

THE KAZAN FEDERAL UNIVERSITY
INSTITUTE OF PHYSICS

**The Methodology of the Experiment on the Dielectric
Spectrometer Novocontrol BDS Concept 80**

**Part 2. WinDETA 5.81 Software to Perform the Experiments
from 10^{-5} Hz to $3 \cdot 10^9$ Hz on the Dielectric Spectrometer
Novocontrol BDS Concept 80**

Study guide

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The aim of this work is to demonstrate the main capabilities of WinDETA 5.81 Software (Novocontrol Technologies GmbH & Co. KG). The basic menu items that are required to perform the experiments from 10^{-5} Hz to $3 \cdot 10^9$ Hz on the dielectric spectrometer Novocontrol BDS Concept80 are explained. This study guide will be useful for students, graduate students, postgraduate students as well as for the researchers in the field of the dielectric spectroscopy of complex systems.

Contents

| | |
|--|----|
| Introduction | 4 |
| 1. General Description | 6 |
| 2. Running a Measurement | 9 |
| 3. Independent or Free Variables | 11 |
| 4. Defining a Measurement with Continuous Temperature | 14 |
| 5. Displaying and Exporting Data | 19 |
| 6. Evaluation of Data | 20 |
| 7. Data Objects and Files | 25 |
| 8. Measurement | 26 |
| 8.1. Sample Specification..... | 26 |
| 8.2. Start Conditions..... | 30 |
| 8.3. End Conditions..... | 31 |
| 8.4. List Order..... | 33 |
| 8.5. Value Lists..... | 34 |
| 8.6. Measurement Points by Steps..... | 37 |
| 8.7. Averaging..... | 37 |
| 8.8. Start Single Sweep..... | 38 |
| 8.9. Start..... | 38 |
| 8.10. Restart..... | 38 |
| 8.11. Stop..... | 39 |
| 8.12. Calibrate ALPHA, BETA, BDC All..... | 40 |
| 9. Temperature Controller | 41 |
| 9.1. Initialize from WinDETA..... | 41 |
| 9.2. Initialize WinDETA from Controller..... | 41 |
| 9.3. Disconnect WinDETA from Controller..... | 41 |
| 9.4. Safety Limits..... | 42 |
| 9.5. Configuration, Quatro V \geq 4.0..... | 43 |
| 10. Axes | 45 |
| 10.1. Online Axes..... | 45 |
| 10.2. Multi Graphics Axes..... | 46 |
| 10.3. Multi Graphics Curves..... | 47 |
| 10.4. 3D Window..... | 50 |
| References | 52 |

Introduction

Dielectric and impedance spectroscopy measures electrical properties of a material as it is subjected to a periodic electrical field to characterise its molecular kinetics. In order to measure as much as quantitative data Novocontrol's hard and software is covering a broad range both frequency and temperature. Due to the high sensitivity of the measuring system, it is possible to detect transitions that can't be detected with other techniques.

Many key aspects of materials properties can be derived from the frequency and temperature dependent measured complex permittivity, conductivity and impedance spectra. For example, time, strength and shape parameters of molecular relaxations, characterization of structural phase transitions over temperature like glass transition, activation energy, charge mobility and diffusion coefficients.

Time dependent measurements of chemical time variant materials provide rates of blending or cure, crystallization and chemical reactions.

Frequency spectra of technical materials such as metal electrolyte interfaces, batteries, proton conducting membranes for fuel cells and doped semiconductors for microelectronics allows to separate molecular effects within several components or layers within batteries, proton conductivity within fuel cells and characterization of deep charge carrier traps in semiconductor metal interfaces.

WinDETA Software performs fully automatic test sequences, capturing impedance information as a function of a variety of free parameters, e.g., frequency, AC/DC voltage, time. WinDETA is the laboratory standard control and evaluation software for broadband dielectric and impedance spectroscopy. Its uniform user interface supports the most important impedance analyzers and temperature controllers. WinDETA transforms your computer into a powerful system with graphical user interface, allowing users to setup and start fully automatic measurements in minutes.

Features of WinDETA:

- laboratory standard control and evaluation multitasking software for dielectric/impedance spectroscopy and electrochemical impedance spectroscopy;
- uniform user interface for various impedance analyzers and temperature controllers nearly independent of hardware;
- flexible experiment setup: control of frequency, temperature, DC bias and time in any multi dimensional arrangement;
- from the measured impedance, more than 30 different electric quantities are evaluated, including permittivity, conductivity, inductance, and many more;
- graphical online display of measured data, temperature curve and system status;

- integrated plot software to display multiple data sets in a single graph, 3D diagrams, Bode and Cole-Cole plots;
- automatic calibration of hardware devices and sample cells;
- exports/imports data in several flexible user defined ASCII formats;
- optional curve fitting software WinFIT for equivalent circuit modelling, data transformations like Havriliak-Negami and time domain conversion.

Applications

Dielectric/impedance spectroscopy and electrochemical impedance spectroscopy are valuable characterization tools for ceramics, glasses, porous materials, polymers, liquid crystals, semiconductors, ionic conductors, batteries, fuel cells, corrosion analysis, biomedical and biological systems, etc.

Various key aspects of materials properties such as molecular relaxations, conductivity, phase separation, phase transitions, activation energy, glass temperature, rate of blending, purity, ageing, curing and many others are easily investigated.

This study guide assumes that readers are familiar with the basic concepts of impedance measurements and dielectric spectroscopy. You may get further information about these topics from [1-5].

1. General Description

WinDETA is a software package that can control complete systems performing electric and magnetic measurements on components, materials, electrochemical samples or interfaces. The measured data are evaluated, displayed and exported to other programs in various fashions. Measurements up to four dimensions are possible [6].

Measurement

Dielectric, conductivity, magnetic and impedance measurements for materials or components are automatically performed in dependence of the five free variables: *frequency*, *temperature*, *time*, *AC/DC voltage*, *AC/DC current*.

For evaluation of electrochemical samples like ion conductors, electrolytes and metal electrolyte interface layers special devices are supported which can apply controlled *DC voltages* in Potentiostat and *current* in Galvanostat mode while the impedance spectra are measured.

Up to four free variables can be arranged in arbitrary order and therefore define more dimensional measurement up to four dimensions. For each free variable, the user may create a value list with linear, logarithmic or arbitrary spacing between data points which will be adjusted in a later measurement.

In addition to fixed setpoints, for the temperature and time list an arbitrary series of temperature ramps or time intervals for continuous measurements can be defined. In order to bring the sample in a defined state before the start and after the end of a measurement, the free variables can be set to user defined start- and end values.

During the measurement, the data are displayed **on-line** as a diagram, and as a numeric table in a **log window**. In addition, all actions of WinDETA and the state of the devices are written into a **message window**. Finally, the actual system state is graphically displayed in a **status window** and **temperature log graphics window**.

Full **automatical control of measurements** is supported. This includes control of the devices by software, calibration of the devices, calibration of the measurement lines and the sample cell.

Data evaluation

The complex dielectric function, specific conductivity and magnetic permeability are evaluated for each data point in dependence of the free variables. The sample impedance Z^* can be evaluated as a complex impedance, admittance, capacity or inductivity. Both parallel and serial circuits are supported.

In addition to the impedance variables the measured time, sample temperature, AC/DC voltages and currents are recorded and displayed in the graphics windows.

Nonlinear Measurements and Higher Harmonics

In addition to the linear terms related to the voltage and current base waves described above, for characterisation of nonlinear sample properties higher harmonic components of the sample voltage and current can be measured, evaluated and displayed for all variables and quantities and in the same way as for the corresponding linear terms.

Data formats

The complete data set of one measurement, which may contain an arbitrary number of data points and up to four free variables in four dimensions is saved as one file in an internal format. The data are exported to other programs as **ASCII tables** in several user defined formats or through the Windows clipboard. Diagrams together with their data are saved as file objects.

Graphics

All evaluated data are **displayed in 2D or 3D diagrams**, which are printed on every printer in highest resolution or exported to other Windows applications. The size of the diagrams is varied by simply changing its window size. Moreover, the user may mark arbitrary areas in the diagrams with the mouse, which afterwards are zoomed to the full window size. All diagrams support manual or auto scaling, linear or logarithmic scaling and axes inversion. 2D diagrams have two different y-axes. An arbitrary number of data curves from one or more measurements or from external ASCII files can be added by the user to each y-axis. The x-axis displays either one of the four free variables or one of the dependent variables. The last option enables for example Cole-Cole Plots (ϵ' as a function of ϵ''). 2D diagrams are saved to disk together with their data curves as own data files.

Up to twelve 3D diagrams may be displayed simultaneously in different windows. For each diagram the user can select two free variables, one of the not free variables, and the orientation of the diagram in arbitrary directions. Data points, which could not be measured are either omitted or interpolated.

Handling

For each WinDETA function, context sensitive on-line help is available, containing the system documentation and hints to solve special problems by using cross references. The system configuration, including the number and position of windows, the selections in dialogs and lists can be saved in **'pre-set' files**. WinDETA supports two kinds of multitasking. First, WinDETA may run together with other Windows applications without blocking the system. Second, WinDETA has internal several tasks, working independent from each other simultaneously. For example, the user is allowed to display data and change configurations while the measurement is running and the temperature control is active.

WinDETA supports three states of measurements [6]:

1. Measurement of the complex impedance Z of an electric sample. Z can be evaluated as a complex impedance, admittance, capacity or inductivity. Both parallel and serial circuits are supported. In addition the absolute values for each of the 4 circuits are available.

2. Measurement of the intrinsic electric properties of a material which can be evaluated either in form of the material dielectric function $\varepsilon = \varepsilon' - i\varepsilon''$ or its specific conductivity $\sigma = \sigma' - i\sigma''$. For this kind of measurement, a sample cell is required in which the material under test is arranged and contacted in a favourable manner. The electric properties are calculated from the sample cell impedance and its geometry. A set of suitable dielectric sample cells is available from NOVOCONTROL.

3. Measurement of the intrinsic magnetic properties of a material which is evaluated as the magnetic permeability $\mu = \mu' - i\mu''$. For this kind of measurement, a magnetic sample cell is required in which the material under test is arranged. The magnetic properties are calculated from the sample cell impedance and its geometry. Magnetic measurements are supported for the RF impedance analyzers Hewlett Packard 4291 and Hewlett Packard 4991 in combination with the magnetic sample cells HP16454S or L.

The impedance, electrical material or electrochemical properties are in general not constant, but depend on physical and chemical conditions. The global purpose of WinDETA is to measure, evaluate and display this dependency. The sample impedance generally varies with frequency f and temperature T of measurement. In addition, WinDETA supports time, AC/DC voltage and AC/DC current dependent measurements which are especially important for electrochemical characterizations. As the quantities in 1. and 2. are functions of f , T , t , these quantities (impedance, electric response) are called **dependent or not free variables**. The variables f , T and t do not depend on others and therefore they are called **independent or free variables**.

Corresponding to the free variables, WinDETA has four **value lists**, in which you may enter the set of values which you intend to measure later on. You may select combination of the lists to be used and the execution order of the value lists. WinDETA treats each value list as a separate dimension. Therefore measurements up to the fourth dimension are possible.

In addition to the fixed setpoints in the value lists, for the temperature and time list, an arbitrary series of temperature ramps or time intervals for continuous measurements can be defined. Quantities or states like e.g. pressure which can not be automatically adjusted by WinDETA can be defined in the **List of Free User Defined Values**.

The measured data can be graphically displayed in a 2D/3D diagrams or exported as ASCII files on disk. The contents of all graphics windows may be send to the printer or copied to the clip board with high resolution.

In addition to the linear measurements described above, WinDETA supports measurement of higher harmonic contributions in the sample response current and voltage. Nonlinear samples create a response current containing in addition to the component at the applied voltage base frequency components at multiples of the base frequency which are called higher harmonics. WinDETA supports higher harmonic characterisation for higher harmonic components in amplitude and phase to the base wave. In addition to the complex measured sample voltage and current components, harmonic components can be expressed in terms of dependent or not free variables.

For electrochemical analysis, impedance measurement systems with Potentiostat and Galvanostat control are supported.

2. Running a Measurement

Before starting a measurement:

- check whether all device connections are OK, a proper sample is connected to the measurement system and all required devices are switched on;
- make sure that all devices are properly calibrated;
- adjust the analyzer specific settings such as **Calibration Options, Coupling Modes, 2, 3, 4 Wire mode, Output Resistance, Measurement Ranges, Limits, Potentiostat, Galvanostat** and **Time Constant** from the corresponding **Configuration** dialog of the **Analyzers, Options** menu;
- internal connect the CE and WE terminals ALPHA-A Analyzer + Electrochemical Interface POT/GAL 15V10A and 30V2A as described in Configuration (ALPHA + POT/GAL).

You can check all configurations for the measurement to be started with **Show Definitions** from the **Measurement** menu [6].

A measurement can be started in three ways:

1. Select **Start** from the **Measurement** menu to start a complete measurement using all **value lists** defined in the **order list, start conditions** and **end conditions**.
2. Select **Start Single Sweep** from the **Measurement** menu to start a one dimensional measurement which uses only the first value list (with the lowest order) defined in the order list. The start and end conditions are ignored.

3. Select **Restart** from the **Measurement** menu to restart a measurement which was aborted before all values could be measured. The values already measured are not cleared and the measurement tasks restarts at the position of the break. A measurement is aborted if you select **Stop** from the **Measurement** menu or if an error occurs.

If the measurement is running, you have the following options to control it:

1. Select **Stop** from the **Measurement** menu to stop the measurement task and reset all devices. A stopped measurement can be re-initialized with **Restart (Measurement menu)**.

2. Select **Pause** from the **Measurement** menu to pause the measurement of impedance points. All devices and initializations remain unchanged, the controllers of the free variables and the measurement task remain active.

3. Select **Resume** from the **Measurement** menu to re-activate a paused measurement.

4. If **Skip Temperature Stabilization** from the **Measurement** menu is selected, WinDETA starts the next impedance measurement block regardless of the process value of the actual temperature.

5. If **Skip to the End of inner List** from the **Measurement** menu is selected, WinDETA marks all the impedance points (**dependent variables**) from the actual point to the last point of the value list with lowest order as **bad points** and proceeds with the next point of the free variables. Note: The lowest value list (**free variables**) remains unchanged.

