MAT 2021

METHODS OF THERMAL ANALYSIS

Students Slide Book | 2021

Teaching staff: Luís Belchior Santos; Carlos Lima

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STUDENTS WORKSHOP 2021

June 9th

List of Topics

https://videoconf-colibri.zoom.us/j/84329558229

Time	STUDENT NAME / TOPICS/THEMATICS
11h00m - 11h25m	Pedro Lopes Metal Ligand Bonding energetics by Solution-Reaction Calorimetry
11h30m - 11h55m	Rui Mendes Molecular Docking study by ITC / VP-DSC
12h00m - 12h25m	Rafael Souza Heat Capacity Measurements by DSC
12h30m - 12h55m	Carlla Vasconcelos Thermal Stability of Ionic Liquids by TGA

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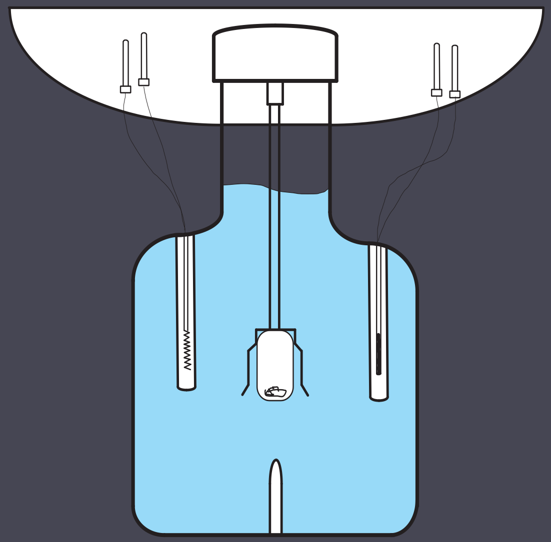

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METHODS OF THERMAL ANALYSIS

METAL LIGAND BONDING ENERGETICS BY REACTION-SOLUTION CALORIMETRY

Pedro Lopes
Bachelor's in Biotechnology, Univ. Aveiro (2020)

Introduction

- **What is calorimetry?**

Heat can't be measured directly. Instead, we measure **Heat Flux** → **Calorimeter**

- Materials characterization
- Energy balance
- Follow chemical reactions
- …

Calorimetry

Calor (Heat) + *Metron* (Measure)

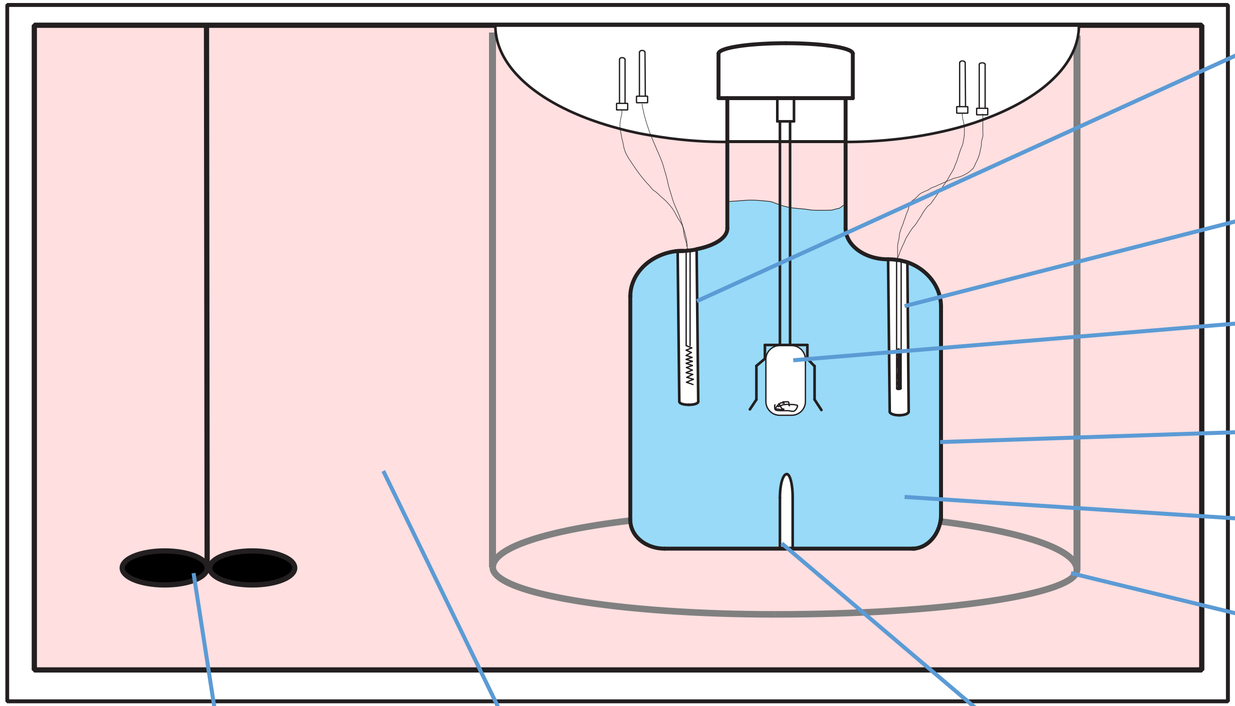
- **What is reaction-solution calorimetry?**

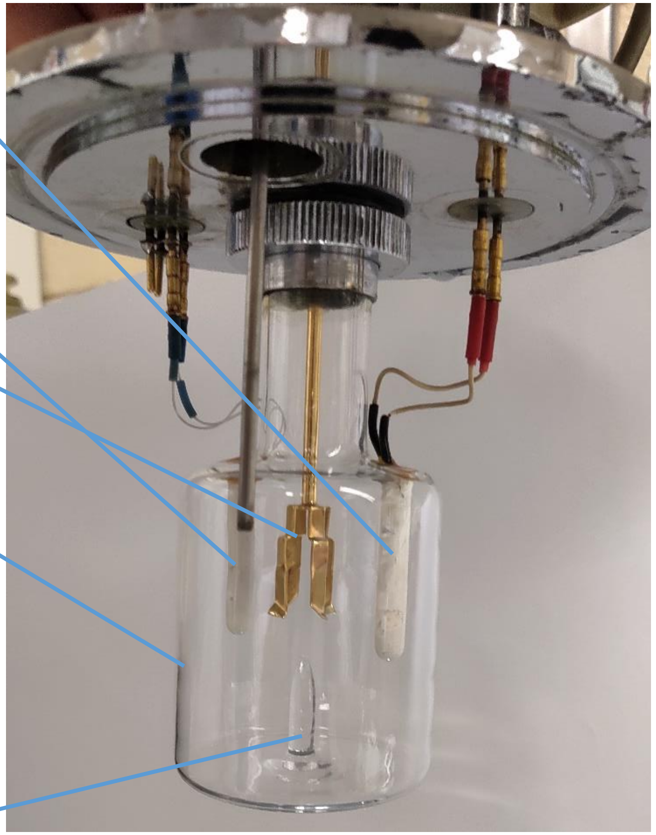
→ Measure the heat flux of the dissolution of a substance in a solvent

→ Measure the heat flux during a reaction

Reaction-Solution Calorimetry

How does it work?





Reaction-Solution Calorimetry

How does it work?

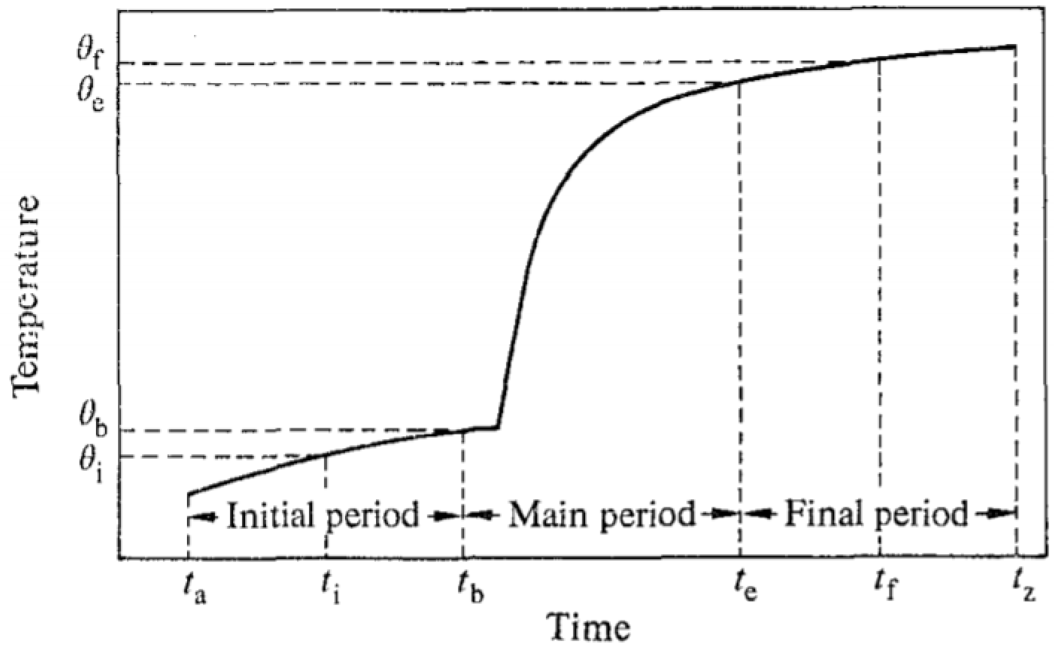
1. Weight the glass ampoule;
2. Weight the sample to be analysed (solid or liquid);
3. Insert the ampoule on the support;
4. Fill rigorously the glass vessel with the appropriated solvent;
5. Insert the ampoule support into the glass vessel;
6. Introduce this assembly into the thermostatic bath;
7. Let the temperature stabilize until thermal equilibrium (about 3000 sec);
8. Break the glass ampoule (into the sapphire breaker).

Calorimeter Functional modes

- Isoperibolic
- Adiabatic
- Pseudo-Adiabatic

Reaction-Solution Calorimetry

How does it work?



Calibration

Electrical	**Chemical**
• Provides heat trough electrical current • Before and/or after the reaction	• Reactants with a very well known energy • Usually, before the reaction

$$d\theta/dt = u + k(\theta_j - \theta)$$
$$u = p/\varepsilon$$
$$\theta_1 = \theta_\infty - (\theta_\infty - \theta_b)\exp\{-k(t - t_b)\}$$

k – thermal leakage modulus
θ_j – jacket temperature
θ_∞ – convergence temperature
p – sum of all constant thermal powers in the calorimeter
ε – energetic equivalent of the calorimeter

S. R. Gunn, *J. Chem. Thermodyn.*, vol. 3, no. 1, pp. 19–34, 1971

Reaction-Solution Calorimetry

What can we measure?

- Heat measurements of processes: Q
- Enthalpies of processes: $\Delta H = Q_p$
- Heat capacities: $C_p = \frac{\Delta H}{\Delta T}$
- Metal Ligand Bonding energetics:

Hypothetical reactions

$$M\,(g) \;+\; L\,(g) \;\rightarrow\; ML\,(g) \qquad \Delta_r H^g = ?$$

⚠ But the compounds are solid or liquid:

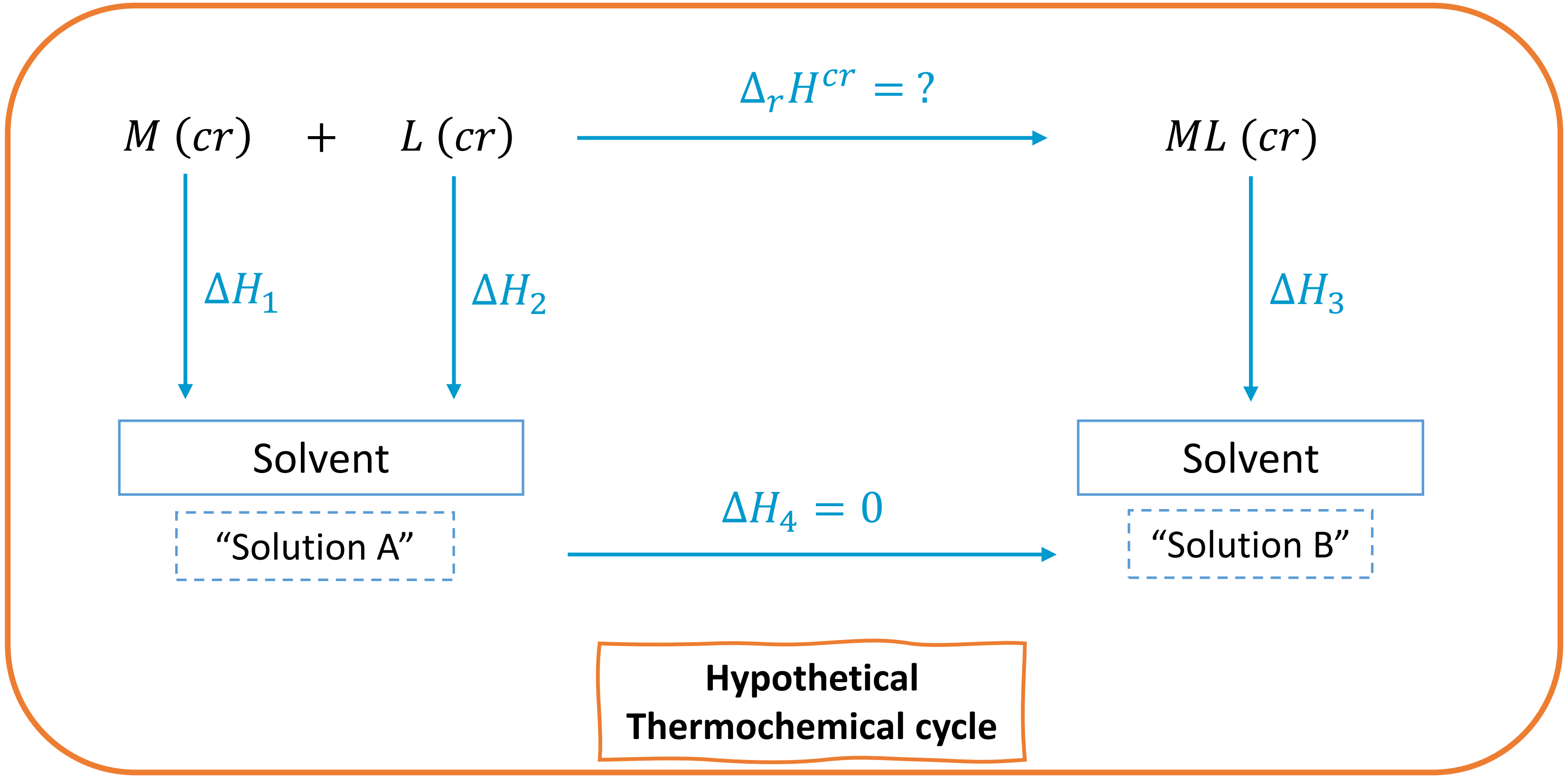
$$M\,(cr) \;+\; L\,(cr) \;\rightarrow\; ML\,(cr) \qquad \Delta_r H^{cr} = ?$$

