



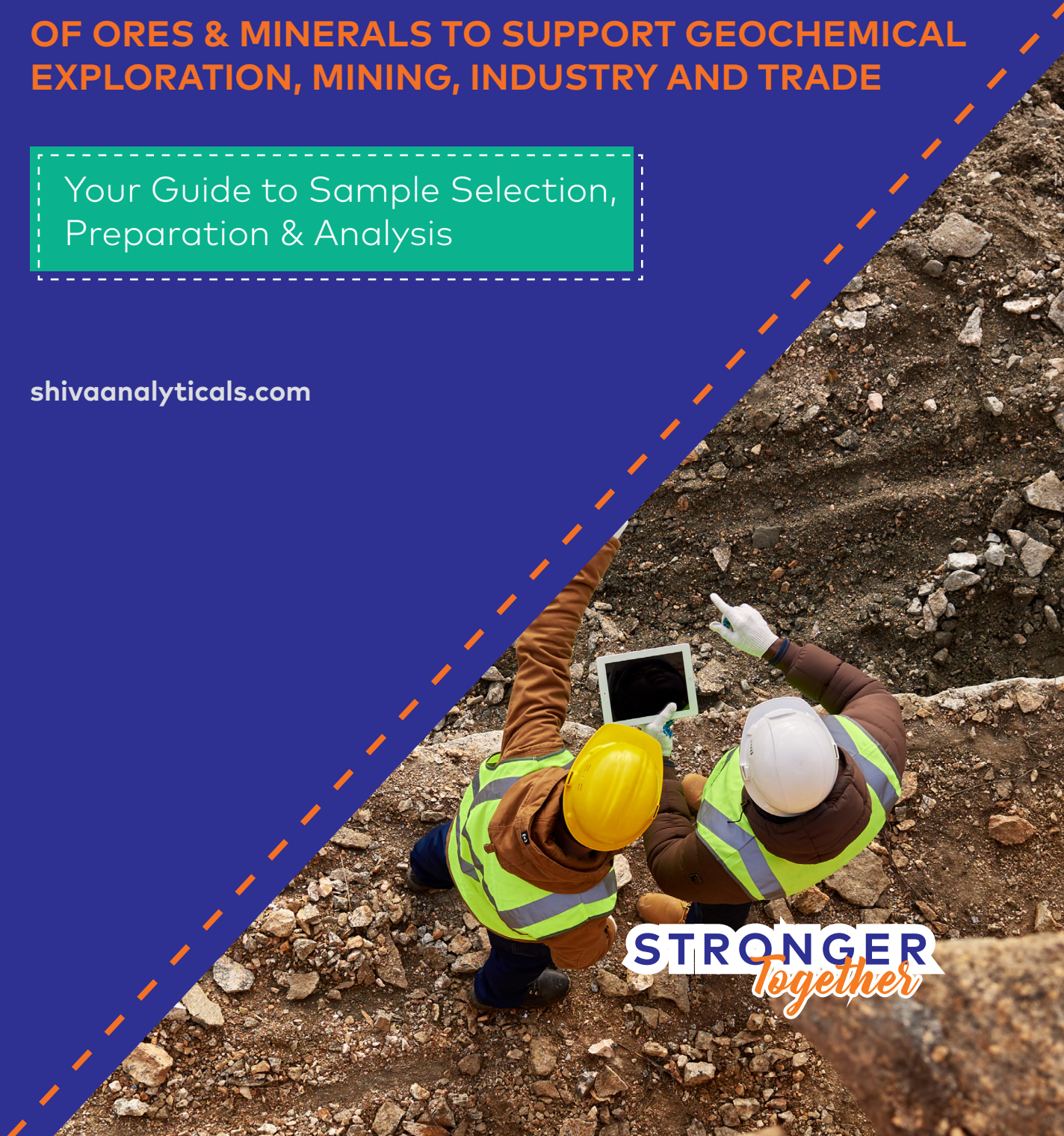
TRUSTED LABORATORY PARTNER FOR ANALYSIS

OF ORES & MINERALS TO SUPPORT GEOCHEMICAL
EXPLORATION, MINING, INDUSTRY AND TRADE

Your Guide to Sample Selection,
Preparation & Analysis

shivaanalytics.com

STRONGER
Together



Shiva Analyticals is one of the largest ores and minerals sample preparation and testing facilities available in Asia; which can handle more than 2000 samples a day which includes sample logging, drying, crushing, rotary division, pulverisation and testing by using world-class sample preparation and testing facilities. Our logistic support helps the customers to ship the samples in shortest possible time from any part of the world.

Selecting the appropriate sample quantity of ores and minerals is critical to capture the variability within the resource of interest and to ensure that the quantity/ scale of the sample collected shall be representative to the average quality of the host material. The sample quantity depends upon geological characteristics (of the ore deposit or mineralized zone) such as mineralogy, texture, grain size, heterogeneity and the choice of sampling method (e.g., grab sampling, channel sampling, drill core sampling). Preferable to conduct pilot sampling to assess the variability and determine an appropriate sample size before collecting a larger number of samples. This allows