Thermogravimetric Analysis and Calorimetry (Mexico)

Unidad de Técnicas Calorimétricas y Termogravimétricas

Activation Energy
Related terms: Arrhenius equation, kinetic analysis, isoconversional method
The activation energy is the minimum energy required for a chemical reaction or a degradation process to occur. In thermogravimetric analysis (TGA) it is derived from the temperature dependence of the rate of mass loss. Commonly, the Arrhenius equation $k = A \cdot \exp(-Ea/RT)$ is rearranged to calculate Ea from a series of TGA runs at different heating rates. Example: a polymer shows a single degradation step; plotting ln(heating rate) versus 1/T at a fixed conversion yields a straight line whose slope gives Ea. Practical challenges include overlapping reactions, baseline drift, and the need for precise temperature calibration.

Baseline
Related terms: instrument drift, reference run, signal correction
The baseline is the instrument response when no sample is present, serving as a reference for subsequent measurements. In both TGA and differential scanning calorimetry (DSC) a stable baseline ensures accurate determination of mass changes or heat flow. For instance, a DSC run of an empty pan should produce a flat line; any curvature indicates instrument drift that must be corrected. Baseline correction is often performed by subtracting a reference run or applying software algorithms. Challenges arise when the baseline varies with temperature, especially at high heating rates, requiring frequent recalibration.

Calibration
Related terms: standard material, temperature accuracy, sensitivity factor
Calibration aligns the instrument’s temperature and signal scales with known standards. For TGA, a certified reference material such as a metal oxide with a defined decomposition temperature is heated to verify temperature accuracy. In DSC, calibration employs substances with well-characterized enthalpy changes, like indium (melting point 156.6 °C, $\Delta H = 28.45\,J \cdot g^{-1}$). Proper calibration eliminates systematic errors, improves reproducibility, and allows comparison across laboratories. Practical issues include aging of thermocouples, changes in furnace atmosphere, and the need to repeat calibration after maintenance.

Derivative Thermogravimetry
Related terms: DTG curve, rate of mass loss, peak identification
Derivative thermogravimetry (DTG) plots the first derivative of mass versus temperature, highlighting the rate at which a sample loses weight. Peaks in a DTG curve correspond to distinct degradation steps, making it easier to resolve overlapping processes. For example, a composite material may show two DTG peaks at 320 °C and 460 °C, indicating separate polymer and filler decompositions. The DTG technique aids in selecting appropriate kinetic models and in estimating activation energies. Interpretation can be complicated by noise, baseline fluctuations, and the need for smoothing algorithms.

Endothermic Process
Related terms: heat absorption, melting, DSC signal
An endothermic process absorbs heat from the surrounding environment, producing a downward peak in DSC (when plotted as heat flow versus temperature). Typical examples include melting, sublimation, and certain phase transitions. In a DSC run of polyethylene, the melting endotherm appears near 130 °C with a characteristic enthalpy change. Recognizing endothermic events is essential for material identification and purity assessment. Challenges involve overlapping exothermic and endothermic signals, baseline drift, and the influence of heating rate on peak shape.

Exothermic Process
Related terms: heat release, crystallization, oxidation
An exothermic process releases heat, generating an upward peak in DSC. Common exotherms include crystallization of polymers, oxidation reactions, and combustion. For instance, the oxidative degradation of a polymer may produce a broad exothermic peak around 350 °C in a TGA-DSC coupled experiment. Exothermic signals help evaluate thermal stability and fire-hazard potential. Interpretation difficulties arise when exothermic and endothermic events overlap, when the sample mass is large, or when the furnace atmosphere changes during the run.

Furnace
Related terms: temperature uniformity, heating element, atmosphere control
The furnace is the component that provides the controlled temperature environment for TGA and DSC measurements. It houses the sample pan, reference pan, and thermocouple. Uniform temperature distribution is critical; gradients can lead to inaccurate mass loss or heat flow data. Modern furnaces allow programmable heating rates, isothermal holds, and rapid cooling. Practical considerations include the choice of heating element (e.g., ceramic vs. metal), the type of gas flow (nitrogen, argon, air), and the maintenance of seals to prevent leaks. Common challenges are thermal lag, especially at high heating rates, and degradation of furnace liners over time.

