



# HIGH-PRESSURE POLYMORPHISM



**Grup de Caracterització de Materials**  
**June 2009**





## 1.- Introduction

- ODIC phases
- Compounds and objectives
- Metastability
- Isomorfism

## 2.- Experimental techniques

- Calorimetry
- Diffractometry
- Dilatometry

## 3.- Pure compounds

- CBr<sub>4</sub>
- Cl<sub>3</sub>CBr

## 4.- Binary System (Cl<sub>3</sub>CBr)<sub>1-x</sub> (CBr<sub>4</sub>)<sub>x</sub>

## 5.- Results

## 6.- Conclusions

## 7.- New Compounds





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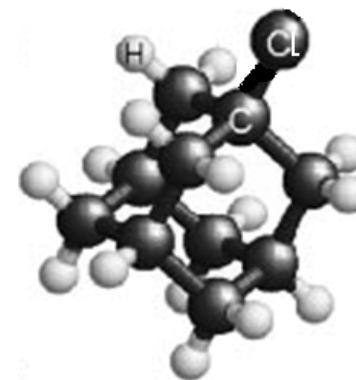
# 1. Introduction

## ODIC phases

- Phases with translational order and no orientational order
- High symmetry structures

## Compounds

- Halogen derivatives of neopentane and ethane
- Cyclic compounds
- Adamantane derivatives





