Test Interfaces for Alpha-A Modular Measurement System

Concepts
Features
Principles of Operation
Application Examples

Introduction

When Novocontrol introduced the Alpha analyzer in 1998, it was soon accepted by most researchers as the most accurate and versatile analysis tool for dielectric, conductivity and impedance spectroscopy. The Alpha analyzer combines a series of exceptional features like ultra wide impedance range, frequency range and high accuracy in a fully automated straightforward to handle instrument [1].

These properties result in a new level of performance especially for dielectric spectroscopy applications which are most demanding to measurement equipment. Especially low loss dielectrics and samples with thermally activated isolator conductor transition can be characterized over a broad frequency and temperature range which was not possible before the Alpha analyzer came to the market.

In addition, the typical functionality of a standard impedance analyzer for low impedance samples is supported. The Alpha analyzer is therefore not only the optimal tool for dielectric spectroscopy but due its exceptional overall performance used by many researches and engineers as an improved analysis tool for other applications, too.

Examples are sensor characterizations where small capacities have to be measured with high precision, analysis of semiconductors, electronic components, bio materials, proteins, organic tissues, cellulose and papers, conductive polymers and organics, ion conductors, fuel cells, power lines, transformer oils and self restoring fuses.

New Alpha-A series combines highest overall performance for both general purpose and special applications

Nevertheless, customers often demanded to combine the Alpha analyzer's exceptional overall performance with additional special functionality like e.g., extended voltage and current range, non linear analysis, high impedance 3- and 4-electrode measurement set-ups, fast measurement rates and DC measurement functionality including potentiostat and galvanostat control functions. From this, the problem arises that due to technical and economical limitations it is difficult to implement this functionality in a single instrument.

With the Alpha-A series which improves the performance of the Alpha analyzer introduced in 1998, Novocontrol now launches a new modular solution which implements all the special applications stated above. The new series is realized as modular measurement system based on an Alpha-A analyzer mainframe unit which can be

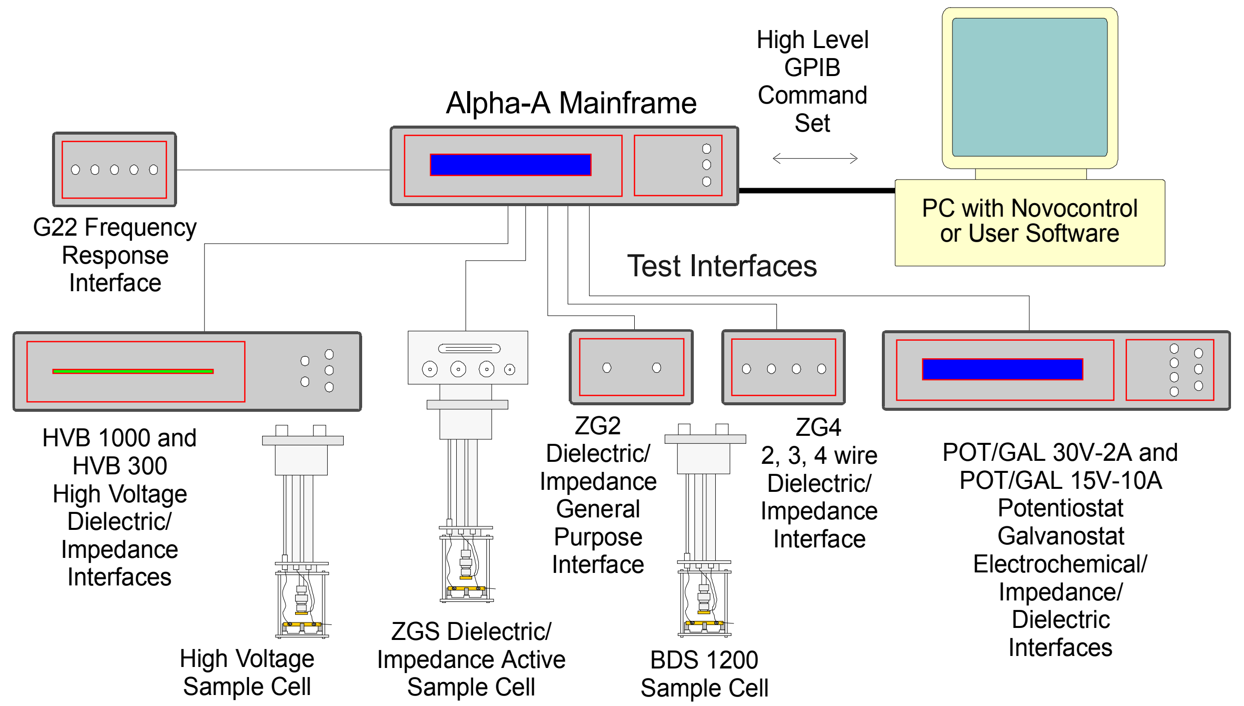


Fig.1. Alpha-A modular series with mainframe and available test interfaces optimized for special applications.

