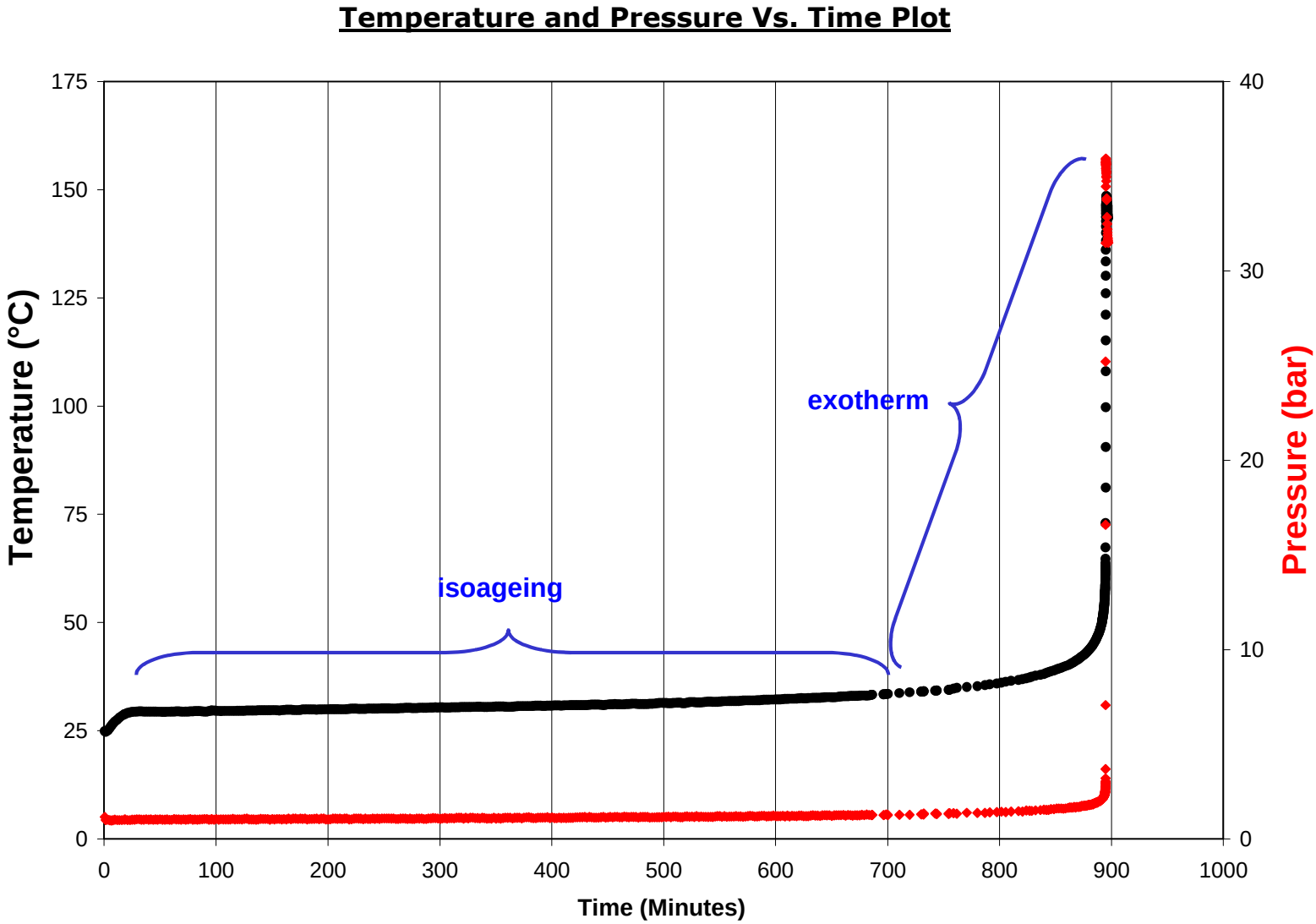
After the near-miss the Company decided to investigate the thermal stability of the final reaction mass



