Quantum Design

# LAB READY SCIENTIFIC INSTRUMENTATION FOR MAGNETIC AND TRANSPORT MEASUREMETNS

**Rick Hapanowicz, Ph.D.**
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# Quantum Design

MPMS®3


DISCOVERY TEACHING LABS

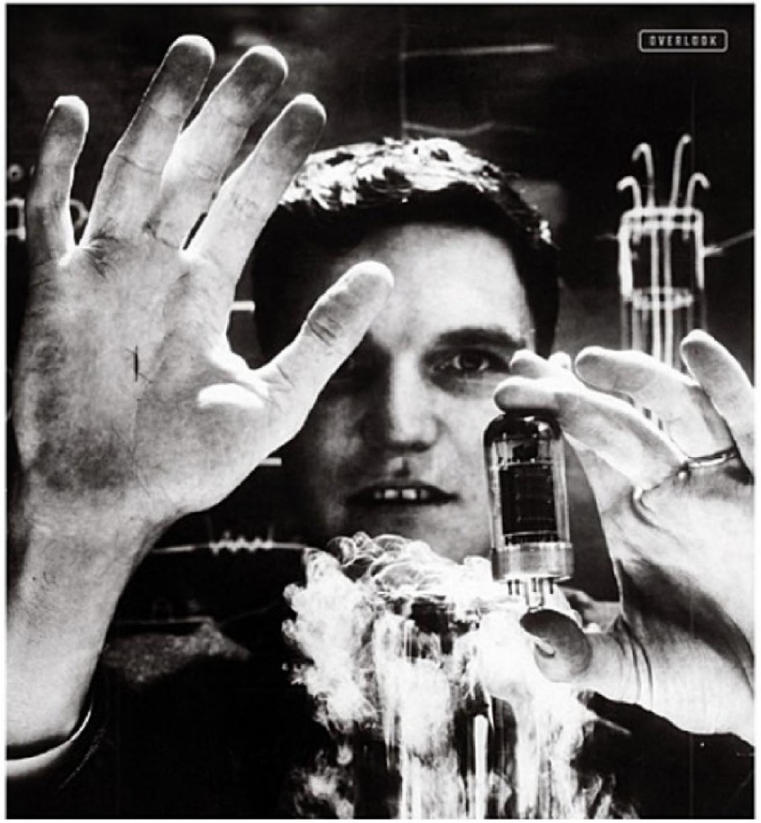
LEARN | MAKE | DISCOVER

PPMS®
DYNACOOL


OptiCool®
By Quantum Design



fusion scope
by Quantum Design

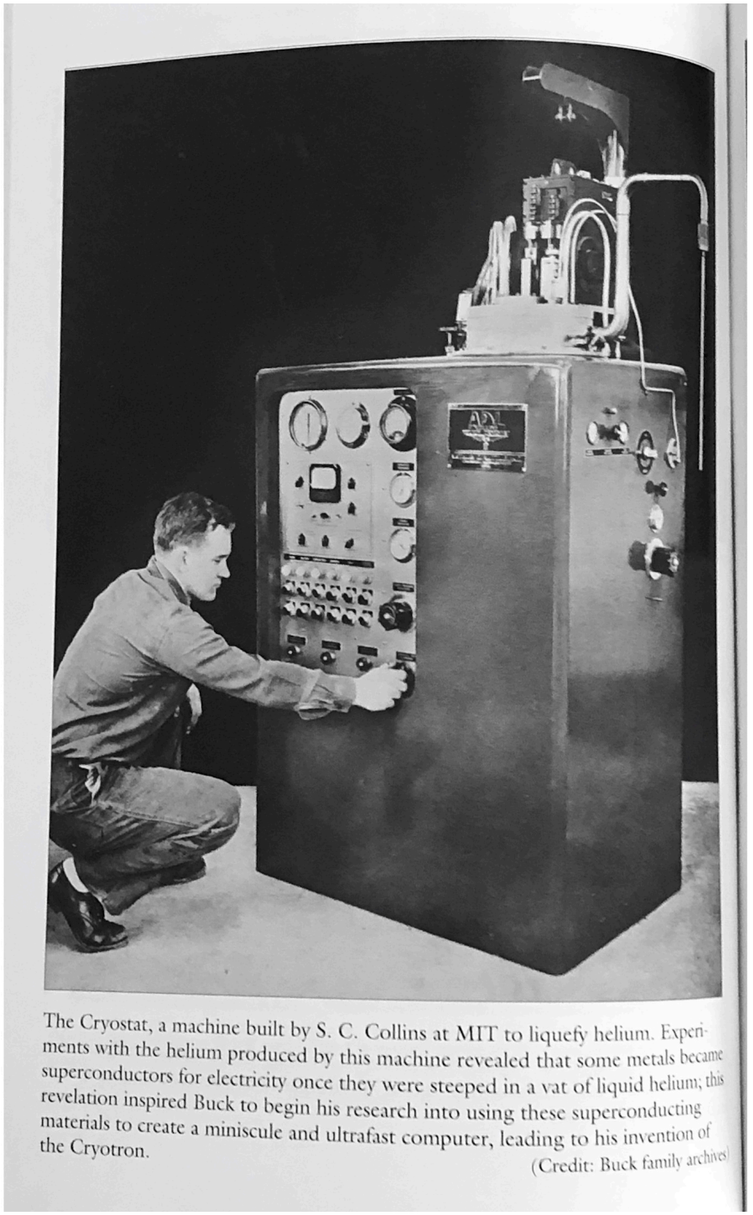



# THE CRYOTRON FILES

## THE UNTOLD STORY OF DUDLEY BUCK
COLD WAR COMPUTER SCIENTIST AND MICROCHIP PIONEER

IAIN DEY AND DOUGLAS BUCK





The Cryostat, a machine built by S. C. Collins at MIT to liquefy helium. Experiments with the helium produced by this machine revealed that some metals became superconductors for electricity once they were steeped in a vat of liquid helium; this revelation inspired Buck to begin his research into using these superconducting materials to create a miniscule and ultrafast computer, leading to his invention of the Cryotron.

(Credit: Buck family archives)

PPMS®
DYNACOOL

## DynaCool Features

- No need for liquid cryogens
- Temperature range of 1.8 K – 400 K
- 9, 12 & 14 tesla magnets available
- Continuous Low-Temperature Control
- Controlled temperature Sweep Mode
- Fully automated operation of available PPMS options

