Instrument Capabilities, Sample Requirements & Data Output

QUALITY CONTROL

Quality control of the analytical method is extremely important for the credibility of the ESRL applications. All users of laboratory facilities will be required to adhere to the ESRL Quality Management System, as outlined in the laboratory quality manual (available on request). Validation of the Zetium calibration and analytical method (to an internationally certified standard) is in progress.

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INSTRUMENTS AND EQUIPMENT

Table 1 -Analytical equipment

Instrument	**Make & Model**
WD XRF	Zetium Wavelength and Energy Dispersive X-Ray Fluorescence
ED XRF	Rigaku NEX CG Energy Dispersive X-Ray Fluorescence
Elemental Analyser	Multi EA 4000
Mercury Analyser	LECO's AMA254 Hg Analyser

Table 2 -Preparation equipment

Instrument	**Make & Model**
Planetary Ball Mill	Retsch Planetary Ball Mill PM400
Pellet Press	Herzog Pellet Press HTP 40
Fusion Instrument	Claisse LeNeo Fusion Instrument

INSTRUMENT DESCRIPTIONS

ED XRF – Rigaku

An analytical tool for the qualitative and quantitative determination of major and minor atomic elements in a wide variety of sample types. Versatile instrument capable of providing rapid, non-destructive, multi-element analyses – from low parts-per-million (ppm) levels to high weight percent (%) concentrations – for elements from Sodium (11Na) to Uranium (92U). The equipment has a capacity of 10 samples.

Samples

For auto-sampling runs (i.e. multiple measurements), samples may be liquid, loose powders, pressed powder pellets, solids or fused beads with a diameter between 32mm - 40mm. For individual analyses, larger (<12 cm) flat and homogeneous samples can be horizontally positioned within the instrument chamber and located using the internal optics (analyses will be close to the sample edge). The internal compartment is a cylindrical chamber and the individual sample cannot exceed the size of 24 cm in diameter to 9 cm in height. Sample materials include, metals (including thin films), cement, ceramic, glass, particulate matter on air filters, and polymers sample material accepted.

Samples must arrive to ESRL either as a liquid, preformed pressed powder pellets/fused beads or as pre-milled material (crushing and grinding facilities in TCD may be available by special request). The ESRL does not have the capacity to prepare fused beads at present.

Benefits

- Very low backgrounds
- Best detection limits for heavy elements
- Quantitative and qualitative analyses
- Stable results over months
- No dissolving and digestive of powder
- Low cost compared to other analytical equipment
- No glassware
- Fast Results
- Non-destructive technique
- Easy sample preparation
- No expensive reagents and waste disposal
- Complimentary to XRD and other analytical techniques

Limitations

- X-ray penetration of the sample is limited to the top 0.01 - 0.1 mm layer
- Light elements (below 13Al) have very limited sensitivity
- Liquids that give off corrosive vapour will not be analysed
- Radiation-sensitive materials may be damaged during analysis
- Magnetic and radioactive materials are unlikely suitable for analysis

Applications

Table 2 -Nex CG Applications (applications in grey may be possible in the future but require further development)

