

High Throughput, Multi-Element Analysis of Effluents by MP-AES

Low-cost method using the Agilent 4210 MP-AES with AVS 4 switching valve in accordance with Brazilian regulations



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Introduction

Effluents are produced by a wide range of industrial, commercial, and agricultural processes. To prevent potentially harmful elements such as As, Cd, Pb, and Hg from contaminating the environment, effluents must be analyzed before they are discharged or treated. The presence of trace amounts of Cd and Pb in surface or ground waters can affect soil fertility and plant growth, as well as accumulating in fish (1-4). Regulations (CONAMA 430/2011) issued by the Brazilian Ministry of the Environment specify maximum permitted concentrations of several elements that must be evaluated in effluents before disposal (5). The list of elements includes Ag, As, B, Ba, Cd, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Se, Sn, and Zn.

Various atomic spectrometric techniques have been used to analyze wastewaters, including Flame Atomic Absorption Spectrometry (FAAS), Graphite Furnace-AAS (GFAAS), Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES), and ICP-Mass Spectrometry (ICP-MS) (6-8). However, as contract and quality

control (QC) laboratories look to improve their efficiency while providing high-quality data, many have added Microwave Plasma-AES (MP-AES) to their range of elemental analysis techniques. MP-AES runs unattended without the need for flammable or expensive gases, or hollow cathode lamps, improving laboratory safety and reducing operating costs (9).

The Agilent 4210 MP-AES uses microwave energy to generate a robust and stable plasma using nitrogen (N_2) gas. The N_2 can be extracted straight from air using the Agilent 4107 Nitrogen Generator or be supplied from a tank or Dewar. Compared to Flame AAS there are considerable savings in operating costs which can be calculated with the online calculator (9). For the analysis of effluents under the CONOMA 430 method, the savings in operating costs means the MP-AES pays for itself in as little as 5 months*. MP-AES is a multi-element technique that is more suited to high throughput applications than FAAS. However, its productivity can be further improved using accessories, such as the Agilent SPS 4 autosampler and Agilent Advanced Valve System (AVS 4), shown in Figure 1.



Figure 1. Agilent 4210 MP-AES with SPS 4 autosampler and AVS 4 switching valve system.

Benefits of productivity accessories

The SPS 4 autosampler and AVS 4 switching valve maximize productivity by allowing unattended analysis. They also ensure a fast and reliable systematic clean-out of the whole sample introduction system between each sample. The AVS 4 only delivers the sample to the MP-AES during the data acquisition step and exposure to high levels of total dissolved solids (TDS). These features ensure excellent long-term stability and maximize the lifetime of consumables.

Experimental

Instrumentation

All measurements were performed using an Agilent 4210 MP-AES equipped with an SPS 4 autosampler and AVS 4 switching valve. The instrument was fitted with a OneNeb Series 2 nebulizer, double-pass glass cyclonic spray chamber, and Easy-fit torch. For the analysis of As, Se, and Hg, the 4210 was fitted with the optional multimode sample introduction system (MSIS) accessory (10). By separating volatile hydride species from the sample or using cold vapor generation, the MSIS provides better performance and lower detection limits than conventional nebulization.

An Agilent 5110 ICP-OES was used to check the accuracy of the MP-AES method for the determination of Ag, B, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sn, and Zn.

Preparation of calibration standards

Procedure 1 for the determination of Ag, B, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sn, and Zn

Multi-element calibration standards were prepared from an intermediate solution, which was obtained by mixing Agilent 1,000 mg/L single standard solutions of Ag, B, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sn, and Zn in 3% (v/v) HNO_3 (Merck, Sao Paulo, Brazil) and ultrapure water (UPW) (Sartorius, Göttingen, Germany). An ionization buffer solution of cesium chloride (CsCl) (Sigma-Aldrich, São Paulo, Brazil) was prepared at 1.0% (w/v) and was continuously added to the sample line using the Agilent internal standard kit (p/n 9910124100).

