JOIDES Resolution:

TECH HELP

A Reference for the Physical Properties Specialist

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Ocean Drilling Program
Texas A & M University

Overview

Tech Help is designed to give the physical properties scientists and technicians a **quick** and **easy** reference to the laboratory. For more detailed instructions it is suggested that the reader refer to the respective 'Cookbook' (where available) or Peter Blum's manual.

Tech Help is separated into sections according to instrument stations: whole-core multi-sensor track (MST), thermal conductivity, velocity & shear strength (V&S), electrical resistivity and index properties (MAD). Each section contains a general overview of the instrument(s) and calibration/measurement procedures.

This manual, however, does not include much information about uploading raw data files to the JANUS database. There is another manual to describe the database, written by IS.

Multi-sensor Track (whole core)

Whole Core Multisensor track
GEOTEK Gamma Ray Attenuation
Bartington MS2C Magnetic Susceptibility
ODP Natural Gamma Radiation
GEOTEK P-wave velocity logger

Discrete Measurements (whole or split core)

TK04 Thermal Conductivity
VSR Track
ODP P-wave velocity (PWS1, PWS2, PWS3)
ODP Vane Shear (AVS)

Discrete Measurments (extracted samples)

Quantachrome penta-pycnometer
Cahn Balance

WC-MST Subsystems

Parker/Compumotor Indexer w/VM50Terminal Strip
Parker/Compumotor Microstep Drive - S Series
EG&G NIM Bin w/12 module Connectors &160 watt Power Supply
EG&G Preamplifier SCA
EG&G MST Module - Multi Channel Buffer w/ Option 5 Card
EG&G High Voltage Power Supply
Bicron Detectors - NAI
Perkin Elmer Internal Card - PCI, Trump-PCI-2K
Magma Bus Expander - 7 slot PCI Expansion System
EG&G ADCAM Multichannel Buffer (917-918A Option E)
Parker/Compumotor Step Motor
National Inst. Signal Breakout Box
EG&G Pulser
Water Pump
EG&G Photomultiplier
EG&G High Voltage Fan-Out
EG&G Dual Spectroscopy Amplifier
EG&G Dual Sum and Inverter Amplifier

VSR-Track Subsystems

Nulogic Motion Control Board - PCI Step-4CL, 4 axis closed loop
Panametrics Ultrasonic Pulser-Receiver
Magnetek Linear Voltage Displacement Transducer (LVDT)
OMEGA Digital Thermometer
National Inst. Oscilloscope Boards
National Inst. PCI 4A Encoder Interface Board
OMEGA Linear Velocity Transducer Signal Conditioner
OMEGA Panel Meter

1.0 Introduction

Welcome to the Physical Properties Lab aboard the R/V JOIDES Resolution. Hopefully it will be a very productive leg with a lot of hard work, co-operation and patience.

Shipboard physical properties data is used:

- to characterize lithologic units
- to correlate stratigraphic units
- to correlate laboratory and wire-line downhole logging data
- to aid in the interpretation of geophysical data

The following is a brief description of the lab routines. Please contact the PP MLS/lab officer/electronics technician before trying any maintenance on the equipment in the lab.

1.1 Before reaching the first site

Become familiar with the various computers and operating systems (MAC OS, Windows 95 and DOS); file servers and the Janus database.

Calibrate all the instruments:

- GRAPE, PWL and NGR on the MST (Section 3.3)
- Thermal conductivity probes for the old thermcon box (if they will be used during the leg (Section 4.41). The TK04 thermcon probes do not require calibration
- PWS 1 and PWS 2 (Section 5.3.1) and PWS 3 (modified Hamilton Frame; Section 5.3.2)
- Electrical resistivity probes (Section 6.2)
- Electronic balance which is done in **port** (Section 7.31)
- Pycnometer (Section 7.32)

1.2 Once on site

- ensure station ***log-sheets*** are completely filled out for each measurement
- ensure that raw data is being written to the server QUAKE:DATA1:JANUS_Q and the local hard-drive (used as a back up) under the JANUS_Q folder
- upload raw data files from QUAKE:DATA1:JANUS_Q to the JANUS database. These files are automatically moved to the TRNFRD folder on DATA1
- Add depths to the raw data (thermcon, resistivity) that are not JANUS supported
- view results and download to a spreadsheet from Netscape (MST/PWS/AVS/MAD)
- Clean your work area before the next shift starts. You will want them to

1.3 Before returning to port

Backup all site data/reports to QUAKE:DATA1 in their respective folder.

During the leg it is essential to explain to the Marine Lab Specialist any changes made (what and why), instrument failures, etc.

Be precise as possible - future legs will rely on these reports to solve similar problems.

2.0 Core Flow

Core flow through the lab is generally straightforward.

The core is first cut on the catwalk into 1.5 meter sections. The sections are scribed and labeled in the core lab and allowed to equilibrate to lab temperature (generally 17 C which takes roughly 2 to 3 hours).

Once the core has equilibrated they are run through the whole-core MST.

Thermal conductivity (*TK04 meter*) measurements are then made prior to splitting the sections if the sediment is soft or after splitting the sections if it is lithified.

The sections are then cut into working and archive halfs.

Discrete velocity, shear strength and electrical resistivity measurements are made on the split working-half.

Index property samples are then taken, prior to shipboard sampling. The index property samples are weighed, dried for 24 hours and dry weight and volumes are then measured.

Once the archive MST is up and running, certain modifications will have to be done to this report!

Core flow is illustrated in *Figure 1*.

3.0 Multi-Sensor Track (MST)

3.1 Introduction

The whole-core Multi-Sensor Track (MST) consists of a *magnetic susceptibility meter*, *GRAPE*, *compressional wave logge*r and a *natural gamma ray unit*. The whole-core MST LabVIEW application coordinates track motion with sensor alignment. This allows for simultaneous measurement and acquisition of magnetic susceptibility, bulk density, P-wave velocity and natural gamma radiation data. The primary goal of the MST is to obtain continuous down core measurements and improve core flow.

3.1.1 Magnetic Susceptibility

The magnetic susceptibility loop on the MST track is connected to a Bartington magnetic susceptibility meter (MS2). The units are set to SI.

3.1.2 Gamma Ray Attenuation Porosity Evaluator (GRAPE)

The measurement of bulk density (g/m^3) by the GRAPE is based on the Compton scattering effect of low energy gamma rays during collisions with other electrons. Gamma rays are transmitted using a ^{137}Cs source through the core and the attenuated signal is detected with a NaI sensor. Attenuation occurs from Compton scattering and from absorption (photoelectric). Compton scattering is the primary process and occurs when the gamma ray (photon) gives up energy upon collision with an electron.

