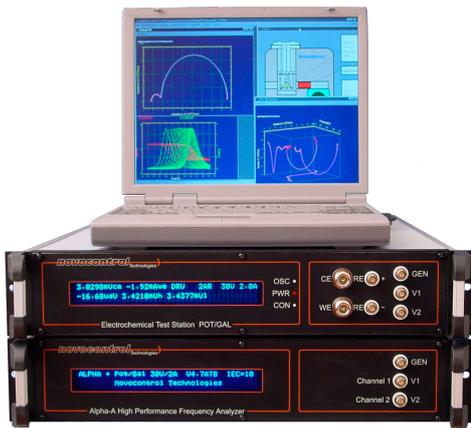


Test Interfaces for the Alpha-A Modular Measurement System

- Concepts
- Features
- Principles of Operation
- Application Examples



Introduction

When Novocontrol introduced the Alpha analyzer, it was soon accepted 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, easy to use instrument [1].

These properties represented a new level of performance, in particular for dielectric spectroscopy with its extraordinary requirements, e.g., an impedance range up to 100 TΩ and loss factor accuracy down to $3 \cdot 10^{-5}$. Over a broad frequency and temperature range, the Alpha enables, e.g., the complete characterisation of low-loss dielectrics and samples with thermally

activated isolator—conductor transition, a difficult if not impossible task before the Alpha analyzer came to the market. The device not only excels in its core area of dielectric spectroscopy but also acts as a standard impedance analyzer in the low impedance range. Due to its exceptional overall performance, the Alpha analyzer is not only the optimal tool for dielectric spectroscopy but an improved analysis tool for all kinds of electric characterisations, like, e.g.,

- sensor characterizations (high precision measurements of small capacities)
- 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
- self restoring fuses.

Alpha-A: highest overall performance for both general purpose and special applications

Nevertheless, customers frequently demanded to combine the Alpha analyzer's exceptional overall performance with additional special functionality like, e.g., extended voltage and current ranges, non-linear analysis, high impedance three- and four-electrode measurements, higher data acquisition rates and dc measurement options, including potentiostatic and galvanostatic control functions. For both technical and eco-

