

**OPTIMIZATION OF NIOBIUM TANTALUM PROSPECTING IN AUSTRIA -
ROUTINES FOR SAMPLING, PREPARATION AND ANALYSIS**

by

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Introduction

Niobium and tantalum are classified as critical raw materials by the European Union and the Austrian government. These metals show an extreme country and market concentration (Herfindahl-Hirschmann-Index (HHI) FeNb & Ta = 8.559), and have to be imported into the EU to 100%. In 2010 Austria imported 1061 t of ferroniobium (FeNb) and 17 t Ta; the latter figure is about 1% of the total world production of Ta (LUIDOLD, 2013). Main ore minerals for niobium and tantalum are columbite and tantalite, which form a solid solution series known as “coltan”-group. About 40% of the global coltan production derives from countries in Central Africa (LINNEN et al. 2014). In these countries coltan is mainly produced by artisanal mining under adverse conditions and might be used to finance military conflicts and civil wars in politically unstable countries for instance in the Democratic Republic of Congo.

Niobium is dominantly used to produce ferroniobium, which is used in high-strength low alloy (HSLA) steels. The light weight and high strength of HSLA steel make it suitable for use in vehicle bodies, ship hulls, railway tracks and oil and gas pipelines. The primary use of Ta is in capacitors, particularly for wireless devices and touch screen technologies (BARDT, 2010). It is also added to superalloys, because of its resistance to high temperature and corrosion and is among other applications used in high-temperature turbines. Furthermore, Ta is biocompatible with human tissue and thus is used in prosthetic joints and pacemakers (LINNEN et al., 2014).

Primary Nb-Ta deposits are mainly associated with carbonatites, alkaline to peralkaline granites and syenites as well as with peraluminous pegmatites and granites (SHAW & GOODENOUGH, 2011; LINNEN et al., 2014). Due to their resistance to chemical and mechanical weathering and high specific gravities tantalum and niobium minerals may also accumulate in alluvial placers.

Coltan in Austria?

As shown in the study on “Critical raw materials for high-technology applications in Austria” there exists a poorly investigated geological potential for coltan in Austria (LUIDOLD et al., 2013). This potential is indicated by the occurrence of spodumene (lithium) pegmatites in some metamorphic units in the Eastern Alps, from which coltan minerals were reported (e.g., at Weinebene). Additionally, the geochemical data sets for stream sediments revealed numerous geochemical niobium anomalies (Ta has not been analysed) within the Eastern Alps and the Bohemian Massif (THALMANN et al., 1989). The geological reasons for these anomalies and their potential use as prospectivity indicators for niobium and tantalum deposits are largely unclear. The ongoing pilot project “Optimization of Nb-Ta prospecting in Austria”, it is funded by the FFG through the FTI initiative “Intelligent Production”, aims for optimizing the exploration methods for coltan deposits and increasing the knowledge about the primary resources of coltan in Austria. In this contribution the sampling strategy, sample preparation and analytical routines, as developed for this project are presented.