3.1.3 Automated Compressional Wave Core Logger

The P-Wave Logger (PWL) measures the travel time of a compressional sound wave through a sediment filled core liner. This travel time combined with the transducer separation is used to calculate the P-wave velocity. The absolute resolution of the PWL is 50 ns in time and 0.04 mm in distance which would typically provide a velocity resolution of 0.1% (~1.5 m/s in unconsolidated sediments). The temperature of the core must be recorded in order to correct velocity measurements to in-situ values. The velocity measurements require good acoustic coupling between the transducers, the core liner and the sediment within the liner. Velocity measurements are therefore only taken on APC cores containing sediment with few voids or cracks.

3.1.4 Natural Gamma Ray measurements

Natural radioactivity is the result of the random and discrete spontaneous decay of atoms. The decay is accompanied by the emission of alpha or beta particles, gamma rays, and the generation of heat. Gamma radiation may be considered as an electromagnetic wave similar to visible light or X-rays, or as a particle or photon emitted from an atomic nucleus during radioactive decay. Its characteristic wave length is 10^{-10} to 10^{-12} cm. In contrast to alpha and beta particles, gamma rays have a high power of penetration and therefore serve as a basis for downhole and core logging techniques.

Radioisotopes with sufficiently long-life, and whose decay produces an appreciable amount of gamma rays, are Potassium (^{40}K), Thorium (^{232}Th) and Uranium (^{238}U). The gamma rays emitted from the three main radioisotopes have a number of discrete energies. Each series has a spectral signature that enables its presence to be discerned. Characteristic peaks are 1.46 MeV for ^{40}K, 2.62 MeV for ^{232}Th and 1.76 MeV for ^{238}U. The relative amplitudes of the three spectra will depend on the proportions of the radioactive components present. A quantitative evaluation of the presence of K, Th and U can be found

by dividing the total spectrum into the three characteristic spectra.

Gamma radioactivity is expressed either in g Ra equivalent/metric ton or in GAPI (Gamma ray American Petroleum Industry) units. NGR logs, including the ones generated during ODP operations, are reported in GAPI units.

NGR tools consist of gamma ray detectors (scintillation) and associated electronics for passing the gamma ray count rate to the recording interface. The scintillation counter contains a doped sodium iodide crystal and a photomultiplier to produce countable pulses. When a gamma ray strikes the crystal, a single photon of light is emitted which strikes a photocathode made from cesium antimony or silver magnesium. Photons hitting the photocathode release bundles of electrons, which are accelerated in an electric field to strike a series of anodes of successively higher potential. A final electrode conducts a small current through a measure resistor to give a voltage pulse, signaling that a gamma ray struck the NaI crystal. The detectors are arranged at 90 angles from each other in a plane orthogonal to the MST track. The peak height of each pulse is measured and stored in the appropriate one of 2048 channels; this, and the EG&G MAESTRO graphical software interface (running on the PC), allow for spectral gamma ray analyses.

3.2 Getting Started

The WHOLE CORE MST CONTROL LabVIEW program is located on the Power Mac (MC 90). Make sure that the only network the MC90 computer is connected to is the DATA1 server on QUAKE. Click and drag all other networks to the trash. Do not have any other programs running.

The program is started by double-clicking on the LabVIEW icon located on the desktop (WholeCoreMST179) and the LabVIEW virtual instrument library files will load. The first panel that appears is the MST TRACK CONFIGURATION screen and it illustrates the relative positions of the various sensors. There should be no need to change the distance configuration of the sensors or the core boat (unless of course a sensor was moved or a new track belt was installed). Errors in relative positions become obvious during calibration procedures. Please do not try to remove the boat's water control standard. The standard is a section of core liner filled with distilled water and can be used for drift correction of the data. At the beginning of the leg you will want to set-up the track and boat to make sure that the left and right limit switches and zero point are set (also everytime the program is restarted). Select **OK** to continue.

A message now appears to load the MST parameter file. The parameter file consists of sample information for each sensor including whether it is on/off, sample interval, sample period and what distance to skip from the top and bottom of each section. Measurement parameters are set from the edit parameter option on the main panel. Select **????.parm** for now and you can set up the parameter file (3.4.1) from the main control panel at a later time.

3.3 Calibrations

3.3.1 Magnetic Susceptibility loop

There is no calibration of the MS2 sensor.

3.3.2 GRAPE

The GRAPE 'calibration standard' consists of a core liner filled with distilled water and a section of aluminum with 6 different diameters. The average density differs within the liner depending on the diameter of the aluminum (See Table 1). The first step in the calibration is to define the GRAPE standard. Select **DEFINE GRAPE STANDARD** from the main control panel and enter densities for five positions along the standard. The first position is water only and the remaining four positions are at the middle of four of the different aluminum diameters. Make sure that you are measuring these positions relative to the top of the standard. Hit **SAVE** to return to the main control panel or if no changes are necessary select CANCEL and CANCEL to the Update Preference File option. Place the standard on the core boat with the blue end cap (top) aft and the clear end cap (bottom) against the water control. Select **CALIBRATE GRAPE** with a 45 second DAQ period and 'Full Liner' and then **GO.** The core boat moves along the track and measures total counts for the five different positions. When the run is completed the total counts vs density is plotted. Select **SAVE** and then **OK** to update the preference file with the new calibration constants.

Aluminum Diameter (cm)	Average Density (g/cc) (Salt Water)	Average Density (g/cc) (Distilled Water)
0	1.024	1.000
1	1.280	1.259
2	1.534	1.518
3	1.790	1.777
4	2.046	2.036
5	2.301	2.296
6	2.557	2.555

Table 1. Aluminum diameters and corresponding densities for the GRAPE standard.

The liner can be filled with distilled water or salt-water but distilled water is preferred. The density of the aluminum is 2.71 g/cm^3.

3.3.3 P-Wave Logger

The P-wave logger set-up/calibration needs to be performed at the beginning of the leg. Select **DEFINE PWL STANDARD** from the main control panel and make sure the ID's and lengths for the PWL standards (there are four acrylic standards) are correct and toggled to 'In Use'. If no changes are necessary select **CANCEL** and **CANCEL** to the Update Preference File option. Select **CALIBRATE PWL**. Settings for the calibration run are: 1) frequency of *500 KHz*; 2) pulse time correction of *2.00 sec*; 3) DAQ time of *5 seconds*; and 4) signal threshold of *30* . Switch the transducer position to CLOSED on the grey box located below the counter-top. Place an acrylic standard between the transducers. Wet the standard/transducer contact to ensure a good signal. If the pointer is set at the right standard position on the front panel select **MEASURE**. Repeat for the next three standards. Make sure

that the number of points to calibrate is set at '*4'* and click the **CALIBRATE** button. Switch the transducer position back to OPEN.
The linear fit and calibration coefficients for the displacement transducer and the electrical delay time (system delay) are displayed with plots of distance vs displacement and time vs distance. Select **SAVE** and **OK** to update the preference file. The calibration should be checked by running the distilled water filled liner through the PWL. If the measured values are correct for distilled water at the present lab temperature the calibration does not need to be corrected. If the velocities are incorrect the calibration needs to be adjusted. Select **CALIBRATE PWL** and manually increase or decrease the electrical delay and run the water control again. Continue this procedure until the measured velocity for the distilled water is correct for the given lab temperature. It may require several control runs to get the correct adjustment for the electrical delay.