$\Delta_{subl} H$

→ **Reaction-Solution Calorimetry**

Reaction-Solution Calorimetry

What can we measure?



$$\Delta_r H^{cr} = \sum \Delta H\ (products) - \sum \Delta H\ (reactants)$$

$$\Delta_r H^{cr} = \Delta H_3 - (\Delta H_1 + \Delta H_2)$$

Case study

Energetics of metal–ligand binding in copper(II) and nickel(II) complexes of two Schiff bases

J. Chem. Soc., Dalton Trans., 1997, Pages 1257–1262

Manuel A. V. Ribeiro da Silva,* Maria D. M. C. Ribeiro da Silva, Manuel J. S. Monte, Jorge M. Gonçalves and Élia M. R. Fernandes

Centro de Investigação em Química, Departamento de Química, Faculdade de Ciências, Universidade do Porto, Rua do Campo Alegre, 687, P-4150 Porto, Portugal

		Metal	
		Cu	Ni
Ligand	H_2acacen	Cu(acacen)	Ni(acacen)
	H_2bzacen	Cu(bzacen)	Ni(bzacen)

H_2acacen = 4,4’-ethylenedinitrilobis(pentan-2-one)
H_2bzacen = 1,1’-diphenyl-3,3’-ethylenedinitrilobis(butan-1-one)

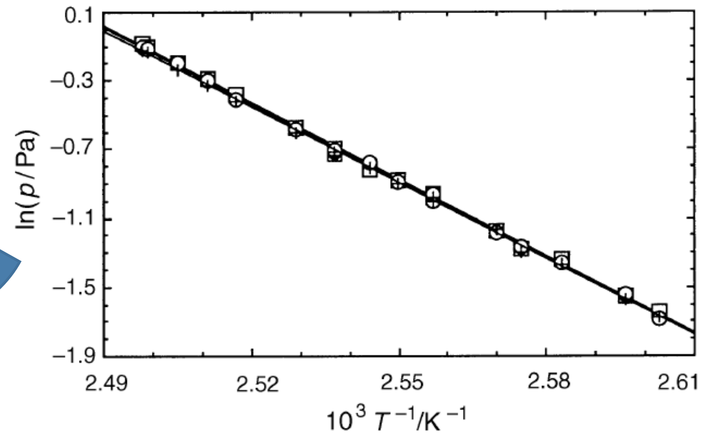
$$\mathrm{Cu(O_2CMe)_2\cdot H_2O\ (cr)} + \mathrm{H_2L\ (cr)} \xrightarrow{\Delta_f H^{cr} = ?} \mathrm{[CuL]\ (cr)} + \mathrm{H_2O\ (l)} + \mathrm{2MeCO_2H\ (l)}$$

$$\mathrm{NiCl_2\cdot 6H_2O\ (cr)} + \mathrm{H_2L\ (cr)} + \mathrm{H_2O\ (l)} \xrightarrow{\Delta_f H^{cr} = ?} \mathrm{[NiL]\ (cr)} + \mathrm{2HCl\cdot H_2O\ (l)}$$

Case study

Energetics of metal–ligand binding in copper(II) and nickel(II) complexes of two Schiff bases

Complex	$\Delta_f H_m^\circ$ (cr)	$-\Delta_f H_m^\circ$ (cr)	$\Delta_{cr}^{g} H_m^\circ$	$-\Delta_f H_m^\circ$ (g)
[Cu(acacen)]	40.866 ± 0.039	385.1 ± 3.2	127.6 ± 0.7	257.5 ± 3.3
[Cu(bzacen)]	34.616 ± 0.052	203.4 ± 5.1	205.6 ± 3.0	-2.2 ± 5.9
[Ni(acacen)]	82.13 ± 0.10	472.6 ± 3.1	129.1 ± 0.9	343.5 ± 3.2
[Ni(bzacen)]	74.59 ± 0.10	292.2 ± 5.1	201.7 ± 2.8	90.5 ± 5.8



Curiosity

1st Solution Calorimeter

Heats of Solution of Group IB Metals in Liquid Tin

by L. B. Ticknor and M. B. Bever

SEPTEMBER 1952, JOURNAL OF METALS—941

An isothermal calorimeter suitable for measurements of heats of solution in liquid tin as solvent is described. Measurements of the heats of solution of gold, silver, copper, and a gold-silver alloy are reported. The heat of formation of this gold-silver alloy is also reported.

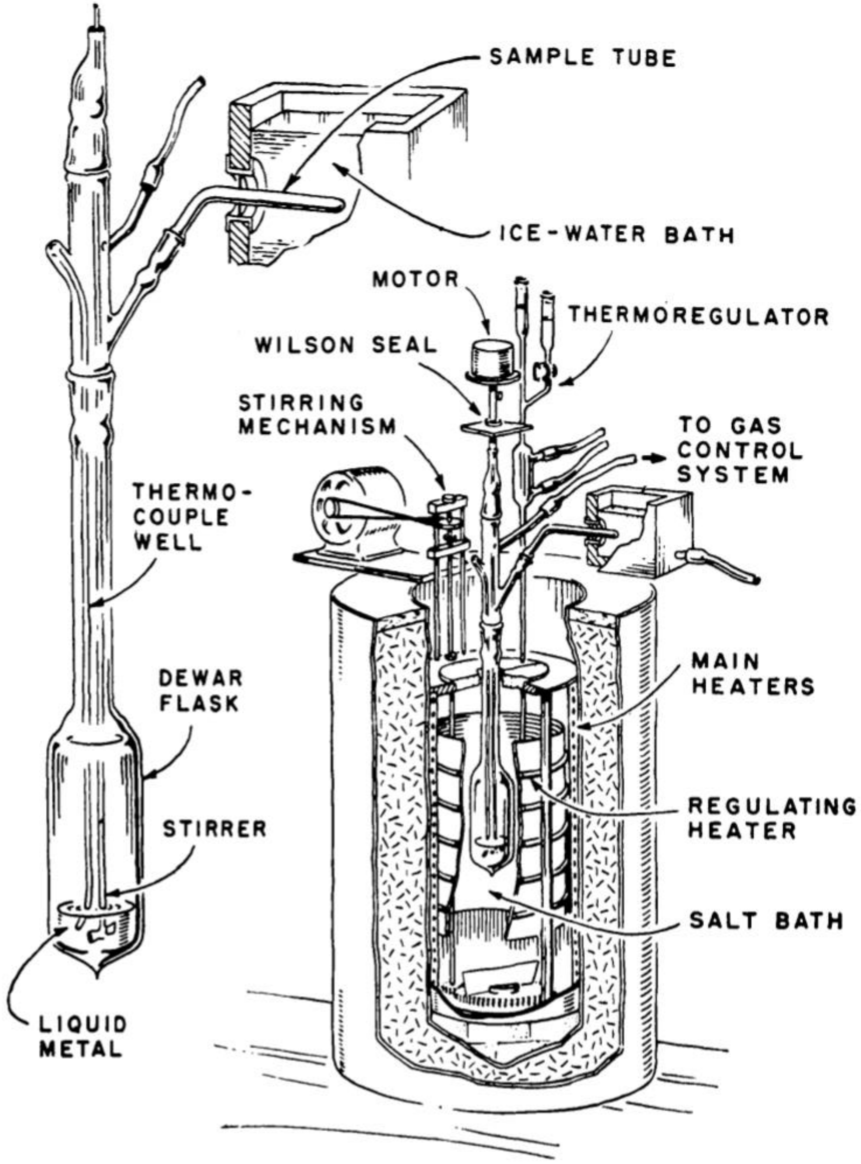


Fig. 1—View of calorimetric equipment.

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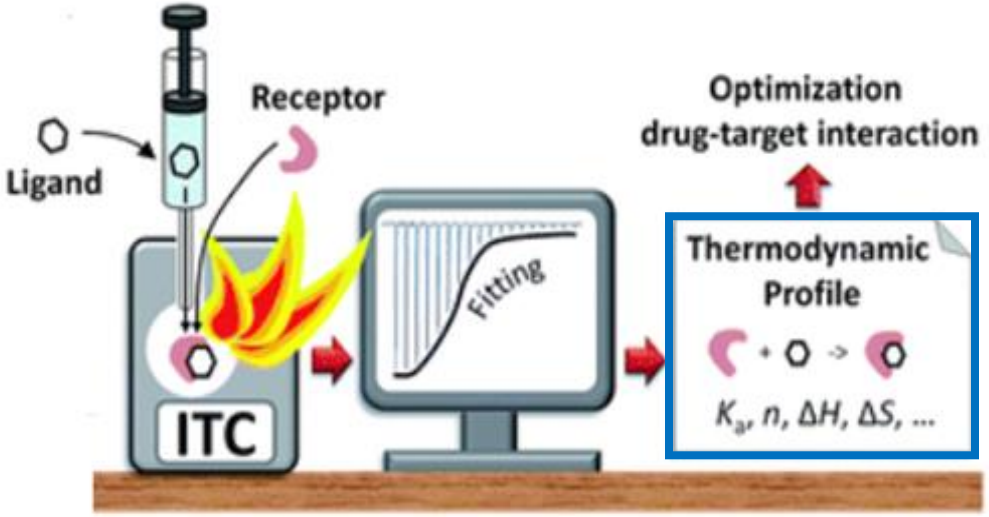
Molecular Docking Study by ITC/VP-DSC

Rui Fernando Alves Mendes
Bachelor´s Degree in Biochemistry, FCUP, 2020

1. Introduction

Isothermal Titration Calorimetry (ITC) is a technique used to measure the heat exchange associated with molecular interactions at constant temperature.

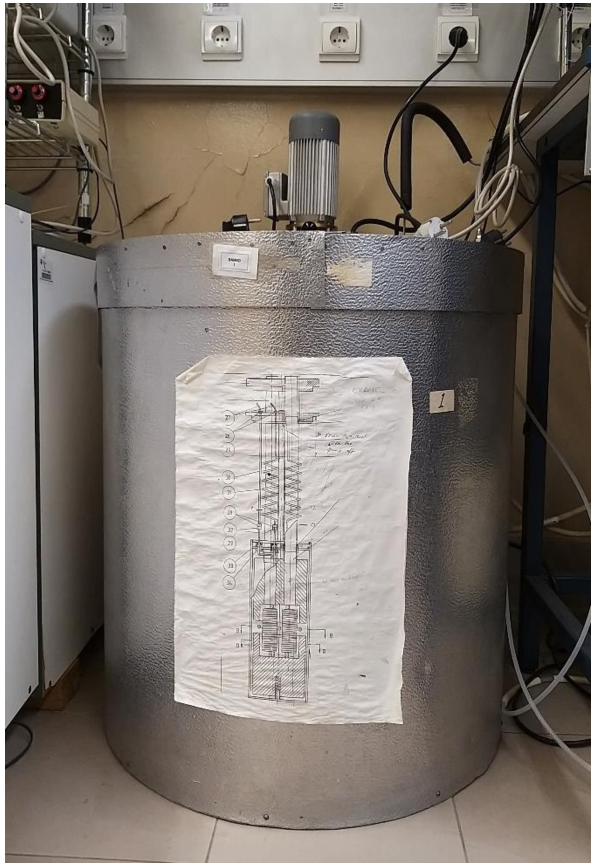


- Binding constant;
- Reaction stoichiometry;
- Enthalpy;
- Entropy;

Complex formation

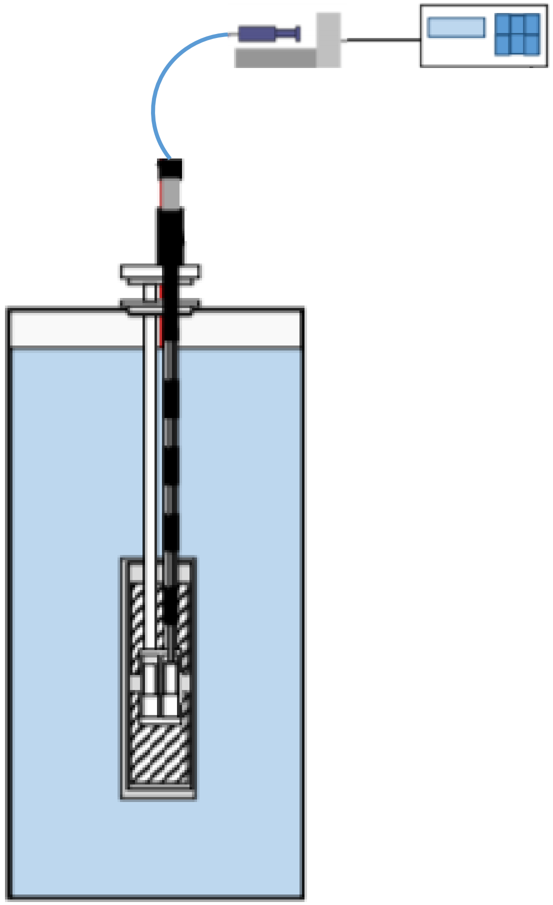
- No need of chemical modification, labelling or immobilization;
- No limitations associated with the sample’s cloudiness or the molecular weight of the structures involved.

