





All samples were weighted into the MAXI-24 HP vessel, approximately 0.5 g (as reported in table 1), then they were fortified with 100 µL of a multi-elemental standard solution (As/Cd/Pb/Ag/ Co/Cu/Ni/Se/Tl/V). For the spiked final concentrations, please, refer to table 3.

| STEP | TIME     | T2     | POWER  |
|------|----------|--------|--------|
| 1    | 00:10:00 | 160 °C | 1800 W |
| 2    | 00:15:00 | 210 °C | 1800 W |
| 3    | 00:10:00 | 210°C  | 1800 W |

5 B C M F " 9 \* ) 1 N J D S P Q X B W F S B N

After microwave digestion, the samples were diluted to 50 mL with DI water and then, analyzed in ICP-OES. Parallel procedure was performed on unspiked samples in order to calculate the recovery rate in relation to the spiked levels.

#### QUANTIFICATION

ICP-OES Instrumental Parameters: RF power (W): 1300; Plasma flow (L/min): 15.0; Auxiliary Flow (L/min): 1.5; Nebulizer Flow (L/min): 0.75; Replicate read time (s): 10; Instrument stabilization delay (s): 15; Sample Uptake Delay (s): 30; Pump Rate (rpm): 15; Rinse Time (s): 10; Replicates: 3.

#### RESULTS AND DISCUSSION

The performance of the Milestone ETHOS UP equipped with MAXI-24 HP rotor was evaluated through a recovery study on samples of interest for the pharma industry. All samples were fortified with a spike solution (Elemental Impurities according to ICH Q3D parenteral, Standard 1, ICP standard. Sigma-Aldrich code 89118-100ML) containing As, Cd, Co, Pb, Hg, Ni, Se, Ag, Tl and V (see spike concentration in Table 3). The samples were digested with Milestone's Ethos UP and subsequently analyzed via ICP-OES.

' J H V S F 9 \* ) 1 . J D S P X B W F 3 V O 3 F ( U F N Q F S B U V S F U S B D F E

Figure 3 shown the temperature profile of the digestion as well as the multiple temperature visualization and recording for all the samples digested.

# INDUSTRY REPORT

## ETHOS UP – MAXI 24 HP | PHARMACEUTICAL



|                           | As    | Cd   | Co   | Pb    | Hg <sup>a</sup> | Ni    | Se    | Ag    | Tl   | V     |
|---------------------------|-------|------|------|-------|-----------------|-------|-------|-------|------|-------|
| Spike value (mg/L)        | 15    | 2    | 5    | 5     | 3               | 20    | 80    | 10    | 8    | 10    |
| <b>Avicel PH-101</b>      |       |      |      |       |                 |       |       |       |      |       |
| Recovery % (n=3)          | 103.7 | 92.5 | 94.3 | 93.1  | 99.6            | 101.1 | 110.9 | 92.5  | 91.7 | 103.6 |
| RSD (%)                   | 0.7   | 1.9  | 1.2  | 2.5   | 1.8             | 2.6   | 2.4   | 1.4   | 2.1  | 2.9   |
| <b>Magnesium Stearate</b> |       |      |      |       |                 |       |       |       |      |       |
| Recovery % (n=3)          | 103.5 | 95.9 | 92.7 | 109.8 | 94.0            | 99.4  | 108.7 | 99.5  | 91.6 | 106.0 |
| RSD (%)                   | 1.5   | 2.1  | 1.3  | 1.1   | 1.8             | 1.5   | 2.3   | 2.4   | 2.1  | 1.2   |
| <b>Ibuprofen</b>          |       |      |      |       |                 |       |       |       |      |       |
| Recovery % (n=3)          | 113.4 | 98.7 | 95.4 | 105.6 | 94.7            | 97.8  | 107.1 | 108.4 | 94.3 | 102.4 |
| RSD (%)                   | 0.9   | 1.5  | 0.8  | 2.2   | 2.1             | 1.7   | 1.4   | 1.8   | 2.1  | 1.2   |
| <b>Salicylic acid</b>     |       |      |      |       |                 |       |       |       |      |       |
| Recovery % (n=3)          | 106.5 | 93.6 | 94.1 | 105.7 | 97.9            | 97.1  | 110.9 | 96.9  | 97.0 | 106.8 |
| RSD (%)                   | 1.2   | 0.9  | 1.4  | 1.7   | 2.5             | 2.1   | 1.4   | 1.1   | 1.0  | 1.6   |

Table 3- Data of the recovery study on spiked samples of interest for Pharma

The analytical results were shown in table 3 with good recoveries of all elements and RSDs below 3%. This demonstrates the robustness and reproducibility of microwave digestion using the ETHOS UP equipped with MAXI-24 HP easyTEMP technology.

### CONCLUSION

The data shown in this industry report demonstrates full recovery of the elements spiked in the samples. The ETHOS UP with MAXI-24 was successfully applied in digesting samples, such as those from the pharmaceutical industry, even in large sample amounts.

Milestone ETHOS UP and MAXI-24 HP rotor provide the highest level of reproducibility, great ease of use and high productivity in alignment with the new USP

requirements. In addition, the Milestone ETHOS UP with easyTEMP control and easyCONTROL software, provides the highest level of reproducibility and traceability in perfect alignment with GMP regulations.

### ABOUT MILESTONE

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C H E M I S T S

MILESTONE Srl - Via Fatebenefratelli, 1/5 - 24010 Sorisole (BG) - Italy  
Tel: +39 035 573857 - Fax: +39 035 575498  
[www.milestonesrl.com](http://www.milestonesrl.com) - email: [analytical@milestonesrl.com](mailto:analytical@milestonesrl.com)