

Materials Science & Engineering UNIVERSITY OF TORONTO

Walter Curlook Materials Characterization Laboratory

Wallberg Building, 184 College Street, Room 147

Walter Curlook Materials Processing Laboratory

Wallberg Building, 184 College Street, Room 53



Introduction

The Walter Curlook Materials Processing and Materials Characterization Laboratories have been established through a generous gift by MSE alumnus distinguished adjunct professor, and former president of Inco Metals, Dr. Walter Curlook (MMS 5T0, MASc 5T1, PhD 5T3).

The laboratories house a range of analytical and process study instruments including X–ray Diffractometer, XRF Spectrometer, ICP–OES, TGA–DTA–DSC Analyzer (STA), and Laser Particle Size Analyzer. Complemented by the sample preparation equipment in other laboratories, they will serve students and faculty with their learning and research needs as well outside users seeking access to the advanced characterization facilities.

The two laboratories are located in the Wallberg Building, Department of Materials Science and Engineering, University of Toronto (184 College St., Toronto). WB147 houses the XRD, XRF, and ICP–OES, WB53 houses the Laser PSA and the STA.



Operation

The laboratories are expected to be fully functional by October 31, 2013. A full–time laboratory manager will be responsible for training the users and performing the regular maintenance.

Laser Particle Size Analyzer

(Horiba Partica LA-950)

Overview

The *Partica LA-950* Laser Diffraction Particle Size Distribution Analyzer is a high end particle sizing instrument. Applications range from studies involving nanoparticles to soils and sediments.

Principle of Operation

The instrument applies the Mie Scattering Theories using a diode laser of 650nm wavelength and a LED of 405nm wavelength for the measurement of particle size distribution of powders, suspensions or emulsions. The existing unit (standard) measures the distribution of particles suspended in a liquid (water, ethanol, and isopropanol), and is capable of performing measurements on dry powders once equipped with a *Dry Measurement Unit.*

partica

HORMBA

Components

The fully integrated system is composed of a measurement unit incorporating a diode laser, LED light source, photo detector array, side and rear scatter detectors, control section, sample chamber and a sample recirculating system (which incorporates an ultrasonic probe, flow cell and a centrifugal circulation pump that stirs, disperses and pumps). A computer system controls the instrument and displays results in a wide range of formats.

Measurement range	0.01 – 3000 µm
Measurement time	< 1 min from the introduction to sample data presentation
Total scan time	Selectable from 1 to 100 seconds
Sample size	10 mg to 5 g per measurement
Ultrasonic dispersion	0–30 min in 1 sec intervals
Circulation speed	0-12,000 ml/min, 15 step selection of speed
Suspension liquids	Compatible with water, ethanol, and isopropanol

ICP- OES (Thermo Fisher Scientific iCAP 6300)

Overview

The iCAP 6300 ICP–OES is a simultaneous spectrometer for rapid analysis of trace elements in a solution down to ppb–ppt levels. The apparatus is widely used in analysis of water and dissolved inorganic materials (soil, minerals, metals, glass, waste, etc).



Principle of Operation

The sample is introduced to the Inductively Coupled Plasma – Optical Emission Spectrometer (ICE–OES) through a nebulizer and is then broken into atoms and ions in a plasma torch, created by purging argon in a RF (radio frequency) generator. Once the exited ions combine with electrons in the cooler regions of the torch and return to their low energy state, light with atom–specific wavelength is emitted. The light from different atoms is separated into its component wavelengths using a diffraction grating. The light intensity for each wavelength is measured with a photomultiplier. By comparing with emissions of a blank, the concentration of the analytes are calculated.

Components

The system includes the ICP spectrometer, chillers, autosampler with probe wash station, and PC with iTEVA program for operating the machine.

Wavelength range	166–847 nm
Detection limits (µg/L)	0.003 (Ca) to 4.1 (Al)
RF source	27.12 MHz solid state (up to 1350 W)
Autosampler capacity	240
Torch configuration	Dual view
Plasma gas	Ar
Purge gas	Ar or N ₂

XRF Spectrometer (Bruker S2 Ranger)

Overview

The *S2 Ranger* energy dispersive XRF analyzer performs multi-element analysis from Carbon (C) to Uranium (U) from 100% down to the ppm-level in solids, powders or liquids with little sample preparation. The application range from ceramics to metals, glass, and construction materials.



