

Thermogravimetric Analysis and Calorimetry (Mexico)

## Unidad de Investigación en Termogravimetría y Calorimetría

Acetylene – Related terms: hydrocarbon, flame synthesis, CVD. A simple alkyne ( $C_2H_2$ ) used as a fuel in thermogravimetric analysis (TGA) for creating reducing atmospheres. Example: heating a sample under acetylene can prevent oxidation of metal oxides. Practical application includes studying catalyst stability during carburization. Challenge: controlling flame temperature to avoid sample damage.

Adsorption – Related terms: physisorption, chemisorption, surface area. The process by which molecules adhere to a solid surface. In TGA, adsorption of moisture can cause apparent weight gain before decomposition. Example: a silica sample may adsorb water, showing a pre-decomposition plateau. Practical use: determining surface area by coupling TGA with BET analysis. Challenge: distinguishing true decomposition from reversible adsorption.

Alloy – Related terms: intermetallic, phase diagram, solid solution. A mixture of two or more metals (or a metal and a non-metal) with a uniform composition. TGA can monitor oxidation kinetics of alloys. Example: studying the protective oxide layer formation on a Ni-Cr alloy. Practical application: assessing high-temperature corrosion resistance. Challenge: overlapping mass-loss events from multiple constituents.

Amorphous – Related terms: glassy, disorder, X-ray diffraction. A non-crystalline solid lacking long-range order. Amorphous polymers often display a glass-transition temperature ( $T_g$ ) detectable by DSC. Example: a polymer film showing a gradual heat-flow change at  $T_g$ . Practical use: designing materials with tailored mechanical flexibility. Challenge: broad transitions that can be obscured by baseline drift.

Atmosphere (TGA) – Related terms: inert gas, oxidative, purge. The gas environment surrounding the sample during analysis. Common atmospheres include nitrogen, argon, and air. Example: running a TGA under nitrogen to prevent oxidation of a polymer. Practical application: studying decomposition pathways under controlled oxygen levels. Challenge: leaks or residual moisture can alter mass-loss curves.

Baseline (DSC) – Related terms: reference pan, calibration, drift. The recorded signal from an empty sample holder used to correct the heat-flow data. Example: a DSC run with a sapphire standard to establish baseline stability. Practical use: improving accuracy of enthalpy calculations. Challenge: baseline drift at high temperatures may mask small transitions.

Calibration (TGA/DSC) – Related terms: standard material, sensitivity, offset. The process of adjusting instrument response using known references. Example: using indium melting to calibrate DSC temperature and enthalpy. Practical application: ensuring quantitative mass-loss measurements. Challenge: frequent recalibration needed for high-precision work.

Carburization – Related terms: carbon diffusion, hardening, steel. The introduction of carbon into a metal

surface to increase hardness. TGA can track weight gain as carbon atoms incorporate. Example: a steel sample exposed to acetylene shows a gradual mass increase. Practical use: optimizing surface treatments for wear resistance. Challenge: controlling carbon depth without causing brittleness.

Catalyst – Related terms: active site, turnover frequency, deactivation. A substance that accelerates a chemical reaction without being consumed. In TGA, catalysts may influence decomposition pathways. Example: a zeolite catalyst reducing the temperature of polymer degradation. Practical application: designing more efficient catalytic processes. Challenge: catalyst poisoning observed as unexpected mass-loss steps.

Char – Related terms: carbonaceous residue, pyrolysis, soot. The solid residue remaining after thermal decomposition of organic material in an inert atmosphere. Example: lignocellulosic biomass yielding 20% char at 500 °C. Practical use: assessing fuel quality and carbon sequestration potential. Challenge: distinguishing char formation from incomplete combustion.

Coefficient of Thermal Expansion (CTE) – Related terms: dilatometry, thermal strain, mismatch. A material property describing dimensional change with temperature. Measured by thermomechanical analysis (TMA) but relevant to TGA when sample size affects heat transfer. Example: a polymer with a high CTE expanding during heating, causing uneven temperature distribution. Practical application: designing composite layers with compatible CTEs. Challenge: anisotropic expansion complicates data interpretation.

