INDIAN INSTITUTE OF TECHNOLOGY (ISM) DHANBAD

Central Research Facility (CRF)

ANALYSIS REQUEST FORM AND SAFETY DATA SHEET-**ICP-OES**

(Please note that only HF-free sample solutions finally diluted in ultrapure water /1% HNO_3) will be accepted for analysis)

User Type: IIT(ISM)/ External University/National Lab/R & D/Industries

Name of the User	
Email	
Contact No	
Name and Address of the institution/organization	
Name of Guide/PI	
Guide/PI Email	
Guide/PI contact No	

1. **Sample Code/Name:** (attach separate sheet if required)

Sr. No.	Sample Code	Nature of sample (rock/water/organic matter/aerosol/mine dust etc.)

Kindly Tick whichever is applicable for the following

2. Analysis required- Qualitative/Quantitative

3. Details of Analysis required (Expected elements) :

4. Expected Concentration: (specify expected/tentative range of concentration of each element)

5. **Sample Preparation:** Kindly mention the acids used for sample preparation in brief ($HCl/HNO_3/HF/H_2SO_4/H_3PO_4/HClO_4/H_2O_2$).

6. **Sample Nature:** Organic/Inorganic/Magnetic/ Non- Magnetic /Any other characteristic nature (Specify)

7. Description of sample preparation followed: (Provide a detailed description on the steps followed to prepare the sample solutions including the acids ($HCl/HNO_3/HF/H_2SO_4/H_3PO_4/HClO_4/H_2O_2$) used during sample preparation)

8. **Volatile organic compound**: Present/ Absent/NA

9. **Specify the Storage and handling conditions** :Room temp/ Refrigeration

10. **Sample Properties**: Carcinogenic(carcinogenicity level) /non-Carcinogenic/

Radioactive/Explosive/Toxic/Corrosive/Flammable/ Non-flammable / Other (specify)

11. **Stability of sample**: Stable under RTP, hygroscopic, sublimes, Reactive in air/moisture/ light/heat

12. **Whether incompatible with any material**- Yes/No (Specify the materials)

13. **Toxicity**: Hazardous/ Non-Hazardous

14. **Health hazards:** Yes/No (irritant to skin/irritant to eyes/harmful to skin/ toxic if inhaled/toxic if ingested)

15. **First aid measures:** Eye/Skin/Inhalation/ Ingestion/Others(specify)

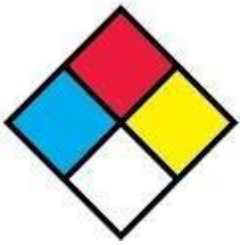
16. **Disposal Method of sample:**

17. **Additional information if any**

18. **All Samples will be discarded within 7 days of analysis. If you wish to collect the samples then you are required to make arrangement for the same. CRF office will not dispatch the same to users under any circumstances.**

19. **MSDS (should be uploaded if available)**

20. Please fill in appropriate numbers in the NFPA diamond if MSDS is available (*Kindly refer the image at the end of the file for reference):



*refer the image below for reference for filling up Point 24:

Declaration

1. **I confirm that the solutions supplied for analysis are HF-free**. Also, I confirm that in circumstances when I have used HF for sample dissolution, the dissolved solute is dried up and again redigested with HCl/HNO_3 to produce desired volume of analytes.
2. **I confirm that the samples submitted for analysis are for research purpose only** and the above furnished details are correct and true to the best of my knowledge. I understand that I will be held responsible for any damages arising from incorrect information provided by me against points 10-15.
3. **I agree to acknowledge CRF, IIT(ISM) Dhanbad** for providing (Instrument name) analytical facility for my research work, in my publications. I also agree to send the publication reference (Journal name/volume number/names of the authors/date of issue of the publication etc) to icp_crf@iitism.ac.in.
4. **I declare that the "Content of this report is meant for our information only and we will not use the content of this report for advertisement, evidence, litigation or quote as certificate to third party**". I accept that all the issued reports/results (Soft/hard) will not carry any Signature or Seal and Stamp of CRF/IIT(ISM) Dhanbad.

Signature of the User

Signature of the In Charge/HOD/PI with College / P.I. / Guide seal / Stamp

Date:
Place:

A general guideline of sample preparation protocol for reference

Rocks. Soils, Ores and Sediments:

These samples can be processed using any one of the following procedures depending upon the requirements of the users and available facilities.

a) Lithium Metaborate Fusion: 0.2 gm of sample and 0.6 gm lithium metaborate taken in a platinum/nickel crucible are heated on a burner till the mixture in the crucible turns into a glassy mass. The crucible is the placed in 30% nitric acid solution and stirred till all the glassy mass dissolves (usually two hours). The solution is then diluted to standard volume with distilled water/ 1% HNO_3.
b) Hydrofluoric Acid Method: 0.5 gm of the sample is taken in a Teflon beaker and 10 mi nitric acid or perchloric acid and 5 ml Hydrofluoric acid are added to it. The solution is heated on a hot plate to dryness. 3 ml of aqua-regia is added to the dry mass and heated till everything dissolves. This step may be repeated multiple time to obtain a clear solution. The solution is diluted to desired volume with distilled water/1% HNO_3. Estimation of Silicon is not possible through this method of sample preparation as silica is lost to the atmosphere as silicon tetra fluoride.
c) Triple Acid Digestion Method: 0.1 gm of sample in taken in Teflon beaker and mixed with $HF:HNO_3:HCl:H_2O$ in 3:3:1:3 proportion. The solution is heated on a hot plate to dryness. 3 ml of aqua-regia is added to the dry mass and heated till everything dissolves. This step may be repeated multiple time to obtain a clear solution. The solution is diluted to desired volume with distilled water/1% HNO_3. Estimation of Silicon is not possible through this method of sample preparation as silica is lost to the atmosphere as silicon tetra fluoride.

