

Standard Conditions for Flame Spectroscopy

1. Ag	Silver	34. Na	Sodium, Natrium
2. Al	Aluminium	35. Nb	Niobium
3. As	Arsenic	36. Nd	Neodymium
4. Au	Gold	37. Ni	Nickel
5. B	Boron	38. Os	Osmium
6. Ba	Barium	39. Pb	Lead
7. Be	Beryllium	40. Pd	Palladium
8. Bi	Bismuth	41. Pr	Praseodymium
9. Ca	Calcium	42. Pt	Platinum
10. Cd	Cadmium	43. Rb	Rubidium
11. Co	Cobalt	44. Re	Rhenium
12. Cr	Chromium	45. Rh	Rhodium
13. Cs	Cesium	46. Ru	Ruthenium
14. Cu	Copper	47. Sb	Antimony
15. Dy	Dysprosium	48. Sc	Scandium
16. Er	Erbium	49. Se	Selenium
17. Eu	Europium	50. Si	Silicon
18. Fe	Iron	51. Sm	Samarium
19. Ga	Gallium	52. Sn	Tin
20. Gd	Gadolinium	53. Sr	Strontium
21. Ge	Germanium	54. Ta	Tantalum
22. Hf	Hafnium	55. Tb	Terbium
23. Hg	Mercury	56. Te	Tellurium
24. Ho	Holmium	57. Ti	Titanium
25. In	Indium	58. Tl	Thallium
26. Ir	Iridium	59. Tm	Thulium
27. K	Potassium, Kalium	60. U	Uranium
28. La	Lanthanum	61. V	Vanadium
29. Li	Lithium	62. W	Tungsten, Wolfram
30. Lu	Lutetium	63. Y	Yttrium
31. Mg	Magnesium	64. Yb	Ytterbium
32. Mn	Manganese	65. Zn	Zinc
33. Mo	Molybdenum	66. Zr	Zirconium

Silver

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Silver metal strip or wire		99.99%
Silver nitrate	AgNO ₃	99.99%

Solution Technique

Dissolve 1.000 g of silver in 20 ml of 1:1 nitric acid and dilute quantitatively to 1 litre to give 1000 µg/ml Ag.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	3 mA
Fuel	acetylene
Support	air
Flame Stoichiometry	oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum/ Working Range µg/ml	Typical Sensitivity µg/ml
328.1	0.2	1 – 5	0.036
338.3	0.2	3 – 12	0.072

Detection Limit

0.003 µg/ml at 328.1 nm using an air-acetylene flame.

Flame Emission

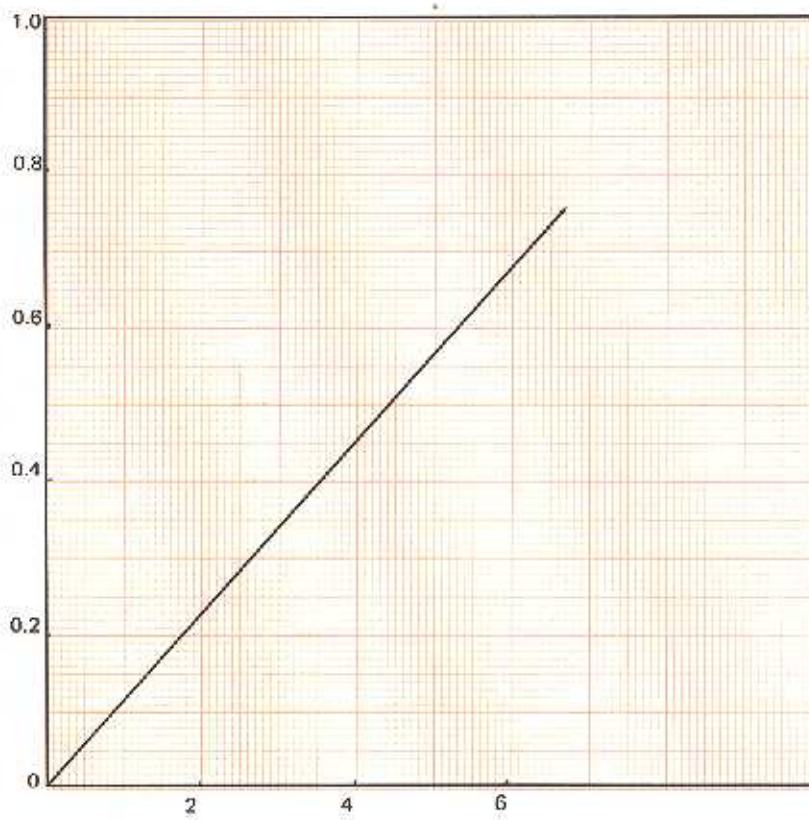
Wavelength	328.1 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit data not available.	

INTERFERENCES

No chemical interferences have been observed in air-acetylene flames.

NOTES

1. Ag Silver



CONCENTRATION $\mu\text{g/ml}$

Aluminium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Aluminium metal (wire form) 99.99%

Solution Technique

Dissolve 1,000 g in 20 ml hydrochloric acid with the addition of a trace of a mercury salt to catalyse the reaction.

Dilute to 1 litre to give 1000 µg/ml Al.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm high.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity <i>A=0.0044</i> µg/ml
309.3	0.2	40-200	0.76
396.1	0.5	50-250	1.1
237.3	0.5	200-800	3.9
236.7	0.5	250-1000	5.3
257.4	0.5	400-1600	8.2
256.8	0.5	650-2600	12.8

Detection Limit

0.04 µg/ml at 309.3 nm using a nitrous oxide-acetylene flame. By reducing the fuel flow slightly and increasing lamp current, an improvement in signal-noise ratio is achieved.

Flame Emission

Wavelength	396.1 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry (Note 1)	reducing; red cone 1-2 cm high
Burner Height (Note 1)	5-8 mm
Detection Limit	0.02 µg/ml

NOTE 1: Adjust for optimum performance.

Aluminium emission appears in the centre of a strongly emitting CN band in the flame. Major factors in the determination at low levels of detection are the adjustment of fuel-support ratio and burner height.

The use of flame emission is only recommended for very low concentrations of aluminium in solutions of simple aqueous matrix.

INTERFERENCES

Aluminium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride to give a final concentration of 2000 µg/ml potassium. The addition of a readily ionizable element such as potassium overcomes enhancement interference from other alkali metals.

Enhancement also occurs with the following elements:—

Aluminium solution 100 µg/ml.

Element	Concentration Range (µg/ml)	% Enhancement
Co	50-5000	3-5
Cr	"	1-14
Fe	"	4-15
Mn	"	4-11
Ni	"	*-26
Ti	"	8-14

* -2% at 50 µg/ml

These interferences are dependent on flame conditions and burner height (1) A fuel rich flame (red cone 1.5-2 cm) decreases the effect.

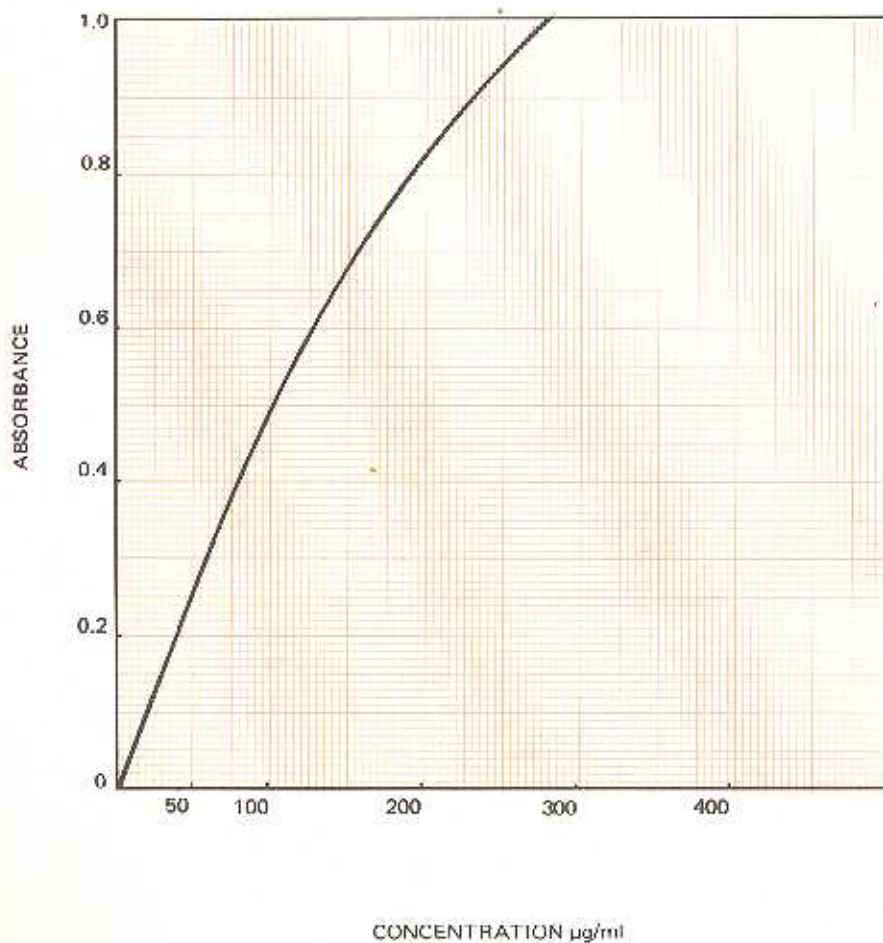
Silicon depresses aluminium absorbance by the formation of an undissociated refractory complex, (2), (3).

The above interference effects can be minimized by matching the sample and standard solutions with respect to the major matrix elements in the sample.

REFERENCES

1. MARKS, J.Y., WELCHER, G.G., *Anal. Chem.* **42**, (9), 1033, 1970.
2. RAMAKRISHNA, T.V. et al. *Anal. Chim. Acta.* **39**, 81, 1967.
3. FERRIS, A.P., et al. *Analyst* **95**, 574, 1970.

2. Al Aluminium



Arsenic

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Arsenious oxide As_2O_3 99.99%

Solution Technique

Dissolve 1.3203 g As_2O_3 in a minimum volume of 20% NaOH and neutralize with nitric acid. Dilute to 1 litre to give 1000 µg/ml As.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current (Note 1)	7mA
Fuel (Note 2)	hydrogen
Support	air
Flame Stoichiometry	reducing

Note 1: Absorbance is critically dependent on lamp current. Lamps must be allowed to warm up for 30 minutes in order to stabilize the line-to-background ratio.

Note 2: Air-hydrogen is recommended in cases where no other species are present. Highly reducing conditions should be employed.

When relatively large amounts of interferent are present, the nitrous oxide-acetylene flame is recommended because it is efficient in preventing chemical interference and has high transparency in the ultraviolet.

The nitrogen-hydrogen entrained air flame can also be used in simple solution matrixes — refer interferences.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
193.7	1.0	50–200	0.78
197.2	1.0	60–250	1.2
189.0	1.0	90–350	1.6

NOTE: The 189.0 nm wavelength is normally used only with an evacuated system because of atmospheric absorption.

Detection Limit

0.25 µg/ml at 193.7 nm using an air-acetylene flame.

A major factor in determining the limit of detection is the change in optical density of the flame as it fluctuates in thermal and environmental air currents. The draught-induced turbulence of the flame is more pronounced with the air-hydrogen flame.

Therefore when carrying out determinations near the limit of detection the use of an air-acetylene flame is recommended. Correction for non-atomic absorption may be necessary.

Flame Emission

Arsenic is not generally determined by flame emission because of the poor emission characteristics of the element.

INTERFERENCES

(a) Spectral Interference

One of the main sources of interference in the determination of arsenic is the molecular absorption of flame gases and solution species at the extreme ultraviolet region of the spectrum where the most sensitive lines for arsenic occur (193.7 nm and 197.2 nm).

This non-atomic absorption can be estimated by means of a continuum light source such as a hydrogen lamp. The element absorbance can then be readily corrected for the non-atomic component.

(b) Chemical Interference

Interference patterns depend largely on the temperature and transparency of the flame. Sensitivity is increased by the use of cool flames such as nitrogen-hydrogen-entrained air but the interferences are in turn markedly increased.

Nitrogen-hydrogen-entrained air flame.

Interferences in this cool flame are susceptible to the type of arsenic species present in solution. Arsenic in the higher oxidation state (e.g. arsenate) shows more severe interference patterns than arsenic in the lower state (arsenious, arsenite). This is due mainly to incomplete dissociation in the cool flame.

Incomplete dissociation of arsenic in any of its forms causes combination of solution ionic species in various sections of the flame and leads to extremely complicated interference patterns. For example, in arsenious solution, nitrate ions depress the absorbance by 18% at pH 1.0 but on increasing the alkalinity to pH 13.0 the absorbance depression increases to 80%.

High molecular absorption effects make interpretation of interference difficult. For example, antimony appears to depress arsenic absorbance by 20% but in fact about 12% of the remaining signal is due to increased non-atomic absorption. That is a total depression of 32%.

Ammonia causes an apparent enhancement but when molecular absorption is taken into account, a net 50% decrease in arsenic absorbance is evident.

In general, the diffusion flame is not recommended for the determination of arsenic except in cases of extremely simple matrix.

Air-hydrogen flame.

Very few chemical interferences occur in this flame. Metals such as copper, nickel, etc. depress the absorbance by 8–10% at concentrations of 10,000 µg/ml.

In complex matrices, correction for non-atomic absorption must be made.

Air-acetylene flame (1) (2)

Aluminium, chromium, manganese, molybdenum, lead and tin all depress the arsenic absorbance from 8–10%. This level of interference can be compensated by matching the matrices of standards and solutions.

Best sensitivity is obtained with a slightly reducing flame.

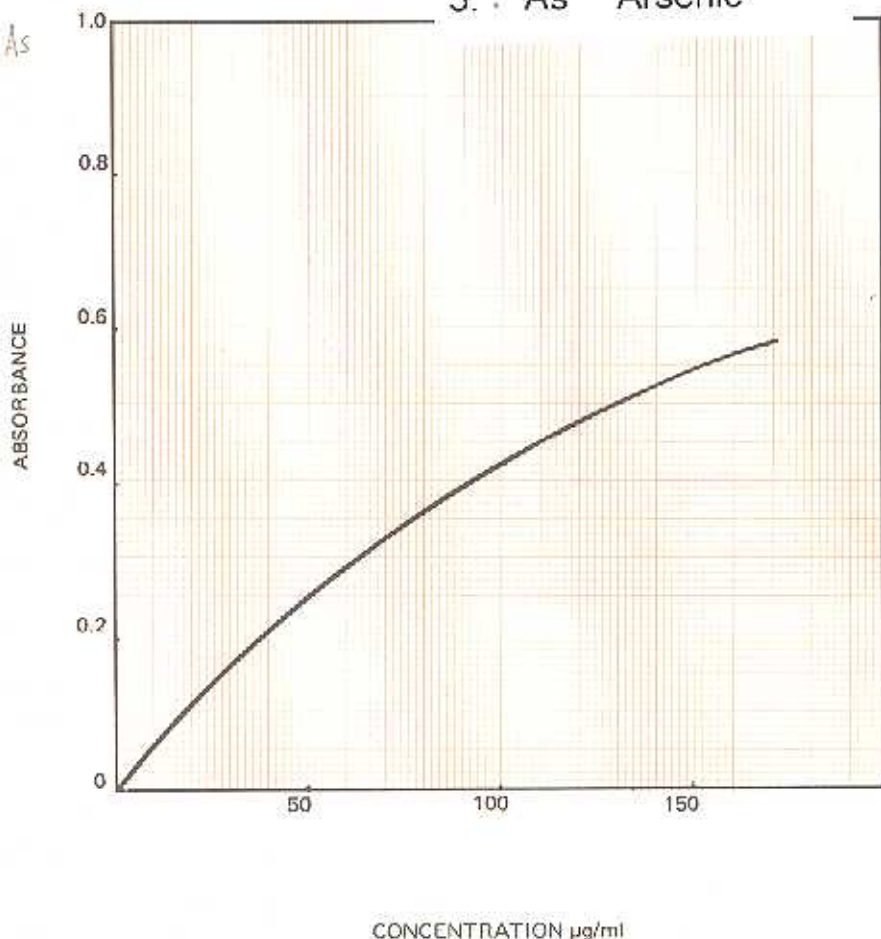
Nitrous oxide-acetylene flame.

No chemical interference has been noted. This flame is recommended for complex matrix solutions.

REFERENCES

1. SMITH, K.E., FRANK, C.W., *Appl. Spectr.* **22** (6), 765, 1968.
2. HWANG, J.Y., SANDONATO, L.M., *Anal. Chem.* **42** (7), 744, 1970.
3. KIRKBRIGHT, G.F., RANSON, L., *Anal. Chem.* **43** (10), 1238, 1971.

3. As Arsenic



NOTES

Gold

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Gold metal 99.9%

Solution Technique

Dissolve 1.000 g of gold foil in a minimum volume of aqua regia. Dilute to 1 litre to give 1000 µg/ml Au.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 4 mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
242.8	0.2	5-20	0.13
267.6	0.2	10-40	0.26

Detection Limit

0.01 µg/ml at 242.8 nm using air-acetylene flame.

Flame Emission

Wavelength 267.6 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection Limit data not available

INTERFERENCES

Few interferences have been noted in air-acetylene flames. However, large excesses of iron, copper, and calcium have been found to suppress the result (1) where gold is extracted into M I B K.

Solvent extraction techniques have been developed (2) which remove any interfering element. The nitrous oxide-acetylene flame will remove any interference but provides a lower sensitivity.

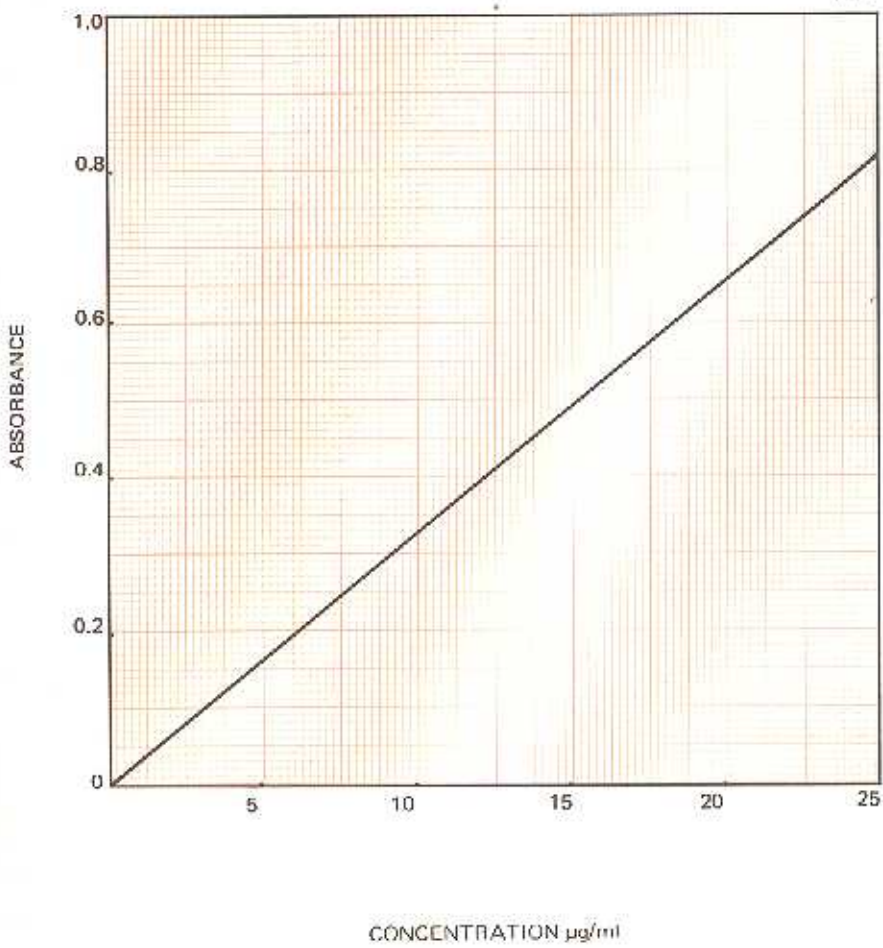
REFERENCES

- (1) HILDON M.A., SULLY G.R., Anal Chim, Acta 54 (1971) 245-251.
(2) ZLATHUIS A., BRUENING W., BAYER E. Anal Chem. 41. 12 (1969), 1693.

NOTES

4. Au Gold

Au



Boron

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Sodium Borate A. R. Grade $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$
Boric Acid A. R. Grade H_3BO_3

Solution Technique

Dissolve 44.095 g of $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ in 500 ml water and dilute to 1 litre to give 5000 $\mu\text{g/ml}$ B.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 15mA
Fuel acetylene
Support nitrous oxide
Flame Stoichiometry reducing: red cone
(Note 1) 2–2.5 cm high

Note 1: Carefully adjust flame stoichiometry and burner position for optimum sensitivity.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	$\mu\text{g/ml}$	$\mu\text{g/ml}$
249.7)	0.1	400–1600	8.4
249.8)			
208.9	0.1	1000–4000	20

Detection Limit

2 $\mu\text{g/ml}$ at 249.7 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength 249.7 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection Limit 5 $\mu\text{g/ml}$

Generally a flame having a red cone 1 cm high with the burner 1-2 mm below the light path will be found to be nearly optimum.

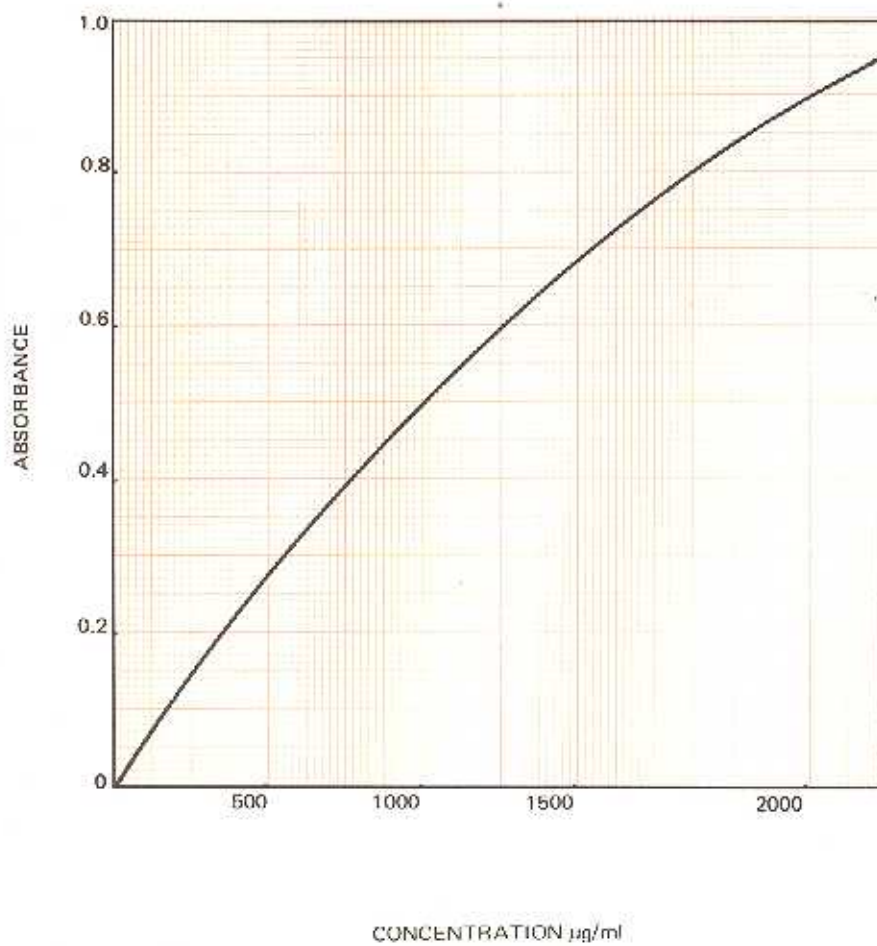
Boron is not usually determined by flame emission because of the poor emission characteristics of the element.

INTERFERENCES

Sodium has been found to cause interference when the ratio of sodium to boron is very high. The effect is usually minimized by adjusting the flame to neutral stoichiometry (red cone 0.5 – 1 cm high) with consequent loss of sensitivity.

NOTES

5. B Boron



Barium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Barium Carbonate A. R. Grade BaCO_3
Barium Chloride A. R. Grade $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$

Solution Technique

Dissolve 1.7785 g of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in water and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Ba.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current*	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing: red cone 2–3 cm high

* Due to the strong emission signals from barium at the resonance wavelength it is recommended that where levels in excess of 10 $\mu\text{g/ml}$ are to be measured the lamp current be increased to 20 mA to minimize photomultiplier noise.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g/ml}$	Typical Sensitivity $\mu\text{g/ml}$
553.6	0.2	10–40	0.20
350.1	0.2	6000–24000	115

Detection Limit

0.01 $\mu\text{g/ml}$ at 553.6 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	553.6 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.004 $\mu\text{g/ml}$

Severe spectral interference will be observed when measuring barium in the presence of calcium due to a strong CaOH band occurring around the same wavelength.

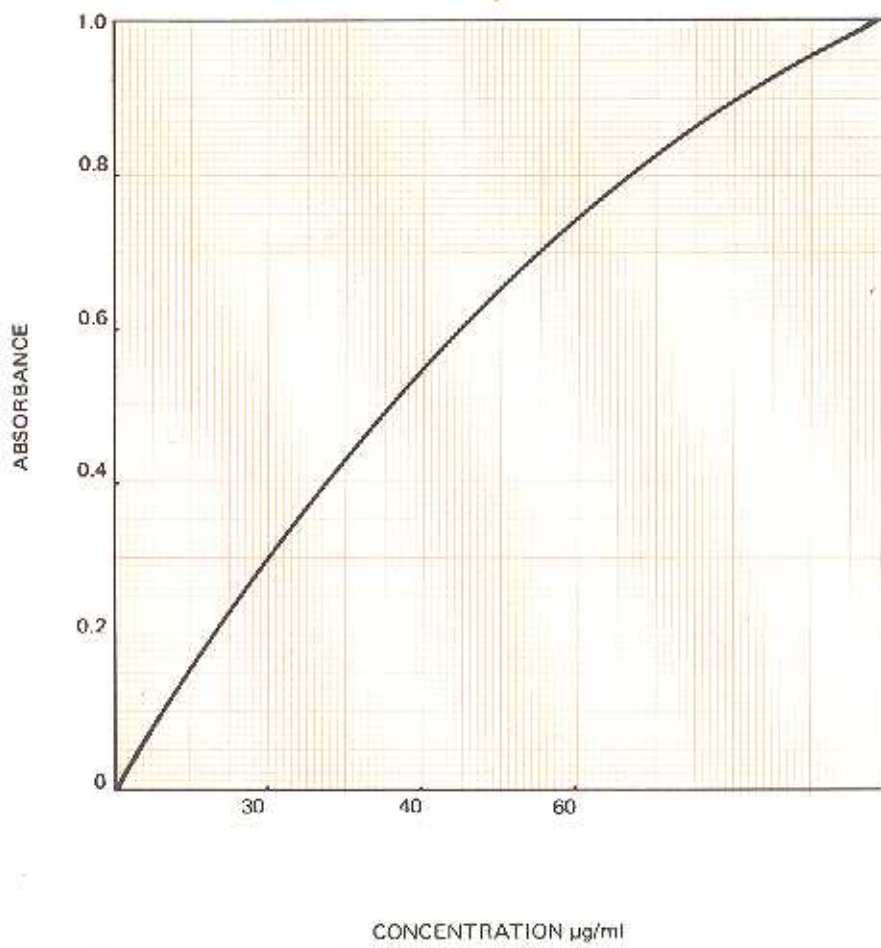
INTERFERENCES

Barium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g/ml}$ potassium in all solutions including the blank. The strong emission from barium falling on the photomultiplier results in considerable increase in shot noise evidenced by increasingly noisy signals as the concentration of barium increases. This may be minimized by working in the HIGH GAIN position and increasing the lamp current to 20 mA. This enables lower photomultiplier gain and hence lower shot noise.

Barium absorbance in an air-acetylene flame is severely depressed by phosphate, silicon and aluminium. This is overcome by the use of a nitrous oxide-acetylene flame.

NOTES

6. Ba Barium



Beryllium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Beryllium metal strip or wire	99.99%
Beryllium oxide	99.99%

Solution Technique

Dissolve 1.000 g of beryllium metal in 20 ml of 1:1 sulphuric acid containing a trace of hydrochloric acid. Dilute to 1 litre to give 1000 µg/ml Be.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	5mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing: red cone 1-2 cm.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	µg/ml	µg/ml
234.9	0.5	1 - 5	.017

Detection Limit

.002 µg/ml at 234.9 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	234.9 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.03 µg/ml

INTERFERENCES

Sodium and silicon at levels in excess of 1000 µg/ml have been found to severely depress beryllium absorbance.

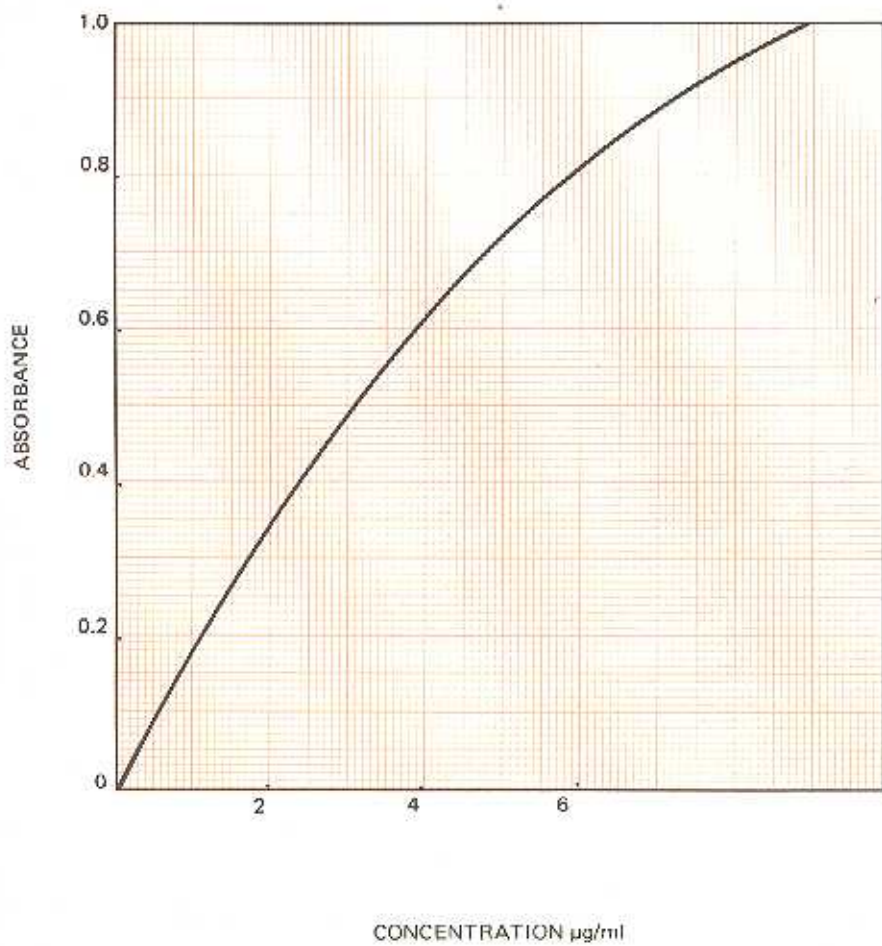
Aluminium has been reported (1, 2) to suppress the absorbance by 25%. This was overcome by the presence of 1.5 g/l of fluoride.