After the measurement is started, WinDETA performs it fully automatically. In general the following steps are executed [6]:

- The **result file** is initialized for the new measurement.
- All **devices** used in the measurement are initialized.
- The **value lists** of the free variables are initialized.
- The x-axis of the **online window** is set to the variable corresponding to the list with lowest order.
- If a temperature list or a **temperature start condition** (not at single sweep) is defined, the active temperature controller is initialized.
- The measurement task is started.
- The controllers are set to the values of the **start conditions**.
- WinDETA waits until all start conditions match.
- LOOP: The setpoints of the controllers are set to the next combination of the free variables.
- WinDETA waits until the process values of all controllers match their setpoints.

- An impedance measurement is started.
- WinDETA waits until the impedance measurement is complete.
- If the measurement is valid, the impedance and the actual time are saved on disk and in the internal measurement object. If temperature control is active, the actual temperature is saved also. The independent variables are calculated and send to the **online** and **log window**.
- If not all points of the value lists have been measured, WinDETA proceeds with LOOP.
- The controllers are set to the values of the **end conditions**.
- WinDETA waits until all end conditions match.
- All devices are reseted, the generator of the impedance measurement system is switched off. If temperature control has been active, and **Heating off (End Conditions, Measurement** menu) is enabled, it is switched off.
- The measurement task terminates.

Each activity of the measurement task is send to the **message window**. The actual status of the system is displayed in the **status window**.

If a temperature log file is enabled, the actual temperature setpoints and process values are send to the temperature log window.

The measurement task works as one of WinDETA's internal multitasking threads, being executed quasi parallel to each other. For internal reasons, you can not load a result file or change some configurations in the **Measurement** menu while the measurement task is active. All other features of WinDETA may be used during the measurement. For example, you may display the data measured so far in the **multi graphics** or **3D window** or change the configuration of the temperature control system.

3. Independent or Free Variables

The main purpose of WinDETA is to measure the electrical response of a component or a material (described by the **dependent or not free variables**) as a function of the independent or free variables [6]. WinDETA supports the next free variables: *frequency, temperature, time, AC/DC voltage, AC/DC current, harmonics and free user defined values*.

For each free variable a **value list** exists in which you may create a set of values, which WinDETA will measure later. Each of the value lists defines a discrete one dimensional measurement space. In addition to fixed setpoints, for the temperature and time list, an **arbitrary series of temperature ramps or time intervals for continuous measurements** can be defined. Dependencies on such quantities as pressure which can not be automatically controlled or measured by WinDETA can be defined by the list of **Free User Defined Values**.

In order to measure more than one free variable, WinDETA allows to combine $n \leq 4$ value lists in one measurement, defining a n-dimensional orthogonal measurement space. For details please see **Defining a Measurement Using Value Lists**.

The free variables have the following meaning:

Frequency of the electric AC field being applied to the sample (component or material) to measure its electric response.

Temperature is applied to the sample during a measurement. The temperature is controlled by a separate temperature control system and a sample cell. In isothermal mode, the temperature controller needs a certain time to settle to a new setpoint. In this time no measurements can be done. You may select the stabilization conditions, which must be met before the next measurement point is started from the entry **Stabilisation Conditions** of the menu **Temp. Controller**.

Time can be used to start repetitive measurements at specified time points to measure time dependent effects like ageing or curing. Each value in the time list denotes a time point to which the start of the next measurement block is delayed. A measurement block includes all measurement points between two succeeding values in the time list (defined by value lists with lower order, refer to **Defining a Measurement Using Value Lists** for details). For example, a measurement block may be a single impedance point, a frequency, temperature AC/DC voltage sweep or any more dimensional combination of sweeps. All time points refer as offsets to a time zero point which is set if the time list is initialized. This happens at the start of a complete measurement (after the measurement has passed the **start conditions**) or if the time list is reset to its first point if executed more than once. If WinDETA could not start a measurement block at an actual time point, the values of all **dependent variables** of this measurement block are marked as **bad points** and not measured. In this case, WinDETA writes the message '**Time over for actual data point**' in the **message window** and proceeds with the next value in the time list. This may happen, if the time for the actual measurement block of a proceeding time point exceeds the intended time point, or if the temperature is not stable at the actual time point, or if other WINDOWS programs are blocking the system at the actual time point.

Remark: In order to have an unique concept of data definition and measurement control, WinDETA treats the time list in the same way as the other value lists. Nevertheless, there are important differences to the other free variables. First, time can not be hold constant during the measurement of the sample's electrical response. Therefore, it only makes sense to attach time points to single measurement blocks if the duration between succeeding time points is long compared to the duration of a measurement block. Second, if you intend to measure the

time dependence of a sample, you should always **place the time list in the highest order**, so that it is only executed once during a complete measurement. Otherwise, if the time list is executed multiple times (as the time list is initialized at every new execution) each value in the time list will correspond to multiple true time points, which are shifted by the duration between two succeeding time lists.

DC Volt is an additional constant DC voltage being superimposed to the AC voltage of the impedance measurement system. As the DC voltage generally is much higher than the AC voltage, it adjusts the total electric field strength in the sample. This may be used to detect non linear effects in the sample's electric response, or to set the electrochemical or molecular structure of the sample to a defined state. For example, the potential of electrochemical cells can be adjusted or the molecular dipoles of a liquid crystal may be arranged by an additional DC voltage in the direction of the field. **As the DC voltage often is high, high currents may be induced in the sample which can not be handled by all impedance measurement devices.** This especially may happen, if the DC resistance of a sample is comparable the AC impedance at the actual measurement frequency. Therefore, using DC voltage is not allowed for all samples. In addition, the setpoint of the DC voltage does not always match the voltage applied to the sample, because the output resistance of the impedance analyser's generator is low but not zero [7, 8].

AC Volt is the voltage of the impedance analysis system frequency generator output, which is applied to the sample. The setpoint of the AC voltage does not always match the voltage applied to the sample, because the output resistance of the impedance analyser's generator is low but not zero. In addition, internal generator current limits may be reached, which may reduce the voltage at the sample.

DC Current is an additional constant DC current being superimposed to the AC current of the impedance measurement system. It is only available for the ALPHA-A Analyzer + Electrochemical Interface POT/GAL 15V10A and 30V2A in Galvanostat mode.

AC Current is the current of the impedance analysis system frequency generator output, which is applied to the sample. It is only available for the ALPHA-A Analyzer + Electrochemical Interface POT/GAL 15V10A and 30V2A in Galvanostat mode.

Harmonics is used to define measurements with higher harmonics components. The numbers in the **Harmonics** list specify the harmonic components which will be measured in addition to the base in a following measurement. Number 0 refers to the base wave, 1 to the first harmonic, 2 to the second,

The list of **Free User Defined Values** is used to measure the dependency of any quantity which can not be automatically controlled or measured by WinDETA, but can be

adjusted manually by the user instead like the cell pressure. For the manual adjustable quantity a name, unit and values can be defined as for the other value lists.

4. Defining a Measurement with Continuous Temperature

Ramps or Time Sweeps

The concept of **Defining a Measurement by Value Lists** in several dimensions is very flexible, but always works with fixed setpoints. For measurements using a **temperature list or time list** this means, that a measurement will only be started, after the temperature has become stable to the actual setpoint of the temperature list or time has reached the actual setpoint of the time list. After the measurement is done, the next setpoint and stabilization cycle will be done and so on.

For measurements with temperature dependence, this procedure results in highest measurement accuracy, as the temperature remains constant during the measurement.

Nevertheless, it is sometimes useful to do continuously measurements while the temperature is changed with a constant rate (temperature ramp).

This will make sense, if

- due to physical reasons for the sample material a constant temperature change is required or
- if a large number of temperatures in a small temperature interval have to be measured (e.g. at a phase transition). In this case the stabilization of each temperature will consume a lot of time so that it is more convenient to do measurements continuously while the temperature slowly changes.

For time dependent measurements, one is often interested to measure continuously as function of time without specifying measurement times for the time list in advance.

In addition, it may be useful to perform temperature ramps or hold times without measuring continuously during the ramp or hold time. Such types of ramps may be included between two fixed temperature points with stabilization to apply a defined temperature time profile for the temperature change from one fixed temperature point to the other.

If the temperature list is defined with highest order (lowest entry in the **order list**), **Temperature Ramp** or **Hold Time** are supported by WinDETA in addition to the standard **fixed temperature setpoints** as follows [6].

A. Temperature Ramp from a Start Temperature to an End Temperature with a Defined Gradient

The ramp start temperature is defined by the actual sample temperature at the time when the ramp will be started. This temperature is usually defined by the temperature list entry which precedes the ramp. In order to create a temperature ramp entry:

- If you want to change or insert a ramp entry at a specific position within the **temperature list**, select its position within the temperature list.

- In the **temperature list**, enter the ramp end temperature in the **Temperature** field.

- Select **Use Temp. Ramp, Gradient** and enter the intended temperature gradient in the field below.

- If you want to perform measurement blocks (like a frequency sweep) continuously while the temperature ramp is active, select **Measure continuously in Temp. Ramps**. Otherwise no measurements will be done during the ramp. The term measurement block is described below under Remarks.

- If you want to define a time interval between continuously measured measurement blocks, enter it in the **Delay Time** field. WinDETA will delay the start of each measurement block within a continuous measurement by this time. This option may be used to avoid too many data to be measured for ramps running a long time.

- Select **Add** to add the ramp entry at the end of the temperature list, or select **Insert** or **Change** to perform these operations.

- If WinDETA reaches a temperature ramp entry, it will start a temperature ramp from the actual temperature to the specified end temperature with the specified gradient.

B. Hold Time at Constant Temperature within the Temperature List

In order to create a hold time entry:

- If you want to change or insert a ramp entry at a specific position within the **temperature list**, select its position within the temperature list.

- In the **temperature list**, enter the intended constant temperature in the **Temperature** field.

- Select **Use Temp. Ramp, Hold Time** and enter the intended value in the field below.

- If you want to perform measurement blocks (like a frequency sweep) continuously while the hold time is active, select **Measure continuously in Temp. Ramps**. Otherwise no measurements will be done during the hold time. The term measurement block is described below under Remarks.

- If you want to define a time interval between continuously measured measurement blocks, enter it in the **Delay Time** field. WinDETA will delay the start of each measurement

block within a continuous measurement by this time. This option may be used to avoid too many data to be measured for long hold times.

- Select **Add** to add the hold time entry at the end of the temperature list, or select **Insert** or **Change** to perform these operations.

- If WinDETA reaches a hold time entry, it will set the temperature setpoint to the constant temperature and wait for the hold time.

C. Time Intervals for Continuous Measurements within the Time List

If the time list is defined with highest order (lowest entry in the **order list**), time dependent continuous measurements are supported by WinDETA. In order to create a continuous time measurement entry:

- If you want to change or insert a time list entry at a specific position within the **time list**, select its position within the time list.

- Select **Continuous Interval**.

- If you want to define a delay time before continuously measured measurement blocks, enter it in the **Delay Time** field. WinDETA will delay the start of each measurement block within the continuous measurement by this time. This option may be used to avoid too many data to be measured for long time intervals.

- Select **Add** to add the continuous time entry at the end of the time list, or select **Insert** or **Change** to perform these operations.

- If WinDETA reaches a continuous time interval in the time list, it will start continuously to execute measurement blocks until the interval end time is reached. The term measurement block is described below under Remarks.

Remarks

- If continuous measurements are defined within a temperature ramp or hold time interval, no stabilization condition will be applied before measurement blocks are started. Instead measurement blocks will run continuously asynchronous and independent from the actual temperature. This is in contrast to fix temperature entries (with **Use Temp. Ramp** disabled) for which a measurement block is only started after stabilization synchronously with the temperature.

- A measurement block includes all measurement points defined by value lists with lower order above the temperature list. For example, a measurement block may be a single impedance point, a frequency, AC/DC voltage sweep or any higher dimensional combination of sweeps. WinDETA will automatically attach the actual temperature or time at the beginning of the measurement to the block. These values will be displayed later in the diagrams or ASCII files.

- Continuous intervals (temperature ramp, hold time interval) may be mixed with fixed time or temperature setpoints in the temperature or time list. In this case fixed setpoints (time or temperature) are synchronized with measurement blocks and continuous intervals are not synchronized with measurement blocks. That means:

1. WinDETA will not start a new measurement block triggered by a fixed setpoint until any preceding measurement block was completed.

2. WinDETA will complete a measurement block triggered by a fixed setpoint, before it proceeds with any next item in the temperature or time list.

3. WinDETA will execute series of continuous intervals according to their defined time or temperature definitions, regardless whether the actual measurement block is terminated at the beginning of a new continuous interval. For example, if two succeeding temperature ramps are defined, WinDETA will not delay the change in ramp speed for the second ramp to the end of the actual measurement block. Instead the ramp speed may change within the block. This concept allows to program a defined temperature-time profile independent from the definitions for measurement blocks above the time list or temperature list. If synchronization between succeeding continuous intervals is required, an additional fixed setpoint between the two continuous intervals should be placed.

Examples: Temperature list with fixed setpoints, continuous temperature ramps and continuous time intervals at fixed temperature. **Measure continuously in Temp. Ramps on.**

| Contents of Temperature List | Comment |
|-------------------------------------|---|
| 20 | Fixed setpoint = 20°C, wait for stabilization, perform measurement block |
| Ramp to 50 with 2K/min | Set ramp speed 2K/min, set ramp end temperature 50°C, start repeating continuously measurement blocks, if setpoint has reached 50°C, stop ramp |
| Ramp to 30 with 1K/min | Do not wait for termination of measurement block in progress, set ramp speed 1K/min, set ramp end temperature 30°C, continue repeating continuously measurement blocks, if setpoint has reached 30°C, stop ramp |
| Stay at 30 for 600 s | Do not wait for termination of measurement block in progress, setpoint = 30°C, continue repeated measurement blocks, after 600 s proceed with next item in list |
| Stay at 100 for 300 s, delay 20 s | Do not wait for termination of measurement block in progress, set temperature setpoint to 100°C, do not wait for stabilization, continue repeated measurement blocks with 20 s delay between the blocks, after 300 s proceed with next item in list |
| 300 | Wait until last measurement has terminated, fixed setpoint = 300°C, wait for stabilization, perform measurement block |
| Ramp to 20 with 3K/min | Set ramp speed 3K/min, set ramp end temperature 20°C, start repeating continuously measurement blocks, if setpoint has reached 20°C, stop ramp at 20°C |

Examples for possible measurement blocks in the **order list** above the temperature list.

