

**Title: Acid Purification Using MILESTONE duoPUR "Subboiling" Distillation System**

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## Acid Purification Using MILESTONE duoPUR "Subboiling" Distillation System

### 1. INTRODUCTION and SCOPE

- 1.1. This procedure is used to produce ultrapure acids by the means of subboiling distillation process which is recognized as the best method to obtain high purity reagents and the lowest blank values for ultratrace analysis.
- 1.2. During the subboiling process the liquid is heated by infrared at the surface to avoid violent boiling and prevent aerosolized particles from depositing on the surface of the condenser and being carried over to the purified acid. The vaporized liquid is collected on an inclined water-cooled condenser and drips into the collection container (PTFE bottle). Ultrapure acids produced have essentially the same concentration as the acids used to produce them.

### 2. APPARATUS

- 2.1. duoPUR Subboiling Distillation System (Milestone, Sorisole, BG, Italy) consists of two high-purity quartz distillation units. Each unit contains two heating elements, a water cooled condenser, reagent addition and collecting bottles, and a drain.
- 2.2. The distillation process is microprocessor controlled, allowing the user to set the distillation time and power level.

### 3. CREATING / EDITING A PROGRAM

- 3.1. Switch on the Control Unit
- 3.2. Create / Edit a Program

A program must be produced in order to use this instrument. Software allows storage of up to 10 independent work programs (named 1-10). Each program can include up to 10 sequential steps each with **time** (hh:mm:ss) and **power** (in %) parameters determined by the user. After the steps are completed, there is always a cooling time during which the power is automatically **OFF**. The program's battery buffers will keep data stored in memory even during a blackout.

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- 3.2.1. Press the **STOP** key for initialization of a newly installed system.
- 3.2.2. Press **F2** to Create or Edit a program.
- 3.2.3. Select the number you wish to assign the program (as a name, 1-10). Then press **F3**- "OK".
- 3.2.4. Press **F2** to edit or **F1** to erase the selected program.
- 3.2.5. Set distillation time and heating power for each step, pressing **F3** after each entry as "OK".
  - 3.2.5.1. A satisfactory purity of low boiling-point chemicals like HCl, HNO<sub>3</sub> (Sdp. 100-125°C) is reached between 50-80% power.
  - 3.2.5.2. When using a duoPUR system, both distillation units can be run simultaneously; therefore the total power of 100% cannot be exceeded.
- 3.2.6. Set the Cooling Time.
- 3.2.7. Press **F3**- program is ready. By pressing **STOP**, the program can be aborted. See Table 1 in Appendix A for the program used for sub-boiling of nitric acid.

#### 4. OPERATING PROCEDURES

- 4.1 Load program to be used
  - 4.1.1. Press **F1** to work with created programs from main menu.
  - 4.1.2. Press **F3** when you want to work with the program selected.
  - 4.1.3. Once a program has been selected, press **F1** to change, or **F3** if OK.
  - 4.1.4. When a different program (i.e. not the previously used program) is selected, the user will be prompted to select which distillation tube to run. The user can toggle through RUN UNIT 1, RUN UNIT 2, and RUN UNIT 1+2 by pressing **F1**. Press **F3** when OK.
- 4.2 Fill Up the Reagent to be Purified
  - 4.2.1. The subboiling system can be filled up to no higher than 2cm below the brim of the distillate collecting tube (500-600 mL). Note: If the liquid level is too high, it will cause the solution to flow directly into the distillate collection vessel.
  - 4.2.2. After filling, close feeder cap, but do not tighten it.
  - 4.2.3. Refill the feeding container before reaching the minimum level of 1-2 cm above the bottom. Warning: Never Run Dry! Never refill before one program is completed or when the heating power is ON.
- 4.3. Press START after selection of a program.

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- 4.4. Turn on cooling water tap.
- 4.5. Turn off the cooling water immediately once the program has finished. Press any key to return to the main menu, or leave it as is.
- 4.7. If you wish to arrest the program before the programmed amount of time, press STOP. Otherwise, the program will continue running until the end of the COOLING step.

**5. CLEANING AND EMPTYING**

- 5.1. When first using the system, clean it by making a pre-run of 10-20 mL with higher power and then discard.
- 5.2. When used for a long period of time, the opening may become blocked (due to the material build-up) resulting with possible damages. When necessary, empty the system after it is completely cooled.
- 5.3. To empty the system, a vacuum pump connected to an exhaust tube can be used. Otherwise, a siphon can be used by placing a thin tube to the bottom of the supply tube. A glass pipette can be used to suck the acid into the tube, the other end of which should be placed in a beaker below the level of the distillation tube.

Note: Make sure to empty system only when it has completely cooled.

**6. SAFETY MEASURES**

- 6.1. Always use standard safety measures for handling chemicals (i.e. lab coat, safety goggles, gloves, etc.).
- 6.2. The Control Unit must NOT be placed under fume hood otherwise it may corrode. Distillation unit should be placed under fume hood.
- 6.3. Cooling Down the System:
  - 6.3.1. Before the distillate is removed, the system must be cooled down; otherwise there may be a release of acid vapours and gas, ex. NO<sub>x</sub>.
  - 6.3.2. Use an additional clean PTFE bottle to reduce the gas load and the risk of contamination when emptying the collection bottle. It provides easy and rapid handling so that "fresh acid" can be distilled immediately.

**7. APPLICABLE SOPs**

SWP-001/\*.\*, "Safe Working Procedures and Policies", Analysis and Air Quality Division.

**8. REVISION**

**December 2004:** Author Heidi Chen. New document 6.15/1.0/S

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**July 2010:** Reviewers: Valbona Celso, Irina Okonskaia

Sections 1 and 2 are modified. Flow charts in Figure 1 and Figure 2 are deleted

**October 2012:** Reviewer: Valbona Celso

Document was reviewed and no changes were made

## 9. REFERENCES

“MILESTONE duoPUR “Subboiling” Distillation System for Ultrapure Reagents User Manual”, Milestone Inc., 2001.

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## APPENDIX A:

*Table 1:* Program used for distillation of 500 mL HNO<sub>3</sub> with HZ1 (Heating Element 1)

<b>Step</b>	<b>Time [hh:mm:ss]</b>	<b>Power HZ1 [%]</b>	<b>Power HZ2 [%]</b>
1	00:05:00	90	0
2	00:30:00	60	0
3	01:00:00	70	0

**COOLING-TIME** 00:20:00

In this program, there is STEP 1 for 5 minutes at 90% power. Then STEP 2 for 30 minutes at 60% power. STEP 3 is for 1 hour at 70% power. The program ends with COOLING-TIME for 20 minutes. HZ2 is not used.

Note: The combination of the power for the two heating elements cannot exceed 100%.