MATRIX REFERENCE MATERIALS
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Methods used by laboratories to analyse samples

1. Aqua regia (2 Acid) digestion (2A_MICP)

Two-acid digests are the weakest of the digestions and will not attack silicate minerals. As such, the two acid leach can only be expected to provide partial results for most elements. . Elements which are hardly (if at all) digested include barite, zircon, monazite, sphene, chromite, gahnite, garnet, ilmenite, rutile and cassiterite. Generally, but not always, most base metals and gold are usually dissolved if the sample is ground finely enough.

After sample digestion, the solution is analysed by either inductively coupled plasma-atomic emission spectrometry (ICP-AES), inductively coupled plasma-mass spectrometry (ICP-MS), atomic absorption spectrometry (AAS) or gravimetric spectrometry. These solutions can also be analysed by hydride atomic absorption spectrometry (AAS) to determine the volatile elements (Sb, As, Bi, Se and Te).

2. Multi acid digestion (4A_ MICP)

Multi-acid (4-acid) digestion is a very effective dissolution procedure for a large number of mineral species and is suitable for a wide range of elements. Multi-acid digestion uses a combination of HNO3 (nitric acid), HF (hydrofluoric acid), HClO4 (perchloric acid) and HCl (hydrochloric acid).

Because hydrofluoric acid dissolves silicate minerals, these digestions are often referred to as “near-total digestions”. However, there can be a loss of volatile elements (e.g. B, As, Pb, Ge, Sb) during this type of digestion and certain minerals (zircon, sphene and magnetite) are only partially digested.

After sample digestion, the solution is analysed by either inductively coupled plasma-atomic emission spectrometry (ICP-AES), inductively coupled plasma-mass spectrometry (ICP-MS), atomic absorption spectrometry (AAS) or gravimetric spectrometry.

3. Fusion (FUS)

Fusion involves the complete digestion of the sample in molten flux. Fusions are generally more aggressive than acid digestion methods and are suitable for many refractory, difficult-to-dissolve minerals such as chromite, ilmenite, spinel, cassiterite and minerals of the tantalum-tungsten solid solution series. Fusion analyses are presumed to provide a complete chemical analysis and are referred to as a “total” analysis. After sample digestion, the solution is analysed by either inductively coupled plasma-atomic emission spectrometry (ICP-AES), inductively coupled plasma-mass spectrometry (ICP-MS), atomic absorption spectrometry (AAS) or gravimetric spectrometry.

3.1 Sodium Peroxide Fusion

Sodium peroxide is a strongly oxidizing flux that is basic, not acidic in nature. It renders most resistant minerals soluble.

The method involves mixing prepared sample with a Sodium Peroxide flux and heating the mixture to separate the sample which is then allowed to cool. The resulting material are dissolved in a weak acid solution and then analysed by ICP-MS and ICP-OES. Elements with low detection limits are available from this digest.

AMIS
(A Division of Torre Analytical Services (Pty) Limited)
(Reg. No. 1989/000201/07)
A: 11 Avalon Road, West Lake View Ext 11, Modderfontein, South Africa
P: PO Box 856, Isando, 1600, Gauteng, South Africa
T: +27 (0) 11 923-0800
W: www.amis.co.za

Directors: JT Botes, R Naidoo, NN Robinson, M Padayachee

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3.2 Lithium borate

Borate fusion involves mixing a solid sample with a solid strong base in a platinum crucible. The crucible containing the sample is then heated at a low temperature for 1-2 mins. The sample mixture is heated to initiate and speed up the oxidation reaction that will quickly follow. Distilled water is gently added to the sample mixture to initiate oxidation between the sample and the base.

Most of the fusion methods employ non-wetting agents that are incorporated into the flux to prevent sticking of the melt to the crucible and improve casting performance of the moulds. Once the sample is oxidized, a non-wetting agent and a lithium-borate flux are added to the oxidized mixture.

The fusion is then carried out at high temperature around 1000°C within an automatic fusion fluxer. At this high temperature, the sample is melted and dissolved by the lithium borate flux to form a perfectly homogeneous mass. The molten mixture is then poured into dilute mineral acid and stirred until the glass flux dissolves. In some cases, the melt is poured into a preheated platinum mould to produce the desired sample for XRF analysis or into an unbreakable beaker with an acid solution for AA or ICP analysis.

