

# The Completed Dissolution of Pharmaceuticals, Using And Omitting HF

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## ABSTRACT

Researchers from CEM have worked to determine if it is possible to recover trace metals without the use of hydrofluoric acid (HF) in sample preparation. Normally, total dissolution of a sample matrix is critical to ensure accurate data, and this study investigated if it was absolutely necessary for a total recovery of analytes of interest. All samples were prepared with a MARS™ 6 microwave digestion unit and analyzed with an ICP-OES, outfitted with an HF introduction system.

## EXPERIMENTAL PROCEDURE

NIST SRM 3280 was ground into a homogeneous mixture, using a mortar and pestle. Then, 0.25g of the sample was transferred to an EasyPrep™ Plus vessel. The sample was either digested using HNO<sub>3</sub>, HCl, and HF, or without the use of HF. The heating program was the CEM One Touch™ Pharmaceutical method (Table 1). After digestion, samples were then quantitatively transferred to a 50 mL class 1 volumetric flask, where they were prepped for analysis on our ICP-OES.

Table 1. Digestion Conditions for MARS 6

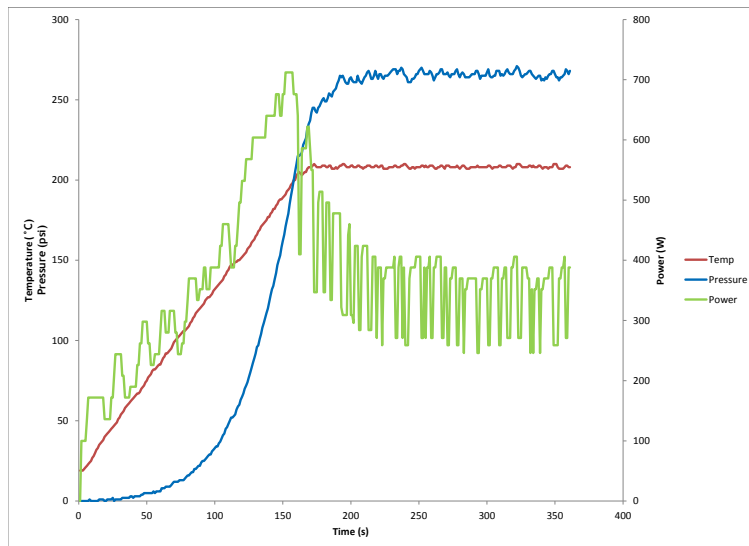
Temperature (° C)	Ramp (mm:ss)	Hold (mm:ss)	Pressure (psi)	Power (W)
210	15:00	15:00	800	1800