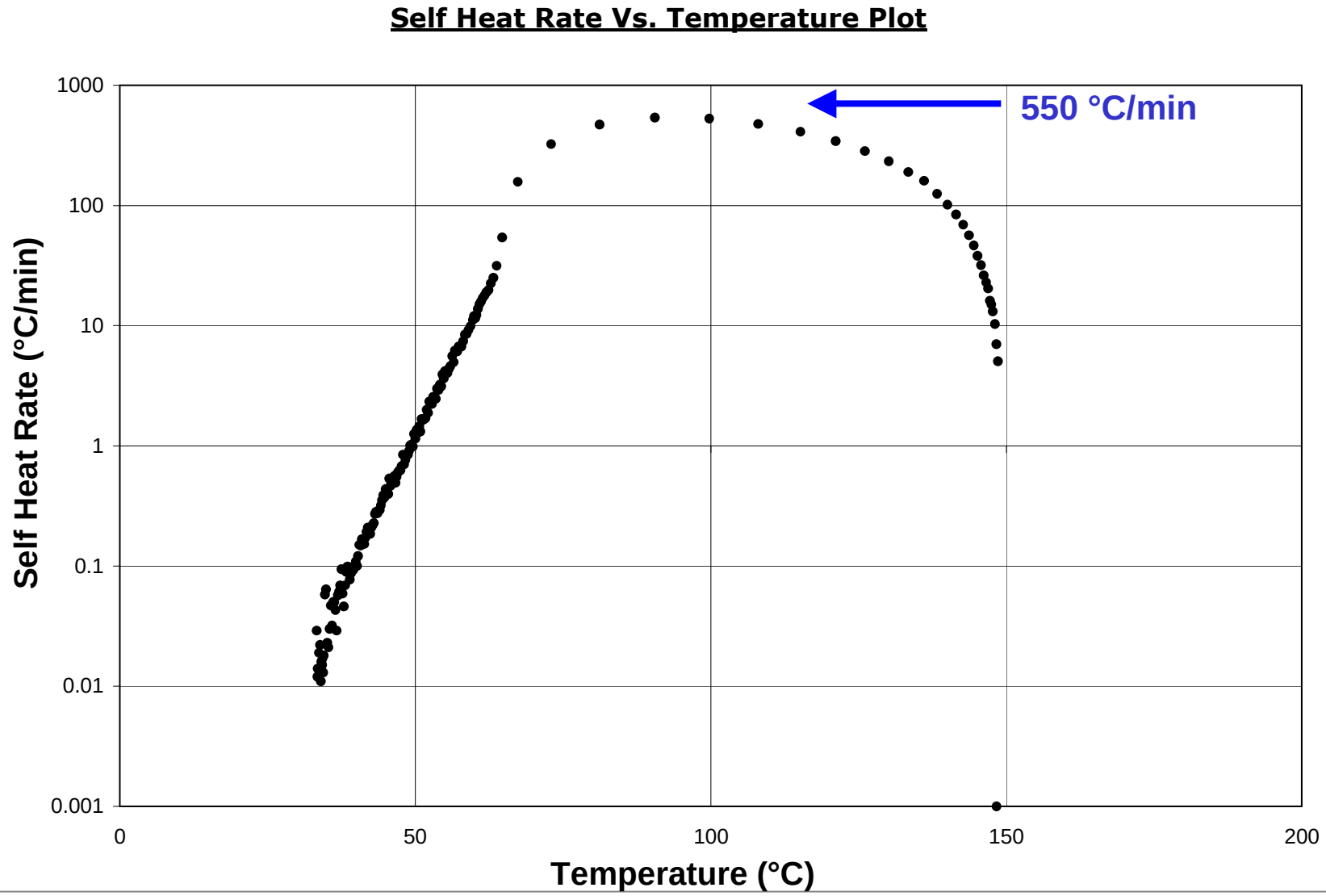
ARC test

Isoageing at room temperature (30 °C)

