

Step Isothermal TGA: Enhanced Component Separation in Thermal Analysis

Overview

In thermogravimetric analysis (TGA), one of the primary objectives is to separate and characterize individual components within a sample based on their thermal decomposition behavior. However, when multiple thermal events occur in close temperature proximity, overlapping mass-loss signals can reduce resolution and limit interpretability.

Factors such as sample mass, purge gas, and heating rate affect this resolution, with **heating rate** being the most impactful. To address these limitations, **AMI has integrated a Step Isothermal function** into the **InfinityPro** software platform for its TGA systems.

What Is Step Isothermal?

Step Isothermal TGA introduces dynamic control over heating conditions by allowing the system to:

- Heat the sample at a defined linear rate
- Monitor real-time derivative weight loss (%/min)
- Automatically switch to an **isothermal hold** once a predefined **Iso Start Threshold** is exceeded
- Resume the temperature ramp once the **Ramp Resume Threshold** is reached

This smart switching improves separation of closely spaced decomposition steps that may otherwise appear merged during traditional linear ramping.

∠Mİ	ADVANCED MEASUREMENT INSTRUMENTS
-----	--

IGA Data Acqu ile Options He	isition - IGA 1000 - Ip	[Setup]							- 0
SETUP	Professees	RT PLOT	Sample		0.00 Time	9)	0	č1:	0.00
	CHUBRATI		Fumace		0.00 Poin	ts:	0 8	Segment	1
Current Method D	Directory: c:\therm	al\data							
Method:	<new experiment=""></new>	•					Ca	dibration	s:
Description 1:	First run using stepw	ise isothermal					(• <u>N</u> one	
Description 2:								Saved	
)perator:	DSM		🔽 Use	e Step Iso	o for this ru	n.		uu <u>t</u> uu	
nstrument ID:	TGA 1000								
Sample wt:	11.46	+ +] Start Temp	End Temp	Heating Rate	Hold Time	Gas	San Rat	nple e
		Segment 1:	25	200	20.0	.0	1	1.00	
las 1:	Nitrogen	Segment 2:	0	0	0.0	.0	1		
ias 2:	Air	Segment 3:	0	0	0.0	.0	1		
		Segment 4:	0	0	0.0	.0	1		
		Segment 5:	0	0	0.0	.0	1		
							<u>S</u> tart E	xperiment	t
			-						
Start CAL	Jata Acquisi							1	2-26 A



How It Works

- The Iso Start Threshold defines the rate of weight loss at which the system will enter an isothermal hold. This is typically set at ~1/10th to 1/15th of the expected peak weight loss rate.
- The Ramp Resume Threshold determines when the system will exit the isothermal condition and resume ramping. It is typically ~1/15th or less of the Iso Start Threshold.

This cycle continues throughout the experiment, enabling adaptive thermal profiling based on the material's real-time behavior.



Iso Start Threshold is usi expected maximum weig heating rate.	ually set at 1 jht loss rate	/10 to 1/15 the at the desired
Iso Start Threshold:	2	%/min
Ramp Resume Thresho less of the Iso Start Thre	ld is usually : shold setting	set a 1/15 or I.

Figure 2: Step Isothermal method setup dialog showing threshold parameters.

Case Study Example

A conventional TGA run on **Barium Chloride** at 20 °C/min reveals two overlapping mass-loss events—one at approximately 80 °C and another around 110 °C. These transitions appear as a single broadened event in standard TGA.



Figure 3: Conventional TGA curve for Barium Chloride showing overlapping events.



Using **Step Isothermal**, the same material yields two **distinct weight-loss steps**, allowing accurate resolution of each component's thermal behavior. The system automatically pauses heating during high-rate decomposition, improving event separation.



Figure 4: Improved separation using Step Isothermal mode on the same material.

Key Benefits

- Enhanced separation of overlapping decomposition or reaction events.
- Automated transition control based on actual weight loss.
- No need for complex scripting or multiple trial runs.
- Especially useful for polymers, composites, hydrates, and materials with closely spaced transitions.



Conclusion

Step Isothermal TGA, standard on all AMI TGA instruments, enables high-resolution thermal analysis for complex materials by combining real-time mass-loss monitoring with intelligent temperature control. This feature adds analytical power and flexibility while remaining fully accessible through AMI's intuitive **InfinityPro** interface.