Note: If continuous intervals are defined, the temperature list has to be placed in the end of the **order list**.

| Contents of Order list | Comment |
|-------------------------------------|---|
| Temperature | Measures only in dependence of temperature, frequency of measurement must be defined by the Start Conditions , measurement block = one temperature point |
| Frequency Temperature | Measures frequency sweeps in dependence of temperature, frequencies must be defined by the frequency list , measurement block = all frequencies in the frequency list |
| DC Volt Frequency Temperature | Measures DC volt sweeps in dependence of frequency. This two dimensional measurement is repeated as a function of temperature. Dc volt values and frequencies must be defined by the DC volt and frequency lists , measurement block = all points defined by the two dimensional DC volt and frequency sweeps. |

Time list example with fixed setpoints and continuous time intervals at fixed temperature.

Note: All time specifications for fixed time points and continuous intervals refer to the start time of measurement.

| Contents of Time List | Comment |
|--------------------------------------|--|
| 30 | Fixed time point = 30 s, wait 30 s after start of measurement, perform measurement block |
| Continuous to 200.0 | Start repeating continuously measurement blocks until 200 s are reached. |
| Continuous to 400.0, delay 10.0 s | Do not wait for termination of measurement block in progress, continue repeating continuously measurement blocks, delay next blocks by 10 s, proceed with next item if time from start of measurement has reached 400 s. |
| 600 | Wait until measurement block in progress has terminated, wait until 600 s from start of measurement has been reached, perform measurement block |

5. Displaying and Exporting Data

The data of the measurement currently in progress is displayed as a 2D graphics in the **Online Window** and as a table of numbers in the **Log Window** [6]. These windows can not display data from files.

In addition, data from WinDETA's measurement object are displayed as 2D multi curve diagrams in the **Multi Graphics Windows** and as 3D surface diagrams in the **3D Windows**.

Select **Load Measurement Result**, to load a measurement from disk in the WinDETA's measurement object.

Note: If a new measurement is started, WinDETA loads the measurement object automatically with the measurement currently in process. The filename of the measurement contained in the measurement object is displayed in the WinDETA main window title bar.

To display data from WinDETA's measurement object in the **Multi Graphics Windows**:

Select **Multi Graphics** from the **Window** menu to open the window.

Select **Multi Graphics Axes** from the **Axes** menu to configure the diagram axes and other diagram options.

Select **Multi Graphics Curves** from the **Axes** menu to add or delete curves from WinDETA's measurement object to the diagram.

Note: The multi graphics window is not cleared, if a new file is loaded in the measurement object. Therefore, curves of several measurements may be displayed together in the multi graphics window.

In order to display data from WinDETA's measurement object as 3D diagrams: Select **New 3D** from the **Window** menu to open a new 3D window.

Select **Configuration (3D)** from the local menu of the 3D window to select data curves and diagram options. The diagram will be displayed after selecting the dialog OK button.

To export data from WinDETA's measurement object as ASCII files: Select **Save Measurement As ASCII** from the **File** menu.

Note: All menu from above are available from **Display or Export Result** (File menu), too.

6. Evaluation of Data

In a dielectric or impedance measurement a voltage U_0 with a fixed frequency $\omega/2\pi$ is applied to a sample cell which contains the sample material under test. U_0 causes a current I_0 at the same frequency in the sample cell. In addition, there will be generally a phase shift between current and voltage (see Fig. 1) described by the phase angle φ [6]:

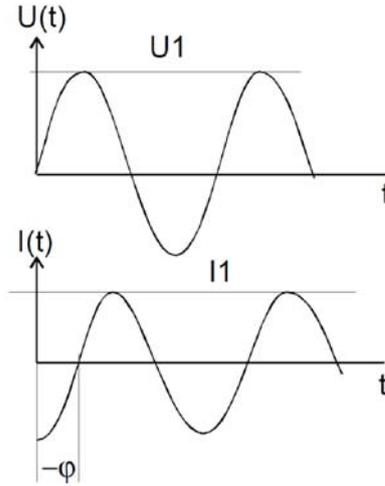


Figure 1. Amplitude and phase relation between voltage and current of a sample capacitor for dielectric measurements.

The ratio of U_1 and I_1 and the phase angle φ are determined by the electromagnetic properties of the sample material and the sample geometry. For easy calculation and representation of the formulas it is convenient to use complex notation.

$$u(t) = U_1 \cos(\omega t) = \text{Re}(U^* \exp(i\omega t)),$$

$$i(t) = I_1 \cos(\omega t + \varphi) = \text{Re}(I^* \exp(i\omega t)),$$

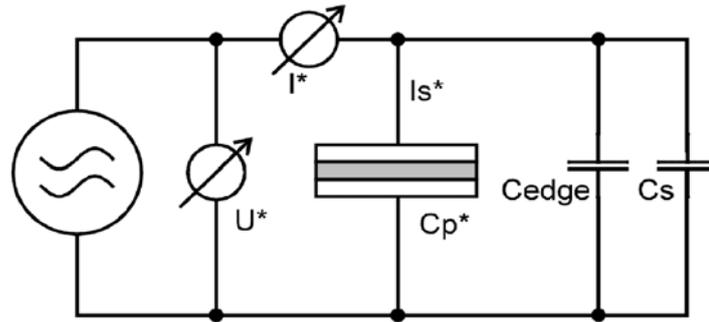
with $U^* = U' + i U''$, $U' = U_1$, $U'' = 0$ and $I^* = I' + i I''$, $I' = I_1 \cos(\varphi)$, $I'' = I_1 \sin(\varphi)$.

For a sample with linear electromagnetic response, the sample impedance $Z^* = U^* / I^*$ depends on the material properties and the sample geometry but not on the voltage amplitude U^* . This information can be expressed as Z^* in various other equivalent representations (**dependent variables**) which are described below in detail.

Note: The values of U^* and I^* directly refer to the voltages and currents at the sample cell. Possible device calibration procedures which consider errors due to non ideal sample cells are already included at this point.

WinDETA supports the following three types of sample cells [6]:

1. Dielectric sample cell with a round plate capacitor filled with the sample material:



C_p^* denotes the complex parallel capacity of the filled sample capacitor without the influence of spacers.

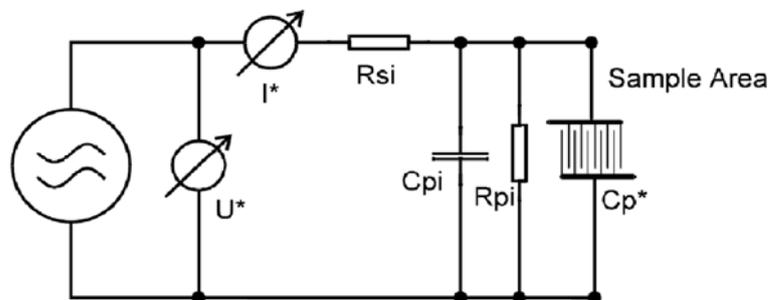
C_s denotes the additional capacity caused by the external sample cell stray capacity and the capacity of optional spacers in the sample capacitor. The sample cell stray capacity is due to the capacity of the electrode connection wires which is about 1 pF. For the liquid sample cell BDS 1308, an additional stray capacity of about 5.5 pF from the Teflon isolation and the upper and lower mounting plates has to be taken into account.

C_{edge} is an additional capacity due to electrical stray fields from the borders of the sample capacitor. C_{edge} will be only considered, if **Use Edge Compensation** in the **Sample Specification** dialog of the **Measurement** menu is active.

The sample capacity is related to the measured voltage and current by

$$C_p^* = -i \frac{I^*}{\omega U^*} - C_{edge} - C_s.$$

2. Dielectric sample cell with an interdigit electrode capacitor covered by the sample material:



The complex capacity C_p^* of the sample area is corrected by the three elements: Pre-Resistance R_{si} corresponding to the resistance of the evaporated electrode material, Parallel

Resistance R_{pi} corresponding to the resistance of the object carrier material, Parallel Capacity C_{pi} corresponding to the parallel capacity due to the object carrier material.

Both for the round plate capacitor and for the interdigit electrode, the sample impedance Z_S^* and the admittance Y_P^* are calculated from complex parallel sample capacity C_p^* by

$$Z_S^* = \frac{1}{Y_P^*} = -\frac{i}{\omega C_p^*}.$$

The complex dielectric constant is ε^* related to the complex sample capacity by

$$\varepsilon^* = \varepsilon' - i\varepsilon'' = \frac{C_P^*}{C_0}$$

with the empty cell capacity C_0 . For a sample cell consisting of round parallel plates with diameter D and spacing d , C_0 is calculated by

$$C_0 = \varepsilon_0 \frac{\pi \left(\frac{D}{2}\right)^2 - A_{spacer}}{d}$$

where A_{spacer} designates the average area inside the sample capacitor occupied by the spacer material. For the interdigit electrode, the value of C_0 has to be measured before the sample material is attached to the electrode.

The complex dielectric modulus is defined as

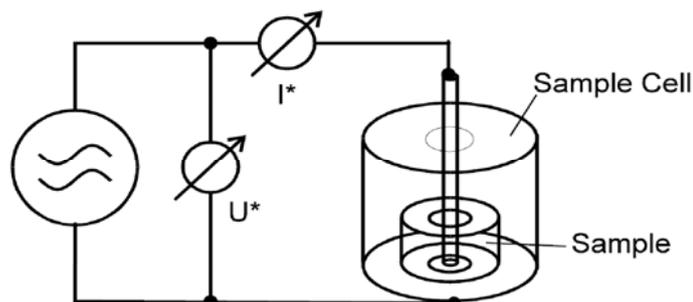
$$M^* = M' + iM'' = 1 / \varepsilon^*.$$

The specific conductivity σ^* is related to the dielectric constant by

$$\sigma^* = \sigma' - i\sigma'' = i2\pi f\varepsilon_0 (\varepsilon^* - 1).$$

The specific resistance of the material is calculated by $\rho = 1 / \sigma'$.

3. Magnetic sample cell partly filled by the sample material:



The sample inductivity L_s^* is related to the measured voltage and current by

$$\frac{U^*}{i\omega I^*} = L_s^* - L_{s0} + L_0$$

where L_0 is the inductivity of the whole empty sample cell volume and L_{s0} is the inductivity of the empty sample cell volume occupied by the sample material. L_0 is determined by a calibration measurement of the empty sample cell.

From this, the sample impedance Z_s^* is calculated by

$$Z_s^* = \frac{1}{Y_p^*} = i\omega L_s^*.$$

The sample permeability is related to the inductivity by

$$\mu^* = \mu' - i\mu'' = \frac{L_s^*}{L_{s0}}.$$

For a magnetic sample with the geometry of an open ring with high h , inner diameter b and outer diameter c

$$L_{s0} = \frac{\mu}{2\pi} h \ln\left(\frac{c}{b}\right).$$

Instead of the complex sample impedance Z_s^* , the electrical response of the sample may be represented by the series or parallel resistance, conductance, capacity or inductivity as shown in Figure 2.

For the serial parts, the components are connected to the sample impedance by

$$Z_s^* = Z_s' + iZ_s'' = \frac{1}{Y_s'} + \frac{1}{Y_s''} = \frac{1}{i\omega C_s'} + i\frac{1}{\omega C_s''} = i\omega L_s' + i\omega L_s''.$$

For the parallel parts, the components are connected to the sample admittance by

$$Y_p^* = \frac{1}{Z_p'} + i\frac{1}{Z_p''} = Y_p' + iY_p'' = i\omega C_p' + i\omega C_p'' = \frac{1}{i\omega L_p'} + i\frac{1}{\omega L_p''}.$$

The absolute values are calculated for Z_s , L_s , Y_p , and C_p .

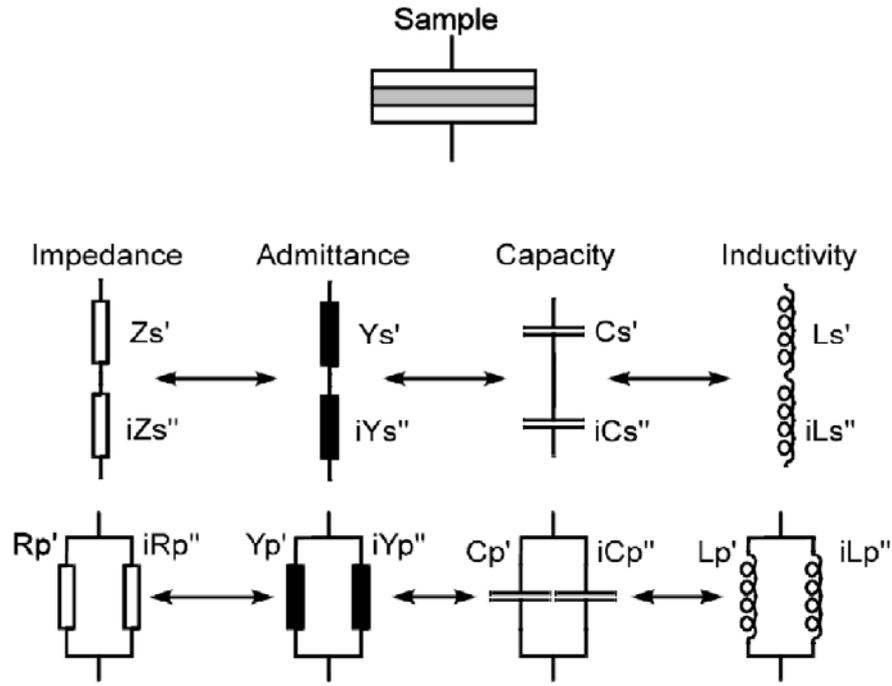


Figure 2. The electrical response of the sample represented by the series or parallel resistance, conductance, capacity or inductivity.

