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# Development and evaluation of a new screening workflow for the determination of the self-accelerating decomposition temperature of solids

*Kai Wegmann, 26.10.2021*



# Content

- Definition of the SADT



- Test methods according to the „orange book“



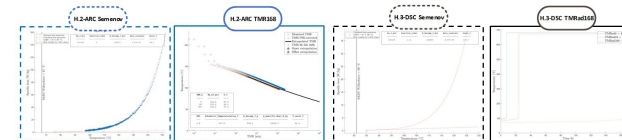
- Motivation and Goals



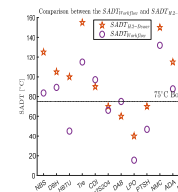
- Instruments



- Demonstration of the new screening workflow on the basis of 16\_DCP

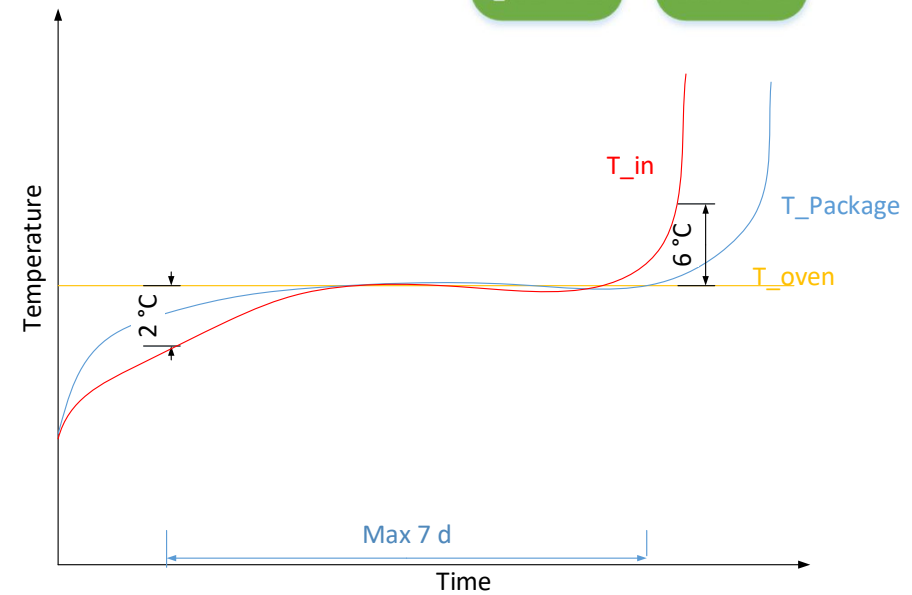
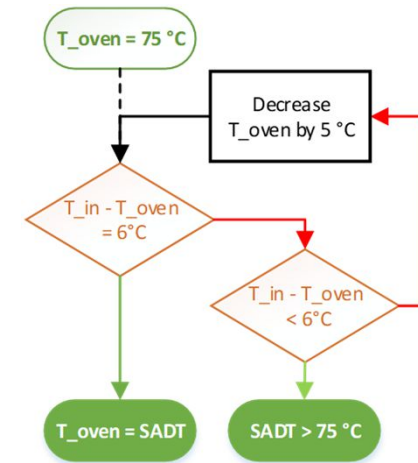
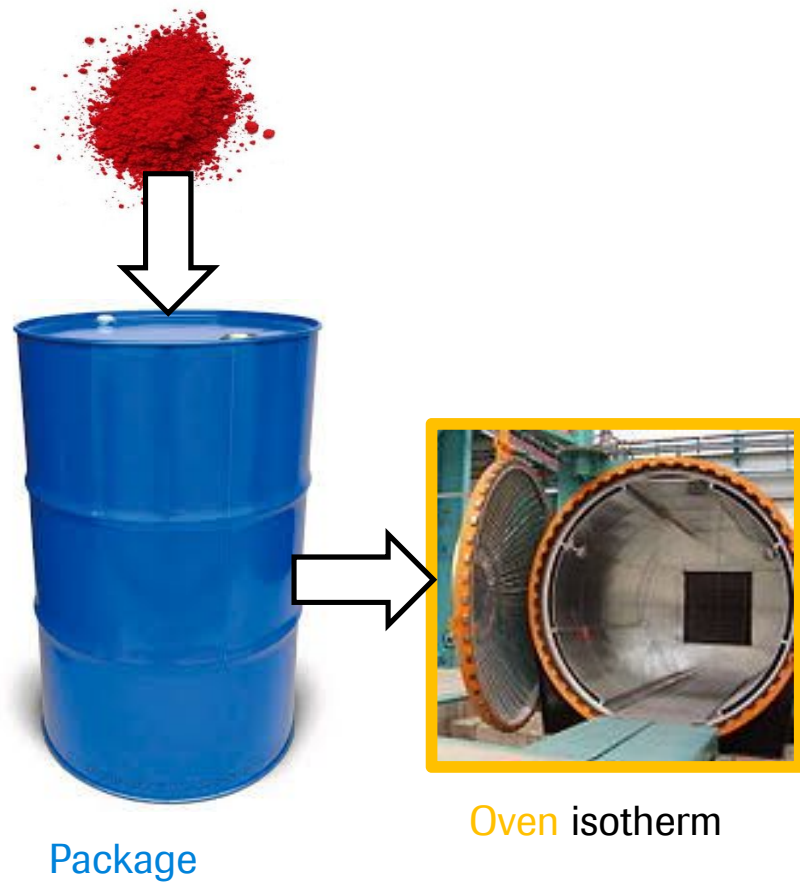


