Probe for electrical measurements in magnetic field and at cryogenic conditions (aka Very low temperature probe - VLTP)

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Location: CEITEC BUT, Laboratory of EPR spectroscopy

Description:

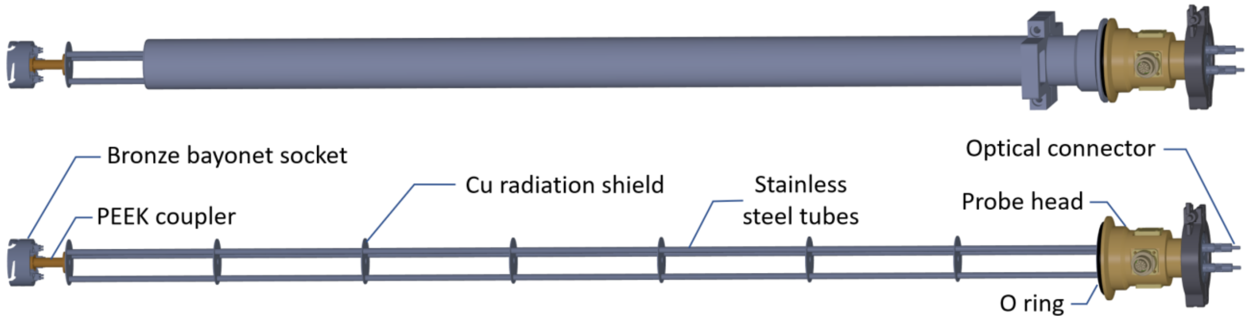
To extend usage options of a Cryogen Free Magnet (CFM) system, located in a laboratory of Electron Paramagnetic Resonance (EPR) spectroscopy at CEITEC VUT, a Very Low Temperature Probe (VLTP) was developed for electronic measurements at cryogenic temperatures. The VLTP was designed to be compatible with a Variable Temperature Insertion (VTI) system which is adjustable cryostat inside the CFM and allows to control temperature of a sample in rage from 2 K to 325 K.

The probe is assembled of a probe head on the top, a body that has a shape of a long prism, an outer tube and a bayonet socket attached to a coupler at the bottom. The probe head has a cylindrical shape and is made of aluminium. On sides of the probe head are four ports, and one port is on top. Two ports on sides are occupied by 11 and 16-pin hermetically sealed connectors. Other ports are sealed by a blank-off covers. The body is made of three hollow nonmagnetic stainless-steel tubes, which have a low thermal conductivity and are placed in a triangle. On one side the tubes are attached to the probe. On another side the body is ended by a copper radiation shield that reflects Infra-Red (IR) radiation. A coupler made of PEEK is placed between this radiation shield and bronze bayonet socket to further reduce a heat transfer. Other radiation shields are placed along the body and serve also as its reinforcement. Thanks to this design, the VLTP can achieve temperature difference of 300 K at length of 94 cm (average thermal gradient is about 3 K/cm). The body is covered by an outer tube when probe is loaded via airlock into the cryostat and provide a hermetical sealing up to a bottom of the probe head. A PEEK coupler is equipped by three 8-pin connectors, and the bayonet socket is compatible with a sample holders used for EPR measurements. Two connectors are connected via phosphor-bronze wires to the hermetic 16-pin connector dedicated for electronic measurements, and the last one is connected to the 11-pin connector usually used for a temperature sensor, field sensor and a heater. Therefore, already made Chip Sample Holder (CHSH) with 16 contacts can be simply used for electronic measurements that require cryogenic temperatures and/or a high magnetic field.

The probe was already tested by measuring of calibration curves of uncalibrated temperature sensors. For this purpose, a special holder with a high thermal conductivity was made. It can hold up to six temperature sensors, however only three were placed at once so far, because in the EPR laboratory are only three suitable instruments. One of the sensors is used as a calibrated standard, and others are uncalibrated. To monitor temperature of a calibrated standard, a temperature controller model 350 from Lake Shore Cryotronics was used. Resistance of other two sensors was measured by a four-wire method source at two source meter units Keithley 2450 from Tektronix. Total uncertainty of measurement at 4.2 K was calculated as 13.2 mK, and at 77 K it was 194.5 mK. In addition, a single purpose software that controls all three instruments and collects a calibration data was made in LabVIEW and is uploaded to a public repository (CEITEC-MOTES-VLTP) on GitHub. Expenses on the probe were already covered by creating of six calibration curves for uncalibrated sensors, because difference in cost of a single calibrated and uncalibrated sensor is about 550 USD.

