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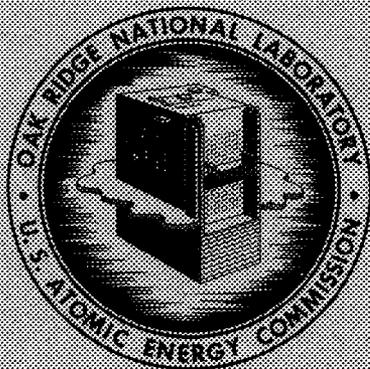


ISOTOPIC MASS SPECTROMETRY OF THE ELEMENTS

Compiled by

E. J. Spitzer

J. R. Sites



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ANALYTICAL CHEMISTRY DIVISION

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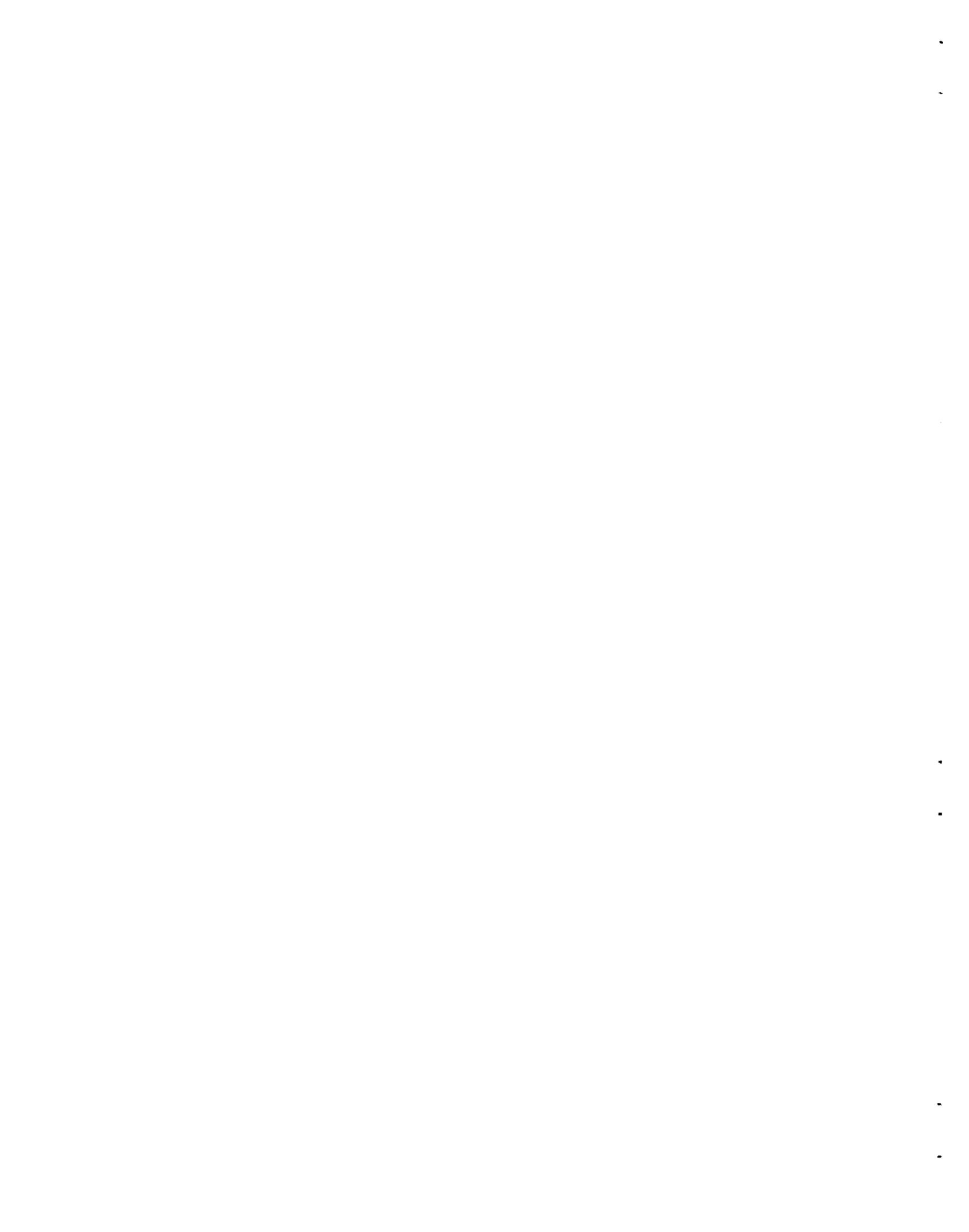
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ABSTRACT

The Analytical Mass Spectrometry Section at Oak Ridge National Laboratory has, for many years, been determining relative isotopic abundances from solid samples. Thus, useful analytical mass spectrometer experience with every pertinent element has been obtained. These include the 55 polyisotopic elements as well as products of irradiation and fission.

The techniques used have been summarized in this report. A separate page for each element lists preferred compound, loading of sample, manner of obtaining a stable ion beam, and solutions of special problems.

These procedures are used daily in the mass spectrometry laboratory, and changes and improvements are frequently being made. They represent a collection of the experimental work of many people. Sometimes several samples a day, each of a different element, must be analyzed on a given mass spectrometer, and it is inefficient not to have a quick-recall aid, such as these procedures. This report certainly does not contain the ultimate nor exhaustive listing of methods but only those currently in use.

GENERAL OPERATING CONDITIONS

Many mass spectrometry laboratories are now determining relative isotopic abundances from solid samples, and other laboratories will be interested in similar analysis in the future. For many years, the Analytical Mass Spectrometry Section of the Oak Ridge National Laboratory has been analyzing isotopes for the Stable Isotopes Separations Group. All 55 of the polyisotopic elements have been separated. The resulting enriched isotopes have been analyzed and made available for loan or sale. Isotopic changes in the products of irradiation, as well as in fission products, are also determined. Thus, useful analytical mass spectrometric experience with every pertinent element has been obtained. It is the purpose of this report to indicate the equipment, outline the general methods used, and itemize the variations in technique required for each element.

All data reported herein were obtained by means of a 60° deflection, sector-type noncommercial metal mass spectrometer. Conventional ion-focusing and accelerating slit systems were used, the ionization chambers being so constructed as to accept expendable bases of the sample-supporting filaments. The filaments are resistance-heated using current-regulated power supplies. The magnet current control permits one to repetitively scan up and down at various rates over a mass range continuously adjustable from a fraction of a mass unit to the full mass range. The magnetically analyzed ion currents, collected on a vane behind an adjustable slit, go

through the 10^{10} -ohm input resistor of a vibrating reed electrometer, and the mass spectra are displayed on a strip chart recorder.

Relative Abundance of Isotopes

The relative abundances of isotopes listed for each element were taken from the *1959 Nuclear Data Tables*.*

Chemical Compound

For the separated stable isotopes, it was hoped that the preferred inventory compound of the element could also be used for the mass analysis. For most elements this is true; however, for some, specially prepared compounds are much better. In a few cases, any simple compound of the element can be used.

Certain physical and chemical properties are desirable. For example, a suitable compound should be chemically stable at room temperature. Also, it is desirable for it to be soluble in water or dilute acid. Sometimes special care is required because the evaporated coating pops off the filament as a result of loss of water of hydration. Stable vaporization may be achieved by using unusual compounds that give thermal decomposition products of high vapor pressure; for example, BaSiF_6 for silicon and Ag_6TeO_6 for tellurium.

Sample Filaments

Several metal filaments are used; however, the most commonly used filament is a $0.001 \times 0.030 \times 0.500$ in. dimpled tantalum ribbon, as shown in Fig. 1. The base used to support the sample filament has a standard octal configuration, with two opposite pins eliminated. The 0.060-in.-diam pins can carry heavy currents, and a single filament is spot-welded across the center pins. The four side pins can be used either for alignment with the sides of the ionization chamber or to support a second filament. These bases cost less than 50¢ each. A long dimple is formed in most of the filaments, greatly aiding in the retention of the sample material in the central portion. Simple hand-drawn pipets used for samples in solution are discarded after use. A micromanipulator is not needed in any of these loading techniques. The same sample filament configuration is used for either thermal or electron-bombardment ionization. The distance between the ionization chamber exit slit and the filament is $\frac{1}{4}$ in., which allows enough space for the electron beam to pass when using bombardment ionization.

Filaments of tungsten, iridium, rhenium, and platinum are also used. Prebaking the filament is necessary for the analysis of potassium, calcium, strontium, and barium. This can be done in

* K. Way (ed.), *1959 Nuclear Data Tables*, Supt. of Documents, U.S. Government Printing Office, Washington 25, D.C.