# Objectives

- **Polymorphism**

→ Thermal and Crystallographic characterization at high pressure

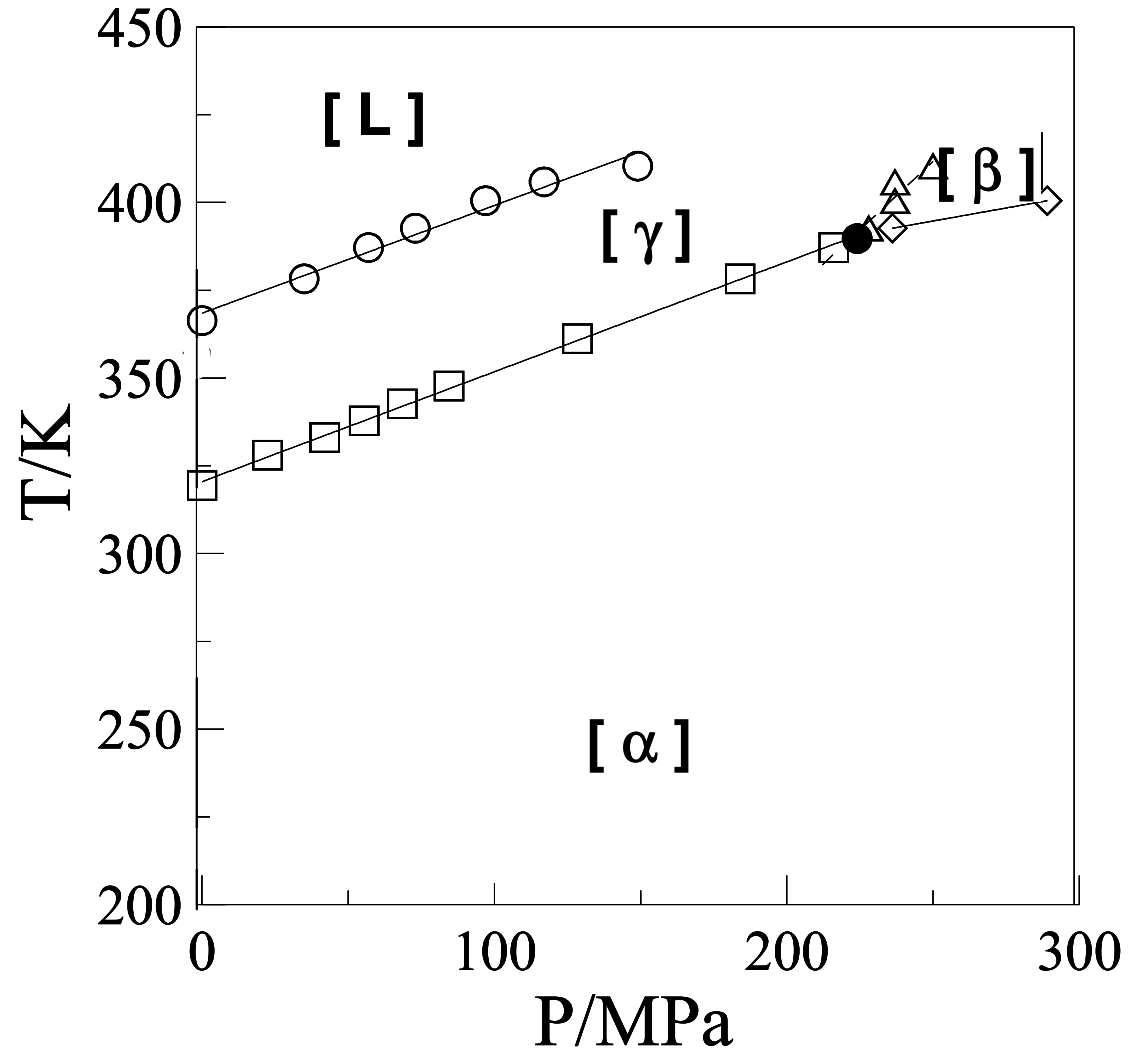
- **Alloy forming between compounds**

→  $p_{\text{normal}}$

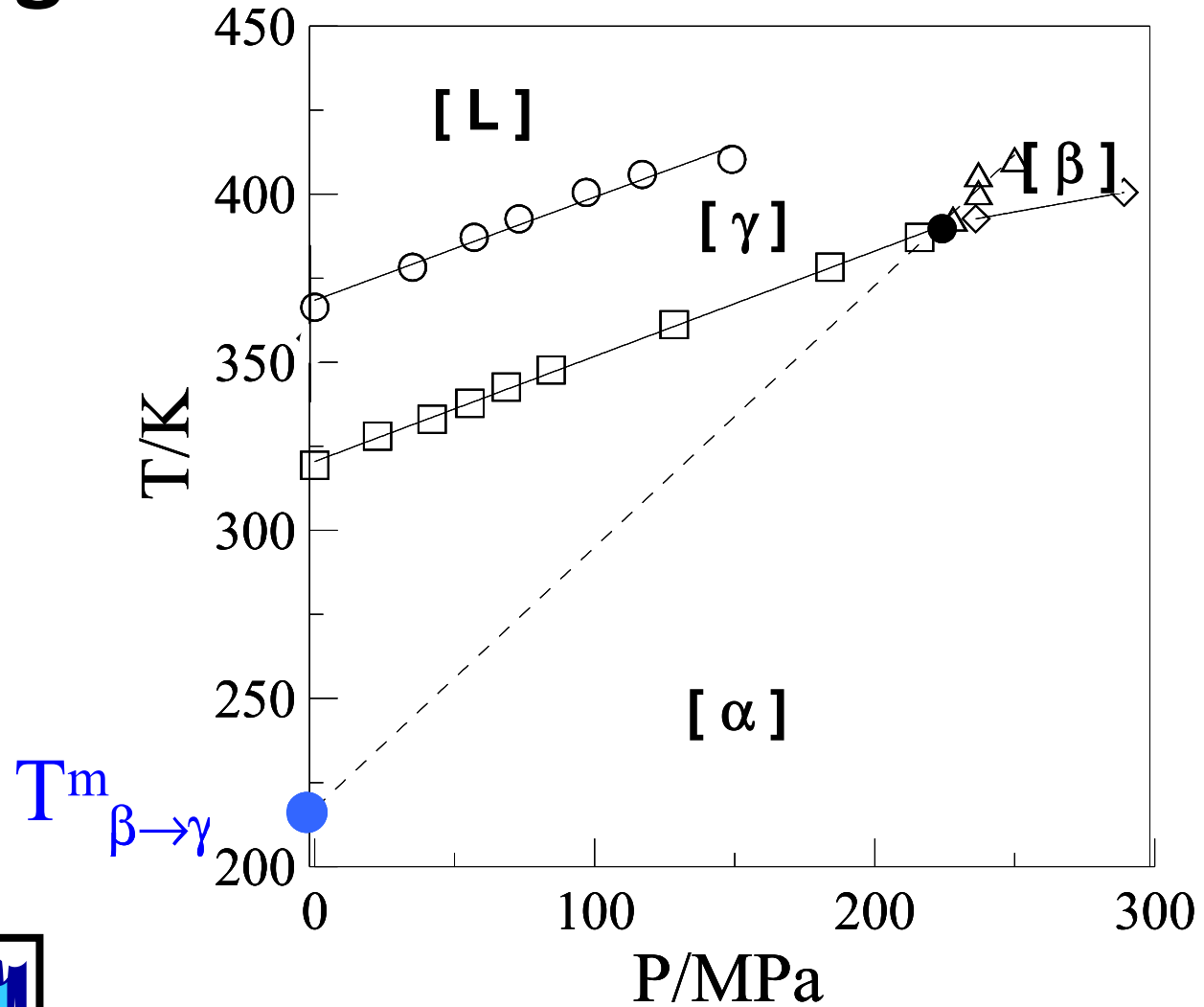
Binary systems at  $p_{\text{normal}}$



# Pressure – Temperature diagrams



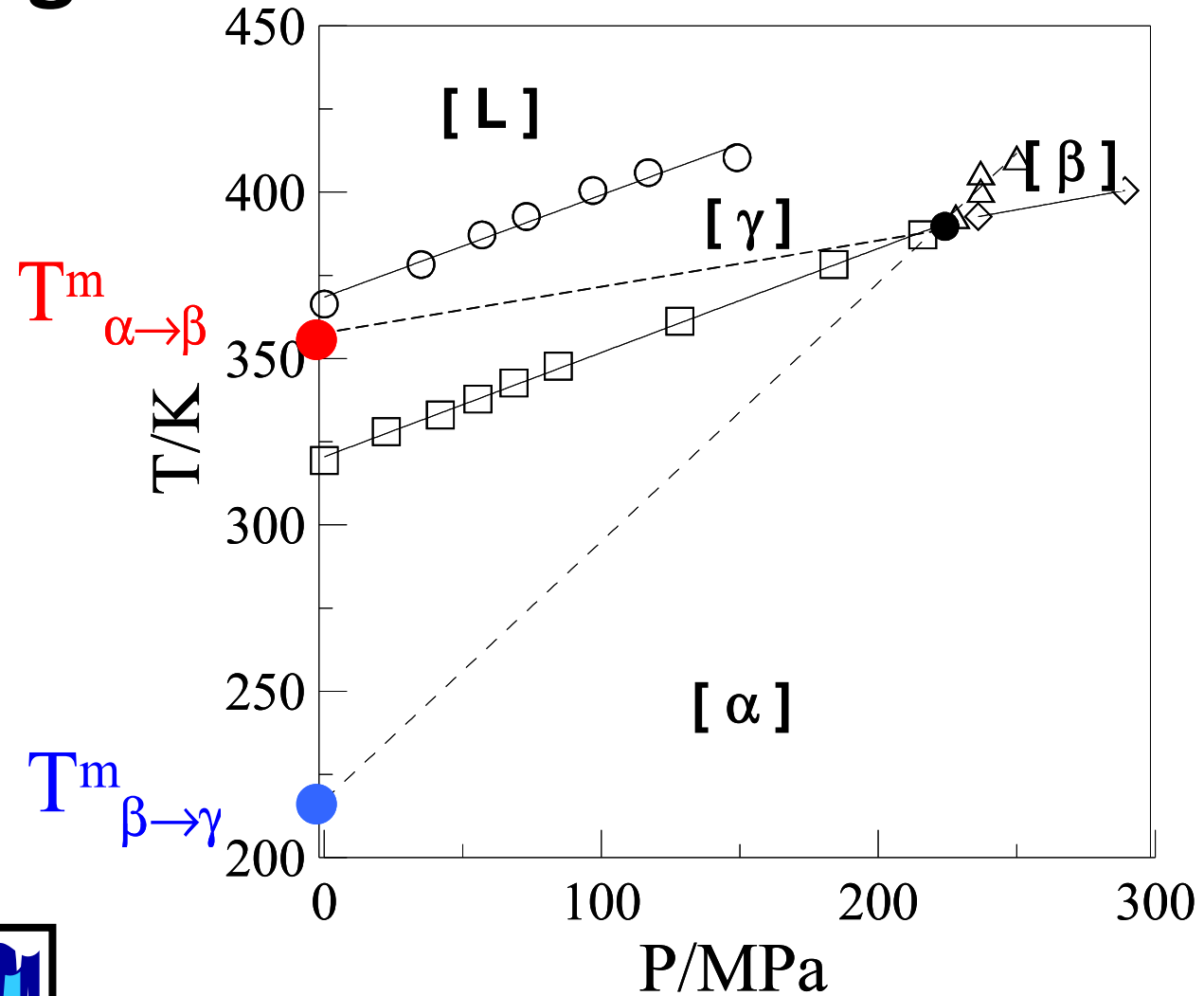
# Pressure – Temperature diagrams





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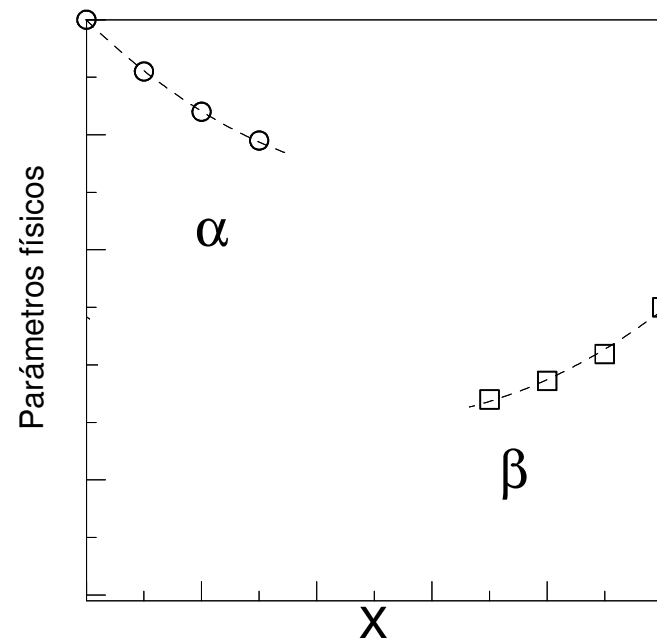
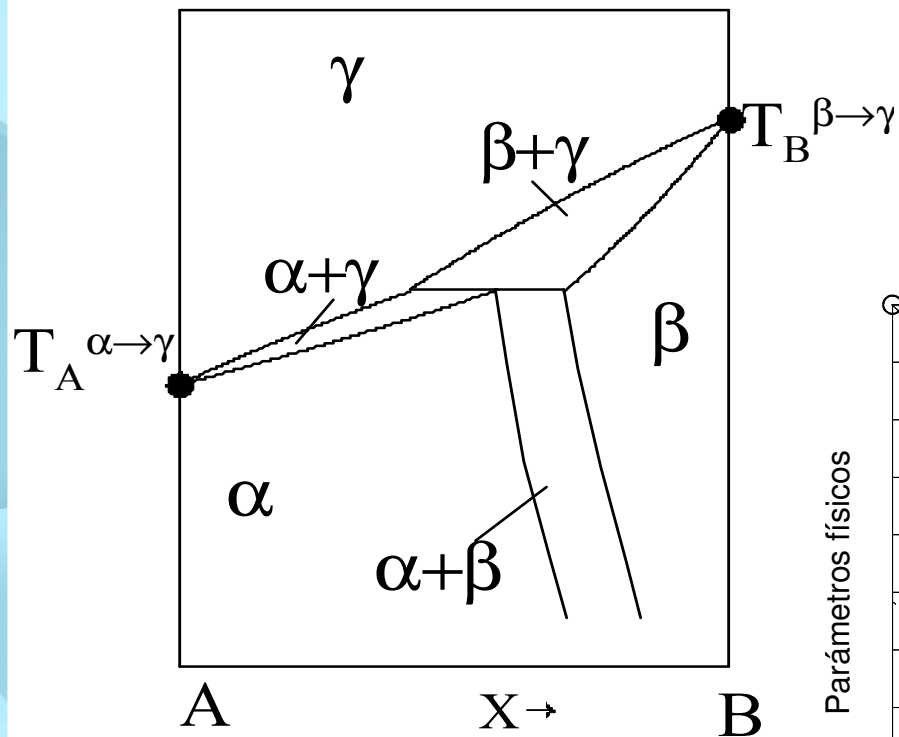
## 1. Introduction