Analysis of air samples:

Air samples can be prepared using following procedure. Air is drawn in at a constant rate through a filter (usually made of cellulose ester or glass fiber). The filter is then digested by following methods:

a) For cellulose ester fiber- A weighed filter paper is taken in a beaker, 10 ml nitric acid is added and heated on a hot plate. While still hot perchloric acid is added drop wise till the organic matter is destroyed. The sample is concentrated further by heating. Finally, solution is diluted with distilled water/1% HNO_3.
b) For glass fiber filters – A weighed filter is taken in a Teflon beaker, 5 ml of HF and 10 ml nitric acid added and heated on a hot plate. Few drops of perchloric acid are added and the solution concentrated to dryness. The residue is dissolved in 10 ml of nitric acid and diluted with distilled with distilled water/1% HNO_3.

Water Sample:

Water samples are analyzed for elements like Ca, Mg, Mn, Fe, Zn, Na, K. But other minor elements need concentration. 500 ml of solution is concentrated to 50 ml with warming of 90^0 C. Volatile elements like mercury may get lost at higher temperature.

Organic Samples:

Samples like blood, urine, polymer etc can be analyzed. For this 10 ml of sample is heated with 10 ml of nitric acid on a hot plate and hot perchloric acid is added drop wise till all the organic matter is destroyed and solution becomes clear. Dilution is done with distilled water/1% HNO_3 to desired volume.

Samples of metals and alloys:

These samples can only be digested using nitric acid or hydrochloric acid or the combination of the two (aqua-regia). Crystalline samples that are water soluble can be submitted in their water dissolved form. Organic solvents cannot be accepted because plasma cannot sustain in presence of organic solvent. Hence the organic solvent must be either volatilized or destroyed.

A Minimum of 30 ml o solution is required for estimation of all elements.

Final sample solution should preferably be diluted in 1%HNO_3 solution for instrument efficiency and maintenance.

HF-HNO_3 decomposition method:

This procedure was used for the basalt samples and andesite AGV-1 where results showed good agreement with HF-$HClO_4$ decomposition. Although insoluble fluorides are a well-known problem with REE determinations, basalts generally give good results with a long (48 hour) HF- HNO_3 digestion (e.g., Jenner et al. 1990). Even through this method has been found to be successful for AGV-1, HF-HNO_3 and HF-$HClO_4$ decomposition are generally inappropriate for felsic samples since these often contain accessory zircon which is relatively insoluble. Lithium borate fusion or sodium peroxide sinter are good decomposition methods for these samples. Powdered sample (100 mg) was weighed into a Savillex vial and moistened with ultrapure water. HF (2 ml) and HNO_3 (0.5 ml) were added to the sample, the vial sealed and placed on a hotplate for 48 hours at 130^0 C. At least twice during the first 24 hours, the container was removed from the hotplate, cooled and placed in an ultrasonic bath for two minutes. After 48 hours the vials were opened and evaporated to incipient dryness. Nitric acid (1 ml) was added and further evaporated to incipient dryness. The reside was dissolved in 2 ml HNO_3 followed by 10-20 ml water, transferred to a polycarbonate container and diluted to 100 ml (1000x dilution of sample). Indium internal standard was also added to give a final concentration of 10 ng ml^{-1}. At last, two reagent blank solutions were prepared with each batch of samples.

Additional Instruction to users:

a) Generally, 30 ml of solution is sufficient for estimation of about 10 to 15 elements.
b) For special samples like rocks / ore samples, appropriate standards along with the samples should be submitted by user.
c) Explosive, poisonous samples and samples giving rise to toxic gases/ fumes cannot be undertaken for ICP-AES analysis.
d) The nebulizer of the instrument is made from glass material; hence samples should not have HF in the solution. Excess HF should be evaporated completely or may be neutralized using Boric Acid.
e) For estimation of silica in rock sample following method of solution preparation is generally used:
   (a) 0.5 gram of rock powder + 1.5 gram of Lithium Meta-Borate is fused in a platinum crucible. After the fusion is complete the crucible is kept on a magnetic stirrer with 30% HNO_3 in it, till the whole thing goes in solution. Same method should be followed for preparation of standard solutions.
   (b) 0.5 gram of rock sample + HF + HNO_3 is heated slowly in a Teflon beaker. On digestion evaporate HF (in which case SiO_2 maybe lost) or neutralizer HF with Boric acid.
   (c) For some of the rocks $HClO_4$ and HNO_3 mixture can be used along with HF. One should remember that same method should be used for preparation of standard as well as blank.
f) Turbid solutions and highly viscous samples will not be entertained.
g) For biological samples organic matter should be destroyed using any of the following methods of solution preparation:
   (a) A known quantity of sample is put in a Quartz/ silica crucible and kept in an incinerator at 800^0 to 900^0 C. The ash is then dissolved in aqua-regia and diluted to a known volume using distilled water.
   (b) A known quantity of sample is taken in a beaker, HNO_3 added to it and heated. When it starts boiling $HClO_4$ is added to it drop-wise, and heating continued till all the organic matter is destroyed. The solution is then diluted to a known volume with distilled water.
h) Blank solution for each batch/set of samples should be provided.