



Ministério do
Desenvolvimento, Indústria
e Comércio Exterior



Nanometrology in Brazil



Carlos Achete

Outline



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government



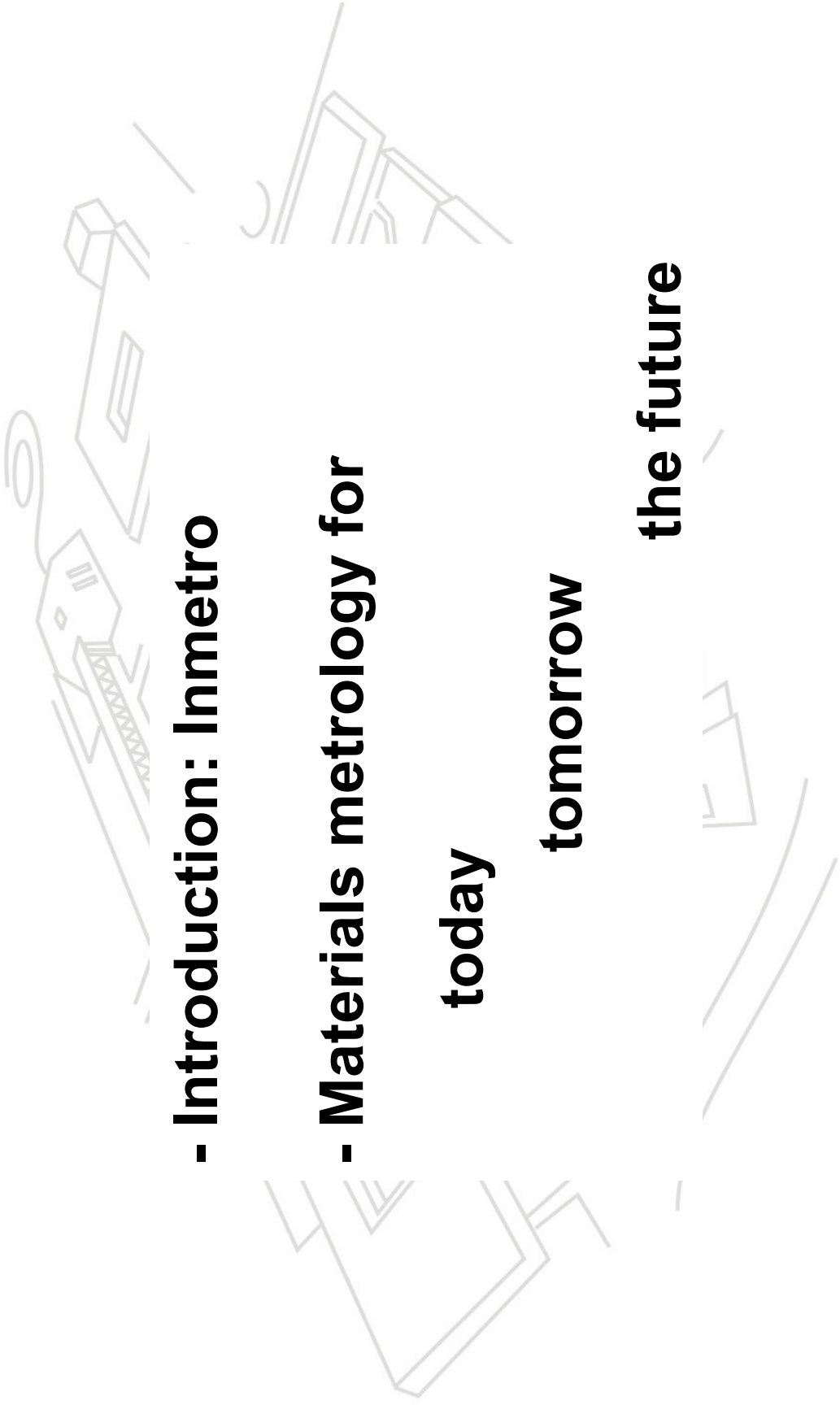
- Introduction: Inmetro

- Materials metrology for

today

tomorrow

the future



Short historic



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

- Early 1900: NIST, PTB, NPL

- 1973: Inmetro

Brazilian products reached quantity level
for gaining the international market. Efforts
have to be made for guaranteeing quality.

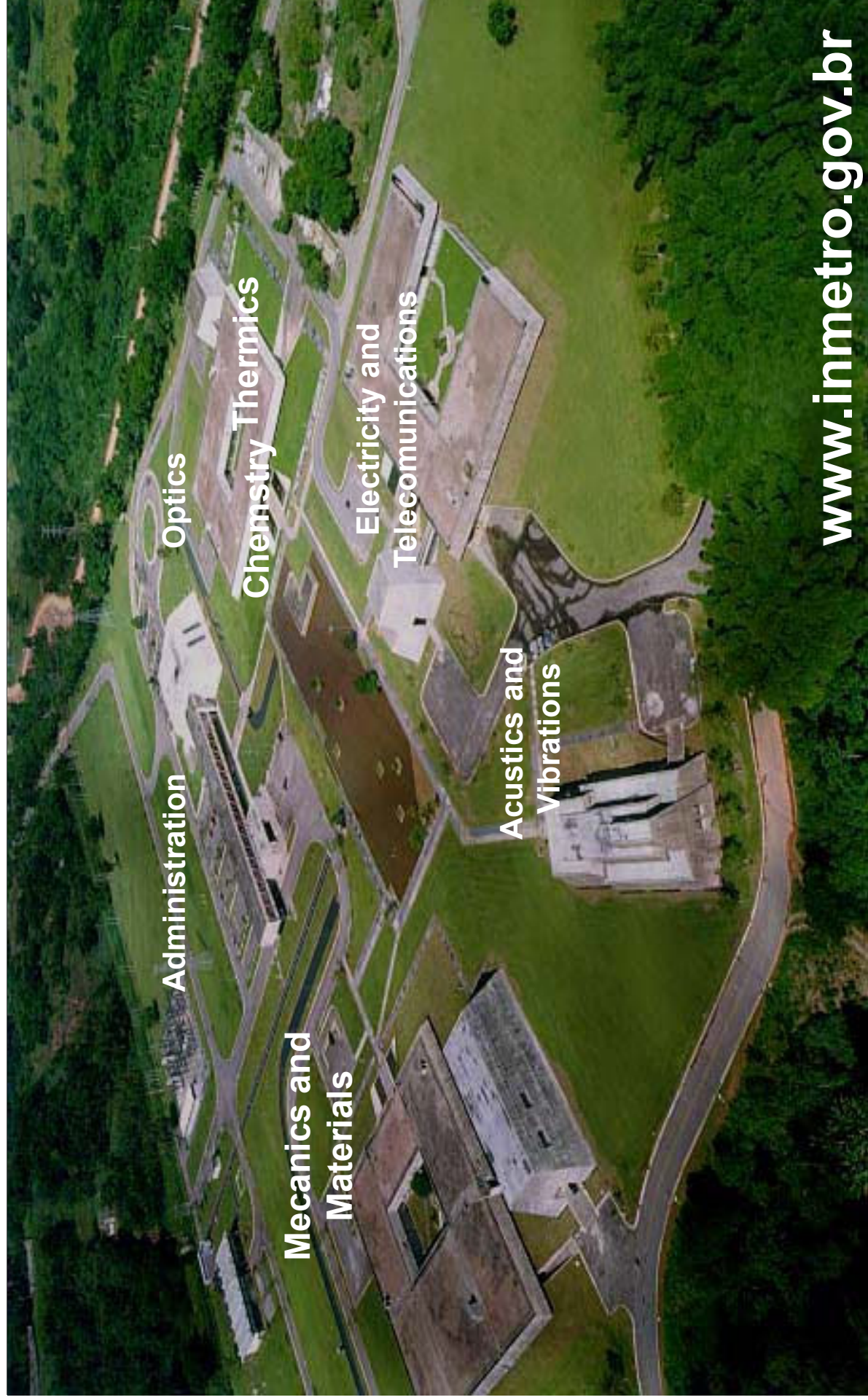
- Materials Division – started 2006/2007

Campus, Xerém - RJ



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government



www.inmetro.gov.br

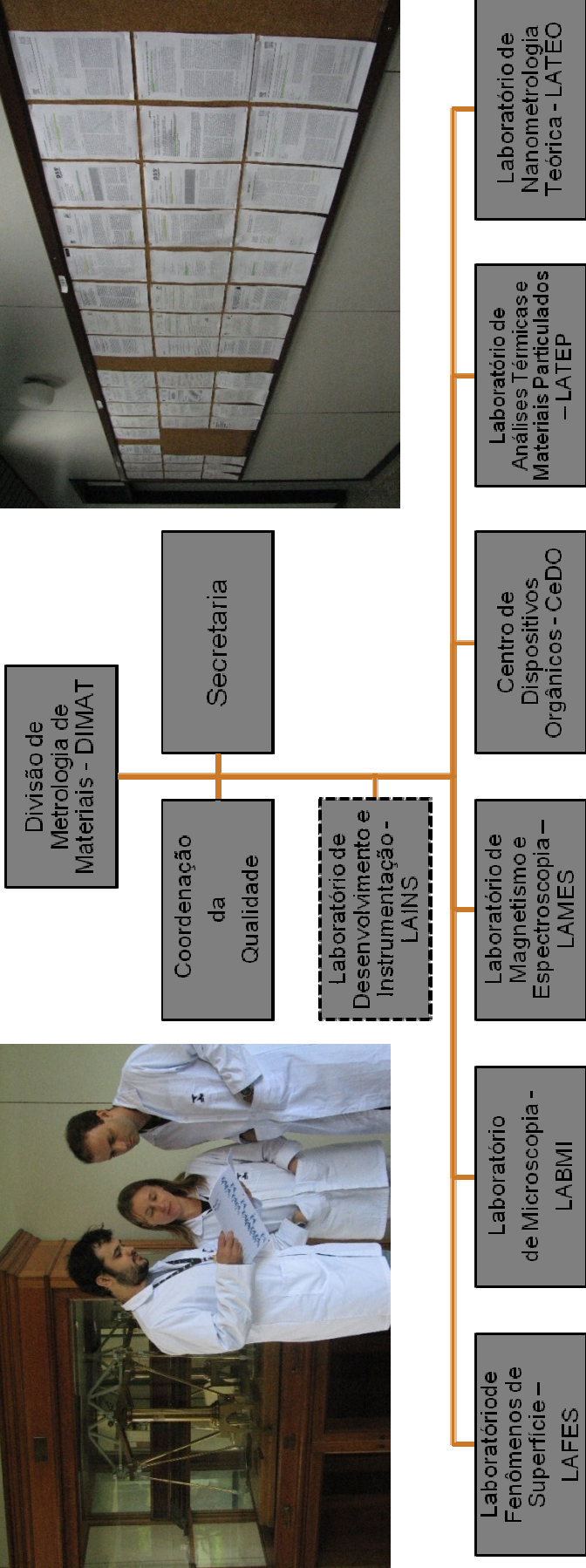
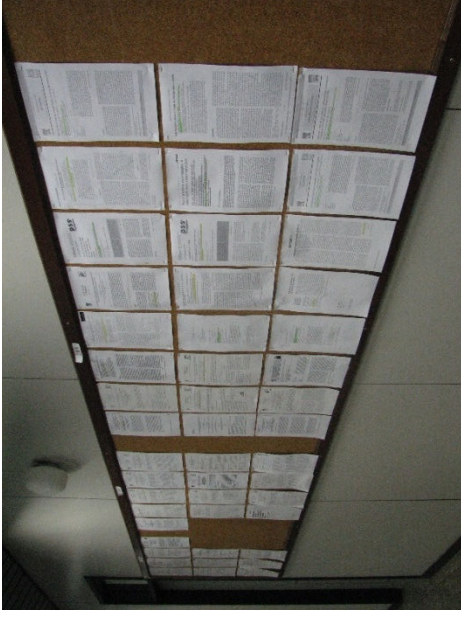


Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

DIMAT

Materials Metrology Division



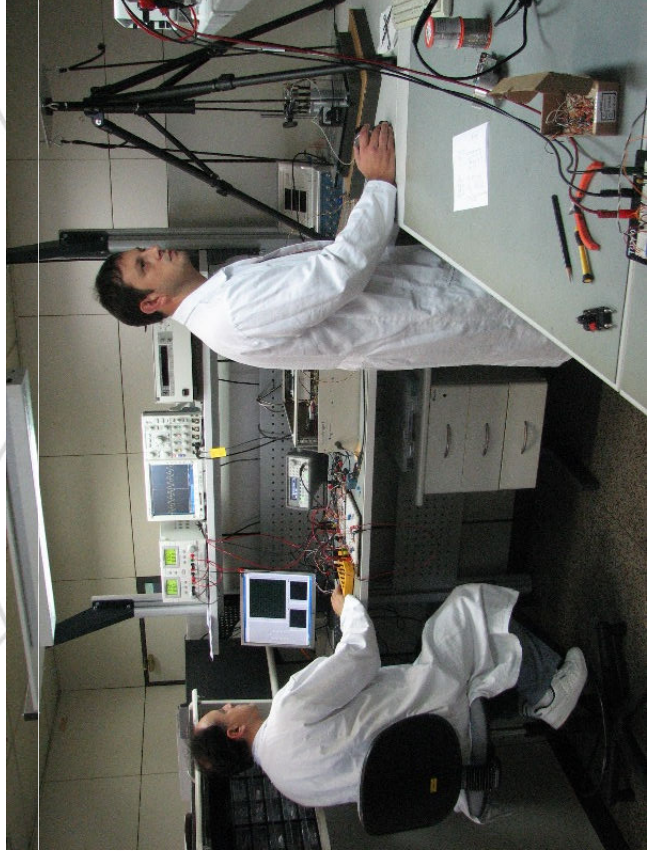


Ministry of
Development, Industry
and Foreign Trade

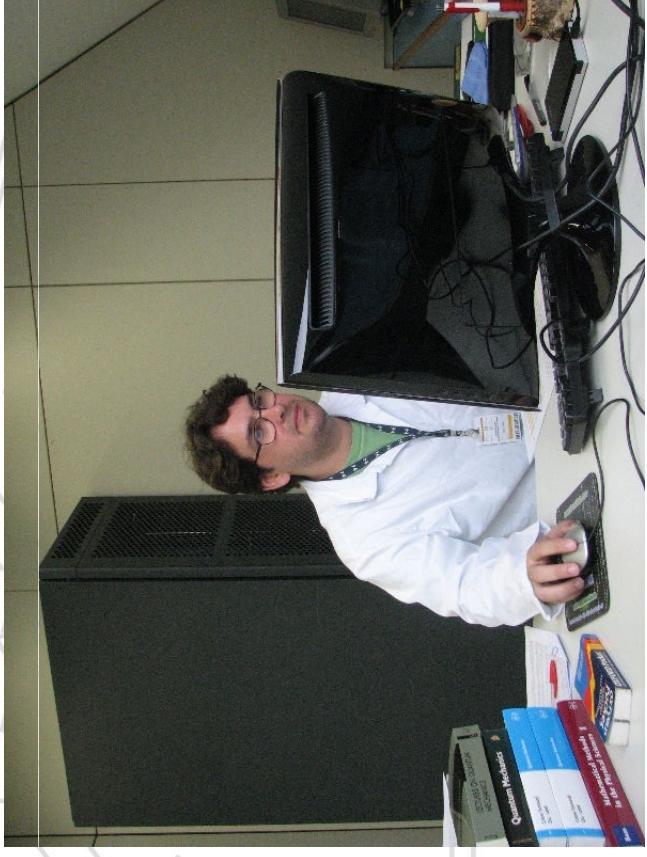
Brazilian Government

Instrumentation and Theory for Nano-metrology

Instrumentation for electronics and computation



Computation cluster



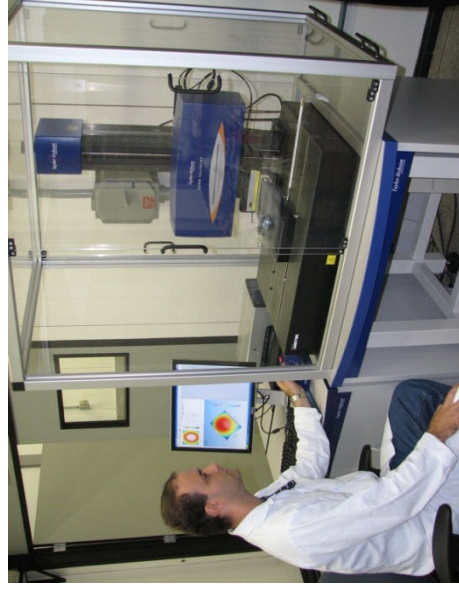
Surface phenomena



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

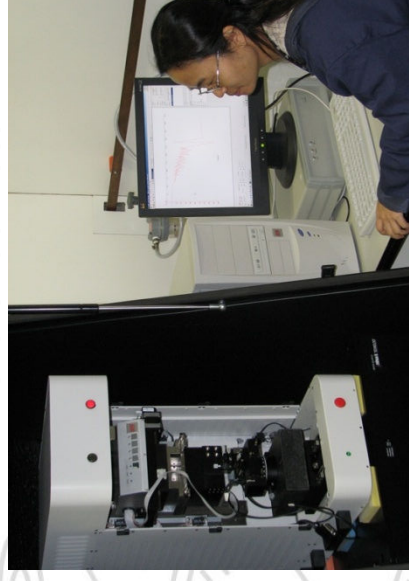
Tribometer



Friction simulator



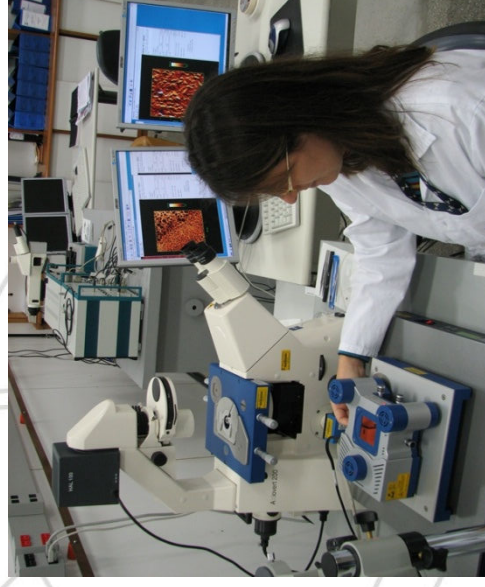
Micro-tribometer



Ultra-high vacuum STM



AFM



XPS + AES + ISS + LEED



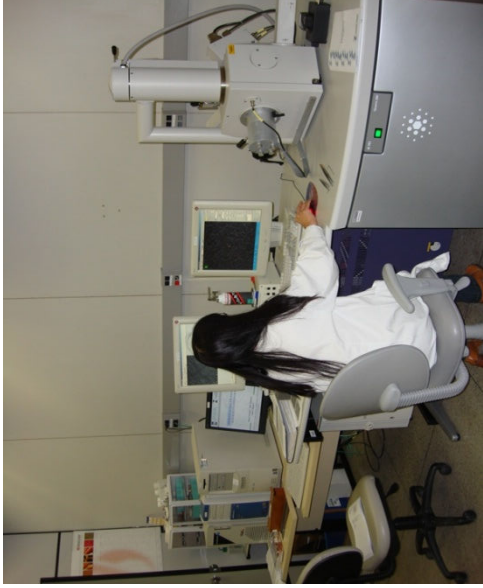
Microscopy



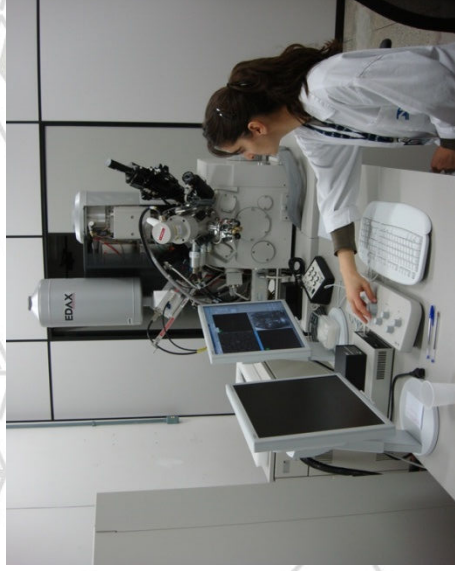
Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

SEM + EDS + EBSD



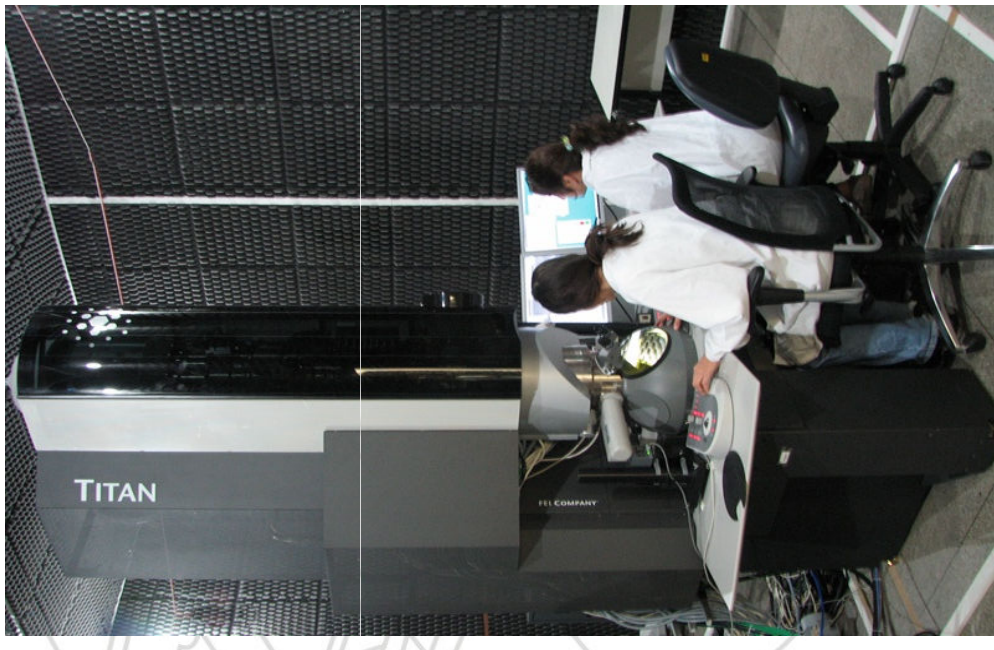
Dual beam – FIB+MEV



TEM



TEM with Cs correction



Organic Devices



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

Produção e caracterização de dispositivos orgânicos, metais e TCOs



Spectroscopy and Diffraction



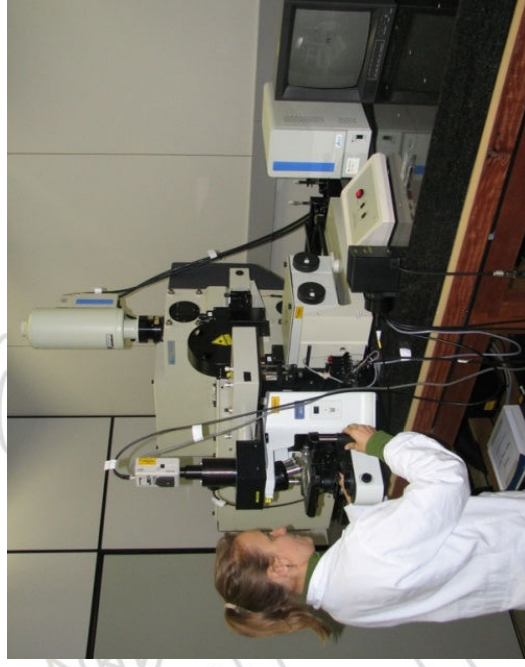
Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

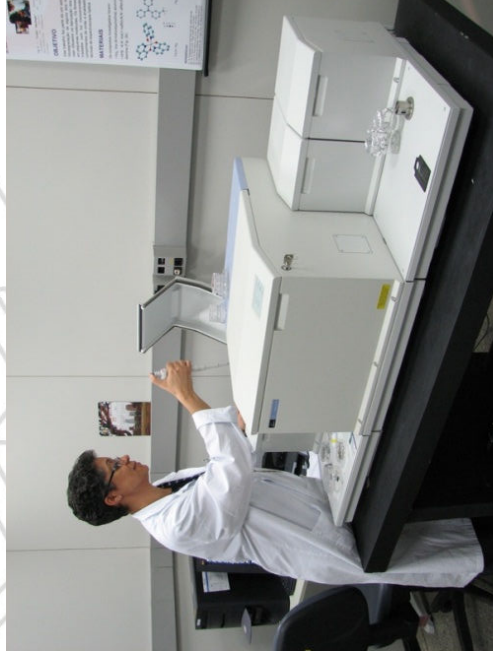
X-ray diffraction



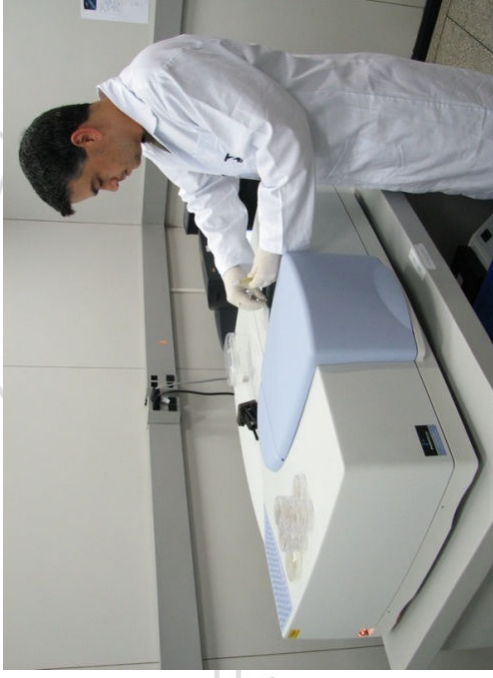
Micro-Raman spectroscopy



IR Espectrosc. ($30-15800\text{cm}^{-1}$)



UV-VIS Espectrosc. (175-3300nm)

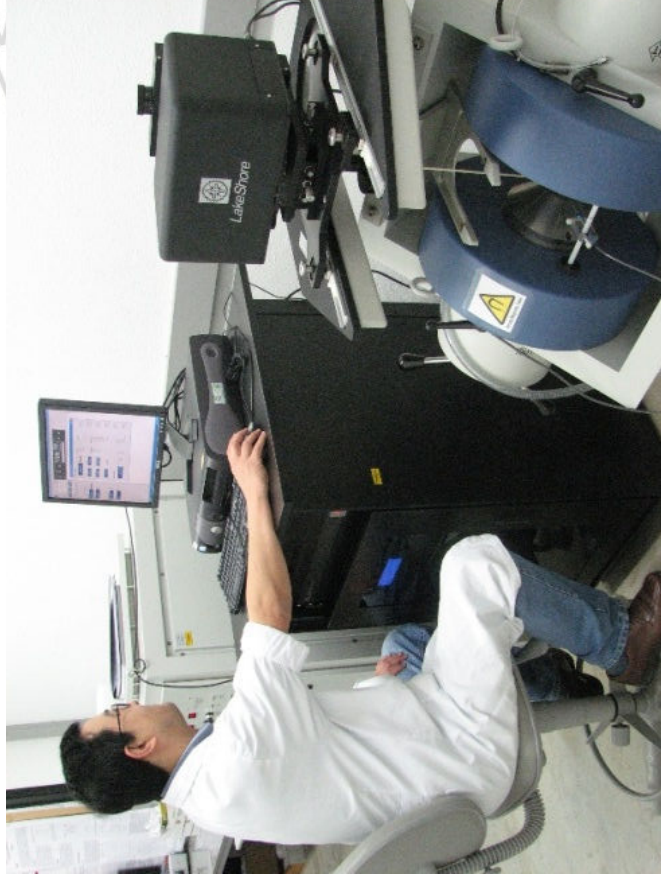




Magnetism

Ministry of
Development, Industry
and Foreign Trade
Brazilian Government

Magnetometer



Histeresimeter



Thermal analysis



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government



Acoustosizer



Autosorb -
BET



Microonda



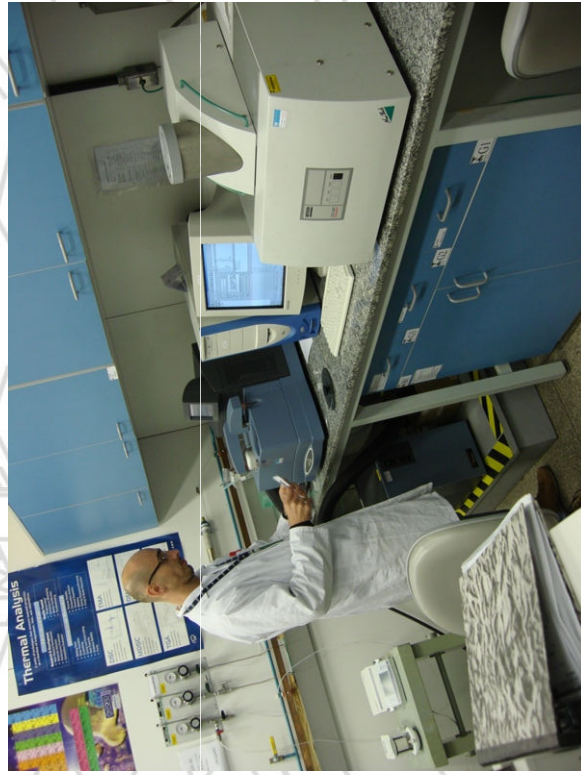
DSC



P-DSC



DSC-PCA



HFM



GHP



NanoFlash



DMA



MS



SDTA



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

Metrology for today

Guarantee for quality and consumer protection

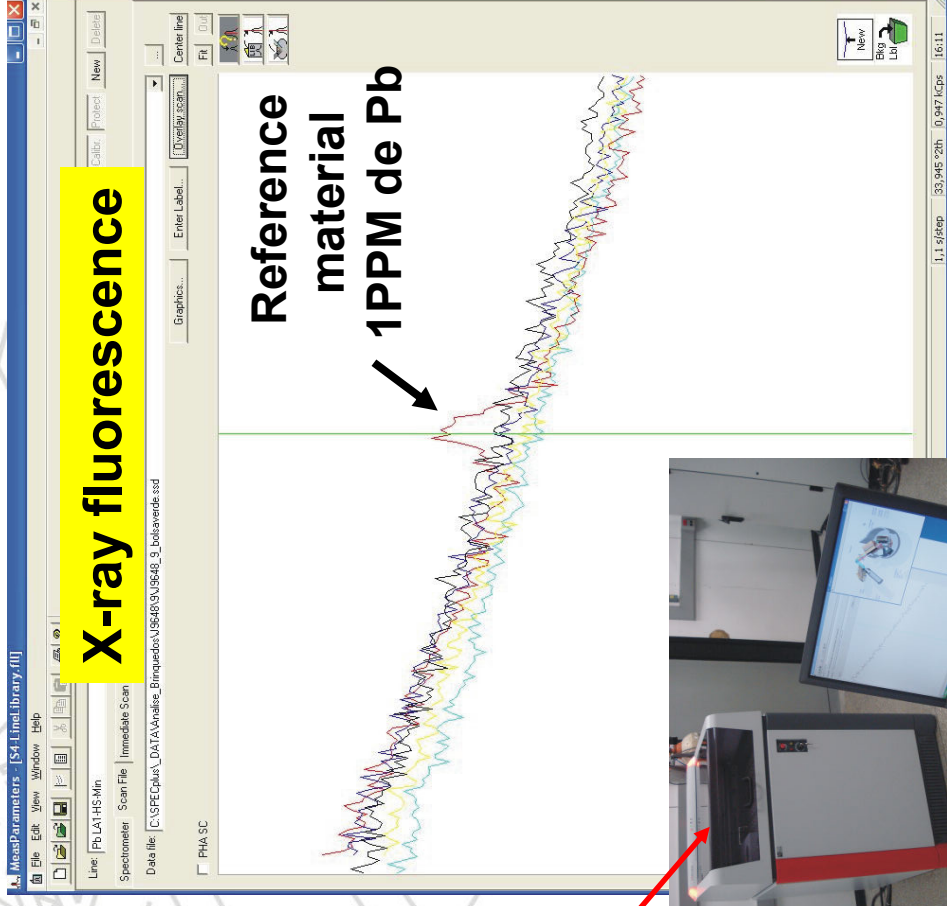
- Emergencies
- National products
- International products