Combustion Analysis – Related terms: elemental analysis, CHNS, oxidation. Determining elemental composition by burning a sample and measuring released gases. Example: a carbon-hydrogen analysis of a polymer to verify formula. Practical use: confirming synthesis purity. Challenge: incomplete combustion leading to erroneous nitrogen values.

Conductivity (Thermal) – Related terms: heat transfer, Fourier's law, insulator. The ability of a material to conduct heat. Influences temperature uniformity in TGA crucibles. Example: a metal crucible with high conductivity provides rapid temperature equilibration. Practical application: selecting crucible material to minimize temperature gradients. Challenge: low-conductivity samples causing lag between setpoint and actual temperature.

Cross-linking – Related terms: network polymer, cure, gel point. Formation of covalent bonds between polymer chains, increasing rigidity. Detected by DSC as an exothermic cure peak. Example: epoxy resin showing a peak at 150 °C corresponding to cross-linking. Practical use: optimizing cure schedules for composites. Challenge: overlapping exotherms with decomposition may obscure kinetic analysis.

Crystallization – Related terms: nucleation, growth, melting point. The process where a liquid or amorphous solid forms an ordered crystal lattice. DSC records an exothermic peak during crystallization from the melt. Example: poly( $\epsilon$ -caprolactone) crystallizing upon cooling at -30 °C. Practical application: controlling polymer morphology for mechanical properties. Challenge: slow crystallization rates requiring long isothermal holds.

Decomposition – Related terms: pyrolysis, thermal degradation, volatiles. The breakdown of a material into smaller fragments upon heating. TGA provides a mass-loss curve indicating decomposition temperature(s). Example: a polymer losing 80% of its mass between 300 °C and 450 °C. Practical use: determining thermal

stability for processing. Challenge: overlapping steps from additives or fillers.

Derivative Thermogravimetry (DTG) – Related terms: rate of mass loss, peak deconvolution, kinetic analysis. The first derivative of the TGA curve, highlighting temperatures where mass loss rate is maximal. Example: a DTG peak at 380 °C indicating the main degradation step of a polyester. Practical application: separating multiple decomposition events. Challenge: noise amplification requiring smoothing.

Diffusion – Related terms: Fick's law, interdiffusion, barrier. Movement of atoms or molecules through a material driven by concentration gradients. In TGA, diffusion controls the rate at which gases escape the sample. Example: oxygen diffusion through a polymer matrix limiting oxidation rate. Practical use: modeling kinetic data with diffusion-controlled models. Challenge: distinguishing diffusion control from chemical reaction control.

Dynamic Mechanical Analysis (DMA) – Related terms: storage modulus, loss modulus,  $\tan \delta$ . A technique measuring material response to oscillatory stress as a function of temperature. Complementary to DSC for identifying glass-transition temperature. Example: a polymer showing a peak in  $\tan \delta$  at 120 °C confirming  $T_g$ . Practical application: predicting performance under mechanical loading. Challenge: correlating DMA transitions with calorimetric data.

Enthalpy ( $\Delta H$ ) – Related terms: heat of reaction, calorimetry, exothermic. The heat content change associated with a process. Measured by DSC as the area under a heat-flow peak. Example: a melting enthalpy of 150 J g<sup>-1</sup> for a low-molecular-weight wax. Practical use: calculating energy requirements for material processing. Challenge: baseline drift can distort small enthalpy values.

Eutectic – Related terms: phase diagram, liquidus, solidus. A specific composition of a binary system that melts at the lowest possible temperature. Detected by DSC as a sharp melting peak. Example: a lead-tin alloy exhibiting a eutectic melt at 183 °C. Practical application: designing solder alloys with precise melting points. Challenge: narrow composition range demanding accurate sample preparation.

Exothermic – Related terms: heat release, combustion, crystallization. A process that releases heat to the surroundings, shown as a downward peak in DSC. Example: the curing of a phenolic resin releasing 200 J g<sup>-1</sup>. Practical use: monitoring cure progress in composite manufacturing. Challenge: overlapping exotherms can mask individual reaction steps.

First-order reaction – Related terms: kinetic model, rate constant, Arrhenius. A reaction whose rate depends linearly on the concentration of one reactant. In TGA, many decompositions follow first-order kinetics. Example: the mass loss of a polymer fitting a first-order model with activation energy 180 kJ mol<sup>-1</sup>. Practical application: predicting lifetime under thermal stress. Challenge: deviations due to diffusion or autocatalysis.

