Teknik Karakterisasi Material

A. Setiabudi

Tujuan Perkuliahan

- Memberi pemahaman tentang prinsip dasar teknik karakterisasi material padat serta dasar-dasar interpretasinya

Undergraduate:

- Basic knowledge in all areas of chemistry
- Intro to problem solving and analytical thinking
- Intro research experience
- Exposure to modern instrumentation and theory
- Exposure to interdisciplinary teams
- Exposure to industry via internship
- Become excited and stay excited about science

Tujuan Karakterisasi Mateial

Menentukan sifat fisika dan kimia (fisiko-kimia)

- Mangetahui jenis zat (atom, molekul, senyawa)
- Mengetahui struktur (Kristal, non kristal, ikatan)
- Mengetahui kemurnian
- Mofologi Permukaan
- Sifat Termal
- Sifat listrik

Characterization of Inorganic Compounds

1. Carry out a new chemical reaction and isolate a new/known compound (organic, inorganic, or organometallic).

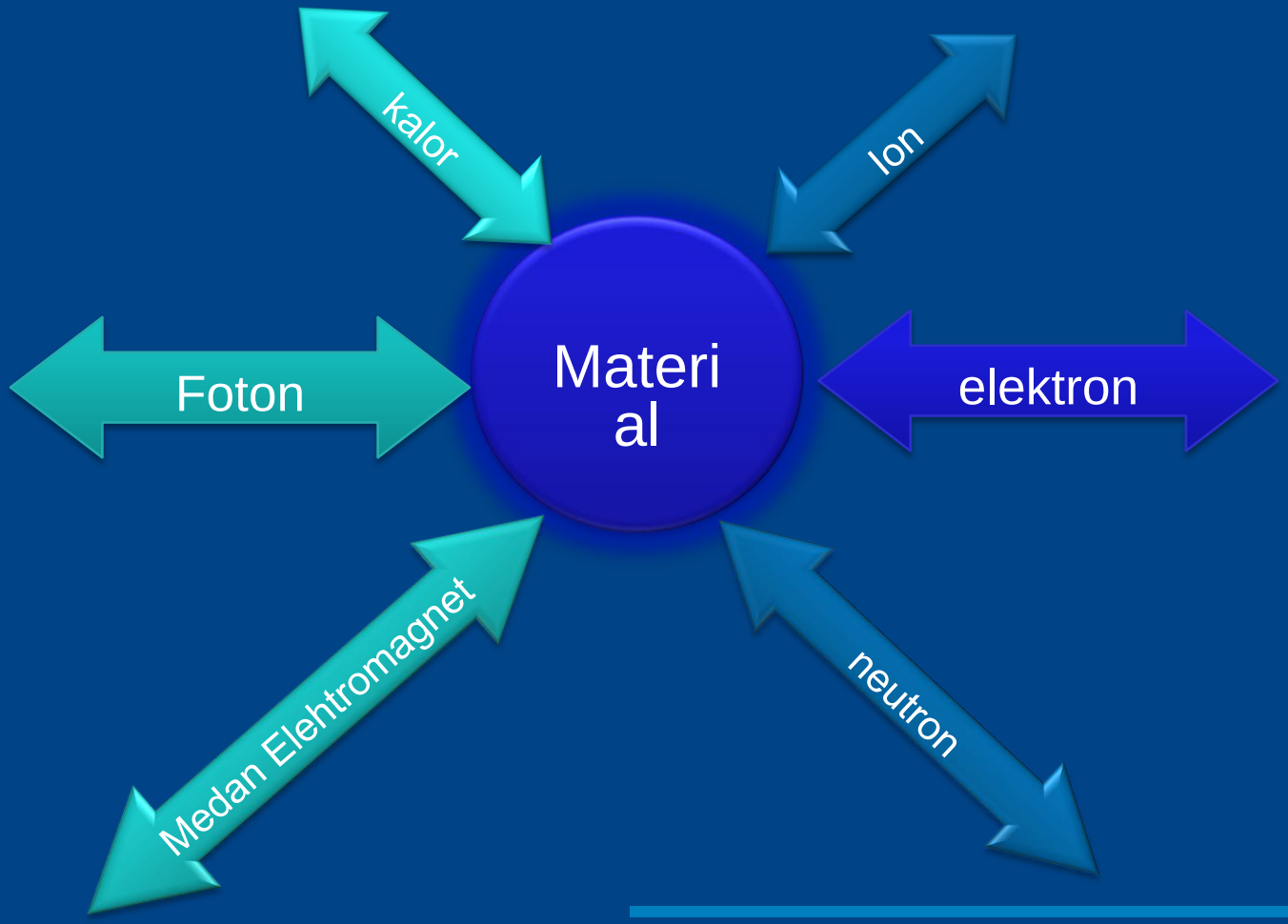
2. How do you know that your reaction has been successful or otherwise?

