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Analytical results for B-horizon soil samples, from the International Falls and Roseau 1°X2° quadrangles, Minnesota/Ontario

By

G.O. Riddle,* A.L. Meier,* J.M. Motooka,* O. Erlich,

J.R. Clark,* J.A. Saunders, D.L. Fey,* and T. Sparks

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*U.S. Geological Survey, DFC, Box 25046, MS 973, Denver, CO 80225

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This report presents the results of a geochemical soil survey of the International Falls and Roseau 1°X2° quadrangles, Minnesota/Ontario. Geochemical soil samples were collected as one of several multi-disciplinary studies associated with the Conterminous United States Mineral Appraisal Program (CUSMAP).

INTRODUCTION

In 1985-1987, the U.S. Geological Survey conducted a reconnaissance geochemical soil survey of the International Falls and Roseau 1°X2° quadrangles, Minnesota/Ontario. The International Falls and Roseau quadrangles comprise about 14,000 mi^2 (36,000 km^2) in northern Minnesota, United States, and northwestern Ontario, Canada. A high-latitude humid climate prevails, and the study area is located along the boundary between taiga and prairie. The study is characterized by large tracts of undeveloped forest and swamp that are interspersed with areas of both active and abandoned farmland. Much of the forested land has mixed stands of evergreen and deciduous trees. Willow, black spruce, and/or labrador tea predominate in the swampy areas. Surface access is provided by numerous federal, state, and provincial highways and county and state-forest roads.

Geology

Most of the southern part of the study area is underlain by Archean metagraywacke, migmatite, and granitoid plutons of the Quetico subprovince (Day and Weiblen, 1986; Percival and Williams, 1989). A small area along the southern edge of these two quadrangles is underlain by metavolcanics of the Vermilion district. The Archean Wabigoon volcanic belt underlies the northern part of the region. Four regional shear zones (Figure 1), the Vermilion fault, the Four Town fault, the Rainy Lake-Seine River fault, and the Quetico fault, cross the study area (Day, 1985, 1987, and in press; Day and others, 1987, and in press; Day and others, 1989). Proterozoic mafic dikes, which generally strike northwest, cut the Archean basement (Southwick and Day, 1983).

Subeconomic massive sulfide deposits have been reported in rocks of the Wabigoon volcanic belt, in the Birchdale-Indus area (Listerud, 1976; Ojakangas and others, 1977; Figure 1). The only gold mine in Minnesota, the Little American mine, is located east of International Falls within the Rainy Lake-Seine River fault zone (Figure 1). The Rainy Lake gold district of Ontario adjoins the northeastern part of the study area (Sims, 1972).

Complex glacially derived overburden covers almost all of the bedrock south of the international border in the International Falls and Roseau 1°X2° quadrangles, Minnesota. Two lodgement tills cover the bedrock in most of the area. The Rainy lobe advanced from the northeast and deposited a noncalcareous till and glaciofluvial sediments. Calcareous till and

