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
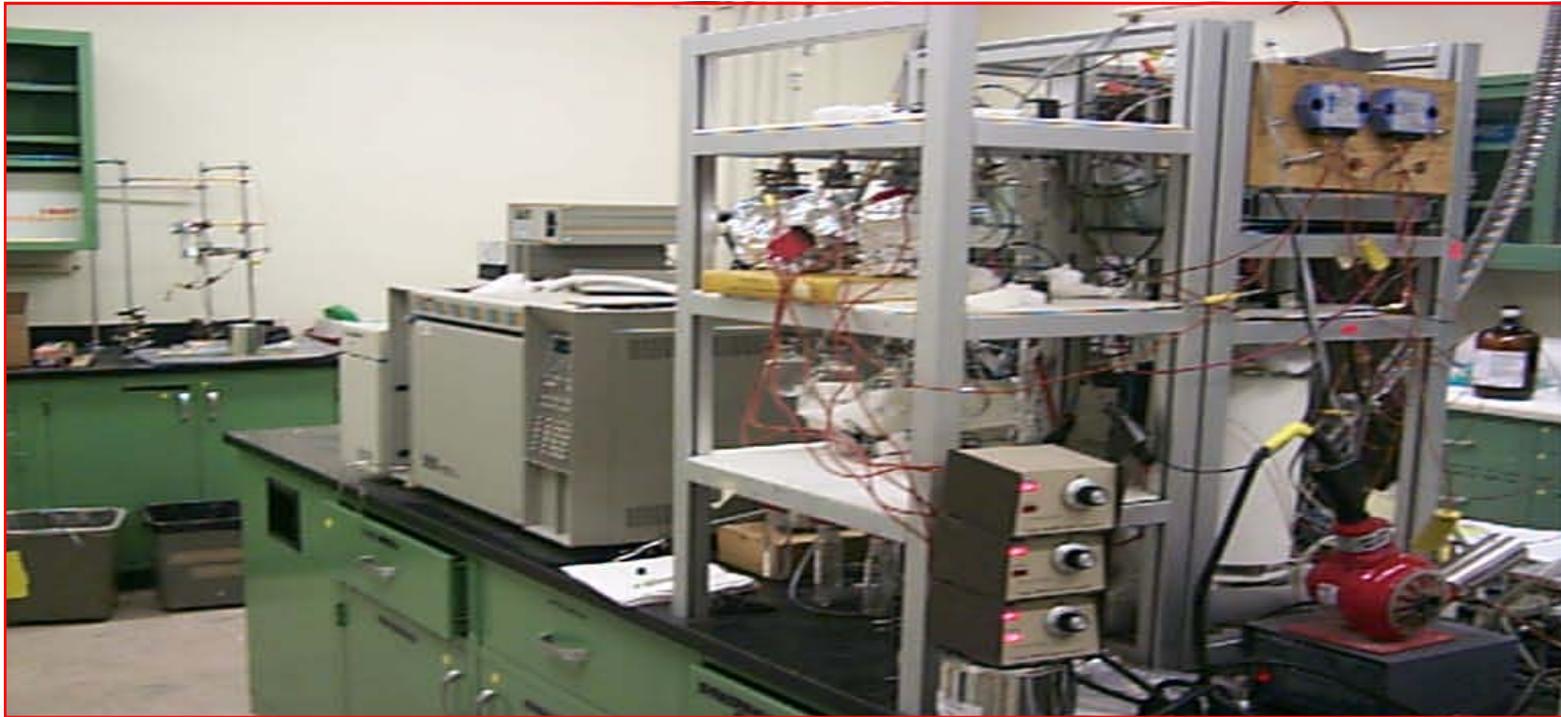
First fully automated vacuum distiller was developed at ESD Las Vegas