# Cross isodimorfism

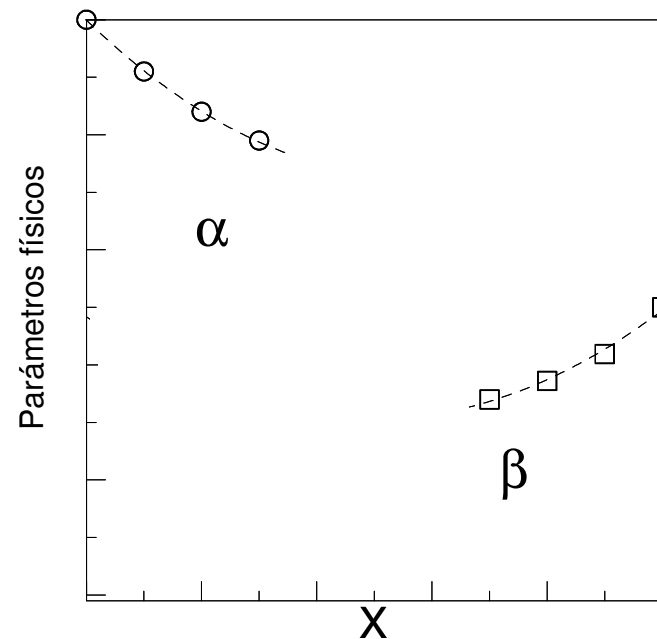
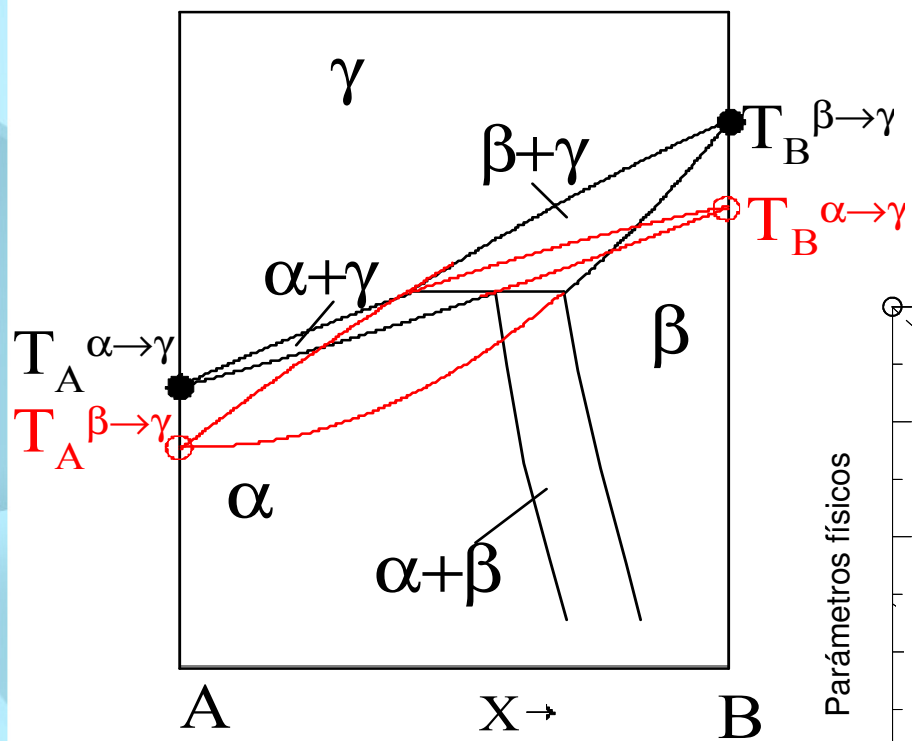


## 1. Introduction





# Cross isodimorfism

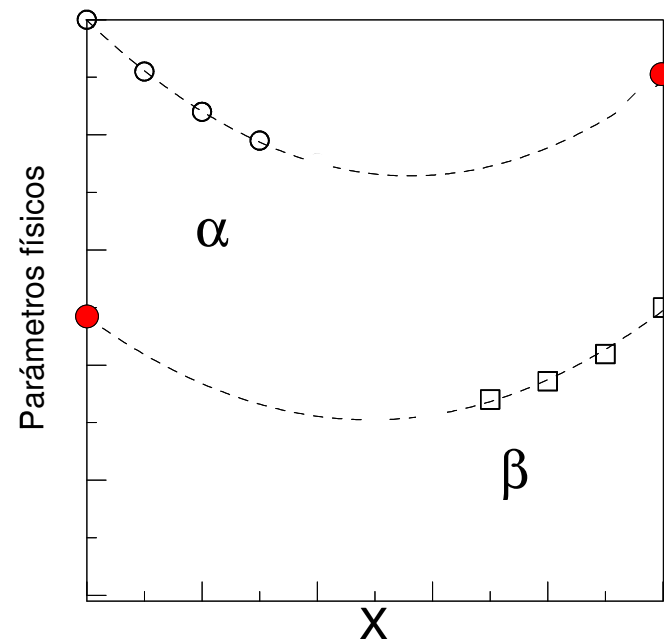
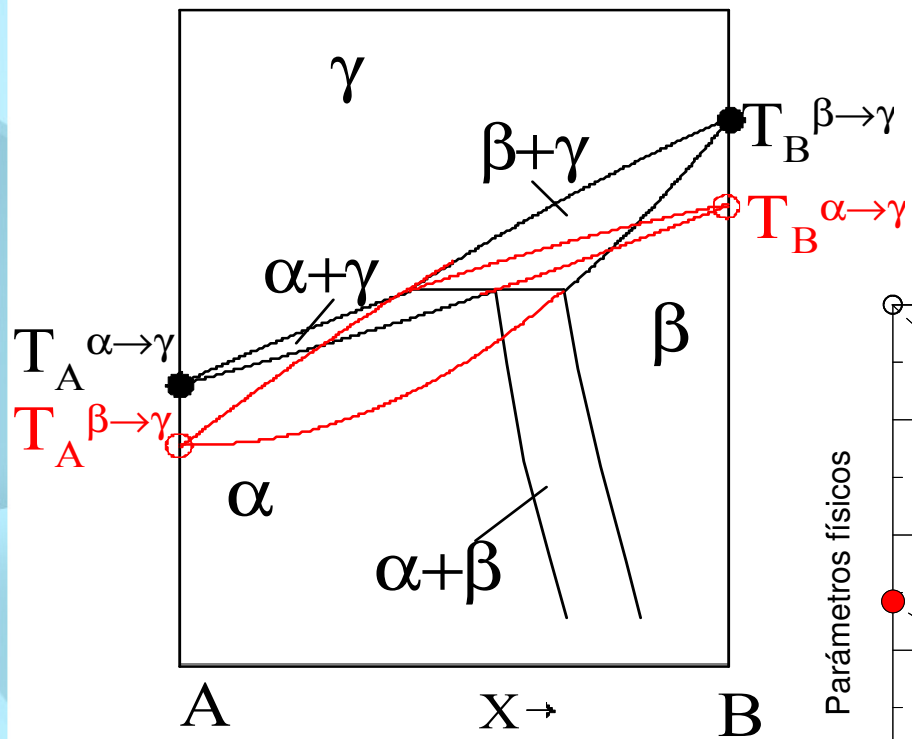


## 1. Introduction





# Cross isodimorfism



## 1. Introduction





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- **Calorimetric**
- **Difractometric**
- **Dilatometric**

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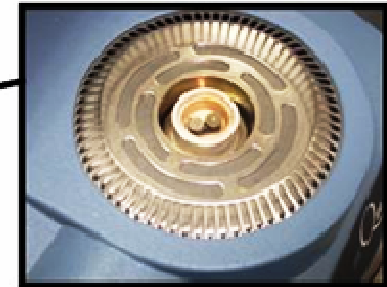
## 7.- New Compounds