3. In other words, how would you characterize the compound that you have isolated?

Common Questions to Ask

1. Does our material contains a known compound?
2. Is it pure?
3. What functional groups does it contain?
4. What is its composition and its molecular weight?
5. How the functional groups are linked to each other (connectivity)?
6. What is the molecular symmetry?
7. What are the bond lengths and angles?
8. What can we say about its electronic structure?

Diagram Interaksi sebagai Dasar Katakteriasi Material

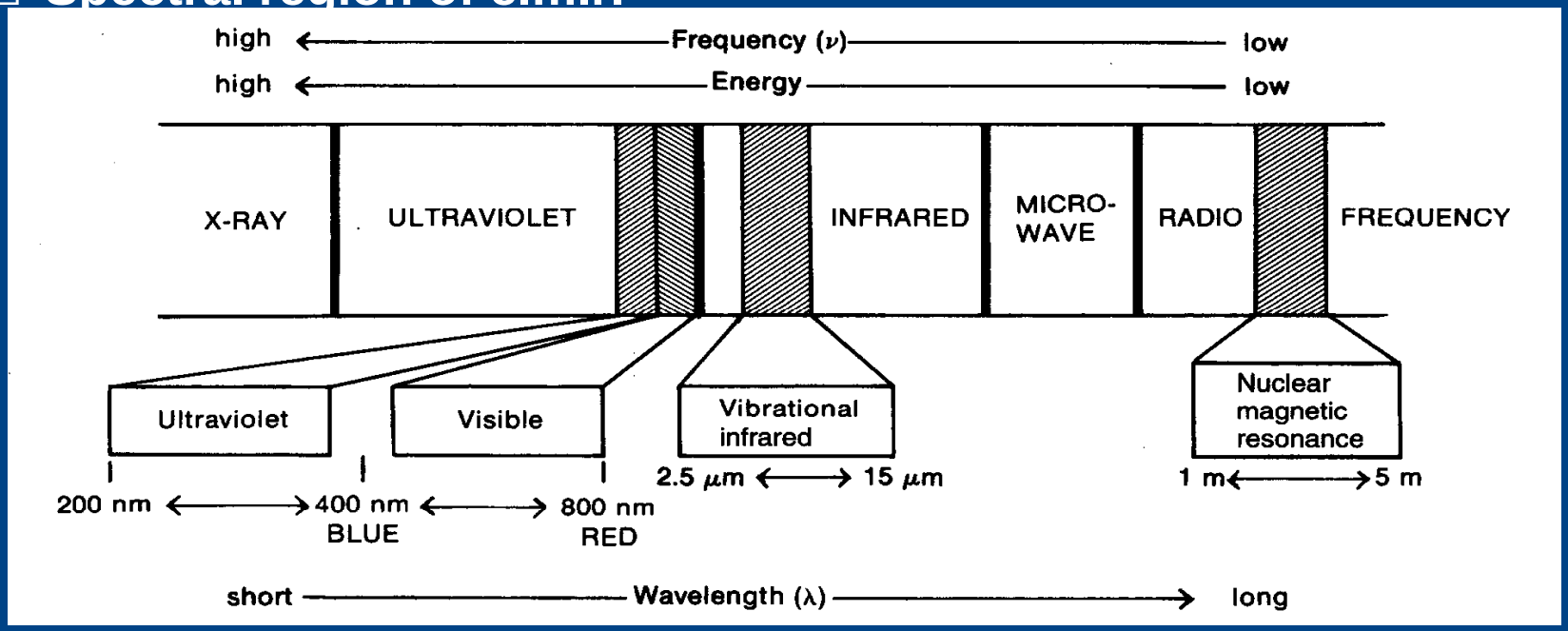


Radiasi Elektromagnetik

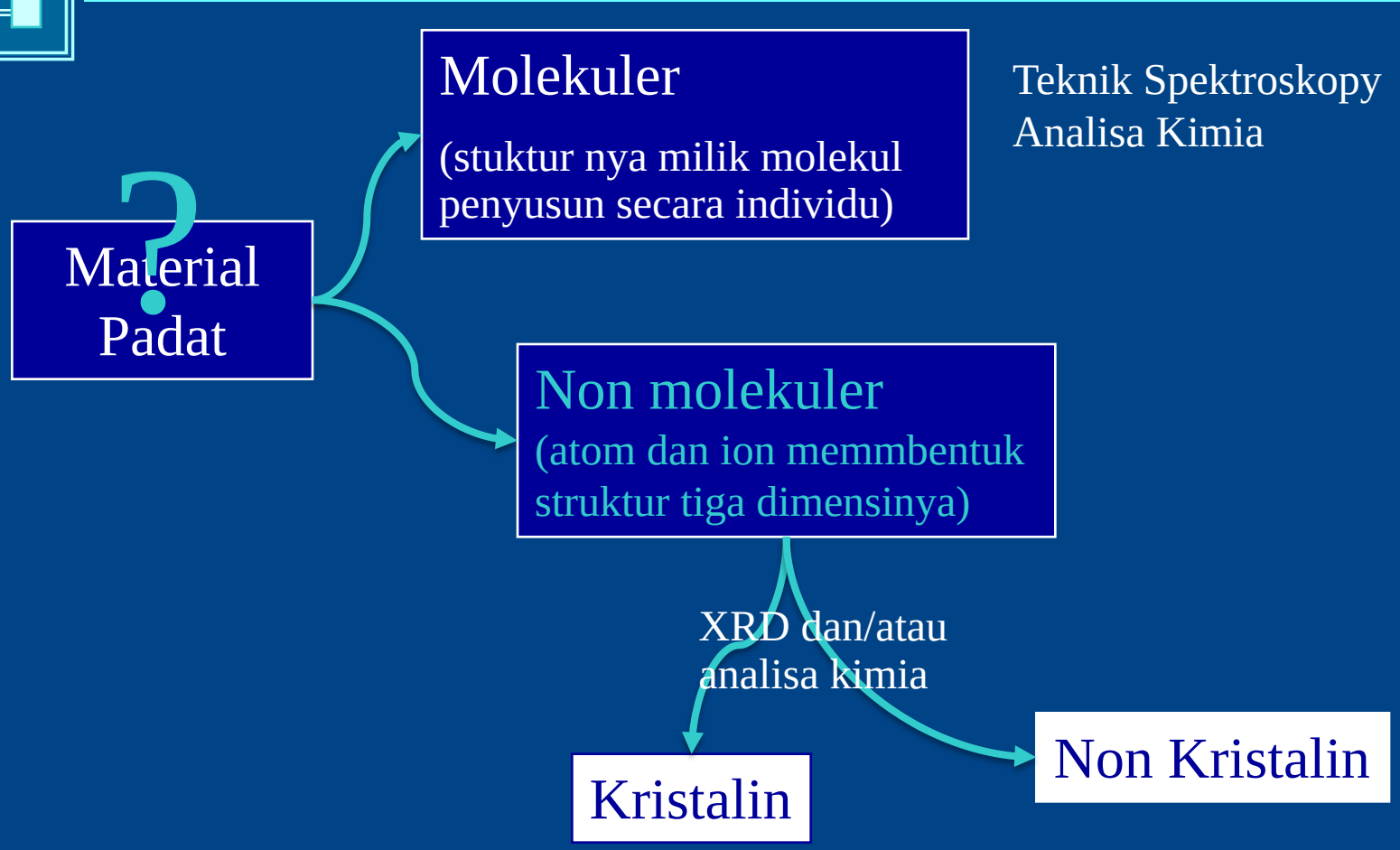
- **Electromagnetic radiation**

  X-ray, light, infra-red, microwave and radio waves are all e.m.r.'s, difference being their frequency thus the amount of energy they possess