connected to a series of test interfaces which are optimized for special functionality. In contrast to many other solutions, all test interfaces are not limited to special functionality but offer highest accuracy, wide impedance and frequency range, too. With this, all interfaces are well suited for a wide range of material measurements from conductors to isolators.

This article gives an overview on the new features of the Alpha-A series with the focus on the test interfaces and some basic applications. A summary including main functions and key specifications can be found in tab. 1 at the end of this article. The basic concepts, features and principles of operation of the Alpha-A series and the economical Alpha and Beta series can be found in the Novocontrol technical brochure "Alpha-A, Alpha and Beta High Performance Dielectric, Conductivity and Electrochemical Impedance Analyzers".

Extended voltage and current ranges

The two high voltage interfaces HVB 300 and HVB 1000 produce output voltage up to ±150 V_P or ±500 V_P respectively. For high current applications, the two POT/GAL 30V-2A with ±2 A and POT/GAL 15V-10A with ±10 A are available. Within the stated limits any combination of AC and DC voltage or current is supported.

Extended output signals maybe advantageously applied for

- non linear dielectric, conductivity and impedance spectroscopy;
- test of materials under stress;
- evaluation of extreme high (high voltage) or low impedance (high current) materials;
- special technical materials like e.g. fuel cell characterization with the POT/GAL 15V-10A interface.

Non linear dielectric, conductivity and impedance spectroscopy

With the new Alpha-A series, Novocontrol introduces for the first time a turnkey commercial solution for non linear dielectric, conductivity and impedance spectroscopy. For this purpose, the Alpha-A mainframe and all test interfaces (except G22) support measurements of the sample voltage and current in terms of DC components, harmonic base waves and higher harmonics up to the interface high frequency limit.

Non linear evaluation is fully supported by the Novocontrol WinDETA software, which reads and graphically displays all voltage and current base, and higher harmonic components. In addition, other parameters like DC material parameters, linear impedance, permittivity, conductivity and the corresponding higher harmonic terms are processed.

The higher harmonic current components $I^*_n(\omega)$ are calculated by the Alpha analyzer like the base wave (n=1) by complex Fourier Transform from the sampled current *i*(*t*), where *n* denotes harmonic n-1

$$I^*_n(\omega) = \frac{2}{nT}\int_0^{nT} i(t)\,\exp(jn\omega t)\,dt \cdot$$

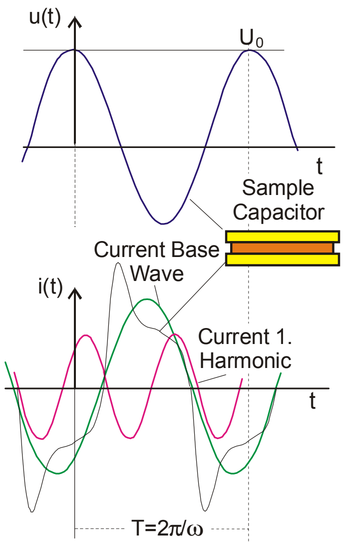


Fig. 2. Sample voltage with non linear response current, current harmonic base wave and first harmonic.

Electrode / interface polarization effects

In a measurement of a material prepared between parallel plate electrodes with spacing *d*, the complex permittivity or conductivity is evaluated from the phase sensitive measurement of the electrodes voltage difference U_S and current I_S. This method presumes that the applied voltage U_S drops homogene-ously within the material by creating a constant electric field $E=U_S/d$.

This presumption does not hold if at the electrode sample interface electrical inhomogeneous layers exist which create a significant voltage drop and by this reduce the E-field in the sample. Such layers may be created by

- accumulation of ions at the electrode for ion conductors like e.g. electrolytes and many liquids (water, electrode polarization);
- bad electrode sample contact connections (contact impedance).