for adjustments to be made based on preliminary results. By considering these factors and applying sound sampling principles, we can select an appropriate sample size that balances representativeness, precision, and practicality in sampling of ores and minerals.

Recommended minimum sample quantities required vs. particle sizes available while the samples collected from host material for analysis:

Sl. No.	Particle/ Grain Size (mm) of the Population	Field Sample Description	Minimum Sample Size (kgs)			Laboratory Process
			for Base Metals, Trace and Rare Earth Elements	Fire Assay for Gold	Fire Assay for Platinum Group Elements	
1	<0.10	Pulverised & Homogenised Representative Sample	0.050	0.100	0.100	Dry & Homogenise before Analysis
2	0.1 - 1	Crushed, Homogenised & Divided Representative Sample	0.250	0.100	0.100	Dry, Homogenise and Pulverise the Entire Sample to <0.075 mm)
3	1 - 2		0.400	0.400	0.400	
4	2 - 5		1.0	1.0	1.0	Dry, Crush the entire sample to <2mm, Homogenise and Rotary Divide to ~500g and Pulverise the Entire 500g Sample to <0.075 mm)
5	5 - 10		2.5	2.5	2.5	
6	10 - 25	Grab Sample/ Field Sample	4.0	4.0	4.0	Dry, Crush the entire sample to <10 mm, Homogenise and Rotary Divide to ~2.5 kg, Crush the entire 2.5 kg of divided sample to <2mm, and Pulverise the Entire 500g Sample to <0.075 mm)
7	25 - 50		6.0	6.0	6.0	
8	50 - 100		10	10	10	
9	>100		10	10	10	