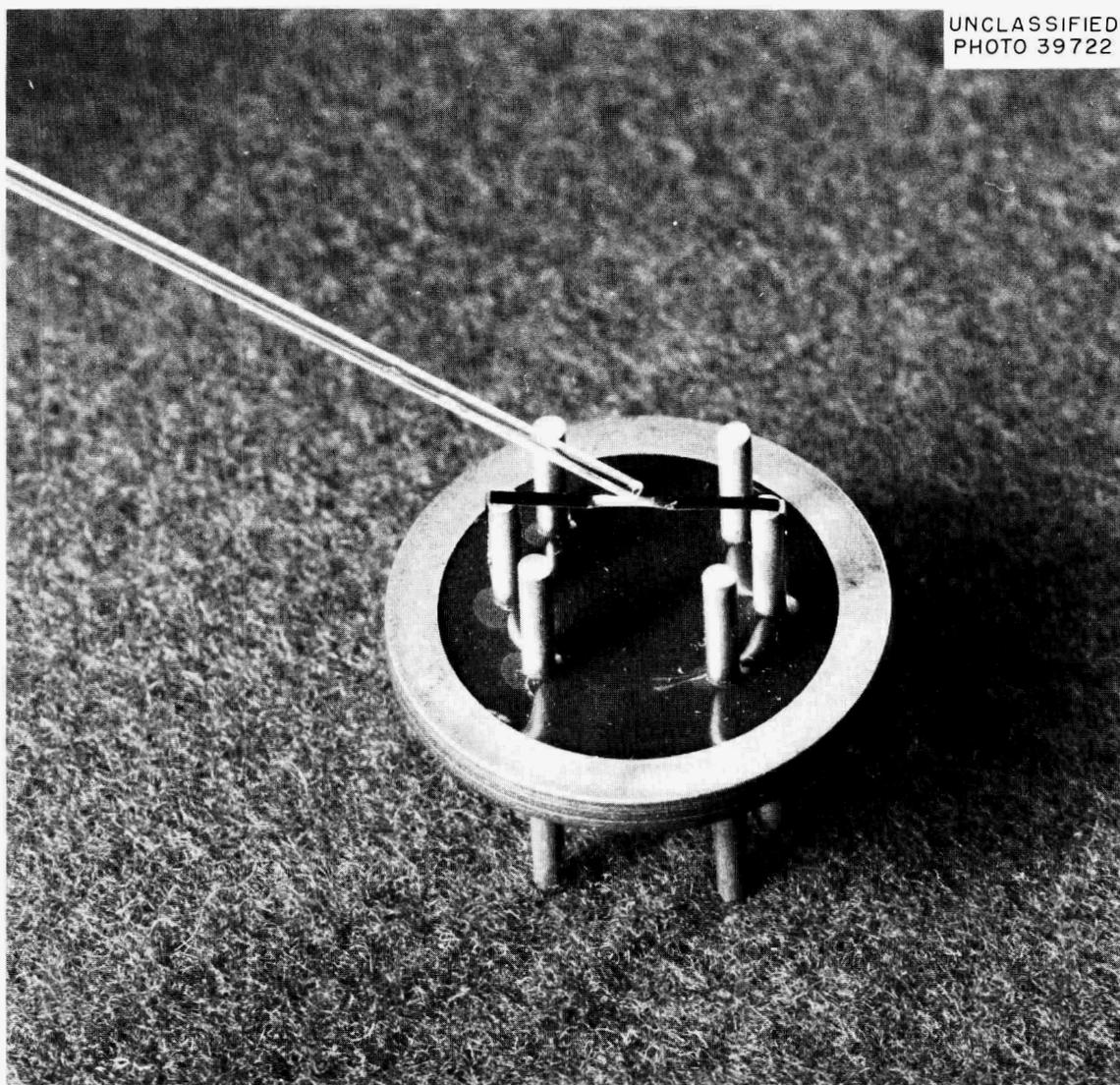


Fig. 1. Sample Filament.

the mass spectrometer to observe the amount of background ion intensity at operating temperatures, or they can be baked in an auxiliary vacuum chamber using a heating cycle that has proved to be satisfactory.

Sample Loading

The sample loading facilities are shown in Fig. 2. The filament base is supported on a small bell-jar flange under the microscope, and a filament can be baked under vacuum to eliminate surface contamination, to fuse a sample, or to reduce an oxide. Accessories are pipets,

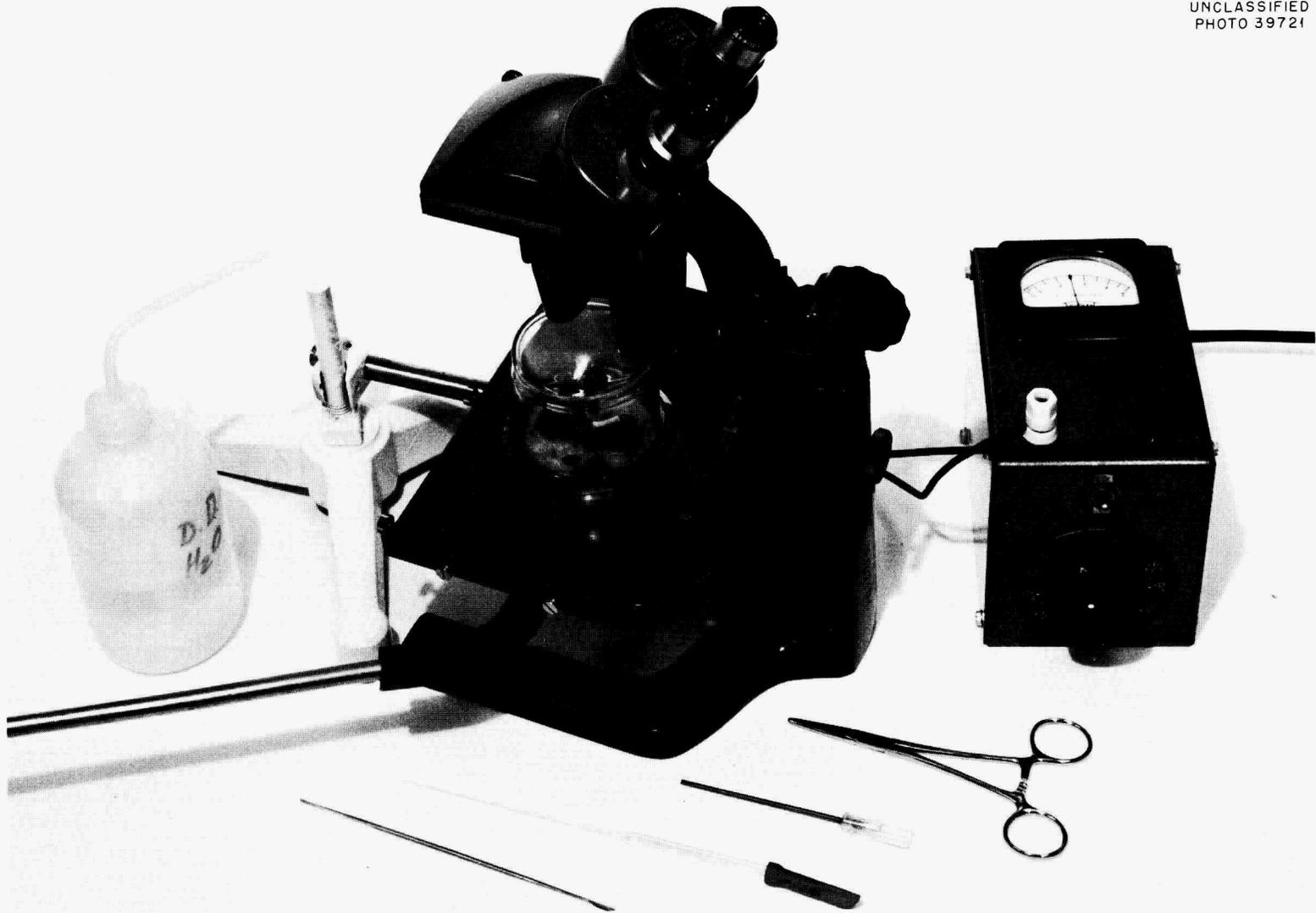


Fig. 2. Sample Loading Facilities.

hemostats, small spatula, and tweezers. Triply distilled water from a polyethylene bottle gives no extra background ions.

The samples placed on the filament vary from a few milligrams to a few nanograms, depending on availability, radioactivity, and volatility which would contribute to memory in the spectrometer source.

Most compounds load very easily on the filament when deposited from a dilute nitrate solution – for example, uranium. A few finely divided powders will slurry with a drop of water or dilute nitric acid, as the rare-earth oxides. Some fuse on the filament, as does NaCl. The usual procedure for loading a platinum-group metal is to fuse it to the proper filament material at a high temperature under vacuum. Boron compounds are added to sodium hydroxide which has been fused on the filament, and further heating forms sodium tetraborate. Fluffy materials and hard materials are crimped into a folded canoe-type filament. Treating CaO and CaCO₃ with HI gives much more steady ion beams of Ca ions. Initial heating of a tantalum filament in air to give a faint blue oxide layer frequently makes the ion beam much more stable. Watching the many loading procedures through the microscope enables one to see phase changes, completion of reactions, or as is sometimes the case only crust from an impure sample.

Temperature Range

The temperature range listed includes the first appearance of the desired ion peaks to the intensity sufficient for obtaining data (approximately 10^{-14} to 10^{-10} amp). The filament size has been selected so that the current needed to reach the maximum temperature is less than 5 amp, and the filament temperature is controlled by a current regulating supply. In some cases, a 0.002×0.060 in. tantalum filament acts as a heat sink. This helps to maintain a slow rate of sample volatilization when the ambient temperature in the source region is too high due to the electron-bombardment filament. In daily practice, the chart shown in Fig. 3 is more convenient to use than an optical pyrometer to determine desired filament temperature.

