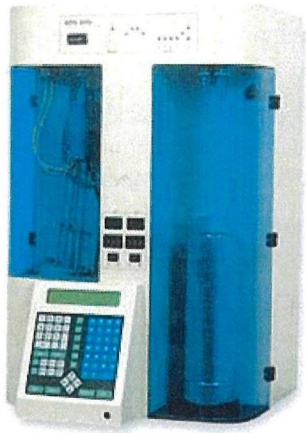
Quantachrome
INSTRUMENTS

NOVA OPERATION MANUAL



HIGH SPEED GAS SORPTION ANALYZER
VERSIONS 10.0-10.05

Instrument Model: 25-E, 26-E and 27-E

P/N 05069 Rev P

QUANTACHROME WARRANTY POLICY

Quantachrome Instruments warrants its instruments to be free from defects in material and workmanship for a period of one year from date of shipment under normal use and conditions.

For the period commencing with the date of shipment and ending one year later, Quantachrome will, at its option either repair or replace any part within an instrument that is found by us to be defective in material or workmanship, without charge to the customer, at our facility or at a customer's facility if the instrument purchased is backed by Quantachrome's on-site warranty as evidenced by the sales contract.

The customer is responsible for all transportation charges to our factory.

Damages during the warranty period resulting from unstable utilities, operator error or unauthorized repairs will not be covered by this warranty.

Parts purchases are warranted to be free of defects for 90 days from shipping date.

The following limits apply to our warranty:

Glassware, including Dewar flasks, is not covered under this warranty except if damaged during shipment. Claims for damage during shipping must be made in writing within 10 days of receipt of the goods.

Expendable items are warranted for 90 days for other than glassware breakage. Such items include, but are not limited to, sample tubes, lamps, fuses, valve plungers, seals, o-rings & other seals, hoses, flexible tubing, thermocouple vacuum tubes, filters, oils and other fluids.

Products sold by Quantachrome under their own brand name are not warranted by Quantachrome, but our best effort will be made to secure repair or replacement if found to be defective.

Warranty becomes void in the event of modifications or repairs by persons other than Quantachrome's service personnel, unless permission is given in writing for such repairs or modifications. Warranty is also void in the event of exposure to corrosive atmospheres of any kind.

Prior authorization must be obtained before returning any item to Quantachrome Instruments. Items must be decontaminated before return to Quantachrome.

Any and all computer program(s), software, firmware, code, data acquisition and/or data reduction methods, computations, graphical and/or tabular data reporting or presentation methods (collectively referred to as software) provided with or in or loaded into any part of the instrument or a computer supplied as part of a Quantachrome Instruments' instrument are provided "as is". Quantachrome Instruments warrants the software will perform substantially in accordance with the accompanying materials for the period of this warranty. All software is licensed and not sold. Quantachrome Instruments retains all rights to all software. You may not reverse engineer, decompile, or disassemble the software. You may not distribute or copy the software, except for the purpose of storing a back-up copy. The exclusive remedy for any software failing to perform substantially in accordance with the accompanying materials shall be, at Quantachrome's option, a) repair or replacement of the software; b) return of the amount paid for the software. You are not entitled to any damages, including but not limited to incidental, punitive, indirect or consequential damages including but not limited to loss of profits, business interruption, personal injury, and other pecuniary losses.

QUANTACHROME INSTRUMENTS' TERMS AND CONDITIONS OF SALE

Terms: Acceptance of this order is conditional on agreement by the Buyer to all terms herein. Buyer's agreement is assumed unless we are notified to the contrary prior to our invoicing. None of the provisions, terms and conditions contained in this document may be added to, modified, superseded or otherwise changed except by a written instrument signed by the Seller's authorized representative, regardless of any contrary or additional provisions contained in any purchase order or other form from the buyer.

FOB Point: The FOB Point is Boynton Beach, FL unless otherwise indicated on our Quotation or agreed to in writing by an authorized Seller representative.

Payment terms: Terms are net 30 days from date of invoice for established accounts were Seller has received adequate financial information to warrant extending credit to Buyer. Purchase orders from customers not meeting with Seller's financial approval are not accepted under credit terms.

Delivery: Delivery dates are Seller's best estimate of the time products will be shipped from our factory and we assume no liability for loss, damage, or consequential damages due to delays. No liability shall result from delay in performance or non-performance of this agreement, directly or indirectly caused by fire, explosion, accidents, flood, labor trouble or shortage, war, act of God or arising from contingencies, happenings, or causes beyond the control of the party affected.

Material Shortages: In the event of inability for any reason to supply the total demands for the materials specified in this order, Seller may allocate its available supply among any or all purchasers, as well as departments and divisions of the Seller, on such basis as it may deem fair and practical, without liability for any failure or performance which may result there from.

Taxes: Buyer shall reimburse Seller for all taxes, excises or other charges which seller may be required to pay to any government (National, State or Local) upon the sale, production or transportation of the commodities sold hereunder and amended from time to time.

Proprietary Information: All specifications, data, drawings, designs and software are proprietary information which are the sole and exclusive property of the Seller and purchaser agrees to retain any and all such proprietary information as Seller may disclose to him in confidence and not disclose it to other parties or use it except in inspection and evaluation purposes in connection with a contract with Seller.

Software License: Title to all software provided as separate modules or embedded in the Products ("Software") shall remain the property of Quantachrome or Quantachrome's licensors. Quantachrome grants to Buyer a non-exclusive, limited license to use the Software together with the Products. Buyer shall not decompile, disassemble or otherwise reverse engineer the Software, and may create derivative works only to the extent permitted by Quantachrome. Buyer shall not sublicense, assign, copy, distribute or disclose any portion of the Software to a third party without the express written consent of Quantachrome. Buyer may transfer or sell its license rights to use the Software only together with the Products to a transferee, which has accepted this provision of Quantachrome's Terms and Conditions of Sale in writing.

Changes: Orders accepted by the Seller cannot be changed or modified in whole or part, except with the written consent of Seller.

Return Policy: Prior approval must be obtained from an authorized Seller representative to return any Product. Seller will assign a return authorization number and record the reason for return. Seller will examine returned part to determine the actual cause, if any, leading to Buyer's return. If Product has a manufacturing defect Seller will, at its discretion, repair or replace with like product.

Restocking charges: Product purchased, under the sole discretion of the Seller, may be returned for a 20% restocking fee and must be returned within 90 days of date of delivery. Product cannot be returned if used or modified.

Claims: Claims for shortages or damage must be reported within 10 business days after receipt of shipment. All claims for loss or damage in transit must be made against the carrier.

Warranty: Quantachrome Instruments warrants all instruments that it manufactures for a period of twelve months from the date of delivery. This warranty includes all parts and labor. Quantachrome does not warrant any product against damage from corrosion, contamination, misapplication, improper specification, or wear and tear and operational conditions beyond the Seller's control. This warranty excludes all glassware and expendable items associated with each instrument. Repairs made during the warranty period are guaranteed until the end of the warranty period or 90 days, whichever is greater.

TABLE OF CONTENTS

This instruction manual refers to any NOVA 10.0 or higher Series instruments, Models 26 (**Helium Mode Capable**) and 25 (**NOT Helium Mode Capable**).

I. TECHNICAL SPECIFICATIONS

NOVA INSTRUMENT

ELECTRICAL

Voltage:	100, 120, 220, 240 V (see nameplate on rear of unit)
Frequency:	50/60 Hz
Power (max):	140 W
Connection:	Grounded, single-phase outlet

PHYSICAL

Height:	79 cm (31 in)
Width:	51 cm (20 in)
Depth:	51 cm (20 in)
Weight:	38 kg (83 lbs.)
Bench space allocation *:	104 cm (41 cm)

* Both doors open fully.

ENVIRONMENTAL

Temperature:	15 °C - 40 °C
Max. Relative Humidity:	80 %

HEATING MANTLE

ELECTRICAL

Voltage:	110-120V
Frequency:	50/60 Hz
Power (±10%):	
Glass-fiber	108W
Quartz-fiber	125W

ENVIRONMENTAL

Temperature:	15 °C - 40 °C
Max. Relative Humidity:	80 % (non-condensing)

II. SAFETY INSTRUCTIONS

SYMBOLS USED IN THIS MANUAL

HOT! This sign denotes a possible hazard to the operator due to high temperatures.

CAUTION! This sign denotes a hazard that could result in damage to the instrument.

WARNING! This sign denotes a hazard that could result in injury to the operator.

NOTE! This sign denotes an important operational detail.

TOOLS This signifies that tools are required for the highlighted action.

SAFETY INSTRUCTIONS FOR THE NOVA

- This instrument has been designed for laboratory use only.
- The NOVA requires a trained operator to use the instrument.
- This instrument must not be used for any application other than that for which it was designed.
- When filling Dewar Flask with liquid nitrogen (LN_2) care must be taken to prevent it from getting between the glass insert and the outer cover. This can cause the glass to implode.

- Because the dewar flask can shatter unexpectedly, a protective shield, safety glasses, and gloves should be worn when filling the flasks.
- When using a gas other than N_2 not at its boiling point, do not use the Calculate P_0 option while calibrating the empty cell or during analysis.

- Never handle hot mantle, cells or clamps with your bare hands.
- Operate this instrument only at the voltage specified on the name plate on the rear of the instrument.
- Inform yourself regarding hazards associated with the sample under test.
- Inform yourself regarding hazards associated with the gas(es) used.
- This instrument must be disconnected from the mains for any cleaning, maintenance or service.
- Do not make any unauthorized modifications to this instrument.
- When attaching a plug to the power cord, be sure to follow the color code shown below:

  Outside North America: **Brown = live, blue = neutral, green/yellow = earth ground**

- Do not operate the instrument if DIP Switch # 8 has been toggled (see Chapter VI Section 4.0 of this manual) as this will bypass the safety abort routine in the event of a power failure to the instrument or an aborted run.

SAFETY INSTRUCTIONS FOR THE HEATING MANTLE

- Quantachrome heating mantles are designed for heating sample cells for the purpose of outgassing solid samples and only on Quantachrome instruments equipped for the same purpose. Use only on instruments with properly functioning, calibrated heating mantle stations.

- Insert the power plug of a heating mantle only into the socket provided for that purpose on a Quantachrome instrument. Do not insert the power plug of a heating mantle into any mains supply socket. Do not insert the plug of any other device into the heating mantle socket on the Quantachrome instrument.

- Insert the thermocouple plug of a heating mantle only into the socket provided for that purpose on a Quantachrome instrument. Do not insert the thermocouple plug of a heating mantle into any other socket. Do not insert the plug of any other device into the thermocouple socket on the Quantachrome instrument.

- If the instrument has more than one heating mantle station, always ensure that the power plug and thermocouple plug of one heating mantle are inserted into the sockets of the same heating mantle station. Do not insert the power plug of a mantle into the power socket of one heating mantle station and the thermocouple plug of the same mantle into the thermocouple socket of a different heating mantle station.

- The heating mantle must have a sample cell in the pocket when in use. Insert only Quantachrome sample cells into a heating mantle. Ensure that no sample adheres to the outside of the sample cell. Do not use wet, broken, cracked or chipped sample cells. Do not insert spatulas, screwdrivers or other objects which are not Quantachrome sample cells into a heating mantle. Never place the sample to be outgassed directly into the heating mantle.

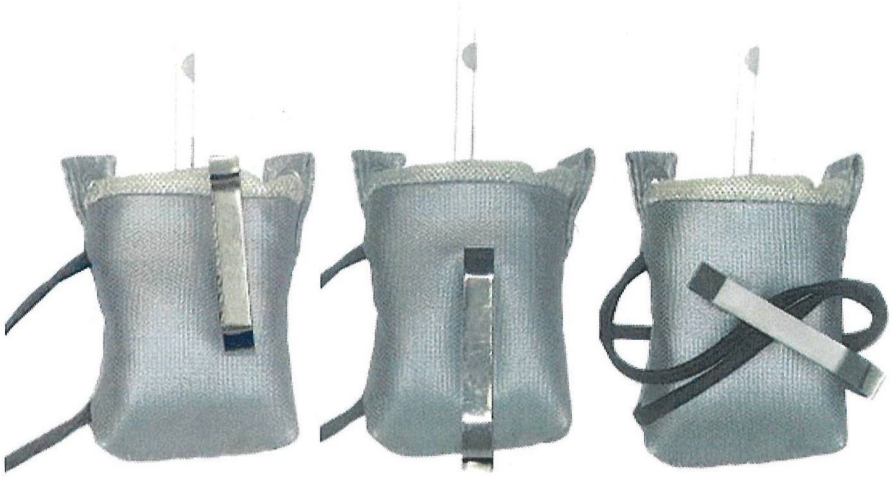
- The outer surfaces of the heating mantle may become hot during use. Do not hold hot heating mantles without wearing protective gloves. Never insert fingers inside the pocket to determine if the mantle is heating up. Do not place a hot heating mantle on a surface which is not heat resistant. Switch off when not in use.

- Do not allow liquids to come into contact with the heating mantle and do not handle heating mantles with wet hands. Do not allow dust to accumulate on, nor in, a heating mantle. Do not expose the heating mantle to a corrosive atmosphere of any kind.

- Do not make any unauthorized modifications to any Quantachrome heating mantle. Do not remove the serial number tag. Removing the tag will void the warranty.

- Make sure that mantle clamps are correctly situated when the heating mantle is on the sample cell (see examples on the next page)

CORRECTLY SITUATED MANTLE CLAMP AND CELL



INCORRECTLY SITUATED MANTLE CLAMP AND CELL

III. INSTALLATION

3.1. UNPACKING THE NOVA

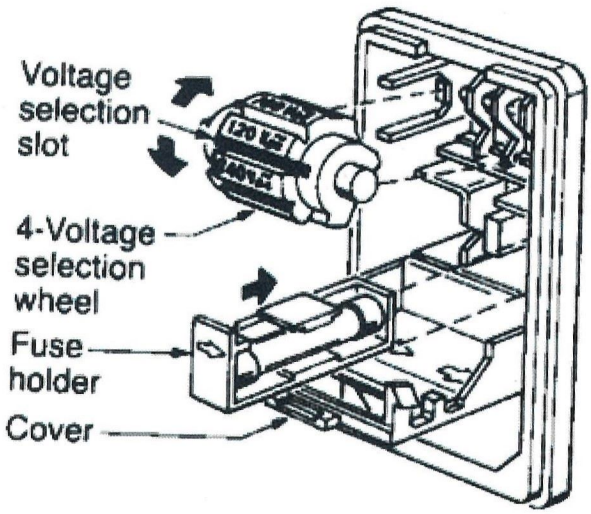
Remove the instrument from its shipping box and place it on the bench where it will be operated. Take care during this operation since the unit weighs 38 kg (83 lbs). Unpack the supplied parts and accessories and compare them to the packing list. If any parts are missing or damaged immediately notify QUANTACHROME or your local authorized representative.

3.2. OPERATION VOLTAGE

Tools required: Flat-head screwdriver, pliers

The NOVA was shipped for operation on the voltage specified in the purchase order. However, it is designed for operation on 100V, 120V, 220V or 240V, 50/60 Hz. The following steps describe how to use the voltage converter.

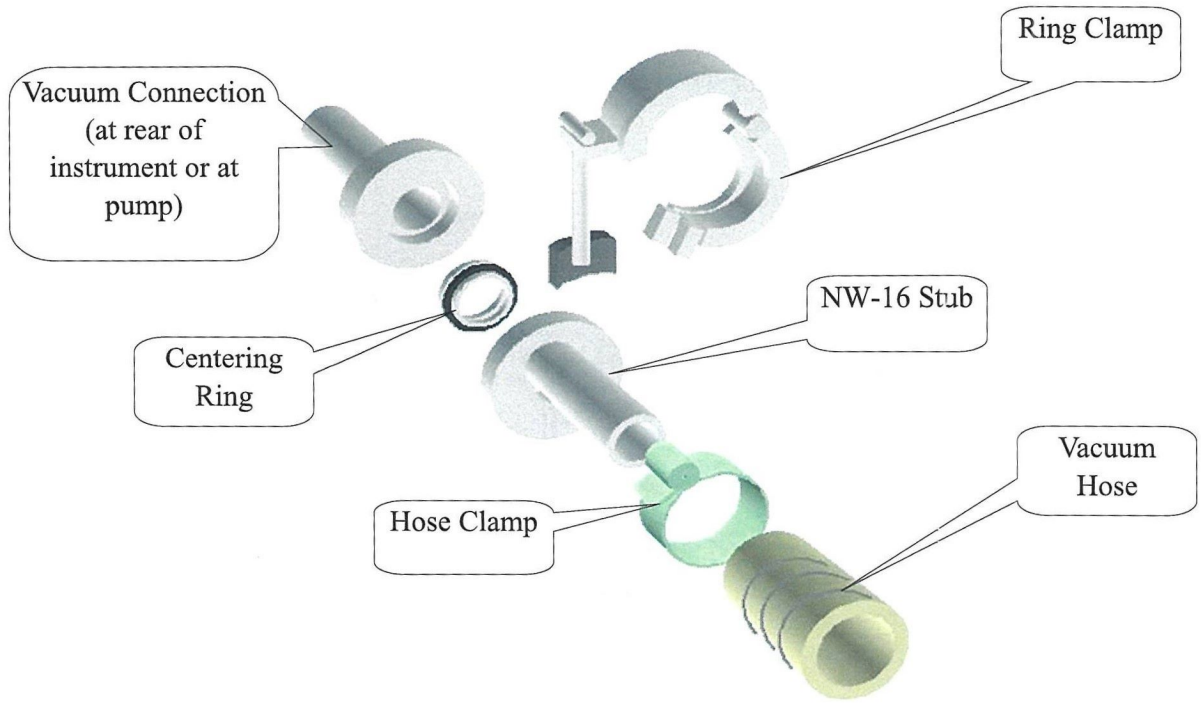
1. Remove the line cord from its socket on the instrument.
2. Pry open the plastic cover by inserting a screwdriver behind the tab on the side away from the socket.
3. Using long nose pliers, gently pull out the wheel located behind the plastic cover.
4. The 4 voltages are printed on the wheel. Reinsert the wheel with the voltage required facing out, toward the operator. Then close the plastic cover.



3.3. VACUUM CONNECTIONS

Tools required: Flat blade or 5/16" wrench

The NOVA is supplied with a KF-16 flange, centering ring and clamp to connect the vacuum hose to the fitting at the rear of the instrument. If the vacuum pump is supplied with the NOVA, a matching set of hardware is supplied to connect the hose to the pump. The vacuum pump should be placed at the rear of the NOVA to facilitate the connection. A pump capable of reaching 10 millitorr should be selected.



3.4. GAS CONNECTIONS

Tools required: 7/16" wrench

Nova 10.0 with Helium Mode option (Helium Mode capable - model 26 only) has two gas input fittings marked ADSORBATE and HELIUM at the rear of the instrument, respectively. Helium gas must be connected for the measurements conducted in the Helium Mode (that is, when the helium void volume measurement is performed during analysis). Model 25 (**NOT** Helium Mode capable) has gas input fitting for ADSORBATE only.

It is recommended to use high purity helium (model 26 only) and adsorbate gases (99.99% or higher). Attach a dual stage regulator with stainless steel diaphragm to the tank. For details on changing the gas tank, see Chapter VI, Section 3.6 of this manual. Connect the regulator to the gas-input connector at the rear of the unit using the 1/8" copper tubing and nut & ferrule set supplied. It may be necessary to obtain an adapter in order to connect the tubing to the regulator. A suitable regulator assembly (P/N 01207) complete with shut-off valve, CGA580 cylinder fitting and 1/8" outlet fitting is available from Quantachrome. The regulator should be set to deliver 10 PSIG (70 kPa) for any gas.

Note: The regulator assembly with part number 01207 or 01207-I is suitable for inert gases such as He, Ar, and N_2. The part number for CO_2 gas is 01207-CO2.

3.5. DEWAR FLASK AND CELL FITTINGS

All NOVA instruments are supplied with the NOVA-e Dewar (P/N 04000-7224), which will hold LN_2 for more than 30 hours. The dewar is placed in the lift-drive cup.

Note: The dewar manufacturer recommends that the following procedure be adopted:

(i) Prior to first use, all the packing material from the inside of the dewar must be removed.

(ii) Wash the dewar with hot, soapy water, rinse with distilled or deionized water, and either air dry or use a lint free towel. This is to ensure a clean, dry, and contaminate free dewar.

3.6. SAMPLE STATIONS AND DEGASSING STATIONS

The sample stations and degassing stations must have the stainless steel dowels inserted in them before turning the NOVA on. The O-Rings and cell adapters for the 9 mm cells are used with the stainless steel dowels.

A liquid nitrogen level sensor is provided. It should be installed in the bayonet style fitting (BNC connector) adjacent to the sample station(s). Take care not to damage the tip.

3.7. ENHANCED KEYBOARD

The NOVA can be used with a standard PS-2 enhanced keyboard (available from Quantachrome; P/N 38056). The connection for the enhanced keyboard is located below the NOVA keypad. Note that the keypad will also be operational when the keyboard is connected to the NOVA.

3.8. NOVAWIN OPERATING SYSTEM / DATA ANALYSIS PACKAGE

NOVAWin is a Windows-based comprehensive program that allows for the integration of the NOVA Series instruments to a remote PC. The program serves the dual function of setting up measurement parameters and providing a platform for enhanced data analysis in the generation of reports including graphical plots and tables. NOVAWin also allows for the direct communication with the NOVA Series instruments and a PC with the automatic uploading of data points as they are acquired throughout the course of the measurement. An enhanced version, NOVAWin-"P" is also available with configurable security features that conform to 21 CFR Part 11 (electronic records) as mandated by the FDA for use in the pharmaceutical industry.

In summary, the following actions are possible using a PC installed with NOVAWin or NOVAWin-P:

- Cell calibrations
- Surface area determinations
- Pore size distribution measurements
- Data archiving
- Report generation including graphical plots and tables

NOVAWin-P incorporates the above features along with advanced security features which include:

- Tamper-evident data files
- Required system login with unique user name and password combination
- Fully detailed audit trail
- 3 access levels programmable by system administrator
- Programmable session time-out (auto logoff)
- Unique report identification

NOVAWin can be installed on any Windows-based PC (Windows 2000 and up). **Make a backup up copy of the CD before proceeding with the installation.** Start the Windows program and click on the START button on the lower left-hand corner of the screen. Select RUN from the menu. This will begin the installation of NOVAWin to your PC. When the installation is successfully completed, remove the backup CD and store it in a safe location (preferably not the same place as the original distribution CD is stored). Refer to the NOVAWin Operation Manual (P/N 05079) for more details on the software installation.

IV. DESCRIPTION OF INSTRUMENT CONTROLS

4.1. POWER SWITCH

The power switch is located on the left side panel of the NOVA. In addition to supplying power to the instrument, the switch may be used to safely abort a run in progress if keypad/keyboard connection is lost by momentarily turning it off and then on. Press 1 to switch ON and O to switch OFF.

4.2. VACUUM FITTING

The vacuum fitting is located at the rear of the NOVA. It is a standard KF-16 fitting. The connecting hose, with the necessary clamp, flange and centering ring is supplied with the instrument. A vacuum pump capable of achieving at least 50 millitorr is required. A 10 millitorr vacuum pump is recommended.

4.3. PRINTER PORT AND SERIAL PORT

A parallel printer port is located at the rear of the instrument. No printer drivers are required. ASCII text is sent to the port. Only DOS-based parallel port printers may be used.

Note: The printer cable is **not** supplied with the NOVA.

A 9-pin RS-232 serial port is located at the rear of the instrument. This port is used to connect the NOVA to a PC for use with the optional NOVAWin/-P software (refer to the NOVAWin Operation Manual, P/N 05079 for details on connecting the NOVA to a PC).

4.4. VIDEO PORT

An SVGA video port has been added to the NOVA which allows you to monitor the instrument during calibration and analysis in a more detailed manner. Video monitor is **not** supplied with the NOVA.

4.5. GAS CONNECTIONS

The NOVA 10.0 series Model 26 (helium mode capable) has two gas input fittings at the rear of the marked ADSORBATE or HELIUM, where the tanks of Adsorbate and Helium are connected respectively. Nova 10.0 series Model 25 (**NOT** helium mode capable) has one gas input fitting and is connected to tank of adsorbate **only**. Six feet (2 m) of copper tubing with a compression fitting on one end are provided with the instrument. The fitting should be attached to the gas-input connector using a 7/16" wrench. Attach the other end of the tubing to the gas regulator on the gas tank using an appropriate nut and ferrule set. If the pressure regulator does not have the proper fitting, an adapter must be obtained. All regulators supplied by Quantachrome are equipped with the proper adapter. Set the regulator to provide 10 PSIG (70 kPa) output to the NOVA.

4.6. DEWAR FLASK

The dewar flask holds the coolant, usually liquid nitrogen, used for the analysis. The flask should be filled to the top prior to each analysis.

4.7. LCD DISPLAY

The LCD display is a 4 X 40 (4 lines by 40 characters) backlit display, located in the control panel. It displays menus, messages and analysis information as necessary. When reading pressure, it shows the manifold pressure (in mm Hg) at the bottom of the display. This allows you to monitor the instrument's progress throughout the analysis.

4.8. KEYPAD

The keypad, located in the control panel, is used for all user input to the NOVA. Menu selections are made by pressing only the number of the menu desired. When data, such as user or sample ID is being entered, the ENTER key must be pressed after the required information has been entered. The 'backspace' key can be used to correct mistakes when entering data, before the ENTER key has been pressed. Pressing "0" or "0" and ENTER will cancel an operation and return you to the previous menu.

4.9. KEYBOARD CONNECTOR

A mini-DIN connector is provided on the front of the NOVA to connect an optional enhanced PS-2 alpha/numeric keyboard (P/N 38056).

4.10. TEMPERATURE CONTROLS AND DISPLAY

The temperature control and display for each degassing station is located on the central divider. The digital selector is used to set the desired degassing temperature in °C. The LED display shows the actual temperature of the heating mantle when the thermocouple is plugged into its socket. The mantles will heat only when the rocker switch is ON. The temperature will still be displayed when the switch is OFF, so that the temperature of the mantle will always be monitored, even when cooling down. The maximum allowable temperature is 350 °C (using standard glass heating mantles as supplied with the instrument). Optional quartz heating mantles (P/N 24021-CE) are available which permit degassing temperatures up to 450 °C.

The heating mantles will not operate if the thermocouple is not plugged into its jack. In this case, the display shows a value above 900, indicating that the thermocouple is not connected or is defective.

4.11. HEATING MANTLES AND OUTLETS

Two heating mantle power outlets are provided in the degassing compartment. The thermocouple plugs are inserted into the jacks located in the same compartment. The heating mantles will not operate unless both the power cord and thermocouple are plugged in.

Note: Always ensure that the power plug and thermocouple plug of one heating mantle are inserted into the sockets of the same heating mantle station. Do not insert the power plug of a mantle into the power socket of one heating mantle station and the thermocouple plug of the same mantle into the thermocouple socket of a different heating mantle station. For safety instructions regarding the heating mantle, see Chapter II.

