ELIMINATION OF THE INTERFERING EFFECTS IN THE DETERMINATION OF STRONTIUM BY FLAME EMISSION SPECTROMETRIC METHOD

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ABSTRACT

In the flame emission spectrometric determination of strontium. the interfering effects of normal aliphatic monocarboxylic and dicarboxylic acids have been studied.

The role of a combination of $MgCl_2$ and NH_4Cl in eliminating aluminium and other inorganic species is also illustrated. Moreover, estimation of strontium in synthetic mixtures was elaborated.

INTRODUCTION

Determination of strontium by a flame spectrophotometric method have been previously described.[1,2,4,11,12]

Elimination of chemical interferences has been reported in a number of papers.[2,3,7,15] Abdallah and Mostafa have explained the mechanism of lanthanum and boric acid combination.

Some authers[3,7,13] have postulated that a high concentration of stronium would release the flame emission of calcium from the inhibition effects of aluminium, and that conversely, a high concentration of calcium would release the flame emission of strontium.

Investigations of the effect of aliphatic acids in determination methods by flame emission spectrometry have been studied extensively.[8,10,13]

The problem has usually been approached in one of two ways; the effect has been studied either at low acid concentrations and called an anion interference[6,14] or at high concentrations and described as changes in flame temperature and the physical properties of the solution.

The purpose of this work was to investigate the effects of aliphatic acids with moderate concentrations to determine, the role of surface tension. Also, the mechanism of a combination of $MgCl_2$ and NH_4Cl in eliminating interference is a new and sensitive releasing agent for the determination of Sr.

EXPERIMENTAL

A Unicam SP 90 Series 2 Atomic Absorption Spectrophotometer was used. Air was supplied through a Unicam SP 93 air compressor. The emission mode was used throughout.

The air flow rate was 5 dm^3 min^{-1} and acetylene was delivered from cylinders at a flow rate of 1 dm^3 min^{-1}, after passing through conc. H_2SO_4 and glass wool for purification.

The optimum instumental parameters were; burner height, 3cm; slitwidth, 0.1 mm ; wavelength 460.7 nm.

All aliphatic acid solutions were prepared from reagent grade acids. Stock solutions of the metals were prepared as chlorides and nitrates Aliquots were suitably diluted to give the desired metal ion concentrations used for the experiments. Direct spectral interferences and flame background changes were eliminated by line scanning and background check on all stock solutions.

RESULTS AND DISCUSSION

Effect of aliphatic acids:

Fig 1. shows the effect of 0.1M solution of monocarboxlic aliphatic acids on the emission intensity of the SrOH band signal. Curve 6 in Fig 1. represents the emission intensity of neutral solution of strontium nitrate. Enhancements of the band emission signal by the use of 0.1M aliphatic monocarboxylic acids are indicated in Fig. 1 (curves 1–5). Figure 2 also shows the effect of increasing amounts of C_{1-5} normal aliphatic acids on the flame emission of Sr $(NO_3)_2$.

The enhancement increases with chain length from formic up to valeric acid and with increasing acid concentration. Figure 3 also shows the effect of some aliphatic dicarboxylic acids where enhancements in SrOH band emission increase from oxalic acid to adipic acid. As described by West,[16] the change in surface tension for monocarboxylic acids is of the following order: valeric < butyric < propionic < acetic <
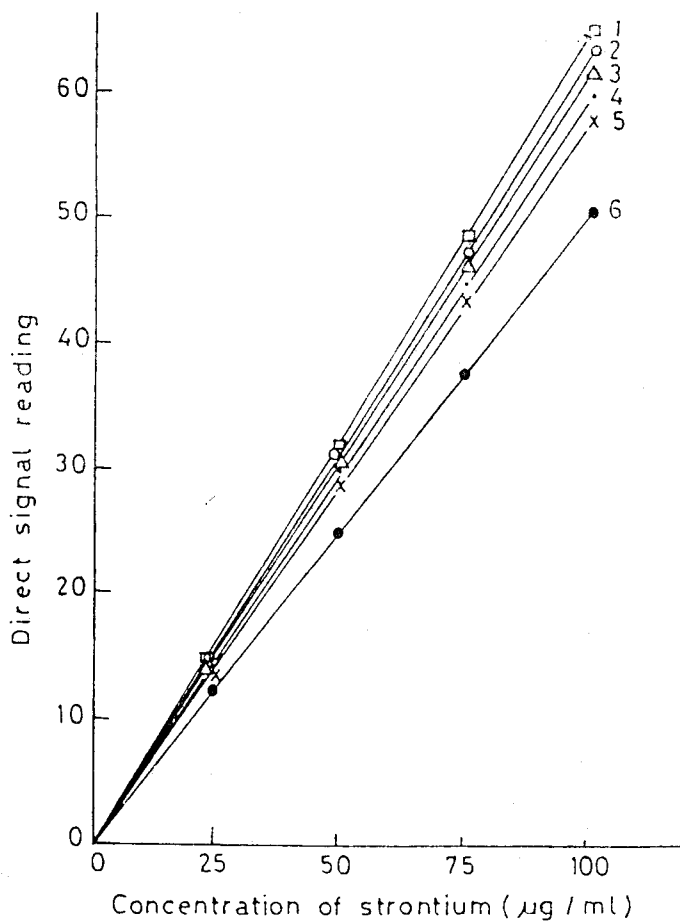
Fig. 1. Influence of 0.1 M solution of different monocarboxylic acid on the emission intensity of SrOH. 1) Valeric acid, 2) butyric acid, 3) Propionic acid, 4) Acetic acid, 5) formic acid and 6) Neutral.