Heating Rate
Related terms: ramp speed, kinetic analysis, thermal lag
Heating rate ($°C\,min^{-1}$) defines how quickly the temperature is increased during a run. Typical rates range from $1\,°C\,min^{-1}$ to $20\,°C\,min^{-1}$, though rapid-scan instruments can exceed $100\,°C\,min^{-1}$. The heating rate influences the position and shape of TGA and DSC peaks; higher rates shift peaks to higher temperatures and can broaden them. In kinetic studies, multiple heating rates are required for isoconversional methods to calculate activation energy. Selecting an appropriate rate balances resolution, analysis time, and sample stability. Excessive rates may cause thermal gradients, while very low rates increase experiment duration and risk of baseline drift.

Isoconversional Method
Related terms: Kissinger-Akahira-Sunose, model-free analysis, conversion degree
The isoconversional method determines kinetic parameters without assuming a specific reaction model. It analyses data at constant conversion levels (α) across different heating rates, plotting ln(heating rate) versus 1/Tα to extract activation energy for each α. Common implementations include the

Kissinger-Akahira-Sunose (KAS) and Flynn-Wall-Ozawa (FWO) approaches. For a polymer, the method may reveal that Ea varies from 150 kJ·mol^{-1} at low conversion to 200 kJ·mol^{-1} at high conversion, indicating a change in degradation mechanism. Limitations involve the need for high-quality data, accurate temperature measurement, and the assumption that Ea is independent of heating rate at a given α.

Kinetic Model
Related terms: reaction order, autocatalytic, nucleation-growth
A kinetic model mathematically describes how a material's mass or heat changes with time or temperature. Common models include first-order, nth-order, diffusion-controlled, and autocatalytic (e.g., Šesták–Berggren). Selecting an appropriate model enables prediction of material behavior under different thermal conditions. For example, the decomposition of a nitrate salt may fit a first-order kinetic model, while polymer cross-linking often follows an autocatalytic scheme. Model selection is guided by fitting residuals, physical plausibility, and comparison with DTG peak shapes. Mis-selection can lead to erroneous lifetime predictions and safety assessments.

Mass Loss
Related terms: percentage weight change, degradation, volatile release
Mass loss quantifies the decrease in sample weight as temperature rises, expressed as a percentage of the initial mass. In TGA, mass loss steps correspond to processes such as moisture evaporation, decomposition, or combustion. For a hydrated mineral, a 5 % loss near 120 °C typically indicates loss of adsorbed water. Accurate mass loss measurement requires stable balance, proper pan selection, and minimal buoyancy effects. Challenges include overlapping events, residual gases affecting balance sensitivity, and the need to correct for baseline drift.

Sample Preparation
Related terms: pan selection, powder grinding, moisture control
Sample preparation ensures reproducible and representative measurements. For TGA, a small amount (2–10 mg) of finely ground material is placed in an alumina or platinum pan. Uniform particle size reduces thermal gradients and improves kinetic analysis. Moisture control is critical; samples are often dried at 105 °C before analysis to avoid spurious mass loss. In DSC, sample mass typically ranges from 5 to 20 mg, and the pan must be sealed to prevent oxidation. Common pitfalls include incomplete drying, uneven spreading, and contamination from the pan.

Thermogravimetric Analysis (TGA)
Related terms: mass change, degradation temperature, kinetic study
Thermogravimetric analysis measures the mass of a sample as a function of temperature or time under a programmed atmosphere. It provides information on thermal stability, composition, and kinetic parameters. For example, a polymer may show a single 80 % mass loss between 300 °C and 450 °C, indicating complete decomposition. TGA data are often coupled with FTIR or mass spectrometry to identify evolved gases. Limitations include the inability to directly measure heat flow, the need for inert atmospheres for non-oxidative studies, and potential sample-size effects.