A bit of experience and finesse is needed in the sequence of raising the filament temperature for some compounds, or the sample may be lost. Also, many analyses are made at a temperature near the melting point of the filament material – for example, some of the platinum-group elements. In some cases, a few sets of data are taken at each increase in ion intensity because the ion beam may deteriorate after the next temperature increase. Frequently, temperature below that for data taking is maintained for a phase change, for background reduction, or for smoother vaporization conditions. Fractional distillation in the spectrometer is sometimes utilized. For example, a small amount of rubidium in a strontium sample will bake out at a lower temperature than that at which the strontium data are taken. Stable ion intensities for some elements, such as the rare-earth elements, can be obtained by setting the filament supply to the proper current.

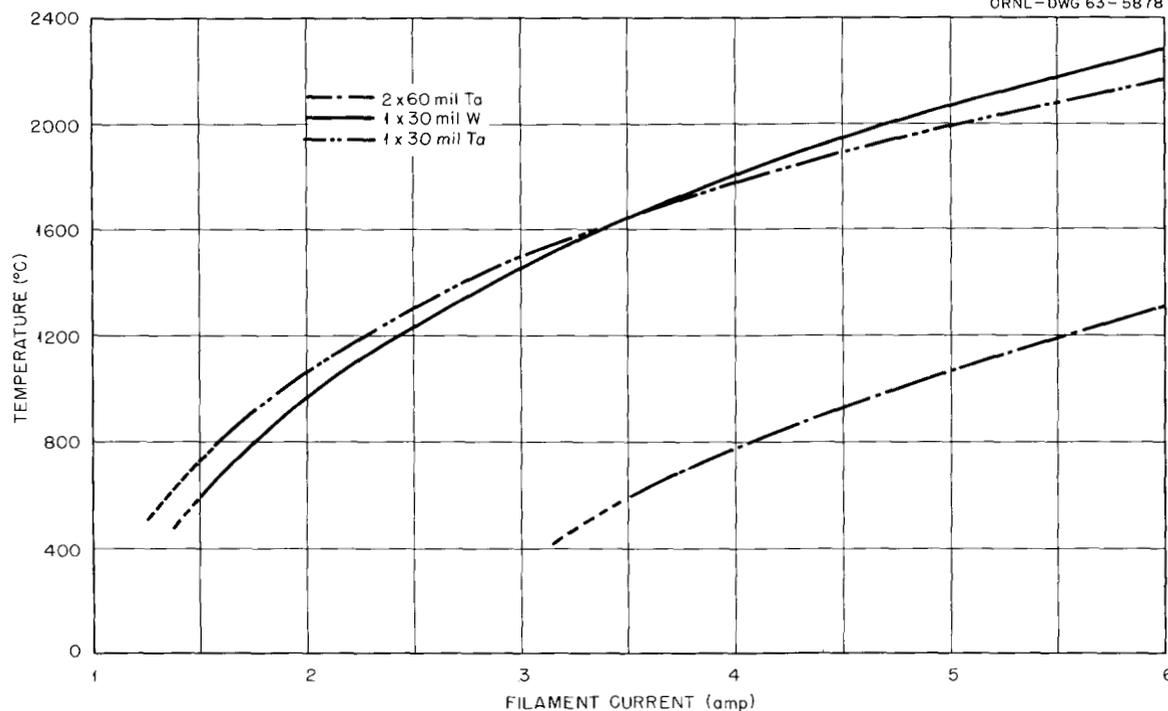


Fig. 3. Filament Temperature Conversion Chart.

Comments

Circumstances continually arise that keep mass spectrometry from becoming too routine. Interfering peaks are often present. For electron-bombardment ion beams, the hydrocarbon background from pump oil or fingerprints may be very persistent. Water in the sample or a small air leak may hinder by keeping the pressure too high.

Corrections for hydrocarbon background often can be made by subtracting a typical background pattern for the mass region, using a nonisorbaric background peak as a base. Corrections for oxygen isotopes are made when necessary, as in analysis of rare-earth oxide ions. All filament materials give sodium, potassium, rubidium, strontium, cesium, and barium ions. Rhenium contains molybdenum, and iridium contains rhodium. Thus, prebaking or proper selection of filament material is required.

The submitted sample may be "clean" to the chemist or the optical spectroscopist and still give bothersome ions of sodium, potassium, etc. In cleaning a uranium solution by extraction, salting with $(\text{NH}_4)_2\text{CO}_3$ instead of CaCO_3 permits steady uranium ion beams of high intensity. Residues of resins and other organics can prevent smooth vaporization and ionization of many compounds.

Isobars can cause errors in peak heights, as among rare-earth elements and for rubidium and strontium. Differences in vapor pressures are enough that isobar interference appears in the form of an ion beam of changing intensity with respect to interference-free ions, and corrections can be made.

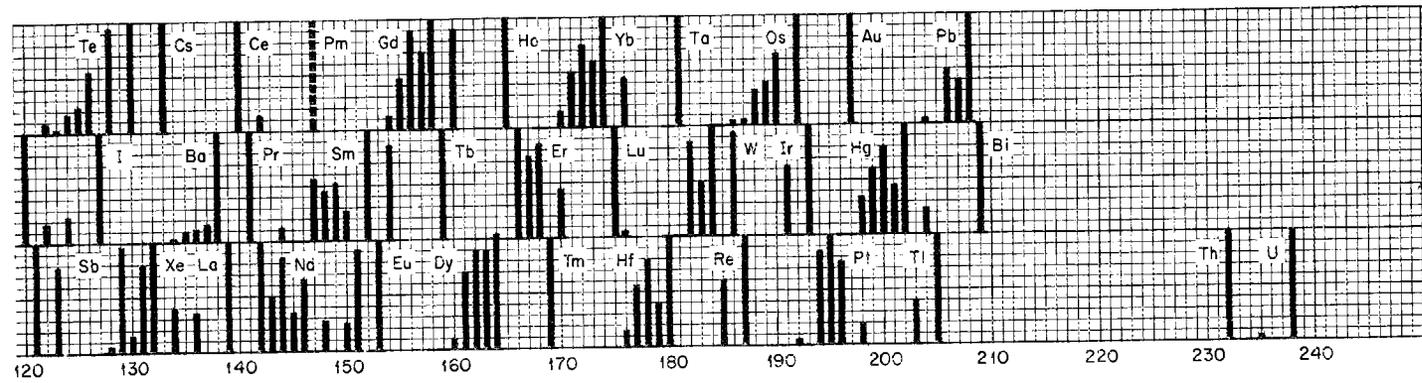
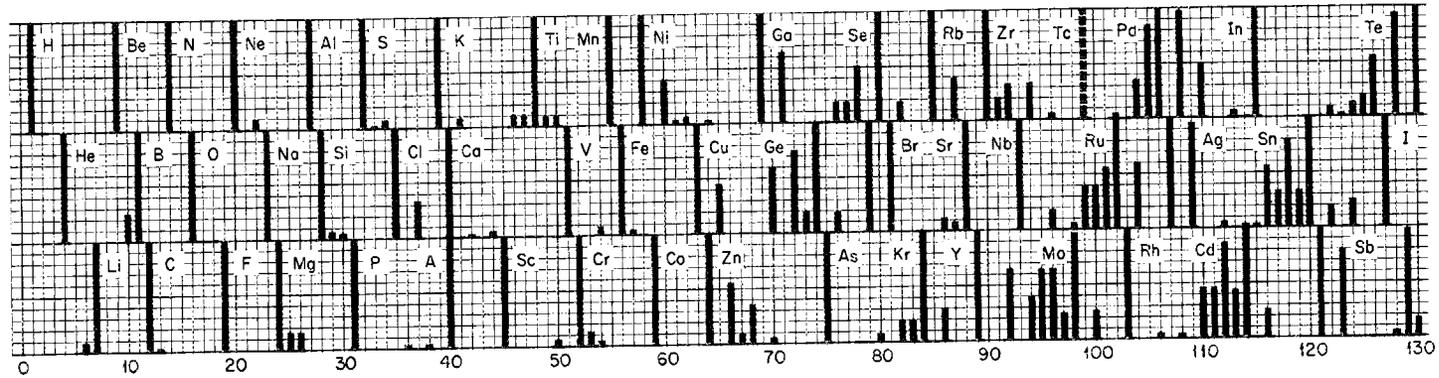
Memory from one run to the next can often be controlled without cleaning the source parts between each analysis by using removable shields, or by analyzing several samples of nearly the same isotopic enrichment. Sometimes, analyzing a "high temperature" sample or two before changing to a different isotope of a "low temperature" set of samples is sufficient.

ACKNOWLEDGMENTS

Many of the compounds used were originally suggested and prepared by W. C. Davis of the Isotopes Division. Most of the procedures have been developed by M. M. Honaker, C. E. Prather, and the compilers, or by other mass spectrometry groups in Oak Ridge. J. R. Walton performed much of the original development. Some of the procedures were taken from the open literature and were adapted to the available equipment.

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Stable Isotope Chart.

