



STAR: Larger Samples/Higher Temperatures

Open Vessel Acid Digestion with Automatic Reagent Addition and Vapor Handling for Samples Requiring High Temperature, Large Sample Sizes, or Serial Reagent Addition

Executive Summary:

The STAR™ Open Vessel Microwave Digestion System allows for digestion of the largest sample sizes with the highest working temperatures, plus the ability to automatically add reagents throughout the digestion process. Individual cells operate independently of each other, allowing different methods to run simultaneously or with staggered start times. The STAR System combines precise microwave heating with independent temperature feedback control, accurate programmable reagent delivery, safe vapor removal, and post-digestion evaporation/concentration. Representative digestions include plant and animal tissues, environmental samples, foodstuffs, oils, polymers, paper, Toray filters, rubber, paints, resins, and adhesives.

Introduction:

Open vessel digestion systems can handle larger sample sizes and those that require high decomposition temperatures. Large organic samples evolve significant volumes of gas upon digestion; however, STAR utilizes an innovative fume scrubber system to contain hazardous vapors. Because high temperature quartz vessels can be used (as well as Pyrex® and Teflon®), the temperature limits are higher. In addition, non-contact IR temperature technology eliminates any temperature limitation imposed by some types of fiber optic probes.

Since some acid will be lost with high operating temperatures and longer run times, the STAR System includes programmable reagent addition to ensure that the sample is continually covered in acid during the digestion process. Automatically adding reagents in small aliquots while the digestion is running also prevents the digestate from cooling.

Digestion of many organic samples is accomplished with a two-stage procedure: charring the sample matrix first with sulfuric acid, followed by oxidation with nitric acid and hydrogen peroxide. In some cases, it is desirable to concentrate or remove excess acid following the digestion.

For these reasons, the STAR System consists of a microwave heating system with individual temperature feedback control for each digestion cell (using patented non-contact IR), individual reagent pumps for each reagent in each cell, and a vapor handling system that captures and neutralizes digestion gases and products. Sample digestion takes place in quartz, Pyrex, or Teflon vessels. Quartz is required for high temperature work.

The result is that up to six independent digestions can be carried out simultaneously or sequentially using the STAR System, with each sample having its own program of multiple ramped heating steps, hold times, acid additions, and finally evaporation/concentration protocols.

In practice, the execution of a digestion is simple. The operator selects the appropriate method, inserts the digestion vessel containing the weighed sample, and presses START. The STAR System performs all other necessary operations unattended.

STAR Open Vessel Digestion System

- Process up to six samples independently using different methods at simultaneous or sequential start times
- Automated reagent addition
- One pump per reagent for each individual cell
- Pre-programmable "Time-to-Temperature" parameters for rapid optimization of methods
- Automated evaporation of sample and reagents to dryness following digestion
- Self-contained scrubber system frees fume hood space
- Easily digest difficult or highly reactive samples in sizes up to 10g
- Meets requirements of USEPA SW-846 Method 3050B



Experimental

There are four generic programs to cover most sample types: Mild Digest, Moderate Char, Rigorous Char, and Super Char. The Mild Digest adds nitric acid, heats the sample to 110 °C, and adds hydrogen peroxide to complete the oxidation. The Char methods first add sulfuric acid and heat to 280 °C (250 °C for Moderate Char) to char the sample matrix. The digest is then cooled to 250 °C (200 °C for Moderate Char) and small aliquots of nitric acid are added. Finally, hydrogen peroxide is added at 200 °C to complete the oxidation. Methods can be easily edited to optimize reagent volume and time to better fit the specific sample matrix or sample size.

STAR System Programs

- Mild Digest - Agricultural, Biological, Environmental, Paper
- Moderate Char - Agricultural & Biologicals with H₂SO₄, Light Oils, Foods, Plastics, Environmental, Paints, Solvents
- Rigorous Char - Oils, Polymers/Plastics, Resins, Solvents, Adhesives, Organic Chemicals, Asphalts, Fuel
- Super Char - Samples larger than 2 grams

Mild Digest Method

Initial Reagent: 10 mL HNO₃

Stage	Ramp Time	Target Temp °C	TAP (min)	Reagent (mL)	Aliquot (mL)	Add at Start
1	3:00	110	10	None	0	No
2	0:01	110	5	10 mL H ₂ O ₂	1	No