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

Accurate chemical analysis

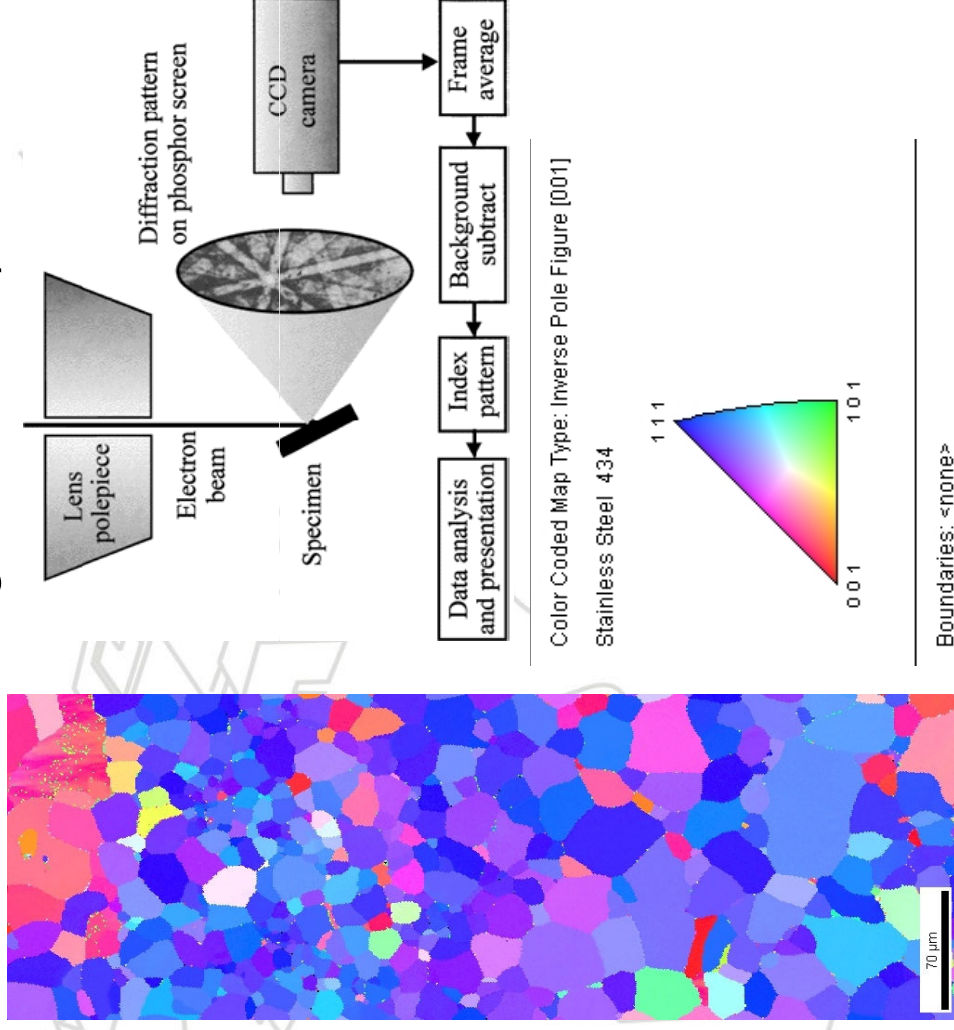


Metallic texture by EBSD

(“electron backscattering diffraction”)

Interlaboratorial

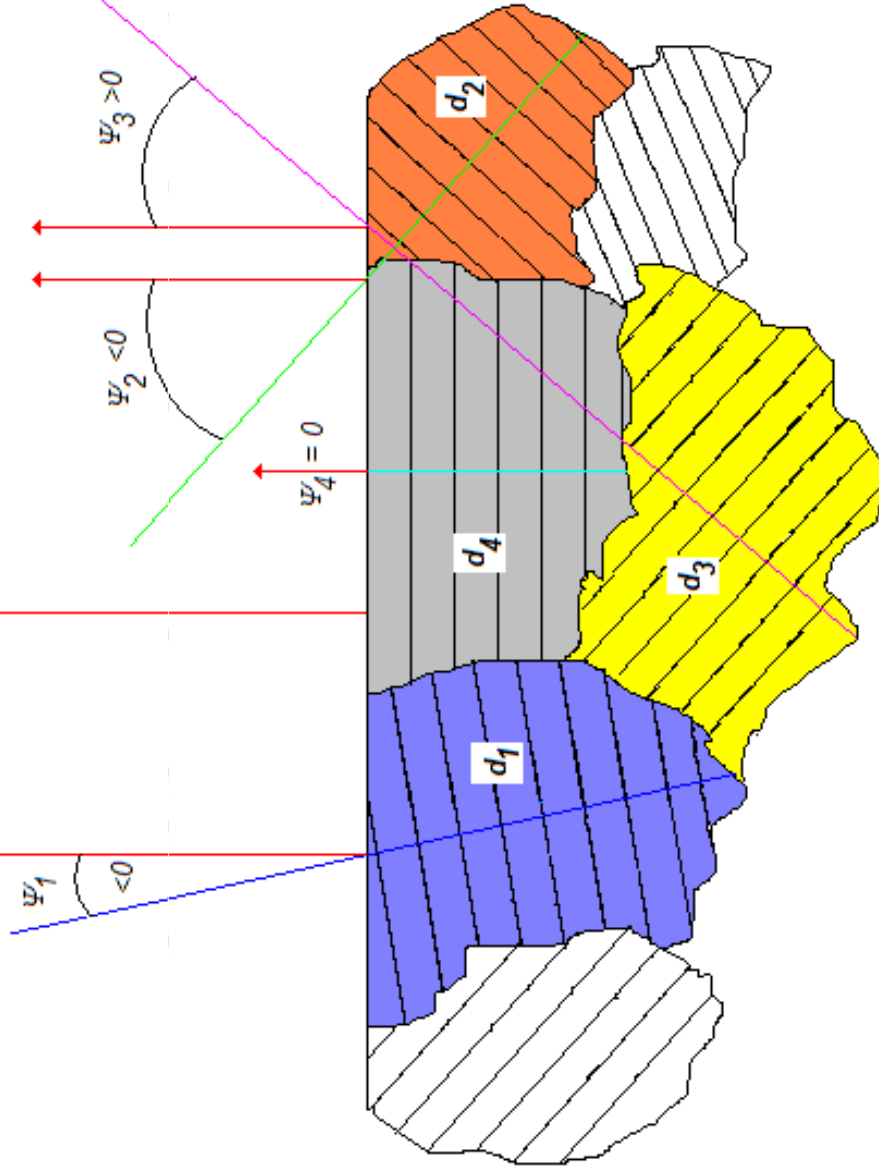
1. Acesita - (TSL)
2. UFSCAR, DEMA - (TSL)
3. USP Politécnica, São Paulo - (TSL)
4. IME - (JEOL / TSL)
5. UFC - (Oxford)
6. UFSCAR, Quimica - (Oxford)
7. Usiminas- (Oxford / HKL)
8. IPT - (JEOL/ HKL)
9. LNLS (JEOL/ HKL)
10. UFOP - (JEOL/HKL)
11. UFRGS - (JEOL/HKL)



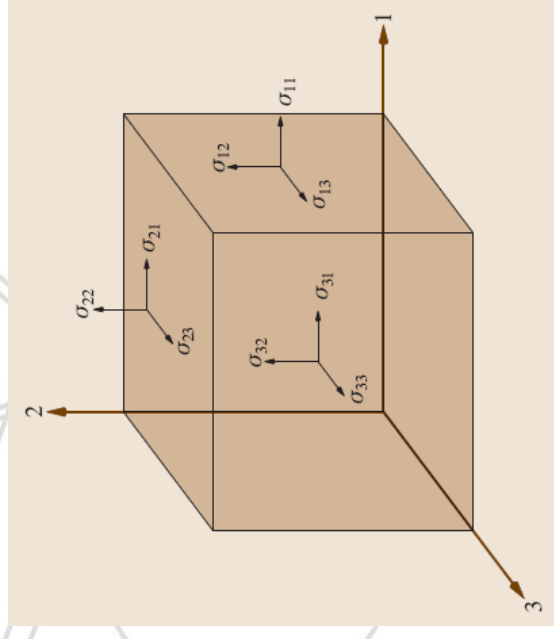
$$\varepsilon_{\psi\varphi}^{hkl} = \varepsilon_{00}^{hkl} + \frac{1}{2} S_2^{hkl} \sigma_{\varphi} \sin^2 \psi + \frac{1}{2} S_2^{hkl} \tau_{\varphi} \sin(2\psi)$$

Surface Normal

$$\psi_2 < \psi_1 < \psi_4 < \psi_3$$



Tensor elástico

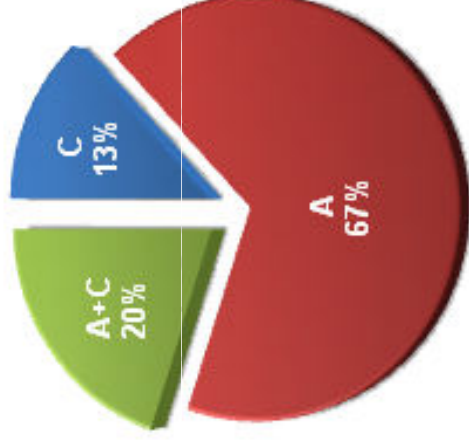
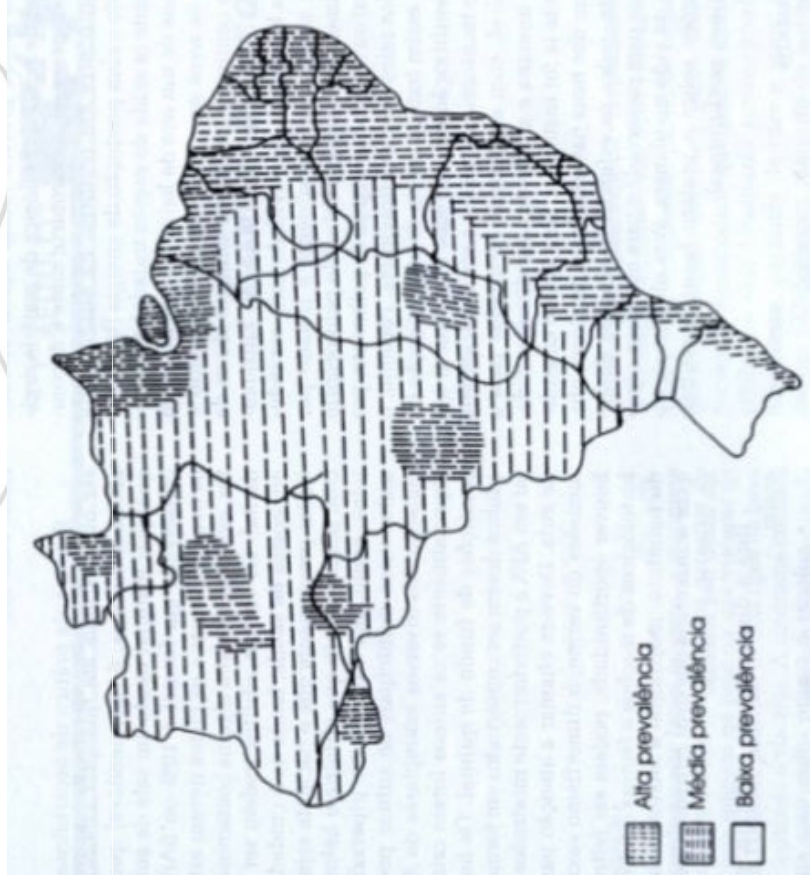


Polimorfism in drugs



Ex: Membendazol – forms A, B e C

Ascariidase in Brazil
(Ministério da Saúde)



Other examples:

Mefenamic acid (ponstan)

Carbamazepine (anti-convulsante)

Tibolone (fem. hormone)

Gestodene (anti-contraceptivo)



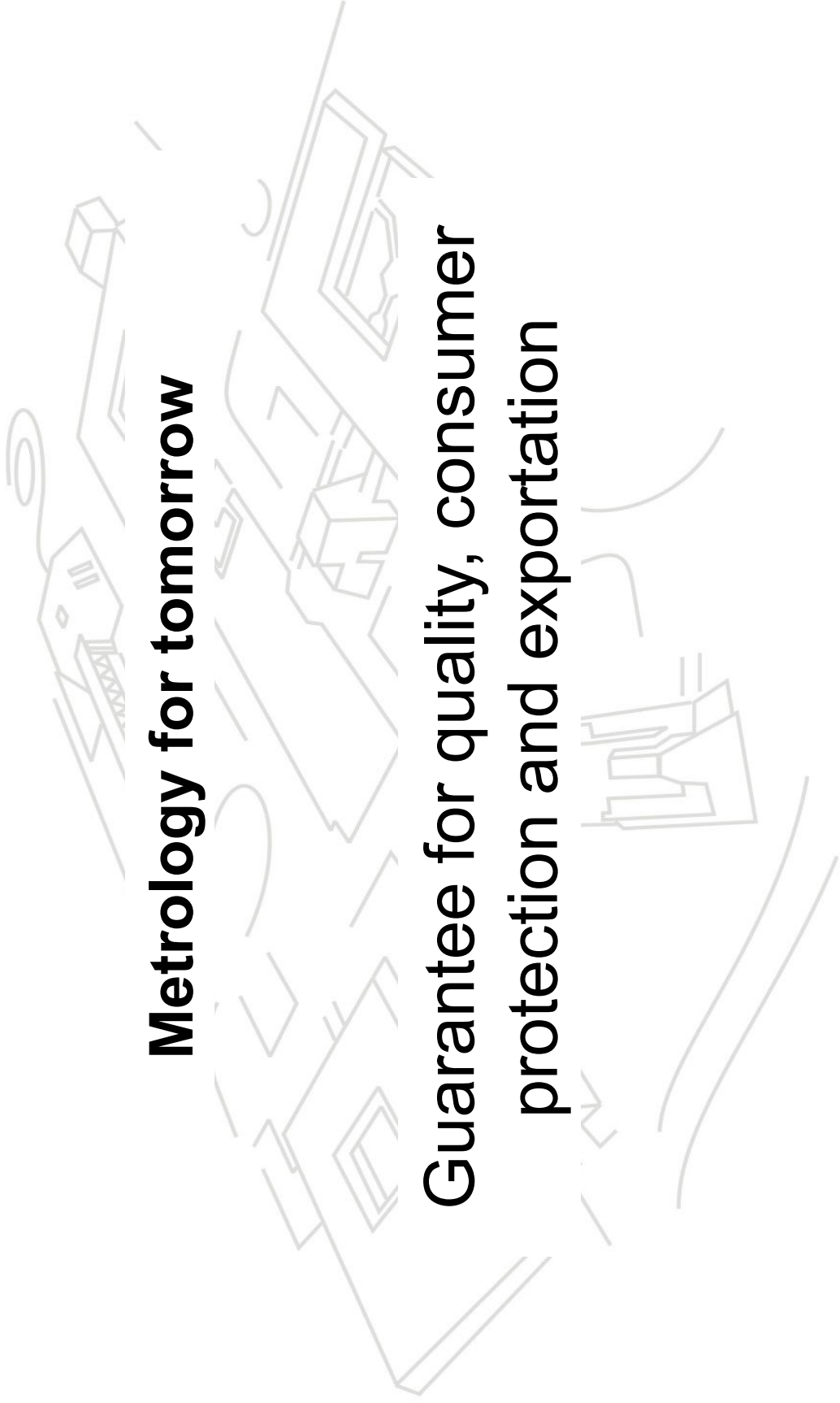
Ministry of
Development, Industry
and Foreign Trade