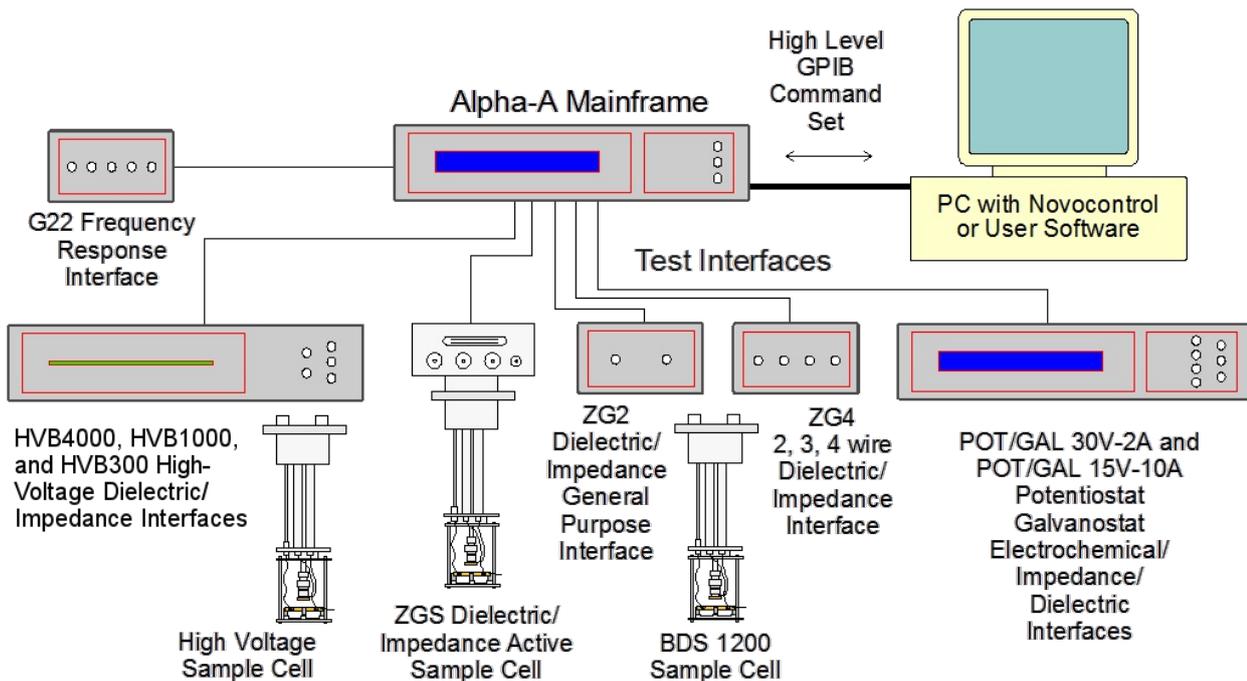


Fig.1. Alpha-A modular series with mainframe and available test interfaces optimized for special applications.

nomical reasons, however, it is difficult to implement all these particular options in one single instrument.

The Alpha-A series not only improves the performance of the earlier introduced Alpha analyzer, but also gives access to all the special applications mentioned above. The new series is realized as a modular measurement system based on an Alpha-A analyzer mainframe unit, connected to a series of test interfaces (one at a time) which are optimized for special functions. In contrast to many other solutions, the test interfaces are not limited to a particular functionality but offer highest accuracy, wide impedance and frequency range, too. As a consequence, all test interfaces are well suited for a wide range of material measurements from conductors to isolators.

This article provides an overview of the new features of the Alpha-A series, focussing 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 high voltage interfaces HVB 300, HVB 1000, and HVB 4000 produce output voltages up to $\pm 150 V_p$, $\pm 500 V_p$, and $\pm 2000 V_p$, respectively. For high current applications, the two POT/GAL 30V-2A with $\pm 2 A$ and POT/GAL 15V-10A with $\pm 10 A$ are available. Within the stated limits, any combination of ac and dc voltage or current is supported. Potential applications of such extended output signals are

- non linear dielectric, conductivity and impedance spectroscopy;
- test of materials under stress;

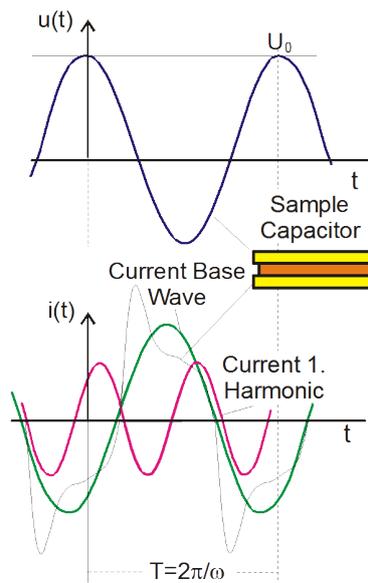


Fig. 2: Sample voltage with non-linear response current, current harmonic base wave and first harmonic.

- evaluation of extreme high or low impedance materials, using high voltages or currents, respectively;
- special technical materials like e.g. fuel cell characterization with the POT/GAL 15 V-10 A interface.

Non linear dielectric, conductivity and impedance spectroscopy

With the new Alpha-A series, Novocontrol introduced the first 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 cor-

$$I_n^*(\omega) = \frac{2}{nT} \int_0^{nT} i(t) \exp(jn\omega t) dt$$

responding higher harmonic terms are processed.