- Results of the validation of the developed screening workflow

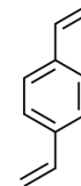


# Definition SADT (Self-accelerating decomposition temperature):

UN Test H.1 United States SADT Test



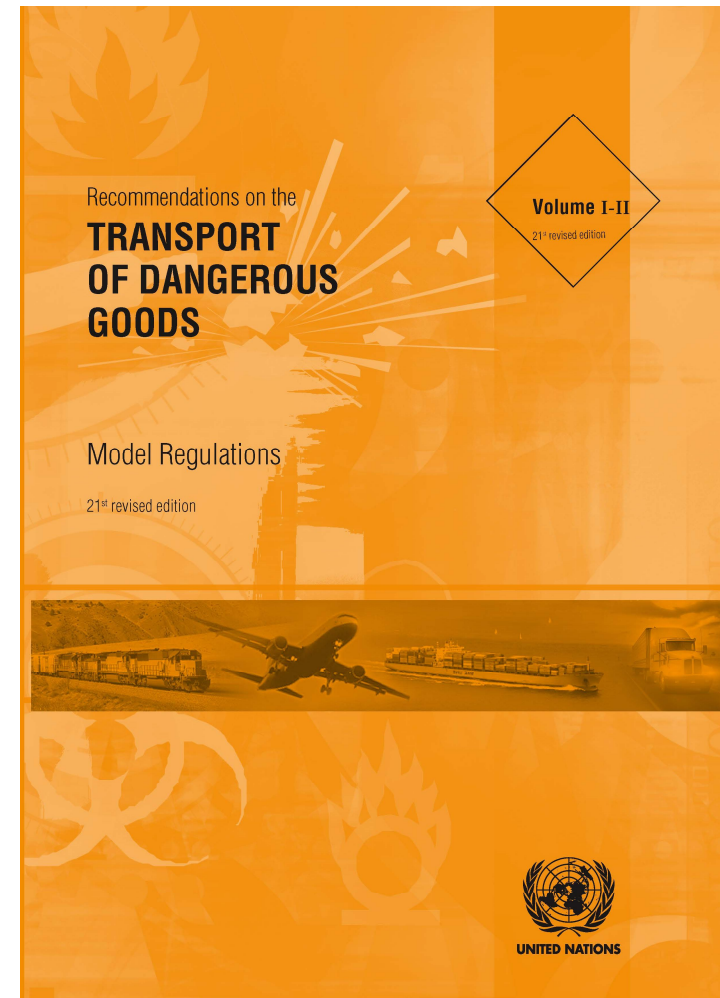
# Wrong SADT...



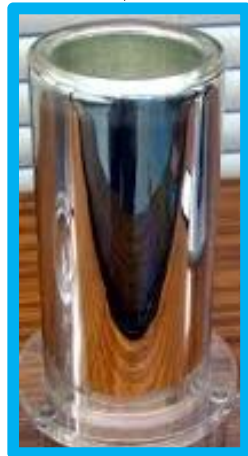
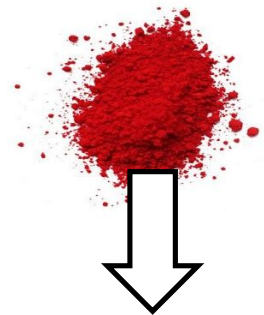
Divinylbenzene

# Evaluation methods for the determination of the SADT

- UN Test H.1: United States SADT Test ✓
- UN Test H.4: Heat Accumulation Test
- UN Test H.2: Adiabatic Storage Test
- ~~UN Test H.3: Isothermal Storage Test~~



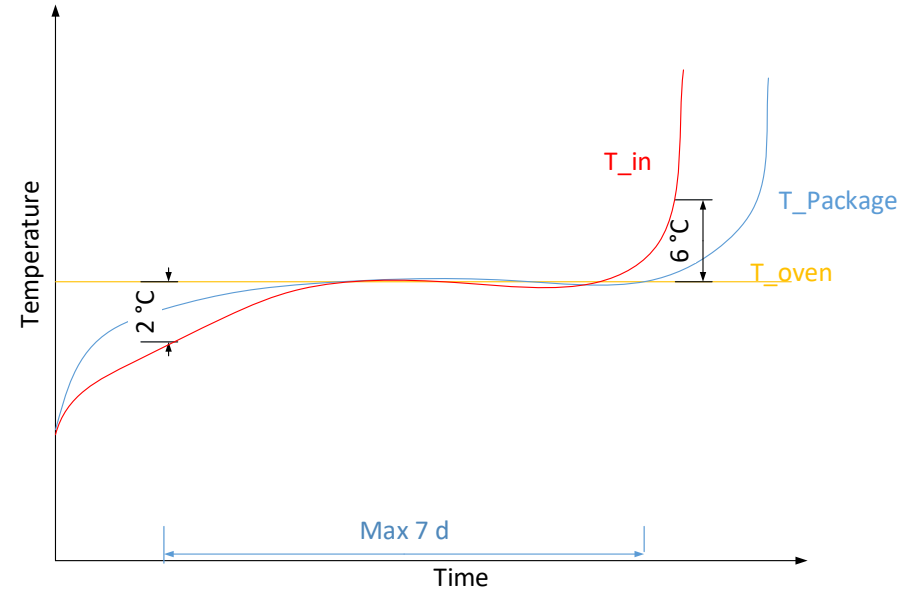
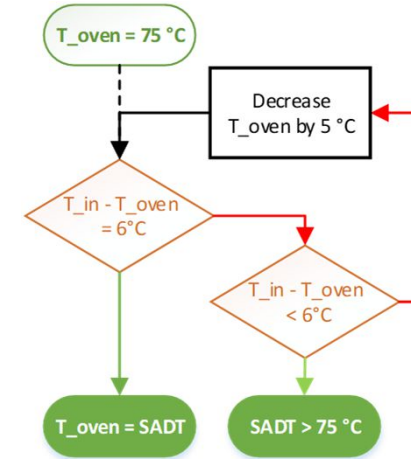
# UN Test H.4 - Heat accumulation Test = H.4 - Dewar Test



