RADOM
INSTRUMENTS

MICAP-OES 1000

RAPID DETERMINATION OF TRACE ELEMENTS IN HOUSEHOLD BLEACH

- Lowest Operating Cost
- Simultaneous Measurement
- Lowest Carbon Footprint
- Smallest Laboratory Footprint

Introduction

The chemical composition for bleach is sodium hypochlorite (NaOCl). Bleach applications prior to Covid were predominantly household laundry and clinical laboratory disinfecting procedures. Post-Covid, bleach is used residentially and commercially for disinfecting activities which increases the demand and therefore, production of the product.

Sodium hypochlorite is only compatible with a few metals - titanium, platinum, gold, silver, and tantalum[1]. All other metals present will cause contamination and increased product decomposition and oxygen gas production. Limited contact with stainless steel, copper, brass, and other alloys should be considered. Copper and steel alloys are commonly used in the manufacturing process of bleach. It has been shown that when nickel and copper are present there is an increase in the overall catalytic reaction rate. The high purity bleach specification is < 50 ppb transition elements. In addition, it is noted that the presence of organic material and suspended solids of calcium and magnesium will increase the rate of decomposition. As a result of these concerns, these elements are also monitored. These factors are considered in manufacturing as well as storage of the final bleach product.

This application brief will outline the process of preparing and analyzing eleven elements in a commercially available bleach. The Radom MICAP-OES 1000 was utilized to determine the eleven target elements: aluminum (Al), calcium (Ca), cobalt (Co), chromium (Cr), copper (Cu), iron (Fe), magnesium (Mg), nickel (Ni), lead (Pb) and zinc (Zn).

MICAP-OES

MICAP-OES 1000 is a Microwave Indicatively Coupled Atmospheric Plasma – Optical Emission Spectrometer with 1 kW power. This innovative nitrogen-based plasma source replaces the traditional argon generated plasma technology. This is only possible by incorporating Radom's CERAWAVE™ technology which replaces the metallic water-cooled coil found in commercially available ICP-OES instruments today.

The independent plasma source is coupled to an echelle based, research-grade spectrometer via a fiber optic connection. The plasma is viewed axially with auto-optimized plasma tail removal. The optimized viewing position is ensured with the torch alignment system. The entire echelle spectrum (194nm to 625 nm + 766nm) is simultaneously captured with each replicate measurement.

MICAP-OES 1000 is a powerful instrumental technique for metal analysis. This instrumentation provides fast, simultaneous sample measurements eliminating multiple preparation steps. In addition, the analysis is performed using instrumentation with low carbon footprint by eliminating the need for combustible gas, sustained usage of argon gas, and chillers. The benefits are a low-cost, ease of use analysis while reducing chemical waste and carbon emissions.

Experimental Design

One bottle of commercial bleach product was purchased for the experiments.

The 5g ± 0.005g of household bleach was weighed in triplicate in 50mL single-use Digitubes. In addition, to verify the preparation procedure, a method blank (MB) and a laboratory control sample (LCS) were prepared and digested in the same process as the samples. The digestion consisted of adding 10mL of de-ionized water, 2.5mL concentrated hydrochloric acid (HCl), and 1mL of 30% hydrogen peroxide (H_2O_2) to each sample tube. The LCS and two of the three bleach samples were spiked with 0.5ppm of the target analytes, aluminum (Al), calcium (Ca), cobalt (Co), chromium (Cr), iron (Fe), magnesium (Mg), manganese (Mn), nickel (Ni), lead (Pb) and zinc (Zn). For matrix matching purposes, 0.57g of 99.999% sodium chloride (NaCl) was added to the MB and LCS to achieve 1.14% NaCl. All five sample tubes were digested for 60 minutes utilizing an SCP Science DigiPrep Jr Hotblock set to 90°C. The digested test solutions were allowed to cool to room temperature, Yttrium (Y) added as an internal standard for a final concentration of 2.0 ppm, and then diluted to 50mL with Type I DI water.