The loss factor $\tan(\delta)$ is calculated by

$$\tan(\delta) = \frac{\varepsilon''}{\varepsilon'} = \frac{\mu''}{\mu'} = \frac{Z_s'}{Z_s''}$$

From the loss factor, the loss angle δ is calculated by

$$\delta = \frac{180^\circ}{\pi} \arctan\left(\frac{\varepsilon''}{\varepsilon'}\right)$$

In contrast to the loss angle δ , the phase angle

$$\varphi = \frac{180^\circ}{\pi} \arctan\left(\frac{Z_s''}{Z_s'}\right)$$

specifies the angle in the complex plane between the applied voltage U^* and the sample cell current I_s^* (without the stray capacity and spacer contribution). φ is shifted by 90° relative to δ .

7. Data Objects and Files

The following diagram shows the most important data objects, data flows (see Fig. 3) and files used by WinDETA and from which menu or dialog they are activated [6].

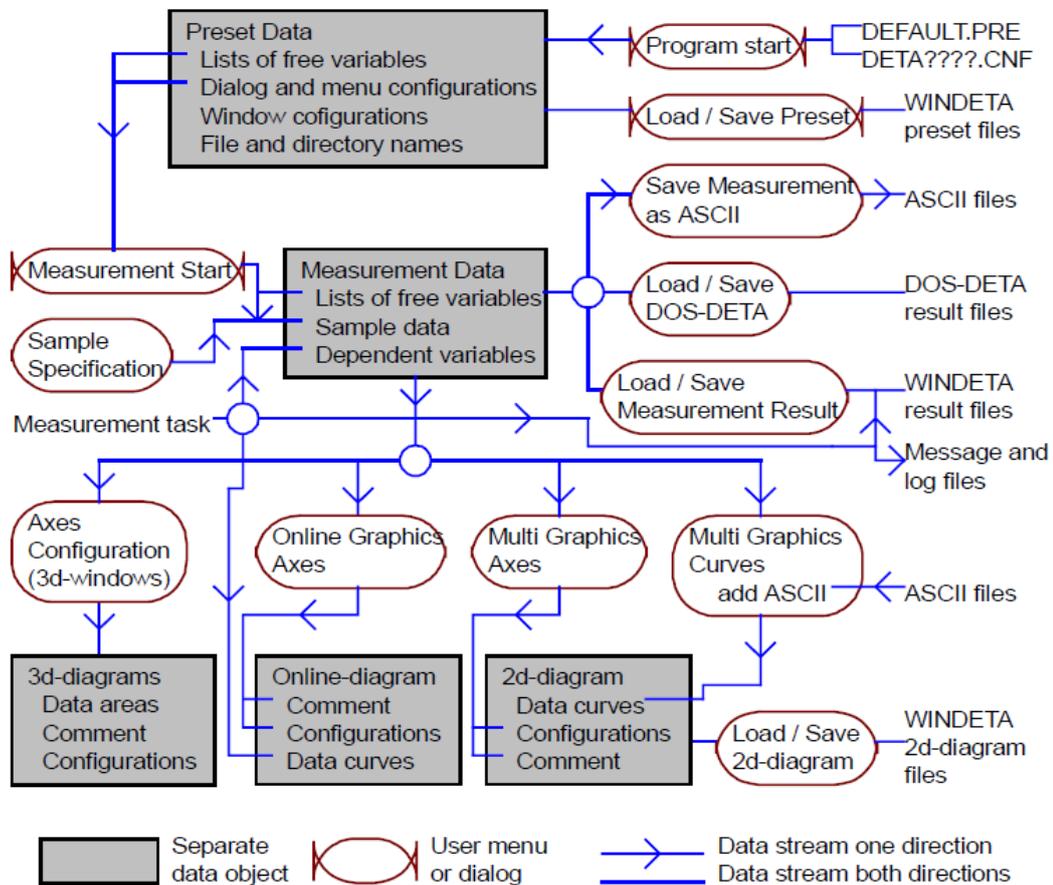


Figure 3. Data flow of WinDETA.

Note that each of the four data objects (Preset data, Measurement data, 2D diagram, 3D diagram) holds an own copy of it's data. For example, existing data in a 2D/3D diagrams don't change if another result file is loaded into the measurement object, allowing you to display more than one measurement simultaneously.

The most important files, WinDETA deals with are [6]:

| Class | Name | Comment |
|---------------------|-------|--|
| WinDETA result file | *.EPS | Saves the complete information of a measurement up to the fourth dimension. |
| Preset file | *.PRE | Saves the preset values of: -All menus, lists and dialogs, with exception of the temperature controller's safety limits . -The number, order and position of open windows, with exception of the 3D windows . -All file and directory names. The file DEFAULT.PRE is loaded at the start of WinDETA. |

| | | |
|----------------------|---------------------|---|
| ASCII file | *.TXT | Allows you to export ASCII-data to- or import ASCII-data from other programs in various formats. |
| 2D diagram file | *.2d | Saves a complete 2D diagram including it's data curves. |
| Message file | *.MSG | Saves all messages being displayed in the messages window . |
| Log file | *.LOG | Saves all text written to the log window . |
| Configuration file | DETA???.CNF | Saves parameters of WinDETA, which can't be altered by menus, like device addresses. Is automatically loaded at program start. |
| DOS-DETA result file | *.126, *.428, *.419 | Saves a single frequency sweep in the DOS-DETA format. A 2D temperature-frequency measurement can be handled as a collection of files in a separate directory. |

8. Measurement

8.1. Sample Specification (Measurement Menu)

Sample Specification

Sample Comment
Glycerol

Dielectric Sample Cell

Sample Diameter [mm] 2.0000e+01

Sample Thickness [mm] 5.0000e-02

Cell Stray + Spacer Capacity [pF] 0.0000e+00

Spacer Area [mm²] 0.0000e+00

Use Edge Compensation

Electrode Thickness [mm] 2.00e+00

C0 [pF] 5.563e+01 Cedge/C0=1.7%

Use Dielectric Sample Cell OK
 Use Dielectric Interdigit Electrode Cancel
 Use Magnetic Sample Cell Help

novoccontrol

Figure 4. Specifies the sample geometry and comment.

In the **Sample Comment** field (see Fig. 4), an arbitrary string may be entered which will be displayed as a header in the graphics windows and the ASCII files.

You can select between three types of sample geometries:

- Select **Dielectric Sample Cell** for the round plate capacitor with the sample material between the plates.

- Select **Dielectric Interdigit Electrode** if you are using this kind of sample geometry.

- Select **Magnetic Sample Cell** for a HP16454 magnetic test fixture (this option is only available for the Hewlett Packard 4291 and 4991).

The round plate capacitor has the parameters **Sample Diameter**, **Sample Thickness**, **Cell Stray + Spacer Capacity** and **Spacer Area**. If **Edge Compensation** is enabled, the **Elelectrode Thickness** is additionally required. These geometrical values are the **calculation base of the dependent variables eps and sig**.

Diameter denotes the diameter of the round sample capacitor plates. If the diameter of the upper electrode is different from the lower electrode, the smaller diameter should be used. If the sample diameter is smaller than the electrodes, it should be used for **Diameter**.

Thickness denotes the spacing between the sample capacitor electrodes.

C_0 denotes the ideal empty cell capacity which is calculated by WinDETA by [6]

$$C_0 = \varepsilon_0 \frac{\pi D^2}{4d} \quad (*)$$

with $\varepsilon_0 = 8.85 \cdot 10^{-12}$ As/Vm, D - electrode **Diameter**, d - electrode spacing (**Thickness**).

Alternative to **Diameter** and **Thickness**, it is allowed to enter a value for C_0 . In this case the electrode **Thickness** will be adjusted by WinDETA according to (*) while leaving the actual electrode **Diameter** unchanged. Directly entering C_0 can be convenient for an electrode geometry different to round plates.

Cell Stray + Spacer Capacity is the sum of two additional capacities which are in parallel with the electrode capacity and which are subtracted from the measured parallel capacity C_p' by WinDETA. These are:

Cell Stray Capacity

- For the BDS 1200 cell and the ZGS active sample cell, it is about 1 pF.

- For the BDS 1308 sample cell it is about 5.5 pF.

- For the RF sample cells BDS 2100 and BDS 2200, 0 pF should be used, as the stray capacity is taken into account by the sample cell open calibration.

Spacer Capacity

If a sample cell with spacers embedded to the sample volume is used (e.g. for measuring liquids), the additional capacity created by the spacers may be specified. The **Spacer Capacity** may be calculated according to [6]

$$C_{spacer} = \varepsilon_s \varepsilon_0 \frac{A_{spacer}}{d_s}$$

where ε_s denotes the average dielectric constant of the spacer material, $\varepsilon_0 = 8.85 \cdot 10^{-12}$ As/Vm, A_{spacer} denotes the average area in the sample cell occupied by the spacers, d_s is the high of the spacer which should be equal to the sample **Thickness**.

The spacer area A_{spacer} is subtracted from the electrode area by WinDETA. If the sample cell without spacers is used, or the errors induced by the spacers can be neglected A_{spacer} should be set to 0.

If **Use Edge Compensation** is enabled, WinDETA subtracts the edge capacity C_{edge} of the sample capacitor from the measured sample capacity C_{sample} . The edge capacity is an additional capacity caused by the electric stray field at the borders of the sample capacitor. C_{edge} is in addition to the cell stray capacity and is calculated for a sample capacitor consisting of two round electrode plates with a round sample between [9]. Both electrodes and the sample must have the same diameter as shown in Figure 5.

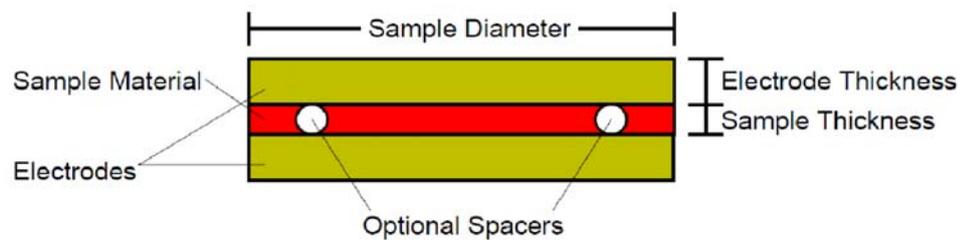


Figure 5. Sample cell.

WinDETA displays the values for

- capacity of the empty electrodes C_0 (vacuum between the electrodes)

and

- the ratio of edge capacity to C_0 (C_{edge}/C_0).

$\frac{C_{edge}}{\varepsilon' C_0}$ can be used to obtain the relative error in the real part of the permittivity caused by edge effects in a measurement.

Note:

- **Edge compensation** should be used with care, as it will correct stray field effects only approximately. This applies especially for large ratios of C_{edge}/C_o . The best method for high accurate results is to choose a sample geometry with a small ratio of $C_{edge}/(\epsilon' C_o)$. As a general rule, the edge effects decrease if the ratio **Sample Thickness / Sample Diameter** is kept small [9].

- **Edge compensation** should not be used for the RF sample cell, as the edge capacity is at least partly already taken into account by the sample RF cell calibration (HP4191), (HP4291) and (HP4991).

- **Edge compensation** should not be used for the BDS1308 liquid sample cell, as the electrodes are surrounded by the cell Teflon isolation and the presumptions for calculating C_{edge} do not apply anymore.

Important: The cell stray capacity + the spacer capacity + C_{edge} may be evaluated from a measurement of the empty cell with electrodes and spacers mounted. For this purpose, the value in the **Cell Stray + Spacer Capacity** field has to be adjusted until a permittivity' (Eps') is measured. This procedure presumes that uncertainty of the electrode **Diameter** and spacing (**Thickness** field) can be neglected. In this case, the procedure will result in best accuracy.

The method may be applied with **Edge compensation** enabled or disabled without an influence on the measured result.

The **Interdigit Electrode** is described by the following parameters:

| | |
|---|--|
| C_o | empty sample capacity |
| Pre-Resistance | resistance in series with the sample capacity due to the evaporated lines |
| Resistance of Object Carrier | resistance parallel to the sample capacity due to the conductivity of the object carrier |
| Basic Capacity of Object Carrier | capacity parallel to the sample capacity due to the object carrier |

For the magnetic test fixture, a sample with the geometry of a flat ring is required. The sample geometry is described by the parameters **Sample High**, **Outer Sample Diameter** and **Inner Sample Diameter**.

Note:

- You may change the sample specification of an existing measurement. This will only affect curves in the **Multi Graphics** and **3D** windows, which are **created after** the change of the sample specification. Existing curves are not altered. Select **Save Measurement Result** from the **Measurement** menu to save the new sample specification in the result file.

- If you change the used sample cell from the dielectric (**Dielectric Sample Cell** or **Dielectric Interdigit Electrode**) to magnetic (**Magnetic Sample Cell**) or the other way around, all curves in the **Online** and **Multi Graphics window** will be cleared and all **3D windows** will be closed.

8.2. Start Conditions (Measurement Menu)

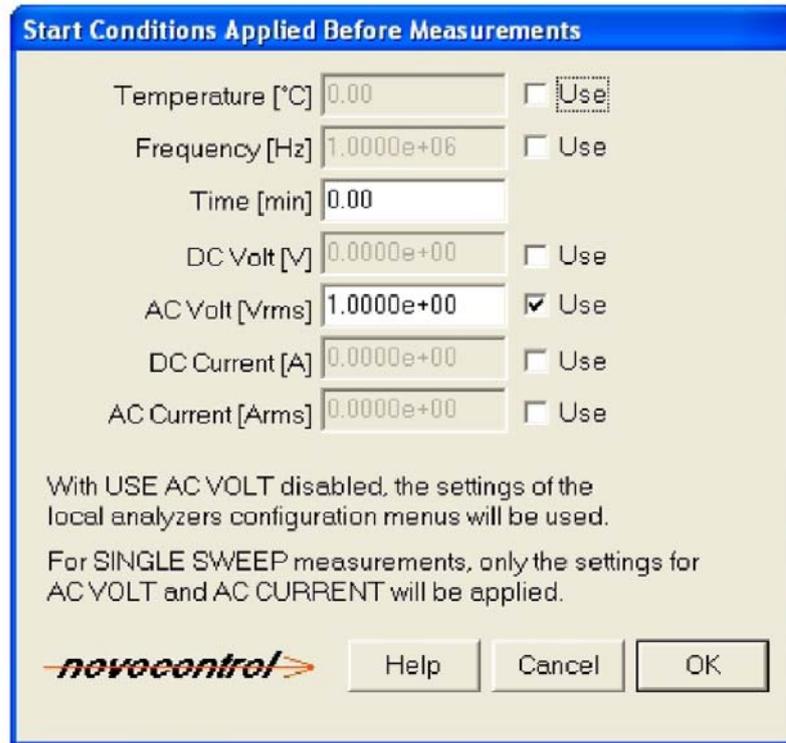


Figure 6. Sets the start up values for the free variables before the start of a measurement.