Differential Scanning Calorimetry (DSC)
Related terms: heat flow, enthalpy change, phase transition

Differential scanning calorimetry records the heat flow difference between a sample and an inert reference as temperature is swept. It detects endothermic and exothermic events, providing transition temperatures and enthalpy values. A typical DSC of a crystalline polymer shows a melting endotherm at 150 °C with $\Delta H = 45\,J \cdot g^{-1}$. DSC can also be used to determine glass transition temperatures (Tg) by observing a step change in heat capacity. Challenges involve baseline stability, overlapping peaks, and the influence of heating rate on peak temperature.

Atmosphere Control
Related terms: inert gas, oxidative environment, purge flow
Atmosphere control dictates the gas composition surrounding the sample during thermal analysis. An inert atmosphere (nitrogen or argon) prevents oxidation, allowing study of intrinsic thermal stability. An oxidative atmosphere (air or oxygen) evaluates combustion behavior and oxidation kinetics. Flow rate (e.g., $50\,mL\,min^{-1}$) must be sufficient to purge evolved gases and maintain uniform composition. Improper control can lead to secondary reactions, such as char oxidation during polymer degradation, skewing mass loss data. Leak detection and proper tubing selection are essential for reliable results.

Baseline Drift
Related terms: instrument stability, temperature compensation, signal correction
Baseline drift refers to gradual changes in the instrument’s zero signal over time, often due to temperature fluctuations, electronic noise, or aging components. In TGA, drift can appear as a slow mass change unrelated to the sample. In DSC, drift manifests as a sloping baseline that complicates enthalpy integration. Regular baseline checks, software correction, and proper instrument warm-up reduce drift. Persistent drift may indicate the need for maintenance or replacement of thermocouples and balance components.

Buoyancy Effect
Related terms: gas density, balance correction, sample volume
During a TGA run, the surrounding gas density changes with temperature, altering the buoyant force on the sample pan. This creates an apparent mass change unrelated to chemical processes. The effect is more pronounced at high temperatures and with large sample volumes. Corrections involve measuring an empty pan under identical conditions and subtracting the buoyancy contribution, or using software algorithms that account for gas density. Failure to correct buoyancy can lead to over-estimation of mass loss, especially for low-mass samples.

Calibration Curve
Related terms: standard reference, linearity, sensitivity factor
A calibration curve plots instrument response versus known quantities of a standard material, establishing the relationship between signal and mass or heat flow. In DSC, a calibration curve using indium and zinc standards verifies both temperature and enthalpy accuracy across a range of temperatures. In TGA, a curve using a series of metal oxides with known decomposition temperatures confirms temperature linearity. The slope of the curve provides the sensitivity factor, which is applied to unknown samples. Regular verification ensures analytical reliability; deviations may indicate sensor drift or fouling.

Decomposition Temperature
Related terms: onset temperature, peak temperature, thermal stability

The decomposition temperature is the temperature at which a material begins to break down chemically, resulting in mass loss or heat evolution. It is often reported as the onset temperature (where the derivative curve first deviates from baseline) or the peak temperature (maximum rate of mass loss). For a cellulose sample, the onset may be around 260 °C, with a peak near 340 °C. Accurate determination requires a stable baseline, appropriate heating rate, and proper sample size. Overlapping reactions can obscure the true onset, necessitating deconvolution techniques.

Derivative Heat Flow
Related terms: dH/dT, peak sharpening, kinetic interpretation
Derivative heat flow represents the rate of change of heat flow with temperature, obtained by differentiating the DSC signal. Peaks in the derivative correspond to rapid thermal events, making it easier to locate transition temperatures precisely. For a polymer with a broad melting range, the derivative heat flow highlights the temperature of maximum melting rate. This information assists in kinetic modeling and in comparing materials with similar DSC profiles. Noise amplification is a common challenge; smoothing algorithms are often applied before differentiation.