4.12. SAMPLE CELLS AND GLASS FILLER RODS

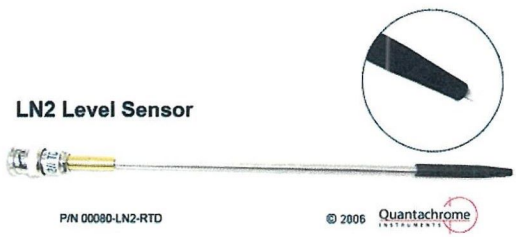
Sample cells are available with outside stem diameters of 6, 9 and 12 mm (internal diameters of 4, 7, and 10 mm respectively). Each cell should be numbered in the space provided (if run in Nova Mode) and used with the appropriate glass filler rod. After performing a cell calibration, you should ensure that the same filler rod is always used for that particular station. For most users, standard (bulbless) cells of a given stem diameter can be given a single identifying number and used interchangeably. Pre-calibrated cell sets are supplied with the NOVA.

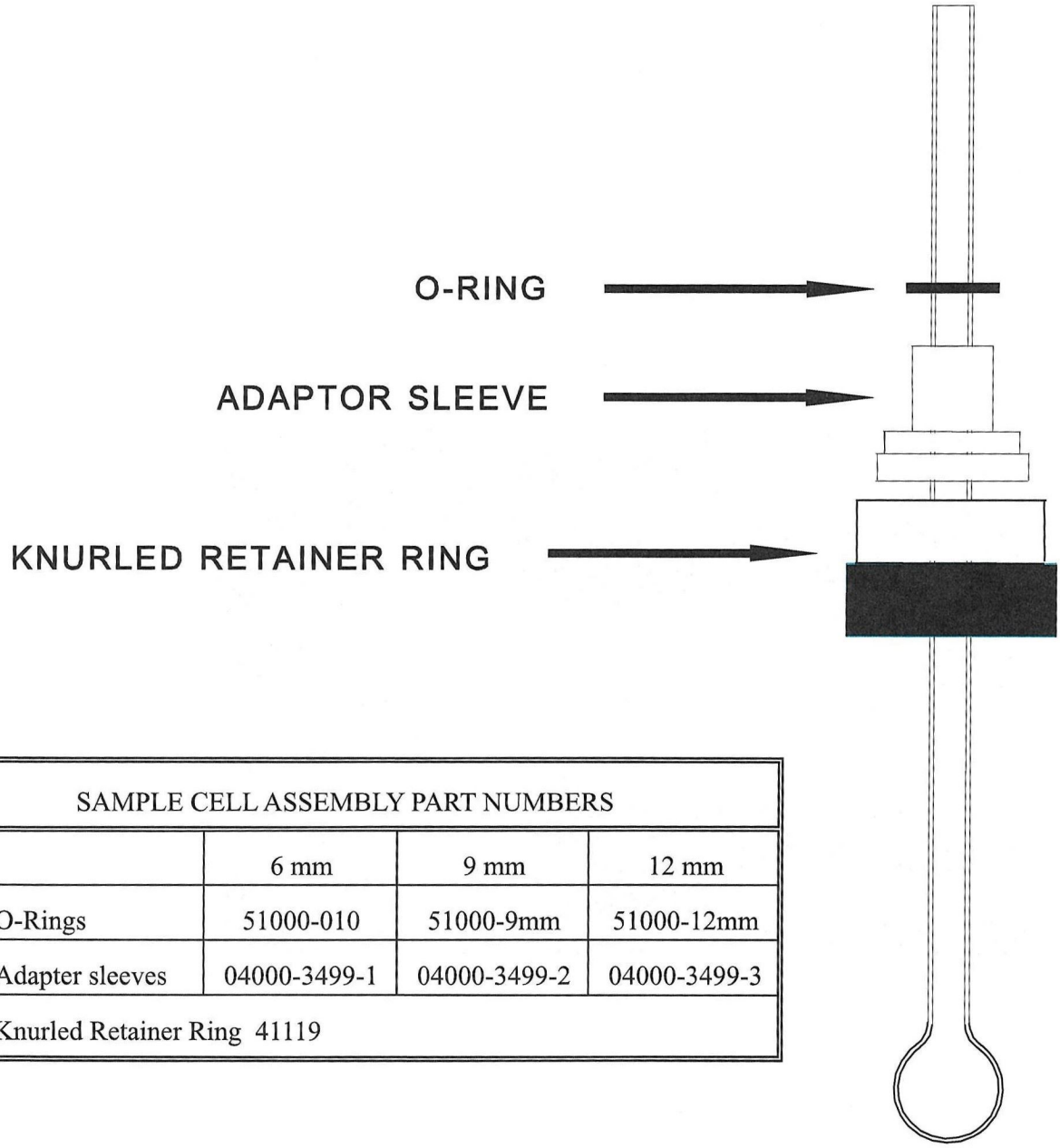
4.13. CELL BULKHEAD FITTINGS

The fittings for holding the sample cells use an adapter sleeve, and an O-Ring and are designed to accept 6, 9, and 12 mm size cells. The correct size O-Ring and adapter must be used with each corresponding cell. Be sure to use only one O-Ring when installing a cell into the fitting. Two O-Rings (of any size) are likely to cause a leak leading to erroneous results.

4.14. COOLANT LEVEL SENSOR

Coolant level is maintained at a constant level around the cell, at a position which minimizes the so-called "cold-zone" of the cell (thereby improving sensitivity). The level is maintained using a coolant level sensor (see diagram on the right) and a circuit which adjusts the dewar height to compensate for the evaporation of coolant during the analysis. The NOVA is supplied with a level sensor suitable for liquid nitrogen. Other level sensors are available to NOVA1200/2200/3200/4200 users for liquid argon coolant, dry ice/acetone slush bath, and a water float sensor for measuring isotherms, for example, at 0 °C using ice/water. NOVA 1000/2000/3000/4000 users only need the LN2 sensor.





SAMPLE CELL ASSEMBLY PART NUMBERS			
	6 mm	9 mm	12 mm
O-Rings	51000-010	51000-9mm	51000-12mm
Adapter sleeves	04000-3499-1	04000-3499-2	04000-3499-3
Knurled Retainer Ring 41119			

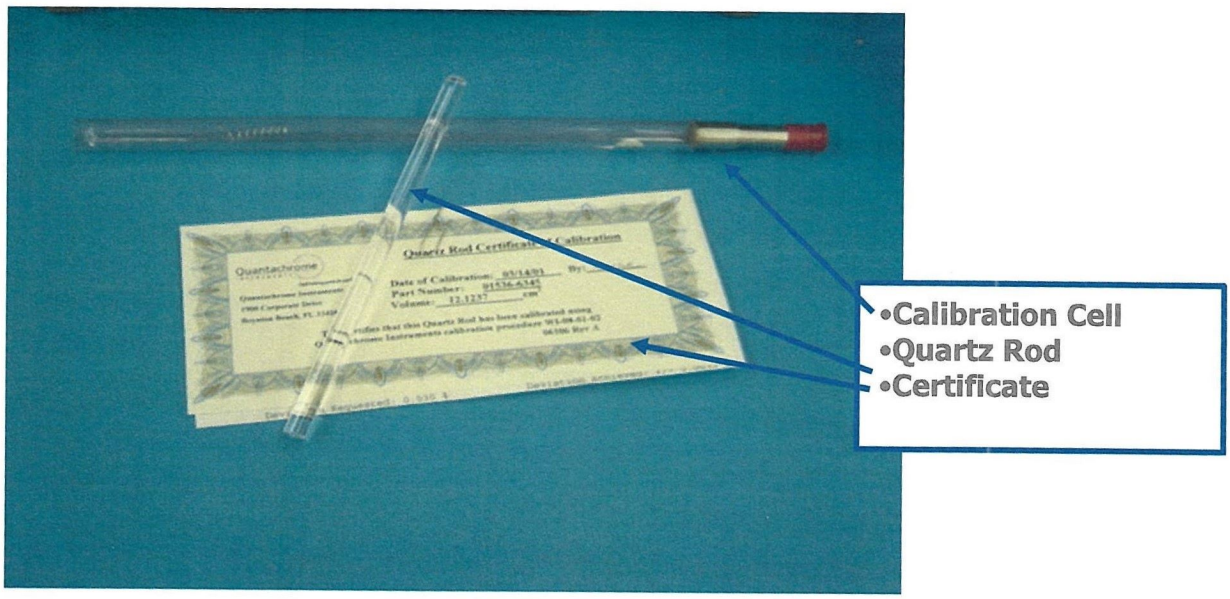
SAMPLE CELL ASSEMBLY

4.15. QUARTZ ROD CALIBRATION KIT

Manifold calibration is performed using Quartz Rod. See Chapter VI, Section 2.3 for manifold calibration instructions.

The calibration kit includes:

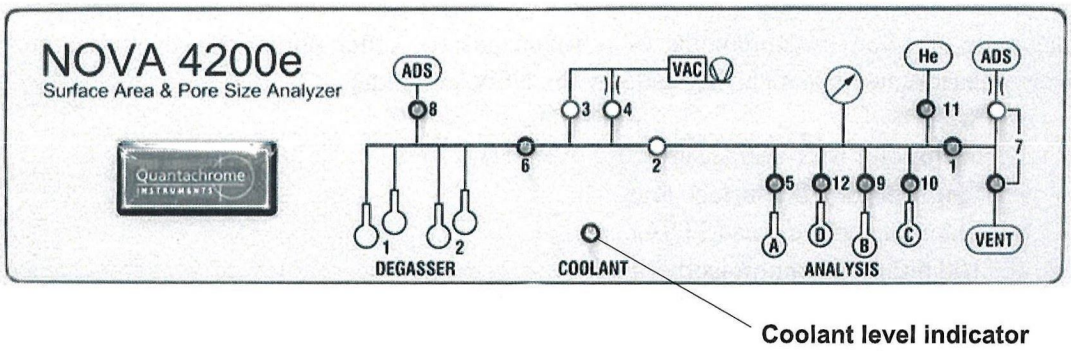
- Calibration cell with spring
- Calibrated quartz rod
- Company certificate for quartz rod volume



4.16. NOVA FLOW DIAGRAM

The diagram is displayed at the top front of the instrument. Valves are represented by LEDs. LED ON indicates an open valve and the LED OFF indicates a closed valve. V7 indicates the status of a 2-way valve, V11 (fine valve) controls the Helium supply to the manifold (helium capable, model 26 only). The coolant level indicator illuminates when the coolant makes contact with the tip of the level sensor.

The flow diagram below is for the NOVA 4200e Series, showing four analysis stations. The NOVA 1000 Series instruments have 1 analysis station, the NOVA 2000 Series instruments have two analysis stations and the NOVA 3000 Series instruments have three analysis stations.



NOVA 4200e SERIES FLOW DIAGRAM

Note for <u>Helium Mode capable</u> Nova 10.0 Series (Model 26) only: When the instrument is operating in the Helium Mode, the valve V11 is shown in the Manual Mode Menu.

V. INSTRUCTIONS FOR USE - OVERVIEW

NOVA is an acronym for **NO** **V**oid **A**nalysis. The NOVA Series instruments will perform rapid and accurate sorption measurements of nitrogen gas (or other non-corrosive gases) on solid surfaces. Measurements that can be made by the NOVA include:

1. Multipoint BET surface area.
2. Single point BET surface area.
3. External surface area (STSA).
4. 100 point adsorption isotherms.
5. 100 point desorption isotherms.
6. Total pore volume.
7. Average pore radius.
8. BJH pore size distribution based on the adsorption or desorption isotherm.
9. Approximate sample volume and density.

Other calculations may be performed using the optional NOVAWin Operating System / Data Analysis Package.

Model 26 of NOVA 10.0 line of instruments offers two modes of the cell void volume determination: Cell Calibration (Nova Mode) as well as the classical void volume determination with Helium gas (Helium Mode). Models 25 of NOVA 10.0 line of instruments support only the Nova Mode (Cell Calibration required). Application of the revolutionary theory in NOVA (**NO** **V**oid **A**nalysis) method (see chapter VIII for the theoretical basis used in the Nova Mode operation) permits measurements omitting pre-determination of cell void volume before each analysis. Additionally, availability of Helium Mode (Model 26 only) in NOVA 10.0 line of instruments expands its feasibility. Consequently, the unique design and dosing algorithms of the NOVA make it an extremely rapid, versatile and accurate sorption analyzer. The NOVA Series consists of 8 different instruments that differ from each other only marginally as far as operation is concerned.

Please read this page carefully before starting any operation with your new NOVA analyzer, and familiarize yourself with the specific functions and capabilities of the instrument model.

The fundamental difference between the 1000, 2000, 3000 and 4000 Series instruments is that they have 1, 2, 3 or 4 analysis stations respectively. While working with the setup and operation menu using the keyboard for the 1000 Series, the instrument will never prompt you to enter the station identification. When using the 2000 Series, the menu will require you to select either/or stations A & B, and for the 3000 Series, the options provided will be Stations A, B, & C. The NOVA 4000 Series instruments have the option of Stations A, B, C, and D. When using the 1000, 2000, 3000, or 4000, the instrument will not require the operator to enter any gas parameters since these instruments are pre-programmed for N_2 gas sorption at liquid nitrogen temperature only. However, for the 1200, 2200, 3200, and the 4200, the instrument will prompt you to enter gas parameters.

8 Different Instruments in the NOVA Series*

Instrument	Number of Analysis Stations	Number of Degas Ports	Gas Compatibility
NOVA 1000	1	2	N_2
NOVA 1200	1	2	Any Gas*
NOVA 2000	2	2	N_2
NOVA 2200	2	2	Any Gas*
NOVA 3000	3	4	N_2
NOVA 3200	3	4	Any Gas*
NOVA 4000	4	4	N_2
NOVA 4200	4	4	Any Gas*

* Any Gas: any non-corrosive gas compatible with materials of construction (glass, copper, Buna).

ANALYZING A SAMPLE

Follow these steps before conducting any analysis. Refer to the appropriate sections mentioned for instructions on conducting the required procedure. Manifold and cell calibrations need only be conducted infrequently. For operation in the Nova Mode, after the instrument/cell sets are properly calibrated, you will only need to degas your sample and run it with the appropriate setup.

5.1. MANIFOLD CALIBRATION

The dosing manifold is factory calibrated. You should check this calibration periodically (for example, once every month) or if changes to the system have been made. However, you should be familiar with the procedure. Refer to Chapter VI for detailed instructions.

Note: There is no need to perform a manifold calibration before every analysis.

5.2. CELL VOID VOLUME DETERMINATION

5.2.1. Helium Mode (model 26 only)

Measuring the void volume of the sample cell immediately prior to sorption measurements - *in the presence of sample* - using non-adsorbing helium is the classical method used in many non-vapor gas sorption instruments (See Section 5.2.1). This method avoids having to calibrate an empty cell in advance of a measurement but does require access to properly pressure regulated high purity helium (at least 99.99 %). If you want to use the Helium Void Volume method, please make sure that a Helium bottle is connected to the Nova 10.0 Model 26.

5.2.2. Sample Cell Calibration

Refer to Chapter VI for detailed instructions on the cell calibration procedure. This needs to be conducted for each sample cell + filler rod + station combination and for each adsorbate/coolant combination. For most users, all standard (bulbless) cells can be considered equivalent (for each diameter). Therefore, one cell/rod/station combination will suffice for a different cell (of the same diameter) with the same rod in the same station.

Note: "Ready Alarm" (series of rapid beeps) will sound when the Cell Calibration is completed.

Note: Once done, there is no need for further calibration for that particular combination.

Note for model 26 only: Cell Calibration option is unavailable during operation in the Helium Mode (as it is not required in this mode).

5.2.2.1. Selecting a Sample Cell

Two factors to consider when selecting a sample cell are stem diameter and sample amount / sample bulb size.

Stem diameter: Choose the narrowest diameter cell that will comfortably admit the sample. For example, a fine powder should be analyzed in a 6 mm outer diameter (o.d.) (4 mm i.d stem cell). Use the 12 mm o.d. stem cells for large pieces that cannot be reduced in size. Larger particles such as granules, and small pellets might require a 9 mm o.d. (7 mm i.d.) diameter stem. Cohesive powders may be analyzed in 9 or 12 mm stem cells to facilitate addition, removal and cleaning.

Sample amount/Bulb size: Always use the smallest bulb that will accommodate the optimal amount of surface area. Larger total surface areas can certainly be analyzed, but they may lengthen the analysis. For surface area determinations only, sample amounts from at least 1 m^2 to 5 m^2 can be analyzed using nitrogen, but careful consideration should be given to proper degassing and equilibrium criteria. If the total area available is less than 1 m^2, then the Low Area (LA) option should be used (if installed). Full adsorption and desorption isotherms should have at least 15 – 20 m^2 in the cell.

Wider stems and larger bulbs can be beneficial in reducing elutriation, see below.

5.3. SAMPLE PREPARATION

Every sample has to be degassed before analysis by flow or vacuum method. Refer to Chapter VI for detailed instructions on the NOVA degassing features.

Note: For all NOVA models, all degassing functions must be accessed via the NOVA keypad / keyboard (i.e. they cannot be accessed via NOVAWin2).

5.3.1. Methods of Sample Preparation

The two methods available for degassing samples on the NOVA are:

(1) Vacuum Degas: Weigh an empty cell, add sample (sufficient for 2-50 m^2 total area), place the sample cell in the pouch of the heating mantle, set clamp in place, insert cell into fitting, tighten fitting and loop elastic cords over hooks provided. Load the degasser and pull vacuum on the sample for at least 10 minutes. Next, set the temperature select to the required degas temperature (see below on choosing an outgassing temperature) and switch the heating mantle on. After sufficient time for complete outgassing, switch the mantle off. Select the backfill gas (Helium or Adsorbate). Allow sample cell to cool. Unload degasser when ready to analyze sample. Remove cell; reweigh to obtain dry, outgassed sample weight.

(2) Flow Degas: The apparatus shown in Figure 1 below (Flow Degasser Assembly) has to be attached for flow degas. Use 6 mm O-Ring and adapter sleeve to mount the metal insert in to the fitting. The gas flow rate can be set by placing the metal flow tube into a beaker of water and adjusting the needle valve to set a flow of 1-2 bubbles per second. The needle valve may have to be readjusted so those particles are not carried out of the sample cells. Turn the valve knob clockwise to reduce gas flow, turn valve counterclockwise to increase gas flow. Weigh an empty cell, add sample. Insert the body of the sample cell into the heating mantle. Place the flow outgas tube into the sample cell. Adjust the collar stop so that the end of the tube is approximately 0.5 cm above the sample with the collar stop resting on the rim of the cell stem. Do not allow the tube to dip into a bed of powder. Load degasser, set the desired outgassing temperature and switch the heating mantle on. After sufficient time for complete outgassing, switch the mantle off. Allow sample cell to cool. Unload degasser when ready to analyze sample (after the outgasssing procedure, user can choose to backfill with adsorbate or with Helium). Remove cell, reweigh to obtain dry, outgassed sample weight.

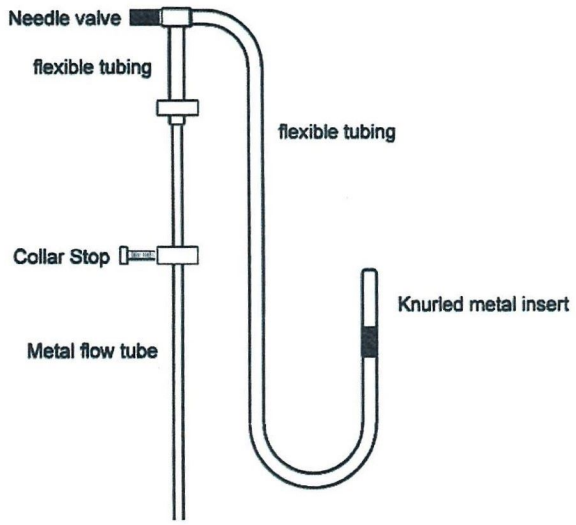


Figure 1: Flow Degasser Assembly

MasterPrep/FloVac Degasser Accessories: A sample can be considered ready for analysis when the sample passes a degas test of no more than 50 microns Hg per minute (at elevated temperature). A sample that cannot pass the same criterion at room temperature may not analyze accurately. Remember, the NOVA must be able to pull, and hold, a vacuum in the sample cell in the presence of sample. A contaminated MasterPrep or FloVac degas station may give artificially high degassing rates during test. You can establish the background pressure rise of a degas station by installing and testing a dowel pin or clean and empty sample cell. A clean system should be able to pass a 20 micron Hg per minute test. Always degas without a filler rod in the cell.

5.3.2. Choosing a Degas Temperature

Samples should be degassed at the highest temperature (up to 350 °C with standard mantles) that will not cause a structural change to the sample. This will accelerate the degassing process. For instance, most carbon samples can also be degassed at 300 °C, as can calcium carbonate. Many hydroxides must be degassed at a lower temperature. Degassing organics must be performed with care since most have quite low softening or glass transition points. For example, magnesium stearate, a common pharmaceutical formulating compound, should be degassed at 40 °C according to the USP.

Loosely bound water ("wet water") will be lost at relatively low temperatures under the influence of vacuum, but strongly bound surface water might require surprisingly high temperatures. Many zeolites, for example, will retain significant quantities of water in their micropores up to 300+ °C.

Use technical reference literature such as the Handbook of Chemistry and Physics (CRC, Boca Raton, Florida) and standard methods such as those published by ASTM to guide your selection of an appropriate degassing temperature.

If you have access to thermal analysis equipment, especially gravimetric, an analysis should be conducted on a separate aliquot of material prior to degassing on the Nova. A suitable degassing temperature would be that which lies in a plateau, or weight-stable region, of the thermogram. Ideally, the thermal analysis should be conducted under vacuum. In general, too low a degassing temperature will cause lengthy preparation, and may result in lower than expected surface areas and pore volumes.

Too high a temperature can cause irreversible damage to the sample, which can result in a decrease in surface area due to sintering, or an increase in surface area due to a thermally induced decomposition.

5.3.3. Degas Time and Testing for Complete Degassing

Time for complete degassing, that is complete removal of unwanted vapors and gases adsorbed on the sample surface, can only be properly determined by conducting a series of tests to determine those conditions of temperature and time which yield reproducible data. As a general guideline however, three hours (at temperature) should be considered a reasonable minimum. IUPAC recommend no less than sixteen hours, which can be conveniently achieved overnight. Samples that require low temperatures generally require the longest outgas times. However, the USP recommended degassing period for magnesium stearate is just two hours at 40 °C.

5.3.4. Elutriation and Its Prevention

Elutriation, or loss of powder out of the sample cell, is caused by too rapid a gas flow out of the cell. It is most problematical for low-density samples, fumed silica for example.

Wider stems and larger bulbs can be beneficial in reducing elutriation. Wider stems reduce the velocity of the gas leaving the cell when evacuation begins and thus it is less likely to entrain powder particles and transport them upwards and out of the cell. The presence of a filler rod significantly increases gas velocity because of the narrowing of the internal dimensions and can exacerbate elutriation. In problematical cases, the filler rod may be dispensed with during analysis, but some loss of resolution and/or sensitivity may result. A larger bulb than necessary significantly reduces gas velocity in the immediate vicinity of the sample (but does increase void volume) and allows the sample to move around without being entrained in the higher velocity up the stem portion.

The most dramatic elutriation problems are encountered during degassing of damp, "light" powders. As the sample heats from ambient, the pressure over the sample decreases due to the action of the vacuum. At some point the water "flashes" into steam. This rapid expansion of gas volume drives powder out of the bulb and up the stem of the cell. This condition can be reduced or eliminated by (i) pre-drying the samples in a conventional drying oven and (ii) raising the temperature of the heating mantle in 20 degree steps. It is recommended that the temperature be "paused" at 60 °C for 30 – 60 minutes under vacuum to allow for a milder removal of moisture before increasing the temperature to 80 °C, then 100 °C and finally maximum degas temperature.

Both degas and analysis stations are fitted with fixed filters that are pressed in to the "bulkhead" adapters. If these become contaminated, or it is desirable to change them for a finer filter, they can be easily removed and cleaned, or replaced entirely. A further flow restriction in the form of a tight fitting 20 micron filter called a "Cell-Seal" can be inserted into the stems of 9 mm and 12 mm sample cells.

In the most difficult cases, and the aforementioned methods have not eliminated the problem it might be necessary to insert a small glass wool plug into the cell stem. This can be held in place between two halves of a cut-in-two glass filler rod. This is the only time that a filler rod should be used in the degasser.

Note: For elutriating samples it is recommended to increase the range of fine evacuation. The Switch Vacuum option (System Manager 3.5.6) allows you to enter the value of pressure at which you wish to change evacuation rate from fine to coarse.

5.3.5. Unloading the Degasser

After the outgasssing procedure, user can choose to backfill with adsorbate or with Helium. Preferably, the adsorbate should be used as backfill gas to prevent or minimize buoyancy errors. A sample cell will weigh less when filled with helium than when filled with air or nitrogen. The error introduced is approximately 1mg per mL of cell volume. This can be significant when using extremely small sample weights (< 50mg).

Allow the mantle to cool below 100 °C before unloading the degasser. Remember, heating mantle clamps may be very hot. A sample cell which feels only warm to the touch whilst still under vacuum can be much hotter to the touch when backfilled with gas. This is particularly true if you are degassing a large mass of metal sample. Exercise caution! A warm sample cell can also introduce weighing errors. The sample cell should be allowed to cool to room temperature before weighing. If sample throughput permits, cool thoroughly while attached to the NOVA, otherwise remove and transfer to a desiccator.

5.4. SETUP ANALYSIS

You must define the analysis type, analysis conditions and the data reduction parameters before running the analysis. Refer to Chapter VI of this manual for details on creating a "SETUP" and/or a "PRESET".

5.5. RUN ANALYSIS

Running an analysis allows you to measure surface area, pore volume, and pore size distribution.

Slide the black Coolant Level Indicator (P/N 04000-7400) onto the lip of the dewar. The indicator hangs on the top lip of the dewar with the arrow-shaped point inside the dewar. Ensure the dewar flask is filled to the bottom point of the indicator. When using liquid nitrogen (or other cryogenic coolants), allow 5 minutes for the coolant to equilibrate for best results. If the nitrogen is still boiling heavily, then the dewar needs to be cleaned before filling it with LN2. If boiling continues after a dry clean dewar is filled for 5 minutes, then you may need to replace the dewar. Due to residual boiling, you may need to top-up to the bottom point again. Remove the indicator after establishing the proper coolant level.

Simply follow the instructions until you are prompted to proceed with the analysis. Transfer the cells from the degas station(s) adding the filler rod(s) so long as the cells were calibrated with them (recommended). Ensure proper alignment of sample cells with the dewar cap. When using sample cells with bulbs, the cells must be inserted through the cap prior to being attached to the analysis stations.

Note: "Ready Alarm" (series of rapid beeps) will sound when the analysis is completed.

