DSC–Thermal Safety, Phase Transitions, Polymorphs

Differential scanning calorimetry (DSC) measures the heat flow from a sample as a function of temperature or time. Differences in heat flow arise when a sample absorbs or releases heat due to thermal effects such as melting, crystallization, chemical reactions, polymorphic transitions, vaporization etc. DSC is also employed as the first in line analytical method for thermal safety analysis and can be used to rapidly assess whether molecules or mixtures are potentially highly energetic, shock sensitive, or explosive.

Mettler Toledo DSC3

Melting, Crystallisation, Evaporation

- Rapid determination of phase transitions such as melting point, glass transitions, crystallisation, vaporization, degradation.
- Suitable for solids and liquids.
- High pressure pans also available
- Temperature range RT to 700 °C
- Maximum heating rate 300 K/min

Polymorphs

- Identification of polymorphs by melting point
- Crystallisation of API from various solvents followed by DSC analysis

Thermal Safety Evaluation

- Thermal safety profiling can be conducted using the DSC to determine the onset of degradation of a chemical or mixture and also to estimate the TD_{24} – the process temperature at which time to maximum reaction rate under adiabatic conditions is 24 hours.

Requirements

5 mg sample

Deliverables

DSC Thermogram, Thermal Safety Profile

charnwooddiscovery.com

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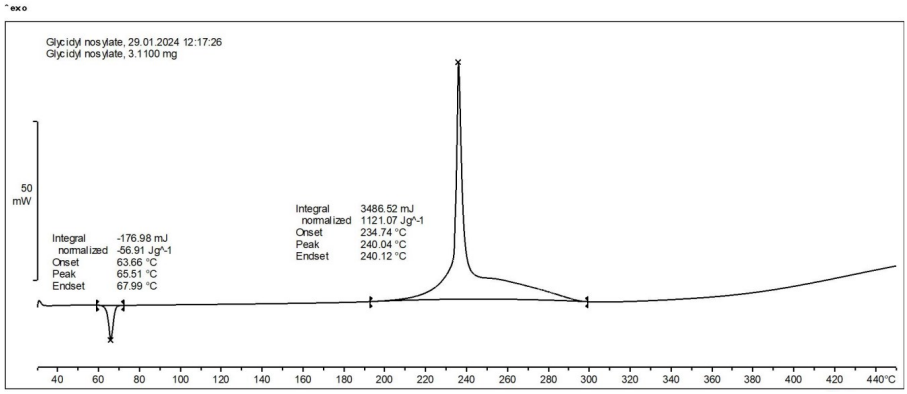
All our assays can be customised to answer specific project questions

Contact us for more information on standard or customised assay formats

DSC–Thermal Safety, Phase Transitions, Polymorphs

Typical DSC Thermogram

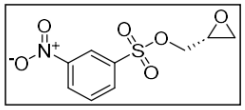


A thermogram generated for chemical compound "glycidyl nosylate". Note the melting point at 64°C and the onset of degradation at 235°C. The thermogram shows a highly energetic decomposition at elevated temperatures.

Subsequent use of glycidyl nosylate by our chemical development team was conducted with a temperature margin of safety window of over 200 °C.

Thermal Safety Evaluation of Glycidyl Nosylate

- Further thermal risk can be conducted to give a more in-depth analysis. In this case, the use of glycidyl nosylate on large scale was restricted due to its "High Hazard" concern

Sample ID	Structure	Formula	Mol Wt
glycidyl nosylate		C9H9NO6S	259.23

Points:	1	2	4	8
Oxygen Balance Hazard				High
Rule of 6 Calculation				Fail
Explosive Functional Group?				Yes
Onset Temperature - DSC				
Scale		Low		
		5-<100 g		

O.R.E.O.S Total			
Points:	Low Hazard	Medium Hazard	High Hazard
			28

Subsequent use of glycidyl nosylate was conducted with a temperature marging of safety window of over 150 °C