



REE Researcher Database Template Instructions

The data collected will be made publicly available through NETL outreach channels such as the NETL REE EDX website (<https://edx.netl.doe.gov/ree/>).

NETL Disclaimer, General Overview and Digestion Procedure Notes:

All results recorded in this template will be made publicly available on NETL's database exchange web portal (<https://edx.netl.doe.gov/ree/>). The focus is to characterize the bulk elemental concentration and mineralogy of rare earth elements in coal and coal by-product material during field sampling in order to provide data needed to determine the best course of action.

The Attributes tab contains definitions and examples that help determine the correct dropdown item/s to select from both of the Field Samples tabs. The Field Samples Dry Mass Basis (DMB) and Dry Ash Basis (DAB) tabs represent the two ways NETL requires the data to be reported. The Instructions tab contains an embedded PDF that provides step-by-step instructions on how to fill out each column within the Field Samples and the Separation – Processing tabs. In order to maintain the NETL database's breadth of data accuracy, researchers should endeavor to accurately and precisely describe all information about the samples that is publicly releasable. Please send all email user questions to NETLREEGROUP@Netl.doe.gov or contact your federal project manager.

Many digestion procedures for analyzing solid coal by-products via inductively coupled plasma mass spectrometry (ICP-MS) or inductively coupled plasma optical emission spectrometry (ICP-OES) analysis (bulk elemental analysis) can be employed. One method employed by NETL-RIC is described below. Note that it is critical that the lab determine the percent recoveries of the rare earths and other elements from certified reference materials to serve as a check on the digestion and analytical procedures. Note as well, that the detection limits of ICP-OES for the rare earths are inferior to those of ICP-MS.

For the NETL digestion procedure, solid samples were prepared by mixing pulverized sample with calcined LiBO_2 (lithium metaborate) at a 1:8 ratio. Prior to fusion, samples were mixed by stirring them with platinum rods in 99.95% platinum crucibles. The open crucibles were heated to 1100°C for five minutes in a CEM - Phoenix microwave muffle furnace, at which point the melt was removed and allowed to cool to a homogeneous glass, which was digested in 5% HNO_3 on low heat while being stirred. The platinum crucibles were rinsed three times with 5% HNO_3 and the samples diluted to a final volume of 100 ml to ensure that the sample glass was completely digested. The samples were further diluted in 2% HNO_3 before analysis to determine the optimal balance between internal standard recovery and limit of detection. Solid samples containing significant carbon content were placed in a LECO Thermogravimetric Analyzer (TGA701) at 107°C for one hour under nitrogen for drying and for five hours at 550°C under normal atmosphere for ashing.

Reference

"Analysis of Rare Earth Elements in Geologic Samples using Inductively Coupled Plasma Mass Spectrometry"

Tracy Bank, Elliot Roth, Phillip Tinker, Evan Granite, DOE Topical Report, available on the NETL REE website - <http://www.netl.doe.gov/research/coal/rare-earth-elements/publications>.

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Template Instructions for Field Sampling (Both Dry Mass Basis & Dry Ash Basis tabs)

Mandatory columns that need to be filled out for “Field Samples” (blue tabs):

Columns	Description
A-C	Sample Ref. #, Sample Type, Description
F-R*	Sample origin and description, Sampling location
AJ-AL	Analysis method/description
BJ	Yttrium concentration ppm
BY-CL	Lanthanides concentration ppm

*Note: Inputs such as *N/A*, *unknown*, or *other* are acceptable, however not preferred.

General Instructions:

Some cells in Row 6 have a red triangle in the right-hand corner. Hover over these cells to reveal additional directions for that column.

Column A: Enter a unique sample reference ID number for the sample that is being analyzed; if running a duplicate of the same sample, enter/write *duplicate* after the unique Reference ID #. If the same sample reference ID number is accidentally entered twice, the cell will highlight yellow to indicate the error. If a sample reference ID number is not added and other cells in the row are populated, the cell will highlight red as a reminder to enter a sample reference ID number.

Column B: Enter generic form of the sample (e.g., core, road cut, or field sample).

Column C: Enter as much detail as possible about the sample.