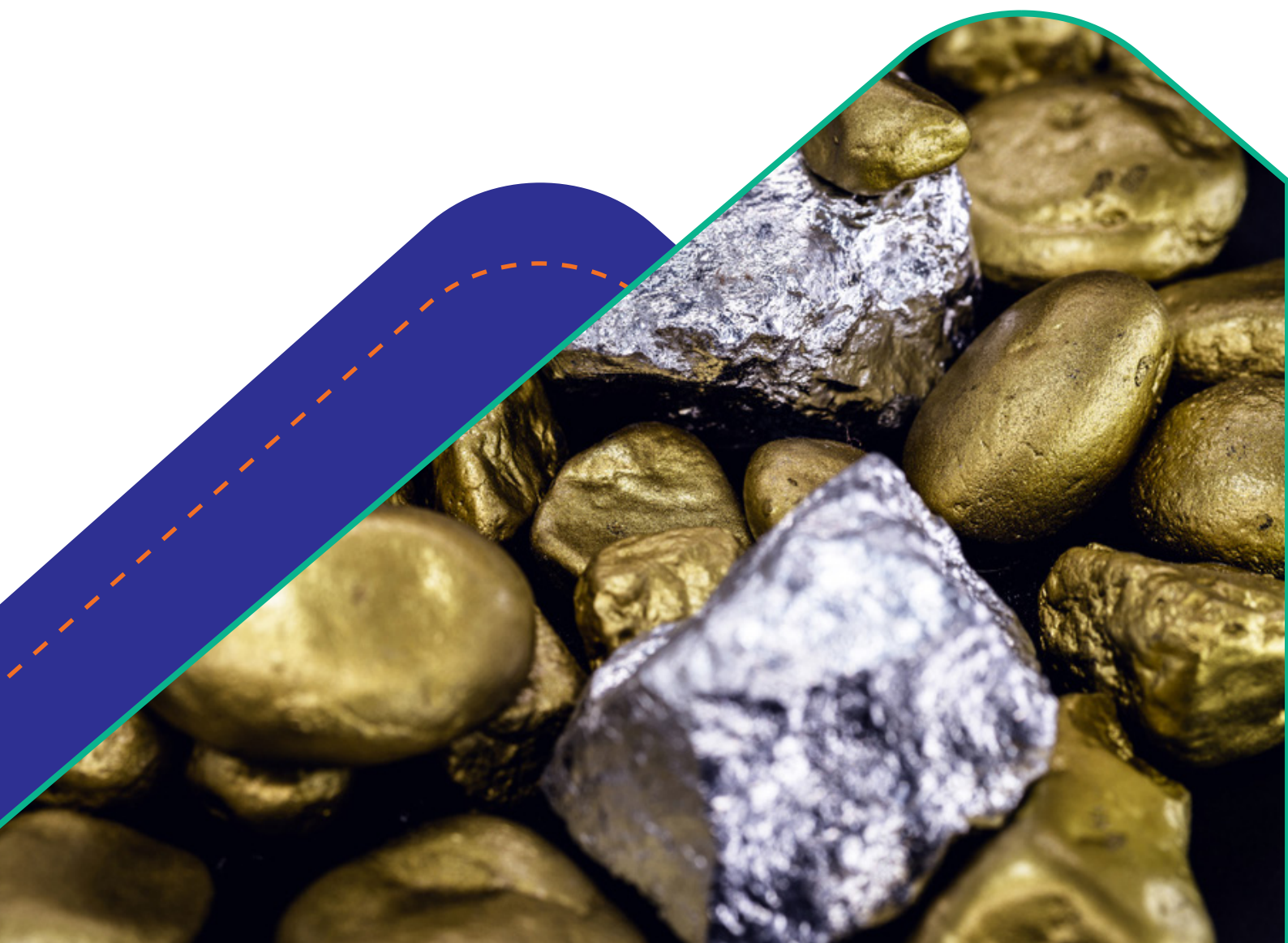
METHOD PACKAGES AND LIST OF THE ELEMENTS

Whole Rock Analysis/Major Oxide Package

SOP/OM/105 _ Lithium Borate Fusion Bead Preparation followed by 4.0 KW WD_XRF Finish.

Elements Tested: SiO₂, Al₂O₃, Fe₂O₃, TiO₂, Cr₂O₃, MnO, CaO, MgO, Na₂O, K₂O, P₂O₅, SO₃ & LOI (LOQ: 0.01%); few trace elements also can be added to this package on request are BaO, SrO, ZrO₂, V₂O₅.

X-Ray Fluorescence Spectroscopy has been widely used in Geochemical Exploration testing for over 75 years. Shiva Analyticals India uses Borate Fusion Bead preparation followed by 4.0KW WD XRF Finish to ensure no interference from the mineralogy of the samples by fusing and converting to a glass bead; which is one of the unique analytical techniques determine SiO₂ and Na₂O simultaneously, accurate, very precise, cost effective, provide quick turnaround and also complete in terms of accounting almost 100% of the chemical composition of the major elements.