0.4 L Dewar  
equal heatloss as  
the transport  
package



Oven isotherm  
Start at 75 °C



# UN Test H.2 – Adiabatic Storage Test = H.2 – Dewar Test



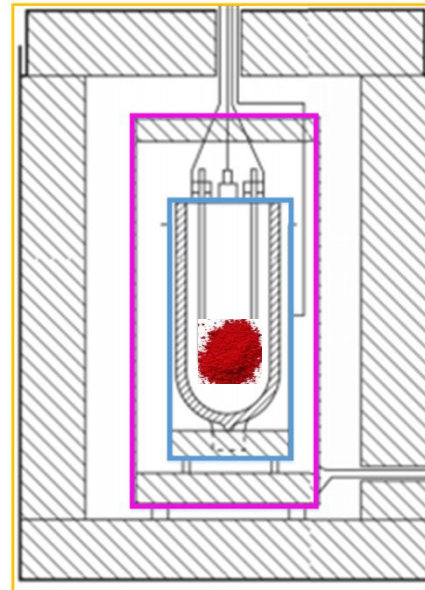
0.2 L Dewar



Autoclave

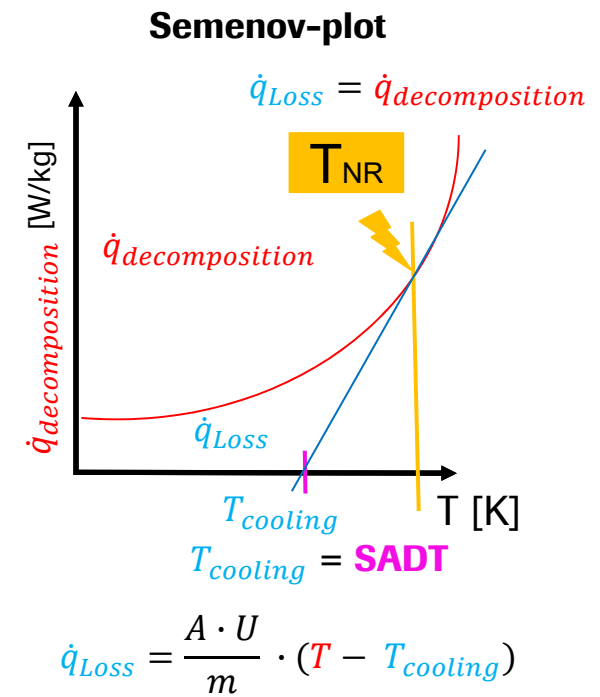
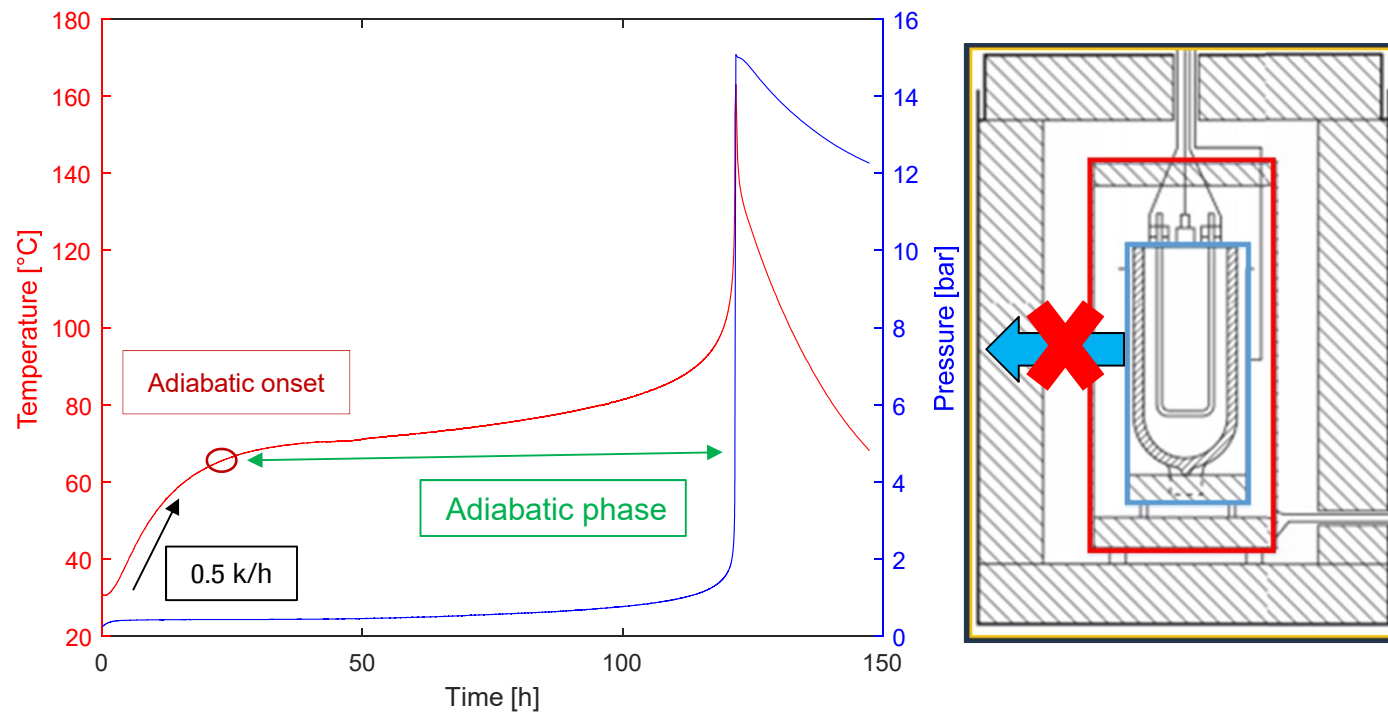


Oven



Cracked Dewar

# UN Test H.2 – Adiabatic Storage Test = H.2 - Dewar



# Motivation

- UN Test H.1 / H.4:

- High amount of substance
- High amount of waste
- Hazardous
- Expensive
- Experimental time (iterative testing)
- SADT is “measured”



