LabTech

E D 3 6 & E H D 3 6

Electrothermal
Hotblock Digester

APPLICATION NOTES

ISO 9001 CE

LABTECH INC.

Your Lab, Our Tech

CONTENT

NOTE: This procedure is a guideline, developed on a specific sample. It may need to be modified or changed to obtain the required results on your sample.

We plan to update EHD36&ED36 application notes periodically. If your interested samples are not included in this application notes, please send your application request to our Customer Service Center. We do our best support you.

Chapter 1

ENVIRONMENTAL

1.1 SOIL

Application field: Soil

Summary

This method provides for the acid digestion of soil sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (PTFE)

Reagent

1. HCl: ρ=1.19g/ml, GR
2. HNO_3: ρ=1.42g/ml, GR
3. HF: ρ=1.49g/ml
4. $HClO_4$: ρ=1.68g/ml, GR

Procedure

5. Add 0.5g sample and 10ml HCl to digestion tube and close the tube.
6. Insert the digestion tube into the cavity of ED36.
7. Setting the temp. of ED36 to 100℃ and run the heating program.
8. Keep 10min at 100℃ after reach 100℃.
9. If the sample do not digest completely. Let the tube cool to ambient temp. and add 5ml HNO_3, 5ml HF and 3ml $HClO_4$.
10. Reset the temp. of ED36 to 170℃.
11. Continuously heating 1h and let the tube cool to ambient temp.
12. Reset the temp. of ED36 to 200℃ and open the tube.
13. Run the heating program till the digestion tube is filled with heavy white smoke.
14. Continuously heating till the solution is approx. 2ml and transparent.

Notes

If the soil sample completely digested, the solution is transparent. Please repeat above procedure if the sample is not completely digested.

Appropriate digestion time and acid proportion are both important for good digestion result due to the organic component are quite various in different soil samples.

ED36 can precisely control the digestion temperature and provide high sample-to-sample uniformity. It's a kind of easy and simple way to digest multiple soil samples.

1.2 WASTE WATER

Application field: Waste water

Summary

This method provides for the acid digestion of waste water sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass or polypropylene)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. H_2SO_4: ρ=1.84g/ml, AR
3. $KMnO_4$: 5%

Procedure

1. Add 20ml sample, 0.5ml HNO_3 and 1ml H_2SO_4 to digestion tube.
2. Add 3ml 5% $KMnO_4$ to digestion tube and let stand for 15min.
3. Add additional 2ml 5% $KMnO_4$ to digestion tube if purple or brown solution fade.
4. Close the digestion tube and keep a little gap for vent.
5. Insert the digestion tube into the cavity of ED36.
6. Setting the temp. of ED36 to 95℃ and run the heating program.
7. Continuously heating 2h and keep the solution un-boiling. Acid refluxing and rinse the wall of tube during the heating program.
8. Let solution cool to ambient temp. and the solution is ready for analysis.

FOOD

1.3 RICE

Application field: Rice

Summary

This method provides for the acid digestion of rice sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. $HClO_4$: ρ=1.68g/ml, GR

Procedure

1. Grind rice sample and pass through 100 screen.
2. Add 1.0g sample and 10ml mixing acids (HNO_3:$HClO_4$=4:1) to digestion tube (horniness glass) and close the tube.
3. Let the sample stand over 12h before insert the digestion tube into the cavity of ED36.
4. Setting the temp. of ED36 to 65℃ and run the heating program.
5. Maintain 15min at 65℃.
6. The tube is filled with red brown color after reach 100℃, then maintain 30min at 100℃.
7. Continuously heating until the solution is transparent and canary.
8. Reset the temp. of ED36 to 190℃.
9. Open the tube and dry the unwanted acids by heating to 190℃.
10. The final solution is 2ml and clear without color.

Notes

The refluxing in the condenser save a large mount of acid and automatically rinse the residual on the wall of digestion tube simultaneously. Circling heating brings

uniformity. Avoid liquid splash and precise control the temperature. It's a kind of easy and simple way to digest multiple rice samples.