Similar effects may be created by none zero impedance of the cables from the electrodes to the analyzer voltage and current terminals for low impedance samples.

These effects can be in principle cancelled out if separate electrodes for the sample current and voltage measurement are used as shown in fig. 3.

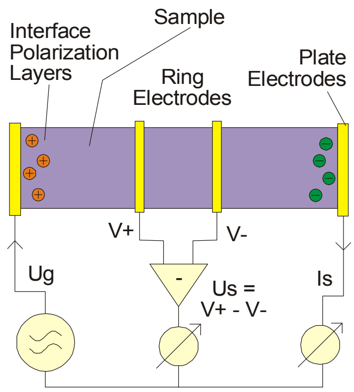


Fig. 3. Principle set-up of a four electrode impedance measurement for electrode - sample interface and cable effects compensation.

The outer two electrodes correspond to the parallel plates of a standard 2-electrode configuration. The voltage is measured by two additional (e.g. ring or needle) electrodes in the inner sample area where no interface effects exist and the field is homogenous.

If the two voltages are measured by an instrument with *infinite input impedance*, no current flows in the voltage electrodes and therefore no ions can accumulate. Due to the zero current, existing contact or cable impedance do not create a voltage drop, too. In this case, the electrical parameters of the material portion between the voltage electrodes can be evaluated without interface polarization and contact or cable effects from the voltage difference between the voltage electrodes and the current flowing through the outer electrodes. The only, but in practice crucial and not always fulfilled presumption is the sufficient high input impedance of the voltage channels.

The ZG4 and POT/GAL interfaces can in addition to 2-wire- be configured for 3-, or 4-wire measurements. Their input impedance is higher as $10^{12}\,\Omega$ in parallel with 10 pF which exceeds the range of most competing instruments by several orders of magnitude and therefore can be seen as a major improvement in broadband 3- and 4-electrode dielectric, conductivity and impedance measurements.

On the other hand, especially for high impedance samples at high frequencies the 10 pF input capacity may be in the same order of magnitude as the sample capacity and may contribute to the measured result. 3- and 4-four electrode measurements therefore in practice always require a detailed analysis of the currents flowing into the voltage terminals and the related voltage drop at contacts or electrode interfaces.

Electrochemical characterizations

3- and 4-electrode arrangements are often used for electrochemical studies too, where one is not so much interested in the bulk electrolyte but especially in the properties of the polarization layer at the metal to electrolyte or ion conductor interface and the related chemical reactions. This is just the opposite to dielectric, conductivity and impedance material spectroscopy where these effects are known as electrode polarization which is unwanted and tried to avoid by e.g. 4-electrode arrangements and special cells.

The measurement set-up for a typical electrochemical 3-electrode measurement is shown in fig. 4. In addition to the two parallel plate electrodes (denoted as Counter and Working electrode), a third voltage reference electrode is placed close to the polarization layer and measures the voltage difference of the polarization double layer capacity to the working electrode. In contrast to material spectroscopy where all electrodes are made of inert metal as e.g. gold, stainless steel or platinum, this applies for the electrochemical cell only for the counter electrode feeding current into the electrolyte.

The working electrode consists of the metal to be characterized in combination with the electrolyte. The reference electrode is usually an open tipped glass capillary filled with a standard electrolyte coupled to a standard metal in order to create a defined electrochemical potential to the electrolyte.

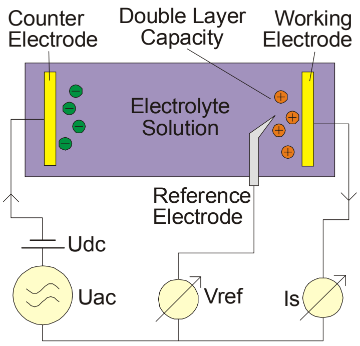


Fig. 4. Principle set-up of a three electrode electrochemical cell for impedance measurement.

The total potential drop across the cell is summed up by all contributions of the chemical process like mass transport, chemical and adsorption steps, electron transfer, etc [2]. By measuring the impedance spectrum $V_{Ref}^{*}(\omega)/I_S^{*}(\omega)$ and fitting it with an equivalent circuit model, the several process contributions can be separated from each other. The typical evaluation includes determination of Warburg impedance related to mass transport, electron transfer resistance, electrolyte resistance and double layer capacity [2].