Dynamic Atmosphere
Related terms: flow switching, programmed gas, reactive analysis
A dynamic atmosphere changes the gas composition during a thermal run, enabling studies of reactions under varying conditions. For example, a TGA experiment may start under nitrogen to assess pyrolysis, then switch to air to evaluate oxidative degradation of the residual char. Programmable gas switching allows investigation of oxidation-reduction cycles, catalyst activation, and moisture sorption. Implementation requires reliable valves, rapid purge, and careful timing to avoid pressure spikes. Mis-timing can introduce artifacts, such as premature oxidation or incomplete gas exchange.

Evolved Gas Analysis (EGA)
Related factors: FTIR coupling, mass spectrometry, gas identification
Evolved gas analysis couples TGA with spectroscopic or mass-spectrometric detectors to identify gases released during thermal events. A TGA-FTIR system records infrared spectra of vapors, revealing functional groups like CO_2, H_2O, or organic fragments. A TGA-MS setup provides molecular weight information, distinguishing between CO, CO_2, and hydrocarbons. EGA is valuable for polymer degradation studies, combustion research, and safety assessments. Challenges include maintaining a leak-free transfer line, calibrating detector response, and dealing with overlapping gas signals at high temperatures.

Heat Capacity
Related terms: Cp, specific heat, DSC baseline
Heat capacity (Cp) is the amount of heat required to raise the temperature of a unit mass by one degree. In DSC, Cp is measured as the baseline slope before any transition occurs. The difference in Cp between phases yields the step change at the glass transition temperature. For amorphous polymers, Cp may increase from $1.2\,J{\cdot}g^{-1}\,K^{-1}$ (glassy) to $1.5\,J{\cdot}g^{-1}\,K^{-1}$ (rubbery). Accurate Cp determination requires a stable baseline, proper reference material, and correction for instrument heat flow. Errors can arise from pan heat loss, thermal lag, and insufficient equilibration time.

Glass Transition Temperature (Tg)

Related terms: amorphous polymer, step change, DSC
The glass transition temperature marks the transition of an amorphous material from a rigid glassy state to a flexible rubbery state. In DSC, Tg appears as a step change in heat flow rather than a peak. For polystyrene, Tg is typically around 100 °C. Determination methods include the midpoint of the step, the extrapolation of baseline slopes, or the maximum of the derivative heat flow. Tg is critical for processing, performance, and storage conditions. Accurate measurement may be hindered by overlapping relaxations, poor baseline stability, or insufficient heating rate.

Instrument Drift

Related terms: temperature offset, signal stability, routine maintenance
Instrument drift denotes gradual changes in temperature or signal accuracy over time, often caused by aging components, thermocouple degradation, or electronic noise. In TGA, drift can shift the apparent decomposition temperature; in DSC, it can alter enthalpy values. Regular calibration, periodic replacement of sensors, and software compensation mitigate drift. Persistent drift may require a full instrument service. Users should monitor drift by running standard materials at the start and end of each batch of analyses.

Isothermal Hold

Related terms: steady-state, kinetic pause, thermal equilibration
An isothermal hold maintains the sample at a constant temperature for a predetermined duration. It is used to study reaction kinetics at a fixed temperature, allowing the determination of rate constants without the influence of temperature ramps. For example, a polymer may be held at 300 °C for 30 minutes to observe the rate of mass loss. Isothermal experiments require precise temperature control and sufficient equilibration time to avoid thermal gradients. Challenges include ensuring the furnace reaches the set temperature quickly and maintaining a stable atmosphere during the hold.

Kinetic Parameter

Related terms: pre-exponential factor, activation energy, reaction order
Kinetic parameters quantify the speed and mechanism of a thermal reaction. The primary parameters are the activation energy (Ea), the pre-exponential factor (A), and the reaction order (n). These values are extracted from TGA or DSC data using model-fitting or model-free methods. For a first-order degradation, the rate constant k follows $k = A \cdot \exp(-Ea/RT)$. Accurate kinetic parameters enable prediction of material lifetime under service conditions. Uncertainties arise from experimental noise, selection of the kinetic model, and assumptions about reaction mechanism.