Fourier Transform Infrared Spectroscopy (FT-IR) Coupled TGA – Related terms: evolved gas analysis, spectroscopic identification, TG-FTIR. A technique linking TGA with FT-IR to identify gases released during heating. Example: detecting CO<sub>2</sub> and H<sub>2</sub>O from polyester degradation. Practical use: elucidating decomposition mechanisms. Challenge: overlapping absorption bands requiring careful deconvolution.

Glass-Transition Temperature ( $T_g$ ) – Related terms: amorphous, specific heat, viscoelastic. The temperature

where an amorphous material transitions from a rigid glassy state to a rubbery state. Detected by DSC as a step change in heat flow. Example: a polymer showing a  $T_g$  at 85 °C. Practical application: setting processing windows for extrusion. Challenge: weak transitions in low-mass samples may be hidden by baseline noise.

Gravimetric Calibration – Related terms: reference weight, sensitivity factor, drift. Adjusting the mass-measurement system of a TGA using certified standards. Example: using a 10 mg nickel standard to verify linearity. Practical use: ensuring quantitative mass loss data. Challenge: thermal expansion of the balance affecting accuracy at high temperatures.

Heat Capacity ( $C_p$ ) – Related terms: specific heat, calorimetric baseline, temperature dependence. The amount of heat required to raise the temperature of a unit mass by one degree. Measured by DSC as the slope of the baseline. Example: a metal with  $C_p \approx 0.5 \text{ J g}^{-1} \text{ K}^{-1}$ . Practical application: designing thermal management systems. Challenge:  $C_p$  changes near phase transitions complicate baseline correction.

Heat Flow (DSC) – Related terms: power input, exotherm, endotherm. The rate at which heat is supplied to or removed from a sample to maintain the same temperature as a reference. Example: a DSC trace showing an endothermic peak for melting. Practical use: quantifying transition enthalpies. Challenge: instrument lag causing peak shifting at high scan rates.

Heterogeneous Catalysis – Related terms: surface reaction, active site, support. Catalysis occurring at the interface between phases, typically solid catalyst and gaseous or liquid reactants. TGA can monitor catalyst deactivation by tracking mass changes due to coke formation. Example: a zeolite catalyst gaining mass from carbon deposits after a reaction cycle. Practical application: scheduling regeneration steps in petrochemical processes. Challenge: differentiating coke from adsorbed reactants.

Isoconversional Methods – Related terms: Kissinger-Akahira-Sunose, Friedman, activation energy. Techniques that calculate kinetic parameters without assuming a reaction model, based on constant conversion levels. Example: applying the Friedman method to a TGA dataset to obtain activation energy as a function of conversion. Practical use: revealing changes in mechanism during decomposition. Challenge: requires high-quality data at multiple heating rates.

Isothermal TGA – Related terms: constant-temperature hold, kinetic study, plateau. A TGA experiment performed at a fixed temperature to observe mass change over time. Example: holding a polymer at 350 °C for 2 h to monitor degradation rate. Practical application: modeling long-term thermal stability. Challenge: maintaining temperature uniformity for extended periods.

Joule Heating – Related terms: resistive heating, electric furnace, power dissipation. The conversion of electrical energy into heat due to resistance. In some DSC instruments, the sample is heated by Joule heating. Example: a DSC with a platinum heating element delivering precise power. Practical use: achieving rapid temperature ramps. Challenge: uneven heating may cause sample hotspots.

Kinetic Model – Related terms: reaction order, autocatalysis, master plot. A mathematical representation describing how a reaction progresses with time and temperature. Example: using the Avrami-Erofeev model to fit a polymer cure curve. Practical application: predicting conversion under different processing conditions. Challenge: selecting the correct model among many possibilities.

**Kissinger Method** – Related terms: peak temperature, heating rate, activation energy. A linear method to estimate activation energy from DSC peak temperatures at various heating rates. Example: plotting  $\ln(\beta/T_p^2)$  versus  $1/T_p$  to obtain  $E_a$  for a decomposition reaction. Practical use: quick kinetic assessment. Challenge: assumes a single, dominant reaction step.