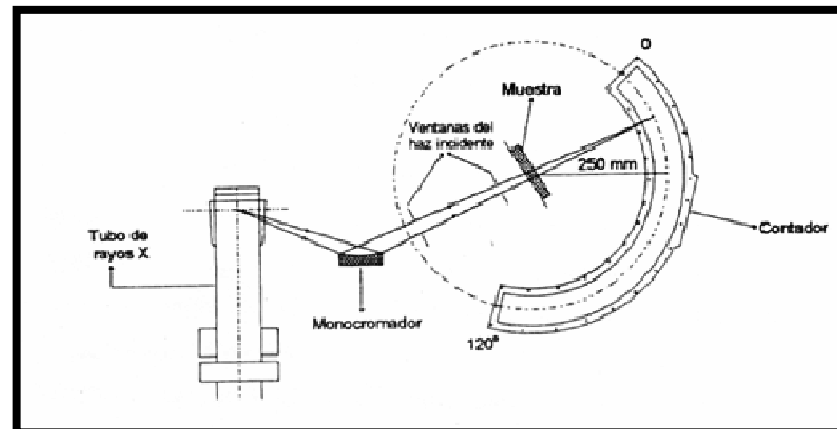


## Diferential Thermal Analysis

- At normal pressure (comercial)
- De 0 a 300 MPa (no comercial)

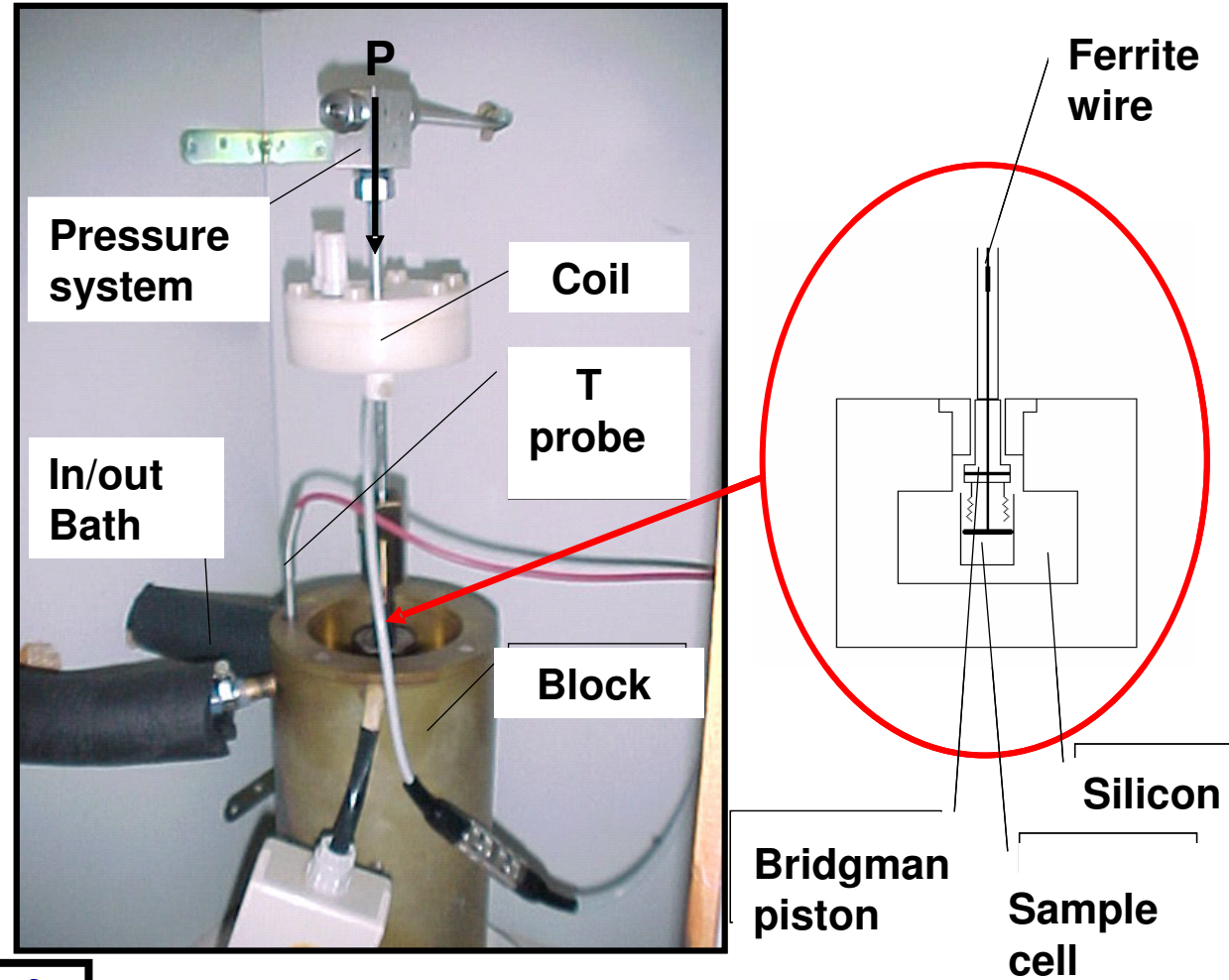


## INEL diffractometer





# High pressure dilatometer





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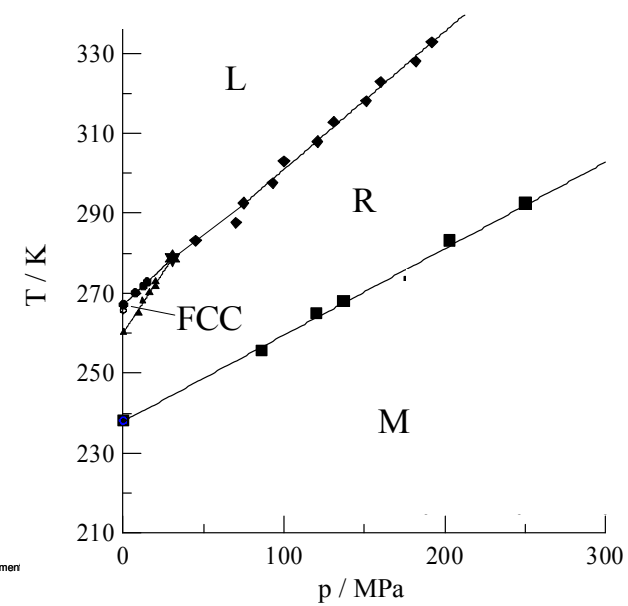
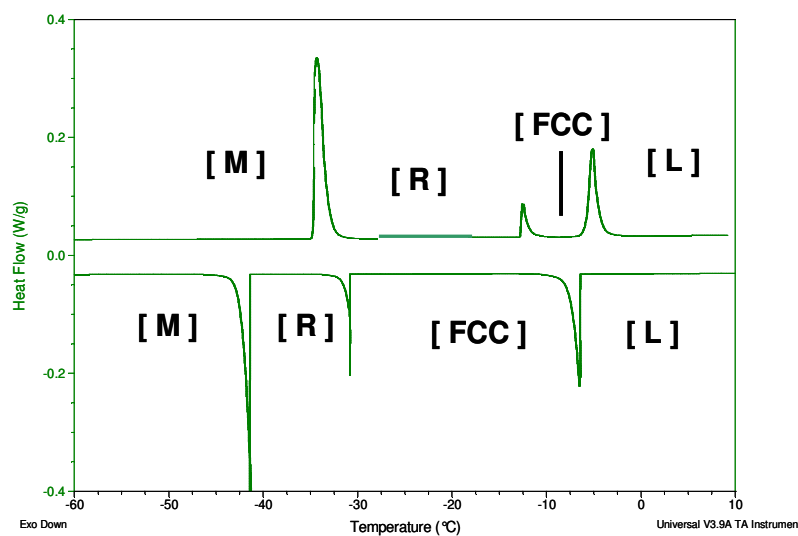
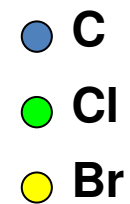
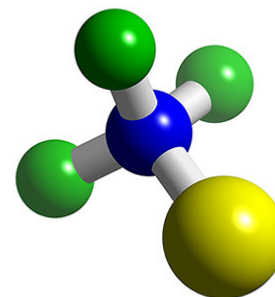
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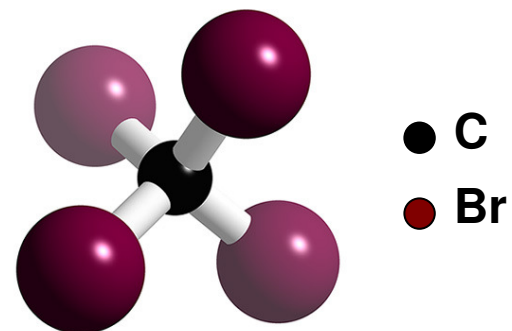
# 3. Pure compounds



