

US EPA ARCHIVE DOCUMENT

Setting Up the Vacuum Distiller to Perform Methods 5032 and 8261

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Introduction

- First distillers have been interfaced to Agilent 5972/5890 and Agilent 5973/6890 systems
- Operating system for distillers require WindowsNT or newer
- Distillers are installed on systems configured for volatile analyses
- The range of analytes determined by method 8261 require sub-ambient cooling



Supplies Needed to Connect Vacuum Distiller

- Gas helium connection (carrier gas to GC/MS)
- Gas nitrogen connection (purging distiller between runs)
- Liquid nitrogen - dewar or central delivery system delivery at 20 psi
- Typical VOA sample prep materials

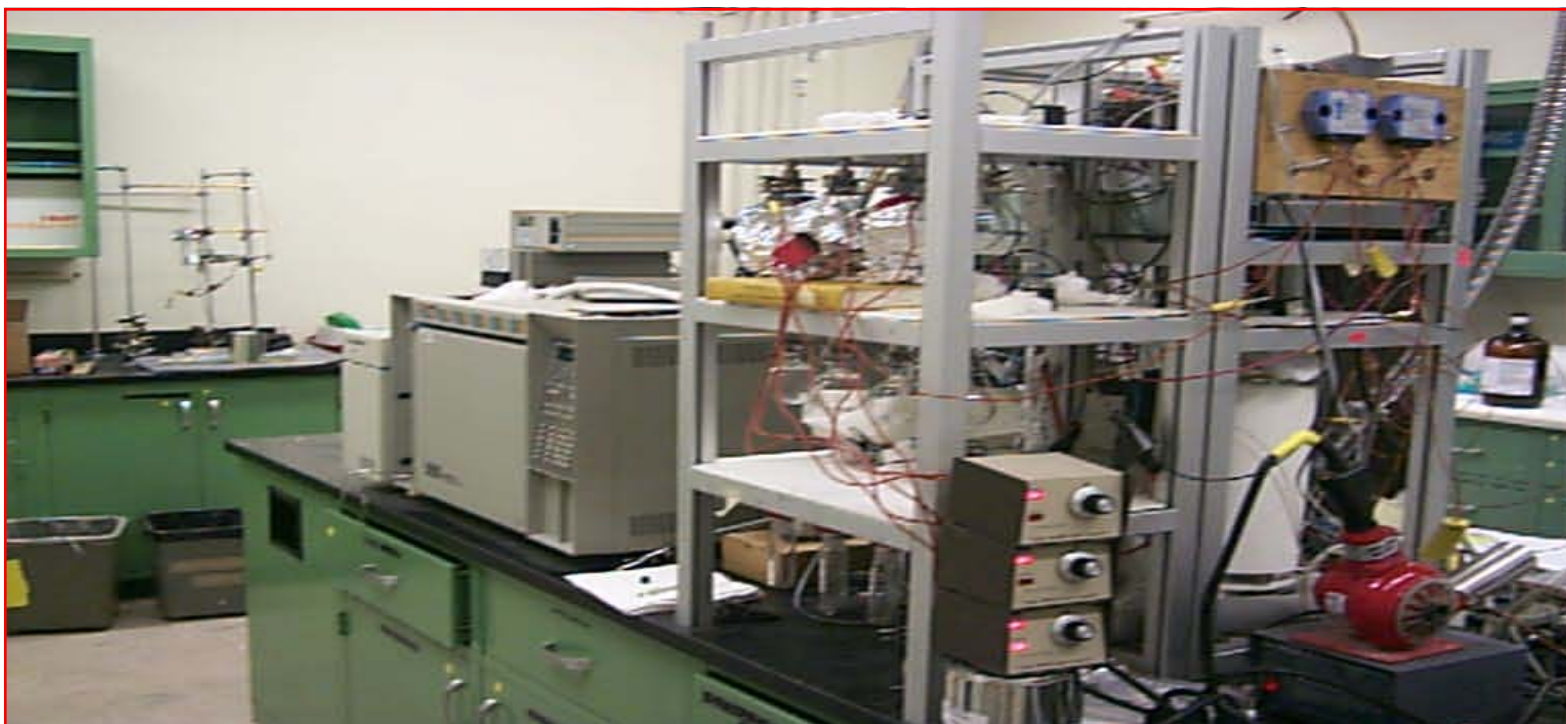


GC/MS Requirements

- Injector: Direct VOA splitter or split/splitless
- Column: Capillary VOA column (60 m x 0.25 mm x 1.4 Fm)
- Oven: Fitted with a 22 psi liquid nitrogen connection
- Data System: Windows NT (Version 4.0) with HP Chemstation
- Command Communications line: COM line between GC/MS/DS and distiller
- GC Start connection for automated start controlled by distiller