**Lag Time (TGA)** – Related terms: thermal inertia, response time, equilibration. The delay between the programmed temperature and the actual temperature of the sample. Example: a lag of 5 °C observed at a 10 °C min<sup>-1</sup> ramp. Practical application: correcting kinetic calculations for accurate temperature values. Challenge: larger lag at high heating rates reduces data reliability.

**Mass Loss (TGA)** – Related terms: decomposition, volatilization, residue. The decrease in sample weight as temperature increases. Example: a 30 % mass loss occurring between 250 °C and 400 °C for a polymer. Practical use: quantifying volatile content. Challenge: overlapping loss events from additives may complicate interpretation.

**Mass Spectrometry (MS) Coupled TGA** – Related terms: evolved-gas analysis, TG-MS, fragmentation pattern. A technique linking TGA with MS to identify gas species released during heating. Example: detecting  $m/z = 44$  peak corresponding to CO<sub>2</sub> during polymer degradation. Practical application: mapping degradation pathways. Challenge: ionization efficiency varies with molecular weight leading to semi-quantitative data.

**Melting Point (T<sub>m</sub>)** – Related terms: solid-liquid transition, enthalpy of fusion, crystallinity. Temperature at which a solid becomes a liquid under atmospheric pressure. Detected by DSC as an endothermic peak. Example: a wax melting at 58 °C with  $\Delta H = 180 \text{ J g}^{-1}$ . Practical use: quality control of pharmaceuticals. Challenge: polymorphic substances may exhibit multiple melting peaks.

**Micro-Calorimetry** – Related terms: high sensitivity, low sample mass, isothermal titration calorimetry (ITC). Calorimetric techniques capable of measuring very small heat flows. Example: a micro-DSC detecting a 0.1 J g<sup>-1</sup> transition in a thin film. Practical application: studying thin-film polymer coatings. Challenge: baseline stability becomes critical at low signal levels.

**Mixture Rule (Heat Capacity)** – Related terms: additive property, rule of mixtures, composite. Approximation that the overall heat capacity of a mixture equals the weighted sum of its components. Example: calculating  $C_p$  of a polymer-filler composite. Practical use: predicting thermal behavior of composites. Challenge: interaction effects may cause deviations.

**Moisture Content** – Related terms: hygroscopic, desorption, water loss. Amount of water physically adsorbed in a sample. In TGA, moisture appears as an initial weight loss below 150 °C. Example: a wood sample losing 8 % mass due to water evaporation. Practical application: pre-drying protocols for accurate thermal analysis. Challenge: high humidity environments can re-adsorb moisture during analysis.

**Monomer** – Related terms: polymerization, repeat unit, initiator. The basic building block that links to form polymers. DSC can detect the exothermic polymerization of residual monomer. Example: residual styrene in a polymer showing a peak at 165 °C. Practical use: ensuring complete cure in composite laminates. Challenge: low monomer concentrations may be below detection limits.

**Multiphase System** – Related terms: heterogeneous, phase separation, interfacial area. A material composed of two or more distinct phases (e.g., polymer blend, composite). DSC may reveal multiple  $T_g$ 's corresponding to each phase. Example: a polymer blend showing  $T_g$  at 70 °C and 130 °C. Practical application: tailoring mechanical properties through phase control. Challenge: overlapping transitions can obscure individual phase behavior.

**Oxidation** – Related terms: combustion, oxidative degradation,  $O_2$  atmosphere. Reaction of a material with oxygen, often leading to weight gain (due to oxide formation) followed by weight loss (due to volatile oxide species). Example: a metal powder gaining mass up to 5% before decomposing at 600 °C in air. Practical use: evaluating corrosion resistance. Challenge: simultaneous oxidation and volatilization complicates kinetic analysis.

**Peak Temperature ( $T_p$ )** – Related terms: DSC, kinetic analysis, Kissinger method. The temperature at which a DSC or DTG peak reaches its maximum rate. Example:  $T_p = 380$  °C for the main degradation of a polyester. Practical application: input for activation energy calculations. Challenge: peak shifting with heating rate requires correction.