Multi Element Geochemical Analysis



Atomic Absorption Spectrometer



Inductively Coupled Plasma Optical Emission Spectrometer



Inductively Coupled Plasma Mass Spectrometer

The current available analytical instrumentation techniques (AAS, ICP OES & ICP MS) and associated sophisticated software can simultaneously determination dozens of elements in a single run of 2 – 5 minutes. These techniques are very rapid, accurate and cost effective. The samples which are aspirated to these instruments shall be in solution form and shall completely dissolve the metals of interest.

Knowledge of the solubilisation properties of the metals including advantages and disadvantages of each of the acid digestion or fusion method packages (proper combination of the acids & fluxes) helps to get complete dissolution of the metals of interest and provide a reliable test data of the resource. List of the metals have the limited solubility or affinity to precipitate during the digestion, fusion and dissolution are Al, Ba, Cr, Hf, Mn, Mo, Nb, Pb, S, Sb, Sn, Si, Ta, Ti, Pb, W, Zr. List of the metals which are volatile and which are partially or fully lost during the digestion, fusion and dissolution are As, B, Bi, Ge, Hg, Pb, S, Sb, Se, Si, Sn, Te.

The test method packages which are offered by Shiva Analyticals India Private Limited ensures each of the metal reported is completely extracted into the solution and the results reported are true representative of the samples submitted to us. We offer the method packages which are thoroughly validated on multiple matrices. We re-validated the extraction method package offered to the customer on the samples submitted by cross verifying a portion of samples by use of alternative extraction methods and alternative instruments without adding any additional cost to the customers. Our test packages do not include any hidden costs such as additional charges for the want of alternative digestion or fusion methods or use of alternative instruments for over ranges or under ranges of the elements.

MULTI-ELEMENT METHOD PACKAGES OFFERED

Aqua Regia/ 2 Acid Digestion is a strong oxidising mixture of acids suitable to dissolve many base metals and sulphide ores. Best suited for reconnaissance and path finding studies as this method facilitates the use of higher sample size 0.25g up to 50g so that ppb and ppt levels of the detection is feasible. 2 acid digestion is low temperature digestion which is preferred for volatile and sensitive elements.

1. 2 Acid Digestion and ICP OES Finish (OM/059): Volatile and Heat Sensitive Elements (Ag, S, As, Bi, Hg, Se, Te) including base metals (Cd, Co, Cu, Mo, Ni, Pb & Zn). The other elements which are part of this package is only for information purpose as their extraction is not complete and only partial/limited in this extraction (are Al, Ba, Be, Ca, Cr, Fe, K, La, Li, Mg, Mn, Na, P, Sb, Sc, Sn, Sr, Ti, V, W, Y, Zr).
2. 2 Acid Digestion and ICP OES & ICP MS Finish (OM/059 & OM/089): Volatile and Heat Sensitive Elements (Ag, S, As, B, Bi, Hg, Se, Te) including base metals (Be, Cd, Co, Cs, Cu, Mo, Ni, Pb, Zn). The other elements which are part of this package is only for information purpose as their extraction is not complete and only partial/limited in this extraction (are Al, Ba, Ca, Ce, Cr, Dy, Er, Eu, Fe, Ga, Gd, Hf, In, K, La, Li, Lu, Mg, Mn, Na, Nb, Nd, P, Pr, Rb, Re, Sb, Sc, Sm, Sn, Sr, Ta, Tb, Th, Ti, Tl, U, V, W, Y, Yb, Zr).

Multi Acid (4 Acid Digestion) is complete digestion methodology for wide range of metal elements. Silica, Boron, Germanium, Mercury are completely lost during the digestion due to the volatile nature of the elements and their reaction products. The other elements which are partially lost during this digestion are As, Sb, Sn, Se, Te. Some of the elements which may have chances to precipitated or hydrolysed during the dilution process are Ba, S, Cr, Hf, Mo, Nb, Ta, Ti, Sb, Sn, W, Zr. Shiva Analyticals laboratory team are knowledgeable and watchful of the signatures of the elements of interest and ensure that all the elements of interest are brought to the solution up to 100% by use of suitable methods and or alternative methods where ever applicable.

