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.

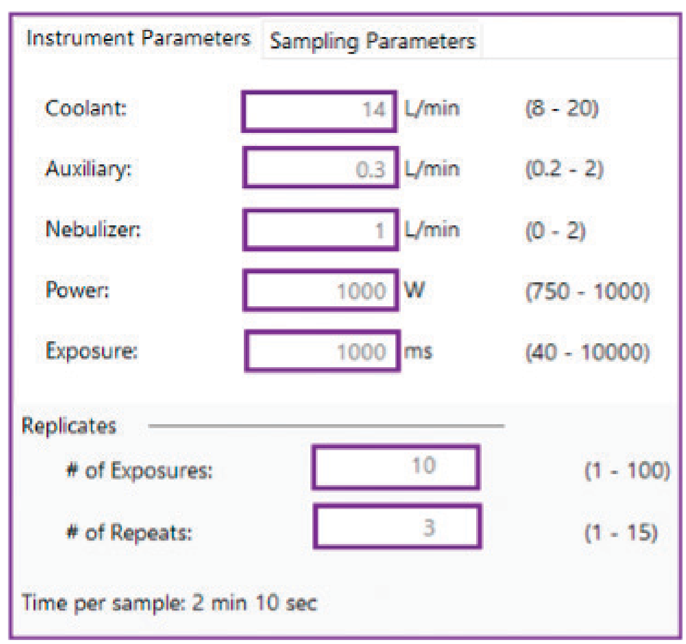
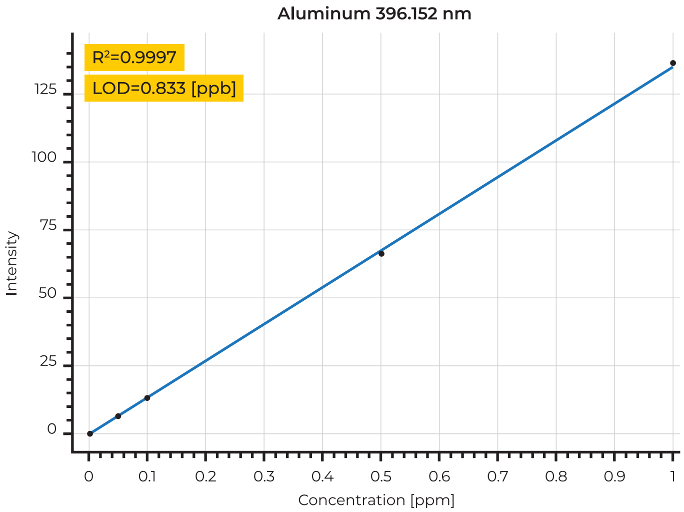
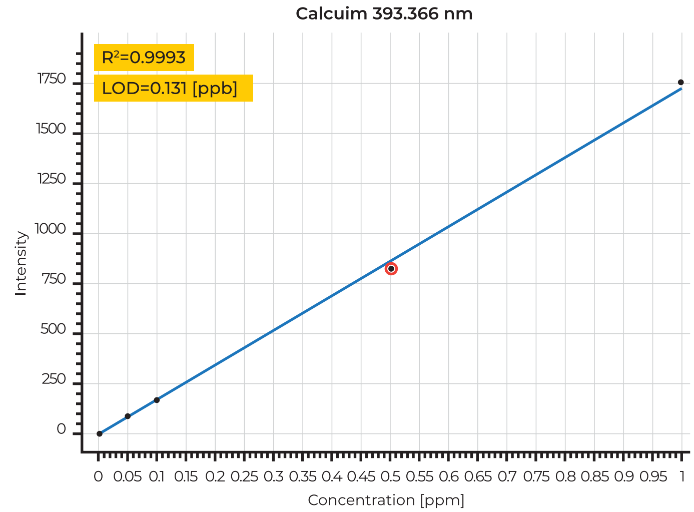
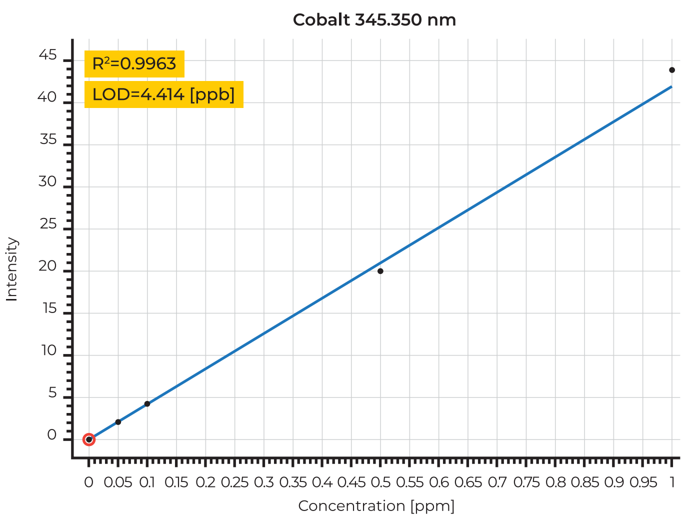
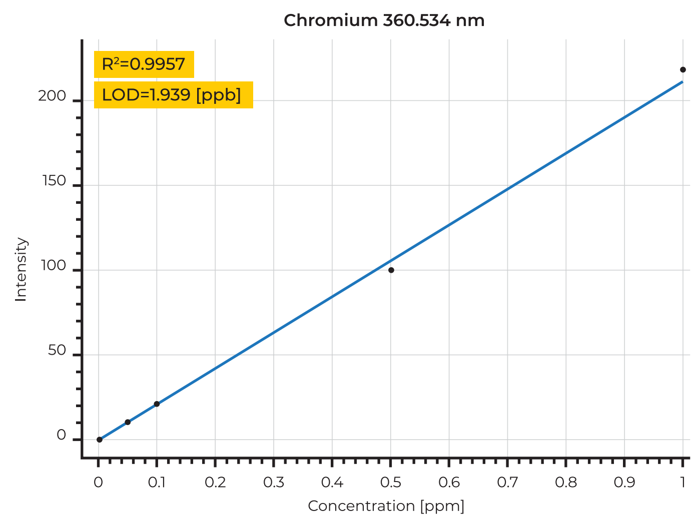


