ZIROX Sensoren und Elektronik GmbH

ZIROX Oxygen Measuring Technology

ZIROX Vacuum probe with electronics for oxygen measurements

XS22.xxH-xxx

Manual

ZIROX Sensoren & Elektronik GmbH
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1 Introduction

1.1 How to use this manual

This manual describes composition, mode of operation and use of the oxygen measuring system for vacuum processes (VMS) developed by ZIROX GmbH.

Address of manufacturer:

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The manufacturer guarantees that this manual was written in accordance with the functional and technical features of the delivered oxygen measuring system.

This manual is not subject to the amendment service. If the manufacturer modifies the VMS with the aim of making technical improvements, the user is responsible for inserting the additional or updated pages supplied.

Proper operation of the VMS can only be ensured if the contents of this manual are known. Therefore, all chapters of this manual must be read carefully prior to operating the VMS.

Pages, charts and figures are numbered consecutively.

1.2 Copyright

1.3

1.4 Commonly used symbols

Symbol for imminent danger:

This symbol refers to imminent danger to persons' life and health.

In case of disregard fatal injuries may result.

danger

Symbol for indirect danger:
This symbol indicates indirect danger.

The degree of the damage depends on the circumstances and the actions of the persons involved.

attention

In case of disregard, destruction or damage of the measuring probe, the electronics assembly or other material assets as well as minor injuries may result.

Symbol for proper handling:

This symbol appears where the manual refers to the adherence to rules, instructions and proper operation.

NOTE

In case of disregard, damage or destruction of the VMS or its single components may result.

1.5 Safety instructions

The following safety instructions inform about potential danger during the operation of the oxygen measuring system (VMS). They must be strictly followed by the responsible staff.

A failure-free and functional operating of the VMS can only be ensured with knowledge of this manual. Please read all the chapters of this equipment manual carefully before the installation and initiation.

attention

The VMS is to be used for the functional operation only (see 1.5).

The VMS is to be installed, operated and serviced by trained staff only.

Special safety instructions for potential danger in certain working processes are given in relevant text passages.

danger

1.6 Application of the measuring system

The measuring system is used for in situ measurements of oxygen in gases.

Typical examples are:

- Measurings in vacuum processes (PVC, CVD, other plasma processes)
- Measurings in process gases conducted under non-standard pressure (e.g. surface treatment processes)
- Measurings in science and research

The measuring gas can be temporarily reducing[1]. It must by no means represent an explosive compound since the probe with its heater works as an ignition source.

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[1] Different conditions of oxygen in the measuring gas must be distinguished:

Free oxygen: Oxygen molecules in the gas are independent without a bond to other gas components (inert gases such as N_2 or Ar). In combustion engines this is called “lean mixture”.

Bound oxygen: Free oxygen molecules do not exist in the gas, only in bound form e.g. as water vapor. Higher temperatures cause a dissociation and oxygen molecules are available. Since the dissociation degree increases with the temperature, the measurement result depends on the temperature. In combustion engines this is called “rich mixture”.

Possibly, free oxygen can react with potential burnable gases at the hot platinum electrode. The result can be a reducing gas.

A consultation with the manufacturer is required before measurings in corrosive gases, the danger of condensate formation or failure of carbon.

1.7 Warranty

ZIROX Sensoren & Elektronik GmbH warrants that the products manufactured and sold are free from manufacturing and material defects at the time of dispatch. In case of defects and faults within 12 months (probe) and 24 months (electronics assembly) respectively after dispatch, ZIROX will clear faults at its own option by repair or replacement. The purchaser must give prompt written notice to ZIROX. The purchaser is not entitled to claim other legal remedies based on this warranty.

The following warranty periods for ZIROX probes apply:

Operation temperature up to 1200°C	*12 months after delivery*
Operation temperature up to 1300°C (high temperature probe)	*6 months after delivery*
Operation temperature up to 1400°C (high temperature probe)	*3 months after delivery*

ZIROX does not warrant supplied products which are subject to normal wear and tear (e.g. reference gas pump).