5.6. PRINTING AND REPORTING OPTIONS

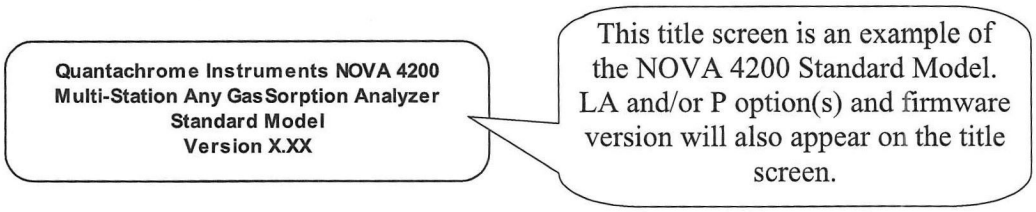
If a printer is attached (and switched ON), the NOVA instrument will automatically generate a printout of the data. For detailed printing of plots and tables you may also use the NOVAWin software.

VI. DETAILED OPERATING INSTRUCTIONS

START-UP

Note: Non NOVA-P instruments cannot be operated without the User Disk in place.

With the NOVA properly installed, vacuum pump connected, dewar flask, *and stainless steel dowels in place*, gas turned on (delivery pressure set to 10 PSIG), turn on the vacuum pump and the NOVA. The LCD window and degassing temperature displays will be tested. The dewar flask will start to rise, and will immediately return to its lower position. The title screen will appear.



After the program has been loaded, the main menu will be displayed. Press the ESC key on the keypad / keyboard to toggle between the Main Menu and Title Screen.

Note: If the power was turned off to abort an analysis or sample cell calibration, and the Dewar had already gone up, the following sequence of events will take place:

- The dewar lift mechanism will remain in its upper position.
- Cells will be evacuated and the lift mechanism will return to its lower position.
- After the NOVA completes evacuation of the cells, it displays the MAIN MENU.

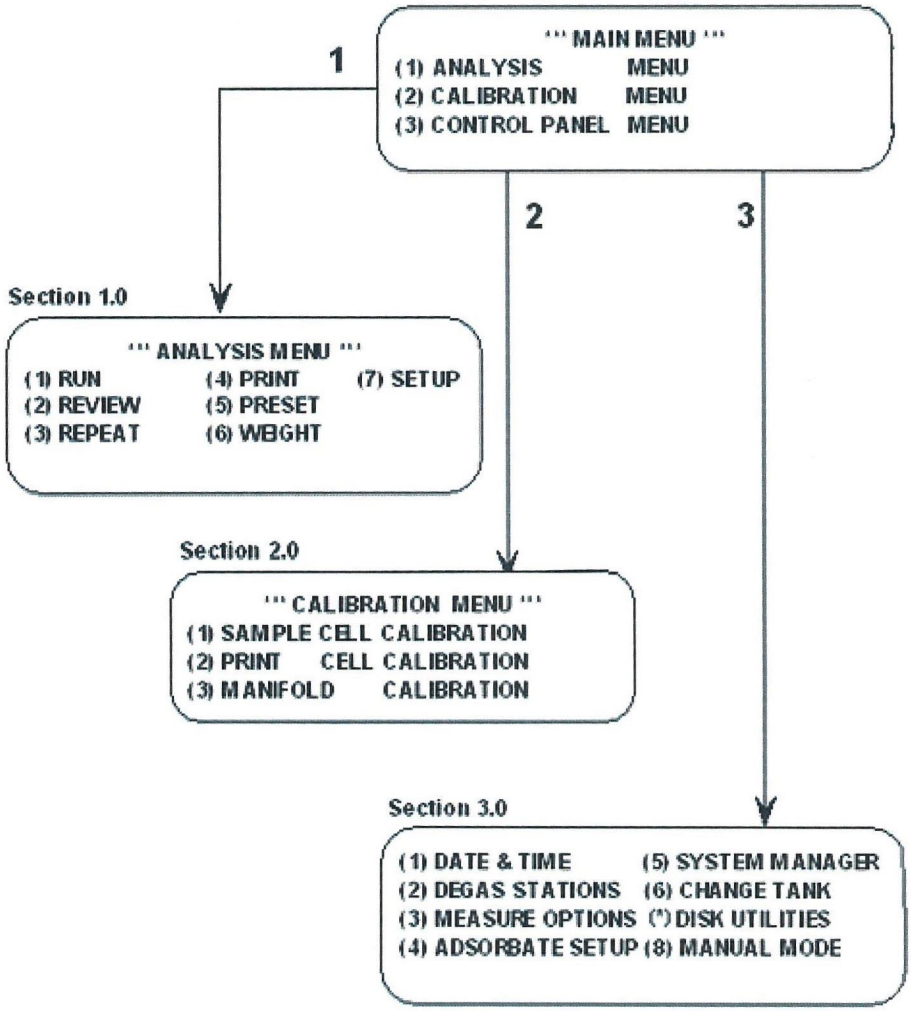
All NOVA operations start with the Main Menu, shown in the flow chart diagram in the following page. Make a selection by pressing the appropriate button on the keypad. The display will present a sub-menu or prompt for input of information as required. Flowcharts have been provided to assist you with proper operation of the instrument. Each flow chart is self-explanatory. Selection of the appropriate number will "branch" you to the desired operation. For example, selecting (1) will display the ANALYSIS sub-menu on the display panel. Detailed descriptions of available choices are given after each flowchart.

Note: Any inaccessible instrument function (according to model number or System Manager lockout) will appear as an asterisk (*) in place of the number shown in the following schematics.

MAIN MENU

The Main Menu has four different sub-menu options:

1. **ANALYSIS**
   For instructions on analyzing samples, see Section 1.
2. **CALIBRATION**
   For instructions on calibrating the sample cells and the manifold, see Section 2.
3. **CONTROL PANEL**
   This sub-menu offers several options and a detailed description is provided in Section 3.



Note: "Upgrade" option is only available if using NovaWin.

Note: Floppy Disk Utility is no longer supported.

1. ANALYSIS MENU

The ANALYSIS MENU contains 7 choices for selection as shown below:

1.1. RUN ANALYSIS

When (1) Run is selected from Analysis Menu, you will be prompted to enter a USER ID. Subsequently, you will need to select the appropriate station(s) for the analysis.

Note: For the 1000 Series, you will not be prompted to specify the station. For the 2000 Series, only STATIONS A & B are available for use, and consequently, the menu displays only the appropriate options. NOVA 3000 Series instruments have STATIONS A, B, and C and NOVA 4000 Series instruments have STATIONS A, B, C, and D available for use.

This manual describes steps involved with the selection of Station A. If you chose more stations, you will need to enter the appropriate information for each station. Upon selecting STATION A, you will be prompted to select an analysis setup by entering the required setup number. Setups can be edited and saved; for detail instructions see section 1.7.

Nova 10.0 Model 25 supports one operation mode: **Nova Mode**. Nova 10.0 Model 26 supports two operation modes: **Nova Mode** and **Helium Mode**. For all the stations used in the run, a common **operation mode** is selected (for detail instructions how to select the mode and/or switch between the Helium and the Nova modes, see Chapter VI - section 3.3. Measure Options).

1. If the instrument is currently in the following operation mode:

(i) "Helium" (model 26 only) – go to step 2. You will NOT be prompted to select the cell number, as in this mode it is not required to use pre-calibrated cells.

(ii) "Nova" - you will need to select a cell for STATION A (and for all additional stations, if applicable). Choose the appropriate cell number (sample cell pre-calibrated using the blank cell calibration) for the **cell/filler rod** combination being used.

2. Subsequently (regardless of the current operation mode) you will be prompted to enter the Sample ID for STATION A (and all additional stations, if applicable) and will be given the option to enter additional comments. The comment is limited to 40 characters in length.

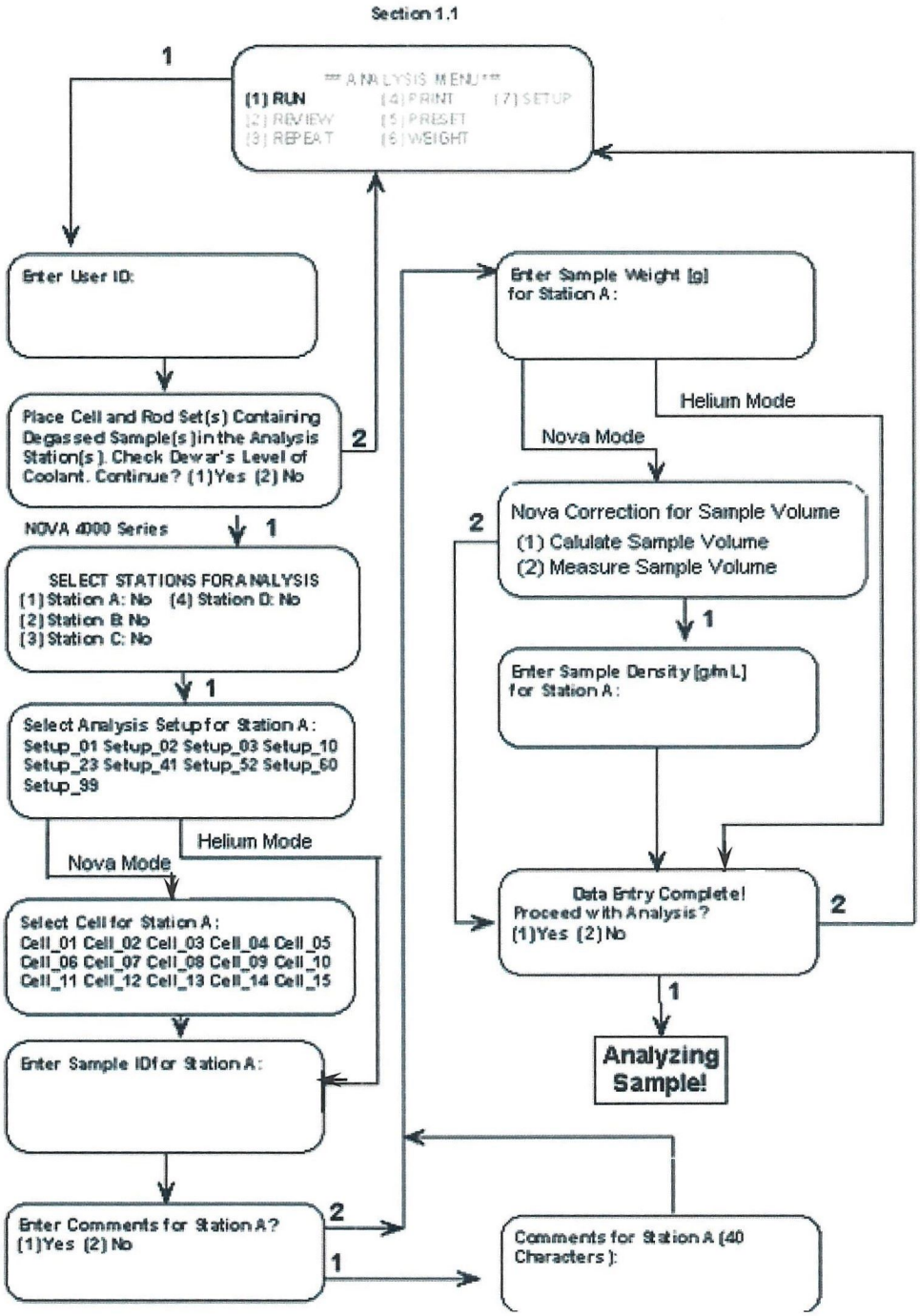
3. Next, you will be prompted to enter the weight of a dry (outgassed) sample (for each active station). If your instrument is in the mode:

(i) "Helium" (model 26 only) - at this point the Data Entry is complete.

(ii) "Nova", you will need to choose to calculate (1) or to measure (2) the sample volume (this selection will apply to all the stations). If you select the first option (1), you are required to enter the sample density (recommended). If the sample density is unknown, select (2) which will allow the instrument to measure the sample volume. After that, Data Entry is complete.

Note: If you are running multiple stations, you may select option (1) only if you know the density for all of the samples in stations used for the run. Otherwise, you must select option (2) - i.e. you can not mix sample volume types.

4. Finally, begin analysis by selecting option (1). If option (2) is selected then all of the entered information will be lost and you will return to the Analysis Menu.


Section 1.1
1
** ANALYSIS MENU **
(1) RUN
(2) REVIEW
(3) REPEAT
(4) PRINT
(5) PRESET
(6) WEIGHT
(7) SETUP
Enter User ID:
Place Cell and Rod Set(s) Containing Degassed Sample(s) in the Analysis Station(s). Check Dewar's Level of Coolant. Continue? (1) Yes (2) No
2
NOVA 4000 Series
1
SELECT STATIONS FOR ANALYSIS
(1) Station A: No (4) Station D: No
(2) Station B: No
(3) Station C: No
1
Select Analysis Setup for Station A:
Setup_01 Setup_02 Setup_03 Setup_10
Setup_23 Setup_41 Setup_52 Setup_60
Setup_99
Helium Mode
Nova Mode
Select Cell for Station A:
Cell_01 Cell_02 Cell_03 Cell_04 Cell_05
Cell_06 Cell_07 Cell_08 Cell_09 Cell_10
Cell_11 Cell_12 Cell_13 Cell_14 Cell_15
Enter Sample ID for Station A:
Enter Comments for Station A?
(1) Yes (2) No
2
1
Comments for Station A (40 Characters):
Enter Sample Weight [g] for Station A:
Helium Mode
Nova Mode
2
Nova Correction for Sample Volume
(1) Calulate Sample Volume
(2) Measure Sample Volume
1
Enter Sample Density [g/mL] for Station A:
Data Entry Complete!
Proceed with Analysis?
(1) Yes (2) No
2
1
Analyzing Sample!

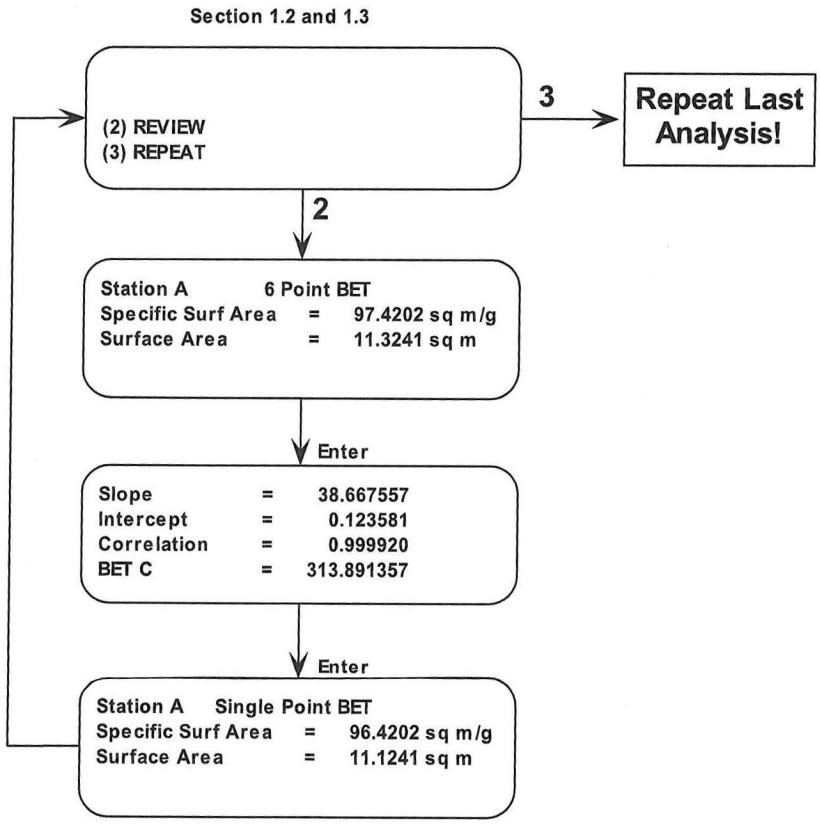
1.2. REVIEW ANALYSIS

Option **(2) Review** (in Analysis Menu) allows you to review all the analysis information from the most recent run. The review shown in the flowchart is for the last run conducted on Station A, and the results for the last run are displayed. Press any key to advance to the next screen. This option remains available even after the power has been cycled OFF/ON.

1.3. REPEAT ANALYSIS

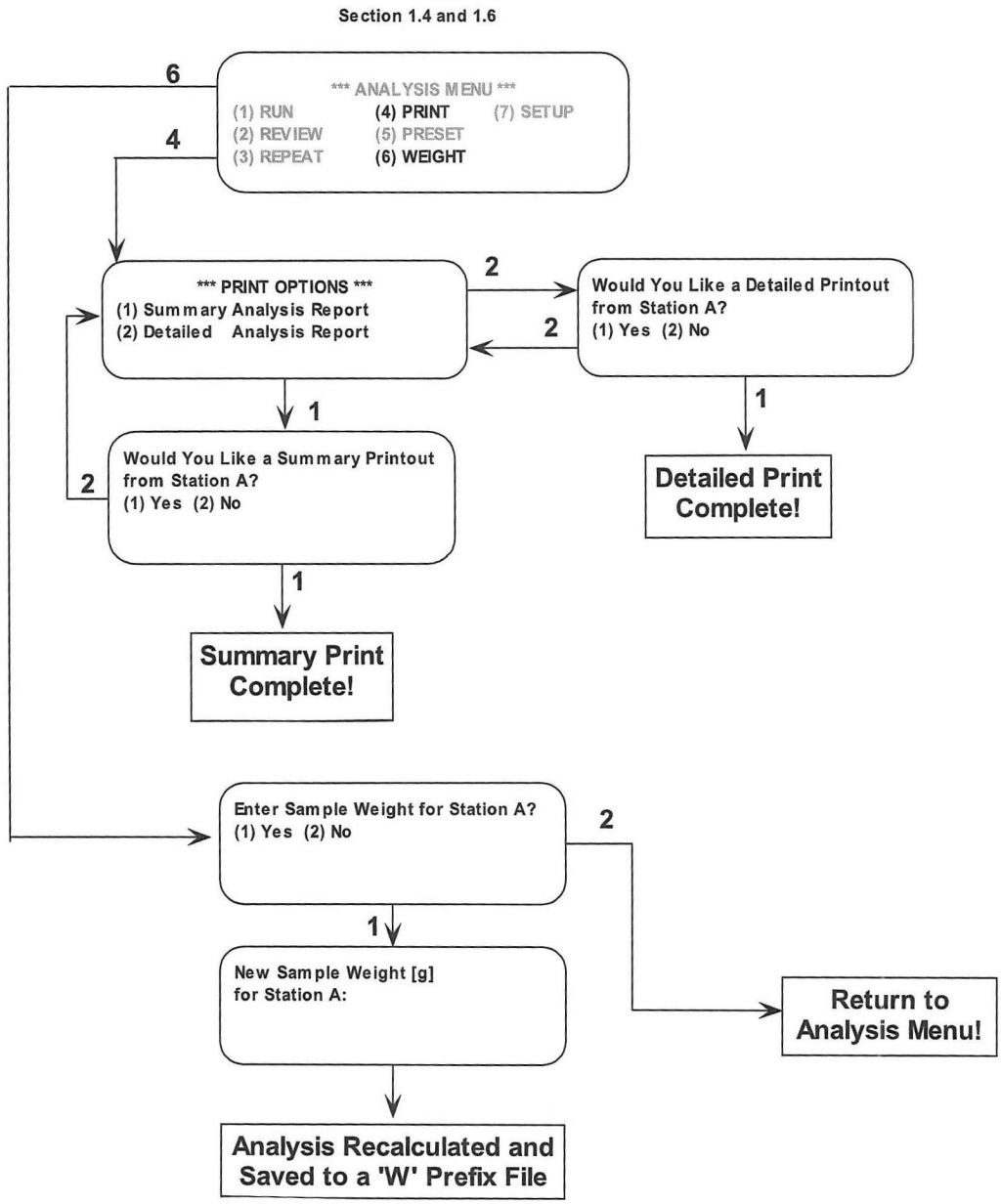
Option **(3) Repeat** (in Analysis Menu) allows you to repeat the last analysis with no additional keypad input. This option is only available immediately after an analysis and will be disabled if the power is cycled OFF/ON or if the analysis was initiated *via* NovaWin. Allow the cell(s) to warm fully to room temperature before repeating analysis. Refill the dewar to the bottom point of the indicator. If there is ice present in the dewar (coolant may be bubbling and may appear cloudy), empty the coolant and replace with fresh coolant. Refill the dewar to the bottom point of the indicator. Remove the indicator after you have set the coolant level.

REVIEW AND REPEAT



1.4. PRINT AND WEIGHT

After selecting option **(4) Print** (from Analysis Menu), the operator will be asked to choose either a Summary Analysis Report or a Detailed Analysis Report. This option remains available even after the power has been cycled OFF/ON. After selecting option **(5) Weight,** for each station, the operator will be asked to enter the sample weight.



1.5 PRESET ANALYSIS

Option (5) **Preset Analysis** helps to streamline analyses when performing similar runs. Selecting Preset Analysis allows you to preset the following features: User ID, stations for analysis, setup files, cell numbers (only when operating in Nova Mode), sample ID numbers, comments, sample weight, and NOVA correction for sample volume (only when operating in Nova Mode). After selecting appropriate information, you can save the preset file and use it for future run. All information that was marked for a prompt will appear when running an analysis. Any messages such as "Fill LN2 in Dewar" or "Place P_0 Cell in Station" will not appear.

1.5.1. Select Preset

The Select Preset option loads a preset run file from the disk into memory. This option minimizes the number of keystrokes required to start an analysis. Enter the name before the ".run" extension. The file name will be displayed next to Run in the Analysis Menu and next to Review and Print in the Preset Run Options Menu.

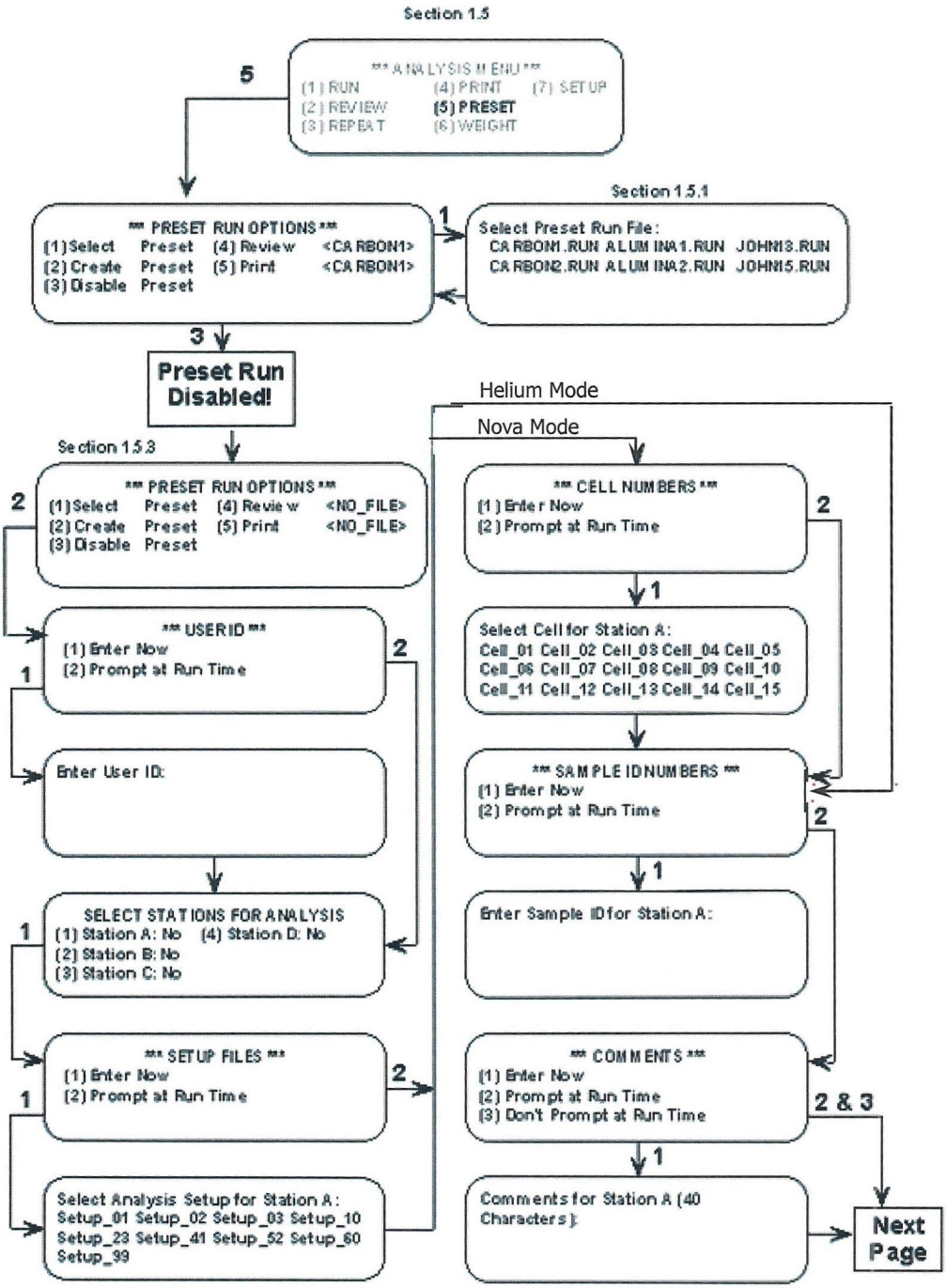
Note: When the NOVA is initialized using NOVAWin, the following will appear automatically next to Run in the Analysis Menu: <NOVAWIN!>.

1.5.2. Create Preset

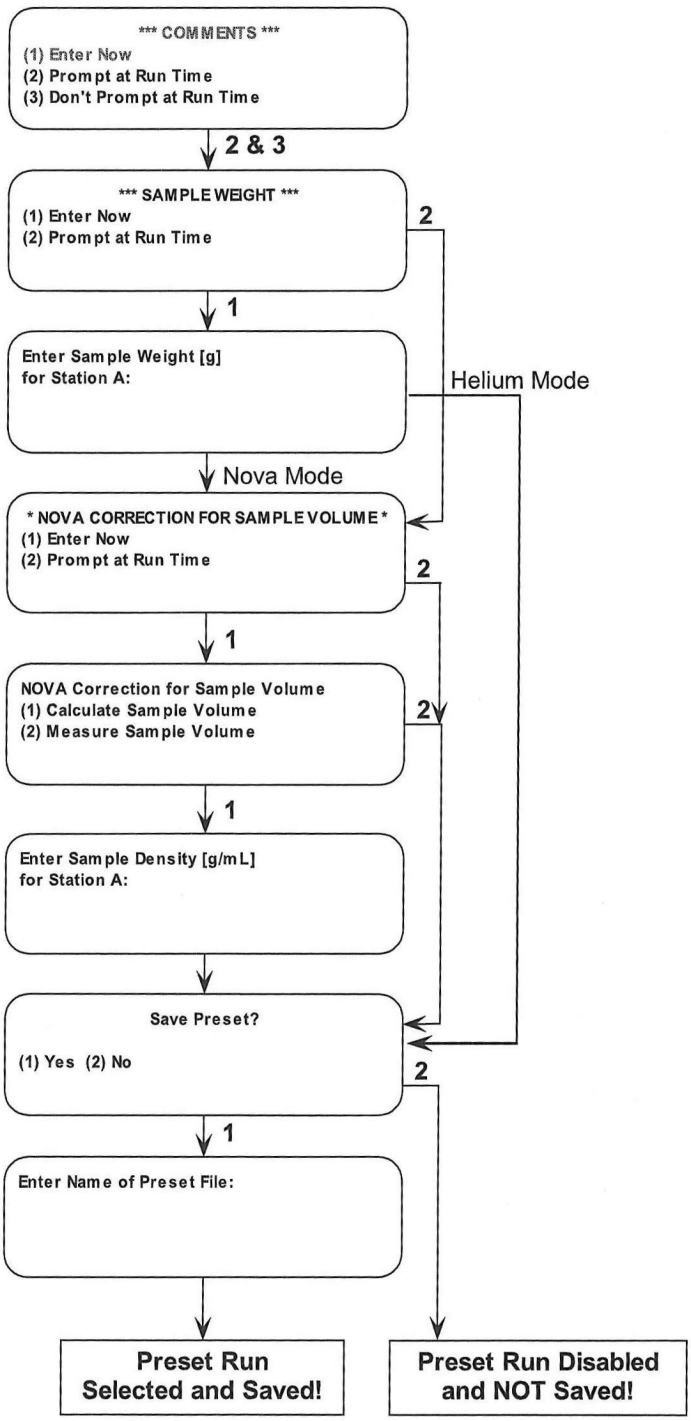
Note: For each sub-level, you will be asked to enter your choice immediately – select option (1) or later - at run time – select option (2).