**Peroxide Initiator** – Related terms: radical generation, cure, exotherm. Chemical compound that decomposes to generate free radicals, initiating polymerization. DSC can monitor the exothermic cure of a peroxide-cured epoxy. Example: a bisphenol A epoxy cured with benzoyl peroxide showing a sharp exotherm at 140 °C. Practical use: controlling cure schedules in resin transfer molding. Challenge: premature decomposition may cause safety hazards.

**Phases (Solid/Liquid/Gas)** – Related terms: phase diagram, transition, equilibrium. Distinct states of matter with uniform physical properties. DSC tracks solid-liquid transitions; TGA can monitor solid-gas volatilization. Example: a solid polymer sublimating directly to gas at 350 °C under vacuum. Practical application: designing processes such as freeze-drying. Challenge: detecting solid-gas transitions without a clear heat-flow signal.

**Polymorphism** – Related terms: crystal forms, metastable, solid-solid transition. Existence of multiple crystal structures for the same chemical composition. DSC reveals polymorphic transitions as endothermic or exothermic peaks. Example: a pharmaceutical displaying a transition from Form I to Form II at 45 °C. Practical use: controlling drug bioavailability. Challenge: polymorph identification requires complementary X-ray diffraction.

**Porosity** – Related terms: void fraction, BET surface area, permeability. The fraction of a material's volume occupied by pores. In TGA, high porosity can accelerate mass loss due to increased surface area. Example: a porous carbon scaffold losing mass faster than a dense counterpart. Practical application: designing catalyst supports with high diffusion rates. Challenge: accurately measuring pore size distribution alongside thermal data.

**Pre-Exponential Factor ( $A$ )** – Related terms: Arrhenius equation, frequency factor, kinetic parameters. The constant representing the frequency of successful collisions in a reaction. Determined from kinetic fits to TGA/DSC data. Example:  $A = 1 \times 10^{13} \text{ s}^{-1}$  for polymer degradation. Practical use: predicting reaction rates at

different temperatures. Challenge: large uncertainties when data are noisy.

Pressure (TGA) – Related terms: vacuum, purge gas, partial pressure. The gas pressure inside the TGA chamber, influencing volatilization and oxidation. Example: running a TGA under 10 mbar to promote complete removal of volatiles. Practical application: studying low-pressure pyrolysis of polymers. Challenge: pressure fluctuations can cause baseline drift.

Primary Decomposition – Related terms: initial breakdown, first-order step, volatile release. The first major mass-loss event in a TGA curve, often corresponding to the cleavage of the weakest bonds. Example: a polymer losing 20% mass at 250°C as the primary decomposition. Practical use: identifying the onset of thermal failure. Challenge: distinguishing primary from secondary reactions when they overlap.

Purging – Related terms: gas flow, inert atmosphere, venting. The process of flowing gas through the TGA chamber to remove released volatiles and maintain a defined atmosphere. Example: a nitrogen purge at 50 mL min<sup>-1</sup> during a polymer degradation run. Practical application: preventing buildup of combustible gases. Challenge: insufficient flow can cause pressure spikes and inaccurate mass measurements.

Quartz Crucible – Related terms: inert container, high temperature, thermal shock. A crucible made of quartz used for TGA/DSC experiments requiring chemical inertness. Example: analyzing a silica sample without contamination from the crucible material. Practical use: studying reactive metals that would corrode metal pans. Challenge: quartz can fracture under rapid temperature changes.

Quench Cooling – Related terms: rapid quench, glass formation, thermal shock. Fast cooling of a sample from high temperature to “freeze” a structure. In DSC, quench cooling can suppress crystallization, allowing observation of the glass transition. Example: cooling a polymer melt at 100°C s<sup>-1</sup> to retain the amorphous state. Practical application: producing amorphous alloys with superior mechanical properties. Challenge: achieving uniform cooling in larger samples.

Reaction Mechanism – Related terms: pathway, intermediate, kinetic model. The step-by-step sequence of elementary reactions leading from reactants to products. TGA-MS or TGA-FTIR data help elucidate mechanisms. Example: identifying CO<sub>2</sub> and CH<sub>4</sub> as primary products of polymer pyrolysis. Practical use: designing inhibitors to improve thermal stability. Challenge: complex mechanisms may involve parallel and consecutive steps.