- UN Test H.2:

- High amount of substance
- High amount of waste
- Expensive
- SADT is modelled
- Experimental time



**Therefore only substances, which have a SADT  $\leq 75$  °C should assessed precisely by a UN Test**

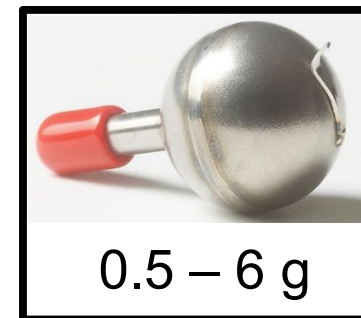
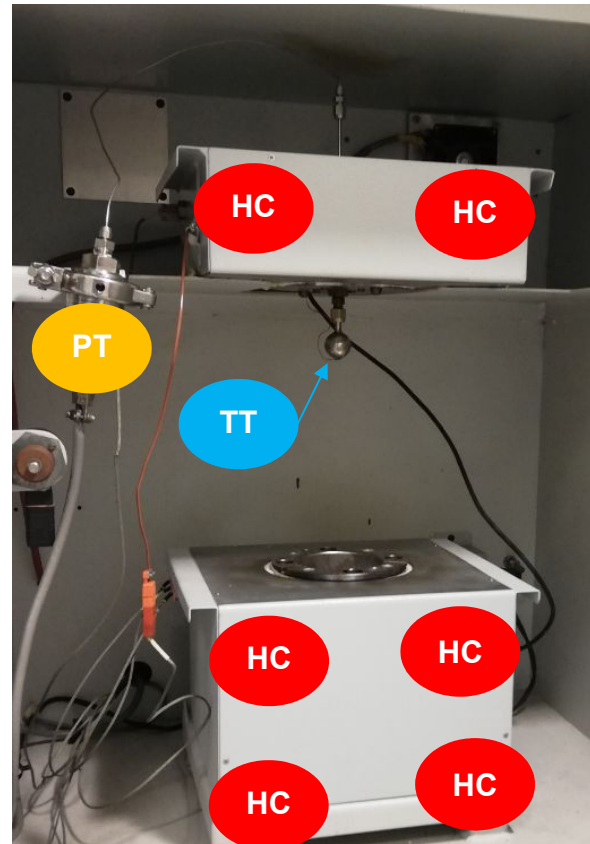
**→ Need for a screening workflow**

# Goals

- Development and validation of a screening workflow for the categorization of substances:
  - Categorize substances into  $SADT \leq 75 \text{ }^\circ\text{C}$  and  $SADT > 75 \text{ }^\circ\text{C}$
  - The categorization shall be performed for a 50 kg transport package
  - Minimal amount of test substance
  - Minimal resources for the measurement
  - Implementable in a safety lab with the available equipment