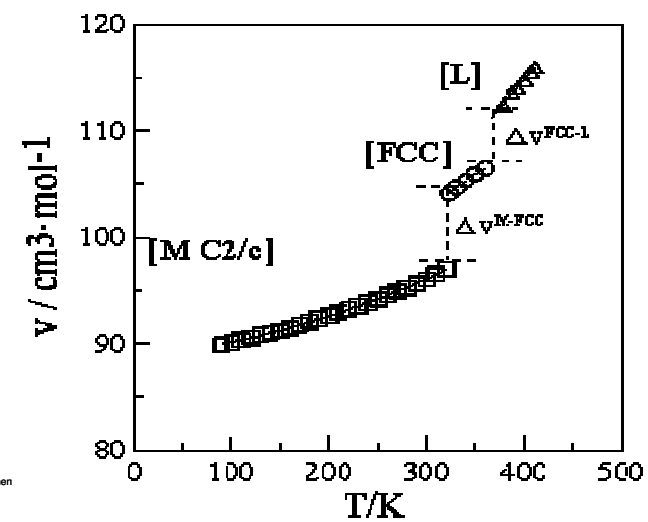
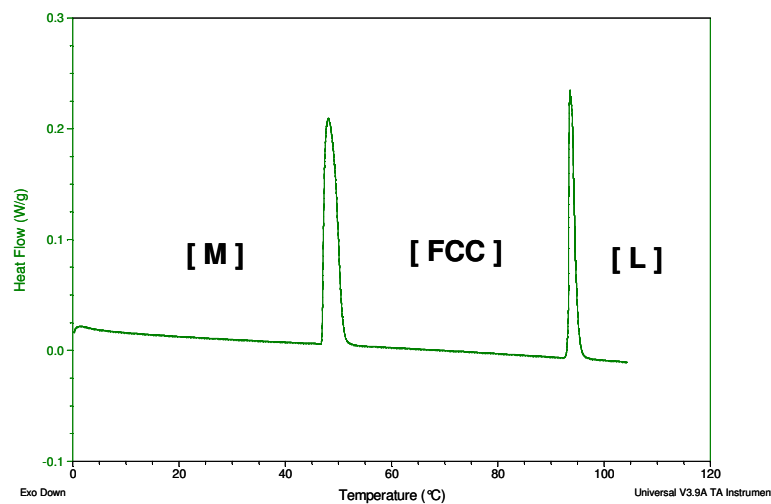