Each free variable activated by the corresponding **Use** switch is set to the specified value.

| | |
|--------------------|--|
| Temperature | Sets the setpoint of the temperature controller and initializes the controller, if Use Temperature is switched on. Otherwise, the state of the temperature controller is not changed and no start up temperature is set. |
| Frequency | Sets the frequency of the impedance measurement system's generator, if Use Frequency is switched on. The start frequency can be used in order to setup fixed frequency measurements without a frequency list defined. |
| Time | Sets a delay time interval. The delay time begins after selecting Start or Restart from the Measurement menu. |
| DC Volt | Applies an additional DC voltage to the sample, which is superimposed to the AC voltage. |
| AC Volt | Sets the amplitude of the AC amplitude of the impedance measurement system's generator. Note: If Use AC Voltage is disabled, no AC start voltage will be applied. If in addition no AC voltage list is defined, the default settings from the Configuration dialogues of the Analyzer menu will be used for the measurement. If Use AC Voltage is enabled, the default settings from the Configuration dialogues will not be used. |

| | |
|-------------------|--|
| DC Current | Applies an additional DC current to the sample, which is superimposed to the AC voltage. |
| AC Current | Sets the AC current amplitude of the impedance measurement system's generator. |

The **measurement task** is not started until the start time interval is over and the temperature has become stable.

All start conditions remain at their values until they are changed by a **value lists of the measurement task**, or by the **end conditions** or if the measurement task is stopped.

Start conditions which are not changed by a value list (no value list defined), remain active during the measurement.

Note: Except for **AC Voltage** and **AC Current** the start conditions are skipped, for measurement started with **Start Single Sweep** from the **Measurement** menu.

8.3. End Conditions (Measurement Menu)

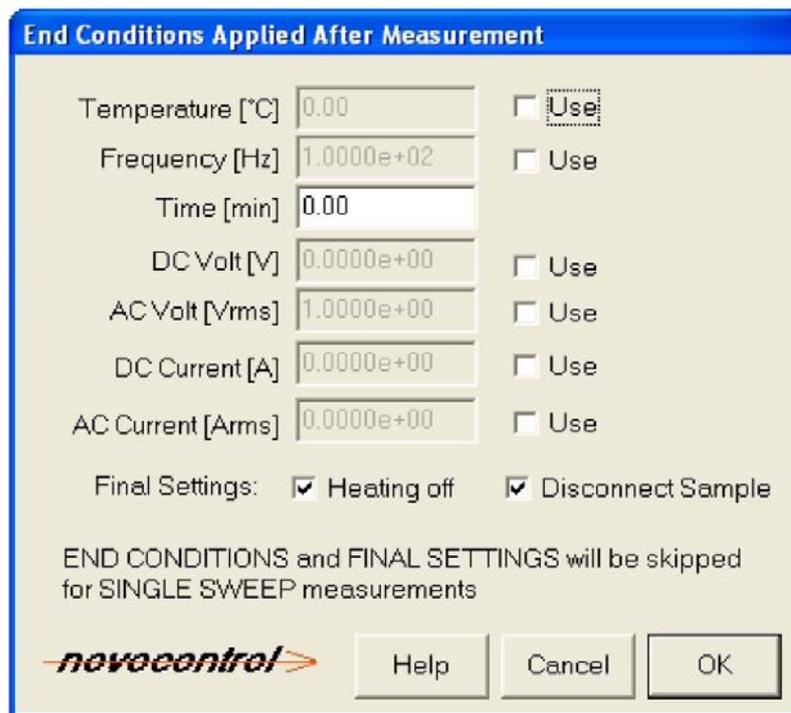


Figure 7. Sets the shut down values for the free variables after all dependent values have been measured.

Each free variable activated by the corresponding **Use** switch is set to the specified value after all points defined in the **Value Lists** were measured.

| | |
|--------------------|--|
| Temperature | Sets the setpoint of the temperature controller. Only available if temperature control has already been used within the measurement or the start conditions . |
| Frequency | Sets the frequency of the impedance measurement system's generator, if Use Frequency is switched on. |
| Time | Sets a delay time interval. The delay time begins after the measurement task as defined by the value lists has terminated. |
| DC Volt | Applies an additional DC voltage to the sample, which is superimposed to the AC voltage. |
| AC Volt | Sets the AC amplitude of the impedance measurement system's generator. |
| DC Current | Applies an additional DC current to the sample, which is superimposed to the AC voltage. |
| AC Current | Sets the AC current amplitude of the impedance measurement system's generator. |

The **measurement task** does not terminate until the time interval is over and the temperature has become stable.

After the **End Conditions** are processed, the **Final Settings** are applied.

Heating off disables the heaters if temperature control has been active.

Disconnect Sample has only an effect for the Alpha and Beta analyzers with firmware version ≥ 4.0 .

- The sample will be disconnected from the analyzer or test interface **Current Input** by an internal switch. In the disconnected state the **Current Input** is at high impedance.

- For a **POT/GAL test interface**, the **CE** terminal will be disconnected from the internal POT/GAL power amplifier, too.

- For a **HVB 4000 test interface**, the **4000 VppOut** terminal will be disconnected from the internal power amplifier, too.

Note:

- For safety reasons it is recommended to disconnect the terminals after a measurement. Disconnecting the **Curr. In** terminal will protect the current input from unintended application of signals. Disconnecting the **CE** or **4000 Vpp Out** terminal (POT/GAL or HVB 4000 only) will protect the electrodes and sample from unintended application of high voltages or currents.

- The terminals should be stay connected after a measurement, only if for physical or chemical reasons it is required to hold the sample at low impedance or at a defined voltage as adjusted in the **End Conditions** dialog of the **Measurement menu**.

- The end conditions including the **Heating off** settings will be skipped, if the measurement was started with **Start Single Sweep** or terminated by **Stop (Measurement menu)**.
- If the measurement was terminated by **Stop**, WinDETA will open a confirmation dialog and ask for the **Heating off** and **Disconnect Sample** settings.
- If a measurement or single sweep has terminated with a **HVB high voltage** or **POT/GAL test interface**, WinDETA will open a confirmation dialog and ask for the **Disconnect Sample** setting, if not already enabled in the **End Conditions** dialog.
- For all other impedance analyzers except the ones stated above, after a single sweep measurement the terminals will be left connected regardless of the **Disconnect Sample** setting in the **End Conditions** dialog.
- Select **Disconnect Sample and Stop** from the **Measurement menu** to manually disconnect the terminals within or after a measurement if still connected.

8.4. List Order (Measurement Menu)

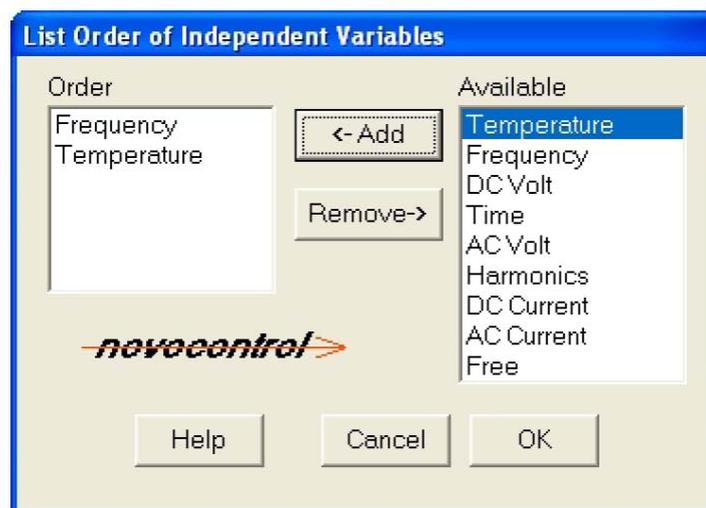


Figure 8. Defines the execution order of value lists for a later measurement.

By selecting the available **independent or free Variables**, you can define which of them are used in the next measurement and the order of the corresponding **value lists**. The variable on top of the left list box presents the list with the lowest order, the one on bottom presents the list with the highest order. For details see **Defining a Measurement Using Value Lists**.

In order to add a free variable to the **Order** list, select it in the **Available** list and press the **←Add** button. In order to delete a free variable from the **Order** list, select it in the **Order** list and press the **Delete** button (see Fig. 8).

8.5. Value Lists (Measurement Menu)

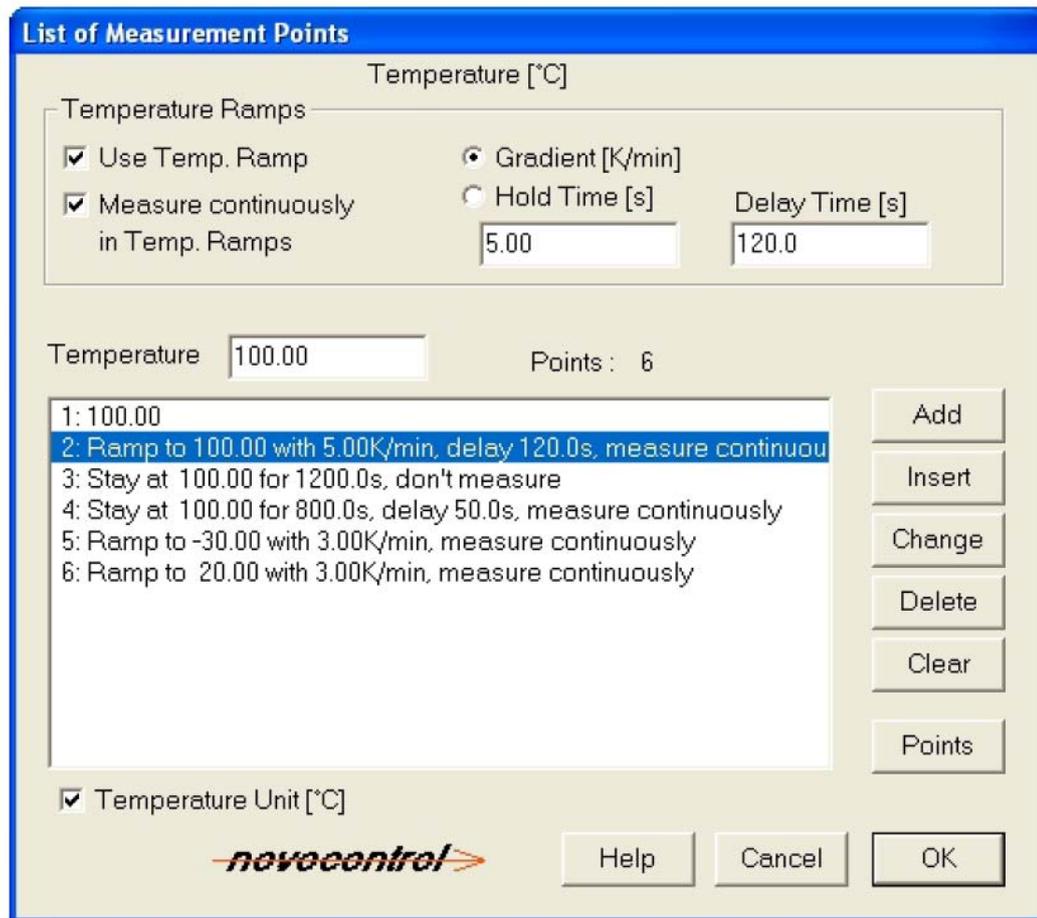


Figure 9. Creates and modifies the value lists.

Creates and modifies the value lists for **Temperature, Frequency, Time, DC Volt, AC Volt, DC Current, AC Current, Harmonics** or **Free User Defined Values**. The value lists define the values of the **Independent or Free Variables** to which the **Dependent or Not Free Variables** in the next measurement are measured.

Enter a value in the **Temperature, Frequency, Time, DC Volt, AC Volt, DC Current, AC Current** input field and press the **Add** button to add the value to the end of the list.

If the list already contains items, select one item with the mouse.

Press the **Insert** button to insert the input field value above the selected item (see Fig. 9).

Press the **Change** button to replace selected item by the input field value. Press the **Delete** button to remove the selected item. Press the **Clear** button to clear all items in the list.

The **Points** button will open the **Measurement Points by Steps Dialog** dialog which lets you create lists automatically.

The **Standard** button will create a standard frequency lists for the actual device combination automatically.

Note: If you are using a Hewlett Packard 4191, the standard frequency list corresponds to the internal calibration frequencies of the HP 4191. Therefore, using the standard frequency list both for the measurement and the sample cell calibration (HP4191) will result in highest measurement accuracy.

If the Temperature List or the Time List are defined with highest order (lowest position in the order list), continuous temperature or time measurements may be defined as described below.

In the **Temperature Ramps** field of the **Temperature List** in addition to fixed temperature setpoints, **temperature ramps or time intervals for continuous measurements** may be defined.

This is done by activating the **Use Temp. Ramp** switch for the actual selected item in the list.

Select the **Gradient** button to define a continuous temperature ramp measurement. Specify the temperature gradient Tgrad (ramp speed) in the field below the **Gradient** button. Specify the ramp end temperature Tend in the **Temperature** field. This will cause WinDETA in later measurement to start a temperature ramp from the actual temperature to Tend with the temperature gradient Tgrad. During the temperature ramp, WinDETA will continuously execute measurement blocks defined by value lists above the temperature list in the **order list**.

Select the **Hold Time** button to define a continuous measurement at constant temperature for a time interval. Specify the time interval Time_dur in the field below the **Hold Time** button. Specify the constant temperature Tstay in the **Temperature** field. This will cause WinDETA in later measurement to set the temperature to Tstay. For the time Time_dur, WinDETA will continuously execute measurement blocks defined by value lists above the temperature list in the **order list**.

In both modes, an optional delay time may entered in the **Delay Time** field. This will delay the start of each measurement block in a continuous measurement by delay time.