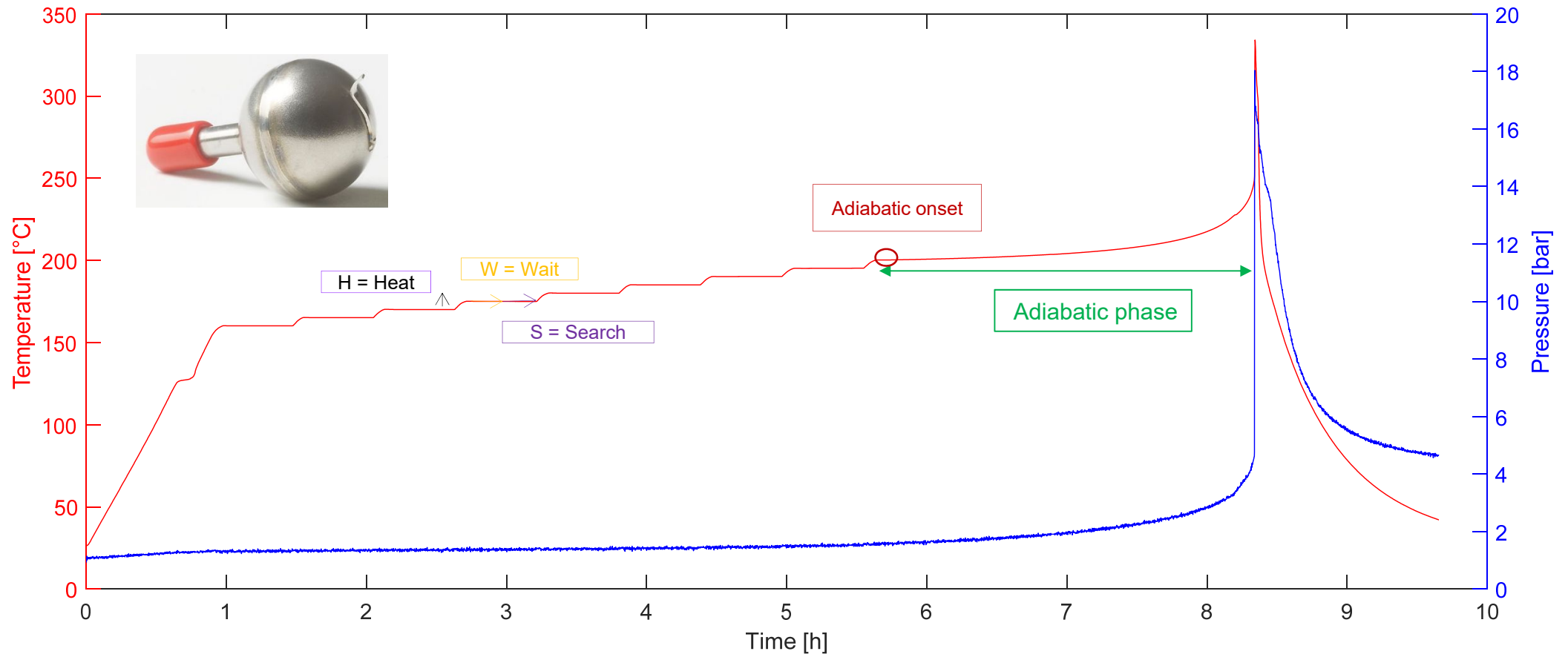


# H.2-ARC / Accelerating rate calorimeter

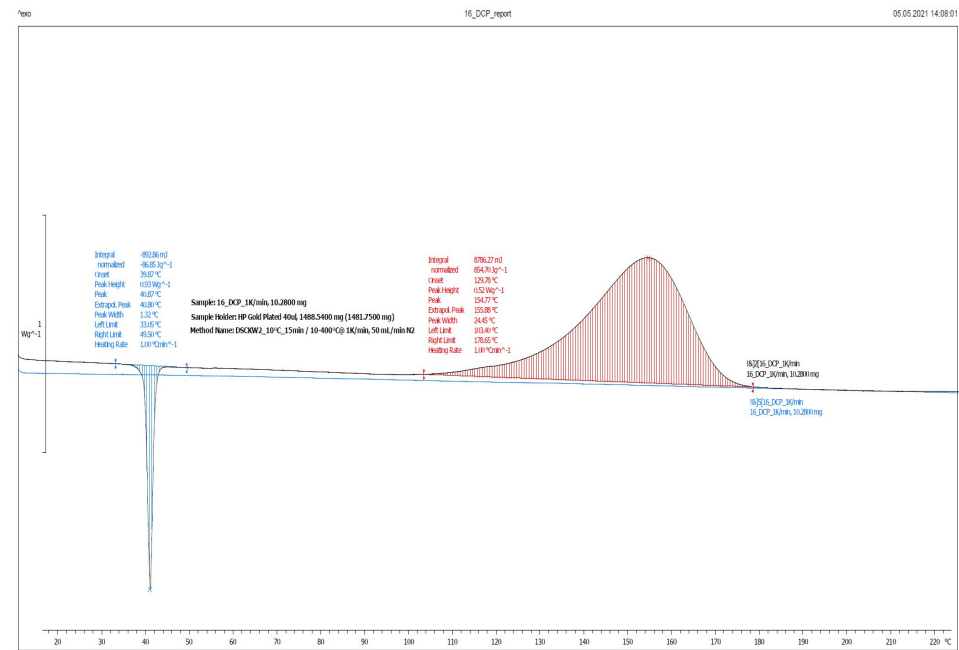
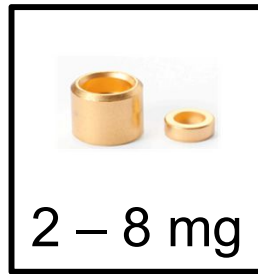


HC	Heat cartridge
TT	Thermocouple
PT	Pressure transducer

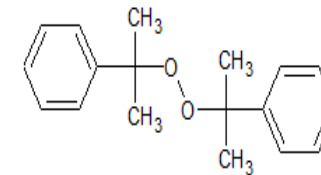
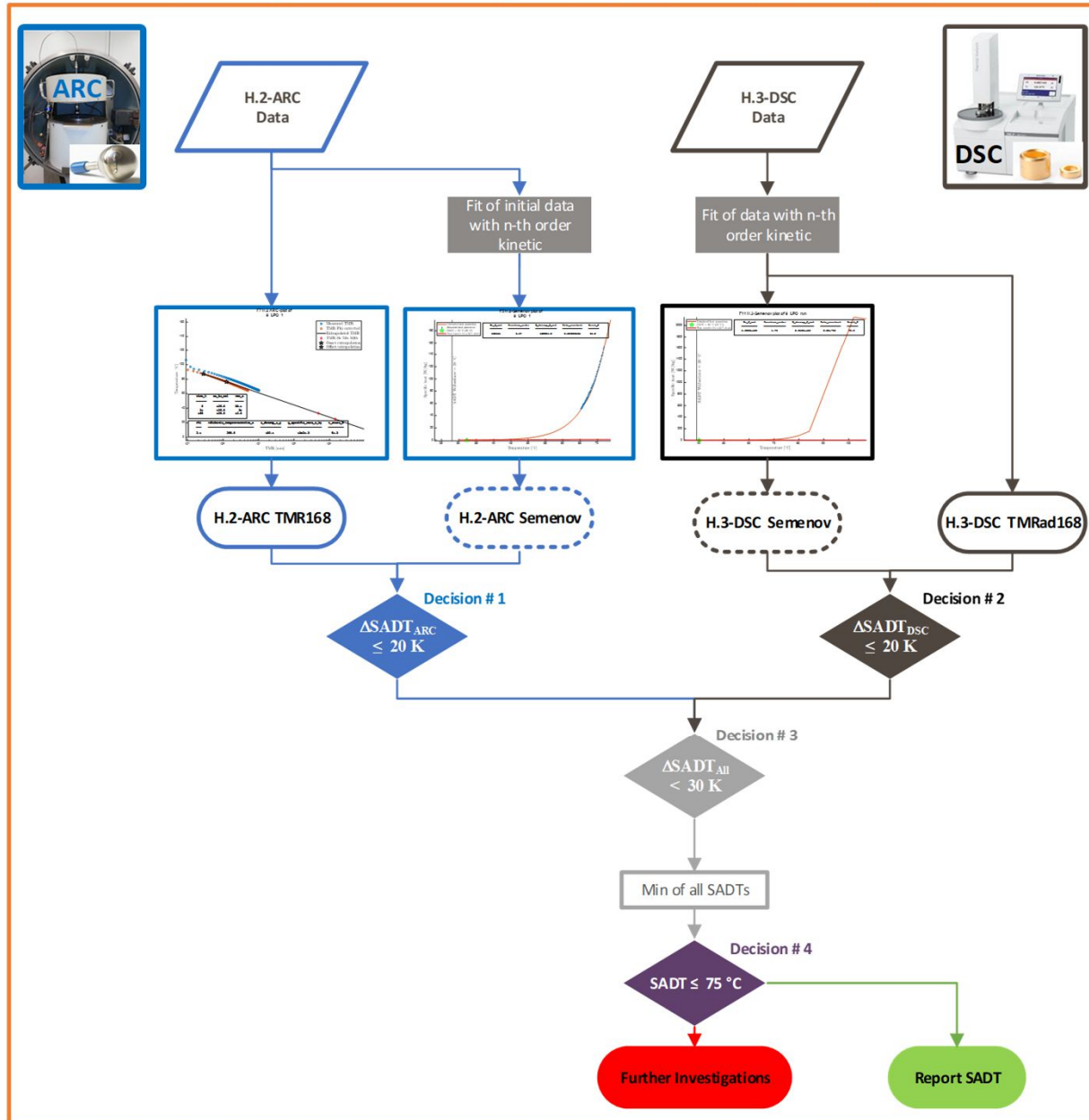
# H.2-ARC / HWS → Heat-Wait-Search