Mass Spectrometry (MS) Detector

Related terms: EGA, ionization, fragment analysis
A mass spectrometry detector identifies gases evolved during TGA by measuring mass-to-charge ratios of ionized fragments. Coupling TGA with MS provides real-time qualitative and quantitative data on decomposition products. For a polymer, MS may reveal peaks corresponding to $m/z = 44$ (CO_2) and $m/z = 28$ (CO). Calibration with known gases improves quantification. Challenges include maintaining a high-vacuum environment, avoiding ion source contamination, and interpreting overlapping fragment patterns.

Modular Furnace Design

Related terms: interchangeable chambers, custom atmospheres, scalability

Modular furnace design allows the user to swap chambers or liners to accommodate different sample sizes, atmospheres, or temperature ranges. In advanced TGA/DSC systems, modules can be configured for high-pressure studies, rapid heating, or combined spectroscopic analysis. This flexibility supports diverse research needs, from polymer degradation to catalyst testing. Mechanical seals and quick-connect gas lines must be reliable to prevent leaks. Modular systems may increase initial cost but reduce long-term maintenance by allowing targeted component replacement.

Multiple Heating Rates
Related terms: Kinetic study, isoconversional analysis, data set expansion
Performing TGA runs at several heating rates (e.g., 5, 10, 15 °C min^{-1}) provides the data needed for model-free kinetic methods. The variation in peak temperature with heating rate reveals the activation energy and reaction mechanism. For a material that decomposes in a single step, the peak temperature shifts linearly with the logarithm of heating rate. Using multiple rates improves the robustness of kinetic parameters but increases experimental workload. Care must be taken to maintain identical sample preparation and atmosphere across all runs to ensure comparability.

Oxidative Degradation
Related terms: air atmosphere, combustion, TGA-DSC coupling
Oxidative degradation occurs when a material reacts with oxygen, leading to mass loss and exothermic heat flow. In TGA, an oxidative run under air shows a rapid mass drop at lower temperatures compared to an inert run, reflecting combustion of the residual char. DSC may record a concurrent exothermic peak. Oxidative studies are essential for fire-safety assessment and for understanding aging of polymers in service. Controlling oxygen concentration, flow rate, and temperature ramp is crucial to avoid runaway reactions. Data interpretation must separate pure thermal decomposition from oxidation effects.

Peak Deconvolution
Related terms: Gaussian fitting, overlapping reactions, software analysis
Peak deconvolution separates overlapping thermal events into individual components using mathematical functions such as Gaussian or Lorentzian shapes. In DTG or DSC curves with multiple overlapping peaks, deconvolution yields the temperature and intensity of each underlying process. For a composite material, deconvolution may reveal three distinct decomposition steps at 280 °C, 350 °C, and 420 °C. Accurate deconvolution requires high-quality data, proper baseline subtraction, and reasonable initial guesses. Over-fitting can produce spurious peaks, so validation against independent techniques (e.g., EGA) is recommended.

Polymer Crystallinity
Related terms: melting enthalpy, DSC, degree of crystallinity
Polymer crystallinity quantifies the fraction of ordered crystalline regions within a polymer matrix. DSC measures the melting enthalpy (ΔHm) of a polymer; dividing ΔHm by the enthalpy of a 100 % crystalline reference yields the degree of crystallinity. For example, a ΔHm of 70 $\mathrm{J{\cdot}g^{-1}}$ for polyethylene (reference ΔHm = 293 $\mathrm{J{\cdot}g^{-1}}$) corresponds to ~24 % crystallinity. Crystallinity influences mechanical properties, barrier performance, and thermal stability. Accurate determination requires baseline correction, proper heating rate, and consideration of re-crystallization during cooling.