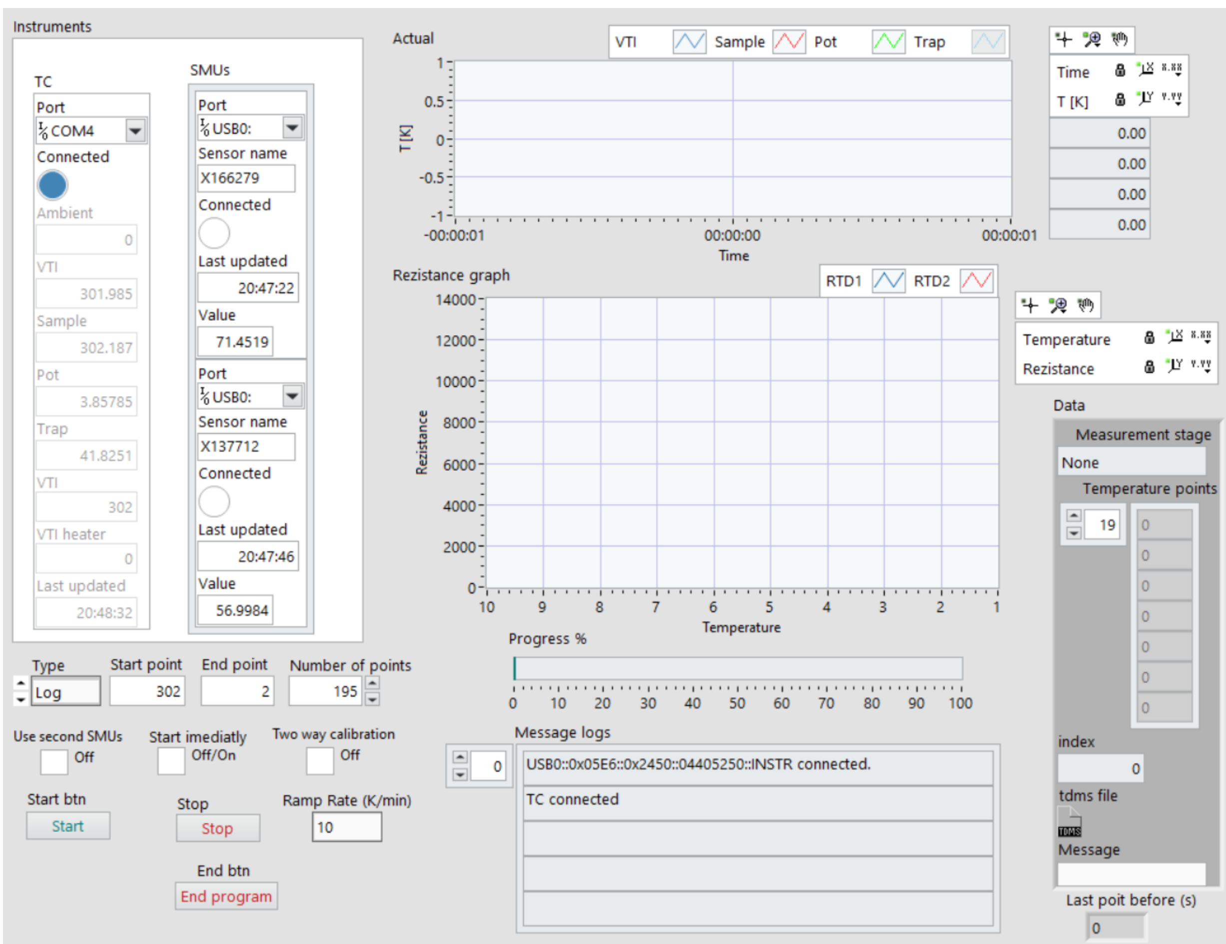
Website: https://github.com/MatSevy/CEITEC-MOTES-VLTP

