

Alternative dissolution methods for analysis of niobium containing samples

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ABSTRACT

Inductively coupled plasma optical emission spectrometry was applied for the qualitative and quantitative analyses of the different high purity Nb containing samples such as Nb metal, Nb₂O₅ and NbF₅. Microwave-assisted digestion of the Nb compounds with concentrated H₂SO₄ in PTFE vessels produced Nb recoveries of 99.90 ± 0.08 % for Nb metal, 98.8 ± 0.7 % for Nb₂O₅ and 100.7 ± 0.4 % for NbF₅. Fusion of Nb₂O₅ with Li₂B₄O₇ flux and the subsequent dissolution of the melt with concentrated H₂SO₄ resulted in the precipitation of H₃BO₃ under these conditions. Methanol was added to the reaction mixture to convert the boric acid into the volatile methyl ester in an effort to remove the excess H₃BO₃. This procedure yielded a recovery of 103 ± 2 % for the pure Nb₂O₅ sample.

KEYWORDS

Niobium samples, microwave dissolution, fusion dissolution, qualitative analysis, quantitative analysis, validation.

1. Introduction

Niobium metal has a number of important applications in modern society and its importance is growing in various fields of modern technology, mainly in metallurgy for the production of hard materials, in optics and electronics.^{1,2} It has also been used extensively in the production of jet and rocket engines,³ used in the frame systems for the Gemini space programme as well as in various sophisticated materials such as superconducting magnets in MRI scanners. The combination of its high mechanical strength, high melting point, resistance to chemical attack and low neutron absorption cross-section, renders it as an ideal metal to be used in the nuclear industry. It is also used in the form of high-purity ferroniobium, nickel-niobium and cobalt-niobium super-alloys for manufacturing of missiles, cutting tools, pipelines and welding rods. Niobium alloys are often used in surgical implants due to its non-toxic and non-reactive biological properties.⁴ Along with titanium, tantalum and aluminium, niobium can also be anodized to produce an array of colours which makes it attractive for the production of jewellery, while hydrated niobium pentoxide, with its high surface acidity has proved to be a suitable catalyst for polymerization of alkenes.⁵

One of the critical steps in the development of new niobium products and their uses is the determination of its purity as well as the presence of any harmful impurities. The first step in the wet analytical evaluation of these mineral ores or products normally involves the complete dissolution of these samples. A limited number of dissolution methods are known for the analysis of niobium and tantalum matrices. Many of these involve the use of extremely harsh and sometimes dangerous chemical reagents such as HF^{6–9} and acid mixtures consisting of HF/HNO₃ or HF/H₂SO₄.^{6,10–14} Attempts to accelerate HF dissolution processes^{4,6,10–15} for pure Nb metal and Nb₂O₅ by combining the acid digestion with microwave digestion proved to be very successful with niobium recoveries in the range 95 to 100%.¹² A decrease in the sample preparation stage to 1 hour was also achieved with this alteration.

Flux digestion has proven to be successful in the dissolution of columbite and tantalite ores. Recent research¹⁶ was focused more on the use of less harmful ammonium bifluoride (NH₄HF₂), NaF and KF as the fluoride sources instead of HF. Results obtained for the NH₄HF₂ process indicate that dissolution of more than 90 % of tantalite/columbite, loparite, pyrochlore/microlite minerals and synthetic tantalum concentrates generated from tungsten carbide scrap with 1–3 % Ta and tin can be achieved with this method. Concentrated KOH¹⁷ was found to successfully dissolve low-grade niobium–tantalum ore with recoveries of 94.7 and 93.6 %, respectively. The use of highly concentrated alkalis and HF has the added drawback in that they damage the glass nebulizers in most modern day equipment such as ICPs. The use of lithium metaborate/tetraborate as flux and the subsequent digestion with HF achieved 100 % decomposition of the beach placers and ferrodiorites containing Ti, Zr, Nb, Hf, Ta, Th, and U.¹¹ In another study, Nb₂O₅ was fused with K₂S₂O₇ followed by the extraction of Nb with concentrated tartaric or oxalic acid. This method (K₂S₂O₇ fusion) was found to be successful for the dissolution of pure Nb₂O₅ only and the niobium ore was dissolved with HF.⁶ The resulting digestions have been analyzed by an ICP-MS,^{10–12} ICP-OES,^{6,11–12} NAAS,¹⁸ FAAS or GFAAS.¹¹

Lately, solid sample introduction techniques^{5,19,20} have received greater attention for Nb and Ta analysis due to the fact that they avoid tedious and time consuming digestion steps as well as the prevention of analyte loss and contamination as is the case during acid digestion or flux dissolution. Although the solid analysis methods are quicker with easy sample preparations, digestions can never be avoided if further processing (such as purification) of niobium compounds is required.