4. Nickel sulphide by fire assay (NiS)

Nickel sulphide is a widely used method to determine platinum group elements. The method involves mixing nickel, sulphur, sodium carbonate, borax and SiO2 together in a clay crucible. Then fusion it at a temperature between 1000˚C - 1300˚C. As the NiS button forms it extracts the PGE from the sample. The button is crushed and dissolved and the residue of extraneous material is filtered leaving the precious metal residue on the on the filter.

Further concentration of precious metals in the nickel button is done by dissolving the powdered button in HCl (hydrochloric acid), filter off the PGE-containing residue on membrane. Then dissolved in aqua regia and finish with ICP-OES or ICP-MS analysis. Another option is to dissolve the powdered button, add tellurium and reduce this to elemental tellurium to concentrate the PGE. The PGE are analysed using ICP-OES or ICP-MS after the digesting of the teluurium precipitate in aqua regia.

5. Lead collection by fire assay (Pb Collection)

Lead collection is widely used to determine gold. This techniques is also effective for platinum and palladium. But for full determination of PGE six-elements, nickel sulphide by fire assay is more suitable. The method involves mixing a sample with a fluxing agent. Lead is added as a collector. The sample is then heated in a furnace at about 1000 degrees. The sample is then fused and the precious metals and the lead separate from the silicate slag to form a button at the bottom of the crucible. This button contains precious minerals.
The precious metals (Au and palladium and Platinum) are dissolved and determined by ICP or AA or Gravimetric Finish.

6. X-Ray fluorescence (XRF)

An XRF spectrometer is an x-ray instrument used for routine, relatively non-destructive chemical analyses of rocks, minerals, sediments and fluids. It works on wavelength-dispersive spectroscopic principles.

The analysis of major and trace elements in geological materials by x-ray fluorescence is made possible by the behaviour of atoms when they interact with radiation. When materials are excited with high-energy, short wavelength radiation (e.g., X-rays), they can become ionized. If the energy of the radiation is sufficient to dislodge a tightly-held inner electron, the atom becomes unstable and an outer electron replaces the missing inner electron. When this happens, energy is released due to the decreased binding energy of the inner electron orbital compared with an outer one.

The emitted radiation is of lower energy than the primary incident X-rays and is termed fluorescent radiation. Because the energy of the emitted photon is characteristic of a transition between specific electron orbitals in a particular element, the resulting fluorescent X-rays can be used to detect the abundances of elements that are present in the sample.

7. Combustion LECO (Comb/LECO)

LECO combustion testing is a technique used to precisely determine the content of carbon, sulfur, nitrogen, oxygen and hydrogen in metals and alloys. When small pieces of metals samples cannot be “sparked” on a spectrograph due to size constraints, LECO combustion can be used analyse the samples at low detection levels.

7.1 Carbon & Sulfur analysis

After weighing the material, it is heated and combusted in the presence of pure oxygen. During the process, carbon and sulfur are oxidized to form CO2 and SO2.

7.2 Oxygen & Nitrogen analysis

Incorporating gas fusion under a flowing stream of helium, then measure combustion gases through infrared absorption and thermal conductivity. During this process, oxygen and carbon combine to form CO2 (carbon dioxide), and nitrogen is released as N2 (nitrogen gas).

7.3 Hydrogen analysis

Incorporating gas fusion under a flowing stream of inert argon gas, then measure combustion gases by infrared absorption and thermal conductivity. During the process, oxygen combines with carbon to form CO2 (carbon dioxide) and hydrogen is released as H2 (hydrogen gas).
With LECO combustion analysis method the following can be tested:

- Carbon steel
- Stainless steel
- Alloys steel
- Cast iron
- Nickel alloys
- Titanium alloys
- Cobalt alloys

8. Specific Gravity determination by pycnometer (SG)

Pycnometer-empty and full. ***NOTE*** *fluid in stopper*

Pycnometer is a flask with a close-fitting ground glass stopper with a fine hole through it, so that a given volume can be accurately obtained. This enables the density of a fluid to be measured accurately, by reference to an appropriate working fluid such as water or mercury, using an analytical balance.

If the flask is weighed empty, full of water, and full of a liquid whose specific gravity is desired, the specific gravity of the liquid can easily be calculated. The particle density of a powder, to which the usual method weighing cannot be applied, can also be determined with a pycnometer. The powder is added to the pycnometer, which is then weighed, giving the weight of the powder sample. The pycnometer is then filled with a liquid of known density, in which the powder is completely insoluble. The weight of the displaced liquid can then be determined, and hence the specific gravity of the powder.