REFERENCES

- (1) RAMAKRISHNA T.V., WEST P.W., ROBINSON J.W. Anal Chim. Acta 39 (1967) 81-87.
- (2) FLEET B., LIBERTY K.V., WEST T.S. Talanta 1970, 17 203-210.

NOTES

7. Be Beryllium



Bismuth

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Bismuth metal strip or wire 99.99%

Solution Technique

Dissolve 1.000 g bismuth metal in a minimum volume of 1:4 hydrochloric acid. Dilute to 1 litre to give 1000 µg/ml Bi.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current (Note 1) 8mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

NOTE 1: Absorbance is relatively independent of lamp current.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
223.1	0.2	10 – 40	0.22
222.8	0.2	40 – 160	0.8
227.7	0.2	300 – 1200	7.0
306.8	0.2	40 – 160	0.9

Detection Limit

0.06 µg/ml at 223.1 nm using an air-acetylene flame.
The use of a slightly more oxidizing flame and increased lamp current will improve signal stability.

Flame Emission

Wavelength 223.1 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous-oxide

Detection limit data not available

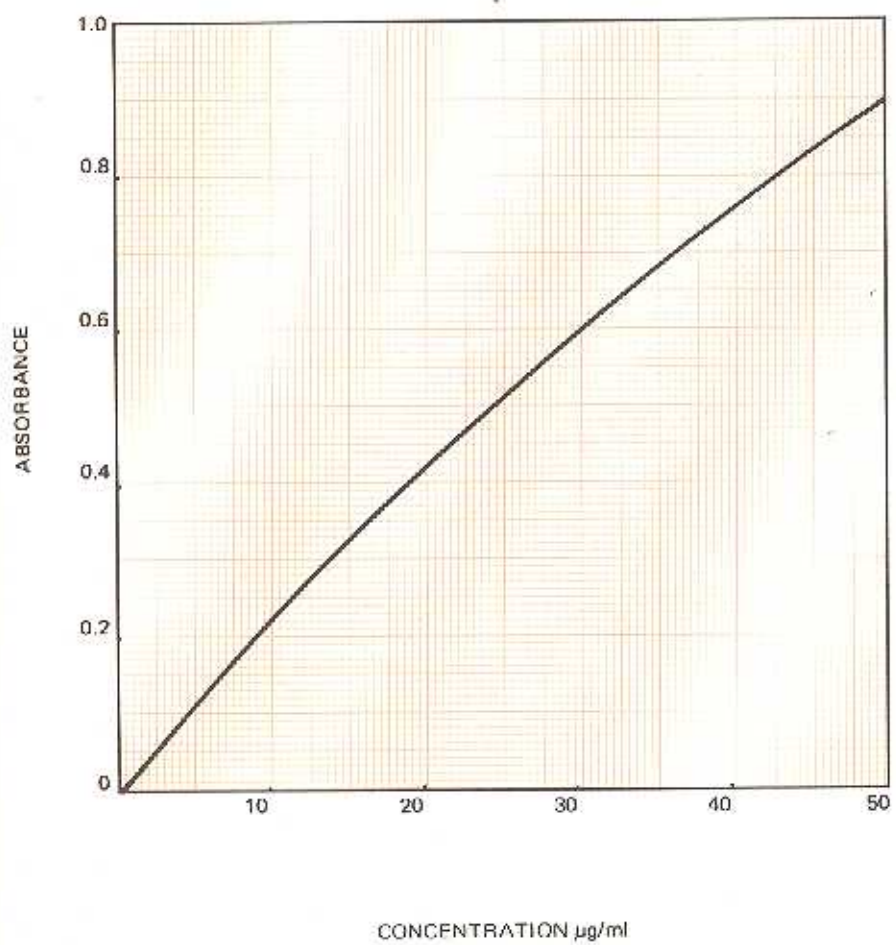
The use of flame emission for bismuth is not recommended because of the poor emission characteristics of the element.

INTERFERENCES

No chemical interference at levels up to 10,000 µg/ml interferent has been reported for the determination of bismuth with an air-acetylene flame.

NOTES

8. Bi Bismuth



Calcium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Calcium carbonate Ca CO₃ 99.9%

Solution Technique

Dissolve 2.497 g of dried calcium carbonate in a minimum volume of 1:4 nitric acid. Dilute to 1 litre to give 1000 µg/ml Ca.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current (Note 1)	3mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing: red cone 1-1.5 cm high

Note 1: For maximum stability increase lamp current to 10–15 mA to overcome photomultiplier shot noise caused by the high emission intensity of calcium. This results in some loss of sensitivity.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
422.7	0.2	1–4	0.021
239.9	0.1	200–800	4.2

Detection Limit

0.0005 µg/ml at 422.7 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	422.7 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.0005 µg/ml

Maximum intensity is obtained with an oxidizing nitrous oxide-acetylene flame (red cone 1 mm high).

INTERFERENCES

Chemical interferences in the air-acetylene flame are pronounced and have been fairly well documented. (1), (2), (3) and (4).

Apart from a 5-10% enhancement caused by the alkali type metals such as sodium, potassium, lithium and magnesium due to suppression of ionization, all other interferences depress the calcium absorbance.

Most of the effects can be reduced somewhat by the use of a lean flame with some loss in sensitivity.

A releasing agent such as strontium (5000 µg/ml) or lanthanum (10,000 µg/ml) will overcome the majority of these effects to a certain extent. Normally this procedure is used in conjunction with the practice of matching sample and standard solutions to obviate combined interference effects.

In the nitrous oxide-acetylene flame the main interference is caused by ionization of calcium itself. This is overcome by the addition of a more readily ionizable element such as potassium (2000–5000 µg/ml).

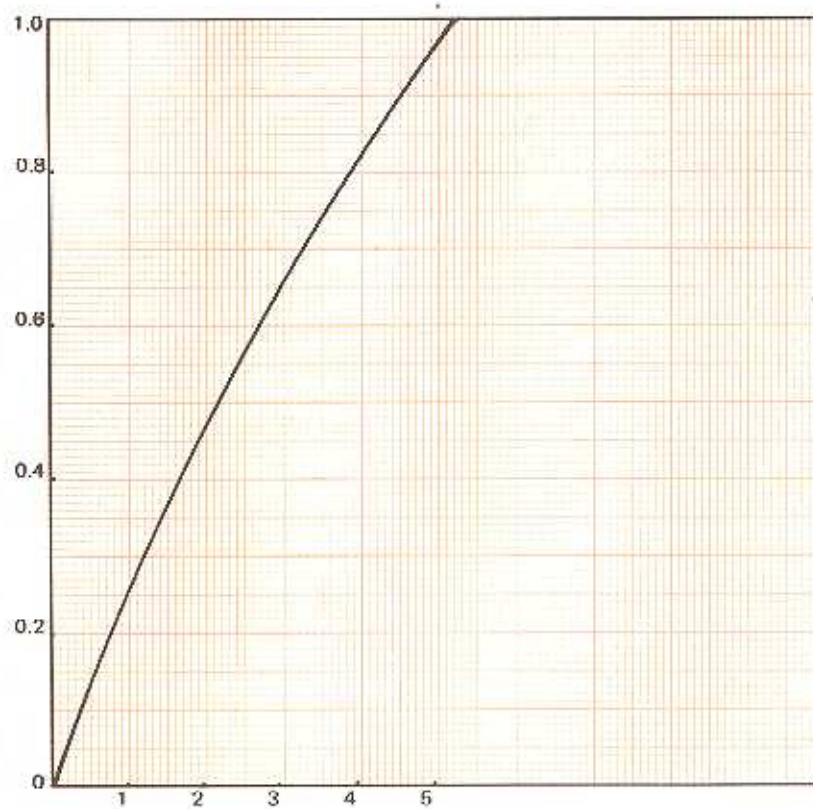
REFERENCES

- ADAMS, P.B., PASSMORE, W.O., *Anal. Chem.* **38**, (4), 630, 1966.
- RAMAKRISHNA, T.V. et al; *Anal. Chim. Acta* **40**, 347, 1968.
- HWANG, J.Y., SANDONATO, L., *Anal. Chim. Acta.* **48**, 188, 1969.
- SASTRI, V.S.; CHAKRABARTI, C.L.; WILLIS, D.E.; *Talanta* **16**, 1093, 1969.

NOTES

9. Ca Calcium

Ca



CONCENTRATION µg/ml

Cadmium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Cadmium metal strip or granules 99.99%
Cadmium sulphate A. R. Grade $3 \text{ Cd SO}_4 \cdot 8 \text{ H}_2\text{O}$

Solution Technique

Dissolve 1.000 g of cadmium in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Cd.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current (Note 1) 3mA
Fuel (Note 2) acetylene
Support air
Flame Stoichiometry oxidizing

Note 1: Absorbance strongly dependent on lamp current.

Note 2: Absorbance strongly dependent on flame stoichiometry. Adjust the fuel flow carefully for maximum sensitivity.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	$\mu\text{g/ml}$	$\mu\text{g/ml}$
228.8	0.5	0.5–2.0	0.011
326.1	1.0	250–1000	4.5

Detection Limit

0.0006 $\mu\text{g/ml}$ at 228.8 nm using an air-acetylene flame.

Flame Emission

Wavelength 326.1 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection limit data not available

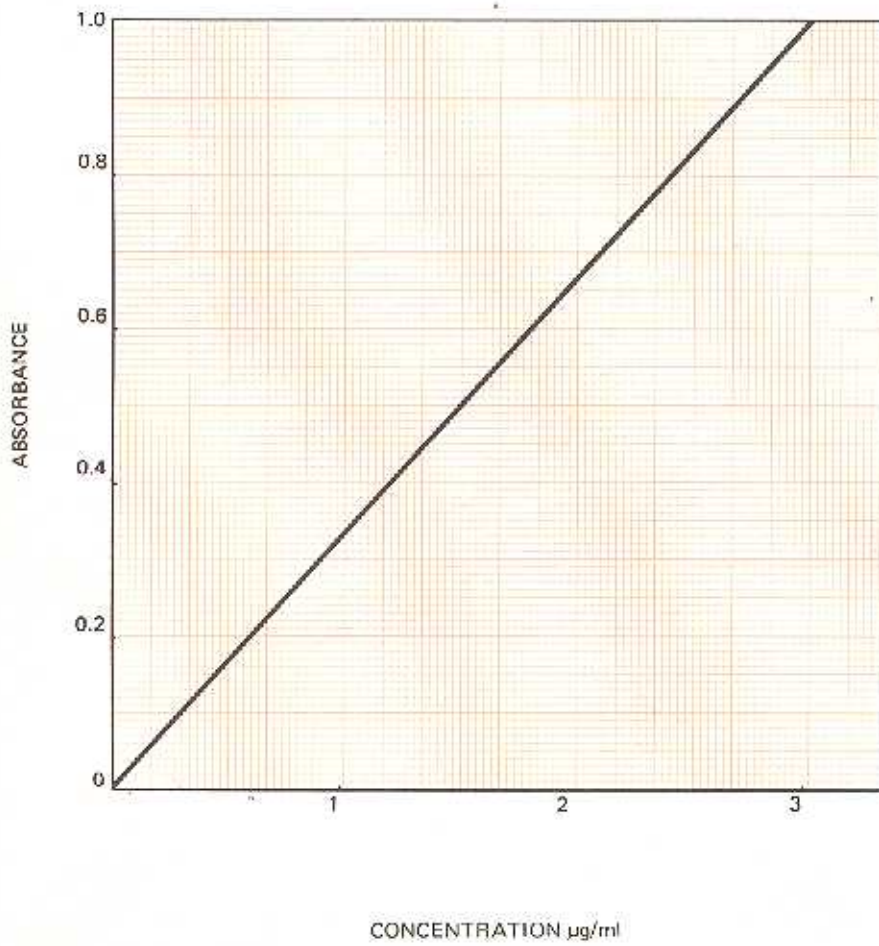
Cadmium is not usually determined by flame emission because of the poor emission characteristics of the element.

INTERFERENCES

No major chemical interference has been reported in the air-acetylene flame.

NOTES

10. Cd Cadmium



Cobalt

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Cobalt metal strip or wire 99.99%

Solution Technique

Dissolve 1.000 g of metal in minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/ml Co.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 5 mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
240.7	0.1	3-12	0.066
304.4	0.2	50-200	1.1
346.6	0.1	100-500	2.6
347.4	0.1	250-1000	4.8
391.0	0.2	7000-30000	147

Detection Limit

0.007 µg/ml at 240.7 nm using an air-acetylene flame.

Flame Emission

Wavelength 345.4 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection limit data not available

INTERFERENCE

Few interferences have been observed for cobalt in an air-acetylene flame.

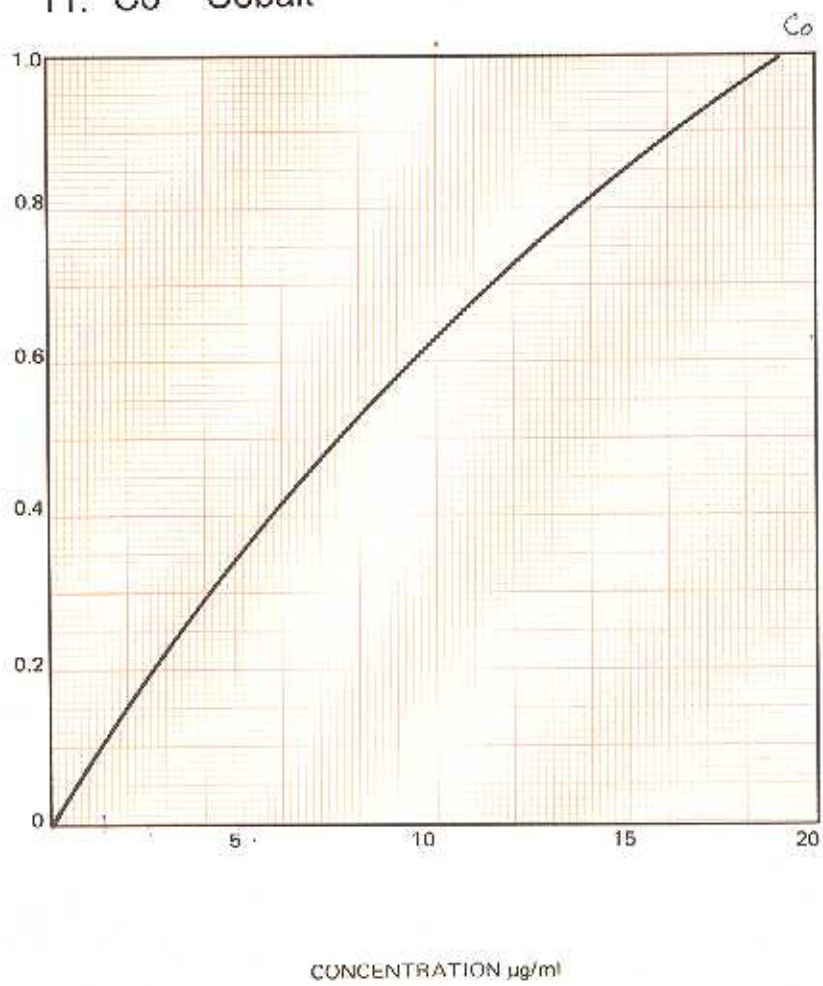
It has been reported that nickel levels in excess of 1500 µg/ml cause severe depression of about 50%. This interference can be avoided by diluting solutions to less than 1500 µg/ml Ni and using the nitrous oxide-acetylene flame. Numerous interferences have been observed in the air-propane flame.

REFERENCES

GINZBERG V.L., SATARINA G.I., Zhv Anal. Chem. **21** 5, 593 1966.

NOTES

11. Co Cobalt



Chromium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Chromium metal strip or wire	99.99%
Potassium Dichromate A. R. Grade	$K_2Cr_2O_7$

Solution Technique

Dissolve 1.000 g of chromium metal in 1:1 hydrochloric acid with gentle heating. Cool and dilute to 1 litre to give 1000 µg/ml Cr.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	5 mA
Fuel	acetylene
Support	air
Flame Stoichiometry	reducing

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	µg/ml	µg/ml
357.9	0.2	2.0-8.0	0.055
425.4	0.2	10-40	0.17
428.9	0.2	25-100	0.41
520.8	0.1	650-2600	11
520.4	0.1	1500-6000	28

Detection Limit

0.005 µg/ml at 357.9 nm using an air-acetylene flame.

Flame Emission

Wavelength	425.4 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection limit data not available	

INTERFERENCE

Cobalt, iron and nickel (particularly in the presence of perchloric acid) have been found to cause depression of chromium absorbance.

This can be overcome by the use of an oxidizing air-acetylene flame or preferably a nitrous oxide-acetylene flame. No ionization suppressant is necessary.

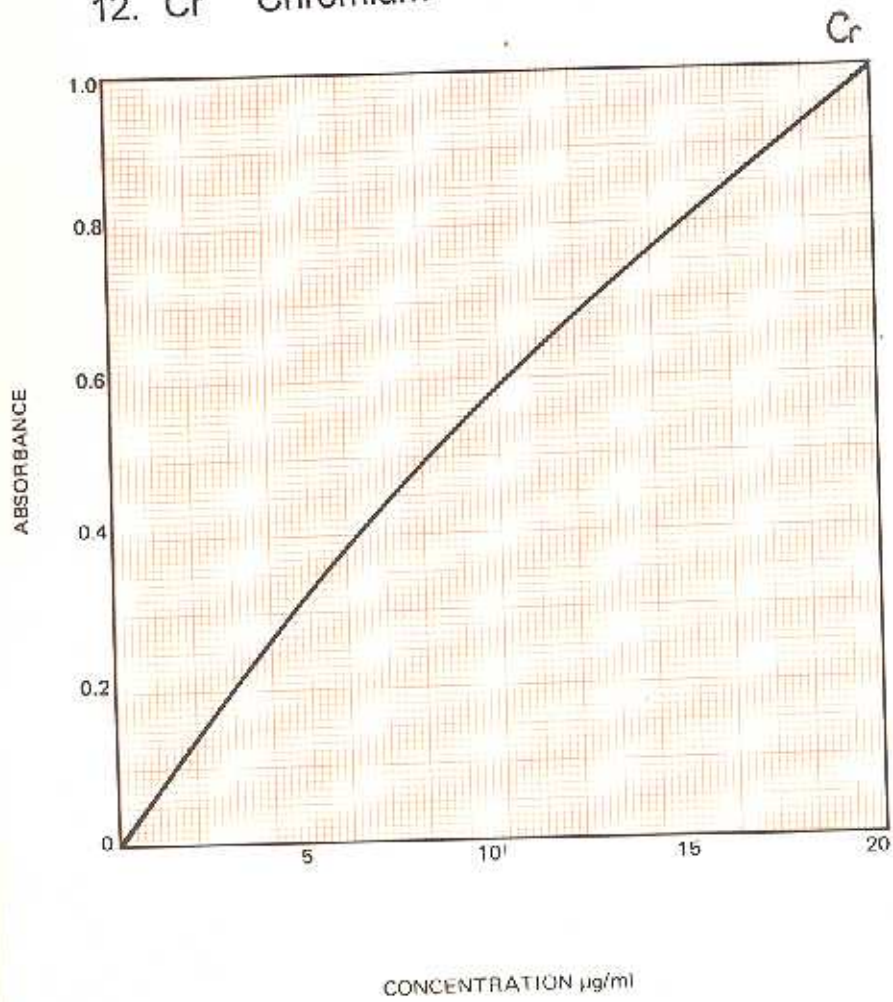
Several authors (1, 2, 3) have found interference in air-acetylene flame from copper, barium, aluminium, magnesium and calcium. The extent of interference is strongly dependent on the flame stoichiometry. Optimization of the stoichiometry or use of the nitrous oxide-acetylene flame can eliminate the interference.

REFERENCES

- 1) YANAGISAWA M., SUZUHRI M., TAKREUCHI T. Anal. Chim. Acta, 52 (1970) 386-389.
- 2) WILSON L., Anal. Chim. Acta, 40 (1968) 503-512.
- 3) TAYLOR R.W. American Laboratory, November 1970, 33-35.

NOTES

12. Cr Chromium



Cesium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Cesium Chloride A. R. Grade CsCl.

Solution Technique

Dissolve 1.2667 g of dried CsCl in water and dilute to 1 litre to give 1000 µg/ml Cs.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20 mA
Fuel	propane
Support	air
Flame Stoichiometry	oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
852.1	1.0	5-20	0.11
894.5	1.0	6-24	0.13
455.5	0.2	300-1200	5.9
459.3	0.1	1000-4000	23

Detection Limit

0.017 µg/ml at 852.1 nm using an air-propane flame. The use of a red sensitive photomultiplier (Hamamatsu R406) is recommended for use above 750 nm.

Flame Emission

Wavelength	852.1 nm
Spectral Band Pass	0.1 nm
Fuel	propane
Support	air

Detection limit data not available

To remove the possibility of second order spectral interference, it is recommended that a filter be used to cut off wavelengths below 600 nm.

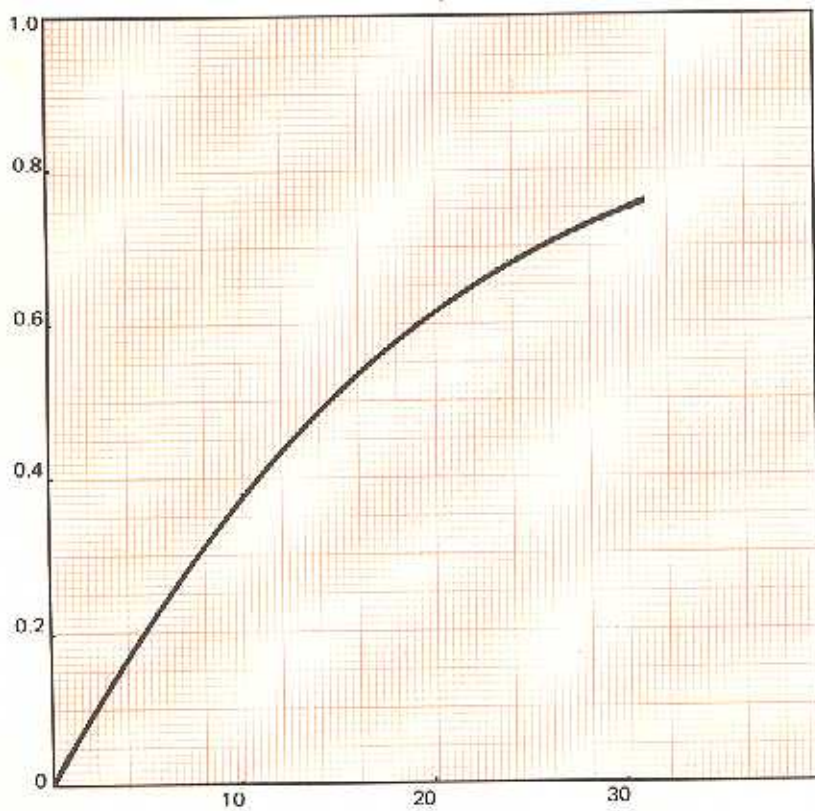
INTERFERENCE

No interference from normal matrix elements has been reported.

Partial ionization of cesium occurs even in the cool air-propane flame; it is recommended that samples and standards should be matched with regard to easily ionized elements such as sodium and potassium to a general level of 1000 - 2000 µg/ml.

NOTES

13. Cs Cesium



CONCENTRATION (µg/ml)

Copper

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Copper metal strip or wire 99.99%

Solution Technique

Dissolve 1.000 g of copper metal in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/ml Cu.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 3mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
324.7	0.2	2-8	0.04
327.4	0.2	6-24	0.14
217.9	0.2	15-60	0.33
218.2	0.2	20-80	0.44
222.6	0.1	70-280	1.5
249.2	0.5	200-800	4.9
244.2	1.0	500-2000	11.2

Detection Limit

0.003 µg/ml at 324.7 nm using an air-acetylene flame.
The use of a very lean (oxidizing) air-acetylene flame will improve signal stability.

Flame Emission

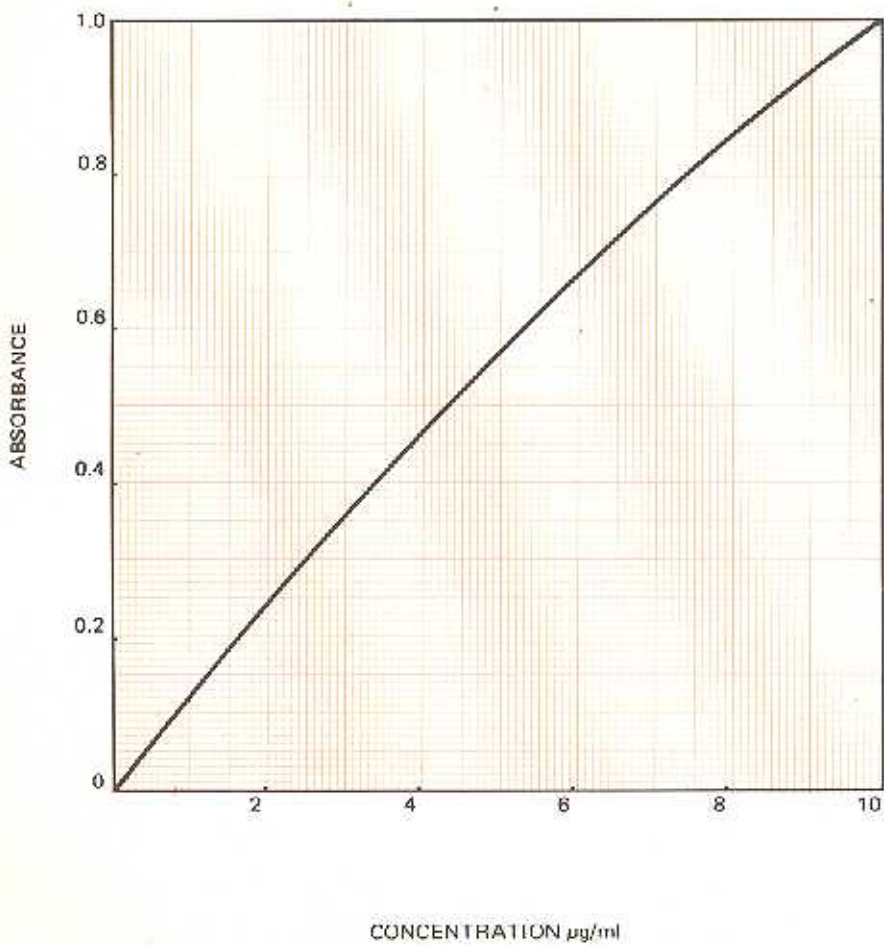
Wavelength 327.4 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection Limit data not available.

INTERFERENCES

No interferences have been reported for copper in the air-acetylene flame but some depression has been noted at high Zn/Cu ratios. This can be minimized by the use of a lean air-acetylene flame or a nitrous oxide-acetylene flame.

NOTES

14. Cu Copper



Dysprosium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Dysprosium oxide Dy₂O₃ 99.9%

Solution Technique

Dissolve 1.148 g of dysprosium oxide in 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/ml Dy.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	15 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing: red cone 1–1.5 cm high.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
421.2	0.1	50–200	0.62
419.5	0.1	65–260	1.4
419.2	0.1	700–2800	16
422.5	0.1	1000–4000	27
421.8	0.1	2500–10000	66

Detection Limit

0.03 µg/ml at 421.2 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	526.5 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.04 µg/ml

Below 10 µg/ml, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

INTERFERENCES

Hydrofluoric acid, aluminium and silicon have been found to depress the absorbance by 90%. The interference is increased by the presence of sodium.

Interferences of this type can usually be overcome by the precipitation of the rare earth oxide and subsequent dissolution in dilute hydrochloric acid.

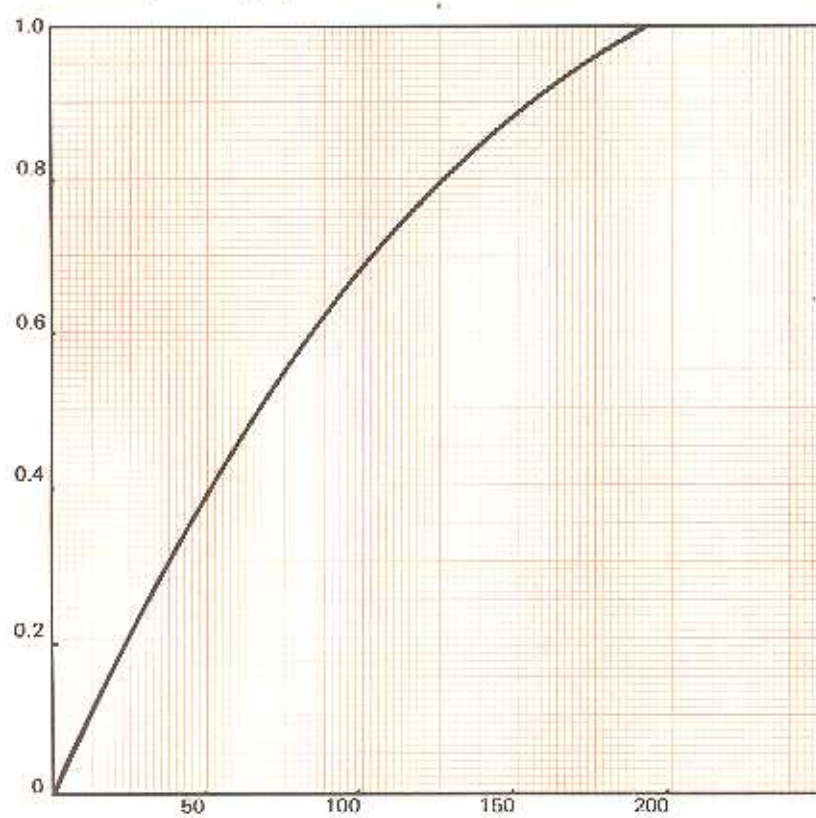
Dysprosium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 µg/ml potassium in all solutions including the blank.

REFERENCES

KINNUNEN O., LINDSJO O., *Chemist-Analyst* 56 (1967) 76–78.