# CBr<sub>4</sub>

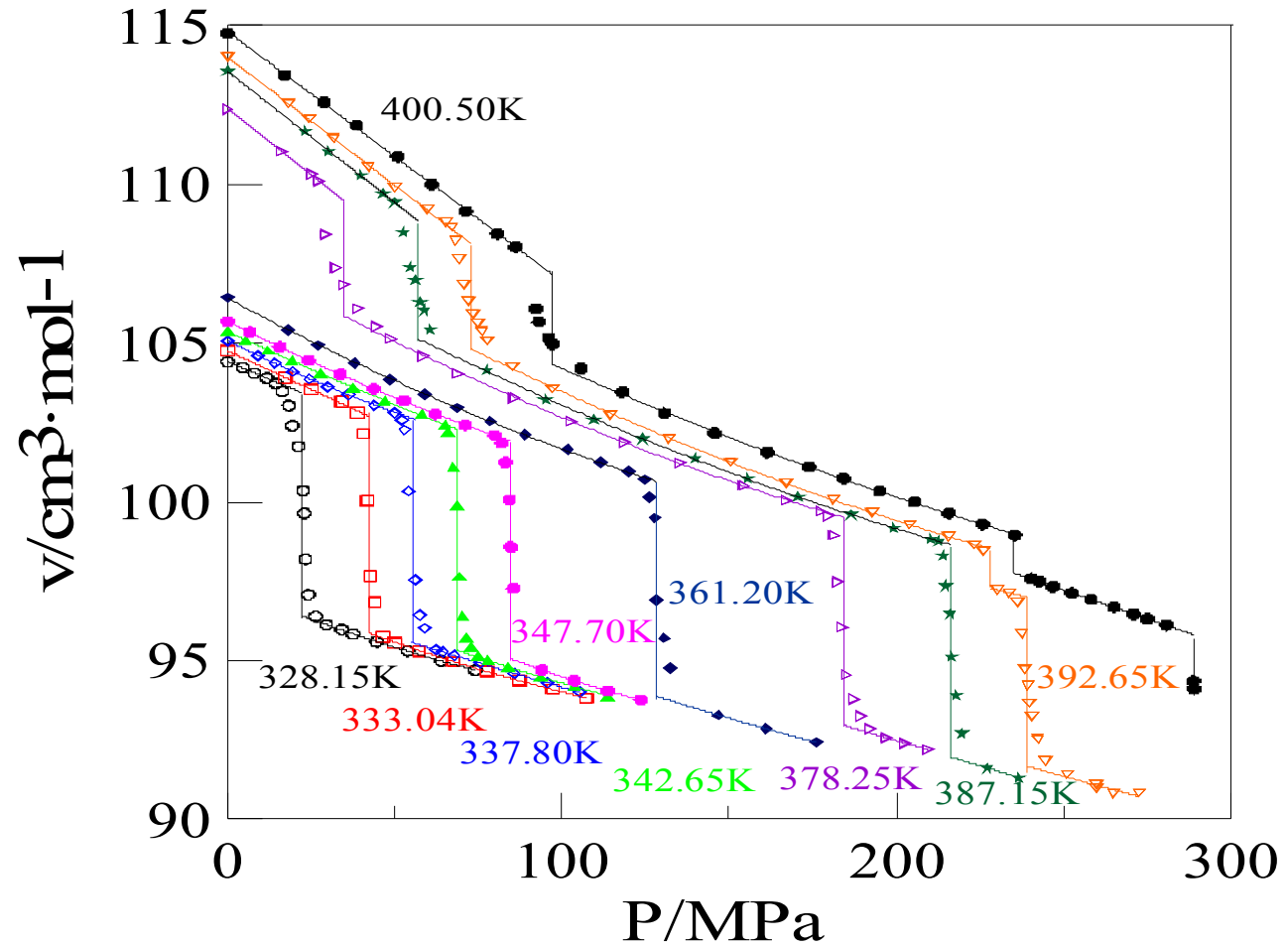


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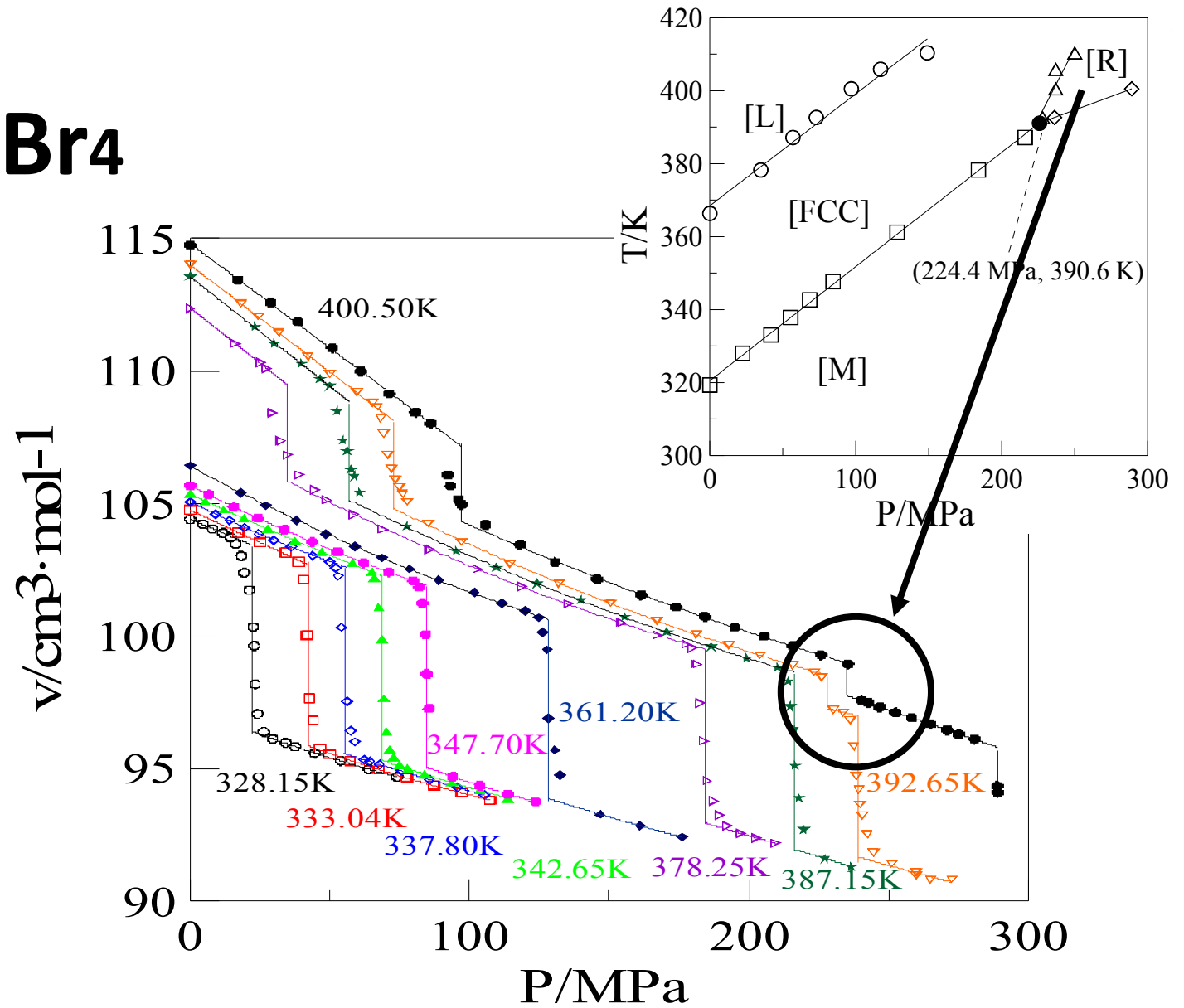
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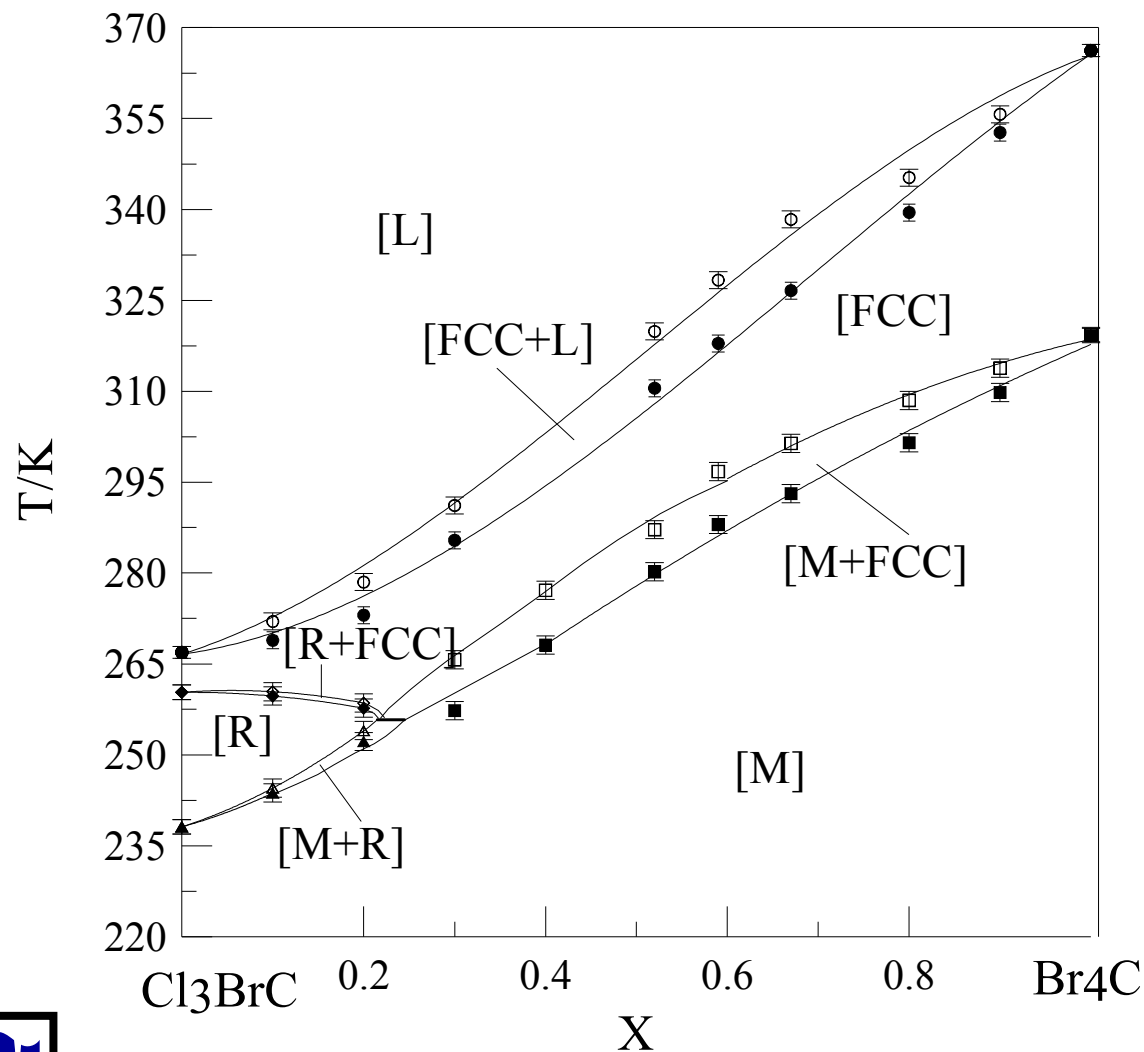
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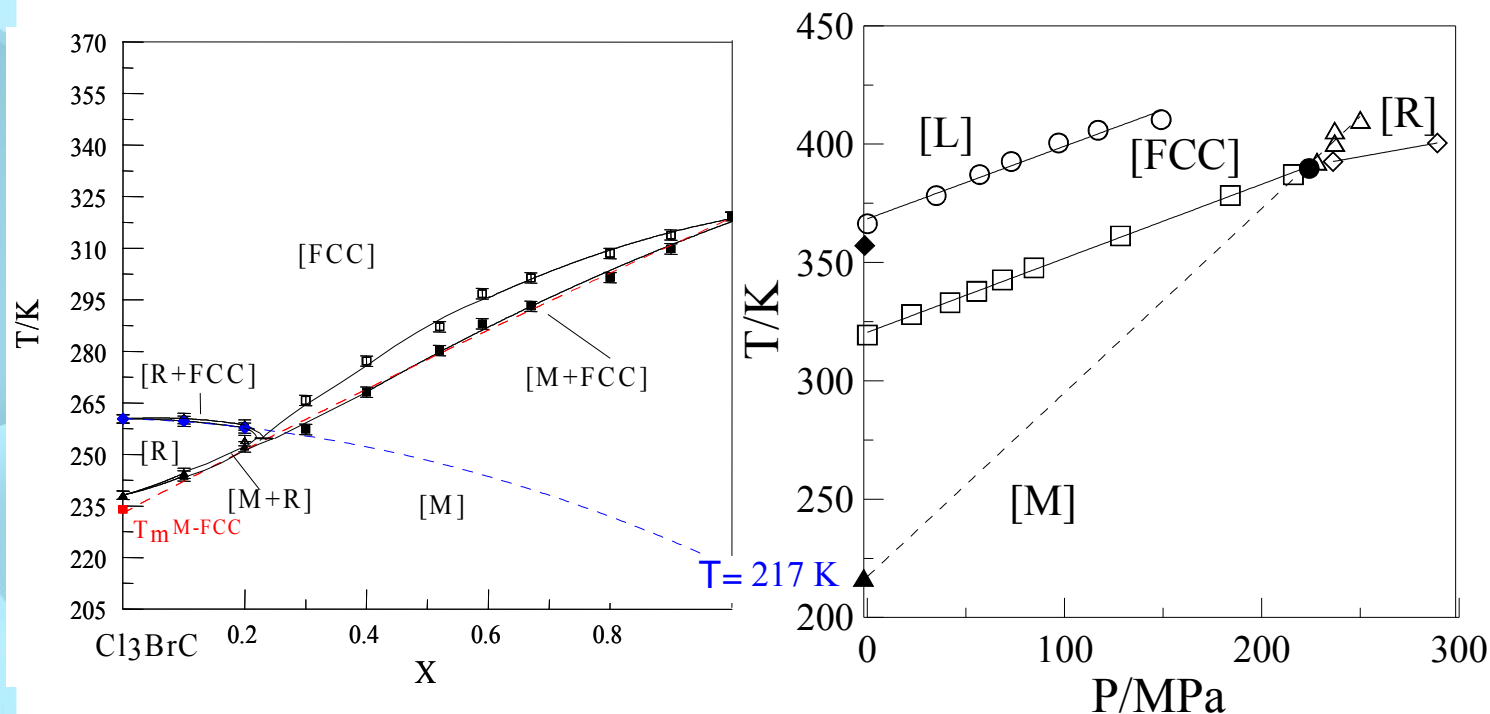
## Diagrama T-X experimental





