

CLASTIC SEDIMENT ELEMENTAL CHEMISTRY DETERMINATION USING X-RAY FLOURESCENCE SPECTROMETRY

Keck Hydrology BWCA 1988
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INTRODUCTION:

A contact between two major bedrock formations is found between the Quetico Provincial Park in Canada and the Boundary Waters Wilderness in Minnesota. They consist of the leucocratic granitic Vermillion Batholith and a metasedimentary granitic mass, respectively. The contact border lies approximately along the border between the U.S. and Canada. A difference in water quality on either side has been noted, and a theory posed that differing bedrock compositions may be reflected in the water and sediment chemistry.

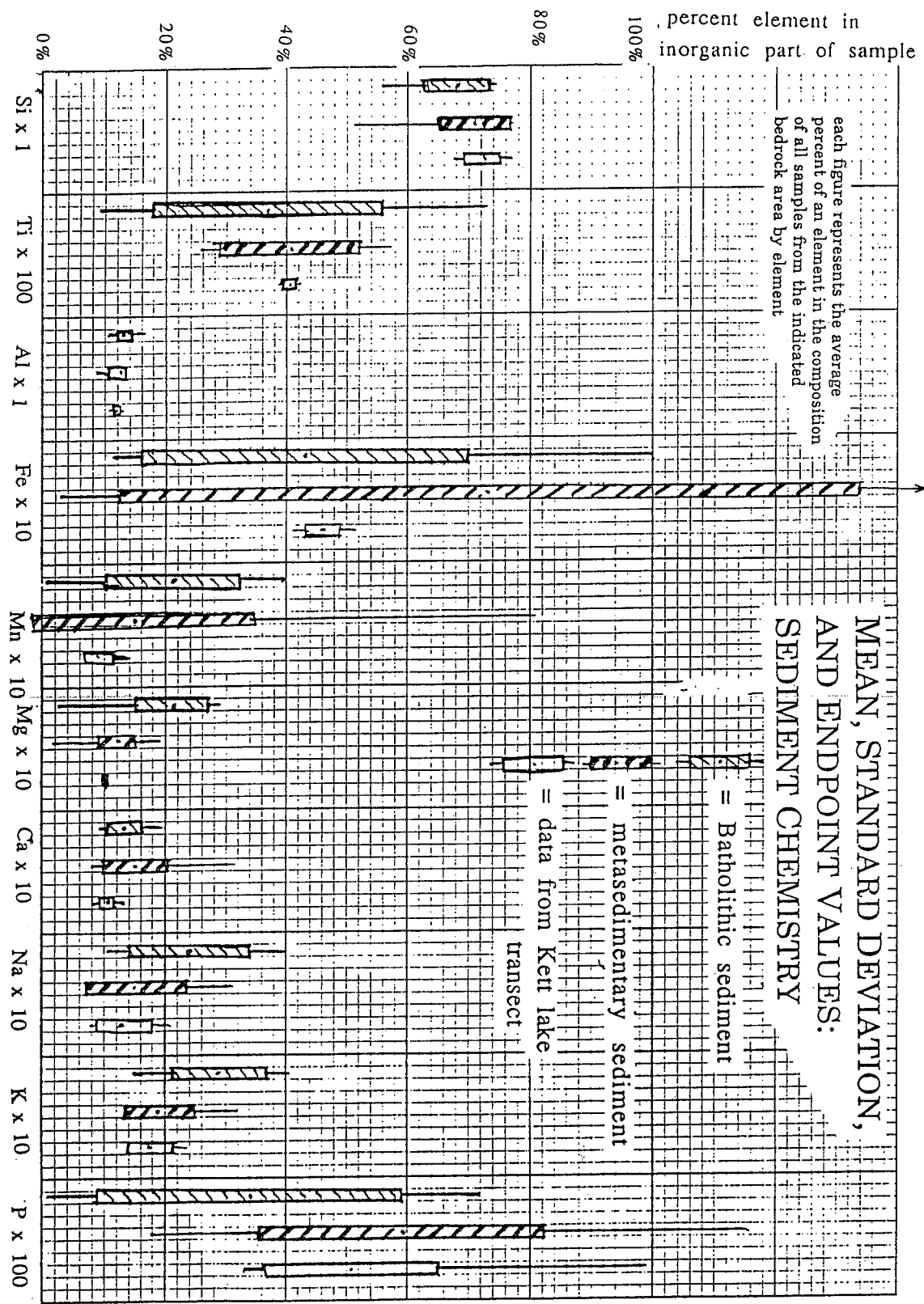
In July and August 1988, the Keck Geology Consortium funded a canoe expedition to the contact zone to study relations between the water, bedrock, and lake sediments. Samples of each from several lakes on either side of the contact were collected for post-field chemistry determination. My part in this effort has been to determine the sediment chemistry, using x-ray fluorescence technique available at Carleton College. I have prepared, as a complement to this abstract, a poster project explaining in detail the method of preparation necessary for determination of clastic sediment composition utilizing XRF technique.