NOTES

15. Dy Dysprosium



CONCENTRATION µg/ml

Erbium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Erbium oxide Er_2O_3 99.9%

Solution Technique

Dissolve 1.1400 g of erbium oxide in 1:1 hydrochloric acid and dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Er.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing red cone 1-2 cm high.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
400.8	0.2	30-120	0.67
389.3	0.2	140-560	3.4
408.8	0.1	250-1000	6.0
402.1	0.1	1000-4000	31

Detection Limit

0.05 $\mu\text{g}/\text{ml}$ at 400.8 nm using nitrous oxide-acetylene flame.

Flame Emission

Wavelength	400.8 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.03 $\mu\text{g}/\text{ml}$

Below 10 $\mu\text{g}/\text{ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from other rare earth elements. At higher concentrations, atomic absorption is normally used.

INTERFERENCES

As in the case of other rare earth elements, severe interference is observed in the presence of hydrofluoric acid, aluminium and silicon, particularly when measured in the presence of sodium.

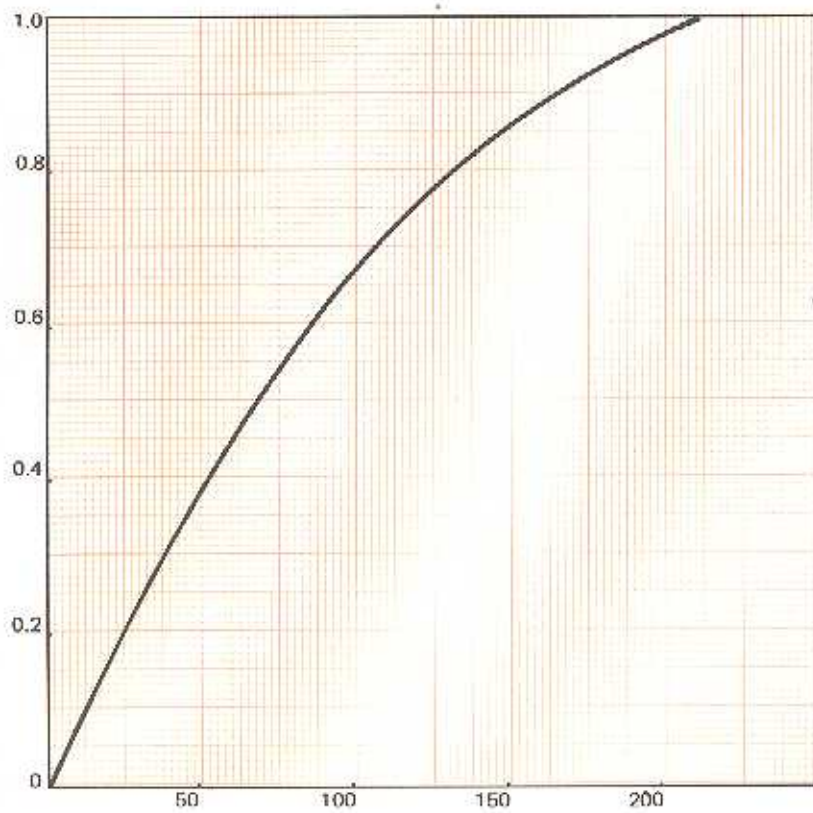
Erbium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank.

REFERENCES

KINNUNEN J., LINDSJO O., *Chemist Analyst* 56 (1967) 76-78.

NOTES

16. Er Erbium



CONCENTRATION $\mu\text{g/ml}$

Europium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Europium oxide Eu_2O_3 99.9%

Solution Technique

Dissolve 1.1579 g of Eu_2O_3 in a minimum volume of 1:1 hydrochloric acid and dilute quantitatively to 1 litre with water to give 1000 $\mu\text{g/ml}$ Eu.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm high.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g/ml}$	Typical Sensitivity $\mu\text{g/ml}$
459.4	0.2	15-60	0.4
333.4	0.2	5000-20000	135

Detection Limit

0.015 $\mu\text{g/ml}$ at 459.4 nm using nitrous oxide-acetylene flame.

Flame Emission

Wavelength	459.4 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.0004 $\mu\text{g/ml}$

Below 10 $\mu\text{g/ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

INTERFERENCE

As in the case of other rare earth elements, severe interference is observed in the presence of hydrofluoric acid, silicon and aluminium when measured in the presence of sodium.

Interference may be minimized by the precipitation of the rare earth oxide and subsequent dissolution in hydrochloric acid.

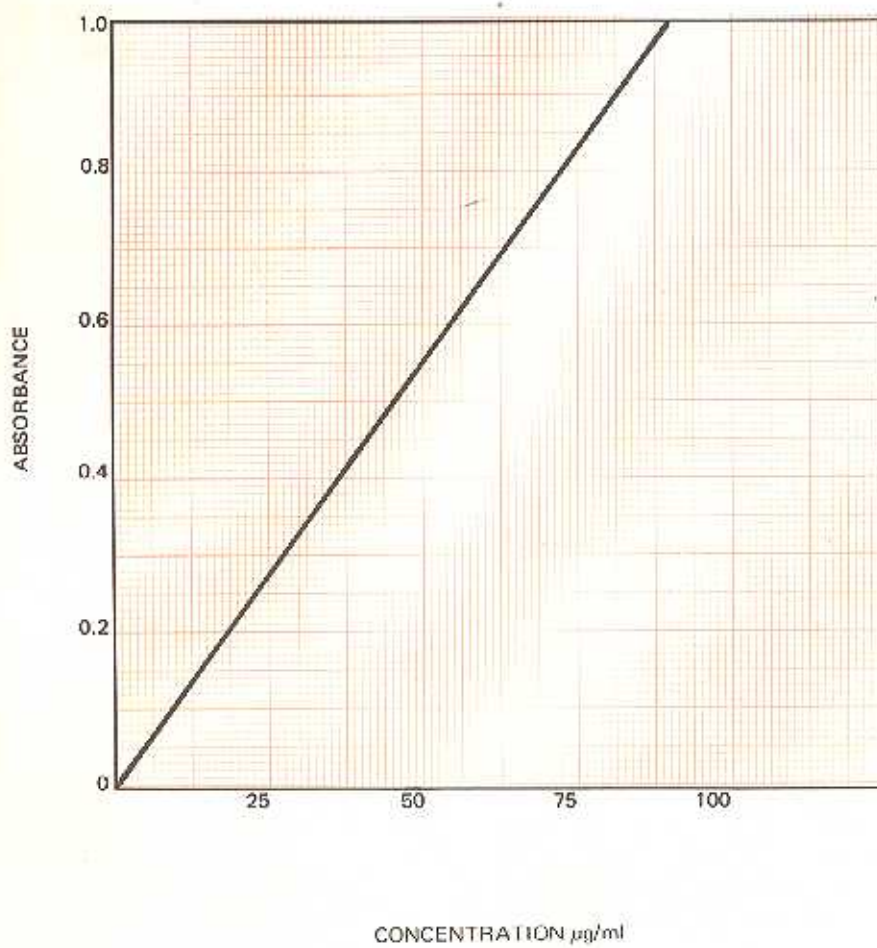
Europium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 $\mu\text{g/ml}$ potassium in all solutions including the blank.

REFERENCES

KINNUNEN J., LINDSJO O., *Chemist-Analyst* 56 (1967) 25-27.

NOTES

17. Eu Europium



Iron

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Iron metal strip or wire 99.9%

Solution Technique

Dissolve 1.000 g of metal in 20 ml of 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/ml Fe.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 5 mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
248.3	0.2	2.5-10	0.062
372.0	0.2	25-100	0.55
386.0	0.2	50-200	0.90
392.0	0.2	800-3200	17

Detection Limit

0.005 µg/ml at 248.3 nm using air-acetylene flame.

Flame Emission

Wavelength 372.0 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support air
Detection Limit data not available

INTERFERENCES

Interference from citric acid has been reported (1) to suppress the absorbance by up to 50% for a citric acid level of 200 µg/ml. The effect is not overcome by adjustment of flame stoichiometry.

The interference has been minimized by measuring the absorption in the presence of phosphoric acid. It is necessary to select an optimum burner height to gain maximum freedom from interference.

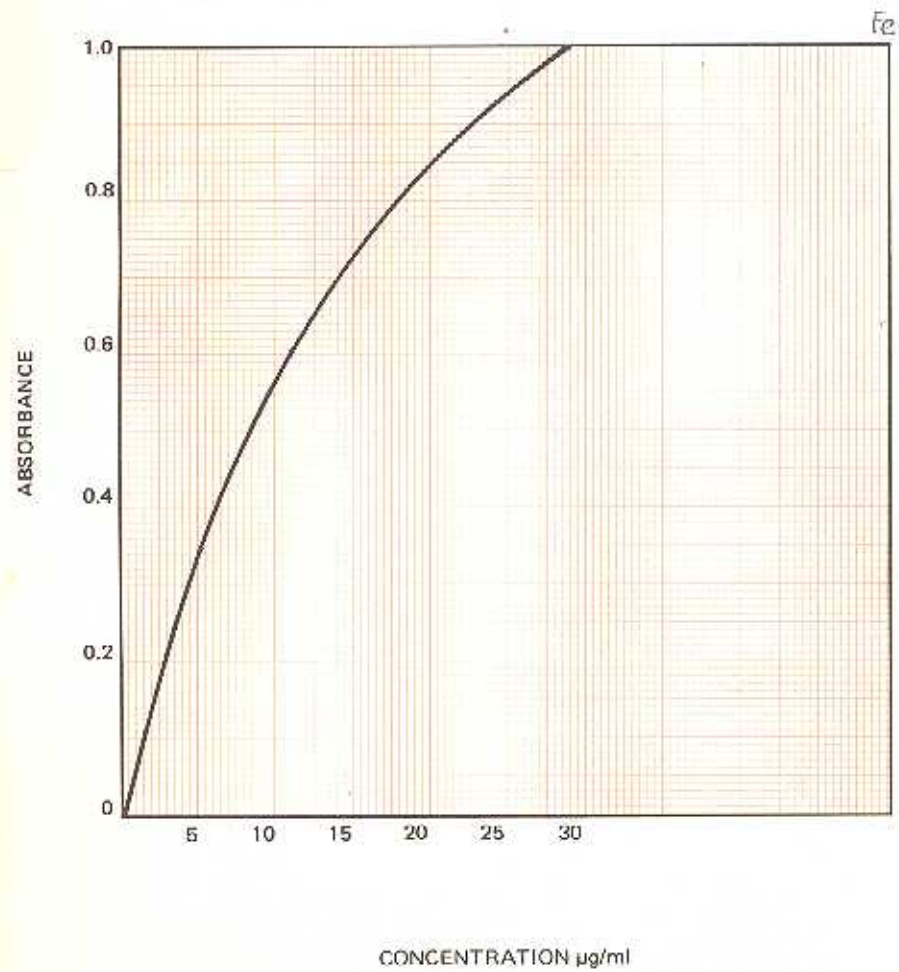
The use of a nitrous oxide-acetylene flame has been found to remove all interference.

REFERENCES

- 1 ROOS J.T.H., PRICE W.J.; Spectrochimica Acta 26B (1971) 279-284.
- 2 VAN LOON J.C., PARISSIS C.M.; Analyst 94 (1969) 1057-1062.

NOTES

18. Fe Iron



Gallium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Gallium metal 99.9%

Solution Technique

Dissolve 1.000 g of gallium metal in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/ml Ga.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current (note 1) 3mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

Note 1: Absorbance is highly dependent on lamp current

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
294.4	0.1	50–200	1.1
287.4	0.2	60–240	1.4
272.0	0.2	1300–5200	32

Detection Limit

0.1 µg/ml at 294.4 nm using an air-acetylene flame.

Flame Emission

Wavelength 403.3 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection Limit data not available

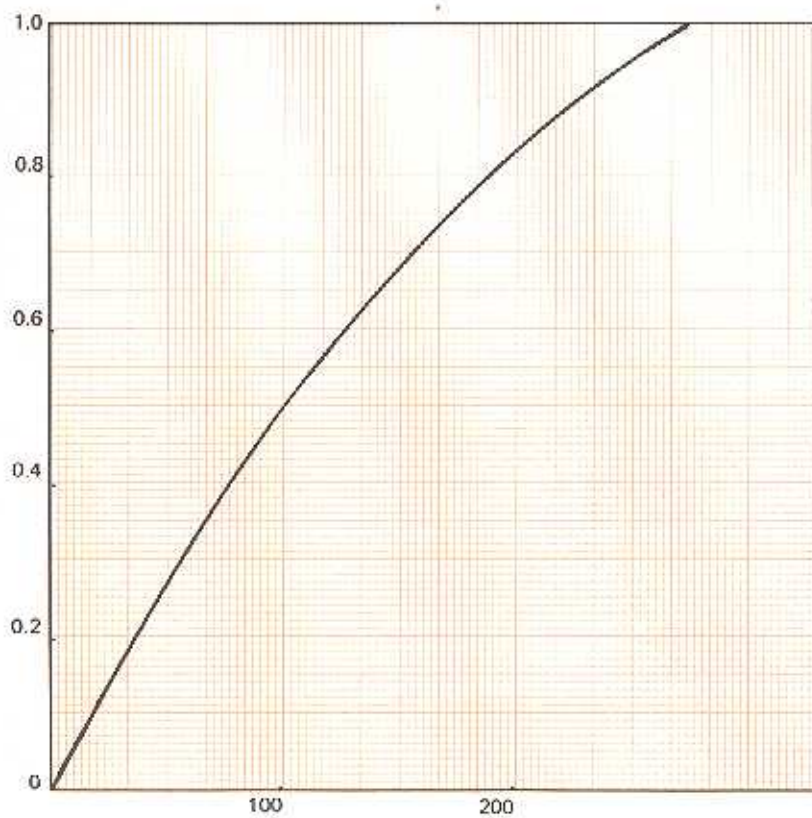
INTERFERENCES

Interferences have not been reported for atomic absorption measurements using the air-acetylene flame. Any interferences may be readily overcome by using the nitrous oxide-acetylene flame with little loss in sensitivity. Gallium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/ml potassium in all solutions including the blank.

For emission measurements, manganese causes spectral interference by emitting at 403.3 nm. This can be overcome by using the 417.2 nm line and establishing the baseline by scanning.

NOTES

19. Ga Gallium



CONCENTRATION µg/ml

Gadolinium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Gadolinium oxide Gd_2O_3 99.9%

Solution Technique

Dissolve 1.1526 g of Gd_2O_3 in a minimum volume of 1:1 hydrochloric acid and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Gd.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	25 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing: red cone 1-2 cm high.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	$\mu\text{g/ml}$	$\mu\text{g/ml}$
368.4	0.1	900-3600	19
405.8	0.1	1500-6000	29
419.1	0.1	4000-16000	74

Detection Limit

3 $\mu\text{g/ml}$ at 368.4 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	461.7 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.1 $\mu\text{g/ml}$

Below 500 $\mu\text{g/ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

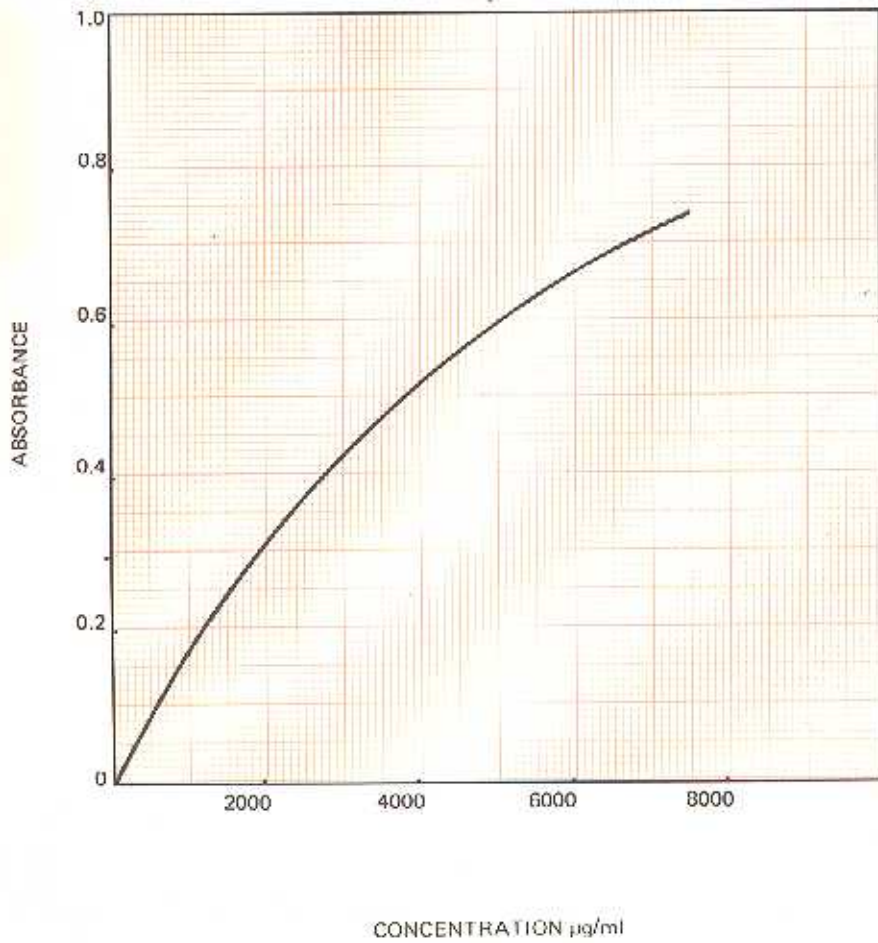
INTERFERENCES

Gadolinium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g/ml}$ potassium in all solutions including the blank.

Hydrofluoric acid, iron, aluminium and silicon each severely depress gadolinium absorbance when present at concentrations in excess of 500 $\mu\text{g/ml}$.

NOTES

20. Gd Gadolinium



Germanium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Germanium metal granules or strip 99.9%

Solution Technique

Dissolve 1.000 g of metal in a minimum volume of aqua regia (1:3 nitric acid: hydrochloric acid) and dilute to 1 litre to give 1000 µg/ml Ge.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current (note 1)	5 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry (note 2)	reducing: red cone 1.2 cm high.

Note 1: Absorbance is slightly dependent on lamp current.

Note 2: Adjust carefully for optimum sensitivity by varying fuel flow.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	µg/ml	µg/ml
265.1)	D 0.5	70–280	1.5
265.2)			
271.0	0.5	150–600	3.3
269.1	0.5	350–1400	7.3
303.9	0.5	1400–4200	30

Detection Limit

0.15 µg/ml at 265.1 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	265.1 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.2 µg/ml

INTERFERENCE

No chemical interferences have been reported.

Hafnium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Hafnium metal (strip) 99.9%

Solution Technique

Dissolve 1.000 g of hafnium metal strip in 5 ml hydrofluoric acid, 5 ml water by the slow DROPWISE addition of 10 ml of concentrated nitric acid. Allow the reaction to subside before each addition. Dilute to 1 litre to give 1000 µg/ml Hf.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working conditions (fixed)

Lamp current 10 mA
Fuel acetylene
Support nitrous oxide
Flame Stoichiometry reducing: red cone
1-2 cm high

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	µg/ml	µg/ml
307.3	0.1	400-1600	10
368.2	0.2	2800-11000	57
377.8	0.2	5000-20000	105

Detection Limit

2.0 µg/ml at 307.3 nm using a nitrous oxide-acetylene flame. The use of a slightly leaner flame (1 cm red cone) can provide a more stable signal.

Flame Emission

Wavelength 368.2 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection Limit 10 µg/ml

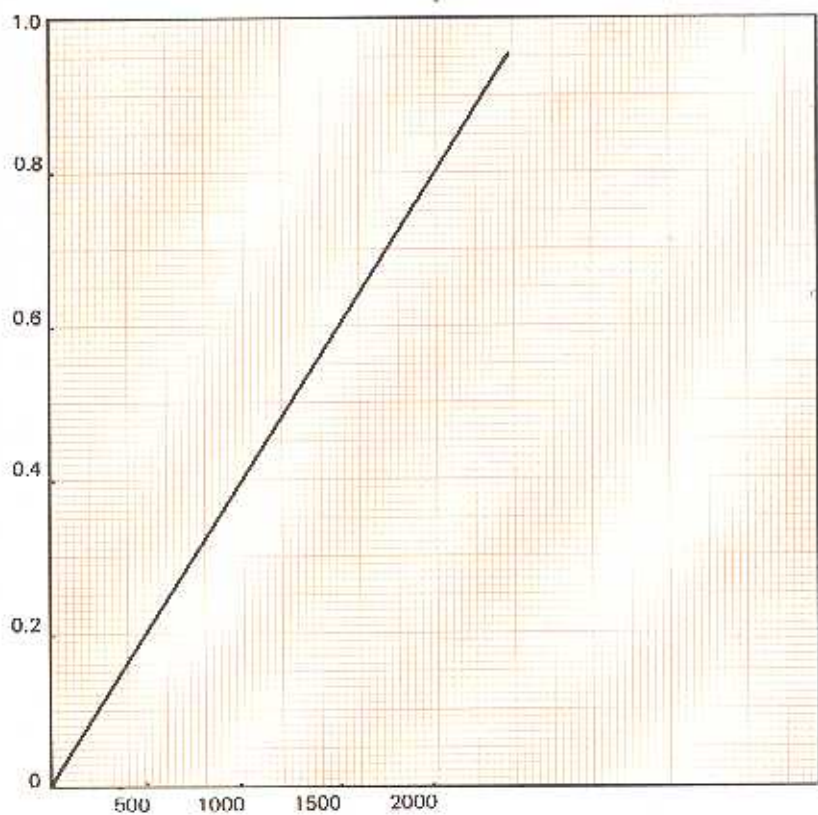
Hafnium is usually determined by atomic absorption.

INTERFERENCES

Hafnium absorbance has been found to be suppressed by sulphuric acid, hydrofluoric acid, alkali metals and alkaline earth metals. Most transition group metals interfere. In many cases the interferents affect the absorbance when present in concentrations as small as 50 µg/ml, with hafnium at 100 µg/ml. The use of an oxidizing nitrous oxide-acetylene flame can minimize most of these effects. However, the development of analytical techniques where the interferent is usually present at minimal concentrations is recommended. Matrices of sample and standard solutions must be carefully matched.

NOTES

22. Hf Hafnium



CONCENTRATION µg/ml

Mercury

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Mercury metal	99.99%
Mercuric Chloride	HgCl ₂ A. R. Grade

Solution Technique

Dissolve 1.354 g mercuric chloride in 10 ml nitric acid. Dilute to 1 litre to give 1000 µg/ml Hg.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	3 mA
Fuel	acetylene
Support	air
Flame Stoichiometry	oxidizing

Working Conditions

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	µg/ml	µg/ml
253.7	0.2	100-400	2.2

Detection Limit

0.2 µg/ml at 253.7 nm using an air-acetylene flame. Using the "cold vapour" technique the detection limit is 0.04 ng/ml or 2 ng absolute.

Flame Emission

Wavelength	253.7 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide

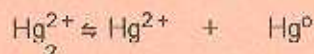
Detection limit data not available

Mercury is not usually determined by flame emission.

INTERFERENCES

Mercury (I) and mercury (II) show different sensitivities in the air-acetylene flame.

Mercury (I) is more sensitive due to the disproportionation reaction.



Elemental mercury is readily atomized with 100% efficiency.

Problems caused by the different sensitivities may be overcome by adding 1 ml of freshly prepared 20% stannous chloride to each solution immediately before analysis. Take note of the dilution of the sample. This method is not recommended unless absolutely necessary as the elemental mercury formed is readily lost by evaporation.

Addition of ascorbic acid has a similar effect, while addition of complexing agents (such as EDTA) assists the disproportionation reaction by removing divalent mercury.

The "cold vapor technique" can be used for trace determinations of mercury. (1) Metallic ions which are reduced to the elemental state by stannous chloride can interfere with the cold vapor method. They can amalgamate with or form stable compounds with mercury. (2) (3)

With both flame and cold vapour techniques it is advisable to check for non-atomic absorption by repeating the analysis with a hydrogen lamp substituted for the hollow cathode lamp.

References:

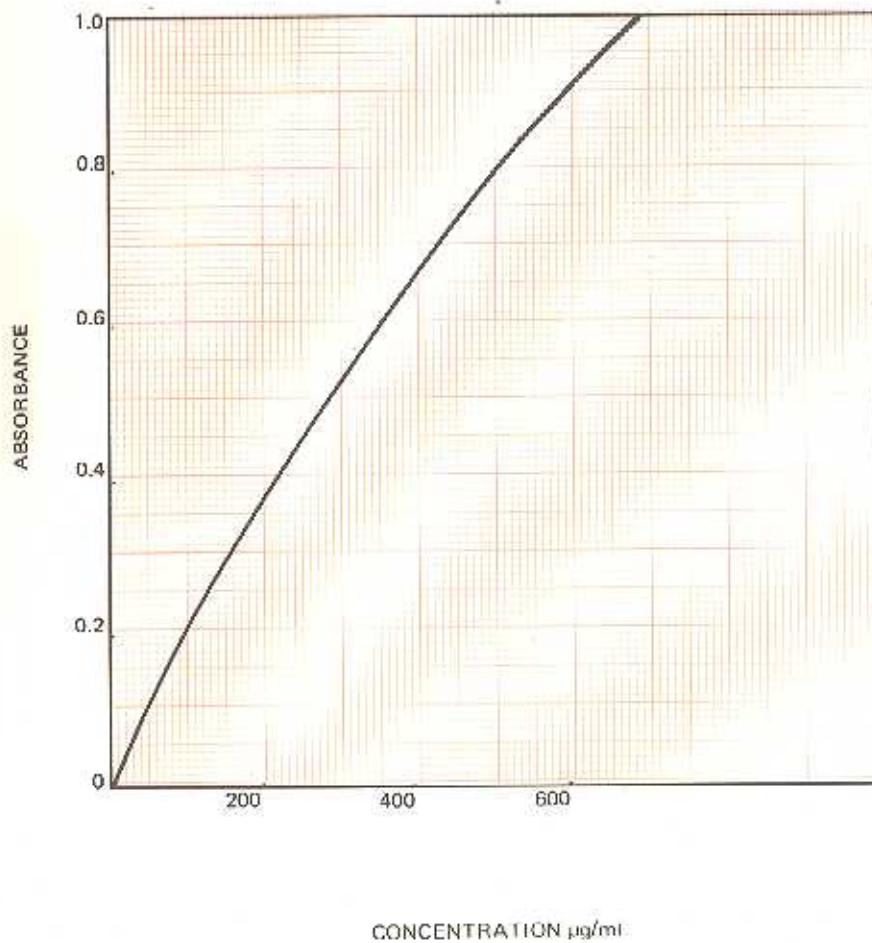
P.T.O.

NOTES

References:

- (1) Stux, R. L., Parker, C. R. "Technical Topics", Varian Techtron Pty. Ltd., June 1972.
- (2) Poleuktov, N.S., Vitkun, R. A., Zhur. Anal. Chim. 18, (1), 37, 1963.
- (3) Poleuktov, N. S., Vitkun, R. A., Zelukova, Y. V., ibid, 19, (8), 937, 1964.

23. Hg Mercury



Holmium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Holmium oxide Ho_2O_3 99.9%

Solution Technique

Dissolve 1.146 g of holmium oxide in a minimum volume of 1:1 hydrochloric acid. Dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Ho.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	15 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing: red cone 1-2 cm high.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	$\mu\text{g}/\text{ml}$	$\mu\text{g}/\text{ml}$
410.4	0.1	40-160	0.8
412.7	0.1	400-1600	8.0
425.4	0.2	3000-12000	66

Detection Limit

0.1 $\mu\text{g}/\text{ml}$ at 410.4 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	559.0 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.02 $\mu\text{g}/\text{ml}$

Below 25 $\mu\text{g}/\text{ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentration atomic absorption is normally used.

INTERFERENCES

Holmium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank. Holmium absorbance has been found to be suppressed by hydrofluoric acid, aluminium, and silicon.

Indium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Indium metal strip or wire 99.9%

Solution Technique

Dissolve 1.000 g of indium metal in the minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/ml In.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 5mA
Fuel (Note 1) acetylene
Support air
Flame Stoichiometry oxidizing

Note 1: Fuel flow must be adjusted carefully for optimum sensitivity and detection limit conditions.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	µg/ml	µg/ml
303.9	0.2	15-60	0.38
271.0	0.1	400-1600	8.3

Detection Limit

0.05 µg/ml at 303.9 nm using an air-acetylene flame.

Flame Emission

Wavelength 451.1 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Flame Stoichiometry oxidizing: red cone 0.5 cm high.
Detection limit 0.04 µg/ml

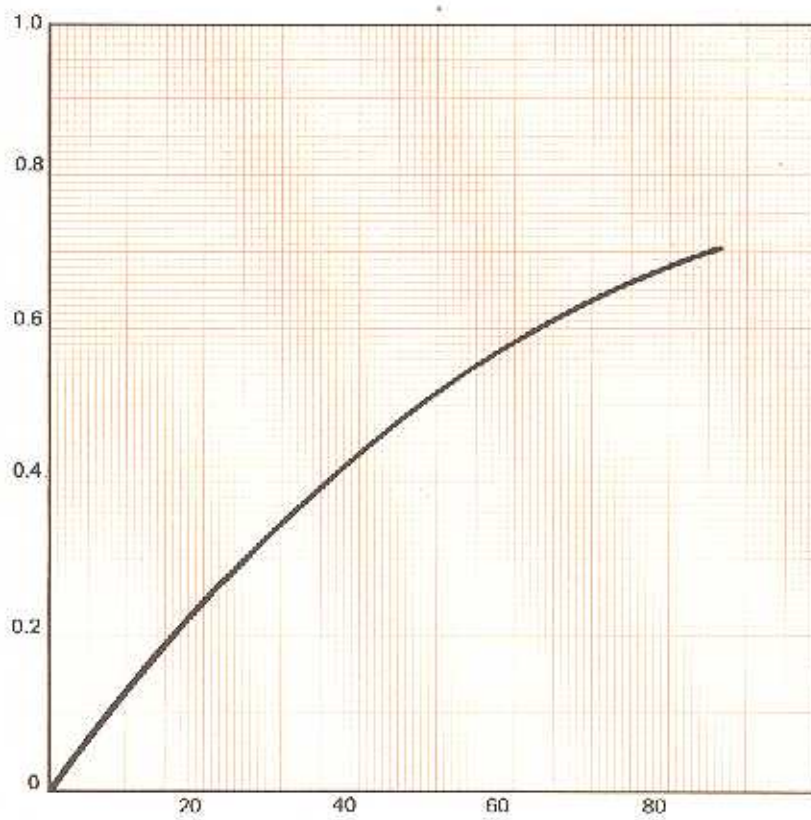
INTERFERENCES

Indium is substantially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/ml potassium in all solutions including the blank.

Elements such as aluminium, silicon, iron, tin and zinc produce small interferences in the nitrous oxide-acetylene flame. These are best overcome by carefully matching the matrix of standards and sample solutions.