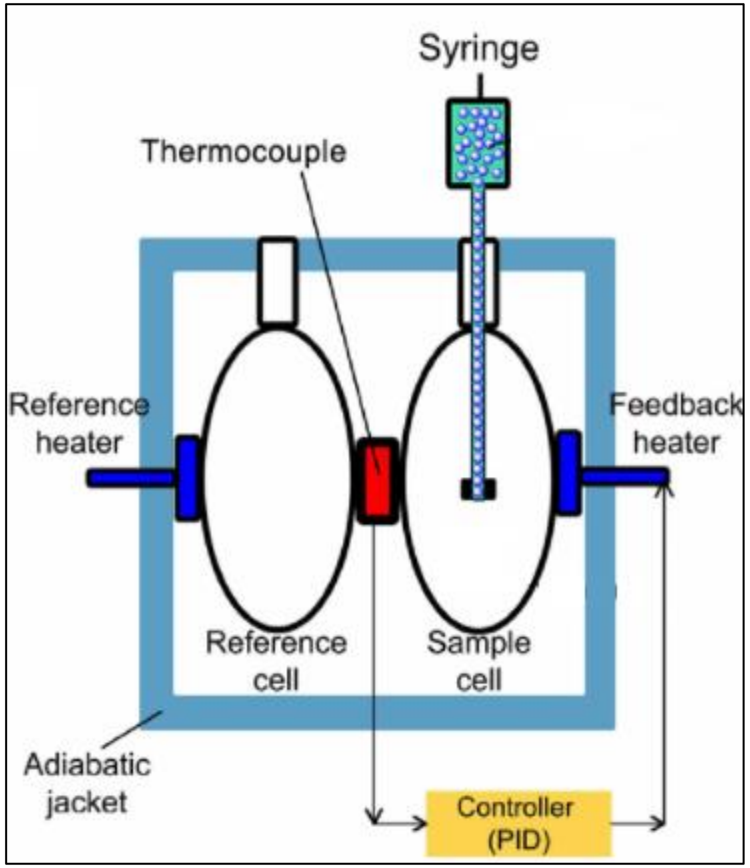
2. How does it work?



ITC by Heat Conduction

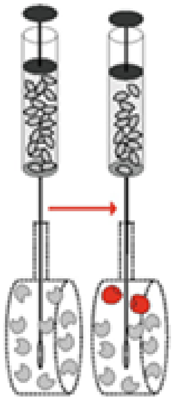
X-Ray

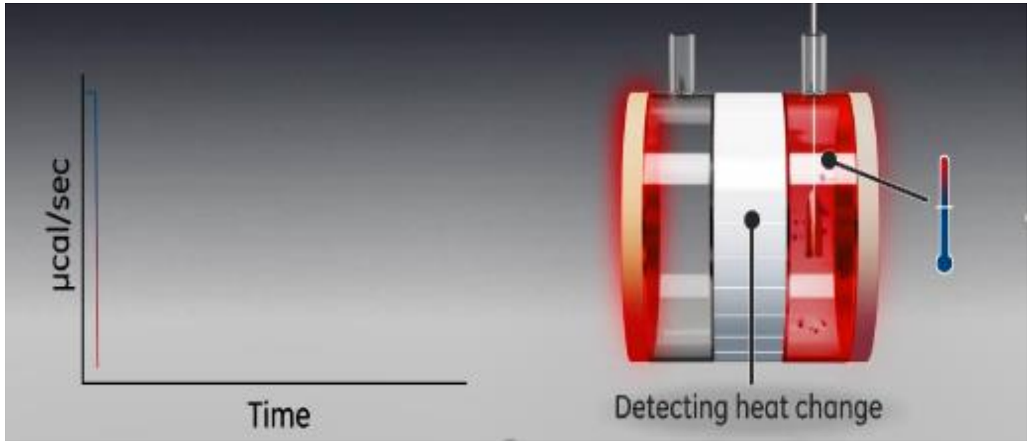


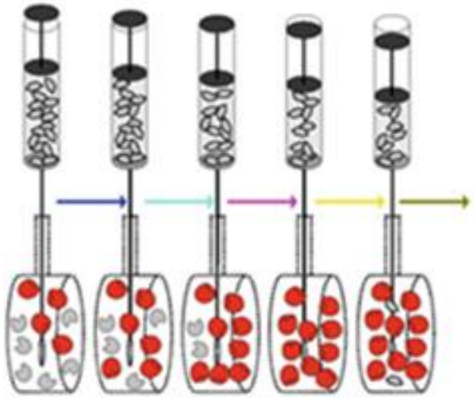


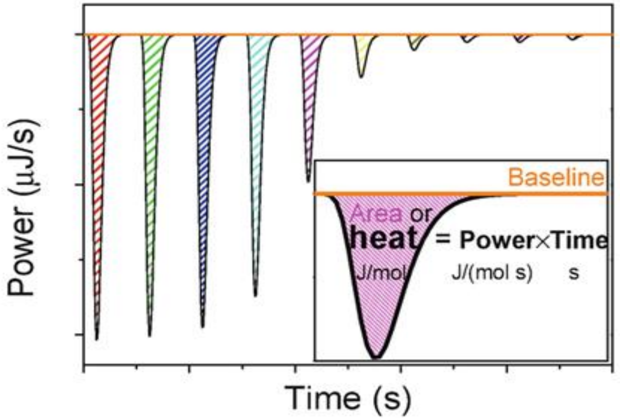
ITC by Power Compensation

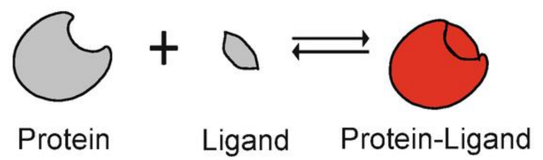
2. How does it work?



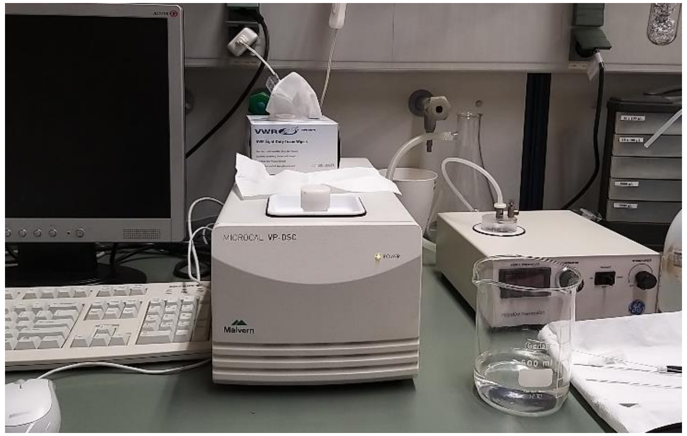








VP-DSC



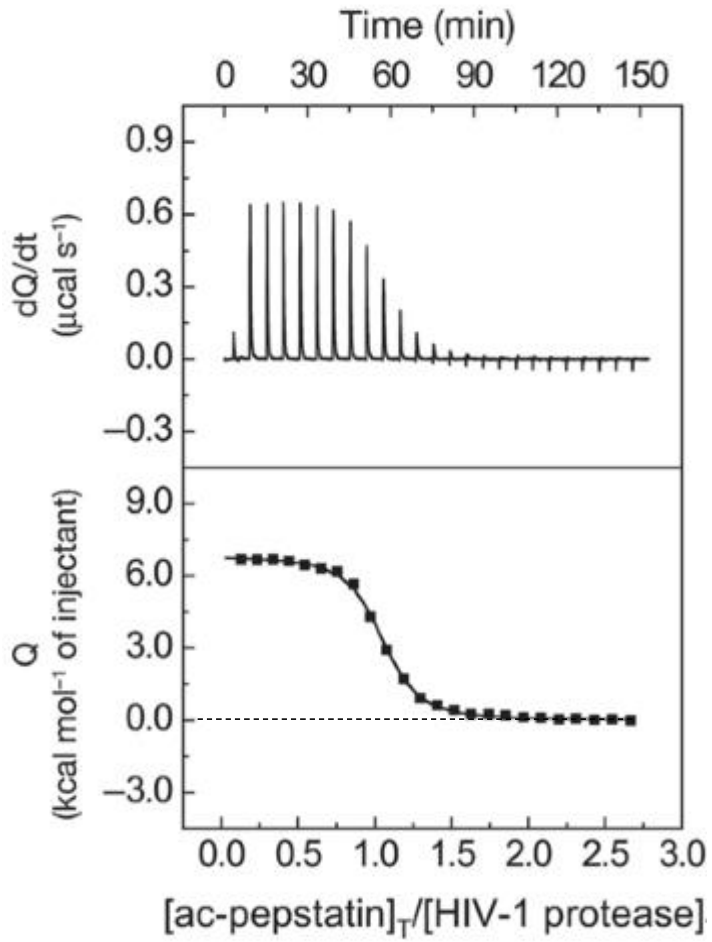
Study of samples in solution

Intramolecular stability of structured macromolecules

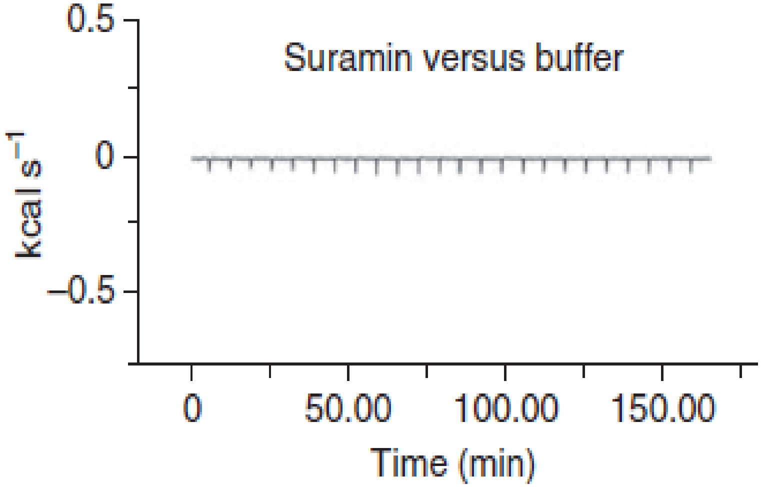
Intermolecular stability of complexes

3. What can we measure?

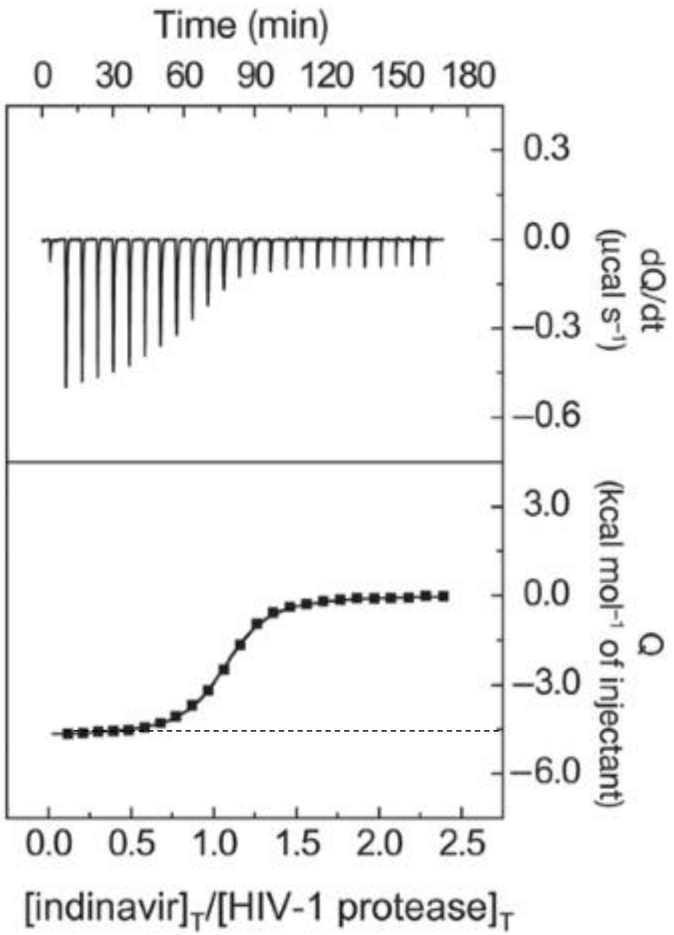
Endothermic Process



No Interaction



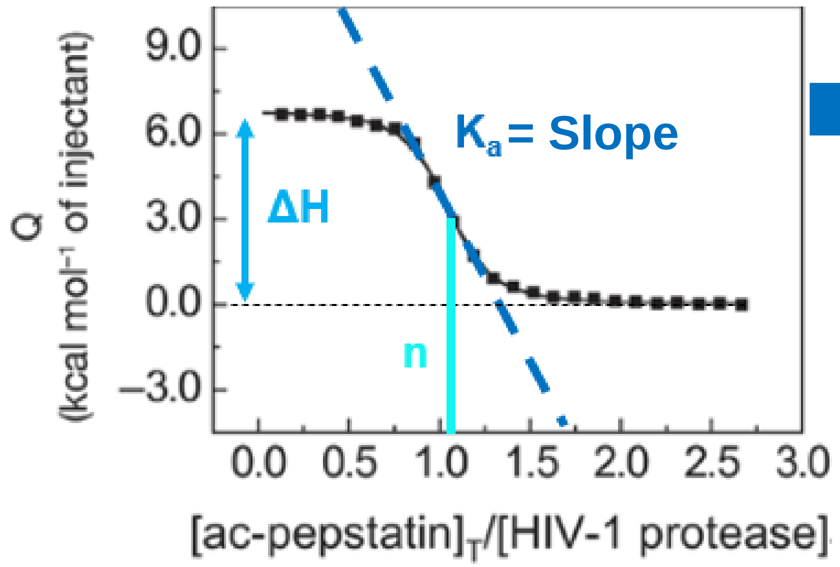
Exothermic Process



Curve optimization

c value

3. What can we measure?



- Binding constant;
- Reaction stoichiometry;
- Enthalpy;

$\rightarrow \Delta G = - RT\ln(K_a)$

$\rightarrow \Delta G = \Delta H - T\Delta S$

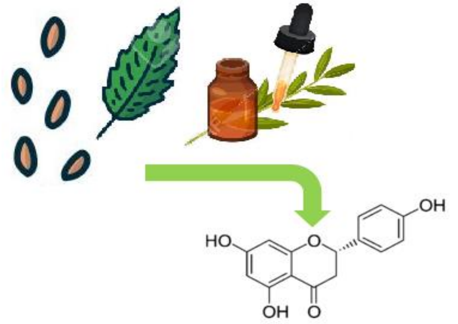
- Gibbs free energy;
- Entropy;