## DynaCool Options

- Heat Capacity
- Thermal Transport
- VSM with Oven up to 1000 K
- Magneto-Optic Measurement (FOSH, Light Source)
- Torque Magnetometry
- AC Susceptibility Option (ACMS II)
- DC Resistivity
- Electrical Transport (ETO)
- Horizontal Sample Rotator
- Multi-Function Probe
- Helium-3 Refrigerator
- Dilution Refrigerator
- High Pressure Cell for Magnetometry
- Hydrostatic Pressure Cell for Electrical Measurements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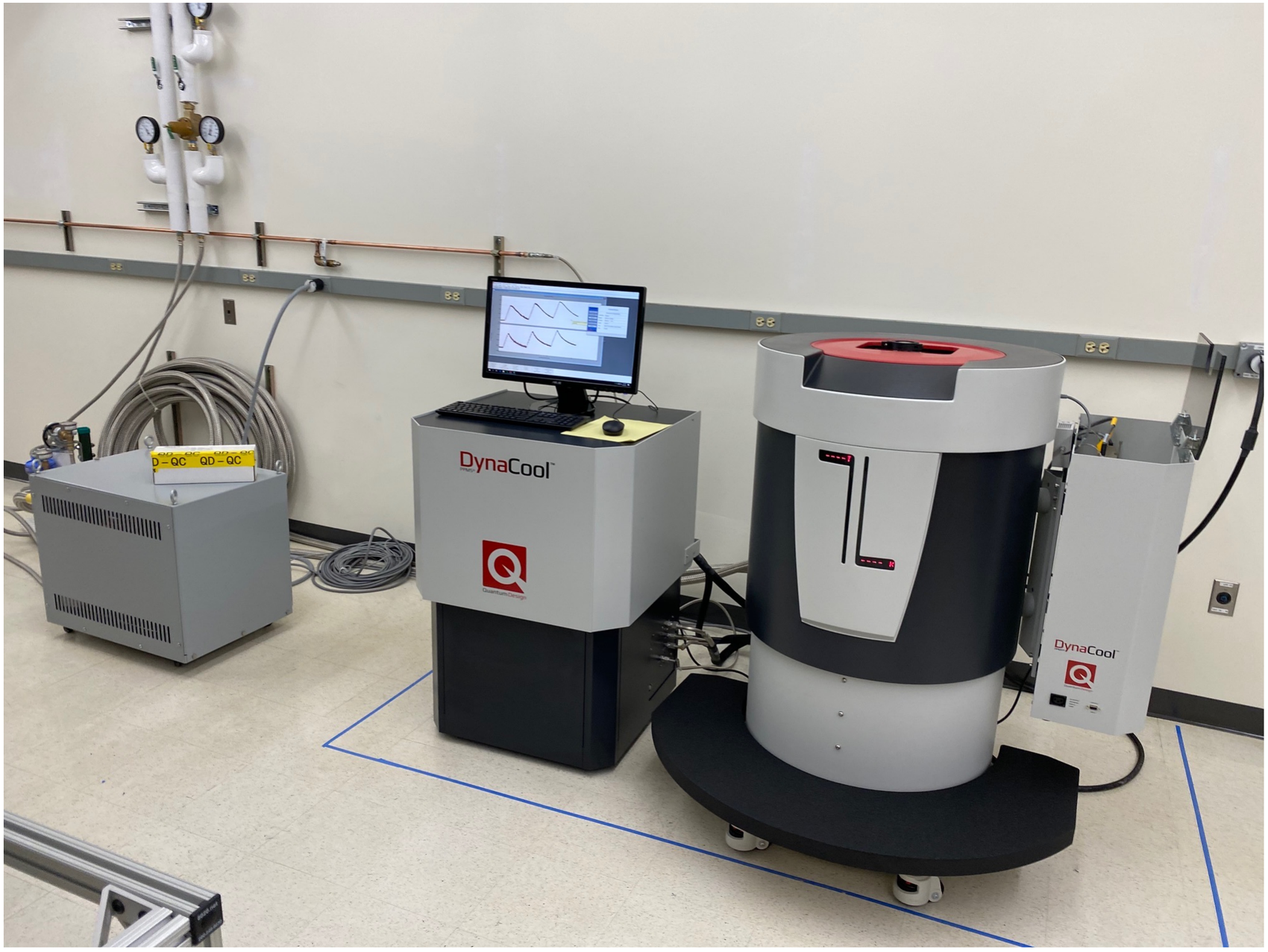


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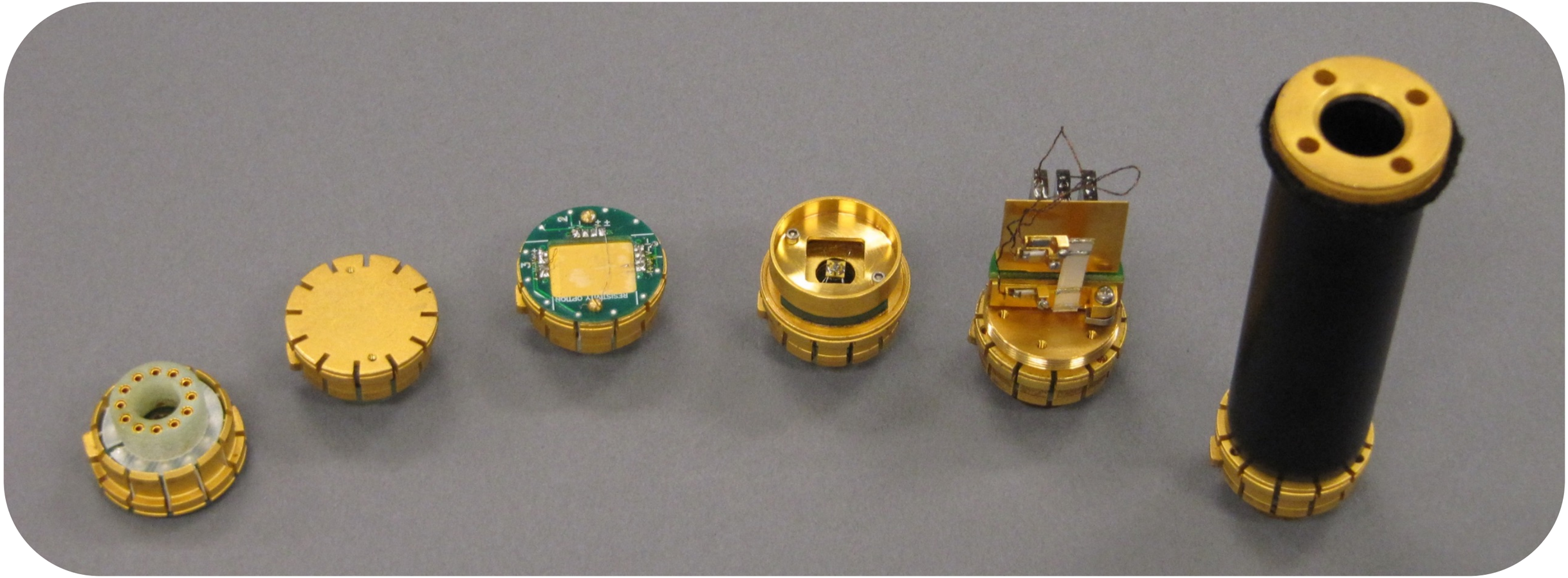
# PPMS MEASUREMENT OPTIONS



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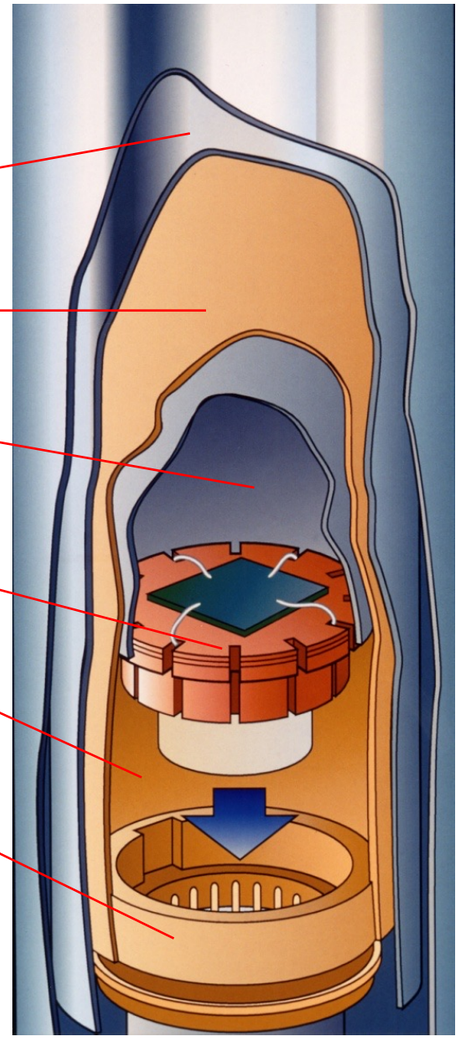
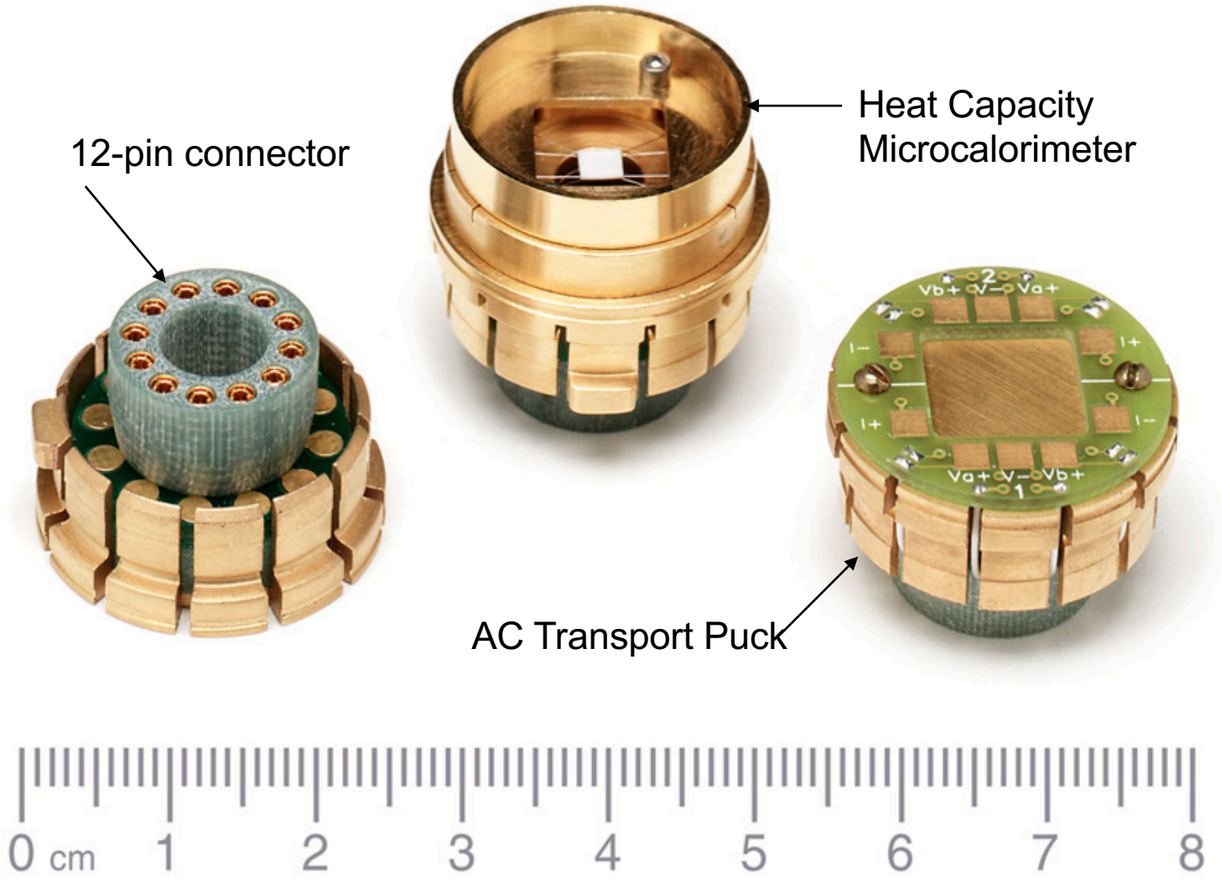
# The Puck

