Setting Up the Vacuum Distiller to Perform Methods 5032 and 8261

Michael H. Hiatt
U.S. Environmental Protection Agency
National Exposure Research Laboratory
Environmental Sciences Division
P.O. Box 93478, Las Vegas, Nevada 89193-3478

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

Commercial Vacuum Distiller

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

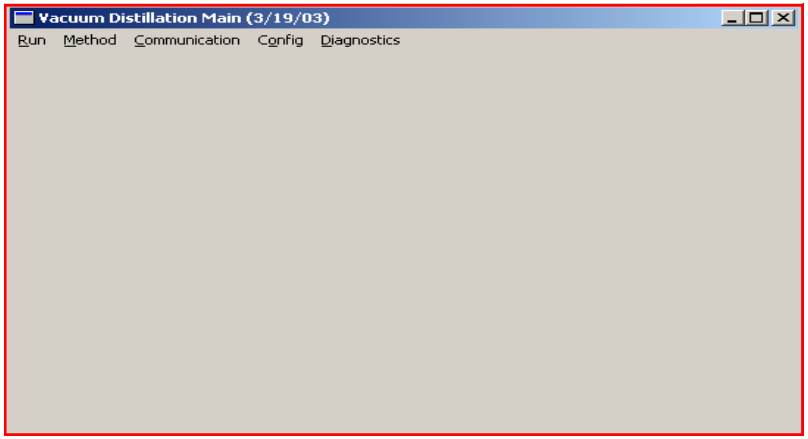
Vacuum Distiller Installation

(follow vendor instructions)

- Prepare bench space (12 1/2” wide and 22” deep), liquid nitrogen & gas supply, and power supply
- Connect and secure LN_2 and gases He_2 & N_2
- 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

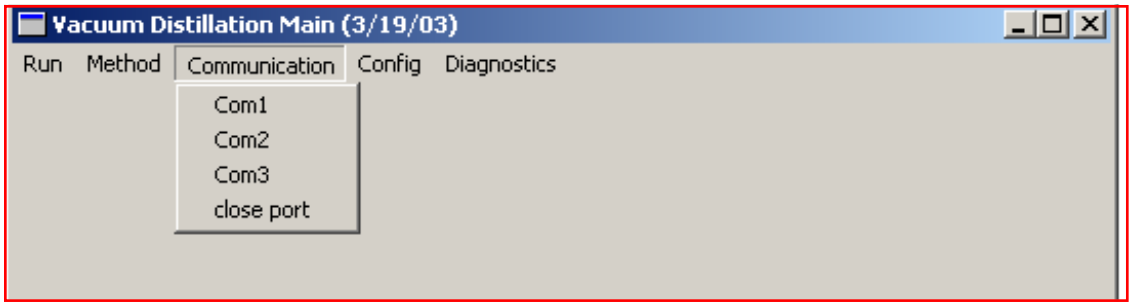
- Power up the vacuum distiller
- Load vacuum distiller operating software according the vendor instructions
- Vacuum distiller operating software will provide screen as below



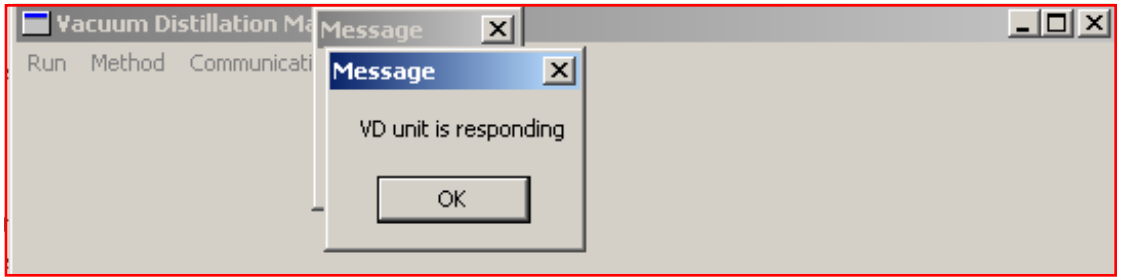
Screen displays shown are for EPA developed system. Commercial display will vary

Verify Connections

- Select the appropriate communications port through Main menu

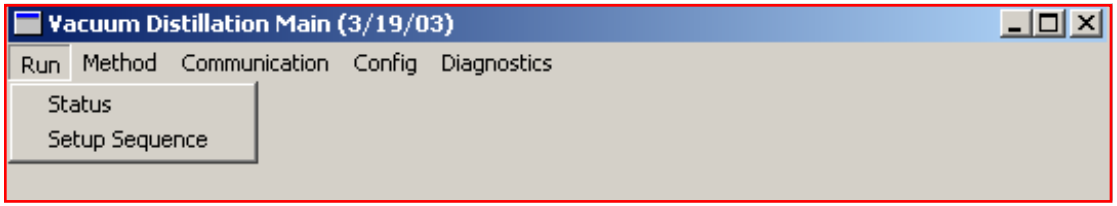


- If communications are established, vacuum distiller will send message to verify system communicating

