USER MANUAL

Acid Purification System for Ultra Acid

OD-98-UAPS

Please read this manual before using the Acid purification system.

ODLAB

Operation Guide

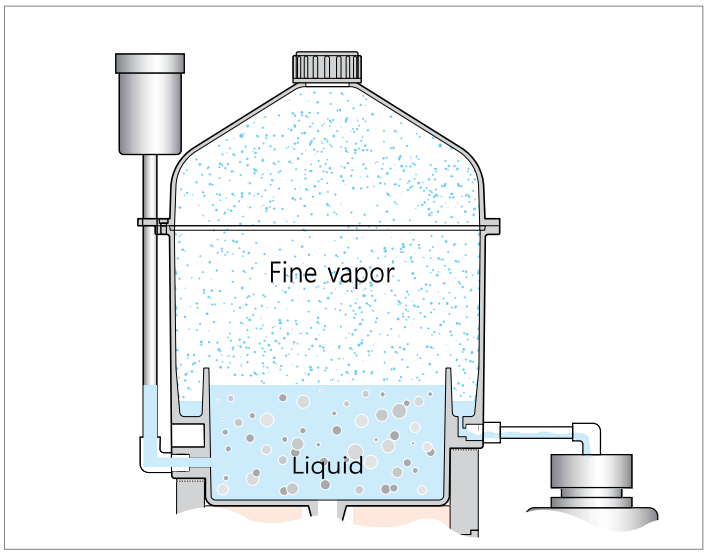
1.Product Features

This Acid Purification System is for purifying acids and some organic solvents by subboiling method. It is made of molding PFA. Each part used in OD-98-UAPS is made of PFA and PTFE. Especially, solutions which will be used for purification will be contact only to PFA parts. The PTFE support is produced by turnery and designed to endure corrode or break by exposition from humic acids for a long time. Heating process is operated by inserting a cartridge heater into a double graphite plate coated with Teflon inside PTFE. The acids will be slowly sub-boiling by heat from the bottom of PFA Container.

PFA Container has a large capacity of 5 liters and has more than 1.4ml (HNO3) purification efficiency per minute. OD-98-UAPS, Its basic purpose is that make customers producing 10ppt of high purity acid with 1ppb acid at customer's laboratory. In order to produce refined acids of less than 10ppt, it is necessary to have clean room of 100class or under and to have a cleaning facility for the trace analysis in the customer's laboratory. Therefore, the laboratory environment should be sufficient for purifying acid, if not you may not obtain high grade acids.

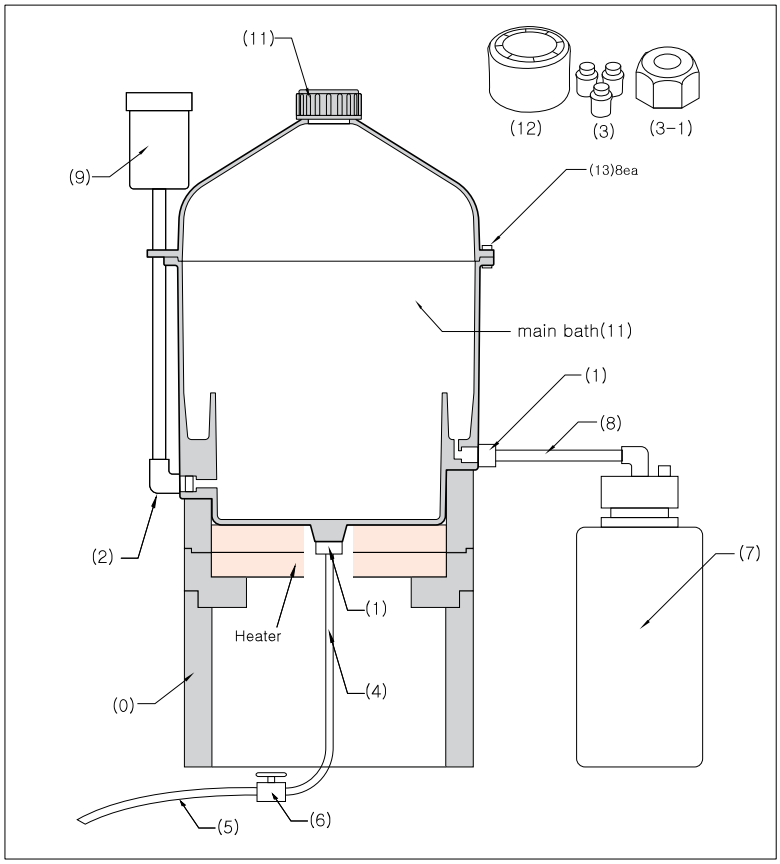
ODLAB's UAPS products are made of Teflon(Metal free features) and can be used in a laboratory dealing with acids. In preparation for not using Purification system anymore, we had planned the PFA Container to have multi-functions from its initial design, which is that PFA Container can be converted to Cleaning system for vials, ICP, ICP-MS. If you no longer use our Purification system, by replacing few parts you can convert it to Cleaning system.