- **Spectral region of e.m.r.**



Contoh Alur Berfikir dalam Karakterisasi



Klasifikasi Teknik Karakterisasi Material

- Teknik Spektroskopy
- Teknik Difraksi
- Teknik Mikroskopy
- Analisa Thermal

Teknik Karakterisasi Material Padat

	Jenis Ikatan	Struktur Elektron	Analisa Unsur	Textur Polikristalin	Struktur Permukaan	Defect Kristal	Struktur Kristal	Unit Sel, Geometri	Kristal Amorf	Identifikasi Fasa
XRD										
Difraksi Elektron dan Microscopy										
Difraksi Neutron										
Mikroscop Optik										
Spektroskopy IR										
Spektroscopy NMR, ESR										
Spektroscopy Elektron: XPS, UPS, AES, EELS										
X-Ray Spektroscopy, XRF, AEFS, EXAFS										
Mosebauer Spektroscopy										

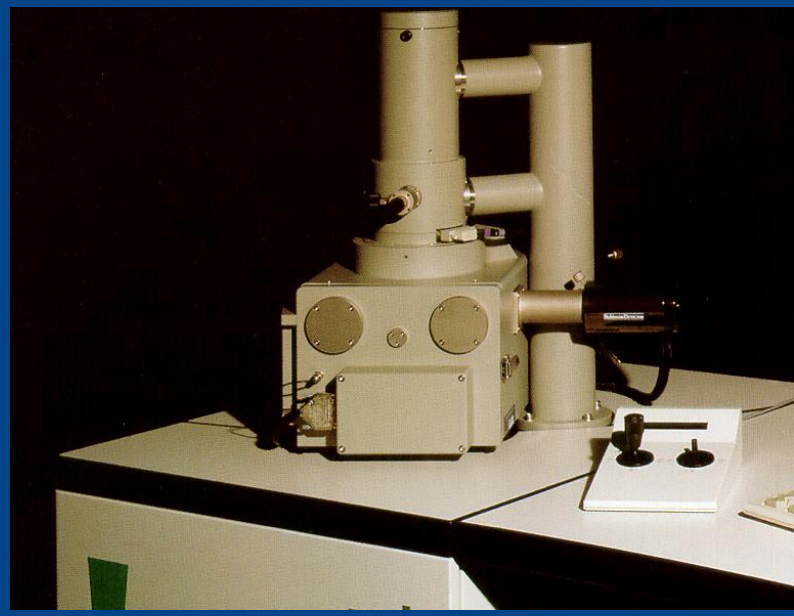
Bahan Perkuliahan

- Sifat Asam Basa Padatan
- Spektroscopy IR untuk padatan
- Teknik XRF
- Teknik SEM dan TEM
- Teknik TG-DTA
- Teknik XRD
- Penentuan Luas Permukaan Padatan