< formic and for dicarboxylic acids the deceasses in surface tension is high as the chain length increased. Since the viscosity and density changes in the solution involved in the present work were small (low acid concentrations), the surface tension may be considered as the principal factor affecting droplet surface area. A decrease in surface tension will increase the efficiency of solvent evaporation, the concentration of metal atoms in the flame, and the emission intensity,
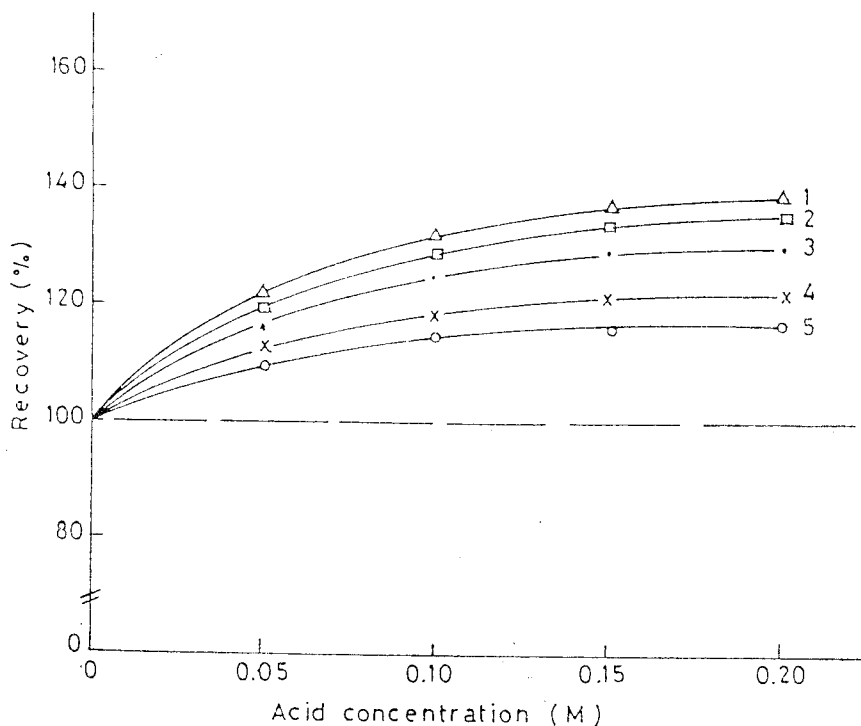
These agree well with data in figs. 1, 2 and 3.

Fig. 2. Effect of various monocarboxylic acid concentrations on SrOH emission from 50 µg Sr /ml.
1) valeric acid, 2) butyric acid, 3) propionic acid, 4) acetic acid and, 5) formic acid.

Elimination of interferences:

Fig. 4 shows the effects of varying concentrations of $MgCl_2$ plus NH_4Cl, $MgCl_2$, Zn, Li, Ni, Fe and Mn on the release of strontium emission from aluminate depression.