After selecting the parameters for the continuous temperature ramp or interval, use the **Add**, **Insert**, and **Change** buttons, to place it to selected position in the temperature list.

Continuous temperature ramp intervals will be displayed in the list as **Ramp to Tend with Tgrad** if no delay time is specified or **Ramp to Tend with Tgrad, delay Time_delay** with specified delay time.

Continuous time intervals at fixed temperature will be displayed in the list as **Stay at Tstay for Time_dur** if no delay time is specified or **Stay at Tstay for Time_dur, delay Time_delay** with specified delay time.

In the **Time List** in addition to fixed time points, **intervals for continuous time measurements** may be defined.

This is done by selecting the **Continuous Interval** button instead of the **Time Point** button (for fixed time setpoints). Specify the end time `Time_end` in the **Time** field. This will cause WinDETA in later measurement to continuously execute measurement blocks defined by value lists above the temperature list in the **order list** until the end time `Time_end` is reached.

An optional delay time may entered in the **Delay Time** field. This will delay the start of each measurement block in a continuous measurement by delay time.

After selecting the parameters for the continuous temperature ramp or interval, use the **Add**, **Insert**, and **Change** buttons, to place it to selected position in the time list.

Continuous time intervals will be displayed in the time list as

Continuous to `Time_end` if no delay time is specified or

Continuous to `Time_end`, delay `Time_delay` with specified delay time.

The **Harmonics** list defines the **Lowest Harmonic > 0** and the **Highest Harmonic** to be measured in the next measurement. The number of the first harmonic (after the base wave) is 1. WinDETA will measure in addition all higher harmonics between **Lowest Harmonic** and **Highest Harmonic**.

The list of **Free User Defined Values** is used to measure dependencies of any quantity which can not be automatically controlled or measured by WinDETA, but can be adjusted manually by the user instead like the cell pressure. For the manual adjustable quantity a name, unit and values can be defined as for the other value lists. In a later measurement WinDETA will ask the user the quantity has to be adjusted to a new value to do so. The advantage of this procedure is, that more dimensional measurements including the manually adjusted quantity can be stored in one file and evaluated, exported and represented like the other automatic value lists.

8.6. Measurement Points by Steps (Sub Dialog of Value Lists)

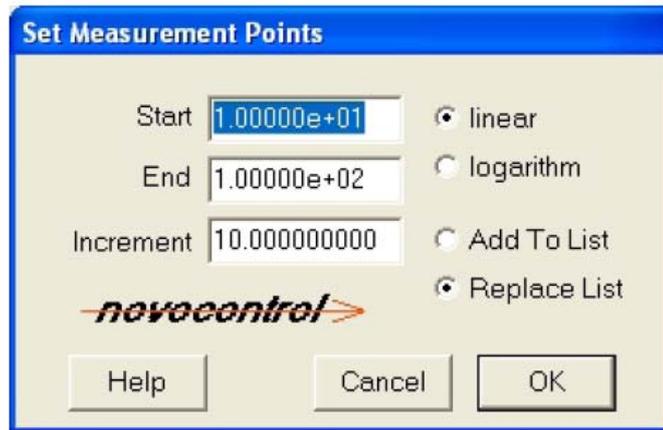


Figure 10. Creates value lists.

Creates **value lists** automatically. It is possible to define the first point, the last point and the factor (logarithm division) or distance (linear division) of your list.

Start: Specifies the first point, which will be automatically created. **End:** Specifies the last point which will be automatically created (see Fig. 10).

If **linear** is selected, the increment between succeeding points except the last point will be constant and equal to the input field **Increment**.

If **logarithm** is selected, the multiplier between succeeding points except the last point will be constant and equal to the input field **Factor**.

If **Add To List** is selected, the created points are added to the end of the actual list, after the OK button is pressed.

If **Replace To List** is selected, the actual list is replaced by the created points, after the **OK** button is pressed.

8.7. Averaging (Measurement Menu)

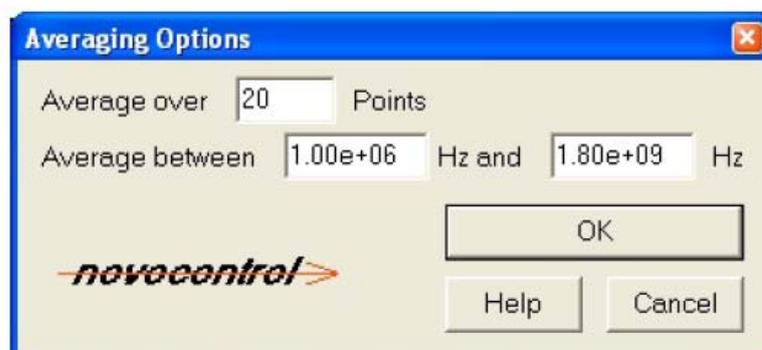


Figure 11. Sets the measurement averaging options.

WinDETA allows averaging over an arbitrary number of points in a specified frequency interval (see Fig. 11).

Average over n points specifies the number of measurement points which are taken at a given frequency. The result is calculated as the arithmetic average of these points.

Average between min Hz and Max Hz specifies the frequency interval, in which averaging is done.

8.8. Start Single Sweep (Measurement Menu)

Before starting a measurement, check whether all device connections are ok, a proper sample is connected to the impedance measurement system and all required devices are switched on. If you have used DC volt or time control see Independent or Free Variables for the restrictions of these measurements.

A single sweep measurement is a one dimensional measurement which uses only the first **value list** (with the lowest order) defined in the **order list**. The **Start Conditions** and most of the **End Conditions** will be ignored.

A single sweep is useful to get a quick overview about the behaviour of the sample without changing the configuration of a more dimensional measurement. It is recommended, to run a single sweep before starting a complete measurement to check whether everything is all right.

8.9. Start (Measurement Menu)

Starts a complete measurement specified by its **Value Lists**, **List Order**, **Start Conditions** and **End Conditions**.

Before starting a measurement, check whether all device connections are ok, a proper sample is connected to the impedance measurement system and all required devices are switched on. If you have used DC volt or time control see Independent or Free Variables for the restrictions of these measurements.

8.10. Restart (Measurement Menu)

Restarts a measurement stopped by user or by an error. The restart is also possible after WinDETA was interrupted.

Before starting a measurement, check whether all device connections are ok, a proper sample is connected to the impedance measurement system and all required devices are switched on. If you have used DC volt or time control see Independent or Free Variables for the restrictions of these measurements.

You may restart a measurement which was aborted before all values could be measured. A measurement is aborted if you select **Stop** from the Measurement menu or if an error occurs.

Possible errors during a measurement may be: loss of power supply, switching off devices or disconnecting lines from devices, device errors, lack of resources (liquid nitrogen for temperature control or space on hard disk), bugs in other programs running at the same time.

In order to restart an interrupted or stopped measurement proceed as follows:

- If the interrupted measurement was the last measurement done by WinDETA, load the preset file **LAST_MES.PRE** with **Load Preset** from the **File** menu. **LAST_MES.PRE** is automatically created if a measurement is started and contains all WinDETA settings of the last measurement.

- If the interrupted measurement was not the last measurement done by WinDETA, make sure that the settings for actual defined temperature controller (**Temp. Controller** menu) and analyzer (**Analyzer** menu) correspond with settings used in the interrupted measurement.

- Reload the interrupted measurement with **Load Measurement Result** from the **File** menu.

- Select **Restart**.

It is also possible to automatically restart the measurement after a power breakdown.

8.11. Stop (Measurement Menu)

The measurement will be aborted. For safety reasons, a confirmation dialog is displayed before stopping.

Before stopping the measurement, you may adjust the **Final Settings** switches:

Heating off disables the heaters if temperature control has been active.

Disconnect Sample has only an effect for the Alpha and Beta analyzers with firmware version ≥ 4.0 .

- The sample will be disconnected from the analyzer or test interface **Current Input** by an internal switch. In the disconnected state the **Current Input** is at high impedance.

- For a **POT/GAL test interface**, the **CE** terminal will be disconnected from the internal POT/GAL power amplifier, too.

- For a **HVB 4000 test interface**, the **4000 VppOut** terminal will be disconnected from the internal power amplifier, too.

Note:

- For safety reasons it is recommended to disconnect the terminals after a measurement. Disconnecting the **Curr. In** terminal will protect the current input from unintended application of signals. Disconnecting the **CE** or **4000 Vpp Out** terminal (POT/GAL or HVB 4000 only) will protect the electrodes and sample from unintended application of high voltages or currents.

- The terminals should be left connected after a measurement only, if for physical or chemical reasons it is required to hold the sample at low impedance or at a defined voltage as adjusted in the **End Conditions** dialog of the **Measurement menu**.

After confirming the dialog with **Yes**, the measurement task will be stopped. A stopped measurement can be re-initialized with **Restart** (Measurement menu).

8.12. Calibrate ALPHA, BETA, BDC All (Analyzers, Options Menu)

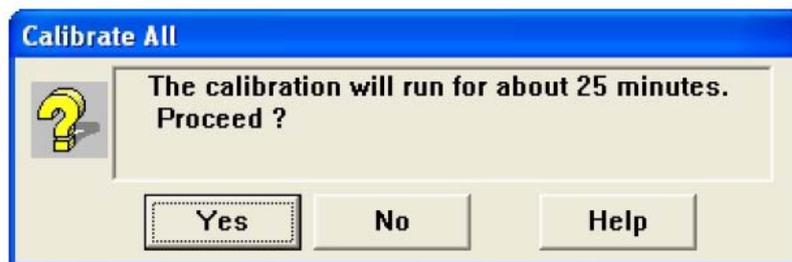


Figure 12. Starts a basic calibration for an ALPHA, BETA Analyzer or BDC.

As low frequencies are involved, this calibration will require a certain amount of time.

Depending on the system the following elements are calibrated:

ALPHA-A, S, -N, -L, -K, or -H in the low voltage mode or BETA:

The internal current ranges with exceptions of the ranges $< 1\text{k}\Omega$ are calibrated.

If low impedance samples $< 1\text{k}\Omega$ have to be measured, a **Load Short Calibration** should be done after the All calibration.

If low capacity samples $< 1\text{pF}$ have to be measured, a **Low Capacity Open Calibration** should be done after the All calibration.

Systems based on the Novocontrol Dielectric Converter BDC:

All internal BDC components are calibrated. No further calibration is required.

The All calibration should be performed (see Fig. 12)

- in intervals of some weeks,

- except for the ALPHA-A and BETA analyzer: before a measurement with the BDC or the ALPHA is started for the first time after WinDETA installation.

Before selecting this menu item be sure to have connected one of the following analyzers in a proper setup and all devices switched on.

9. Temperature Controller

9.1. Initialize from WinDETA (Temp. Controller Menu)

Initializes the active temperature controller to start the communication with WinDETA. The settings of the temperature controller are initialized from the settings of WinDETA's dialogs.

The QUATRO safety limits are always initialized by the default values of WinDETA, which are **not** saved in the preset files [6, 8].

If temperature logging is enabled, the **Temperature Log Window** and the **temperature log file** will be initialized. If the initialization is unsuccessful, the **status window** will get out of work and measurement using temperature control will not be started.

If you have temporarily disconnected the temperature controller from the PC, you may use this item to re-initialize.

9.2. Initialize WinDETA from Controller (Temp. Controller Menu)

Initializes the active temperature controller to start the communication with WinDETA. The settings of WinDETA's dialogs are initialized from the settings of the temperature controller.

If temperature logging is enabled, the **temperature log window** and the **temperature log file** will be initialized. If the initialization is unsuccessful, the **status window** is out of operation and measurements using temperature control will not be started.

If you have temporarily disconnected the temperature controller from the PC, you may use this item to re-initialize.

9.3. Disconnect WinDETA from Controller (Temp. Controller Menu)

Causes WinDETA to stop the communication with the temperature controller. This option has to be activated, before WinDETA will allow to select another active temperature controller. The option is also useful, if you intend to switch off the temperature controller before leaving WinDETA.

9.4. Safety Limits (Temp. Controller Menu)



Figure 13. Sets the Quatro temperature controller safety limits.

The limits are not saved in the preset file, and therefore are set to their default values at the start of WinDETA and at the start of each measurement (see Fig. 13).

Dewar empty -200.0 to 400.0 °C

If the temperature at the dewar evaporator exceeds this limit, the dewar control as well as the gas heating are switched OFF because the dewar is likely to get empty.

Factory set value: -170°C.

You can override this limit function by setting this value to -200°C.

Low pressure 0.0 to 1000.0 mBar

If the pressure inside the dewar falls below this limit, the gas heating is switched off.

Factory set value: 50 mBar.

WARNING: Don't set this value below 10 mBar if you want prevent your gas heating from severe damage! You can explicitly override this limit function by setting this value to 0 mBar.

Over pressure 0.0 to 1000.0 mBar

To protect the dewar, the pressure control as well as the gas heating control are switched off, if the pressure exceeds this value.

Factory set value: 500 mBar.

You can override this limit function by setting this value to 1000 mBar. The dewar is then protected by its built in over pressure valve.

Start temperature -200.0 to 400.0 °C

This limit functions ensures N2 flow before the gas heating is powered. If the gas heating is switched on, the DC power outlet is disabled until the gas temperature falls below this value.

Factory set value: -20°C.

It may be necessary to increase this limit value if you operate the gas heating with N₂ from your laboratory mains instead of the liquid N₂ in the dewar. In this case, only the pressure limits are active. Be sure to set it (see above) to a safe value [6].