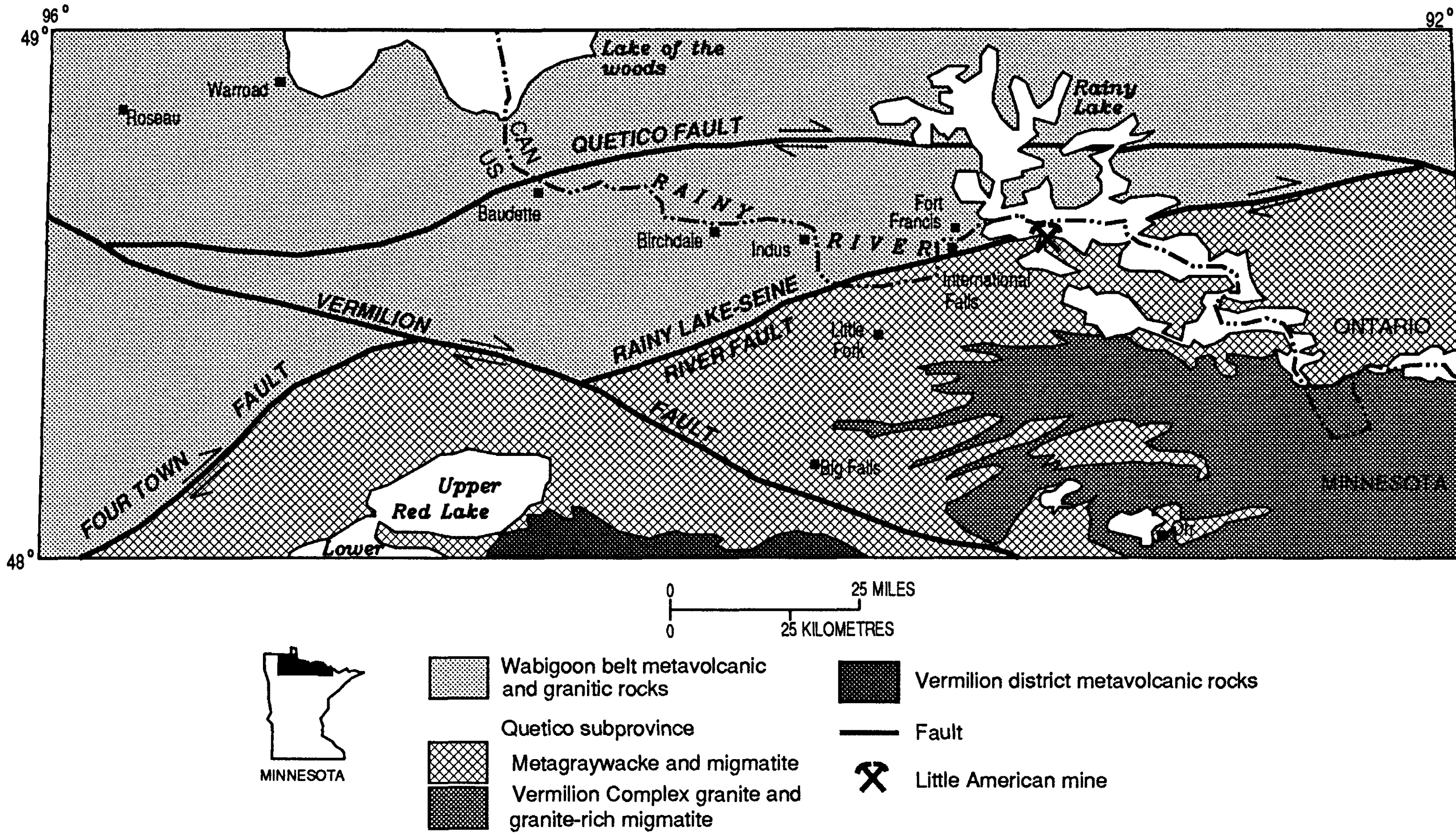

Figure 1. Generalized geological map of the Roseau and International Falls 1°X2° quadrangles, Minnesota (from maps provided by W.C. Day).

glaciolacustrine sediments were deposited as a result of the subsequent advance of the Koochiching lobe from the northwest. Sediments were deposited in proglacial lakes, which formed along the front of both of these lobes. During deglaciation, theBeltrami arm of Glacial Lake Agassiz deposited lacustrine sediments over much of the study area (Wright, 1972; Martin and others, 1988; Horton and others, 1989; Martin and others, 1989). Lake sediment thicknesses in much of the study area range between 1 and 10 meters (G.N. Meyers, oral communication, 1990). Thus, glacial overburden at a given location might consist of two sheets of basal (lodgement) till, multiple layers of outwash, and lake sediments. This can be further complicated by the occurrence of Holocene flood-plain alluvium and peat bogs (Eng, 1980; Horton and others, 1989).

Hummocky terrain with numerous lakes and streams characterizes the eastern half of the International Falls quadrangle. The western half of the International Falls quadrangle and the Roseau quadrangle are part of a largely featureless, poorly dissected, lake bed resulting from Glacial Lake Agassiz. There are a few large lakes, natural drainage is very poorly developed, and part of this area has been described as, "probably the largest uninterrupted wetland in the world" (Wright, 1972). The water table is very shallow throughout most of the study area.

METHODS

Sampling

B-horizon soil samples were chosen as the sample medium for the project. This sample medium was found to produce predictable results, it can be found throughout almost all of both quadrangles, and the work could be performed within the budgetary constraints of the project. Samples were collected at approximate one-mile (1.6 km) intervals, or at sites where B-horizon soils were available, along accessible roads in most of the International Falls quadrangle and the northeastern part of the Roseau quadrangle. Many areas within both quadrangles were so swampy that there was no access, and sample coverage in those areas is poor. Where conditions were more swampy or overburden thickness was generally over 30 meters, an approximate one-mile (1.6 km) spacing was maintained along north-south roads, a direction which transects the regional trend of the bedrock, and an approximate two-mile (3.2 km) spacing was used along east-west roads. A total of 1799 sites were sampled. At each site, the soil profile was exposed, and the sample was collected from the upper 1 foot (0.3 meter) of the first soil horizon that contained visible oxides of ferric iron (the B horizon). At some sites, B-horizon soil material could not be collected, and only A-horizon samples were collected. Data for 1672 B-horizon soils samples is presented in this report. The A-horizon samples were used only in the pilot phase of the study, and were found to not be as reliable a media as the B-horizon soils.

Sample Preparation

Soil samples were air dried and sieved for the minus-0.25 mm fraction (minus-60-mesh fraction, fine sand and smaller). Stainless-steel screens were used for sieving.