Standard Preparation

Five standards were prepared in single use, 50mL tubes. The standards were prepared from a multi-element standard. The diluent was 5% HCL and 1.14% NaCl. Yttrium (Y) was added to each test solution and standard at a concentration of 2.0 ppm. Note, an on-line addition can also be considered for internal standard addition.

Table 1 presents the standard concentrations prepared for analysis.

The MICAP sample introduction assembly components utilized for this work are summarized in Table 2.

Standard 1	0.00
Standard 2	0.050
Standard 3	0.10
Standard 4	0.50
Standard 5	1.0

Table 1. Working Standards (ppm)

Component	Description
Torch	Radom 1.5 mm injector
Spray Chamber	Single pass cyclonic
Nebulizer	High efficiency polymeric nebulizer
Sample Tubing	Black/Black Solva Flex (0.76mm ID)
Drain Tubing	Blue/Yellow Solva Flex (1.52mm ID)

Table 2. MICAP-OES 1000 Sample Introduction Assembly

The Plasma and Measurement Conditions are presented in Figure 2. The total sample measurement time is 2 minutes and 10 seconds.

Representative calibration curves with figures of merit for each wavelength are presented in Figure 3. The graphs Included represent the appropriate concentration range per wavelength.

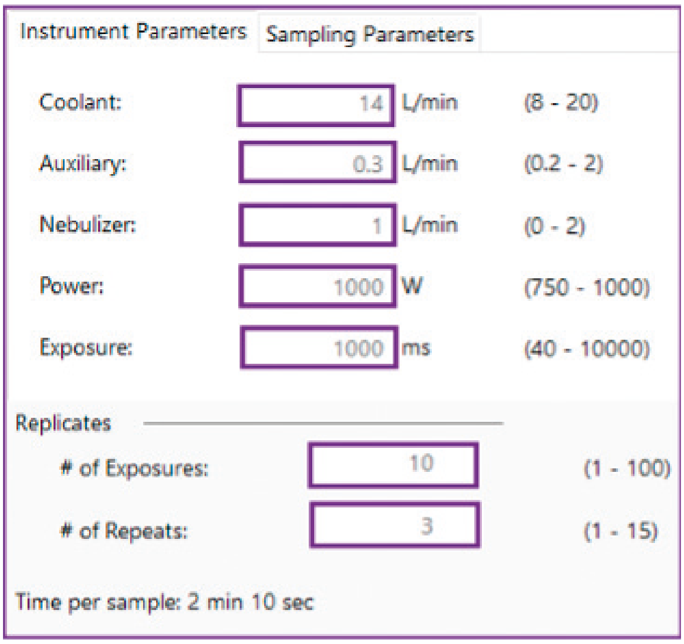
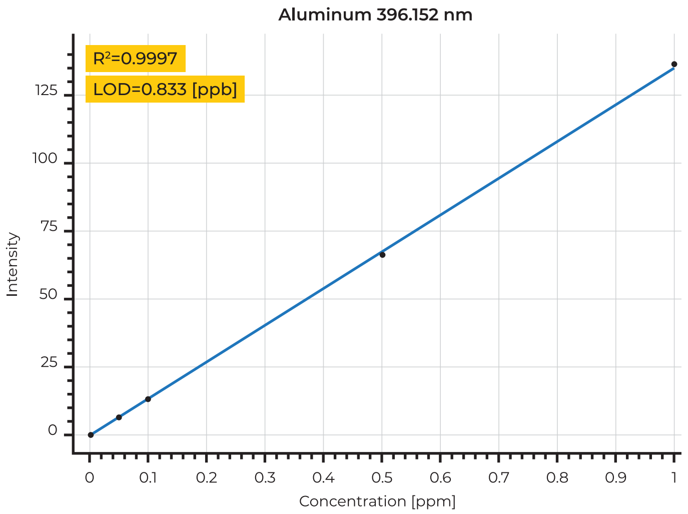
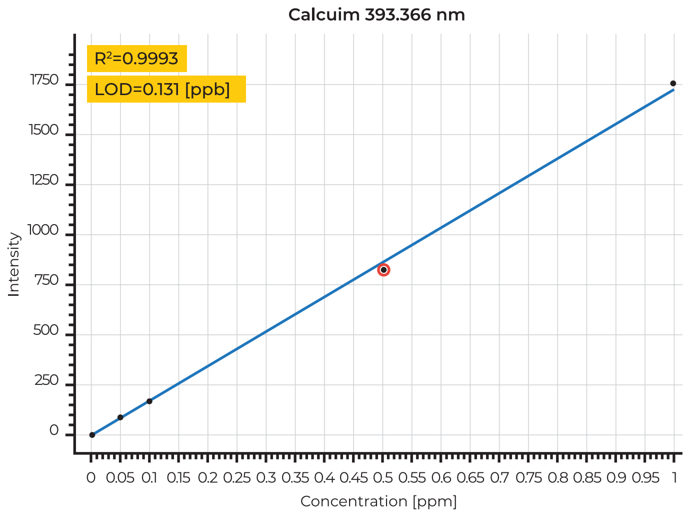