9.5. Configuration, Quatro V ≥ 4.0 (Temp. Controller Menu)

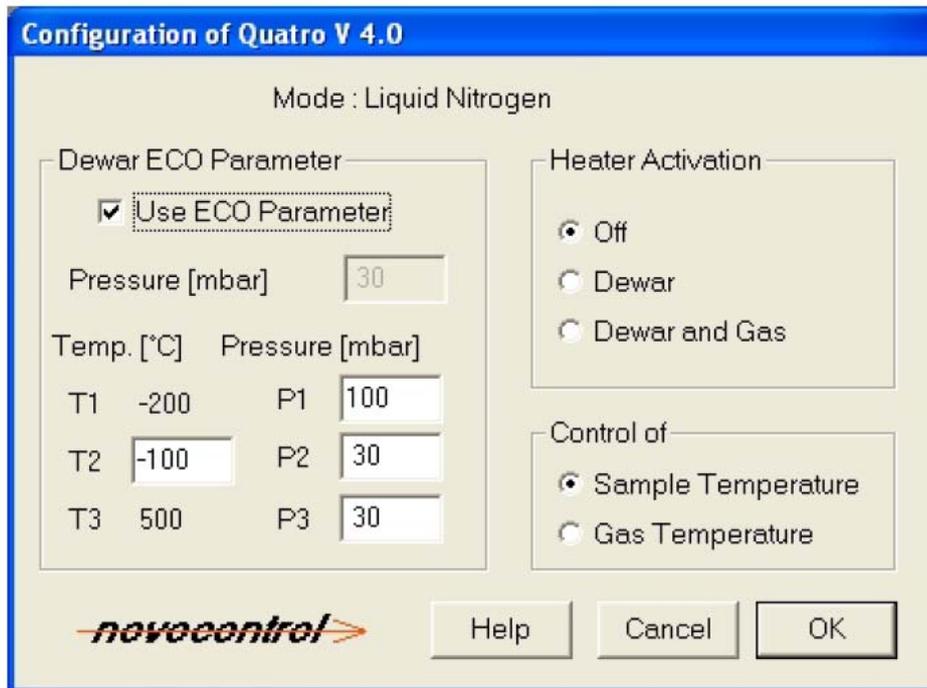


Figure 14. Sets the Quatro temperature controller version ≥ 4.0 configurations.

Heater Activation

Off: Switches all heatings off.

Dewar: Switches the dewar heating on.

Dewar and Gas: Switches the dewar and gas heating on.

Note: The gas heating will only be activated, if all **safety limits** match. Therefore, the gas heating generally cannot be switched on at the same time as the dewar heating, because the system will need some time to reach the safety limits. The dewar heating is turned on automatically if a measurement is started, which needs temperature control (see Fig. 14). If the dewar pressure is about its low pressure limit, also the gas heating is turned on. If **heating of** in the **End Conditions** dialog (menu Measurement) has been selected, both heatings are turned off at the end of the measurement.

In the **Control of...** field, the temperature source being used by the controller can be selected.

Sample Temperature selects control of the sample temperature. In this case, temperature control will be very accurate, but the stabilization will require some time.

Gas Temperature selects control at the temperature at the gas heater. In this case, temperature control will be very fast, but the sample temperature generally will not match to the temperature setpoint of the **temperature list**, because there will be always a difference between gas and sample temperature.

The field **Dewar ECO Parameter** controls the pressure in the liquid nitrogen dewar.

If **Use ECO Parameter** is not selected (not crossed), the dewar pressure will be set to the constant value specified in the **Pressure** field.

If **Use ECO Parameter** is selected (crossed), the dewar pressure will depend on the actual setpoint of the temperature controller. The dependence pressure - setpoint is specified by the four fields T2, P1, P2, and P3 defining two lines in a pressure - temperature diagram as shown in Figure 15:

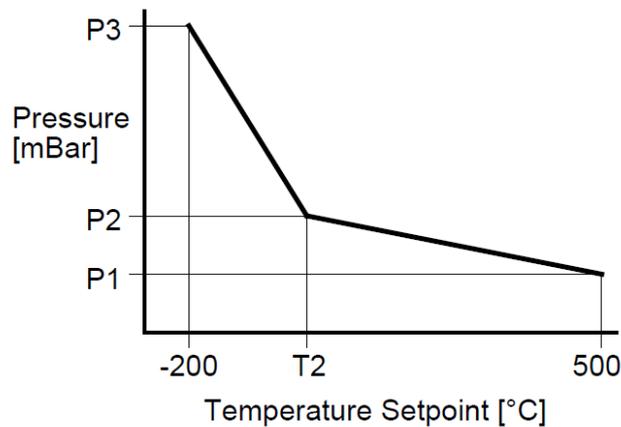


Figure 15. The pressure - temperature setpoint diagram.

This concept allows to use high pressure at low temperatures to get a large cooling power to reach lowest temperatures. On the other hand, at high temperatures the pressure can be reduced. This will save liquid nitrogen and heating energy. Moreover the system reaches higher end temperatures at lower pressure, because a smaller amount of cold nitrogen has to be heated.

Standard values are [6]:

| P1 | T2 | P2 | P3 |
|-----------|-----------|-----------|-----------|
| 180mBar | -100°C | 30mBar | 30mBar |

10. Axes

10.1. Online Axes (Graphics Menu)

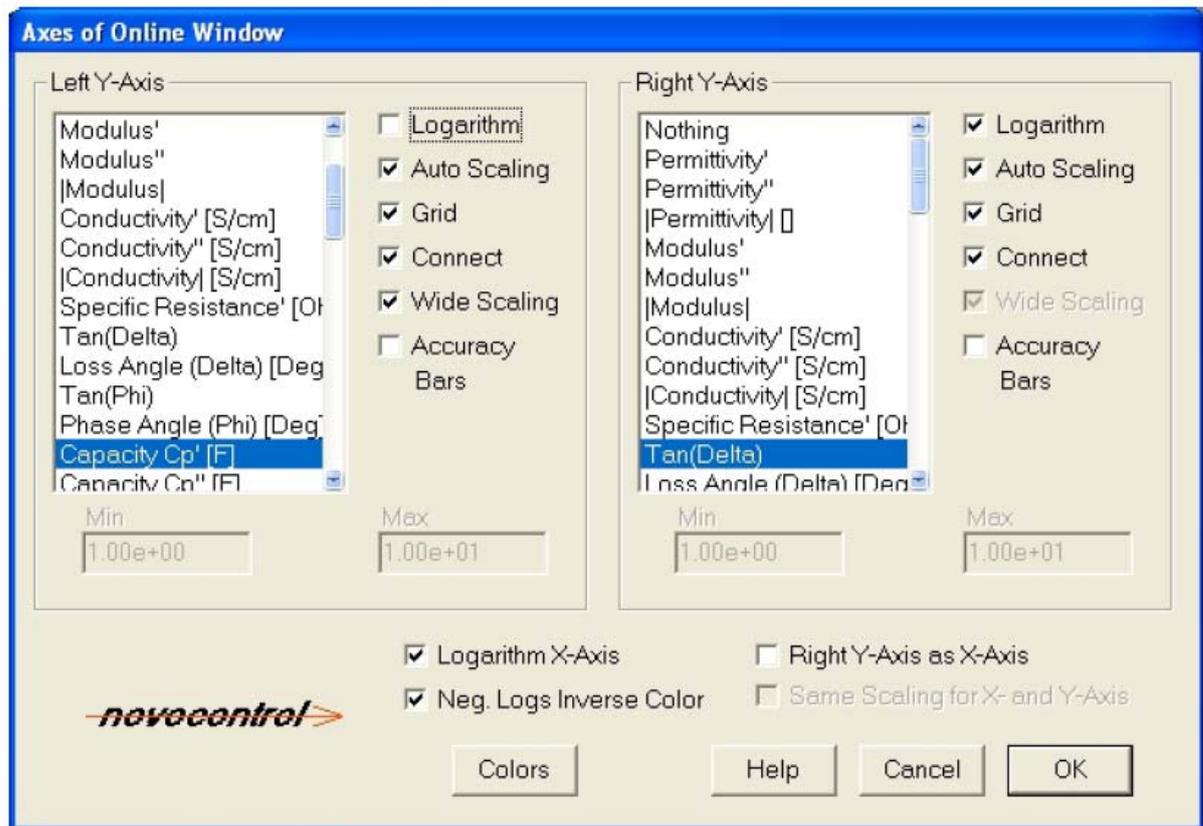


Figure 16. Sets the online window configurations.

The **Y-Axis** lists select the **dependent variables** of the y-axes, the switches define the curve presentations (see Fig. 16). The variable of the x-axis is same of the inner list of the measurement **list order**.

Switches:

| | |
|-------------------------|--|
| Logarithm | Causes the axis division to be logarithm, else linear. If Logarithm is switched on, negative values are converted to positives. |
| Auto Scaling | Causes the axis scaling to be done automatically, else within minimum and maximum. |
| Grid | Draws a division grid. |
| Connect | Connects data points to a curve. |
| Wide Scaling | Wide scaling causes the y-axis to show a larger area around the curve for flat curves. If this option is disabled, flat curves will be scaled from their minimum to their maximum value so that even small deviations are shown. |
| Accuracy Bars | Displays bars showing the measurement accuracy for each measured point. This option is not available for all device combinations and for all dependent variables. |
| Logarithm X-Axis | Displays the x-axis in logarithmic representation. |

| | |
|--|---|
| Neg. Logs Inverse Color | Shows negative values on a logarithmic y-axis in inverse color. |
| Right Y-Axis as X-Axis | Draws the variable selected for the right y-axis on the x-axis. This allows you to create diagrams showing a curve of two dependent variables in dependence of a free variable which is not plotted. Example: Cole-Cole plot: ϵ' against ϵ'' with frequency as the parameter. |
| Same Scaling for Y- and X- Axis | Creates identical scaling and axis length for the X- and Y-axis. This option can be used e.g. in combination with the option above in Cole Cole plots in order not to distort semicircles. |

Min, Max: Selects an invisible interval to which the data are clipped and the axis is automatically scaled, if **Auto Scaling** is off.

The **magnifier mode** is not available while a measurement is running.

10.2. Multi Graphics Axes (Graphics Menu)

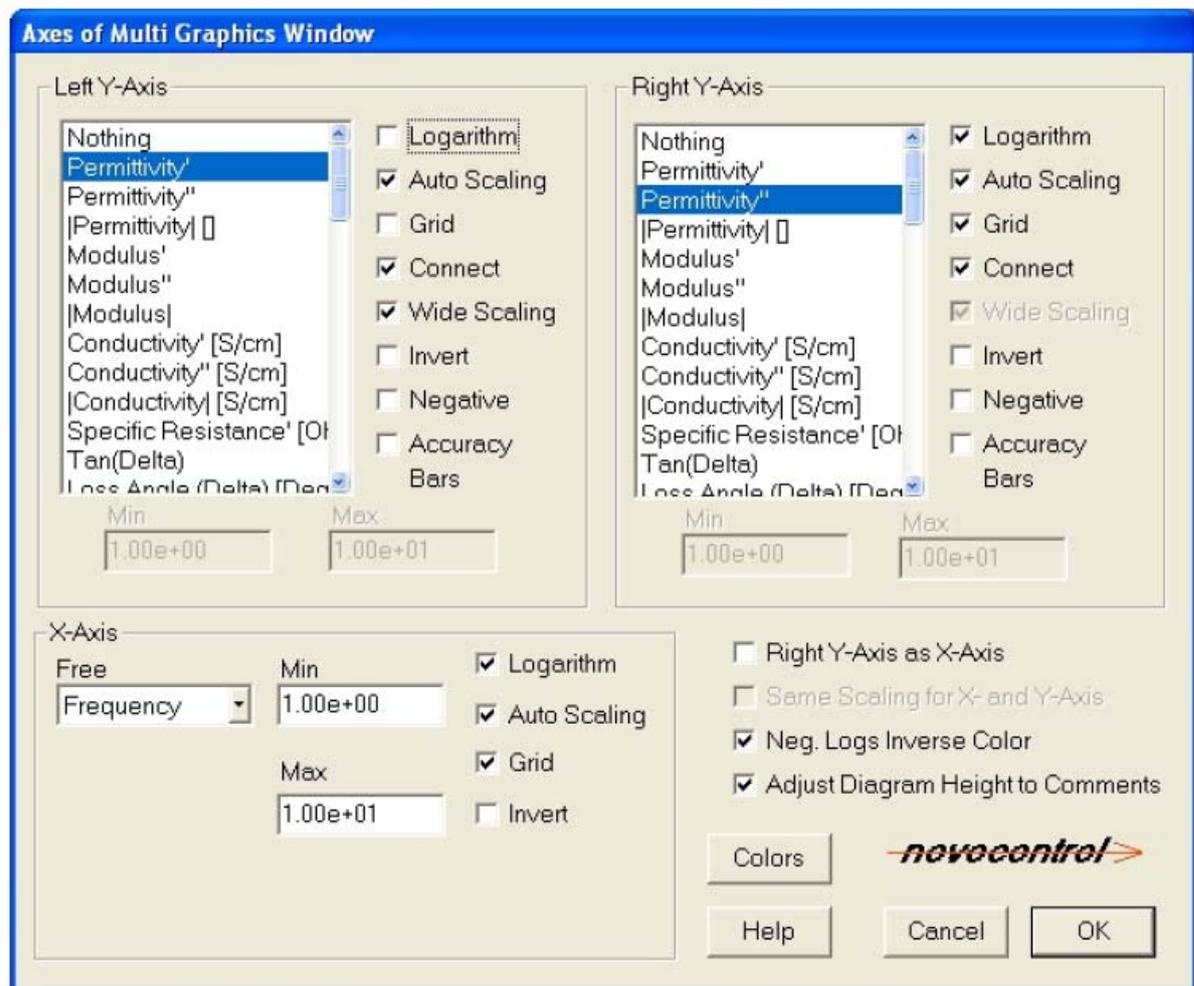


Figure 17. Sets the multi graphics window configurations.

The **Y-Axis** lists select the corresponding **dependent or not free variables**. The switches define the curve presentations. The **independent or free variable** of the x-axis is set

by the **X-Axis Free** list (see Fig. 17). If the variable on the x-axis is changed, all existing curves in the multi graphics diagram are cleared.