Note that the power button for the switches controlling the automatic movement of the PWL transducers is beside the power cord connection on the back of the grey box.

3.3.4 Natural Gamma Ray

The physical properties Marine Lab Specialist should perform this calibration.

The calibration of the natural gamma ray unit consists of two stages. First it is necessary to ensure that corresponding energy events detected by the four sensors are counted by similar channels. The MAESTRO graphical user interface is used to check to see that only one peak is present for the corresponding standards. Take the two thorium sources and place them in the center of the NGR. Disconnect sensors two through four. Start the MAESTRO program located on PC 25 and select **START** from the **ACQUIRE** menu. Adjust the gain on the amplifier of the connected sensor until there is a single peak at 2.62 MeV (plus low energy counts).

Repeat for the other sensors and then recheck the peak with the four sensors connected.

The second part of the calibration involves assigning the proper channels to the LabVIEW application. Select **CALIBRATE NGR** from the main control panel. Place the thorium sources in the NGR unit and select **Start Calibration DAQ** and continue collecting data until you see a defined peak near channel 211. Place the curser in the center of the peak and select **ASSIGN**. Repeat this procedure for the KCl source and then select **CALIB & SELECT WINDOW**. Select **SAVE** and **OK** to update the preference file.

The background value for the NGR is also measured. Select **BACKGROUND NGR** from the main control panel. You can either use the water standard or no standard at all (air) to do the reference

(background) measurement. Then select **START BACKGROUND DAQ** and **SAVE THE BKGD** when finished. Select **OK** to update the preference file.

3.4 Measurements

Running a section on the MST is, for the most part, a straight forward task.

The MST data and control files are assigned run numbers and saved in the QUAKE:DATA1:JANUS_Q directory and the local hard drive in the JANUS_Q directory. Each sensor has its own folder (WC-MS, GRAPE, PWL, NGR) and within each sensor folder there are CALIBRATION, CONTROL and SiteHole folders. The files are assigned a file name corresponding to the run number on the MST (ie. DATA1:JANUS_Q:GRAPE:GRAPE *SiteHole\ Grape run #.* dat). There is no reference to core or section numbers. All calibration and control files are placed in calibration and control folders with no SiteHole designation
(ie. DATA1:JANUS_Q:GRAPE:GRAPE CALIBRATION:*Grape run #.* cal).

A log sheet is kept by the MST Control Center to record sample ids, run number and comments. This sheet is used to locate data files which need to be edited (generally sample ID) or deleted (bad runs) prior to data upload to Janus. It is therefore essential to keep a record of each run and note factors affecting data quality and sections that were rerun.

3.4.1 Sample Parameters

The sampling parameter requirements for the sensors need to be defined prior to collecting measurements on the MST. The scientific party will decide on sampling parameters based on expected core recovery, leg objectives and time limitations. Select **EDIT PARAMETERS** from the main control panel.
The options for each sensor include:

- sensor on/off?
- sample interval (cm)
- sample period (sec)
- avoid the top and bottom length of each section (used to avoid the section end caps)
- set the signal threshold (for the PWL)

The **PLOT AND TIME ESTIMATE** utility displays the relative sample positions selected for each sensor and the estimated time required to scan a 150cm core section.

The boat's water control should ALWAYS be measured; so make sure it is toggled to collect data.

The selected parameters may be saved to site specific or hole specific parameter files. This is a convenient method to record sampling parameters as defined by the scientific objectives. It also allows any operator to load the correct sampling parameters if the program has to be restarted. To save a parameter file select **DONE** and then **SAVE**. Select **LOAD PARAMETER FILE** to load or edit an existing parameter file. Remember to Update Preference File after a parameter file has been saved.

3.4.2 Data Collection

Place the core section on the core boat with the bottom (clear or yellow cap) tight against the core boat water control. The **CHANGE SITE** utility on the main control panel is used to set leg#/site#/hole#.

Select **FULL SCAN** and the SECTION INFORMATION control panel will appear. Enter the sample ID (Core#/core type/section#), check the temperature of the core and of the ambient air and the sensors which are turned on. Select **NORMAL** or **CONTROL** for measurement scan type. Normal scan is used for core sections. The control scan is used for measurements on a distilled water filled core liner.

The LINER CONTROL toggles between **FULL**, **HALF** (hard rock sections) or **NO** liner while CORE CONTROL enables you to select **FULL** or **HALF** core.

If a whole round sample was removed from the top of the section, you must enter its length in the **TOP OF SECTION MISSING** utility. The MST software assumes that the physical top of the section is 0cm. You can also avoid measuring any empty liner at the end of the section (hard rock sections) by entering a value in the **Ignore Bottom of Section** control. Record the appropriate information on the MST log-sheet and ENSURE that the section ID corresponds to the correct run number.The section number is automatically incremented by one after each run and the core number will increment after the section number has reached seven; we do not usually run core catchers on the MST.

Select the **GO** button and the MAIN CONTROL panel appears and the core boat moves aft (to the left) until the top of the section breaks the optical beam. At this point the boat stops, the software determines the section length, calculates the track motion profile and sensor control and takes necessary background measurements. The boat then moves to its first measurement position and begins the scan. The scan can be stopped by selecting the **ABORT** button on the main control panel or by hitting one of the two hard wired ***EMERGENCY STOP*** switches located on the shelve aft and forward of the four sensors. The program reacts quicker to the ***EMERGENCY STOP*** switches; therefore, use these if there is a possibility that the sensors or track will be damaged.

When the scan is complete, the core boat moves to the unload position. Remove the core section HERE

and then save the data files by selecting **SAVE**. If for some reason you did not want to save the data for a particular sensor, toggle to **DON'T SAVE DATA**. Select **RETURN CORE** to return the core boat to the load position and prepare another section to be run.

3.4.2.1 Discrete Scan

The Discrete Scan option does not use the current parameter settings but allows the operator to select:

- specific points within the section to measure
- the core diameter at that point
- which sensors to use

The discrete scan is usually used for hard rock sections with large gaps between pieces.

3.4.3 Front Panel plots

Data collected by each sensor is plotted in real time on the main control panel. The y-axis of the plots are auto scaling while the x-axis is set at 155cm.