LIST OF INCLUDED ELEMENTS

Aluminum	Neptunium
Antimony	Nickel
Barium	Osmium
Boron	Palladium
Bromine	Platinum
Cadmium	Potassium
Caesium	Praseodymium
Calcium	Rhenium
Cerium	Rubidium
Chlorine	Ruthenium
Chromium	Samarium
Copper	Selenium
Dysprosium	Silicon
Erbium	Silver
Europium	Sodium
Gadolinium	Strontium
Gallium	Sulfur
Germanium	Tantalum
Hafnium	Technetium
Holmium	Tellurium
Indium	Terbium
Iodine	Thallium
Iridium	Thorium
Iron	Thulium
Lanthanum	Tin
Lead	Titanium
Lithium	Tungsten
Lutetium	Uranium
Magnesium	Vanadium
Mercury	Ytterbium
Molybdenum	Zinc
Neodymium	Zirconium

Aluminum

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Al_2O_3	27	100
Sample filament	Tungsten		
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1300–1600°C		
Ion measured	Al^+		

Sample Loading

Place finely divided aluminum oxide on the filament and slurry with distilled water. Dry the sample with a gentle heat.

Comments

The temperature at which Al^+ first appears is unpredictable and may vary over a wide range. Interference from previous aluminum samples has not been observed.

Antimony

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Sb	121	57.25
Sample filament	Tantalum	123	42.75
Filament size	1 × 30 mils		
Ionization	Electron bombardment		
Temperature range	<400–750°C		
Ion measured	SbO ⁺		

Sample Loading

Place antimony on the filament; increase the temperature slowly until sample melts.

Comments

The optimum temperature must be approached slowly after SbO⁺ first appears.

The ratio of Sb⁺ to SbO⁺ is approximately 1:10.

Interference from previous antimony samples is a problem.

Correction for O¹⁸ may be necessary.

Barium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Ba(NO ₃) ₂	130	0.101
Sample filament	Tantalum	132	0.097
Filament size	1 × 30 mils	134	2.42
Ionization	Thermal	135	6.59
Temperature range	1300–1650°C	136	7.81
Ion measured	Ba ⁺	137	11.32
		138	71.66

Sample Loading

Place barium nitrate on the filament; increase the temperature slowly until the sample melts. If the temperature is increased too rapidly the sample will "pop" off the filament. Melting Ba(NO₃)₂ will require ~3 amp when heated in air.

Comments

Most tantalum filaments contain residual barium. The major part of the impurities can be removed by prebaking the filament at 6.0 amp for 2 min at a pressure of $<5 \times 10^{-5}$ torr.

When the sample is submitted as BaCO₃, load it on the filament and then add a drop of concentrated nitric acid and complete the preparation as described for the nitrate.

The temperature at which Ba⁺ first appears is unpredictable and may vary over a wide range.

The temperature must be increased slowly because excessive heat generally produces an erratic beam.

Interference from previous barium samples is a problem.

The intensity ratio of Ba⁺ to BaO⁺ varies widely and is not constant.

Boron

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	$\text{Na}_2\text{B}_4\text{O}_7$	10	19.61
Sample filament	Tantalum	11	80.39
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	750--1000°C		
Ion measured	Na_2BO_2^+		

Sample Loading

Place sodium tetraborate on the filament; increase the temperature slowly until the sample melts.

Comments

Two micrograms of boron, as $\text{Na}_2\text{B}_4\text{O}_7$, have produced an ion current of 5×10^{-12} amp with very slow decay (~5% in 10 min).

B^{10} and B^{11} , as Na_2BO_2^+ , have masses at 88 and 89.

Boron metal or compounds, such as B_2O_3 , H_2BO_3 , and B_4C , are converted to sodium tetraborate by fusing them in a small pellet of NaOH. This is done on the filament, and the temperature is then increased to a red heat (~750°C).

Interference from previous boron samples has not been observed.

Sr^{88} can give interference. Correction for O^{17} may be necessary.

Bromine

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	NaBr	79	50.537
Sample filament	Tantalum	81	49.463
Filament size	1 × 30 mils		
Ionization	Electron bombardment		
Temperature range	400–1000°C		
Ion measured	NaBr ⁺		

Sample Loading

Place sodium bromide on the filament; increase the temperature slowly until the sample melts.

Comments

The NaBr⁺ intensity will decrease soon after it first appears. Additional increases of temperature will produce a stable and higher ion current.

Cadmium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	CdO	106	1.215
Sample filament	Tantalum	108	0.875
Filament size	1 × 30 mils	110	12.39
Ionization	Electron bombardment	111	12.75
Temperature range	800–1100°C	112	24.07
Ion measured	Cd ⁺	113	12.26
		114	28.86
		116	7.58

Sample Loading

Place finely divided cadmium oxide on the filament and slurry with distilled water. Dry the sample with a gentle heat.

Comments

The Cd⁺ intensity will decrease soon after it first appears. Additional increase of temperature will produce a stable and higher ion current.

Interference from previous cadmium samples is a serious problem.

Palmer* reported that CdSO₄ dissolved in borax gave thermal ions (Cd⁺) at 900°C from a tungsten filament.

*G. H. Palmer, in *Advances in Mass Spectrometry*, ed. by J. D. Waldron, Pergamon Press, New York, 1959.

Caesium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Cs_2SnCl_6	133	100
Sample filament	Tantalum		
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	600–800°C		
Ion measured	Cs^+		

Sample Loading

Place caesium chlorostannate solution on the filament and dry with a gentle heat.

Comments

Some tantalum filaments contain residual caesium. The major part of the impurities can be removed by prebaking the filament at 6.0 amp for 1.5 min at a pressure of $<5 \times 10^{-5}$ torr.

The temperature must be increased slowly because excessive heat generally produces an erratic beam.

Interference from previous caesium samples is a problem.

Interference from Ba^+ is possible, but Cs^+ usually appears at a lower temperature than Ba^+ .

Many other Cs salts readily ionize and can be used for mass analysis.

Calcium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	CaCO ₃	40	96.97
Sample filament	Tantalum	42	0.64
Filament size	1 × 30 mils	43	0.145
Ionization	Thermal	44	2.06
Temperature range	1400–1700°C	46	0.0033
Ion measured	Ca ⁺	48	0.185

Sample Loading

Place calcium carbonate on the filament and slurry with hydriodic acid. Dry the sample with a gentle heat and drive off excess iodine; then increase the filament temperature to a dull-red heat (~750°C). Heating will first convert the sample to CaI₂ and then to CaO. The sample is then tightly bound to the filament.

Comments

One microgram of calcium will produce an ion current of 10⁻¹⁰ amp for more than 20 min.

The Ca⁺ beam is stable over long periods of time.

Interference from K⁴⁰ is possible, and data is only taken when K⁴¹ is very much less than the major Ca isotope.

Interference from previous calcium samples has not been observed.

CaO, Ca(NO₃)₂, or CaI₂ may be used in place of CaCO₃.

Cerium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	CeO ₂	136	0.193
Sample filament	Tantalum	138	0.250
Filament size	1 × 30 mils	140	88.48
Ionization	Thermal	142	11.07
Temperature range	1300–1600°C		
Ion measured	CeO ⁺		

Sample Loading

Place finely divided cerium oxide on the filament and partially digest with a drop of concentrated nitric acid. Dry the sample with a gentle heat; then increase the filament temperature to a dull-red heat (~750°C).

Comments

One microgram of cerium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Ce⁺ to CeO⁺ is 1 : >100.

Interference from previous cerium samples has not been observed.

Corrections for O¹⁷ and O¹⁸ may be necessary.

Chlorine

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	NaCl	35	75.529
Sample filament	Tantalum	37	24.471
Filament size	1 × 30 mils		
Ionization	Electron bombardment		
Temperature range	700–1400°C		
Ion measured	NaCl ⁺		

Sample Loading

Place sodium chloride on the filament; increase the temperature slowly until the sample melts. If the temperature is increased too rapidly the sample will "pop" off of the filament.

Comments

Interference from previous chlorine samples has not been observed at either the NaCl⁺ or Na₂Cl⁺ positions. These ions have approximately equal intensity.

Data can be taken at Cl⁺ and HCl⁺; however, interference from previous samples is serious at this position.

The temperature at which NaCl⁺ first appears is unpredictable and may vary over a wide range.

Data have been obtained using the negative ions of chlorine.

The Cl⁻ data are relatively free from interference.

Chromium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Cr_2O_3	50	4.31
Sample filament	Tungsten	52	83.76
Filament size	1 × 30 mils	53	9.55
Ionization	Thermal	54	2.38
Temperature range	1300–1600°C		
Ion measured	Cr^+		

Sample Loading

Place finely divided chromium oxide on the filament. Introduce the loaded filament into a vacuum chamber and pump down to $<20 \mu$ pressure. Increase the filament temperature until the sample begins to fuse.

Comments

The Cr^+ beam is stable over long periods of time.

Interference from previous chromium samples has not been observed.

Copper

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	CuO	63	69.09
Sample filament	Tungsten	65	30.91
Filament size	1 × 30 mils		
Ionization	Electron bombardment		
Temperature range	1000–1400°C		
Ion measured	Cu ⁺		

Sample Loading

Place finely divided cupric oxide on the filament and slurry with distilled water. Dry with a gentle heat. Cover with a vacuum chamber and pump down to <20 μ pressure. Increase the filament temperature until sample reduces to copper metal and melts.

Comments

Electron bombardment intensities are a factor of 10 greater than thermal intensities. Interference from previous copper samples has not been observed.