10.3. Multi Graphics Curves (Graphics Menu)

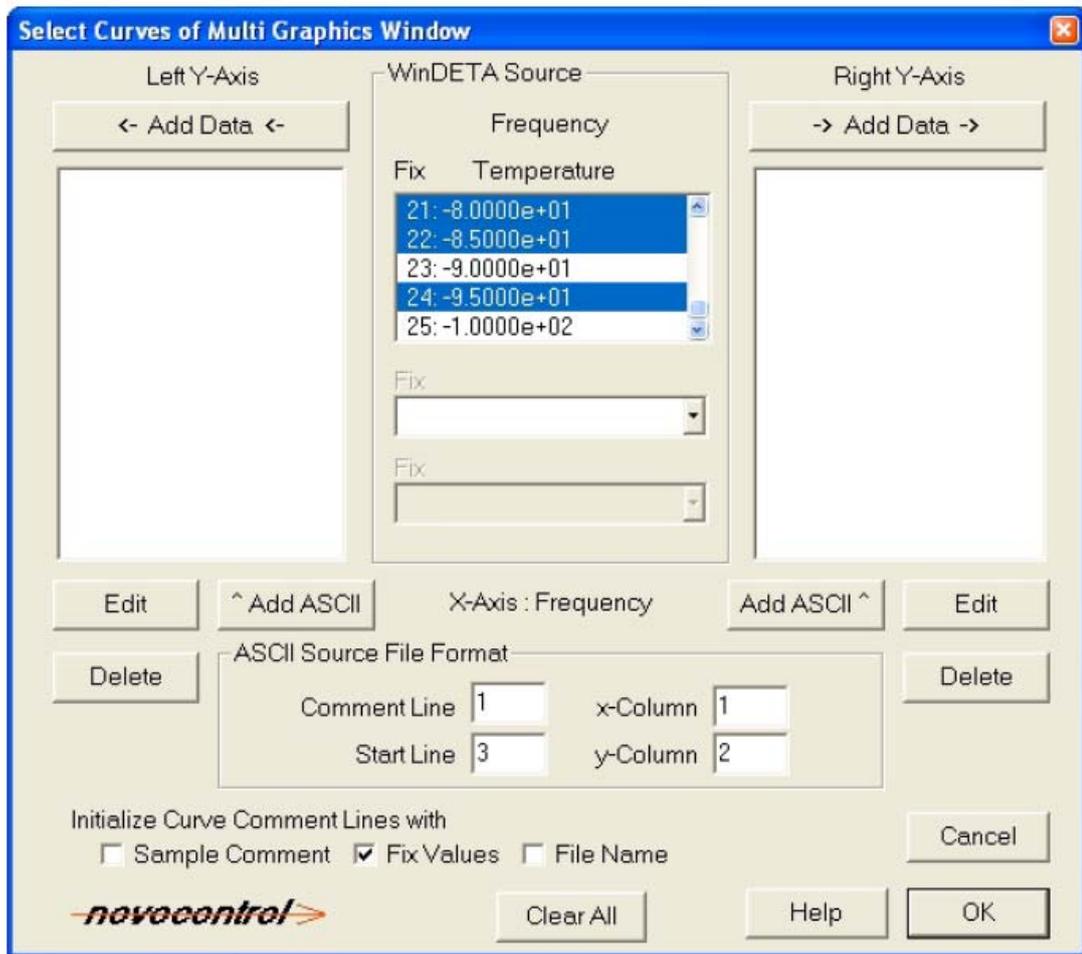


Figure 18. Adds or removes data curves of the multi graphics window.

The comments of all data curves shown in the multi graphics window are listed in the **Left Y-Axis** and **Right Y-Axis** lists.

In order to display data in the multi graphics window, you must add data curves to the **Left-Y Axis** and **Right Y-Axis** lists (see Fig. 18). Before doing so, you may select the diagram configurations and especially the x-axis variable by the **Multi Graphics Axes** dialogue from the **Graphics** menu.

Data curves are added from the WinDETA internal measurement object. It contains either the current measurement in progress or a measurement loaded from a WinDETA binary result file by **Load Measurement Result** (**File** menu).

The free variable available to add is displayed in the first line of the **WinDETA Source** field.

Select the **Add Data** buttons of the **Left Y-Axis** or **Right Y-Axis** fields to add data curves.

Note:

- If the internal measurement object holds data with more than one dimensions (more than one lists defined in the **order list**), select the fix values from the **Fix** lists in the **WinDETA Source** field, in order to get a defined selection before pressing the **Add** buttons. The fix values specify values for the free variables contained in the measurement, but not selected as the diagram x-axis. For details see **Selecting Data from a Measurement**.

- The contents of the multi graphics window is not cleared if a new result file is loaded in the internal measurement object by **Load Measurement Result** (**File** menu). This allows to add and display data curves from several files simultaneously.

To delete a curve from the diagram, select it in the **Left Y-Axis** or **Right Y-Axis** lists and press the corresponding **Delete** button.

Press the **Clear All** button to delete all curves in the diagram.

If a data curve is added with **Add Data** into the diagram, WinDETA will attach an unique plot symbol to each curve to separate it from other data curves. In addition each curve symbol with a corresponding curve comment line text is displayed in the diagram header. WinDETA initializes the curve comment line according the settings of **Initialize Curve Comment Lines with**

- **Sample Comment:** The **Sample Comment** from **Sample Specification** of the **File** menu;

- **Fix Values:** The values of the **free variables** which are either defined in **Start Conditions** of the **File** menu and / or

free variable values defined in a **value list** of the **File** menu which are constant for the selected curve;

- **File Name:** The *.eps data file name from which the curve was added into the diagram.

To edit a curve comment line, select it in the **Left Y-Axis** or **Right Y-Axis** lists and press the corresponding **Edit** button.

You may also add curves from **ASCII files** to the diagram. For this purpose, the ASCII file must contain the values for the x-axis and y-axis in separate columns. It may contain other columns with data and optional comment lines before the data columns.

In order to add a curve from an ASCII file, proceed as follows:

- Enter the format of the ASCII file by using the following input fields

Comment line: Specifies the line in the file from which WinDETA reads the comment for the curve. Enter 0 for no comment.

Start Line: Specifies the row in the file where the data columns start.

x-Column: Specifies the column in the file from which WinDETA reads the values for the x-axis.

y-Column: Specifies the column in the file from which WinDETA reads the values for the y-axis.

- To add a curve to the diagrams left or right y-axis, press the corresponding **Add ASCII** button.

- Select the name of the ASCII file from the opened file select box.

The comment of the curve will be displayed in the **Left Y-Axis** or **Right Y-Axis** lists.

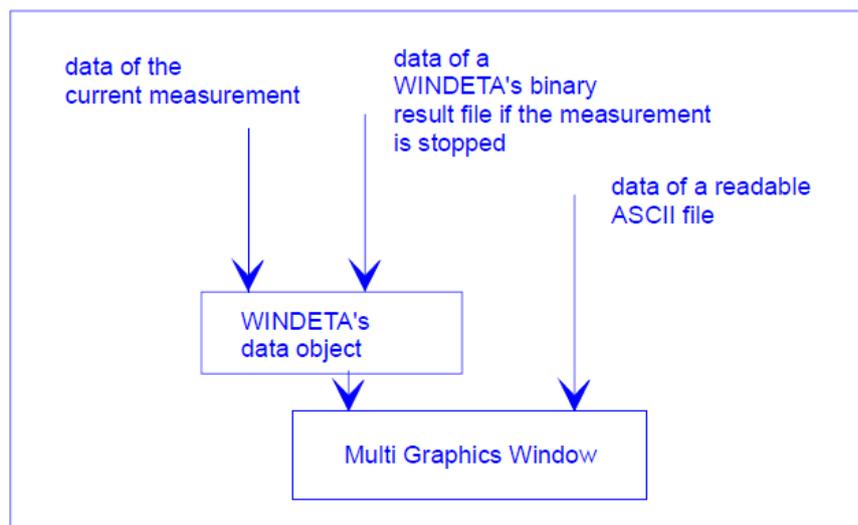


Figure 19. Data flow of WinDETA.

To save or load the complete contents of the multi graphics window see **Load 2D diagram** or **Save 2D diagram** from the **File** menu.

Note:

If data are imported from ASCII files, the following format rules apply.

- The ASCII file must be arranged in lines. Each line corresponds to one data point. The lines must be separated by new lines (ASCII 10) chars.

- Lines specified as data lines must contain at least two numbers arranged in columns.

Two columns must be selected for the x and y values of the data point as described above.

- The columns must be separated by an arbitrary number of the following white space characters:

'space' (ASCII 32), 'tab' (ASCII 9), ';' (ASCII 59).

The start of a new column is recognised by the first character which is not a white space character.

- Each column selected for data must start with a number.
- Examples for numbers are 1, 1.2, 1.23e6, 123e4.

10.4. 3D Window (3D Window Graphics Menu)

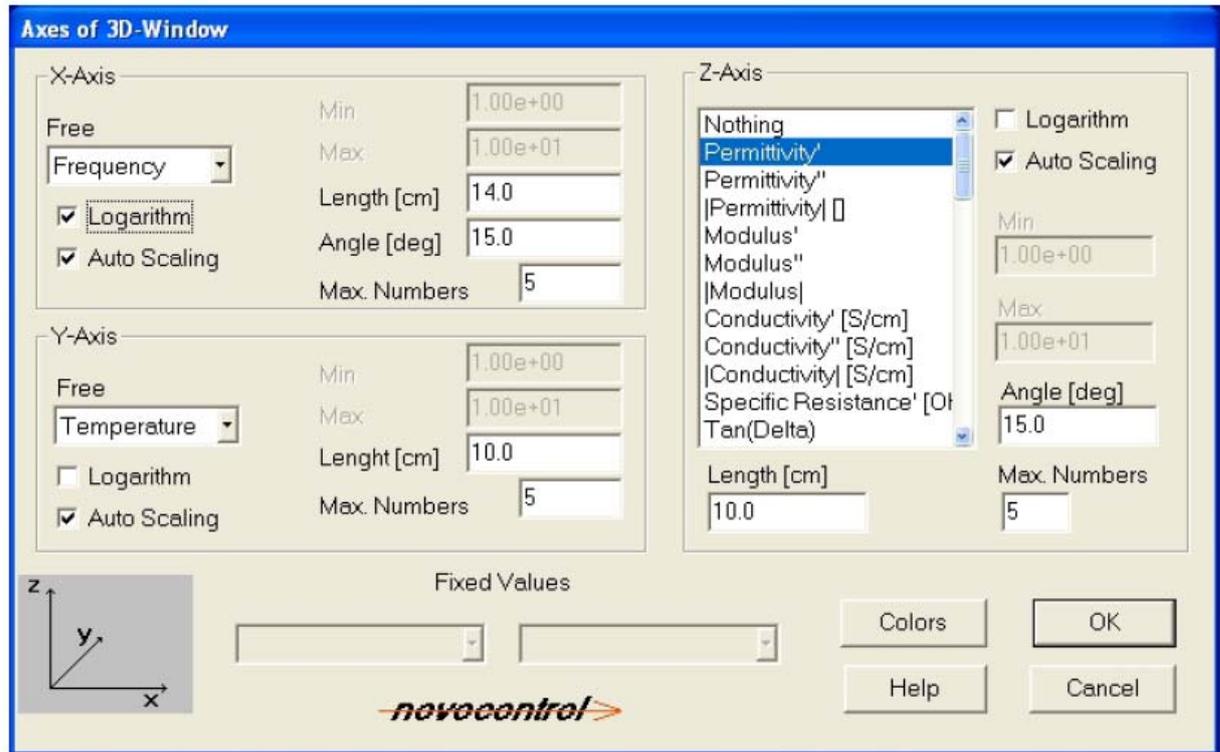


Figure 20. Sets configurations of the actual 3D window.

A 3D plot can only be created if the actual loaded measurement includes two or more **independent variables**, for instance frequency and temperature. These two free independent variables are selected by means of the x- and y- **Free** combo boxes (see Fig. 20). If they are more as two independent variables it is necessary to choose the measurement points of them at which the surface will be drawn. By using the **Fix Values** combo boxes you can define the points respectively.

If the measurement is running or was stopped WinDETA's internal data object contains the data measured so far, which can be shown in the 3D diagram. Another source is a WinDETA binary result file. If the measurement is not running it is possible to **load the result file** in the internal data object.

Note: The data from the internal data object are copied to the 3D diagram when the OK button of the **Configuration** dialog is pressed. The diagrams do not change if the internal data

object is loaded with new data. This enables to display several measurements in several 3D windows simultaneously.

Switches:

| | |
|---------------------------------|--|
| Nothing... [Inductivity] | Defines which Dependent or Not Free Variables will be shown at the z-axis. |
| Logarithm | Causes the axis division to be logarithm, else linear. If Logarithm is switched on, negative values are converted to positives. |
| Auto Scaling | Causes the axis scaling to be done automatically, else within minimum and maximum. |

Min, Max: Selects an invisible interval to which the data are clipped and the axis is automatically scaled, if **Auto Scaling** is off.

Length: Specifies the length of an axis which is not rotated. The unit [**cm**] is accurate in print outs.

Max. Numbers: Specifies the number of numbers drawn on the axis by the auto sealer. You may use this, to avoid over writing of axis numbers by others.

Angle: Specifies the rotation angle of the axis.

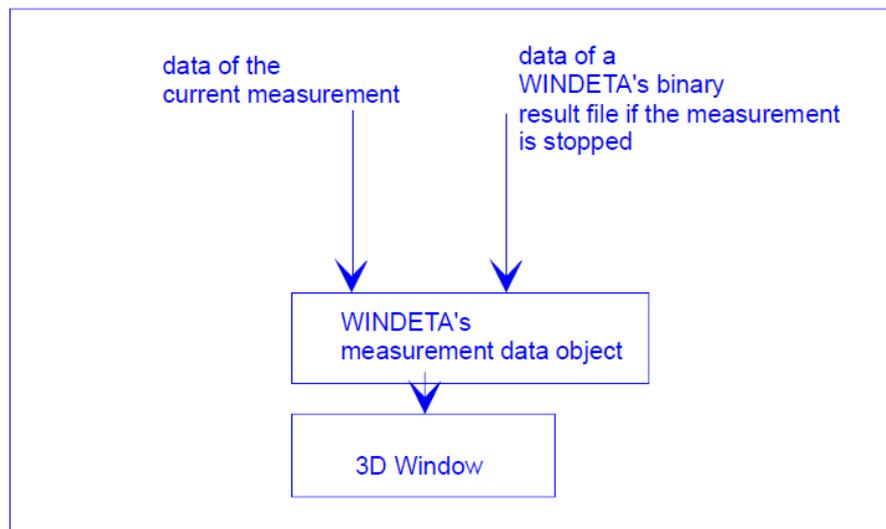


Figure 21. Data flow of WinDETA.

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