By performing titrations at a range of temperatures, the ΔC_p for an interaction can be determined.

$$\Delta C_p = \frac{\partial \Delta H}{\partial T}$$

- Strength of interaction;
- Stability of biological complex;
- Number of binding sites;
- Reaction's spontaneity;

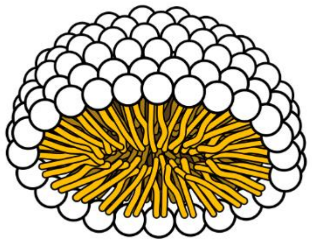
4. Application Note



Natural Product Reports
CrossMark
Cite this: *Nat. Prod. Rep.*, 2016, **33**, 881

Application of isothermal titration calorimetry as a tool to study natural product interactions

O. Callies and A. Hernández Daranas*



Use of isothermal titration calorimetry to study surfactant aggregation in colloidal systems☆

CrossMark

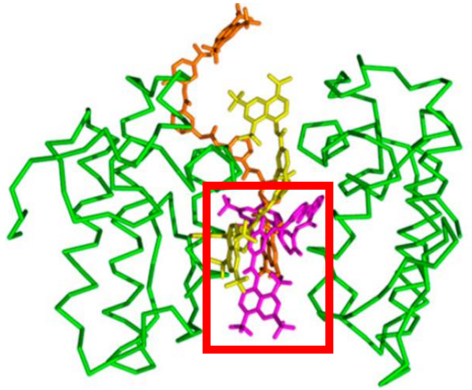
Watson Loh [a,*], César Brinatti [a], Kam Chiu Tam [b]

[a] *Institute of Chemistry, University of Campinas (UNICAMP), CP 6154, CEP 13083-970, Campinas, SP, Brazil*
[b] *Department of Chemical Engineering and Waterloo Institute for Nanotechnology, University of Waterloo, 200 University Avenue West, Waterloo, ON N2L 3G1, Canada*

Application on Drug Discovery

4. Application Note

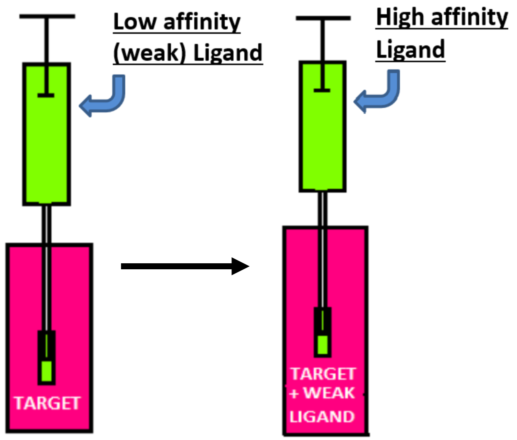
Identification of Target Proteins



Suramin

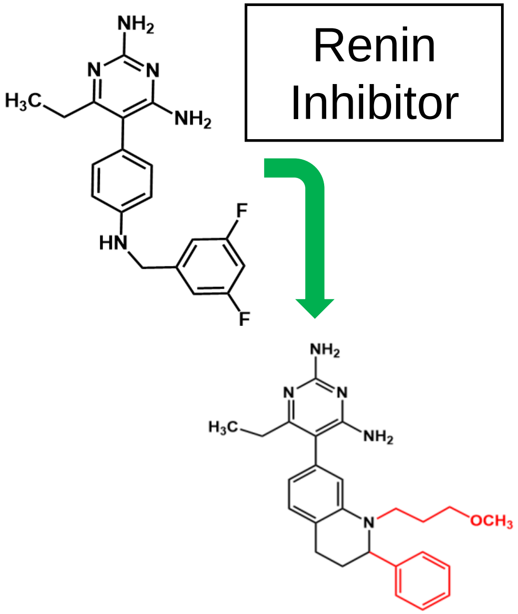
Zhou, X.; *et al*. *Nat. Protoc*. **2011**, 6, 158-165.

Drug-Macromolecule Binding Studies



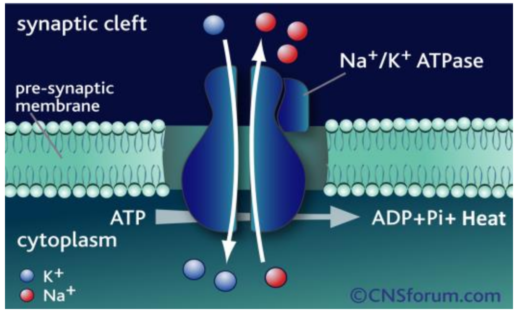
Velazquez-Campoy, A.; *et al. Nat. Protoc*. **2006**, 1, 186-191.

Lead Optimization



Sarver, R. W.; *et al. Anal. Biochem*. **2007**, 360, 30-40.

Enzyme Kinetics

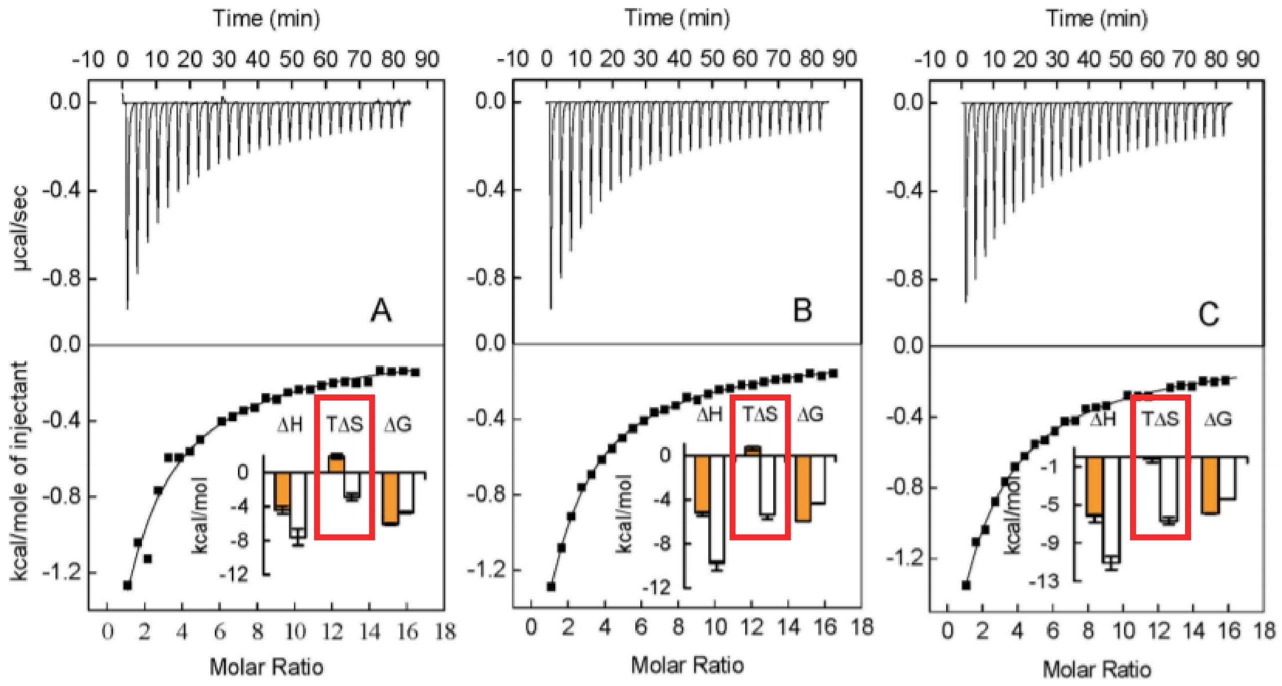


Noske, R.; *et al. Bioph. Acta*. **2010**, 1797, 1540-1545.

5. Case Study

A Comprehensive Insight into Binding of Hippuric Acid to Human Serum Albumin: A Study to Uncover Its Impaired Elimination through Hemodialysis

Nida Zaidi, Mohammad Rehan Ajmal, Gulam Rabbani, Ejaz Ahmad, and Rizwan Hasan Khan *

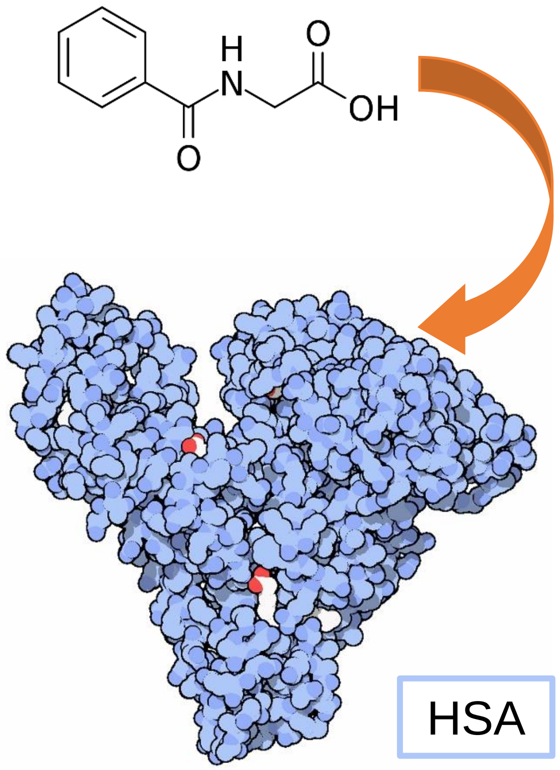


High affinity site
Low affinity site

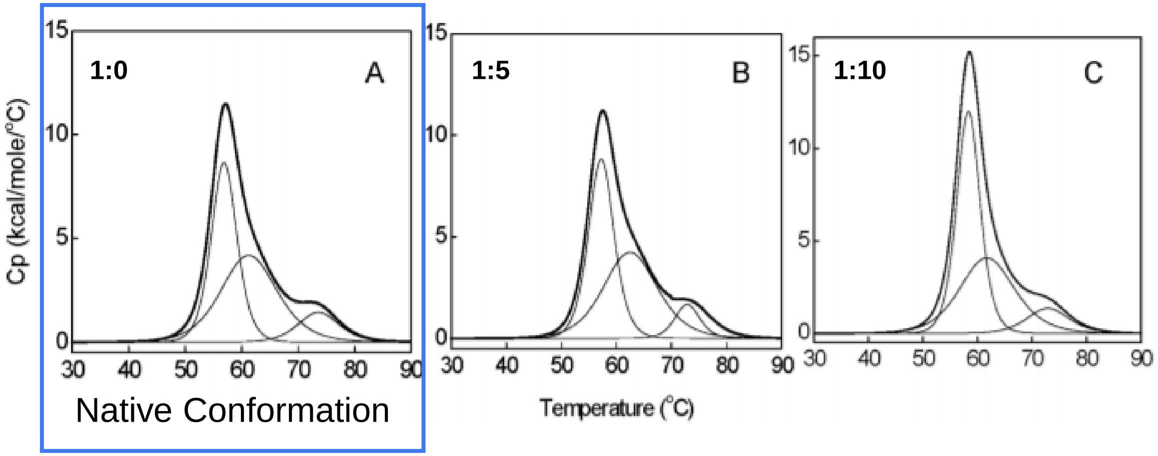
ITC

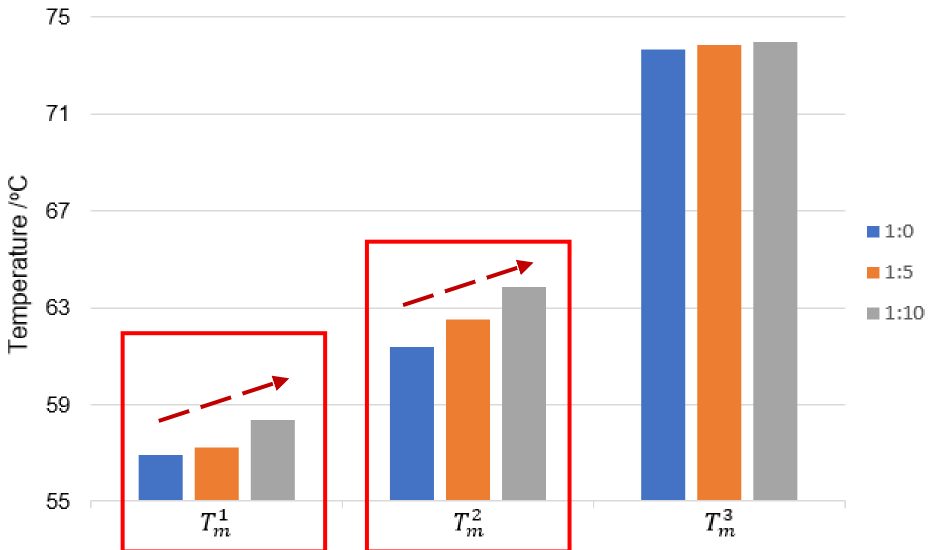
Binding Displacement Measurement

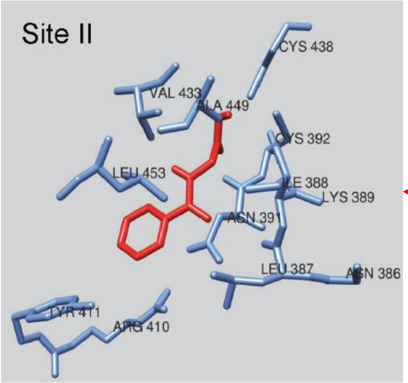
2 structuraly different binding sites



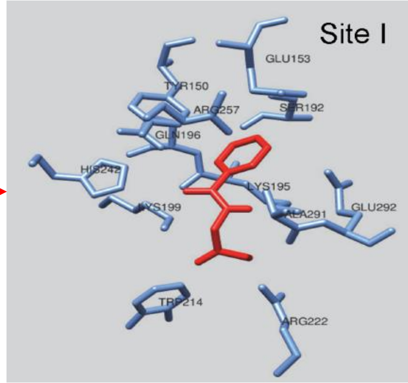
5. Case Study







Domain III



Domain II

Study Conclusions:

- ✓ HA markedly binds to both binding sites of HSA;
- ✓ Binding is a spontaneous, enthalpically driven, entropically opposed process;
- ✓ HA elimination through hemodialysis may hinder.