As on the working electrodes an electrochemical reaction takes place, it is necessary to keep the DC potential V_{Ref} at a defined value or alternatively apply a constant DC current to the cell. This can be done by a Potentiostat / Galvanostat DC circuit as shown in fig. 5. The voltage amplifier connected to CE electrode compares in potentiostat mode the differential voltage $V_{Prc} = V_{RE+} - V_{RE-}$ of both reference electrodes with the intended voltage V_{setDC}. The amplifier adjusts its output voltage until V_{Prc} and V_{setDC} match resulting in a constant and sample impedance independent reference voltage differential which can be adjusted by Vsetdc. In galvanostat mode V_{Prc} is created proportional to the measured cell current by a current to voltage converter (V<-I), resulting in constant sample cell current. In both modes, the variable capacitor C_t adjusts the control loop time constant in order to avoid free high frequency oscillations caused by too high open loop gain.

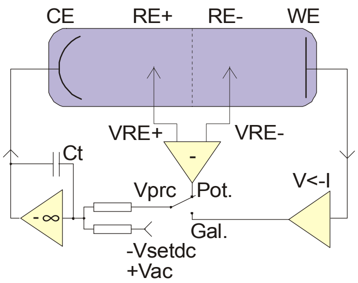


Fig. 5. Principle of Potentiostat / Galvanostat circuit with differential reference voltage inputs.

For impedance measurement an additional small AC voltage V_{AC} is superimposed to V_{setDC} and the AC response is measured as according to fig. 4.

The two new POT/GAL interfaces are optimized for impedance measurements of electrolytes with superimposed controlled voltage or current as described above. The POT/GAL 15V-10A provides up to ±10 A output current and with this is especially suited for fuel cell characterization. POT/GAL 30V-2A is a high quality general purpose instrument. In addition to DC control, the DC voltages and current at the four electrodes are continuously measured and displayed.

In contrast to existing potentiostats /galvanostats for impedance measurement, the POT/GAL interfaces are not restricted to electrolyte characterization but can be used for nearly all other kind of materials, too. E.g. both interfaces support a reference capacitor technique for accurate and broadband measurement of high impedance low loss isolators.

For samples not requiring controlled DC signals, beside the potentiostat and galvanostat a third direct voltage mode is included which bypasses the control amplifier and allows operation like a standard dielectric, conductivity and impedance analyzer with extended current and voltage range. As a further improvement and new feature, the bandwidth for impedance measurement is not limited by the selected time constant of the DC control amplifier (fig. 5). Instead for any DC bandwidth like e.g. 1 Hz, the full AC impedance frequency range up to 1 MHz is available in both DC controlled modes. Last but not least, both POT/GAL interfaces support fast continuously adjustable simultaneous voltage and current limits of the power amplifier output implemented independent from the DC control loop, working even in the case of oscillation or reference electrode cable damage in order to protect sensitive samples.

Characterization of fast time dependent processes

One major area of dielectric, conductivity and impedance spectroscopy is monitoring of time variant samples, which is usually due to chemical reactions or crystallization. Examples are time dependent measurements of e.g. epoxy curing or material ageing. Until now, most dielectric analysis systems had data rates in the range of several seconds. This limit is significantly reduced by the new Alpha-A mainframe which is available with a fast data rate option and can measure (and continuously send via the GPIB bus) up to 157 impedance data points per second. This allows process monitoring on time scales below 10 ms. The fast data rates are supported for all test interfaces, but not available for all operation modes like e.g. reference measurement mode or at frequencies < 200 Hz.

For the mainframe standard version (without the fast option) the maximum data rate is up to 10 data points per second.

Two electrode standard configurations

For most dielectric, conductivity and impedance measurements polarization or contact effects as discussed above are less prominent. In this case, the standard configuration with the sample material prepared between two parallel plate electrodes is most advantageous. This arrangement is most simple and allows easy and flexible sample preparation. Nevertheless, the design requirements to an appropriate sample cell operating over a wide frequency, impedance and temperature range without sacrificing the Alpha analyzer's high accuracy are extraordinary demanding.

Such a sample cell in realized as a part of the ZGS interface which is the successor of the popular Alpha-S analyzer active sample cell [1]. In addition to the sample cell, ZGS includes an impedance converter electronics and reference capacitors in the cell head. This arrangement avoids cables in the impedance path between the sample electrodes and analyzer input terminals. Cables may limit the usable high frequency range due to inductance and contribute low frequency noise for high impedance samples.