In the absence of the releasers, the recovery (%) of strontium emission which is caused by aluminium is 8 %. The main problem for aluminium is the formation of mixed spinels (SrO. Al_2O_3) of high melting point[9]. The improvement in SrOH signal in solution containing Al plus Zn, Li, Ni, Fe and Mn in fig. 4 (curves 3–7) can be attributed to partial replacement of Sr ion by such ions in the Sr –O– Al lattice.

Since the atomic radii* of all these ions are relatively small than that of strontium, the releasing action of $MgCl_2$ can be described by means of the following equation:

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*Atomic radii are 1.35, 1.53, 1.62, 1.72, 1.79 and 2.45 A° for Zn, li, Ni, Fe, Mn and Sr respectively.
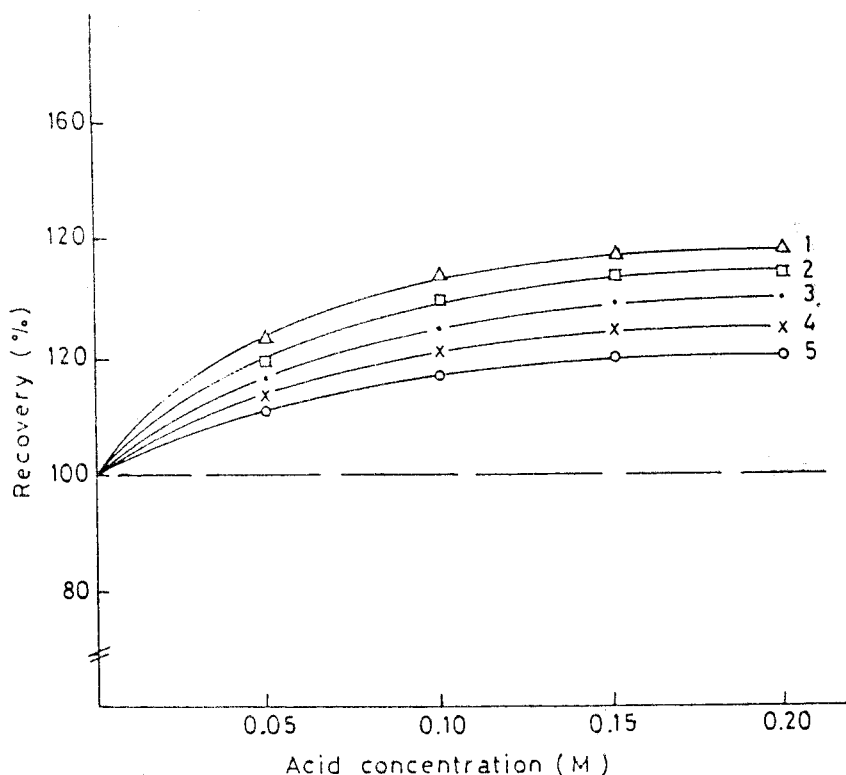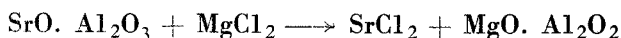
Fig. 3. Effect of various dicarboxylic acid concentrations on SrOH emission form 50μg Sr/ml.
1) Adipic acid, 2) Gluateric, 3) Succinic, 4) Malonic and 5) oxalic acid.

$$SrO. \ Al_2O_3 + MgCl_2 \longrightarrow SrCl_2 + MgO. \ Al_2O_2$$

Thus leaving Sr to form the more readily dissociated compound ($SrCl_2$). NH_4Cl is used as a flame buffer, thus protect the formation of SrOH. The effectiveness of $MgCl_2$ and NH_4Cl in releasing stronium from aluminium interference can be successfully explained in terms of the displacement of the chemical equilibrium in the above equation towards $SrCl_2$.

The interfering effects of cation and organic acid mixtures in the presence of 1mg/ml for both $MgCl_2$ and NH_4Cl are summarized in Table 1. The interfering effects of all studied species are completely eliminated. The accuracy of the method is shown by the absolute and