An intermediate standard solution was prepared by mixing and diluting Agilent single element standard solutions of 1,000 mg/L As and Se. Calibration standards were then prepared from 0.2 to 1.0 mg/L.

Procedure 2 for the determination of As and Se

The MSIS was used in hydride generation mode, using 1.2% (w/v) $NaBH_4$ (Sigma-Aldrich, São Paulo, Brazil) in 0.5% (w/v) NaOH (Sigma-Aldrich, São Paulo, Brazil) as the reductant (Figure 2). The calibration standards were prepared in 50% (v/v) HCl (Merck, São Paulo, Brazil) and submitted to a pre-reduction step using the Multiwave GO microwave digestion system (Anton-Paar), as described in Table 1.

Table 1. Pre-reduction step for As and Se standards using microwave digestion.

| Step | Ramp Time (min) | Temperature (°C) | Hold Time (min) |
|------|-----------------|------------------|-----------------|
| 1 | 10 | 110 | 10 |

*When running 200 samples per week, compared to flame AAS.

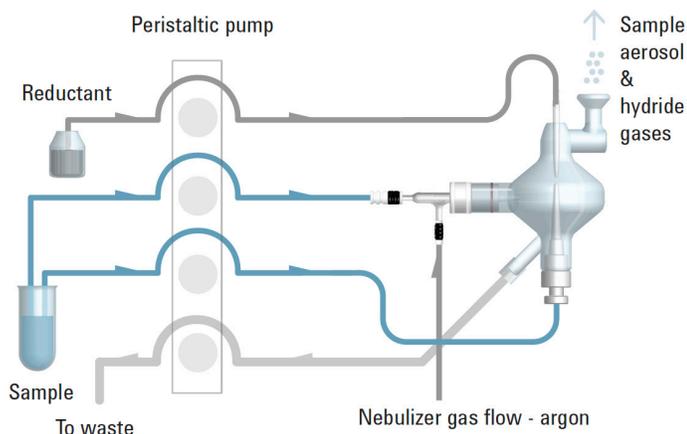


Figure 2. MSIS in hydride mode using the three-channel peristaltic pump.

Procedure 3 for the determination of Hg

Mercury calibration standards were prepared from 0.005 to 0.025 mg/L in 0.1% (v/v) HCl and 0.1% (v/v) HNO₃ using an Agilent single standard solution of Hg at 1,000 mg/L.

The MSIS was used in cold vapor generation mode using 25% (w/v) SnCl₂ (Química Moderna, Barueri, SP) in 20% (v/v) HCl as the reducing agent.

Agilent 4210 MP-AES instrument operating conditions are detailed in Table 2 and the calibration concentration ranges for all elements are given in Table 4.

Table 2. 4210 MP-AES operating parameters.

| Parameter | Calibration Procedure 1 | Calibration Procedure 2 | Calibration Procedure 3 |
|-----------------------------|---|-------------------------------|-------------------------|
| Element and Wavelength (nm) | Ag (328.068), B (249.772), Ba (455.403), Cd (228.802), Cr (425.433), Cu (324.754), Fe (371.993), Mn (403.076), Ni (352.454), Pb (405.781), Sn (317.505), Zn (213.857) | As (193.695) and Se (196.026) | Hg (253.652) |
| Background Correction | Auto | | |
| Read Time (s) | 3 (10 for Pb) | 10 | 20 |
| Viewing Position | 0 | | |
| Spray Chamber | Double-pass | MSIS | |
| Nebulizer | OneNeb Series 2 with Nitrogen Humidifier | MiraMist (PTFE) | |
| Nebulizer Flow (L/min) | 0.50 | 0.40 | 0.75 |
| Replicates | 3 | 5 | |
| Pump Speed (rpm) | 15 | | |

| Pump Tubing | Orange/green (CsCl 1.0%), orange/white (sample), blue/blue (waste) | Black/black (sample and reductant), blue/blue (waste) | |
|------------------------|--|---|------|
| Uptake Time (s) | 20 (with fast pump on) | 25 (with fast pump on) | |
| Switch Delay (s) | 20 | NA | |
| Rinse Time (s) | 30 (with fast pump on) | | |
| Stabilization Time (s) | 10 | 25 | |
| Calibration Fit | Linear | | |
| Weighted Fit | On | | |
| Run Time (min) | 3.30 | 3 | 2.20 |