3.5 Limited Trouble Shooting

If the core boat does not respond to the Whole Core MST program:

- Call for Help. If no-one responds...cry and scream and jump up and down
- Check to make sure that the optical switches are aligned
- Go to **TRACK UTILITIES** (main panel) and select **ZERO BOAT**
- Go to **CONFIGURE TRACK** and select **TRACK SET UP** and the core boat will scale the steps between the limit switches at both ends of the track
- Quit the program and restart it
- Ensure the QUAKE:DATA1 server is on the desktop
- If the **EMERGENCY STOP** button was pressed, make sure that you pull it back out...heh

4.0 Thermal Conductivity

4.1 Introduction

Thermal conductivity (Thermcon/TC) measurements and downhole temperature measurements are required for accurate geothermal heat flow determinations. The thermal conductivity (**k**) of a material is the amount of energy or heat per second needed to make the material, some distance from the heat source, increase in temperature by some amount. More quantitatively, **k** is the coefficient which gives the rate of heat transfer (**Q**) across a given steady-state temperature difference (**T**) over a given distance (**x**) in a material:

$$\mathbf{Q = k (\ T/\ x)} \qquad \mathbf{(1)}$$

Thermal conductivity is derived from the slope of the temperature vs. the logarithm of time plot. Units of **Q** are $\mathbf{W\ m^{-2}}$ (SI system), so that units of **k** are $\mathbf{W\ m^{-1}\ (\ C)^{-1}}$ (SI system).

The full-space needle-probe method (soft sediments) of measuring **k** uses transient heating which is a result of a steady line source of heat from a probe. It is assumed in this method that the thermal properties of the material being measured are uniform and isotropic, that the line heater is infinitely long in an infinitely extended material, and that the temperature of the material does not change, independent of the heat input.

The half-space needle-probe method (used for lithified sediments and hard rock samples) assumes that the same theory applies, but requires that a correction factor be included to account for the geometry of the experiment, since the medium is assumed to be semi-infinite rather than fully-infinite.

There are two thermal conductivity meters in the PP lab. The 'old' TC box, developed by Woods Hole, and the TK04 meter, developed for the German DSDP program. There are several advantages to using the TK04 meter: 1) there is a noticeable reduction in data scatter; 2) the meter requires no probe calibration, while it may take 2 days to calibrate the probes for the old box; and 3) the software requires less data entry. Although the TK04 meter can only accommodate one probe, compared to five for the old box, using the TK04 meter does not affect core flow.

4.2 Sample Preparation

The sediment cores must equilibrate to lab temperature; so a good time to take a measurement will be after the section has been run through the MST. The TC software program will not begin collecting data until the temperature drift is within a predetermined range. The thermcon measurements are corrected

for the drift determined immediately prior to the start of the thermcon measurements.

Thermcon measurements can be made using either the full-space or half-space probes:

- The full-space method is used in soft sediment prior to splitting the core. The needle-probe is inserted through a drilled hole in the *working* half section of the sediment core. The probes break and bend easily, therefore DO NOT FORCE the probe into the sediment.

- The half space method is used on a polished (240 grit) half of hard rock sample or lithified sediment. Half-space measurements are taken with the sample and probe submerged in a salt water bath which reduces the effect of temperature variations.

- Thermal joint compound can be used to insure good thermal contact with the probe and the sediment or rock sample. Check with the other scientists regarding any concerns (geochemists, petrologists etc.) about using the thermal joint compound.

- It is recommended that a consistent sampling interval be used, unless lithology or core conditions dictate otherwise. In this way you will be able to avoid intervals designated for other studies, provide for more evenly spaced sampling intervals and significantly reduce the amount of data entry.

4.3 TK04 Thermal Conductivity Meter

The full-space line source (**VLQ**) and the half-space line source (**HLQ**), use different applications, boundary conditions and evaluation procedures. There are two batch jobs that set the appropriate measuring and evaluation parameters for each method.

The VLQ (full space) parameters should be set at **3** for the number of measurements, **2** W/m for the heating power and **150** seconds for the measuring time. The data is evaluated by the 'Conventional Method' (CON) (see TK04 manual) with time limits of 40 and 300 seconds and an evaluation time interval of 240 seconds.
The HLQ (half space) parameters should be set at **3** for the number of measurements, **3** W/m for the heating power and **80** seconds for the measuring time. The data is evaluated with the 'Special Approximation Method' (SAM) (see TK04 manual) with time limits of 20 and 80 seconds and an evaluation time interval of 25 seconds.

Current adjustments required when changing heating power or between full and half space measurements are made using the current adjust dial on the TK04 unit. Recommendations for heating power for a wide range of materials are contained in the TK04 manual. These values can be changed in

the **TKSAM.INI** and **HALFSP.INI** files, however, it is not recommended to change them. To change site/hole or test and evaluation parameters, type **resetpar** in the c:\TK04\ directory on the PC105 computer.

To run the VLQ method (full space) while in the TK04 directory type:

needle *site/hole* eg. **needle 1074A**

To run the HLQ method (half space) while in the TK04 directory type :

halfsp *site/hole* eg. **halfsp 1075B**

The main menu (TKMEAS) appears:

1) Probe # : ####
2) Data directory : **c:\tk04\SiteHole**
3) Root name : **core core type section-**
4) Serial No. Start at : **1**
5) No. of measurements: (at least **2**)
6) Heating power [W/m] : (usually start around) **2** *(full space)* **3** *(half space)*
7) Measuring time (sec): **150** *(full space)* **80** *(half space)*
8) Save data? (**Y**/**N**)
9) Comments: **sample interval, observances, etc.**

A data directory, named after the site/hole, is created within the TK04 directory. Two data files are created for each measurement. The **.dwl* file contains the raw data including temperature, time and resistance while the **.lst* file contains the thermal conductivity value and other semi-useful information. The file TC-LIST.DAT within the SiteHole directory is the collection of all the data for a particular SiteHole.

4.3.1 Adding Sub-Bottom Depths

Presently, the thermal conductivity station is not supported by JANUS; therefore, depths need to be added to the sample intervals. To add depths, create an EXCEL spreadsheet with the thermcon values and use the JANUS depths utilities option. The spreadsheet must have no header information and the first eight columns must contain
leg / site / hole / core / core type / section / top interval / bottom interval. The thermcon measurement is placed in the ninth column. Save the file as 'text' format. Log into JANUS and select **DEPTHS** under the **PREFERENCES** menu. Choose the depth scale required and then select **DEPTH UTILITY** under the **REPORT** menu. Select the thermcon spreadsheet file (no depths) and also a file to be created (depths added) and run the program.

4.4 OLD Thermcon Box

The old thermcon box can accommodate 5 probes, therefore 5 thermcon measurements can be made simultaneously. The needles will burn out during a run if they are left exposed and therefore must be either inserted into a sample or a standard. A standard is measured during each run. Rotating the probes in the standard allows for early detection of a failing probe and drift correction of data.

There is a large amount of flexibility inherent in the procedures required for data processing. Familiarity with the test procedures and limitations and the properties of the standards will help the user to apply good judgement in selecting samples and time intervals for processing. The physical property scientists should determine the parameters for data processing in order to ensure consistency.