Corrosive gases and solid particles may cause damage and require repair or replacement due to normal wear and tear.

The contact of the products with explosive gas mixtures, halogens in high concentrations and sulphuric gases (e.g. SO_2) is not permitted.

The contact of the products with siliconic or phosphoric compounds is not permitted either.

A connection of ZIROX and non-ZIROX products voids any warranty claims.

Warranty and liability claims for damage to persons and/or property are void if they are subject to the following:

- Normal wear and tear
- Improper use of the product
- Disregard of the manual’s instructions
- Improper installation, initiation, operation and maintenance of the product
- Operation of the product without protective measures
- Unauthorized functional and technical modification of the product
- Dismantling of parts as well as installation of spare parts or additional units which are not delivered or permitted by the manufacturer
- Improper repairs or faulty operation
- External impact
- Acts of God

• Overview of the measuring system

The measuring system serves the measuring of the oxygen partial pressure in vacuum. It consists of a probe and an integrated control and signal processing electronics. The probe can be directly installed in vacuum systems.

The probe is provided with a KF40/KF25 flange (others available on request).

The integrated electronics represents a minimal system. It merely arranges the heater regulation of the sensor element, its reference air supply and the voltage-/ current converter for the cell voltage. The measurement range can be converted from 400 mV to 1200 mV. In addition, a signal is provided which shows the stand-by of the probe (heater regulation is in steady state).

2 Probe

2.1 General description

The sensor element of the vacuum probe consists of a partially closed solid electrolyte tube. The reference electrode and a resistance heater are located inside the tube. The measuring electrode outside the solid electrolyte tube contains a thermocouple for the temperature regulation of the sensor element. The measuring electrode is on the outer face of the solid electrolyte tube and thus directly in the measuring gas area.

For normal use the probe is delivered with a shielding cylinder which has a flattening effect on the measuring signal on one hand and protects the measuring electrode from parasitical coating on the other hand. Only for measurings with an extremely high speed response the probe should be used without a shielding cylinder.

The connecting head of the probe contains the complete electronics with the reference gas pump.

In operating status the ceramic front heats up to a temperature of about 700 °C. Burn hazard!	⚠ danger

2.2 Construction and application of the probe

The following illustration shows the scale drawing of the probe:

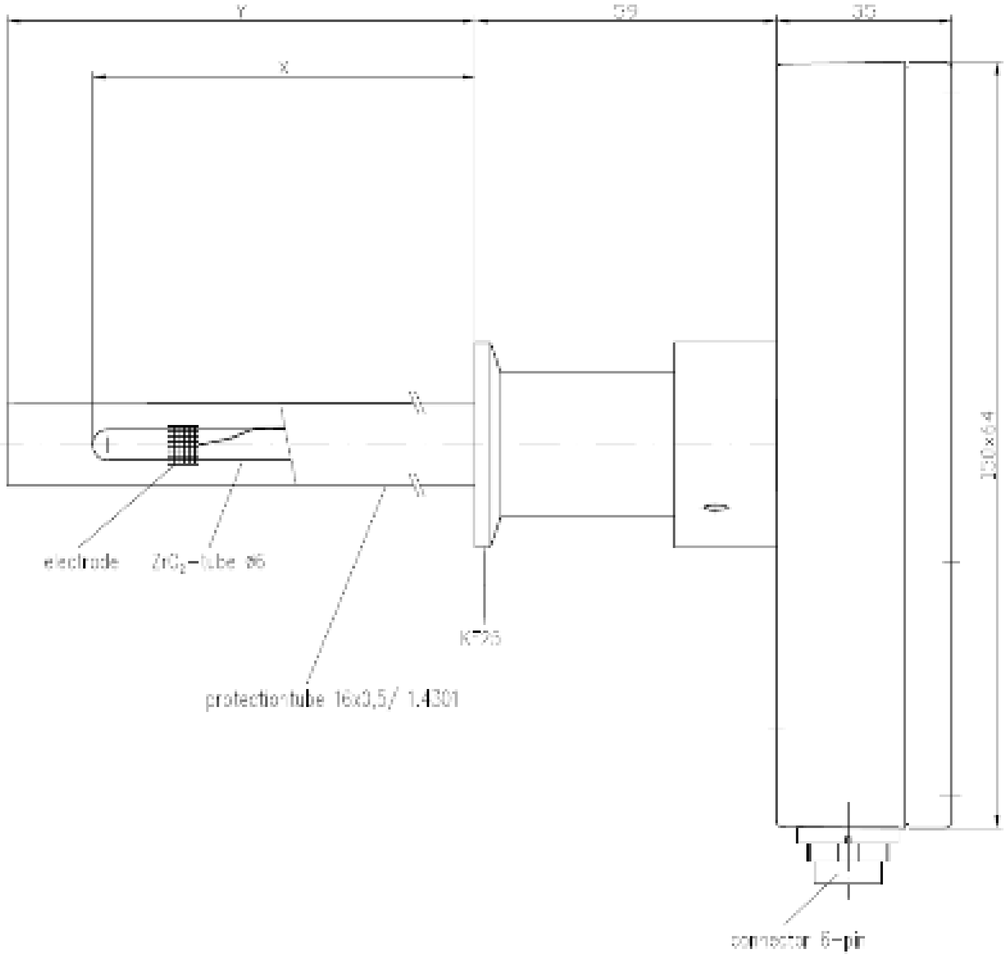


Figure 1: Scale drawing of the probe

The ZrO_2 sensor element is normally located under a removable shielding cylinder (shield tube). For high measuring dynamics the tube is removable. Handle the solid electrolyte tube with care. It is fragile. The measuring cell is heated inside the sensor tube by an electrical heater. The installed thermocouple measures the temperature of the measuring cell.

Unlike probes which are used under normal pressure, vacuum probes have to be calibrated. Reasons are the inside heater of the sensor tube used in this probe and the energy removal of the heated sensor element depending on the pressure and the ambient gases. Due to the lack of isothermal electrodes, the validity of the Nernst equation is not given. Usually, running the production unit with empirically determined sensor signals is sufficient for the process management.

For measurings of oxygen partial pressure the probe must be calibrated by means of test measurings. The cell voltage in ambiant air has to be determined under normal pressure. This value is the so-called asymmetry voltage. The asymmetry voltage has to be subtracted from the cell voltage occurring in other oxygen concentrations.

2.3 Technical data of the probe

Output range	0…10 V (XS22.5x) 0… 5 V (XS22.9x) 0… 20 mA (XS22.3x, XS22.7x)	To be specified in your order!
Pressure range	$1.5 \cdot 10^{5} \ldots 1 \cdot 10^{-5}$ Pa	1.5 bar…$1 \cdot 10^{-7}$ mbar
Measuring range	$1 \cdot 10^{5} \ldots 1 \cdot 10^{-21}$ Pa	
Diameter of the probe	6 mm	Additional protection tube (diameter 16 mm, others on request)
Length of the probe	140…300 mm	User-specific
Thermocouple	Type B	
Heater voltage	24 V	Approx. 30 W
Heater resistance	20 Ω	
Installation requirements	DN40KF (XS22.x2) DN25KF (XS22.x1)	To be specified in your order! Other flanges on request
Temperature of electrodes	700 °C	Controlled by internal electronics
Temperature at the installation flange	Approx. 60 °C	Dependent on installation situation and length of the sensor
He-leak rate	$<10^{-8}$ mbar l/s	
Reference air flow	5 ⋯ 10 l/h	Delivered by internal pump
Offset voltage with protection tube	-15...-20 mV	

3 Evaluation electronics

3.1 General description

The required electronics for the steady operation of the probe is located in the connecting head of the probe. The connecting head contains a diaphragm pump which supplies the reference electrode with air. The electronics ensures a steady sensor temperature of 700° C and converts the EMF of the sensor into an isolated output signal (e.g. 0-20 mA). The range of the cell voltage can be converted from 400 mV to 1200 mV.