In contrast to other interfaces and solutions from other manufactures, ZGS is specified at the sample electrodes position and therefore users do not have to care about artefacts by cell contributions to the measured results. The ZGS interface is therefore the optimal and turnkey solution for dielectric, conductivity and impedance material measurements not requiring three or four electrode configurations.

The ZG2 interface contains the same impedance converter and reference capacitors as ZGS, but no integrated sample cell. ZG2 can be connected by BNC terminals to any passive sample cell like e.g. Novocontrol BDS 1200 or any impedance under test. Compared to ZGS, the combination ZG2 + BDS 1200 is somewhat more flexible but not as accurate due to the additional cables between BDS 1200 and ZG2.

New test interface concept improves flexibility and performance

For the test interfaces a new system concept is realized combining the advantages of a stand alone dielectric, conductivity and impedance analyzer with the specialized functionality of traditional combined analyzer - interface combinations.

Compared to traditional set-ups the new Alpha-A series has a different system connection topology starting with one out of a series of interfaces which is connected to the Alpha-A mainframe. The mainframe operates the entire system including low level interface control and high level result evaluation. For this purpose the mainframe is operated by a PC via a set of high level GPIB commands in a flexible and simple fashion.

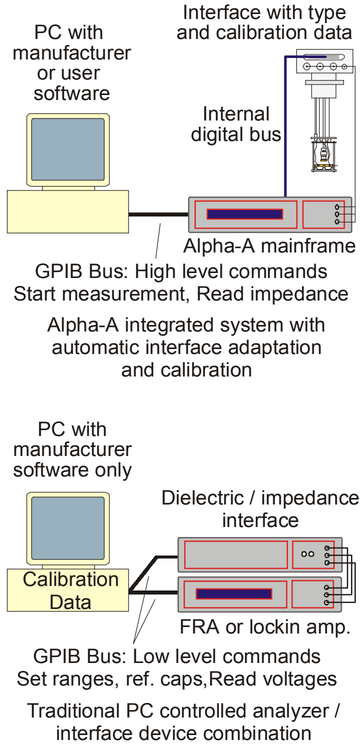


Fig. 6. System topology for the Alpha-A series and a traditional PC - FRA - interface system

E. g. a typical command sequence could be: adjust AC voltage and frequency, start measurement, read result as complex impedance or capacity. Depending on the interface type, the mainframe automatically adapts its functionality and command set. E.g. the voltage adjust command will accept voltages up to ±500 V for a HVB 1000 high voltage interface but only ±40V for a ZG2 interface, or special commands for DC polarization control will be available for the POT/GAL interfaces.

The adaptive interface concept allows

- fast low level data exchange and control between the Alpha mainframe and the interface;

- simple and straightforward integration of the Alpha-A system into own written control software or system control packages of other manufacturers.

Though the Alpha-A and all interfaces except G22 are completely integrated in the Novocontrol WinDETA software, operation by other software may be required if the analyzer shall be operated in combination with other equipment (e.g. a synchrotron ring).

This option is an advantage over traditional device combinations for dielectric, or electrochemical impedance measurements, where a frequency response analyzer and an interface need to be controlled by a dedicated PC software and the system can not operate without this software as shown in fig. 6.

Frequency response, gain phase measurements

In impedance mode used for dielectric, conductivity and impedance spectroscopy the Alpha analyzer measures the response voltage and current of a material sample to an applied sine wave signal.

In gain phase mode, a second voltage is measured instead of the current. This allows to measure two response voltages to an applied sine wave driver signal at two arbitrary points of a system under test. The two voltages are measured with the Alpha frequency response analyzer channels CH1 and CH2. The applied sine wave is created by the Alpha sine wave generator.

Gain phase measurements are supported by the Alpha-A mainframe standalone or in combination with any test interface except the two HVB which are for impedance mode only. The G22 interface is for gain phase measurements only and optimized for those applications.

A typical application would be to e. g. measure the transfer function of an amplifier or transformer. In this case, the Alpha generator output and input CH1 have to be connected to the amplifier input and CH2 to the amplifier output. The amplifier complex transfer function defined by V_{out}^*/V_{in}^* is determined by measuring the voltages at CH2 and CH1.