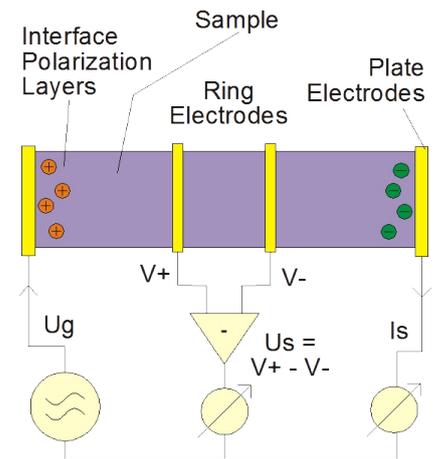


Fig. 3: Principle set-up of a four electrode impedance measurement for electrode-sample interface and cable effects compensation.

responding higher harmonic terms are processed.

Base wave $I_n^*(\omega)$ and higher harmonic current components $I_{n>1}^*(\omega)$ are calculated by complex Fourier Transform

from the sampled current $i(t)$, where n denotes harmonic $n-1$.

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 assumes that the applied voltage U_s drops homogeneously within the material by creating a constant electric field $E=U_s/d$.

This assumption, however, does not always hold. Electrically inhomogeneous layers at the sample-electrode interface may create a significant voltage drop and thus reduce the effective electric field in the sample. Such layers may be due to, e.g.,

- 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 when measuring low-impedance samples due to non-negligible impedance of

cables connecting the electrodes to the analyzer voltage and current terminals, respectively.

These effects may be compensated by using separate electrodes for the sample current and voltage measurements, respectively, as shown in Figure 3.

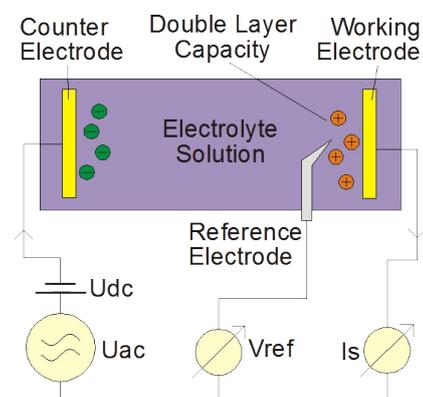


Fig. 4: Principle set-up of a three electrode electrochemical cell for impedance measurement.

The outer two electrodes correspond to the parallel plates of a standard two-electrode configuration. The voltage, however, is measured by two additional (e.g. ring or needle) electrodes in the inner sample area where the electric field is homogenous since there are no interface effects present.

If the two voltages are measured by an instrument with infinite input impedance, no current flow appears in the voltage electrodes, and thus no ions can accumulate. Due to the negligible current, contact or cable impedance contributions cannot create a voltage drop as well. The electrical parameters of the material portion between the voltage electrodes can thus be determined without contributions from interface polarization and contact or cable effects, simply by measuring the voltage between the inner electrodes and the current flowing through the outer electrodes. The only, but in practice crucial and rarely well fulfilled presumption is the sufficiently high input impedance of the voltage channels.

In addition to the standard two-wire set-up, the ZG4 and POT/GAL interfaces can be configured for three-, or four-wire measurements. Their input

impedance is higher than $10^{12} \Omega$ parallel to 10 pF. These parameters exceed the range of most competing instruments by several orders of magnitude and therefore must be considered as a major improvement in broadband dielectric, conductivity and impedance measurements using three- or four-wire configurations.

When measuring high impedance samples at high frequencies, however, the 10 pF input capacity may be of the same order of magnitude as the sample capacity and may thus contribute to the measured result. Three- and four-electrode measurements there-

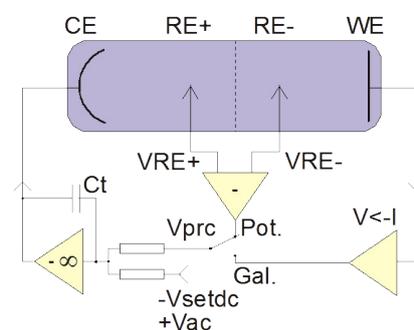


Fig. 5: Principle of Potentiostat / Galvanostat circuit with differential reference voltage inputs.

fore 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

Three- and four-electrode arrangements are often used for electrochemical studies as well. Here we are typically less interested in the bulk electrolyte but rather 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 of typical dielectric, conductivity and impedance material spectroscopy where these effects are considered as undesirable electrode polarization which can be suppressed by using, e.g., four-electrode arrangements and special cells.