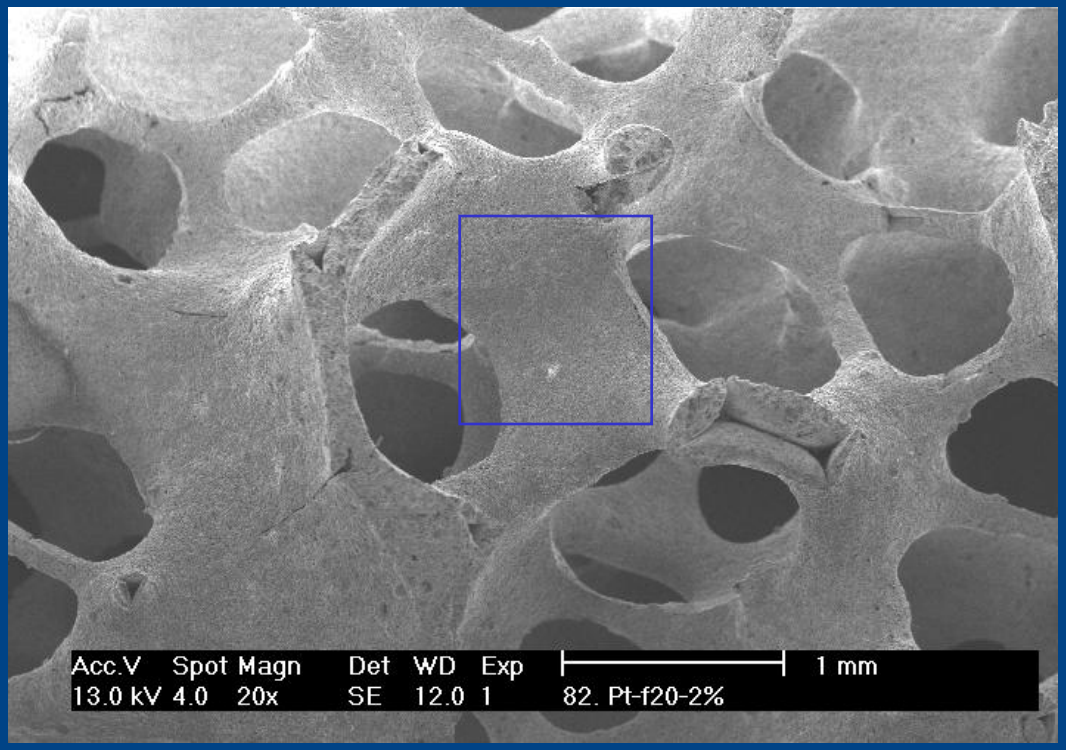
Buku Sumber

- Anthony R. West, *Solid state chemistry and its applications,* John Willey and Sons, New York (1989)
- J.W. Niemansverdriet, *Sepectroscopy in Catalysis*, Willey-VCH, New York (2000)
- Sumber-sumber di internet

