Total Sulfur

With ion chromatography

And a unique sample digestion apparatus

A new and powerful analytical technique combining the newly patented RADOX digestion method from Analytical Sciences Limited with Ion Chromatography technology – now being successfully used in the nuclear power generation industry to measure Total Sulfur in ultrapure water where the UPW beds employ sulfonated co-polymers of styrene and DVB and the condition of the beds needs critical monitoring

the Radox® TS

Sulfonates and UPW

A fast, automated and accurate method for measuring Total Sulfur (TS) in ultra pure water (UPW) has long been a goal of analysts in this field. Cation exchange resin beds used to produce UPW are generally constructed from sulfonated materials - generally sulfonated styrene or divinyl-benzene (DVB). These materials will eventually leach into the UPW product and because they have a multitude of molecular weights are not susceptible to analysis using conventional HPLC or elemental assay. An obvious solution is to digest the samples converting the sulfur present to SO_4 and then use Ion Chromatography to obtain a quantitative analysis.

Until now, the digestion has been very time consuming with little confidence that the digestion is complete.

Efficient Conversion to Sulfate

Tests show that the new Radox® digestor quickly and efficiently mineralizes all organic sulfonated material as well as other organic species; the digestion system is as efficient with the more difficult to oxidize organics, such as acetic acid.

Ion Chromatography

By combining an Ion Chromatograph (IC) with the novel patented digestion technique developed in Cambridge by Analytical Sciences, laboratories worldwide now have a powerful new tool - the Radox® digestor. The Radox® digestor can be operated stand-alone, or it may be combined with an Ion Chromatograph to measure Total Sulfur and other important analytes such as Total Phosphorus.

Field tested at Sizewell "B"

The Radox® digestor has now been successfully tested and approved and is now being used routinely by Sizewell "B" nuclear power station, monitoring their secondary cooling water for sulfonate leaching. Sizewell "B", located in Suffolk, England is a PWR reactor.

Fig.1
Flow Schematic

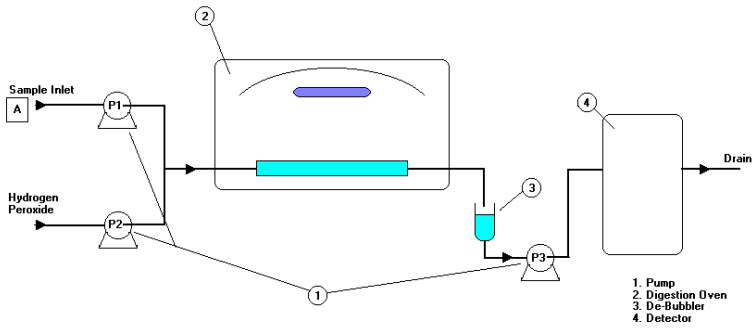
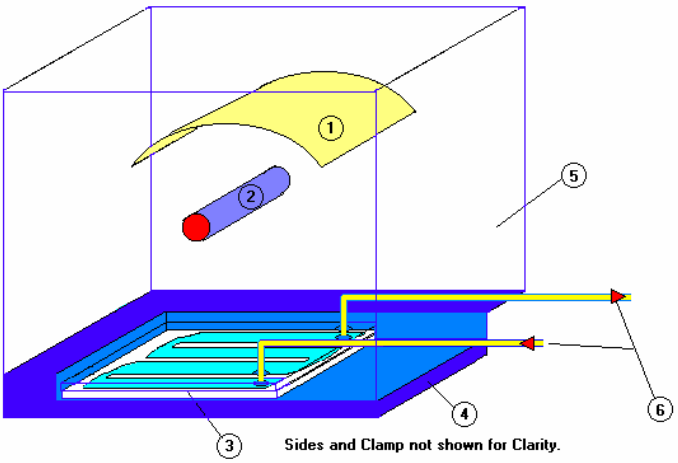


Fig.2
Digestion Oven



1. Dichroic Reflector
2. Medium Pressure Mercury Vapour Lamp
3. Digestion Module
4. Cold Sink Block
5. Sealed Enclosure, cooled
6. Sample Inet & Outlet

The Radox®

The Radox® works by using intense electromagnetic radiation across a broad spectrum including UV and infrared. The sample is passed through a heat-controlled conduit where it is subject to both thermal and UV oxidation. The radiation source is a medium pressure mercury vapor lamp having an output of at least 600 watts.

Radiation Source

Conventional UV digestion equipment tends to use low pressure UV lamps (usually operating at less than 40 watts) which produce ultraviolet radiation mainly in the region of 253.7nM. It has been found that, by exposing the sample to a broader range of radiation frequencies (and higher intensity), the Radox® is able to digest a sample far more efficiently than conventional UV digestion instruments.

The radiation source used for the Radox® emits a broad spectrum from below 185nM to above 2000nM. The first band has a wave length (in free space) of less than 250nM (UVC) and the second band (UVA) includes a peak in the region of 360nM. In addition to producing ozone, hydroxyl ions and other free radicals the radiation, in combination with the heat generated, is sufficient to excite and break many of the molecular bonds present in organic material. The radiation is also sufficient to penetrate to a depth of at least 500 microns through semi-opaque and turbid liquid samples.