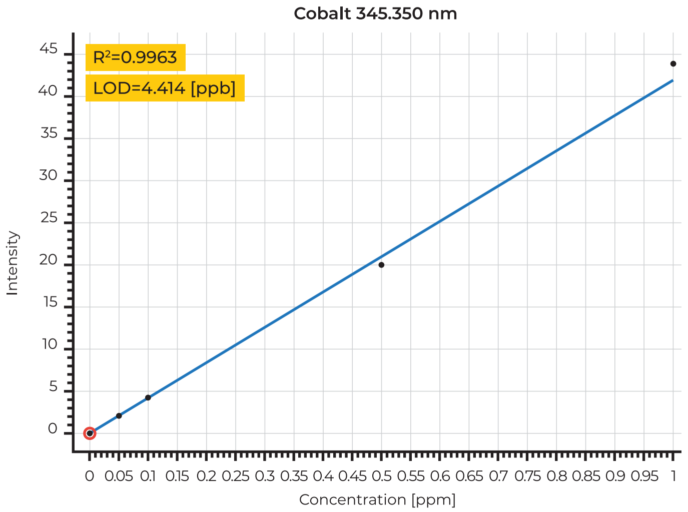
Figure 2. Plasma and Measurement Conditions



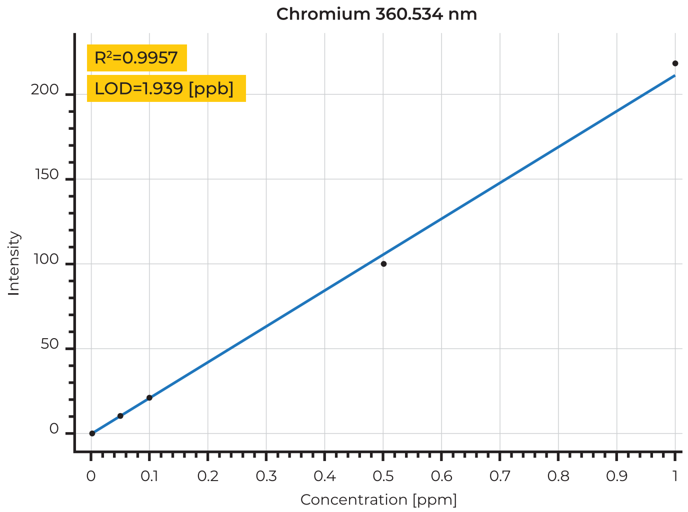
Aluminum 396.152 nm
R²=0.9997
LOD=0.833 [ppb]
Intensity
Concentration [ppm]



