

AE-163

Flame Spectrophotometric Determination
of Strontium in Water and
Biological Material

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AKTIEFÖRETAGET ATOMENERGI

STOCKHOLM, SWEDEN 1964

FLAME SPECTROPHOTOMETRIC DETERMINATION OF
STRONTIUM IN WATER AND BIOLOGICAL MATERIAL

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Summary

A flame spectrophotometric method has been developed for the determination of strontium in biological material and water samples.

Strontium is determined in the presence of calcium at a wavelength of 4607 Å.

The intensity of the strontium emission from the sample is increased if n-butanol is added to a solution of the sample in water. With a 6 vol. % solution of n-butanol in water, an optimum intensity of 3.5 times that obtained with pure water solution is obtained.

Anions and alkali metals which might interfere with the flame spectrophotometric determination are separated from the sample by a simple ion exchange operation.

The method allows determination of strontium in solutions down to 0.1 µg/ml. In this case the standard deviation is 3.1 % and with a strontium concentration of 1 µg/ml the deviation is 0.9 %.

This method has been used for the determination of strontium in samples of varying composition such as bone, meat and skin from fishes, samples of human bones, shell-fish, milk, and water, in which case Sr quantities of 5 µg were determined with an analytical error of less than 5 % and Sr quantities greater than 10 µg with an error of less than 3 %.

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1. Introduction

In order to study the enrichment of strontium in water organisms from the surroundings, an investigation is being made at AB Atomenergi (1) based on the determination of stable strontium in bone, meat and organs from fish, shell-fish, and water. An analytical method was required for this investigation which enabled very small quantities of strontium and calcium to be determined in an extensive sample material of varying composition. As it was also desirable to determine the individual variation of the strontium content in fish of the same kind, it was essential that the method should be extremely sensitive and accurate.

Activation analysis according to Harrison & Dymond (2) and similar methods are characterized by extremely high sensitivity in determination of, for instance, barium and strontium in different biological samples. Spectrometric analysis with emission-spectrograph (3) or X-ray fluorescence (4) has also been used for determination of strontium in similar connections. For routine determination of strontium alone in an extensive sample material, however, flame-photometric methods combined with more or less complete chemical separation of strontium (5), (6), (7) are generally used.

According to earlier experience when developing a flame-photometric method for determination of strontium and calcium in different mole ratios in calcium-strontium apatite (8), one can determine strontium flame-photometrically without prior separation of calcium. Instead the quantity of calcium in the analytical sample is adjusted to a suitable value and used as a so-called radiation buffer. The practical work was therefore directed to investigating various means of increasing the sensitivity of the strontium determination. By utilizing the full sensitivity of the instrument and making suitable organic additions to the sample, it proved possible to determine down to 0.1 ppm strontium in the analytical sample. The influence of other

materials occurring in sample materials used for the strontium determination was also investigated. As a result of the practical work a simple but very sensitive method was arrived at, comprising separation of interfering anions and alkali metals by means of ion exchange followed by flame-photometric determination of strontium. The method was later applied to fish bone, fish meat, milk, shell-fish, samples of human bone and water samples with strongly varying salt content.

2. Examination of method

2.1. Apparatus and reagents

The investigation was made with a Beckman Du spectro-photometer equipped with photomultiplier (IP28), flame attachment and Spectral Energy Recording Attachment (SERA). For the excitation a hydrogen-oxygen or acetylene-oxygen flame was used.

Reagents used in the investigation were of p. a. quality, except Ca and Mg solutions, which were prepared from "specpure" salts*.

2.2. Determination of strontium in the presence of calcium

The analytical work when determining strontium in water and biological material is considerably simplified if the determination can be performed without prior separation of calcium. In these samples the Ca:Sr ratio may be as great as 5000:1, which makes it difficult to separate these closely related elements completely. The experimental tests were therefore directed towards determining strontium in the presence of quite large quantities of calcium. For this purpose a number of test solutions containing strontium in the concentration range 0.5 - 5 $\mu\text{g}/\text{ml}$ and with additions of calcium between 1000 - 2000 $\mu\text{g}/\text{ml}$ were prepared. Strontium was determined by automatic recording of the 4580 - 4625 Å range of wave-

* Johnson, Matthey & Co.

lengths, after which the intensity of the Sr emission in the 4607 A line was estimated according to figure 2a.