Dysprosium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Dy ₂ O ₃	156	0.0524
Sample filament	Tantalum	158	0.0902
Filament size	1 × 30 mils	160	2.294
Ionization	Thermal	161	18.88
Temperature range	1400–1500°C	162	25.53
Ion Measured	Dy ⁺	163	24.97
		164	28.18

Sample Loading

Place finely divided dysprosium oxide on the filament and partially digest with a drop of concentrated nitric acid. Dry sample with a gentle heat; then increase the filament temperature to a dull-red heat (~750°C).

Comments

One microgram of dysprosium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Dy⁺ to DyO⁺ is 10:1.

Interference from previous dysprosium samples has not been observed.

Erbium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Er ₂ O ₃	162	0.136
Sample filament	Tantalum	164	1.56
Filament size	1 × 30 mils	166	33.41
Ionization	Thermal	167	22.94
Temperature range	1400–1700°C	168	27.07
Ion measured	Er ⁺	170	14.88

Sample Loading

Place finely divided erbium oxide on the filament and partially digest with a drop of concentrated nitric acid. Dry sample with a gentle heat; then increase the filament temperature to a dull-red heat (~700°C).

Comments

One microgram of erbium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Er⁺ to ErO⁺ is >100:1.

Interference from previous erbium samples has not been observed.

Europium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Eu_2O_3	151	47.82
Sample filament	Tantalum	153	52.18
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1400--1700°C		
Ion measured	Eu^+		

Sample Loading

Place finely divided europium oxide on the filament and partially digest with a drop of concentrated nitric acid. Dry sample with a gentle heat; then increase the filament temperature to a dull-red heat ($\sim 750^\circ\text{C}$).

Comments

One microgram of europium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Eu^+ to EuO^+ is $>100:1$.

Interference from previous europium samples has not been observed.

Gadolinium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Gd ₂ O ₃	152	0.200
Sample filament	Tantalum	154	2.15
Filament size	1 × 30 mils	155	14.73
Ionization	Thermal	156	20.47
Temperature range	1400–1700°C	157	15.68
Ion measured	GdO ⁺	158	24.87
		160	21.90

Sample Loading

Place finely divided gadolinium oxide on the filament and partially digest with a drop of concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

One microgram of gadolinium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Gd⁺ to GdO⁺ is 1 : >100.

Interference from previous gadolinium samples has not been observed.

Corrections for O¹⁷ and O¹⁸ may be necessary.

Gallium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Ga_2O_3	69	60.4
Sample filament	Tungsten	71	39.6
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	700–1200°C		
Ion measured	Ga^+		

Sample Loading

Place gallium oxide on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

The use of small samples improves the stability of the ion beam.

Interference from previous gallium samples has not been observed.

Germanium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	BaGeF ₆	70	20.52
Sample filament	Tantalum	72	27.43
Filament size	1 × 60 mils	73	7.76
Ionization	Electron bombardment	74	36.54
Temperature range	750–1200°C	76	7.76
Ion measured	GeF ₃ ⁺ or GeF ⁺		

Sample Loading

Place barium fluogermanate on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

BaGeF₆ is prepared by dissolving GeO₂ in HF followed by precipitation with BaCl₂. The salt is isolated by centrifugation.

Ions Ge⁺, GeF⁺, GeF₂⁺, GeF₃⁺, GeOF₃⁺, and GeOF₄⁺ are noted but the best data are obtained at GeF₃⁺.

SiF₃⁺ usually appears in the ion spectrum but does not isotopically interfere.

If Ge⁺ and GeF⁺ are as intense as GeF₃⁺, the compound apparently has not been prepared properly and a new sample must be submitted.

The GeF₃⁺ intensity will decrease soon after it first appears. Additional increase of temperature will produce a stable and higher ion current.

Masses of Isotopic Ions

Ge ⁺	GeF ⁺	GeF ₂ ⁺	GeF ₃ ⁺	GeOF ₃ ⁺	GeOF ₄ ⁺
70	89	108	127	143	163
72	91	110	129	145	164
73	92	111	130	146	165
74	93	112	131	147	166
76	95	114	133	149	168

Hafnium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	HfO ₂	174	0.18
Sample filament	Tantalum	176	5.20
Filament size	1 × 30 mils	177	18.50
Ionization	Electron bombardment	178	27.14
Temperature range	1700–1900°C	179	13.75
Ion measured	HfO ⁺	180	35.24

Sample Loading

Place finely divided hafnium oxide on the filament and slurry with distilled water. Dry sample with a gentle heat. Cover with a vacuum chamber and pump down to <20 μ pressure. Increase filament temperature until the sample begins to reduce to hafnium metal and melts. Do not completely convert to hafnium metal.

Comments

The observed ratio of Hf⁺ to HfO⁺ is approximately 1:5.

Thermal ion intensities are very low compared to electron-bombardment intensities.

Ta⁺, TaO⁺, and W can give interference. Interference from previous hafnium samples has not been observed.

Corrections for O¹⁷ and O¹⁸ may be necessary.

Holmium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Ho_2O_3	165	100
Sample filament	Tantalum		
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1400–1600°C		
Ion measured	Ho^+		

Sample Loading

Place finely divided holmium oxide on the filament and partially digest with a drop of concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat ($\sim 750^\circ\text{C}$).

Comments

One microgram of holmium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Ho^+ to HoO^+ is 10:1.

Interference from previous holmium samples has not been observed.

Indium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	In_2O_3	113	4.28
Sample filament	Tantalum	115	95.72
Filament size	2 × 60 mils		
Ionization	Thermal		
Temperature range	700–1000°C		
Ion measured	In^+		

Sample Loading

Place indium oxide on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

The temperature must be increased slowly because excessive heat generally produces an erratic beam.

When In^+ first appears, the intensity will increase slowly for a few minutes and then rapidly.

Filament temperature must be reduced to avoid losing the sample.

InO^+ appears briefly at low temperatures, but decreases as In^+ increases.

Interference from previous indium samples is a problem.

Iodine

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	NaI	127	100
Sample filament	Tantalum		
Filament size	2 × 60 mils		
Ionization	Electron bombardment		
Temperature range	<200°C		
Ion measured	NaI ⁺		

Sample Loading

Place the sodium iodide solution on the filament and dry with a gentle heat.

Comments

Data can be obtained from <1 μg of iodine.

Temperature must be increased slowly because excessive heat generally produces an erratic beam.

Interference from previous iodine samples is a very serious problem.

This procedure has frequently been used for I¹²⁵, I¹²⁷, and I¹²⁹.

Iridium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Ir	191	37.3
Sample filament	Tungsten	193	62.7
Filament size	1 × 30 mils		
Ionization	Electron bombardment		
Temperature range	1500–1800°C		
Ion measured	Ir ⁺		

Sample Loading

Place finely divided iridium metal on the filament. Cover with a vacuum chamber and pump down to $<20 \mu$ pressure. Increase filament temperature until sample begins to melt.

Comments

A large sample is required (>1 mg).

Optimum temperature must be approached very slowly after Ir⁺ first appears.

Interference from previous iridium samples has not been observed.

Data were not obtained when using a tantalum filament.

Iron

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Fe_2O_3	54	5.82
Sample filament	Tantalum	56	91.66
Filament size	2×60 mils	57	2.19
Ionization	Electron bombardment	58	0.33
Temperature range	$<200^\circ\text{C}$		
Ion measured	FeI^+		

Sample Loading

Place iron oxide on the filament and add hydriodic acid. Dry sample with a gentle heat; then increase the temperature slowly until sample melts. Melting the iron iodide will require ~ 4 amp when heated in air.

Comments

FeI_2 is very hygroscopic.

Interference from previous iron iodide samples is a very serious problem.

Fe metal can be ionized thermally, but ion intensities are relatively low compared to electron bombardment. Fe_2O_3 can be converted to Fe metal as follows: Load finely divided iron oxide on a tantalum filament. Cover with a vacuum chamber and pump down to $<20 \mu$ pressure. Increase temperature until the sample melts. A temperature range of $1200\text{--}1400^\circ\text{C}$ is required to obtain Fe^+ .

Mn^+ is frequently observed in the ion spectrum but does not interfere.

FeCl_2 has been used for analysis.

Masses of Isotopic Ions

Fe^+	FeI^+	FeI_2^+
54	181	308
56	183	310
57	184	311
58	185	312

Lanthanum

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	La ₂ O ₃	138	0.089
Sample filament	Tantalum	139	99.911
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1300–1600°C		
Ion measured	LaO ⁺		

Sample Loading

Place finely divided lanthanum oxide on the filament and partially digest with concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

One microgram of lanthanum produces an ion current of 10^{-10} amp for an hour.

The observed ratio of La⁺ to LaO⁺ is 1:>100.

Interference from previous lanthanum samples has not been observed.

Correction for O¹⁷ may be necessary.

Lead

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	$\text{Pb}(\text{NO}_3)_2$	204	1.48
Sample filament	Tungsten	206	23.6
Filament size	1 × 30 mils	207	22.6
Ionization	Thermal	208	52.3
Temperature range	1000–1400°C		
Ion measured	Pb^+		

Sample Loading

Place the lead nitrate solution on the filament and add a drop of saturated boric acid solution.