2.Features of Sub-boiling



Sub-boiling is a method of refining micro-vaporized gaseous particles by condensing them with room-temperature cooling on the surface of the PFA Container by heating a solution at below boiling point. Heating below the boiling point, the element which is sensitive to the temperature change could be high purity acid without vaporization. In addition, there is no splashing from the surface of the solution, acid purification can be done without contamination. Our heating system is consisted with graphite material with good thermal conductivity to transfer heat evenly inside the container so that you can obtain high purity acid.

3.Product Composition



(1) Male connector 1/4"- 3/8".....2ea
(2) Male elbow 1/4"-3/8".....2ea
(3) Sleve cap for cleaning PFA container.....3ea
(3-1)Sleve cap Nut.....1ea
(4) Flexible Tube 500mm for draining...1ea
(5) Flexible Tube 1500mm for draining.1ea
(6) Ball valve 1/4" PTFE for draining.....1ea
(7) PFA Wide -mouth Bottle 1Liter with Cap having air vent.....1Bottle
(8) PFA Tube 100mm for connecting with 1Liter bottle.....1ea
(9) Acid pour tube with PTFE Cap.....1ea (funnel type)
(11) Cap for main PFA container.....1ea
(12) Hand wrench for cap.....1ea
(13) Peek bolt and nut.....8ea

4. Warning Label

◆ Be sure to use the product properly to prevent any danger or property damage.

Caution

Failure to observe this warning may result in death or serious injury. Be sure to take precautions to avoid accidents

Warning label advises to avoid possible burns. Please pay attention to slight burns during heating, do not touch on the middle side of the PTFE surface

This warning label is attached inside beneath of PTFE graphite heater. While operating this product it may cause possible burning please do not touch PTFE part.

5. Cleaning before first use

In order to use OD-98-UAPS, the inside of the PFA container and the inside of the PFA bottle should be cleaned with high purity DI Water and 10% Hydrofluoric acid or 10% Nitric acid beforehand.
Close the three opened PFA connectors with enclosed sleve caps and fill it with DIWater and shake container for cleaning.
Then add 10% hydrofluoric acid solution or nitric acid solution and shake the inside in the same way.
After that add 10% hydrofluoric acid or nitric acid, and heat the product to 150 ℃ as refining the actual product, it is to wash the inside with acid fine vapor Continue to repeat this procedure until result data satisfies you.


1. Close PTFE with caps


2. Close Drain PTFE with cap


3. Close Container cap


4. shake and wash

6. Installation

Protect your hands with clean safety gloves and assemble the product. Connect the PFA connectors to both sides of the PFA Container.(1,2) Connect a funnel type Tube for injecting acid and Cap.(4,5,6)
Assemble a drain male connector on the bottom and connect the 500mm PFA flexible tube.(7,8) (Using two s panners to hold and turn the male connectors).(9)
Pass the tube to the center hole on the graphite plate and seat it on the Container PTFE support.(10, 11)
After connecting the PFA flexible tube to the PTFE Ball valve (12,13), assemble the PFA Cap on the top of the container(14)
Connect the container and 1 liter bottle with a PFA Tube 100mm.(15)