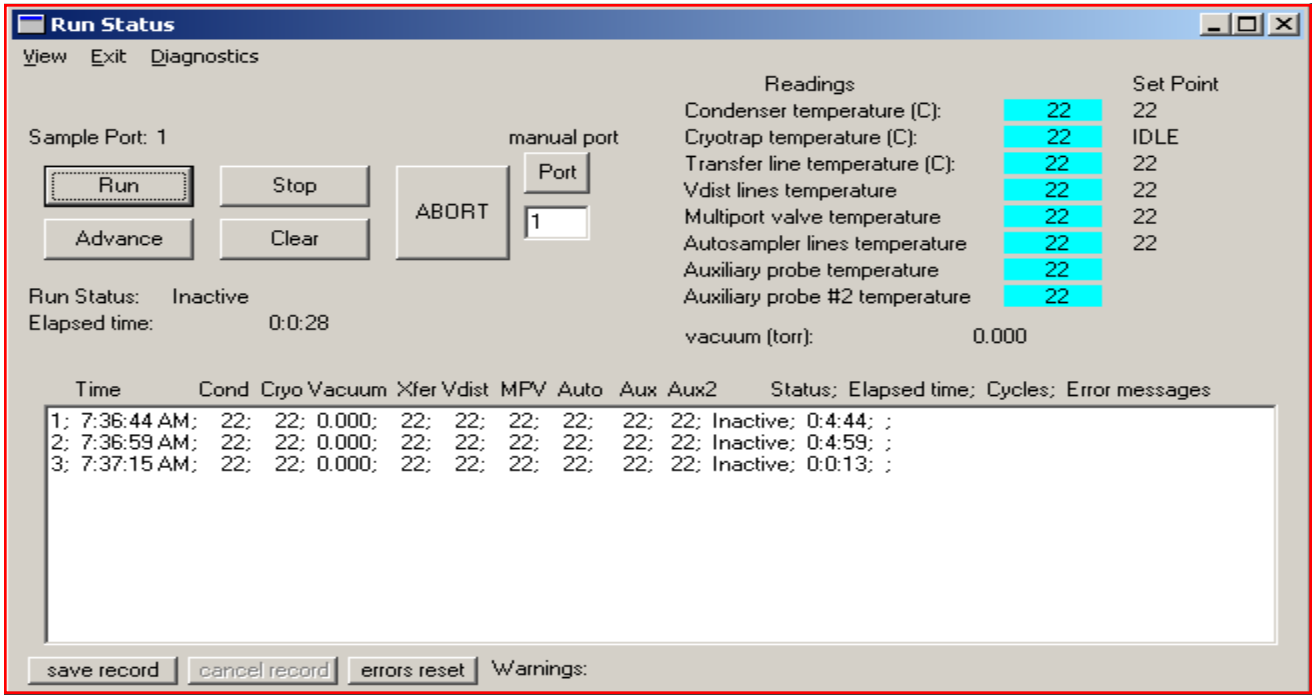


Display Instrument Readings

- 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

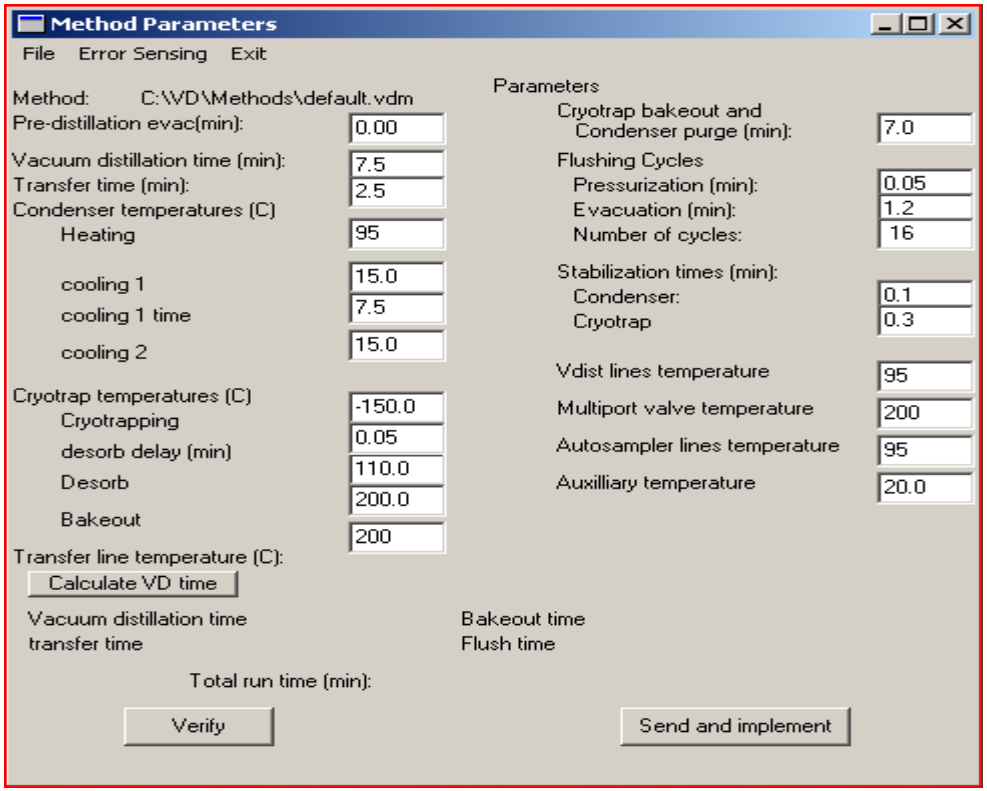
- All zones will have temperature readings if system working properly
- Vacuum reading should be less than 0.4 torr

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



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”)