The aim of the present study was the development and optimization of alternative, economically viable and ecologically friendly digestion methods for the dissolution of niobium containing samples using the high purity niobium(V) oxide (Nb₂O₅), niobium(V) fluoride (NbF₅) and niobium metal. ICP-OES was selected as the preferred quantification method due to its high sample throughput rate, single set of standards

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Table 1 ICP-OES operating conditions.

R.F. power	1.2 kW
Coolant gas flow	14.0 L min ⁻¹
Plasma gas flow	1.2 L min ⁻¹
Carrier gas flow	0.7 L min ⁻¹
Sample uptake method	Peristaltic pump
Spray chamber	Glass cyclonic spray chamber with concentric nebulizer

needed with standard addition, low level of interference, extensive linear dynamic range and multi-element detection capability.

2. Experimental

2.1. Apparatus

A Shimadzu ICPS-7510 ICP-OES sequential plasma spectrometer was used for the wet chemical analysis of the samples under investigation. The emission intensity measurements were made under constant conditions indicated in Table 1. An Anton Paar Perkin-Elmer MULTIWAVE 3000 microwave reaction system equipped with an 8SXF100 rotor and eight polytetrafluoroethylene (PTFE) reaction vessels was used for the acid dissolution of samples. Flux fusions were performed in a high temperature oven supplied by Labequip. Grade B volumetric flasks and glassware bought from Merck were used in this study.

2.2. Reagents

Separate standard solutions containing 1000 ppm Th and U were bought from De Bruyn Spectroscopic Solutions while 1000 ppm Nb, 1000 ppm Ta, 1000 ppm W, 1000 ppm Si, 1000 ppm Ti, 1000 ppm Sn and 1000 ppm Y were bought from Merck. The multi-element standard No. XXVI containing 1000 ppm each of Mn, Al, Fe and Ca was also bought from Merck. The high purity niobium foil (99.8 %, 0.127 mm thick), niobium(V) oxide powder (99.99 %) and niobium(V) fluoride powder (98 %) were sourced from Sigma-Aldrich. All the acids (95–98 % sulphuric acid A.C.S. reagent bought from Sigma-Aldrich, 32 % hydrochloric acid, 65 % nitric acid and 85 % ortho-phosphoric acid bought from Merck) used in this study were of analytical grade. Lithium tetraborate was bought from Johnson Matthey Materials Technology, lithium metaborate (>98 %) A.C.S. reagent was bought from Sigma-Aldrich and potassium pyrosulphate and sodium hydroxide were bought from Merck. Double distilled water which was checked for contaminants by qualitative analysis using ICP-OES was used for dilution of the standard and sample solutions.

2.3. Experimental Procedures

2.3.1. ICP-OES Instrument Parameters

The operating parameters of the instrument were continuously monitored to ensure the maximum performance and reliability of the ICP-OES results. All the analyses were performed 20 to 30 min after the spectrometer was turned on to achieve a stable plasma as well as constant and reproducible sample introduction.

2.3.2. Microwave Digestion General Procedure and Parameters

The polytetrafluoroethylene (PTFE) reaction vessels used for this digestion were cleaned by steaming the vessels with 8 mL of 55 % nitric acid in microwave-heated sealed vessels for 15 min followed by rinsing several times with double distilled water and drying of the vessels before use. Niobium samples accurately weighed (to 0.1 mg) in vials were quantitatively transferred into

Table 2 Microwave operating conditions used for the acid-assisted digestion.

Power	1200 W
Pressure	60 bar (maximum)
Temperature	240 °C (maximum)
Acid volume	10 mL

each pre-cleaned vessel and 10.00 mL analytical grade acids were pipetted into each reaction vessel. The vessels were then placed into the ceramic jackets and transferred to a rotor of the microwave digestion system, sealed, tightened and finally taken to the microwave reaction system. The microwave parameters shown in Table 2 were kept constant for all the digestions of the niobium samples. Except for the effect of irradiation time studies, an irradiation time of 45 min was used.