4.4.1 Calibration

The thermcon needles should be calibrated at the beginning of each leg. There are four standards used for calibration. Black rubber has a thermal conductivity of 0.54 and is designated as STD 2, gelatin has a thermal conductivity of 0.67 and is designated as
STD 4; red rubber has a thermal conductivity of 0.96 and is designated as STD 1; macor has a thermal conductivity of 1.61 and is designated as STD 3; and basalt (used only for half space probes) has a thermal conductivity of 2.05 and is designated as STD 5.

The first step is to change the old calibration values for the needles. Do this by editing the PROBES.DAT file in the C:\TCPROG directory on the PC21 computer. Change the seventh column to 1.00 (slope) and the eighth column to 0.00 (y intercept) for each needle to be calibrated. The first column is the probe ID and the sixth column indicates if the needle is used for full-space (0) or half-space (-1).

Measure each standard at least five times with all the probes. Plot the measured values vs the known values for each standard and perform a linear regression. Edit PROBES.DAT again and enter the new slope and y intercept from the regression. It is recommended that the scientists run the standard measurements and process the data to become familiar with the procedure.

4.4.2 Measurements

To begin measurements, insert the needles you want to use into the back of the old Thermcon box in ascending order (this reduces confusion when probe position is required). The position designation corresponds to the port on the back of the box (position 1 is K1).

Access the thermcon program by typing **TCMENU** at the **C:\TCPROG** prompt. The TC menu will appear with three choices:

1) COLLECT THERMCON DATA
2) PROCESS THERMCON DATA
3) EXIT TO DOS

Select 1 for data collection. The program will prompt the operator for general information including leg, site, hole, core, run number and number of probes in use for this run. The value in the brackets [ ] is the default. The length of the TC run is always six minutes.

Enter the needle number positions and corresponding sample information including section and interval. When measuring standards enter "ST" for the section number and the corresponding standard designation for standard number.

Once all the sample information is entered press the reset button on the thermcon box when prompted by the program. The program now conducts a drift study and displays the temperature drift at each sample interval. After each position has equilibrated the drift study can be terminated by pressing 'O'. Next press the reset button on the thermcon box when prompted by the computer and data collection will begin.

4.4.2 Processing the Data

Enter '**2**' at the main menu for data processing and specify the run number to process. Next enter the position to process and answer yes for drift correction.

A graph of the temperature and time will appear. The vertical cursors enclose the values that will be used to calculate the thermal conductivity. The interval is generally between 60 and 240. The cursors can be moved manually if the program selection is not optimal. Press the return key once and then select **Y** if the data is to be used. A hard-copy printout is produced for possible later time-series analysis. Make sure the printer is 'on-line' or the computer will freeze up. Note on the log sheet any high error or drift warnings. If the data appears to follow a non-linear path, don't use the run, and remeasure the sample interval.

4.4.3 Adding Sub-Bottom Depths

The thermal conductivity station at present is not supported by JANUS; therefore, depths need to be added to the sample intervals. To add depths create an EXCEL spreadsheet with the thermcon values and use the JANUS depths utilities option. The spreadsheet must have no header information and the

first eight columns must contain
leg / site / hole / core / core type / section / top interval / bottom interval. The thermcon measurement is placed in the ninth column. Save the file in 'text' format. Log into JANUS and select **DEPTHS** under the **PREFERENCES** menu. Choose the depth scale required and then select **DEPTH UTILITY** under the **REPORT** menu. Select the thermcon spreadsheet file (no depths) and also a file to be created (depths added) and run the program.

5.0 Velocity and Shear Strength (V&S)

5.1 Introduction

V & S is a LabVIEW program for the Digital Sediment Velocimeters (DSV), the modified Hamilton Frame and the Wykeham-Farrance vane apparatus. The measurements are made on the split working sections, before sampling, which ensures an undisturbed section for the measurements.

The DSV is a digital data acquisition system developed to measure interval velocities and attenuation in sediment cores. The DSVs are designated as PWS1 and PWS 2 (P-Wave Station). The velocity calculation is based on the travel time of an impulsive acoustic signal travelling between two piezoelectric (acoustic) transducers of a known separation (PWS 1 & 2). Velocity measurements can be made longitudinal (Z direction) or transverse (Y direction) to the core axis. The blades containing the ultrasonic transducers are imbedded directly into the core. A high voltage pulse is then applied to the transmitter transducer. The compressional waveform received, after transmission across the core material, is digitized by an oscilloscope.

The modified Hamilton frame (PWS 3) measures velocity 'into' the split core (X direction on a normal working half) and is used for stiffer sediment or hard rock samples. PWS 3 can also measure wrt any orientation with the cutting of a cube out of a section. The transducer separation is measured with a sliding scale. The pressure applied, to the sample as the top transducer is lowered to come in contact with the sample, is also measured.

The Wykeham-Farrance vane remains a reliable measurer of sediment shear strength. This technique assumes an "undrained" fine-grained marine sediment (unlithified) sample, and accordingly, employs a very rapid vane shear speed of 90 degrees of torque per minute. The torque applied to the vane is measured, just prior to failure, by a calibrated spring stress, from which the sediment's shear strength can be calculated. A thorough discussion of operation and theory is presented in Boyce's (1976) DSDP Initial Reports paper (see Vane Shear folder). Once a sediment has become 'biscuited', the vane shear tests are not meaningful.

Shear strength can also be measured with hand-held Torvanes and pocket penetrometers. Adaptor heads can be used with the Torvanes to obtain a wider range of sensitivity. These devices are used in sediment which is too stiff to measure with the vane blades.

5.2 Getting Started

The computer controlling the V&S is the PowerMac (MC 58). The only network that the computer should be connected to is DATA 1 and make sure that no other programs are running.

To start the LabVIEW program, double-click on the VSR CONTROL LabVIEW icon on the desktop. Refer to the V&S CookBook for specific details on anything in this chapter.

The SYSTEM INFORMATION panel now appears, illustrating the transducer orientations and vane blade rotation rate. There is no need to modify the system set up. Click on **OK**. Next appears the EQUIPMENT STATUS panel where the GPIB addresses and serial port connections are configured. Select **OK** to proceed.

The TRACK CONFIGURATION panel now appears. The optical position encoder at the aft end of the

track enables sample position at each sensor to be automatically determined. The track has to be calibrated **whenever** the V&S program is started. Mark off a distance of 100cm along the track using a meter stick (mark the 0 and 100 cm points on the track for future calibrations). Move the cylinder so its clip is at the 0cm mark and select **ZERO** on the panel. Repeat for the 10 cm mark but click on the **100** button. Now move the cylinder so that the clip is at the center of each sensor, selecting the appropriate button for each sensor. Select **OK** when finished.

The LEG SITE AND HOLE panel now appears. Enter the appropriate leg site and hole data and select **DONE**. The main V&S control panel is now assessable.