The measurement set-up for a typical electrochemical three-electrode

measurement is shown in Figure 4. In addition to the two parallel plate electrodes (denoted as Counter and Working electrodes, respectively), 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.

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 using an equivalent circuit model, the several process contributions can be separated from each other. The typical evaluation includes determination of a 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 achieved by a Potentiostat/Galvanostat dc circuit as shown in Figure 5. In potentiostat mode, the voltage amplifier connected to the CE electrode compares 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 V_{setdc} . 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.

For impedance measurement an additional small AC voltage V_{ac} is superimposed to V_{setdc} and the ac response is measured according to Figure 4.

The two 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 thus is particularly well suited for fuel cell characterization. The 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 and 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 as well. Both interfaces, e.g., support the reference capacitor technique for accurate and broadband measurement of high impedance low loss isolators.

For samples not requiring controlled dc signals, a third direct voltage mode is included which bypasses the control amplifier and allows operation as 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. Finally, both POT/GAL interfaces provide sophisticated means of protecting sensitive (and po-

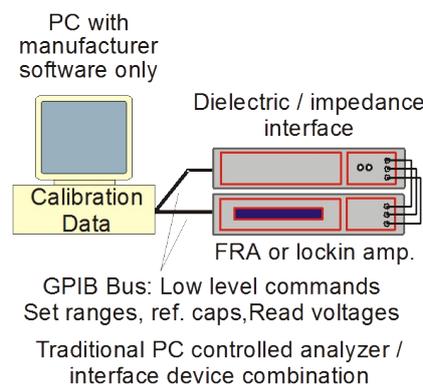
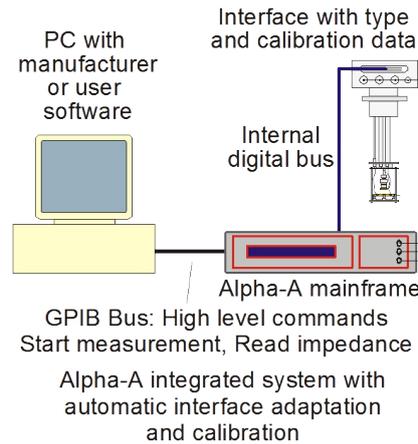


Fig. 6: System topology for the Alpha-A series and a traditional PC - FRA - interface system

tentially expensive) samples. This is accomplished by voltage and current limiters which are

- operating independently of the dc control loop
- fast and continuously adjustable
- operative even in case of oscillation or reference electrode cable damage.

Characterization of fast time-dependent processes

One major area of dielectric, conductivity and impedance spectroscopy is monitoring samples with time variant properties which are usually due to chemical reactions or crystallization. Examples are measurements of, e.g., epoxy curing or material ageing. Most dielectric analysis systems had data rates in the range of several seconds. This limit is significantly reduced by the new Alpha-A mainframe: its fast data rate option can measure 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 below 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 allows easy and flexible sample preparation. Nevertheless, the design requirements for a suitable sample cell, operating over wide frequency, impedance and temperature ranges without sacrificing the Alpha analyzer's high accuracy are extraordinary demanding.

Such a sample cell is 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, the ZGS houses an impedance converter and reference capacitors in its top part. 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, the ZGS is specified at the sample electrodes position: users do not have to care about artefacts by cell contributions to the measured results. The ZGS interface is thus the optimal and turnkey solution for dielectric, conductivity and impedance material measurements for two-electrode configurations.