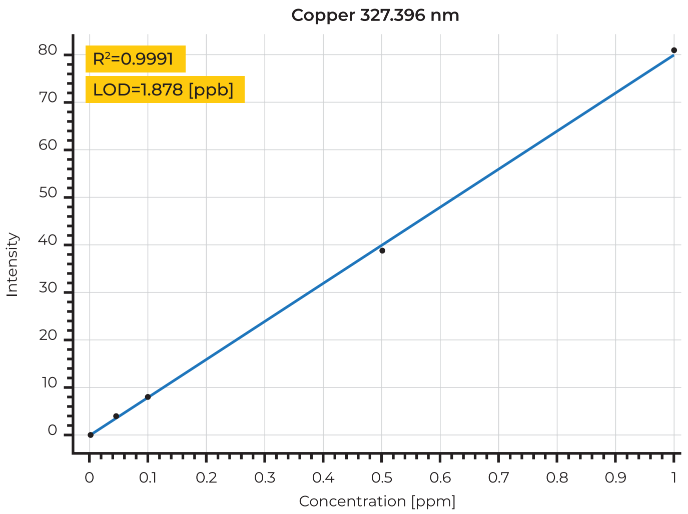
Calcuim 393.366 nm
R²=0.9993
LOD=0.131 [ppb]
Intensity
Concentration [ppm]



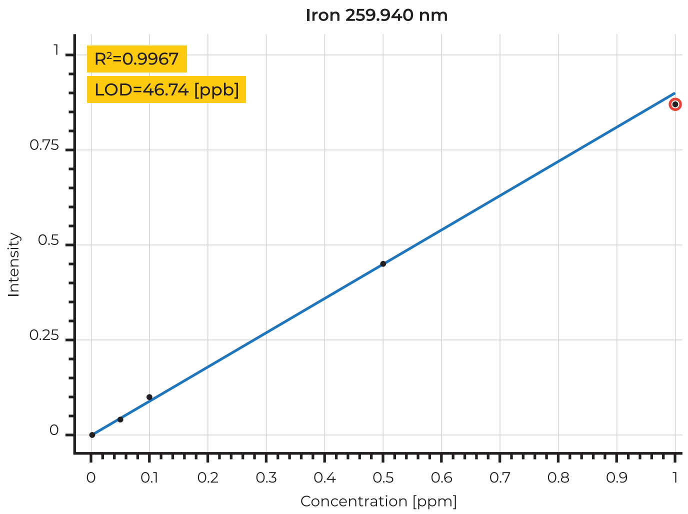
Cobalt 345.350 nm
R²=0.9963
LOD=4.414 [ppb]
Intensity
Concentration [ppm]



Chromium 360.534 nm
R²=0.9957
LOD=1.939 [ppb]
Intensity
Concentration [ppm]



Copper 327.396 nm
R²=0.9991
LOD=1.878 [ppb]
Intensity
Concentration [ppm]



Iron 259.940 nm
R²=0.9967
LOD=46.74 [ppb]
Intensity
Concentration [ppm]