NOTES

25. In Indium



CONCENTRATION $\mu\text{g/ml}$

Iridium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Ammonium chloroiridate A. R. $(\text{NH}_4)_2 \text{IrCl}_6$
Iridium powder 99.9%

Solution Technique

Dissolve 2.294 g of $(\text{NH}_4)_2 \text{IrCl}_6$ in a minimum volume of 10% hydrochloric acid. Dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Ir.

RECOMMENDED STANDARD MATERIALS

Atomic Absorption

Working Conditions (fixed)

Lamp current	20 mA
Fuel (note 1)	acetylene
Support	air
Flame Stoichiometry	reducing; slightly luminous

Note 1: Absorbance is highly dependent on the flame stoichiometry and the acetylene flow rate must be carefully optimized.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
208.9	0.1	40-160	0.77
164.0	0.1	120-480	2.5
266.5	0.1	140-560	2.8
254.4	0.1	180-720	3.8

Detection Limit

0.4 $\mu\text{g}/\text{ml}$ at 208.9 nm using an air-acetylene flame. Fuel flow must be adjusted carefully for optimum conditions.

Flame Emission

Wavelength	380.0 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide

Detection Limit data not available

Iridium is usually determined by atomic absorption.

INTERFERENCES

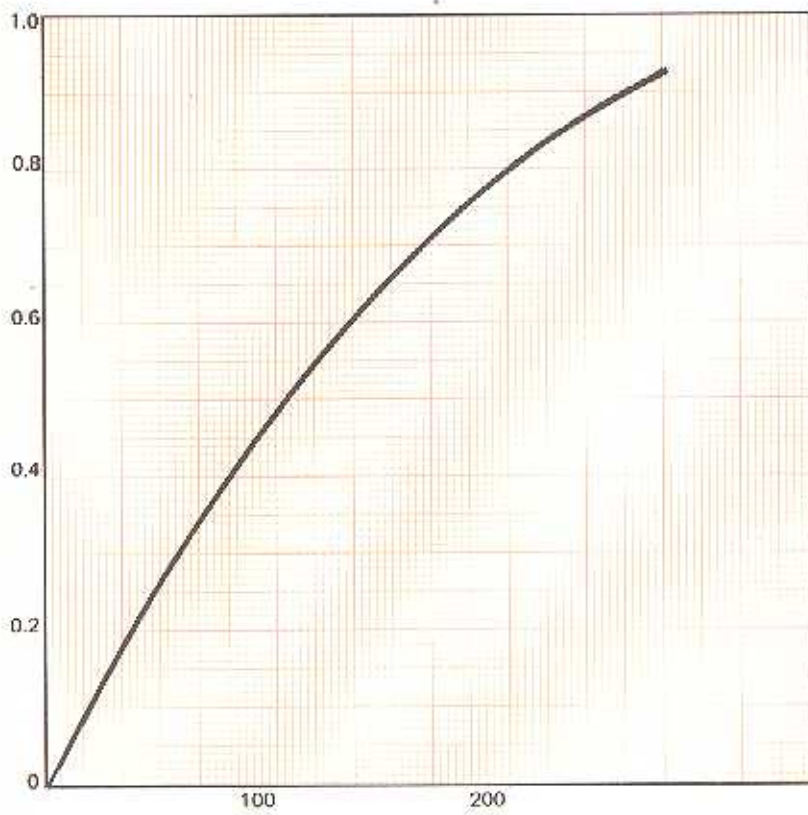
Chemical interference in the air-acetylene flame is extremely diverse (1). In simple solutions, the overall effect of a particular element is markedly dependent on the concentration ratio of iridium to interferent. In general, aluminium, copper, lead, platinum, sodium, potassium, enhance the absorbance by over 50%; titanium, tin, nickel, iron, palladium, tend to depress the signal by amounts greater than 30%. The interference pattern in the complex matrix of ores, minerals etc., is extremely difficult to categorise. Fortunately, an empirical means of overcoming the major interference effects has been reported (2). Although (in common with many releasing agents) the chemistry of the reaction has not been investigated, it has been found that a copper-sodium mixture (7000 $\mu\text{g}/\text{ml}$ Cu; 3000 $\mu\text{g}/\text{ml}$ Na) is very effective in counter-acting the interferences. The mixture is prepared from the sulphate salts of the respective elements. Copper nitrate, at a much higher level (20,000 $\mu\text{g}/\text{ml}$ Cu) has also been used to overcome these interferences (3).

REFERENCES

- (1) VAN LOON J.C., Atomic Abs. Newsletter. **8**, (1) 6, 1969.
- (2) GRIMALDI, F.S., SCHNEPFE M.M., Talanta **17**, 617, 1970.
- (3) HOUZE A.A.G., Journal South African Chem. Inst. **23**, 115, 1970.

NOTES

26. Ir Iridium



CONCENTRATION µg/ml

Potassium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Potassium chloride KCl A. R. Grade

Solution Technique

Dissolve 1.907 g of dried potassium chloride in water and dilute to 1 litre to give 1000 µg/ml K.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	5 mA
Fuel (Note)	propane
Support	air
Flame Stoichiometry	oxidizing

Note: An air-acetylene flame is normally used because interferences are reduced and the signal/noise ratio is improved.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
766.5	0.5	0.5 – 2.0	0.01
769.9	0.5	1.5 – 6.0	0.03
404.4	0.1	200 – 800	3.7

A red-sensitive photomultiplier (Hamamatsu R406) is recommended for determinations at 766.5 nm and 769.9 nm.

Detection Limit

0.003 µg/ml at 766.5 nm using an air-propane flame.

Flame Emission

Wavelength	766.5 nm
Spectral Band Pass	0.1 nm
Fuel	propane
Support	air

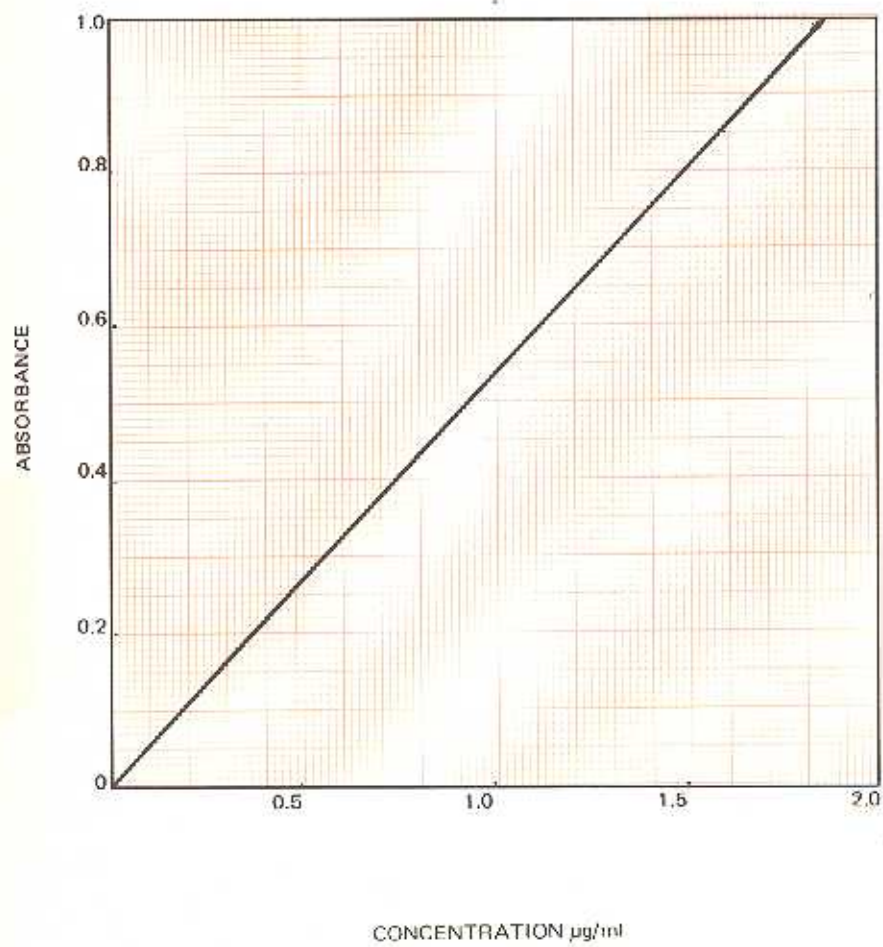
The flame emission determination of potassium is limited by flame stability and by "pick up" of potassium from the air and storage vessels. Air-acetylene can be used if an ionization suppressant is added. At very low levels air-propane is preferred because the suppressants necessary with air-acetylene may be contaminated with potassium.

INTERFERENCES

Potassium is partially ionized in the air-acetylene flame. To suppress ionization, add cesium nitrate or chloride solution to give a final concentration of 1000 µg/ml cesium in all solutions including the blank. The purest available cesium compound must be used to avoid potassium contamination. In the air-propane flame ionization is negligible.

NOTES

27. K Potassium, Kalium



Lanthanum

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Lanthanum oxide La_2O_3 99.99%

Solution Technique

Dissolve 11.730 g of La_2O_3 in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 10000 $\mu\text{g}/\text{ml}$ La.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1.5-2 cm

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
550.1	0.2	2500-10000	48
403.7	0.2	6000-24000	117
357.4	0.2	14000-52000	260

Detection Limit

3 $\mu\text{g}/\text{ml}$ at 550.1 nm using a nitrous oxide-acetylene flame.

The strong emission from the flame creates shot noise in the photomultiplier which limits detection below this moderate concentration. Shot noise is minimized by using the lowest possible photomultiplier voltage and the high gain electronic circuit where available.

Flame Emission

Wavelength	441.7 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.5 $\mu\text{g}/\text{ml}$

The flame emission signal for lanthanum is stronger than the atomic absorption signal at all concentrations. Determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements.

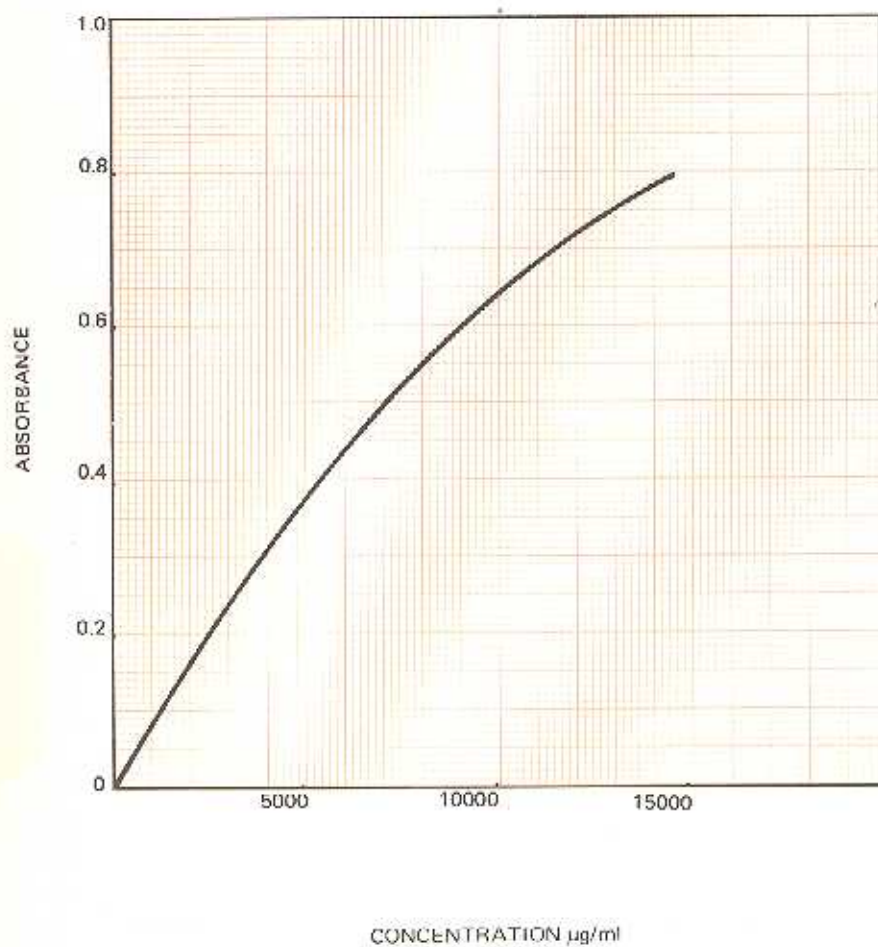
INTERFERENCES

Lanthanum is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 5000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank.

Both absorption and emission signals for lanthanum are depressed in the presence of phosphates, fluorides, silicon, aluminium and iron, and by other rare earths. Many solvent extraction procedures can be found in the literature; one simple method is extraction at pH 7 with 0.1 M cinnamic acid in hexane. Otherwise rigorous matching of sample and standard solutions is necessary to control interferences. No chemical releasing agent has been discovered.

NOTES

28. La Lanthanum



Lithium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Lithium carbonate Li_2CO_3 99.9%

Solution Technique

Dissolve 5.324 g of Li_2CO_3 in a minimum volume of 1:5 nitric acid and dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Li.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 5 mA
Fuel propane
Support air
Flame Stoichiometry oxidizing

An air-acetylene flame can be used although lithium is appreciably ionized in this hotter flame.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
670.8	0.5	1-4	0.017
323.3	0.2	500-2000	8.6
610.4	0.5	8000-32000	162

At 670.8 nm it is advisable to use a sharp cutoff filter to avoid second-order interference from the neon 335.5 nm line emitted by the lamp.

Detection Limit

0.0015 $\mu\text{g}/\text{ml}$ at 670.8 nm using an air-propane flame.

Flame Emission

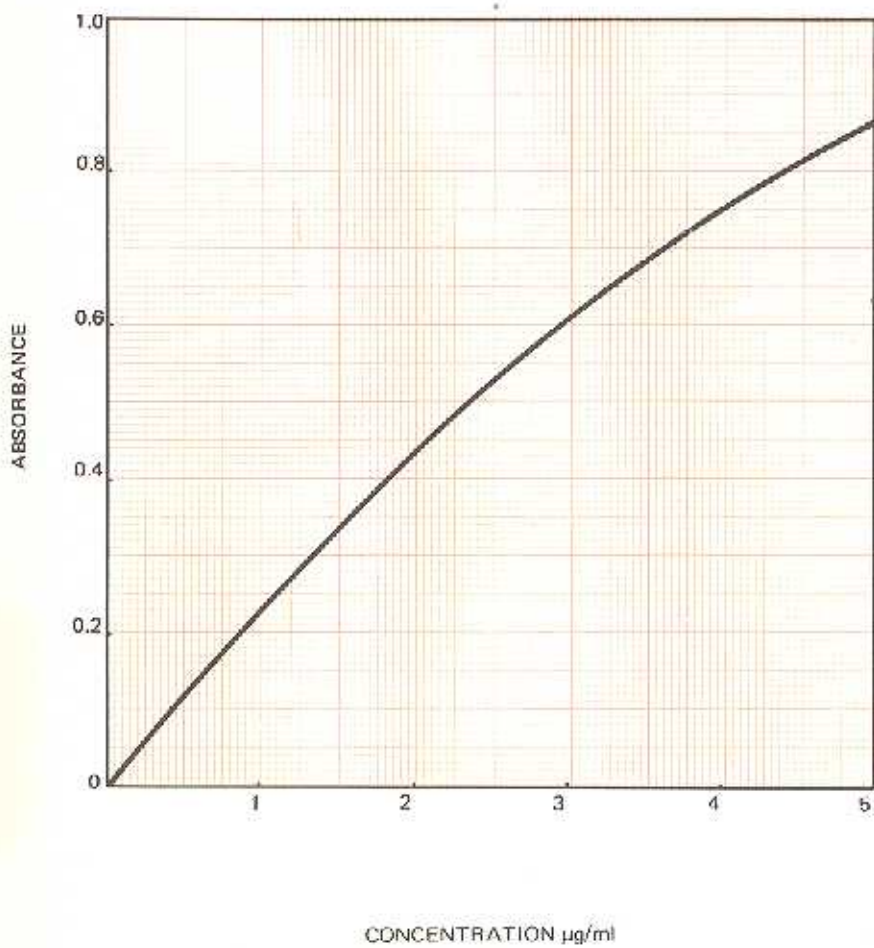
Wavelength 670.8 nm
Spectral Band Pass 0.1 nm
Fuel propane
Support air
Detection limit data not available.

INTERFERENCES

In an air-propane flame, lithium ionization is negligible. In the hotter air-acetylene flame ionization is appreciable and it is necessary to match all solutions with respect to easily ionized elements such as Na, K, Rb, Cs, Ca, Sr, Ba. Ionization is suppressed if all solutions are made to contain 2000 $\mu\text{g}/\text{ml}$ potassium. No chemical interferences are known.

NOTES

29. Li Lithium



Lutetium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Lutetium oxide Lu_2O_3 99.9%

Solution Technique

Dissolve 1.137 g of Lu_2O_3 in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Lu.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
336.0	0.2	400-1600	7.9
356.8	0.2	600-2400	13
331.2	0.2	800-3200	18
337.7	0.2	900-3600	21

Detection Limit

1.0 $\mu\text{g}/\text{ml}$ at 336.0 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	466.2 nm or 451.9 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.15 $\mu\text{g}/\text{ml}$

Note that the emission at 466.2 nm arises from the LuO band emission, while at 451.9 nm the emission is a line due to Lu atoms. The detection limits are comparable at either wavelength and spectral interferences are severe.

The flame emission signal for lutetium is stronger than the atomic absorption signal, especially at low concentrations. Below 400 $\mu\text{g}/\text{ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

INTERFERENCES

Lutetium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank.

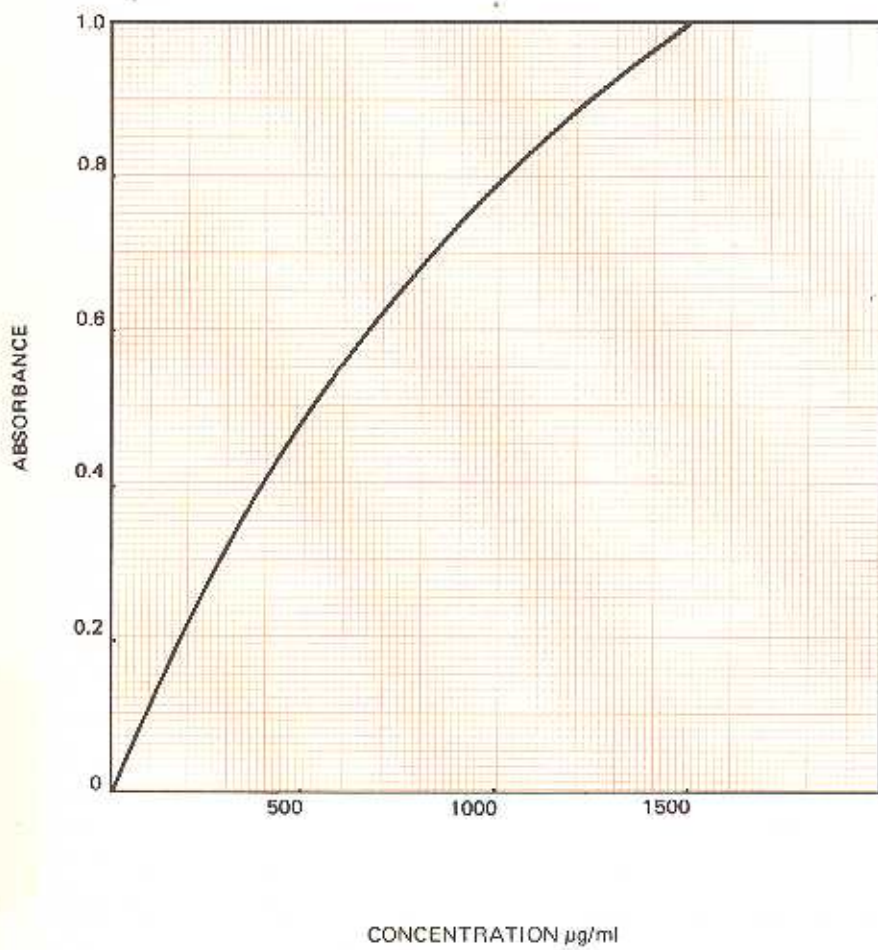
Absorbance and emission signals are depressed in the presence of phosphates, fluorides, silicon, aluminium and iron, and the other rare earth elements. Non-specific solvent extraction methods are available to circumvent some of these interferences. No chemical releasing agents are known.

REFERENCES

RAINS T.C., HOUSE H.P., MENIS O., Anal. Chim. Acta. **22**, 315, 1960

NOTES

30. Lu Lutetium



Magnesium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Magnesium metal strip or wire 99.99%

Solution Technique

Dissolve 1.000g of magnesium in 1.4 nitric acid. Dilute to 1 litre to give 1000 µg/ml Mg.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	3 mA
Fuel	acetylene
Support	air
Flame Stoichiometry	oxidizing

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	µg/ml	µg/ml
285.2	0.5	0.1–0.4	0.003
202.5	1.0	5–20	0.09

Note: Sensitivity is strongly dependent on lamp current.

Detection Limit

0.0003 µg/ml at 285.2 nm using an air-acetylene flame.

Flame Emission

Wavelength	285.2 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection limit data not available.	

INTERFERENCES

The extent to which chemical interferences affect absorbance is dependent on the type of flame used. Interferences in the air-propane flame are many and interdependent (1) (2).

This flame has been largely superseded by the air-acetylene flame in which few interferences are significant in the optimum working range (3).

The most common interferences in air-acetylene can be overcome by the addition of a known excess of a releasing agent such as strontium (1000-5000 µg/ml) or lanthanum (10,000 µg/ml). Solutions containing 200 µg/ml interferent in 0.4 µg/ml Mg show the following interference pattern:

Mg Absorbance	Mg Absorbance
Al – 24%	SiO ₃ – 42%
Li + 10%	CO ₃ – 17%
Ti – 16%	SeO ₃ – 14%
Zr – 9%	

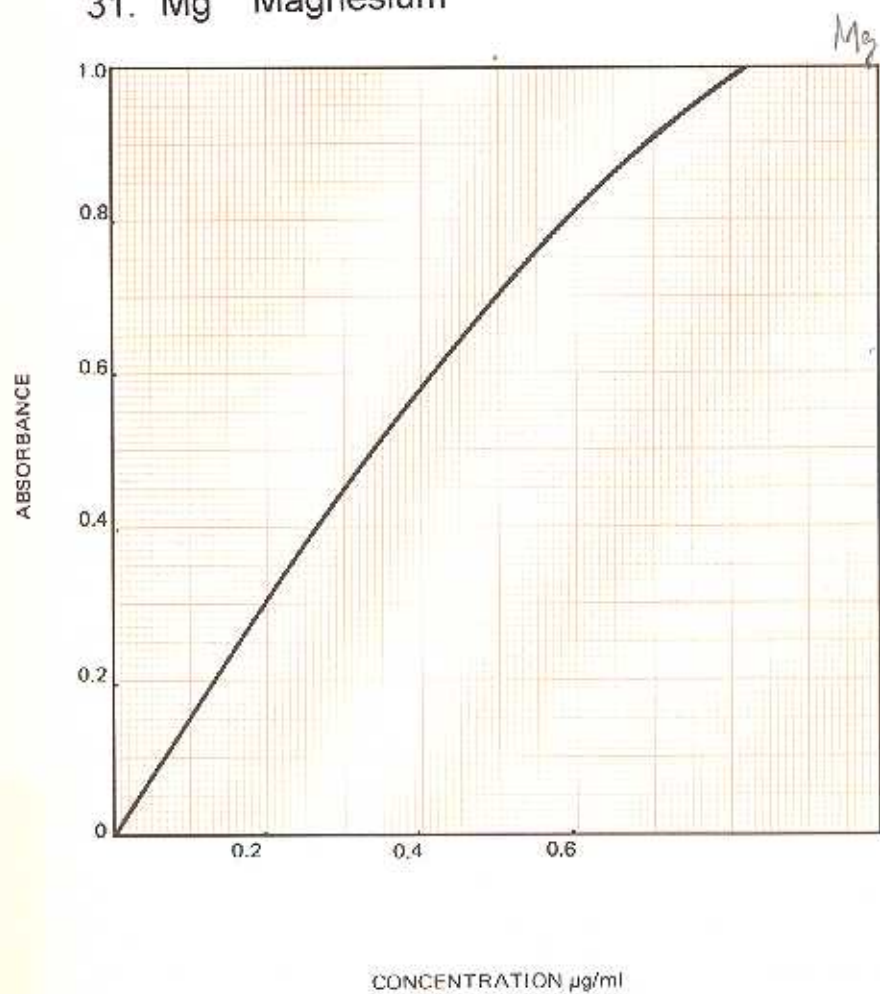
The nitrous oxide-acetylene flame shows no interference except a general absorbance enhancement of 15% by the alkali metals due to suppression of ionization.

REFERENCES

- (1) HALLS D.J., TOWNSHEND A., Anal. Chim. Acta **38** 278, 1966
- (2) FIRMAN R.J., Spectrochim. Acta **21**, 341, 1965
- (3) RAMAKRISHNA T.V., WEST P.W., ROBINSON J.W., Anal. Chim. Acta **40**, 347, 1968.

NOTES

31. Mg Magnesium



Manganese

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Manganese metal strip or wire 99.99%

Solution Technique

Dissolve 1.000 g manganese in a minimum volume of 1:1 nitric acid and dilute to 1 litre to give 1000 µg/ml Mn.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 5 mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

A nitrous oxide-acetylene flame can also be used but sensitivity is poorer.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
279.5	0.2	1.0–4.0	0.024
403.1	0.2	15–60	0.30
321.7	0.1	3500–14000	65

Detection Limit

0.003 µg/ml at 279.5 nm using an air-acetylene flame.

Flame Emission

Wavelength 403.1 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide

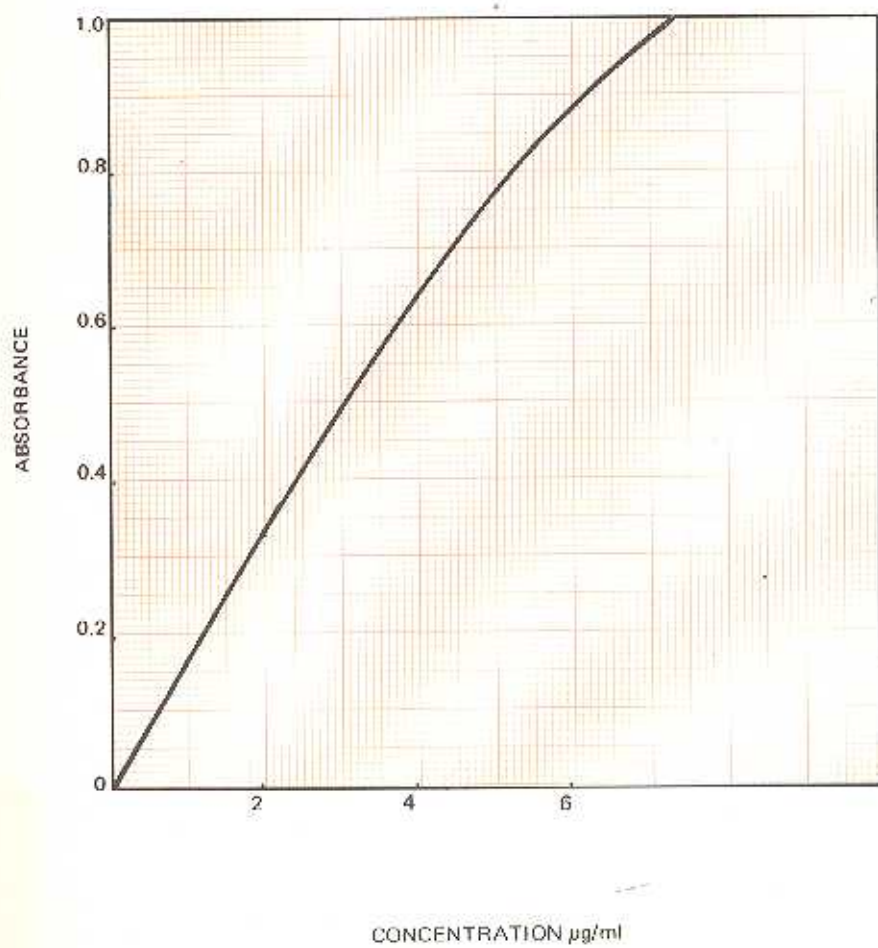
Detection limit data not available. An air-acetylene flame can be used with poorer sensitivity.

INTERFERENCES

In a reducing air-acetylene flame the absorbance is depressed in the presence of phosphate, perchlorate, iron, nickel and cobalt. In an oxidizing air-acetylene flame or a nitrous oxide-acetylene flame these interferences do not arise. No releasing agent is usually necessary.

NOTES

32. Mn Manganese



Molybdenum

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Molybdenum metal strip or wire 99.99%

Solution Technique

Dissolve 1.000 g molybdenum in hot concentrated nitric acid, cool and dilute to 1 litre to give 1000 µg/ml Mo.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	5 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	strongly reducing; red cone 2-3 cm.

A brightly luminous air-acetylene flame can be used with poorer sensitivity and increased interference effects.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
313.3	0.2	15-60	0.33
320.9	0.1	250-1000	4.3

Many other wavelengths can be used — see reference (1).

Detection Limit

0.04 µg/ml at 313.3 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	390.3 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.8 µg/ml

INTERFERENCES

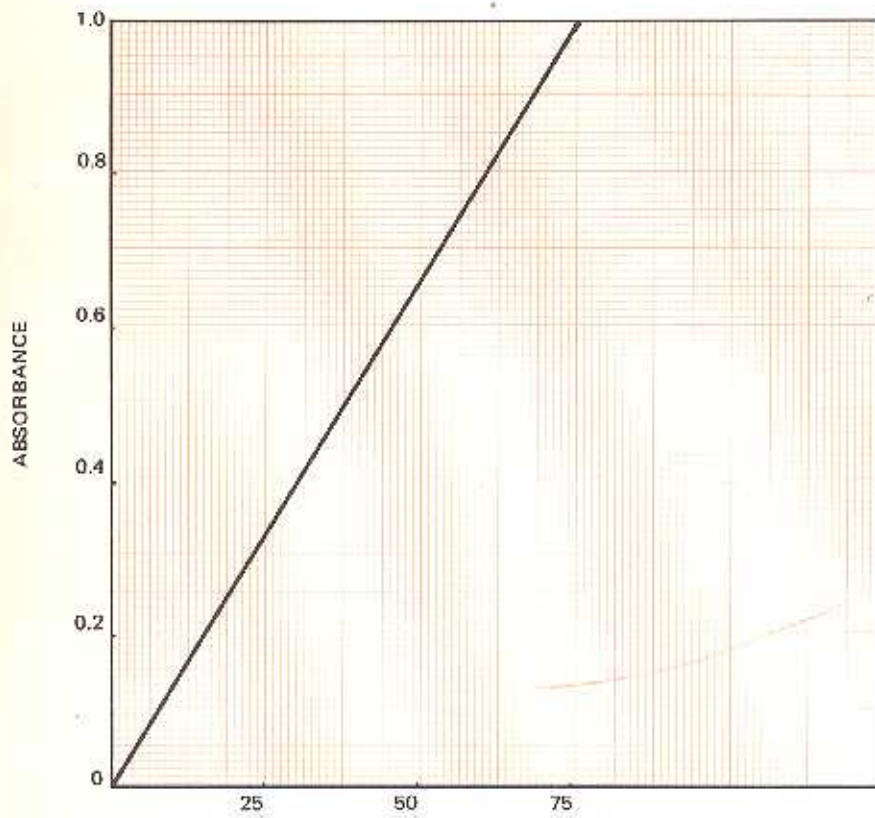
Conflicting statements have been made on interferences in molybdenum atomic absorption. Various workers have found either no interferences or severe interferences from all metals, apparently depending on the solution conditions. In the recommended nitrous oxide-acetylene flame interferences can be suppressed by adding an excess of a refractory element (1000 µg/ml Al).