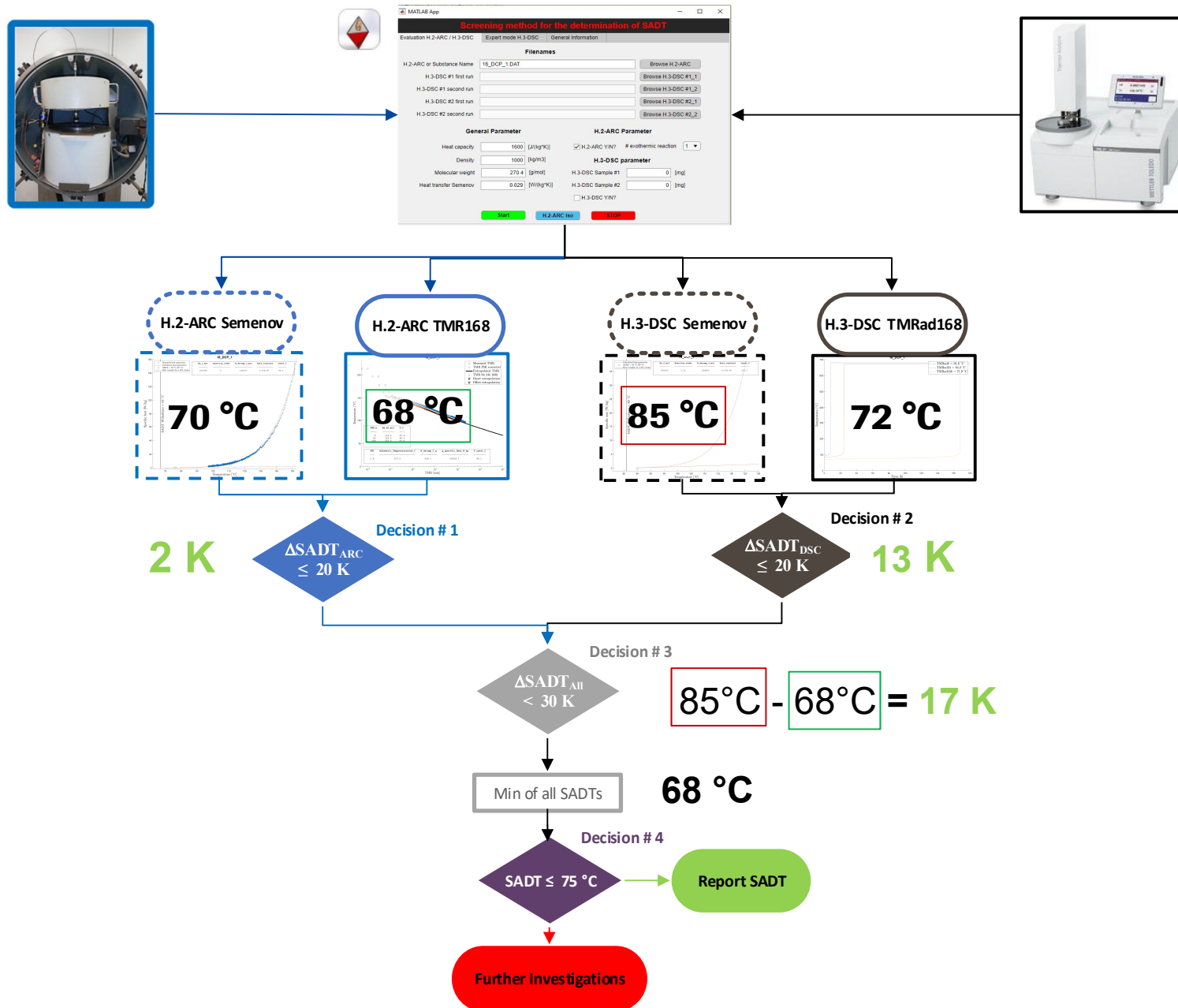
# H.3-DSC / Differential Scanning Calorimeter



