APPLICATION NOTE

Food Safety

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The Analysis of Common Salt by ICP Optical Emission Spectrometry

Introduction

The analysis of sodium chloride, or common cooking salt is one of the most difficult and demanding analyses. This is mainly because of the high sodium content in the dissolved sample, and the high degree of ionization of this element when it is introduced into a flame or plasma. Furthermore, the analytical requirements with respect to this sample are very tough, mainly because very high purity salt is desirable for many industrial processes, the most important of these being the production of chlorine and all related products. As saturated concentration of 360g/L, no more NaCl will dissolve and the solution is on the point of crystallizing out. This physical property has many consequences in the ICP since solutions that are on the point of saturation tend to be dried at two different points in the sample introduction system and crystals can easily be formed. These two points are at the nebulizer tips as the solution is aspirated and at the injector tip. Just before entering the plasma the aerosol must travel along the injector and through the final orifice before reaching the plasma. This injector is close to the plasma and will often be hot. Any heating of the aerosol at this point is dangerous because the water droplets may be vaporized and dry crystals can lodge at the injector orifice. These crystals quickly grow to form a “tree“ which blocks the tip totally.

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There are several approaches which can reduce the chances of blocking some part of the sample introduction system, these are; diluting the solutions so that they are not close to the saturation point, use of an argon humidifier to wet the argon and prevent drying at the nebulizer tips, and the use of a quartz injector or other poorly conducting material, placed as far as possible from the base of the plasma, to prevent the injector becoming too hot. In this work the salt solutions were diluted to 125g/L or 12.5% NaCl which is somewhat less concentrated than the usual 200-250 g/L used by the industry.

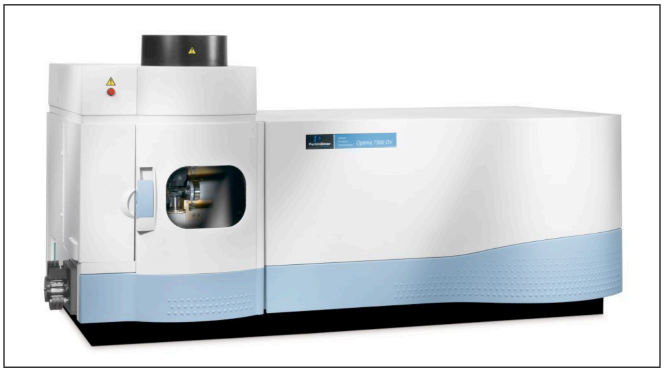

Fig. 1 Optima

However, axial observation of the plasma has been used throughout the work. This gives up to a factor 10 improvement in the sensitivity and detection limits of the plasma system compared to the traditional radial system. A new design of Ar humidifier has been used in this work. It is a simple device constructed of glass which is half filled with deionized water. The argon is passed through the humidifier and bubbled through water just prior to the nebulizer. A glass frit ensures small bubbles of argon so that the maximum amount of gas comes into contact with the water.

A graduated quartz injector tube with a 2mm I.D. at the tip has been used. This does not get hot during analysis and reduces the risk of salt crystals blocking the injector.

Plasma Operation

The plasma operating conditions were optimized for the best signal to background ratio for a 1mg/L Mn solution and were as follows:

Plasma gas:	15-20 L/min
Auxilliary gas:	1 L/min
Nebulizer gas:	1.1 L/min
Sample uptake rate:	1 ml/min

Results

The ICP was calibrated with matrix matched solutions of 12.5% ultrapure sodium chloride to which multi-element standards had been added. An internal standard of 1mg/L Sc was added to all blanks, standards and samples.

Axial Detection Limits in Salt Solution (3XSD of 10 repeat measurements of a blank salt solution).

Element, nm	DL ug/L 12.5% NaCl	DL mg/kg in the salt	Element, nm	DL ug/L 12.5% NaCl	DL mg/kg in the salt
Ag328	0.3	0.003	Ni232	1.8	0.015
Al396	0.6	<0.005	P213	21	0.18
As188	15	0.12	Pb220	12	0.095
Ba455	<0.1	0.0006	Pt265	5	0.04
Ca422	<0.1	0.0009	S180	10	0.08
Cd228	0.9	0.006	Sb206	10	0.08
Co238	1.5	0.012	Si251	1.5	0.012
Cr267	1.8	0.013	Sr407	<0.1	0.0003
Cu327	0.9	0.007	Ti334	0.3	0.003
Fe238	0.9	0.006	V272	0.9	0.007
Mg279	<0.1	0.0003	Y371	0.3	0.0012
Mn257	<0.1	0.0012	Zn213	1.2	0.01
Mo204	2.7	0.02			

Summary

Detection limits relative to the sample of solid salt have been improved by the use of more dilute solutions coupled with axial viewing of the plasma.

The use of salt solutions at 125g/L allows the solutions to be analyzed unattended without any danger of blocking the sample introduction system while at the same time giving very good detection limits relative to the solid sample.

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