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METHODS OF THERMAL ANALYSIS

Heat Capacity Measurements by DSC (Differential Scanning Calorimetry)

RAFAEL GOMES DE SOUZA
Bachelor's Degree in Chemical Engineering, Instituto Mauá de Tecnologia, São Caetano do Sul (2017)

DQB... 2020-2021

Introduction

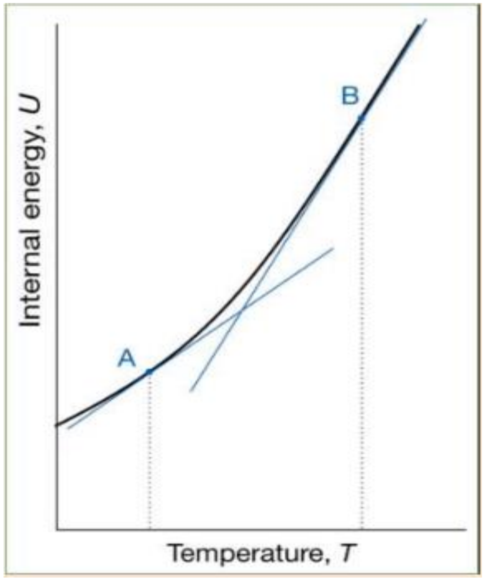
Heat Capacity (Cp)

Intensive Property

Definition: Energy necessary to raise the temperature of a system by 1 K.

- U = Q + W
- H ≡ U + PV

$$\frac{dQ}{dt}$$

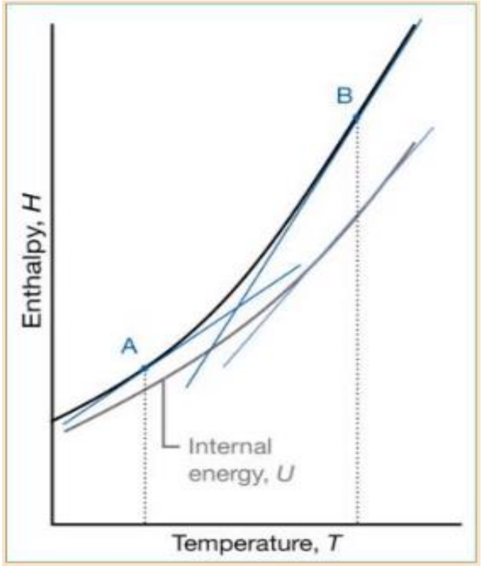


At constant volume and for infinitesimal process:

$dq_V = dU$

Hence:

$$C_v = \left(\frac{\partial U}{\partial T}\right)_V$$



At constant pressure and for infinitesimal process:

$dq_P = dH$

Hence:

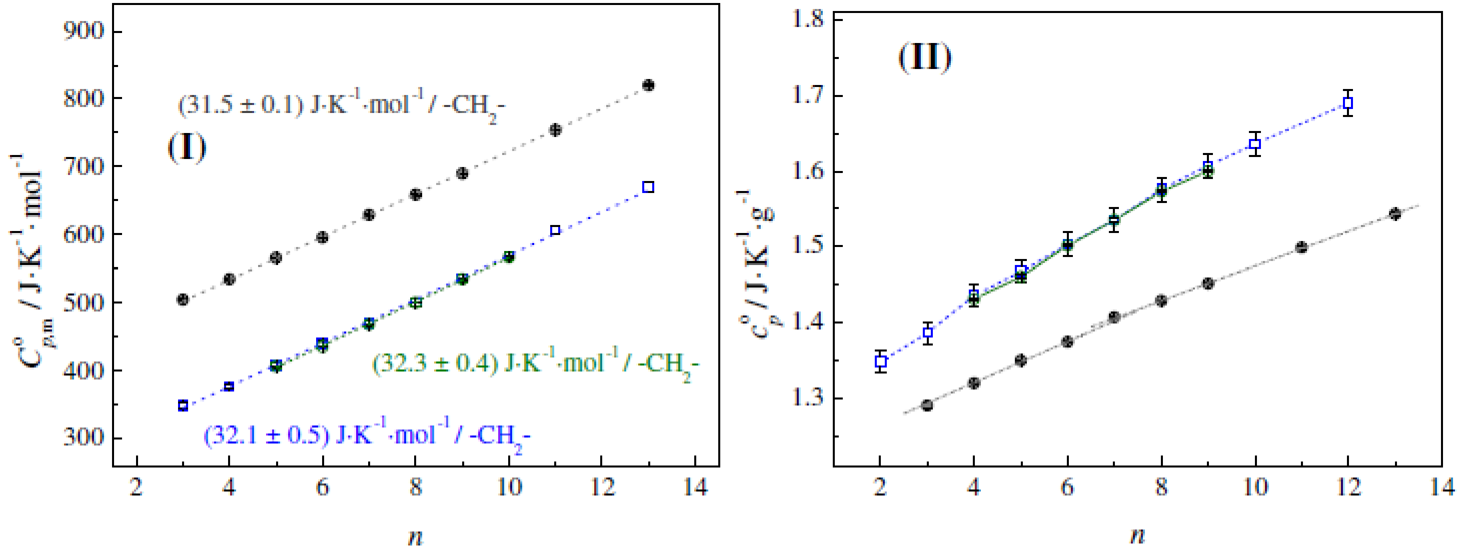
$$C_p = \left(\frac{\partial H}{\partial T}\right)_P$$

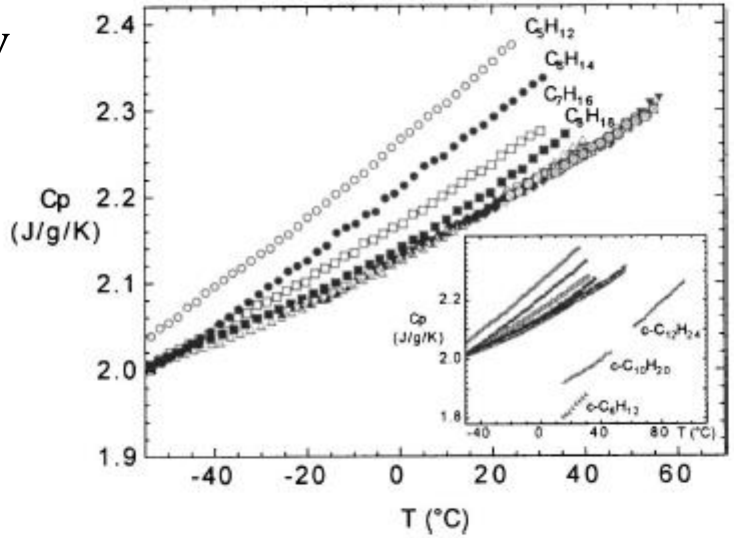
Levine, Ira N.; Physical Chemistry; McGRAW-HILL INTERNATIONAL EDTIONS; New York ; 1995

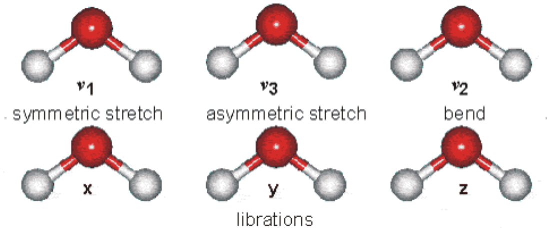
Introduction

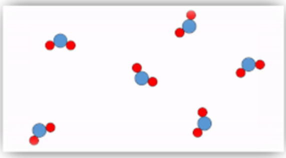
Heat capacity can be converted into extensive thermodynamic property (Molar, volumic and Specific Heat Capacity):

These properties change with the increase in temperature: c_v { Translational Energy, Rotational Energy, Vibrational Energy, Eletronic Energy, Intermolecular }







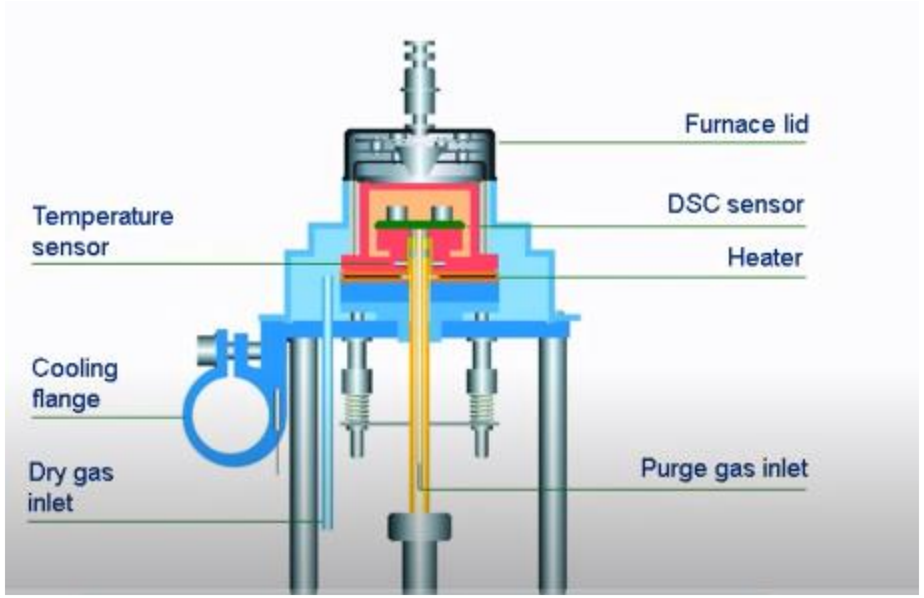


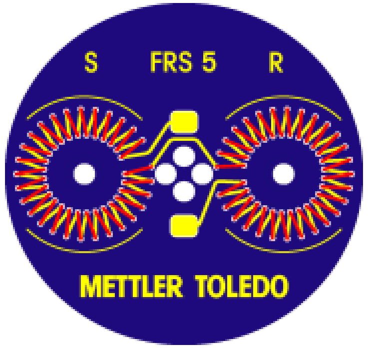
Molar Heat Capacity (J/K·mol) and Specific Heat Capacity (J/K· g) as a function of the number of carbons in the alkyl chain of ionic liquids.

HUANG D., SIMON S. L. , MCKENNA G. B.; Chain length dependence of the thermodynamic properties of linear and cyclic alkanes and polymers; The Journal of Chemical Engineering
SERRA P. B. P, RIBEIRO F. M. S., ROCHA M. A. A., FULEM M., Růžička K., COUTINHO J. A. P., SANTOS L. M. N. B. F; Solid-liquid equilibrium and heat capacity trend in the alkylimidazolium PF6 series; The Journal of Chemical Physics

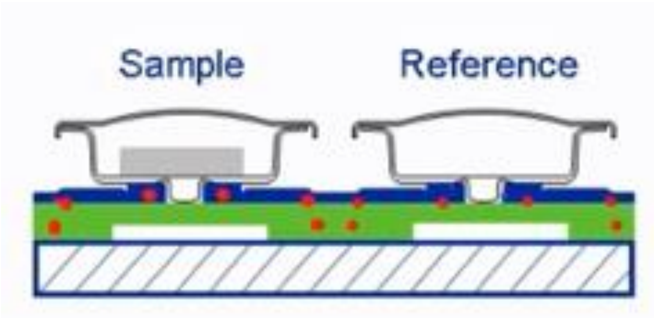
How does it work

How does Differential Scanning Calorimetry Works?





- -Samples and Reference are heated;
- -Both sorrounded by a furnace;
- -Reference crucible empty;
- -Star shaped sensors → heat flow
- -Crucible is placed on the top of the sensor

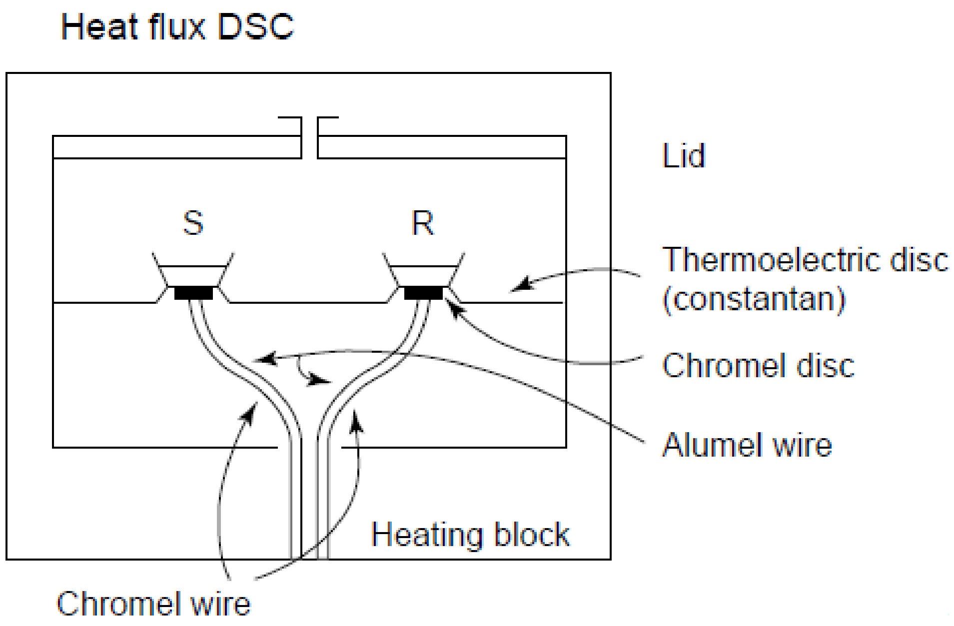


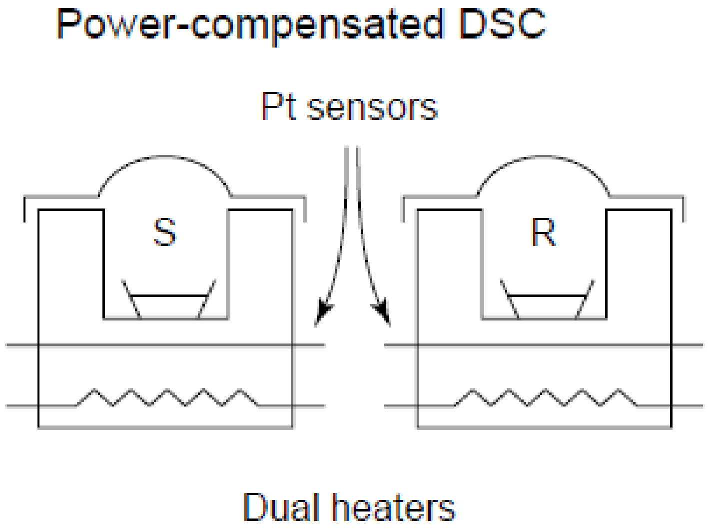
https://www.youtube.com/watch?v=MRJXMEIpmpU