Pre-exponential Factor (A)
Related terms: frequency factor, Arrhenius equation, kinetic modeling
The pre-exponential factor represents the frequency of successful collisions leading to reaction in the Arrhenius expression. It combines molecular orientation and vibrational contributions. In kinetic analysis of TGA data, A is obtained alongside Ea by fitting a chosen model to the experimental conversion versus temperature data. Typical values range from 10^6 to $10^{13}\,s^{-1}$ for solid-state reactions. Accurate A estimation depends on correct model selection; an inappropriate model can inflate or deflate A dramatically. Validation against literature values helps assess plausibility.

Reference Material
Related terms: standard, calibration, known transition
A reference material is a substance with well-characterized thermal properties used for instrument calibration. In DSC, metals such as indium, zinc, and tin serve as standards for temperature and enthalpy. In TGA, certified oxides or polymers with known decomposition temperatures provide temperature verification. The reference should be homogeneous, stable, and free of contaminants. Regular use of reference materials ensures consistent performance across different laboratories and over time. Degradation of the reference itself can introduce systematic errors, so periodic replacement is advised.

Reproducibility
Related terms: repeatability, standard deviation, method validation
Reproducibility assesses the degree to which repeated measurements under unchanged conditions yield the same result. In thermal analysis, it is expressed as the relative standard deviation of peak temperatures, enthalpy values, or mass loss percentages across multiple runs. High reproducibility ($\leq 2\,\%$ RSD) indicates reliable methodology and instrument stability. Factors influencing reproducibility include sample heterogeneity, pan sealing, gas flow consistency, and operator technique. Documenting all experimental parameters and adhering to standard operating procedures enhances reproducibility.

Sample Pan
Related terms: aluminum pan, platinum crucible, sealed pan
The sample pan holds the material during TGA or DSC measurements. Choice of pan material depends on the temperature range, reactivity, and required sealing. Aluminum pans are common for low-temperature TGA; platinum crucibles are used for high-temperature or oxidative studies due to their inertness. Sealed pans prevent loss of volatile components and are essential for moisture-sensitive samples. Pan geometry influences heat transfer; larger surface area improves temperature uniformity but may increase buoyancy effects. Proper cleaning and handling prevent cross-contamination.

Scanning Rate
Related terms: temperature ramp, data resolution, kinetic influence
Scanning rate, synonymous with heating rate, determines how quickly temperature is increased during a run. Faster rates reduce experiment time but can broaden peaks and shift transition temperatures to higher values. Slower rates improve resolution but increase susceptibility to baseline drift and prolong analysis. Selecting an optimal scanning rate balances these factors and aligns with the objectives of the study—whether qualitative identification or quantitative kinetic modeling. Consistency of scanning rate across

comparative runs is essential for reliable interpretation.

Thermal Lag
Related terms: temperature gradient, response time, furnace-sample equilibration
Thermal lag describes the delay between the programmed furnace temperature and the actual temperature of the sample. It becomes significant at high heating rates or when large sample masses are used. Lag leads to apparent shift of transition temperatures and can distort kinetic calculations. Mitigation strategies include using small sample masses, reducing heating rates, and employing thermocouples positioned close to the sample. Software compensation may also be applied, but accurate physical measurement remains the most reliable approach.

Thermal Conductivity
Related terms: heat transfer, sample geometry, furnace design
Thermal conductivity is the ability of a material to conduct heat. In calorimetric analysis, it influences how quickly a sample reaches the furnace temperature. Low-conductivity samples may exhibit temperature gradients, causing broadened peaks and inaccurate kinetic data. Selecting thin, uniformly distributed samples and using high-conductivity pans (e.g., platinum) improve thermal equilibration. For highly insulating polymers, a pre-heating step may be employed to reduce lag. Understanding conductivity helps in designing experiments that minimize artifacts.

Thermal Stability
Related terms: decomposition temperature, TGA, service life
Thermal stability refers to a material’s resistance to chemical change at elevated temperatures. It is assessed by TGA through the onset of mass loss and by DSC through exothermic degradation peaks. Materials with high thermal stability, such as polyimides, show decomposition temperatures above 500 °C, whereas low-stability polymers may degrade below 200 °C. Stability is a key factor in material selection for high-temperature applications. Factors affecting stability include molecular structure, presence of additives, and atmospheric conditions. Accurate stability assessment requires careful control of heating rate and atmosphere.