REFERENCES

- (1) DAVID D.J., *Analyst* **86**, 730, 1961.
- (2) RAMAKRISHNA T.V., WEST P.W., ROBINSON J.W., *Anal. Chim. Acta.* **44**, 437, 1969.
- (3) KIRKBRIGHT G.D., SMITH A.M., WEST T.S., *Analyst* **91**, 700, 1966.

NOTES

33. Mo Molybdenum



CONCENTRATION $\mu\text{g/ml}$

Sodium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Sodium chloride NaCl	99.99%
Sodium carbonate Na ₂ CO ₃	99.99%

Solution Technique

Dissolve 2.542 g of dried NaCl in water and dilute to 1 litre to give 1000 µg/ml Na.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	5 mA
Fuel	propane
Support	air
Flame Stoichiometry	oxidizing: blue cone 1½ cm.

An air-acetylene flame can also be used to provide an improved signal/noise ratio.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
589.0	0.2	0.15 – 0.60	0.003
589.6	0.2	0.5 – 2.0	0.008
330.2 } 330.3 }	0.2	100 – 400	1.6

Detection Limit

0.0003 µg/ml at 589.0 nm using an air-propane flame.

Flame Emission

Wavelength	589.0 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	air
Detection limit data not available.	

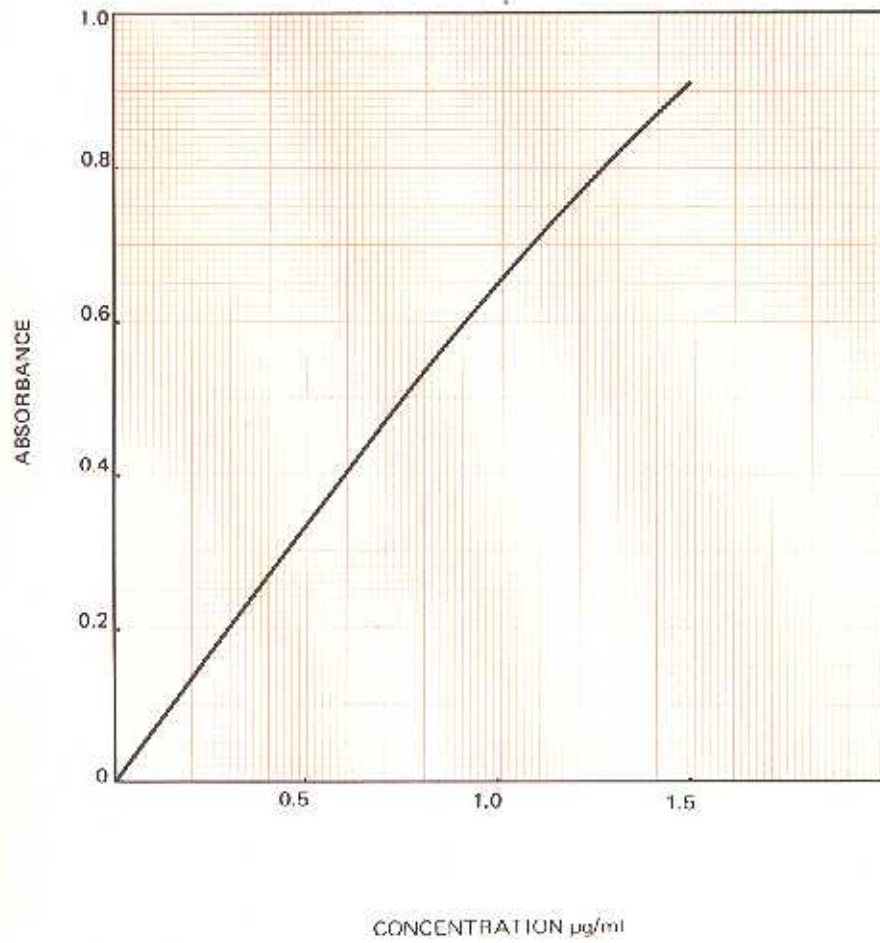
INTERFERENCES

Sodium is partially ionized in the air-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/ml potassium in all solutions including the blank. In the air-propane flame ionization is negligible. No chemical interferences have been reported.

NOTES

34. Na Sodium, Natrium

Na



Niobium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Niobium metal (strip) 99.99%

Solution Technique

Dissolve 5.000 g of niobium metal strip in 5 ml hydrofluoric acid, 5 ml water by the slow DROPWISE addition of 10 ml of concentrated nitric acid. Dilute to 1 litre to give 5000 µg/ml Nb.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
334.9	0.2	1000-4000	21
358.0	0.2	1000-4000	23
408.0	0.2	1100-4500	25
405.9	0.2	1100-4500	25

Detection Limit

2 µg/ml at 334.9 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	405.9 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	1.0 µg/ml

INTERFERENCES

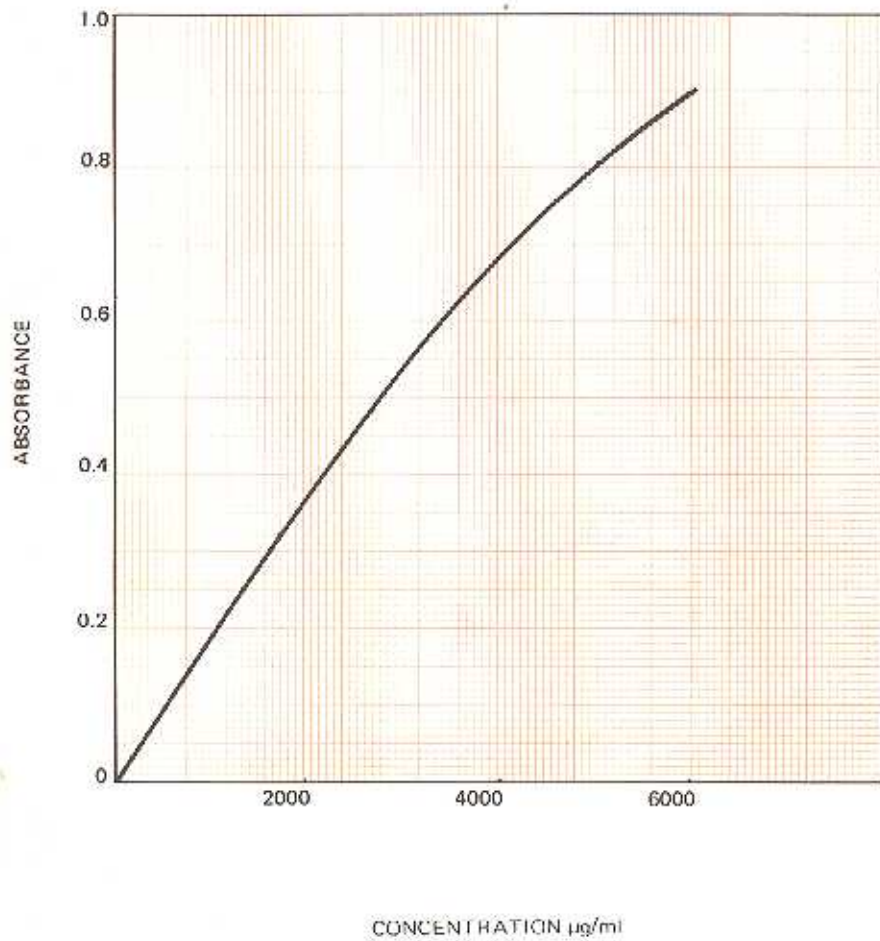
No systematic studies have been published specifically on niobium interferences. Hydrofluoric acid increases the absorbance signal up to 2% HF, but greater concentrations depress the signal. Sodium in the presence of 2% HF depresses the signal. By analogy with zirconium it is expected that 0.1M NH₄F would eliminate some of these interferences and enhance the signal, although data for Nb have not been published.

REFERENCES

THOMAS P.E., PICKERING W.F., *Talanta* **18**, 127, 1971.
BOND A.M., *Anal. Chem.* **42**, 932, 1970.
SASTRI V.S., CHAKRABARTI C.L., WILLIS D.E., *Talanta* **16**, 1093, 1969.

NOTES

35. Nb Niobium



Neodymium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Neodymium oxide Nd_2O_3 99.99%

Solution Technique

Dissolve 1.167 g of Nd_2O_3 in 1:1 nitric acid and dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Nd.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	$\mu\text{g}/\text{ml}$	$\mu\text{g}/\text{ml}$
492.5	0.2	350-1400	8
486.7	0.1	2500-10000	54

Detection Limit

1.6 $\mu\text{g}/\text{ml}$ at 492.5 nm using a nitrous oxide-acetylene flame. To reduce shot noise in the photomultiplier the lamp current should be increased, the photomultiplier gain reduced and "high gain" electronics used where available.

Flame Emission

Wavelength	660.8 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.08 $\mu\text{g}/\text{ml}$

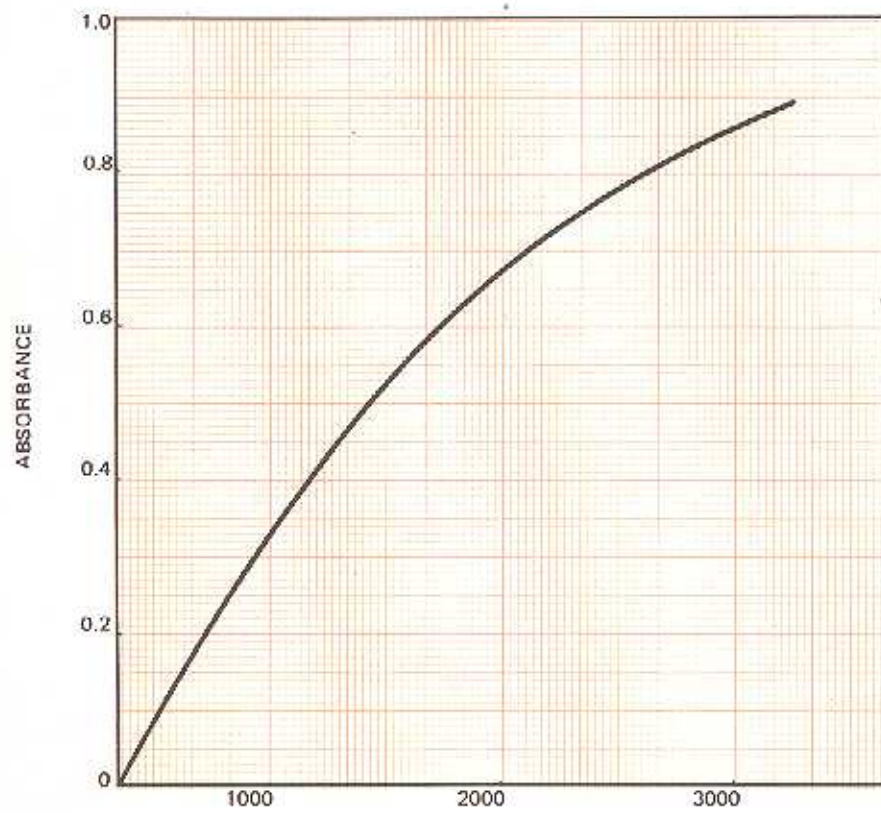
The flame emission signal for neodymium is stronger than the atomic absorption signal, especially at low concentrations. Below 350 $\mu\text{g}/\text{ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

INTERFERENCES

Neodymium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank. The signal is depressed in the presence of 500 $\mu\text{g}/\text{ml}$ silicon, aluminium, iron, titanium or fluoride. A more oxidizing flame reduces these effects.

NOTES

36. Nd Neodymium



CONCENTRATION µg/ml

Nickel

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Nickel metal strip or wire 99.99%

Solution Technique

Dissolve 1.000 g of nickel in 1:1 nitric acid and dilute to 1 litre to give 1000 µg/ml Ni.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 5 mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

A nitrous oxide-acetylene flame can also be used, with poorer sensitivity.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
232.0	0.2	3 – 12	0.066
341.5	0.2	15 – 60	0.34
352.4	0.2	15 – 60	0.34
351.5	0.2	35 – 140	0.72
362.5	0.2	2000 – 8000	36

Note The 352.4 nm line is preferred because the calibration is more nearly linear over the working range and the signal is less susceptible to non-atomic absorption than at the more sensitive 232.0 nm line.

Detection Limit

0.008 µg/ml at 232.0 nm using an air-acetylene flame.

Flame Emission

Wavelength 341.5 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide

Detection limit data not available.

An air-acetylene flame can be used with poorer sensitivity.

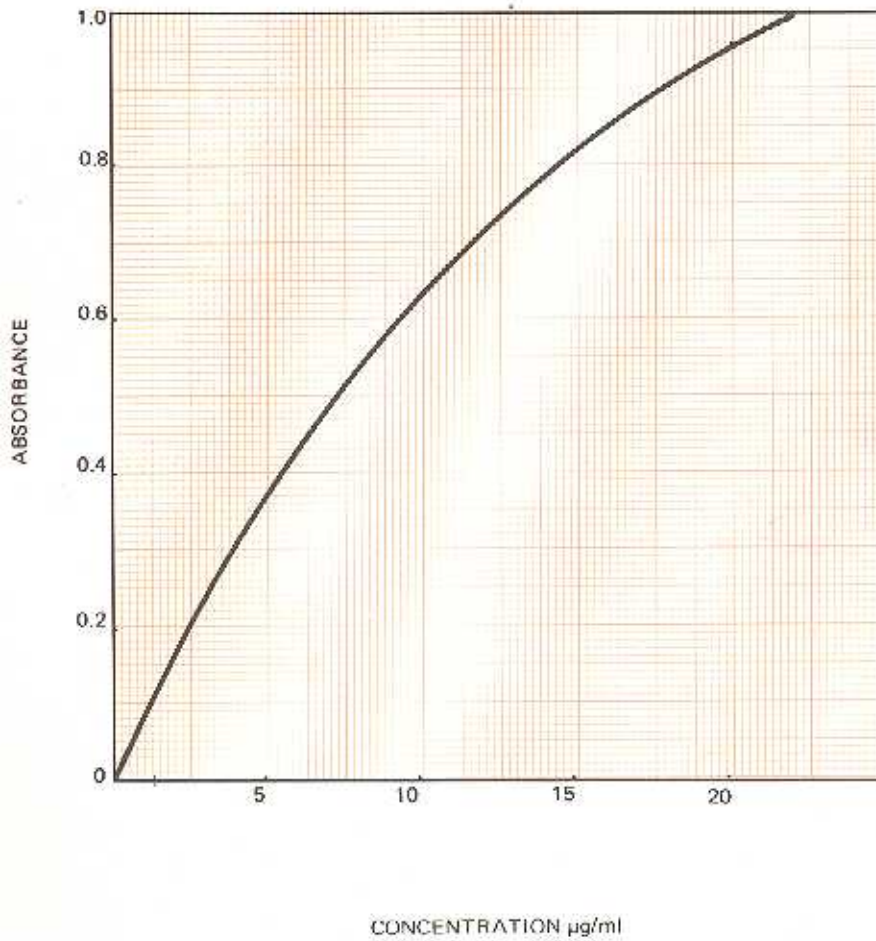
INTERFERENCES

At 232.0 nm wavelength, non-atomic species in the flame absorb strongly. Where the sample has a high concentration of dissolved solids it is necessary to correct for non-atomic absorption by using the 231.7 nm nickel non-absorbing line. At 352.4 nm, this effect is negligible even for high matrix solutions.

In hydrochloric and perchloric acid solution, a slight (5%) absorbance depression has been observed in the presence of iron, cobalt and chromium. In a more oxidizing flame the effects are minimized and in the nitrous oxide-acetylene flame no interferences are observed.

NOTES

37. Ni Nickel



Osmium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Osmium tetroxide OsO_4 , resublimed

Solution Technique

Dissolve 1.340 g OsO_4 in 100 ml water with the addition of a sodium hydroxide pellet to assist dissolution. Dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Os.

CAUTION: Osmium solutions and vapour are toxic.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm

An air-acetylene flame can also be used but with poorer sensitivity.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
290.9	0.2	50-200	1.2
426.1	0.2	800-3200	19

Detection Limit

0.12 $\mu\text{g}/\text{ml}$ at 290.9 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	426.1 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.3 $\mu\text{g}/\text{ml}$

INTERFERENCES

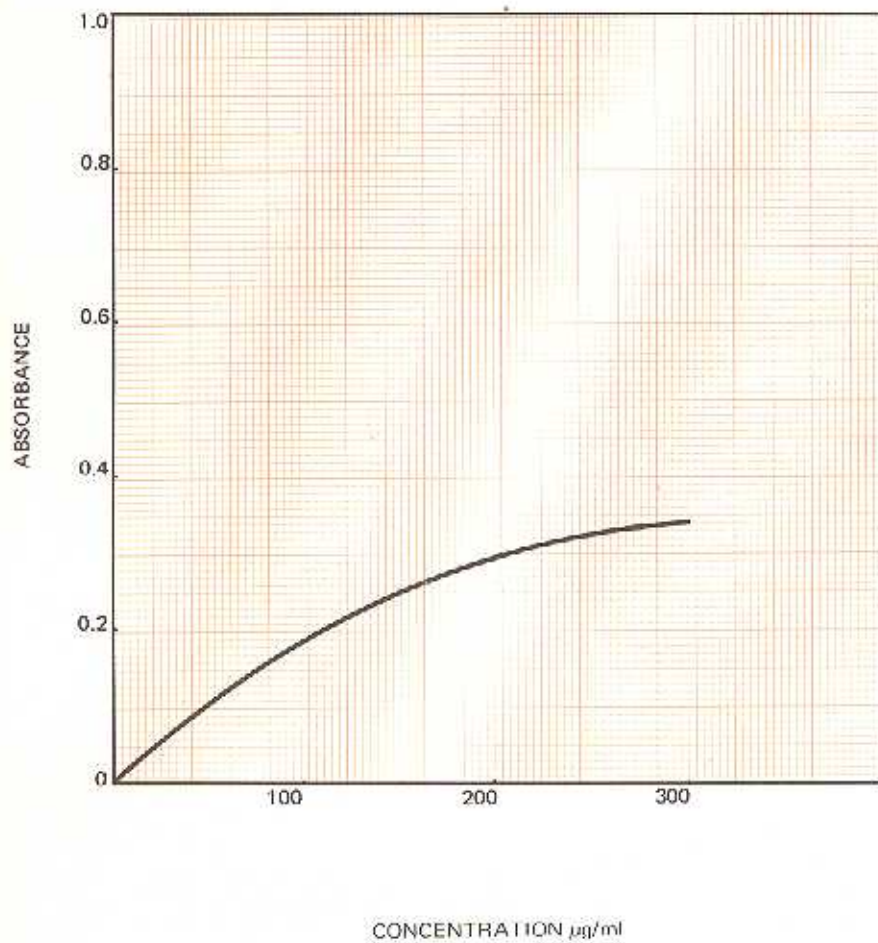
It has been reported that in an oxidizing flame or in the presence of peroxides in solution the signal is suppressed because OsO_4 formation removes free osmium atoms. No studies using reducing agents have been reported.

REFERENCE

OSOLINSKI T.W., KNIGHT N.H., Chem.Eng.News Oct. 16, 1967.

NOTES

38. Os Osmium



Lead

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Lead metal strip or wire 99.9%

Solution Technique

Dissolve 1.000 g of lead in 1:1 nitric acid. Dilute to 1 litre to give 1000 µg/ml Pb.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 6 mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
217.0	1.0	5-20	0.11
283.3	0.2	10-40	0.23
261.4	0.2	200-800	4.0
202.2	0.2	250-1000	5.6
205.3	0.2	2000-8000	38

Detection Limit

0.02 µg/ml at 217.0 nm using an air-acetylene flame.

Flame Emission

Wavelength 405.8 nm
Spectral Band Pass 0.1 nm
Fuel Acetylene
Support Nitrous Oxide
Detection Limit data not available

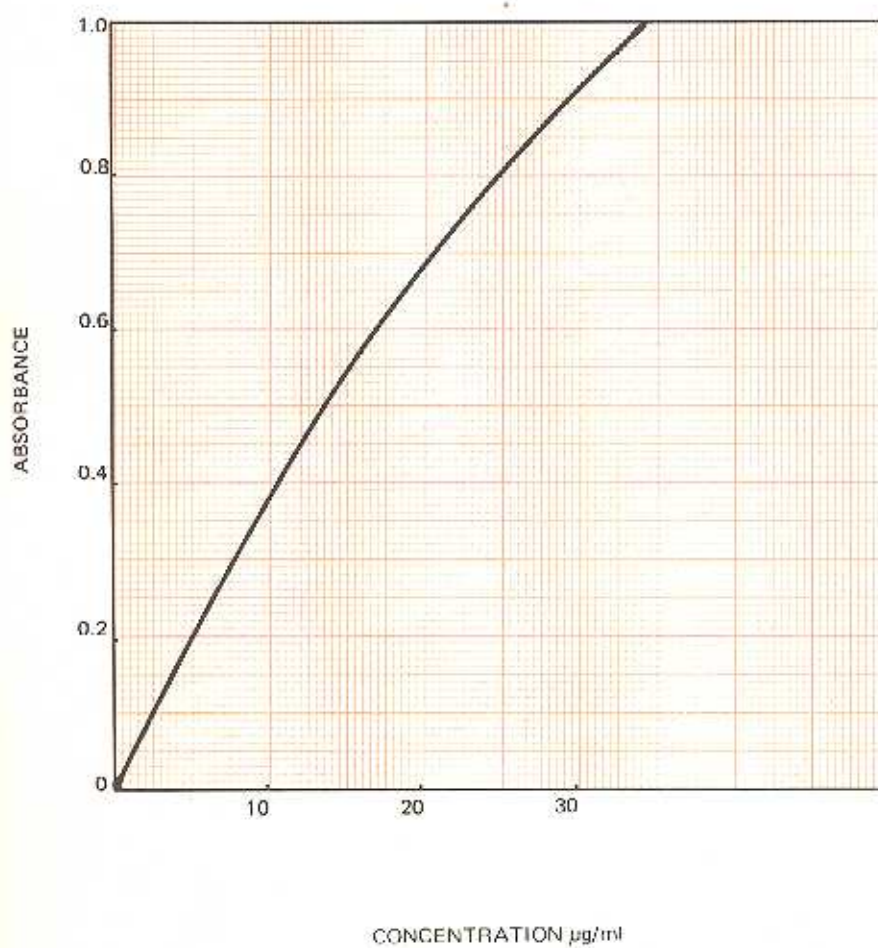
INTERFERENCES

No cationic interferences have been reported for the air-acetylene flame, however a number of anionic interferences have been reported. Phosphate, carbonate, iodide, fluoride and acetate suppress lead absorbance significantly at concentrations ten times greater than lead. These interferences can be largely overcome by addition of EDTA solution so that the sample solutions are 0.1 Molar with respect to EDTA.

At the 217.0 nm wavelength, non-atomic species in the flame absorb strongly. Where the sample has a high concentration of dissolved solids it is necessary to correct for non-atomic absorption using a hydrogen continuum lamp.

NOTES

39. Pb Lead



Palladium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Ammonium chloropalladite, $(\text{NH}_4)_2\text{PdCl}_4$ 99.9%

Solution Technique

Dissolve 2.672 g of ammonium chloropalladite in water and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Pd.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	5 mA
Fuel	acetylene
Support	air
Flame Stoichiometry	oxidizing

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	$\mu\text{g/ml}$	$\mu\text{g/ml}$
244.8	0.2	4–16	0.092
247.6	0.2	7–28	0.13
340.5	1.0	35–140	0.63

Detection Limit

0.02 $\mu\text{g/ml}$ at 244.8 nm using an air-acetylene flame.

Flame Emission

Wavelength	363.5 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.04 $\mu\text{g/ml}$ (see reference)

INTERFERENCES

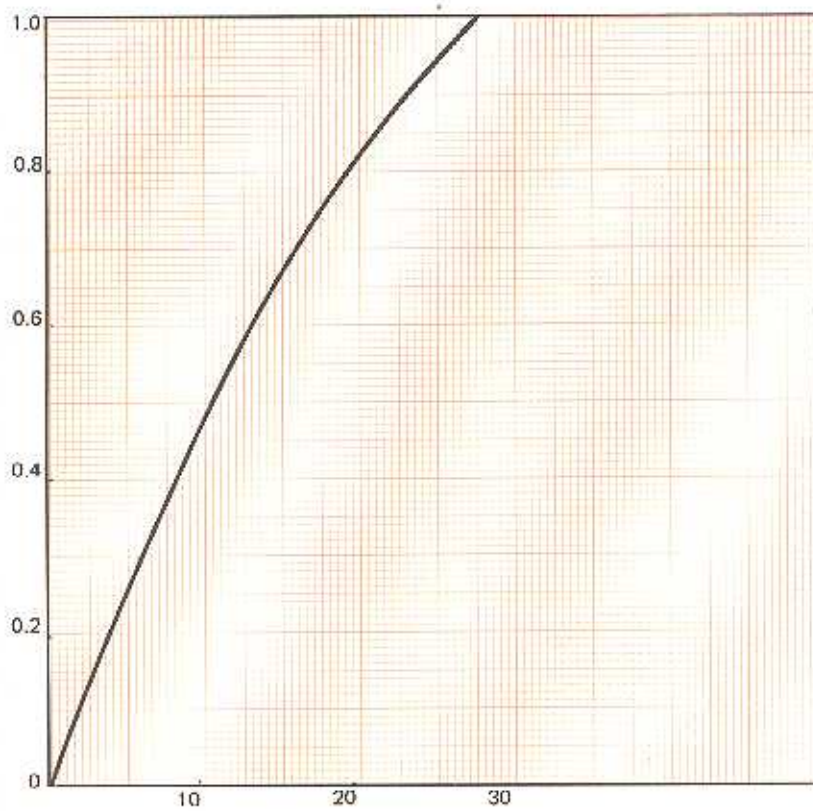
In the air-acetylene flame the atomic absorption signal is depressed in the presence of aluminium, cobalt or nickel at all concentrations and by hydrofluoric acid. The depression is eliminated in the presence of lanthanum (5000 $\mu\text{g/ml}$ as chloride) or EDTA (0.01M). The interference can be reduced by using a more oxidizing flame and by taking measurements higher in the flame. A nitrous oxide-acetylene flame can be used to overcome the interferences but the sensitivity is much poorer.

REFERENCE

SYCHRA V., SLEVIN P.J., MATOUSEK J., BEK F., *Anal. Chim. Acta* **52**, 259, 1970.

NOTES

40. Pd Palladium



CONCENTRATION µg/ml

Praseodymium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Praseodymium oxide Pr_2O_3 99.9%

Solution Technique

Dissolve 1.170 g of Pr_2O_3 in 5 ml hydrochloric acid and dilute to 100 ml to give 10000 $\mu\text{g}/\text{ml}$ Pr.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current (Note)	8 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm

Note: Absorbance is much lower at increased lamp current.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
495.1	0.2	800-3200	18
513.3	0.2	2000-8000	39

Note: Praseodymium in the flame emits strongly at both wavelengths. To reduce noise in the photomultiplier, the "high gain" setting should be used.

Detection Limit

10 $\mu\text{g}/\text{ml}$ at 495.1 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	284.0 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	2.0 $\mu\text{g}/\text{ml}$

The flame emission signal for praseodymium is stronger than the atomic absorption signal, especially at low concentrations. Below 800 $\mu\text{g}/\text{ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

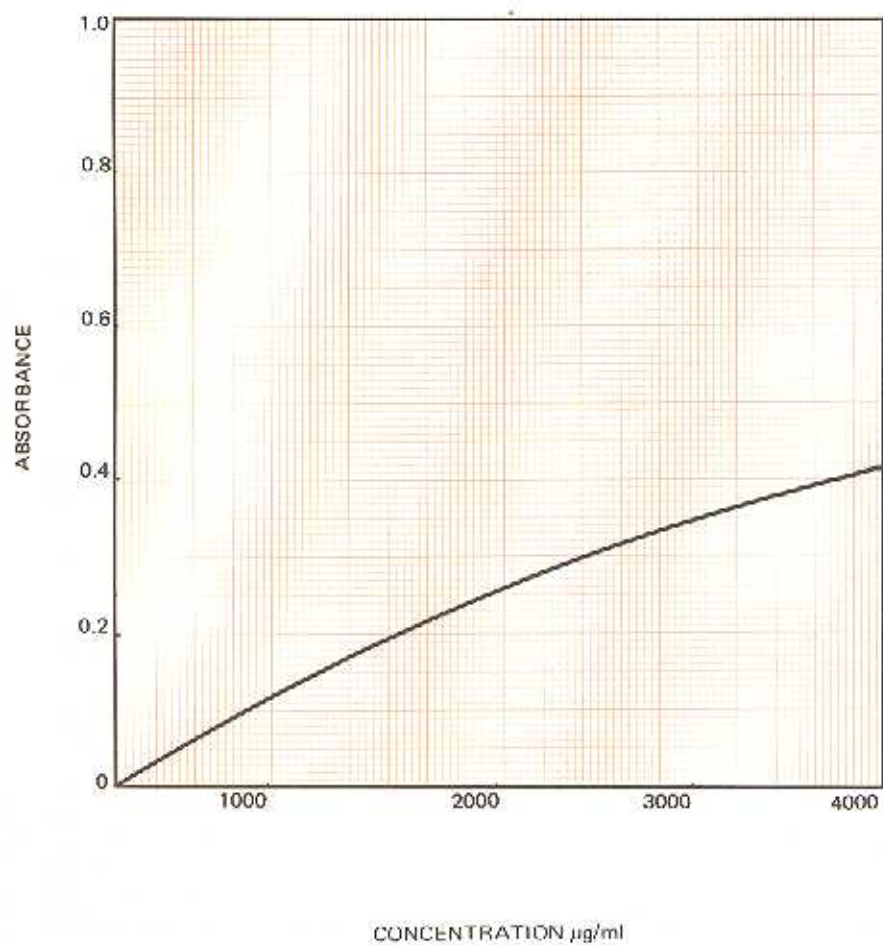
INTERFERENCES

Praseodymium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank.