HÖHNE G. W., HEMMINGER W. F., FLAMMERSHEIM H. J.; Differential Scanning Calorimetry; 2nd. Ed. Berlin, Heidelberg, New York: Springer-verlag, 2003

How does it work

Types of Differential Scanning Calorimeters



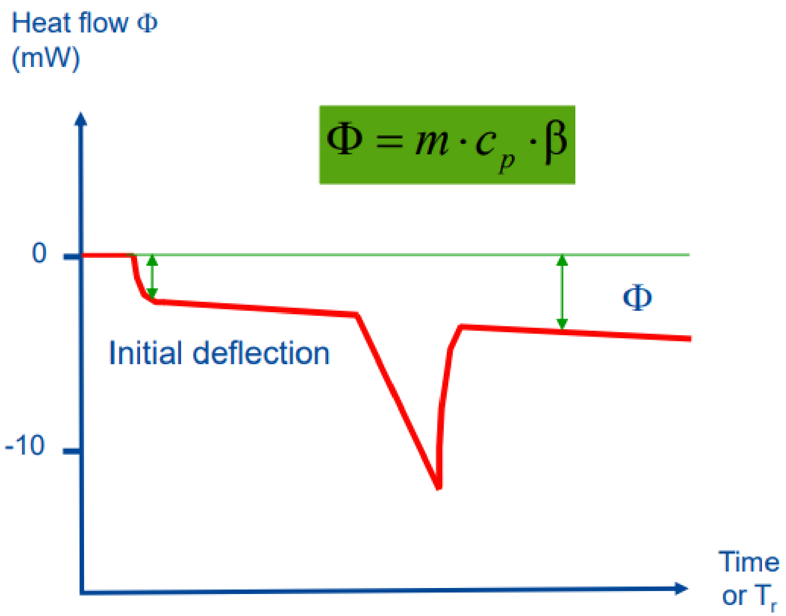


- Individual furnace;
- No direct contact with thermocouple;
- Temperature difference is monitored.

- 2 independent furnaces and sensors;
- Small furnaces (quick cooling/heating);
- Heat compensation is monitored (ΔT ≈0).

SKOOG, D. A., LEARY, J. J. Thermal Methods. In: PRINCIPLES OF Instrumental Analysis. Fortworth, Saunders College Publishing, 1992. Cap. 23, p. 569-577
BROWN, M. E. Introduction to Thermal Analysis. London, Chapman and Hall, 1988, p. 1-6, 23-49
LUND, D. B. Appücations of Differential Scanning Calorimetry infoods. In: PELEG, M & BAGLEY, E. B, ed. Physical Properties ofFoods. Westport, A VI, 1983. Cap. 4, p. 125-143.
MA, C. -Y., HARWALKAR, V. R., MAURICE, T. J. Instrumentation and techniques of thermal analysis infood reserch. In: HARWALKAR, V. R & MA, C. -Y., ed. Thermal analysis offoods. London, Elsevier, 1990. Cap. 1, p. 1-15.

What can we measure



$$\Phi = m \cdot c_p \cdot \beta$$

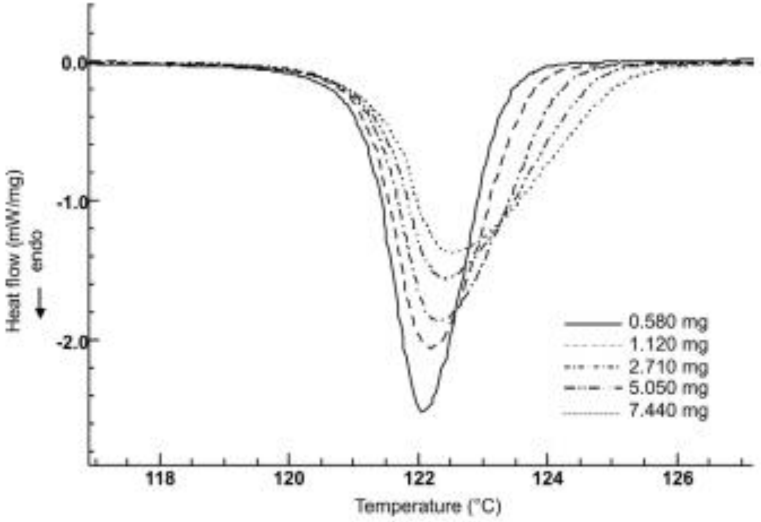
Where:

m is the sample mass

c_p is the heat capacity of the sample

β is the heating rate.

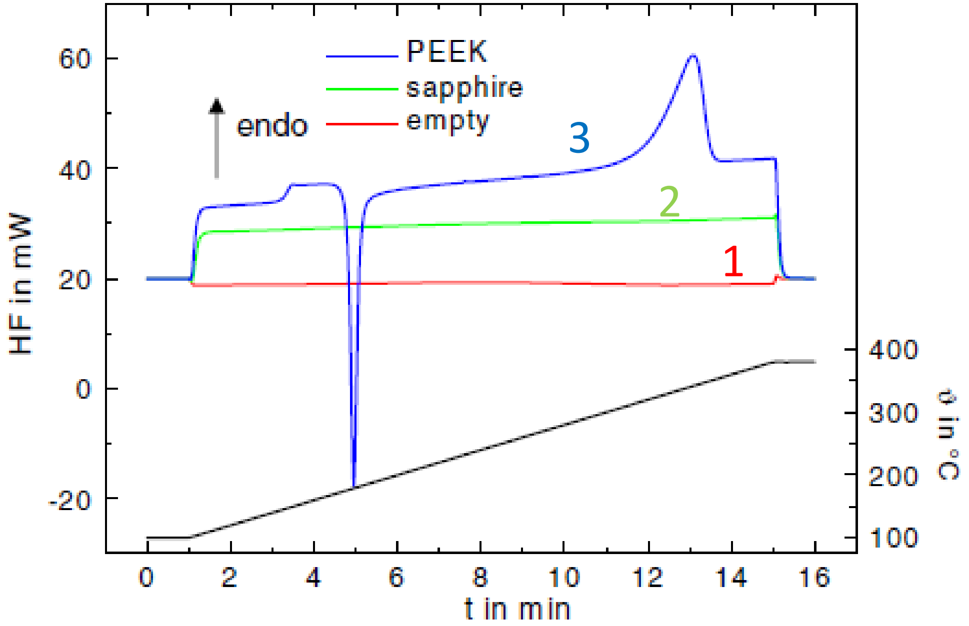
↑ m ↑ φ ↓ signal



DSC curves of AZT (zidovudine) using different masses recorded in a dynamic nitrogen atmosphere.

Mettler Toledo; Introducción a la técnica DSC
ARAÚJO A. A. S., BEZERRA M. S., STORPIRTIS S., MATOS J. R.; Determination of the melting temperature, heat of fusion, and purity analysis of different samples of zidovudine (AZT) using DSC. Brazilian Journal of Pharmaceutical Sciences

What can we measure



1 – Scanning through empty crucibles (base line)

2 – Scanning for calibration

3 – Scanning for sample

4 – Measure ranges and calculate cp:

$$c_p(T) = c_{p\ sapphire}(T)\,\frac{m_{sapphire}\cdot\beta}{m_{sample}\cdot\beta}\cdot\frac{\phi_{sample}(T)-\phi_{empty}(T)}{\phi_{sapphire}(T)-\phi_{empty}(T)}$$

With: $$K(T) = c_{p\ sapphire}(T)\,\frac{m_{sapphire}\cdot\beta}{\phi_{sapphire}(T)-\phi_{empty}(T)}$$

Hence: $$c_p(T) = K(T)*\frac{\phi_{sapphire}(T)-\phi_{empty}(T)}{m_{sample}\cdot\beta}$$

Where:

c_p – heat capacity for the sample
$c_{sapphire}$ – heat capacity for sapphire;
m_{sample} – sample mass;
$m_{sapphire}$ – sapphire mass;
Φ_{sample} – heat flux for the sample;
$\Phi_{sapphire}$ – heat flux for sapphire;
Φ_{empty} – heat flux for empty crucible.

SCHICK, Christoph; Glass transition under confinement-what can be learned from calorimetry; The European Physical Journal Special Topics

Application note

Journal of chemical & engineering data

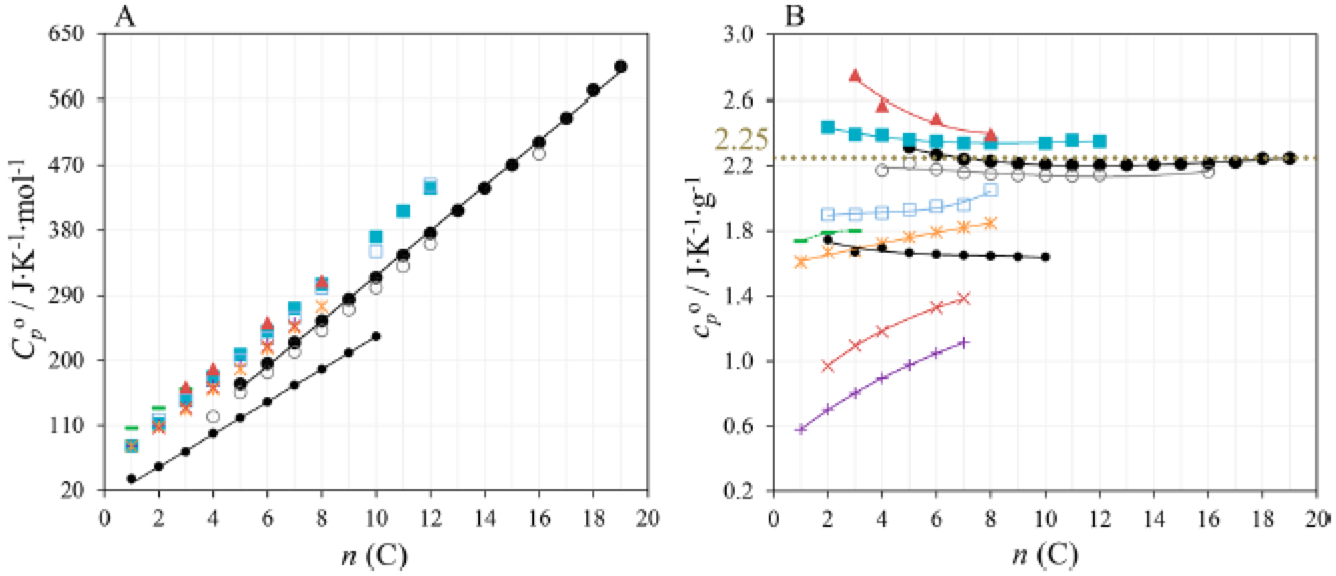
Review

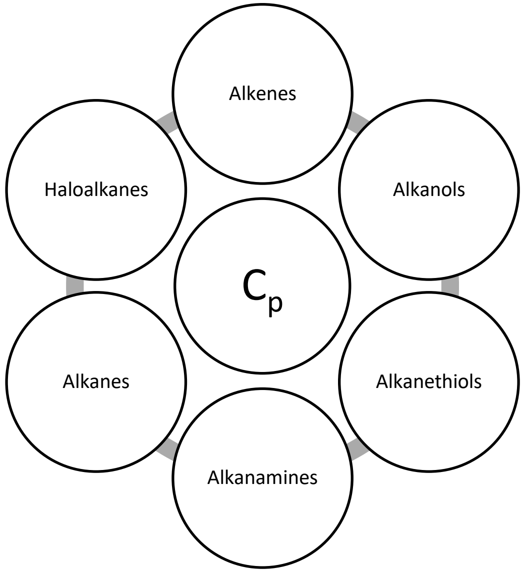
Cite This: *J. Chem. Eng. Data* 2018, 63, 1–20

pubs.acs.org/jced

Chain Length Dependence of the Thermodynamic Properties of *n*-Alkanes and their Monosubstituted Derivatives

José C. S. Costa,*[,†,‡] Adélio Mendes,[‡] and Luís M. N. B. F. Santos[†]