Reference Material (DSC) – Related terms: calibration standard, indium, sapphire. A material with known transition temperatures and enthalpies used to calibrate DSC instruments. Example: indium melting at 156.6°C with  $\Delta H = 28.45 \text{ J g}^{-1}$ . Practical use: ensuring temperature accuracy across labs. Challenge: degradation of the reference over repeated cycles.

Residual Stress – Related terms: thermal strain, warpage, annealing. Stress locked into a material due to non-uniform cooling or curing. Detected indirectly by DSC as shifts in T<sub>g</sub> after annealing. Example: a cured composite showing a higher T<sub>g</sub> after a post-cure heat treatment. Practical application: reducing warpage in molded parts. Challenge: measuring stress quantitatively requires complementary techniques.

Rheology – Related terms: viscosity, shear rate, flow behavior. Study of material flow under applied stress.

While not a thermal analysis technique, rheology data complement DSC/TGA for processing polymers. Example: a polymer melt viscosity dropping sharply near its  $T_g$ . Practical use: optimizing extrusion parameters. Challenge: correlating rheological changes with thermal transitions.

Sample Preparation – Related terms: grinding, drying, mass selection. The steps taken to ready a specimen for TGA/DSC measurement. Example: grinding a polymer into a fine powder and drying at 80 °C for 2 h before analysis. Practical use: minimizing thermal gradients and ensuring reproducibility. Challenge: avoiding contamination or altering the material's intrinsic properties.

Secondary Decomposition – Related terms: char oxidation, volatile evolution, multi-step. A later mass-loss event following the primary decomposition, often associated with the breakdown of residues. Example: a polymer char oxidizing between 600 °C and 750 °C, causing a second DTG peak. Practical application: estimating total combustible loss. Challenge: overlapping with oxidation of the sample holder.

Self-Heating – Related terms: exothermic runaway, adiabatic, safety. The phenomenon where a material's own exothermic reaction raises its temperature, accelerating the reaction. Detected in DSC as a rapidly rising baseline. Example: a peroxide-cured resin exhibiting self-heating at 150 °C. Practical use: designing safe processing windows. Challenge: preventing thermal runaway in large batch reactors.

Signal-to-Noise Ratio (SNR) – Related terms: baseline noise, detector sensitivity, smoothing. A measure of data quality, defined as the ratio of the true signal amplitude to the background noise level. Example: a DSC trace with SNR = 20 for a small melting peak. Practical application: determining minimum detectable transition. Challenge: low SNR may require signal averaging or slower scan rates.

Sintering – Related terms: densification, particle coalescence, grain growth. The process of compacting powder particles into a solid mass through heat without melting. DSC can detect the onset of sintering as an exothermic event. Example: ceramic powders beginning to sinter at 1200 °C. Practical use: producing high-strength ceramics. Challenge: controlling shrinkage to avoid dimensional defects.

Solvent Evaporation – Related terms: drying, volatilization, residual solvent. Loss of solvent from a sample during heating, often observed as a low-temperature mass loss. Example: a polymer film losing 5% mass between 50 °C and 120 °C due to residual THF. Practical application: verifying complete removal of processing solvents. Challenge: overlapping solvent loss with moisture desorption.

Specific Heat ( $C_p$ ) – Related terms: heat capacity, enthalpy, temperature dependence. The heat required to raise the temperature of one gram of a substance by one kelvin. Measured by DSC as the slope of the baseline. Example: water having  $C_p \approx 4.18 \text{ J g}^{-1} \text{ K}^{-1}$ . Practical use: calculating energy balance in thermal processes. Challenge:  $C_p$  may change sharply near phase transitions, requiring piecewise fitting.

Standard Deviation ( $\sigma$ ) – Related terms: reproducibility, statistical analysis, error bars. A statistical metric indicating the spread of repeated measurements. Example: three DSC runs of a polymer melting point showing  $\sigma = 0.3 \text{ }^\circ\text{C}$ . Practical application: assessing instrument precision. Challenge: low sample mass can increase  $\sigma$  due to instrumental noise.

Stoichiometry – Related terms: reaction balance, mole ratio, limiting reagent. The quantitative relationship

between reactants and products in a chemical reaction. In TGA, stoichiometry helps predict expected mass loss. Example: a polymer with formula  $C_6H_{10}O_5$  losing 60% of its mass upon complete oxidation to  $CO_2$  and  $H_2O$ . Practical use: designing experiments to achieve complete conversion. Challenge: side reactions may alter the observed mass change.