The atomic absorption signal is depressed in the presence of silicon at high concentrations (>2000 $\mu\text{g}/\text{ml}$).

NOTES

41. Pr Praseodymium



Platinum

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Ammonium chloroplatinate $(\text{NH}_4)_2 \text{PtCl}_6$ 99.9%

Solution Technique

Dissolve 2.275 g of $(\text{NH}_4)_2 \text{PtCl}_6$ in water and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Pt.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA
Fuel	acetylene
Support	air
Flame Stoichiometry	oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g/ml}$	Typical Sensitivity $\mu\text{g/ml}$
266.0	0.2	50–200	1.2
299.8	0.2	300–1200	6.6

Detection Limit

0.12 $\mu\text{g/ml}$ at 266.0 nm using an air-acetylene flame.

Flame Emission

Wavelength	266.0 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection limit data are not available.	

INTERFERENCES

The atomic absorption signal is depressed in the presence of most other noble metals, and also in the presence of acids. The interferences can be reduced (but not eliminated) by using a more oxidizing flame. Interferences are eliminated if all solutions are made 2% in copper or 1% in lanthanum.

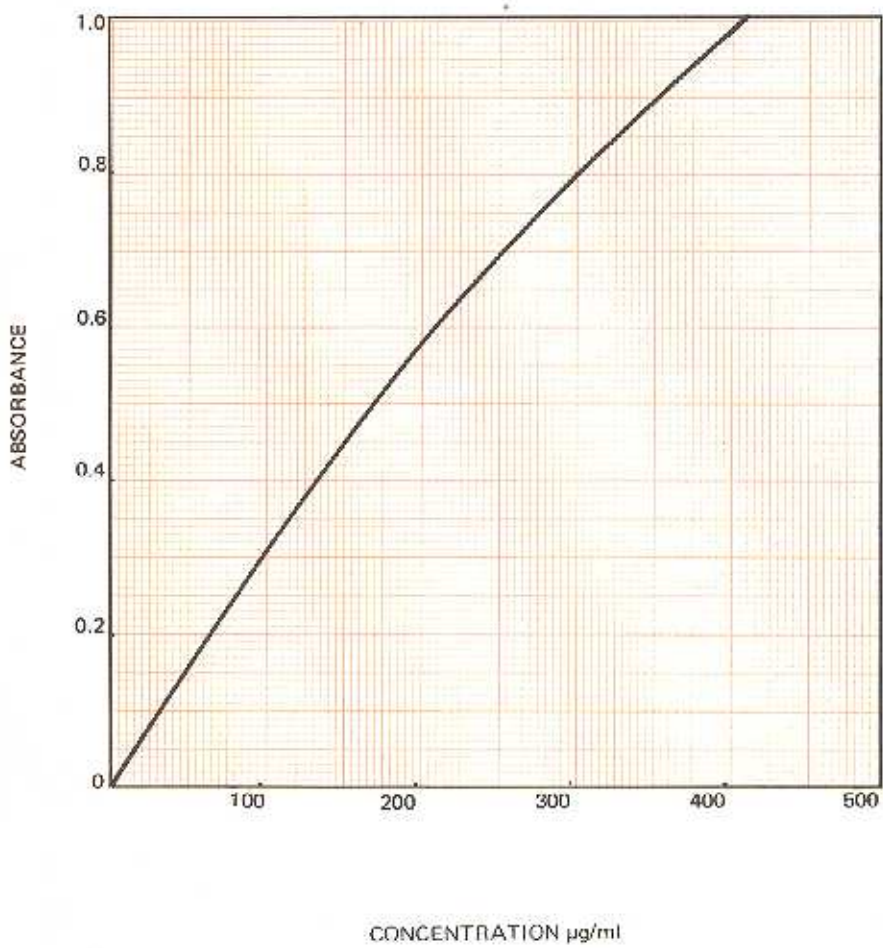
A nitrous oxide-acetylene flame can be used to overcome the interferences but the sensitivity is poorer.

REFERENCES

- SCHNEPPE M.M., GRIMALDI F.S., *Talanta* **16**, 591, 1969.
 STRASHEIM A., WESSELS G.J., *Appl. Spectrosc.* **17**, 65, 1963.
 PITTS A.G., VAN LOON J.C., BEAMISH F.E., *Anal. Chim. Acta* **50**, 181, 1970; **50**, 195, 1970.

NOTES

42. Pt Platinum



Rubidium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Rubidium chloride, RbCl 99.9%

Solution Technique

Dissolve 1.415 g of dried RbCl in water and dilute to 1 litre to give 1000 µg/ml Rb.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 15mA
Fuel (note) propane
Support air
Flame Stoichiometry oxidizing

Note: An air-acetylene flame can also be used. Chemical interference is reduced but sensitivity is poorer and ionization is increased.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
780.0	0.2	2-10	0.042
794.8	0.2	5-20	0.11
420.2	0.2	200-800	4.1
421.6	0.2	550-2200	12.5

A red-sensitive photomultiplier (Hamamatsu R406) is preferred for determinations at 780.0 and 794.8 nm.

Detection Limit

0.003 µg/ml at 780.0 nm using an air-propane flame.

Flame Emission

Wavelength 780.0 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support air

Detection limit data not available
A red sensitive photomultiplier should be used.

INTERFERENCES

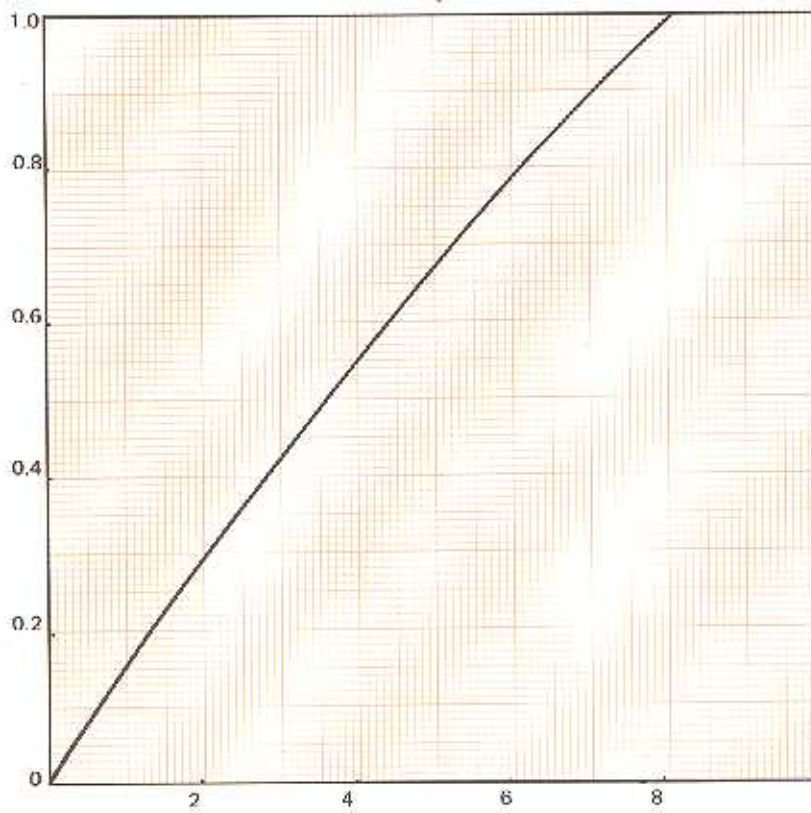
Rubidium is 5 - 10% ionized in the air-propane flame and 40 - 50% ionized in the air-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 µg/ml potassium in all solutions including the blank.

In both flames hydrochloric and other acids (0.1 M) depress the atomic absorption signal in the lower regions of the flame but not at higher levels. Aluminium depresses the signal at all heights in an air-propane flame, but not in air-acetylene. Any ionizable metal will increase the signal unless excess potassium is added to all solutions.

All interference effects except ionization are reduced in the air-acetylene flame.

NOTES

43. Rb Rubidium



CONCENTRATION µg/ml

Rhenium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Potassium perrhenate $KReO_4$ 99.9%

Solution Technique

Dissolve 1.554 g of $KReO_4$ in water and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Re.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g/ml}$	Typical Sensitivity $\mu\text{g/ml}$
346.1	0.1	400-1600	10
346.5	0.2	1000-4000	17
345.2	0.2	1300-5200	26

Note: The use of maximum lamp current and minimum spectral band pass is recommended for best results.

Detection Limit

0.9 $\mu\text{g/ml}$ at 346.1 nm using a nitrous oxide-acetylene flame.

Flame Emission

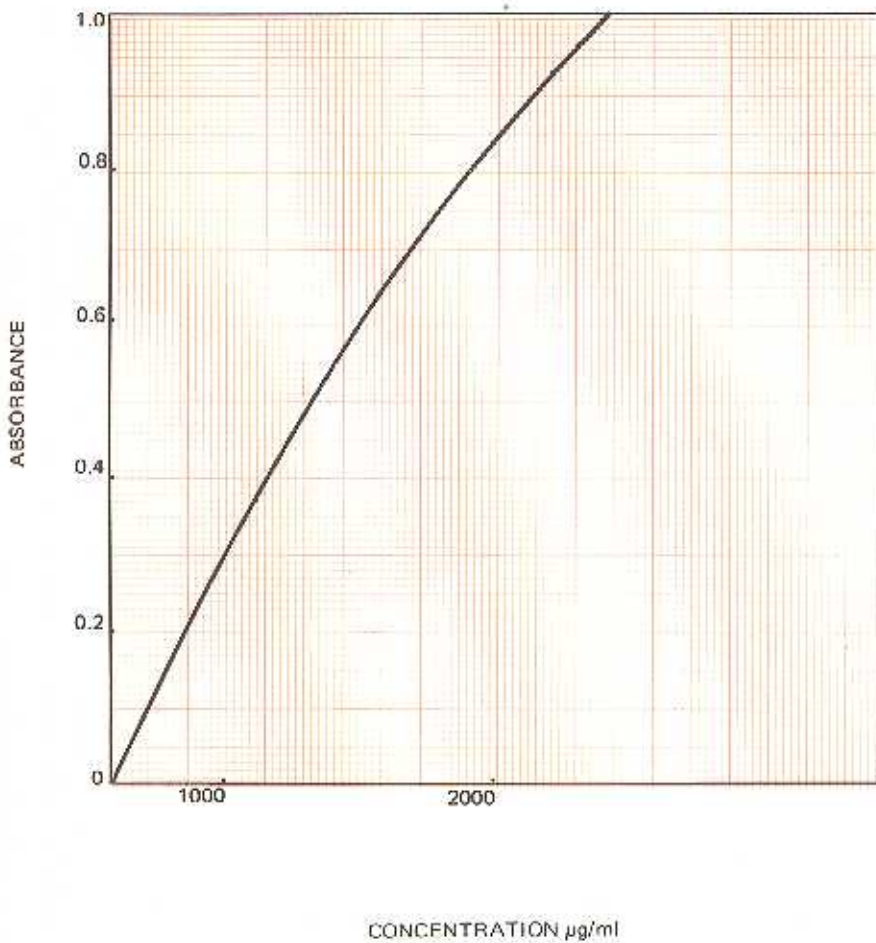
Wavelength	346.1 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection limit data not available.	

INTERFERENCES

The atomic absorption signal for rhenium is enhanced in the presence of sulphuric acid (0.1%-5%) and depressed in the presence of calcium, barium or magnesium (>100 $\mu\text{g/ml}$). Most transition group metals depress the signal slightly at any concentration. All of these interferences can be minimized by using a slightly more oxidizing flame. No comprehensive studies of interferences have been published.

NOTES

44. Re Rhenium



Rhodium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Ammonium chlororhodite, $(\text{NH}_4)_3\text{RhCl}_6 \cdot 1\frac{1}{2}\text{H}_2\text{O}$
Rhodium metal 99.9%

Solution Technique

Dissolve 3.860 g of $(\text{NH}_4)_3\text{RhCl}_6 \cdot 1\frac{1}{2}\text{H}_2\text{O}$ in a minimum volume of 10% hydrochloric acid and dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Rh.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	5mA
Fuel	acetylene
Support	air
Flame Stoichiometry	oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
343.5	0.5	5–25	0.15
328.1	0.2	400–1600	8.7

Many other wavelengths can be used (see reference 4)

Detection Limit

0.006 $\mu\text{g}/\text{ml}$ at 343.5 nm using an air-acetylene flame.

Flame Emission

Wavelength	369.2 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection limit data not available.	

INTERFERENCES

In an air-acetylene flame, most other elements interfere and the interference depends unpredictably on their concentration. Phosphoric and sulphuric acids depress the signal (constant above 10% concentration). Alkali metal sulphates, on the other hand, enhance the signal strongly although other sulphates give mixed results.

Releasing agents mentioned in the literature include:

- 3% NaHSO_4 in 10% HCl (1)
- 1% La as $\text{La}_2(\text{SO}_4)_3$ in 2% HCl (2)
- 1 mg/ml U as uranyl nitrate (3)

although these reagents are not completely effective in the presence of the other noble metals.

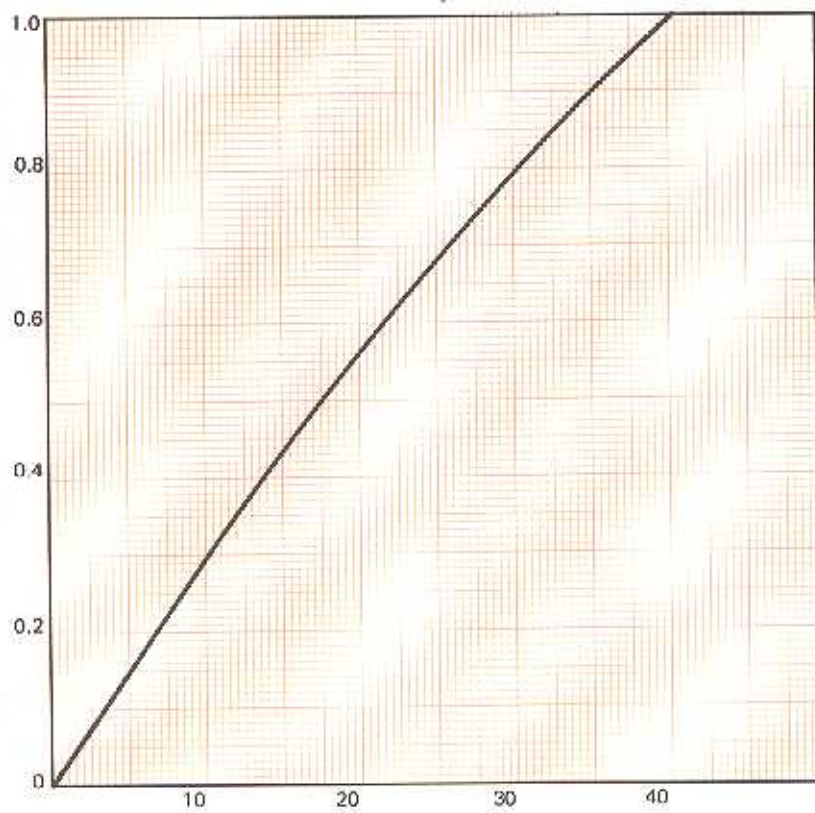
In a nitrous oxide-acetylene flame most of these interferences do not arise; only ruthenium and iridium interfere and 0.5% Zn suppresses these effects (4).

References

- (1) KALLMANN S., HOBART E.W., Anal. Chim. Acta **51**, 120, 1970
- (2) SCHNEPFER M.M., GRIMALDI F.S., Talanta **16**, 1461, 1969.
- (3) SCARBOROUGH J.M., Anal. Chem. **41**, 250, 1969
- (4) ATWELL M.G., HEBERT J.Y., Appl. Spectrosc. **23**, 480, 1969.

NOTES

45. Rh Rhodium



CONCENTRATION µg/ml

Ruthenium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Ruthenium chloride, $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$ A. R. Grade

Solution Technique

Dissolve 2.052 g of $\text{RuCl}_3 \cdot 3\text{H}_2\text{O}$ in 50 ml 1:4 hydrochloric acid and dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Ru.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10mA
Fuel	acetylene
Support	air
Flame Stoichiometry	oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
349.9	0.2	30–120	0.72
392.6	0.2	400–1600	8.5

Detection Limit

0.16 $\mu\text{g}/\text{ml}$ at 349.9 nm using an air-acetylene flame

Flame Emission

Wavelength	372.8 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	air
Detection limit data not available	

INTERFERENCES

In an air-acetylene flame most other elements and most acids interfere in an unpredictable manner. The only releasing agent discovered so far is uranyl nitrate (4% W/V U), which overcomes all interferences except the depression due to Ti (III).

The nitrous oxide flame is not recommended as it does not entirely remove the interferences and gives poorer sensitivity.

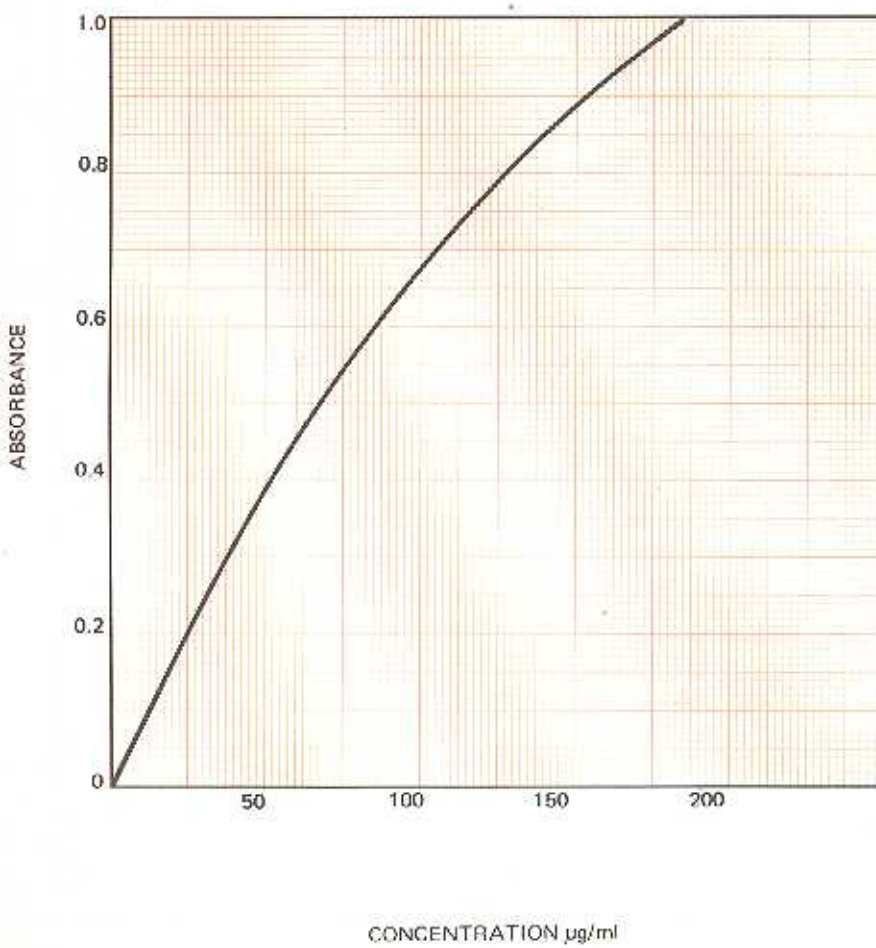
Mutual interference with the other noble metals has been observed in the air-acetylene flame. Uranium reduces these interferences.

References

MONTFORD B., CRIBBS S.C., *Anal. Chim. Acta* **53**, 101, 1971.
SCARBOROUGH J.M., *Anal. Chem.* **41**, 250, 1969.

NOTES

46. Ru Ruthenium



Antimony

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Antimony metal granules	99.99%
Potassium antimonyl tartrate	99.99%

Solution Technique

Dissolve 1.000 g antimony in 100 ml hydrochloric acid containing 2 ml nitric acid, and dilute to 1 litre to give 1000 µg/ml Sb as Sb (V) in 10% hydrochloric acid. Make all further dilutions with 10% hydrochloric acid, or add 1% tartaric acid to prevent precipitation of antimony oxychlorides.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10mA
Fuel (Note)	acetylene
Support	air
Flame Stoichiometry	oxidizing

Note: Air-propane flames may be used with greater sensitivity but increased interference effects; and nitrous oxide-acetylene flames with reduced sensitivity and no interference effects.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
217.6	0.2	10-40	0.29
206.8	0.2	15-60	0.35
231.2	0.5	25-100	0.68
212.7	1.0	100-400	2.0

Detection Limit

0.07 µg/ml at 217.6 nm using an air-acetylene flame.

Flame Emission

Wavelength	259.8 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection limit data not available	

INTERFERENCES

In an air-acetylene flame, copper and nickel depress the signal, especially in a reducing flame. A more oxidizing flame removes the effect.

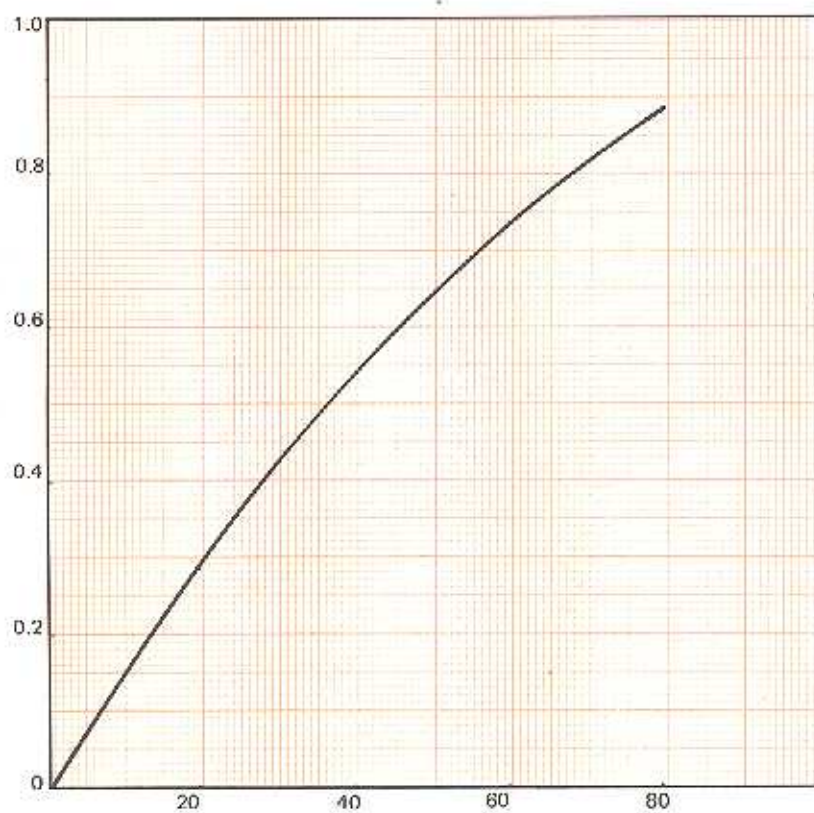
It has been suggested that the presence of excess oxidant in the solution depresses the absorbance, although for pure solutions in either oxidation state no differences in absorbance have been found. It is essential during sample preparation to avoid boiling antimony solutions containing chloride, as otherwise some antimony will be lost.

REFERENCES

- YANAGISAWA M., SUZUKI M., TAKEUCHI T., *Anal. Chim. Acta.* **47**, 121, 1969.
 MOSTYN R.A., CUNNINGHAM A.F., *Anal. Chem.* **39**, 433, 1967.

NOTES

47. Sb Antimony



CONCENTRATION µg/ml

Scandium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Scandium oxide Sc_2O_3 99.9%

Solution Technique

Dissolve 1.534 g of ignited scandium oxide in a minimum volume of dilute hydrochloric acid (1:1) and make up to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Sc.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-1.5 cm.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	$\mu\text{g}/\text{ml}$	$\mu\text{g}/\text{ml}$
391.2	0.2	15-60	0.35
327.4	0.2	50-200	1.0
326.9	0.2	80-320	1.8

Detection Limit

0.05 $\mu\text{g}/\text{ml}$ at 391.2 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	402.0 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.6 $\mu\text{g}/\text{ml}$

INTERFERENCES

Scandium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank.

Sulphate or fluoride anions strongly suppress scandium absorption even when these interferents are only present in similar quantities to scandium. Many cations suppress or enhance the absorption when present in one hundred fold excess.

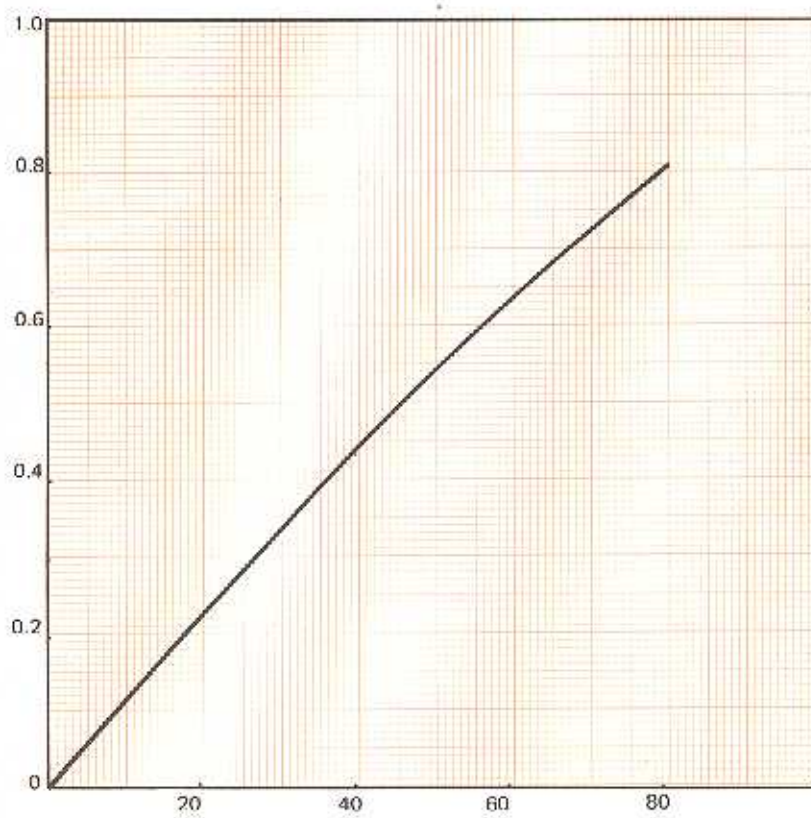
To minimize the effects of these interferences, avoid using sulphate or fluoride where possible and ensure that the standard solutions contain the same reagents and major matrix elements at approximately the same concentration.

REFERENCE

KRIEGE O.H., WELCHER G.G., *Talanta* **15**, 781 (1968).

NOTES

48. Sc Scandium



CONCENTRATION $\mu\text{g/ml}$

Selenium

SELENIUM STANDARD CONDITIONS

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Selenium metal pellets 99.9%

Solution Technique

Dissolve 1.000 g of selenium metal in 80 ml of 1:1 nitric acid, heat gently to initiate the reaction – cool if the reaction proceeds too vigorously. Cool and dilute to 1 litre to give 1000 µg/ml Se.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 10 mA
Fuel hydrogen
Support air
Flame Stoichiometry highly reducing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
196.0	1.0	20–80	0.48
204.0	0.3	300–1200	6.8

Detection Limit

1 µg/ml at 196.0 nm using an air-hydrogen flame. The use of a photomultiplier having good U.V. response improves the signal stability (e.g. Hamamatsu R106). The use of an air-acetylene flame also results in steadier absorption signals.

Flame Emission

Selenium is not generally determined by flame emission because of the poor emission characteristics of the element.

INTERFERENCES

At 196.0 nm and 204.0 nm, non-atomic species in the flame absorb strongly. Use a hydrogen continuum lamp to measure the absorbance signal caused by these species and subtract this signal from the apparent absorbance signal for each sample and standard solution.

Chemical interference from most elements is within an allowable 5% in the air-hydrogen flame. Only barium, lead, lithium, sodium and strontium depress the selenium absorbance by 25, 55, 12, 6 and 15% respectively.

The air-acetylene flame reduces all of these interferences except those of sodium and strontium which have a 23–14% enhancing effect on the absorbance. (c.f. the depressing effect of the air-hydrogen flame).

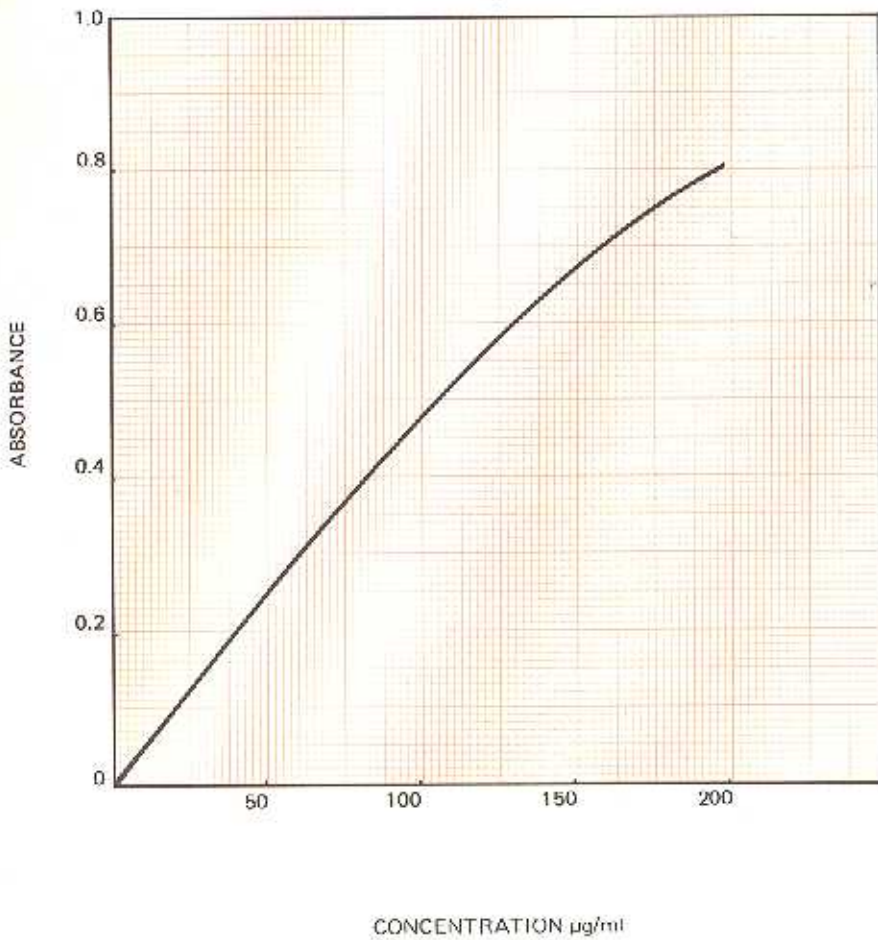
With the nitrous oxide-acetylene flame all interferences are overcome but the sensitivity is reduced.