The tests showed that strontium in this concentration range in the presence of the Ca buffer could be measured with high precision and, moreover, that the intensity of Sr emission was increased by the buffer by about 15 %. Using this measuring technique there is no influence from the calcium (background radiation), and the net emission from strontium in the 4607 A line is constant in the presence of 200 - 5000 μg Ca/ml (7).

It was also shown that the influence of a number of other elements decreased greatly on addition of the Ca buffer (table 3).

2.3. Influence of organic admixtures on the intensity of Sr emission

In order to increase the sensitivity of flame photometry, organic solutions have recently been used as solvents in place of water. For a large number of elements the sensitivity of the determination has thereby been improved, sometimes by a factor of up to 100. Additions of organic solutions to samples in water solution have also given good results (9), (10).

So far as is known, however, there has been no report of any method for strontium which makes use of this effect.

In order to find a suitable solvent, experiments were performed with additions of acetone, acetone + amyl alcohol, propanol and n-butanol to samples in water solution. The results of these experiments are presented in table 1.

As appears from the table an increase in the intensity of Sr emission was obtained for all the additions investigated, n-butanol being most effective. The subsequent investigation was therefore carried out only with addition of n-butanol.

Solvent	Intensity of emission from 1 μg Sr/ml % T
Water	25
20 vol. % acetone - 80 vol. % water	70
" " " acetone + amyl alcohol (4:1) - 80 vol. % water	78
10 vol. % propanol - 90 vol. % water	71
6 vol. % n-butanol - 94 vol. % water	87

Table 1. The influence of different solvents on the intensity of emission from strontium.

Figure 1 shows the influence of the n-butanol on the intensity of Sr emission both for $\text{C}_2\text{H}_2\text{-O}_2$ flame and $\text{H}_2\text{-O}_2$ flame. As is seen, the increase in intensity is greatest for the acetylene oxygen gas flame. With a $\text{H}_2\text{-O}_2$ flame the absolute sensitivity of the determination is almost the same, however, since the lower background of the $\text{H}_2\text{-O}_2$ flame in the region of the Sr line permits a higher amplification than during measurement with the acetylene oxygen gas flame.

μg Sr/ml*	Gas	Standard deviation %	No. of measurements
0.1	$\text{H}_2\text{-O}_2$	3.1	10
0.2	$\text{H}_2\text{-O}_2$	2.6	"
0.6	$\text{C}_2\text{H}_2\text{-O}_2$	1.4	"
1.0	$\text{C}_2\text{H}_2\text{-O}_2$	0.9	"

Table 2. Standard deviations determined for different Sr concentrations in the presence of 1 mg Ca/ml.

* 6 vol. % n-butanol water solution.

2.4. Elimination of interference from other elements

Sodium and potassium usually occur in water and biological material in such quantities that the flame emits continuous Na and K radiation and the ionization state for strontium is affected. The change in ionization makes it necessary to measure the Sr emission against references having about the same Na and K contents as the analytical sample.

A simple way of removing alkali metals from the sample is to trap the cations in a cation exchanger (Dowex 50W x 8), after which the alkali metals are removed with 0.6 N HCl. Strontium is then removed with 3.5 N HCl. By removing interfering phosphate and sulphate ions from the sample at the same time, the analytical work is considerably simplified and, if ion exchange columns are used as in fig. 4, the procedure needs no special supervision.

The influence of other elements on the intensity of Sr emission both in the presence of Ca buffer and without Ca buffer in the test material is seen in table 3.

3. Procedure

3.1. Reagents

SrCl₂ solution, 20 µg Sr/ml

CaCl₂ -" - , 20 mg Ca/ml (specpure CaCO₃)

HCl p. a., NH₄OH p. a., n-butanol p. a.

Deionised H₂O, distilled twice.

Ion exchange resin - Dowex 50 W x 8/H⁺, 50 - 100 mesh/

Ion exchange column - The column is prepared (ion exchanger layer 10 x 160 mm) and washed with water (pH 5 - 6).