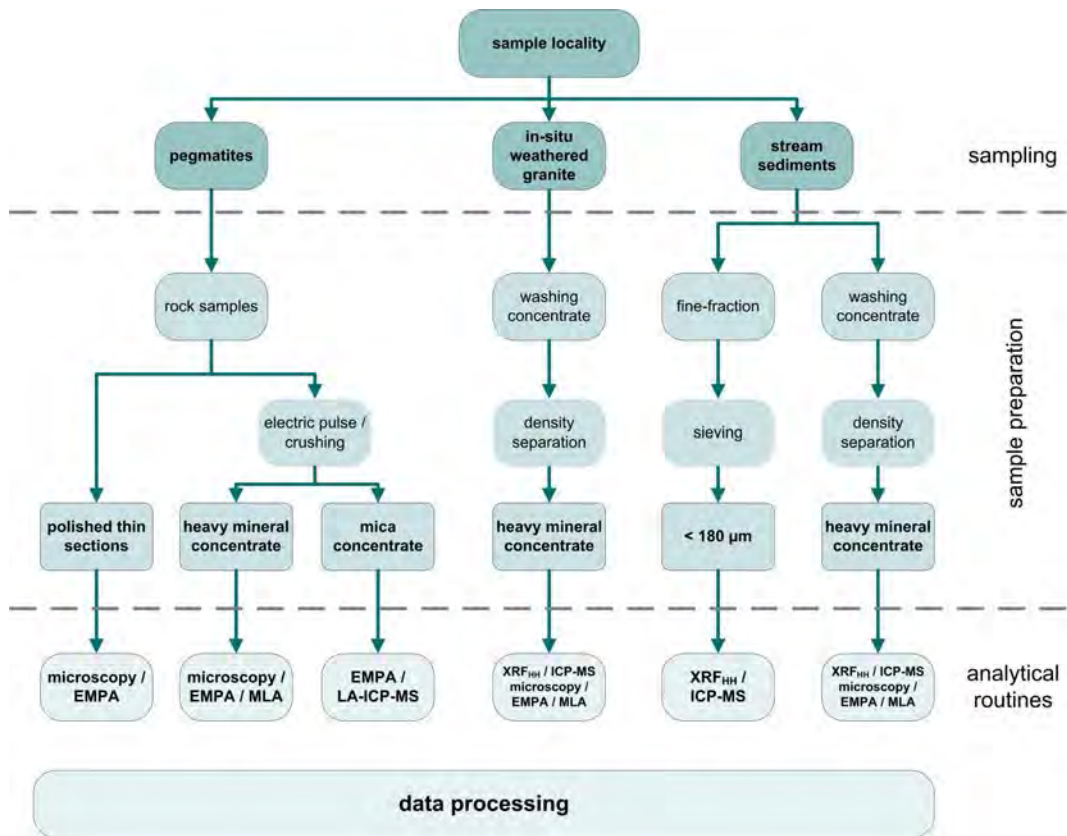


Fig. 1
Overview of preparation and analytical routines applied to the three types of media.

Sampling, preparation and analytical routines

The routines developed for sampling, preparation and mineralogical and chemical analyses are summarized in Fig. 1. For the purpose of verifying and following up known geochemical anomalies and checking pegmatites for Nb-Ta mineralization three types of media were sampled: pegmatite rock, stream sediments (heavy mineral concentrate, fine fraction < 180 µm) and eluvium material formed by the in-situ weathering of granites ("Granitgrus").

Within the Bohemian Massif stream sediments and granite eluvium were sampled in three areas: north of Sandl and south of Weitra (see LEGERER et al., 2014) and in an area north of Heidenreichstein and Litschau. Pegmatite samples were taken at Widy Quarry, Königswiesen and Reinolz.

Studied pegmatite localities within the Eastern Alps include Hohenwart, Lachtal, Mitterberg/Übelbach, Garrach/St. Radegund and the Weinebene spodumene deposit. A few stream sediment samples were collected in the Hohenwart area and around Übelbach. The routines, which were applied for pegmatite samples are reported in more detail in AHRER & RAITH (2014).

The routines as applied to stream sediments and eluvium are as follows: At each sampling site the fine fraction (SF) and a washed heavy mineral concentrate (SM) were collected. The samples were obtained from point bars along stream channels. The SF samples were gained using a small shovel to take the upper approx. 2 cm of the stream bar after removing organic material by dispersing in shallow water. Upon retrieval, the sediment samples were transferred into paper bags, decanted and dried. If possible, multiple subsamples were aggregated to form a single sample for that site. To obtain heavy mineral concentrates, an amount of 15-20 l of active river sediment was collected into a bucket and sieved < 1.4 mm to remove coarser material. Afterwards the sample was wet panned in the field to preconcentrate the heavy minerals and to remove organic matter and clay material. The remaining sample was dried and passed through a 355 µm sieve. To remove remaining minerals of low density, the sample was treated by heavy liquid separation using sodium-poly-tungstate ($\rho=2.95$ g/cm). After careful washing with bidistilled water the remaining sample was split into two halves. One portion was ground in an agate vibrating cup mill prior to chemical analysis, the other one was used for mineralogical studies. Eluvial material was dealt with in the same way as described above for heavy mineral concentrates although the processing started from about the double amount of material (about 30-40 l).