Control elements on the printed circuit board:

- With the slide switch the maximum input voltage processed by the voltage/current converter can be converted from 400 mV to 1200 mV.
- The regulator (called offset) allows the tuning of the sensor's offset voltage in the range of ±50 mV, i.e. with this regulator the current output can be tuned at 0 mA.
- The regulator next to the pump engine (called pump) serves for the tuning of the pumping power.
- At the pin contacts (test point U) a voltage of 0-1 V can be measured which is proportional to the output signal.

The following visual signaling is provided:

- The red light-emitting diode signalizes a 24 V/DC operating voltage.
- The green light-emitting diode signalizes the operating temperature (700° C).

The electronics is protected by a resettable fuse.

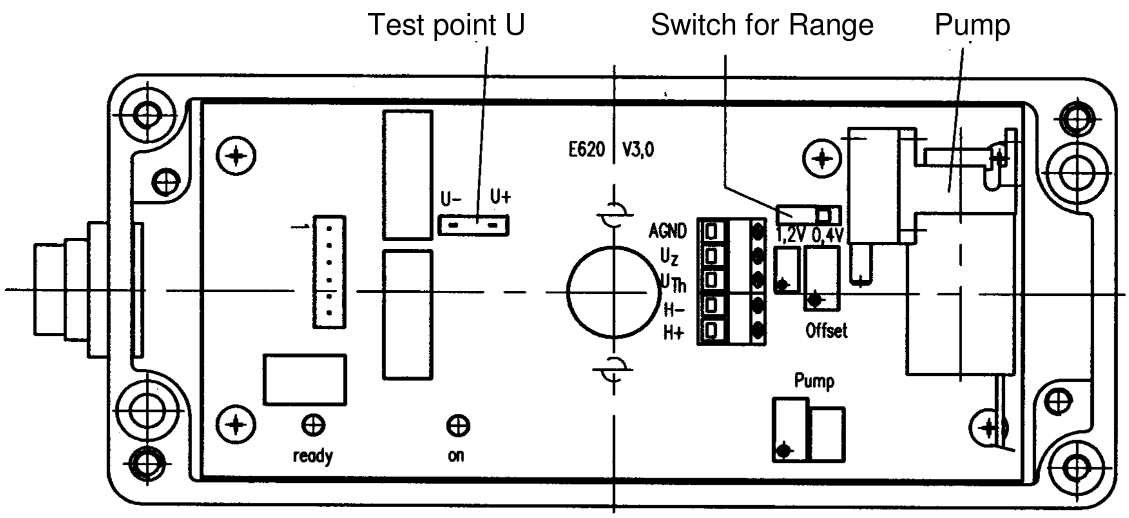


Figure 2: View of electronics E620

3.2 Technical data

Dimensions L x W x H	150 x 63 x 35 mm^3	
Operating voltage	24 V/DC ±10%	
Current consumption	1.2 A	
Electric capacity	Approx. 30 W	
Output signal	0 – 20 mA	0-10 V / 0-5 V on request
	400 or 1200 mV max. cell voltage	Change range via switch
Reference air flow	5 – 10 l/h	
Plug assignment: 1-pink 2-blue	 + 24 V GND	Options: Cable type: LIYD11Y 7 x 0.25 Type of plug: 423 6 pin (Binder) Order-No.: 99-5622-15-06
3-green 4-yellow	+ I_A - I_A	400 mV corr. 20 mA or 1200 mV corr. 20 mA Depending on range
5-brown 6-white	Ready contact (potential-free)	60 V 1 A/DC or 125 V 1 A/AC

4 Initiation

When the operating voltage is switched on, the heater needs approx. 5 minutes to heat up the sensor element to 700° C. The green LED signalizes that the temperature is reached. The current output delivers a signal corresponding to the cell voltage. Its accuracy, however, does not comply with the technical data. Only after additional 15 minutes, the thermal conditions are in steady state and the probe is ready for measuring.

