

APPLICATION NOTE

NIST 1515 - Apple Leaves

SUMMARY

The application note summarizes the digestion of NIST 1515, an apple leaves standard reference material using ColdBlock™ Digestion Pro Series Technology.

Instrument: ColdBlock CBM (with quartz test tubes), chiller, ICP-MS & ICP-OES

Published: July 2024

Digestion Time: 20 Minutes

Acid Used: HNO₃ & H₂O₂

Average ColdBlock Recovery vs. CRM:

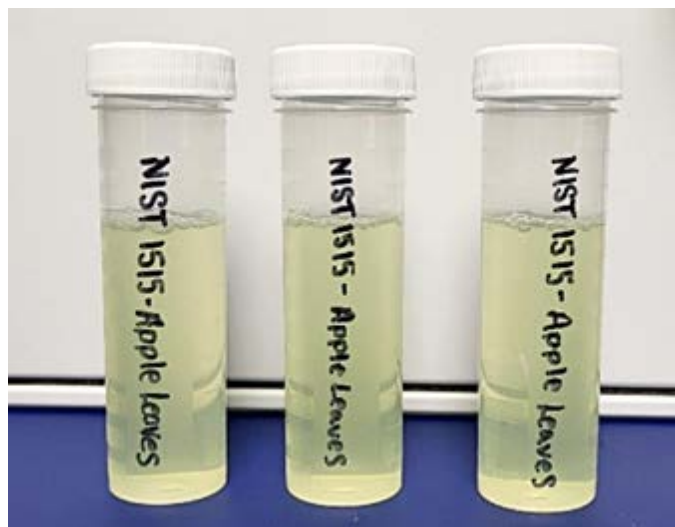
- 103% Boron
- 100% Phosphorus
- 99% Potassium

METHODOLOGY

1. Chiller temperature was set to -5°C
2. 0.5g of each sample was weighed and placed into a quartz ColdBlock™ Digestion vessel
3. 10 mL of HNO₃ was added
4. Sample was digested at 65% power for 20 minutes
5. 2mL of ≥ 30% H₂O₂ was added slowly
6. Samples were cooled and bulked to 40mL using 2% HNO₃_{v/v}

DISCUSSION

- Samples were digested in triplicate
- Samples were filtered prior to analysis by ICP-MS
- The NIST methods used for determination of certified Aluminum values included INAA & ICP-OES using HNO₃/HF/HClO₄ digestion, so Aluminum recovered slightly low (~70%) using HNO₃ & H₂O₂
- To improve the recovery the Aluminum, a second method was examined that included the addition of 1mL HF (10mL HNO₃ + 1mL HF 65% power for 20 minutes, then slowly add 2mL of 30% H₂O₂) See Table 2 for results
- NIST 1515 consists of dried apple leaves of the Golden Delicious and Rome varieties



NIST 1515 after bulk up to 40mL

NIST 1515 - Apple Leaves

Results

Table 1 - Results of HNO₃ & H₂O₂ digestion - NIST 1515 Apple Leaves

Method:	0.5g	10mL HNO ₃ 65% power for 20 minutes, then slowly add 2mL of 30% H ₂ O ₂							
Element	Consensus Value (ppm)	+/-	Sample A	Sample B	Sample C	Average (ppm)	Stdev	RSD	Recovery
B	27.6	2.8	26.8	29.0	29.7	28.5	1.3	4.4%	103%
Ba	48.8	2.3	44.8	51.8	48.2	48.3	2.8	5.9%	99%
Ca	15250	100	14001	16218	15042	15087	906	6.0%	99%
Cd	0.0132	0.0015	0.0148	0.0137	0.0125	0.0137	0.001	6.9%	104%
Cu	5.69	0.13	5.81	5.74	5.44	5.66	0.2	2.8%	100%
Fe	82.7	2.6	82.6	85.3	92.5	86.8	4.2	4.8%	105%
Hg	0.0432	0.0023	0.0423	0.0418	0.0426	0.0422	0.0004	0.9%	98%
K	16080	210	14670	17110	15878	15886	996.2	6.3%	99%
Mg	2710	120	2510	2929	2699	2713	171	6.3%	100%
Mn	54.1	1.1	59.1	59.4	55.0	57.8	2.0	3.5%	107%
Mo	0.095	0.011	0.108	0.106	0.105	0.106	0.001	1.2%	112%
P	1593	68	1572	1585	1617	1591	18.9	1.2%	100%
Pb	0.47	0.024	0.48	0.48	0.44	0.47	0.02	4.0%	99%
Rb	10.2	1.6	9.5	9.5	9.2	9.4	0.1	1.4%	92%
Sr	25.1	1.1	21.9	25.5	23.9	23.8	1.5	6.1%	95%
V	0.254	0.027	0.300	0.282	0.298	0.293	0.01	2.8%	115%
Zn	12.45	0.43	13.20	12.28	12.28	12.59	0.4	3.4%	101%

Table 2 - Results of HNO₃ + HF & H₂O₂ digestion (to improve recovery of Aluminum)

Method:	0.5g	10mL HNO ₃ + 1mL HF power for 20 minutes, then slowly add 2mL of 30% H ₂ O ₂							
Element	Consensus Value (ppm)	+/-	Sample A	Sample B	Sample C	Average (ppm)	Stdev	RSD	Recovery
Al	284.5	5.8	249.8	285.2	246.1	260.4	17.6	6.8%	92%