Dry this mixture with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

Analyses have been made on less than 10 μg of lead.

Interference from previous lead samples has not been observed.

PbO or PbCrO_4 can be ionized with electron bombardment. These compounds should be loaded on tantalum filaments and data taken at Pb^+ .

Lithium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	LiI	6	7.42
Sample filament	Tantalum	7	92.58
Filament size	2 × 60 mils		
Ionization	Electron bombardment		
Temperature range	<500°C		
Ion measured	Li ₂ I ⁺		

Sample Loading

Place lithium iodide solution on the filament and dry with a gentle heat.

Comments

Most Li salts can be converted to LiI with the addition of hydriodic acid.

Li salts (Li₂SO₄, Li₂CO₃, and LiOH) can be ionized thermally and analysis made at Li⁺. This can induce errors due to the large percentage difference in mass.

Interference from previous lithium samples is a serious problem.

Mass	Ion
139	(Li ⁶ Li ⁶ I ¹²⁷) ⁺
140	(Li ⁶ Li ⁷ I ¹²⁷) ⁺
141	(Li ⁷ Li ⁷ I ¹²⁷) ⁺

Lutetium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Lu_2O_3	175	97.41
Sample filament	Tantalum	176	2.59
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1400–1800°C		
Ion measured	Lu^+		

Sample Loading

Place finely divided lutetium oxide on the filament and partially digest with concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat ($\sim 750^\circ\text{C}$).

Comments

One microgram of lutetium produces an ion current of 10^{-10} amp for more than an hour. The observed ratio of Lu^+ to LuO^+ is $>100:1$.

Interference from previous lutetium samples has not been observed.

Magnesium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	MgO	24	78.70
Sample filament	Tungsten	25	10.13
Filament size	1 × 30 mils	26	11.17
Ionization	Thermal		
Temperature range	1400–1500°C		
Ion measured	Mg ⁺		

Sample Loading

Place finely divided magnesium oxide on filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

The optimum temperature must be approached very slowly after Mg⁺ first appears.

If Na²³ or Al²⁷ impurity ions are of sufficient intensity, the "tails" of these beams may interfere at the Mg²⁴ or Mg²⁶ mass positions.

Interference from previous magnesium samples has not been observed.

Mercury

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Hg(NO ₃) ₂	196	0.146
Sample filament	Tantalum	198	10.02
Filament size	2 × 60 mils	199	16.84
Ionization	Electron bombardment	200	23.13
Temperature range	< 500°C	201	13.22
Ion measured	Hg ⁺	202	29.80
		204	6.85

Sample Loading

Place mercuric nitrate solution on the filament and dry with a gentle heat.

Comments

Interference from previous mercury samples has been observed.

Samples of <2 mg of mercury in gold foil can be loaded by crimping the 2 × 60 mil tantalum filament around a sliver of foil.

Data have been obtained from mercuric sulfide.

Molybdenum

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	MoO ₃	92	15.84
Sample filament	Tungsten	94	9.04
Filament size	1 × 30 mils	95	15.72
Ionization	Electron bombardment	96	16.53
Temperature range	700–1000°C	97	9.46
Ion measured	MoO ₂ ⁺	98	23.78
		100	9.63

Sample Loading

Place finely divided molybdenum oxide on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

The following ions are observed: Mo⁺, MoO⁺, MoO₂⁺, and MoO₃⁺, with an intensity ratio of 2, 5, 10, and 3 respectively.

Thermal intensities are very low compared to electron-bombardment intensities.

Interference from previous molybdenum samples has not been observed.

Corrections for O¹⁷ and O¹⁸ may be necessary.

Masses of Isotopic Ions

Mo ⁺	MoO ⁺	MoO ₂ ⁺	MoO ₃ ⁺
92	108	124	140
94	110	126	142
95	111	127	143
96	112	128	144
97	113	129	145
98	114	130	146
100	116	132	148

Neodymium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Nd ₂ O ₃	142	27.11
Sample filament	Tantalum	143	12.17
Filament size	1 × 30 mils	144	23.85
Ionization	Thermal	145	8.30
Temperature range	1300--1600°C	146	17.22
Ion measured	NdO ⁺	148	5.73
		150	5.62

Sample Loading

Place finely divided neodymium oxide on the filament and partially digest with concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

One microgram of neodymium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Nd⁺ to NdO⁺ is 1:>100.

Interference from previous neodymium samples has not been observed.

Corrections for O¹⁷ and O¹⁸ may be necessary.

Neptunium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	$\text{NpO}_2(\text{NO}_3)_2$	237	—
Sample filament	Tantalum		
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1700–1900°C		
Ion measured	NpO_2^+		

Sample Loading

Place the neptunium nitrate solution on the filament and dry with a gentle heat.

Comments

The observed ratio of $\text{Np}^+ : \text{NpO}^+ : \text{NpO}_2^+$ is approximately <1 : 10 : 20.

Nickel

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Ni	58	67.88
Sample filament	Tungsten	60	26.23
Filament size	1 × 30 mils	61	1.19
Ionization	Electron bombardment	62	3.66
Temperature range	1000–1400°C	64	1.08
Ion measured	Ni ⁺		

Sample Loading

Place finely divided nickel metal on the filament. Cover with a vacuum chamber and pump down to $<20 \mu$ pressure. Increase filament temperature until sample melts.

Comments

A serious background peak is frequently found at mass 64. This is eliminated by baking the loaded filament at approximately 850°C overnight.

Thermal ionization intensities are very low ($<1:10$) compared to those produced by electron bombardment.

Interference from previous nickel samples has not been observed.

Osmium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	$\text{OsO}_2 \cdot X\text{H}_2\text{O}$	184	0.018
Sample filament	Tantalum	186	1.59
Filament size	2 × 60 mils	187	1.64
Ionization	Electron bombardment	188	13.3
Temperature range	< 500°C	189	16.1
Ion measured	OsO_4^+	190	26.4
		192	41.0

Sample Loading

Place finely divided hydrated osmium dioxide on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

OsO_4 melts at 45° and boils at 100°C, yielding a very poisonous vapor which causes pathological disturbances, even to the extent of blindness.

The optimum temperature must be approached very slowly after OsO_4^+ first appears.

The following ions are observed: Os^+ , OsO^+ , OsO_2^+ , OsO_3^+ , and OsO_4^+ with an intensity ratio of 3, 2, 4, 3, and 10 respectively.

Interference from previous samples is a problem. Corrections for O^{17} and O^{18} may be necessary.

Masses of Isotopic Ions

Os^+	OsO^+	OsO_2^+	OsO_3^+	OsO_4^+
184	200	216	232	248
186	202	218	234	250
187	203	219	235	251
188	204	220	236	252
189	205	221	237	253
190	206	222	238	254
192	208	224	240	256

Palladium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Pd	102	0.96
Sample filament	Iridium or rhenium	104	10.97
Filament size	1 × 30 mils	105	22.23
Ionization	Electron bombardment	106	27.33
Temperature range	>1000°C	108	26.71
Ion measured	Pd ⁺	110	11.81

Sample Loading

Place finely divided palladium metal on the filament. Cover with a vacuum chamber and pump down to <20 μ pressure. Increase filament temperature until sample melts.

Comments

The Pd⁺ beam is stable over long periods of time. Interference from previous palladium samples has not been observed.

Platinum

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Pt	190	0.0127
Sample filament	Tungsten	192	0.78
Filament size	1 × 30 mils	194	32.9
Ionization	Electron bombardment	195	33.8
Temperature range	900–1500°C	196	25.3
Ion measured	Pt ⁺	198	7.21

Sample Loading

Place finely divided platinum metal on the filament. Cover with a vacuum chamber and pump down to $< 20 \mu$ pressure. Increase filament temperature until sample melts.

Comments

WO⁺ frequently appears. As the intensity of WO⁺ begins to decrease Pt⁺ will appear.

Hg⁺ and WO⁺ can give interference. Interference from previous platinum samples has not been observed.

Data were not obtained when using a tantalum filament.

Potassium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	K_3PO_4	39	93.10
Sample filament	Tantalum	40	0.01181
Filament size	1 × 30 mils	41	6.88
Ionization	Thermal		
Temperature range	600–800°C		
Ion measured	K^+		

Sample Loading

Place potassium solution on the filament and dry with a gentle heat.

Comments

All tantalum filaments contain residual potassium. The major part of the impurities can be removed by prebaking the filament at 6.0 amp for 2 min at a pressure of $<5 \times 10^{-5}$ torr.

Data have been obtained on $<1 \mu\text{g}$ of potassium.

Temperature must be increased slowly because excessive heat generally produces an erratic beam.

Interference from previous potassium samples is a very serious problem.

Praseodymium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Pr_2O_3	141	100
Sample filament	Tantalum		
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1300–1600°C		
Ion measured	PrO^+		

Sample Loading

Place finely divided praseodymium oxide on the filament and partially digest with concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat ($\sim 750^\circ\text{C}$).

Comments

One microgram of praseodymium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Pr^+ to PrO^+ is 1 : >100.

Interference from previous praseodymium samples has not been observed.

