Using ICP scan analysis as a powerful diagnostic and troubleshooting tool in analytical laboratories

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Introduction

Contemporary market requirements pose an ever-increasing demand on testing laboratories to cope with more and more non-standard investigations. These may relate to a wide spectrum of materials and matrices, ranging from simple-matrix aqueous samples (e.g. surface, ground & drinking waters, wastewaters) to very complex matrices of different samples sizes and textures like solid deposits, semi-solid residues, building materials, soils, sediments, sludge & biological samples etc.
Modern laboratory instrumentation has been providing laboratories with cutting-edge technology to help them engage efficiently and effectively in forensic-type investigations, which in small countries, like Cyprus, form a substantial part of our workload, not relating to routine and standardized laboratory activities.
In the context of our investigation, ICP scanning provides a qualitative analysis covering the entire spectrum of metals, metalloids, alkalies & alkaline earth elements of the periodic table (over 70 elements), in a fraction of the time required by other techniques. As such, it can be applied in problem-solving enquiries as an extremely efficient diagnostic tool, either on its own or complementing other analytical techniques.
However, deciding whether to select ICP scanning over other analytical techniques or in combination with them, selecting appropriate sample preparation techniques, and most importantly interpreting the results in an appropriate manner that will be of real value to the customer, requires extensive analytical experience, excellent customer feedback, solid scientific background and the ability to handle and present data in a statistically sound and scientifically justified manner. Therefore, the assessment of the overall quality and integrity of the produced data, forms an integral part and indispensable requirement of adopting this analytical technique, including the ability to decide whether to reject outliers and provide additional instrumental justification/verification.

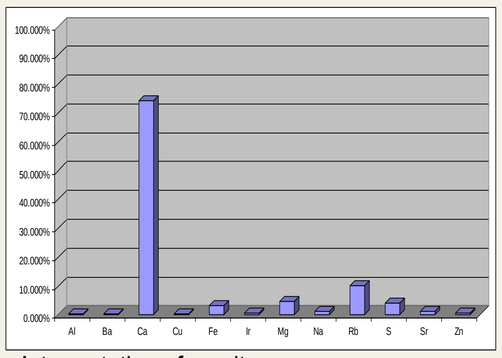
Methodology

In the context of this poster presentation a series of specific case studies relating to the chain of production, distribution and use of drinking water will be presented. Also, some additional independent studies will be included.
In all of above examples, solid, semi-solid and liquid samples have been dried & homogenised (where applicable) and treated with three alternative techniques. Mild nitric acid digestion, aqua-regia digestion and microwave-assisted aqua-regia digestion have been employed to disperse and fully digest the samples before measuring qualitatively on a vacuum-type ICP OES spectrometer. A Shimadzu multi-type emission spectrometer equipped with qualitative database calibration has been used to carry out a high precision qualitative analysis. This renders a reliable qualitative result while at the same time eliminates the need for time-consuming comparative examination with the standard sample.

Case Study #1

Brief description
Following complaints to a local water authority that a village was experiencing a gradually decreasing water supply, an on-site investigation was carried out and it was found that the reason behind this was a series of blockages in the distribution network.
Certain pipe-lengths were isolated and we were called in to provide analytical services with the aim to investigate the source of the problem.
A typical example of a blocked pipe section is shown below as well as the subsequent results of the ICP scan analysis on the residue that caused the blockage.