1. Assemble an Elbow connector

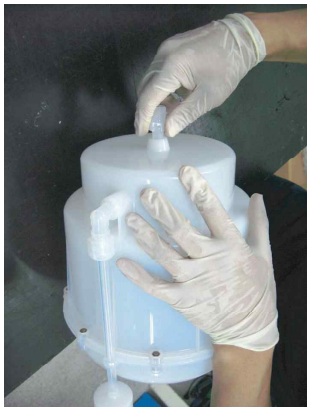

2. Assemble a Male connector


3. Assembled product


4. Assemble Acid pour tube


5. Assemble PFA Hopper


6. Assembled product


7. Assemble Drain male connector

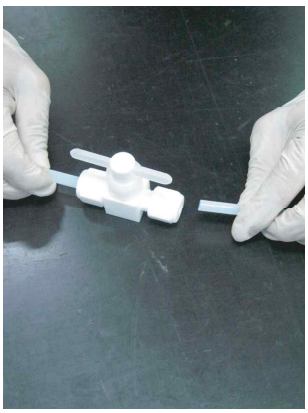

8. Connecting 50CM Drain Tube


9. Hold and turn with Spanner


10. Pass a drain tube through the hole


11. Pass a drain tube through PTFE hole


12. Assemble 50cm Tube


13. Connect PTFE with Tube


14. Close a cap on the top


15. Connect a 100mm tube with a bottle


16. Assembly completed

7. Analysis result after refining (70% Nitric acid purification results)

Below chart shows analysis result in 100 class cleanroom after cleaning for 8 days with above mentioned cleaning method.

(unit:ppt)

	1ppb HNO_3	180℃ 1st	180℃ 2th	180℃ 3th	150℃ 1st	150℃ 2th
Ag	1041.984	11.129	13.952	0.146	1.116	1.8
Al	1010.15	8.168	6.859	3.015	0.281	5.865
As	1130.676	0.725	10.506	1.088	1.565	0.629
Au	1007.292	1.1	0.112	1.72	0.798	1.15
Ba	1013.484	2.224	1.209	1.724	0.762	3.11
Be	1002.588	ND	ND	0.079	ND	ND
Bi	1055.214	ND	ND	0.08	ND	0.077
Ca	1003.648	65.426	44.445	19.233	38.743	24.902
Cd	1110.065	ND	0.442	ND	ND	ND
Co	1009.257	3.33	7.228	4.301	1.192	0.784
Cr	1045.312	92.603	82.981	180.123	23.683	13.259
Cu	1108.596	2.264	8.818	ND	ND	0.518
Fe	1285.48	38.101	30.612	26.798	22.302	27.069
Ga	1397.828	ND	0.262	0.412	0.113	0.332
Ge	1158.644	ND	1.452	ND	ND	ND
Hf	1016.524	ND	ND	0.196	0.112	0.286

(unit:ppt)

	1ppb HNO_3	180℃ 1st	180℃ 2th	180℃ 3th	150℃ 1st	150℃ 2th
Hg	1005.517	0.042	ND	2.051	0.602	0.948
In	1124.38	ND	0.036	0.015	ND	0.006
K	1011.544	3.502	4.266	ND	ND	ND
Li	1541.648	ND	0.249	ND	0.015	0.004
Mg	1116.444	3.231	3.486	1.19	2.031	4.832
Mn	1042.256	ND	ND	6.738	0.64	4.243
Mo	1199.344	0.074	1.686	1.977	1.107	1.817
Na	1218.94	2.995	15.911	0.828	0.991	3.36
Nb	1183.092	ND	ND	0.044	ND	ND
Ni	1101.812	6.944	8.138	10.42	8.485	12.751
Pb	1067.104	1.147	0.299	ND	2.942	0.315
Sb	1002.588	ND	0.474	ND	ND	ND
Sn	1005.992	8.071	7.834	4.07	2.706	11.449
Sr	1321.288	0.123	0.206	0.174	0.026	0.499
Ta	1064.14	ND	0.004	ND	0.035	ND
Th	1000.428	0.154	0.245	0.239	8.567	ND
Ti	1026.384	37.928	39.093	69.398	8.567	5.726
Tl	1025.308	ND	ND	ND	ND	ND
U	1031.276	0.041	0.004	0.016	ND	0.005
V	1000.752	ND	ND	7.914	ND	4.308
W	1162.116	ND	0.054	ND	0.055	1.279
Zn	1010.816	5.197	2.216	ND	ND	ND
Zr	1197.308	1.414	0.03	0.311	0.395	0.897

As shown in the above analysis results, Ca and Fe concentrations are higher than other elements, but it is expected to be improved to below 10ppt. Also, Ti and Cr concentration is lowered at the 150° C. Agilent ICP-MS used and showed a purification efficiency of 1 Liter / 7hour at 150°C.

8. Results of cleansing solution analysis

The table below shows the analysis results of the hydrofluoric acid solution after washing it with 10% hydrofluoric acid solution.