Application	**Geological Film**	**Geological Fusion**	**Geological Pellet**	**Other**	**Scan**
Analysis	Major oxides	Major oxides	Major oxides	Major oxides	Screening
Sample preparation	Loose powder	Fused glass beads	Pressed powder	Pressed powder, fused glass beads, solids or loose powder.	Pressed powder, fused glass beads, solids or loose powder.
Results unit	%	%	%	%	%
Sample type	Solid geological	Solid geological	Solid geological	Minerals, metals, cement, ceramic, glass, particulate matter on air filters, biological materials and polymers.	Minerals, metals, cement, ceramic, glass, particulate matter on air filters, and polymers.
Method	ED XRF + Fundamental Parameters	ED XRF + Fundamental Parameters	ED XRF + Fundamental Parameters	ED XRF + Fundamental Parameters	ED XRF
Analytes	Al_2O_3[5], CaO[5], Fe_2O_3[3], K_2O[2], MgO[7], MnO[1], Na_2O[1], P_2O_5[3], SiO_2[6], SO_3[4], TiO_2[1].	Al_2O_3[9, 11], CaO[8,10], Fe_2O_3[8, 11], K_2O[8,10], MgO[8,10], MnO[8,10], SiO_2[9,12,13], SO_3[8,10], TiO_2[8,10].	Al_2O_3[14,19,22], CaO[14,18,22], Fe_2O_3[14,19,22], K_2O[14,17,22], MgO[14,20,22], MnO[14,17,22], Na_2O[15,17,22], SiO_2[16,21,23] TiO_2[14,17,24].	Potential analyses requiring development and/or validation	Energy range (keV): 1 to 40
LLD	< 0.1 %	[8]: < 0.1 % [9]: < 0.3 %	[14]: < 0.1 % [15]: < 0.2 % [16]: < 0.5 %		Semi-quantitative
Maximum Concentration	[1]: < 10 % [2]: < 20 % [3]: < 40 % [4]: < 50 % [5]: < 60 % [6]: < 70 % [7]: > 90 %	[10]: < 10 % [11]: < 20 % [12]: > 70 %	[17]: < 10 % [18]: < 20 % [19]: < 30 % [20]: < 50 % [21]: < 80 %		
Deviation	≤ 0.6 %	≤ 0.06 % [13]: < 0.7 %	[22]: ≤ 0.07 % [23]: < 0.12 % [24]: < 0.3 %		
Average Precision	0.05	0.03	0.01		
Average Accuracy	0.09	0.04	0.09		
Approximate analysis time per sample	15 min	8 min	11 min	20 min	20 min

WD XRF and ED XRF- Zetium

The Zetium XRF spectrometer is the market leader in high-quality design and innovative features for sub-ppm to percentage analysis of 4Be to 241Am. The instrument is equipped with a barcode scanner and capable of operating autonomously, with a capacity of 114 samples.

Samples

Geological material only (excluding ore type samples or radioactive material) in the form of pressed powder pellets or fused beads with an analysis diameter between 27mm - 40mm.

Samples must arrive to ESRL either as preformed pressed powder pellets/fused beads or as pre-milled material (crushing and grinding facilities in TCD may be available by special request). The ESRL does not have the capacity to prepare fused beads at present.

Benefits

- High resolution for all elements
- Quantitative and qualitative analyses
- Stable results over months
- No dissolving and digestive of powder
- Low cost compared to other analytical equipment
- No glassware
- Fast Results
- Non-destructive technique
- Easy sample preparation
- No expensive reagents and waste disposal
- Complimentary to XRD and other analytical techniques

Limitations

- X-ray penetration of the sample is limited to the top 0.01 - 0.1 mm layer
- Light elements (below 13Al) have limited sensitivity.
- Liquids and loose powder cannot be analysed
- Radiation-sensitive materials may be damaged during analysis
- Magnetic and radioactive materials are unlikely suitable for analysis.

Applications

Table 3 - Zetium Applications (applications in grey may be possible in the future but require further development)

Application	Pro-Trace	Geology PP	Ag-Ce	WROXI	Fused beads	Omnian
Analysis	Trace element	Major oxides	Trace element	Major oxides	Major element	Screening
Sample preparation	Pressed powder	Pressed powder	Pressed powder	Fused glass beads	Fused glass beads	Pressed powder, fused glass beads or solids.
Results unit	ppm	%	ppm	%	%	ppm or %
Sample type	Solid geological	Solid geological	Solid geological	Solid geological	Requires development	Minerals, metals, cement, ceramic, glass, particulate matter on air filters, and polymers.
Method	WDXRF	WDXRF	EDXRF	WDXRF	ED or WD	WDXRF
Analytes	Ag[25,29], As[25,28], Ba[26,29], Bi[25,28], Br[25,29], CaO[27,30], Cd[25,28], Ce[26,28], Co[25,28], Cr[25,28], Cs[25,29], Cu[25,28], Fe_2O_3[27,30], Ga[25,28], Ge[25,29], Hf[25,29], Hg[25,29], I[25,29], La[26,28], Mn[25,30], Mo[25,29], Nb[25,28], Nd[25,28], Ni[25,28], Pb[25,28], Rb[25,28], Sb[25,28], Sc[25,29], Se[25,28], Sm[25,29], Sn[25,28], Sr[25,29], Ta[25,28], Te[25,28], Th[25,28], TiO_2[25,30], Tl[25,29], U[25,29], V[25,28], W[25,28], Y[25,28], Yb[25,29], Zn[25,28], Zr[25,28].	Al_2O_3[34], CaO[34], Cl[31], Fe_2O_3[33], K_2O[32], MgO[35], MnO[31], Na_2O[32], P_2O_5[33], SiO_2[35], SO_3[31], TiO_2[32].	Ag[36], Ba[39], Cd[36], Ce[38], Cs[37], I[36], La[37], Sb[36], Sn[38], Te[36]	Al_2O_3[43], BaO[41], CaO[43], Cr_2O_3[40], CuO[40], Fe_2O_3[43], HfO_2[40], K_2O[41], MgO[43], Mn_3O_4[43], Na_2O[42], NiO[40], P_2O_5[41], PbO[40], SiO_2[43], SO_3[42], SrO[41], TiO_2[41], V_2O_5[40], ZnO[40], ZrO_2[42].	Requires development	Angle range (°2T): 14° to 147.06°.
LLD	[25]: < 5 ppm, [26]: < 10 ppm [27]: < 20 ppm	< 0.01 %	< 3 ppm	< 0.01 %		Semi-quantitative
Maximum Concentration	[28]: < 1000 ppm [29]: < 4000 ppm [30]: < 4000 ppm	[31]: < 3% [32]: < 15% [33]: < 40% [34]: < 60% [35]: > 90%	[36]: < 10ppm [37]: < 50 ppm [38]: < 150 ppm [39]: >1000 ppm	[40]: ≤10% [41]: ≤ 40% [42]: < 60% [43]: > 60%		Semi-quantitative
Validation	In progress	In progress	In progress	In progress		Semi-quantitative
Approximate analysis time per sample	1h 15min	3 min	10 min	9 min		21 min