relative errors (Table 2). Precision is expressed by the standard and relative standard deviations. It is apparent that the results agree well mutually, and the method is advantageous from the view point of both precision and accuracy.
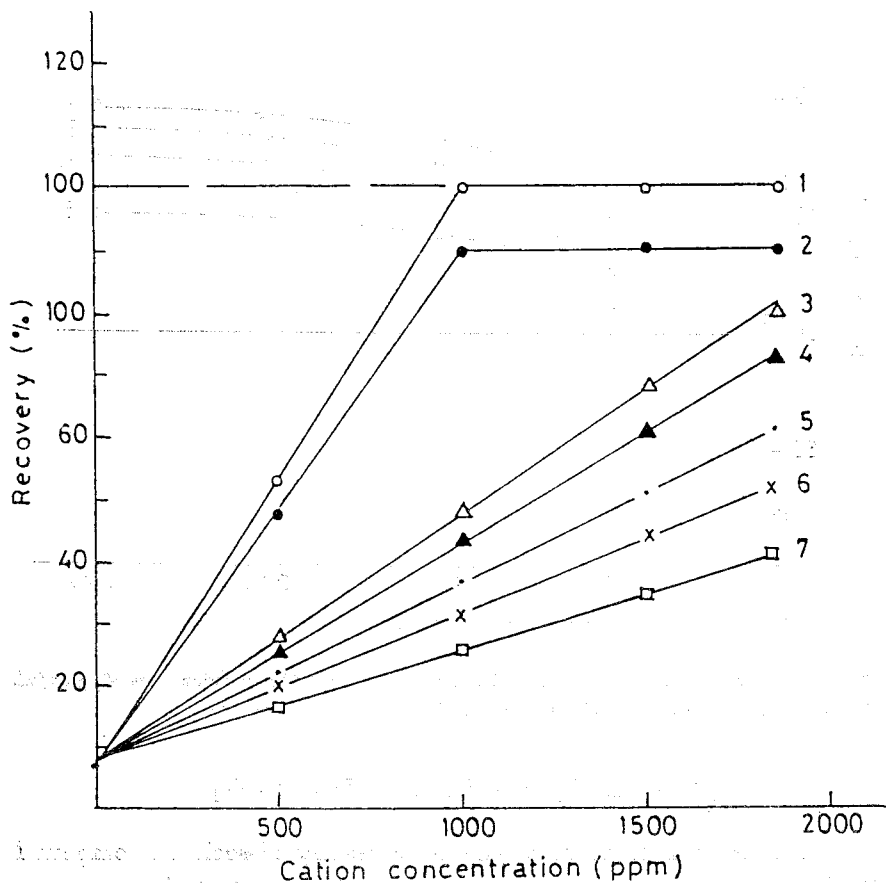

Fig. 4. Use of some cations in eliminating of 100 μg / ml Al on the emission of 50 μg / ml Sr at 460.7 nm,
1) MgCl₂ – NH₂Cl, 2) MgCl₂, 3) Zn 4) Li, 5) Ni, 6) Fe and, 7) Mn.

Table 1. Interferences of various cation and organic compounds on 50ppm Sr emission without and with addition of 1mg / ml $MgCl_2$ + 1mg / ml NH_4Cl.

No Added species 100; /ppm	Recovered percentage of Sr	
	without adding $MgCl_2$ + NH_2Cl	with adding $MgCl_2$ + N_2HCl
1 Mn + In	40	100
2 Mn + Tl	90	98
3 Mn + Cr	118	98
4 Mn + Cu	54	98
5 Al + Zn	28	100
6 Al + In	32	100
7 Al + Tl	34	100
8 Cr + Fe	80	102
9 Cr + Ni	75	102
10 acetic + valeric	118	102
11 oxalic + malonic	120	98
12 oxalic + succinic	134	100
13 oxalic + maleic	130	100
14 oxalic + gluateric	120	100
15 oxalic + adipic	118	98
16 malonic + succinic	130	98
17 malonic + maleic	115	100
18 malonic + gluateric	122	98

Table 2. Comparative results for analysis of Sr in synthetic solutions using 1mg / ml $MgCl_2$ + 1mg / ml NH_4 Cl.

No	Added Cations	Sr (PPM)			Absolute error	Relative error	Standard** deviation —	Relative Standard deviation %
		Added	Found* without treatment	Found with treatment				
1	50Sr+100Al+100Mn+100Ni	50	37	50	0	0	0.25	0.50
2	100Sr+100Al+100Ba+100Fe	100	68	100	0	0	0.33	0.33
3	150Sr+100Al+100Mn+100Ba	150	115	148	-2	-1.35	0.36	0.24
4	200Sr+150Al+100Ni+100Co	200	148	198	-2	-1.01	0.46	0.23

These results are taken from direct aspiration of synthetic mixtures without any treatment and recoreded here for the sake of comparison.

The number of analyses involved in the determination of standard deviation is 20.

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