Thermogravimetric-Differential Scanning Calorimetry (TG-DSC) Coupling
Related terms: simultaneous analysis, heat flow, mass loss
TG-DSC coupling records mass change and heat flow simultaneously on the same sample. This dual data set provides a comprehensive view of thermal events, linking endothermic or exothermic processes to corresponding mass variations. For a polymer, the TG-DSC trace may show a mass loss at 350 °C accompanied by an exothermic peak, indicating oxidative degradation of the residue. Coupled analysis improves interpretation of complex reactions and aids in mechanistic studies. Instrument synchronization, proper calibration of both channels, and careful baseline management are essential for reliable results.

Thermal Event
Related terms: transition, reaction, DSC peak
A thermal event is any observable change in a material’s physical or chemical state as temperature varies. Examples include melting, glass transition, crystallization, decomposition, and oxidation. In DSC, events appear as peaks or steps in the heat flow curve; in TGA, they manifest as mass loss steps. Identifying and

characterizing thermal events enable material identification, quality control, and performance prediction. Overlapping events can complicate analysis, requiring deconvolution or complementary techniques such as EGA.

Unidad de Técnicas Calorimétricas y Termogravimétricas
Related terms: Thermal analysis laboratory, Mexican university, TGA-DSC facility
The Unidad de Técnicas Calorimétricas y Termogravimétricas (UCTT) is a specialized research unit within Mexican higher-education institutions that provides training and services in calorimetry and thermogravimetry. It offers state-of-the-art TGA, DSC, and coupled TG-DSC instruments, along with expertise in kinetic modeling, EGA, and material characterization. Students enrolled in the Thermogravimetric Analysis and Calorimetry course conduct hands-on experiments under the guidance of faculty, covering sample preparation, instrument calibration, data acquisition, and interpretation. The unit also supports industrial collaborations, assisting companies in polymer stability testing, additive evaluation, and failure analysis. Challenges faced by the UCTT include maintaining instrument precision in a high-usage environment, updating software to meet international standards, and integrating emerging techniques such as modulated DSC. Nonetheless, the unit remains a pivotal hub for advancing thermal analysis education and research across Mexico.

Validation Protocol
Related terms: method verification, standard operation, quality assurance
A validation protocol establishes that a thermal analysis method reliably produces accurate and repeatable results. It includes steps such as calibration with reference materials, repeatability tests using the same sample, reproducibility checks across different operators, and comparison with literature values. For a new DSC method, the protocol may require three independent runs of a certified polymer, each yielding ΔH within ± 5 % of the certified value. Documentation of all parameters—heating rate, atmosphere, pan type—is essential. Successful validation ensures compliance with quality standards and supports regulatory submissions.

Weight Percent Conversion (α)
Related terms: degree of conversion, fractional mass loss, kinetic plotting
Weight percent conversion, denoted α, represents the fraction of the original sample mass that has been transformed at a given temperature. It is calculated as $\alpha = (m_0 - m)/ (m_0 - m\_f)$, where m_0 is initial mass, m is mass at temperature T, and m_f is final mass after complete reaction. In kinetic analysis, α serves as the independent variable for isoconversional methods. For a polymer that loses 80 % of its mass, α reaches 0.8 at the end of the degradation. Accurate determination of α requires precise baseline correction and accounting for buoyancy effects.

Zero-Shift Correction
Related terms: temperature offset, calibration, baseline alignment
Zero-shift correction adjusts the temperature axis to compensate for systematic offsets between the programmed furnace temperature and the actual sample temperature. It is performed by measuring a known transition (e.g., indium melting) and applying the difference to all subsequent runs. For example, if indium melts at 157.5 °C instead of 156.6 °C, a –1.0 °C correction is applied. This correction improves the

accuracy of reported transition temperatures and kinetic parameters. Failure to apply zero-shift can lead to misinterpretation of thermal stability and erroneous activation energy calculations.