1.4 FISH

Application field: Fish

Summary

This method provides for the acid digestion of fish sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. $HClO_4$: ρ=1.68g/ml, GR

Procedure

1. Dry the fish sample at 120℃ and grind the sample by agate mortar.
2. Add 0.5g sample and 8ml HNO_3 to digestion tube (horniness glass) and close the tube.
3. Insert the digestion tube into the cavity of ED36.
4. Setting the temp. of ED36 to 130℃ and run the heating program. Approx. 15min from ambient temp. to 130℃.
5. After reach 130℃, make the acid refluxing and rinse the wall of tube.
6. Keep 20min refluxing and the solution basically clear.
7. Let the tube cool to ambient temp. and add 2ml $HClO_4$.
8. Reset the temp. of ED36 to 190℃ and place the tube into the cavity of ED36.
9. Continue heating sample to no white smoke and the solution is 2ml, transparent and colorless.

Notes

If the fish samples are totally digested, the solution is transparent. If used for AAS&ICP-AES analysis, please choose appropriate acidity of medium. It's available for testing when the medium is diluted to scale.

The refluxing in the condenser save a large mount of acid and automatically rinse the residual on the wall of digestion tube simultaneously. Circling heating brings uniformity. The total procedure is approx. 1 hour by LabTech ED36 hot block digester. It's a kind of very easy and simple way to digest multiple fish samples

1.5 MILK

Application field: Milk

Summary

This method provides for the acid digestion of milk sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. $HClO_4$: ρ=1.68g/ml, GR

Procedure

1. Add 0.5g sample and 10ml HNO_3 to digestion tube and close the tube.
2. Insert the digestion tube into the cavity of ED36.
3. Setting the temp. of ED36 to 120℃ and run the heating program. Approx. 15min from ambient temp. to 120℃.
4. Acid refluxing and rinse the wall of tube during the heating program.
5. Maintain 60min after reach 120℃ until the solution basically clear.
6. Let the tube cool to ambient temp. and add 2ml $HClO_4$. Then insert the digestion tube into the cavity of ED36.
7. Reset the temp. of ED36 to 190℃ and run the heating program until the solution no white smoke.

Notes

The grease, which comes from milk digestion, reacts to $HClO_4$ will bring explosion. Compare with hotplate beaker digestion, when the residual solution is small, the sample could not be vaporized to solid phase due to the small bottom of digestion tube.

Prolong the digestion time at 120℃ will dissolve most part of organic substances. Then add $HClO_4$ to avoid explosion.

If using H_2SO_4 to carbonize the sample, and then add a little of H_2SO_4 and $HClO_4$ to dissolve carbonization substance. The result is also good.

1.6 RAISIN

Application field: Raisin

Summary

This method provides for the acid digestion of raisin sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

HNO_3: ρ=1.42g/ml, GR

Procedure

1. Add 0.5g sample and 8ml HNO_3 to glass digestion tube and close the tube.
2. Insert the digestion tube into the cavity of ED36.
3. Setting the temp. of ED36 to 130℃ and run the heating program. Approx. 15min from ambient temp. to 130℃.
4. Acid refluxing and rinse the wall of tube 30min after reach 130℃.
5. Run the heating program until the solution is clear.

Notes

The sample amount, reagent amount, setting temp., etc. are all reference data. Please adjust the digestion temp., digestion time and acid concentration to guarantee the best digestion result for specific sample.

AGRICULTURAL

1.7 VEGETABLE

Application field: Vegetable

Summary

This method provides for the acid digestion of vegetable samples using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. $HClO_4$: ρ=1.68g/ml, GR

Procedure

1. Take the edible part of fresh vegetable and clean it by tap water and quadric distilled water successively. Dry the surface of sample and place it in oven for drying at 100℃. Then grind the sample and place it on desiccator.
2. Add 0.5g sample and 15ml mixing acids (HNO_3:$HClO_4$=4:1) to digestion tube and close the tube.
3. Let the sample stand over 12h before insert the digestion tube into the cavity of ED36.
4. Setting the temp. of ED36 to 65℃ and run the heating program.
5. Maintain 10min acid refluxing and rinse the wall of tube.
6. Reset the temp. of ED36 to 100℃ and run the heating program.
7. Keep 30min under 100℃ after the digestion tube is filled with red brown color.
8. If the sample do not digest completely. Let the tube cool to ambient temp. and add 5ml mixing acid (HNO_3:$HClO_4$=4:1).
9. Continuously heating until the solution is transparent and canary.
10. Let the tube cool to ambient temp. and add 2ml distilled water.
11. Reset the temp. of ED36 to 190℃and run the heating program till white smoke appears. The solution is colorless or tiny yellow.