3. 4 Acid Digestion and ICP OES Finish (OM/051): Major Elements (Al, Fe, Ti, Ca, Mg, Na, K, P, S, Mn); Minor and Trace Elements (Ag, Ba, Bi, Cd, Cr, Co, Cu, La, Li, Mo, Ni, Pb, Sb, Sc, Sn, Sr, V, W, Y, Zn, Zr)
4. 4 Acid Digestion and ICP OES & ICP MS Finish (OM/051 & OM/052): Major Elements (Al, Fe, Ti, Ca, Mg, Na, K, P, S, Mn); Minor and Trace Elements (Ag, Ba, Be, Bi, Cd, Ce, Cs, Cr, Co, Cu, Ga, Hf, In, Li, Mo, Nb, Ni, Pb, Rb, Sb, Se, Sn, Sr, Ta, Te, Tl, V, W, Y, Zn, Zr); Rare Earth Elements (Cerium (Ce), Dysprosium (Dy), Erbium (Er), Europium (Eu), Gadolinium (Gd), Holmium (Ho), Lutetium (Lu), Neodymium (Nd), Praseodymium (Pr), Samarium (Sm), , Terbium (Tb), Thulium (Tm), Ytterbium (Yb), Thorium (Th), Uranium (U), Scandium (Sc), Yttrium (Y), Lanthanum (La))

SODIUM PEROXIDE FUSION PACKAGE

Sodium Peroxide fusion is widely used geochemical methodology for the complete solution of multi elements including major oxide elements, volatile elements and refractory minerals containing Si, Ti, Cr, Sn, Nb, Ta, W, Sb.

The advantages of sodium peroxide fusion are:

- > Low temperature digestion which does not volatilise the elements As, Bi, Sb & keeps Silica into the solution whereas the other acid digestion methodologies cannot determine Silica along with other metals;
- > single methodology can be used for the analysis of few volatile elements, major oxide elements including silica, minor and trace elements including rare earth elements.

The disadvantages of sodium peroxide fusion are:

- > Sodium cannot be determined by this method and need to use other methods for sodium.
- > High concentration of flux in the solution necessitates the dilution of the samples and hence the detection limits for trace and rare earth elements are much higher than which can be achieved by acid digestion methodologies for some of the elements.
- > Few volatile elements B, Hg, Se, Te cannot be determined by this method.

5. Sodium Peroxide Fusion and ICP OES Finish (OM/056): Major Elements (Al, Fe, Si, Ti, Ca, Mg, K, P, S, Mn); Minor and Trace Elements (Ag, As, Ba, Bi, Cd, Cr, Co, Cu, La, Li, Mo, Ni, Pb, Sb, Sc, Sn, Sr, V, W, Y, Zn, Zr)
6. Sodium Peroxide Fusion and ICP OES & ICP MS Finish (OM/056 & OM/091): Major Elements (Al, Fe, Si, Ti, Ca, Mg, K, P, S, Mn); Minor and Trace Elements (Ag, Ba, Be, Bi, Cd, Ce, Cs, Cr, Co, Cu, Ga, Hf, In, Li, Mo, Nb, Ni, Pb, Rb, Sb, Sn, Sr, Ta, Tl, V, W, Y, Zn, Zr); Rare Earth Elements (Cerium (Ce), Dysprosium (Dy), Erbium (Er), Europium (Eu), Gadolinium (Gd), Holmium (Ho), Lutetium (Lu), Neodymium (Nd), Praseodymium (Pr), Samarium (Sm), , Terbium (Tb), Thulium (Tm), Ytterbium (Yb), Thorium (Th), Uranium (U), Scandium (Sc), Yttrium (Y), Lanthanum (La))

VOLATILE HYDRIDE ELEMENTS PACKAGE

Arsenic as As, Bismuth as Bi, Selenium as Se, Antimony as Sb, Mercury as Hg are hydride forming elements determined by low temperature aqua regia digestion and AAS Finish (OM/084). This methodology has much better detection levels (0.10 mg/kg to 0.20 mg/kg) when compared with flame AAS and ICP OES and a lower alternative to ICP MS multi element option.