Brazilian Government



Metrology for tomorrow

Guarantee for quality, consumer protection and exportation



BIOFUEL



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government



Girassol



Soja



Soja

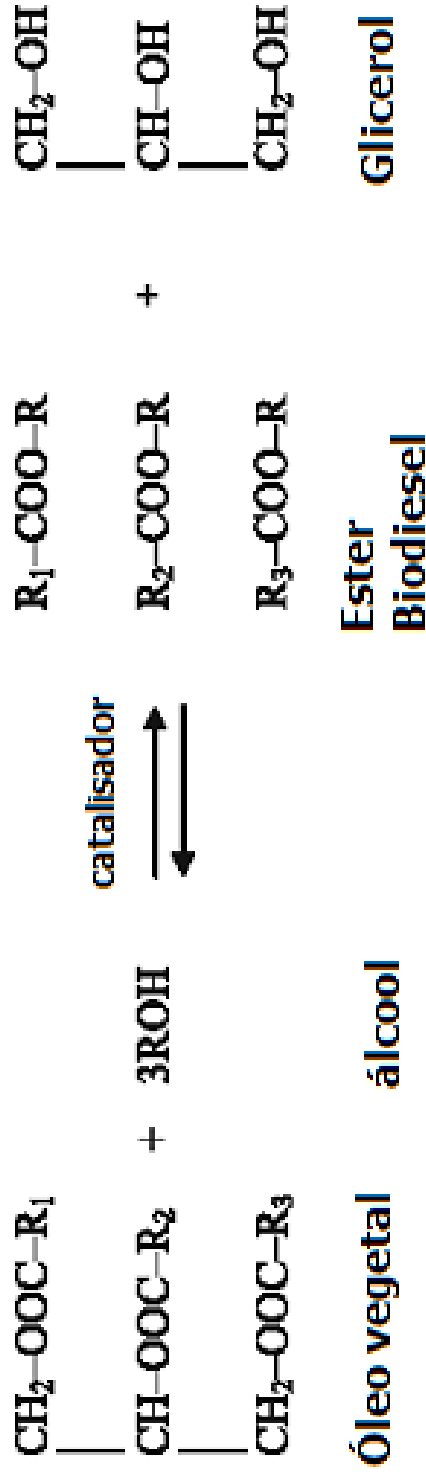


Canola



Canola

Transesterification



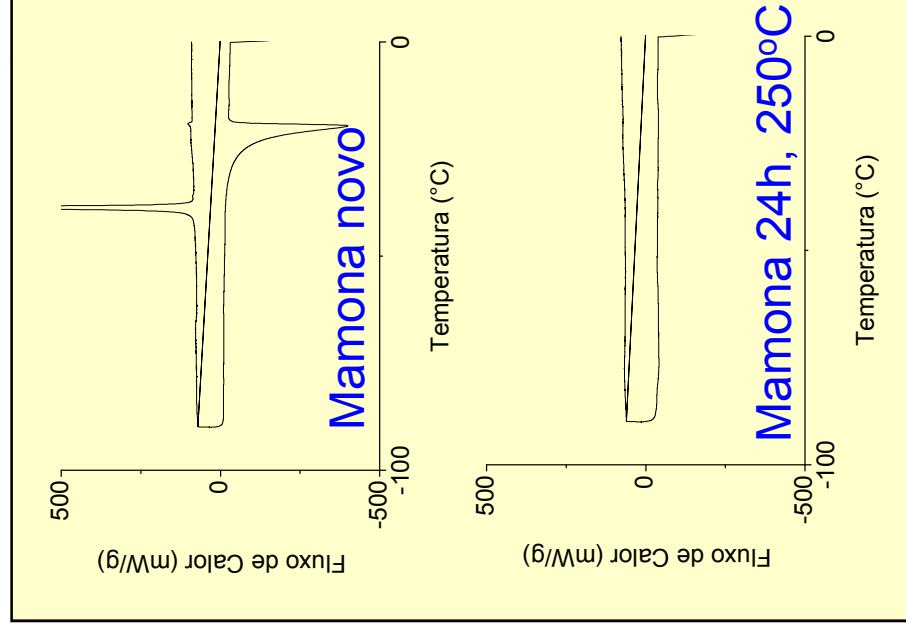
Development of Certified Reference Material



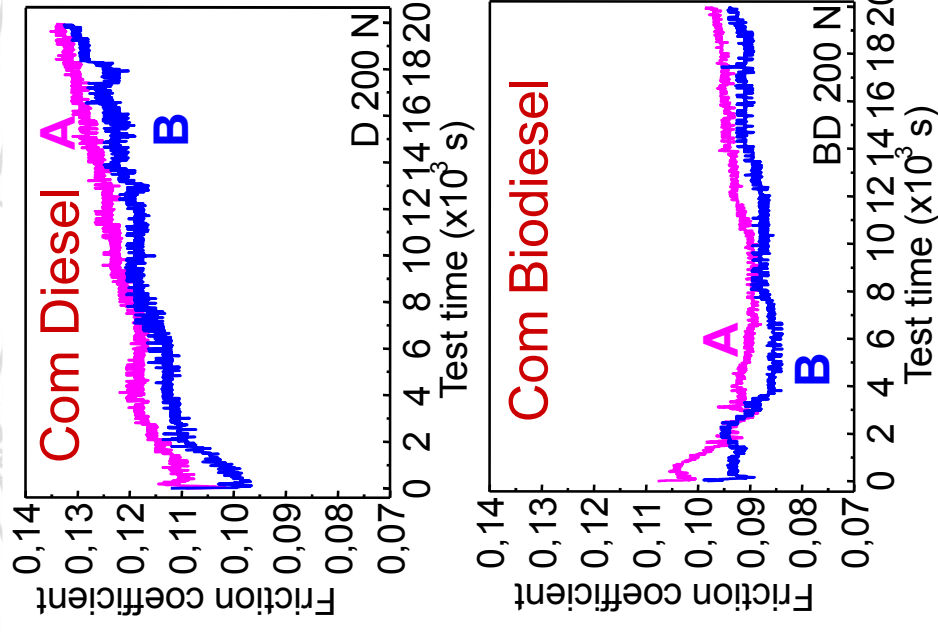
Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

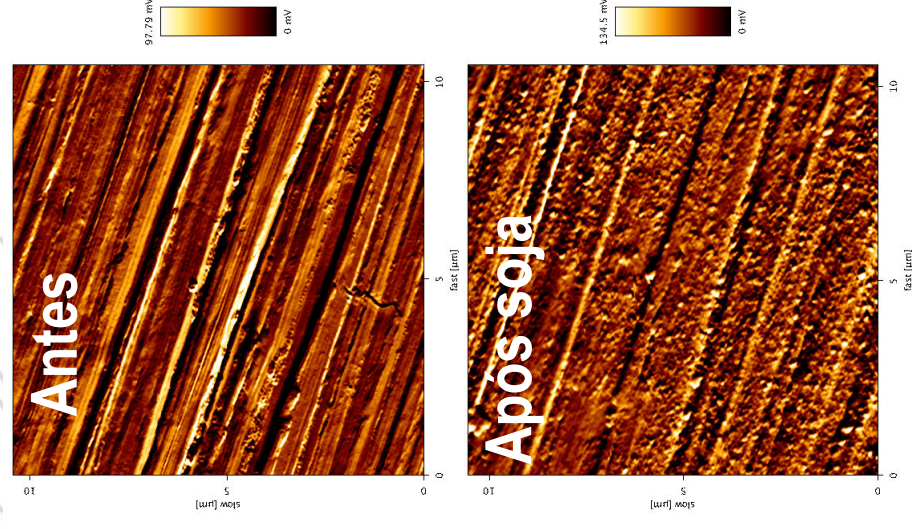
Thermal stability DSC And purity



Lubricity



Interaction with carbon steel



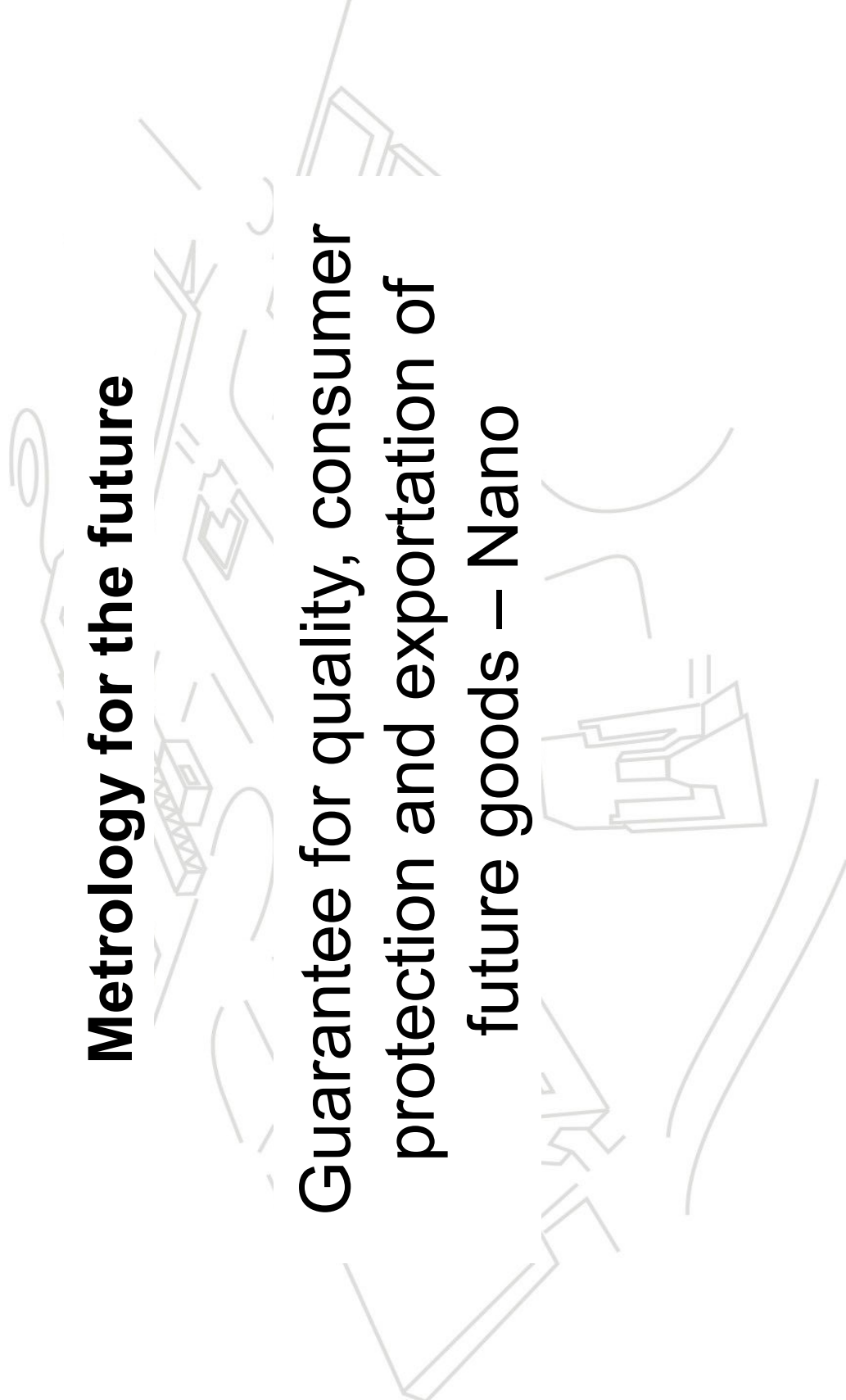


Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

Metrology for the future

**Guarantee for quality, consumer
protection and exportation of
future goods – Nano**

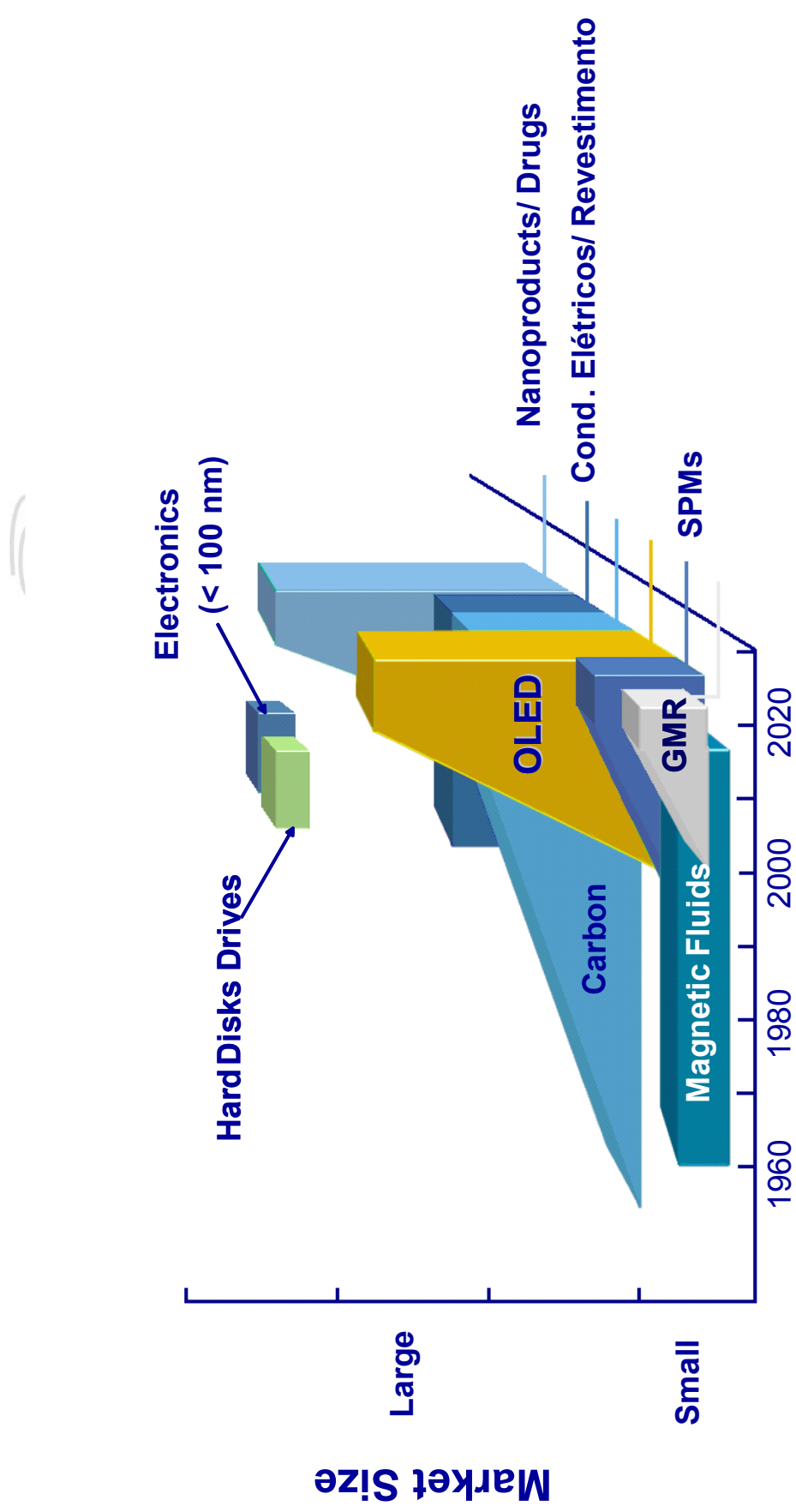


International market



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government



From Nanotechnology World, 2005



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

NANOMETROLOGY

- **Nanoparticles**
- Nanocatalysis
- Organic semiconductors
- Carbon

Nanofluids: nano-particles suspension in liquid medium

- Lubricants
- Covering
- Pollution cleaning
- Drug delivery
- Functional tissue-cell interaction
- Pigments and coloring
- **Heat Transfer**