Prototype Vacuum Distiller



First fully automated vacuum distiller was developed at ESD Las Vegas

It is shown configured with Agilent 5972/5890 GC/MS



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Commercial Vacuum Distiller



Commercial vacuum distiller configured with Agilent 5973/6890 GC/MS



Vacuum Distiller Installation

(follow vendor instructions)

- Prepare bench space (12 1/2" wide and 22" deep), liquid nitrogen & gas supply, and power supply
- Connect and secure LN₂ and gases He₂ & N₂
- Connect vacuum pump
- Connect transfer line to the GC Connect "GC Start Cable" to GC
- Connect communication cable to computer (COM1 or 2)
- Connect distiller and vacuum pump power cords to a 120 volt outlet (10 amp)



Vacuum Distiller Software Install

- Power up the vacuum distiller
- Load vacuum distiller operating software according the vendor instructions
- Vacuum distiller operating software will provide screen as below

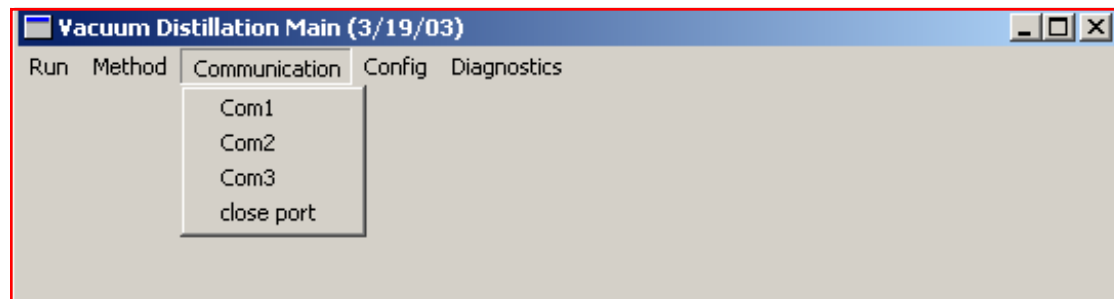


Screen displays shown are for EPA developed system. Commercial display will vary

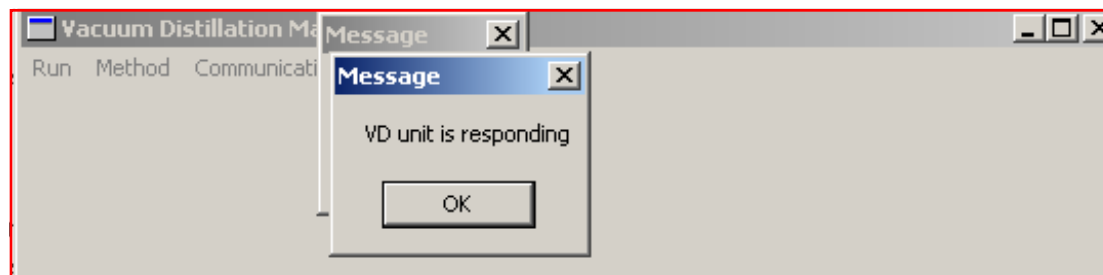


Verify Connections

- Select the appropriate communications port through Main menu

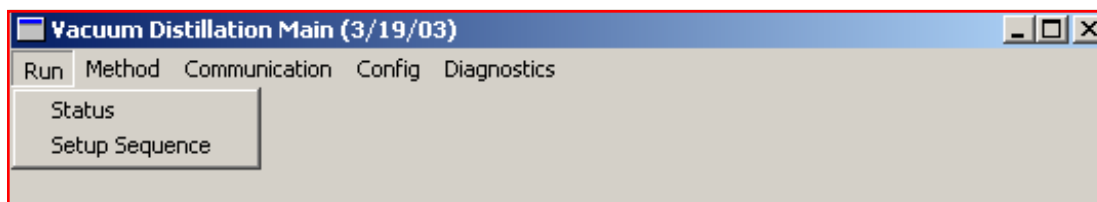


- If communications are established, vacuum distiller will send message to verify system communicating

