



# New Techniques for Preparing Plastics and Polymers by Microwave Sample Preparation

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## Abstract

Traditional acid digestion of many polymers have typically involved the use of a sulfuric acid char step in order to dehydrate the polymer and break the cross linkages. This is followed by a second step of oxidation with nitric acid. This is a tedious process and the sulfuric acid step raises the viscosity of the liquid which is not friendly to ICP-MS analysis. Other techniques such as dry ashing samples prior to taking up in dilute acids and very costly high pressure apparatus have also been attempted but each has their own difficulties.

We will prepare a variety of polymeric samples by traditional cost effective microwave digestion without the use of sulfuric acid or dry ashing steps. Analysis will be reported using ICP-OES and ICP-MS analysis. Spike recovery data as well as data derived from standard reference materials will presented and discussed.

## The Solutions

### Mars 6 iPrep



- System can operate at high temperatures and pressures without sulfuric acid.
- Easy to assemble, frame, sleeve, plug and cap.
- Operates by infrared sensor only - no fiber optic to worry about
- Can run up to 12 vessels simultaneously
- Teflon® vessels allow for low level detection limit.

### iPrep vessel



- Digests the most difficult organic samples
- Twice the sample size for most organics
- Precisely regulates venting to ensure volatile elements are retained

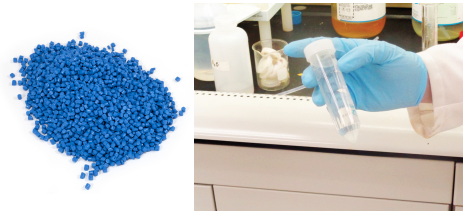
## Samples Investigated

- Low Density Polyethylene- 680K and 681K
- Additive Elements in Polyethylene – (NIST SRM 2855)
- Polyvinyl Chloride

## Method Parameters

Sample Type	Final Hold Temperature	Total Run Time MARS 6 iPrep
LDPE - 680 K, 681 K	200 °C	40 min.
PVC	210 °C	45 min.
PE (NIST 2855)	200 °C	40 min.

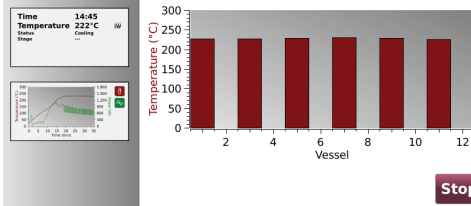
## Completely Digested Sample



Before digest

After digest

## Run - PET 230C



2:46 PM

## PE and PVC results

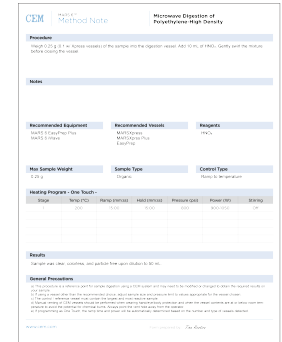
10 ppm spike	As 188,980	As 193,696	Cd 214,439	Cd 226,502	Cr 205,560	Cr 267,716	Pb 182,143	Pb 220,353
PE	8,928	8,468	8,802	8,832	8,904	9,112	9,140	9,028
PE	9,520	9,109	9,101	9,085	9,214	9,384	8,884	9,204
PE	8,938	9,574	8,748	8,726	8,932	9,117	9,241	8,968
PVC	10,092	9,888	10,120	10,070	10,136	10,268	10,242	10,259
PVC	9,526	9,309	9,496	9,451	9,501	9,596	9,648	9,521
PVC	9,626	9,183	9,596	9,543	9,561	9,690	9,556	9,596
True Value	10	10	10	10	10	10	10	10
Average	9.44	9.26	9.31	9.28	9.37	9.53	9.45	9.43
% Recovery	94.4	92.6	93.1	92.8	93.7	95.3	94.5	94.3
STD DEV	0.444	0.480	0.527	0.504	0.464	0.434	0.477	0.479
% RSD	4.71	5.18	5.66	5.42	4.94	4.55	5.05	5.08

LDPE 680 K	Cd 110	Cd 111	Pb 207	Pb 208
mg/kg				
Sample 1	20.53	20.36	12.47	12.98
Sample 2	20.83	20.45	12.03	12.52
Sample 3	19.99	19.64	12.48	12.83
True Value	19.6	19.6	13.6	13.6
Average	20.45	20.15	12.33	12.77
% Recovery	104.3	102.80	90.7	93.9
STD DEV	0.428	0.442	0.256	0.235
% RSD	0.0041	0.0043	0.0028	0.0025

EC 681 K	As 188,980	As 193,696	Cd 214,439	Cd 226,502	Pb 182,143	Pb 220,353
LDPE	26,609	25,665	123,335	122,583	89,002	90,084
LDPE	30,101	28,624	142,260	141,010	102,124	102,727
LDPE	31,159	30,071	139,073	137,918	99,499	101,245
True Value	29.1	29.1	137	137	98	98
Average	29.29	28.12	134.89	133.84	96.88	98.02
% Recovery	101	96.7	98.4	97.7	98.9	100
STD REV	2,381	2,246	10,132	9,868	6,944	6,912
% RSD	8.128	7.986	7.512	7.373	7.168	7.051

Run by ICP-OES

## Method Note



## LDPE, PE and PVC Results

Run In Triplicate	As 188,980	Cd 214,439	Cr 267,716	Fe 238,204	Pb 220,353	Se 198,026
LDPE	1,858	2,151	1,6336	1,9778	1,8729	1,8714
PE	1,917	2,196	1,6602	2,0127	1,9175	1,9221
PVC	1,9543	2,2609	1,7206	2,0525	1,8854	2,031
Average	1,91	2,20	1,67	2,01	1,89	1,94
% Recovery	95.5	110.00	83.5	100.5	94.5	97
STD DEV	0,049	0,055	0,045	0,037	0,023	0,082
% RSD	0,0508	0,0502	0,0534	0,0372	0,0243	0,0841

Run by ICP-MS

## Conclusions

- Samples were completely digested in all cases after dilution.
- Larger samples may be run, but a slight color may be present.
- Results were consistent with certified or spiked values.
- Low blank values of the MARS 6 iPrep vessel allow for trace analysis.
- MARS 6 iPrep provides an excellent option for acid digestion of difficult matrices.