It should be noted, that in gain phase mode the Alpha can be operated like a digital lock-in amplifier with two input channels. The main function of a lock-in amplifier is to apply a signal with a defined frequency to a system under test and to measure a response signal. As a further lock-in amplifier feature, only the frequency component of the applied generator signal is detected in the response signal. As usually most of the response signals noise and DC errors are at other frequencies, those will therefore be suppressed. The Alpha uses the same principle, but has in addition a second voltage channel, wider bandwidth and better accuracy as lock-in amplifiers.

Gain phase measurements are therefore not specially related to dielectric, conductivity and impedance spectroscopy but may be of general interest if

- a very small or noisy signal has to be measured with high precision;
- the response of any kind of system to an applied signal in the frequency domain has to be measured.

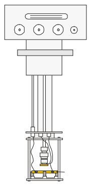
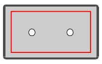
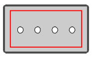
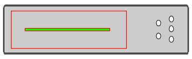
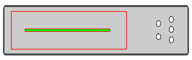
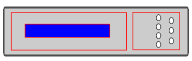
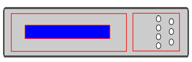
References

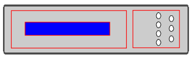
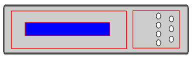
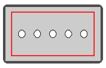
[1] G. Schaumburg, Novocontrol, Dielectrics Newsletter, Issue May 1999

[2] Electrochemistry, Carl H. Hamman et. al, Wiley-VCH, 1998, ISBN 3-527-29096-6

Gerhard Schaumburg
Novocontrol Technologies
GmbH & Co. KG

Test Interfaces Overview and Key Specifacitons

Test Interface	Especially Recommended	Special Features	Key Specification
ZGS 	Dielectric or conductive samples which can be prepared between parallel plate electrodes like e.g. polymers, semiconductors, glasses, and liquids or powders with not to high ion conductivity (electrode polarization). Optimized for broadband measurements of low loss dielectrics over a huge temperature range. Most accurate and straightforward solution for parallel plate electrode configurations.	Included active parallel plate sample cell, specification includes cell effects.	ZGS specs. apply at sample cell electrode position F 3 µHz - 20 MHz Z_R 10 mΩ - 10^{14} Ω tan(δ) $3 \cdot 10^{-5}$; Pa 2 m° V_{AC} ±4,2 V_P V_{DC} ±40 V_P, 70 mA max Z_O 50 Ω
ZG2 	General purpose interface for 2-wire measurements of • dielectric or conductive material samples in combinations with the Novocontrol BDS 1200 parallel plate sample cell; • passive customer own made sample cells; • electronic components.	Fixed 2-wire measurement configuration. Economical version of ZG4 without differential voltage inputs for 3- or 4-wire measurements.	F 3 µHz - 20 MHz Z_R 10 mΩ - 10^{14}Ω tan(δ) $3 \cdot 10^{-5}$; Pa 2 m° V_{AC} ±4.2 V_P V_{DC} ±40 V_P, 70 mA max Z_O 50 Ω
ZG4 	Dielectric or conductive samples with • significant electrode contact impedance or electrode sample interface polarization like e.g. electrolytes, liquids with ion conductivity (e.g. water based); • low impedance samples below 1 □like e.g. strong electrolytes, heavily doped semiconductors, metals, superconductors; • electronic components or networks.	Selectable 2-, 3- or 4-wire measurement configuration with additional high input impedance and driven shielded V_{high} and V_{low} differential voltage inputs.	F 3 µHz - 20 MHz Z_R 1 mΩ - 10^{14} Ω tan(δ) $3 \cdot 10^{-5}$; Pa 2 m° V_{AC} ±4.2 V_P V_{DC} ±40 V_P, 70 mA max Z_O 50 Ω Z_{VI} 10^{12} Ω ‖ 10 pF
HVB300  **HVB1000** 	Dielectrics, semiconductors or electronic components at high AC and/or DC voltages: • non linear dielectric/impedance spectroscopy • characterization of materials or components under stress; • extreme high impedance samples exceeding 10^{14} Ω̃ Optimized for broadband high voltage measurements of low loss dielectrics. Operation with Novocontrol High Voltage Sample Cell recommended.	High AC and/or DC output voltage. Protected against sample breakdowns and permanent shorts.	Z_R 1 Ω - 10^{15} Ω tan(δ) $3 \cdot 10^{-5}$; Pa 2 m° **HVB300** F 3 µHz - 1 MHz $V_{AC}+V_{DC}$ ±150 V_P 70 mA max Z_O 200 Ω **HVB1000** F 3 µHz - 10 kHz $V_{AC}+V_{DC}$ ±500 V_P, 3.3 mA max Z_O 150 kΩ
POT/GAL 30V-2A  **POT/GAL15V-10A** 	• Electrochemical cell reactions, metal electrolyte interfaces, ion conductors , conductive liquids for both low and high impedance samples up to 10^{13} Ω; • general purpose low impedance samples or electronic components at high currents. Optimized for low impedance samples with strong electrode sample interface polarization effects.	High power superimposed fixed or controlled (Potentiostat) dc voltage or constant DC current (Galvanostat). 2-, 3-, or 4-wire input configuration with driven shields. Simultaneous measurement of DC voltage and current at the four sample terminals. Continuously adjustable output voltage and current limit.	F 3 µHz - 1 MHz tan(δ) 10^{-4}; Pa 6 m° Z_R 1 mΩ - 10^{13} Ω **POT/GAL 30V-2A** $V_{AC}+V_{DC}$ ±30 V_P, ±2 A_P Z_O 1 Ω - 1 kΩ Z_{VI} 10^{12} Ω ‖ 10 pF **POT/GAL 15V-10A** $V_{AC}+V_{DC}$ ±15 V_P, ±10 A_P Z_O 0,1 Ω - 1 kΩ Z_{VI} 10^{12} Ω ‖ 10 pF