Design:



Actual construction:

Screenshot from software for measurement of RTD:



Technical parameters:

Total length	106 cm	Active length	94 cm
Probe head flange	7.5 cm / 5 cm (outer / inner diameter)		
Used ports	2 (11-pin DBEE-104A056 + 16-pin SFE-104A086)		
Blank ports:	2 on sides of probe head + 1 on top of probe head		
Maximum temperature	325 K (approx. 52 °C)		
Achieved temperature	< 2 K (sample holder)		

Economical aspects (so far):

Material cost:

Aluminium head: 1 000 CZK

Nonmagnetic stainless-steel tubes: 1 000 CZK

Radiation shields: 500 CZK

Holder mount (bay): 500 CZK

Sensor holder: 500 CZK

Temperature sensor: 20 000 CZK

Phosphor bronze wires: 5 000 CZK

Contact pins: 500 CZK

Hermetic connectors: 2 000 CZK
(Fisher Connectors: SFE-104A086-130 + DBEE-104A056-130)

(Total: 31 000 CZK)

Development time:

Design: 24 h

Construction: 54 h

Programming: 24 h

(102 h * 180 CZK/h = 18 360 CZK)

Savings so far: 4x (calibrated sensor – uncalibrated sensors) = 6x (781 USD – 231 USD) = 2200 USD = approx. 81 500 CZK

(Valid on Sep. 5th 2022.)

Appendix A - Uncertainty of calibration

Temperature Controller 350, uncertainty of sensor, SourceMeter Keithley 2450

Uncertainty Temperature Controller 350

For 4,2 K and 77 K

Temperature Controller 350 – 4,2 K

Range: 0-10 kΩ

Accuracy: ±1 Ω ±0,04% of rdg

Resistance at 4,2 K is 3507,2 Ω.

$\Delta_{max} = \frac{0{,}04 \times 3507{,}2\ \Omega}{100} + 1\ \Omega = \pm 2{,}4\ \Omega$ for 25 °C

$u_B(R) = \frac{\Delta_{max}}{\sqrt{3}} = \frac{2{,}4}{\sqrt{3}} = 1{,}39\ \Omega$

For 95% reliability:

$U(R) = u_B(R) \times k = 1{,}39\ \times 2 = 2{,}77\ \Omega$

Uncertainty converted to temperature:

$$u_{TC350}(T) = u(R) \times \frac{dT}{dR} = 1{,}39\ \Omega \times \frac{1}{973{,}09}\ \frac{\mathrm{K}}{\Omega} = 1{,}43 \times\ 10^{-3}\ K = \pm \boldsymbol{1{,}43\ mK}$$

Temperature Controller 350 – 77 K

Range: 0- 300 Ω

Accuracy: ±0,01 Ω ±0,04% of rdg

Resistance at 77 K is 205,67 Ω.

$\Delta_{max} = \frac{0{,}04 \times 205{,}67\ \Omega}{100} + 0{,}01\ \Omega = \pm 0{,}092\ \Omega$ for 25 °C

$u_B(R) = \frac{\Delta_{max}}{\sqrt{3}} = \frac{0{,}092}{\sqrt{3}} = 0{,}053\ \Omega$

For 95% reliability:

$U(R) = u_B(R) \times k = 0{,}053\ \times 2 = 0{,}107\ \Omega$

Uncertainty converted to temperature:

$$u_{TC350}(T) = u(R) \times \frac{dT}{dR} = 0{,}053\ \Omega \times \frac{1}{2{,}8137}\ \frac{\mathrm{K}}{\Omega} = 0{,}019\ K = \pm \boldsymbol{19\ mK}$$

Uncertainty SourceMeter 2450

For 4,2 K and 77 K

SourceMeter 2450 – 4,2 K

Range: 0-20 kΩ

Accuracy: ±1 Ω ±0,043% of rdg

Resistance at 4,2K is 3507,2 Ω.