Pegmatite samples were collected considering zonation of pegmatite bodies. Depending on the mean crystal size up to 20 kg of material was collected from each zone to obtain representative samples. To separate heavy minerals from pegmatite matrix minerals the samples were crushed by electric pulse disaggregation and/or a jaw crusher, treated by heavy liquid separation and/or wet panned. Additionally, mica concentrates were prepared from crushed pegmatite material by handpicking.

Polished (thin) sections were prepared from rock samples and heavy mineral concentrates for polarisation microscopy (transmitted and reflected light) as well as for electron microprobe analysis (EMPA; electron imaging, qualitative EDS and quantitative WDS analysis, element distribution maps). EMPA analyses were done using the Superprobe JEOL JXA 8200 of UZAG Steiermark at Montanuniversitaet Leoben. This allowed mineralogical phase identification and mineral chemical analysis of Nb-Ta and associated phases.

In addition, heavy mineral concentrates obtained from stream sediments and pegmatites were analyzed by automated MLA-techniques (heavy mineral concentrates of stream sediments, TU Bergakademie Freiberg/Germany; concentrates from pegmatites, BGR Hannover/Germany). With this technique quantitative information about the mineralogical composition of samples was obtained. Combined LA-ICP-MS and EMPA analyses were applied for analysis of the main and trace element composition of micas. LA-ICP-MS analyses of micas were done at the Department of Chemistry and Department of Earth Sciences, Karl – Franzens – University, Graz using a New Wave UP 213 laser unit combined with an ICP-MS Agilent 7500 ICP quadrupole mass spectrometer. Each processing step of stream sediments and eluvium granite material was monitored by handheld XRF analysis using a Niton XL3t RF Analyzer at the Chair of General and Analytical Chemistry, Montanuniversitaet Leoben. Chemical compositions were then determined by ICP-MS and ICP-ES techniques at AcmeLabs/Canada.

By applying this multi-method mineralogical and chemical approach it could be demonstrated that geochemical anomalies of Nb-Ta-(REE±W±Sn etc.) in the Bohemian Massif reflect different sources and provenance. Whereas some regional anomalies like the one N of Sandl are caused by incorporation of higher contents of Nb and Ta in Ti oxides (see LEGERER et al., 2014), others like the one north of Litschau in the Rottal area are caused by presence of distinct Nb-Ta phases. There, tiny inclusions of columbite-tantalite were observed within cassiterite as well as single larger grains of columbite (Fig. 2a, 2b).