- Vacuum Space
- Cooling Annulus
- Sample Insertion Tool
- Sample “Puck”
- Sealed Sample Chamber
- 12-pin Connector



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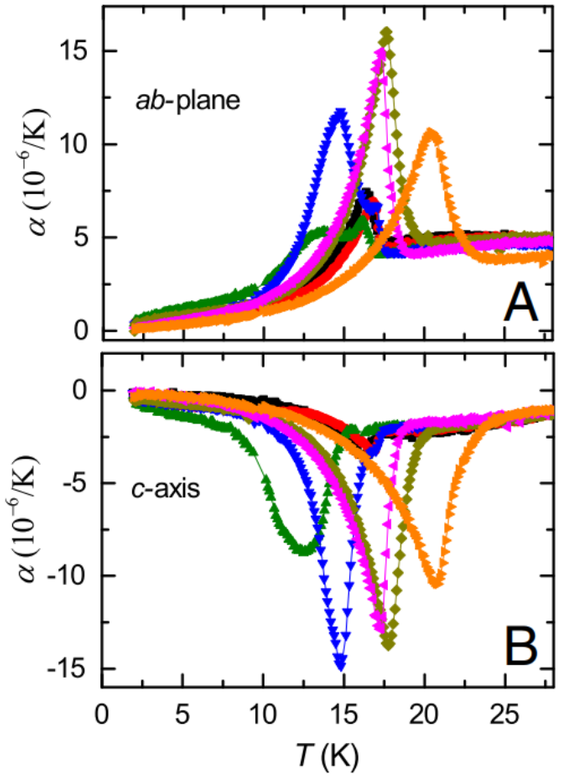
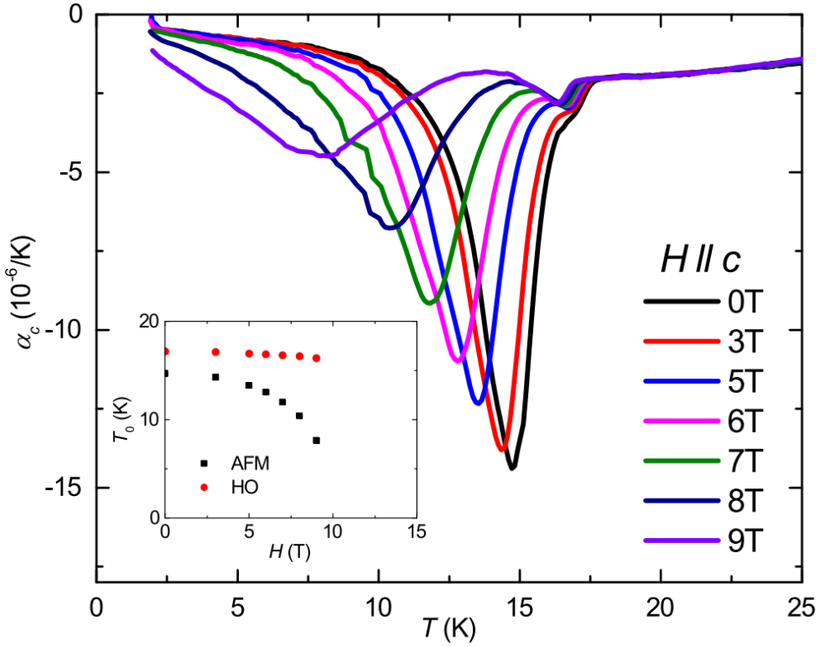
Randy K. Dumas--dumas@qdusa.com

# DilatoMeter

**Sample Size:**
2mm +/- 0.05
**Temperature Range:**
1.8 - 400 K
**Magnetic Field:**
Up to 16T
**Noise Level:**
70 ppm at 300 K
20 ppm at 2 K




*Phase diagram and thermal expansion measurements on the system $URu_{2-x}Fe_xSi_2$*
*Sheng Ran, et al., PNAS **113**, 13348 (2016)*