Addition $\mu\text{g/ml}$	Recovery ($0.8 \mu\text{g Sr/ml}$), %			
	without Ca buffer		with Ca buffer, $1000 \mu\text{g/ml}$	
	hydrogen gas oxygen gas	acetylene oxygen gas	hydrogene gas oxygen gas	acetylene oxygene gas
5 aluminum	38	46	99	100
10 aluminum	30	36	98	100
10 barium	100		100	100
10 iron	83		100	100
100 iron	64		100	100
300 iron			100	100
10 calcium	105		100	100
400 calcium	115		100	100
20 potassium			101	102
80 potassium	110		105	105
300 potassium			116	
10 cobalt	95		101	
10 copper	83		95	100
10 chromium	91		98	99
20 lithium	100		100	100
10 manganese	87		100	
50 magnesium			100	
100 magnesium	101		100	
300 magnesium			100	
20 sodium			100	100
100 sodium			104	104
10 zinc	96		100	

Table 3. Effect of other cations on the Sr recovery.

3.2. Sample preparation

3.2.1 Water sample

A suitable quantity of sample (lake water 500 ml, sea water 25 ml) is measured into a beaker.

3.2.2 Biological material

The material is dried and then burnt in an oven at 500 °C for 8 hours. Part of the ash (bone ash 100 mg, meat ash 300 mg) is weighted into a 50 ml beaker, the sample is damped with water, and 5 ml 6 N HCl is added. The sample is heated to boiling point and carefully boiled dry (the treatment with HCl is repeated). The sample is then dissolved in 0.5 ml 0.1 N HCl and 25 ml water.

3.3. Ion exchange

The pH-value of the prepared sample solution is adjusted to 3 - 4 and the sample is allowed to pass the ion exchange column at a rate of 2 - 3 ml/min. The resin is washed with 170 ml of 0.65 N HCl and the effluent is discarded. Then the strontium is removed by percolating 120 ml of 3.5 N HCl. The Sr fraction is evaporated to dryness and the residue dissolved in 10 ml of water.

3.4. Working solution

A suitable amount of the Sr fraction (not more than 8 ml) and 0.6 ml n-butanol are transferred to a 10 ml graduated cylinder (the working solution should contain 0.8 - 1.6 mg Ca/ml and, if necessary, specpure calcium is added). The solution is diluted with water to 10 ml volume and is shaken vigorously.

3.5. Flame spectrophotometric measurement

Strontium in the working solution is determined by direct comparison with references. The emission in the range of wavelengths

4580 - 4630 A is measured by automatic scanning and recording, and the intensity of the Sr emission at the 4607 A wavelength is calculated according to fig. 2a.

For preparation of the calibration curve (fig. 5) a series of references containing 0.1 - 0.8 μg Sr/ml, 1.2 mg Ca/ml and 6 vol. % n-butanol is prepared.

4. Results

The method has been tested as follows:

A known quantity of strontium was added to meat, bone and water samples, previously analysed with respect to strontium, and the total Sr quantity was subsequently determined.

Comparison with results obtained by another method (11).

Strontium was determined in samples synthesized with a known Sr content.

The results in table 4 show that the method can be used for a sample material of greatly varying composition. The high sensitivity of the method permits determination of 5 μg Sr with an analytical error of less than 5 %. For Sr quantities greater than 10 μg the error is less than 3 %.