SEM

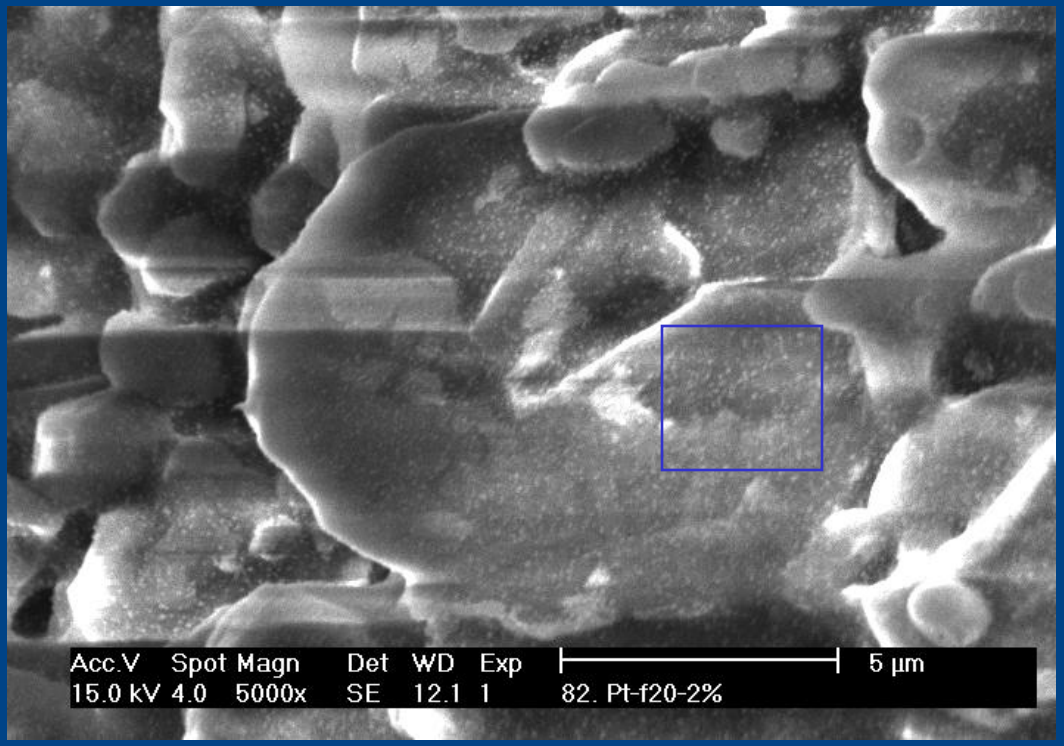


Pengantar: SEM



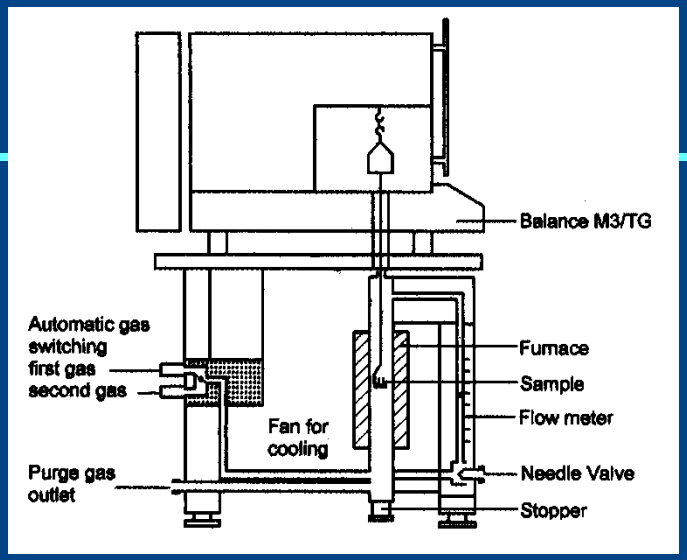
- Ceramic foam
- Perbesaran 20x

Pengantar: SEM



- Ceramic foam
- Perbesaran 5000x
- Partikel dapat dilihat pada skala micron

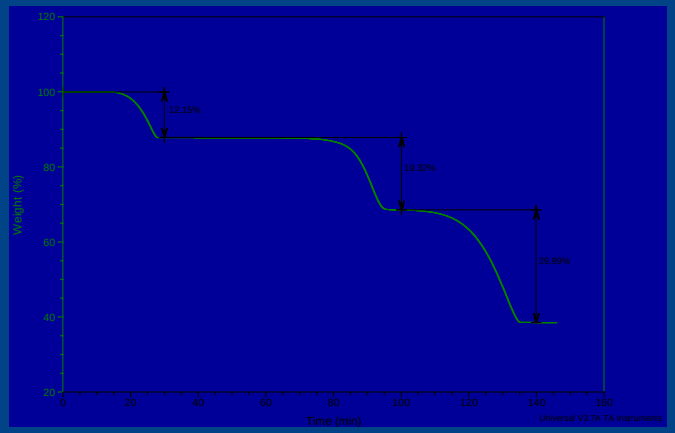
Thermogravimetric Analysis (TGA)



- thermobalance allows for monitoring sample weight as a function of temperature
- two most common instrument types