In delivery condition all required reference values are adjusted. The measuring range is set at 400 mV. After a longer operation time the reference values should be checked.

Balancing the offset voltage: In a measuring range of 400 mV the output current has to be balanced to 0 mV by the regulator “offset”. The measuring electrode must be located in normal air with a reference air flow of 5 ··· 10 l/h.

Adjustment of the reference air flow: A flow meter has to be used between the pump and the probe. A flow rate of 5… 10 l/h has to be adjusted with the regulator “pump”.

5 Potential fault causes and fault clearance

The following list presents an overview of potential fault appearances. Faults not in the list require a consultation with the manufacturer.

Symptoms	Fault causes	Fault clearance
Red LED off (no supply voltage)	No supply voltage at the probe	Check power supply
	Internal fuse switched off	Send probe to service
Green LED off (no ready-signal)	Probe is too cooled by gas flow	Install probe flow-protected
	Heater of the probe defective	Send probe to service
	Thermocouple defective	Send probe to service
Output signal > range	Current / voltage transformer for cell voltage is overdriven	Convert amplifier to higher measuring range 1200 mV

6 Details for the use of ZrO_2 probes in vacuum technology

6.1 Theoretical relations

Potentiometric ZrO_2 probes measure the difference of the oxygen partial pressure at both electrodes.

One electrode is fixed inside, the other one outside the ZrO_2 tube. Air as reference gas flows around the inner electrode.

The NERNST equation applies:

$$U = \frac{RT}{4F} \ln \frac{p_{O_2}(air)}{p_{O_2}(measuring\ gas)}$$ (Nernst equation)

Condition: isothermal electrodes

U – cell voltage

R – molar gas constant, R = 8.31 J/(mol · K)

T – Kelvin temperature

F – Faraday constant, F = $9.64 \cdot 10^4$ C/mol

p_{O2} (air) – partial pressure of oxygen at reference electrode

p_{O2} (measuring gas) – partial pressure of oxygen at measuring electrode

After inserting the known values of the natural constants the following applies:

$$p_{O_2}(measuring\ gas) = p_{O_2}(reference\ gas) \cdot \exp[-46.42 \cdot \frac{U/mV}{T/K}]$$

$$p_{O_2}(measuring\ gas) = 0.2064 \cdot p_L \cdot \exp[-46.42 \cdot \frac{U/mV}{T/K}]$$

0.2064 refers to the oxygen concentration in air at 50 % relative humidity and p_L is the air pressure. The pressure unit of p_{O2} (measuring gas) and p_L can be chosen user-defined but must be identical for both pressures. The numerical value in this equation requires the probe signals U used in mV and T in Kelvin.

For the calculation of the oxygen concentration φ_{O2} in vacuum, which is the ratio of the number of oxygen molecules to the total of gas particles, the total pressure in the vacuum chamber is necessary.

The following applies:

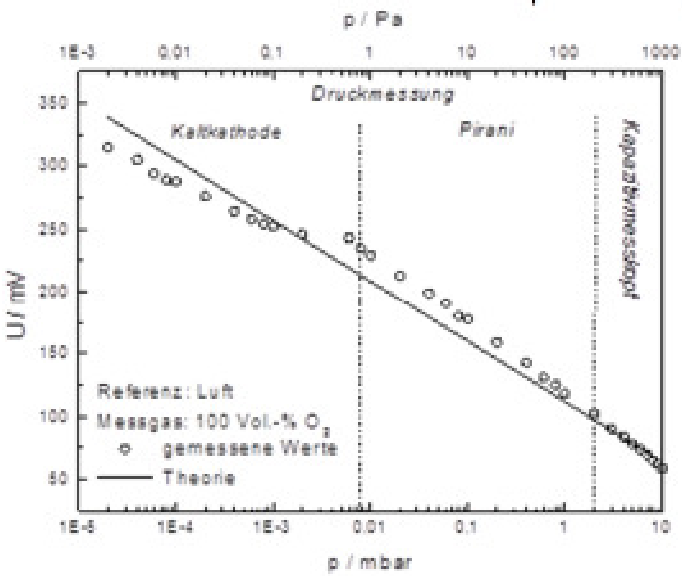
$$\varphi_{O_2} = \frac{0.2064\, p_L}{p} \cdot \exp\left[-46.42 \cdot \frac{U/mV}{T/K}\right] .$$