5.3 Calibrations

It is essential (prior to calibrating and taking measurements) that the proper time sequence is followed when creating standards, calibration and data files. The standards files contain one time stamp corresponding to the time the files were created. The calibration files contain two time stamps - one for the time they were created and the second corresponding to the time stamp for the standards file. This second time stamp must be the same as the time stamp in the standards file. The data files also contain two time stamps - one corresponding to the time it was created and one to the time of the last calibration. The calibration files are uploaded to JANUS only if the corresponding standards files are in JANUS. The data files are uploaded to JANUS only if the corresponding calibration files are in JANUS. Confused? Refer to V&S manual or get the PP tech. It is a good idea to create new PWS standards and controls files at the beginning of each leg, prior to calibrations and measurements.

Select **LINER AND CONTROL STANDARDS** from the main V&S control panel. There should be no need to make changes and simply choose **SAVE** for the water control and the liner control. Select **DONE** to return to the main panel. Now select **PWS STANDARDS** and the PWS DESIGN A STANDARD panel appears. Make sure each standard is named, material is defined and control buttons are set to
'IN-USE'. Select **SAVE** and return to the main V&S control panel.

Calibrating PWS 1, PWS 2 and PWS 3 consist of measuring the electrical delay time for the three systems.

5.3.1 PWS 1 and PWS 2

Select **PWS 1 CALIBRATION**. Set the DTL (Detection Threshold Limit) to '*1'*. Lower the transducers (using the hydraulic lever) into a half core liner filled with distilled water and also insert the thermocouple into the water outside of the transducers. Adjust the lower knob on the O-scope to highlight the first arrival with the blue cursor. Select **DOWNLOAD**. The computer will pick the first arrival time. The delay time is then calculated using the velocity of sound in distilled water for a given temperature. The first arrival time can also be picked manually by dragging the vertical yellow cursor to the appropriate position. The computer recalculates the delay time if the cursor is moved. For a check, a table of P-wave velocities in distilled water at various temperatures is located on the wall next to the Janus upload computer. Select **SAVE** when finished. Repeat the above for PWS 2.

5.3.2 PWS 3 (Hamilton Frame)

Calibrating PWS 3 is a little more involved, but just as easy. A series of measurements are made using distance standards. The standards are polycarbonate mini-cores, found in the Hamilton Frame drawer.

First, zero the sliding scale by lowering the top transducer so it is in contact with the lower transducer. Note the pressure applied and use this pressure (2000 ± 40 generally works ok) for future measurements.

Select **PWS 3 CALIBRATION** from the main V&S control panel. Spray both transducer surfaces with distilled water. Place a standard on the lower transducer and screw down the top transducer, applying the same pressure used to zero the sliding scale. Press the green DATA button on the MIG-1 (GPIB-9) to download the separation distance from the sliding scale. Adjust the lower knob on the oscilloscope to highlight the first break. Select **DOWNLOAD** and the computer will pick the first arrival time. Ensure the distance and time values are correct for the marked standard and select **ASSIGN**. Repeat the above procedure for the remaining three standards.

Enter the number of standards used for the calibration in the "**USE ONLY THE FIRST...**" box. Select **CALIBRATION** and the delay time is calculated. Select **SAVE** when finished.

5.3.3 Shear Vane

There are spring and vane calibrations for the automated shear vane. The spring calibrations were performed prior to shipment to the JR. Select **SPRING CALIBRATION** to see the spring constants.The vane calibrations require measuring the height and diameter of the blades on the vanes. Select **VANE CALIBRATION** to see the vane calibration data. There should be no need to change these values.

5.4 Measurements

5.4.1 PWS 1 and PWS 2

PWS 1 and PWS 2 are used only in soft sediment or until the sediment pulls apart when the blades are inserted. Position the working half under the PWS at the desired sample interval. Lower the transducers into the sediment. Insert the thermocouple close to the transducers, but not in-between the transducer blades.The temperature is automatically recorded, however the velocity is not corrected to in-situ temperatures values. Adjust the lower knob on the oscilloscope to highlight the first arrival with the blue cursor. Select **DOWNLOAD**. The computer will usually pick the first arrival time but it can also be manually picked by dragging the yellow vertical cursor to the position. Note the velocity on the paper logsheet. Select **SAVE** and then **DONE/CANCEL** to return to the main V&S control panel.

5.4.2 PWS 3

PWS 3 is used for stiffer sediment or hard rock. The measurements can be made while the core is in or out of the liner or on mini-cores/cubes. The mini-cores/cubes can also be used for index property measurements.

Screw down the top transducer to the predetermined pressure. Press the green DATA button on the MIG-1 (GPIB-9). Ensure that the orientation and liner correction switches are set for the appropriate measurement. Use 2.54 mm for liner thickness if you are using the liner correction. Adjust the lower knob on the oscilloscope to highlight the first break. Select **DOWNLOAD** and the computer will pick the first arrival time and calculate the velocity. Note the velocity on the paper logsheet. Select **SAVE** to save the data and **DONE/CANCEL** to return to the main panel.

If measurements are being taken on mini-cores or cubes cut out of the working half sections of the core, care must be taken to make parallel faces along each orientation. This will facilitate a good contact

between the transducers and some meaningful measurements. Also make sure to toggle to 'Manual Position Input' so you can manually input the sample interval.

5.4.3 Shear Vane

Position the core under the vane apparatus. Lower the vane into the sediment using the hydraulic lever. Select **AVS** from the main panel. A screen with a list of springs and vanes appears. Choose the spring and vane presently in use. Select **GO** and the AVS will begin rotating. The data is displayed on the stress (kPa) vs degrees of rotation plot. Select **STOP** after the peak and residual strengths are defined.

Select **SAVE RAW DATA** to save the raw data (all data points). Select **USE RESIDUAL STRENGTH** to save the residual strength value. Note the strengths on the paper logsheet. Selecting **SAVE** will save the peak shear strength value and other data specified. Back the vane blade out of the sample, and gently tamp down the rough spots on the sediment surface. Return the core section to the proper place on the working table. Wash the vane blades with the old toothbrush and some distilled water.

5.4.4 Data Files

The data files are written to the JANUS_Q directory on DATA1 and on the local hard drive (as a back up). Each sensor has its own folder (PWS 1, PWS 2, PWS 3, AVS) and within each sensor folder there are CALIBRATION and SiteHole folders. The file names correspond to the run number (incremented automatically) on the V&S program. There is no reference to the core or section number (ie. DATA1:JANUS_Q:AVS:AVS *SiteHole*:AVS *run number*.dat). All calibration files are placed in calibration and folders with no SiteHole designation (ie DATA1:JANUS_Q:PWS 1:PWS 1 CALIBRATION:PWS 1 *run number*.cal).

5.5 Limited Trouble Shooting

- To reset the O-scope, select the **REINITIALIZE** button while you are in the main V&S control panel and toggle to reset the O-scope. This feature will become VERY handy when you start playing with the different settings, knobs and dials on the O-scope.