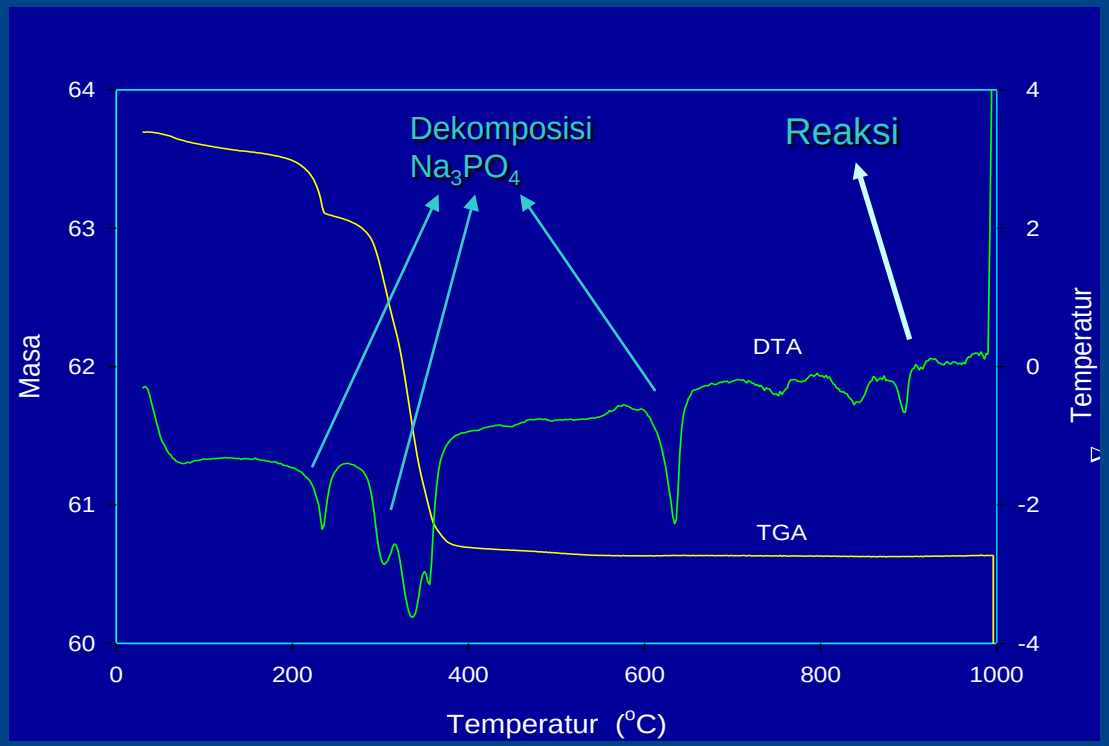
  reflection
  null

- weight calibration using calibrated weights
- temperature calibration based on ferromagnetic transition of Curie point standards (e.g., Ni)
- larger sample masses, lower temperature gradients, and higher purge rates minimize undesirable buoyancy effects



TG curve of calcium oxalate

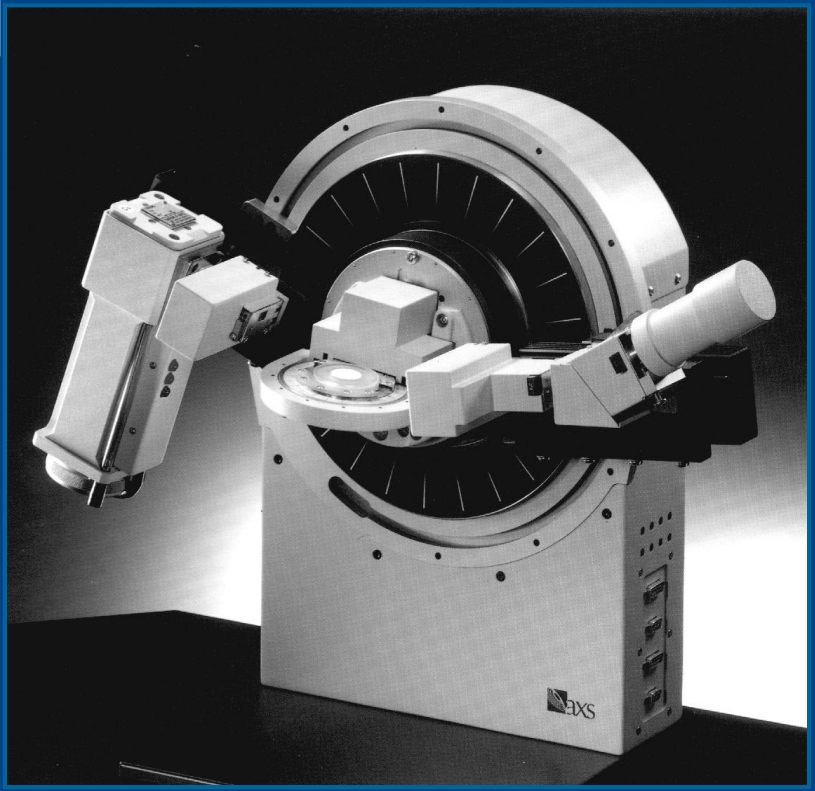
Hasil; TGA



- **Perubahan-perubahan pada t < 850 ^{0}C serupa dg pemanasan Na_3PO_4**
- **Reaksi Na_3PO_4 dg $ZrSiO_4$ kemungkinan terjadi pada suhu > 900 C**