To create a new Preset Run File, follow the instructions below:

1. First, you will be asked to enter a User ID: (1) now or (2) at run time.
2. Subsequently, you will be prompted to select the station(s) to be used (for NOVA 2000, 3000, and 4000 Series only) for the analysis.
3. Next, select an existing Setup. Choose option (1) only if you have already created a **Setup** for this analysis. Then enter the Setup number for the analysis.
4. Subsequently, if your instrument is in the Helium Mode (model 26 only), you will go directly to step 5 (the selection of cells is unnecessary in this case, and it is not available). However, if your instrument is in the Nova Mode, the following screen will prompt you to enter a cell(s) number(s). If you know the cell/filler rod combination(s) - Cell Number(s) you plan to use for the analysis on each station, select option (1) and enter appropriate Cell Number(s).
5. Next, you will be prompted to enter an ID for the sample, and the following screen will allow you to enter comments immediately or later. If you do not want to put any comments, choose option (3).
6. Subsequently, choose (1) to enter sample weight right away or (2) to do it at run time (recommended). Use the weight of the sample after degassing and enter this value as the sample weight. For best results, weigh the sample immediately after degassing and transfer the cell (with filler rod, if used) directly into the sample station for analysis.


Section 1.5
*** ANALYSIS MENU ***
(1) RUN (4) PRINT (7) SETUP
(2) REVIEW (5) PRESET
(3) REPEAT (6) WEIGHT
5
Section 1.5.1
*** PRESET RUN OPTIONS ***
(1) Select Preset (4) Review <CARBON1>
(2) Create Preset (5) Print <CARBON1>
(3) Disable Preset
1
Select Preset Run File:
CARBON1.RUN ALUMINA1.RUN JOHN13.RUN
CARBON2.RUN ALUMINA2.RUN JOHN15.RUN
3
Preset Run Disabled!
Helium Mode
Nova Mode
Section 1.5.3
*** PRESET RUN OPTIONS ***
(1) Select Preset (4) Review <NO_FILE>
(2) Create Preset (5) Print <NO_FILE>
(3) Disable Preset
2
*** USERID ***
(1) Enter Now
(2) Prompt at Run Time
1
2
Enter User ID:
SELECT STATIONS FOR ANALYSIS
(1) Station A: No (4) Station D: No
(2) Station B: No
(3) Station C: No
1
*** SETUP FILES ***
(1) Enter Now
(2) Prompt at Run Time
1
2
Select Analysis Setup for Station A:
Setup_01 Setup_02 Setup_03 Setup_10
Setup_23 Setup_41 Setup_52 Setup_60
Setup_99
*** CELL NUMBERS ***
(1) Enter Now
(2) Prompt at Run Time
2
1
Select Cell for Station A:
Cell_01 Cell_02 Cell_03 Cell_04 Cell_05
Cell_06 Cell_07 Cell_08 Cell_09 Cell_10
Cell_11 Cell_12 Cell_13 Cell_14 Cell_15
*** SAMPLE ID NUMBERS ***
(1) Enter Now
(2) Prompt at Run Time
2
1
Enter Sample ID for Station A:
*** COMMENTS ***
(1) Enter Now
(2) Prompt at Run Time
(3) Don't Prompt at Run Time
2 & 3
1
Comments for Station A (40 Characters):
Next Page

PRESET ANALYSIS continued from previous page



7. Next, if your NOVA is currently in the Helium Mode (model 26 only), you will go directly to the – final step. However, if your instrument is in the Nova Mode, first you will be asked to choose option for the **NOVA Correction for Sample Volume**. You can: (1) Enter the correction now, or (2) be prompted to do so at Run Time. The correction is based on (1) calculation of a sample volume, in which case you must input sample density (in g/mL) or (2) measurement of a sample volume at Run Time.
8. To finish, choose (1) to save PRESET settings or (2) not to save the changes. During saving, a file name must be entered (up to 8 characters).

1.5.3. Disable Preset

This option will disable using a preset run file and all analysis information will have to be entered at the beginning of a run. The PRESET RUN OPTIONS menu will show <NO_FILE> next to the Review and Print commands. The ANALYSIS MENU will not display a PRESET file name next to the Run command in the ANALYSIS MENU.

1.5.4. Review Preset

This allows you to review all the parameters in the *.run file selected above before conducting an analysis.

1.5.5. Print Preset

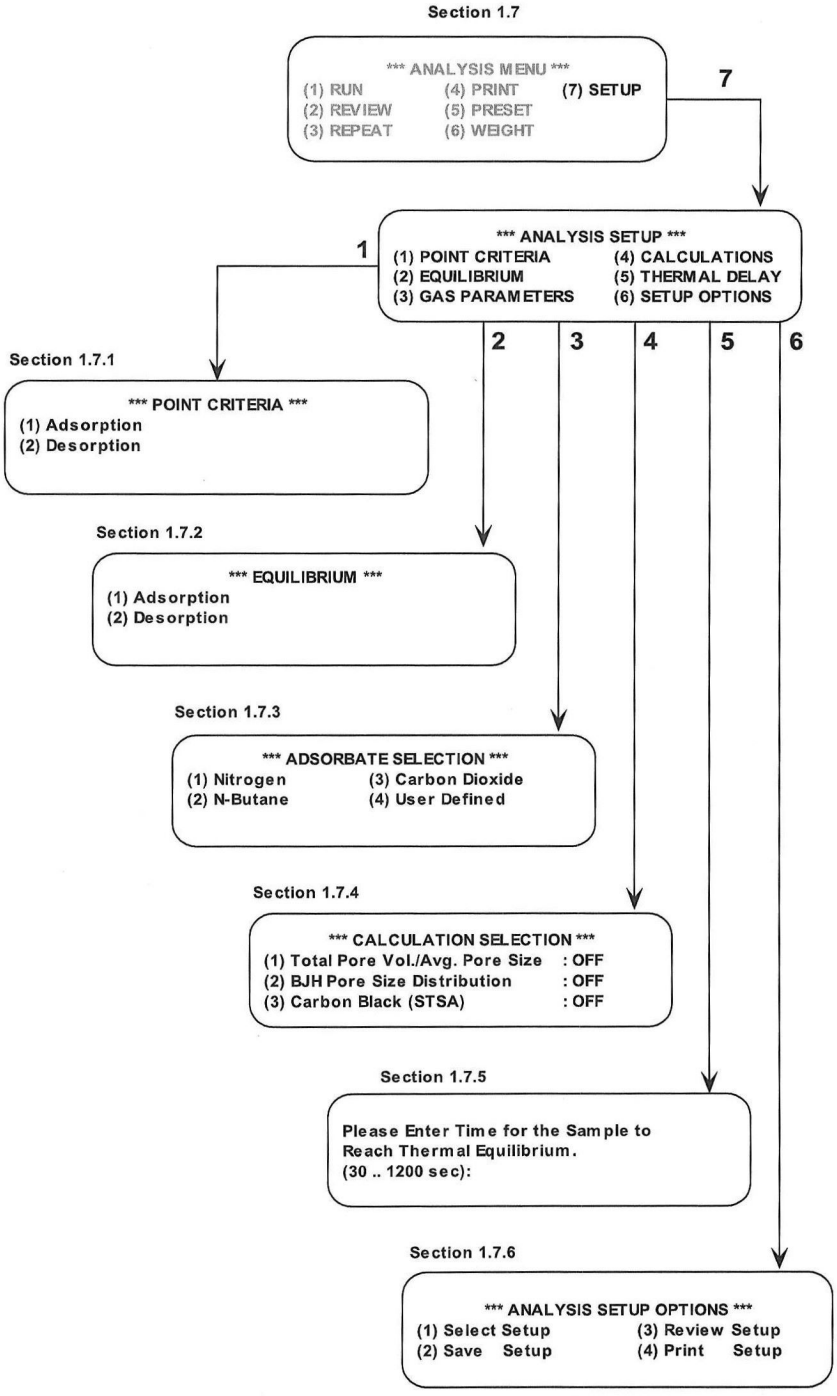
This option allows you to print the *.run file selected above provided a compatible printer is attached to the instrument.

1.6. WEIGHT

After an analysis is complete, you may want to weigh the sample again. If you want to change the weight, select the weight option. The user can change the weight and the NOVA will recalculate the analysis results. This will create a new file that can be printed or uploaded. The new file will have a 'W' prefix instead of the 'N' prefix to identify altered data. This option is only available immediately after an analysis and will be disabled if the power is cycled OFF/ON.

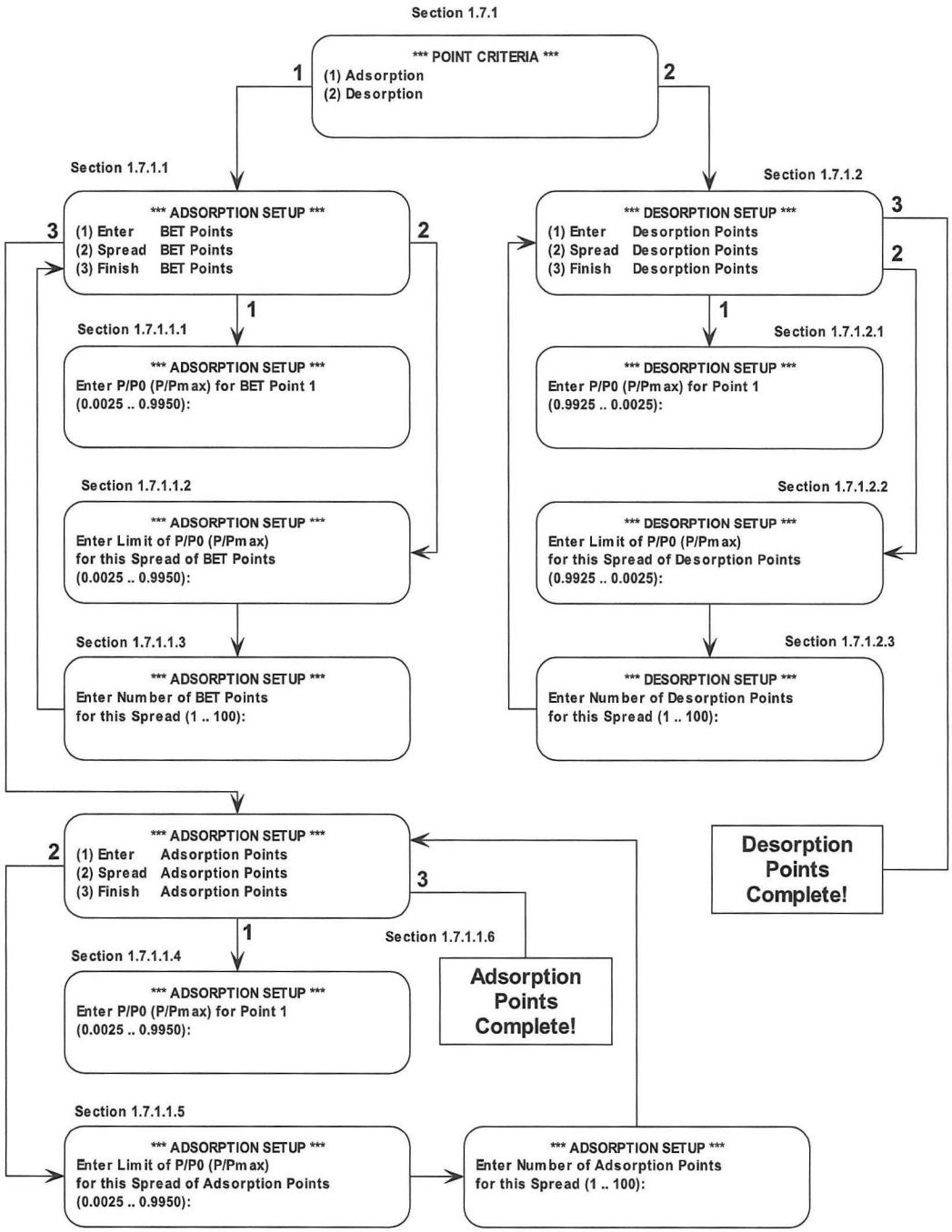
1.7. SETUP ANALYSIS

The SETUP Menu provides 6 additional sub-options and they will be discussed in detail in the following sections.


Section 1.7
*** ANALYSIS MENU ***
(1) RUN
(2) REVIEW
(3) REPEAT
(4) PRINT
(5) PRESET
(6) WEIGHT
(7) SETUP
7
*** ANALYSIS SETUP ***
1
(1) POINT CRITERIA
(2) EQUILIBRIUM
(3) GAS PARAMETERS
(4) CALCULATIONS
(5) THERMAL DELAY
(6) SETUP OPTIONS
2
3
4
5
6
Section 1.7.1
*** POINT CRITERIA ***
(1) Adsorption
(2) Desorption
Section 1.7.2
*** EQUILIBRIUM ***
(1) Adsorption
(2) Desorption
Section 1.7.3
*** ADSORBATE SELECTION ***
(1) Nitrogen
(2) N-Butane
(3) Carbon Dioxide
(4) User Defined
Section 1.7.4
*** CALCULATION SELECTION ***
(1) Total Pore Vol./Avg. Pore Size : OFF
(2) BJH Pore Size Distribution : OFF
(3) Carbon Black (STSA) : OFF
Section 1.7.5
Please Enter Time for the Sample to
Reach Thermal Equilibrium.
(30 .. 1200 sec):
Section 1.7.6
*** ANALYSIS SETUP OPTIONS ***
(1) Select Setup
(2) Save Setup
(3) Review Setup
(4) Print Setup

1.7.1. Point Criteria

This selection allows you to set (i) adsorption points and (ii) desorption points. After entering all adsorption points first, you will enter desorption points.



1.7.1.1. ADSORPTION SETUP

1.7.1.1.1. Enter BET Points

The instrument will display the acceptable range for the points to be entered. The minimum relative pressure difference between points is 0.0025 (separate points are entered for the BET analysis and the other adsorption points). Select the desired relative pressures to be used for the surface area analysis. As each point is entered, the display will update the point number and the acceptable range for the next point. When all of the desired BET points have been entered, enter "0" to exit from entering BET Points.

1.7.1.1.2. Spread BET Points

The Spread BET Points function (recommended) is an efficient alternative to entering the BET points individually in that you can evenly space the desired number of data points over the requested linear range. The BET range extends from P/P_0 0.05 to 0.3. When using the Spread function, manually enter the first BET point (0.05) for the measurement (see Section 1.7.1.1.1 above). Next, choose the Spread function and enter a value for the top limit for the BET spread (0.3). Finally, choose the amount of data points for the spread. For example, for a seven-point BET measurement, enter 6 for the amount of data points in the spread (recall that the first data point, 0.05, was entered manually).

1.7.1.1.3. Finish BET Points

This ends the BET setup and allows you to select points beyond the BET range (i.e. the remainder of the adsorption isotherm), if so desired. Selected BET points will be used for an automated surface area calculation performed by the NOVA according to the BET theory.

1.7.1.1.4. Enter Adsorption Points

The lowest relative pressure that can be entered must be above the highest BET point selected. The display will show the acceptable range. The points may be entered individually and on completion, enter "0" followed by ENTER to return to the ADSORPTION SETUP MENU.

1.7.1.1.5. Spread Adsorption Points

As with the BET menu, you can allow the instrument to select P/P_0 points by itself (only above the BET range). It is recommended to go directly into this menu choice after BET point selection is completed (see section 1.7.1.1.3). Select the desired maximum P/P_0 adsorption point (the instrument will provide the lowest point possible beyond the earlier selected BET range). This should be followed by entering the required number of points required (the combined total for adsorption plus BET points selected earlier is 100).

1.7.1.1.6. Finish Adsorption Points

Selecting this option will return you to the POINT CRITERIA Menu (1.7.1). Press ESC to return to the ANALYSIS SETUP MENU (1.7) if no desorption points are required.

1.7.1.2. DESORPTION SETUP

Select (2) from the POINT CRITERIA Menu (1.7.1) to choose DESORPTION SETUP. As with adsorption, you will have to select the desorption points required.

1.7.1.2.1. Enter Desorption Points

Selecting (1) will allow you to enter the desired relative pressures to be used for the desorption analysis. The minimum relative pressure between points must be 0.0025. As each point is entered, the display will update the point number and the acceptable range for the next point. When all of the desired points have been entered, enter "0" to return to the Desorption Setup Menu.

1.7.1.2.2. Spread Desorption Points

As with the Adsorption Menu, you can allow the instrument to select evenly spaced P/P_0 points in a requested linear range. Select the desired minimum P/P_0 desorption point (the instrument will provide the lowest point possible beyond the earlier selected range). This should be followed by entering the required number of points for the linear spread (individually entered desorption points and spread desorption points cannot exceed 100).

1.7.1.2.3. Finish Desorption Points

This selection will bring you back to the POINT CRITERIA menu (1.6.1). Press ESC or "0" to return to the Analysis Setup Menu.

1.7.2. EQUILIBRIUM

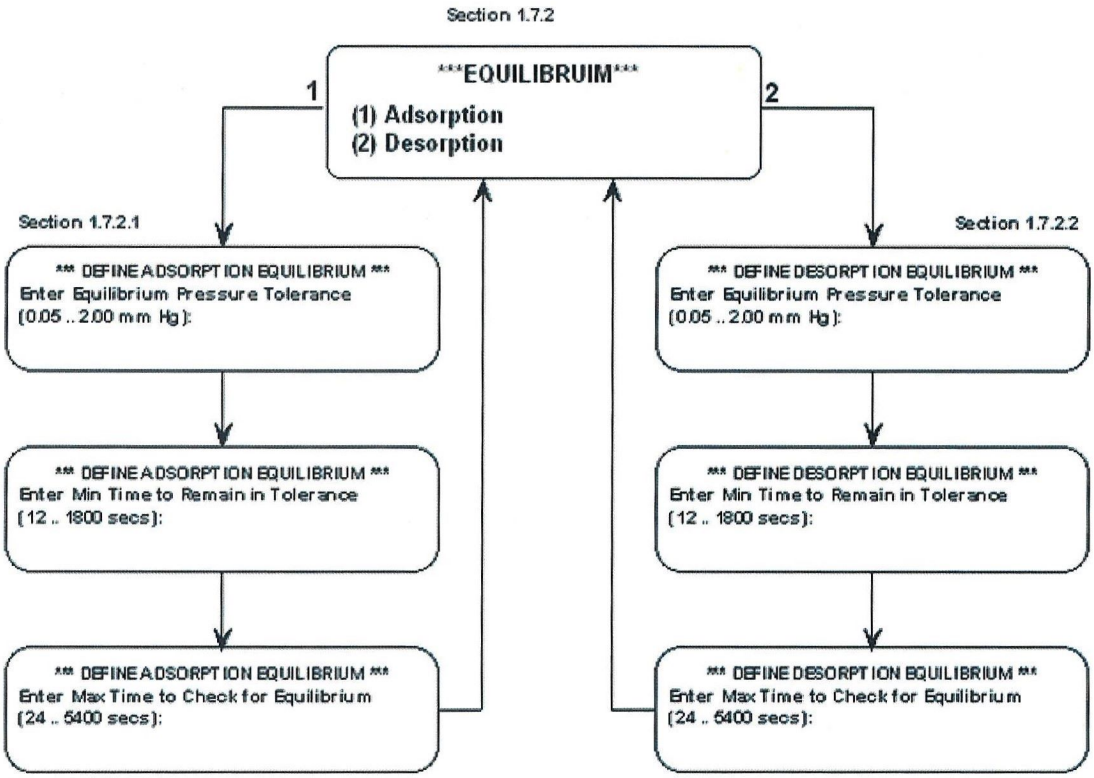
1.7.2.1. DEFINE ADSORPTION EQUILIBRIUM

Selecting (1) prompts you to enter the equilibrium pressure tolerance for adsorption. Enter the maximum acceptable change in pressure (mm Hg) over the specified equilibrium time tolerance.

Note: A value of 0.1 mm Hg will be sufficient for most samples.

When the equilibrium pressure tolerance has been entered, the display will prompt you to enter the time to remain in equilibrium. Enter the time the pressure must remain in equilibrium before a data point is accepted.

Note: A value of 60 seconds has been found to be sufficient for most samples. Longer times may be necessary for relatively large sample weights (> 1g) and for samples which have low thermal conductivity.



When the time tolerance has been entered, the display will prompt you to enter a maximum time to check for equilibrium. This dwell time is a "time out" value which determines when a data point will be accepted if the pressure does not reach equilibrium and does not fall out of the pressure limit range. The acceptable time range is from twice the equilibration time to 5400 seconds.

Note: The recommended value is three times the equilibrium time. Longer times may be necessary for relatively large sample weights (> 1g) and for samples which have low thermal conductivity, high surface area and/or large pore volume.

1.7.2.2. DEFINE DESORPTION EQUILIBRIUM

Selecting (2) will present similar choices as with the Adsorption Menu. Enter the maximum acceptable change in pressure (mm Hg) over the specified equilibrium time tolerance. For most samples, a value of 0.1 mm Hg will be sufficient.

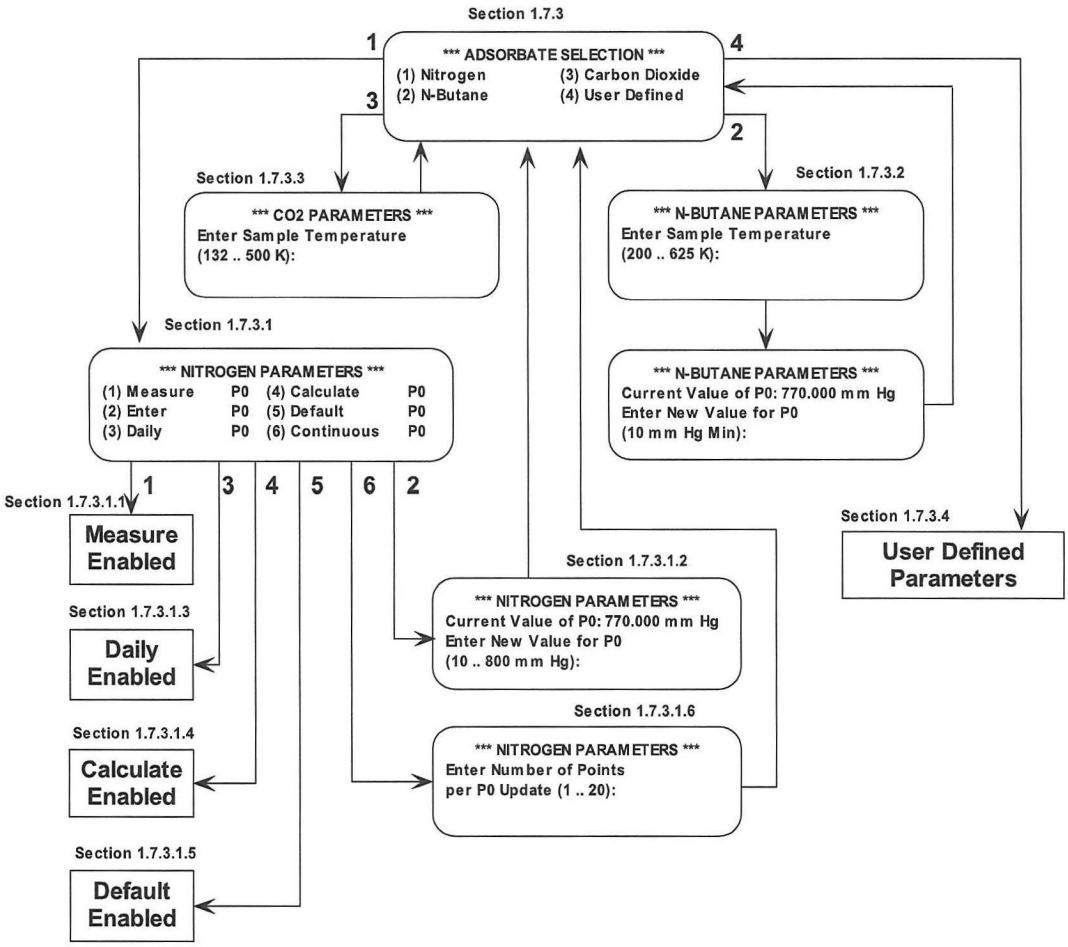
When the equilibrium pressure tolerance has been entered, the display will prompt the user to enter the time to remain in equilibrium. Enter the time the pressure must remain in equilibrium before a data point is accepted. A value of 60 seconds has been found to be sufficient for most samples. Longer times may be necessary for relatively large sample weights (> 1g) and for samples which have low thermal conductivity.

When the time tolerance has been entered, the display will prompt you to enter a maximum time to check for equilibrium. This dwell time is a "time out" value which determines when a data point will be accepted if (i) the pressure does not reach equilibrium and (ii) does not fall out of the pressure limit range. The acceptable time range is from a minimum of twice the equilibration time to a maximum of 5400 seconds.

Note: The recommended value is three times the equilibrium time. Longer times may be necessary for relatively large sample weights (> 1g) and for samples which have low thermal conductivity, high surface area and/or large pore volume.

1.7.3. ADSORBATE SELECTION

Select the appropriate gas that will be used as the adsorbate.



1.7.3.1. NITROGEN PARAMETERS

Selection (1) will allow you to enter the appropriate parameters for nitrogen.

Note: For the 1000, 2000, 3000, and 4000, the software will not present any gas options other than nitrogen. Only for the 1200, 2200, 3200, and the 4200 will the software provide adsorbate options besides nitrogen.

AVAILABLE P_0 OPTIONS

Nova instruments allow various methods for the evaluation of the saturation pressure (P_0).

Note: **Calculate P_0** option is recommended for routine sample measurements using adsorbate at its boiling point temperature, such as N_2 at 77 K or Ar at 87 K (for details see 1.7.3.1.4.). For the most accurate pore size analysis results, it is recommended to use **Continuous P_0** option (option is unavailable in the case of NOVA 1000 series; for details see 1.7.3.1.6.).

1.7.3.1.1. Measure P_0

This selection instructs the instrument to measure P_0 during analysis by condensing nitrogen in the sample cell on the sample prior to adsorption run. This method is lastly recommended and is to be used for certain analyses only. The default station for **P_0 Measurement (prior to analysis)** is station A.