The ZG2 interface contains the same impedance converter and reference capacitors as the ZGS, but no integrated sample cell. The ZG2 can be connected by BNC terminals to any passive sample cell like, e.g., the Novocontrol BDS 1200 or any impedance under test. Compared to the ZGS, the combination ZG2 + BDS 1200 is more flexible but not as accurate due to the additional cables between the BDS 1200 and the 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. To this end, the mainframe is operated by a PC via a set of high level GPIB commands in a flexible and simple fashion.

A typical command sequence could look like this:

- 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. The voltage adjust command, e.g., will accept voltages up to ± 500 V for a HVB 1000 high voltage interface but only ± 40 V for a ZG2 interface; 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;

- easy 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 is to be operated in combination with other equipment (e.g., at 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 Figure 6.

Frequency response, gain phase measurements

In impedance mode, typically used for dielectric, conductivity and impedance spectroscopy, the Alpha-A analyzer measures the voltage and response 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-A 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 both stand-alone or in combination with any test interface, except the three HVB test interfaces which are for impedance mode only. The G22 interface is for gain phase measurements only and optimized for those applications.

A typical application of a gain-phase measurement is, e.g., 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 or transformer input and CH2 to the amplifier or transformer output. The complex

transfer function defined by V_{out}^*/V_{in}^* is determined by measuring the voltages at CH2 and CH1.

It should be emphasized that in gain phase mode, the Alpha-A 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. Since most of the signal noise and DC errors appear at other frequencies, these contributions will be suppressed with very high efficiency. The Alpha-A uses the same principle, but offers an additional voltage channel, wider bandwidth and better accuracy than standard lock-in amplifiers.

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

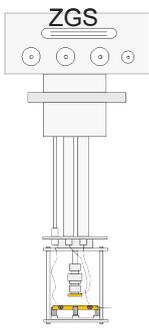
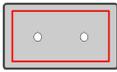
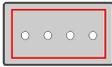
- a very small or noisy signal has to be measured with high precision;
- the response of any kind of system to an applied signal in the frequency domain has to be measured.

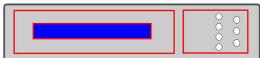
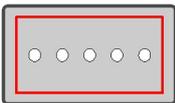
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Test Interfaces: Overview and Specifications

 <p>ZGS</p>	<p>Dielectric or conductive samples which can be prepared between parallel plate electrodes like, e.g., polymers, semiconductors, glasses, and liquids or powders with not too 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.</p>	<p>Included active parallel plate sample cell, specification includes cell effects.</p>	<p>F 3 μHz – 40 MHz Z_R 10 mΩ – 10^{14} Ω $\tan \delta$ $3 \cdot 10^{-5}$; Pa 2 m$^\circ$ V_{AC} $\pm 4.3 V_P$ V_{DC} $\pm 40 V_P$ 70 mA max Z_O 50 Ω</p>
<p>ZG2</p> 	<p>General purpose interface for 2-wire measurements of</p> <ul style="list-style-type: none"> dielectric or conductive material samples in combinations with the Novocontrol BDS 1200 parallel plate sample cell; passive customer own made sample cells; electronic components. 	<p>Fixed two-wire measurement configuration. Economical version of ZG4 without differential voltage inputs for two- or four-wire measurements.</p>	<p>F 3 μHz – 40 MHz Z_R 10 mΩ – 10^{14} Ω $\tan \delta$ $3 \cdot 10^{-5}$; Pa 2 m$^\circ$ V_{AC} $\pm 4.3 V_P$ V_{DC} $\pm 40 V_P$ 70 mA max Z_O 50 Ω</p>
<p>ZG4</p> 	<p>Dielectric or conductive samples with</p> <ul style="list-style-type: none"> significant electrode contact impedance or electrode sample interface polarization like, e.g., electrolytes, ion-conducting liquids (e.g. water based); low impedance samples below 1 Ω like, e.g., strong electrolytes, heavily doped semiconductors, metals, superconductors; electronic components or networks. 	<p>Selectable two-, three-, or four-wire measurement configuration with additional high input impedance and driven shielded V_{high} and V_{low} differential voltage inputs.</p>	<p>F 3 μHz – 40 MHz Z_R 0.1 mΩ – 10^{14} Ω $\tan \delta$ $3 \cdot 10^{-5}$; Pa 2 m$^\circ$ V_{AC} $\pm 4.3 V_P$ V_{DC} $\pm 40 V_P$ 70 mA max Z_O 50 Ω</p>
<p>HVB 300 HVB 1000 HVB 4000</p> 	<p>Dielectrics, semiconductors or electronic components at high AC and/or DC voltages:</p> <ul style="list-style-type: none"> non linear dielectric/impedance spectroscopy characterization of materials or components under stress; extreme high impedance samples exceeding 10^{14} Ω <p>Optimized for broadband high voltage measurements of low-loss dielectrics. Operation with Novocontrol High Voltage Sample Cell recommended.</p>	<p>High AC and/or DC output voltage. Protected against sample breakdowns and permanent shorts.</p>	<p>$\tan \delta$ $3 \cdot 10^{-5}$; Pa 2 m$^\circ$ HVB 300: F 3 μHz – 1 MHz Z_R 1 Ω – 10^{15} Ω $V_{AC}+V_{DC}$ $\pm 150 V_P$ 70 mA max Z_O 200 Ω HVB 1000: F 3 μHz – 10 kHz Z_R 100 Ω – 10^{15} Ω $V_{AC}+V_{DC}$ $\pm 500 V_P$ 3.3 mA max Z_O 150 kΩ HVB 4000: F 3 μHz – 10 kHz Z_R 1 kΩ – 10^{15} Ω $V_{AC}+V_{DC}$ $\pm 2000 V_P$ 2.7 mA max Z_O 750 kΩ</p>

