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Distribution of niobium in heavy-mineral-concentrate  
samples from the Charlotte 1° x 2° quadrangle,  
North Carolina and South Carolina

by

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This map is a product of a geochemical survey of Charlotte 1° x 2° quadrangle, North Carolina and South Carolina, beginning in 1978 that is part of a multidisciplinary study to determine the mineral potential of the area. Correlative studies are the completion of a geologic map of the quadrangle and aeromagnetic, aeroradiation, and gravity surveys (Wilson and Daniels, 1980).

The Charlotte quadrangle provides a nearly complete section across the Piedmont: its northwestern corner is in the Blue Ridge, its southeastern corner is over a basin of Triassic sedimentary rocks only a few miles from the Coastal Plain. All of the quadrangle except the southeastern corner is underlain by crystalline rocks of Precambrian and Paleozoic age metamorphosed to greenschist facies in the Slate Belt and to amphibolite facies farther west. Both premetamorphic and post metamorphic intrusive rocks are present. The rocks have been weathered to permeable saprolite reaching depths of 200 feet (60 meters) in the Inner Piedmont. Because of the thorough leaching, most soils are acidic.

In making the geochemical survey, we took samples of sediment within a few miles of the heads of major streams and of the tributaries of these streams keeping the size of the drainage basin small. By doing so, we usually reduce the variety of rocks that contribute detritus to the sample, thus facilitating a correlation between sample composition and the geology of the drainage basin. At the same time we reduce the chance that a localized cloudburst has buried the sample site with sediment from a small part of the drainage basin, thus reducing the validity of the sample as an approximate composite of the rocks of the whole basin. Nevertheless, the samples are not all geologically and geochemically equivalent. For instance, at some sites in the mountainous area in the northwestern part of the quadrangle, many clasts in the stream sediment are several yards (meters) across and collection of fine detritus suitable for a sample required a 1/2-hour search. Not far to the east the finer sediment was abundant.

In the Piedmont, the usual procedure was to sample rather coarse sediment--pebble- or cobble-containing gravel--and to dig deeply to the bottom of the alluvial bed or to a compact clay layer. The coarsest particles in the gravel--boulders, cobbles and coarse pebbles--were excluded from the sample, which then consisted of about 10 lbs (4 1/2 kg) of clay to granule or fine gravel sized material. The heavy minerals were extracted from this unsifted material at the sample site with a gold pan. Samples taken in the same manner on earlier projects were also used to get better coverage of the Inner Piedmont than we would have had otherwise.

The quartz, feldspar, and other minerals of specific gravity below 2.89 were removed from the pan concentrate by floating them with bromoform. The heavy-mineral concentrate cleaned in that way was then separated magnetically into four fractions. The first was removed with a hand magnet, or an equivalent instrument, and not studied. The remaining concentrate was passed through a Frantz Isodynamic Separator at successive current settings of 0.5 ampere and 1 ampere with 15° side slope and 25° forward slope. The material removed from the sample at 0.5 ampere and 1 ampere will be referred to as the M.5 and M1 concentrates or fractions, respectively, and the nonmagnetic material at 1 ampere will be referred to as the NM concentrate or fraction. Most common ore minerals occur mainly in the NM fraction, making them and their contained metals easier to find and to identify. The NM fraction also

contains zircon, sillimanite, kyanite, spinel, apatite, sphene, and the  $\text{TiO}_2$  minerals. It is generally the most useful fraction. The M1 fraction is largely monazite in the Inner Piedmont. Because of interferences caused by cerium during spectrographic analysis and the high content of radiogenic lead in the monazite, it was necessary to remove it from the bulk concentrates to improve the quality of analyses and to permit recognition of lead, possibly derived from ore deposits, in the NM and M.5 fractions. East of the Inner Piedmont the M1 concentrate contained very abundant epidote, clinozoisite, mixed mineral grains, including ilmenite partly converted to leucoxene, staurolite, and locally abundant spinel. The M.5 concentrate contains abundant garnet in the Inner Piedmont, dark ferromagnesian minerals in the Charlotte Belt, and ilmenite in most provinces.

Mineral proportions in each magnetic fraction were estimated using a binocular microscope. Minerals of special interest were identified optically or by X-ray diffraction. The time available did not permit a thorough mineralogic study of all concentrates. Metal-rich minerals were sought in all samples that were shown by the spectrograph to contain metal in unusually high concentrations. After the presence of a metal-rich mineral was established, the variations in metal contents among the concentrates were inferred to indicate variations in the content of metalliferous minerals.

Each fraction was analyzed semiquantitatively for 31 elements using a six-step, D.C. arc, optical-emission spectrographic method (Grimes and Marranzino, 1968). The semiquantitative spectrographic values are reported as one of six steps per order of magnitude (1, 0.7, 0.5, 0.3, 0.2, 0.15, and multiples of 10 of these numbers) and the values are the approximate geometric midpoints of the concentration ranges. The precision of the method has been shown to be within one adjoining reporting interval on each side of the reported values 83 percent of the time and within two adjoining intervals on each side of the reported value 96 percent of the time (Motooka and Grimes, 1976).

The lower limits of spectrographic determination for the elements that are mentioned in this report are: niobium, 50 ppm; beryllium, 2 ppm; cobalt, 10 ppm; and tin, 10 ppm.

All analytical data for sample material other than concentrates are taken from a report by Ferguson (1979). Such sample material is referred to as "silt" in this report.