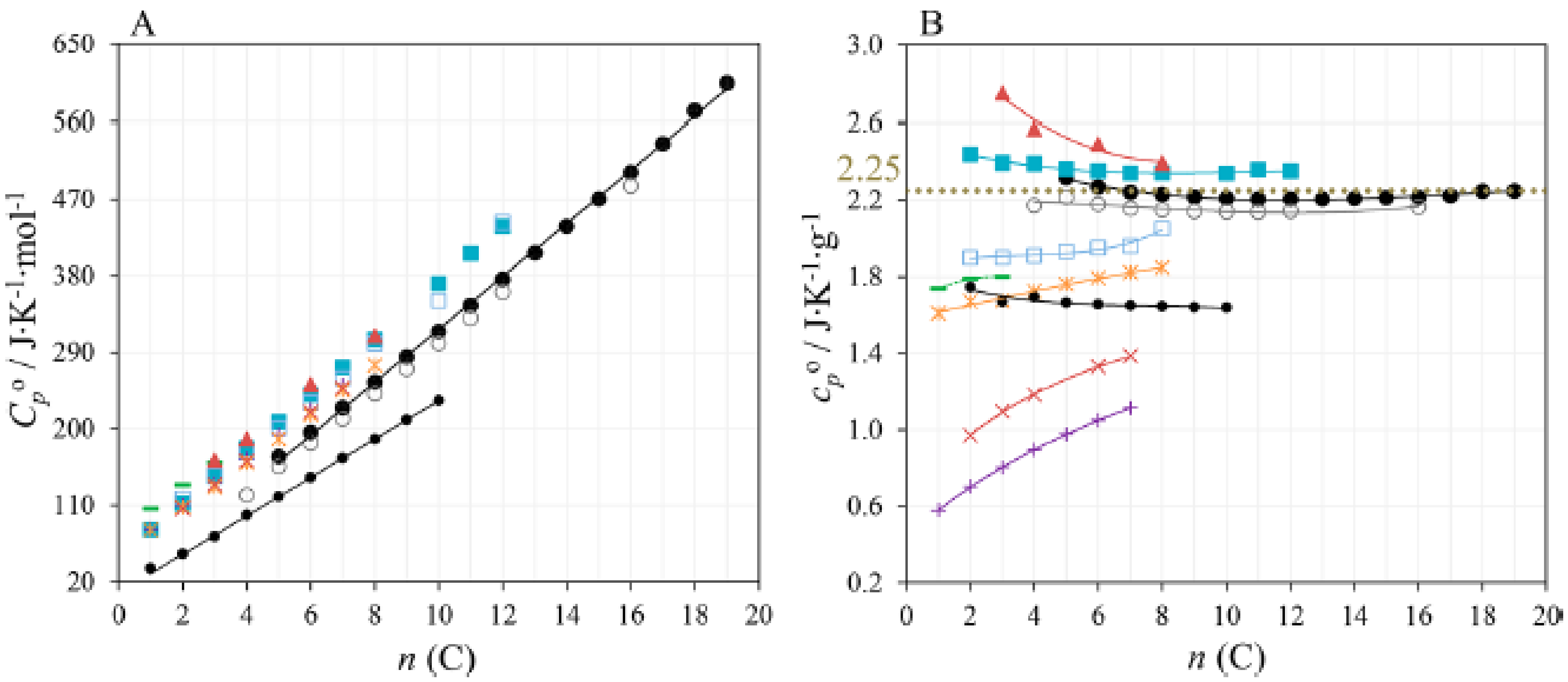




Other termal properties were studied:

- $T_{boiling}$ and $T_{melting}$;
- ΔH, ΔS and ΔG of fusion, vaporization and sublimation

Application note



n-alkanes (large solid black circle)
n-alkanols (blue solid square)
n-alkanamines (red solid triangle)
n-chloroalkanes (yellow asterisk)
n-iodoalkanes (purple plus)

n-alkenes (grey open circle)
n-alkanethiols (blue open square)
n-nitroalkanes (green dash)
n-bromoalkanes (red ×)
n-alkanes on gás phase (small solid black circle)

- Additive group contribution (-CH_2-) → change in C_p (becomes similar to alkanes)
- Contribution from functional groups
- Contribution from physical state
- Specific Heat Capacity → Lower for heavier atoms
- Cp converges to constant values at higher temperatures

Curiosity/particularity

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Contents lists available at ScienceDirect
Thermochimica Acta
journal homepage: www.elsevier.com/locate/tca
thermochimica acta

Measurement of specific heat capacity via fast scanning calorimetry—Accuracy and loss corrections

C.R. Quick[a], J.E.K. Schawe[b], P.J. Uggowitzer[a], S. Pogatscher[a,*]
[a] Chair of Nonferrous Metallurgy, Department of Metallurgy, Montanuniversitaet Leoben, Franz-Josef-Str. 18, 8700 Leoben, Austria
[b] Mettler-Toledo GmbH, Heuwinkelstrasse 3, 8606 Nänikon, Switzerland

- Measurements of specific heat capacity in metals via fast scanning calorimetry
- Introduces a correction fator (small mass rates) → $\varphi = mc_p\beta + \varphi_{loss}$

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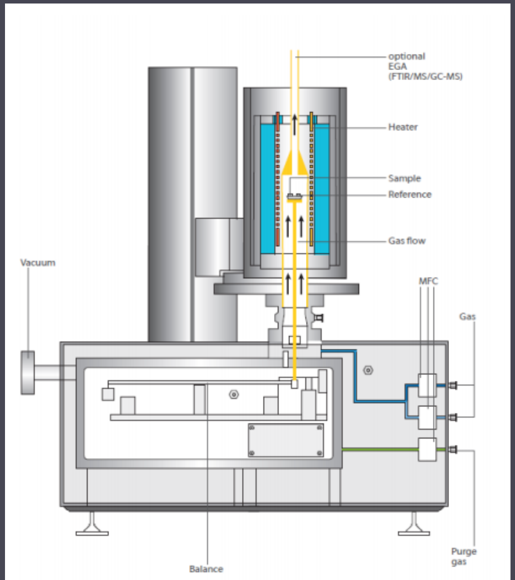


MAT 2021

METHODS OF THERMAL ANALYSIS

Thermal Stability of Ionic Liquids by TGA

Carlla Lorena Façanha Silva de Vasconcelos
Industrial Chemistry – UFC

MAT2021

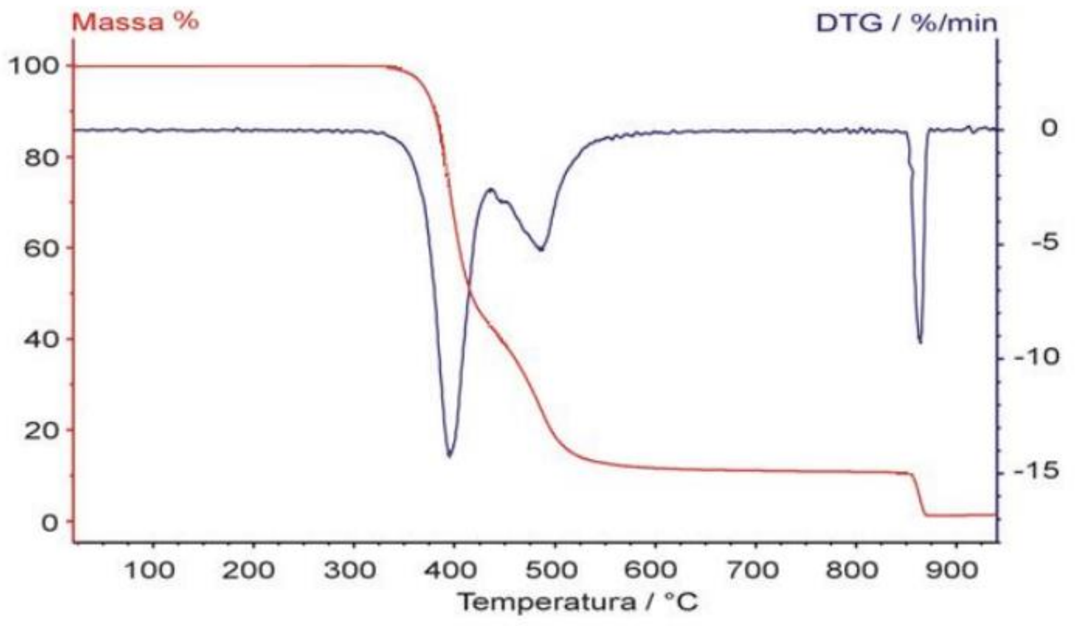
Métodos de Análise Térmica
Thermal Analysis Methods

Introduction

TGA

Sample mass variation by:
- Temperature programming function
- Atmosphere variation

- Study of the thermal decomposition of substances
- Studies on corrosion of metals in atmospheres
- Study of dehydration and hygroscopicity



Graph 1 - Graph of TGA (red) and its derivative, DTG (blue)

Wust, K. M. Estabilidade térmica, oxidativa e suporte em sílica mesoporosa de líquidos iônicos derivados do ibuprofeno. Santa Maria. 2018

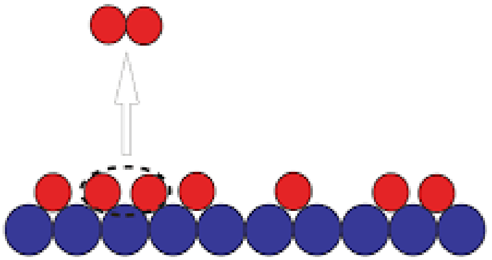
Introduction

Mass loss

- Decomposition
- Desorption
- Dehydration
- Desolvation
- Volatilization

Mass gain

- Oxidation
- Adsorption



Principles of Technique

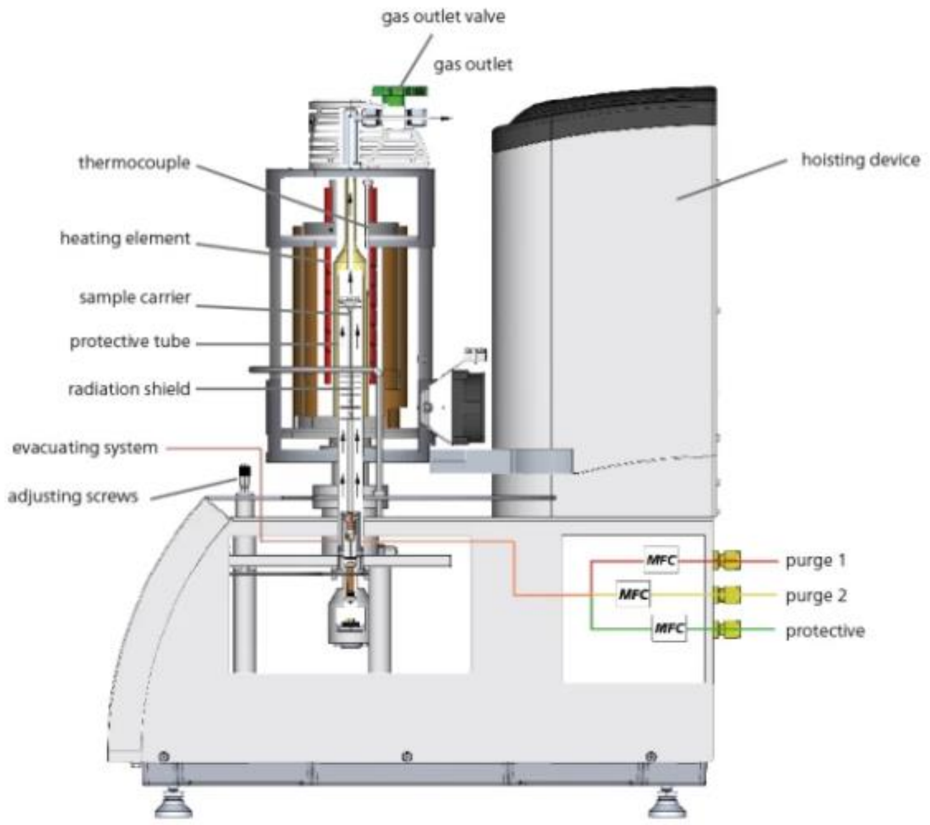
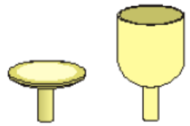


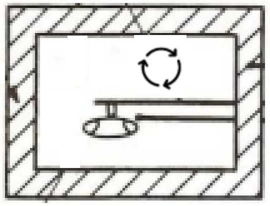
Figure 1 - Schematic of a TGA instrument

Table 1 - Main factors influencing TGA analysis :

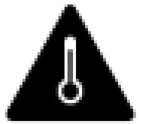
Instrumental Factors
Oven heating ratio
Paper registration speed
Oven atmosphere
Sample support geometry
Scale sensitivity
Composition of the sample holder
Sample Factors
Sample quantity
Solubility of the gases involved
Particle size and the heat of reaction
Sample packaging
Nature of the sample
Thermal conductivity



sample support



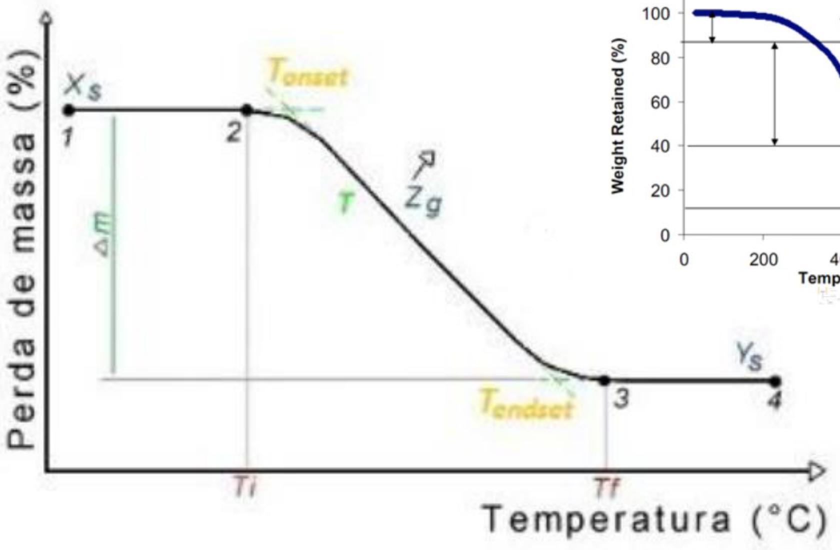
Oven atmosphere

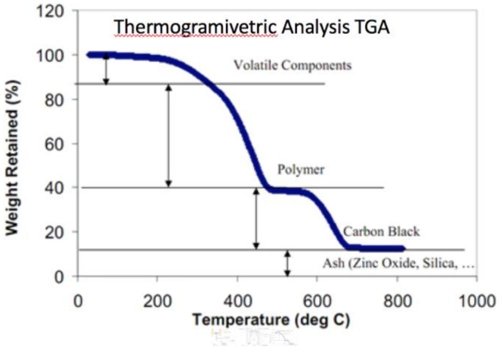


Temperature variation

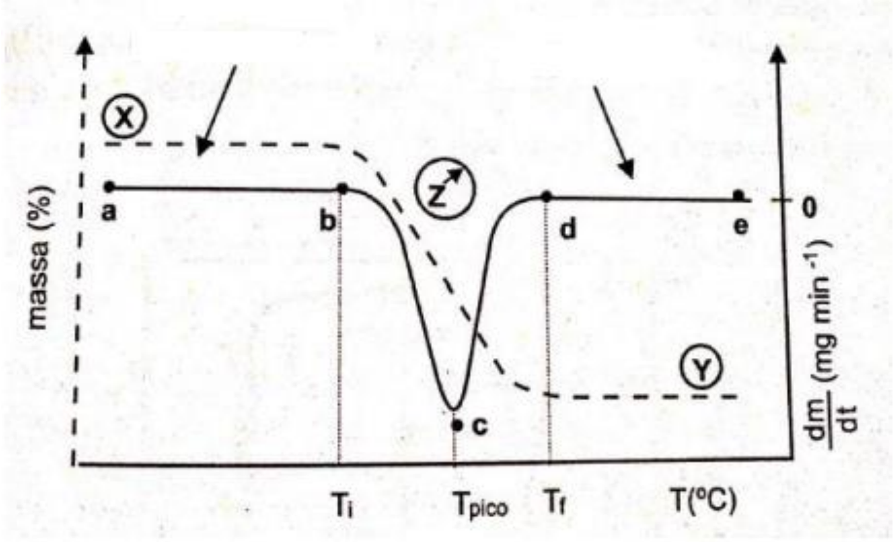
Denari, G.B; Cavalheiro, E, T.G. Principios e aplicações de análise térmica. São Carlos, 2012