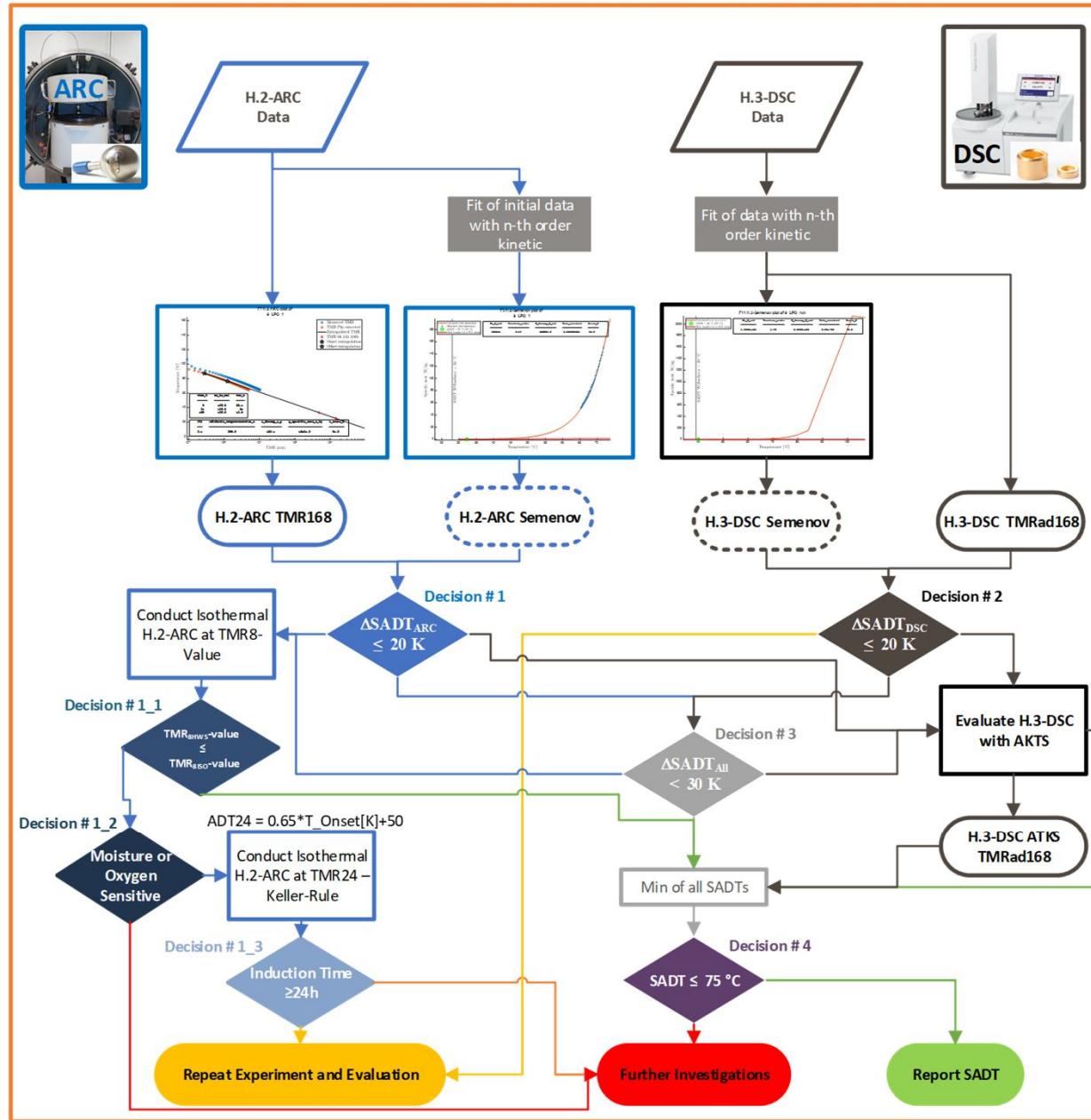
### Screening of self-accelerating decomposition temperature (SADT)



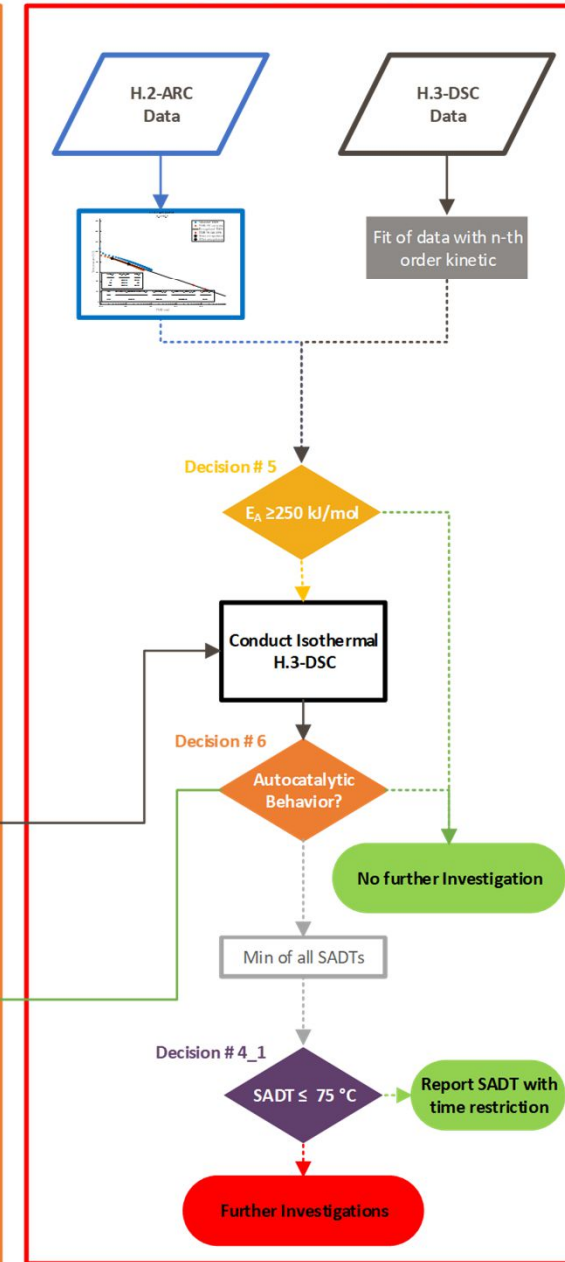
16\_DCP  
(Dicumyl peroxide)

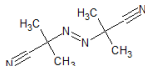
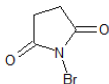

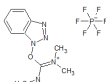

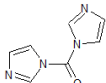
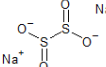

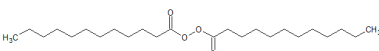
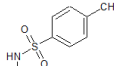