Notes
The refluxing in the condenser save a large mount of acid and automatically rinse the residual on the wall of digestion tube simultaneously. Circling heating brings uniformity. It's a kind of easy and simple way to digest multiple vegetable samples.

1.8 EXTRACTIVE FROM THE LEAF OF GINGKO

Application field: Extractive from the Leaf of Gingko

Summary
This method provides for the acid digestion of extractive from the leaf of gingko using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

HNO_3: ρ=1.42g/ml, GR

Procedure
1. Add 0.5g sample and 8ml HNO_3 to digestion tube and close the tube.
2. Insert the digestion tube into the cavity of ED36.
3. Setting the temp. of ED36 to 120℃ and run the heating program. Approx. 12~15min from ambient to setting point.
4. After reach 120℃, make the acid refluxing and rinse the wall of tube.
5. Keep 20min refluxing and the solution basically clear.
6. Continue heating 1.5~2h until the solution changes to canary and transparent.
7. Reset the temp. of ED36 to 160℃ and drive away the acid.
8. Let the digestion tube cool to ambient temp. Then fix the volume of the sample.

Notes
This method is also available for the digestion of Chinese traditional medicine. The result will be better if soak the Chinese traditional medicine in nitric acid before digestion. Digest sample at 95℃ low temperature for testing Hg element. For Pb, Cd, etc. heavy mental elements, you can increase temperatue and make them dissolving totally. At the same time, please adjust the proportion of oxidant and add appropriate H_2O_2 (30%) and $HClO_4$ according to sample and interested element.

1.9 TEA LEAF

Application field: Tea leaf

Summary

This method provides for the acid digestion of tea leaf sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. $HClO_4$: ρ=1.68g/ml, GR
3. H_2O_2: 30%, AR

Procedure

1. Grind tea-leaf sample and pass through 120 screen.
2. Add 0.5g sample and 10ml HNO_3 to digestion tube (horniness glass) and close the tube.
3. Place the sample over 12h before insert the digestion tube into the cavity of ED36.
4. Setting the temp. of ED36 to 65℃ and run the heating program.
5. Maintain 15min at 65℃ after reach 65℃.
6. Reset the temp. of ED36 to 135℃and maintain 2h at 135℃ after reach 135℃.
7. Let the tube cool to ambient temp. if the solution is not clear, add 5ml mixing acids (HNO_3:$HClO_4$=4:1) or H_2O_2.
8. Continuously heating until the solution is transparent.
9. Reset the temp. of ED36 to 160℃.
10. Open the tube and dry the unwanted acids by heating at 160℃.
11. The final solution is 2ml and clear.

Notes

The refluxing in the condenser save a large mount of acid and automatically rinse the residual on the wall of digestion tube simultaneously. Circling heating brings uniformity. Avoid liquid splash and precise control the temperature. It's a kind of easy and simple way to digest multiple tea-leaf samples.

CHEMICAL

1.10 COSMETIC

Application field: Cosmetic

Summary

This method provides for the acid digestion of cosmetic sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech EHD36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. H_2SO_4 : ρ=1.84g/ml, GR

Procedure

1. Add 1.0g sample into digestion tube and close the tube.
2. Insert the digestion tube into the cavity of EHD36.
3. Setting the temp. of EHD36 to 90℃. Run 5-10min to volatilize the organic solvents.
4. Let the digestion tube cool to ambient temp. Add 5ml water and 10~15ml HNO_3. Then close the digestion tube.
5. Insert the digestion tube into the cavity of EHD36 and waiting for several minutes.
6. Continuously heating 5min on 90℃.
7. Let the digestion tube cool to ambient temp. Add 5ml H_2SO_4 and insert tube into the cavity of EHD36.
8. Reset the temp. to 350℃ and continuously heating sample till the tube filled with white smoke.
9. Let the digestion tube cool to ambient temp. and fix the volume of solution.

Notes

The cosmetic samples ordinarily have organic compounds, such as ethanol and glycerin. Need to volatilize organic solvents firstly.