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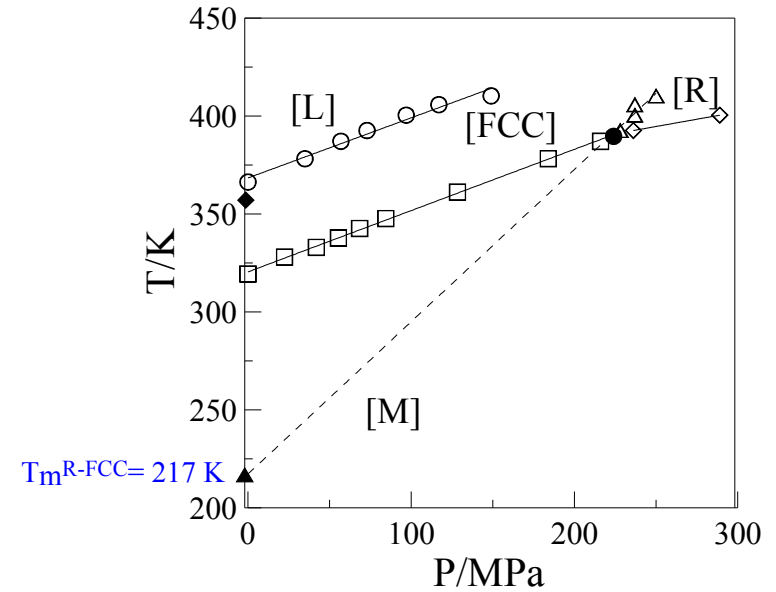
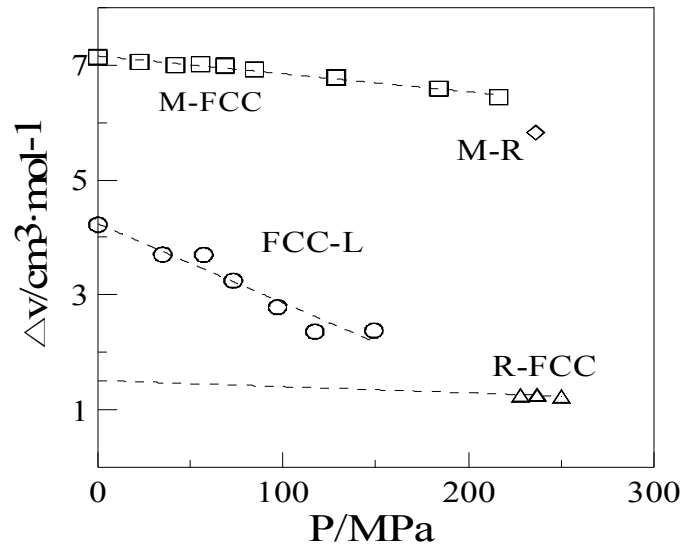
## 7.- New Compounds



# 5. Results interpretation



## T-X diagram discussion



$$R \rightarrow FCC \left\{ \begin{array}{l} \Delta v^{Ext} = 1.41 \text{ cm}^3/\text{mol} \\ \left( \frac{dP}{dT} \right)^{P-T} = 1.64 \text{ MPa/K} \\ \Delta H = 0.50 \text{ kJ/mol} \end{array} \right.$$

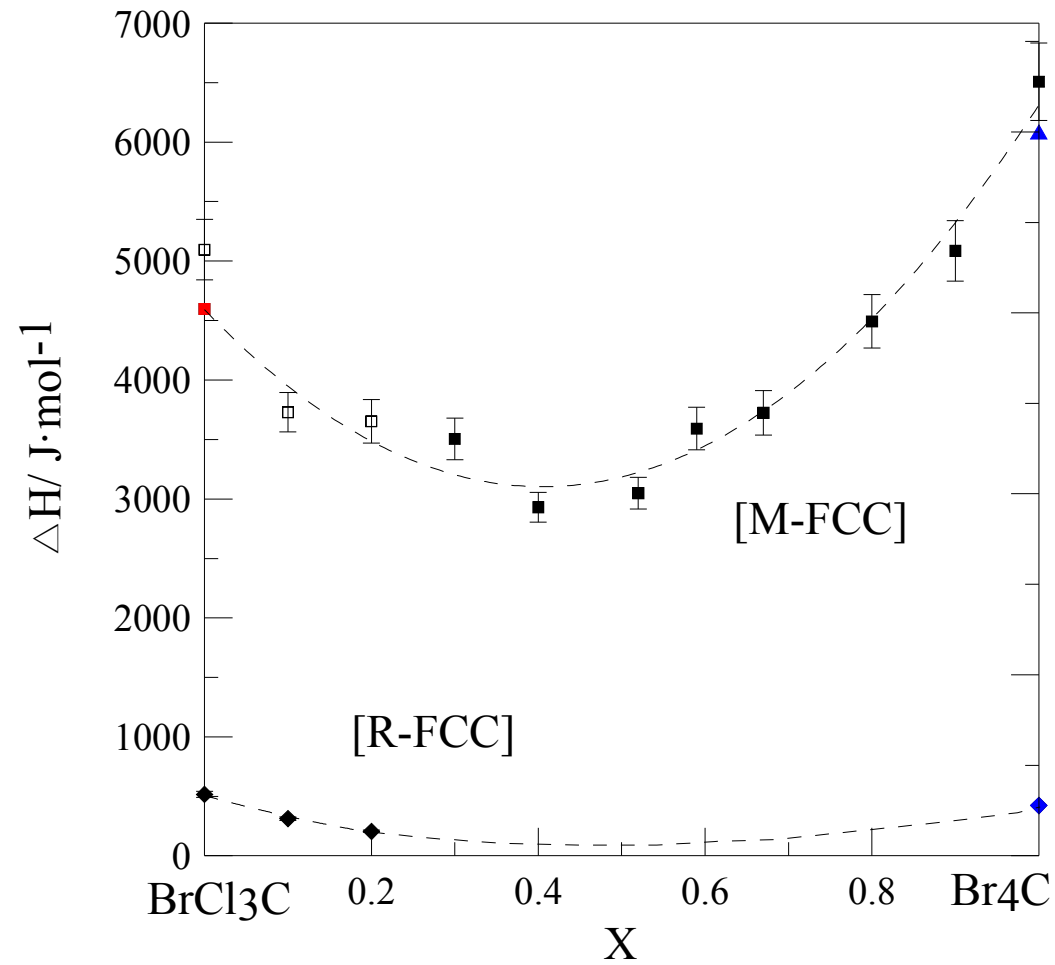
$$\frac{dP}{dT} = \frac{\Delta H}{T \cdot \Delta v}$$





# 5. Results interpretation

## Enthalpic interpolation





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## 6. Conclusions



- **Se ha determinado el diagrama experimental PVT y el diagrama experimental PT del  $\text{Br}_4\text{C}$ , con la aparición de una nueva fase de alta presión con simetría romboédrica ODIC.**
- **Se ha determinado experimentalmente el sistema binario  $\text{BrCl}_3\text{C}-\text{Br}_4\text{C}$ , comprobando el isomorfismo entre sus fases monoclinicas y FCC; y mediante el formalismo del isodimorfismo cruzado se infiere una fase de alta presión con simetría romboédrica y orientacionalmente desordenada, estudiándose la coherencia del sistema binario con el compuesto puro.**





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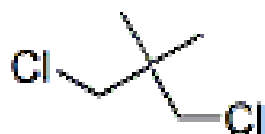
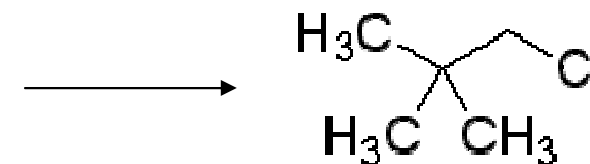