Sample	Sample quantity	Natural Sr in the sample μg	Added $\mu\text{g Sr}$	Calculated $\mu\text{g Sr}$	Found $\mu\text{g Sr}$	Deviation %
Water, Ulkesjön	1 l	36.4	6.0	42.4	43.7	+ 3.1
" Möllesjön	1 l	40.2	6.0	46.2	47.0	+ 1.7
" Tvären	25 ml	41.0	10.0	51.0	50.0	- 2.0
" Nittsjön	250 "	30.0	20.0	50.0	50.0	\pm 0
" Glisstjärn	250 "	93.8	50.0	143.8	141.0	- 1.9
Meat ash A, fish	302 mg	11.6	6.0	17.6	18.1	+ 2.8
" " B, fish	308 "	27.8	6.0	33.8	34.2	+ 1.2
Bone ash A, fish	100 mg	20.1	20.0	40.1	39.8	- 0.8
" " B, fish	141 "	29.7	20.0	49.7	49.7	\pm 0
" " C, human	215 mg	36.3	20.0	56.3	56.0	- 0.5
" " D, human	176 "	9.37	20.0	29.37	28.75	- 2.1
Bone ash, synth.	(125 $\mu\text{g Sr}$ + 140 mg Ca + 20 mg Na + 100 mg H_3PO_4)				126.5	+ 1.2
Meat ash, "	(10 $\mu\text{g Sr}$ + 5 mg Ca + 10 mg K)				10.3	+ 3.0
Water sample, "	(50 $\mu\text{g Sr}$ + 6 mg Ca + 100 mg Na + 15 mg Mg)				50.0	\pm 0
Water sample, "	(5 $\mu\text{g Sr}$ + 6 mg Ca + 10 mg Na)				5.2	+ 4.0

Determination of the precision

	Sr, determined	$\mu\text{g Sr/g ash}$	
	141 mg	29.7 μg	211
	275 "	57.5 "	209
	276 "	57.5 "	208
Bone ash B, fish	219 "	44.6 "	204
	239 "	50.0 "	209
	275 "	56.7 "	206
	276 "	59.2 "	214
			Mean: 209
	Standard deviation:		3.3

Table 4. Results of flame photometric determination of strontium in different test materials.

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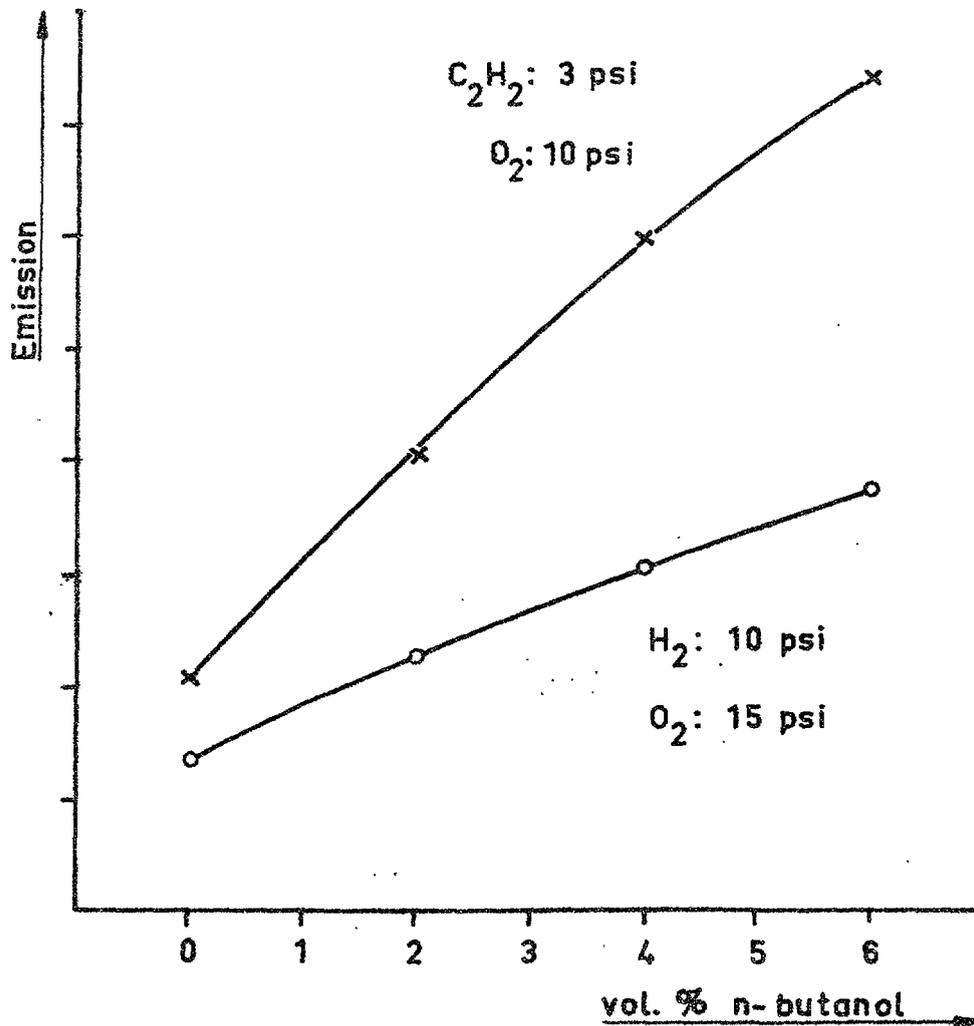