Samples and sample preparation

Ten industrial and domestic effluents were prepared for analysis. Since CONAMA 430 defines limits for dissolved Cu, Fe, and Mn, each sample was filtered using a 0.45 µm syringe filter (Agilent p/n 5190-5266). As, Ba, B, Cd, Pb, Cr, Sn, Hg, Ni, Ag, Se, and Zn are measured in the sample without filtration (so defined as total). Filtered and raw samples were acid-digested using the Multiwave GO microwave digestion system program described in Table 3. This program was used for all elements. 5 mL of sample and 0.3 mL of HNO₃ were added to each vessel. After sample digestion and cooling, the solution was diluted to 10 mL, making the final acid concentration 3% (v/v). For the determination of As and Se, a different acid concentration was required for the digestion. 5 mL of concentrated HCl and 0.1 mL of concentrated HNO₃ were added to 5 mL of sample, making the final acid concentration approximately 50% HCl (v/v) and 1% HNO₃ (v/v).

Table 3. Microwave-assisted digestion program used for sample preparation.

| Step | Ramp Time (min) | Temperature (°C) | Hold Time (min) |
|------|-----------------|------------------|-----------------|
| 1 | 10 | 180 | 10 |

Results and discussion

Calibration

Calibration curves for all 15 elements specified in CONAMA 430 showed good linearity across the concentration range, as indicated by correlation coefficients > 0.999 (Table 4). Representative calibration curves for some of the most challenging elements of the analysis (Cd, Pb, As, and Hg) are shown in Figure 3.

The method limit of quantitation (MLOQ) were calculated using 10 times the standard deviation of seven blank measurements, multiplied by the dilution factor. The MLOQs for all elements were lower than the maximum permitted concentration specified in CONAMA 430, as shown in Table 4.

Table 4. Correlation coefficients, MLOQs, and CONAMA regulated upper concentration limits.

| Element and Wavelength (nm) | Standard Conc Range (mg/L) | Correlation Coefficient (R) | MLOQ (mg/L) | CONAMA 430/2011 Maximum Permitted Concentration (mg/L) |
|-----------------------------|----------------------------|-----------------------------|-------------|--|
| Ag (328.068) | 0.1 – 0.5 | 0.9999 | 0.07 | 0.1 |
| ^a As (193.695) | 0.2 – 1.0 | 0.9996 | 0.05 | 0.5 |
| B (249.772) | 2.0 – 10 | 0.9999 | 0.07 | 5.0 |
| Ba (455.403) | 2.0 – 10 | 0.9999 | 0.01 | 5.0 |
| Cd (228.802) | 0.1 – 0.5 | 0.9999 | 0.08 | 0.2 |
| Cr (425.433) | 0.05 – 0.25 | 0.9999 | 0.03 | 0.1* |
| Cu (324.754) | 0.5 – 2.5 | 0.9999 | 0.02 | 1.0 |
| Fe (371.993) | 5 – 25 | 0.9999 | 0.06 | 15 |
| ^b Hg (253.652) | 0.005 – 0.025 | 0.9995 | 0.008 | 0.01 |
| Mn (403.076) | 0.5 – 2.5 | 0.9999 | 0.01 | 1.0 |
| Ni (352.454) | 0.5 – 2.5 | 0.9999 | 0.08 | 2.0 |
| Pb (405.781) | 0.2 – 1.0 | 0.9999 | 0.15 | 0.5 |
| ^a Se (196.026) | 0.2 – 1.0 | 0.9993 | 0.05 | 0.3 |
| Sn (317.505) | 2.0 – 10 | 1.0000 | 0.17 | 4.0 |
| Zn (213.857) | 2.0 – 10 | 0.9999 | 0.04 | 5.0 |

*Cr(VI)
 a Determined using MSIS in hydride generation mode.
 b Determined using MSIS in cold vapor mode.