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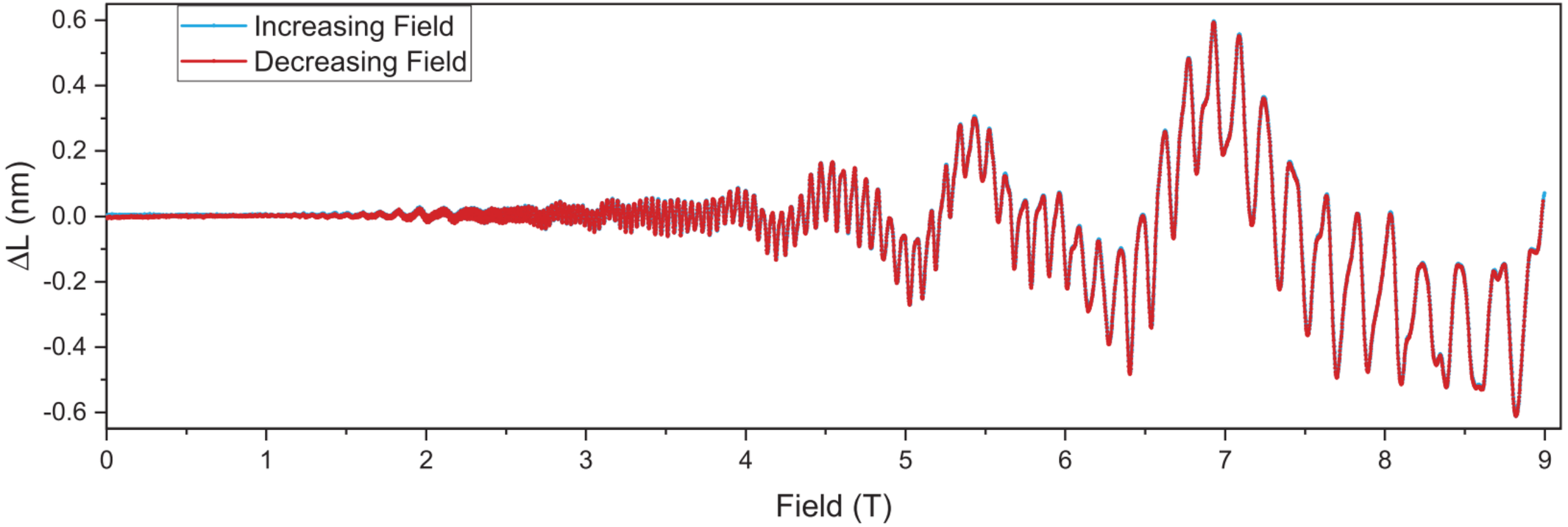
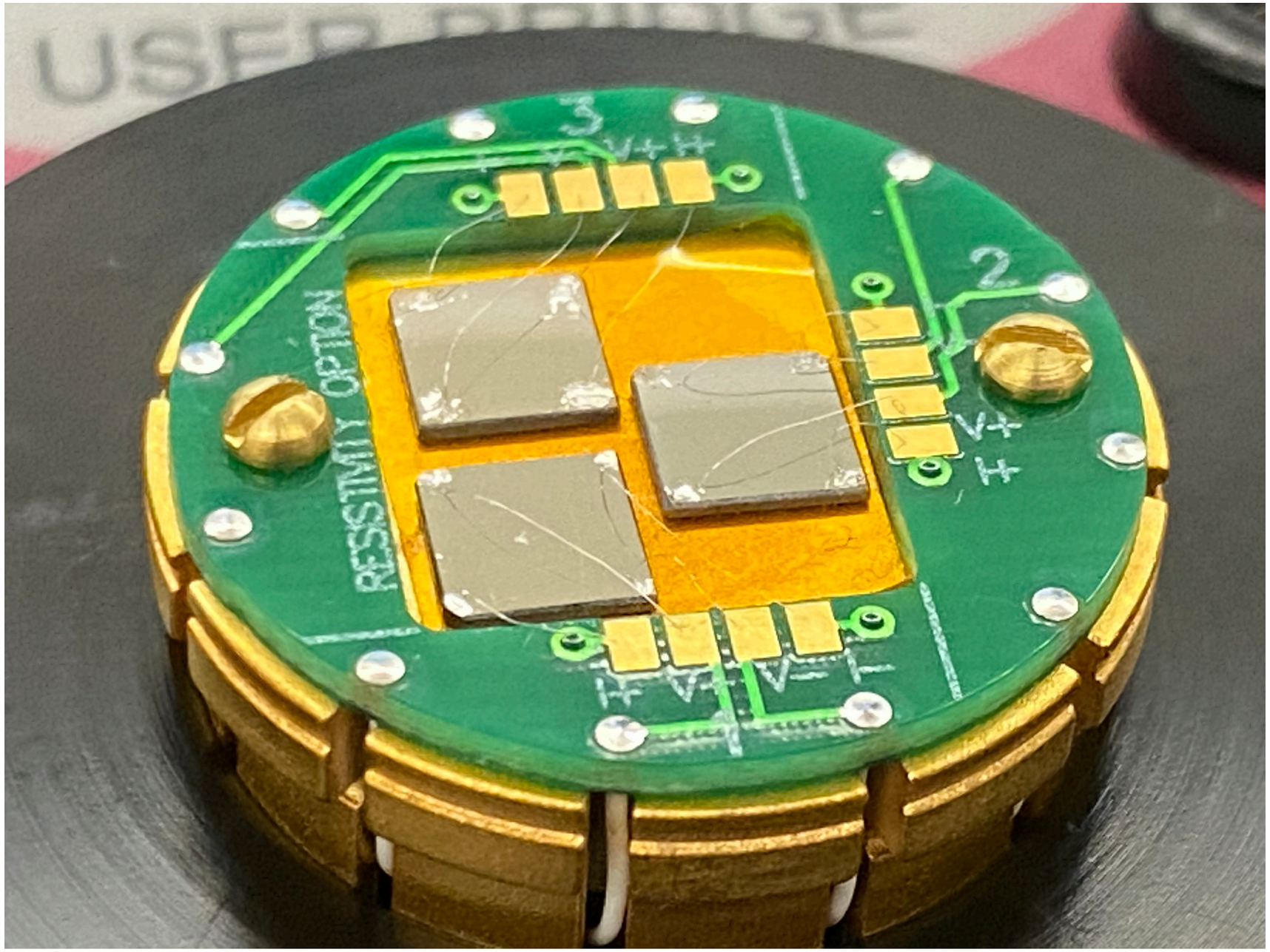
# DilatoMeter

**Sample Size:**
2mm +/- 0.05
**Temperature Range:**
1.8 - 400 K
**Magnetic Field:**
Up to 16T
**Noise Level:**
70 ppm at 300 K
20 ppm at 2 K

## Aluminum magnetostriction at 2.0 K showing the De Haas-van Alphen Effect



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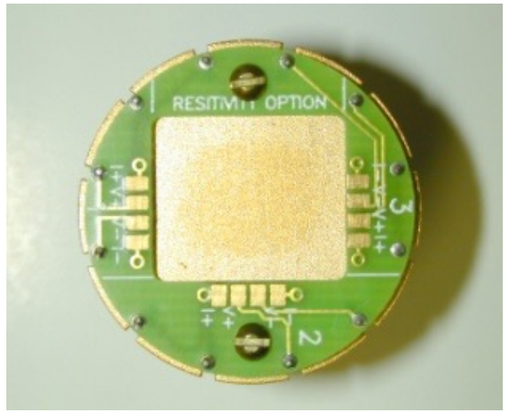
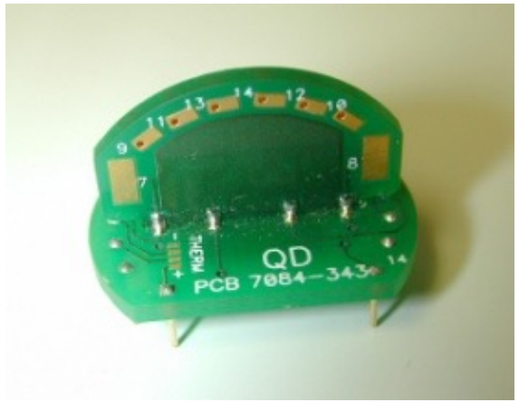
# PPMS TRANSPORT SAMPLE HOLDERS




**SAMPLE PUCK**


**ROTATOR SAMPLE BOARDS**

**HELIUM-3 SAMPLE BOARD**

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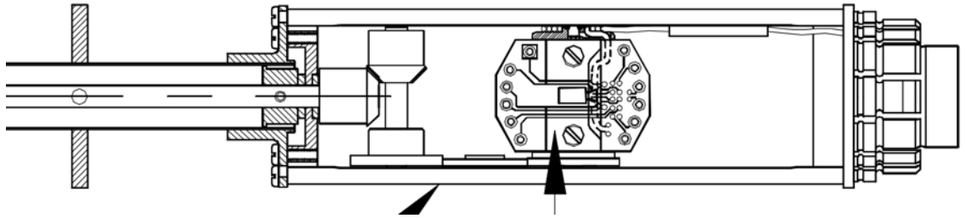
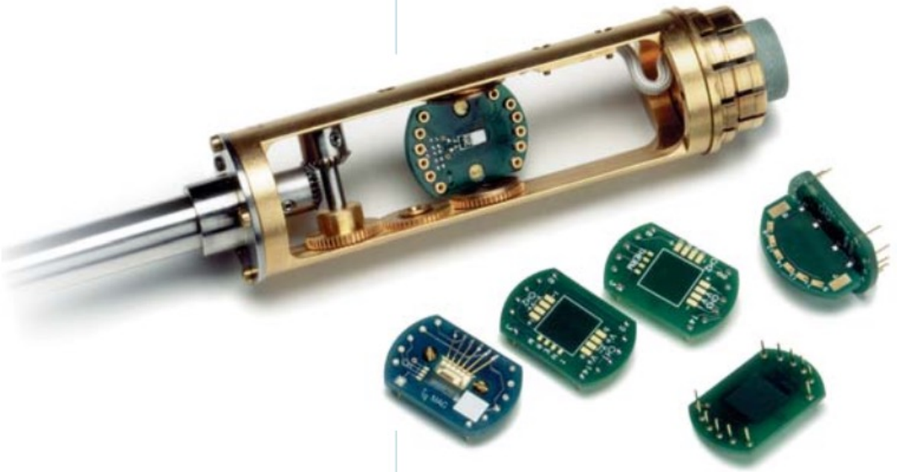
# HORIZONTAL ROTATOR
# APPLICATION: ANISOTROPIC MAGNETORESISTANCE