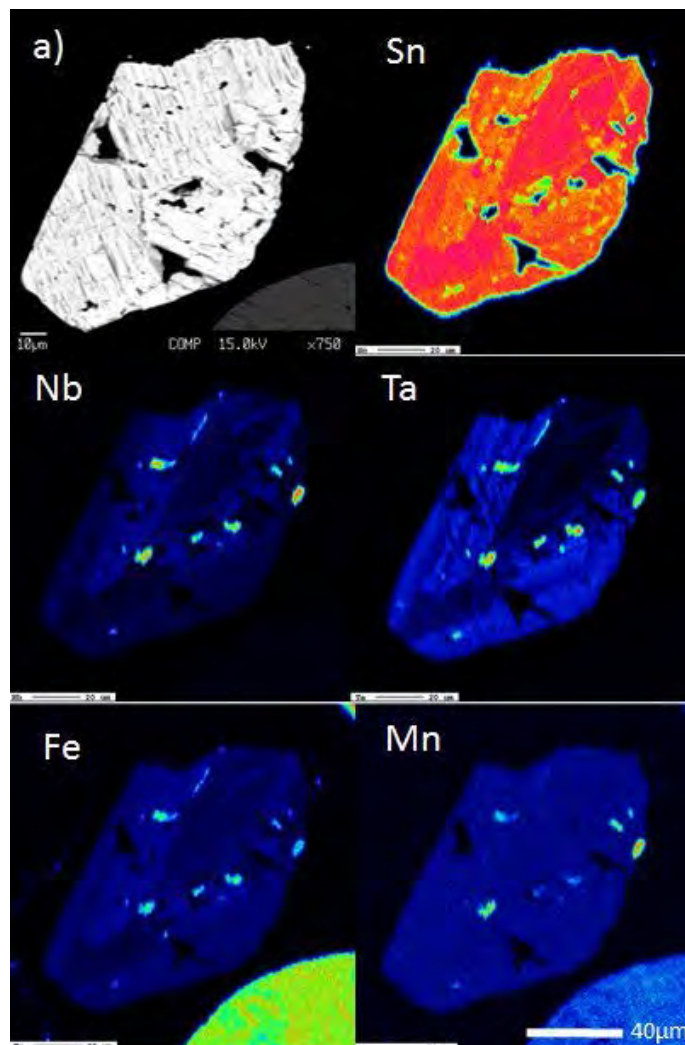


Fig. 2a
Element distribution map.
Cassiterite with tiny inclusions of
columbite-tantalite,
Rottal/Litschau.

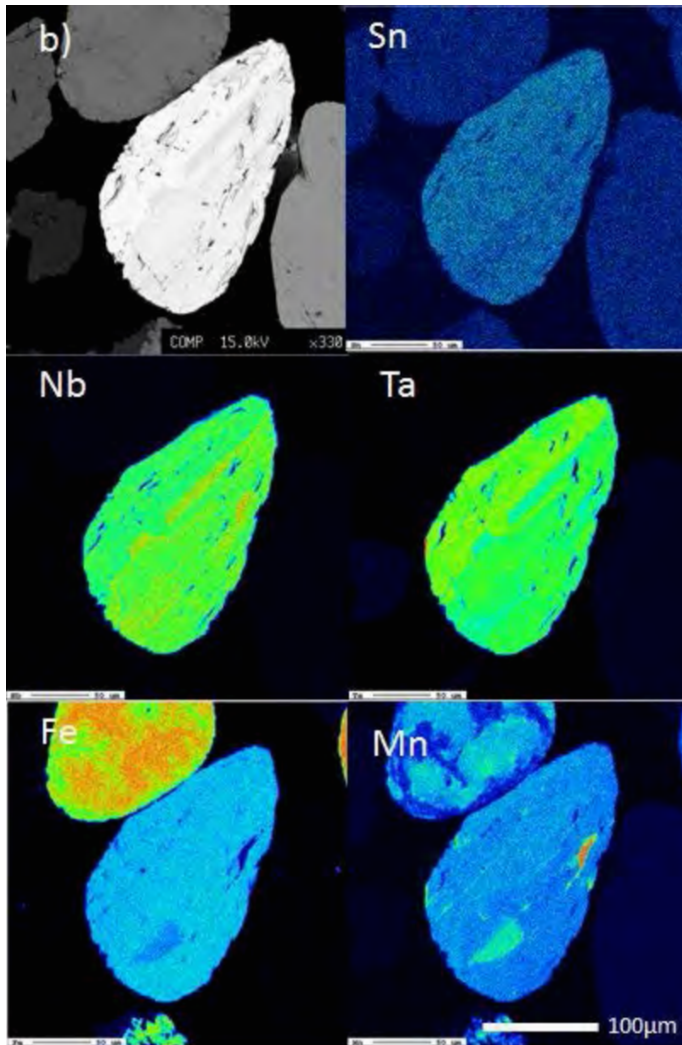


Fig. 2b
Element distribution map.
Larger columbite grain, Rottal-
/Litschau.

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