(i) If the cell in the station A is a sample cell (contains the sample to be analyzed), then after the P_0 measurement, the adsorbate will be evacuated (dewar will jolt) prior to resuming the analysis.

(ii) If the cell in the station A is not a sample cell (empty cell designated for the P_0 measurement), the dewar will remain up and this cell will be cleaned of adsorbate at the end of analysis.

1.7.3.1.2. Enter P_0

This will display the current value of P_0 and will you to enter a new value for P_0. Ambient pressure in mm Hg plus 10 should be entered. As an example, if the ambient pressure is 757 mm Hg, then the user should enter a value of 767 mm Hg.

1.7.3.1.3. Daily P_0

Under Measure Options in the control panel, select Daily P_0. Insert a 9-mm bulbless cell (do not include filler rod) in Station A. Upon starting the measurement, the cell will be evacuated, the dewar will rise, and the cell will be filled with LN2. It will proceed to measure the daily P_0. Once this is done a P_0 value resides in memory and is used each time Daily P_0 is selected for an analysis. That value is stored in the instruments memory and will not change until a new Daily P_0 is measured. Daily P_0 can only be measured *via* the Nova instrument keypad, however this measured value can be accessed and used in analysis initiated *via* both Nova and Nova Win.

1.7.3.1.4. Calculate P_0

If Calculate P_0 is selected, at the beginning of the analysis, the manifold is pressurized and atmosphere is measured in the manifold. The value measured is ambient pressure and an offset 10 mm Hg is added to that value. **Calculate P_0** option is recommended for routine sample measurements (using adsorbate at its boiling point temperature, such as N_2 at 77 K or Ar at 87 K).

1.7.3.1.5. Default P_0

Default P_0 uses the P_0 value measured or entered from the cell calibration being used.

1.7.3.1.6. Continuous P_0

Continuous P_0 measures P0 the same way as Daily P_0. However, this measurement is done during the run and requires the use of one station during analysis. The NOVA 1000 series does not have this option. The NOVA 2000 series can only run one sample station if Continuous P_0 is selected. The NOVA 3000 series can only run two sample stations if it is selected. **Continuous P_0** provides the analysis with a continual update of P_0 throughout the entire run. The value of P_0 can be updated for every 1 - 20 points. However, for the most accurate results, we recommend to update P_0 for every 1-3 data point(s).

Users of NOVA 2000, **3000 and 4000 Series** may only choose **Station A** to conduct the P_0 measurement during the run (even if the cell in this station contains the sample). In the case of **NOVA 3000 and 4000 Series the highest remaining station letter available will be the dedicated station for the Continuous P_0 measurements.** As an example, if you are using a NOVA 4000 and you wish to use Stations B and D as the analysis stations, Station C will be the station for the Continuous P_0 measurements. For NOVA 3000 Series users, Station C is the default station for the P_0 measurements using Continuous P_0 while for NOVA 4000 Series users, Station D is the default station for the Continuous P_0 measurements. Continuous P_0 measurements should be made using an empty 9-mm bulbless cell without filler rod.

1.7.3.2. N-BUTANE PARAMETERS

This selection will prompt you to enter the sample analysis temperature. This will be followed by a window that displays the current value of P_0 with the option to enter a new value of P_0.

1.7.3.3. CARBON DIOXIDE PARAMETERS

This selection prompts you to enter the bath temperature. After entering the bath temperature, will be returned to the ADSORBATE SELECTION menu. The effecive P_0 is calculated by the NOVA. Data points are selected based on P_{MAX} rather than P_0.

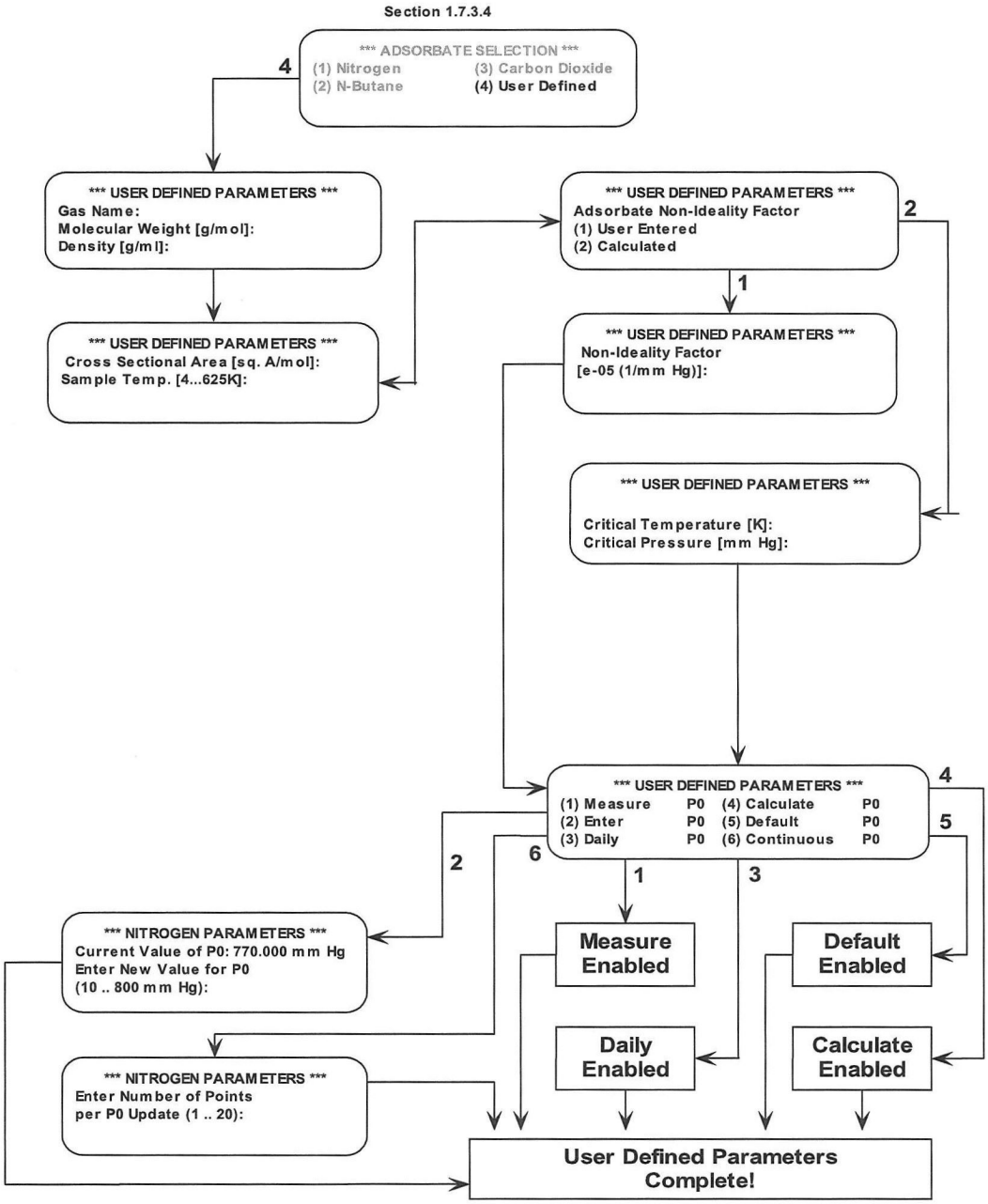
1.7.3.4. USER DEFINED ADSORBATE PARAMETERS

Enter the gas name, its molecular weight, density, cross-sectional area, and sample temperature. Next, enter the adsorbate non-ideality factor. There are two options:

1. Upon selection of (1), you are prompted to enter the value followed by prompts to select P_0 options identical to Sections 1.7.3.1 - 1.7.3.6.
2. Calculated factor which will prompt you to enter the value of the critical temperature of the adsorbate in Kelvin followed by the value of the critical pressure (mm Hg). You will then proceed to the section where it is required to select P_0 options identical to Sections 1.7.3.1 - 1.7.3.6.

Caution: When using a gas other than N_2 which is not at its boiling point, do not use the Calculate P_0 option while calibrating the empty cell or during analysis. As an example, when using Ar at 77 K (which is the boiling point of nitrogen; the boiling point of Ar being 87 K), use the Enter P_0 option during the analysis setup and use a value of 205 mm of Hg for Ar. In this case, the saturation pressure of argon corresponds to a solid phase and amounts to 205 Torr for an ambient pressure of 760 Torr.

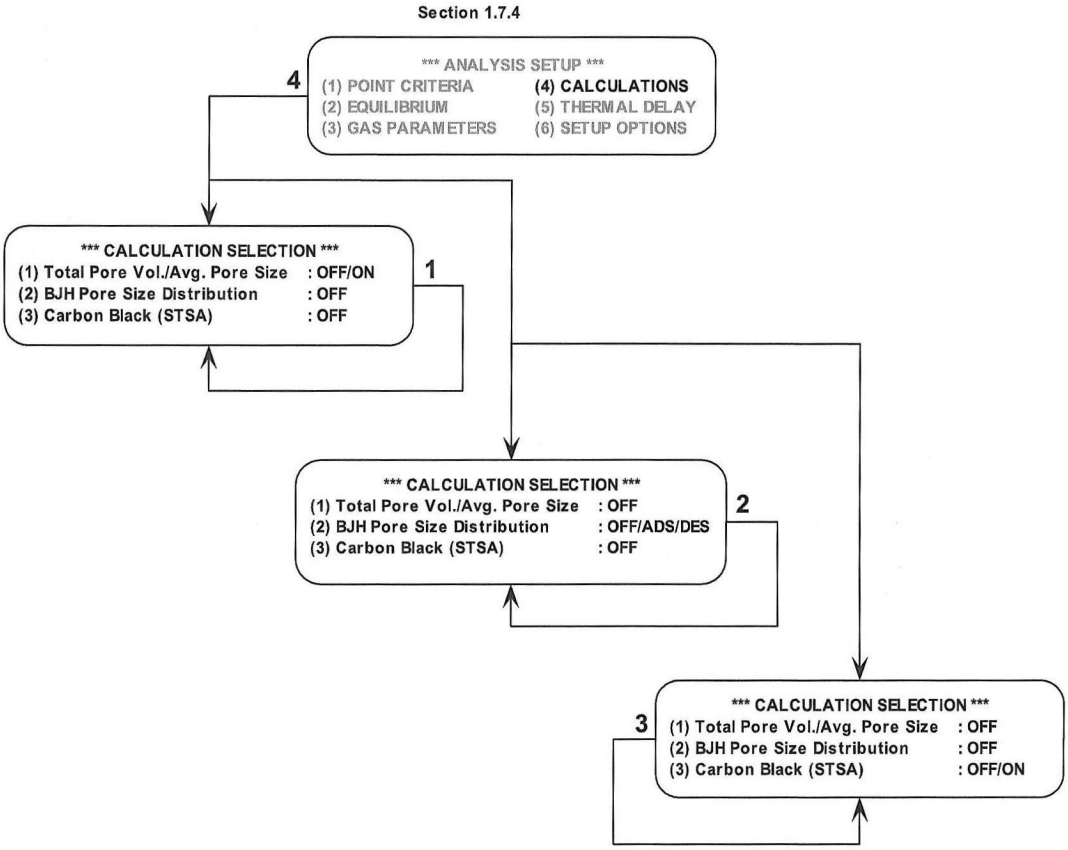
ADSORBATE SELECTION
User Defined



1.7.4. CALCULATIONS

For each selection, a toggle "switch" provides for the appropriate selection.

Note: You may bypass this section if you are using the NOVAWin2 software for data reporting and printing.



1.7.4.1. Total Pore Vol. / Ave. Pore Size

Press (1) to toggle between ON and OFF. If you would like for the NOVA to calculate these values (i.e. appear on the printout) then the toggle must be set to ON.

1.7.4.2. BJH Pore Size Distribution

Press (2) to toggle between OFF, ADSORPTION, or DESORPTION. The selection of ADSORPTION will provide for pore size distribution using the adsorption isotherm data. Selecting DESORPTION provides pore size distribution from the desorption isotherm data.

1.7.4.3. Carbon Black (STSA)

Press (3) to toggle between OFF and ON. Use this to determine external surface area (STSA) by ASTM D5785. You must select BET points in an appropriate range (typically P/P_0 of 0.05 - 0.25) and adsorption data points up to 0.5 P/P_0.

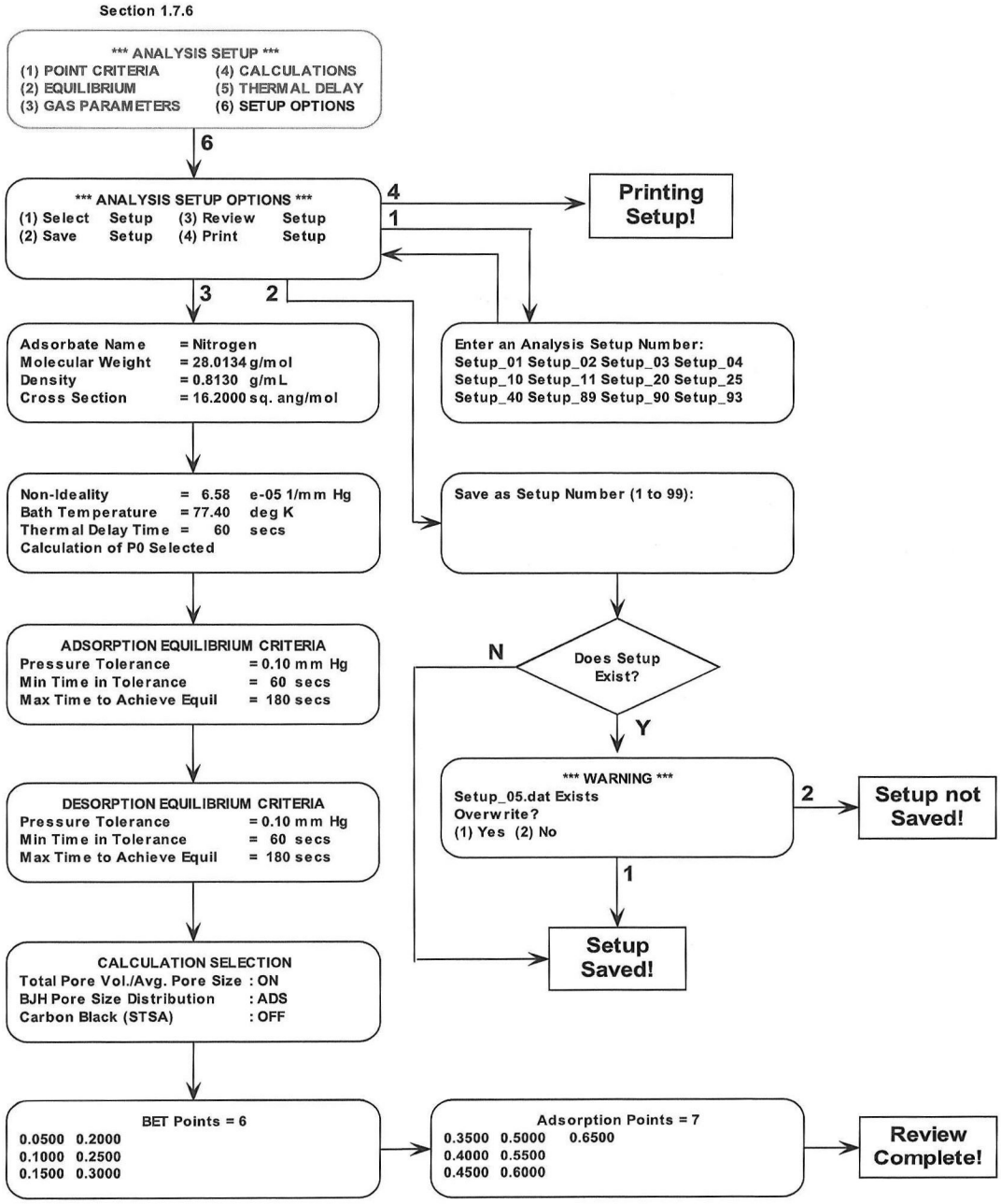
1.7.5. THERMAL DELAY

You can select any time interval (in the range 180 – 1200 seconds) for the sample to reach thermal equilibrium (i.e. the minimum time after the dewar is raised before the first data point is measured).

Note: It is recommended to use a shorter Thermal Delay time for standard (bulbless) cells, and longer for large bulb cells. Additional time may be necessary for large masses of low surface area material.

Note: Various time (thermal delay before the measurement) might be required to uniformly cool-down samples with different thermal conductivities, as materials with low thermal conductivity will require more time to achieve a stable, uniform temperature.

1.7.6. SETUP OPTIONS



1.7.6.1. Select Setup

Select Setup prompts you to enter an analysis setup number. Enter one of the numbers show below on the display ranging from 1 to 99. The selected setup (i.e. Setup_01) is loaded into memory for viewing or printing it. If you want to change that setup, then press "0" or ESC to change parameters in the Analysis Setup Menu. The altered setup can be saved as another setup if so desired.

1.7.6.2. Save Setup

Save Setup prompts the user to save a setup with a number from 1 to 99. If the number already exists from a previous saved setup, the instruments will ask to overwrite (1) Yes (2) No. If you chose not to save and you want to exit the Analysis Setup Menu, the instrument will inform you that the setup is not saved. If you chose not to save and leave the Analysis Setup Menu, then all residing information associated with the setup will be erased.

1.7.6.3. Review Setup

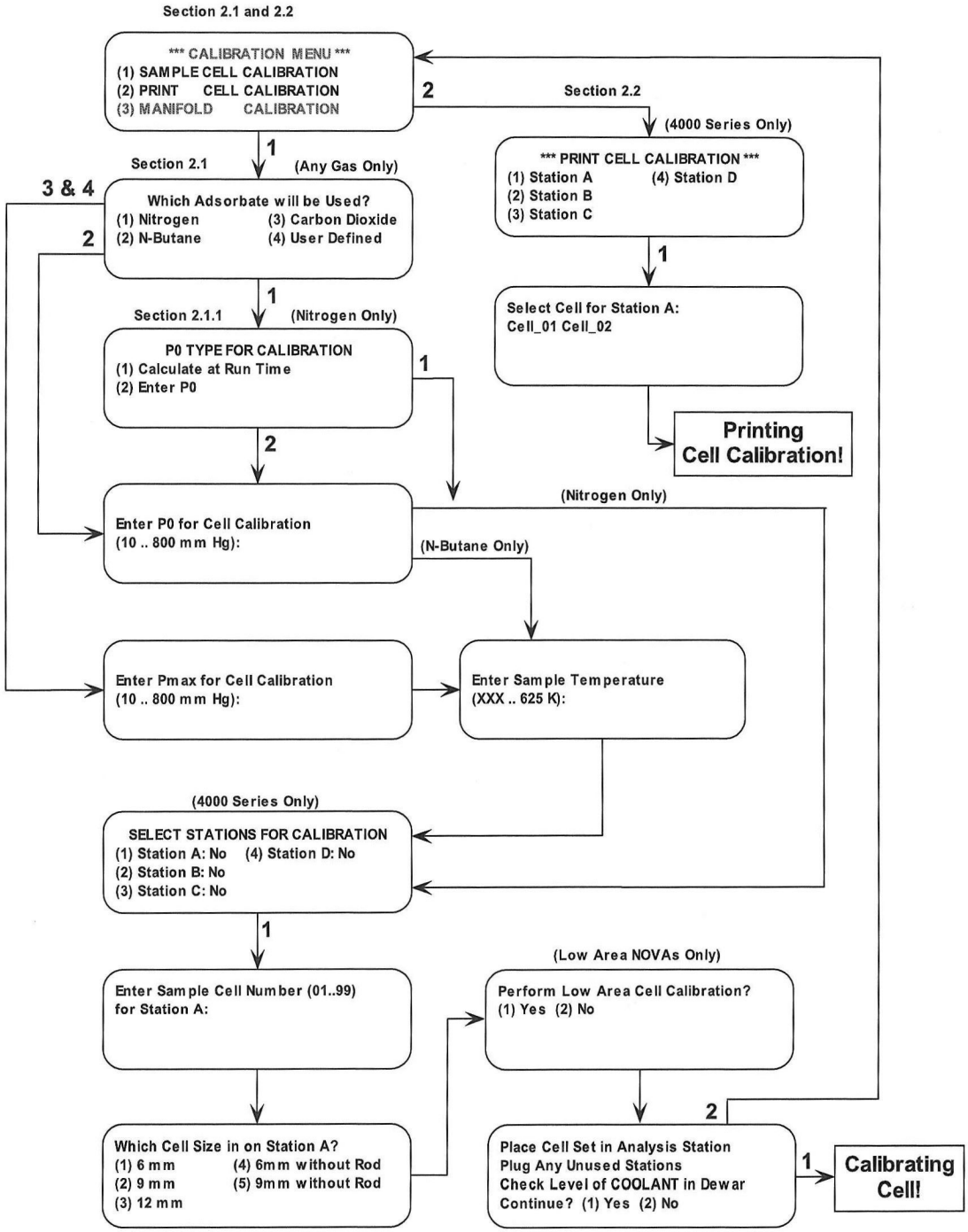
This displays a sequence of windows that allows you to review setup options such as adsorbate parameters, adsorption/desorption criteria, and calculation options selected. Press any key to display each window. This is provided to assist in determining if the appropriate selections/options have been selected before analysis is performed.

1.7.6.4. Print Setup

This option prints the setup options selected provided a compatible printer is attached to the instrument.

2. CALIBRATION MENU

Depending on the current operation mode: Helium Mode (model 26 only) or Nova Mode, the options: (1) Sample Cell Calibration and (2) Print Cell Calibration are not or are available, respectively.



2.1. SAMPLE CELL CALIBRATION

This procedure is not necessary when instrument operates in the Helium Mode (model 26 only); therefore it is only available during operation in the Nova Mode. If the NOVA model is a 1200, 2200, 3200, or 4200, there are 4 selections for setting up adsorbates.

2.1.1. Nitrogen

Pressing (1) selects nitrogen as the adsorbate. Then user must choose between calculating P_0 during Run Time or entering a P_0 value. Choosing the former will prompt to select the Station for calibration. The illustration is shown for selecting Station A.

Choosing the "Enter P_0" option will require to enter a value of P_0 (in mm of Hg), which is then followed by the prompt to enter to select a station which will be used for calibration.

Then, user is prompted to enter a unique cell number to identify that particular sample cell. This will be the number used in analysis conducted later using that particular sample cell. The instrument requires a one-time calibration of a particular sample cell. Once the calibration is performed on a cell, you must invoke the cell number at analysis time.

Next, user must enter the size of the sample cell used. Enter the number that corresponds to the sample cell to be calibrated. Then user will be required to place the sample cell in the Station (here we have selected Station A for illustration purposes).

Subsequently, the instrument requires confirmation that user checked the level of coolant in the dewar. Slide the Coolant Level Indicator (P/N 04000-7400) onto the lip of the dewar. The indicator hangs on the top lip of the dewar with the arrow-shaped point inside the dewar. Ensure the dewar flask is filled to the bottom point of the indicator. Remove the indicator after establishing the proper coolant level. Selecting (1) will calibrate the cell while choosing (2) will return the operator to the CALIBRATION MENU.

When calibrating sample cells, cell numbers may be selected only once (they are not specifically assigned to the station in which they were calibrated), set to a specific cell type (6 mm, 9 mm, or 12 mm) and whether it was calibrated with or without a filler rod. Calibrated sample cells can be used in any of the stations for sample analysis.

2.1.2. N-Butane

The sequence of steps when selecting n-butane as an adsorbent is similar to that for selecting Nitrogen as explained above. The only additional requirement is that you must enter P_{MAX} for cell calibration (as opposed to P_0 for Nitrogen) and the temperature (in K). Be sure to enter a value of P_{MAX} which will not cause butane to be condensed in the cell during calibration. P_{MAX} is simply the maximum pressure (in mm of Hg) up to which the NOVA will calibrate the cell.

2.1.3. Carbon Dioxide

The steps are identical to the ones discussed above for N-Butane.

Note: For most applications, it is recommended to enter P_{MAX} as 800.

2.1.4. User Defined

The steps are identical to the ones discussed for N-Butane and Carbon Dioxide above.

Caution: When using a gas other than N_2 which is not at its boiling point, do not use the Calculate P_0 option while calibrating the empty cell or during analysis. As an example, when using Ar at 77 K (which is the boiling point of nitrogen; the boiling point of Ar being 87 K), use the Enter P_0 option during the analysis setup and use a value of 205 mm of Hg for Ar. In this case, the saturation pressure of argon corresponds to a solid phase and amounts to 205 Torr for an ambient pressure of 760 Torr.

2.2. PRINT CELL CALIBRATION

This provides the option of printing cell calibration information. Choose the station and cell number for which the calibration information is required.

Note: Make sure that a printer is connected to the NOVA and switched ON before requesting this print function.

Note: The NOVA 1000 does not prompt for Station information since only 1 station is available on the 1000 Series.

Note: Cell calibrations can only be printed using this function. It is not available in NOVAWin2.

2.3. MANIFOLD CALIBRATION

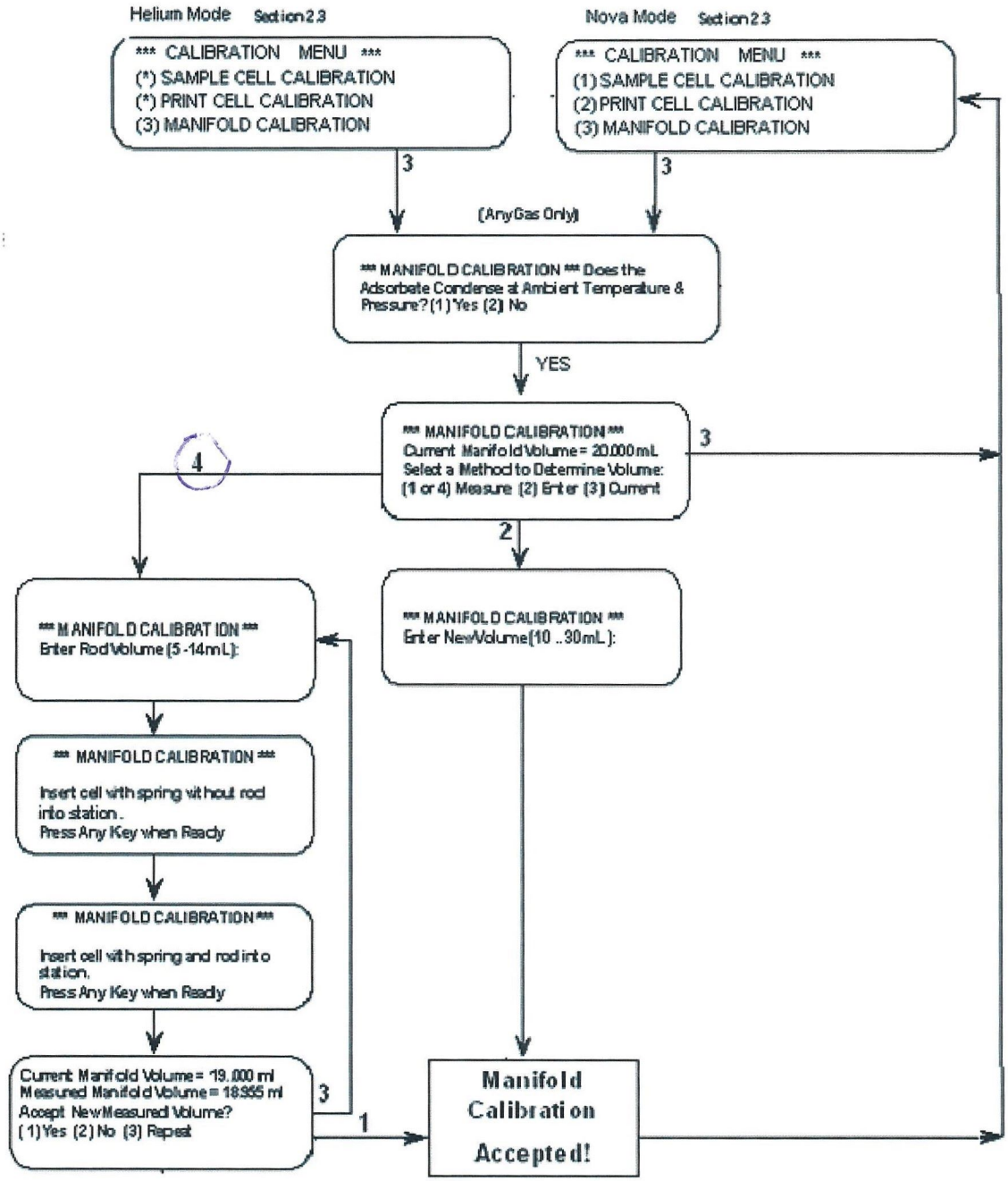
Flow chart for manifold calibration is shown on the next page. For manifold calibration select option (3) on the CALIBRATION MENU. First, you will be prompted to indicate whether the adsorbate condenses at a room temperature.