Rhenium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	$\text{NH}_4\text{Re}_2\text{O}_7$	185	37.07
Sample filament	Tantalum	187	62.93
Filament size	2×60 mils		
Ionization	Electron bombardment		
Temperature range	$<200^\circ\text{C}$		
Ion measured	Re^+		

Sample Loading

Place ammonium perrhenate on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

In addition to Re^+ the following ions are observed: ReO^+ , ReO_2^+ , ReO_3^+ , and ReO_4^+ . Each of these ions can unite with H to give an additional peak one mass higher.

Interference from previous rhenium samples is a problem.

Masses of Isotopic Ions

Re^+	ReO^+	ReO_2^+	ReO_3^+	ReO_4H^+
185	201	217	233	250
187	203	219	235	252

Rubidium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	RbCl	85	72.15
Sample filament	Tantalum	87	27.85
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	900–1300°C		
Ion measured	Rb ⁺		

Sample Loading

Place the rubidium chloride solution on the filament and dry with a gentle heat.

Comments

Most tantalum filaments contain residual rubidium. The major part of the impurities can be removed by prebaking the filament at 6.0 amp for 1.5 min at a pressure of $<5 \times 10^{-5}$ torr.

Data have been obtained on $<1 \mu\text{g}$ of rubidium.

Interference from previous rubidium samples is a very serious problem.

Ruthenium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Ru	96	5.51
Sample filament	Tungsten	98	1.87
Filament size	1 × 30 mils	99	12.72
Ionization	Thermal	100	12.62
Temperature range	1600–2000°C	101	17.07
Ion measured	Ru ⁺	102	31.61
		104	18.58

Sample Loading

Place finely divided ruthenium metal on the filament. Cover with a vacuum chamber and pump down to $<20 \mu$ pressure. Increase temperature until sample melts.

Comments

The optimum temperature must be approached very slowly after Ru⁺ first appears.

Ru metal will alloy with the tungsten filament, causing it to melt. The Ru⁺ beam becomes unstable just before this occurs.

Interference from previous ruthenium samples has not been observed.

Samarium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Sm ₂ O ₃	144	3.09
Sample filament	Tantalum	147	14.97
Filament size	1 × 30 mils	148	11.24
Ionization	Thermal	149	13.83
Temperature range	1400–1700°C	150	7.44
Ion measured	Sm ⁺	152	26.72
		154	22.71

Sample Loading

Place finely divided samarium oxide on the filament and partially digest with concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat (~750°C)

Comments

One microgram of samarium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Sm⁺ to SmO⁺ is >100:1.

Interference from previous samarium samples has not been observed.

Selenium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	PbSeO ₄	74	0.87
Sample filament	Tantalum	76	9.02
Filament size	2 × 60 mils	77	7.58
Ionization	Electron bombardment	78	23.52
Temperature range	400--700°C	80	49.82
Ion measured	SeO ₂ ⁺	82	9.19

Sample Loading

Place lead selenate on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

The filament should be prebaked to help reduce any hydrocarbon background, as it frequently causes interference. Se⁺, SeO⁺, and SeO₂⁺ have relative intensities of 1, 2, and 3, respectively. Se₂²⁺ gives interference at Se⁺.

There is no interference from previous selenium samples at SeO⁺ and SeO₂⁺.

Corrections for O¹⁷ and O¹⁸ may be necessary.

Lead selenate is prepared by dissolving elemental selenium in ammonia and hydrogen peroxide and precipitating the selenium by adding lead acetate and then acetic acid.

Masses of Isotopic Ions

Se ⁺	SeO ⁺	SeO ₂ ⁺
74	90	106
76	92	108
77	93	109
78	94	110
80	96	112
82	98	114

Silicon

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	BaSiF ₆	28	92.21
Sample filament	Tantalum	29	4.70
Filament size	2 × 60 mils	30	3.09
Ionization	Electron bombardment		
Temperature range	<200°C		
Ion measured	SiF ₃ ⁺		

Sample Loading

Place barium fluorosilicate on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

Optimum temperature must be approached very slowly after SiF₃⁺ first appears.

BaSiF₆ is prepared by dissolving 1–2 mg samples of SiO₂ in diluted HF followed by a precipitation with BaCl₂. The sample is recovered by centrifuging in plastic.

Masses of Isotopic Ions

Si ⁺	SiF ⁺	SiF ₂ ⁺	SiF ₃ ⁺	SiF ₄ ⁺
28	47	66	85	104
29	48	67	86	105
30	49	68	87	106

Silver

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	AgNO ₃	107	51.35
Sample filament	Tungsten	109	48.65
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1100–1400°C		
Ion measured	Ag ⁺		

Sample Loading

Place the silver nitrate solution on the filament and add a drop of saturated boric acid solution.
Dry mixture with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

Interference from previous silver samples has not been observed.

Ag metal can be converted to nitrate on filament by addition of a drop of concentrated HNO₃ and heating.

AgI can be used for analysis using the following conditions:

2 × 60 mil tantalum filament

Electron bombardment

<200°C filament temperature

Data taken at Ag⁺

Other ions observed are Ag₂⁺, Ag₃⁺, AgI⁺, Ag₂I⁺, and AgI₂⁺.

Sodium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	NaOH	23	100
Sample filament	Tantalum		
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	<800°C		
Ion measured	Na ⁺		

Sample Loading

Place sodium hydroxide solution on the filament and dry with a gentle heat.

Comments

Most tantalum filaments contain residual sodium. The major part of these impurities can be removed by prebaking the filament at 6.0 amp for 1.5 min at a pressure of 5×10^{-5} torr.

The temperature at which Na⁺ first appears is unpredictable and may vary over a wide range.

Temperature must be increased slowly because excessive heat generally produces an erratic beam.

Interference from previous sodium samples is a very serious problem.

Strontium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotopes	Atom %
Compound	$\text{Sr}(\text{NO}_3)_2$	84	0.56
Sample filament	Tantalum	86	9.86
Filament size	1 × 30 mils	87	7.02
Ionization	Thermal	88	82.56
Temperature range	1200–1500°C		
Ion measured	Sr^+		

Sample Loading

Place strontium nitrate solution on the filament and dry with a gentle heat.

Comments

Most tantalum filaments contain residual rubidium and sometimes strontium. The major part of the impurities can be removed by prebaking the filament at 6.0 amp for 1.5 min at a pressure of $<5 \times 10^{-5}$ torr.

Interference from previous strontium samples is a problem.

If Rb^{85+} appears, a correction for Rb^{87+} is necessary.

Data can be obtained from 10 nanograms of $\text{Sr}(\text{NO}_3)_2$ or SrSO_4 . SrCl_2 requires a 100-nanogram sample for comparable data.

Sulfur

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	As_2S_3	32	95.0
Sample filament	Tantalum	33	0.760
Filament size	1 × 30 mils	34	4.22
Ionization	Electron bombardment	36	0.0136
Temperature range	<500°C		
Ion measured	AsS^+		

Sample Loading

Place arsenic trisulfide on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

Ions AsS^+ , As_2S^+ , As_2S_2^+ , and As_2S_3^+ are noted, but the best data is obtained at AsS^+ .

Tantalum

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Ta ₂ O ₅	180	0.0123
Sample filament	Rhenium	181	99.9877
Filament size	1 × 15 mils		
Ionization	Electron bombardment		
Temperature range	> 1500°C		
Ion measured	TaO ₂ ⁺		

Sample Loading

Place finely divided tantalum oxide on the filament and partially digest with 1:1 hydrofluoric acid. BE VERY CAREFUL WHEN HANDLING HF. Dry sample with a gentle heat. Cover with a vacuum chamber and pump down to <20 μ pressure. Increase filament temperature until sample begins to reduce to tantalum metal and melt.

Comments

The observed ratio of Ta⁺:TaO⁺:TaO₂⁺ is approximately 1:2:8. Interference from previous tantalum samples has not been observed.

When at low isotopic concentrations, base-line corrections for mass 212 (Ta¹⁸⁰O₂¹⁶)⁺ can be made by comparison with mass 214 and 215 [(Ta¹⁸¹O₂¹⁷)⁺ and (Ta¹⁸¹O₂¹⁸)⁺].

Correction for O¹⁷ may be necessary.

Technetium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	NH_4TcO_4	99	
Sample filament	Iridium		
Filament size	1 × 30 mils		
Ionization	Electron bombardment		
Temperature range			
Ion measured	Tc^+		

Sample Loading

Evaporate a solution of ammonium pertechnetate on the filament and reduce to metal by heating in H_2 , or electroplate technetium on the filament and heat in H_2 to form a tightly adhering layer of technetium.

Comments

Technetium will produce Tc^+ thermally from iridium filaments.

See ORNL-1327, *Mass Spectrometer Study of Technetium*, by J. R. Sites, C. R. Baldock, and L. O. Gilpatrick (August 26, 1952).

Tellurium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Ag_6TeO_6	120	0.089
Sample filament	Tantalum	122	2.46
Filament size	2×60 mils	123	0.87
Ionization	Electron bombardment	124	4.61
Temperature range	$<200^\circ\text{C}$	125	6.99
Ion measured	TeO_2^+	126	18.71
		128	31.79
		130	34.48

Sample Loading

Place silver tellurate on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

Te^+ , TeO^+ , and TeO_2^+ appear. The highest ratio of ion current to hydrocarbon background is at TeO_2^+ .

Ag^+ , AgO^+ , AgO_2^+ and Ag_2^+ are observed but do not interfere.