Elemental Analyser – Multi EA

Multi EA 4000: a versatile tool for the automated determination of TS (Total Sulphur), TC (Total Carbon), TOC (Total Organic Carbon), TIC (Total Inorganic Carbon) and TC-EC (Determination of carbon after pyrolysis) samples, with autosampler control in a single analysis process.

Samples

Macro elemental analyses of loose soil samples, sediments, construction materials, ashes, polymers, wastes, catalysts, minerals and fertilizers. Sample particle sizes <200 µm are recommended for more uniform combustion and better repeatability between measurements. However, analyses are possible with particle sizes <1 mm.

Benefits

- Sample weights up to 3 g
- Simultaneous detection of carbon and sulfur
- Low operating costs
- Automated solids sampler
- Wide-range NDIR detector for carbon and sulfur

Limitations

- Flammable or explosive materials are prohibited.
- No corrosive substances (e.g. concentrated acids) can be analysed.

Applications

Table 4 - EA4000 Applications (applications in grey may be possible in the future but require further development)

Application	**TC+TS_inorg_ with_TIC_auto**	**TS_inorg_ with_TIC_auto**	**TOC_Difference _inorg**	**TIC_auto**	**EC_TC_inorg_ with_TIC_auto**
Results unit	%	%	%	%	%
Sample type	Solid geological, construction materials, ashes, polymers, wastes, catalysts, minerals and fertilizers	Solid geological, construction materials, ashes, polymers, wastes, catalysts, minerals and fertilizers	Solid geological, construction materials, ashes, polymers, wastes, catalysts, minerals and fertilizers	Solid geological, construction materials, ashes, polymers, wastes, catalysts, minerals and fertilizers	Solid geological, construction materials, ashes, polymers, wastes, catalysts, minerals and fertilizers
Analytes	Total Surfur and Total Carbon	Total Surfur	Total Inorganic Carbon and Total Carbon	Total Inorganic Carbon	Potential analyses requiring development and/or validation
LLD	393.83 µg (TC) and 39.24 µg (TS)	39.24 µg	55.06 µg (TIC) and 393.83 µg (TC)	55.06 µg	
Maximum Concentration	100%	100%	100%	100%	
Validation	In progress	In progress	In progress	In progress	
Analysis time per sample	10 min	10 min	20 min	10 min	

Mercury Analyser – Hg Analyser

The AMA254 technique offers direct analysis of trace Hg in a variety of sample media. A combustion/catalyst tube decomposes samples in an oxygen-rich environment, removing interfering elements. An Au amalgamator trap then collects all Hg from the evolved gases and a dual-path length cuvette/spectrophotomer specifically determines Hg concentrations over a wide dynamic range.