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

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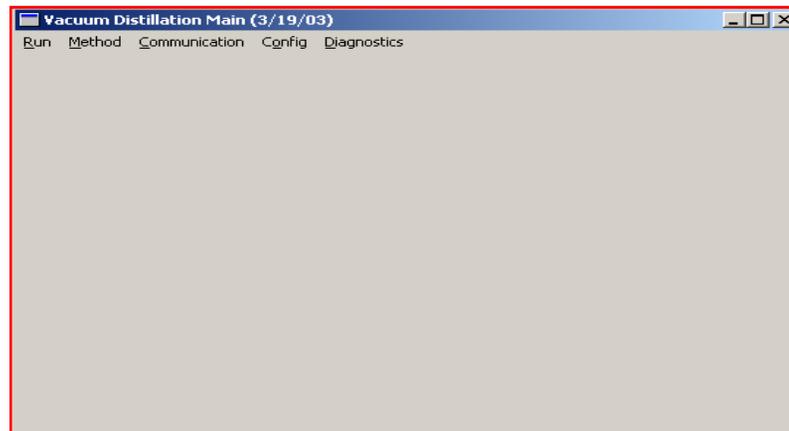
Screen displays shown are for EPA developed system. Commercial display will vary
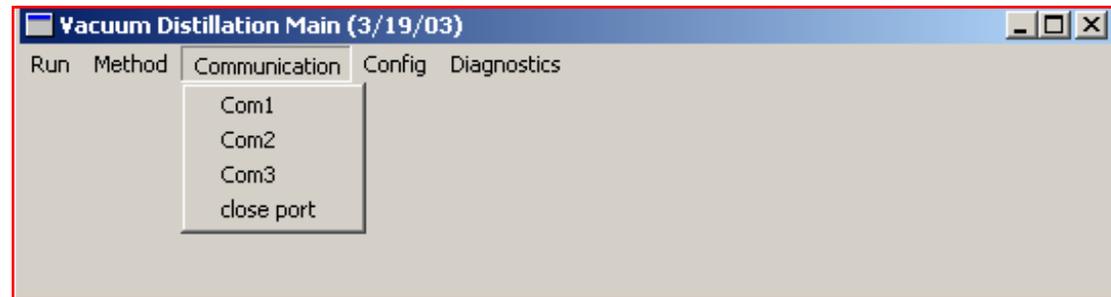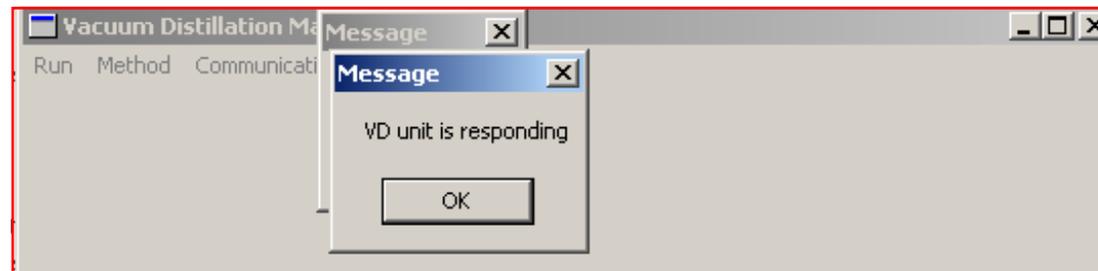
Verify Connections

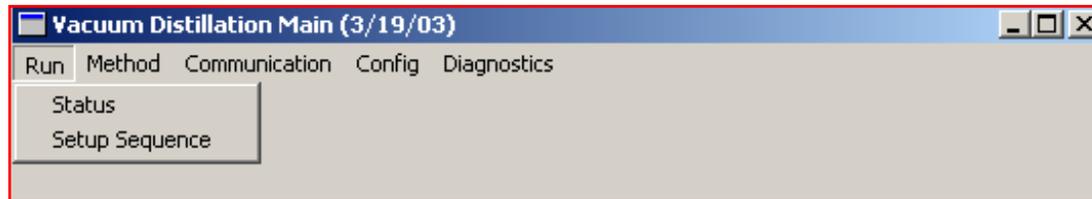- Select the appropriate communications port through Main menu