Fig. 1. Influence of n-butanol on the intensity of the emission from 1 μg Sr/ml at the wavelength 4607 A.

Buffer: 1000 μg Ca/ml

Instrument setting

Amplification; 8 (1P 28)

Slit width; 0.025 mm

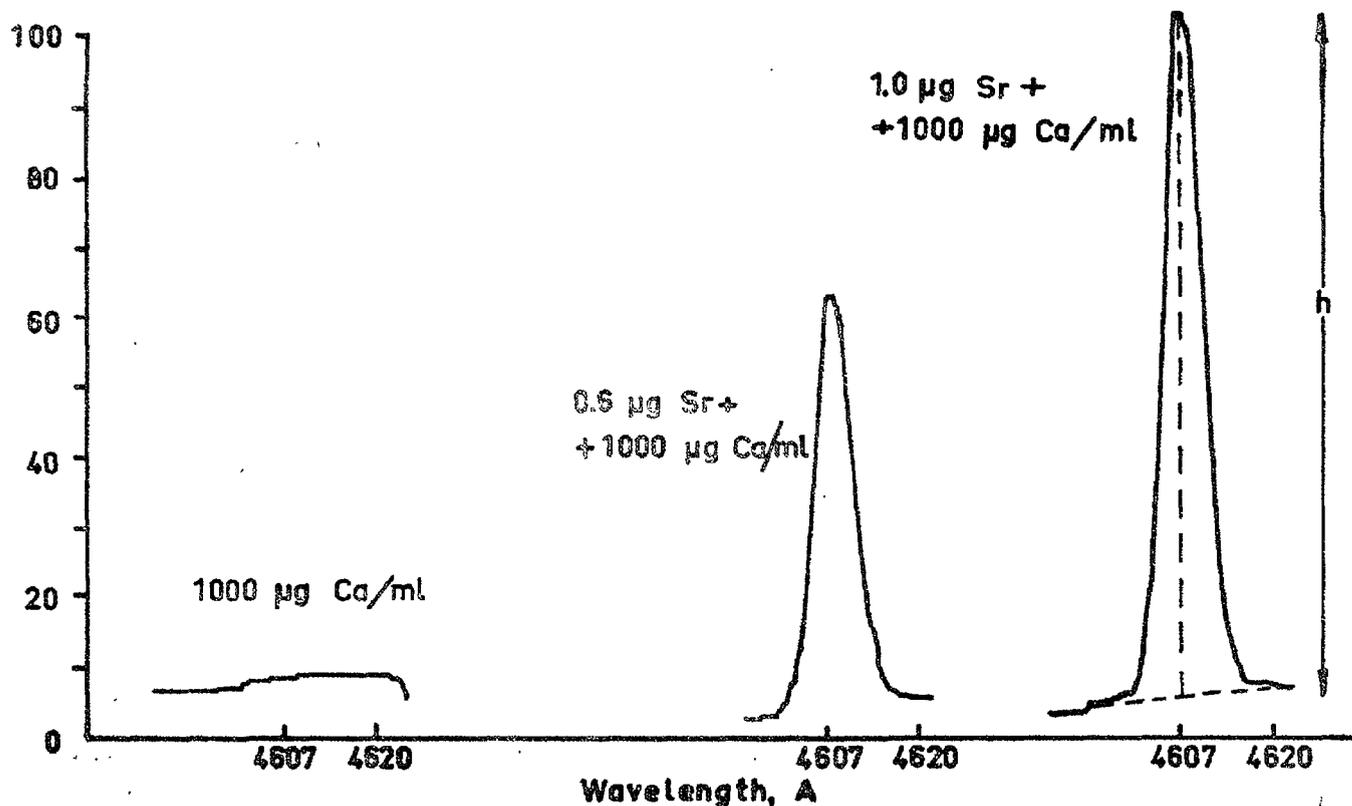


Fig. 2a. Flame emission spectra
 Solvent: 6 vol. % n-butanol-water solution