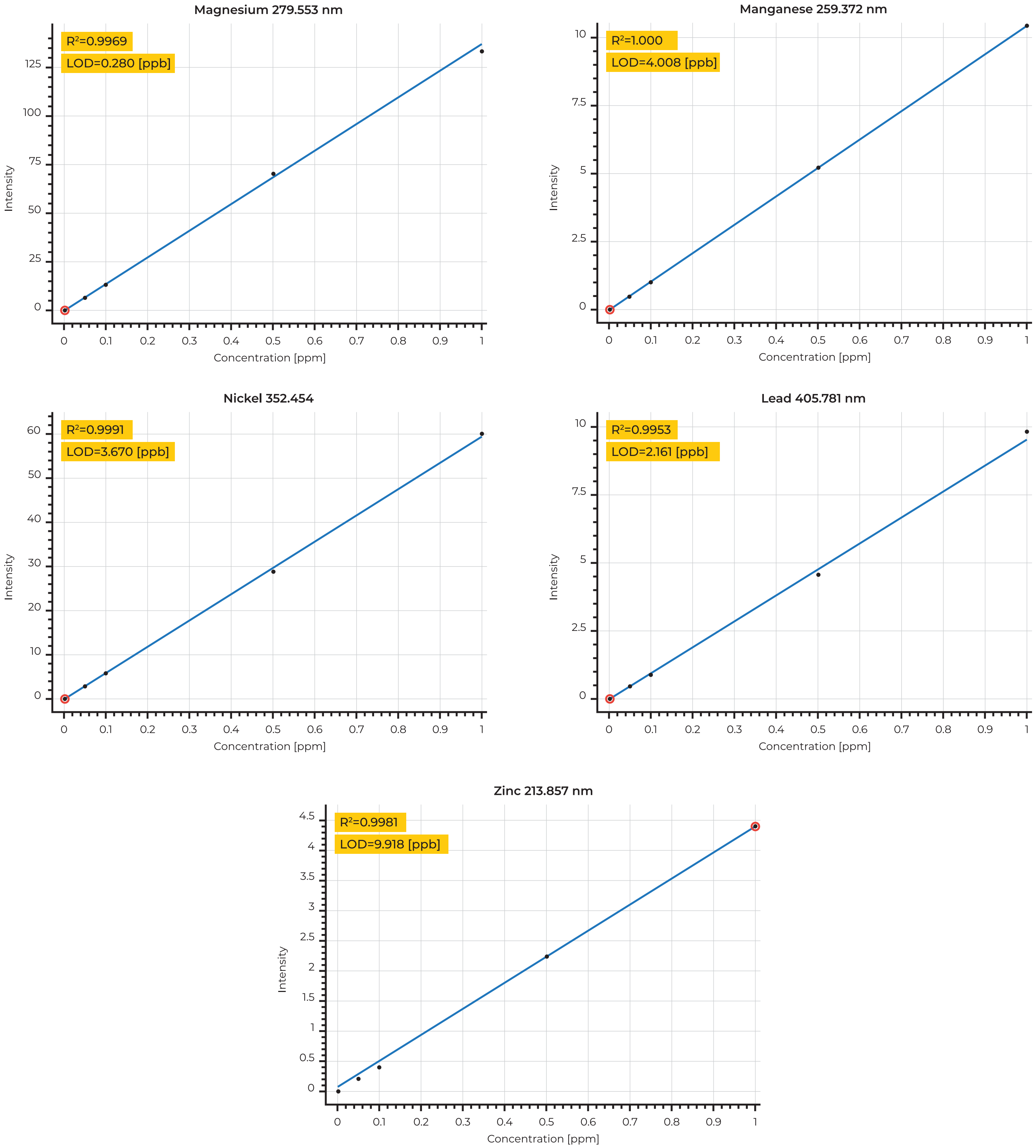


Figure 3. Typical Calibration Curves Examples

Result Summary and Discussion

The household bleach product sample weight and final volume weight were entered into the software. MICAP-OES 1000 is a simultaneous, echelle polychromator with CMOS detector. As a result, multiple wavelengths can be selected with no disadvantage to the required sample volume or time of analysis. Two wavelengths were selected for each target element. The results determined for calcium, chromium, lead, magnesium, and nickel were reported from the primary and secondary wavelengths as an example of performance from multiple emission lines.

Multiple quality control (QC) checks were prepared and analyzed to confirm the results, as no certified reference material was available. These included a Matrix Blank (MB), Laboratory Control Sample (LCS), a Matrix Spike (MS) and Matrix Spike Duplicate (MSD). The test solution analysis was completed by reanalyzing the standards as Continuing Calibration Verification (CCV) solutions.

Radom Intuitive Software (RIS) can insert these quality control solutions into the Session and automate the monitoring process. Figure 4 presents the QC Checks used in this method.