(i) Press (3) to use a value currently in memory from an earlier calibration. Display returns to the CALIBRATION MENU.

(ii) Press (2) to enter a value for the manifold volume (in mL). Display returns to the CALIBRATION MENU.

You will be prompted to:

- Enter the rod volume – enter the value provided by the company certificate.
- Insert cell with spring without rod into station – attach cell provided with the calibration kit to station 1.
- Insert cell with spring and rod into station – use the quartz rod provided with the calibration kit and attach cell with rod to station 1.



The LCD panel then displays the values of the current manifold volume and the new measured volume. Select either one as required or press (3) to measure the volume again.

Note: It is recommended to repeat the Manifold Calibration procedure at least five times in succession. Eliminate the lowest and highest values and take the average of the remaining values.

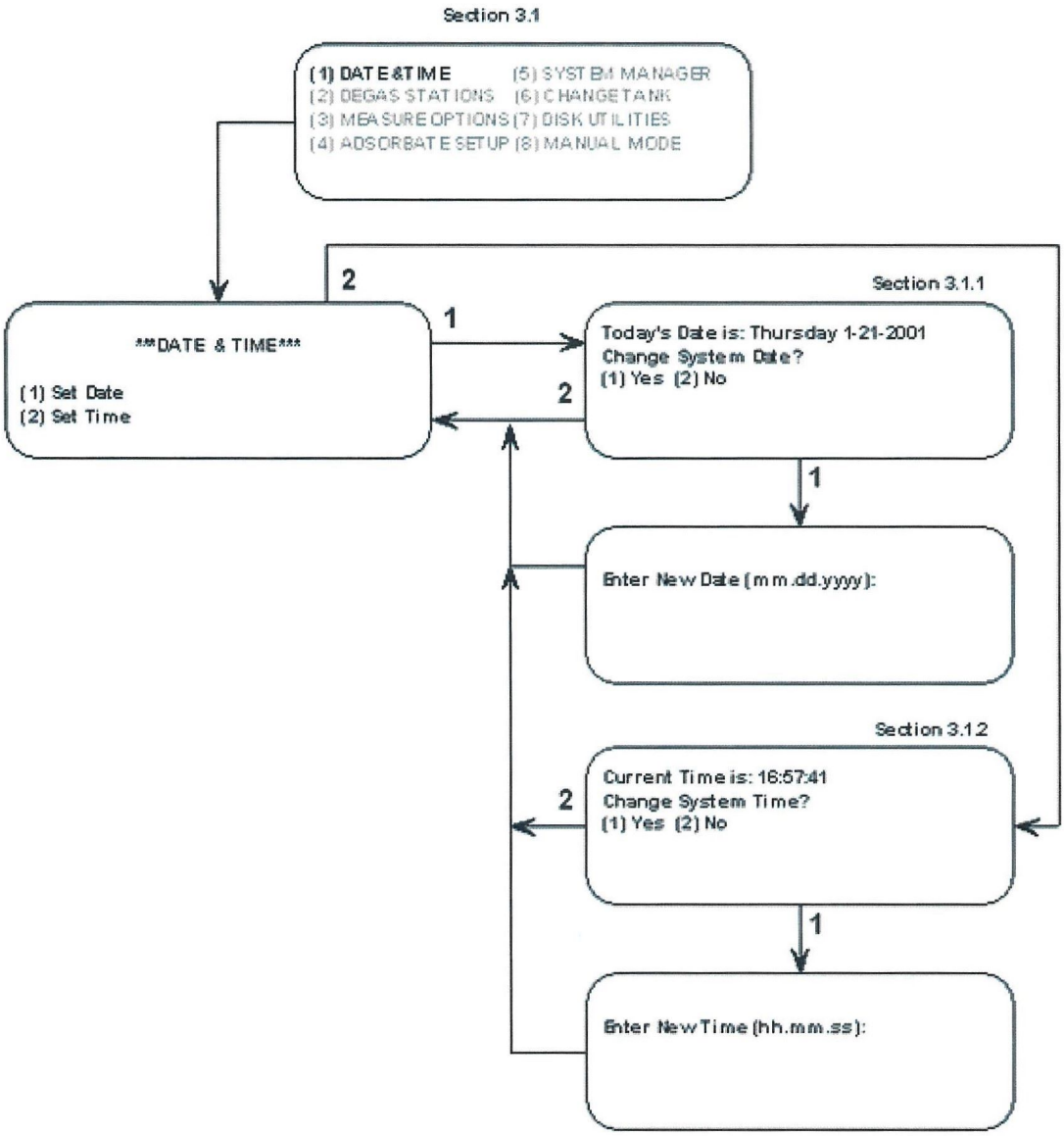
The actual total manifold volume should be within 17-22 ml (value varies between different models). Unless service/maintenance work has been performed on the manifold, the manifold calibration should yield a value of within 0.1 mL of one previously measured. If not, the manifold may have been or is now contaminated, the vacuum level may be insufficient (check fittings and pump oil, for example), or pressure transducer needs to be re-aligned.

Note: It is recommended to repeat the Manifold Calibration procedure at least five times in succession. Eliminate the lowest and highest values and take the average of the remaining values.

3. CONTROL PANEL MENU

This Menu provides several Sub-menus that are listed in order below:

3.1. DATE AND TIME



3.1.1. Set Date

Press (1) to change the date. The required format is (m)m.(d)d.yyyy where (m)m represents the month (eg. 5 for May and so on), (d)d is for the day (eg. 25 and so on) and yyyy represents the year in that specific format (i.e. 4 digits, eg.1999 has to be entered as 1999 and not as 99).The decimal point is used as the delimitator between the month, day and year. Do not add leading zeros for values below 10. Press (2) to accept the date.

Note: All three time units must be entered before the new values are accepted.

3.1.2. Set Time

Press (2) to change the time using a 24 hour, (h)h.(m)m.ss format. The decimal point is used as the delimitator between the hour, minute and seconds. Do not add leading zeros for values below 10. Press (2) to accept the current time.

Note: All three time units must be entered before the new values are accepted.

3.2. DEGAS STATIONS

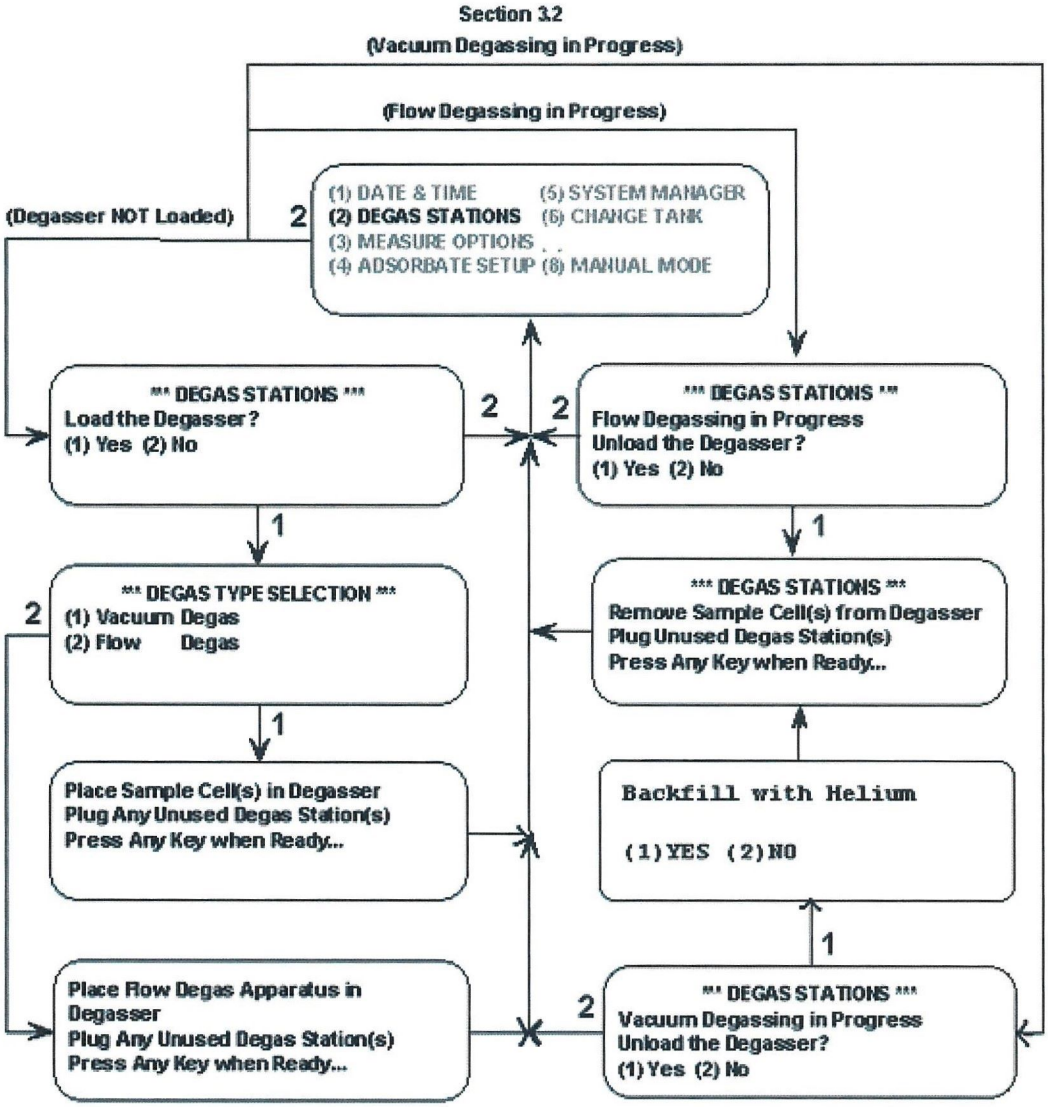
This option must be used to load/unload the degasser station before adding/removing sample cells to/from the degas fittings.

If the instrument is not degassing when this selection was made: press (1) for Vacuum Degas or (2) for Flow Degas. Place the sample cell in the degas station(s) (with any unused stations being plugged). Refer to Section V "INSTRUCTIONS FOR USE – OVERVIEW in this manual for proper use of either the vacuum degas or the flow degas option. Press any key to start degassing.

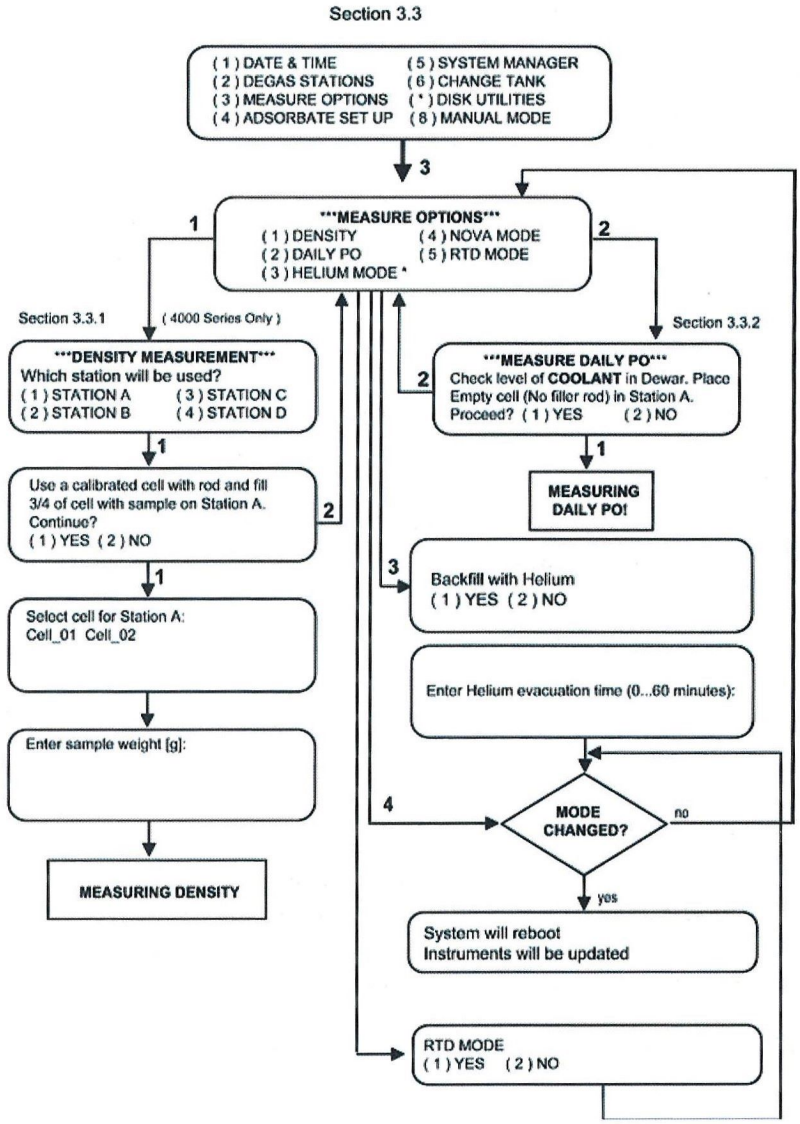
If this selection was made during degassing: the NOVA will prompt user to either (1) Unload or (2) No action. After selecting **(1) Unload, user** must choose to backfill cell with Helium – option **(1) YES**; or with the adsorbate – option **(2) NO;** and subsequently, to remove any sample cells from the degas stations (also plugging any unused stations). Press any key to start the degas process. This option is provided for both Vacuum Degas and/or Flow Degas, in progress.

Note: Heating mantles are controlled *via* the temperature set and heater on/off switches. Keyboard entries do not affect heating mantle operation.

DEGAS MENU can be accessed during an analysis by pressing the 'Backspace' or the "P" key.


Section 3.2
(Vacuum Degassing in Progress)
(Flow Degassing in Progress)
(Degasser NOT Loaded)
2
(1) DATE & TIME
(2) DEGAS STATIONS
(3) MEASURE OPTIONS
(4) ADSORBATE SETUP
(5) SYSTEM MANAGER
(6) CHANGE TANK
(8) MANUAL MODE
*** DEGAS STATIONS ***
Load the Degasser?
(1) Yes (2) No
2
2
*** DEGAS STATIONS ***
Flow Degassing in Progress
Unload the Degasser?
(1) Yes (2) No
1
1
2
*** DEGAS TYPE SELECTION ***
(1) Vacuum Degas
(2) Flow Degas
*** DEGAS STATIONS ***
Remove Sample Cell(s) from Degasser
Plug Unused Degas Station(s)
Press Any Key when Ready...
1
Place Sample Cell(s) in Degasser
Plug Any Unused Degas Station(s)
Press Any Key when Ready...
Backfill with Helium
(1)YES (2)NO
1
Place Flow Degas Apparatus in
Degasser
Plug Any Unused Degas Station(s)
Press Any Key when Ready...
2
*** DEGAS STATIONS ***
Vacuum Degassing in Progress
Unload the Degasser?
(1) Yes (2) No

3.3. MEASURE OPTIONS



If there is an asterisk next to the RTD mode option, the CLC mode is enabled. Analysis will proceed as long as coolant is detected (RTD probe is required).

If the asterisk is not displayed, it means that the RTD mode is disabled. In such case the analysis will proceed but the user must first remove the RTD probe. During the analysis, the dewar will be raised to the top-most position and TempComp will be used.

3.3.1. Density

The NOVA will recall the void volume of a calibrated cell. When a sample is placed into the cell, void volume is reduced by the amount of that sample's volume. The NOVA can either measure the volume or calculate it. If the NOVA is instructed to calculate the void volume, it needs to have the sample weight and sample density. If sample density is unknown, then user can perform the density measurement using the NOVA. A calibrated large bulb cell set will be required to make the density measurement. For best results, a cooled degassed sample should fill ¾ of a large bulb cell. Increasing the amount of sample will increase your accuracy. Since the density value is solely used for recalculating the void volume, it does not need to measure densities as accurately as a gas pycnometer. The design of the NOVA was never intended for this purpose.

3.3.2. Daily P_0

The measure daily P_0 option allows the user to establish a new P_0 value in memory as infrequently as once a day. The user can select daily P_0 from the "analysis setup-P_0 selection menu" during analysis setup (see 1.7.3.1.3). The same daily P_0 value will be used in all analyses that have this option selected. When this option is selected, the user will be prompted to place an empty sample cell in the sample station and fill the dewar with liquid nitrogen. (Only station A can be used to acquire a new *Daily* P/P_0). The operator should use a 9 mm O.D. bulbless cell with no filler rod for this measurement. When this has been done, press (1) and the NOVA will setup and measure the new *Daily* P_0.

If you do not update the P_0 value each day, then the last value measured will be used for P_0.

3.3.3. RTD Mode

The Firmware version 10.03 allows the user to run analysis with RTD coolant level control (CLC) turned on or off. To enable or disable RTD, go to the entry screen of the instrument and:

- press <esc>
- In "Main Menu" press 3 for "control panel menu"
- Then press 3 for "measure option"
- In "measure option" menu, there will be either:
  - "(5) RTD mode*" if CLC is enabled or,
  - "(5) RTD mode" if CLC is disabled.

To enable or disable CLC enter 5 and then choose 1 to enable (RTD mode) or 2 to disable CLC (no RTD mode). If the mode changes, the instrument will reboot.

To fully disable the CLC, the RTD mode must be off and the RTD probe must be removed from the instrument.

If there is an asterisk next to the RTD mode option, the CLC mode is enabled. Analysis will proceed as long as coolant is detected (RTD probe is required). If the asterisk is not displayed, it means that the RTD mode is disabled. In such case the analysis will proceed but the user must first remove the RTD probe. During the analysis, the dewar will be raised to the top-most position and tempcomp will be used.

3.4. ADSORBATE SETUP

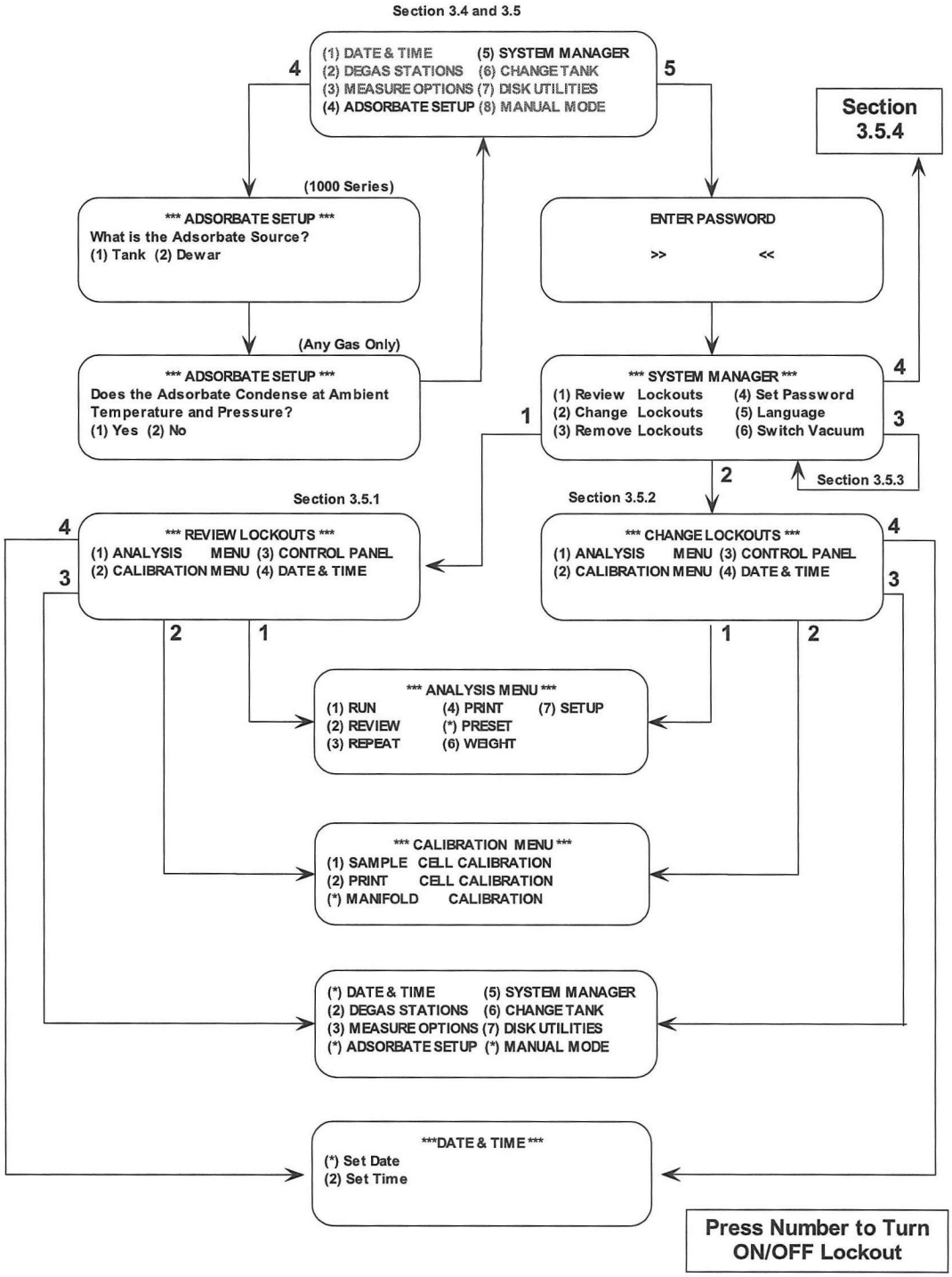
All NOVA models have this option except for the NOVA 2000, 3000 and 4000. The Adsorbate Setup Option accomplishes two purposes. If a NOVA 1000 Series instrument is being used, then it will ask to use the gas tank or the dewar as the gas input line. If dewar is selected, then the gas is taken right from the LN2 in the dewar through a pick-up tube. If tank is selected, then the gas input is taken right from the tank. All other models do not have the dewar option. The Any Gas models such as the NOVA 1200, NOVA 2200, NOVA 3200, and NOVA 4200 use the Adsorbate Setup Option for another reason. It will ask if the adsorbate condenses at ambient temperature and pressure. If it does, then your not using nitrogen but some other gas such as n-butane that requires some changes in the instrument's mode of operation. Nitrogen users should say "No" to this question (the default setting is no). Only the NOVA 1200 will ask both questions mentioned above.

3.5. SYSTEM MANAGER

System Manager allows a laboratory manager to lockout various menus from unauthorized users and to change the language of the display and printouts. The manager may want to customize what a user can or cannot access. For example, there could be a Preset or Setup that the manager wants the operator to always use. If a password has not been entered, the manager can do so after entering System Manager. Once it is set, System Manger can only be accessed by use of the password. This gives the laboratory manager control over the instruments operations.

Note: NOVAWin2-P users must use the System Manager lockouts for the software to be 21 CFR Part 11 compliant.

ADSORBATE SETUP AND SYSTEM MANAGER



3.5.1. Review Lockouts

This allows the manager to review the lockout options for the following menus: (1) Analysis Menu, (2) Calibration Menu, (3) Control Panel, and (4) Comm/Date/Time. Any item that has a lockout is identified by an asterisk (*), those with a number are available to the user.

3.5.2. Change Lockouts

This option allows a manager to change the items that are locked out. To lockout an item, press the appropriate number. That number will be replaced by an asterisk (*) and lockout that item. To unlock an item, press the number the asterisk would be and that number will reappear.

3.5.3. Remove Lockouts

Selecting this option removes lockouts for all items.