PRECIOUS METAL ANALYSIS PACKAGES



Precious metals required to be determined to sub ppm and ppb levels. Lead fire assay pre-concentration method is followed over a century for the determination of precious metals Gold, Platinum and Palladium. About 50g of sample is used for Fire Assay precious metal pre-concentration process; unlike acid digestion multi element base metal methodologies where a fraction of a gram of sample is used for the acid digestion and assay of dozens of metals in ppm levels. Lead fire assay and by AAS finish can achieve (0.01 ppm, 10 µg/kg), by ICP OES finish can achieve (0.005 ppm, 5 µg/kg) and by ICP MS finish can achieve (0.001 ppm, 1 µg/kg). Shiva Analyticals India has 3 decades of track record for fire assay with the capacity of about 1000 fire assay analysis a day coupled with the state of the art sample preparation facility.

- 7.** Gold as Au by Lead Fire Assay and AAS Finish (OM/058, LOQ: 0.01 mg/kg)
- 8.** Gold as Au by Lead Fire Assay and ICP OES Finish (OM/053G, LOQ: 0.005 mg/kg)
- 9.** Gold as Au by Lead Fire Assay and ICP MS Finish (OM/057G, LOQ: 0.001 mg/kg)
- 10.** Gold as Au, Platinum as Pt and Palladium as Pd by Lead Fire Assay and ICP OES Finish (OM/053P, LOQ: 0.005 mg/kg)
- 11.** Gold as Au, Platinum as Pt and Palladium as Pd by Lead Fire Assay and ICP MS Finish (OM/057P, LOQ: 0.001 mg/kg)
- 12.** Gold as Au by Cyanide Leach and AAS Finish (OM/095, LOQ: 0.05 mg/kg)
- 13.** Platinum Group Elements (Pt, Pd, Rh, Ru, Ir, Os) by Nickel Sulphide Fire Assay and ICP MS Finish (OM/055, LOQ: 0.001 mg/kg)

SINGLE ELEMENT PACKAGES

Shiva analyticals offer several tailor made multi element packages which include various physicochemical /wet chemistry methods like Specific Gravity, Bulk Density, Apparent Density, FeO, Cl, F, C_Graphitic, C_Organic, C_Inorganic, SO₄, SO₃, S__Sulphide, S_Elemental, Iodine, Bromine etc.

Specific Gravity/Relative Density, Bulk Density/Apparent Density: These physical properties are dependent on the form of the sample and form of the sample on which the testing is performed. We shall be very clear on the form of the sample (Original Rock form or Powdered form) on which these properties are to be determined. The specific gravity on the original rock form cannot be repeated once the sample is pulverised.