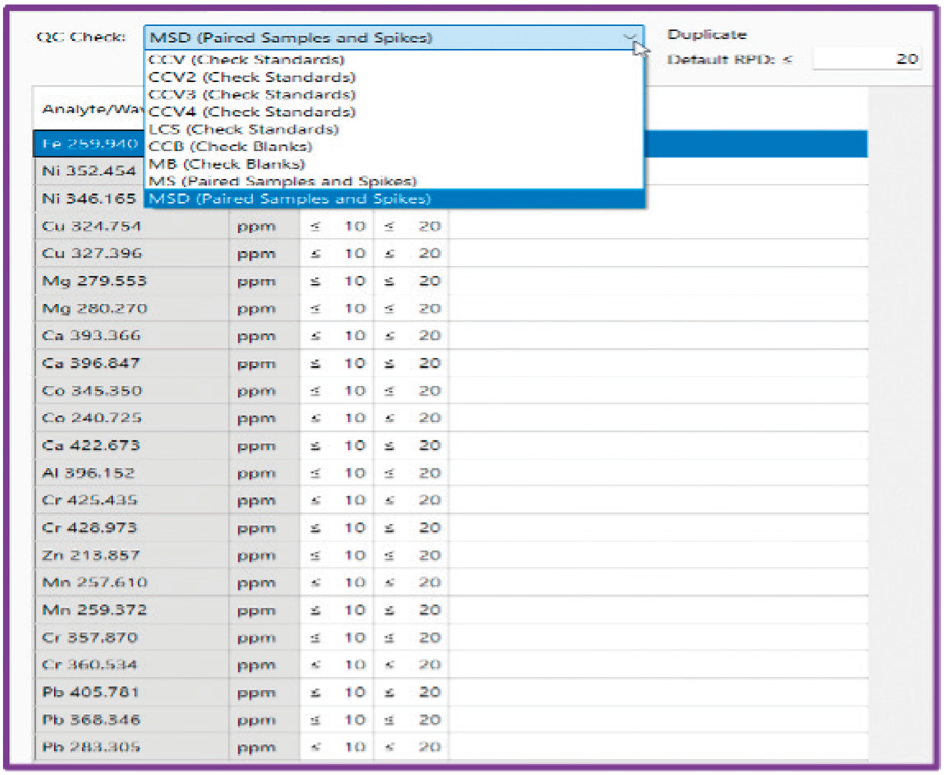


Figure 4. Quality Control Solutions Selected for Result Verification

The results determined for the eleven target elements in the commercial bleach product sample plus the Matrix Spike and Matrix Spike Duplicate are presented in Table 3. The matrix spike recovery was specified as ± 20% with the relative percent difference between the Matrix Spike and Matrix Spike Duplicate criterion was ± 20%. However, the typical recovery for all elements was less than ± 10% with an RPD of less than ± 3%.

Element and Wavelength (nm)	Matrix Blank MB	Matrix Blank Spike LCS	LCS Recovery (%)	Bleach Sample Conc. (ppm)	Bleach Sample Matrix Spike		Bleach Sample Matrix Spike Duplicate	
					Conc. (ppm)	% Recovery	Conc. (ppm)	% Recovery
Al 396.152 nm	< 0.050	0.454	91	0.207	5.884	114	6.118	118
Ca 393.366	0.054	0.525	105	0.405	5.506	102	5.619	104
Ca 396.847	0.054	0.517	103	0.390	5.437	101	5.531	103
Co 345.350	< 0.050	0.451	90	0.187	5.167	100	5.342	103
Cr 360.534 nm	< 0.050	0.443	89	0.103	5.58	110	5.829	115
Cr 428.973 nm	< 0.050	0.447	89	0.097	5.741	113	5.96	117
Cu 327.396	< 0.050	0.46	92	0.087	5.046	99	5.195	102
Fe 259.940	< 0.050	0.51	102	< 0.050	4.348	87	4.226	85
Mg 279.553	< 0.050	0.527	105	< 0.050	4.666	94	4.667	94
Mg 280.270	< 0.050	0.515	103	< 0.050	4.478	90	4.455	89
Mn 259.372 nm	< 0.050	0.52	104	< 0.050	4.601	90	4.537	89
Ni 346.165	< 0.050	0.466	93	< 0.050	4.922	99	5.039	101
Ni 352.454	< 0.050	0.467	93	< 0.050	4.922	101	5.055	104
Pb 283.305 nm	< 0.050	0.462	92	0.147	4.945	96	5.290	103
Pb 405.781 nm	< 0.050	0.441	88	0.121	4.967	97	5.233	102
Zn 213.857 nm	< 0.050	0.518	104	< 0.050	4.736	101	5.011	104

Table 3. Commercial Bleach Product Sample and MS/MSD Recovery Results

The standard readback recovery is presented in Table 4 below with most recovery within ± 10%. There was a slightly higher recovery for cobalt and iron observed, which likely occurred in the preparation of the solutions.