6.0 Electrical Resistivity

6.1 Introduction

The resistivity measurements utilizes a four probe configuration (Wenner spread) with two current and two potential electrodes. An alternating current is applied to the two outer electrodes and the potential drop (resistance) across the two inner electrodes is measured by the Wayne-Kerr instrument.

Resistivity (**r**) is defined as the resistance of a unit area across a unit length:

$$\mathbf{r = R\ (A/l)} \qquad \mathbf{(2)}$$

where **R** = resistance in **ohms ( )**
r = resistivity in **ohm-m**
A/l = area/length parameter in $\mathbf{m^2/m}$ = **C**

6.2 Calibrations

Conversion of the measured resistance value to resistivity requires determining the probe's geometric factor. The geometric factor can be determined by calibration of the probe using a known fluid (seawater) or by knowing the separation distance between the electrodes on the probe. This distance can be measured on the larger probes and the geometric factor (**G**) can be found by:

$$\mathbf{G = 2*\ \ *l} \qquad \mathbf{(3)}$$

where **l** = the measured separation distance (SI units). Resistivity is then found by:

$$\mathbf{r = R_{meas} * G} \qquad \mathbf{(4)}$$

Prior to the probe calibration and/or sample measurements the Wayne-Kerr bridge should be set up. Connect the color coded cables from the probe to the Wayne-Kerr box. Make sure the settings are:

15 KHz testing frequency
10 mA current
BIAS is OFF
Impedance selector on Z
AUTO range selection
Repetitive measurements at a NORMal rate

Press the Trim O/C button on the Wayne-Kerr. Wait a couple of seconds and press the trigger button as instructed on the monitor. Connect the four electrodes with a piece of wire (paper clip). Press the Trim s/c button and then the trigger button as instructed by the monitor.

The other calibration procedure finds the area/length parameter (**C**) for the specific probe geometry. The calibration is performed by placing the probe in a saltwater filled half-liner at a known temperature (**T** : C) and measuring the resistance. Take quite a few calibration measurements, both longitudinal and transverse. Salt water resistivity (salinity = 35 ppm) is calculated by:

LINESPACE 150 r_{ known} ~ = ~ (2 . 803~ + ~ 0 . 0996~T)^{ - 1} **(5)**

The seawater resistivity ($\mathbf{r_{known}}$) and the measured resistance ($\mathbf{R_{meas}}$), are used to calculate the **A/l** parameter (**C**):

$$\mathbf{C = r_{known}/R_{meas}} \qquad \mathbf{(6)}$$

Note the values for G and C should be approximately the same.

6.3 Measurements

The resistivity of an interval is therefore determined by:

$$\mathbf{r = R_{meas} * C} \qquad \mathbf{(7)}$$

The resistivity measurements can be used to determine the formation factor and to estimate porosity. The formation factor is the ratio of the resistivity of the saturated sediment to the resistivity of the pore fluid. The coefficients for each lithology are normally determined by plotting the formation factor vs. measured porosity and fitting an exponential curve to this data.

6.3.1 Soft sediment

Measurements should be made with the probes aligned along (longitudinal) and/or across (transverse) the core axis. Anisotropic effects may be determined if both measurements are made at each interval. Measurements across bedding planes should be avoided. The sediment temperature at the test interval must also be recorded.

There are probes of different geometries. The two larger probes provide more consistent results then the smaller probes. It is suggested to calibrate one or two different probes in order to be familiar with the Wayne-Kerr instrument. Try measuring resistivity on different saltwater salinities. Compare the resistivity values obtained from using the measured distance and the calibration value.

Carefully push the probe electrodes into the working half of the section. Make sure to record the appropriate data on the log-sheet. The data has to be manually entered into a computer spreadsheet - no nice LabView set-up here. The resistance values fluctuate (count down). This is probably occurring since current is flowing through the pins, the sediment between the pins will be heated, and the resistance of the sediment changes with its temperature. Therefore, wait about 2 minutes for the readings to stabilize. It is very important to standardize the measurement procedure among operators. The

temperature is measured by inserting the thermocouple into the sediment. Note the sediment temperature, resistance value, orientation and interval on the paper logsheet.

6.3.2 Hard rock

The hard rock resistivity method utilizes two probes which measure the resistance across a mini-core sample. The Wayne-Kerr instrument should be set to measure the resistance at 50 KHz instead of the 15 kHz setting used for soft sediment. The sample is placed inside the plastic holder. It may be helpful to apply an electrically conductive paste to each end of the sample, at the electrode contact interface, to ensure good signal. The area (**A**) and length (**l**) of the sample are measured with calipers. Resistivity is then calculated using Eq. 2. Resistivity should be in SI units therefore use ohms for **R** and meters for **A/l**.

6.4 Adding Sub-Bottom Depths

The electrical resistance station at present is not supported by JANUS; therefore, depths need to be added to the sample intervals. To add depths create an EXCEL spreadsheet with the resistivity values and use the JANUS depths utilities option. The spreadsheet must have no header information and the first eight columns must contain
leg / site / hole / core / core type / section / top interval / bottom interval. The resistivity measurement is placed in the ninth column. Save the file in 'text' format. Log into JANUS and select **DEPTHS** under the **PREFERENCES** menu. Choose the depth scale required and then select **DEPTH UTILITY** under the **REPORT** menu. Select the resistivity spreadsheet file (no depths) and also a file to be created (depths added) and run the program.

7.0 Index Properties

7.1 Introduction

The index properties measured/derived include bulk density, grain density, dry density, water content, porosity and void ratio. Three methods are used to determine index properties. The three methods differ in the way that the wet volume is determined:

- *Method A* - the wet volume is measured using a sampling ring or constant volume sampler of known volume
- *Method B* - the wet volume is measured using the pycnometer
- *Method C* - the wet volume is derived from the measured wet mass, dry mass and dry volume. The method of choice is C because the pycnometer is considered by some to be inaccurate when measuring saturated sediments.

The index properties measuring station is controlled by the MAD (Moisture and Density) LabVIEW program. This program communicates with the balance and pycnometer allowing for automatic entry of volume and mass data. A second LabVIEW program, MAD Sample Download retrieves sample id information from the Janus database after it has been entered at the sample table work station. Sample ids can also be entered by scanning the bar codes on the sample labels or by manually entering the sample id via the SAMPLES menu.

It is **EXTREMELY** important to keep organized paper logsheets because you will find that you have to do a lot of manual MAD editing.

7.2 Getting Started

To load sample ids from the JANUS database to the MAD program, you must run the MAD Sample Download program and constantly run the program when new samples are entered into the database.