METHOD:

XRF requires that samples be in glass disc form. Clastic sediment, with its higher percentage of unbound water and organic content than normal rocks, must undergo special preparation. All equipment mentioned is in the x-ray lab, Mudd Hall, Carleton College. The mud samples were first dried in glass test tubes in a 250 F drying oven for 8 hours apiece, to remove unbound water. I did not measure water loss, because the water content of a specimen would seem to depend on stratigraphic position, and not on bedrock composition. I would estimate that the average amount of water in specimens was 65-95%. The dried mud was then placed in a weighed ceramic crucible and freed of organics in an 1800 F ignition furnace, and the weight percent of organic material lost was noted. I then mixed what was left of the sample with lithium tetraborate flux, placing it in a platinum crucible over a large air-fed Bunsen burner for 15 minutes until a clear liquid formed. I then cooled it in a disc-shaped mold, and marked it with its sample I.D. number. A few samples were too sandy for fusion, and had to be mashed, ground, and passed through an 80-rated mesh. The glass discs were inserted into the spectrometer for composition determination. Elemental results were reported by the spectrometer computer in the form of oxides, as it is assumed that these major elements will occur naturally in such a state, though the spectrometer actually measured only the element listed.

FINDINGS:

Attached is a graph showing average values and standard deviations from them for the ten major elements tested. These are in percentages of the inorganic part of the sediment sampled. I obtained the mean by determining an average value for the group of percentages for each element in each sample group, sample groups being determined by bedrock type. The standard deviation from the mean indicates the range into which two-thirds of the individual sample values fall. The lines protruding from the deviation symbol show where the highest and lowest readings in a group for that element were. The standard deviations of each of these type groups overlap for most elements, which tells us that the difference in, for example, silicon or titanium levels between sediments over the batholith and sediments over the metasediments is not statistically of interest. The only possibly significant differences in composition between the batholithic- and metasedimentary-underlain sediments are found in Mg, and to a lesser extent in Al, Fe, Na, K, and P. Al and Mg percentages are lower in the batholith. Silicon percentages are basically the same (about 70%) in all samples. All these elements are needed by plants, animals, and microorganisms in the systems, and as the BWCA side seemed more biologically productive, I would say that the ratios and levels of individual elemental percentages in those sediments reflect organic processes in their ecosystems. Elements available in the BWCA may be utilized biologically to a larger extent than those over the batholith.