Samples

LECO's AMA254 determines trace amounts of Hg in various materials, including soils, biological samples, coal, combustion residues and other solid/liquid samples. The dried and powdered samples can be weighed directly into the boat placed on the balance.

Benefits

- Determination of mercury in liquid and solid samples
- Fast, safe and accurate determination of traces of mercury
- Results in ten minutes
- No sample pre-treatment required.
- Automated sampler on the combustion/catalyst tube.

Limitations

- Flammable or explosive materials are prohibited.
- No corrosive substances (e.g. concentrated acids) can be analysed

Applications

Table 5 - AMA254 Applications

Application	**Determination of Mercury in Plant Tissue**	**Determination of Mercury in Sewage Sludge**	**Total Mercury in Soils**
Results unit	ppm	ppm	ppm
Sample type	Plant Tissue (Vegetable Food)	Sewage Sludge	Soils
Analytes	Mercury	Mercury	Mercury
Validation	Potential analyses requiring development and/or validation		
Analysis time per sample	10 min	10 min	10 min

Planetary Ball Mill – PM400

The PM400 has four grinding stations capable of grinding up to 8 samples at a max speed of 400rpm. The PM 400 has the following adjustable grinding parameters: grinding time, speed, interval, directional reversal and pause time. The ESRL is equipped with 8 agate grinding vessels (80ml) and 2 complete sets of agate grinding balls (ø36mm and ø20mm).

Samples

The Planetary Ball Mill is designed to grind and mix, soft, medium hard and extremely hard, brittle and fibrous materials, wet or dry, quickly and with high reproducibility. Samples that carry the risk of explosion or fire are prohibited.

Only geological materials (excluding ore samples), are permitted for grinding in the ESRL at present. Sample material must have a hardness <7mohs. The sample volume per grinding vessel is between 10 – 35ml with a feed size of up to <4mm depending on the material. Samples may need to be pre-milled/crushed before arriving to the ESRL depending on the material (crushing and grinding facilities in TCD may be available by special request).

Output

Fine, homogenised, powdered material (grain size dependent on the material and grinding parameters chosen).

Pellet Press – HTP 40

The pellet press was developed specifically for pressing to form pellets and ensures high level or reproducibility of the sample preparation. The Pellet Press is equipped for free pressing and pressing in aluminium cups with a max press force of 400kN.

Samples

Ferroalloys, geological samples, cement raw meal, clinker, cement, sinter, slag, refractory materials, ceramics and glass in a dry powdery form (max 10µm, max 9mohs) are all suitable sample materials for pressing into pellets. Liquids, volatile or explosive substances are prohibited from entering the pellet press.

Only Geological materials (excluding ore samples) are permitted for pressing in the ESRL at present. A wax pellet binder or similar may be required depending on the material type. Samples may need to be pre-milled/crushed before arriving to the ESRL depending on the material.

Output

Samples are pressed to form 40mm diameter pellets suitable for XRF analysis.

Fusion Instrument – Claisse LeNeo

LeNeo fusion instrument prepares glass disks for XRF analysis as well as borate and peroxide solutions for AA and ICP analysis. LeNeo is a versatile instrument providing excellent reproducibility allowing the obtention of high-quality analytical results. Note, only glass disk preparation is currently available in the ESRL.

This automatic electric instrument has one fusion position and a heating chamber that heats up to 1200°C. With fully automated pouring and automatically locking safety doors, LeNeo provides safe cold-to-cold operation. Programmable fusion parameters include: temperature, duration, heating rate, crucible rocking speed, cooling airflow, pouring modes and magnetic stirring. Absolute control of the fusion temperature and the additional parameters optimises oxidation and fusion success.

Samples

The Claisse LeNeo is a sample preparation instrument used to transform powders of cement, lime, catalysts, mining and geological samples, refractories, glass, silica, bauxite, alumina, and many others into either glass disks, borate solutions or peroxide solutions. Samples that carry the risk of explosion or fire are prohibited.

Only geological materials, excluding samples with a large proportion of reduced phases including ores, sulphide rich samples, metals/alloys, or metal-rich samples, are permitted for fusion in the ESRL at present. Sample material should be finely powdered (nominally to <75 µm or to achieve a smooth talc-like feel between gloved fingers) and dry. A sample mass of 1.00g ± 0.005g is required per ESRL standard operating procedure.

Output

Glass disks (40mm diameters) for XRF analysis.