Stable
physically

Stable
chemically

Small size particles
100 nm

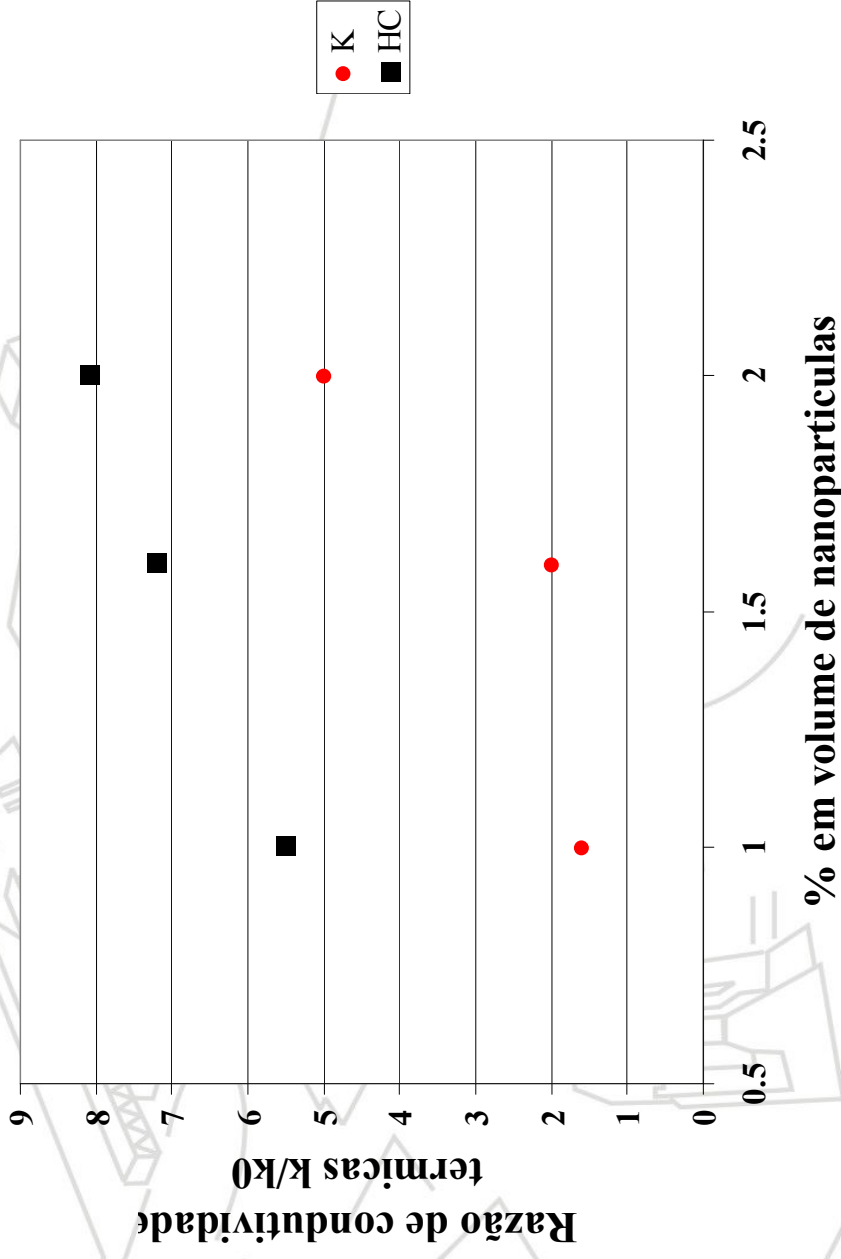
ZnO, γ -Al₂O₃, CuO (Petrobrás)



Increase in Heat conductivity

Reported values:

- 4 % vol Al₂O₃ → 8 % k
- 4 % vol CuO → 12 % k
- 10⁻⁴% vol Thiol-Au → 10 % k
- 0,5 % vol Fe → 18 % k
- 4 % vol Cu → 20 % k
- 0,1 % vol SWCN → 150 % k



Nano-particles size



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

Scherrer Equation (1918)

$$B = \frac{K\lambda}{L \cdot \cos \Theta}$$

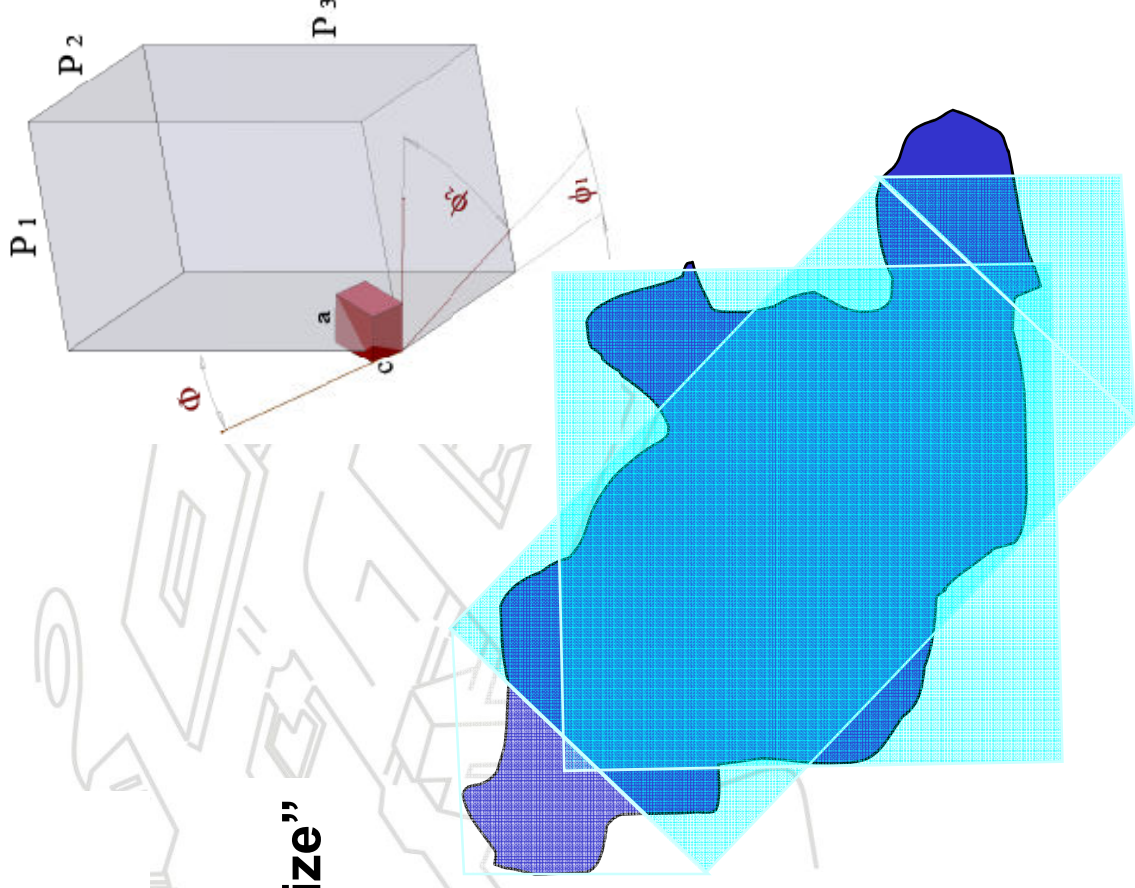
B – FWHM

L – crystallite “size”

“apparent” size

Approximations:

1. Cubic with same dimensions
2. K arbitrary



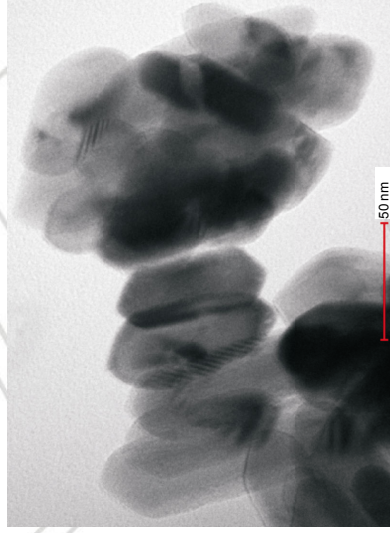
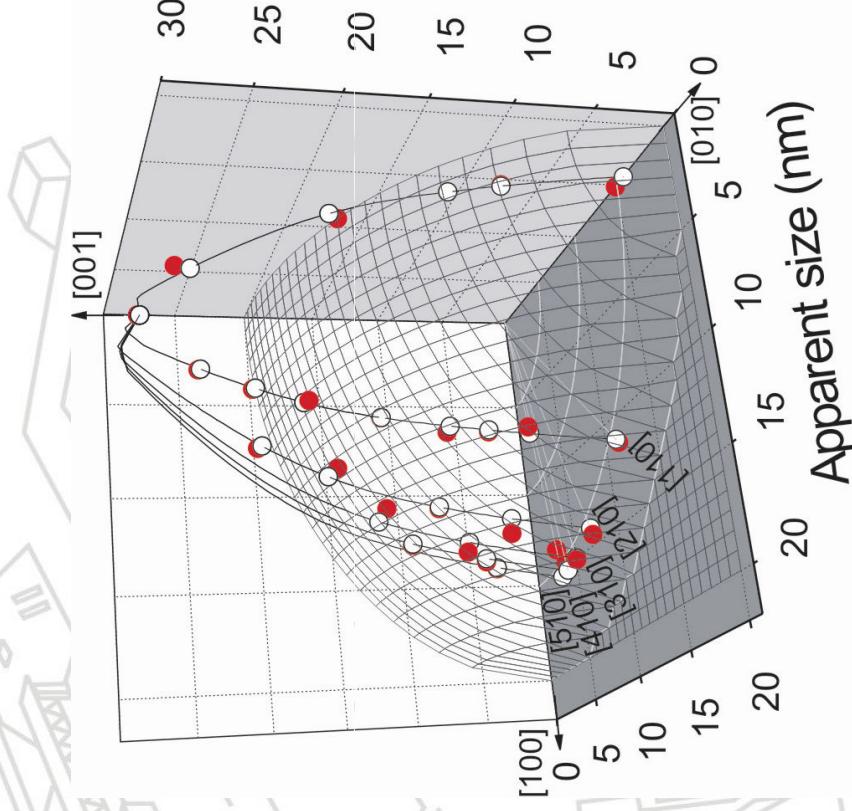
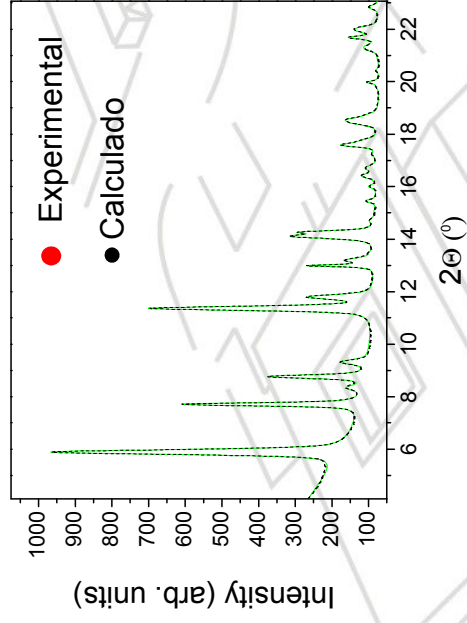
Modeling shape of Nano-rutile (TiO₂)



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

Dedicated software based on several $(h k l)$ and $\beta = (\text{area}/\text{height})$



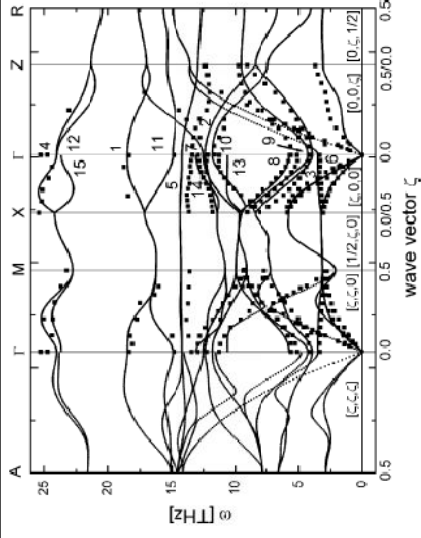
Nano-materials calibrated by TEM

NanoRutile-TiO₂ size from Raman spectroscopy



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

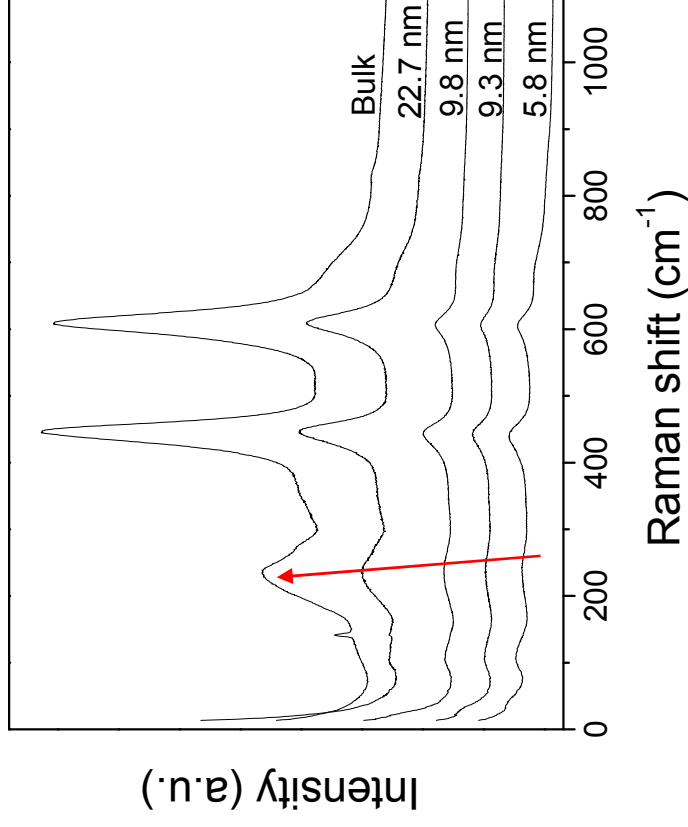
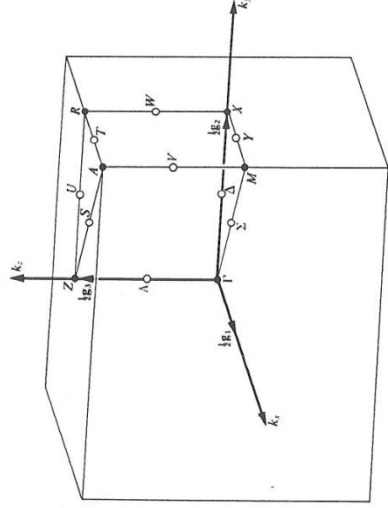


Frequency and width depends
on particle size

Uncertainty principle

$$\Delta p_x \cdot \Delta x \sim \hbar \Rightarrow \Delta q \sim \frac{1}{\Delta x}$$

$$I(\omega) \propto \iiint_{BZ} |C(\vec{q})|^2 d^3q \frac{1}{[\omega - \omega(q)]^2 + (\Gamma/2)^2};$$