Isoageing at 30 °C – T, P Vs. t

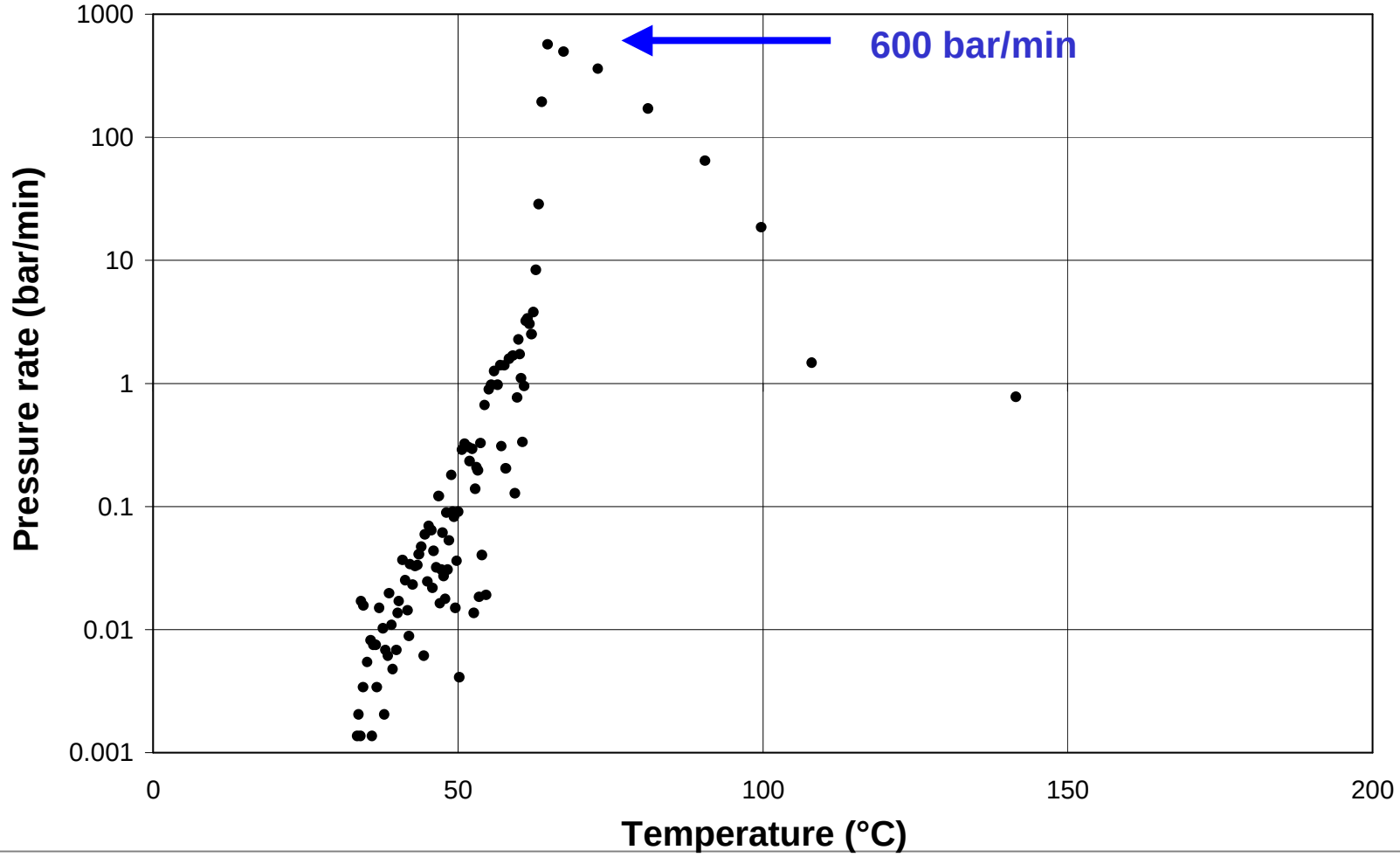


Isoageing at 30 °C - SHR

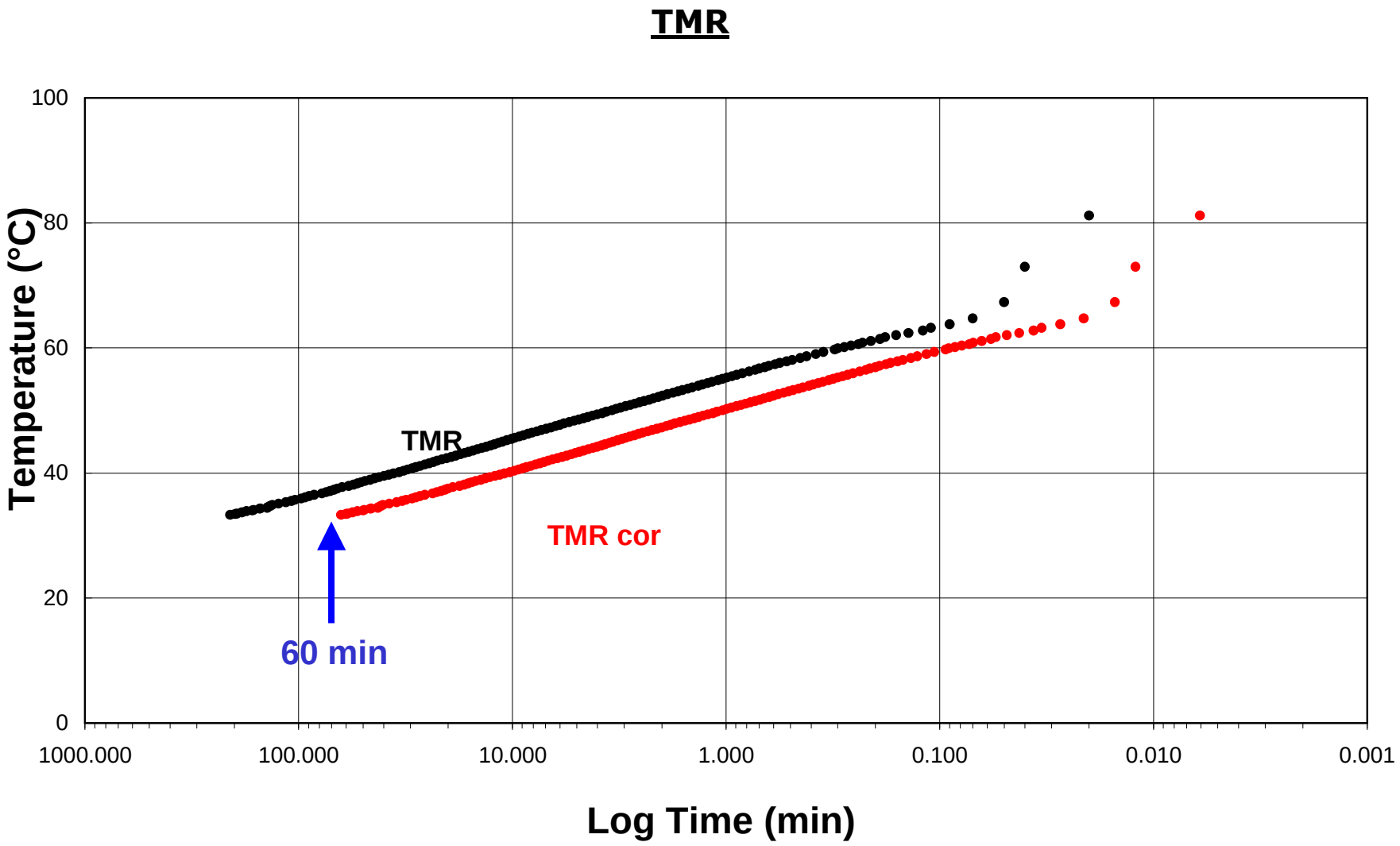


Isoageing at 30 °C - P_{rate}

Pressure Rate Vs. Temperature Plot



Isoageing at 30 °C - TMR



Isoageing at 30 °C - Results

Maximum Self Heating Rate (Φ corrected) = 1800 °C/min

Adiabatic Temperature Rise (Φ corrected) = 400 °C/min

T_{max} = 430 °C

P_{max} = 35 bar (fill level 0.3 g/ml)

TMR (33 °C) = 60 min

Conclusion

The reaction mass decomposes at room temperature

Isolation and purification have to be done immediately after the reaction

The Customer knew it, but he didn’t say anything

The Company studied the synthesis reaction but not the thermal stability of the final mixture