Analytical Methods

Pilot studies were performed to determine which analytical methods most clearly defined hydromorphic-dispersion anomalies. Very-weak partial leaching methods were found to give the best anomaly to background contrast in soil samples from the study area (Clark and others, 1990). Each sample was analyzed by three methods, which are briefly described below. In the following summary, the water that was used had been purified to a resistance of 16 Megohm/cm. First, 1 gram of sieved sample material was washed with 15 mL of water for one hour (the "water" leach). In the second method, 1 gram of sample was leached for one hour with 15 mL of 1% (w/v) dextrose in water and 0.1 mL of 5% (w/v) glucose oxidase powder in water (the ENZ leach). In the third method, 1 gram of sample was leached for 24 hours, with 15 mL of 1% (w/v) dextrose and 0.1% ascorbic acid in water and 0.1 mL of 5% glucose oxidase powder in water (the ENZ+ASC leach). After the prescribed leaching time, 10 mL of the leach solution was removed with a pipette and set aside. Leach solutions were immediately stabilized with 0.1 mL Merck Suprapure nitric acid, and 0.1 mL formaldehyde was added to prevent the growth of fungi in the leach solutions. A one microgram terbium spike was also added to serve as an internal standard for the ICP/MS determinations.

Determinations were made by ICP/MS for V, Cr, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Rb, Mo, Ag, Cd, In, Sn, Sb, Te, W, Tl, Pb, Bi, and U. Inductively coupled plasma/atomic emission spectrometry (ICP/AES) was used to determine Al, Ca, Fe, Mg, K, Mn, Na, Ba, La, P, Sr, Ti, and Y. Because each sample was analyzed by three leach procedures, 108 variables were generated for each B-horizon sample. The lower determination limits for ICP/MS are listed in table 1a, and those for ICP/AES are listed in table 1b. Table 2 is a summary of the leaching methods used in this study. A more detailed description of the leaching procedures and instrumental conditions will be included in forthcoming publications.

The data for each leach was scanned for unusually high Pb values. Samples in which high Pb was accompanied by anomalous Sn and/or Sb were assumed to be contaminated with bird shot, and that line of data was deleted from the accompanying tables.

DESCRIPTION OF DATA DISKETTE AND TABLES

The accompanying IBM-formatted diskette contains a self-extracting archive file, INL-ROS.EXE. This text and tables 3-8 are included as compressed files within the archive file. Approximately 6.3 Mb of disk space are required to extract the archive file. Tables 3-8 are included in two file formats; ASCII text files with a .TXT extension and Lotus spreadsheet files with a .WK1 extension. This text is included as a text file (INLROS.TXT) and as a WordPerfect 5.0 file (INLROS.WP5). A separate README file is also on the diskette that provides a description of the INL-ROS.EXE file and the procedure for decompressing the archive. Tables 3 and 4 contain data produced with the water leach, tables 5 and 6 contain data produced with the ENZ leach, and tables 7 and 8 contain data produced with the ENZ+ASC leach. The data are arranged so that column 1 contains the USGS-assigned sample number, and the next two columns list the latitude and longitude for each sample site. The three letter prefix of each sample number indicates the 1°X2° quadrangle within which the sample site is located. Tables 3, 5, and 7 contain the results for determinations made by ICP/MS, and the elements are listed in the order of increasing mass number. Tables 4, 6, and 8 contain the data generated by ICP/AES, and the major and minor elements are listed in alphabetical order followed by trace elements in alphabetical order. In the ICP/MS analyses, if an element was observed but was below the lower determination limit, a "less than" symbol (<) was entered in the tables in front of the lower limit of determination. In the ICP/AES analyses, the letters "ND" in the tables indicates that a given element was looked for but not found at the lower limit of determination shown for that element in table 1b. The values in tables 3-8 are accurate to two significant digits.

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Table 1a. Limits of determination for partial analysis of soils with inductively-coupled plasma/mass spectrometer.

Element	Determination Limit[1]
V (ppb)	1
Cr (ppb)	130
Co (ppb)	2
Ni (ppb)	10
Cu (ppb)	3
Zn (ppb)	20
Ga (ppb)	6
Ge (ppb)	30
As (ppb)	5
Se (ppb)	30
Rb (ppb)	1
Mo (ppb)	3
Ag (ppb)	0.3
Cd (ppb)	1
In (ppb)	0.3
Sn (ppb)	10
Sb (ppb)	3
Te (ppb)	3
W (ppb)	2
Tl (ppb)	0.3
Pb (ppb)	2
Bi (ppb)	1
U (ppb)	1

[1] Determination limit in the soil sample.

Table 1b. Limits of determination for partial analysis of soils with inductively-coupled plasma/atomic emission spectrometer.

Element	Determination Limit[1]
Al (ppm)	4
Ca (ppm)	1
Fe (ppm)	0.8
Mg (ppm)	1
K (ppm)	5
Mn (ppm)	0.05
Na (ppm)	9
Ba (ppm)	0.04
P (ppm)	7.5
Sr (ppm)	0.01
Ti (ppm)	0.2
Y (ppm)	0.02

[1] Determination limit in the soil sample.

Table 2. Chemical methods used.

Leach Method	Reagents	Duration	Phases Leached
Water leach	water	1 hour	Water-soluble salts, ion-exchangeable surfaces
ENZ leach	dextrose glucose oxidase water	1 hour	Amorphous MnO_2 coatings, a small portion of ferric hydroxides, water-soluble salts, ion-exchangeable surfaces
ENZ+ASC leach	dextrose glucose oxidase ascorbic acid water	24 hours	Amorphous and semi-amorphous MnO_2 coatings, ferric hydroxide coatings water-soluble salts ion-exchangeable surfaces