Must adjust an appropriate time for volatilization due to the content of organic component are different in cosmetic samples. If the cosmetic samples are cream, please heating the sample by low temp. firstly and melt the sample adhere in the wall to the bottom of digestion tube.

In addition, please add acid slowly if the cosmetic samples include calcium carbonate, etc. power. This manipulation can prevent the violent expansion of caused carbon dioxide gas.

INDUSTRIAL

1.11 PAINT CHIP

Application field: Paint Chip

Summary

This method provides for the acid digestion of paint chip sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. $HClO_4$: ρ=1.68g/ml, GR
3. H_2O_2: 30%, AR

Procedure

1. Add 0.5g dried sample and 10ml HNO_3 to digestion tube and close the tube.
2. Insert the digestion tube into the cavity of ED36.
3. Setting the temp. of ED36 to 160℃ and run the heating program.
4. During the heating process, add 1~2ml H_2O_2 time after time. Finally add 1ml $HClO_4$ until the sample basically dissolving.
5. Reset the temp. of ED36 to 180℃.
6. Continue heating sample until the solution is small. Then fix the volume of the sample.

Notes

The finally solution is turbid maybe due to some inorganic additives in paint chip sample, which available for testing after filtration.

1.12 OIL

Application field: Oil

Summary

This method provides for the acid digestion of oil sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, AR
2. H_2SO_4 : ρ=1.84g/ml, AR
3. $HClO_4$: ρ=1.68g/ml, AR
4. H_2O_2: 30%, AR

Procedure

1. Add 0.5g sample, 10ml mixing acids (HNO_3:H_2SO_4=3:1), and 1.5ml $HClO_4$ to digestion tube (horniness glass) and close the tube.
2. Let the sample stand over 12h.
3. Add 2 pcs of furbished beading to the digestion tube and insert the digestion tube into the cavity of ED36.
4. Setting the temp. of ED36 to 50℃ and run the heating program. Slowly heating the sample and see the bubble boiling.
5. Keep 20min under 50℃. Make sure the bubble boiling is complete and this will prevent the sample splash from the digestion vessel.
6. Reset the temp. of ED36 to 120℃ and continuously heating 30min.
7. Reset the temp. of ED36 to 145℃ and run the heating program.
8. Let the digestion tube cool to ambient temp. if the solution darken and unclear.
9. Add 5ml mixing acids (HNO_3:$HClO_4$=4:1) or H_2O_2 to digestion tube.
10. Continuously heating till the solution is transparent.
11. Reset the temp. of ED36 to 150℃ and run the heating program until the solution is 2ml.

Notes

ED36 can precisely control the digestion temperature and provide high sample-to-sample uniformity. It's a kind of easy and simple way to digest multiple soil samples.

GEOLOGICAL

1.13 COAL ASH

Application field: Coal Ash

Summary

This method provides for the acid digestion of coal ash sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. H_2SO_4 : ρ=1.84g/ml, AR
3. HCl : ρ=1.19g/ml, AR
4. HF: ρ=1.49g/ml
5. H_3BO_3

Procedure

1. Add 0.5g sample, 10ml mixing acids （HNO3:HCl=1:3）and 4ml HF to digestion tube and close the tube.
2. Insert the digestion tube into the cavity of ED36.
3. Setting the temp. of ED36 to 100℃ and run the heating program for approx. 120min.
4. Let the tube cool to ambient temp. and add 2g H_3BO_3 to it.
5. Insert the digestion tube into the cavity of ED36 and continuously run the heating program for approx. 10min.
6. The solution is available for analysis after filtration.

CLINICAL

1.14 BLOOD

Application field: Blood

Summary

This method provides for the acid digestion of blood sample using hot block digester for the mental determination of spectroscopic methods.

Equipment

LabTech ED36 hot block digester and digestion tube (glass)

Reagent

1. HNO_3: ρ=1.42g/ml, GR
2. $HClO_4$: ρ=1.68g/ml, GR

Procedure

1. Take 20ml sample and place it on 37℃ water bath for 10min.
2. Transfer sample to centrifuge and run 10min under 3000rpm. Collect serum for following digestion.
3. Add 5ml serum to digestion tube, and add 2ml mixing acids (HNO_3:$HClO_4$=20:1) successively. Shaking the tube slowly when add the acid.
4. Place 10min after insert the digestion tube into the cavity of ED36.
5. Setting the temp. of ED36 to 120℃ and run the heating program.
6. After nitric acid break down, reset the temp. of ED36 to 190℃.
7. Continue heating until no white smoke and solution changes to small volume.
8. Add 1ml distilled water and drive away the acid by heating solution.
9. Turn off ED36 and take away the digestion tube.
10. Let the tube cool to ambient temp. and add distilled water to dissolve residual substance.
11. Shake up the solution before analysis.