- The Horizontal Rotator option gives the user fine control, using a variety of sample mounts and a high resolution motor, sample position relative to the applied field



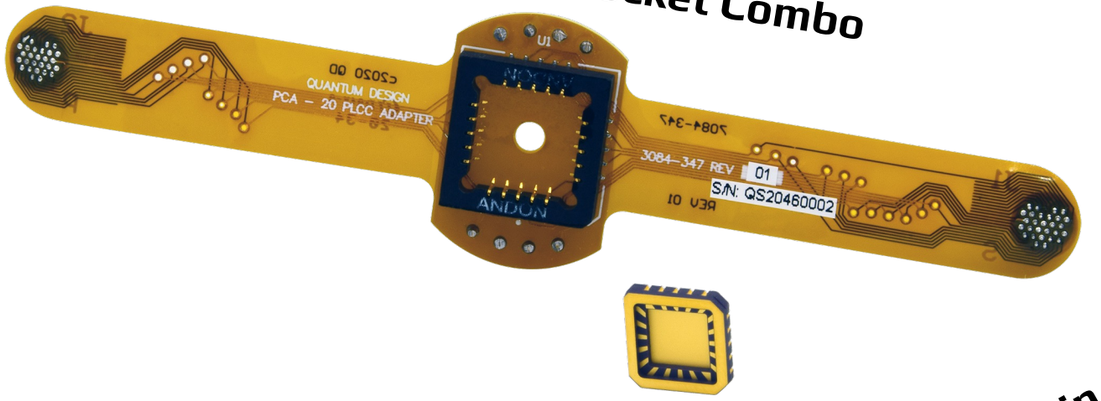
- ETO data can be collected as the sample's orientation is systematically varied

- Any anisotropic response in magnetoresistance can provide information about crystal structure, inter-layer coupling, etc.

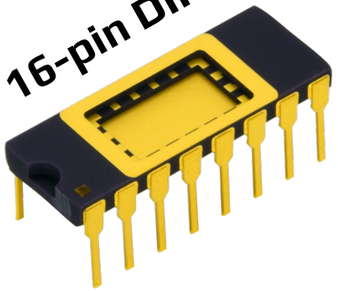


# MULTIFUNCTION PROBE MFP

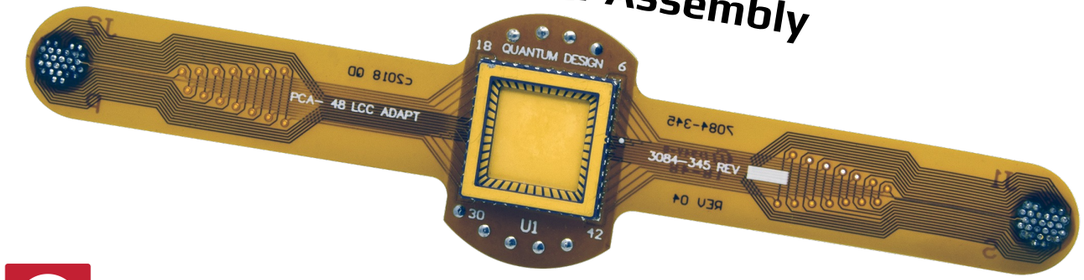
## 20-pin LCC
## Chip Carrier/Socket Combo



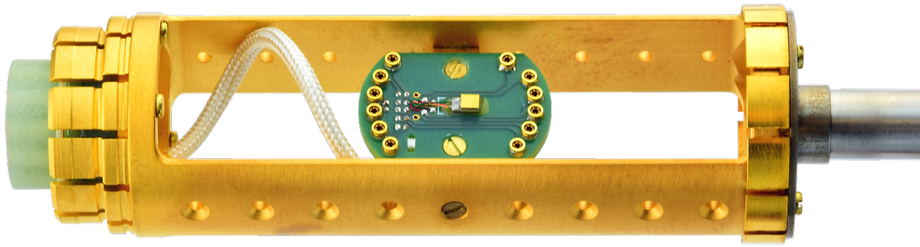

## 16-pin DIP



## 48-pin
## Chip Carrier Assembly

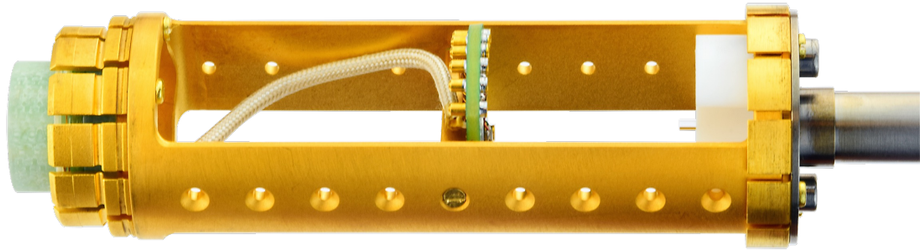



Type-A

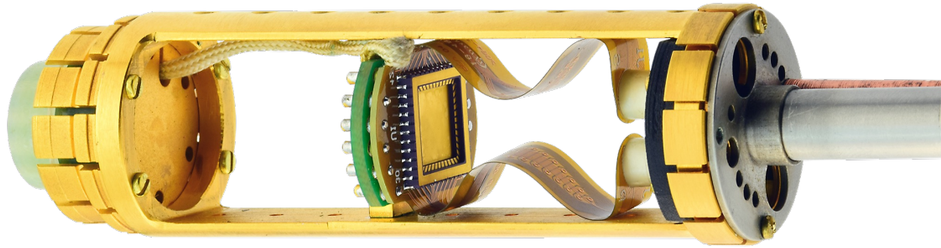


Type-B

Photoconductivity



Type-M



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# 16-Pin Dual Inline Package (DIP)

**Type-B** (P450B/D450B/V450B)
8087-402-01 (PPMS)
8087-402-03 (DynaCool)
8087-402-02 (VersaLab)

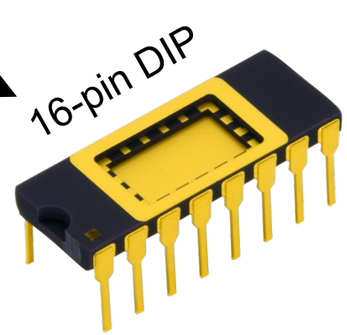
Only 12 of the 16 connections are used
Standard sample chamber thermometry
Standard sample chamber wiring
→ via standard grey LEMO connection

**Type-G** (P450G/D450G/V450G)
8087-405-01 (PPMS)
8087-405-03 (DynaCool)
8087-405-02 (VersaLab)

All 16 connections are used
→ 16 pin Fischer connection at top of probe
Calibrated Cernox thermometer at sample location

No electrical connections on top of probe

2 Included for Type-G: CSB01655





Many other 16-pin DIP variants available:
https://www.spectrum-semi.com/products/sidebraze

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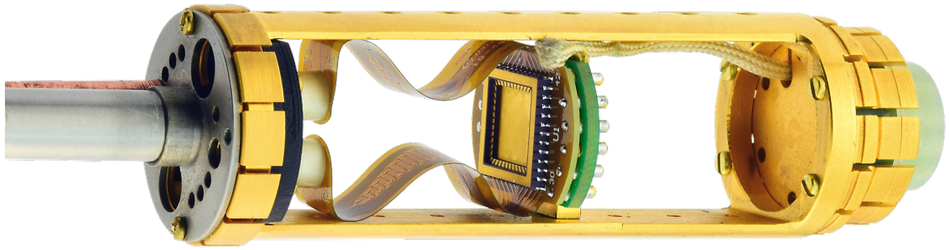
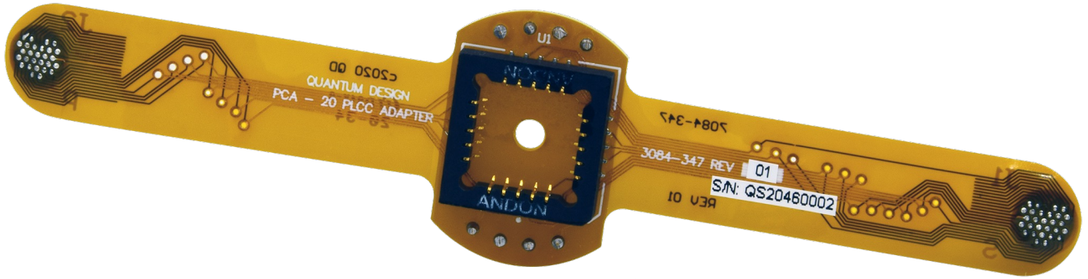
# Leadless Chip Carrier (LCC)