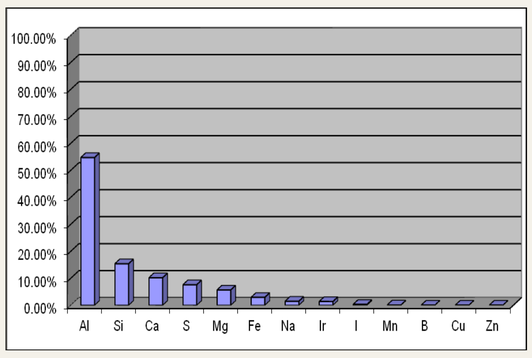


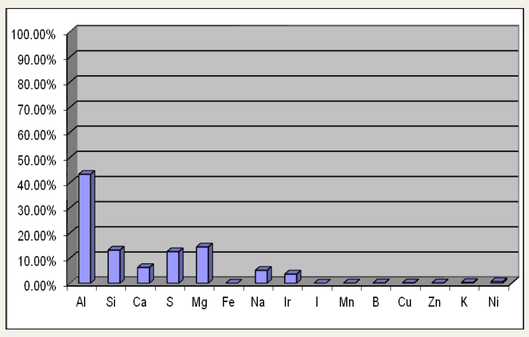



Interpretation of results
Excessive scale deposition is confirmed to be the cause of the blockage, possibly as carryovers from larger diameter pipes which were gradually concentrating in the restricted flow area. ICP scan verifies that the origin of the depositions is primarily limestone (CaCO3) and also sulphate-based minerals to a smaller extent. There is also a clear indication of corrosion taking place within the distribution network (presence of iron and zinc) and of the alum-based flocculants used in the water refinery process.

Case Study #2

Brief description
Potable water supplies on the island are mainly dependent on rain water collected in reservoirs, with contribution from seawater desalination plants during the last two decades.
This investigation relates to sludge samples received from two separate water refinery plants in the course of optimising the dosing of flocculants, following elevated aluminium findings in mains water supplies (150 – 250 ppb against the regulated limit of 200ppb).

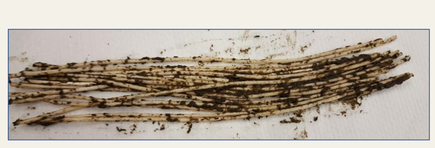


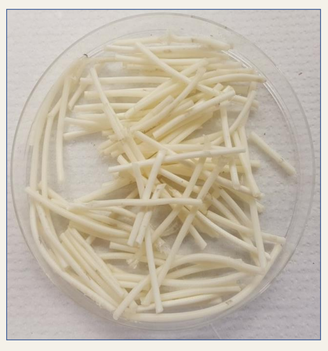
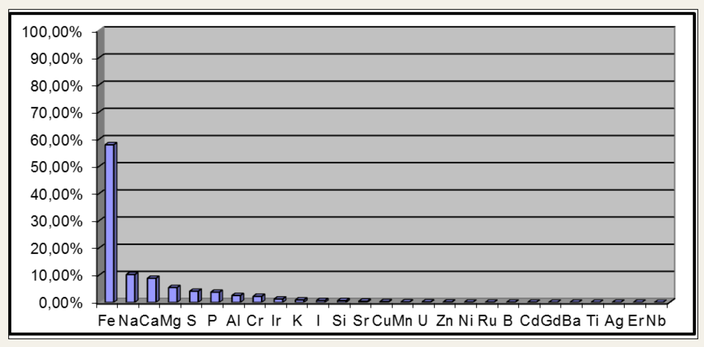


Interpretation of results
It is evident that both refineries had been using the flocculant (alum sulphate at the time) in excess, with no optimisation on the dosing. As a result of this study, an alternative chemical was introduced (PAC). This proved to be equally effective, with lower aluminium content.

Case Study #3

Brief description
The tertiary treatment process of a Sewage Treatment Plant (STP) that employs the membrane technique for bacterial removal had been experiencing problems of reduced throughput which coincided with the need for more frequent cleaning of the membrane modules.
In the process of investigation, hollow-fibre membrane sections were delivered to the lab for analysis, as shown below (before & after sample preparation).