 Amplification: full (1P 28) Half intensity band width: 5 Å
 Scanning speed: 27 Å/min Chart speed: 25.5 mm/min
 Gas Hydrogen: 10 p. s. i
 Oxygen: 15 "

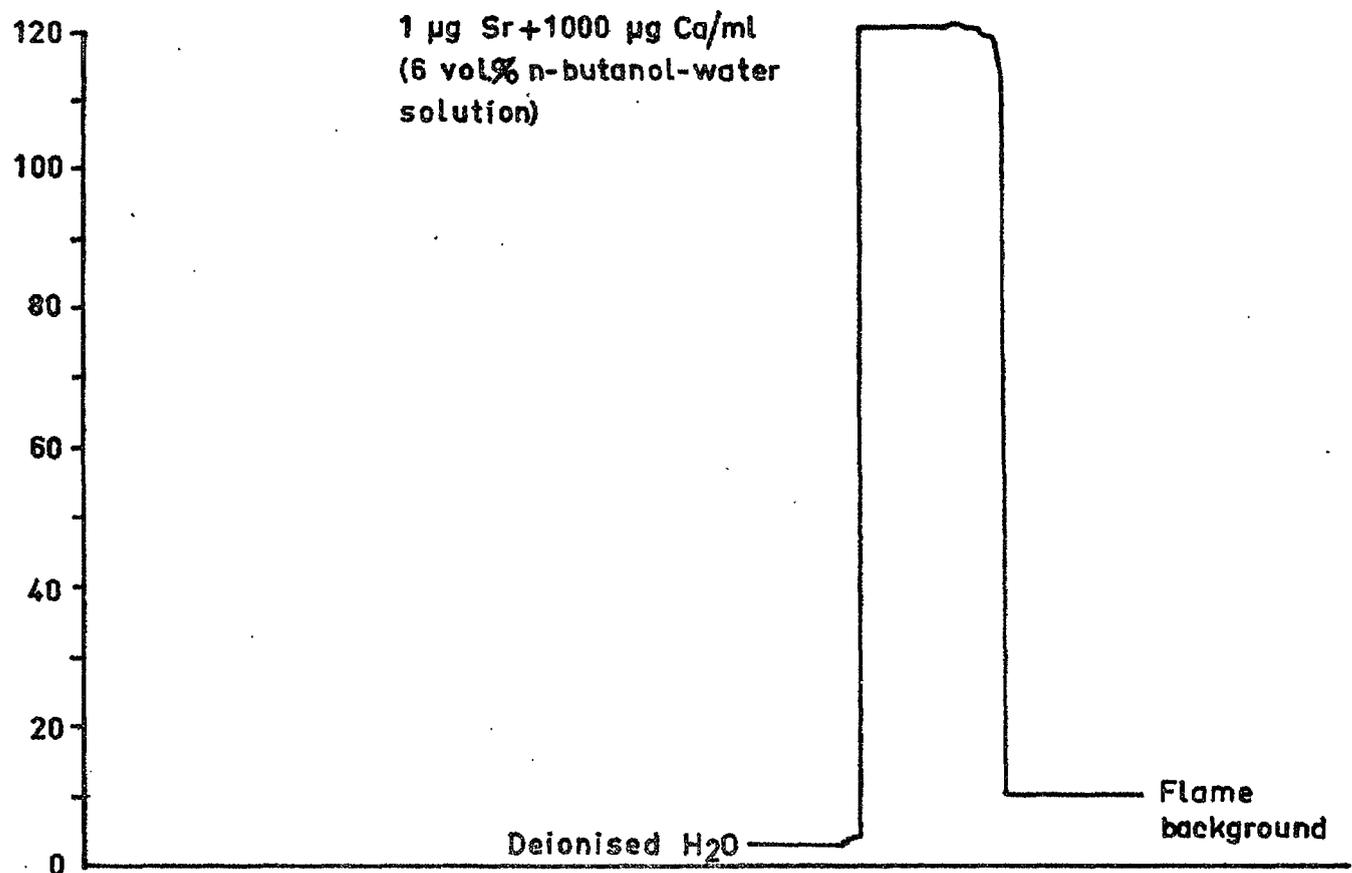


Fig. 2b. Continuous recording at the 4607 Å wavelength
 Instruments setting and gas see fig. 2a.