Columns D-E: If the sample being reported originates from a previously recorded sample (furthest upstream sample), enter the furthest upstream sample Reference # (column A) in column D. For example, if reporting a fly ash sample that was generated from burning coal, and the feed coal had been previously analyzed and reported, enter the coal Reference # in columns D and E. If samples have been collected and measured from three or more upstream process steps (for example, if you are reporting fly ash and have also collected and reported the raw coal from the mine and the clean coal from a prep plant that produced that specific ash), report the Ref # of the raw coal in column D (furthest upstream sample) and the clean coal (previous upstream sample) in column E.

Columns F-N: Refer to “Overview and Explanations” and “Attributes” tabs as reference and record the location or origin of the sample in these columns. For example, where the coal, mine drainage, etc., was mined or formed, not where it was processed, cleaned, burned, treated or disposed of.

Columns O-R: Enter the sampling location. For example, the prep plant, power plant, treatment plant, disposal site, etc.

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Columns S-T: If the sample has a known size fraction, enter that information in column S. Enter the % moisture as-received in column T.

Column U: Enter the % ash after the sample was dried and the moisture was removed. % Ash on a dry basis.

Columns V-Z: If Proximate Analysis was conducted, enter the information in columns V-Z.

Columns AA-AE: If Ultimate Analysis was conducted, enter the information in columns AA-AE.

Columns AF-AI: If CHNS Analysis was conducted, enter the information in columns AF-AI.

Column AJ: Enter the method used to digest the sample, if applicable.

Column AK: Enter the analytical technique used to conduct elemental analysis of the sample.

Column AL: The Basis of analysis reporting (Either Dry Mass Basis or Ash Basis depending upon which tab you are on)

Columns AM-CQ: Enter the concentration(s) of the element sample analyzed in ppm (mass). The reported values should be elemental, not oxides.

Columns CR-CS: These are calculated fields and are the sum of the values entered for Y and the lanthanides (REE).

Column CT: Enter any notes that may be relevant to the analysis, such as potential errors, accuracy, problems, or interesting observations.

Columns CU-DN: If a secondary analysis was conducted on the sample (i.e., ICP-OES) to determine the concentrations of major elements, enter the information in columns CU-DN.

Columns DO-DR: If XRD was run on the sample, enter the major, minor, and trace phases/minerals identified (best judgement) and any notes relevant to the measurement, such as the experimental procedure or interesting observations.

Columns DS-DY: If SEM/EDX was used to analyze the sample, enter the minerals/particles that were identified and their corresponding sizes. For example, monazite was identified and was 5–20 μm in size. Enter any notes that were relevant to the sample or technique.

Spreadsheet Instructions for Separation-Processing (Purple tabs)

If any separation, processing, or characterization was conducted that alters the concentrations of the REEs in the sample, report the analysis in this tab. For example, if density separation was conducted and the concentration of REEs in the heavy and light fractions of a field sample was measured, the data would be entered in this tab

If data is entered in this tab, the following table displays the mandatory columns to be filled out:

Columns	Description
A-C	Sample Ref. #, Sample Type, Description
D-E	Original Field sample Reference # and previous sample reference #
F-H*	Process/separation/characterization steps and description
Z-AB	Analysis method/description
AZ	Yttrium concentration ppm
BJ-BW	Lanthanides concentration ppm
CE	Total weight percent recovery of the sample compared to the original field sample

*Note: Inputs such as *N/A*, *unknown*, or *other* are acceptable, however not preferred.

Columns A-C: Same as Field Sampling Tab.

Columns D-E: Column D denotes the original field sample source reported in the Field Samples Tab column A (Reference #). The Reference # in column D must exactly match the Reference number in column A of the Field Samples Tab to enable the calculations in the spreadsheet for REE+Y concentration factor and REE+Y percent recovery.

Columns F-H: Report the processing/separation/characterization steps used. If multiple steps such as density, size, and magnetic separations were conducted, enter the steps sequentially in column G. In column H enter as much information about the process as possible.

Columns I-AA: Same as Field Sampling Tab instructions for columns S-KK.

Column AB: Report the concentrations on the same basis as the original field sample you have done processing steps on. This will depend upon what tab you select, DMB or DAB.

Columns AC-CD: Same as Field Sampling Tab instructions for columns AM-CQ. The reported values should be elemental, not oxides.

Column CE: Total weight percent recovery of the sample compared to that of the original field sample.

Columns CF-CG: Automatically calculated.

Columns CH-DM: Same as Field Sampling Tab instructions for columns CT-DY.

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