**Thermal Conductivity** – Related terms: heat transfer, Fourier's law, insulator. Ability of a material to conduct heat. Influences temperature uniformity within a TGA crucible. Example: a metal sample with high conductivity equilibrating quickly, while a polymer sample lags behind. Practical application: selecting crucible materials to minimize gradients. Challenge: low-conductivity samples may require longer equilibration times.

**Thermal Decomposition** – Related terms: pyrolysis, volatilization, char formation. The breakdown of a material into smaller fragments when heated. TGA provides the temperature range and extent of decomposition. Example: a polymer decomposing between 300 °C and 500 °C, yielding a 20% char residue. Practical use: evaluating suitability of materials for high-temperature applications. Challenge: overlapping decomposition steps from additives.

**Thermal Diffusivity** – Related terms: heat capacity, conductivity, penetration depth. Measure of how quickly heat spreads through a material. Influences the rate at which a sample reaches the programmed temperature. Example: a low-diffusivity polymer requiring longer dwell times for temperature equilibration. Practical application: designing heating profiles for uniform processing. Challenge: calculating diffusivity from limited experimental data.

**Thermal Gravimetric Analysis (TGA)** – Related terms: mass loss, DTG, kinetic study. An analytical technique that records the change in mass of a sample as a function of temperature or time. Example: a TGA run from 30 °C to 800 °C at 10 °C min<sup>-1</sup> revealing three distinct mass-loss steps. Practical use: determining moisture content, decomposition temperatures, and residual ash. Challenges: baseline drift, overlapping events, and instrument lag.

**Thermal Stability** – Related terms: decomposition temperature, onset, lifetime. The resistance of a material to thermal degradation. Assessed by the temperature at which significant mass loss begins in TGA. Example: a polymer with an onset of degradation at 350 °C considered thermally stable for processing at 250 °C. Practical application: selecting materials for aerospace components. Challenge: additives may mask the true stability of the base polymer.

**Thermal Transition** – Related terms: glass transition, melting, crystallization. Any temperature-induced change in a material's physical state detected by DSC. Example: a polymer showing both T<sub>g</sub> at 80 °C and melting at 150 °C. Practical use: defining processing windows for injection molding. Challenge: weak transitions may be hidden by baseline noise.

**Thermogravimetric Curve** – Related terms: TGA plot, mass versus temperature, residual mass. The graphical representation of sample weight change during a TGA experiment. Example: a curve with a flat plateau up to 200 °C followed by a steep decline. Practical application: visual identification of decomposition steps. Challenge: interpreting small inflections without derivative analysis.

**Thermodynamic Equilibrium** – Related terms: Gibbs free energy, phase stability, reversible. The state where a system's macroscopic properties remain constant over time. In DSC, reversible transitions (e.g.,  $T_g$ ) are near equilibrium, while irreversible reactions (e.g., oxidation) are not. Example: a polymer reaching equilibrium between amorphous and crystalline domains during a slow DSC scan. Practical use: predicting material behavior under slow heating. Challenge: rapid scan rates drive the system out of equilibrium, altering observed transitions.

**Thermodynamic Parameters** – Related terms:  $\Delta H$ ,  $\Delta S$ ,  $\Delta G$ . Quantities describing the energetics of a process. DSC directly provides  $\Delta H$ ; entropy change  $\Delta S$  can be derived from temperature and  $\Delta H$ . Example: melting of a crystal with  $\Delta H = 120 \text{ J g}^{-1}$  and  $\Delta S = 0.4 \text{ J g}^{-1} \text{ K}^{-1}$ . Practical application: assessing spontaneity of phase changes. Challenge: accurate entropy determination requires precise baseline.

**Time-Temperature Superposition (TTS)** – Related terms: master curve, viscoelasticity, shift factor. A principle allowing data from different temperatures to be superimposed onto a single curve by horizontal shifting. Example: constructing a master curve for polymer creep using DSC-derived  $T_g$  data. Practical use: predicting long-term behavior from short-term tests. Challenge: non-linear shift factors for complex materials.