3.5.4. Set Password

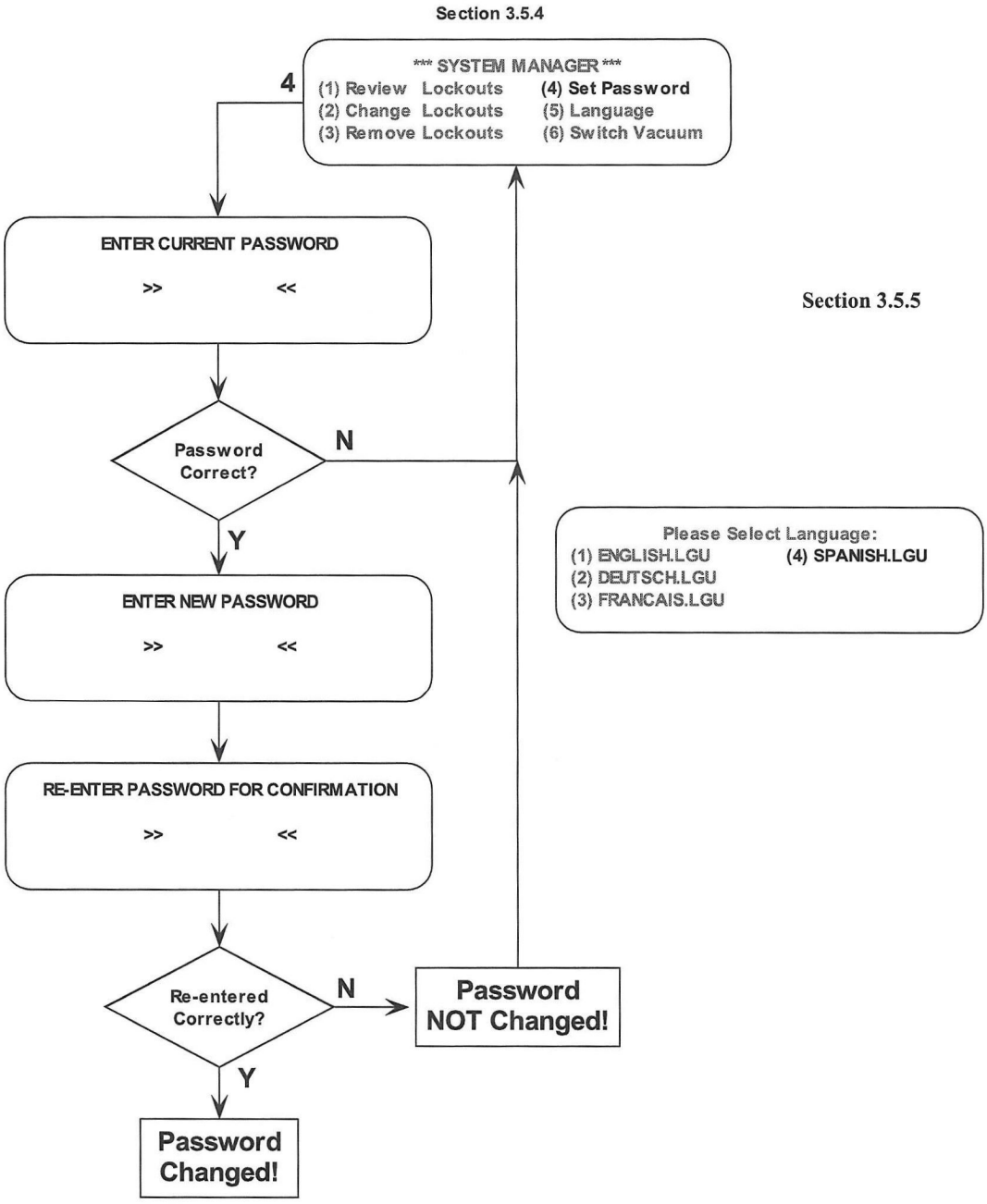
Selecting this option prompts you to enter the current password. Only the correct password will allow you to select a new password. You will be required to re-confirm the new password before it is accepted. If any change to the lockouts has occurred, then the manager will be prompted to save upon exiting System Manager.

3.5.5. Language

Select the Language option if you wish to change the language of the NOVA display. English, German, French and Spanish are supported by the NOVA. The NOVA LCD and printouts (from a printer connected to the NOVA) will reflect this language change.

3.5.6. Switch Vacuum

This option allows you to enter the value of pressure at which you wish to change evacuation rate from fine to coarse.

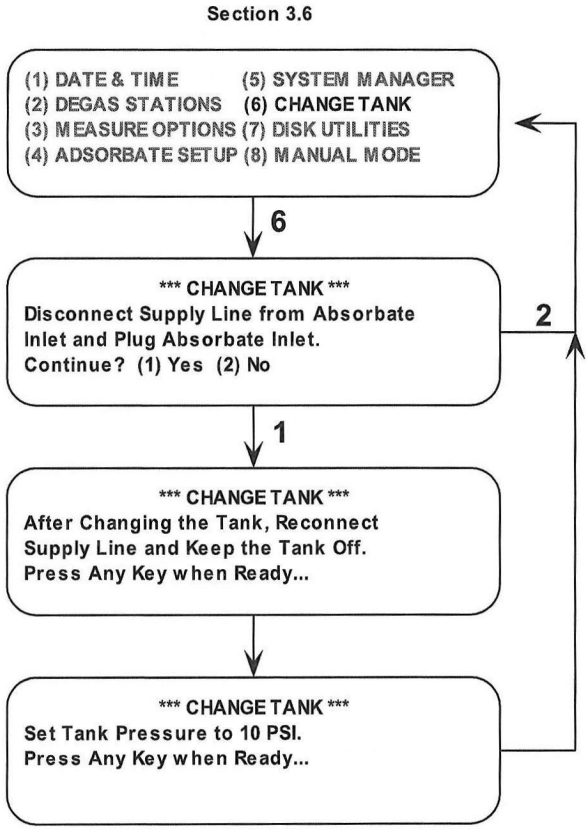

Section 3.5.4
*** SYSTEM MANAGER ***
(1) Review Lockouts
(2) Change Lockouts
(3) Remove Lockouts
(4) Set Password
(5) Language
(6) Switch Vacuum
4
ENTER CURRENT PASSWORD
>> <<
Password Correct?
N
Y
ENTER NEW PASSWORD
>> <<
RE-ENTER PASSWORD FOR CONFIRMATION
>> <<
Re-entered Correctly?
N
Y
Password NOT Changed!
Password Changed!
Section 3.5.5
Please Select Language:
(1) ENGLISH.LGU
(2) DEUTSCH.LGU
(3) FRANCAIS.LGU
(4) SPANISH.LGU

3.6. CHANGE TANK

This option should be used when changing the adsorbate gas tank. Selecting this option prompts you to disconnect the supply line and plug the adsorbate inlet. After this, select

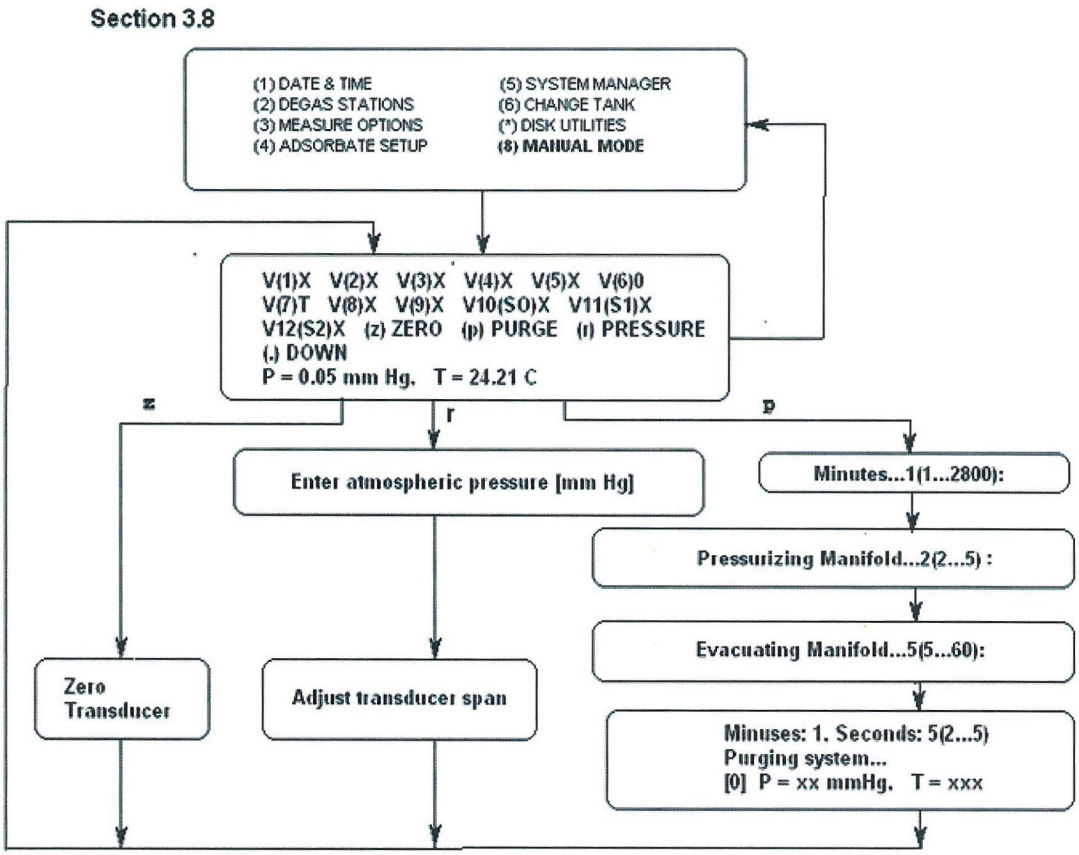
1. Continue the process of changing the gas tank.
2. Return to Section 3.6.

Upon selecting (1), you are prompted to change the tank and reconnect the supply line with the gas flow valve (on the regulator) turned OFF. Press any key after this is complete. Next, set the gas pressure to 10 PSIG (70 kPa) followed by opening the gas flow valve and pressing any key when this is complete.



3.7. MANUAL MODE

The Manual mode allows the operator to operate the valves and the dewar drive directly using the keypad/keyboard. It is intended for troubleshooting and system verification only.



Note: Only experienced NOVA users should access this option. Access to MANUAL MODE can be locked out using the SYSTEM MANAGER lockouts.

Note: Low area option is no longer supported.

Note for Model 26 only: Valve 11 controls the helium supply to the manifold. When the instrument is set for helium mode, Valve 11 is shown in the manual mode menu.

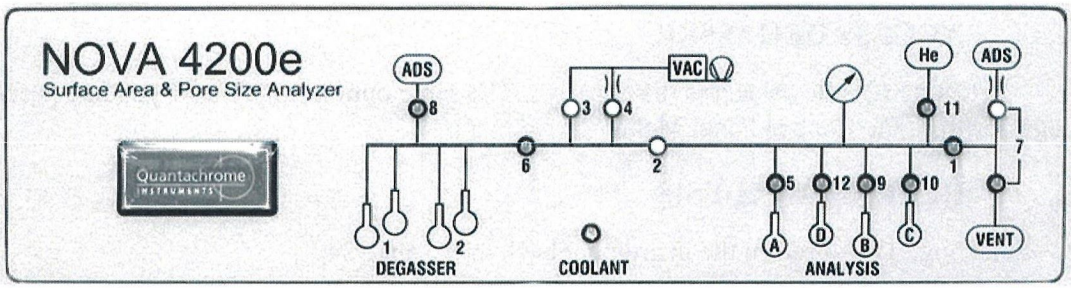
The display panel shows the status of the valves, the dewar, and the adsorbate/vent line. Each of the options TOGGLES between OPEN and CLOSED for the valves. 'X' indicates a closed valve whereas 'O' indicates an open valve. In the illustration V(1)X indicates a CLOSED Valve 1. Choosing (1) would open Valve 1.

Selecting (.) will bring the Dewar either UP or DOWN. Selecting (7) will choose between the adsorbate line or vent. Valves 10, 11 and 12 can be opened by pressing Shift 0, Shift 1 and Shift 2 on the keypad/keyboard., respectively.

Selecting (z) will zero the pressure transducers, selecting (r) will adjust the pressure transducer's span.

Pressing (p) will purge the system. System purge consists of sequences of pressurization period followed by evacuation period. Prior to starting purge, set all valves except for 1, 2, 3, 4 and 11 to purge selected area of the system. Then, specify purge duration from 1 to 2800 minutes, pressurization period 2 to 5 seconds and evacuation period 5 to 60 seconds.

Note: If a sample cell contains sample, do not open the coarse vacuum on cell while pressure is above 77 mm Hg in manifold. That could contaminate your system with sample.



4. RUN TIME OPTIONS

4.1. ABORT ANALYSIS

To abort an analysis, press '.' (the period key). After 3 seconds confirm abort by pressing (1) or press (2) to continue the analysis. If the keypad (or keyboard) is inoperative, switch the NOVA OFF using the main power supply switch. Wait for 3 seconds and switch the unit back on again. Any data points acquired before the abort can still be retrieved. Turn on the instrument; the data will be loaded from memory into the instrument and saved in a file on the NOVA User Disk. The data can then be analyzed using the optional NOVAWin2 software. The last analysis can be reviewed or printed from the ANALYSIS MENU.

4.2. PAUSE ANALYSIS

To pause an analysis or cell calibration to access the degasser simply press the 'backspace' or the 'P' key. The LCD will display "Please Wait for Dose to Complete" to access the PAUSE MENU. Once the dose has completed, the PAUSE MENU will have the following options: (1) Access the Degasser and (2) Return to Analysis.

4.2.1. ACCESS DEGASSER

Selecting (2) allows access the degasser. The same options apply as if you had accessed the degasser from the Control Panel Menu.

4.2.2. RETURN ANALYSIS

Selecting (3) will return the instrument back to the analysis.

VII. MAINTENANCE AND REPAIR

7.1. CLEANING

The sample cells must be cleaned and dried after each use. Use regular tap water and a mild detergent to clean the sample cells using the stem cleaner provided. Flush several times (with tap water); rinse few times (with distilled or deionized water), and subsequently dry in an oven. An ultrasonic bath can be used to clean the fittings and o-rings (always rinse with distilled water before drying). Ensure that all parts are completely dried before use.

Heating mantles must be disconnected from the NOVA before cleaning. To remove dust from the outside of the mantle - use a dry, soft cloth. To remove inert, non-conductive sample which may have inadvertently entered the sample cell pocket, invert the heating mantle and gently tap the mouth of the pocket on a firm surface to dislodge the foreign matter. A heating mantle which has become contaminated by flammable material, liquids or conductive particles, e.g. metal powder, must not be used and should be discarded.

The instrument must be disconnected from the mains supply before cleaning.

To remove dust from the outside of the instrument, use a dry soft cloth.

Do not use acids, alkalis, or solvents to clean any part of the instrument.

Observe local regulations regarding handling and disposal of waste samples.

7.2. SPARE PARTS AND OPTIONAL PARTS LIST[1]

Description	Part Number	Quantity supplied with the NOVA			
		NOVA 1000 Series	NOVA 2000 Series	NOVA 3000 Series	NOVA 4000 Series
CE, Outgasser Mantle Assembly	01313-E	2	2	2	2
Input Line, Copper, 5'	01003	1	1	1	1
Heating Mantle Clamp	01053	2	2	2	2
1/8" Nut & Ferrule Set, Brass	01071	1	1	1	1
Long Flow Outgasser Adapter Assembly	01308-3941L	2	2	4	4
Glass Filler Rod Long 6 mm X 268.5 mm	74105-L	1	2	3	4
Glass Filler Rod Short 6 mm X 131.5 mm	74105-S	Opt	Opt	Opt	Opt
Glass Filler Rod Long 3 mm X 268.5 mm	74104-L	Opt	Opt	Opt	Opt
Glass Filler Rod Short 3 mm X 131.5 mm	74104-S	Opt	Opt	Opt	Opt
Glass Filler Rod Long 9 mm X 268.5 mm	74106-L	Opt	Opt	Opt	Opt
Glass Filler Rod Short 9 mm X 131.5 mm	74106-S	Opt	Opt	Opt	Opt
9 mm Cell Adapter	04000-3499-2	3	4	7	8
6 mm Cell Adapter	04000-3499-1	2	2	4	4
12 mm Cell Adapter	04000-3499-3	1	1	1	1
Sample Cell Funnel, Delrin	04000-3595	1	1	1	1

1 Not all parts are supplied with the instrument

Description	Part Number	Quantity supplied with the NOVA			
		NOVA 1000 Series	NOVA 2000 Series	NOVA 3000 Series	NOVA 4000 Series
Dewar	01603-7486-2	1	1	1	1
Coolant Level Sensor	00080-LN2-RTD	1	1	1	1
Fuse, 5 Amp, 5X20mm	24074	2	2	2	2
Line Cord, Domestic	26007	1	1	1	1
Line Cord, Foreign	26069	Opt	Opt	Opt	Opt
NOVAWin2-P Software	36041-2.0NWCFRCD	Opt	Opt	Opt	Opt
NOVAWin2 Software	36042-2.0NWCD	Opt	Opt	Opt	Opt
NOVAWin2 Instruction Manual	05079	Opt	Opt	Opt	Opt
Flange, Brass, Stub	44019	1	1	1	1
Centering Ring, KF16	44028	1	1	1	1
Clamp, Alum. KF16	44030	1	1	1	1
Vardex ¾" I.D. Vacuum Tubing	46025	5'	5'	5'	5'
O-Ring Buna, 010	51000-010	1 pair	1 pair	1 pair	3 pair
Clamp, Hose, 1" I.D.	71018	1	1	1	1

Description	Part Number	Quantity supplied with the NOVA			
		NOVA 1000 Series	NOVA 2000 Series	NOVA 3000 Series	NOVA 4000 Series
Nova, 9mm, large bulb, long cell	74064	2	3	4	5
Nova, 9 mm Large Bulb short cell	74064-1	Opt	Opt	Opt	Opt
Nova, 9 mm Small Bulb long cell	74063	Opt	Opt	Opt	Opt
Nova, 9 mm Small Bulb short cell	74063-1	Opt	Opt	Opt	Opt
Nova, 6 mm Large Bulb long cell	74062	Opt	Opt	Opt	Opt
Nova, 6 mm Large Bulb short cell	74062-1	Opt	Opt	Opt	Opt
Nova, 6 mm Small Bulb long cell	74061	Opt	Opt	Opt	Opt
Nova, 6 mm Small Bulb short cell	74061-1	Opt	Opt	Opt	Opt
Nova, 12 mm Large Bulb short cell	74066-1	Opt	Opt	Opt	Opt
Nova, 12 mm Small Bulb long cell	74065	Opt	Opt	Opt	Opt
Nova, 12 mm Small Bulb short cell	74065-1	Opt	Opt	Opt	Opt
LN2, Pick Up Tube (Long)	74069	1	N/A	N/A	N/A
LN2, Pick Up Tube (Short)	74069-1	Opt	N/A	N/A	N/A
Nova, Std. 9 mm Pel. Long	74099	4	5	8	9
Nova, Std. 9 mm Pel. Short	74099-S	Opt	Opt	Opt	Opt
Nova, Std. 6 mm Pellet Cell Long	74098	Opt	Opt	Opt	Opt
Nova, Std. 6 mm Pellet Cell Short	74098-S	Opt	Opt	Opt	Opt
Nova, Std. 12 mm Pellet Cell Long	74100	Opt	Opt	Opt	Opt
Nova, Std. 12 mm Pellet Cell Short	74100-S	Opt	Opt	Opt	Opt
6mm X 12" Stems Cleaner (1 Doz.)	96036	1 Pk.	1 Pk.	1 Pk.	1 Pk.
104 Keyboard (PS-2)	38056	Opt	Opt	Opt	Opt

7.3. SERVICE AND REPAIR

Service and repairs shall be conducted only by personnel trained for this purpose.

In North America contact:

Quantachrome Instruments
1900 Corporate Drive, Boynton Beach, FL 33426
tel (561) 731 4999
fax (561) 732 9888
email qc.service@quantachrome.com
URL: www.quantachrome.com

Outside North America contact your local authorized Quantachrome representative.

Do not return a unit for service or repair without prior authorization.

Decontaminate units before return. Do not return any units which have been radioactively, biologically or microbiologically contaminated. **These are not covered under warranty**.

VIII. NOVA OPERATING THEORY

The operating theory of the NOVA is outlined below.

Let η = moles of nitrogen gas transferred from a manifold of volume V_M at a temperature T_a into an empty sample cell partly immersed in liquid nitrogen. Then

$$\eta = \eta_c + \eta_w \tag{1}$$

where,

η_c = moles transferred to the cell cold zone
η_w = moles transferred to the cell warm zone

Assuming ideal conditions

$$\frac{\Delta P_M V_M}{R T_a} = \frac{\Delta P_c V_c}{R T_c} + \frac{\Delta P_w V_w}{R T_w} \tag{2}$$

Here, ΔP_M is the change in manifold pressure when gas is transferred to the cell. $\Delta P_w = \Delta P_c = \Delta P$ is the change in the pressure within the warm and cold zones of the sample cell and because they are equal, equation (2) can be written as

$$\frac{\Delta P_M V_M}{R T_a} = \frac{\Delta P}{R}\left(\frac{V_c}{T_c} + \frac{V_w}{T_w} \right) \tag{3}$$

Although there exists a temperature gradient between the cold and warm zones of the sample cell, the volume of gas within the cell can be treated as though it were apportioned to a warm and cold zone with an infinitely steep gradient between zones, i.e., no intermediate temperatures, without introducing any error in these calculations. Correcting equation (3) for gas nonideality in the cold zone leads to

$$\frac{\Delta P_M V_M}{R T_a} = \frac{\Delta P}{R}\left(\frac{V_w}{T_w} + \frac{V_c (1 + \alpha P)}{T_c} \right) \tag{4}$$

where P is the pressure within the cell and α is the nonideality correction factor of 6.6×10^{-5} $torr^{-1}$ for nitrogen.

When a sample is placed in the cell the volume in the cold zone, V_c, is reduced by the sample volume, V_s or $M_s \rho_s^{-1}$ where M_s and ρ_s are the sample mass and density, respectively. Then equation (4) can be written as

$$\frac{\Delta P_M V_M}{R T_a} = \frac{\Delta P}{R}\left(\frac{V_w}{T_w} + \frac{(V_c - M \rho^{-1})(1 + \alpha P)}{T_c} \right) \tag{5}$$

Solving equation (4) for the number of moles leaving the manifold, η_A, required to achieve a specified cell pressure yields

$$\eta_A = \frac{\Delta P}{R}\left(\frac{V_w}{T_w} + \frac{V_c\,(1 + \alpha P)}{T_c} \right) \tag{6}$$

and similarly, the number of moles leaving the manifold to achieve the same cell pressure when a sample is present but no adsorption occurs is given by

$$\eta_B = \frac{\Delta P}{R}\left(\frac{V_w}{T_w} + \frac{(V_c - M\rho^{-1})(1 + \alpha P)}{T_c} \right) \tag{7}$$

Subtracting equation (6) from equation (7) yields

$$\eta_B = \eta_A - \frac{\Delta P}{R}\,\frac{(M\,\rho^{-1})(1 + \alpha P)}{T_c} \tag{8}$$

Rewriting equation (8) for the corresponding gas volumes at standard temperature and pressure (STP) yields

$$V_B = V_A - \frac{\Delta P\, M\, \rho^{-1}\,(1 + \alpha P)}{760}\,\frac{273.15}{77.4} \tag{9}$$

where 77.4 is the standard boiling point (in Kelvin) of liquid nitrogen.

The NOVA stores a curve of equation (6) made on an empty cell in memory. When the operator enters the sample volume, or its density and mass, the computer calculates the curve corresponding to equation (9). If these curves are called the A and B curves respectively, their plots would appear as in Figure I.

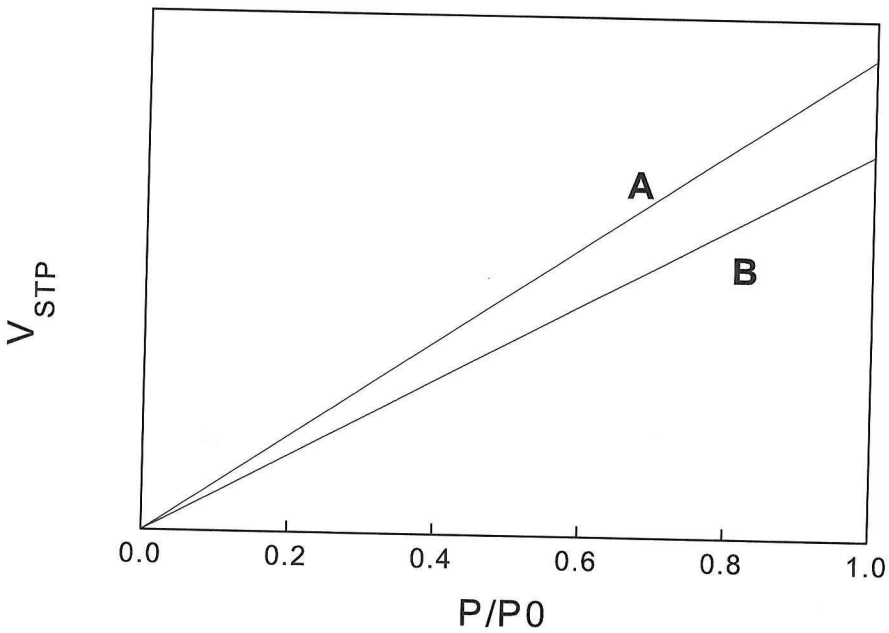


Figure I

The A curve is constructed using 25 data points and knowing the sample volume, it calculates the B curve value which is the true void volume of the sample cell.

The corresponding value on the B curve is subtracted from the actual adsorption or desorption volumes. Thus adsorption or desorption data are compensated for non-ideal gas behavior of only the gas volume in the cold zone of the sample cell and for adsorption on the cell walls.

When subtracting the B curve from the actual adsorption data the computer interpolates the data to ensure that the subtraction is performed at exactly the same pressures.

Once obtained, the A curve for any cell is stored on disk until the cell is recalibrated with a new A curve. Thereafter, you need only enter the sample mass and density in order to calculate a new B curve which is done automatically as adsorption or desorption data are acquired.

IX. GAS SORPTION THEORY

In this chapter, various theories for surface area and porosity measurements are discussed.

9.1. SURFACE AREA

The Brunauer-Emmett-Teller (BET) method[1] is the most widely used procedure for the determination of the surface area of solid materials and involves the use of the BET equation (1).

$$\frac{1}{W\left(\left(P_0/P\right)-1\right)} = \frac{1}{W_m C} + \frac{C-1}{W_m C}\left(\frac{P}{P_0}\right) \qquad (10)$$

in which W is the weight of gas adsorbed at a relative pressure P/P_0 and W_m is the weight of adsorbate constituting a monolayer of surface coverage. The term C, the BET C constant, is related to the energy of adsorption in the first adsorbed layer and consequently its value is an indication of the magnitude of the adsorbent/adsorbate interactions.

9.1.1. MULTIPOINT BET METHOD

The BET equation (10) requires a linear plot of $1/[W(P_0/P)-1]$ vs P/P_0 which for most solids, using nitrogen as the adsorbate, is restricted to a limited region of the adsorption isotherm, usually in the P/P_0 range of 0.05 to 0.35. This linear region is shifted to lower relative pressures for microporous materials. A typical BET plot is shown in Figure II.

