Application Brief

Environmental

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Analysis of Industrial Waste Samples using the Agilent 5800 VDV ICP-OES

Smart tools for operational and sample insight

Analyzing complex sample digests

Chinese method HJ 781-2015 outlines the procedure for the determination of 22 elements in solid waste samples, prepared using a four-acid microwave digestion program. Such complex sample digests often contain elements that are not targetted in the method. These additional (and often unknown) elements can lead to spectral interference if their presence is not considered. Spectral interference can cause erroneous results and force the remeasurement of samples, causing wasted time.

The Agilent IntelliQuant function automatically identifies likely spectral interferences and unexpected elements, providing valuable insight into the sample composition. IntelliQuant can identify up to 70 elements in a sample within seconds. The resultant data can be filtered, showing only the results (elements) that are of interest.

In this study, instrument performance was demonstrated by analyzing industrial waste NIST SRM 2782. The results are shown in Table 1. Recoveries within ±10% were achieved.

Table 1. Results for nine of the 22 elements measured for NIST SRM 2782, using a method compliant with Chinese Standard HJ 781-2015, on an Agilent 5800 ICP-OES. The full list of results is available in Agilent document number 5994-1542.

Element and Wavelength (nm)	MDL (mg/kg)	HJ 781 MDL Specification (mg/kg)	Measured SRM (mg/kg)	Expected (mg/kg)	SRM Recovery (%)
As 188.980	0.0711	-	164	166	99
Cd 228.802	0.0103	0.1	3.82	4.17	92
Cr 267.716	0.0395	0.5	102	109	94
Cu 327.395	0.0337	0.4	2690	2594	104
Fe 273.358	1.96	8.9	267000	269000	99
Mo 204.598	0.0299	-	9.98	10.07	99
Ni 221.648	0.0247	0.4	151	154.1	98
Pb 220.353	0.0571	1.4	533	574	93
Zn 206.200	0.0216	1.2	1200	1254	96

Sample insights using IntelliQuant

The use of the Agilent IntelliQuant function helped deliver the excellent recoveries achieved for the SRM analysis. The HJ-781 method specifies three possible Zn emissions lines to use: 213.857, 202.548, and 206.200 nm. A scan of the industrial waste SRM digests, using IntelliQuant, identified interferences on two of these lines (213.857 and 202.548 nm), as indicated by the one-star confidence rating IntelliQuant gave to those lines in Figure 1. IntelliQuant suggested five other possible wavelengths to use for the analysis of Zn (as show in Figure 1). One of the wavelengths specified in the method, Zn 206.200 nm, as well as Zn 334.502 nm were given the highest confidence rating (five stars). However, Zn 206.200 nm was identified as more sensitive than 334.502 nm and would thus provide a better detection limit. Zn 210.442 was considered a concentration outlier and given only a one star rating. Analytical results generated using this wavelength would be unreliable.

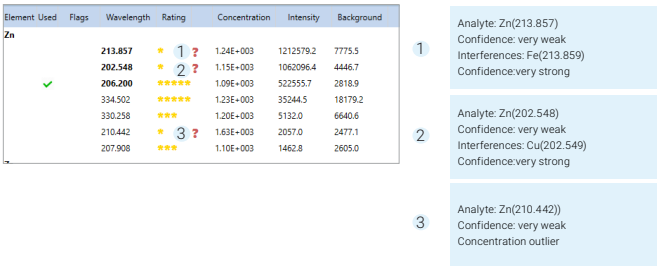

Figure 1. The IntelliQuant star ranking system uses data analytics to rank different emission wavelengths for the same element. Clicking on the "?" symbol displays reasons for the poor rating on a wavelength.

Instrument maintenance schedule determined by sample load

Analyzing complex samples, such as industrial waste, can be tough on the sample introduction system of an ICP-OES. This can result in deteroriating analytical performance, high consumable costs, and unplanned instrument downtime. Scheduling maintenance tasks according to the number of solutions measured, rather than elapsed time can reduce these impacts.

The Agilent 5800 and 5900 instruments have an Early Maintenance Feedback (EMF) function that allows the user to setup an alert to prompt maintenance after a specified number of samples. Recommended alert settings for specific sample types can be generated automatically. This will result in more frequent instrument maintenance when measuring complex samples, ensuring continued good analytical performance.

Flag outlier results automatically

To assess the quality of the analytical data, it was useful to compare results obtained from different wavelengths for the same element. The Outlier Conditional Formatting (OCF) software function was used to do this.

The OCF function found that the difference between the results obtained using the three Pb wavelengths exceeded the precision threshold specified. The results were flagged for review (Figure 2).

With three slightly different results for the same element, IntelliQuant was used to identify the result to report. In this case, the 220.353 nm Pb emission line was given the highest confidence rating by the IntelliQuant algorithm, so that result was deemed to be the most accurate.

Solution Label	Outlier Summary	Pb 220.353 nm ppm	Pb 283.305 nm ppm	Pb 405.781 nm ppm
Sample 1	F	184.5	390.0	206.5
Sample 2	F	4799.4	5512.0	5402.1
Sample 3	F	466.5	1251.5	502.4
Sample 4	F	374.7	603.1	427.3
Sample 5	F	480.0	1265.6	504.4
Sample 6	F	4890.5	5625.9	5511.1

Figure 2. All six results were flagged in the Outlier Summary column. This informs the analyst that there is a difference in the Pb concentration determined using the different wavelengths.

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