Test Interface	Especially Recommended	Special Features	Key Specification
POT/GAL 30V-2A  **POT/GAL15V-10A** 	• Electrochemical cell reactions, metal electrolyte interfaces, ion conductors , conductive liquids for both low and high impedance samples up to $10^{13}\ \Omega$; • general purpose low impedance samples or electronic components at high currents. Optimized for low impedance samples with strong electrode sample interface polarization effects.	High power superimposed fixed or controlled (Potentiostat) dc voltage or constant DC current (Galvanostat). 2-, 3-, or 4-wire input configuration with driven shields. Simultaneous measurement of DC voltage and current at the four sample terminals. Continuously adjustable output voltage and current limit.	F 3 µHz - 1 MHz tan(δ) 10^{-4}; Pa 6 m° Z_R 1 mΩ - 10^{13} Ω **POT/GAL 30V-2A** $V_{AC}+V_{DC}$ ±30 V_P, ±2 A_P Z_O 1 Ω - 1 kΩ Z_{VI} 10^{12} Ω ‖ 10 pF **POT/GAL 15V-10A** $V_{AC}+V_{DC}$ ±15 V_P, ±10 A_P Z_O 0,1 Ω - 1 kΩ Z_{VI} 10^{12} Ω ‖ 10 pF
G22 	High accuracy frequency response and gain phase measurements. Measures the amplitudes and phase shift $V1_{high}$-$V1_{low}$ and $V2_{high}$-$V2_{low}$ of two voltage channels. Similar functionality to a double input channel lockin amplifier with improved accuracy.	Two high sensitive voltage channels with high impedance driven shielded differential inputs. Not for impedance measurements.	F 3 µHz - 20 MHz Pa 10 m° V_{AC} ±4.2 V_P V_{DC} ±40 V_P, 70 mA max Z_O 50 Ω Z_{VI} 10^{12} Ω ‖ 10 pF

Tab. 1.
Alpha-A analyzer test interfaces overview. All test interfaces except G22 support automatic reference capacitor measurements and are suited for both dielectric and conductive samples but optimized for special applications. The Alpha mainframe standalone and all interfaces except HVB support two channel high accuracy frequency response or gain phase measurements.

Ranges:
F: Frequency, Z_R: Impedance, V_{AC}: AC output voltage, V_{DC}: DC output voltage, $V_{AC}+V_{DC}$: Any combination of |AC output voltage| + |DC output voltage|

Absolute Accuracies: tan(δ): loss factor, Pa: Phase angle

Other: Z_{VI}: Differential voltage terminals input impedance, Z_O: Power amplifier output impedance

NOVOCONTROL Technolgies GmbH & Co. KG

Obererbacher Strasse 9
D-56414 Hundsangen
Germany

Phone: ++(0)6435 - 96230
Fax: ++(0)6435 - 962333

Mail: novo@novocontrol.com
WWW: http://www.novocontrol.com