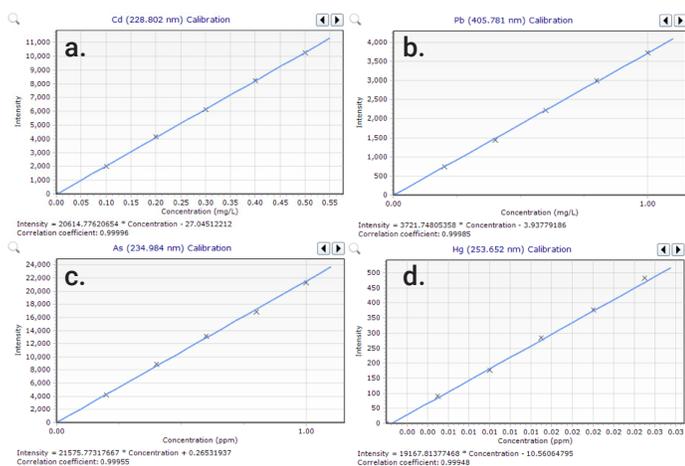


Figure 3. Calibration curves for a. Cd, b. Pb, c. As, and d. Hg.

Sample analysis

The elemental composition of effluents depends on how they are produced. The treatment of effluents relies on the fast, reliable quantitation of the sample constituents. The results from the analysis of 10 effluents analyzed using MP-AES are shown in Table 5. Data is provided for B, Ba, Cr, Cu, Fe, Mn, Ni, and Zn, since the concentrations of Ag, As, Cd, Hg, Pb, Se, and Sn were below the MLOQ in all samples. Only boron in sample 7 exceeded the CONAMA 430/2011 maximum permitted concentration of 5.0 mg/L, indicating that this effluent required further treatment before disposal. If the total chromium result of any sample exceeded 0.1 mg/L, Cr(III) and Cr(VI) would need to be determined by UV-Vis (11) or HPLC-ICP-MS (12).

Table 5. Results of the analysis of 10 effluents using the 4210 MP-AES with the SPS 4 and AVS 4. No results are provided for Ag, As, Cd, Hg, Pb, Se, and Sn, as the concentrations were below the MLOQ in all samples.

| Sample | Concentration (mg/L) | | | | | | |
|--------|----------------------|-------|-------|------|------|-------|-------|
| | B | Ba | Cu* | Fe* | Mn* | Ni | Zn |
| 1 | 0.09 | 0.03 | 0.05 | 1.2 | 0.05 | <MLOQ | 0.39 |
| 2 | <MLOQ | 0.09 | 0.20 | 5.2 | 0.08 | <MLOQ | 0.25 |
| 3 | 0.21 | <MLOQ | <MLOQ | 3.2 | 0.07 | <MLOQ | 3.5 |
| 4 | <MLOQ | 0.02 | <MLOQ | 0.08 | 0.01 | <MLOQ | <MLOQ |
| 5 | <MLOQ | 0.10 | 0.06 | 0.33 | 0.20 | <MLOQ | 0.49 |
| 6 | 0.22 | 0.14 | 0.27 | 5.6 | 0.10 | <MLOQ | 1.5 |
| 7 | 8.4 | 0.10 | 0.05 | 5.8 | 0.82 | <MLOQ | 0.31 |
| 8 | <MLOQ | 0.04 | 0.03 | 0.38 | 0.03 | <MLOQ | 0.06 |
| 9 | 2.5 | 0.16 | <MLOQ | 0.27 | 0.02 | 0.12 | <MLOQ |
| 10 | <MLOQ | 0.03 | 0.02 | 0.16 | 0.02 | <MLOQ | 1.3 |

*Dissolved content

Long-term stability (LTS)

To check instrument stability during the long-term measurement of Ag, B, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sn, and Zn, a spiked effluent sample was analyzed repeatedly for almost 8 hours. Using a humidifier for the nebulizer gas flow, the long-term measurement precision was < 1.5% RSD for all elements. The LTS test results demonstrate that the 4210 MP-AES with the SPS 4 and AVS 4 can operate for a full working day with excellent working precision and stability.