6.2 Instructions for calibration

When operating in vacuum differences in the electrodes' temperatures usually cause indeterminable thermoelectric voltage which depends on the respective production unit. This thermoelectric voltage adds to the signal voltage which corresponds to the Nernst equation.

For exact measuring of the oxygen partial pressure the measuring system must be calibrated with a pressure gauge at the production unit filled with a known gas (preferably pure oxygen).

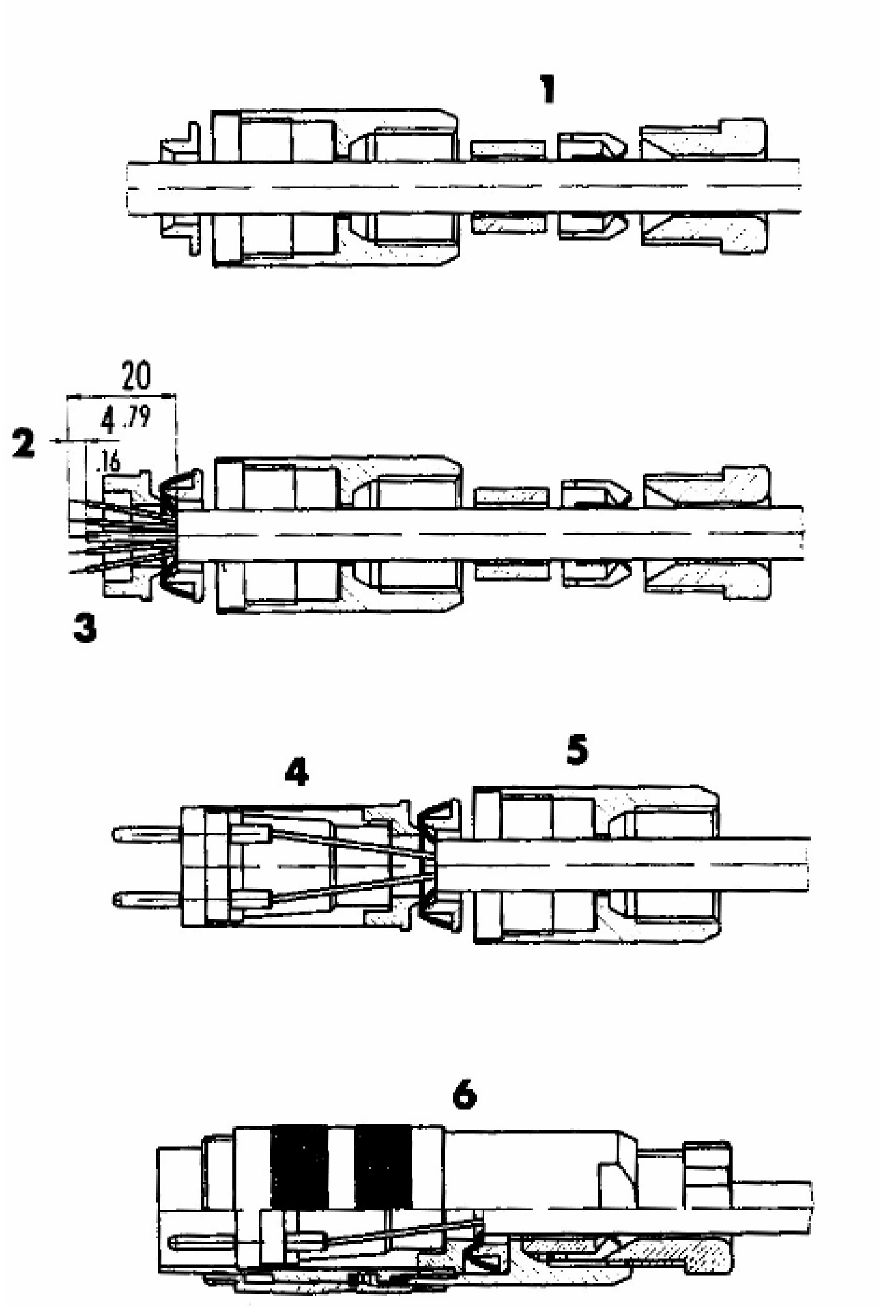
The line represents the signal voltage U which is expected for the oxygen pressure scale (x-coordinate) at the probe with air as reference gas. The measured values represent the probe signals after the gradual evacuation with different pumps over the pressures measured in a higher pressure range with a capacity meter, in the medium pressure range with the Pirani device and in the lower pressure range at a cold cathode. The jumps between the different ranges show that the pressure gauges were not adjusted and provide faulty results themselves. It shows, however, that the probe works as expected in a range of 6 decimal powers. The displayed range extends from approximately a hundredth of the normal pressure to 10 billionths of this pressure (corresponding to 10 Vol.-ppb).