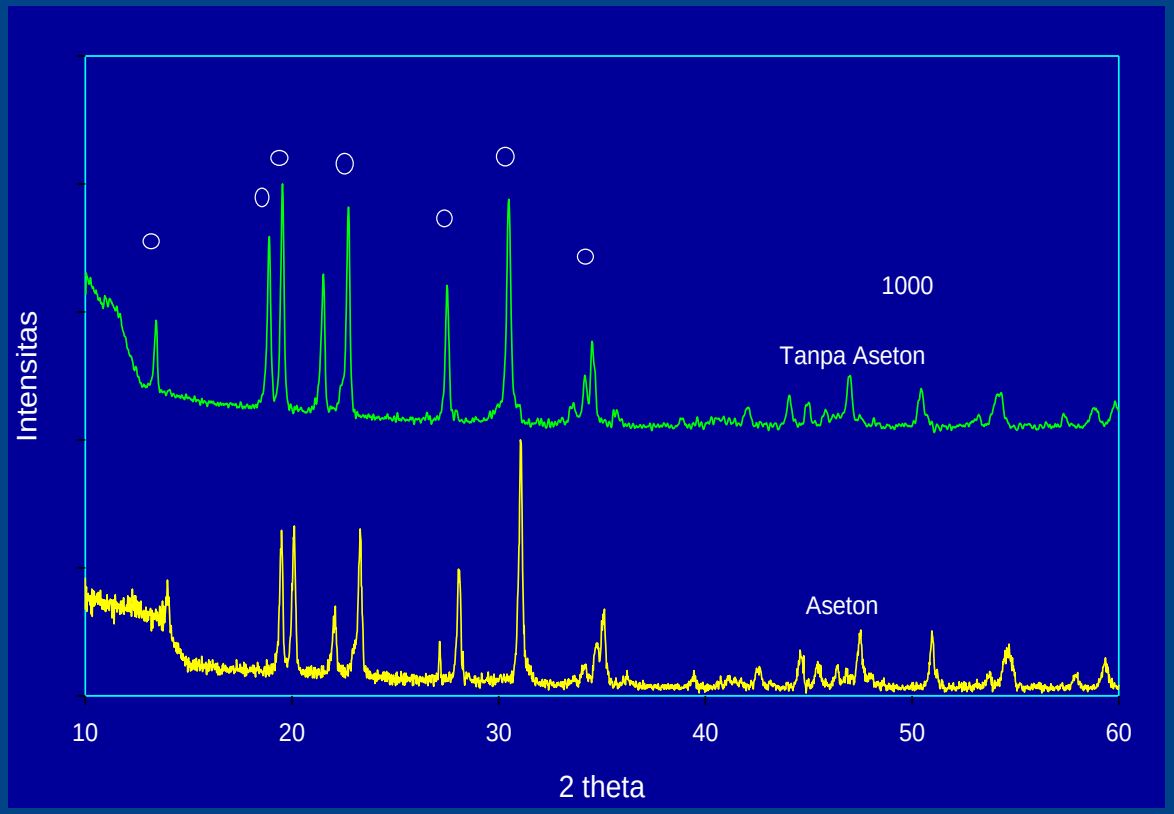
Kurva TG-DTA pemanasan Campuran Na_3PO_4-$ZrSiO_4$

A modern Diffractometer



Hasil

XRD: Aditif Proses Pencampuran



Tujuan: menambah homogenitas campuran pra-sintering

Tidak ada perbedaan signifikan antara sampel yang dipreparasi dalam bentuk serbuk dan yang diberi tekanan 60 psi