Test Interface	Recommended Applications	Special Features	Key Specifications
<p>POT/GAL 30 V-2A</p>  <p>POT/GAL 15 V-10A</p> 	<ul style="list-style-type: none"> Electrochemical cell reactions, metal electrolyte interfaces, ion conductors, conductive liquids for both low and high impedance samples up to 1013 Ω; general purpose low impedance samples or electronic components at high currents. <p>Optimized for low impedance samples with strong electrode sample interface polarization effects.</p>	<p>High power superimposed fixed or controlled (Potentiostat) dc voltage or constant dc current (Galvanostat). Two-, three-, or four-wire input configuration with driven shields.</p> <p>Simultaneous measurement of DC voltage and current at the four sample terminals. Continuously adjustable output voltage and current limit.</p>	<p>F 3 μHz – 1 MHz tan δ 10⁻⁴; Pa 6 m° Z_R 1 mΩ – 10¹³ Ω</p> <p>POT/GAL 30V-2A V_{AC}+V_{DC} ±30 V_P, ±2 A_P Z_O 1 Ω– 1 kΩ Z_{VI} 10¹² Ω 10 pF</p> <p>POT/GAL 15V-10A V_{AC}+V_{DC} ±15 V_P, ±10 A_P Z_O 0.1 Ω– 1 kΩ Z_{VI} 10¹² Ω 10 pF</p>
<p>G22</p> 	<p>High accuracy frequency response and gain phase measurements. Measures the amplitudes and phase shift V1high-V1low and V2high-V2low of two voltage channels. Similar functionality to a double input channel lockin amplifier with improved accuracy.</p>	<p>Two highly sensitive voltage channels with high impedance driven shielded differential inputs. Not for impedance measurements.</p>	<p>F 3 μHz – 40 MHz Pa 10 m° V_{AC} ±4.3 V_P V_{DC} ±40 V_P 70 mA max Z_O 50 Ω Z_{VI} 10¹² Ω 10 pF</p>

Tab. 1: Alpha-A analyzer test interfaces overview. All test interfaces except the G22 support automatic reference capacitor measurements and are suited for both dielectric and conductive samples but optimized for special applications. The Alpha-A mainframe standalone and all interfaces except HVB support dual-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: tanδ: loss factor, Pa: Phase angle

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



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