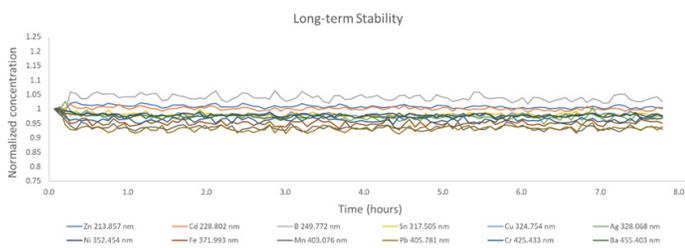


Figure 4. Long-term stability for Ag, B, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sn, and Zn. The maximum %RSD was 1.2% for Pb 405.781 nm over almost eight hours of continuous operation.

Accuracy check: ICP-OES analysis and spike recovery

To check the accuracy of the MP-AES results for the 12 elements analyzed per Procedure 1 (Table 1), the same 10 effluent samples were measured using the 5110 ICP OES. The MP-AES and ICP OES results for elements with concentrations higher than the MLOQ (Table 5) have been compared in Figures 5a to 5f. The coefficient of linearity for B, Ba, Cu, Fe, Mn, and Zn is >0.99 in all cases, showing good agreement between the results obtained from both instruments.

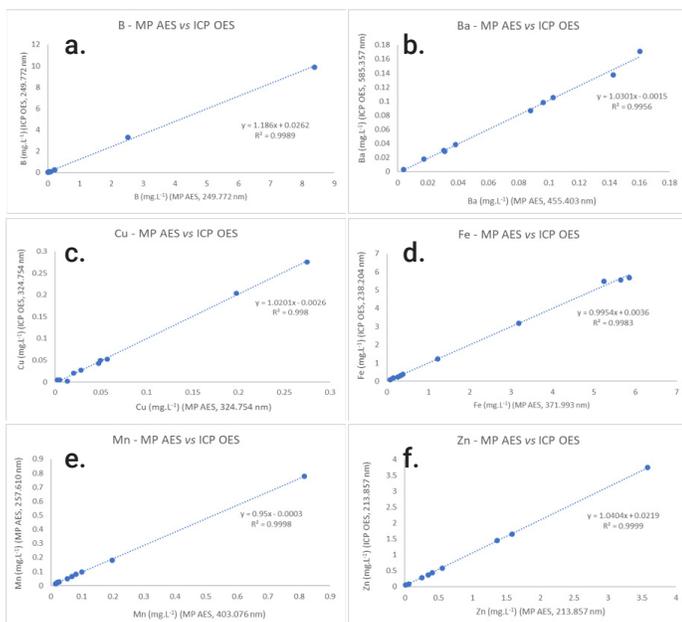


Figure 5. Comparison between MP-AES and ICP-OES results for the measurement of a. B, b. Ba, c. Cu, d. Fe, e. Mn, and f. Zn in 10 effluent samples.

Sample 4 was selected for the spike recovery test. The low-level and high-level spike concentrations of each analyte (Table 6) were added to the sample before sample preparation. Recoveries for all analytes were within $\pm 10\%$, as shown in Table 6.

MP Expert Utilities: Quick Read and Dashboard

Analysts can run a quick read scan to better understand a sample before developing the full method. Effluents often contain high levels of group 1A and 2A elements and major concentration analytes, such as Na, K, Mg, and Ca. These easily ionized elements (EIEs) can cause analytical problems such as ionization variation, depending on the element. Performing a quick read scan of the samples before setting up a method can reduce time spent on further dilution steps and give an insight into any potential interferents.

