

Analysis of Soils, Sediments, and Sludges by ICP-OES per US EPA 6010D

Improved data quality with the use of smart tools

Analyze sample digests with more certainty

The US Environmental Protection Agency (EPA) 6010D method is widely used for the determination of 31 elements in soils, sediment, and sludges by ICP-OES. To ensure an accurate analysis, Agilent 5800 and 5900 ICP-OES instruments use smart tools that simplify method development, monitor instrument performance, and generate more reliable analytical results.

An Agilent 5800 ICP-OES fitted with an Agilent AVS 7 switching valve was used to analyze soil samples per the 6010D method. Agilent ICP Expert 7.5 control software with IntelliQuant Screening and early maintenance feedback (EMF) was used to provide valuable insights into the samples and track instrument operational performance.

The method was validated by analyzing an industrial waste standard reference material, NIST 2711a. Excellent recoveries were achieved for all elements, as shown in Table 1. See the application note for the full-data set (7).

Table 1. US EPA 6010D performance validation data for selected elements.

	MDL	NIST 2711a (n=6)		
	(mg/L)	Measured (mg/kg)	Expected (mg/kg)	Recovery (%)
As 188.980	0.00449	90.3	89	101
Cd 214.439	0.000122	49.4	47	105
Co 230.786	0.000711	7.25	7.5	97
Cr 205.560	0.000458	15.5	15	103
Cu 327.395	0.000839	121	130	93
Hg 194.164	0.000967	6.7	7.4	91
Ni 231.604	0.000693	15.5	15	103
Pb 220.353	0.00249	1230	1300	95
Se 196.026	0.0092	1.69	1.7	99
Tl 190.794	0.00548	2.12	2.1	101
Zn 206.200	0.000328	363	350	104

Intelligent method development

IntelliQuant Screening was used to assist with method development, particularly for wavelength selection. Based on the analysis of two soil samples, As 188.980 nm received the best star ranking, as shown in Figure 1. This ranking is likely due to an interference from Al causing a background shift on the more commonly used 193.696 nm wavelength. V 197.199 nm interferes with As 197.198 nm and received a 1-star ranking.

Element	Used	Flags	Wavelength	Rating	Concentration	Intensity	Background
As			188.980	★★★★★	63.74	3200.2	1950.6
			193.696	★★★	80.14	3045.7	4197.4
			197.198	★	66.29	2743.6	3529.5
			200.334	★★	52.13	737.9	2455.0

Figure 1. IntelliQuant Screening selects the best wavelengths for your analysis.

Automatically flag outlier results

The outlier conditional formatting (OCF) function of the ICP Expert software was used to help with the QC process. The element concentration %RSD flag (F) identified an issue with arsenic, as shown in Figure 2. Specifically, As 193 and 197 reported lower results than As 188. Further information can be obtained from the result concentration % RSD flag (B). The B flag indicates when the replicate %RSD is greater than the set value. In this example, %RSD was set to 3%.

Solution Label	Outlier Summary	As 188.980 nm ppm	As 193.696 nm ppm	As 197.198 nm ppm
SRM 2781 1	B F	8.141439	6.540163	5.805309

Figure 2. Examples of two flags that help the analyst to quality control the data.

Base your instrument maintenance schedule on quality feedback

Analyzing complex sample types can be tough on the instrument's sample introduction system. To maximize analytical performance and minimize unplanned instrument downtime, scheduling maintenance tasks by the number of solutions measured rather than time can be beneficial. The user

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can configure the maintenance counters, or default counters for specific sample types can be generated automatically (Figure 3). The AVS 7 extends the life of the torch allowing the counter to be set at a relatively high number of samples.

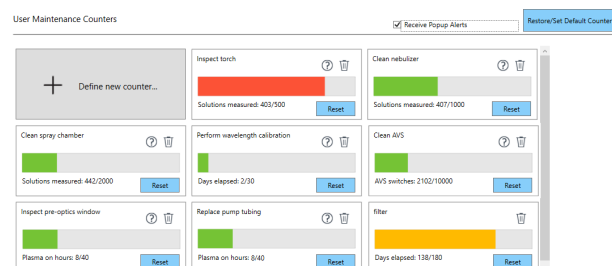


Figure 3. Maintenance page in ICP Expert 7.5 – early feedback can prevent unexpected downtime and reduce consumable costs.

High-quality results

Smart tools such as IntelliQuant Screening, OCF, and EMF help analysts to achieve a high level of performance and reduce the need to remeasure samples, improving productivity. To test the stability of the 5800, the initial calibration verification standard was measured every 10 samples over a six-hour period. All measurements passed the US EPA 6010D specification of +/-10% without the need to recalibrate or remeasure samples (Figure 4). The RSD over six hours was less than 2% for all elements.

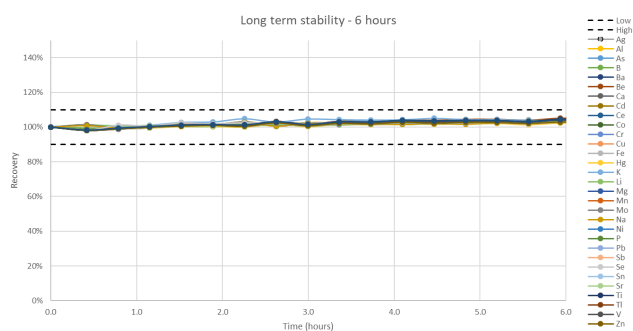


Figure 4. Long-term stability over six hours for all elements specified in EPA 6010D.

More information

Analysis of Waste Samples According to US EPA Method 6010D, Agilent publication, [5994-2027EN](https://www.agilent.com/chem/5994-2027EN)