2.3.3. Calibration Curve for Microwave-assisted Acid Digestion Analyses

The calibration curve solutions were prepared by introducing 0.1, 0.2, 0.3, 0.5 and 1.0 mL of the 1000 ppm ICP-OES standard solution/s into five 100.00 mL volumetric flasks using a micro-pipette. To these, 10.00 mL of acid that was used to digest the sample was added. The solutions were then diluted to the mark with distilled water to prepare 1.0, 2.0, 3.0, 5.0, and 10.0 ppm concentrations of the elements of interest. The blank solutions were prepared by diluting 10.00 mL of the acid being used to the 100.00 mL mark of the volumetric flask and were used for background correction.

2.3.4. Standard Addition Calibration Curve for Fusion Digestion Analysis

The standard addition calibration method was used for the analyses of all the solutions that were obtained by flux fusion digestion methods to compensate for potential matrix effects. 10 mL aliquots of a sample solution were added to the five 100.00 mL volumetric flasks followed by a 5.00 mL addition of the acid or base used to dissolve the melt and then the standard solutions were added to make 0.0, 1.0, 3.0, 5.0 and 10.0 ppm standard solutions. The solutions were then diluted to the 100.00 mL volumetric flask mark with distilled water.

2.3.5. Microwave-assisted Dissolution of Nb₂O₅ Oxide

Aliquots of approximately 0.01 to 0.10 g of niobium sample were accurately weighed (to 0.1 mg) into the dry glass vials and quantitatively transferred into pre-cleaned PTFE vessels of the microwave. The microwave dissolution procedure was followed as outlined in Section 2.3.2 for the dissolution of high purity Nb₂O₅, Nb metal and NbF₅. The clear solutions obtained after digestion were quantitatively transferred to 100.00 mL volumetric flasks and diluted to the mark with distilled water for ICP-OES analysis using an external calibration curve.

2.3.6. Fusion Dissolution of Nb₂O₅ using Li₂B₄O₇ as Flux

Approximately 0.1 g of Nb₂O₅ sample was (accurately weighed to 0.1 mg in a platinum crucible) thoroughly mixed with approximately 2.0 g of Li₂B₄O₇ flux in a platinum crucible. The mixture was placed in the high temperature oven, set to 1100 °C, for 30 min. A clear melt was obtained for both fluxes and the hot crucible was immediately placed in a cold water bath to crack the glassy melt to speed up the dissolution process. 10 mL of concentrated H₂SO₄ were added to the cold melt followed by immediate but cautious addition of approximately 30 mL of methanol. The solution was heated to ca. 40 °C with constant

Table 3 Limits of detection at the three most sensitive line orders.

Element	Limits of detection (ppm) at three sensitive wavelengths		
	Line order 1	Line order 2	Line order 3
Nb	0.0094	0.0093	0.0100
Ta	0.0240	0.0047	0.0240
Li	0.0002	0.0100	0.0043
Na	0.0034	0.0066	0.0310
Mg	0.0004	0.0002	0.0200
Al	0.0055	0.0074	0.0130
Ca	0.0004	0.0011	0.0025
Ti	0.0015	0.0015	0.0015
Mn	0.0003	0.0004	0.0007
Fe	0.0035	0.0080	0.0024
Ni	0.0029	0.0083	0.0029
Cu	0.0020	0.0017	0.0058

stirring. The addition of methanol to the solution containing the high boron concentration facilitated the production of the highly volatile B(OMe)₃ ester (see Eq. 2, presence of green flame) and the addition of the methanol led to the gradual disappearance of the white precipitate (H₃BO₃). Both the boron ester (absence of green flame) and the excess methanol were finally removed by heating the solution at elevated temperatures for 90 min.

The solution was quantitatively transferred to 100.00 mL volumetric flask and diluted to the mark with distilled water. The analysis was then performed as indicated in Section 2.3.4.

2.3.7. Determination of Limits of Detection for Selected Elements

Five 100 mL standard solutions containing 0.4, 1.0, 3.0, 5.0, and 10.0 ppm of the elements in Table 3 and the blank solutions were prepared in 10 mL H₂SO₄. The intensity measurements were performed at the three most sensitive analytical lines of each element. The intensities used for the calculation of the LOD are the average of three replicate measurements in the standard solution and ten replicate measurements in the blank solution for each element.