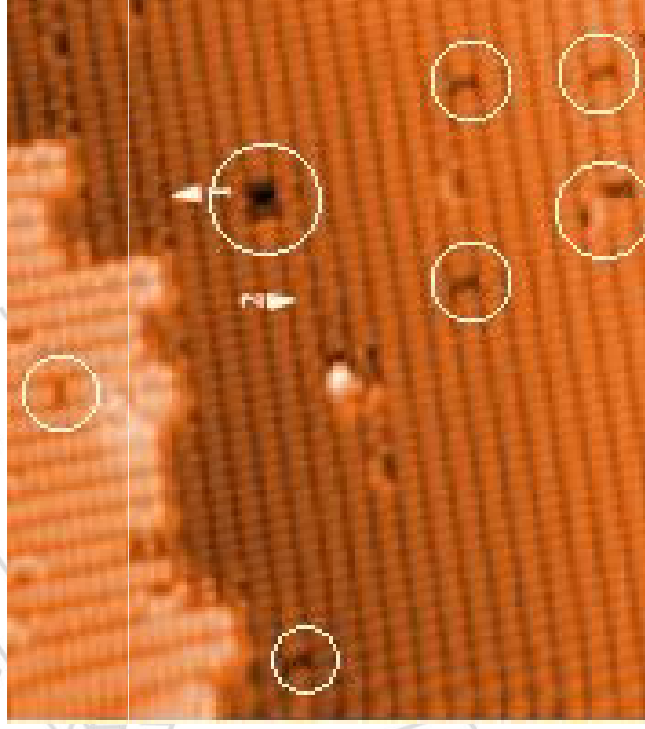
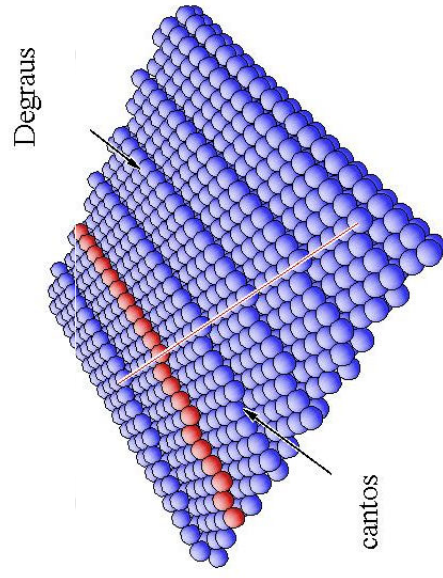
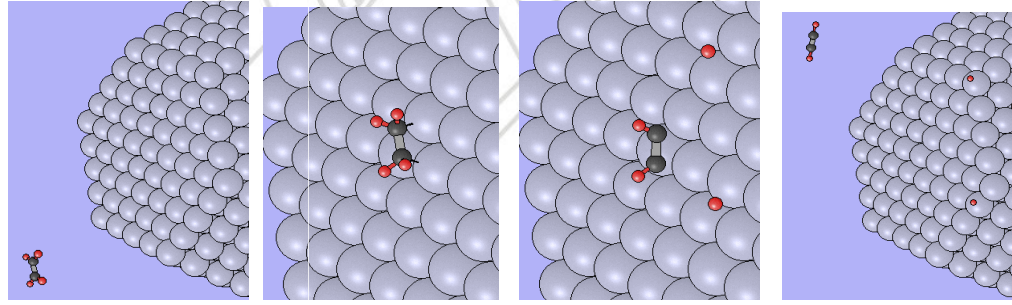
Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

NANOMETROLOGY

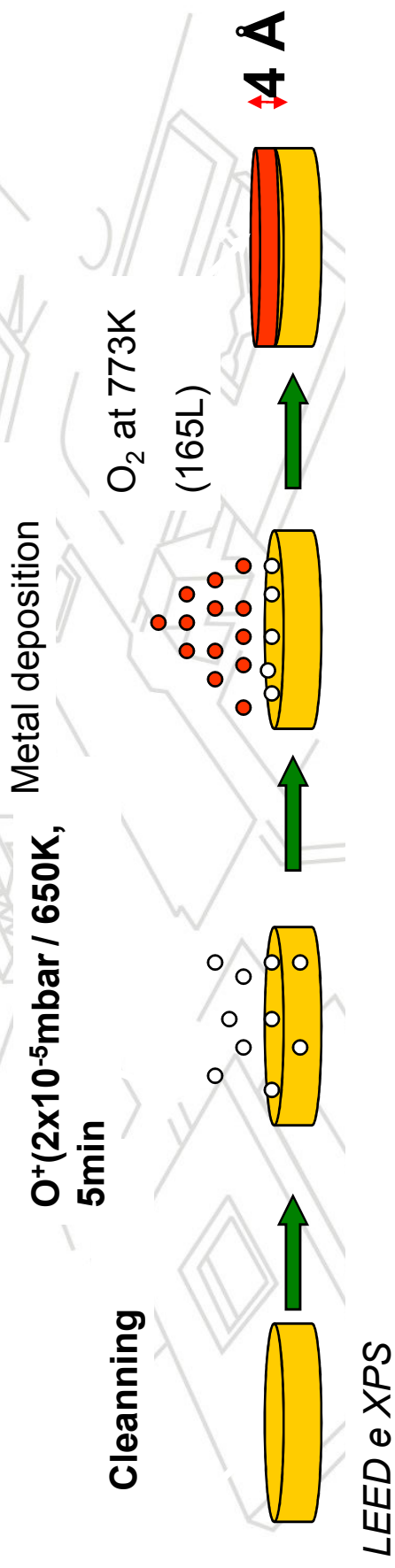
- Nanoparticles
- **Nanocatalysis**
- Organic semiconductors
- Carbon (see 11AM presentation)

Interface between surface science and catalysis



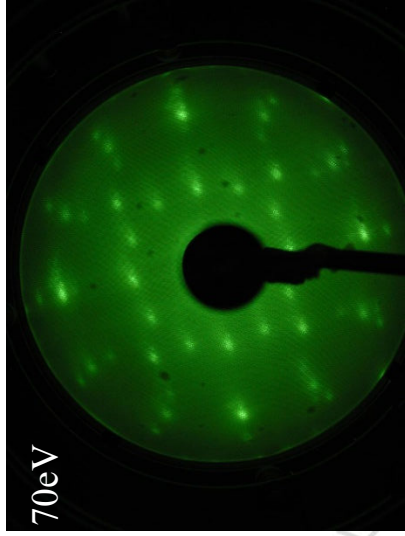
Building the thin film oxide...

MoOx/Cu₃Au (100)

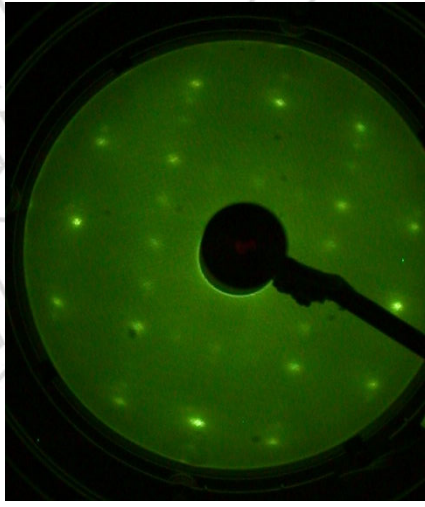


Two films can be made

LEED: simetry: retangular + hexagonal

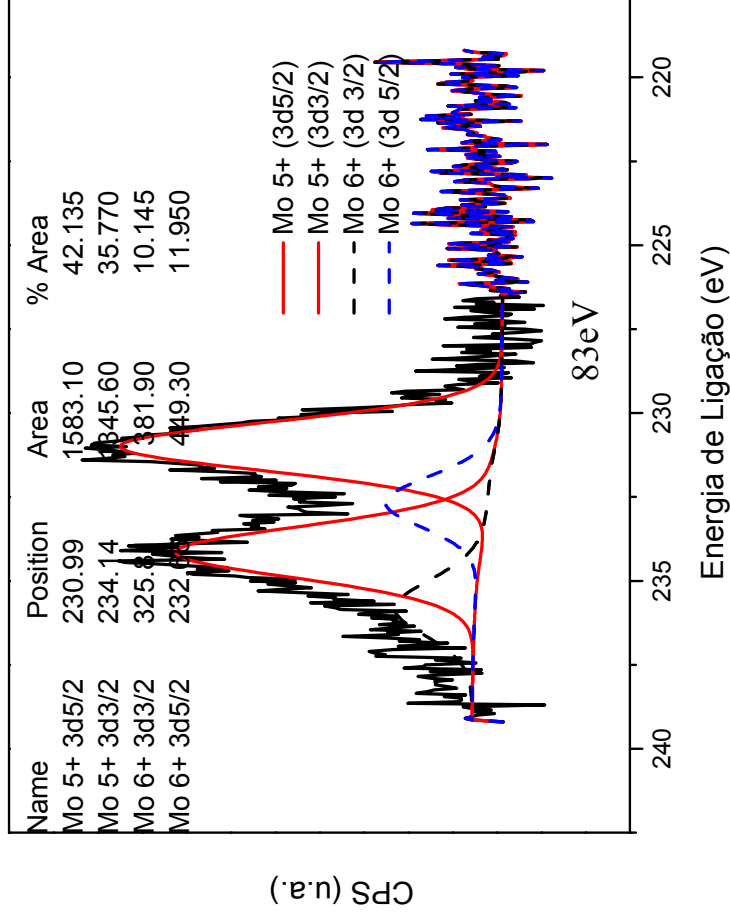


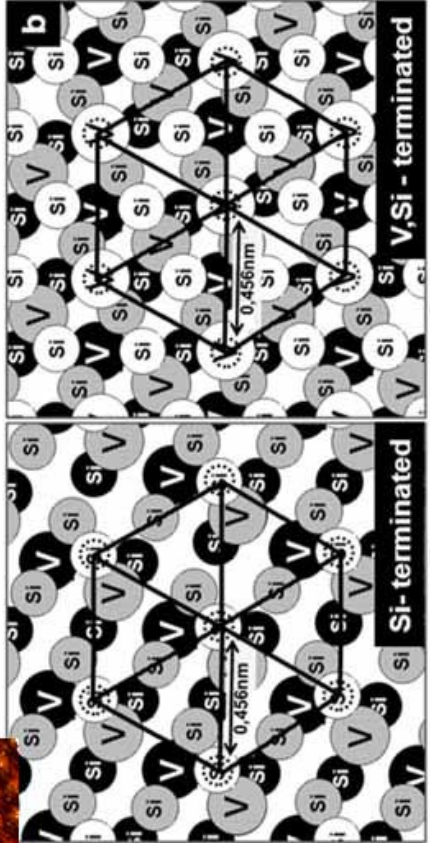
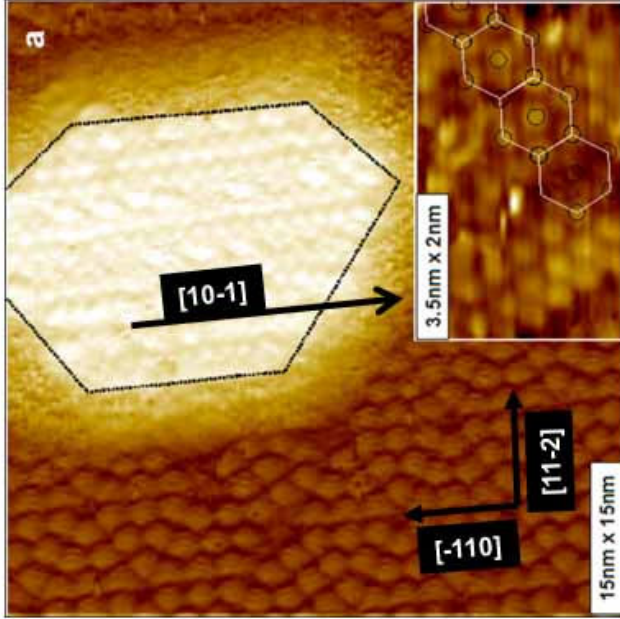
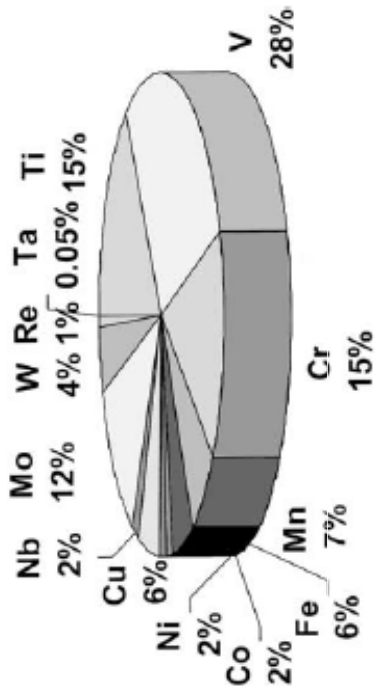
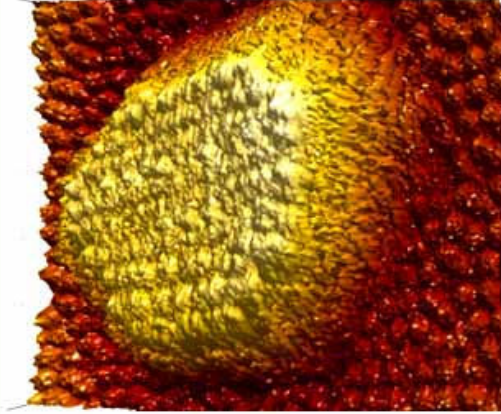
(Mixture Mo^0 , Mo^{4+} , Mo^{5+} , Mo^{6+})



(predominance Mo^{5+})