Sample Exposure System

The sample is passed through a sample guide (conduit) underneath the light source. This conduit is formed into a glass base plate with a quartz cover transparent to UV light. The capillary has an inner diameter of 1mm, and the track is 0.3mm deep. The shape of the conduit is serpentine in all cases.

The sample is introduced into the exposure system by a peristaltic pump and flushed through under pressure during which time it is exposed to both heat and intense UV light. Flushing between samples ensures integrity from one sample to the next.

Reflection

The Radox® has a dichroic reflector placed above the light source which reflects the upward traveling UV radiation but is substantially transparent to infrared radiation thus allowing the heat to rise through it to be drawn away via the heat-sink/fan assembly.

Digestion and Control Units
The Radox® Basic Specification

The digestion system is housed in an enclosed oven compartment made from a special heat-sink components surrounded by high quality low-noise fans for conducting heat away from the interior. This has the benefit of sealing the ozone produced within the oven and prevents UV light escaping, which could otherwise prove dangerous.

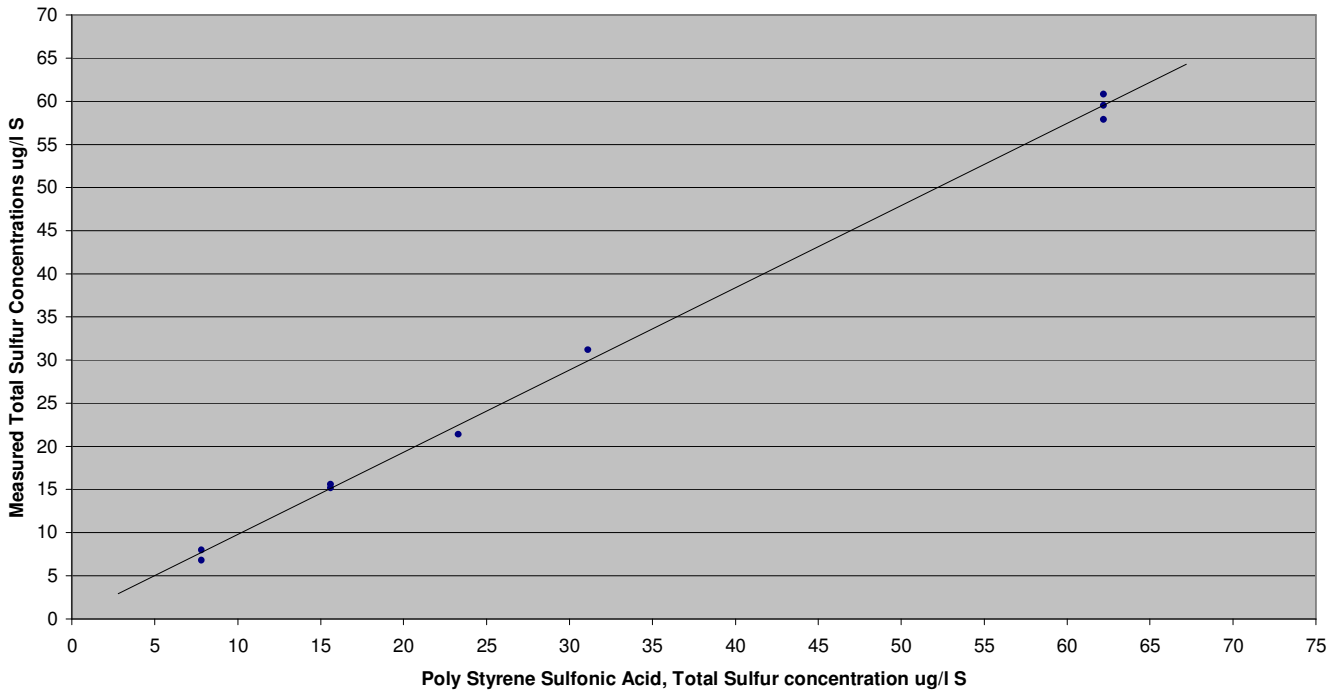
As well as the digestion assembly the Radox® has a separate small unit which houses the control circuitry, power supply, light ballast system and peristaltic pump. This whole may be used stand alone, or linked to a PC for automated control of batch runs.

Ion Chromatograph

For measurements, the samples digested by the Radox® are degassed and may be transferred by a peristaltic pumping system to an ion chromatograph (IC).

Flow Rate, digestant	Typically > 1.5ml/min
Blank	<1.0 ug/l S
Digestion efficiency	>95% for Sulfonates
Detector	Dionex® Ion Chromatograph
Injection Loop Volume	100ul
Column	Dionex® AS14
Oxidant conc	16.5mM H_2O_2
Power	240VAC, 1.0KVA

Measured Concentrations of sulfur using an IC after digestion of sulfonate samples using the RADOX



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