3. Results and discussion

3.1 Analytical Line Selection

The analytical lines for the analytes under investigation were selected carefully to avoid any spectral interference from possible impurities. The line selection was made on the basis of the LOD (Table 3) and the complexity of the matrix of the niobium sample under investigation. The niobium intensity measurements for the pure niobium sample solutions (Nb₂O₅, Nb metal, and NbF₅) prepared by microwave and acid digestion were made at 309.418 nm (line order 1) while 316.340 nm (line order 2) was used for the analysis of solutions prepared by flux digestion. All the experimental LODs (determined by ICP) for the wavelengths selected in this study were below the literature^{21,22} estimated LODs.

3.2. Validation

Validation of the methods for niobium determination was performed on high purity niobium samples dissolved by microwave digestion and fusion digestion. Validation parameters such as accuracy, percentage recovery, precision and sensitivity were all used to evaluate the success of the different analytical determinations.²³ The slope of the calibration curve was used as a measure of the sensitivity of the instrument to the analyte concentrations. The *y*-intercept provides an estimate of the

variability of the intensity measurements as well as acceptable background corrections (zero intercept for the normal calibration curve) made for each analytical determination. These parameters were used to evaluate the effectiveness (accuracy and precision) of the different analytical determinations that were made in this study.

3.2.1. Linearity

Calibration curves were constructed by plotting the analyte concentration (ppm) versus emission intensities of the elements at the selected analytical lines. The calibration curves obtained for the Nb determination using either the microwave or the flux fusion procedure all showed excellent linearity with correlation coefficients (*R*² better than 0.9997). The *t*-test was used to determine the significance of any differences between the obtained *y*-intercept of the calibration curve and the theoretical *y*-intercept. The *t*-value of 1.726 was obtained for microwave digestion of Nb metal which is less than the theoretical *t*-value of 3.182 (*t*_{*n*-2} = 3) at 95 % confidence level. This indicated that the obtained *y*-intercept is not significantly different from zero.

3.2.2. Sensitivity

The sensitivity (slope, *m* of the calibration curve) obtained for Nb was greater than 1.74. ICP-OES measurements of the intensities of selected analytes were made for the calculation of the LOD and limits of quantification (LOQ). The limits of detection were calculated as three times the standard deviation of the analyte intensity obtained from 10 replicates of the blank and devised by the slope, as indicated by the following equation:²⁴

$$\text{LOD} = \frac{k \cdot s_{bl}}{m}, \quad (1)$$

where *k* is a numerical factor chosen in accordance with the confidence level (the value of *k* = 3 which corresponds to a 98.3 % confidence level was used) desired, *m* is the slope of the calibration graph and *s*_{bl} the standard deviation of the blank responses. The LODs for Nb 309.418 nm and Nb 316.079 nm obtained were 0.0094 and 0.0093 ppm, respectively. The detection limits of the elements that were chosen for this study are reported Table 3. The LOQ was calculated as ten times the LOD. All the LOQs obtained are below 1 ppm, which easily allows for trace level elemental analysis for all the elements that were detected in the respective Nb compounds that were studied.

3.2.3. Precision

The precision of the microwave-assisted digestion was evaluated by performing five replicate digestions and analyses. The average recovery results with standard deviation and relative standard deviation are summarized in Table 4. Low RSD values obtained showed good reproducibility of the measurements with this digestion method.

3.2.4. Percentage Recovery and Accuracy

3.2.4.1. Microwave dissolution of Nb₂O₅ with different acids

CRMs for Nb were not commercially available and the recovery

Table 4 Analytical results using the microwave-assisted digestion.

Sample	% Recovery	SD	RSD	<i>t</i> -value
Nb ₂ O ₅	98.8	0.7	0.7	-3.00
Nb metal	99.90	0.08	0.08	2.35
NbF ₅	100.7	0.4	0.4	0.01

Conditions: λ = 309.418 nm, digestion time = 45 min, 10 mL H₂SO₄.

Table 5 Analytical results for % Nb recoveries from different digestion periods.

Sample name	Irradiation time	
	45 min	60 min
Nb ₂ O ₅	98.8	98.3
% Nb recovery	98.8	98.3
SD*	0.7	0.6
RSD	0.7	0.7

Conditions: $\lambda = 309.418$ nm, 10 mL H₂SO₄

*Standard deviations are based on $n = 4$ measurements

of the metal was performed on high purity metal and inorganic samples. The influence of different acids in the microwave dissolution of Nb₂O₅ was studied using 32 % HCl, 95–98 % H₂SO₄, 85 % H₃PO₄, 65 % HNO₃ and *aqua regia*. The microwave digestion period was set for 30 min. Recovery results obtained for HCl, HNO₃ and *aqua regia* were 2.27 ± 0.01 %, 0.012 ± 0.001 % and 0.09 ± 0.02 % Nb, respectively. More encouraging results were obtained for H₃PO₄ (84.3 ± 0.1 % Nb) and H₂SO₄ (86.9 ± 0.3 % Nb) which indicated that both H₃PO₄ and H₂SO₄ can be used for digestion. In this study, H₂SO₄ was chosen because of its low viscosity (27 cP) compared to H₃PO₄ (65 cP).²⁵ High viscosity requires larger dilution volumes to ensure efficient flow rate in the ICP nebulizer.