**Thermal Runaway** – Related terms: exothermic reaction, safety, adiabatic. Uncontrolled acceleration of a reaction due to the heat generated exceeding the system's ability to dissipate it. Detected in DSC as a rapidly rising baseline that may exceed instrument limits. Example: a peroxide-cured epoxy heating uncontrollably at  $160^\circ\text{C}$ . Practical use: designing safety interlocks for reactors. Challenge: predicting onset requires accurate kinetic parameters.

**Transition Metal Catalyst** – Related terms: coordination complex, oxidation state, activity. Metals such as Fe, Ni, or Pt that facilitate chemical reactions. In TGA, transition metal catalysts can lower degradation temperatures of polymers. Example: a nickel catalyst causing polymer depolymerization at  $250^\circ\text{C}$  instead of  $350^\circ\text{C}$ . Practical application: catalytic recycling of plastics. Challenge: catalyst deactivation via sintering or coke formation.

**Tri-dimensional (3D) Printing Materials** – Related terms: additive manufacturing, filament, cure. Materials designed for use in 3D printers, often thermoplastics or photopolymers. DSC determines melting or curing behavior; TGA assesses thermal stability. Example: a PLA filament melting at  $180^\circ\text{C}$  with negligible mass loss up to  $250^\circ\text{C}$ . Practical use: selecting feedstock for high-temperature extrusion. Challenge: additives (colorants, plasticizers) may introduce extra thermal events.

**Vacuum TGA** – Related terms: low pressure, desorption, pyrolysis. Performing TGA under reduced pressure to enhance removal of volatiles. Example: a polymer heated under 5 mbar showing earlier mass loss compared to ambient pressure. Practical application: simulating space-environment degradation. Challenge: maintaining consistent pressure throughout the run.

**Viscosity** – Related terms: flow resistance, shear rate, rheology. Resistance of a fluid to deformation. While not directly measured by TGA/DSC, viscosity changes near  $T_g$  are important for processing. Example: a polymer viscosity dropping by three orders of magnitude when passing its  $T_g$ . Practical use: determining optimal extrusion temperatures. Challenge: correlating viscosity data with calorimetric transitions for

complex blends.

Weight Percent (wt%) – Related terms: composition, mass fraction, concentration. The proportion of a component expressed as a percentage of the total mass. Used in TGA to report residual ash or char. Example: a sample leaving 12 wt% residue after complete decomposition. Practical application: comparing purity of different batches. Challenge: accurate weighing of small samples (Zero-Order Reaction – Related terms: constant rate, kinetic model, surface reaction. A reaction whose rate is independent of reactant concentration. In TGA, surface-controlled oxidation may follow zero-order kinetics. Example: a metal oxide gaining mass linearly with time at a fixed temperature. Practical use: simplifying kinetic analysis for surface reactions. Challenge: real systems often deviate due to diffusion limitations.

Zona de Investigación en Termogravimetría y Calorimetría (Unidad de Investigación en Termogravimetría y Calorimetría) – Related terms: research unit, thermogravimetric analysis, calorimetry, Universidad Nacional Autónoma de México (UNAM). A specialized research group within the Mexican university system focusing on advanced thermal analysis techniques, including TGA, DSC, TG-MS, and TG-FTIR. The unit conducts fundamental studies on polymer degradation, catalyst stability, and material characterization, providing training for graduate students and collaborating with industry for process optimization. Practical applications include developing high-temperature polymers for aerospace, designing corrosion-resistant alloys, and improving waste-to-energy conversion processes. Challenges faced by the unit involve acquiring high-resolution instruments, managing sample preparation for heterogeneous materials, and integrating multi-modal data (thermal, spectroscopic, and kinetic) to build comprehensive degradation models. The unit also emphasizes the importance of accurate calibration, reproducibility, and safety protocols when handling exothermic reactions and high-temperature operations.

Zero-Shift Correction – Related terms: baseline alignment, temperature offset, calibration. Adjusting the temperature axis of a DSC/TGA run to compensate for systematic deviations. Example: applying a  $-2^{\circ}\text{C}$  correction after observing the indium melting point consistently  $2^{\circ}\text{C}$  high. Practical use: ensuring comparability across instruments. Challenge: determining the correct shift when multiple reference points are unavailable.