REFERENCES

NAKAHARA, T. et al; Anal. Chim. Acta. **50**, 51, 1970.

NOTES

49. Se Selenium



Silicon

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Silicon dioxide 99.99%

Solution Technique

Fuse 2.14 g of silicon dioxide with 8 g of sodium hydroxide in a zirconium crucible at dull red heat until a clear melt is obtained. Cool, dissolve the cake in 100 ml of 1:3 hydrochloric acid and make up to 1 litre to give 1000 µg/ml Si.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 15 mA
Fuel acetylene
Support nitrous oxide
Flame Stoichiometry strongly reducing;
yellow outer edge.
red cone: 2-3 cm high.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
251.6	0.2	70-280	1.6
250.7	0.2	200-800	4.6
251.4	0.2	250-1000	5.6
252.4	0.2	250-1000	5.7
288.2	0.2	1000-4000	27

Detection Limit

0.3 µg/ml at 251.6 nm using a nitrous oxide-acetylene flame.

The use of a less reducing flame (as described in Interferences) results in more stable absorption signals and eliminates any memory effect due to formation of silicon carbide on the edges of the burner slot.

Flame Emission

Wavelength 251.6 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection Limit 4.0 µg/ml

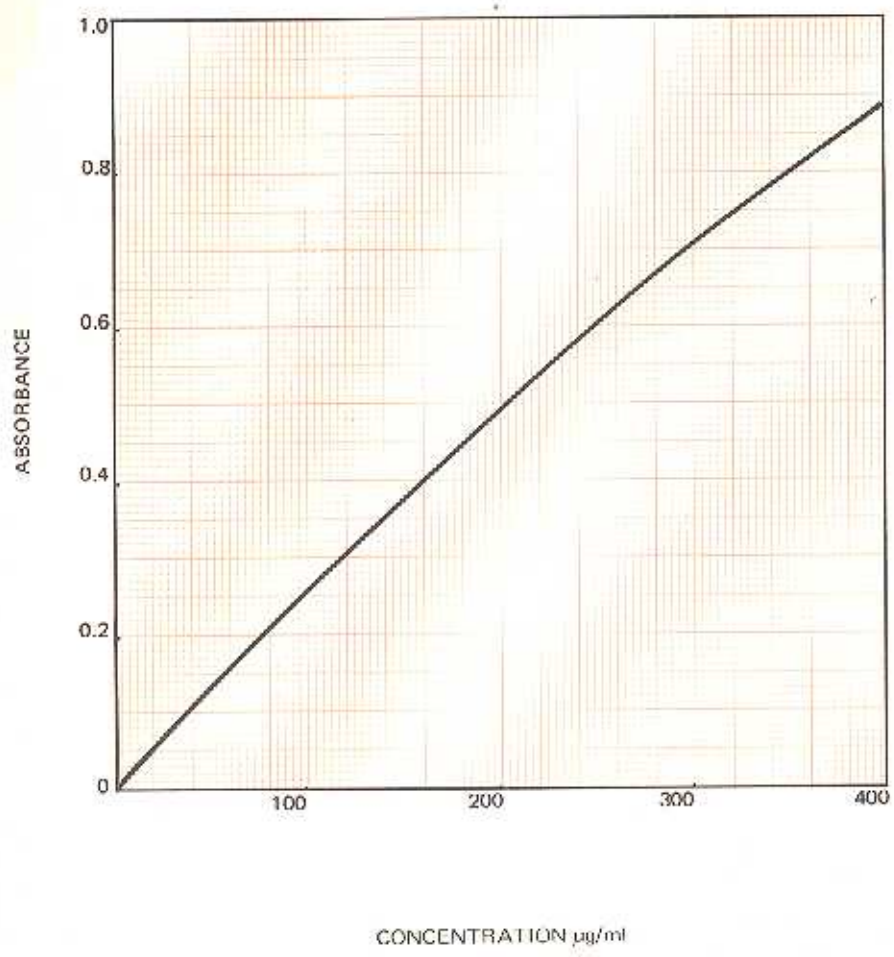
INTERFERENCES

Severe depression of silicon absorbance has been observed in the presence of hydrofluoric acid, boric acid and potassium at significant levels (1%).

The effect is minimised by adjusting the flame to neutral stoichiometry (red cone 0.5 - 1 cm high), with consequent loss of sensitivity.

NOTES

50. Si Silicon



Samarium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Samarium oxide Sm_2O_3 99.9%

Solution Technique

Dissolve 1.159 g of Sm_2O_3 in 10 ml 1:1 hydrochloric acid and dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Sm.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-1.5 cm

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
429.7	0.2	300-1200	6.9
476.0	0.2	600-2400	13

Detection Limit

4.0 $\mu\text{g}/\text{ml}$ at 429.7 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	442.4 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.06 $\mu\text{g}/\text{ml}$

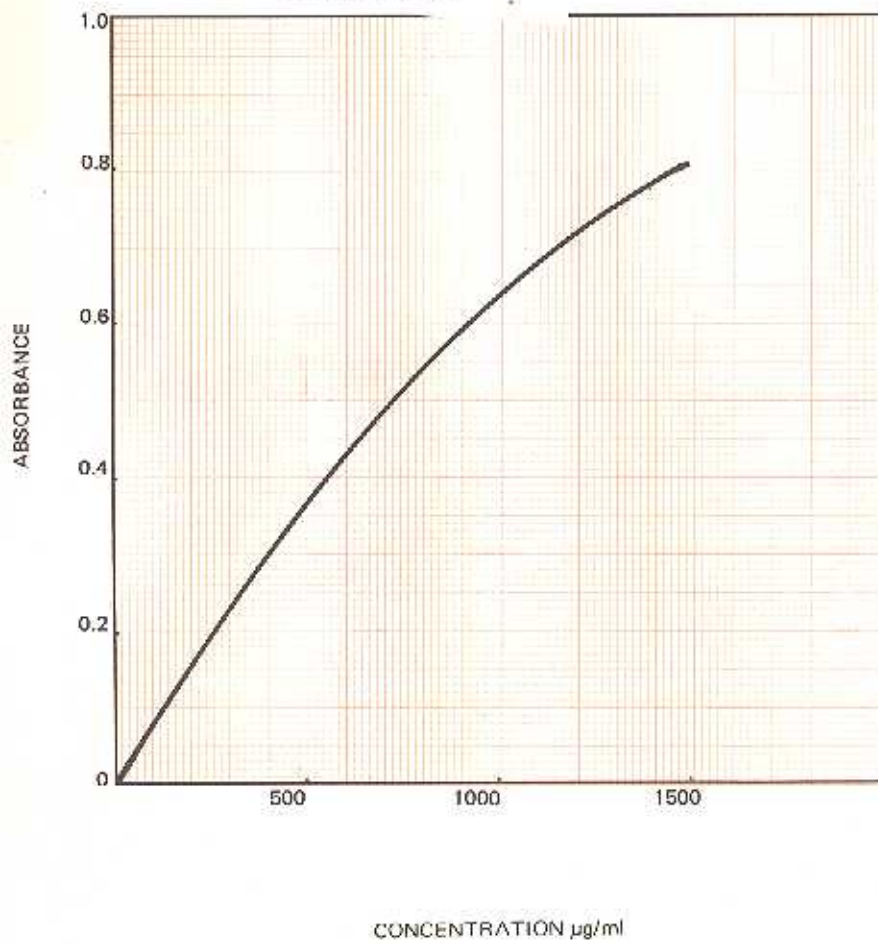
The flame emission signal for samarium is stronger than the atomic absorption signal, especially at low concentrations. Below 300 $\mu\text{g}/\text{ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

INTERFERENCES

Samarium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank.

NOTES

51. Sm Samarium



Tin

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Tin metal granules 99.9%

Solution Technique

Dissolve 1.000 g of tin in 100 ml hydrochloric acid (warm to 60°C if necessary). Cool and dilute to 1 litre to give 1000 µg/ml Sn in 1:9 hydrochloric acid. All further dilutions are made with 1:9 hydrochloric acid or 1% tartaric acid.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 8 mA
Fuel (Note 1) acetylene
Support (Note 1) nitrous oxide
Flame Stoichiometry reducing; red cone
2 cm

Note 1: The nitrous oxide-acetylene flame is recommended because interferences are minimized in this flame. Greater sensitivity is obtained in the air hydrogen flame, and this is recommended when no other elements are present. Air-acetylene is not recommended.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
286.3	0.2	50–200	1.0 (a)
	0.2	200–800	4.0 (b)
224.6	0.1	25–100	0.46 (a)
	0.1	100–400	2.0 (b)
233.5	0.2	25–100	0.6 (a)
266.1	0.2	800–3200	18.7 (a)
235.4	0.2	50–200	1.1 (b)

(a) Air-hydrogen flame

(b) Nitrous oxide-acetylene flame.

Note: With the nitrous oxide-acetylene flame, the 235.4 nm line is preferred because it provides greater sensitivity and good linearity over a wide concentration range.

However, the 286.3 nm line should be used when background absorption is encountered at the lower wavelengths.

Detection Limit

0.03 µg/ml at 224.6 nm using an air-hydrogen flame.

Flame Emission

Wavelength 284.0 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection Limit 0.7 µg/ml

A strongly reducing flame is required. $\times 10^{-4}$.

INTERFERENCES

In the air-hydrogen flame, Juliano and Harrison observed many cationic and anionic interferences. In particular, the alkali and alkaline earth metals affect tin absorbance in any concentration, tending towards a constant elevation or depression of signal at surprisingly low interferent concentration.

These can be summarised; for 50 µg/ml Sn

Metal	Effect on Sn absorbance	Constant between
Li	Depressed 10%	50–2000 µg/ml
Na	Depressed 10%	500–2000 µg/ml
K	Elevated 20%	50–2000 µg/ml
Rb	Elevated 20%	100–2000 µg/ml
Cs	Elevated 25%	100–2000 µg/ml
Mg	Elevated 15%	500–2000 µg/ml
Ca	Elevated 10%	200–2000 µg/ml
Sr	Elevated 25%	500–2000 µg/ml
Ba	No effect	

NOTES

The transition series metals showed varied effects. For copper the interference was a 20% elevation at 200–1000 $\mu\text{g/ml}$, decreasing to a 10% elevation at 2000 $\mu\text{g/ml}$. For cobalt continuously increasing elevation was observed with increasing concentration, while for zinc a slight depression increased continuously with concentration.

For aluminium the effect was markedly dependent on the height of observation in the flame, becoming negligible at high aluminium concentrations high in the flame. In the presence of titanium a spectacular enhancement was recorded in the lower regions of the flame.

Phosphoric acid gave a depression of the signal the effect decreasing somewhat in a more reducing flame. The depression attributed to sulphuric acid was not reduced in the same way. Other acids gave small effects.

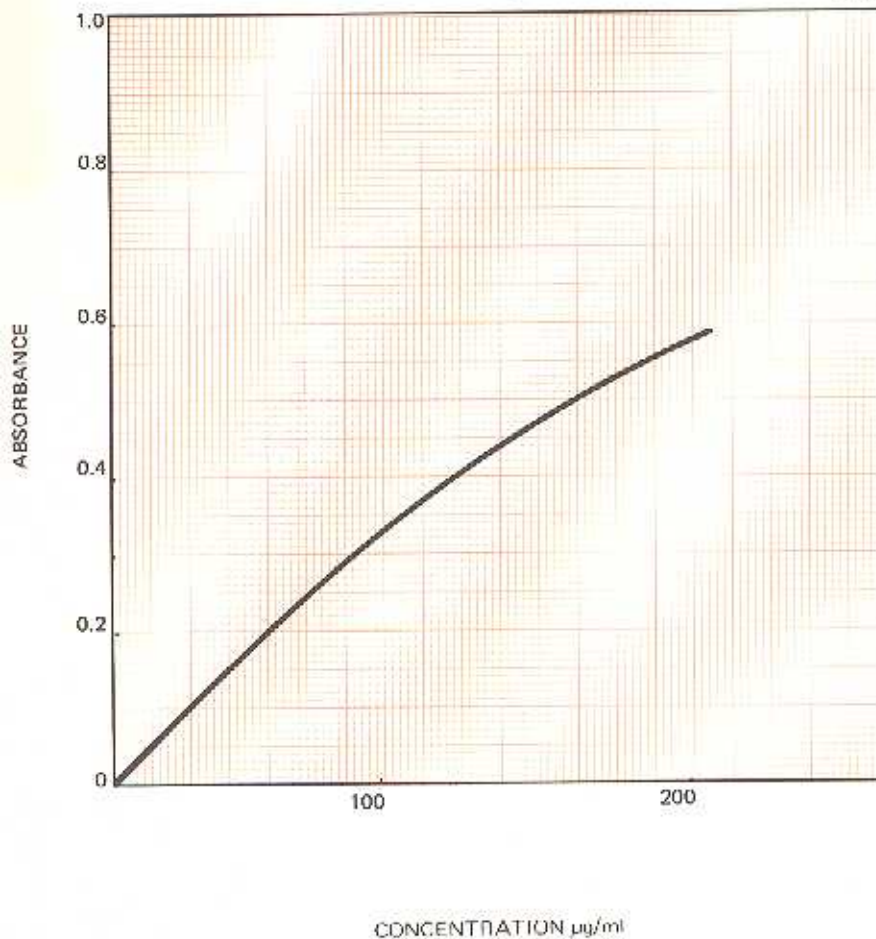
All these interferences are also found in the air-acetylene flame although the extent is reduced. In the nitrous oxide-acetylene flame, however, no interferences have been reported.

REFERENCE

JULIANO P.O., HARRISON W.W., *Anal. Chem.* **42**, 84, 1970

52. Sn Tin

Sn



Strontium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Strontium carbonate SrCO_3 99.99%

Solution Technique

Dissolve 1.685 g SrCO_3 in 10 ml 1:1 nitric acid and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Sr.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA.
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	strongly oxidizing; red cone 0.25 cm.

An air-acetylene flame can also be used but the sensitivity is poorer and chemical interferences are significant.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	$\mu\text{g/ml}$	$\mu\text{g/ml}$
460.7	0.2	2-10	0.044

Detection Limit

0.004 $\mu\text{g/ml}$ at 460.7 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	460.7 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.001 $\mu\text{g/ml}$

INTERFERENCES

In the air-acetylene flame, silicon, aluminium, titanium, zirconium, phosphate and sulphate depress the signal at all concentrations. These effects can be removed by adding 1% lanthanum as nitrate or chloride to all solutions, or 0.01 M EDTA with 2000 $\mu\text{g/ml}$ lanthanum.

In the nitrous oxide-acetylene flame these interferences do not occur.

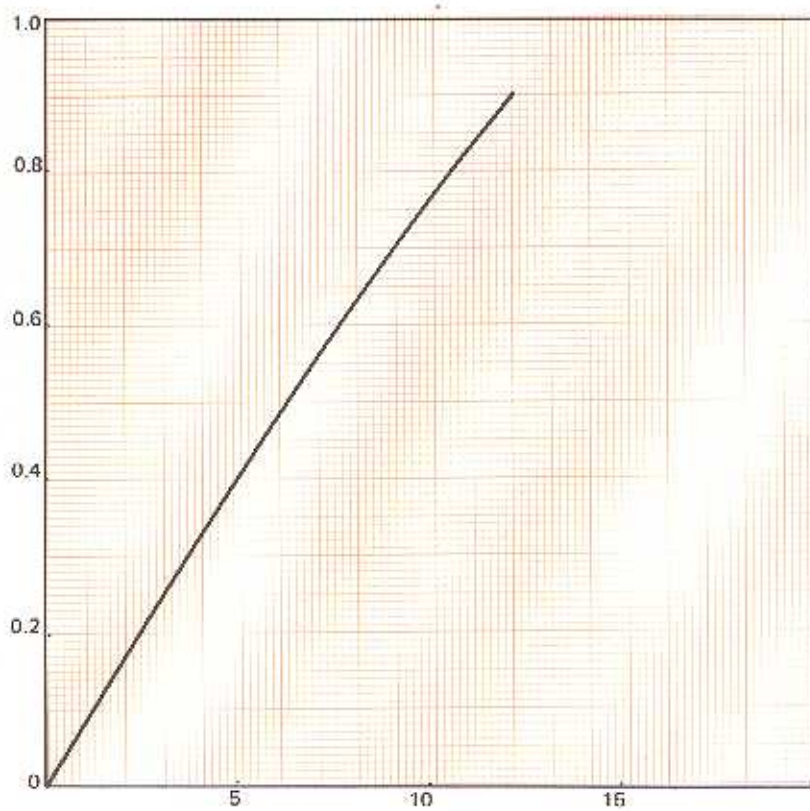
Strontium is partially ionized in all flames. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g/ml}$ potassium in all solutions including the blank.

REFERENCES

- WILLIAMS C.H., *Anal. Chim. Acta.* **22**, 163, 1960.
 ADAMS P.B., PASSMORE W.O., *Anal. Chem.* **38**, 630, 1966.
 AMOS M.D., WILLIS J.B., *Spectrochim. Acta.* **22**, 1325, 1966.

NOTES

53. Sr Strontium



CONCENTRATION µg/ml

Tantalum

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Tantalum metal (strip) 99.9%

Solution Technique

Dissolve 1.000 g of tantalum metal strip in 10 ml hydrofluoric acid, 5 ml water by the slow DROPWISE addition of 10 ml of concentrated nitric acid. Allow the reaction to subside before each addition. Dilute to 1 litre to give 1000 µg/ml Ta.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 20 mA.
Fuel acetylene
Support nitrous oxide
Flame Stoichiometry reducing;
red cone 1-2 cm.

Working Conditions (variable)

Wavelength	Spectral Band Pass	Optimum Working Range	Typical Sensitivity
nm	nm	µg/ml	µg/ml
271.5	0.2	500-2000	11
275.8	0.2	2500-10000	58

Detection Limit

2.1 µg/ml at 271.5 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength 481.3 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection Limit 3 µg/ml

INTERFERENCES

Fluoride and ammonium ions in the absence of alkali metals increase the absorbance signal. In the presence of alkali metals and fluoride the absorbance is depressed. Sulphate depresses the absorbance, and phosphate enhances it up to 2% phosphoric acid - higher concentrations of phosphate depress the absorbance.

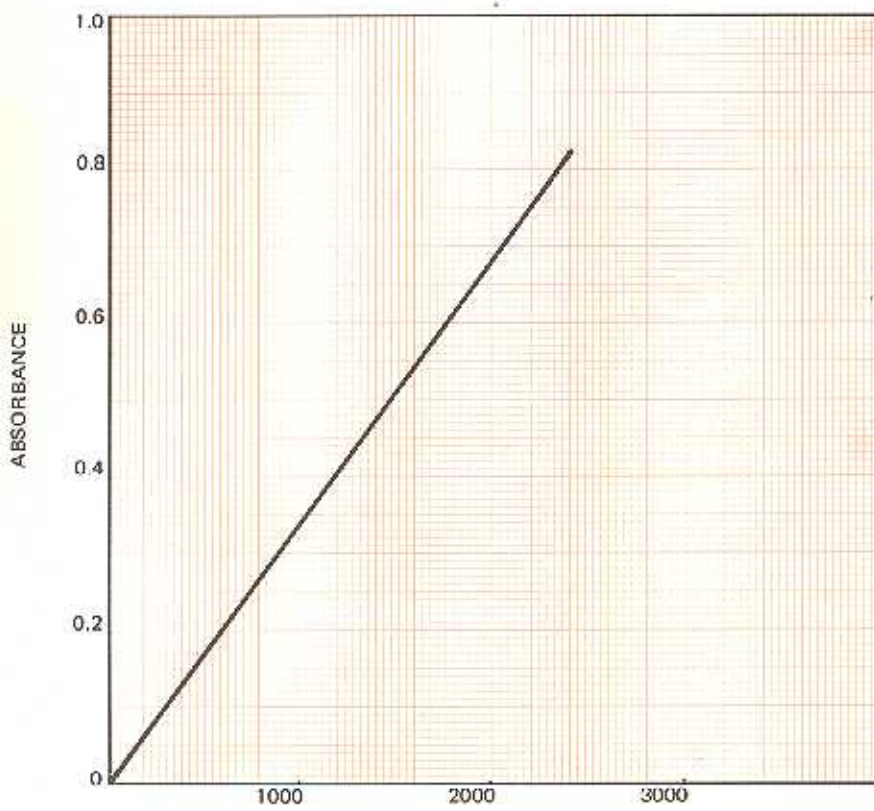
In the absence of alkali metals, the addition of ammonium fluoride to all solutions (0.1M) gives improved sensitivity and reduces acid interferences. In the presence of alkali metals no releasing agents are known. Tantalum (V) cupferrate can be extracted from 0.5% tartrate systems at pH 0 with isoamyl alcohol; Tantalum as the fluoride is extracted from 6M sulphuric acid, 10M hydrofluoric acid + 2.2M ammonium fluoride medium into methyl isobutyl ketone.

REFERENCES

- THOMAS P.E., PICKERING W.F., *Talanta* **18**, 127, 1971.
BOND A.M., *Anal. Chem.* **42**, 932, 1970.
STARY J., *The Solvent Extraction of Metal Chelates*, Pergamon Press, Oxford, 1964.
MILNER G.W.C., BARNETT G.A., SMALES A.A., *Analyst* **80**, 380, 1955.

NOTES

54. Ta Tantalum



CONCENTRATION µg/ml

Terbium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Terbium oxide Tb_4O_7 99.99%

Solution Technique

Dissolve 5.881 g of Tb_4O_7 in 20 ml of 1:1 hydrochloric acid and dilute to 1 litre to give 5000 $\mu\text{g/ml}$ Tb.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	15 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-1.5 cm

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g/ml}$	Typical Sensitivity $\mu\text{g/ml}$
432.7	0.2	500-2000	11
431.9	0.2	1000-4000	21
433.9	0.2	1300-5200	28

Detection Limit

0.5 $\mu\text{g/ml}$ at 432.7 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	596.6 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.2 $\mu\text{g/ml}$

Emission at 596.6 nm is band emission and severe spectral interference may be expected. An alternative is to use the line emission at 432.6 nm where a detection limit of 1 $\mu\text{g/ml}$ has been measured.

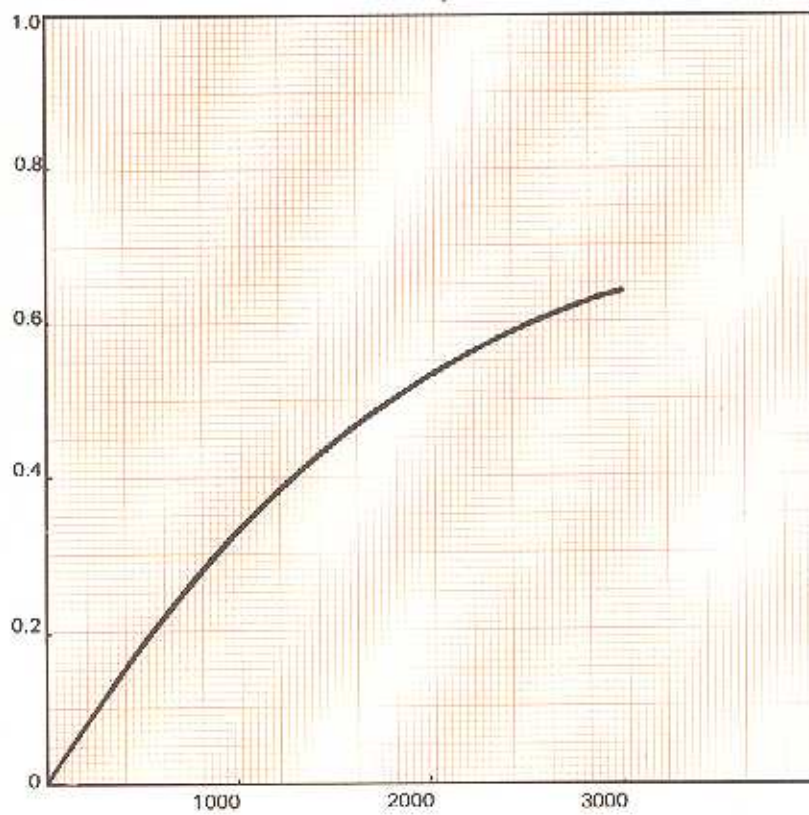
INTERFERENCES

Terbium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g/ml}$ potassium in all solutions including the blank.

Iron, aluminium, silicon and hydrofluoric acid all cause depression of the terbium absorbance. Standard solutions should therefore contain these elements and reagents in approximately the same concentration as in the sample solutions.

NOTES

55. Tb Terbium



CONCENTRATION $\mu\text{g/ml}$

Tellurium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Tellurium metal granules 99.99%

Solution Technique

Dissolve 1.000 g of tellurium in 20 ml of 1:1:1 nitric acid:hydrochloric acid:water, without boiling. Dilute to 1 litre to give 1000 µg/ml Te.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 8 mA
Fuel acetylene
Support air
Flame Stoichiometry highly oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
214.3	0.2	10-40	0.26
225.9	0.5	200-800	4.2
238.6	0.2	2000-8000	37

Detection Limit

0.12 µg/ml at 214.3 nm using an air-acetylene flame. Refer to interferences.

Flame Emission

Wavelength 214.3 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide

Detection Limit data not available

The determination of tellurium by flame emission is not recommended.

INTERFERENCES

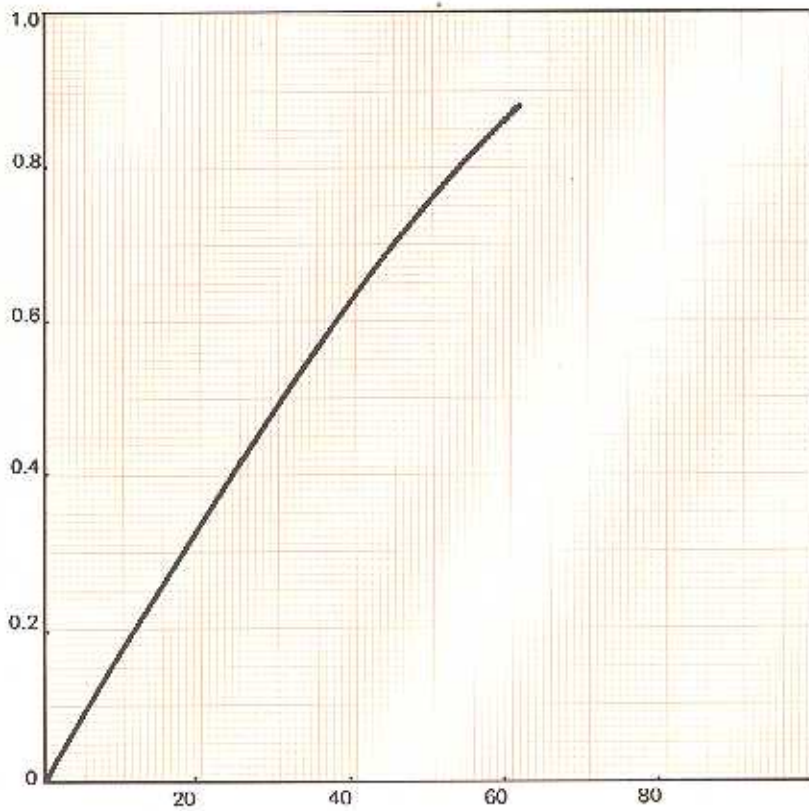
No severe interferences have been encountered in the air-acetylene flame.

The use of air-hydrogen can lead to interference in samples having very high matrix levels of acids.

At low tellurium concentrations, acids and dissolved carbon dioxide can absorb significant amounts of radiation. Use a hydrogen continuum lamp to measure the absorbance signal caused by these species and subtract this signal for each sample and standard solution.

NOTES

56. Te Tellurium



CONCENTRATION µg/ml

Titanium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Titanium metal (strip) 99.99%

Solution Technique

Dissolve 1.000 g of titanium metal strip in 10 ml of hydrofluoric acid, 10 ml water by the slow DROPWISE addition of 20 ml nitric acid. Allow the reaction to subside before each addition. Dilute to 1 litre to give 1000 µg/ml Ti.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-1½ cm.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
364.3	0.2	60-240	1.4
365.4	0.2	100-400	1.9
399.0	0.2	200-800	4.1

Detection Limit

0.1 µg/ml at 364.3 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	399.8 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	1 µg/ml

INTERFERENCES

Most metallic elements enhance the titanium absorption signal at concentrations above 500 µg/ml, probably by competing with titanium for the available oxygen in the flame and reducing the refractory titanium oxide. Sodium above 1000 µg/ml depresses the signal; fluoride, chloride and ammonium ions enhance it. Releasing agents include:

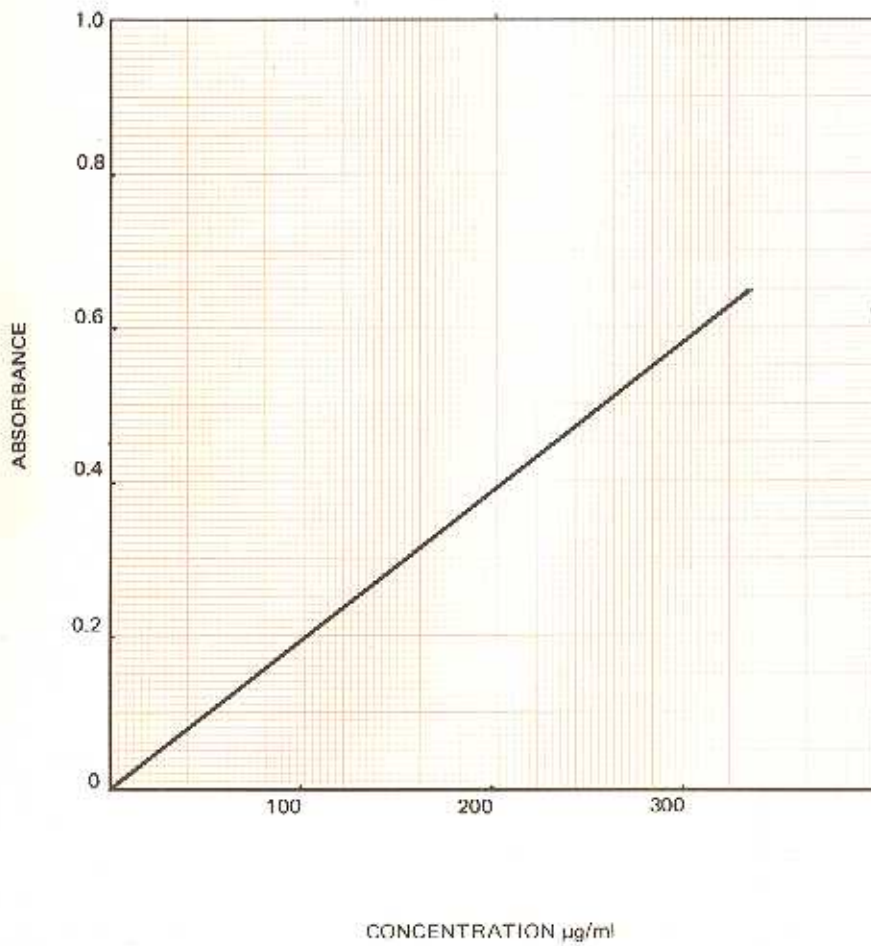
1000 µg/ml Al in HCl
2000 µg/ml KCl
0.1 M NH₄F

REFERENCES

THOMAS P.E., PICKERING W.F., *Talanta* **18**, 127, 1971.
WILLIS J.B., *Appl. Opt.* **7**, 1295, 1968.
BOND A.M., *Anal. Chem.* **42**, 932, 1968.
MOSTYN R.A., CUNNINGHAM A.F., *At. Abs. News* **1**, **6**, 86, 1967.
SASTRI V.S., CHAKRABARTI C.L., WILLIS D.E., *Talanta* **16**, 1093, 1969.