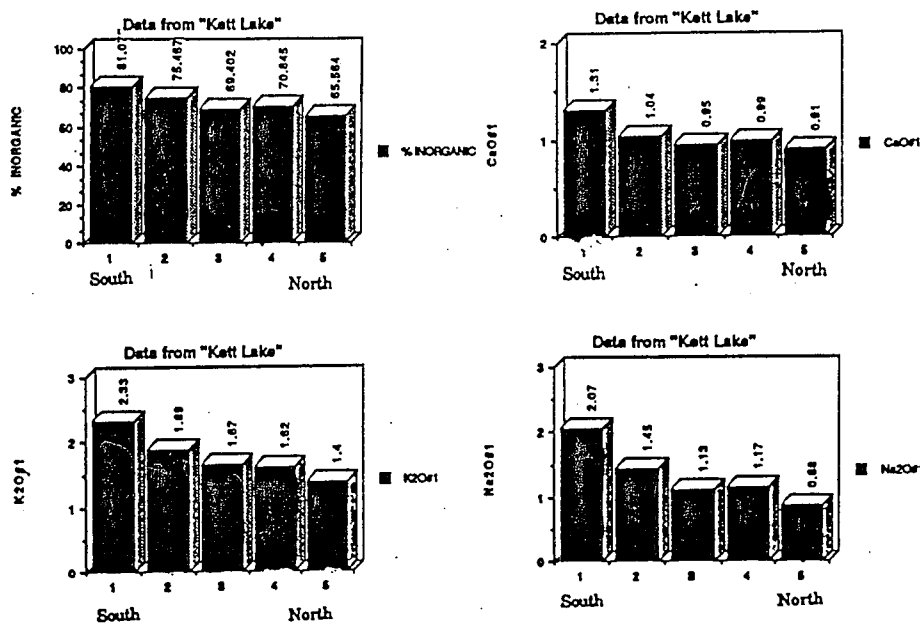
Ca has a larger range of values in the BWCA, and its upper values exceed those of the Quetico. This compliments the determination of more calcium in the water in the BWCA. The difference may be due to the more eutrophic nature of the BWCA lakes; more microorganisms are utilizing Ca in that system.

Higher values and a broader range of values for Fe are also found in the metasedimentary sediments.

These suggest that the BWCA possesses a more diverse chemical makeup than the Quetico. I agree with this; I have noticed more species of trees, more varied soil types, and evidence of a higher level of eutrophication in the BWCA.

KETT LAKE:

Kett Lake is a long narrow North-South oriented lake which straddles the contact. I chose it for a transect to see if there are significant differences in the chemistry within a lake containing both major bedrock types. The North is composed of metasediments, the south of batholith.



K, Na, Ca, and the percent of inorganics all decrease to the northern (metasedimentary) end of the lake, while levels of P, Mn, and Fe rise. Sediments on metasedimentary rock generally have lower Na and K values. The decrease of Ca in the northern end of Kett is in conflict with the general tendency of Ca to be more abundant over metasediments. Perhaps sedimentary calcium depends to some extent on the environment of sediment deposition: the north end of Kett is deeper by several meters and narrower than the relatively Ca-rich south, and Batholithic lakes (Ca-poor) tended to be deeper than their metasedimentary neighbors. There was also a higher percent of organic matter in the north, which also supports the theory that the local environment of deposition may have been similar to that of a batholithic lake, in which organic content was often lower.

There is a jump in the value of P, Mn, and Al at the center of the lake, where the contact is assumed to be. Ti and Mg percentages remain fairly constant, but also rise in the middle of the lake. Mg was the only element present in very different levels between the two lake groups, and it is interesting that within Kett its levels are similar over both bedrocks. Perhaps, Kett lake being, I assume, a basically closed system, a certain amount of mixing and redistribution of elements may go on which cannot between the other separate lakes studied.

CONCLUSION:

X-ray fluorescence spectrometry is a reliable, accurate method for determination of bulk elemental chemistry of clastic sediment. Samples can be prepared by utilizing a modification of standard technique used on rocks. Organics are unfortunately destroyed in the ignition furnace, and in areas with high organic content (50% or more) one may want to invest time in microscopy. Most of the sediment I worked with were 60-90% inorganic. Fluorescence is a quick method, and extremely accurate. Each sample requires only about 30 minutes in the radiation chamber after preparation, (Prep for each is about 11 hours, but they can be dried and fused in batches.) The spectrometer reports to 0.01%, and can be set up to measure trace minerals.

The chemistry of sediments on either side of the BWCA contact zone, with the exception of Mg, do not differ to a statistically significant degree. They reflect the general chemistry of their areas.

A future study focusing on comparing the ecology of both regions for clearer information as to the physical basis for the biological diversity differences would be interesting.