- 14.** Specific Gravity by Water Displacement Method (OM/125): This test is done on the original rock samples received in the laboratory on as it is form. $\text{Specific Gravity} = \frac{\text{Weight of the Dry Sample, g}}{\text{Weight of the Water, g}}$. The specific gravity is a unit less value.
- 15.** Bulk Density by Water Displacement Method (OM/126): This test is done on the original rock samples received in the laboratory on as-it-is form. $\text{Bulk Density (g/ml)} = \frac{\text{Weight of the Dry Sample, g}}{\text{Volume of the Water, ml}}$.
- 16.** Specific Gravity of Powder Samples by using a suitable Solvent and Pycnometer (OM/127): This test is done on fine powdered analysis samples. The value of specific gravity on the pulverised samples differ from the values obtained on the original rock form and hence the laboratory shall be informed before the samples are crushed and pulverised.
- 17.** Bulk Density (g/ml) of Powder Samples by volume measurement (OM/128): This test is done on fine powdered analysis samples. The value of Bulk Density on the pulverised samples differ from the values obtained on the original rock form and hence the laboratory shall be informed before the samples are crushed and pulverised.
- 18.** Bulk Density (kg/m³) of a consignment (OM/129): Bulk Density value measured under this requirement differs from the values reported under sl.no. 15 and sl.no.16. This parameter requires > 200 kg of the representative sample. Stowage factor (1/bulk density) is used in the shipping industry to determine the amount of cargo that can be stored in a given space.
- 19.** FeO (Ferrous Oxide): Ferrous Oxide is determined by Oxidation and Reduction Method (OM/096)
- 20.** Chloride as Cl (OM/050): Determined by aqueous extraction and Mohr Titration or nitric oxide extraction and Volhard Titration.
- 21.** Fluoride as F (OM/075): Fluoride is determined by Distillation and Specific Ion Selective determination.
- 22.** Forms of Sulphur (total sulphur content provides an overall measure of sulphur presence, understanding the distribution and forms of sulphur within a material is vital for predicting behaviour during processing, assessing environmental impact, ensuring product quality, and meeting regulatory requirements). Total Sulphur (OM 092), Sulphide (088), Sulphate (076).

23. Carbon (In minerals, carbon can exist in various forms, including organic carbon, inorganic carbon, and elemental carbon. Each form has its own significance and requires specific analytical methods for accurate determination) Organic (OM/084B), Carbon Total (OM/084C), Carbon Inorganic/Graphitic (OM/084A).

24. Copper by Electro gravimetry (OM/130): Electro gravimetric determination of copper is a technique used to quantitatively analyze the concentration of copper ions in a solution by quantitative depositing the copper as solid metal onto an electrode under the influence of an electric current. Electro gravimetric determination is accurate & selective for copper ions, meaning it can specifically measure copper concentration in the presence of other metal ions. This selectivity is important in complex samples where accurate quantification of copper is required.

MINERALOGY PACKAGE

X-ray diffraction (XRD) and petrology studies are both valuable techniques used in geology and materials science for analyzing minerals and rocks, but they serve different purposes and offer unique advantages. Here's how XRD is superior to petrology studies in certain aspects. XRD excels in identifying crystalline minerals present in a sample. It provides precise information about the crystal structure and lattice parameters of minerals, which can be challenging to ascertain solely through petrological observations. XRD allows for quantitative analysis of mineral phases present in a sample, providing accurate estimates of mineral composition based on peak intensities. Petrology studies, while descriptive, may not always provide quantitative data on mineral abundance. XRD is highly sensitive and can detect trace amounts of minerals present in a sample, even at concentrations as low as a few percent. Powder Diffraction (XRD) Database, contains a comprehensive collection of more than 6000 diffraction patterns for various materials. Researchers use this resource for identifying unknown substances, confirming crystal structures, and conducting material characterization. Shiva Analyticals team has decades of experience on XRD studies. Accurate chemical assay coupled with reliable mineralogy information is vital in resource characterisation.



LITHIUM TESTING SERVICES BY SHIVA ANALYTICALS



Shiva Analyticals, part of the Cotecna Group, offers one of the largest exploration testing facilities. The lab offers lithium testing services such as sample collection, logistics, crushing, drying, division, pulverization and testing. We handle samples including Stream Sediments, Soils, Rock Chips, Rocks and Drill Cores.

Shiva is based in India and has been operating in India and Africa for over 3 decades. To ensure a quick turnaround while protecting sample integrity, Shiva partners with Cotecna offices across Africa for sample collection and testing. In addition, Shiva works closely with Cotecna South Africa and its Richards Bay laboratory.

Global lithium production has increased by about 40% during the year 2023 when compared to the year 2022. The production during the year 2023 is close to 1 million MT. Contrary to the increase in the production the prices of lithium products are soared by 100% during the same period. The lithium exploration and mineral beneficiation activity is ramped up to meet the sudden surge in the lithium demand.