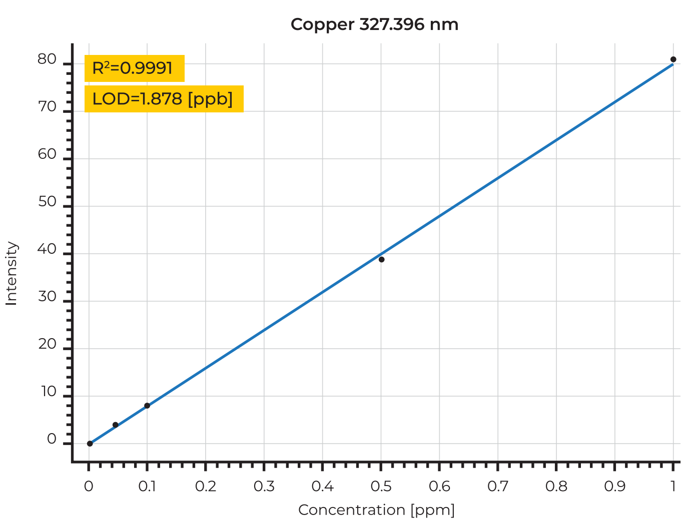

**Figure 2. Plasma and Measurement Conditions**

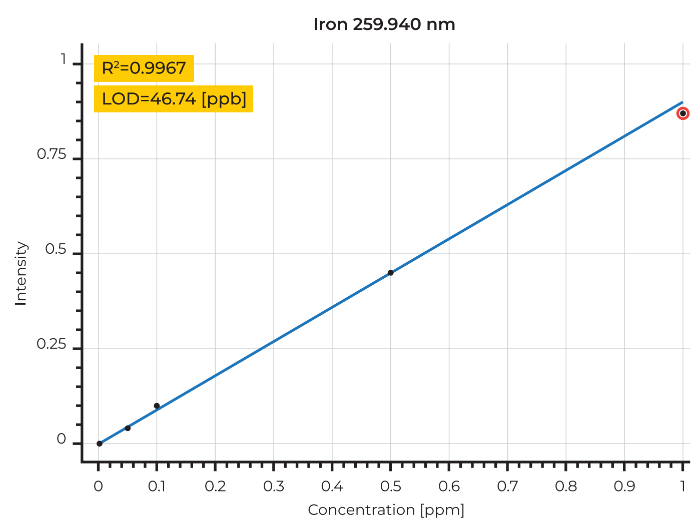

Aluminum 396.152 nm
R²=0.9997
LOD=0.833 [ppb]
Intensity
Concentration [ppm]