Principles of Technique





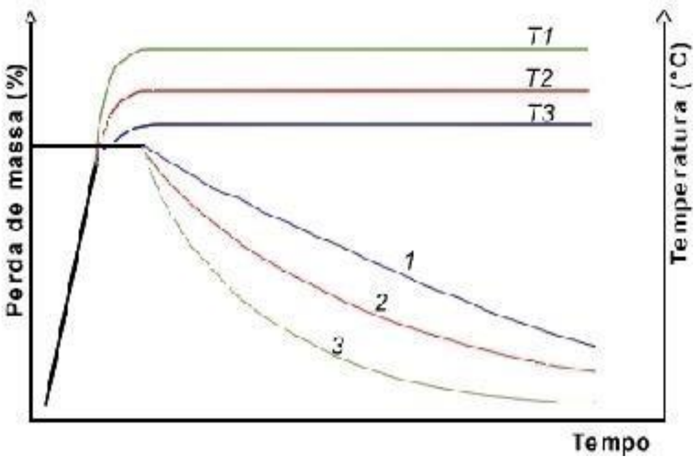
- Xs is a intial mass
- 2 – start of thermal decomposition
- 3 – end of thermal decomposition
- Line T, extrapolated Tonset and Tendset
- 1, 2, 3 and 4 we have the amount of mass released from the sample.



- X is the initial mass
- b - start of thermal decomposition
- c - temperature at which the reaction speed is maximum
- d – end of thermal decomposition
- bcd - area proportional to mass loss

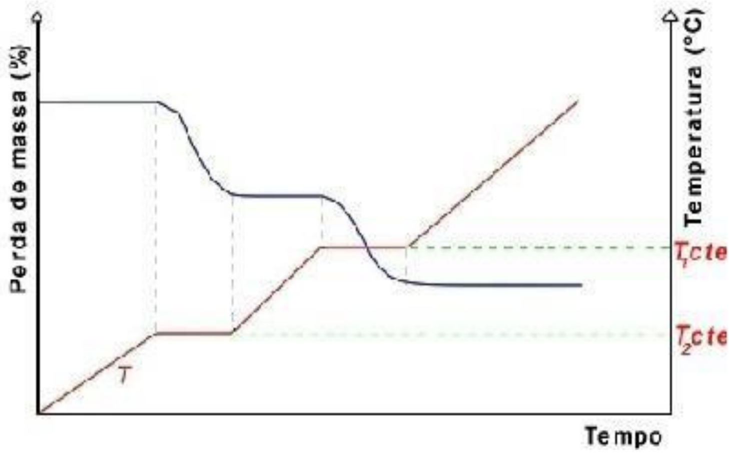
IONASHIRO, M. Giolito: Fundamentos da Termogravimetria, Análise Térmica Diferencial, Calorimetria Exploratória Diferencial. São Paulo: Giz, 2005.

Principles of Technique



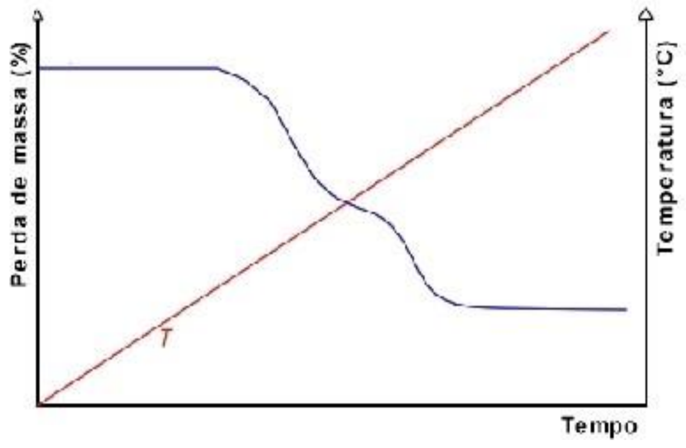
Graph 4- isothermal TG

- Constant temperature



Graph 5- almost isothermal

- Constant temperature until mass variation, then it's held constant until stabilization, and then the temperature goes back up until the next mass change

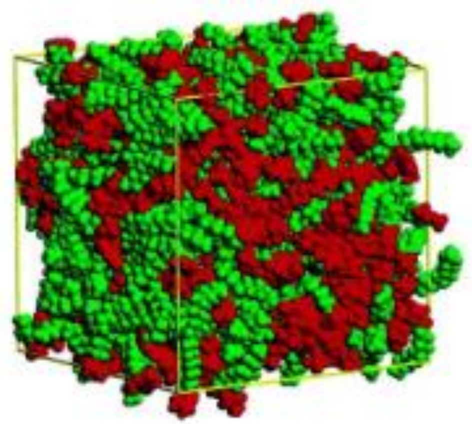


Graph 6- conventional TG

- The temperature increases or decreases linearly

Novelli, M. Estudo comparativo de borrachas utilizadas como guarnição em carenagens de geradores de energia a diesel. São Carlos, 2015.

Application: Thermal stability analysis of ionic liquids by TGA



Organic salts found in the liquid phase at temperatures below 100°C

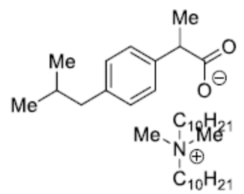
Bulky and asymmetric cations and anions

↓

Reduce crystal lattice energy

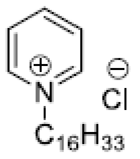
1 trillion combinations

- **Pharmaceutical industry**

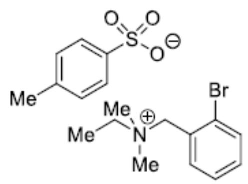


Ibuprofenato de dicecilmetilamônio

$[N_{10,10,1,1}][IBU]$

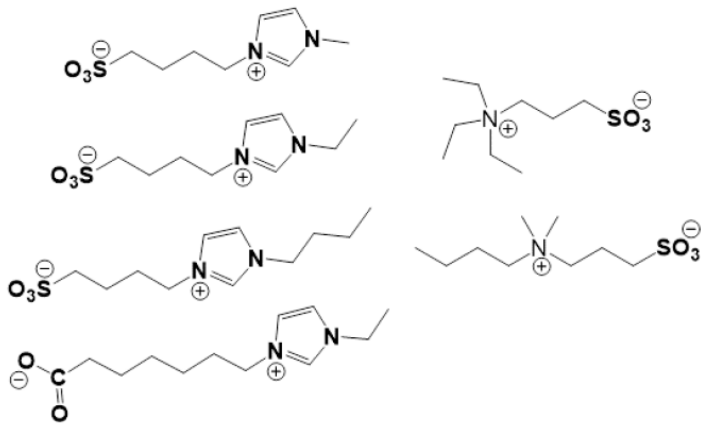


Cloreto de cetilpiridínio



Tosilato de bretílio

- **Materials industry**



MAT2021
Métodos de Análise Térmica
Thermal Analysis Methods

Application: Thermal stability analysis of ionic liquids by TGA

Pharmaceutical Industry

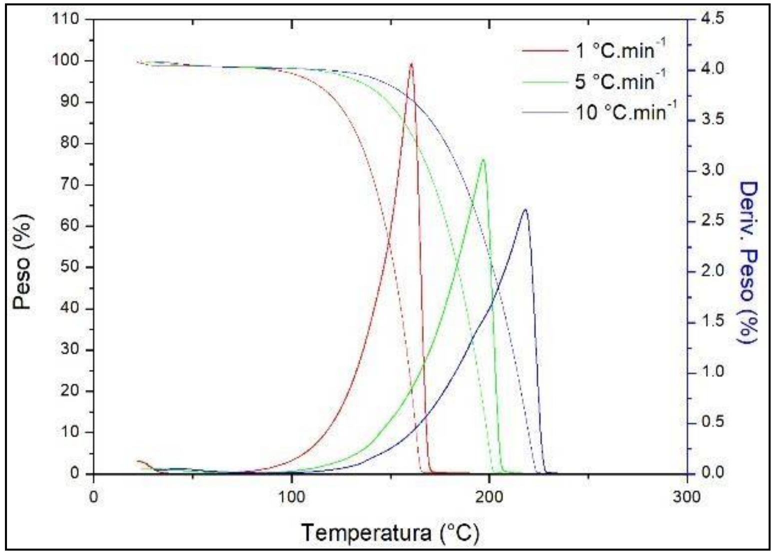
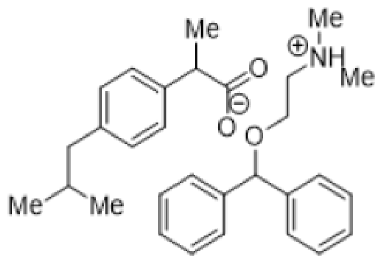


Figure 3 - Profile of [DIF][IBU] decomposition at rates 1, 5 and 10 °C min-1.



[DIF][IBU]

LI - IFAS (cations or anions of pharmacologically active ingredients)

Relevancy:.

- Transport and Storage;
- Technological transformations;
- Development of formulations;
- Decomposition of contaminants;

Wust, K. M. Estabilidade térmica, oxidativa e suporte em sílica mesoporosa de líquidos iônicos derivados do ibuprofeno. Santa Maria. 2018

Application: Thermal stability analysis of ionic liquids by TGA

Materials Industry

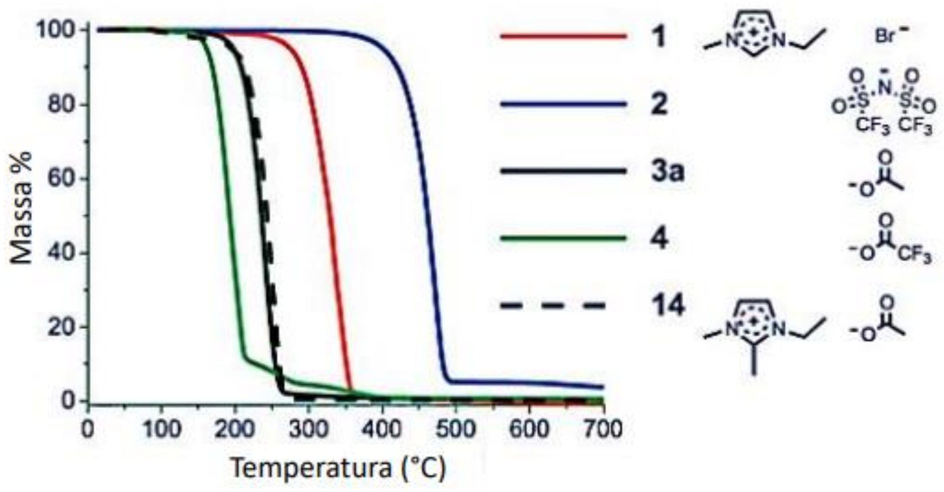
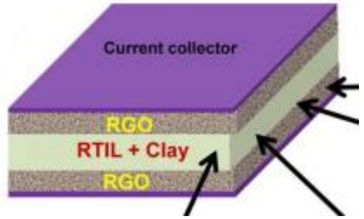


Figure 5- Thermogravimetric analysis of some ionic liquids



LI for supercapacitor production

Relevancy:

- Devices that operate at high temperatures

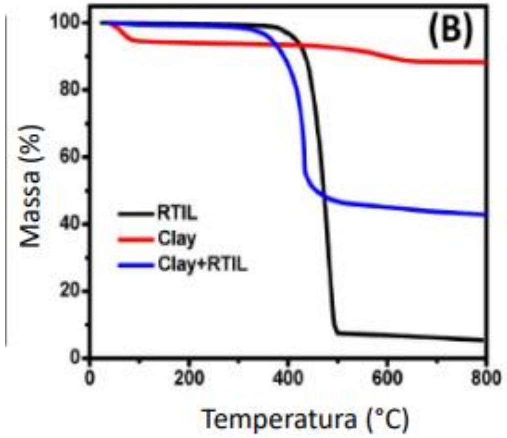


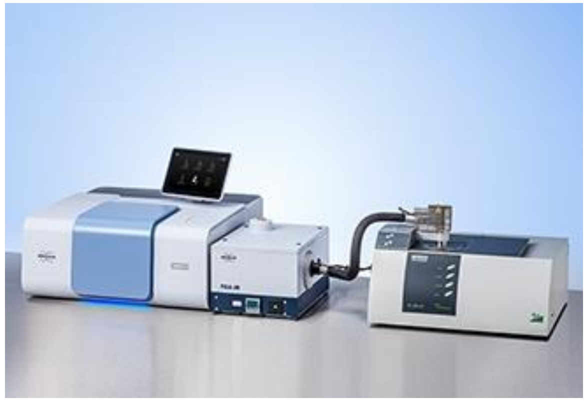
Figure 6- Thermogravimetric analysis of ionic liquid, BMMI-TFSI ionic liquid with clay and pure clay.

Borges, R. S.; Reddy, A.L.M; Rodrigues, M. T.; Gullapalli, H. Balakrishnan, K. Silva, G. G. ; Ajayan, P. M. Supercapacitor Operating At 200 Degrees Celsius. SCIENTIFIC REPORTS | 3 : 2572 |2013

Curiosities and particularities

Coupling with:

- Fourier Transform Infrared Spectroscopy (FTIR)
- Mass Spectroscopy (MS)



TGA with FTIR

TGA with MS

TG-TDA