Principle of Operation

The sample is exposed to high energy X–ray, resulting in ionization of the atoms. Characteristics Secondary X–rays are emitted from the material when the electron reconfiguration lowers the energy of the exited material. These characteristic radiations are detected and quantified, pointing to the type and quantity of the sample constituents.

Components

The system includes X–ray tube, detector, vacuum pump, sample tray, and sample loading mechanism, built–in processor, screen, and printer. A separate computer may be used for more advanced applications such as development of the calibration methods.

X–ray tube	50 W (50 kV max, 2 mA max) with Pd anode
Detector	xFlash® LE Silicon Drift Detector
Elemental range	Carbon to uranium
Concentration range	ppm to 100%
Sample preparation	Direct, sample cups, pressed pellets, fused beads
Sample size	Liquids and loose powders: up to 50 ml Solids: 40 mm diameter
Sample changer	28 position tablet

X–ray Diffractometer (Rigaku MiniFlex 600)

Overview

The *Miniflex 600* is a benchtop general purpose X-ray diffractometer that can perform qualitative and quantitative analysis of polycrystalline materials. It is an essential tool in phase identification and quantification and crystal structure characterization for a wide range of applications such as materials, geology, forensic, food, pharmaceutical, and environmental studies.



Principle of Operation

The X-ray is generated in a Cu x-ray tube, filtered by monochromator to increase sensitivity, collimated to concentrate, and is directed towards the sample. The X-ray is diffracted by the atomic layers of the crystal, and the diffraction pattern is used for the analysis based on the Bragg's diffraction law $(n\lambda=2d \sin\theta)$. The 2θ positions of the diffraction peaks provide a unique fingerprint of the phases present and the intensity and spread of the peaks are used to obtain the quantity of each phase based on the Rietveld Quantitative Analysis method.

Components

The system is equipped with a 2.0 kW Cu X–ray tube, Nal scintillation counter detector, graphite monochromator, and an automated 6–position sample changer with sample spinner. Instrument control and data processing are carried out using a PC. Rigaku's PDXL Comprehensive Analysis Package is used for data reduction, background elimination, search and peak match with ICDD PDF databases, crystallite size determination, % crystallinity determination, and quantitative analysis.

Generator power	600 W (tube voltage 40 kV, current 15 mA)	
Goniometer	Scan range (20):	–3 to 145 deg.
	Scan speed:	0.01–100 °/min (20)
	Minimum step width:	0.005 ° (20)
Detector	Nal scintillation counter	

Simultaneous Thermal Analyzer

(NETZSCH STA 449 F3 Jupiter)

Overview

The STA 449 F3 Jupiter is a powerful state-of-the-art thermal analysis tool for simultaneous Thermograivemetry (TG) and Differential Scanning Calorimetery (DSC) analyses on a sample. It measures the mass change and the heat released/absorbed by the sample material during heating/cooling for such studies as: melting/ crystallization behavior, phase transitions, oxidation/reduction behaviour, decomposition, thermokinetics, glass transition, etc.



Principle of Operation

The STA system is equipped with a microbalance that measure the mass of the sample as it experiences a prescribed heating/cooling regime. The sample temperature is also precisely measured and compared with a reference material. The data are processed to provide two spectrums, presenting the mass change, and the heat flow to (from) the sample.

Components

The STA includes a high temperature graphite furnace, furnace control and power supply, gas control system, vacuum pumps, top–loading microbalance, TG–DSC sample carrier, and refrigerated bath circulator.

Measurement modes	TG, DTA, DSC
Temperature range	25–2000 °C
Sample weights	Up to 200 mg
TG resolution	1 µg
Atmosphere	Static and dynamic, inert, reducing, oxidizing
Vacuum	<10 ⁻² mbar

Need more info?

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