XPS, Mo(V) identified

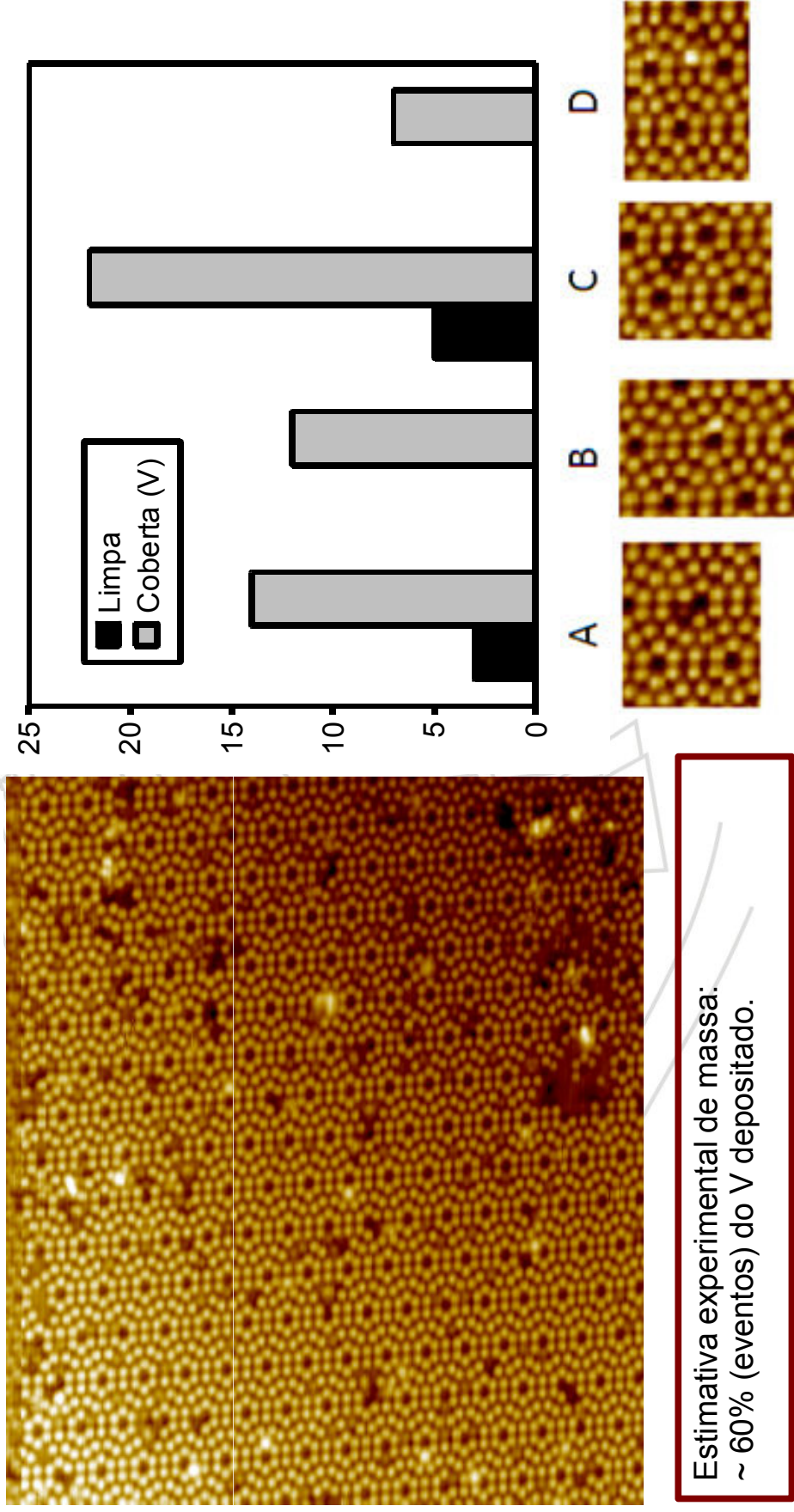




Vanadium in Silicon

STM - V = + 1.4 V

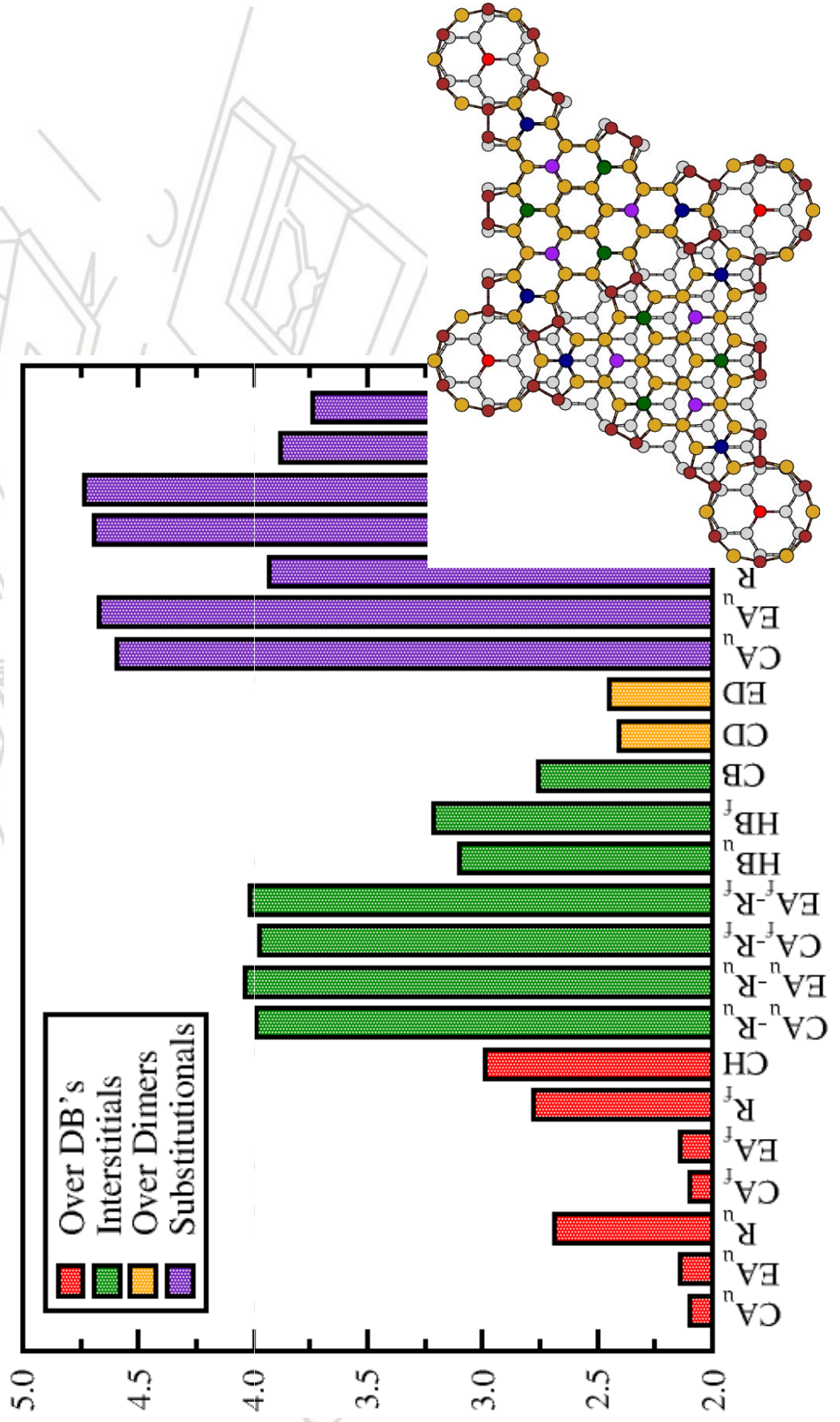
eventos STM (80%)
Baixa-cobert.: $0.012 \text{ MC} = 7.9 \times 10^{14} \text{ at/cm}^2$



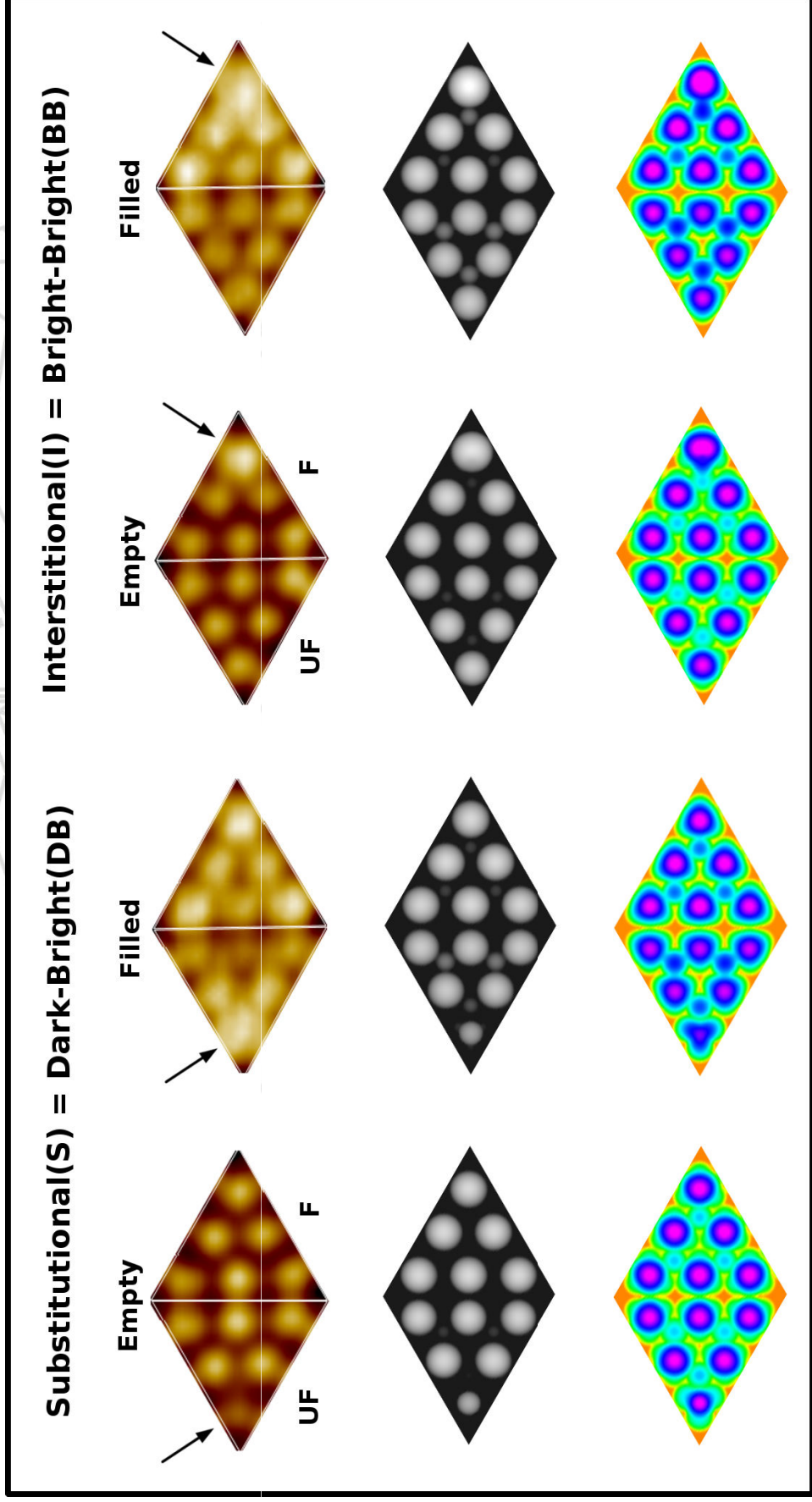
Estimativa experimental de massa:
~ 60% (eventos) do V depositado.



Energetics (eV):

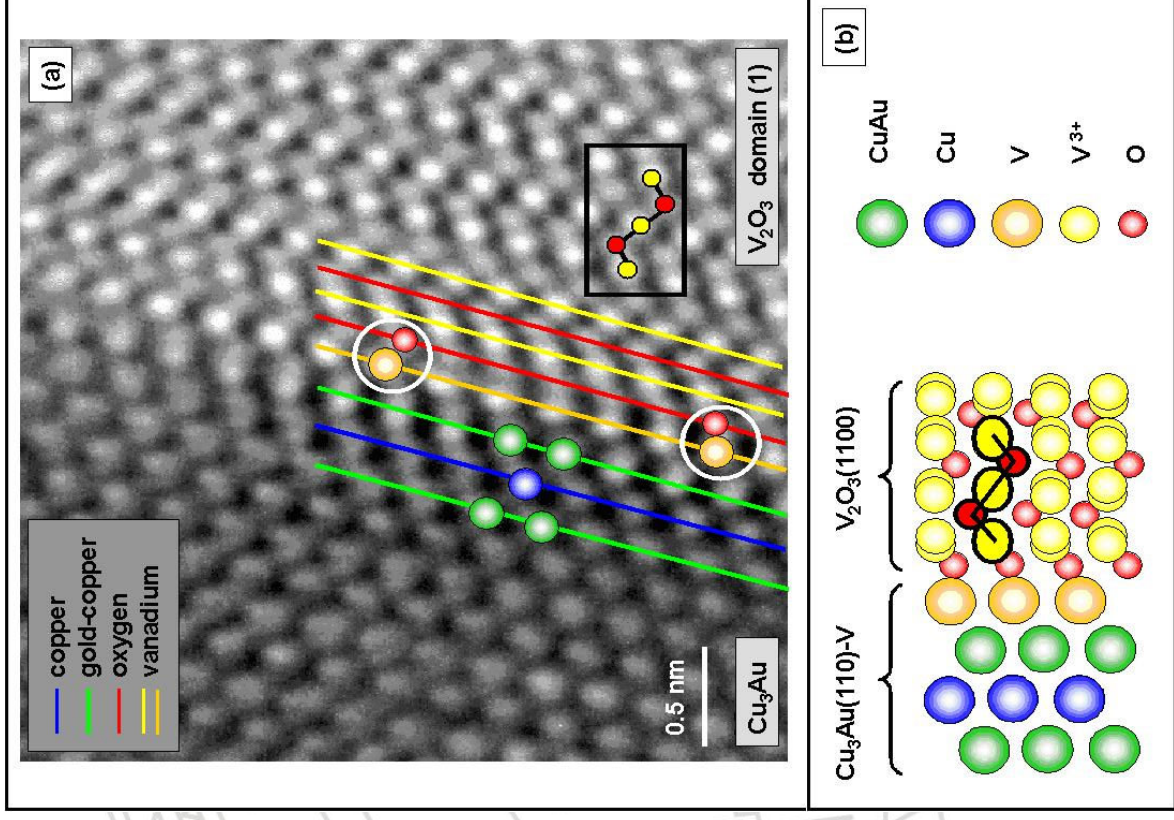


“Bias” polarity effect:



HR-TEM

Interface $V_xO_y - Cu_3Au$



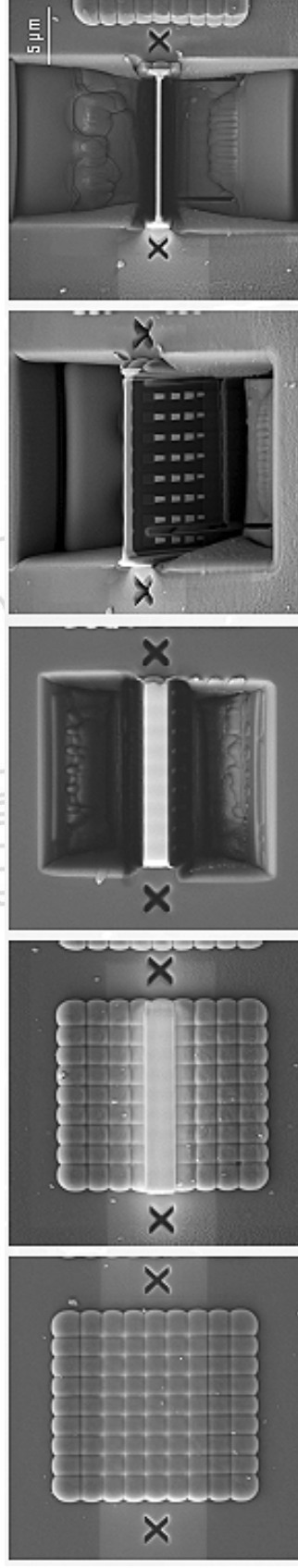
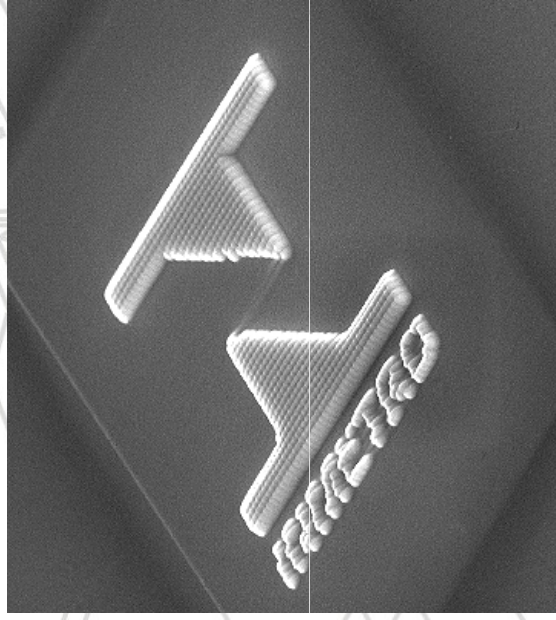
Nanofabrication and lithography



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

Dual beam – FIB+MEV





Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

NANOMETROLOGY

- Nanoparticles
- Nanocatalysis
- **Organic semiconductors**
- Carbon (see 11AM presentation)