## BORIC ACID MAY BE NEEDED

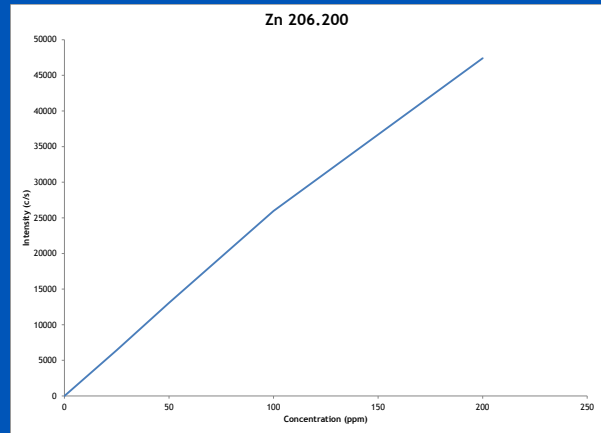
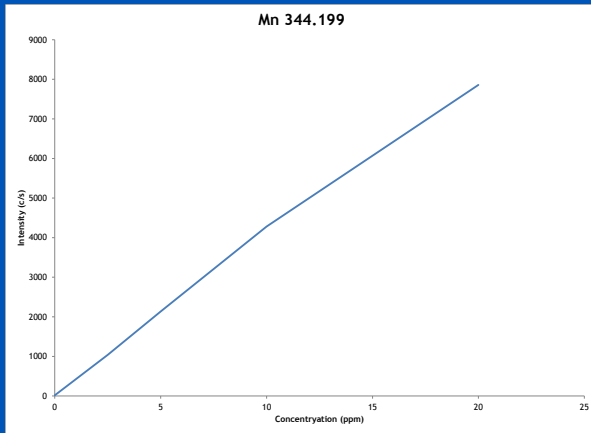
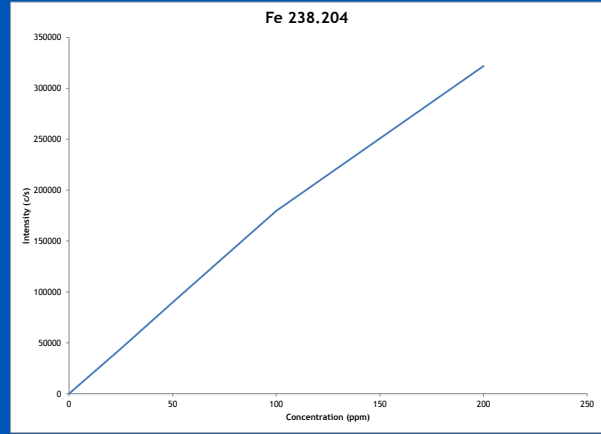
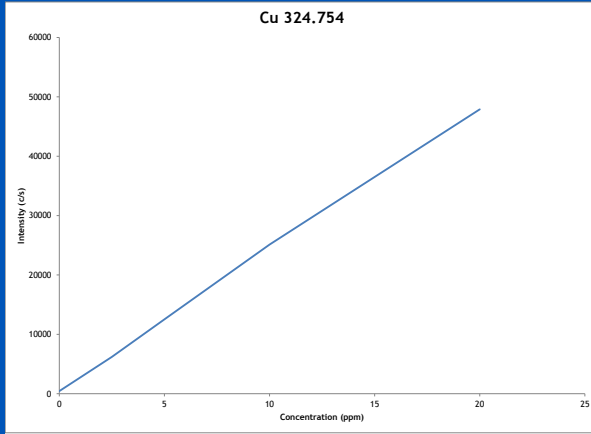
After digestion of samples with HF, there were particles left. These particles could be due to elemental interactions with the F- ion, specifically Ca and Al. The use of H<sub>3</sub>BO<sub>3</sub> was employed to clear up the solution for total digestion. This will yield the total digestion that is called for in USP 232. Iron seemed to be bound as an Iron fluoride compound, as shown in the data, resulting in poor recoveries. Once the boric acid was added, recoveries went back to the acceptable range.



## TYPICAL HEATING CURVE FOR THE MARS 6 METHOD USED



# CALIBRATION CURVES



## DATA

	Cu 324.754	Fe 238.204	Mn 344.199	Zn 206.200
<b>HNO<sub>3</sub> and HCl</b>				
Sample 1	1271.78	11711.30	1388.59	9672.4
Sample 2	1334.13	12203.00	1642.18	10208.6
Sample 3	1211.63	11311.40	1303.61	9622.93
Average	1272.51	11741.90	1444.79	9834.64
% Recovery	90.89	95.08	100.33	96.89
STDEV	61.25	446.59	176.14	324.80
%RSD	4.81	3.80	12.19	3.30
<b>HNO<sub>3</sub>, HCl, and HF</b>				
Sample 1	1224.21	8692.63	1406.75	10713.1
Sample 2	1304.00	8539.03	1629.47	10400.5
Sample 3	1327.40	8465.76	1409.07	10519.7
Average	1285.20	8565.81	1481.76	10544.43
% Recovery	91.80	69.36	102.90	103.89
STDEV	54.10	115.78	127.92	157.76
%RSD	4.21	1.35	8.63	1.50
<b>HNO<sub>3</sub>, HCl, HF, and H<sub>3</sub>BO<sub>3</sub></b>				
Sample 1	1334.56	11086.90	1335.31	10121.8
Sample 2	1522.89	11237.90	1463.37	10142.4
Sample 3	1401.45	13620.40	1587.63	10231.5
Average	1419.63	11981.73	1462.10	10165.23
% Recovery	101.40	97.02	101.53	100.15
STDEV	95.47	1421.13	126.16	58.31
%RSD	6.73	11.86	8.63	0.57

## CONCLUSION

For these elements of interest and within this sample matrix, there doesn't seem to be any benefit from adding in the hydrofluoric acid. In fact, the HF seems to actually cause a hindrance to the recoveries of some elements, because of interactions with the fluoride ion, causing an insoluble fluoride, in this case iron. High % RSD for the last set of data may be due to the high dissolved solids, caused by the addition of the boric acid.

In the path forward, more elements will be looked into and different sample matrices will be examined to see if this trend continues. These samples have presented their own unique challenges, which must be addressed by follow-up studies.