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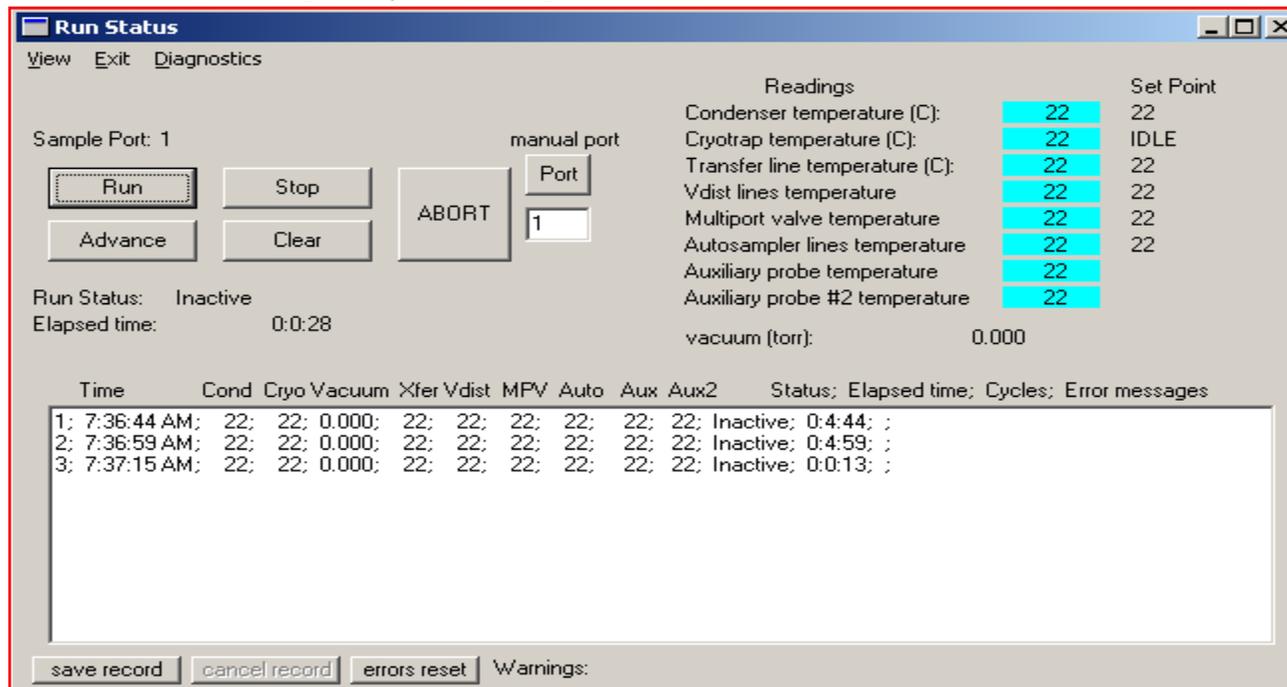
Display Instrument Readings

- View system status (Run ->Status)

Vacuum Distillation Main (3/19/03)

Run Method Communication Config Diagnostics

   Status

   Setup Sequence

- Software will display temperatures and vacuum

Run Status

View Exit Diagnostics

Sample Port: 1

manual port

Port

1

| Run | Stop | ABORT |
| Advance | Clear | |

Run Status: Inactive

Elapsed time: 0:0:28

Readings

	Readings	Set Point
Condenser temperature (C):	22	22
Cryotrap temperature (C):	22	IDLE
Transfer line temperature (C):	22	22
Vdist lines temperature	22	22
Multiport valve temperature	22	22
Autosampler lines temperature	22	22
Auxiliary probe temperature	22	
Auxiliary probe #2 temperature	22	

vacuum (torr):   0.000

Time	Cond	Cryo	Vacuum	Xfer	Vdist	MPV	Auto	Aux	Aux2	Status; Elapsed time; Cycles; Error messages
1; 7:36:44 AM;	22;	22;	0.000;	22;	22;	22;	22;	22;	22;	Inactive; 0:4:44; ;
2; 7:36:59 AM;	22;	22;	0.000;	22;	22;	22;	22;	22;	22;	Inactive; 0:4:59; ;
3; 7:37:15 AM;	22;	22;	0.000;	22;	22;	22;	22;	22;	22;	Inactive; 0:0:13; ;

| save record | cancel record | errors reset | Warnings: |

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")