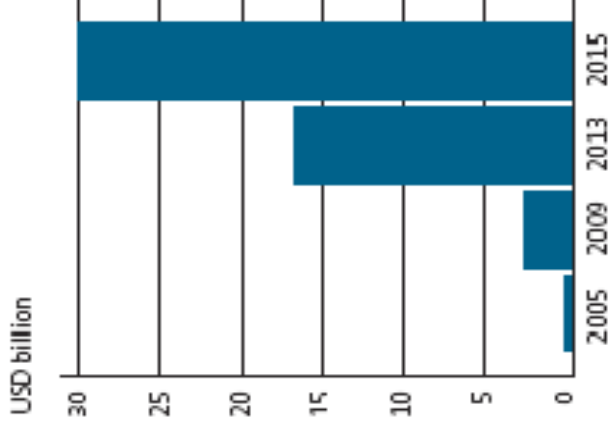
Market analysis



Ministry of
Development, Industry
and Foreign Trade

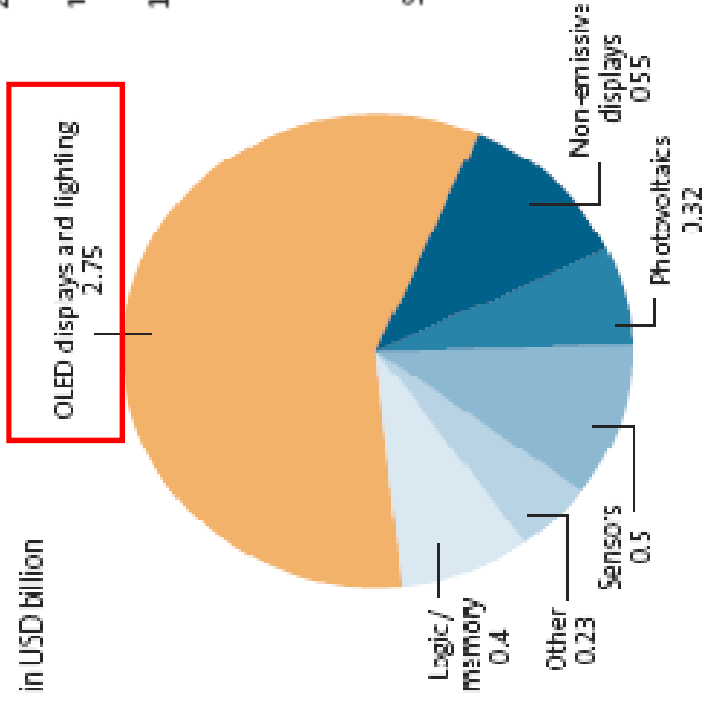
Brazilian Government

Global market of organic electronics 2005–2015



Source: IDTechEx 2006

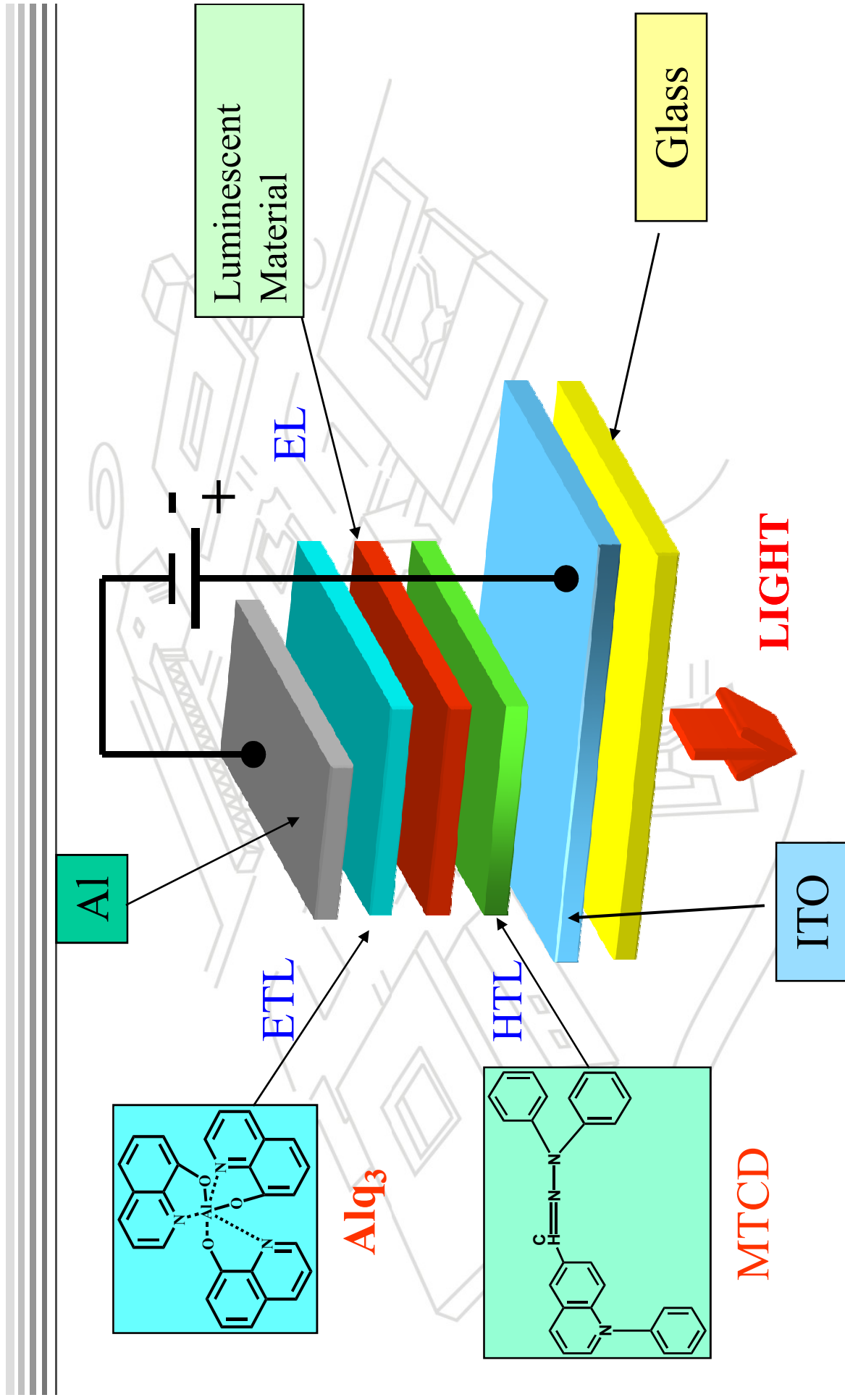
in USD billion



Source: IDTechEx 2006



OLED structure

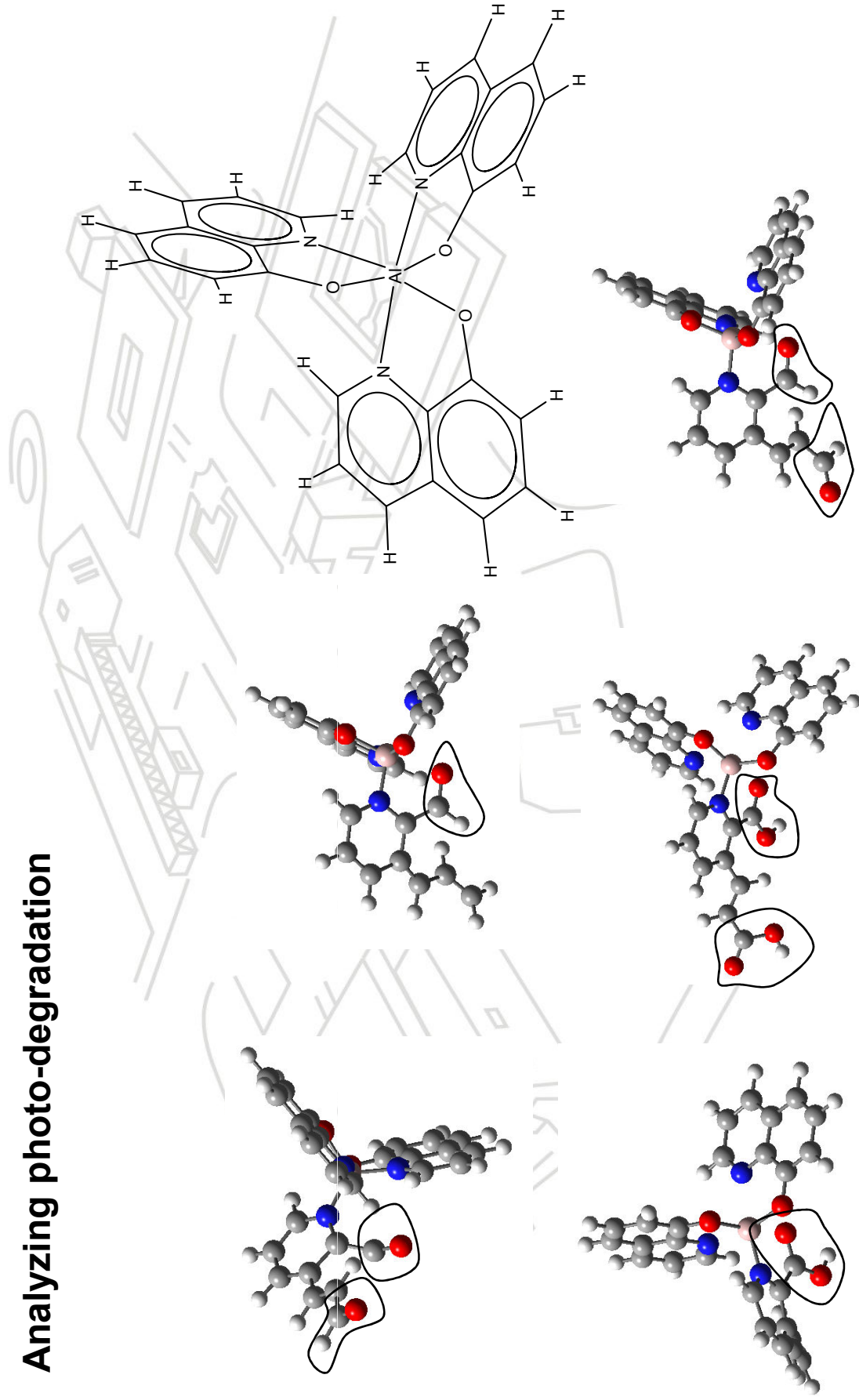




Ministry of
Development, Industry
and Foreign Trade

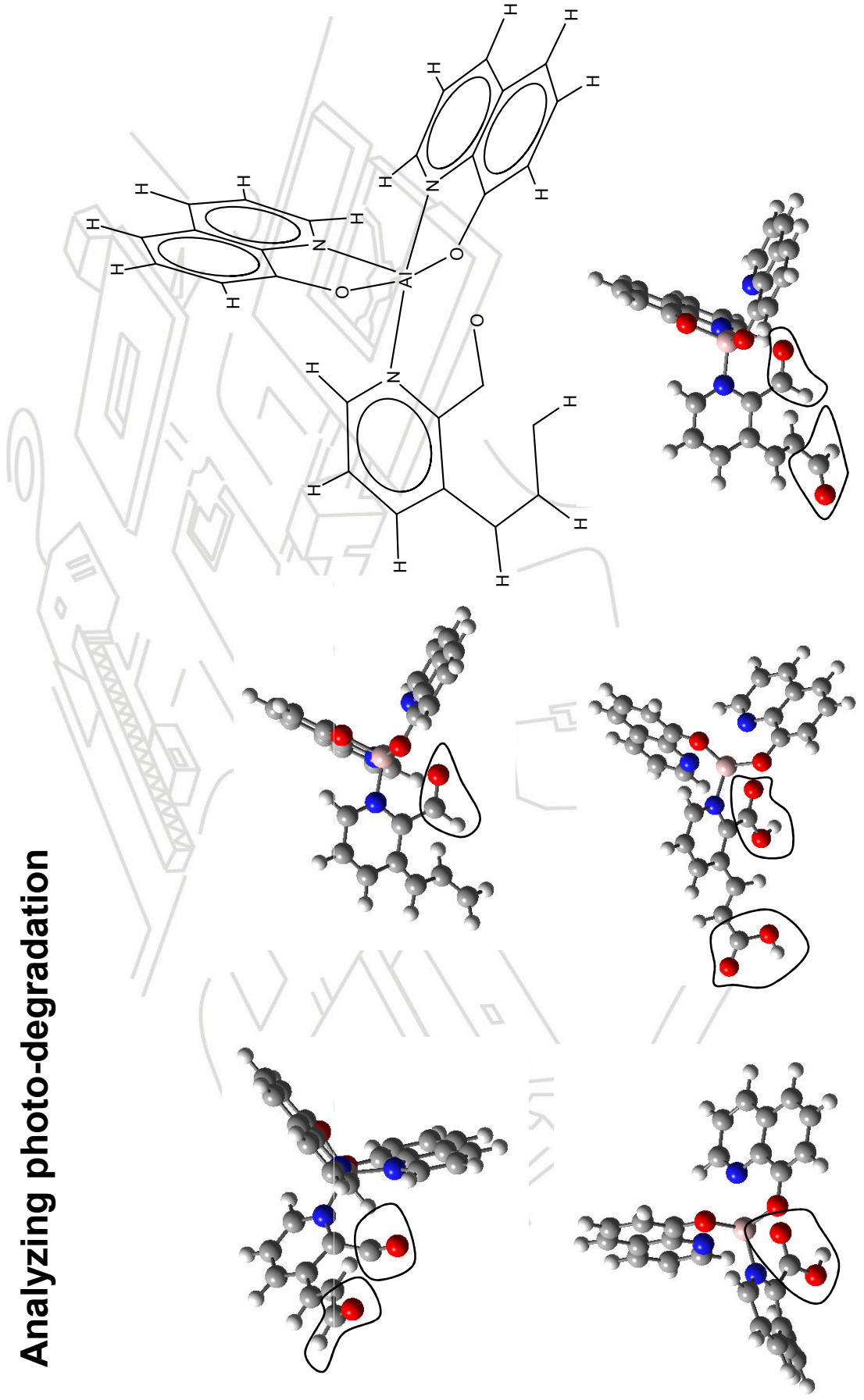
Brazilian Government

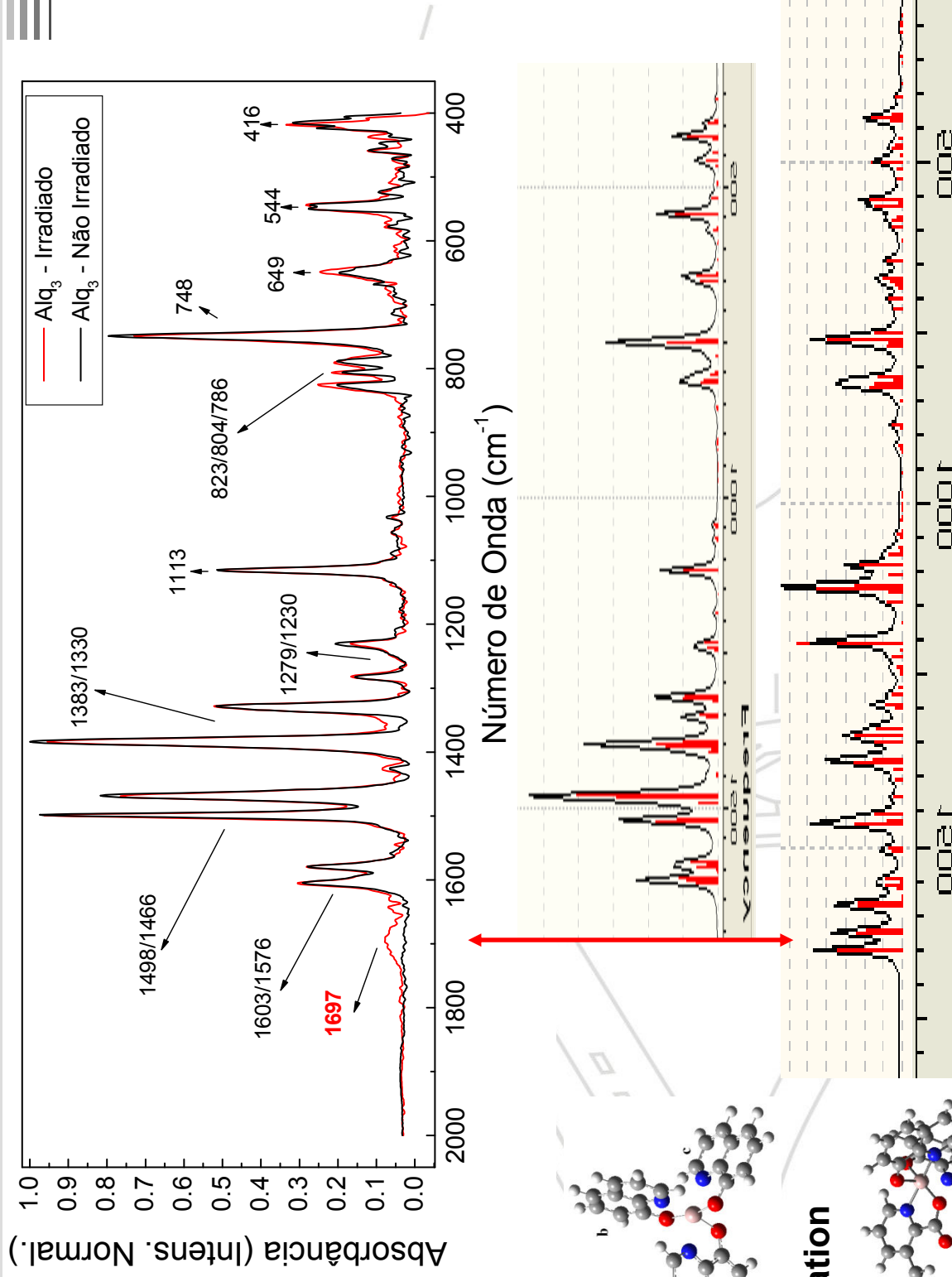
Analyzing photo-degradation





Analyzing photo-degradation





Theoretical spectra for Alq₃

Equip



Ministry of
Development, Industry
and Foreign Trade

Brazilian Government