Analysis results of 10% HF cleansing solution

(unit:ppt)

	Cleaning solution 1st	Cleaning solution 2st		Cleaning solution 1st	Cleaning solution 2st
Ag	ND	0.175	Ca	719.219	356.146
Al	90.945	18.126	Cd	0.21	0.23
As	ND	9.052	Co	6.762	10.923
Au	8.59	1.53	Cr	326.999	35.405
Ba	13.388	1.663	Cu	38.546	32.536
Be	ND	ND	Fe	337.691	42.508
Bi	0.073	ND	Ga	0.235	0.041

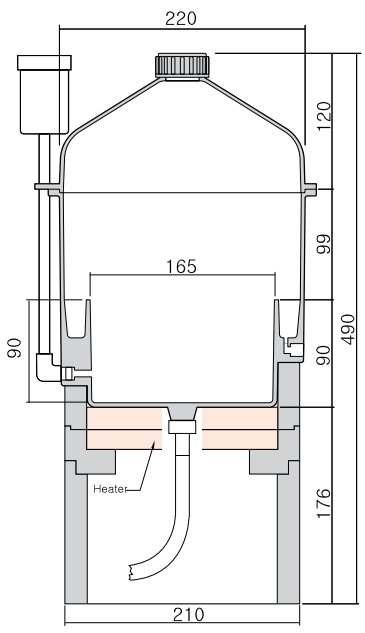
(unit:ppt)

	Cleaning solution 1st	Cleaning solution 2st		Cleaning solution 1st	Cleaning solution 2st
Ge	ND	3.095	Sb	0.879	0.197
Hf	0.342	ND	Sn	7.805	12.17
Hg	ND	ND	Sr	1.378	2.783
In	ND	0.006	Ta	ND	0.006
K	45.424	24.397	Th	ND	ND
Li	0.762	0.297	Ti	143.991	37.957
Mg	45.148	191.989	Tl	ND	0.053
Mn	ND	0.543	U	ND	ND
Mo	10.713	2.012	V	ND	ND
Na	50.079	15.317	W	2.019	0.159
Nb	0.047	ND	Zn	38.577	200.688
Ni	47.82	12.931	Zr	1.854	2.542
Pb	3.879	1.517			

As shown in the above analysis result, all commercially available PFA injection products shows high values from elements Al, Ca, Co, Cr, Cu, Fe, Na, Mg. However, these elements can be clean with acid cleaning its surface

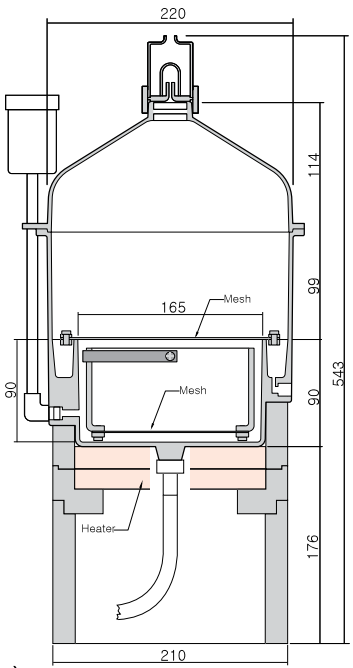
9. Dimension

(Acid Purification System)



Change of usage

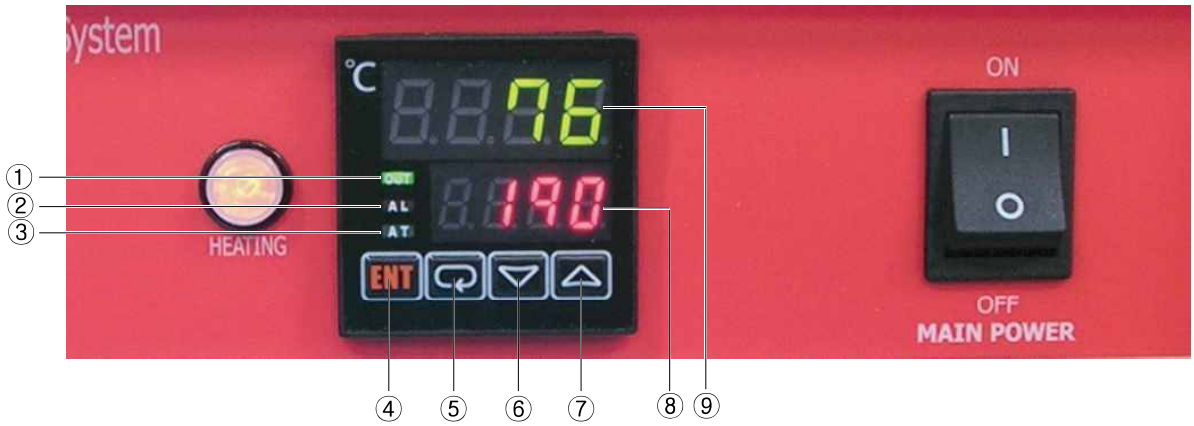
(Acid Cleaning System)