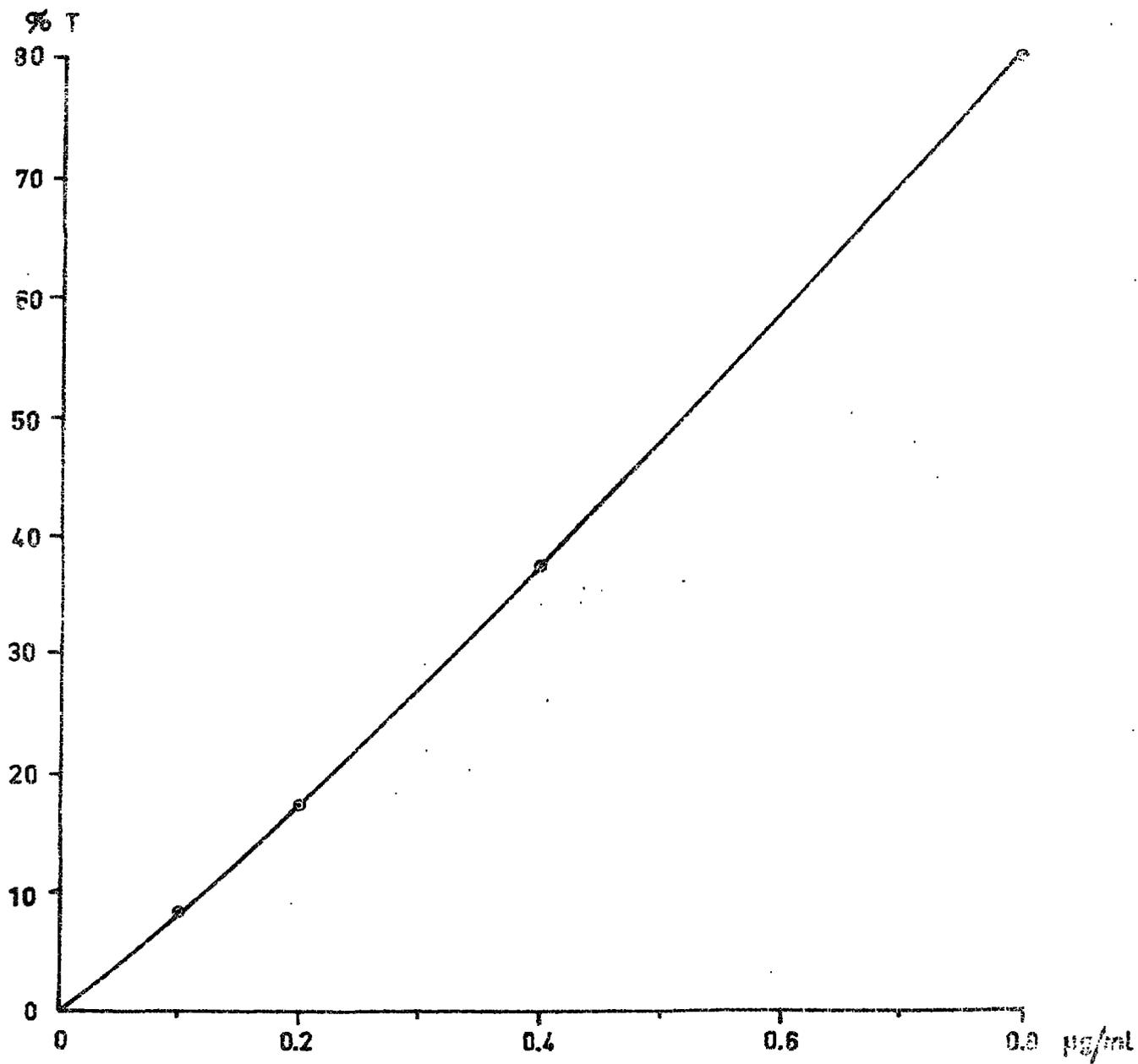


Fig. 3. Calibration curve for strontium in the concentration range

0.1 - 0.8 µg/ml

Solvent: 6 vol. % n-butanol-water solution