3.2.4.2. Microwave dissolution of Nb₂O₅ with H₂SO₄ at different irradiation times.

The effect of time on the dissolution of 0.10 g Nb₂O₅ with 10 mL of 95–98 % H₂SO₄ in the microwave oven was evaluated by performing independent digestions using 30, 45 and 60 min digestion times. During the 30 min digestion time, a maximum of 86.9 ± 0.3 % Nb recovery was achieved. Increasing irradiation time to 45 min produced a recovery of Nb in excess of 98 % and no further dissolution (98.3 ± 0.6 %) was observed for the 60 min irradiation time (see Table 5). These results clearly indicate that Nb₂O₅ can be completely dissolved after 45 min irradiation in sulphuric acid using the microwave conditions as indicated in Table 2.

3.2.4.3. Microwave dissolution of Nb metal and NbF₅ with H₂SO₄

Nb metal (foil) and NbF₅ were both irradiated in the microwave oven for 45 min using H₂SO₄ as acid. Visual inspection indicated that all the Nb foil and NbF₅ dissolved after 45 min of microwave digestion. The solutions were then quantitatively transferred to the 100.00 mL volumetric flasks and diluted to the mark with distilled water for ICP-OES analysis. The % Nb recoveries obtained on analysis by ICP-OES were all in excess of 99 % Nb which indicates that the use of microwave-assisted dissolution is extremely successful for the dissolution of the metal in H₂SO₄. The reproducibility of the measurements (expressed as RSD) was also very good. This method therefore points to an alternative method for Nb metal and avoids the use of HF in the digestion process. The average recovery of four independent analyses for Nb₂O₅, Nb metal and NbF₅ dissolved by microwave digestion are summarized in Table 4. All the t -values obtained are below the theoretical value of ± 3.182 at 95 % confidence limit for three degrees of freedom which shows good accuracy of the results.

3.2.4.4. Fusion dissolution of Nb₂O₅

Three different fluxes namely NaOH, Li₂B₄O₇ and LiBO₂ were investigated for the fusion digestion of high purity Nb₂O₅. The

contents were stirred until none of the glassy melt was visually observed but a white precipitate formed during this dissolution step. The precipitate was removed by filtration and the solution was analysed by ICP-OES. The % Nb recoveries obtained from this study indicated that the Nb dissolution was the highest for the Li₂B₄O₇ flux (22.7 ± 0.2 %) and the lowest for LiBO₂ (0.51 ± 0.08 %) while NaOH as flux reagent yielded only 9.16 ± 0.09 % Nb recovery. Even the relatively high recovery of niobium (~ 23 %) with Li₂B₄O₇ compared to the other fluxes did not provide a satisfactory recovery of Nb. A change from 3.25 % HNO₃ to 5 % HCl and 5 % H₂SO₄ acids also did not remove the precipitate nor improve the Nb recovery (14.6 ± 0.3 and 4.10 ± 0.06 %, respectively). The white precipitate that forms during the dissolution of the Nb₂O₅/flux melt was identified as H₃BO₃ by means of IR spectra. The IR spectrum of the white precipitate gave stretching frequencies at 3190, 1416 and 1058 cm⁻¹ which corresponds very well with those that were found for a pure H₃BO₃ sample (3191, 1416 and 1048 cm⁻¹) and those cited in literature (3193,²⁶ and 1476–1415 and 1039 cm⁻¹).^{27,28}

In order to try to increase the dissolution of the melt and therefore the recovery of Nb from the dissolution step, the effect of acid concentration, using 0.26, 3.25, 16.25, 32.5 and 65 % HNO₃, on the Nb₂O₅/Li₂B₄O₇ melt was investigated. Results obtained from this variation showed that the Nb recovery increased with the increase in the HNO₃ concentration from 0.70 ± 0.09 to 37.4 ± 0.5 %. This observation prompted the use of high acid concentrations in the following studies to improve dissolution of the melt and more importantly the recovery of Nb. It was also decided to try and prevent H₃BO₃ precipitation at high HNO₃ and H₂SO₄ concentrations by converting the H₃BO₃ which is slightly soluble^{29,30} under these reaction conditions into the volatile methyl ester as indicated by the following equation:³¹