Using the quick scan pane shown in Figure 6, the analyst selects which element and wavelength to include in the scan. Once selected, the scan takes only a few seconds. The quick read scan results for Na (588.995 nm), K (766.491 nm), Mg (285.213 nm), and Ca (393.366 nm) in Sample 4 are shown in Figure 7. Quick read is a diagnostic tool that allows the analysis of specific elements in order to check peak shapes and intensities, without the need to setup and develop a full method.

In this study, an ionization buffer was used to overcome EIE effects. This approach was effective, as shown by the recovery data in Table 6.

Table 6. Recovery of low- and high-concentration level spikes (mg/L) of sample 4 by MP-AES.

| Analyte | Sample 4 Measured Conc | Low Level Spike | Low-Level Spiked Sample Conc | Recovery (%) | High-Level Spike | High-Level Spiked Sample Conc | Recovery (%) |
|-----------------|------------------------|-----------------|------------------------------|--------------|------------------|-------------------------------|--------------|
| Ag | <0.07 | 0.200 | 0.180 | 90 | 0.400 | 0.411 | 103 |
| As ^a | <0.05 | 0.200 | 0.202 | 101 | 0.800 | 0.759 | 95 |
| B | <0.07 | 4.00 | 4.07 | 102 | 8.00 | 8.21 | 103 |
| Ba | 0.02 | 4.00 | 4.06 | 101 | 8.00 | 8.43 | 105 |
| Cd | <0.08 | 0.200 | 0.203 | 102 | 0.400 | 0.415 | 104 |
| Cr | <0.03 | 0.100* | 0.097 | 97 | 0.200 | 0.213 | 107 |
| Cu | <0.02 | 1.00 | 0.999 | 100 | 2.00 | 2.10 | 105 |
| Fe | 0.08 | 10.0 | 9.89 | 98 | 20.0 | 21.4 | 107 |
| Hg ^b | <0.008 | 0.010 | 0.009 | 90 | 0.020 | 0.020 | 100 |
| Mn | 0.01 | 1.00 | 0.986 | 99 | 2.00 | 2.17 | 109 |
| Ni | <0.08 | 2.00 | 2.01 | 100 | 4.00 | 4.21 | 105 |
| Pb | <0.15 | 0.400 | 0.393 | 98 | 0.800 | 0.855 | 107 |
| Se ^a | <0.05 | 0.200 | 0.218 | 109 | 0.800 | 0.804 | 101 |
| Sn | <0.17 | 4.00 | 4.00 | 100 | 8.00 | 8.53 | 107 |
| Zn | <0.04 | 4.00 | 4.02 | 100 | 8.00 | 8.24 | 103 |

*Cr(VI)

^a Determined by using MSIS in hydride generation mode.

^b Determined using MSIS in cold vapor mode.

The screenshot shows the 'Selected element Na' interface. On the left is a periodic table with 'Na' selected. Below it is a 'Read' button and a wavelength input field showing '588.995'. On the right is a table of spectral data for Na:

| Wavelength (nm) | Ion | Intensity | Order |
|-----------------|-----|-----------|-------|
| 588.995 | I | 108731.2 | 1 |
| 589.592 | I | 81616.0 | 2 |
| 330.237 | I | 597.4 | 3 |
| 568.820 | I | 516.8 | 4 |
| 568.263 | I | 254.0 | 5 |
| 330.298 | I | 239.4 | 6 |
| 616.075 | I | 95.1 | 7 |
| 498.281 | I | 68.7 | 8 |
| 315.953 | II | 50.4 | 9 |
| 615.423 | I | 48.3 | 10 |

Figure 6. The quick read pane on MP Expert version 1.6 (or later) software. Using sodium as an example, wavelengths can be selected within the pane shown on the right. Each selected wavelength will then to be scanned using quick read to check the peak shape and intensity, as shown in Figure 7.