The maximum deviations from the theoretical run were approximately 25 mV or half a decimal power of the oxygen partial pressure. Since the deviations are both positive and negative, a distinct explanation is impossible. In the lower pressure range the deviation points to the range which is expected when oxygen moves from the reference air side to the measuring gas side. Therefore, calibrations should only be conducted with reliable pressure gauges in the field of probe operation.

In practice, if the probe is needed for the adjustment and replication of defined conditions only, it is sufficient to determine the probe signals empirically in a successful trial. The operating mode has to be set up so as to receive the requested probe signal. Due to different parameter impacts on the raw signal of the probe this is the easiest and in some case the only way. Receiving theoretical values is only possible under highly simplified (lab) conditions for the most part.

7 Assembly instructions for the plug



1. Thread parts
2. Strip the insulation and widen shield
3. Assemble shield clamping ring
4. Solder wire, attach distance bush
5. Cut off lapping shield
6. Assemble remaining parts according to plan

8 Declaration of conformity

EG - Konformitätserklärung

Dokument- Nr.: **12** **17. Juni 2003**

Hersteller: **Zirox Sensoren & Elektronik GmbH**

Anschrift: **Am Koppelberg 21**
D - 17489 Greifswald

Produktbezeichnung: Vakuumsonde XS22H mit E 620

Die Übereinstimmung des bezeichneten Produktes mit den Vorschriften der Richtlinie des Rates
89/336/EWG (zuletzt geändert: 93/68/EWG)
wird nachgewiesen durch:

Der Hersteller hat die in der Richtlinie 89/336/EWG genannten harmonisierten Normen angewandt und die Übereinstimmung des Produktes festgestellt.

harmonisierte europäische Normen:

Nummer:	Text:	Ausgabedatum:
DIN EN 61000-6-4	Elektromagnetische Verträglichkeit (EMV);Teil 6-4 Störaussendung für Industriebereich	08.2002
DIN EN 61000-6-2	Elektromagnetische Verträglichkeit (EMV);Teil 6-2 Fachgrundnorm: Störfestigkeit für Industriebereich	08.2002

Diese Erklärung bescheinigt die Übereinstimmung mit der genannten Richtlinie, beinhaltet jedoch keine Zusicherung von Eigenschaften. Die Sicherheitshinweise der mitgelieferten Produktdokumentation sind zu beachten.

Aussteller: **Zirox Sensoren & Elektronik GmbH**

Ort, Datum: Greifswald 18.06.03

Rechtsverbindliche Unterschrift: ....