The lithium is extracted from the following 3 major sources:

1. Underground Lithium Containing Brine Reservoirs (Global Production ~64%)
2. Lithium Bearing Clays (lepidolite (polyolithionite, trilithionite), zinnwaldite, masutomilite, swinefordite, hectorite, cookeite and jadarite); Lepidolite and zinnwaldite have Li_2O levels of 1.2–5.9% and 2–3%, respectively. (Global Production ~7%)
3. Mica Schist (Lepidolite) & Pegmatite Rock Formations (spodumene (5.8–8.1% Li_2O)) (Global Production ~29%)

Occurrence of the Lithium rich Brine Reservoirs and Lithium Bearing Clays is limited to certain geological regions and hence hard rock pigmetite formations are targeted globally as part of the lithium exploration projects. The following 5 mineral phases are more focussed as viable sources for the lithium extraction.

1. **Spodumene [$\text{LiAlSi}_2\text{O}_6$]**: a pyroxene mineral that typically contains between 5% and 6% lithium oxide (Li_2O) and is the primary source of lithium in hard rock mining operations.
2. **Petalite [$\text{LiAlSi}_4\text{O}_{10}$]**: a lithium aluminum silicate mineral that contains between 3% and 4% lithium oxide and is found in granitic pegmatites.

3. **Lepidolite** $[K(Li,Al,Rb)_2(Al,Si)_4O_{10}(F,OH)_2]$: a lithium mica mineral that typically contains between 3% and 4% lithium oxide and is found in granite pegmatites and some lithium-rich granites.
4. **Amblygonite** $[(Li,Na)AlPO_4(F,OH)]$: a phosphate mineral that typically contains between 4% and 6% lithium oxide and is found in pegmatites.
5. **Jadarite** $[LiNaSiB_3O_7OH]$: a borosilicate mineral discovered in 2007 in Serbia that contains high concentrations of lithium and boron. A sodium-lithium-borate-hydrate mineral that was recently discovered in Serbia and is currently being evaluated as a potential new source of lithium

The extraction process for Li from mineral forms like spodumene, lepidolite, petalite and zinnwaldite is well established. Of the approximately 140 known minerals containing lithium, only 4, that is, Lepidolite ($KLi_{1.5}Al_{1.5}[Si_3O_{10}][F,OH]_2$), Spodumene ($LiO_2 \cdot Al_2O_3 \cdot 4SiO_2$), Petalite ($LiO_2 \cdot Al_2O_3 \cdot 8SiO_2$) and Amblygonite ($LiAl[PO_4][OH,F]$) are known to occur in quantities sufficient for commercial interest as well as industrial importance.

Characterisation of the Lithium resources in terms of levels of Lithium (Li_2O) and associated elements like SiO_2 , Al_2O_3 , Na_2O , K_2O , Rb_2O , P_2O_5 , Nb, Ta and other minor, trace and rare earth elements by using ICP OES and ICP MS techniques followed by mineralogy by using XRD technique play a major role in planning the beneficiating and extraction process. The exact estimate of lithium content plays a key role as the levels of lithium in these minerals is low and beneficiation and extraction process are very cost intensive.



Shiva Analyticals India Private Limited has been supporting the Geological Exploration, Mining and Trade for the last 3 decades in the sample preparation and testing of ores and minerals for the customers from India, Africa, Middle East and Australia. The ISO/IEC 17025: 2017 accredited facility of Shiva Analyticals has been supporting the Lithium exploration fraternity in terms of very precise and quick estimation of Lithium and other associated elements and also providing the support on XRD investigation for the type of the mineralogy of the Lithium resource.

STRONGER
Together



SHIVA
Part of the Cotecna Group

CONTACT DETAILS

SHIVA ANALYTICALS (INDIA) PRIVATE LIMITED,

Plot No: 24D(P) & 34D,
KIADB Industrial Area, Hoskote,
Bangalore - 562 114, Karnataka, India
Tel : +91 80 28015333,
Mob: +91 8296 4220 72 & +91 6366 9234 08
Email : info@shivaaanalytics.com