**Type-M** (P450M/D450M/V450M)
8087-404-01 (PPMS)
8087-404-03 (DynaCool)
8087-404-02 (VersaLab)



**Sample holders not included** → Order Separately

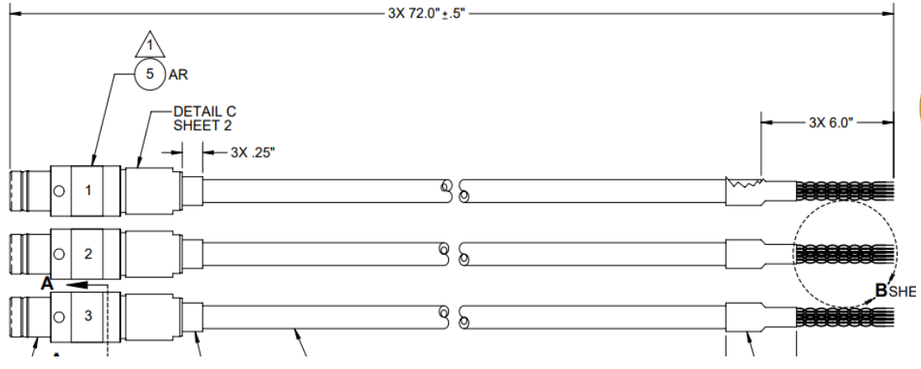
20-pin LCC Socket Adapter (4087-459)



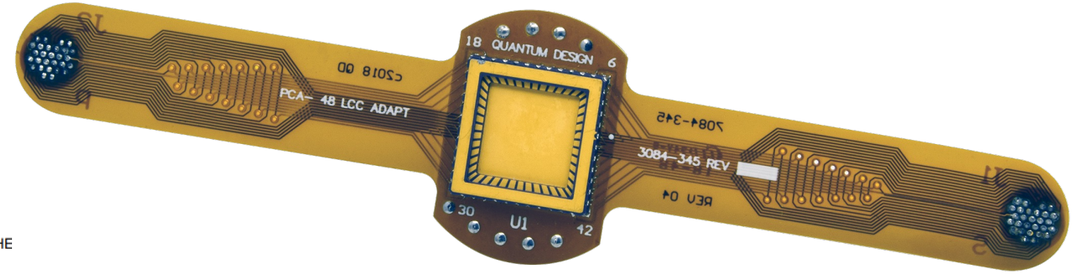

Up to 48 connections routed to the top of the probe
→ Three (3) 16-pin Fischer connections at top of probe
Calibrated Cernox thermometer at sample location

Not included: LCC02034 →

**3 cables included:**
Fischer-to-pigtail cable kit (3084-381)




48-pin Chip Carrier Assembly (4087-446)



# MULTIFUNCTION PROBE FOR CUSTOM EXPERIMENTS

- ESD-sensitive sample measurements
- Dielectric constant measurements
- Resistance, Hall Effect and Seebeck up to 1000 K
- Ferromagnetic Resonance
- Photoconductivity
- Probe with your dream wiring.

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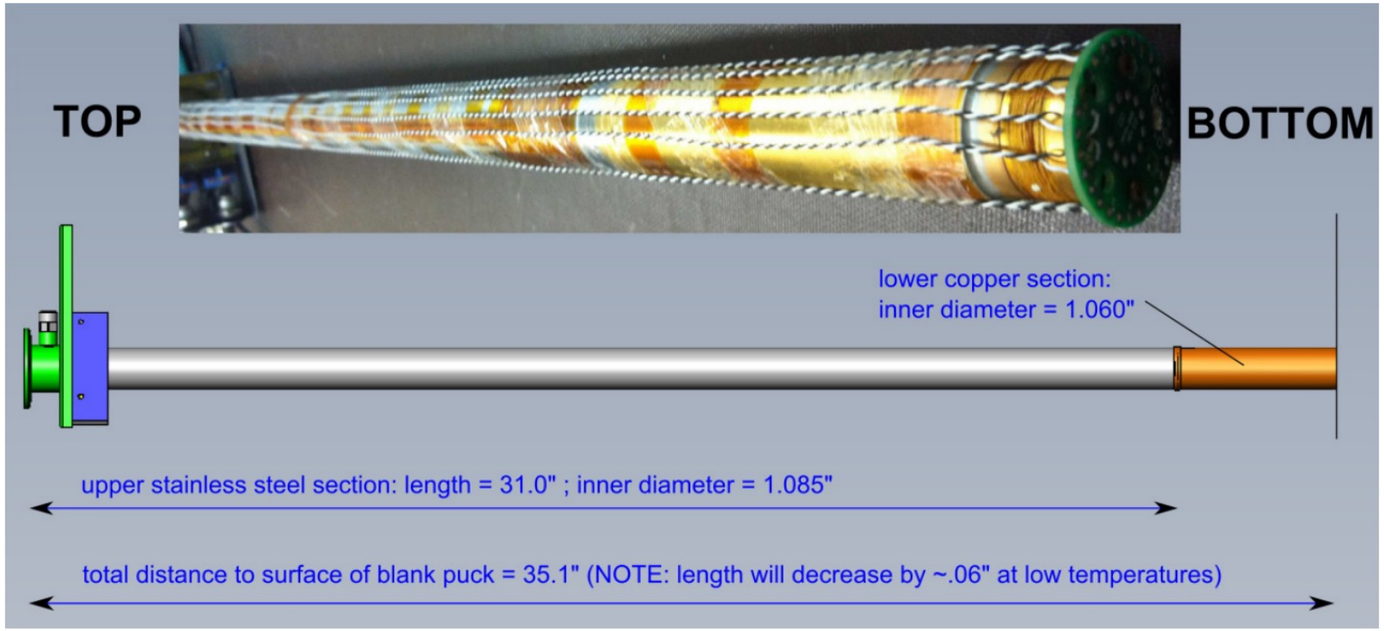
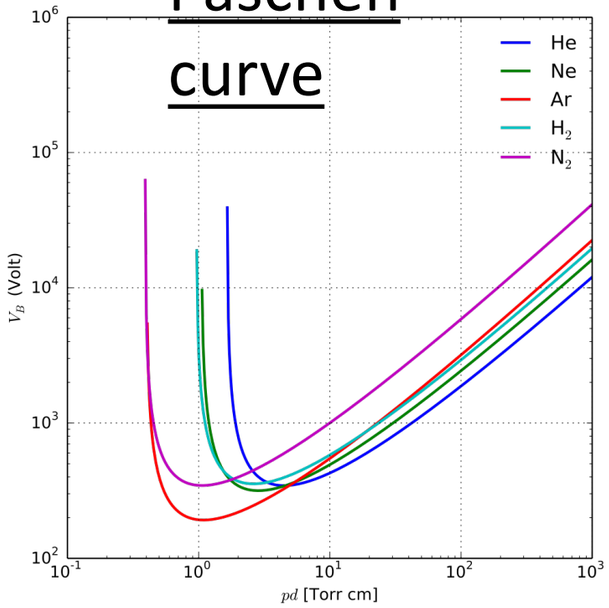
# Sample Chamber: Electrical Considerations




| parameter | value | comments |
|---|---|---|
| wire type | copper alloy | similar resistivity to Cu at 300 K; typical RRR ~ 3 - 10 |
| gauge | 28 AWG (.0126" diam) | twisted pairs are taped down to outside of chamber – see photo above |
| length | ~36" | twisted pairs along 34.2" length |
| | | |
| calculated resistance | R = 0.40 Ω | at 300 K ; roundtrip along two wires, assuming perfect short at bottom of sample chamber |
| measured resistance | R ~ 1Ω | at 300 K ; roundtrip along two wires with shorted puck at bottom; additional ~0.6 ohms from contacts at grey Lemo connector, vacuum interface board, heater block, and blank puck (4084-100); there are a total of 16 joints (either solder or connectors) in the roundtrip circuit including the shorted puck. |
| capacitance to ground | ~100 pF | for one wire to ground |
| | | |
| max. voltage | 50 V DC | this is a conservative limit; note that helium gas pressure in annulus around chamber is in the range of ~10-760 torr and minimum He breakdown voltage occurs near 1 torr |
| max. current | 500 mA | continuous; up to 2 A used in short pulses in PPMS AC Transport |

