

Revising ASTM D5673 Standard Test Method for Elements in Water by Inductively Coupled Plasma—Mass Spectrometry to Include Interference Removal Technology and a Few Extra Metals for Grins

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Why should we update ASTM D5673?

Shares validation data with EPA 200.8, however only approved at Part 136

Data published in JAOAC VOL. 77, NO. 4, 1994

Method used by other ASTM Committees (example = ores and minerals)

Data to be shared and included in SM 3125, also approved only at Part 136

Need an ICP-MS method that allows interference removal with various interference checks. IRT allowed by Part 136.6 but not described in the methods

More elements are possible if interferences are removed

Description of the validation study (EPA 200.8. AOAC 993.14 and ASTM D5673)

Started in 1990, methods published in ~1994

20 elements and 3 matrices. Optional wastewater and optional digestion.

13 labs returned data

9 labs did the optional wastewater

Study based on ASTM D2777 Youden Pairs

Concentrations distributed in seven samples; low, medium and high

Description of the validation study (200.8, AOAC, and D5673) - continued

Digest spiked samples, analyze, and report numerical value	
HNO ₃ plus HCI digestion	
ICP-MS 0.75 amu at 5% peak height, 5 – 250 amu	
No interference removal technology used at the time	
No Interference Check Standard listed or tested in validation.	
Interference equations, and some interferences listed in the methods	

Updates to D5673 methods since initial publication

As and Se in mine influenced water

- Added an interference check standard
- New Interlab study was conducted

Gold in CN solutions

Interlab study conducted

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Proposed updates to ASTM D5673



Existing elements validated in ASTM D5673



Existing elements validated in ASTM D5673 and proposed additional elements



Additional elements will require text to describe potential issues



New calibration solutions added, old ones updated

Working with reagent and standard providers to update recipes in method This includes which elements are combined and at what concentrations

Spectral and REE interference check solutions added

Example – Calibration solutions so far



Silver Thallium Thorium Uranium Vanadium Zinc



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Re-write description of apparatus to allow other than quadrupole



The ions are extracted from the plasma through a differentially pumped vacuum interface and mass-to-charge ratio separated by a quadrupole mass spectrometer.





Interferences must be recognized and corrected for



Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions



IRT technology minimizes the impact of ionic interferences

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Interference Removal Technology (IRT)

Rather than Explain "collision or reaction cell", the method will allow any technology that removes interferences.

Interference check solutions with criteria added to method

How you remove an interference is up to you (or the manufacturer)

Harmonize method with ASTM D7439 Determination of Elements in Airborne Particulate Matter by Inductively Coupled Plasma–Mass Spectrometry

SHIMADZU Update list of potential internal standards with limitations

Internal Standard	Mass	Possible Limitation	
Lithium	7	May be present in samples	
Scandium ^A	45	Polyatomic ion interference	
Yttrium ^A	89	May be present in samples	
Rhodium	103		
Indium ^A	115	Isobaric interference by Sn	
Terbium ^A	159		
Holmium	165		
Lutetium	175		
Platinum	195	May be present in samples	
Bismuth ^A	209	May be present in samples	

Provide interference check solutions and explanation as to why they are used.

General purpose Spectral Interference Check (SIC) solution – suitable for use when analyzing most wastewaters.

This solution contains known concentrations of interfering elements and is used to verify that the method corrects the data to within acceptable QC limits.

Chloride in the SIC solution evaluates corrections for chloride-related interferences such as ³⁵Cl₁₆O⁺ on 51V + and ⁴⁰Ar₃₅Cl⁺ on ⁷⁵As⁺.

Iron is used to demonstrate adequate correction for the determination of manganese.

Molybdenum indicates the correction of molybdenum oxide effects on cadmium isotopes.

The other components help evaluate correction for various molecular-ion isobaric interferences.

The SIC is used to verify that the method corrects by the data to within acceptable QC limits.

SHIMADZU SIC Check solution Concentration – preparation described in method

Solution component	Concentration (mg/L)	
Al	100	
Ca	300	
Fe	250	
Mg	100	
Na	250	
Р	100	
К	100	
S	100	
С	200	
Cl	2000	
Мо	2	
Ti	2	

For water with TDS < 500 mg/L prepare a 1/10 dilution

Assume detections < RL. Limits determined by ILS

REE interference check solution

Rare Earth Element Spectral Interference Check (REE-SIC) solution - contains Samarium, Dysprosium, Neodymium and Gadolinium to evaluate double charged interference on arsenic and selenium when REEs are suspected to be present in the samples.

REE-SIC Working Solution – analyze each stock solution separately or combine into concentrations of 50 µg/L each or concentrations expected to approximate the highest concentration of the REE in samples.

Use the REE-SIC working solutions to establish corrections on potential As and Se interferences.

May need to scan all new sample types, or add REE into the routine method

There are other potential interferences not included in the draft yet

Mine dewatering Interference check already validated



ICSA working Solution – combine 10 ml of the 1000 mg/L calcium stock standard and 5 mL of the 1000 mg/L chloride stock standard in a 100 mL volumetric flask. Dilute to the mark with reagent water and mix.



ICSB working solution - combine 10 ml of the 1000 mg/L calcium stock standard and 5 mL of the 1000 mg/L chloride stock standard in a 100 mL volumetric flask and add 20 microliters each of 100 mg/L As and Se. Dilute to volume with water and mix.

Summary of updating ASTM D5673

New elements are being added	IRT being added to address REE interference, but also add to method since it was never there	IRT enables new elements to be added	Technical changes to the method require a new Inter-lab study
The new study will not invalidate the old one	Interference check solutions are added	The method will not restrict the mass spectrometer or the IRT used	The updated method is in collaboration with SM 3125

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Thank You



A special Thank You to all the D19 members that have contributed so far.



If you would like to participate:

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Questions?

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