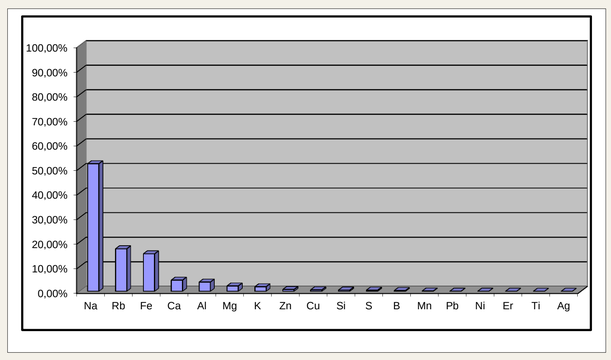


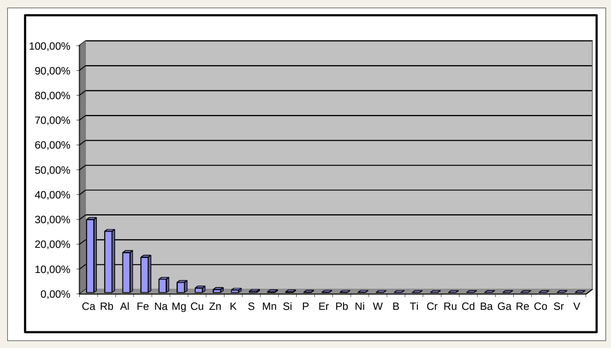




Interpretation of results
The quality of the mains supply in the district where this STP operates was raising concerns that this might be caused by excessive scale deposition on the membranes. The ICP scan had actually revealed another source of concern which seemed to be linked with the ferrous chloride used as a flocculant/coacculant in the primary treatment section of the station.

Case Study #4

Brief description
A seaside holiday residence was experiencing problems with highly coloured water in the mains supply. One water sample from the mains supply (roof storage tank) and one salts residue sample from the surface of the paved area cleaned with the mains water, were analyzed with results as shown.

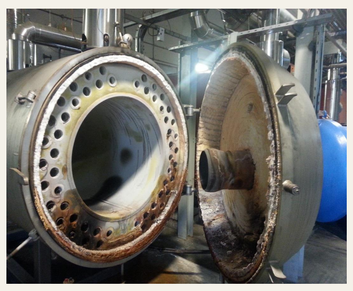


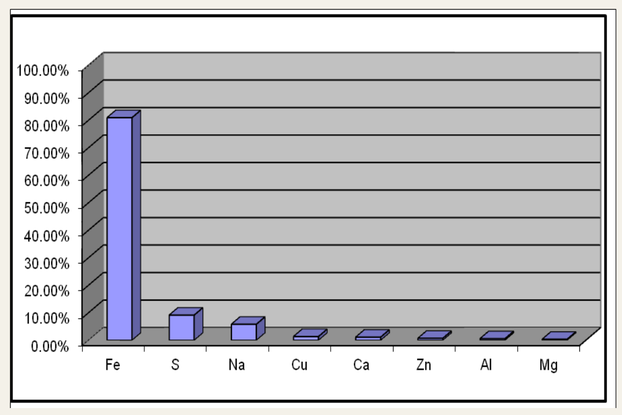


Interpretation of results
The mains supply water indicates a SWRO plant product passing though a water refinery and exhibits a corrosive tendency, as indicated by the presence of iron. The salts residue composition shows the same origin, after weathering due to successive evaporation /concentration cycles.

Case Study #5

Brief description
A firetube boiler failure at a large commercial complex comprising of offices and shops, was investigated. Salt deposits from the inner surface of the boiler near the areas of failure were examined in order to ascertain the real cause of the problem.





Interpretation of results
As expected, the predominant ion present was iron and also copper at lower levels, signifying excessive corrosion within the boiler. However, an additional indication was provided by the presence of sulfur which was suspected to be linked to the HFO used as a fuel. This might be a significant contributor to the corrosion observed as also verified by subsequent sulfur determination on the fuel which rendered sulfur results ranging between 2,5 – 3%.

Bibliography

1. Laboratory analytical records
2. SHIMADZU Multitype plasma spectrometer ICPE-9000 Instrument specifications