Interference from previous tellurium samples is a serious problem.

Corrections for O^{17} and O^{18} may be necessary.

Ag_6TeO_6 is prepared by dissolving elemental tellurium in ammonia and hydrogen peroxide, and precipitating tellurium with silver ions from a neutral solution.

Terbium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Tb ₂ O ₃	159	100
Sample filament	Tantalum		
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1400–1700°C		
Ion measured	TbO ⁺		

Sample Loading

Place finely divided terbium oxide on the filament and partially digest with concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

One microgram of terbium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Tb⁺ to TbO⁺ is 1 : >100.

Interference from previous terbium samples has not been observed.

Thallium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	$\text{Tl}(\text{NO}_3)_3$	203	29.50
Sample filament	Tungsten	205	70.50
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1000--1400°C		
Ion measured	Tl^+		

Sample Loading

Place the thallium nitrate solution on the filament and add a drop of saturated boric acid solution. Dry this mixture with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

The Tl^+ beam will decrease soon after it first appears. Additional increase of temperature will produce a stable and higher ion current.

Any Pb contamination will appear as the Tl ions die away.

Interference from previous thallium samples has not been observed.

Tl_2O_3 is converted to nitrate on the filament by addition of nitric acid. Proceed as described for nitrate.

Thorium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	ThO ₂	232	100
Sample filament	Tantalum		
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	> 1700°C		
Ion measured	ThO ⁺		

Sample Loading

Place finely divided thorium oxide on the filament and partially digest the sample with a drop of concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

Interference from previous thorium samples has not been observed.

Thulium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Tm ₂ O ₃	169	100
Sample filament	Tantalum		
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1400–1700°C		
Ion measured	Tm ⁺		

Sample Loading

Place finely divided thulium oxide on the filament and partially digest with concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

One microgram of thulium produces an ion current of 10^{-10} amp for an hour. The observed ratio of Tm⁺ to TmO⁺ is >100:1.

Interference from previous thulium samples has not been observed.

Tin

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Sn	112	0.96
Sample filament	Tungsten	114	0.66
Filament size	1 × 30 mils	115	0.35
Ionization	Electron bombardment	116	14.30
Temperature range	800–1200°C	117	7.61
Ion measured	Sn ⁺	118	24.03
		119	8.58
		120	32.85
		122	4.72
		124	5.94

Sample Loading

Place finely divided tin metal on the filament. Cover with a vacuum chamber and pump down to 20μ pressure. Increase filament temperature until sample melts.

Comments

The Sn⁺ intensity will decrease soon after it first appears. Additional increase of temperature will produce a stable and higher ion current.

Memory from previous tin samples has not been observed.

The ratio of Sn⁺ to SnO⁺ varies widely.

Titanium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	TiO ₂	46	7.93
Sample filament	Tantalum	47	7.28
Filament size	1 × 30 mils	48	73.94
Ionization	Thermal	49	5.51
Temperature range	1300–1600°C	50	5.34
Ion measured	TiO ⁺		

Sample Loading

Place finely divided titanium oxide on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

The ratio of Ti⁺ to TiO⁺ is approximately 1:5.

Interference from previous titanium samples has not been observed.

Corrections for O¹⁷ and O¹⁸ may be necessary.

Tungsten

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	WO ₃	180	0.135
Sample filament	Tantalum	182	26.41
Filament size	1 × 30 mils	183	14.40
Ionization	Electron bombardment	184	30.64
Temperature range	900--1200°C	186	28.41
Ion measured	WO ₂ ⁺		

Sample Loading

Place finely divided tungsten oxide on the filament and slurry with a saturated boric acid solution. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

The WO₂⁺ beam will decrease soon after it first appears. Additional increase of temperature will produce a stable and higher ion intensity. The H₃BO₃ tends to stabilize the ion beam.

Interference from previous tungsten samples has not been observed.

Corrections for O¹⁷ and O¹⁸ may be necessary.

G. H. Palmer* reports that the thermal ion, Na₂WO₄⁺, has been obtained from sodium tungstate on a tantalum filament.

Masses of Isotopic Ions

W ⁺	WO ⁺	WO ₂ ⁺
180	196	212
182	198	214
183	199	215
184	200	216
186	202	218

*G. H. Palmer, in *Advances in Mass Spectrometry*, ed. by J. D. Waldron, Pergamon Press, New York, 1959.

Vanadium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	V_2O_5	50	0.24
Sample filament	Tantalum	51	99.76
Filament size	1 × 30 mils		
Ionization	Thermal		
Temperature range	1400–1600°C		
Ion measured	V^+		

Sample Loading

Place vanadium oxide on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

Interference from previous vanadium samples has not been observed.

The ratio of V^+ to VO^+ is approximately 10:1.

Uranium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	U_3O_8	234	0.0056
Sample filament	Tantalum	235	0.7205
Filament size	1 × 30 mils	238	99.2739
Ionization	Thermal		
Temperature range	1500–1700°C		
Ion measured	UO_2^+		

Sample Loading

Place finely divided uranium oxide on the filament and partially digest the sample with a drop of concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat ($\sim 750^\circ\text{C}$).

Comments

Approximately 50 μg of uranium is normally used for analysis.

Data have been obtained from $<10\text{-}\mu\text{g}$ samples.

When UO_2^+ first appears, the intensity sometimes increases slowly for a few minutes and then rapidly. If this occurs the filament temperature must be reduced to avoid losing the sample.

Interference from previous uranium samples has not been observed.

Corrections for O^{17} and O^{18} may be necessary.

Small samples (<1 to 10 nanograms) are analyzed using an electron multiplier receiver. The sample is loaded as uranium nitrate on a tungsten filament. When the pressure in the source area is approximately 1×10^{-7} torr, benzene vapor is introduced to increase the pressure to 5×10^{-5} . The sample and filament are then conditioned by increasing the filament temperature to 1600°C for 30 sec. U^+ is the ion measured, and the temperature range is 1750 to 1850°C .

Ytterbium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	Yb ₂ O ₃	168	0.135
Sample filament	Tantalum	170	3.03
Filament size	1 × 30 mils	171	14.31
Ionization	Thermal	172	21.82
Temperature range	1300–1700°C	173	16.13
Ion measured	Yb ⁺	174	31.84
		176	12.73

Sample Loading

Place finely divided ytterbium oxide on the filament and partially digest with concentrated nitric acid. Dry sample with a gentle heat; then increase filament temperature to a dull-red heat (~750°C).

Comments

One microgram of ytterbium produces an ion current of 10^{-10} amp for more than an hour.

The observed ratio of Yb⁺ to YbO⁺ is >100:1.

Interference from previous ytterbium samples has not been observed.

Zinc

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	ZnO	64	48.89
Sample filament	Tantalum	66	27.81
Filament size	2 × 60 mils	67	4.11
Ionization	Electron bombardment	68	18.57
Temperature range	800–1200°C	70	0.62
Ion measured	Zn ⁺		

Sample Loading

Place zinc oxide on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

The Zn⁺ beam will decrease soon after it first appears. Additional increase of temperature will produce a stable and higher ion current. Hydrocarbon background frequently causes interference.

Interference from previous zinc samples is a very serious problem.

ZnI₂ and ZnF₂ are compounds that have been used for analysis.

Zirconium

Preferred Conditions		Relative Abundance of Isotopes	
		Isotope	Atom %
Compound	ZrO ₂	90	51.46
Sample filament	Tantalum	91	11.23
Filament size	1 × 30 mils	92	17.11
Ionization	Thermal	94	17.40
Temperature range	1600--1900°C	96	2.80
Ion measured	ZrO ⁺		

Sample Loading

Place finely divided zirconium oxide on the filament and slurry with distilled water. Dry sample with a gentle heat.

Comments

The ZrO⁺ beam will decrease soon after it first appears. Additional increase of temperature will produce a stable and higher ion current.

The observed ratio of Zr⁺ to ZrO⁺ is approximately 1:10.

Interference from previous zirconium samples has not been observed.

Corrections for O¹⁷ and O¹⁸ may be necessary.

Preferred Conditions	Relative Abundance of Isotopes	
	Isotope	Atom %
Compound		
Sample filament		
Filament size		
Ionization		
Temperature range		
Ion measured		

Sample Loading**Comments**

Preferred Conditions	Relative Abundance of Isotopes	
	Isotope	Atom %
Compound Sample filament Filament size Ionization Temperature range Ion measured		

Sample Loading

Comments

Preferred Conditions	Relative Abundance of Isotopes	
	Isotope	Atom %
Compound		
Sample filament		
Filament size		
Ionization		
Temperature range		
Ion measured		

Sample Loading

Comments

Preferred Conditions	Relative Abundance of Isotopes	
	Isotope	Atom %
Compound		
Sample filament		
Filament size		
Ionization		
Temperature range		
Ion measured		

Sample Loading**Comments**

Preferred Conditions	Relative Abundance of Isotopes	
	Isotope	Atom %
Compound		
Sample filament		
Filament size		
Ionization		
Temperature range		
Ion measured		

Sample Loading

Comments

Preferred Conditions	Relative Abundance of Isotopes	
	Isotope	Atom %
Compound Sample filament Filament size Ionization Temperature range Ion measured		

Sample Loading

Comments



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