TORT-2 Lobster Hepatopancreas - 1 gm*

	STAR System™ ppm	Certified ppm	Uncertainty
As	21.0	21.6	1.8
Cd	25.8	26.7	0.6
Cr	1.11	0.77	0.15
Co	0.54	0.51	0.09
Cu	103	106	10
Fe	97.4	105	13
Pb	0.33	0.35	0.13
Mn	12.3	13.6	1.2
Hg	0.31	0.27	0.06
Ni	2.79	2.50	0.19
V	1.7	1.64	0.19

*Air condenser

Montana Soil I - SRM 2710

Method 3050 w/HCl
1 Gram Sample : n = 6

	Al Wt%	As PPM	Cd PPM	Cr PPM	Cu PPM	Fe Wt %	Mn PPM
Average	1.34	553.0	18.20	18.20	2811	2.43	6267
Std Dev.	0.21	35.1	1.20	3.86	201	0.20	319
Low Ref	1.2	490.0	13.0	15	2400	2.2	6200
High Ref	2.6	600.0	26.0	23	3400	3.2	9000

Moderate Char

Initial Reagent: 10 mL HNO₃
5 mL H₂SO₄

Stage	Ramp Time	Target Temp °C	TAP (min)	Reagent (mL)	Aliquot (mL)	Add At Start
1	3	120	1	None	0	No
2	3	250	5	10 mL HNO ₃	1	No
3	0	200	10	20 H ₂ O ₂	1	No

Bovine Muscle SRM-1572

n = 6

	Certified Value	SD	Recovered Value	SD
Cu	2.84	0.45	2.77	0.13
Fe	71.2	9.2	65.6	2.2
Mn	0.37	0.09	0.31	0.01
Ni	0.05	0.04	0.09	0.06
Pb	0.38	0.24	0.33	0.05

Rigorous Char

Initial Reagent: 10 mL HNO₃
10 mL H₂SO₄

Stage	Ramp Time	Target Temp °C	TAP (min)	Reagent (mL)	Aliquot (mL)	Add At Start
1	3:00	120	1:00	None	0	No
2	3:00	250	0:10	2 mL HNO ₃	2	No
3	1:00	280	0	None	0	No
4	0	250	10:00	20 mL HNO ₃	1	No
5	0	200	10:00	20 mL H ₂ O ₂	1	No

Wear Metals in Oil - SRM 1083a 5 grams*

	STAR System™ ppm	Certified Value ppm	Range
Cu	96.0	98	94 - 102
Mg	95.7	98	94 - 102
Mo	95.7	97	92 - 102
Ni	96.3	101	97 - 105
Ti	98.4	99	98 - 100

*Air condenser

Conostan Oil Metallo-Organic Standard 100 ppm Arsenic - 1g*

Replicate	STAR System™ As (ppm)
1	98
2	101
3	100
4	99
5	100
Average	100
Std. Dev.	0.86
%RSD	0.9

*Air condenser

Super Char

Initial Reagent: 10 mL HNO₃; 10mL H₂SO₄

Stage	Ramp Temp °C(min)	Target (mL)	TAP (mL)	Reagent Start	Aliquot	Add At	Time
1	3:00	120	1:00	None	0	No	
2	1:30	150	5:00	10 mL HNO ₃	1	No	
3	1:30	175	5:00	10 mL HNO ₃	1	No	
4	1:30	200	5:00	10 mL HNO ₃	1	No	
5	1:30	225	5:00	10 mL HNO ₃	1	No	
6	1:30	250	5:00	10 mL HNO ₃	1	No	
7	2:00	280	0:00	1.5mL HNO ₃	1.5	Yes	
8	0:00	250	10:00	20 mL HNO ₃	1	No	
9	0:00	200	10:00	20 mL H ₂ O ₂	1	No	

Infant Formula 2 grams*

	STAR ppm	RSD %	Labeled Value
Ca	548	0.7	493
Cu	0.75	1.3	0.61
Fe	12.9	2.2	12.2
K	985	1.1	710
Mg	57.3	40.6	
Na	172	1.5	183
P	406	9.2	379
Zn	8.2	21	5.1

*Air condenser

Low Density PolyEthylene (LDPE) Plastic - 1g*

	STAR System™ ppm	QC Value ppm	Uncertainty
Al	474.4	466.5	4.3
Ca	36.9	32.0	0.6
Mg	9.6	9.6	0.1
Ti	21.1	20.9	0.3

*Air condenser

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