Notes

This method is also available for the digestion of urine sample.

ACID SPECIFICATION

The sample amount, reagent amount, setting temp., etc. are all reference data. Please adjust the digestion temp., digestion time and acid concentration to guarantee the best digestion result due to the diversity of sample.

1.15 EASY SAMPLE

Sample Type	Drinking-water, waste water, etc.		
Sample Amount	Solid sample ＜2g and liquid sample is 20ml		
Reagent Amount	8-10ml		
Acid	HCl	HNO_3	HCl+HNO_3
Acid Type	Strong non-oxidizing mineral acid	Strong oxidizing mineral acid	Extremely strong oxidizing mineral acid
Digestion Temp.	65-95℃	65-95℃	65-95℃
Setting Temp.	70-100℃	70-100℃	70-100℃
Available Digestion Vessel	Glass and Polypropylene	Glass and Polypropylene	Glass and Polypropylene
Available Digester	ED36/EHD36	ED36/EHD36	ED36/EHD36
Note	Especially suitable for Hg determination		

1.16 COMMON SAMPLE

Sample Type	Vegetable, soil, food, etc.		
Sample Amount	＜2g		
Reagent Amount	8-10ml		
Acid	HCl	HNO_3	HCl+HNO_3
Acid Type	Strong non-oxidizing mineral acid	Strong oxidizing mineral acid	Extremely strong oxidizing mineral acid
Digestion Temp.	＜80℃	80-120℃	95-110℃
Setting Temp.	90-95℃	90-130℃	100-120℃
Available Digestion Vessel	Glass, Polypropylene and PTFE	Glass	Glass
Available Digester	ED36/EHD36	ED36/EHD36	ED36/EHD36
Note	If the sample is not digested completely, please let the vessel cool to ambient temp. and add H_2O_2, then repeat former steps.		

1.17 DIFFICULT SAMPLE

Sample Type	Feed, oil, polymer and component unknown sample						
Sample Amount	＜2g						
Reagent Amount	10ml						
Acid	HCl	HNO_3	HCl+HNO_3	H_2SO_4	HF	H_2SO_4+ HNO_3	HNO_3+$HClO_4$
Acid Type	Strong non-oxidizing mineral acid	Strong oxidizing mineral acid	Extremely strong oxidizing mineral acid	Strong oxidized and dehydrated acid	Strong non-oxidizing mineral acid ideal for samples such as SiO_2	Charring firstly, and then digestion	Extremely strong acid
Digestion Temp.	＜80℃	80-120℃	95-110℃	230℃以上	＜95℃	Add H_2SO_4 at 220℃ firstly, and then 80-120℃	145-200℃
Setting Temp.	90-95℃	90-130℃	100-120℃	260-400℃	＜110℃	260℃firstly and then 90-130℃	160-210℃
Available Digestion Vessel	Glass and PTFE	Glass	Glass	Glass	PTFE	Glass	Glass
Available Digester	ED36/ EHD36	ED36/ EHD36	ED36/ EHD36	EHD36	ED36/ EHD36	EHD36	ED36/ EHD36
Note	The dosage of acid is quite big during high temp. digestion. Please add acid according to specific sample. If the sample is not digested completely, please add acid to dissolve the sample time after time.						

Chapter 9

SERVICE

The LABTECH worldwide Technical Support network consists of highly trained Field Service Engineers, Technical Support Specialists and Service Coordinators who are ready to quickly assist customers with answers and resolutions to service needs and application questions.

If you need any help, please feel free to tell LABTECH, and LABTECH will do best to support you.

LABTECH
Customer Service Center
89 October Hill Road, Suite 5A
New Englander Industrial Park
Holliston, MA 01746
U.S.A.

Tel : 1-508 893 8966/1-508 283 3515
Fax : 1-508 893 8967
E-mail: support@labtechgroup.com
Http: www.labtechgroup.com