# Neopentane Chlor Derivatives

## 7. New Compounds

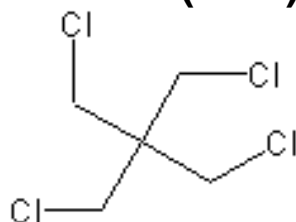
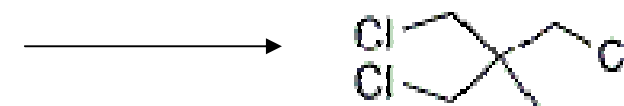


1-chloro-2,2-dimethyl-propane  
(1CL)



2,2-Dimethyl-1,3-Dichloropropane  
(2CL)

1,1,1-Tris(chloromethyl)ethane  
(3CL)



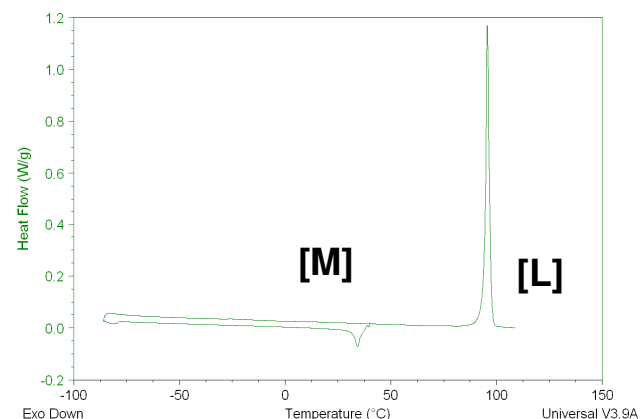
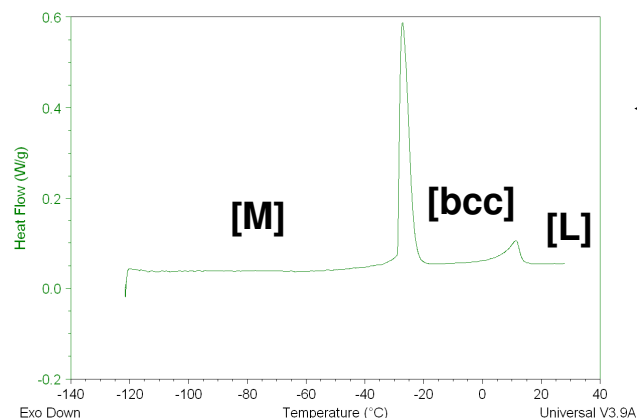
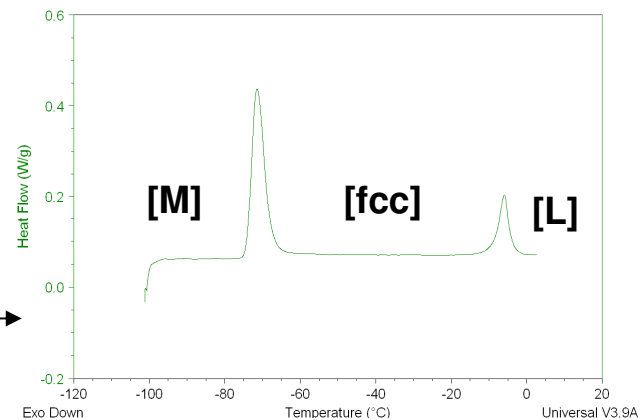
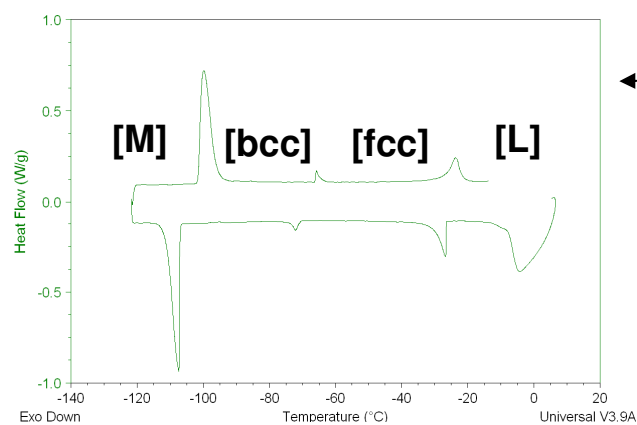
Pentaerythrityl tetrachloride  
(4CL)





# Thermal analysis

## 7. New Compounds





# Binary Systems

2CL – 3CI

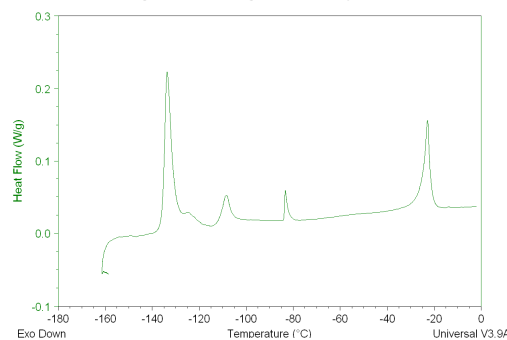
1CL - 2CL

1CL – 3CI

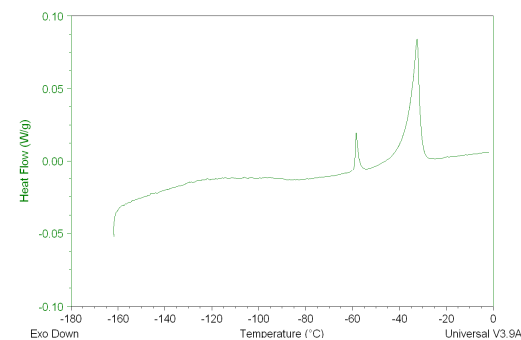
3CL - 4CL

## Examples

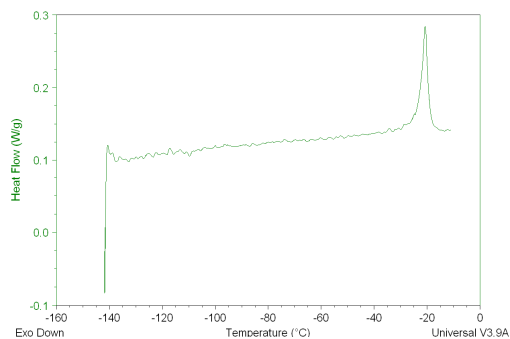
1CL-2CI 10%



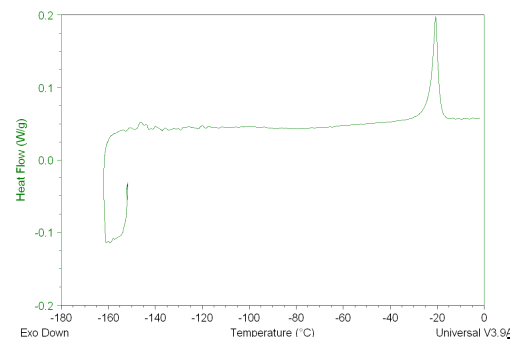
1CL-3CI 10%



1CL-2CI 30%



1CL-3CI 30%

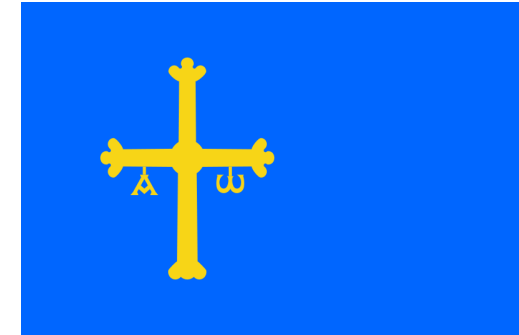
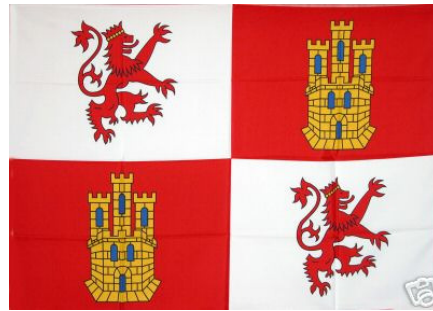


7. New Compounds





# Gracias por su atención



*La força d'un sentiment*