NOTES

57. Ti Titanium



Thallium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Thallos nitrate $TlNO_3$ 99.99%

Solution Technique

Dissolve 1.303 g of dried $TlNO_3$ in 20 ml 1% nitric acid and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Tl.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 20 mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g/ml}$	Typical Sensitivity $\mu\text{g/ml}$
276.8	0.2	10–50	0.28
258.0	0.5	1000–4000	21

Other lines are available at 377.6 nm and 237.9 nm of intermediate sensitivity.

Detection Limit

0.02 $\mu\text{g/ml}$ at 276.8 nm using an air-acetylene flame.

Flame Emission

Wavelength 535.1 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection limit data not available.

INTERFERENCES

No interferences have been reported in an air-acetylene flame.

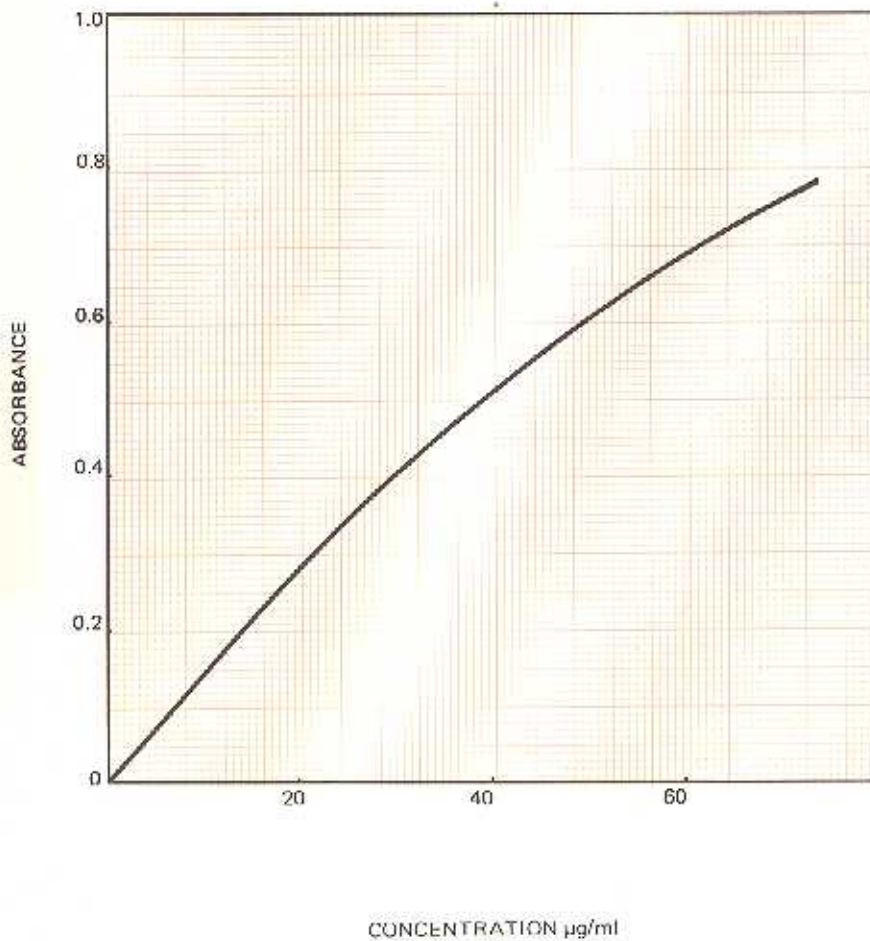
Thallium is partially ionized in the nitrous-oxide-acetylene flame.

To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g/ml}$ potassium in all solutions including the blank.

In the air-acetylene flame ionization is negligible.

NOTES

58. Tl Thallium



Thulium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Thulium oxide Tm_2O_3 99.9%

Solution Technique

Dissolve 1.142 g of Tm_2O_3 in 20 ml 1:1 hydrochloric acid and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Tm.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	15 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g/ml}$	Typical Sensitivity $\mu\text{g/ml}$
371.8	0.2	10-50	0.29
420.4	0.5	40-160	0.90
436.0	0.2	100-500	2.7
530.7	0.5	250-1000	5.2

Detection Limit

0.02 $\mu\text{g/ml}$ at 371.8 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	410.6 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.03 $\mu\text{g/ml}$

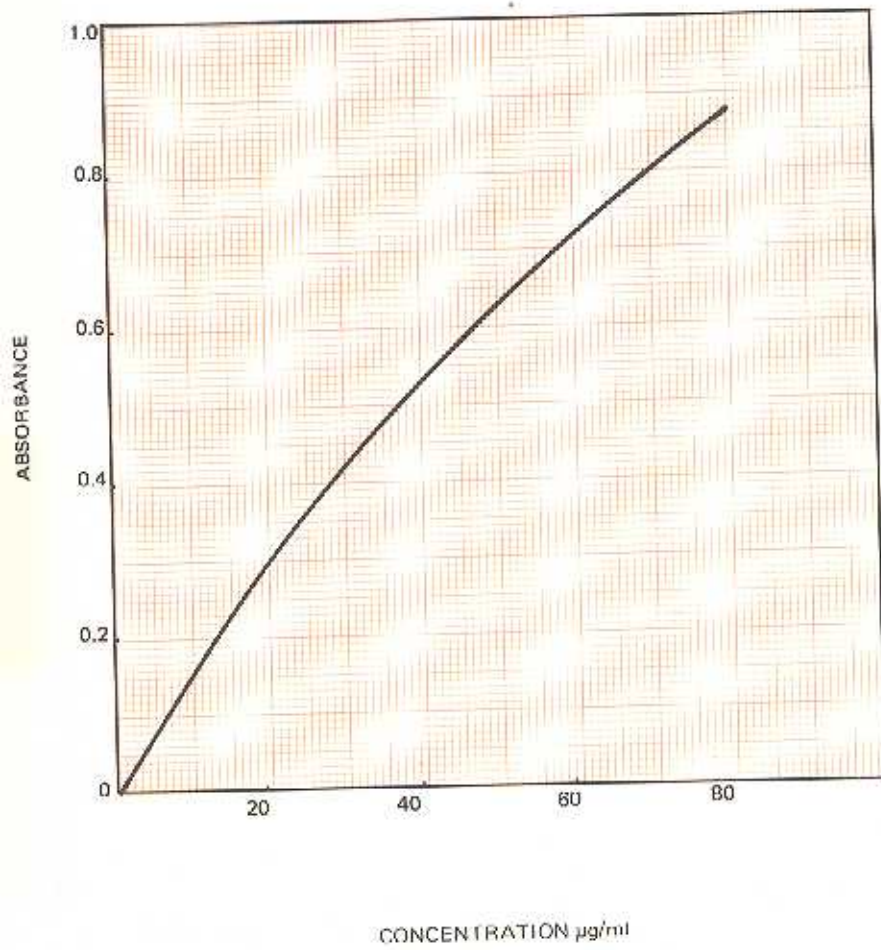
The flame emission signal for thulium is just as strong as the atomic absorption signal, especially at low concentrations. Below 10 $\mu\text{g/ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

INTERFERENCES

Thulium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g/ml}$ potassium in all solutions including the blank.

NOTES

59. Tm Thulium



Uranium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Uranyl nitrate, $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ A. R. Grade

Solution Technique

Dissolve 21.10 g of uranyl nitrate in water and dilute to 1 litre to give 10000 $\mu\text{g}/\text{ml}$ U. Normal radio-chemical precautions are necessary.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-1.5 cm.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
358.5	0.1	5000-20000	113
356.7	0.1	8000-32000	170
351.5	0.1	10000-40000	220
348.9	0.1	15000-60000	330

Detection Limit

39 $\mu\text{g}/\text{ml}$ at 358.5 nm using a nitrous oxide-acetylene flame.

Flame Emission

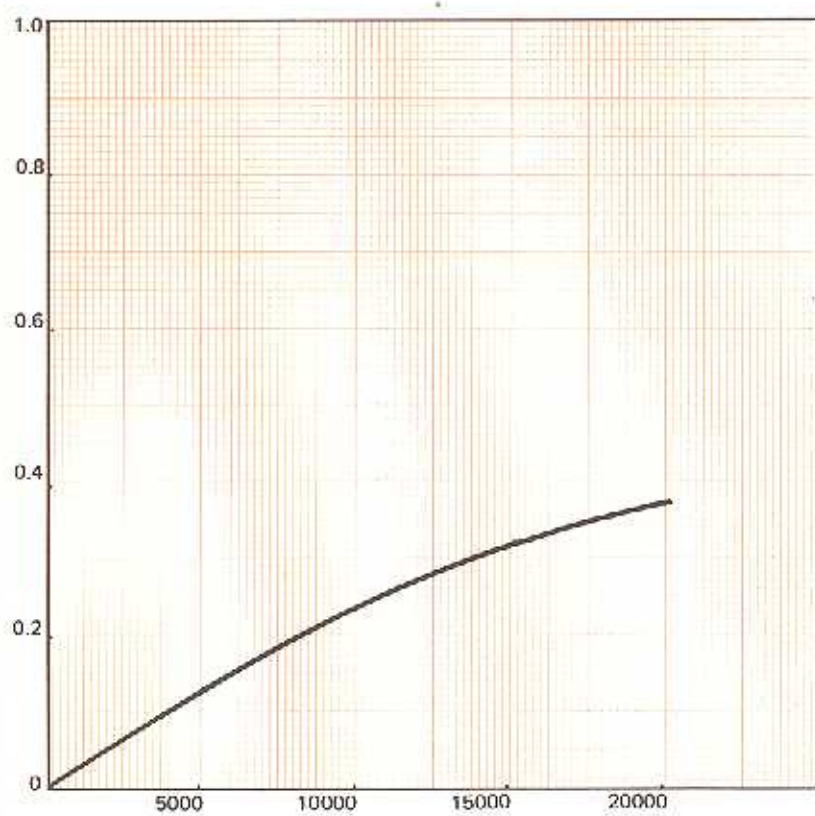
Wavelength	591.5 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	50 $\mu\text{g}/\text{ml}$

INTERFERENCES

Uranium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank.

NOTES

60. U Uranium



CONCENTRATION $\mu\text{g/ml}$

Vanadium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Vanadium metal granules 99.99%

Solution Technique

Dissolve 1.000 g of vanadium in 40 ml nitric acid and dilute to 1 litre to give 1000 µg/ml V.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-1½ cm.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
318.5	0.1	40-120	0.88
318.4	0.1	60-240	1.4
318.3			
306.6	0.2	150-600	3.9
439.0	0.5	350-1400	7.8

Detection Limit

0.11 µg/ml at 318.5 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	437.9 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.3 µg/ml

INTERFERENCES

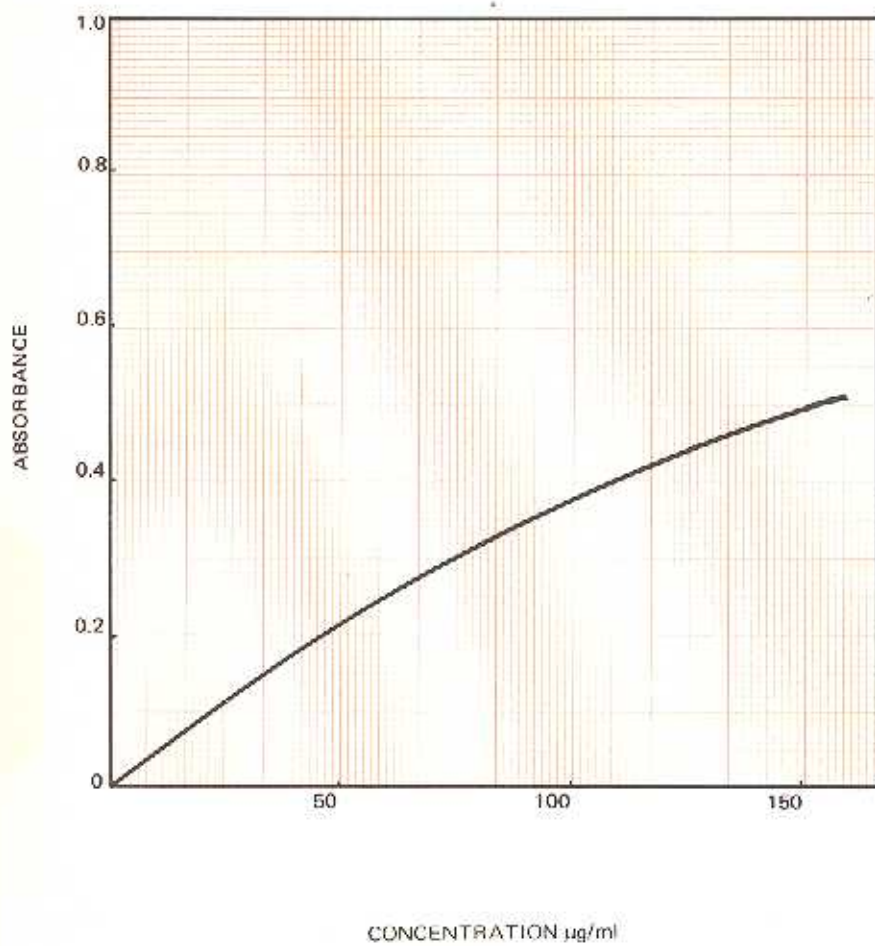
Vanadium is not as greatly affected as niobium or tantalum by alkali metal fluorides. Ammonia (0.1M NH₄F) enhances the signal, as do a large range of other species. The interferences are removed by adding aluminium (2:1 excess or 2000 µg/ml).

REFERENCES

SACHDEV S.L., ROBINSON J.W., WEST P.W., *Anal. Chim. Acta.* **37**, 12, 1967.
BOND A.M., *Anal. Chem.* **42**, 932, 1970
THOMAS P.E., PICKERING W.F., *Talanta* **18**, 127, 1971.

NOTES

61. V Vanadium



Tungsten

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Tungsten metal rod	99.99%
Sodium tungstate A. R. Grade	Na_2WO_4

Solution Technique

Dissolve 1.000 g tungsten metal in 20 ml nitric acid and 10 ml hydrofluoric acid in a PTFE beaker. Dilute to 1 litre in a plastic volumetric flask to give 1000 $\mu\text{g}/\text{ml}$ W.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1-2 cm.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
255.1	0.1	250-1000	5.8
400.9	0.5	1000-4000	22
407.4	0.5	2000-8000	48

Detection Limit

1.2 $\mu\text{g}/\text{ml}$ at 255.1 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	400.9 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection limit	3 $\mu\text{g}/\text{ml}$

INTERFERENCES

In phosphoric or sulphuric acid media, tungsten absorbance depends on the presence or absence of iron, cobalt, copper, potassium and various other species. In hydrofluoric acid media, potassium (at least) has no effect. Ammonium fluoride is reported to have no effect on standards containing sodium and potassium. No comprehensive interference studies have been published.

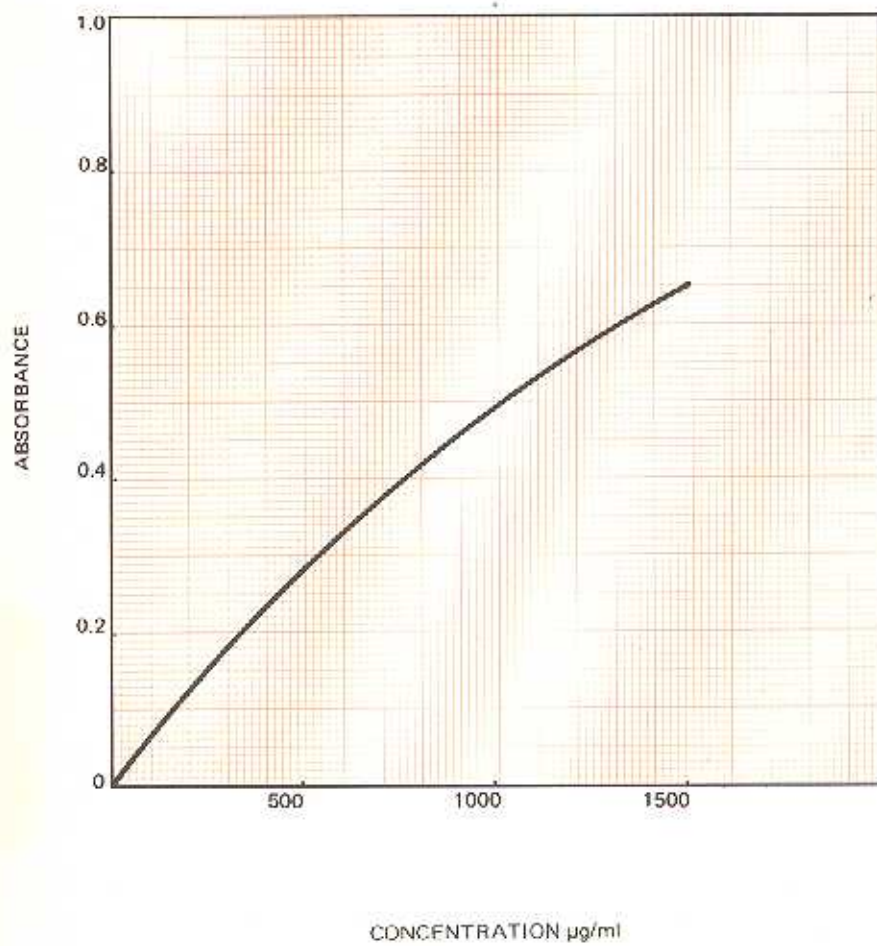
REFERENCES

THOMAS P.E., PICKERING W.F., *Talanta* **18**, 127, 1971.

BOND A.M., *Anal. Chem.* **42**, 932, 1970.

NOTES

62. W Tungsten, Wolfram



Yttrium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Yttrium oxide Y_2O_3 99.99%

Solution Technique

Dissolve 1.270 g Y_2O_3 in 20 ml hydrochloric acid and dilute to 1 litre to give 1000 $\mu\text{g/ml}$ Y.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	10 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1.5-2 cm.

Working Conditions (Variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g/ml}$	Typical Sensitivity $\mu\text{g/ml}$
410.2	0.5	200-800	4.8
414.3	0.2	300-1200	7.2

Detection Limit

0.5 $\mu\text{g/ml}$ at 410.2 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	407.7 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	5 $\mu\text{g/ml}$

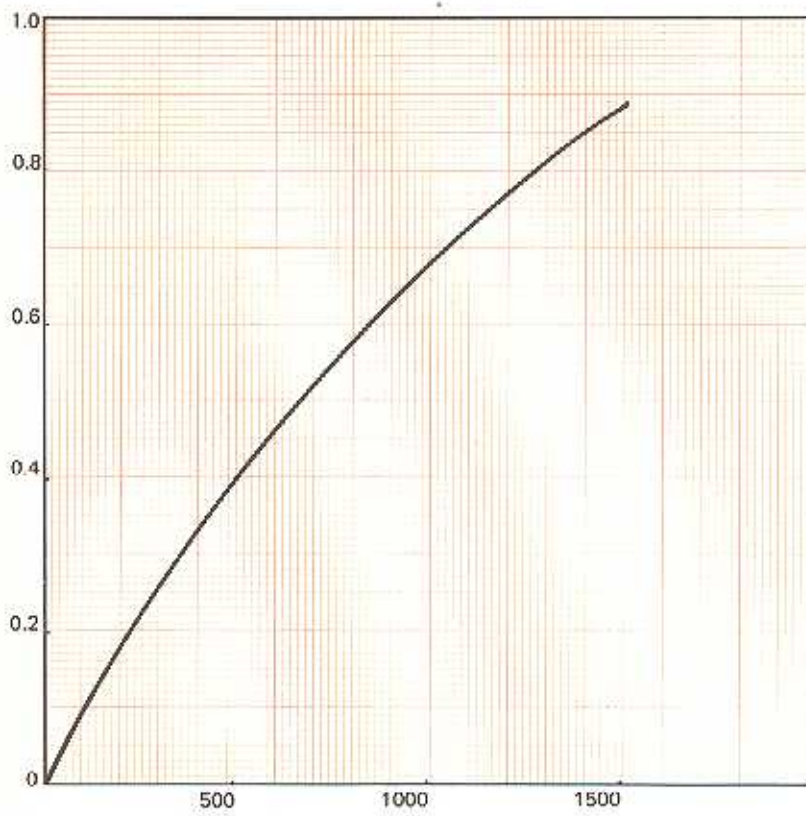
INTERFERENCES

The absorbance signal is depressed in the presence of aluminium, potassium and phosphoric acid at all concentrations.

Yttrium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 4000 $\mu\text{g/ml}$ potassium in all solutions including the blank. The absorbance depression due to potassium is not severe at these concentrations.

NOTES

63. Y Yttrium



CONCENTRATION µg/ml

Ytterbium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Ytterbium oxide Yb_2O_3 99.9%

Solution Technique

Dissolve 1.139 g of Yb_2O_3 in 20 ml hydrochloric acid and dilute to 1 litre to give 1000 $\mu\text{g}/\text{ml}$ Yb.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	5 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1.5-2 cm.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range $\mu\text{g}/\text{ml}$	Typical Sensitivity $\mu\text{g}/\text{ml}$
398.8	0.5	3-12	0.077
246.5	0.2	100-400	2.8
267.3	0.1	1000-4000	27

Detection Limit

0.013 $\mu\text{g}/\text{ml}$ at 398.8 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	398.8 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit	0.0005 $\mu\text{g}/\text{ml}$

The flame emission signal for ytterbium is stronger than the atomic absorption signal, especially at low concentrations. Below 3 $\mu\text{g}/\text{ml}$, determination by flame emission is preferred, although the wavelength required must be accurately isolated to avoid spectral interference from the other rare earth elements. At higher concentrations, atomic absorption is normally used.

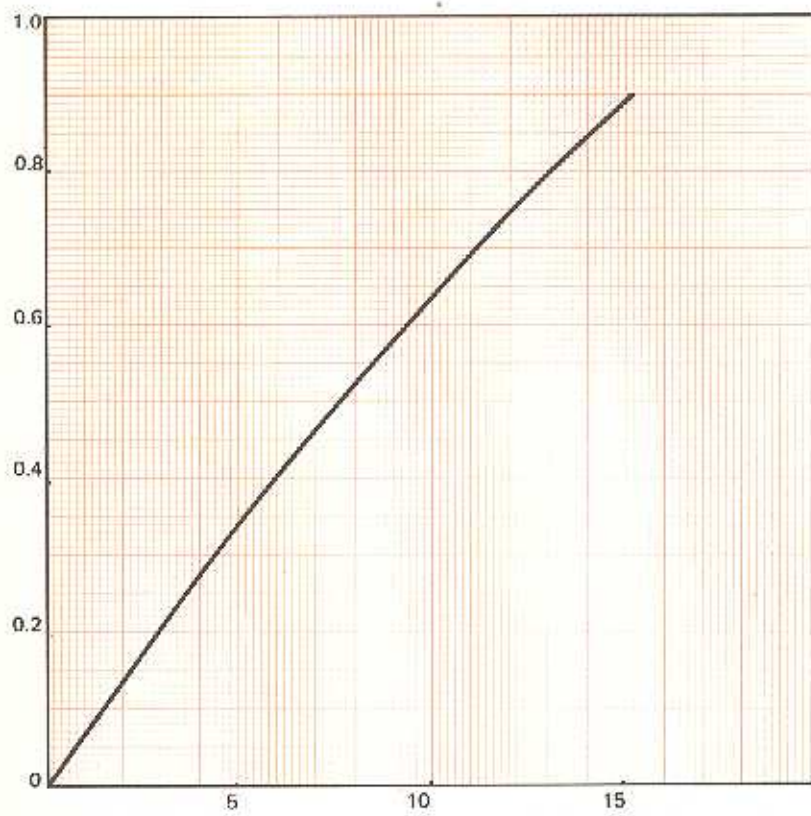
INTERFERENCES

Ytterbium is partially ionized in the nitrous oxide-acetylene flame. To suppress ionization, add potassium nitrate or chloride solution to give a final concentration of 2000 $\mu\text{g}/\text{ml}$ potassium in all solutions including the blank.

Ytterbium absorption and emission signals are depressed in the presence of hydrofluoric acid, iron, aluminum and silicon at all concentrations.

NOTES

64. Yb Ytterbium



CONCENTRATION µg/ml

Zinc

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Zinc metal granules 99.99%

Solution Technique

Dissolve 1.000 g of zinc in 40 ml 1:1 hydrochloric acid and dilute to 1 litre to give 1000 µg/ml Zn.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current 5 mA
Fuel acetylene
Support air
Flame Stoichiometry oxidizing
All other conventional flames can be used.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
213.9	0.2	0.4–1.6	0.009
307.6	0.5	3500–14000	76

Detection Limit

0.002 µg/ml at 213.9 nm using an air-acetylene flame.

Flame Emission

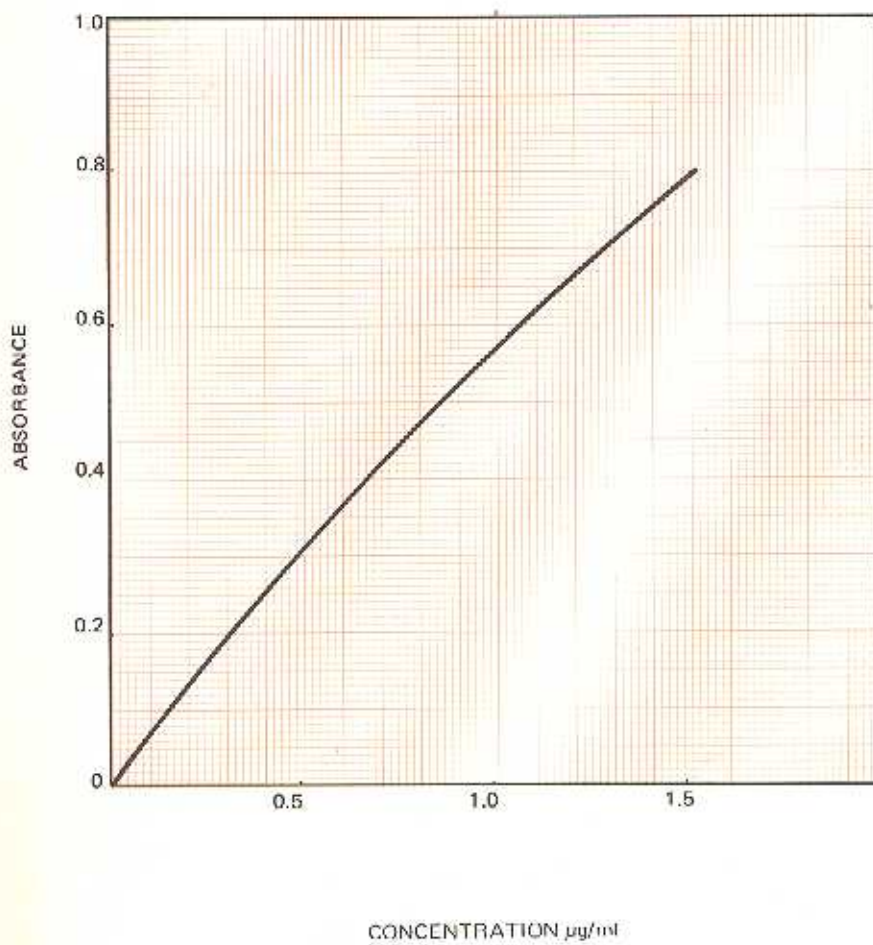
Wavelength 213.9 nm
Spectral Band Pass 0.1 nm
Fuel acetylene
Support nitrous oxide
Detection Limit data not available.

INTERFERENCES

No chemical interferences have been found in an air-acetylene flame. When working with biological samples, ashing is necessary to avoid the physical effects of protein molecules on nebulizer performance. At the 213.9 nm wavelength, non-atomic species in the flame absorb strongly. Where the sample has a high concentration of dissolved solids it is necessary to correct for non-atomic absorption using a hydrogen continuum lamp.

NOTES

65. Zn Zinc



Zirconium

PREPARATION OF STANDARD SOLUTIONS

Recommended Standard Materials

Zirconium metal (strip) 99.99%

Solution Technique

Dissolve 1.000 g of zirconium metal strip in 10 ml of hydrofluoric acid, 10 ml water by the slow DROPWISE addition of 20 ml nitric acid. Allow the reaction to subside before each addition. Dilute to 1 litre to give 1000 µg/ml Zr.

RECOMMENDED INSTRUMENT PARAMETERS

Atomic Absorption

Working Conditions (fixed)

Lamp Current	20 mA
Fuel	acetylene
Support	nitrous oxide
Flame Stoichiometry	reducing; red cone 1½-2 cm.

Working Conditions (variable)

Wavelength nm	Spectral Band Pass nm	Optimum Working Range µg/ml	Typical Sensitivity µg/ml
360.1	0.1	400-1600	9.1
468.8	0.1	4000-16000	75

Detection Limit

1.0 µg/ml at 360.1 nm using a nitrous oxide-acetylene flame.

Flame Emission

Wavelength	360.1 nm
Spectral Band Pass	0.1 nm
Fuel	acetylene
Support	nitrous oxide
Detection Limit data not available	

INTERFERENCES

Most metallic elements depress the absorbance signal, especially at concentrations greater than 1000 µg/ml. Hydrofluoric acid (3%) enhances the signal but at higher concentration gives a slight depression. Ammonium ion and chloride ion (0.1 M) and neutral fluoride compounds give enhancement and remove at least some of the metallic interferences. The addition of ammonium fluoride (0.1 M) has been recommended. The effects are minimized with a more oxidizing flame but sensitivity is reduced.

REFERENCES

THOMAS P.E., PICKERING W.F., *Talanta* **18**, 127, 1971.
BOND A.M., *Anal. Chem.* **42**, 832, 1970.

NOTES

66. Zr Zirconium