Material: PFA(Container), PTFE(Support), Heater(Graphite)
Power: 110~240VAC, 50/60Hz
Power consumption: 500W
Temperature Control: PID type Manual controller or Touch Screen Controller(option item)

Operation Guide

■Product name and Function



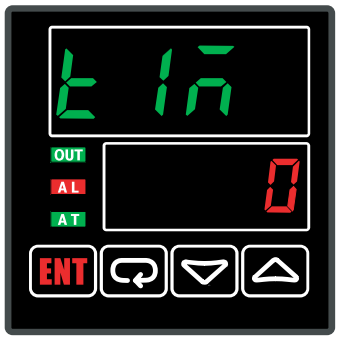
① OUT Lamp	The lamp light goes on when the graphite block is powered up. (during heating).
② AL Lamp	Alarm Function. (Not activated for Cleaning)
③ AT Lamp	Light goes on during auto tunning operating.
④ Enter Key	This key is for setting temperature and time.
⑤ Mode Key	This key is for entering each mode. (Some modes are not available)
⑥ Down Key	This key is for lower setting temperature and time.
⑦ Up Key	This key is for upper setting temperature and time.
⑧ Setting time & Time display(SV)	Display setting temperature and set time.
⑨ Present Temp(PV)	Display current temperature.

■How to set the temperature

Turn on the power switch and set the temperature by pressing ⑥Down key and ⑦Up key. After setting, be sure to press the ④ Enter key ENT

■How to set the time

After setting the temperature, press ⑤mode key 8 times to display. And then, press the ⑥Down key or ⑦Up key to set the time.
The time can be set from 1 minute to 99 hours and 59 minutes.
Press ④enter key ENT and hold for 5 seconds to complete the setting.
When setting is completed, set temperature and set time will be displayed in ⑧window alternately (setting in the option variable group)



◀ Display mode during time setting

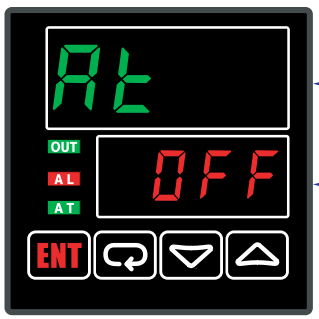
◀ Time display (minutes)

Note: Temperature cannot be set after setting the time, be sure to set the time after setting the temperature.

After start heating, timer starts counting down when the temperature reaches 90% of set temperature.
For example, when you set the temperature to 200°C and heating started, the timer starts counting down when the temperature reaches 180°C. Timer starts counting down during operation.
Timer stops when heating is terminated and timer display will be shown as below image.
Press the ④Enter key ENT for 3 seconds to initialize the settings.

■ Auto Tunning(AT)

Our product is auto tuned from factory. However, since the electrical environment differs from customer's laboratory, it is recommended to auto-tune again at the laboratory for your use. This should be done when the current temperature is not stable according to set temperature.
Auto tunning shall be set at the temperature you prefer and press the⑤Mode key for 9 time.
When 'AT' is displayed, press ⑦Up key to turn it on, and press ⑤Mode key again for 3 seconds, then AT starts operating. The actual temperature and set temperature are displayed on the screen, and the ③AT lamp lights up. ③AT lamp is turned off automatically when AT is finished.



◀ Auto tunning display (AT)

◀ Normally OFF indication / Press ⑦Up key to turns ON

Note: While auto tunning, the temperature setting is not working. The time required depends on the temperature you want to set. (30 minutes to 1 hour)

■What is Auto Tunning?

It is a controller function to set the PID automatically.
(PDI is the way controls the Proportional operation, Integral operation, Differential operation at once.)

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