Most samples were taken by J. W. Whitlow and W. R. Griffiths. Lesser numbers were taken by D. F. Siems, A. L. Meier, and K. A. Duttweiler. The mineral analyses were made by W. R. Griffiths, K. A. Duttweiler, J. W. Whitlow, and C. L. Bigelow, with special mineral determinations by Theodore Botinelly. All spectrographic analyses were made by D. F. Siems, in part from plates prepared by K. A. Duttweiler. Steve McDanal and Christine McDougal were responsible for entering and editing the locality and spectrographic data in the RASS computer file. Many maps were subsequently plotted from this file by H. V. Alminas, L. O. Wilch, J. D. Hoffman, and T. L. Marceau. Most mineral distribution maps were plotted by K. A. Duttweiler.

The only niobium minerals identified in the Charlotte quadrangle are columbite  $[\text{Fe}(\text{NbO}_3)_2]$  and ixiolite (Ta, Nb, Sn, Fe, Mn oxide). Columbite-tantalite is in the pegmatites of the tin-spodumene belt, in the Brown Mountain granite near the northwestern corner of the quadrangle and in the Wilson Creek gneiss just north of the quadrangle, west of Brown Mountain; ixiolite is known only in the granite pluton south of Salisbury. In our separations, columbite is in the M.5 concentrates and ixiolite mainly in M1 concentrate. Some concentrates with high niobium contents may contain niobium minerals that have not yet been identified. The niobium is in part incorporated in minerals of which it is not ordinarily a major component, particularly titanium minerals.

The titanium minerals in the Charlotte quadrangle, as elsewhere, contain varying amounts of niobium. Thus rutile contains as little as 70 ppm Nb in concentrates from the Advance and Olive Branch quadrangles in the Charlotte Belt, and Ashford 7 1/2 minute quadrangle in the Blue Ridge and more than 5000 ppm in concentrates from the Blacksburg North 7 1/2 minute quadrangle in the Inner Piedmont. Rutilites from the Troutman, Boiling Springs South, Blacksburg North, Hiddenite, and Shepherds quadrangles contain 1000 ppm or more of Nb. Inner Piedmont ilmenite from Boiling Springs South, Hiddenite, and Gaffney quadrangles contains 300, 150, and 1000 ppm respectively, thereby contributing Nb to the M.5 concentrates. Sphene contains 100 to 200 ppm Nb.

Some other minerals also contain Nb. Among them are cassiterite from Blacksburg North quadrangle in the Inner Piedmont (1000 ppm Nb), garnet from the same quadrangle (100 ppm Nb) and from the Glen Alpine quadrangle (50 ppm Nb), spinel from the Calahaln quadrangle in the Charlotte Belt (700-1000 ppm Nb), and staurolite from the Blacksburg North Quadrangle (100 ppm).

High niobium values are found in all three magnetic fractions in a cluster at the northern end of the Brown Mountain pluton near the northwestern corner of the Charlotte quadrangle; niobium is lowest in the NM fraction. High to moderately high values are found in and west of the southern part of the Salisbury pluton; again the values are lowest in the NM fraction, but they are higher than those in NM samples collected in the surrounding territory. Niobium in the tin-spodumene belt shows on maps for all three fractions, although the highest niobium values in the different fractions are not necessarily found at the same sample sites.

The nonmagnetic samples from the Inner Piedmont Belt are rather consistently moderately rich in niobium. This probably results from the formation of rutile during metamorphism, incorporating much of the niobium of the rocks. Ilmenite may similarly have incorporated niobium during metamorphism in the Inner Piedmont Belt.

The Charlotte and Carolina Slate Belts yielded concentrates with rather low contents of niobium in most places. A cluster of samples taken southeast of Charlotte and west of the Gold Hill fault zone contains moderately high Nb contents in both the NM and, the M.5 fractions. Samples from the southeast corner of the quadrangle have moderately high niobium contents in the M1 and M.5 fractions, probably as recycled grains from old alluvium.

In general, the niobium contents of the M1 and especially the M.5 concentrates, are most closely related to known sources of niobium minerals.

Niobium in the silt samples shows a prominent belt with about 5 ppm that trends northwest from the southeastern corner of the quadrangle. Within this belt and over the Salisbury pluton the niobium contents rise from about the usual 5 ppm to 35 to 70 ppm, but only over the southern one-half of the intrusive, which Fullagar and others (1971) report to contain abnormal amounts of niobium. An area with 95 to 190 ppm Nb in Iredell and Alexander Counties lies in a region long known to have been rather weakly mineralized, as is indicated by occurrences of quartz crystals (some of them rutilated), rutile crystals, and hiddenite.

Silt in the rest of the Inner Piedmont is generally Nb-poor, as is that of the Blue Ridge Belt, except for a few samples at the southern end of the Brown Mountain pluton that probably reflect the unusual composition of the granite in that pluton. Another cluster of niobium-rich silt samples, with 40 to 60 ppm, south of Albermarle, in the Carolina Slate Belt, is not spacially associated with known geologic features except small mafic intrusive bodies--improbable sources--so these samples may reflect recycled old sediment.

Niobium from the tin-spodumene belt does not show very well in the silts, although its central part yields samples that contain 80 to 185 ppm niobium.

Niobium is accompanied by tin and beryllium in most places within the Charlotte 1° x 2° quadrangle. South of Salisbury the mineral ixiolite contains both tin and niobium. In other areas those two metals are in separate minerals, columbite and cassiterite. Beryllium is not found in significant concentrations in either columbite or cassiterite. Rather unexpectedly, niobium is associated with cobalt near the Salisbury pluton, an association that is yet unexplained.

#### References

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