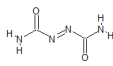
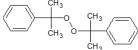


### Screening of self-accelerating decomposition temperature (SADT)



### Identification of autocatalytic behavior

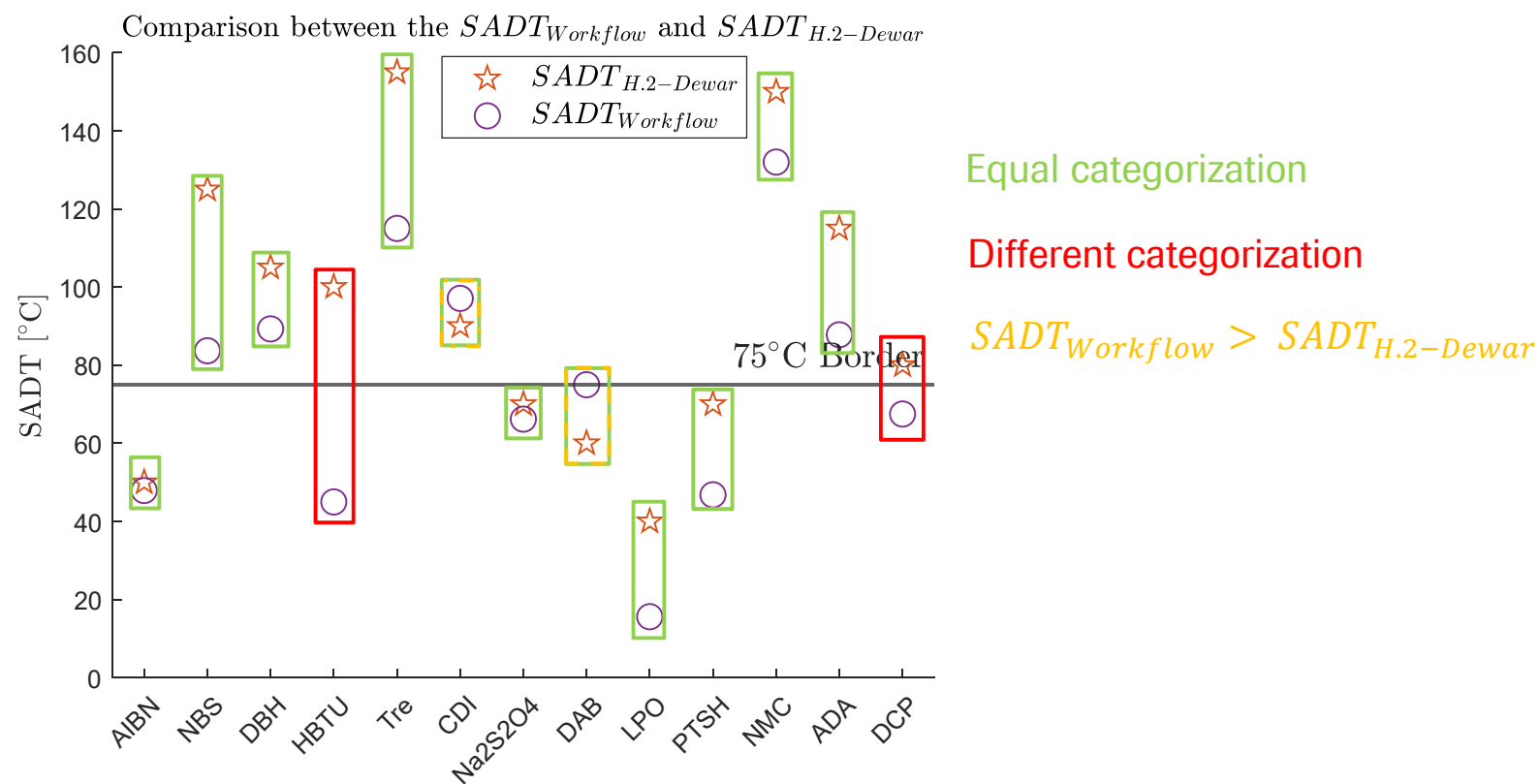


Abbreviation	Substance
	AIBN Azobisisobutyronitrile
	NBS N-Bromosuccinimide
	DBH 1,3-Dibrom-5,5-dimethylhydantion
	HBTU 2-(1H-Benzotriazol-1-yl)-1,3,3-tetramethyluronium-hexafluorophosphat
	Tre D-(+)-Trehalose dihydrat
	CDI 1,1-Carbonyldiimidazol
	Na <sub>2</sub> S <sub>2</sub> O <sub>4</sub> Natriumdithionit
	DAB 1,2-Diamino-4-brombenzol
	LPO Lauroyl peroxid
	PTSH p-Toluenesulphonyl hydrazid
	NMC 3-methyl-4-nitrophenole
	ADA Azodicarbonamide
	DCP Dicumylperoxid

# Results

## Screening of self-accelerated decomposition temperature (SADT)

Decision # 4  
 $SADT \leq 75^\circ\text{C}$



# Development and evaluation of a new screening workflow for the determination of the self-accelerating decomposition temperature of solids.

Development and validation of a screening workflow for the determination of the SADT:

- Categorize substances into  $SADT \leq 75\text{ °C}$  and  $SADT > 75\text{ °C}$
- The categorization shall be performed for a 50 kg transport package
- Minimal amount of test substance
- Minimal resources for the measurement
- Implementable in a safety lab with the available equipment



# Outlook



VALIDATED



?



**F. Hofmann-La Roche Ltd**

Dr. Klaus Schwenzfeuer, Christoph Kuratli, Xuong Nghiem, Dr. Michael Pfammatter, Kim Zulliger, Corinna Faber, Flavio Lack, Nikolaus Martin, Dr. Roland Zurbrügg

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**TÜV-SÜD Schweiz AG**

Mischa Schwaninger, Delphine Berset

**Consilab Anlagensicherheit GmbH**

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