The addition of methanol to the tertaborate flux and the subsequent heating to remove both the boron ester and excess methanol was extremely successful with no precipitation formation and the Nb recovery increased from 37.5 ± 0.2 % (65 % HNO₃) to 103 ± 2 % Nb with 95–98 % H₂SO₄.

3.3. Trace Element Determination

Qualitative and quantitative analyses were also performed with the ICP-OES to determine the type and amount of impurities (trace elements) present in Nb metal, Nb₂O₅ and NbF₅ solutions prepared by microwave digestion. An element was considered as present in the solution if it showed well defined emission peaks on at least three of its most sensitive analytical lines. The presence of some of the elements were questionable since some of their characteristic peaks at their most sensitive analytical lines were not well defined (in all the questionable cases one line for the element in question had excessive noise associated with it) while the other two lines were present and better defined. Quantitative results, using calibration curves with very low concentrations, were subsequently used to try and confirm or reject the presence of these questionable elements in the samples.

For some elements it was difficult to make an accurate decision about its presence from the qualitative data due to the possible spectral interferences at the sensitive lines of the element. For example, the determination of B at the 249.687 nm analytical line (due to Nb 249.697 nm), Cu at 324.754 nm (due to Nb 324.747 nm), and B at 182.640 nm (due to S 182.037 and 182.625 nm) would be greatly affected by the presence of Nb in the H₂SO₄ matrix. The elements cited in Table 6 were detected in

Table 6 ICP-AES determination of the trace elements in niobium metal, niobium oxide and niobium fluoride (%).

Element	Wavelength (nm)	Nb ₂ O ₅	Nb metal	NbF ₅
Na	588.995	0.01	–	–
Cu	327.396	0.30	0.38	0.35
Si	251.612	0.68	0.52	1.49
Mo	203.845	–	–	–
Ti	336.121	–	–	–
Ca	396.847	–	–	–
Fe	239.562	0.01	0.01	–
B	208.959	–	–	0.22
Zn	288.215	0.06	0.05	0.06
Ba	233.527	–	–	0.11
Total %		1.06	0.96	2.23

the three different samples (Nb₂O₅, Nb foil and NbF₅) and were subsequently quantified. From the quantitative analyses some elements such as Ti and Ca were confirmed as absent with their concentrations calculated as zero as within experimental error, while other elements' (such as Ba) concentrations were below their LODs at their selected emission lines. The total of all the impurities in the three samples were higher than expected according to their specification (see Section 2.2 above). This could be due to reagent contamination (such as H₂SO₄), spectral interferences for some elements, a systematic error in the sample preparation or glassware contamination. The purity of these commercial samples could also be questioned since there was no certificate of analysis provided for their purity.

4. Conclusion

Microwave-assisted dissolution using H₂SO₄ of high purity Nb metal, Nb₂O₅ and NbF₅ resulted in the complete dissolution of these highly unreactive compounds and resulted in accurate recoveries and excellent precision in all the analyses using ICP-OES as analytical technique. These results indicate that microwave-assisted digestion using H₂SO₄ can be used as an alternative to HF digestion for niobium and its compounds. The drawbacks of the microwave technique are constraints such as relatively small sample sizes that can be digested per run and the vessel cleaning time which can prolong the sample preparation. Furthermore, lack of knowledge of the chemical composition of the sample can make the choice of solvent (acid) difficult and vessel explosions may result.

The fusion digestion method for pure Nb₂O₅ with Li₂B₄O₇ and subsequent dissolution of the melt with H₂SO₄/methanol mixture was highly successful. A change to an ICP-OES standard addition method for the fusion digestion was also very successful since no significant chemical interferences from the fluxes were observed in the final results. The drawbacks of the fusion method are that, it takes more time (ca. 3 h) than the microwave-assisted dissolution, large quantities of methanol are used and the use of H₂SO₄ requires the solutions to stand overnight before accurate measurements can be made.

The high sensitivity as well as good percentage recoveries obtained in this study indicates the ability of the ICP-OES as analytical method to accurately qualify and quantify niobium containing samples.

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