Paschen curve

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# CUSTOM EXPERIMENT NOTES

- **WIRE PAIRING –** Align your experiment wiring pairs (e.g., V+/V-) with system pairs (3/4) (5/6) to minimize inductive crosstalk.
- **GROUNDING -** Cryostat (including puck and chamber) is grounded through the vacuum pump so is NOT a good ground reference for sensitive measurements - Capacitive coupling of the twisted pair wiring to chamber ground
- **GROUND LOOPS** - Experiment wiring should be connected only at the electronics and not at the cryostat.
- **USE AT LOW TEMPERATURES –** Differential Thermal Expansion; For low current applications, consider alloys such as phosphor bronze, Constantan, or Manganin - QD Conductance Calculator on Website

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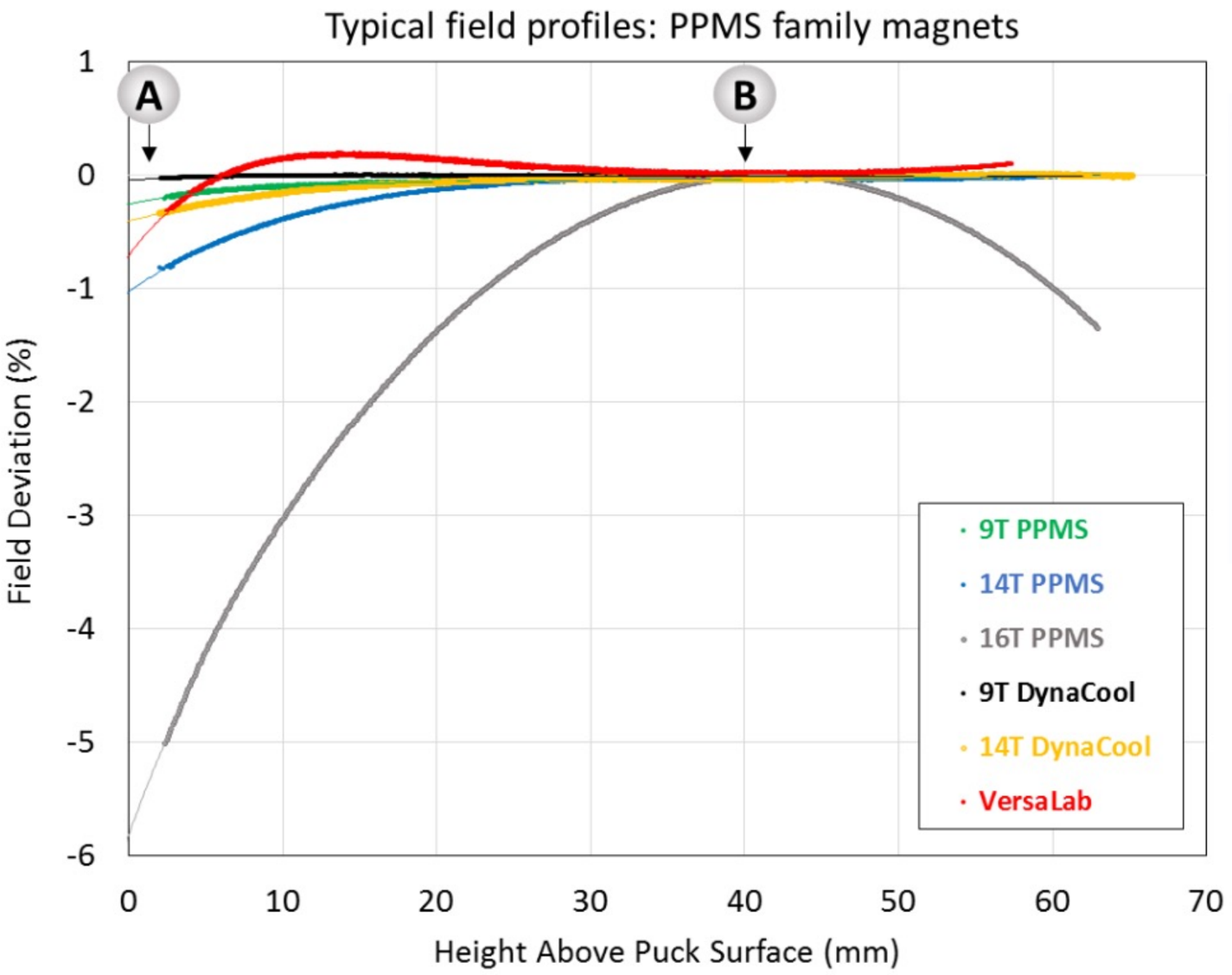
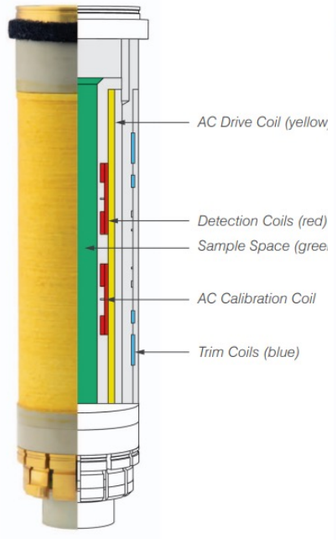
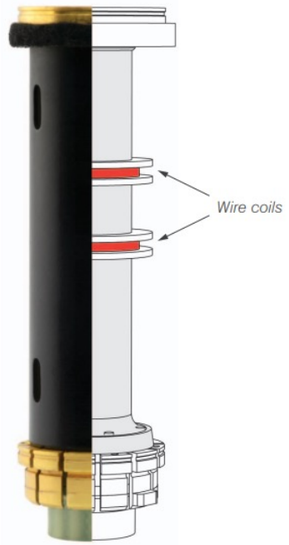
# Superconducting Magnets: Uniformity