To start the MAD program, double click on the Moisture Density LabVIEW icon on the desktop. The MAIN MENU panel will appear. The chooses on the main menu include: **VOLUME**, **MASS**, **CALIBRATE BALANCE**, **CALIBRATE PYCNOMETER**, **SAMPLES**, **REPORTS**, **OPTIONS** and **QUIT**.

Select **OPTIONS** to enter balance and pycnometer parameters. Balance parameters include number of measurements, time for measuring and allowable standard deviation. The pycnometer parameters include purge time, number of measurements and standard deviation of the last three measurements. If three continuous measurement are within .1% then the run for that cell is completed.

7.3 Calibrations

7.3.1 Scientech 202 Electronic Balance

The balance must be calibrated in port, while the ship is "motionless". Calibration is performed by measuring a series of calibration weights of known mass, over the expected differential mass range (about 5 gram), thus giving a linear relationship between mass and voltage.

Select **CALIBRATE BALANCE** from the MAIN MENU panel and follow the balance calibration procedure outlined on the CALIBRATE BALANCE panel.

7.3.2 Pycnometer

There are two calibrations to be performed. The internal reference cell (V_A) and the five individual cells (V_C). The V_A and V_c are calibrated at the beginning of the leg. The V_c calibration is checked throughout the leg by measuring the sphere standard in one cell per pycnometer run and then rotating the sphere between cells after each run. Repeat the V_c calibration for a specific cell if the measured volume is ±0.02 cm^3 of the known sphere volume (7.0699 cm^3). The control measurements are conducted the same as a sample measurement (see below).

Select **CALIBRATE PYCNOMETER** from the MAIN MENU panel and follow the instructions provided on the PYCNOMETER CALIBRATION panel. The MAD program assumes small as the size of the cell holders. Calibration of the pycnometer for large size sample holders therefore must be completed using the pycnometer's keypad. It is a good idea if time permits to calibrate the large sample holders in case they are required during the leg.

7.4 Measurements

The discrete index property measurements are taken as soon as the core is split. The samples are placed in a 10ml Pyrex beaker to about 3mm below the rim (to prevent material losses during handling). The beaker is covered with parafilm to prevent moisture loss before the wet mass is found. Once the wet mass is measured the samples are oven dried at 105 C for 24 hours and then re-weighed and the dry volume measured with the pycnometer. All index properties are calculated from this raw data. The sample residues are then bagged for future additional analysis, primarily carbonate content.

7.4.1 Mass Measurements

The ship is an environment of cyclically changing gravity which affects the weight of the samples. To offset the ship's motion, the sample mass is determined using a technique of differential counterbalancing on twin Scientech 202 electronic balances. The balances are configured with an analog 0-5 volt output. The maximum sample mass determined by the weighing station is approximately 33g.

Select **MASS** from the MAIN MENU panel. The options for this panel include: 1) type of measurement (wet/dry/control); 2) reference mass; 3) beaker id (manually entry or choose from the beaker list); 4) sample id (manual or use list option); 5) new sample (if sample id not already in data base); 6) tare the balance; 7) start measuring; and 8) save.

7.4.2 Volume Measurements

Volumes are found using a Quantachrome Penta-Pycnometer (the Pig) which measures volume using Archimedes' principle of fluid displacement. The displacing fluid is Helium, which assures penetration into crevices and pores approaching one Angstrom (10^{-10} m) in dimension. Purge times of 3 minutes are used to obtain a helium saturated steady-state condition. The Pig measures volumes to an approximate precision of ± 0.02 cc.

Select **VOLUME** from the MAIN MENU panel. The options for this panel include: 1) cells used for the pycnometer run; 2) measurement type (wet/dry/control); 3) beaker number or standard used; 4) **MEASURE** (to begin a Pig run); 5) **RETRIEVE** after the run is completed; and 6) **SAVE** to save the data.

The program does not prompt you when the pycnometer run has been completed. This information is obtained from the display on the pycnometer. Also the retrieval of the data takes up to 30sec per cell, therefore, ***do not*** select save until the display on the pycnometer has returned to the main menu. Selecting save prior to completion of the data retrieval results in the previous volumes being saved. To obtain a hard-copy printout of your cell volumes, press the "PRINT" button on the pycnometer keypad.

7.5 Drying Methods

7.5.1 Mechanical convection oven

Place the sample beakers in the oven at 105 C (± 5 C) for 24 hours after obtaining wet mass. After the material has dried to a constant mass, the beaker is removed from the oven and transferred to the desiccator, where it will come to room temperature, so the operation of the balance will not be affected by convection currents. This method is the method of choice.

7.5.2 Freeze drying

Freeze dry samples are quickly frozen using either liquid nitrogen or propane and are cooled by liquid nitrogen. It is desirable that freezing be done at temperatures less than -130 C to avoid the formation of crystalline ice. Sublimation of the frozen water is then carried out at temperatures between -50 C and -100 C. Times ranging upward from 24 hours are required, with the time increasing with decreasing temperature.

7.6 Data Upload

The index properties data files have to be created prior to uploading to JANUS. Select **OPTIONS** from the MAIN MENU panel and then select **UPLOAD SAMPLES**. A list of samples appear which have not been uploaded to the database. Highlight (use shift key) all the samples from the list and then select **UPLOAD**. A file containing the raw data for the selected samples is written to JANUS_Q:MAD on the local drive. The file designation is year/month/day/number. The number designation begins at 00 and is incremented if more then one sample upload is made on the same day. Note that the beakers, calibration and controls files are written to the MAD folder every time you upload sample data.

Next, log into JANUS using *phys111* for user and password. Select **Physical Properties** from **Applications** menu. Under **Transfer** select **Moisture and Density Files**. The data, beaker, calibration and controls files appear. Highlight the data file and select Upload Selected File. The other files do not need to be uploaded every time.

7.7 Reports

A report of the calculated index properties can be created by selecting the **REPORTS** option on the MAIN MENU panel.

7.8 Equations

To view the equations used to calculate index properties select **REPORTS** and then **VIEW EQUATIONS**.

7.9 Helpful hints

- Covers must be screwed down tightly to achieve metal to metal contact. Any dirt on the cell lip or on the cover, which makes contact with the cell lip, will introduce errors. These surfaces must be kept clean. A light smear of vaseline jelly on the o-ring helps improve the quality of the seal.

- Maximum accuracy is achieved if the cells are calibrated when the control measure is off. This allows for volumetric changes in components with ambient temperature variations.

- Be sure to wipe the outside of the beaker clean!

- To prevent desiccation place a small sheet of parafilm over the beaker if the samples will be sitting out before measuring wet mass or wet volume.

- Accuracy depends upon the applicability of the ideal gas equation of state. Samples which are severely contaminated by volatile materials may produce adequately high vapor pressures to cause deviations from the ideal gas laws. It is recommended that these materials be purged for longer periods of time and that repetitive analysis be performed until constant results are obtained.

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Version: 179
Review by: Patrick Riley
Review on: Nov. 2003
Supervisor sign off: _____