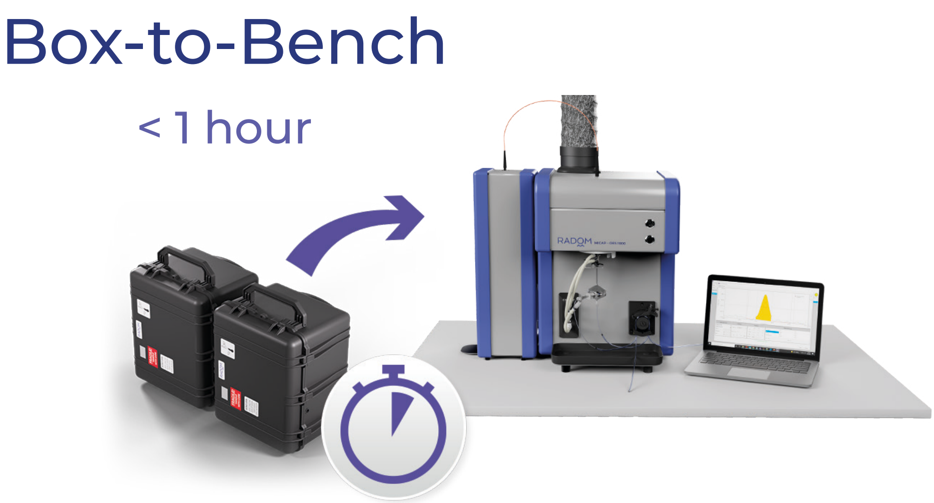
Element and Wavelength (nm)	% Recovery of Standard Readback Solution								
	CCV 1.00 (ppm)	% Recovery	CCV 0.500 (ppm)	% Recovery	CCV 0.100 (ppm)	% Recovery	CCV 0.050 (ppm)	% Recovery	CCB (ppm)
Al 396.152 nm	0.919	92	0.476	95	0.11	110	0.051	102	< 0.050
Ca 393.366	0.992	99	0.491	98	0.105	105	0.051	102	< 0.050
Ca 396.847	0.967	97	0.487	97	0.109	109	0.049	98	< 0.050
Co 345.350	0.96	96	0.466	93	0.108	108	0.057	114	< 0.050
Cr 360.534 nm	0.955	96	0.463	93	0.109	109	0.052	104	< 0.050
Cr 428.973 nm	0.902	90	0.47	94	0.111	111	0.054	108	< 0.050
Cu 327.396	0.961	96	0.47	94	0.104	104	0.053	106	< 0.050
Fe 259.940	1.045	105	0.534	107	0.092	92	0.050	101	< 0.050
Mg 279.553	0.985	99	0.528	106	0.099	99	0.049	98	< 0.050
Mg 280.270	1.019	102	0.507	101	0.099	99	0.05	100	< 0.050
Mn 259.372 nm	1.028	103	0.523	105	0.101	101	0.052	104	< 0.050
Ni 346.165	0.966	97	0.479	96	0.109	109	0.055	110	< 0.050
Ni 352.454	0.975	98	0.478	96	0.099	99	0.054	108	< 0.050
Pb 283.305 nm	0.981	98	0.476	95	0.102	102	0.050	100	< 0.050
Pb 405.781 nm	0.948	95	0.462	92	0.111	111	0.046	92	< 0.050
Zn 213.857 nm	0.994	99	0.507	101	0.110	110	0.044	88	< 0.050

Table 4. Standard Readback Recovery

Conclusions

MICAP-OES 1000 powered by Cerawave™ technology is designed to generate a robust, stable nitrogen plasma even with tough matrices. The bleach matrix and NaCl matrix matching (1.14%) provides a great example of this robustness.

The MICAP system is customer installable with its "Box-to-Bench" design. The optimized method for the determination of Al, Ca, Co, Cr, Cu, Fe, Mg, Ni, Pb and Zn in commercial bleach products has already been created. The only component missing is you.



References

1. ProTank Storage Tanks and Specifications; https://www.protank.com/sodium-hypochlorite; Alek Eccles, Protank Chemical Extraordinaire

Scan the code to access our website for additional information and our resources

For any questions or additional resources:

inquiries@radomcorp.com or call 877-977-2366