Wavelength: 4607 Å

Buffer: 1000 µg Ca/ml

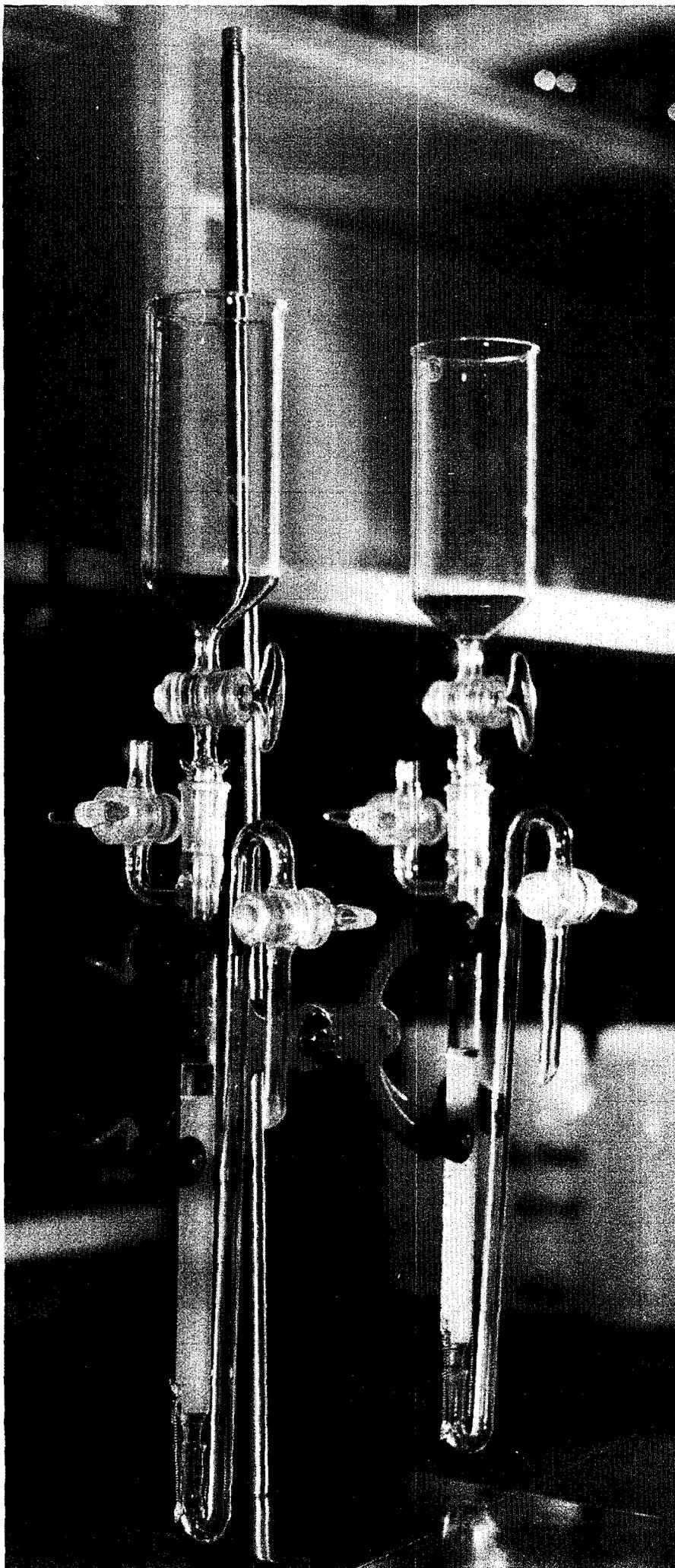


Fig. 4.

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