The standard multipoint BET procedure requires a minimum of three points in the appropriate relative pressure range. The weight of a monolayer of adsorbate W_m can then be obtained from the slope s and intercept i of the BET plot. From equation (10):

$$s = \frac{C-1}{W_m C} \tag{11}$$

$$i = \frac{1}{W_m C} \tag{12}$$

Thus, the weight of a monolayer W_m can be obtained by combining equations (11) and (12).

$$W_m = \frac{1}{s+i} \tag{13}$$

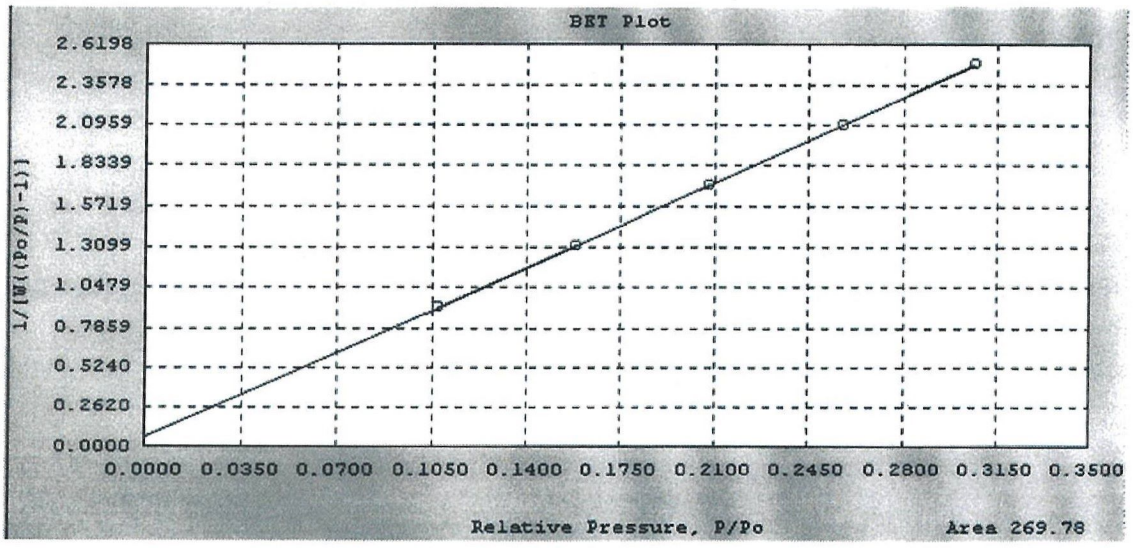


Fig. II. Typical BET plot

The second step in the application of the BET method is the calculation of the surface area. This requires a knowledge of the molecular cross-sectional area A_{cs} of the adsorbate molecule. The total surface area S_t of the sample can be expressed as:

$$S_t = \frac{W_m \, N \, A_{cs}}{M} \tag{14}$$

where N is Avogadro's number (6.023×10^{23} molecules/mol) and M is the molecular weight of the adsorbate. Nitrogen is the most widely used gas for surface area determinations since it exhibits intermediate values for the C constant (50-250) on most solid surfaces, precluding either localized adsorption or behavior as a two dimensional gas. Since it has been established[2,3] that the C constant influences the value of the cross-sectional area of an adsorbate, the acceptable range of C constants for nitrogen makes it possible to calculate its cross-sectional area from its bulk liquid properties. For the hexagonal close-packed nitrogen monolayer at 77 K, the cross-sectional area A_{cs} for nitrogen is 16.2 $Å^2$.

The specific surface area S of the solid can be calculated from the total surface area S_t and the sample weight w, according to equation (15):

$$S = S_t / w \tag{15}$$

9.1.2. SINGLE POINT BET METHOD

For routine measurements of surface areas a simplified procedure may be applied, using only a single point on the adsorption isotherm in the linear region of the BET plot. For nitrogen the C value is usually sufficiently large to warrant the assumption that the intercept in the BET equation is zero. Thus, the BET equation (10) reduces to

$$W_m = W\left(1 - P / P_0\right) \tag{16}$$

By measuring the amount of nitrogen adsorbed at one relative pressure (preferably near P/P_0 = 0.3) the monolayer capacity W_m can be calculated using equation (16) and the ideal gas equation.

$$W_m = \frac{P \, V \, M}{R \, T}\left(1 - P / P_0\right) \tag{17}$$

The total surface area then can be obtained from equation (14). That is,

$$S_t = \frac{P \, V \, N \, A_{cs}\left(1 - P / P_0\right)}{R \, T} \tag{18}$$

9.1.3. MULTIPOINT/SINGLE POINT COMPARISON

$$W_m = W\left(\frac{P_0}{P}-1\right)\left[\frac{1}{C}+\frac{C-1}{C}\left(\frac{P}{P_0}\right)\right] \qquad (19)$$

The relative error introduced by the single point versus the multipoint method for determining surface area is a function of the BET C constant and the relative pressure used. The magnitude of the error in the single point method can be determined from a comparison of the monolayer weight obtained from the BET equation (10) and the single point equation (16). Solving equation (1) for W_m gives:

$$W' = W\left[\left(P_0/P\right)-1\right]P/P_0 \qquad (20)$$

Rewriting the single point equation (16), gives the relative error inherent in the single point method, then, is

$$\frac{W_m - W_{m'}}{W_m} = \frac{1 - P/P_0}{1+\left[P/P_0\left(C-1\right)\right]} \qquad (21)$$

Equation (21) indicates that for a given C value, the relative error decreases with increasing relative pressure. Therefore, a relative pressure as high as possible, yet still in the linear region of the BET plot, should be chosen for single point surface area determinations. For all except microporous samples a P/P_0 of about 0.3 is preferable. For single point determinations on microporous samples a relative pressure as high as possible on the linear BET plot should be chosen.

Table I gives the relative error for various C values calculated from equation (13) using P/P_0 of 0.3. When the C constant is 100, a 2 percent error is indicated.

TABLE I

SINGLE POINT/MULTIPOINT COMPARISON

C CONSTANT	RELATIVE ERROR
1	0.70
10	0.19
50	0.04
100	0.02
1000	0.002
∞	0

Prior to using the single point method for the determination of surface area, the C constant can be evaluated from a multipoint BET plot. That is,

$$C = (s/i)+1 \qquad (22)$$

where s and I are the slope and intercept, respectively, of the BET plot. Subsequently, the single point method can be used on materials having the same composition. For greater accuracy, if the C constant is known, the single point result may be corrected using equation (21).

9.2. POROSITY BY GAS ADSORPTION

It is expedient to characterize pores according to their sizes.

a) Pores with openings exceeding 500 Å in diameter are called "macropores".
b) The term "micropores" describes pores with diameters not exceeding 20 Å.
c) Pores of intermediate size are called "mesopores".

Porosity of powders and other porous solids can be conveniently characterized by gas adsorption studies. Two common techniques for describing porosity are the determination of total pore volume and pore size distribution. For the evaluation of the porosity of most solid materials, nitrogen at 77 K is the most suitable adsorbate.

9.2.1. TOTAL PORE VOLUME AND AVERAGE PORE RADIUS

The total pore volume is derived from the amount of vapor adsorbed at a relative pressure close to unity, by assuming that the pores are then filled with liquid adsorbate. For a discussion of the relationship between pore size and relative pressure, see Section 3. If the solid contains no macropores the isotherm will remain nearly horizontal over a range of P/P_0 approaching unity and the pore volume is well defined. However, in the presence of macropores the isotherm rises rapidly near $P/P_0 = 1$ and in the limit of large macropores may exhibit an essentially vertical rise. In this case the limiting adsorption cannot be identified reliably with the total pore volume. The volume of nitrogen adsorbed (V_{ads}) can be converted to the volume of liquid nitrogen (V_{liq}) contained in the pores using equation (23). That is,

$$V_{liq} = \frac{P_a \; V_{ads} \; V_m}{R\,T} \qquad (23)$$

in which P_a and T are ambient pressure and temperature, respectively, and V_m is the molar volume of the liquid adsorbate (34.7 cm^3/mol for nitrogen).

Since pores which would not be filled below a relative pressure of 1 have a negligible contribution to the total pore volume and the surface area of the sample, the average pore size can be estimated from the pore volume. For example, assuming cylindrical pore geometry (type A hysteresis), the average pore radius r_p can be expressed as

$$r_p = \frac{2V_{liq}}{S} \tag{24}$$

where V_{liq} is obtained from equation (23) and S is the BET surface area. For other pore geometries a knowledge of the shape of the hysteresis in the adsorption/desorption isotherm is required.

9.2.2. PORE SIZE DISTRIBUTIONS (MESOPORE)

The distribution of pore volume with respect to pore size is called a pore size distribution. It is generally accepted that the desorption isotherm is more appropriate than the adsorption isotherm for evaluating the pore size distribution of an adsorbent. The desorption branch of the isotherm, for the same volume of gas, exhibits a lower relative pressure, resulting in a lower free energy state. Thus, the desorption isotherm is closer to true thermodynamic stability. In certain cases, for example, samples exhibiting type E hysteresis, the adsorption isotherm is recommended for pore size distribution determinations. The NOVA offers the capability of using either branch of the isotherm for the calculation. Since nitrogen has been used extensively in gas adsorption studies, it has been well-characterized and serves as the most common adsorbate for pore size distribution determinations. Therefore, the following discussion will apply to the use of nitrogen as the adsorbate.

Mesopore size calculations are made assuming cylindrical pore geometry using the Kelvin equation in the form

$$r_K = \frac{-2\gamma V_m}{RT \ln(P/P_0)} \tag{25}$$

where

γ = the surface tension of nitrogen at its boiling point (8.85 ergs/cm^2 at 77 K).
V_m = the molar volume of liquid nitrogen (34.7 cm^3/mol).
R = gas constant (8.314x10^7 ergs/deg/mol).
T = boiling point of nitrogen (77 K).
P/P_0 = relative pressure of nitrogen.
r_K = the Kelvin radius of the pore.

Using the appropriate constants for nitrogen, equation (25) reduces to

$$r_K = \frac{4.15}{\log\left(P_0 / P\right)} \tag{26}$$

The Kelvin radius r_K (in Å) is the radius of the pore in which condensation occurs at a relative pressure of P/P_0. Since, prior to condensation, some adsorption has taken place on the walls of the pore, r_K does not represent the actual pore radius. Conversely, during desorption an adsorbed layer remains on the walls when evaporation occurs. The actual pore radius r_p is given by

$$r_p = r_k + t \tag{27}$$

where t is the thickness of the adsorbed layer. This statistical t can be considered as 3.54 Å (V_{ads} / V_m) in which 3.54 Å is the thickness of one nitrogen molecular layer and V_{ads} / V_m is the ratio of the volume of nitrogen adsorbed at a given relative pressure to the volume adsorbed at the completion of a monolayer for a nonporous solid of the same composition as the porous sample. A more convenient method for estimating t (in Å) was proposed by Halsey[4] in the form of equation (28) and is used by default for pore size distribution calculations in the NOVA.

$$t = 3.54 \left[\frac{5}{2.303 \log\left(P_0 / P\right)}\right]^{1/3} \tag{28}$$

The NOVA data reduction system computes the pore size distribution using the method proposed by Barrett, Joyner and Halenda[5] (BJH).

9.2.3. BJH METHOD

Assuming that the initial relative pressure $(P/P_0)_1$ is close to unity, all pores are filled with liquid. The largest pore of radius r_{p1} has a physically adsorbed layer of nitrogen molecules of thickness t_1. Inside this thickness is an inner capillary with radius r_K from which evaporation takes place as P / P_0 is lowered. The relationship between the pore volume V_{pl} and the inner capillary (Kelvin) volume V_K is given by

$$V_{p1} = \frac{V_{K1}\, r_{p1}^2}{r_{K1}^2} \tag{29}$$

When the relative pressure is lowered from $(P/P_0)_1$ to $(P/P_0)_2$ a volume V_1 will desorb from the surface. This liquid volume V_1 represents not only emptying of the largest pore of its condensate but also a reduction in the thickness of its physically adsorbed layer by an amount Δt_1. Across this relative pressure decrement the average change in thickness is $\Delta t_1 / 2$. The pore volume of the largest pore may now be expressed as :

$$V_{p1} = V_1 \left(\frac{r_{p1}}{r_{K1} + \frac{\Delta t_1}{2}} \right)^2 \tag{30}$$

When the relative pressure is again lowered to $(P/P_0)_3$ the volume of liquid desorbed includes not only the condensate from the next larger size pores but also the volume from a second thinning of the physically adsorbed layer left behind in the pores of the largest size. The volume V_{p2} desorbed from pores of the smaller size is given by:

$$V_{p2} = \left(\frac{r_{p2}}{r_{K2} + \frac{\Delta t_2}{2}} \right)^2 \left(V_2 - V_{\Delta t_2} \right) \tag{31}$$

An expression for $V_{\Delta t_2}$ is

$$V_{\Delta t_2} = \Delta t_2 \; Ac_1 \tag{32}$$

where Ac_1 is the area exposed by the previously emptied pores from which the physically adsorbed gas is desorbed.

Equation (32) can be generalized to represent any step of a stepwise desorption by writing it in the form

$$V_{\Delta t_n} = \Delta t_n \sum_{j=1}^{n-1} Ac_j \tag{33}$$

The summation in equation (33) is the sum of the average area in unfilled pores down to, but not including, the pore that was emptied in the desorption. Substituting the general value for $V_{\Delta t_n}$ into equation (31) results in an exact expression for calculating pore volumes at various relative pressures.

$$V_{pn} = \left(\frac{r_{pn}}{r_{Kn} + \frac{\Delta t_n}{2}} \right)^2 \left(\Delta V_n - \Delta t_n \sum_{j=1}^{n-1} Ac_j \right) \tag{34}$$

Since the area (Ac) for any one size empty pore is not a constant but varies with each decrement of P/P_0, this term must be evaluated.

The area of each pore A_p is a constant and can be calculated from the pore volume, assuming cylindrical pore geometry. That is,

$$A_p = \frac{2\ V_p}{r_p} \tag{35}$$

Then the pore areas can be cumulatively summed so that for any step in the desorption process A_p is known. The BJH method offers a means of computing $\Sigma\ A_{c_j}$ from A_p for each relative pressure decrement as follows:
It is assumed that all pores emptied of their condensate during a relative pressure decrement have an average radius $\underline{r}_p$ calculated from the Kelvin equation (25) radii at the upper and lower values of P/P_0 in the desorption step. The average capillary (core) radius is expressed as

$$\bar{r}_c = \bar{r}_p - \bar{t}_r \tag{36}$$

where $\bar{t}_r$ is the thickness of the adsorbed layer at the average radius in the interval in the current pressure decrement and is calculated from equation (28).

The term "c" in equation (33) then is given by

$$c = \frac{\bar{r}_c}{\bar{r}_p} = \frac{\bar{r}_p - \bar{t}_r}{\bar{r}_p} \qquad (37)$$

Equation (34) now can be used in conjunction with equation (37) as an exact expression for the computation of pore size distributions.

9.2.4. EXTERNAL SURFACE AREA (STSA)

The NOVA applies, as an option, a t curve derived for carbon blacks[6] to the evaluation of external surface area (or statistical thickness surface area, STSA) according to de Boer's method[7]. This method involves the measurement of nitrogen adsorbed by the sample at various low pressure values. The procedure is the same as that employed in the BET surface area measurement, but it extends the pressure range to higher pressures to permit calculation of the matrix surface area, that is, the non-micro porous part of the material. A t-plot is a plot of the volume of gas adsorbed versus t, the statistical thickness of an adsorbed film. In the NOVA the t (in Å) values are calculated by default as a function of the relative pressure using the carbon black equation,

$$t_{CB} = 0.88\left(P/P_0\right)^2 + 6.45\left(P/P_0\right) + 2.98 \qquad (38)$$

Alternative t curves and calculations are available through an optional NOVA data reduction software package.

Typical t-plots are shown in Figures III, IV and V, representing various possible pore sizes. Figure III is a t-plot of a sample having no micropores, as evidenced by the ability to extrapolate the line to the origin, since the slope represents the total surface area S_t of all the pores, that is,

$$S_t = \frac{V_{ads}^{STP}\ (15.47)}{t} \qquad (39)$$

where V_{ads}^{STP} is the volume of gas adsorbed corrected to standard conditions of temperature and pressure and the constant 15.47 represents the conversion of the gas volume to liquid and t is in Å. Using the slope, s, of the plot in Figure III, equation (39) reduces to

$$S_t\ (m^2/g) = s \times 15.47 \qquad (40)$$

In the absence of micropores there is good agreement between the t-area, S_t, and the surface area determined by the BET method.

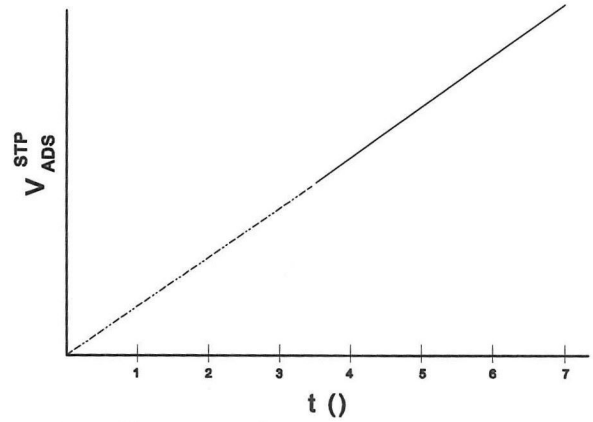


Fig. III. t-plot of a mesoporous material.

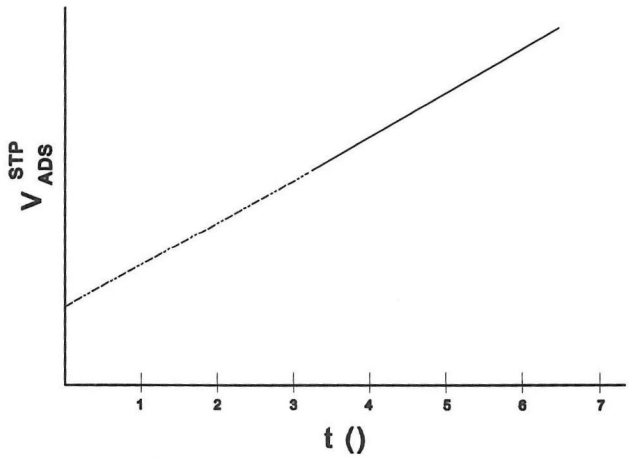


Fig. IV. t-plot of a microporous sample.

APPENDIX A. NOVA DOSING ROUTINE

The procedure used by the NOVA to dose and obtain a data point is described below. The explanation is for acquisition of adsorption data, although a desorption point is obtained in a similar manner.

DEFINITION OF TERMS

P_{max}

P_{max} is the maximum working pressure for acquiring data. When the adsorbate used is nitrogen, P_{max} is equal to the working value of P_0. Otherwise, the value of P_{max} is 800 mm Hg. If P_0 is greater than P_{max} then P_{max} = 800 mm Hg. If P_0 is less than 800 mm Hg then $P_0 = P_{max}$.

TARGET PRESSURE

The target pressure is the desired relative pressure (P/P_0), defined by the user, where a data point should be taken.

EQUILIBRIUM PRESSURE TOLERANCE

The pressure tolerance is the user defined range (mm Hg), within which the pressure in the cell must remain for the defined equilibration time in order that the point be accepted as a valid data point. The acceptable range is 0.05 to 2 mm Hg.

EQUILIBRIUM TIME TOLERANCE

The equilibration time is the user defined time during which the pressure in the cell must not change by more than the defined pressure tolerance in order that the point be accepted as a valid data point. The acceptable range is 18 to 1800 seconds.

DWELL TIME

If the pressure tolerance is not met, and further, if the pressure does not fall below the lower pressure limit (upper limit for desorption), then the dwell time limit will be invoked and will
cause the data point to be taken. The acceptable dwell time range is from twice the equilibration time to 5400 seconds.

PRESSURE LIMITS

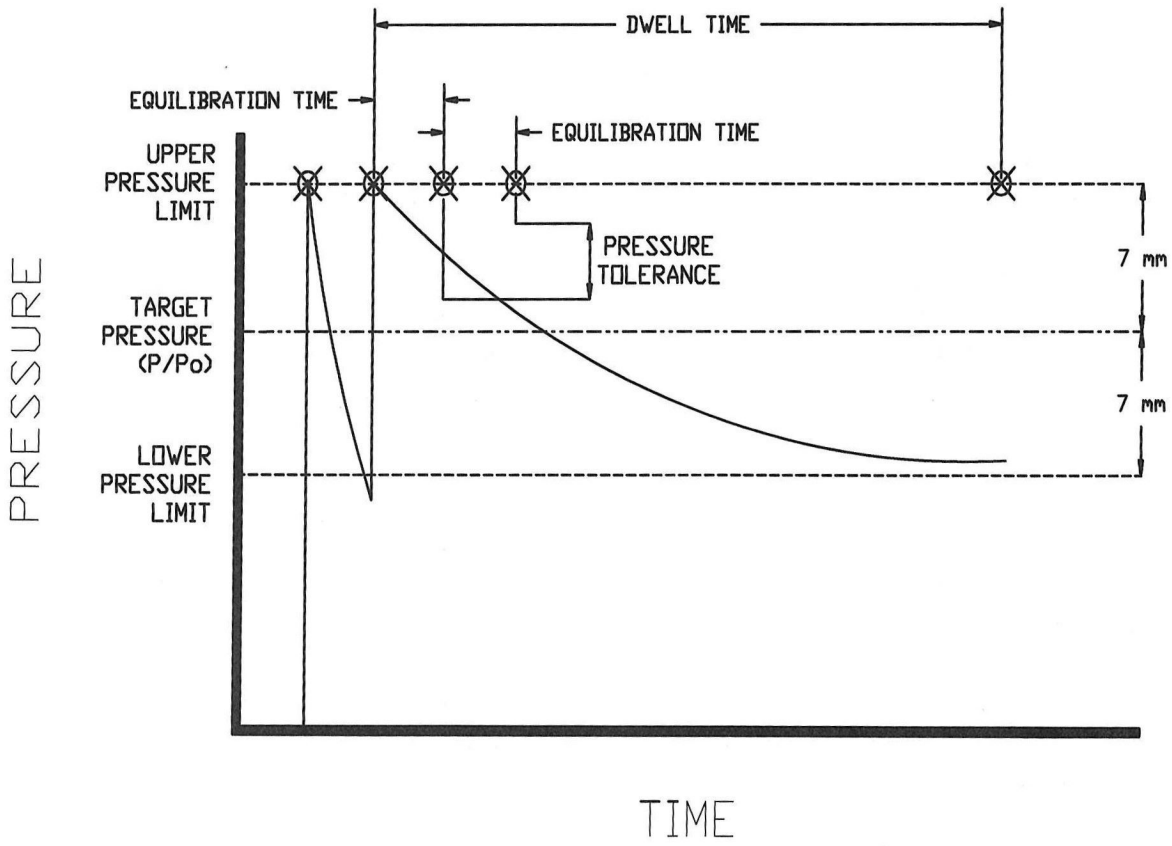
The pressure limit is a window of 7 mm above and below the target pressure.

PROCEDURE

Adsorbate is introduced into the manifold until the pressure is 7 mm above the target pressure (upper pressure limit, see Figure below). The valve to the cell is then opened and the adsorbate is admitted to the sample cell. If the pressure falls below the lower pressure limit, the dosing procedure is repeated. If the pressure does not fall below the lower pressure limit, the pressure is measured every 6 seconds. If the pressure does not change by more than the user defined pressure tolerance for the equilibration time specified, the data is accepted and stored as a valid data point.

If the pressure does change by more than the user defined pressure tolerance, the instrument will continue to monitor the pressure until it meets the pressure and time requirements. If the pressure again falls below the lower pressure limit, the instrument will redose the sample and the process will start over. If the pressure does not fall below the lower pressure limit and does not meet the pressure tolerance requirements, the point will be accepted as valid data when the dwell time limit has been reached. If more than one sample station is used, the same procedure is used to acquire a data point on the second station after a point has been obtained for the first station.

The NOVA also employs a "MAXI-DOSE" routine which monitors the rate of adsorption and adjusts the dose to bring the sample to equilibrium more quickly.


DWELL TIME
EQUILIBRATION TIME
EQUILIBRATION TIME
UPPER PRESSURE LIMIT
PRESSURE TOLERANCE
7 mm
TARGET PRESSURE (P/Po)
7 mm
LOWER PRESSURE LIMIT
PRESSURE
TIME

APPENDIX B. CORRECTION OF ERRORS ASSOCIATED WITH LONG ANALYSIS

During the course of any analysis the bath level will continuously decrease due to evaporation of the liquid coolant. An analysis which takes substantially longer to complete than a cell calibration, such as a complete adsorption and desorption isotherm, will be accompanied by a much larger change in coolant level than the change associated with the sample cell set calibration. This will result in warming of part of the volume within the groove in the filler rod and the annulus between the rod and the glass wall. Therefore, less gas will be contained in this volume at any given pressure. Thus, through any cycle such as

$$P/P_0 = 0.1 \rightarrow P/P_0 = 0.9 \rightarrow P/P_0 = 0.1$$

The warming of the sample cell will require that more adsorbate gas be removed from the cell than was introduced. This can cause the desorption isotherm to fall slightly below the adsorption isotherm.

To compensate for this, Quantachrome has incorporated a routine called TempComp™ (US Patent 6,387,704). TempComp has been incorporated into the firmware routine and is used for all measurements.

You must ensure that the liquid nitrogen level in the dewar is always at the bottom point of the Coolant Level Indicator before the start of any calibration or analysis.

APPENDIX C. RECOMMENDATIONS FOR OPTIMAL ANALYSIS

The following mode of operation is recommended to ensure greater accuracy using the NOVA:

1. During calibration, ensure that all stations have a sample cell attached to them even if calibrating only one station. For greater accuracy, use same type of cells in all stations (if using a 9 mm small bulb long stem cell in Station A of a Nova 3000, then use 9 mm small bulb long stem cells in the other two stations).
2. During analysis, all stations should have sample cells attached to them even if you are performing an analysis on one station only. As above, use same cell combinations in all stations to obtain greater accuracy in measurements.
3. Fill the liquid nitrogen (or other cryogenic liquid) to about 2.5cm (1 inch) from the top before starting any analysis or calibration. If there is ice present in the dewar (coolant may be bubbling and may appear cloudy), empty the coolant and replace with fresh coolant.

APPENDIX D. RECOMMENDED VALUES FOR COMMON ADSORBATES

GAS	TEMP (°C)	α FACTOR X 10^{-5} (1/mm Hg)	CROSS SECTIONAL AREA ($Å^2$/molecule)	MOLECULAR WEIGHT (g/mol)
Ar	-195.8	11.4	14.2	39.948
	-183	3.94		
CO_2	-78	2.75	19.5	44.01
	-25	1.55		
	0	1.75		
CO	-183	3.42	16.3	28.01
N_2	-195.8	6.58	16.2	28.0134
	-183	3.78		
O_2	-183	4.17	14.1	31.9988
C_4H_{10}	0	14.2	46.9	58.12
	25	4.21		

APPENDIX E. TROUBLESHOOTING GUIDE

Symptom	Reason	Action
Open isotherm (adsorption / desorption loop does not close i.e. desorption branch always lies above adsorption branch even at low relative pressure.	Under-equilibrated conditions.	If isotherm is *unexpectedly* open, extend the desorption equilibration time. Microporous materials often need longer desorption equilibration times.
	Incomplete outgassing.	Extend outgassing time and/or increase out gassing temperature.
Crossed isotherm.	Leaks.	Check integrity of o-ring on sample cell. Make sure o-ring is lightly greased. Make sure that the bulkhead is free from other o-rings.
	Incorrect cell geometry defined during calibration.	Choose proper cell calibration file. Sample measurement conditions must be identical (temperature, use of filler rod) to the cell calibration conditions.
	System contamination.	Pump down unit overnight.
Dewar goes up but analysis hangs.	Upper limit switch not hit.	Check for obstruction to dewar travel.
Unit fails vacuum integrity test on boot up.	Vacuum hose and/or fittings leak.	Check hose and tighten fittings. Replace as necessary.
Mantle not hot.	Thermocouple not plugged in.	Ensure cables are plugged into correct outlets.
	Mantle power cable not plugged in.	
Po cell blows out.	LN_2 evaporated below Po cell.	Shorten analysis time: change equilibration conditions, reduce amount of sample etc. Replace faulty dewar.
Sample cell blows out.	Same as for Po cell.	
Sample elutriates during outgassing.	See 5.3.4 Elutriation and Its Prevention.	
Series of rapid beeps sound during calibration or analysis run.	This feature called "Ready Alarm" indicates that the calibration or analysis run is completed.	

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