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]

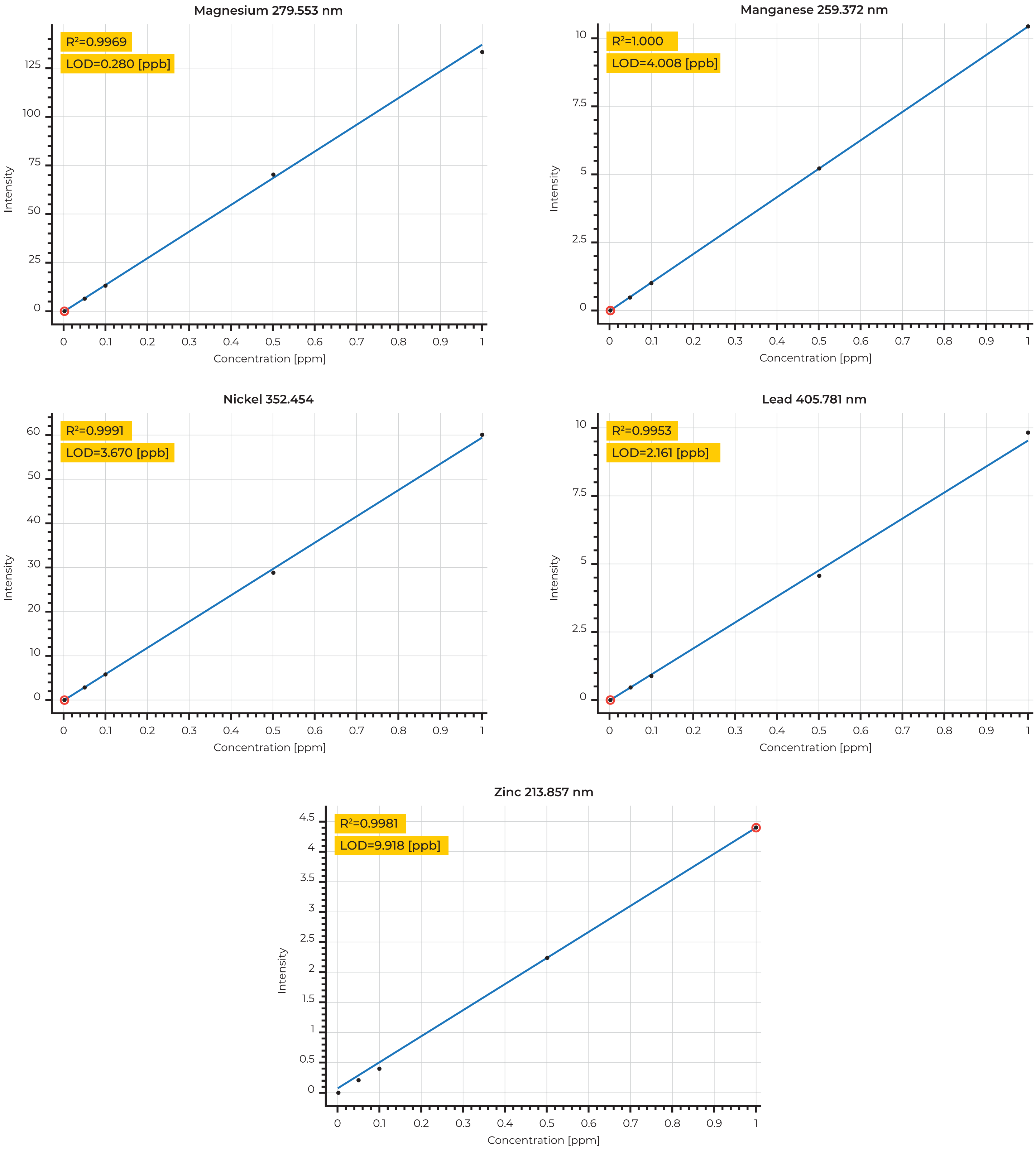



**Figure 3. Typical Calibration Curves Examples**

## 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.

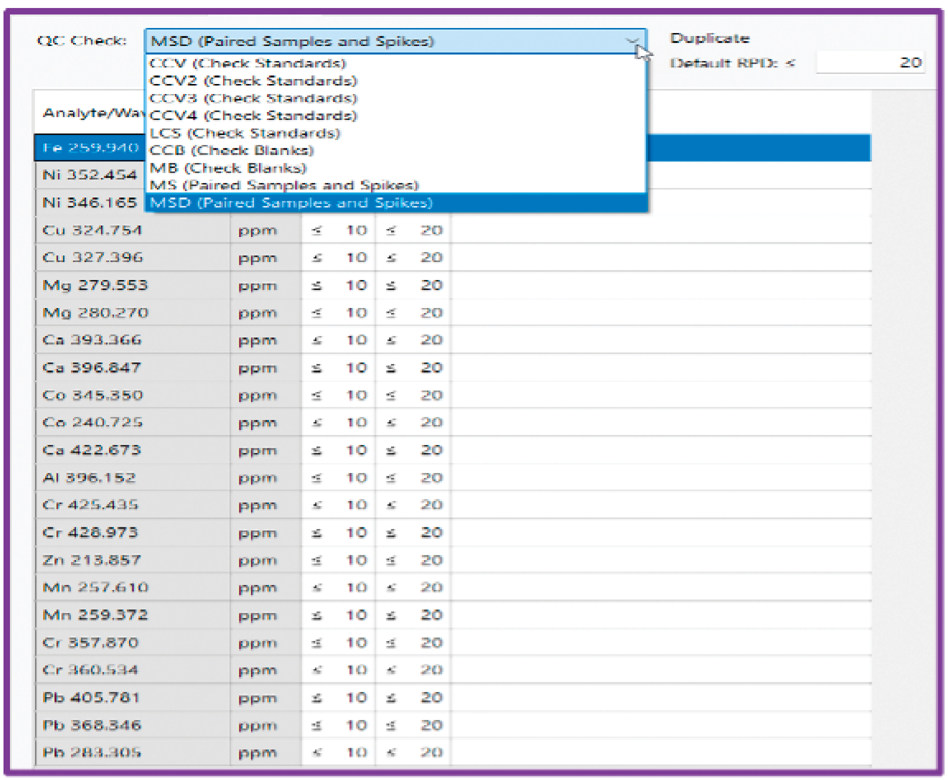



**Figure 4. Quality Control Solutions Selected for Result Verification**

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.