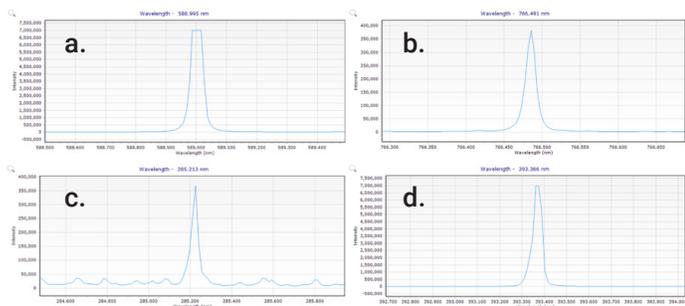


Figure 7. Primary emission lines for a. Na (588.995 nm), b. K (766.491 nm), c. Mg (285.213 nm), and d. Ca (393.366 nm) obtained from a quick read scan of Sample 4.

Being able to check instrument operating conditions quickly and easily is a simple way to keep the MP-AES running safely, efficiently, and productively. The MP-AES Dashboard shows readings from several instrument parameters, such as mains voltage and frequency, temperature measurements, gas flow rates, and gas pressure (Figure 8). Monitoring these parameters allows preventative maintenance to be undertaken, ensuring consistent and reliable instrument performance.

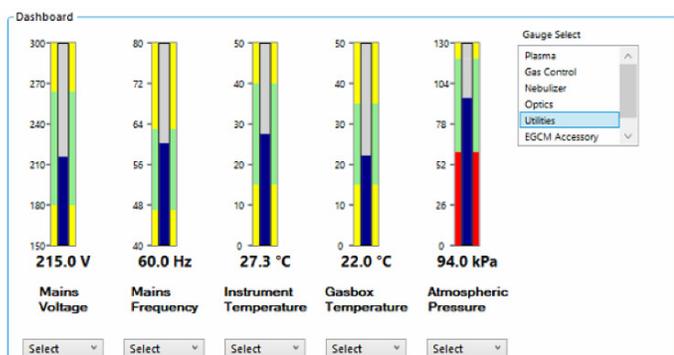


Figure 8. Overview of instrument parameters using the dashboard feature of the instrument control software. Each parameter reading (blue bar) should be within the recommended (green) range.

Conclusion

The Agilent 4210 MP-AES with the SPS 4 and AVS 4 is suitable for the elemental determination of effluents in accordance with the regulations specified in CONAMA 430/2011.

The MP-AES results for Ag, B, Ba, Cd, Cr, Cu, Fe, Mn, Ni, Pb, Sn, and Zn in 10 effluent samples were in good agreement with results obtained by ICP-OES. A spike recovery test using the

same elements in an effluent sample further confirmed the accuracy of the method. Both low-level and high-level spikes were measured within 10% of the expected concentrations. Only boron in one of the samples was found to exceed the upper concentration limits specified in the Brazilian regulations.

For the analysis of As, Se, and Hg, the 4210 MP-AES was fitted with a multimode sample introduction system (MSIS). As, Se, and Hg were measured in hydride or cold vapor generation mode.

The quick read scan feature of MP Expert software was used to provide valuable information about the samples before the full method was developed. Because EIEs can cause interferences, quick read was used to scan the samples for Na, K, Mg, and Ca, with each scan taking only a few seconds. The scan data was used to check the peak shapes and intensities of each element without the need to develop a full method.

The Agilent 4210 MP-AES with the SPS 4 and AVS 4 can run unattended batches of samples over long periods with good accuracy and stability with %RSD < 1.5%. The method meets the needs of QC and contract laboratories looking for a fast, reliable, and low-cost alternative to FAAS for the elemental analysis of effluents and wastewaters.

Acknowledgments

The authors would like to thank Camila Costa Petr leo, C tia Cristina Guzela, and Val ria Rafashi from the National Industrial Apprenticeship Service (SENAI Mario Amato, S o Bernardo do Campo, S o Paulo, Brazil) for providing the effluent samples.

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Note: the AVS 4, quick read scan, and dashboard require MP Expert 1.6.X (or later) version software. An MP Expert PRO license is needed to enable automatic and periodic analysis of QC standards.

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