$\Delta_{max} = \frac{0,043 \times 3507,2\ \Omega}{100} + 1\ \Omega = \pm 2,5\ \Omega$ for 23 ± 5 °C, 1 year after calibration

$u_B(R) = \frac{\Delta_{max}}{\sqrt{3}} = \frac{2,5}{\sqrt{3}} = 1,45\ \Omega$

For 95% reliability:

$$U(R) = u_B(R) \times k = 1,45\ \times 2 = 2,9\ \Omega$$

Uncertainty converted to temperature:

$$u(T) = u(R) \times \frac{dT}{dR} = 1,45\ \Omega \times \frac{1}{973,09}\frac{\mathrm{K}}{\Omega} = 1,49 \times\ 10^{-3}\ K = \pm 1,49\ mK$$

SourceMeter 2450 – 77 K

Range: 0-2 kΩ

Accuracy: ±0,1 Ω ±0,045% of rdg

Resistance at 77 K is 205,67 Ω.

$\Delta_{max} = \frac{0,045 \times 205,67\ \Omega}{100} + 0,1\ \Omega = \pm 0,19\Omega$ for 23 ± 5 °C, 1 year after calibration

$u_B(R) = \frac{\Delta_{max}}{\sqrt{3}} = \frac{0,19}{\sqrt{3}} = 0,11\ \Omega$

For 95% reliability:

$$U(R) = u_B(R) \times k = 0,11\ \times 2 = 0,22\ \Omega$$

Uncertainty converted to temperature:

$$u(T) = u(R) \times \frac{dT}{dR} = 0,11\ \Omega \times \frac{1}{2,8137}\frac{\mathrm{K}}{\Omega} = 0,079\ K = \pm 79\ mK$$

Uncertainty Sensor Cernox 1050

For T = 4,2 K

$$u_{Cernox}(T) = \pm 6,1\ mK$$

For T = 77 K

$$u_{Cernox}(T) = \pm 50\ mK$$

Uncertainty of calibration – 4,2 K and 77 K

$$u_{cal4,2}(T) = \sqrt{u_{TC350}(T)^2 + u_{SM2450}(T)^2 + u_{Cernox}(T)^2} = \sqrt{1{,}43mK^2 + 1{,}49mK^2 + 6{,}1mK^2} = \mathbf{6,44\ mK}$$

$$u_{cal77}(T) = \sqrt{u_{TC350}(T)^2 + u_{SM2450}(T)^2 + u_{Cernox}(T)^2} = \sqrt{19mK^2 + 79mK^2 + 50mK^2} = \mathbf{95,4\ mK}$$

For 95% reliability:

$$U_{cal4,2}(T) = 2 \times u_{cal4,2}(T) = 12{,}9\ mK$$

$$U_{cal77}(T) = 2 \times u_{cal77}(T) = 190{,}8\ mK$$

Uncertainty of measurement

Temperature Controller 350, uncertainty of calibration

Uncertanty Temperature Controller 350

For 4,2 K

$$u_{TC350}(T) = 1{,}43\ mK$$

For 77 K

$$u_{TC350}(T) = 19\ mK$$

Uncertainty of calibration

For 4,2 K

$$u_{cal4,2}(T) = 6{,}44\ mK$$

For 77 K

$$u_{cal77}(T) = 95{,}4\ mK$$

Uncertainty of measurement – 4,2 K and 77 K

$$u_{meas4,2}(T) = \sqrt{u_{TC350}(T)^2 + u_{Cal}(T)^2} = \sqrt{1{,}43mK^2 + 6{,}44mK^2} = 6{,}6\ mK$$

$$u_{meas77}(T) = \sqrt{u_{TC350}(T)^2 + u_{Cal}(T)^2} = \sqrt{19mK^2 + 95{,}4mK^2} = 97{,}27\ mK$$

For 95% reliability:

$$U_{meas4,2}(T) = 2 \times u_{meas4,2}(T) = 13{,}2\ mK$$

$$U_{meas77}(T) = 2 \times u_{meas77}(T) = 194{,}5\ mK$$