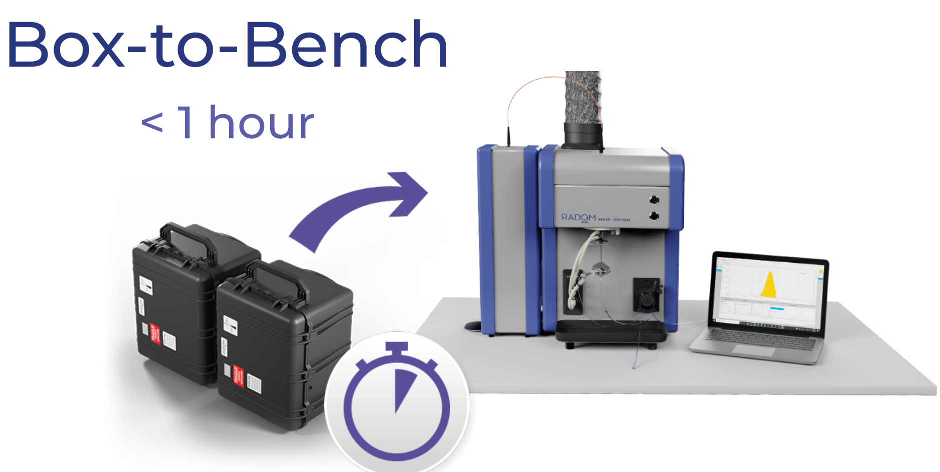
| Element and Wavelength (nm) | % Recovery of Standard Readback Solution | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | CCV 1.00 (ppm) | % Recovery | CCV 0.500 (ppm) | % Recovery | CCV 0.100 (ppm) | % Recovery | CCV 0.050 (ppm) | % Recovery | CCB (ppm) |
| Al 396.152 nm | 0.919 | 92 | 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