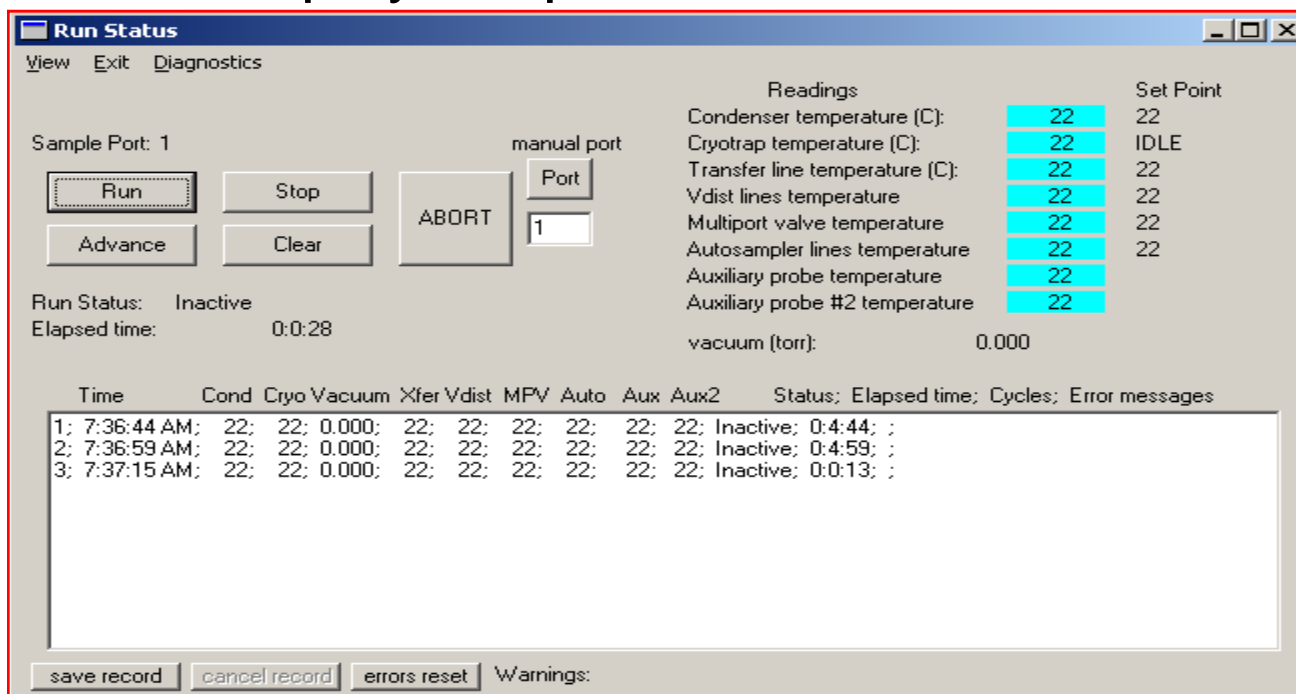


Display Instrument Readings

- View system status (Run ->Status)



- Software will display temperatures and vacuum



Note:system temperatures are set to ambient on power-up or if PC is not communicating



Verify System

- All zones will have temperature readings if system working properly
- Vacuum reading should be less than 0.4 torr



Vacuum Distiller Zone Temperatures

- Set vacuum distiller lines, valves to 95 °C through Methods Parameter Menu
- Condenser (Heating) 95 °C
- GC transfer line 200 °C
- Vdist lines 95 °C
- Autosampler lines 95 °C
- Multiport valve 200 °C



Method Parameters Menu (Main->Method->Run Method)

- After entering zone heating target temperatures from previous slide, hit **Send and Implement**

Method Parameters	
Method:	C:\WD\Methods\default.vdm
Pre-distillation evac(min):	0.00
Vacuum distillation time (min):	7.5
Transfer time (min):	2.5
Condenser temperatures (C)	
Heating	95
cooling 1	15.0
cooling 1 time	7.5
cooling 2	15.0
Cryotrap temperatures (C)	
Cryotrapping	-150.0
desorb delay (min)	0.05
Desorb	110.0
Bakeout	200.0
Transfer line temperature (C):	
Calculate VD time	
Vacuum distillation time	
transfer time	
Total run time (min):	
Verify	
Parameters	
Cryotrap bakeout and	
Condenser purge (min):	7.0
Flushing Cycles	
Pressurization (min):	0.05
Evacuation (min):	1.2
Number of cycles:	16
Stabilization times (min):	
Condenser:	0.1
Cryotrap	0.3
Vdist lines temperature	95
Multiport valve temperature	200
Autosampler lines temperature	95
Auxilliary temperature	20.0
Bakeout time	
Flush time	
Send and implement	



Vacuum Distiller Operational

- Temperature zones are at set temperatures
- Vacuum is $<.4$ torr
- System is communicating and past readings are displayed in Run Status Window
- Next step is to set up distiller conditions (see presentation “Tuning the Vacuum Distiller Optimizing Analyte Response and Chromatography”)