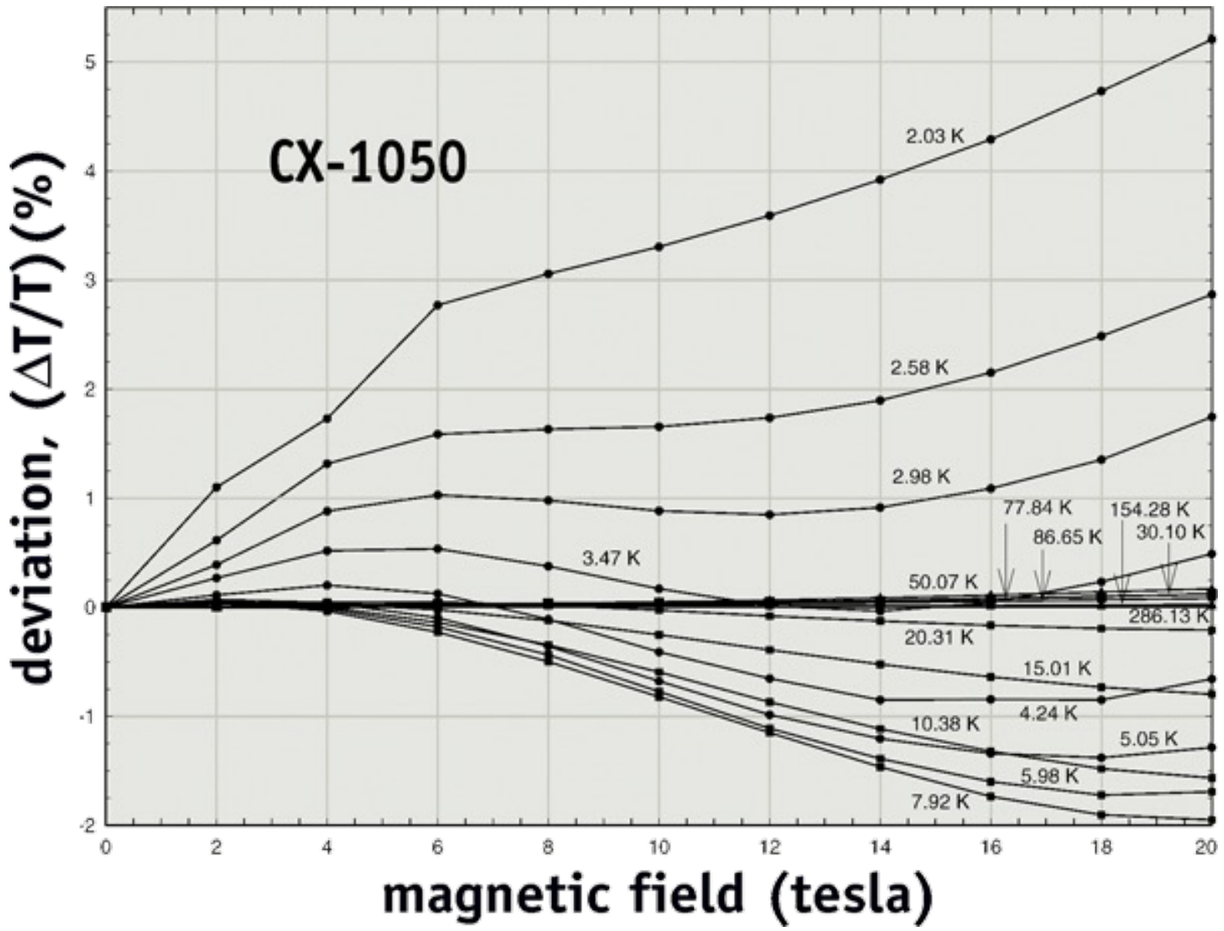


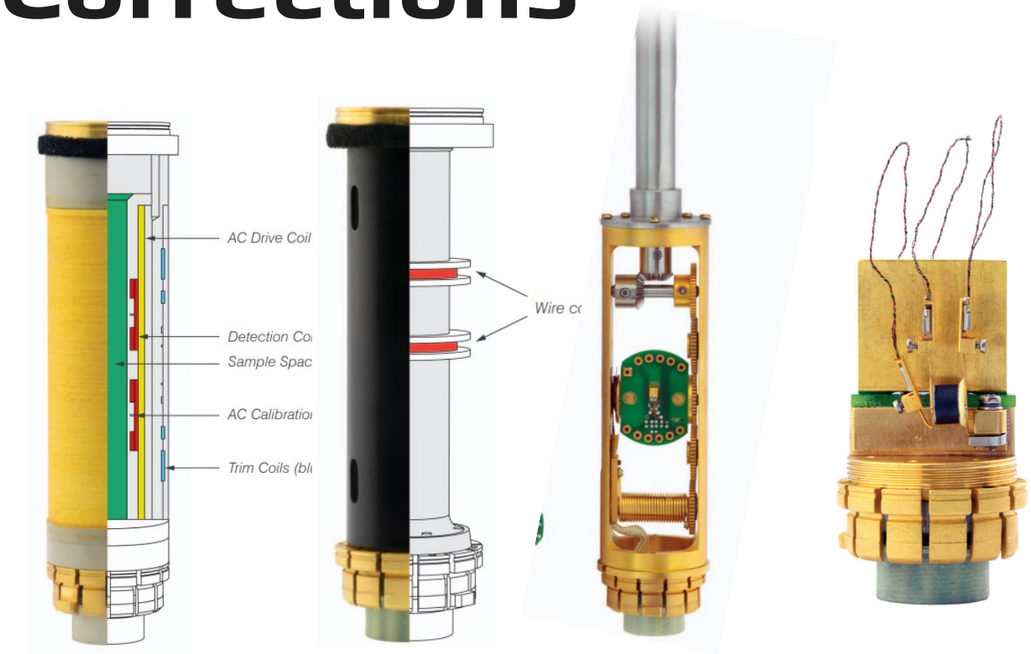



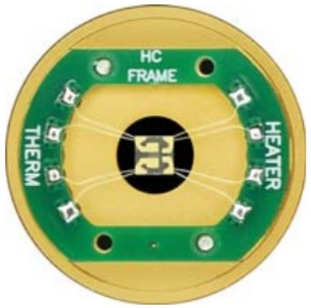





# Thermometry: MR Corrections




**Well defined protocols in the Heat Capacity Option Software**

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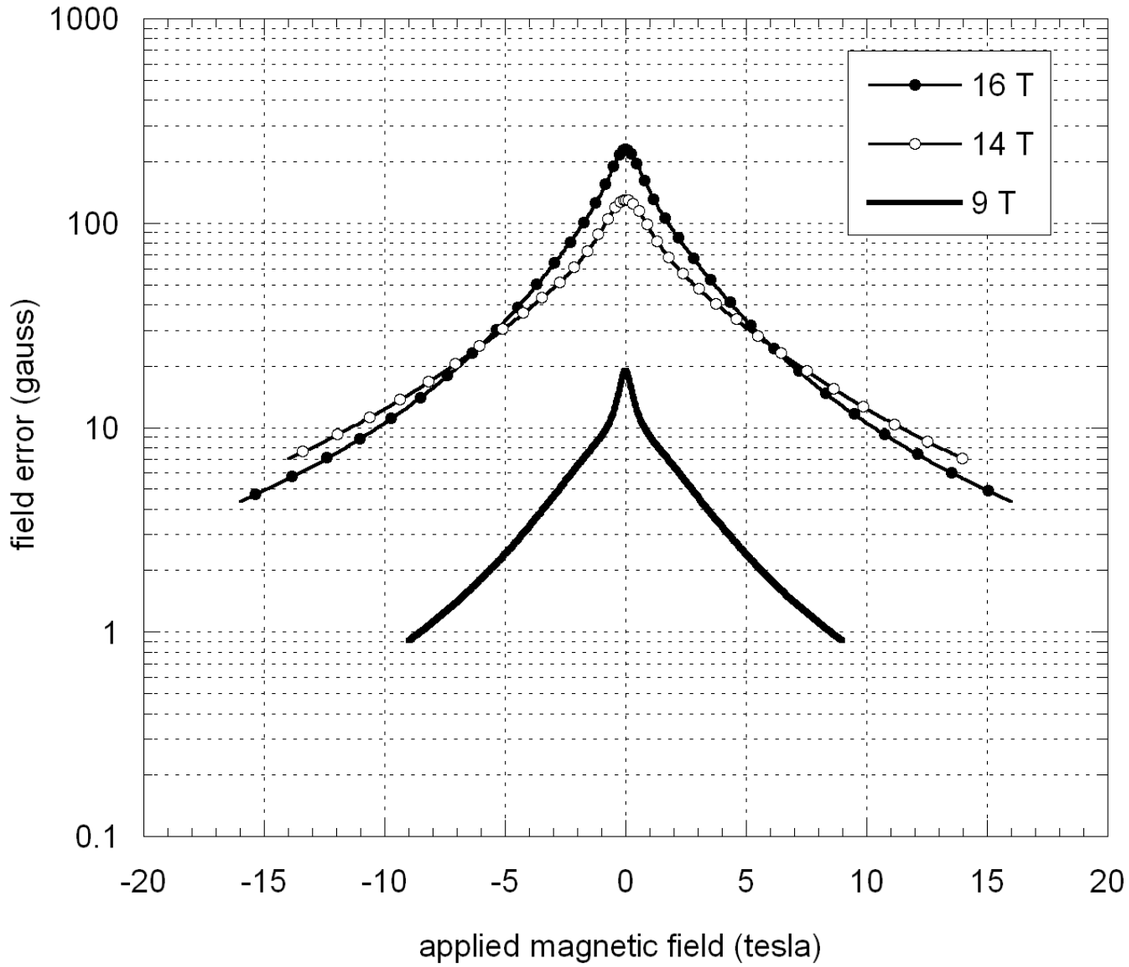
# Superconducting Magnets: Remanent Fields

**Reported field based only**
**on current in magnet power supply**
**-no field sensor in chamber**

**Accuracy issue for B < 1 T**

**Application Note: 1070-207**




**Measure the Pd standard using VSM to correct**
**Application Note 1500-021**

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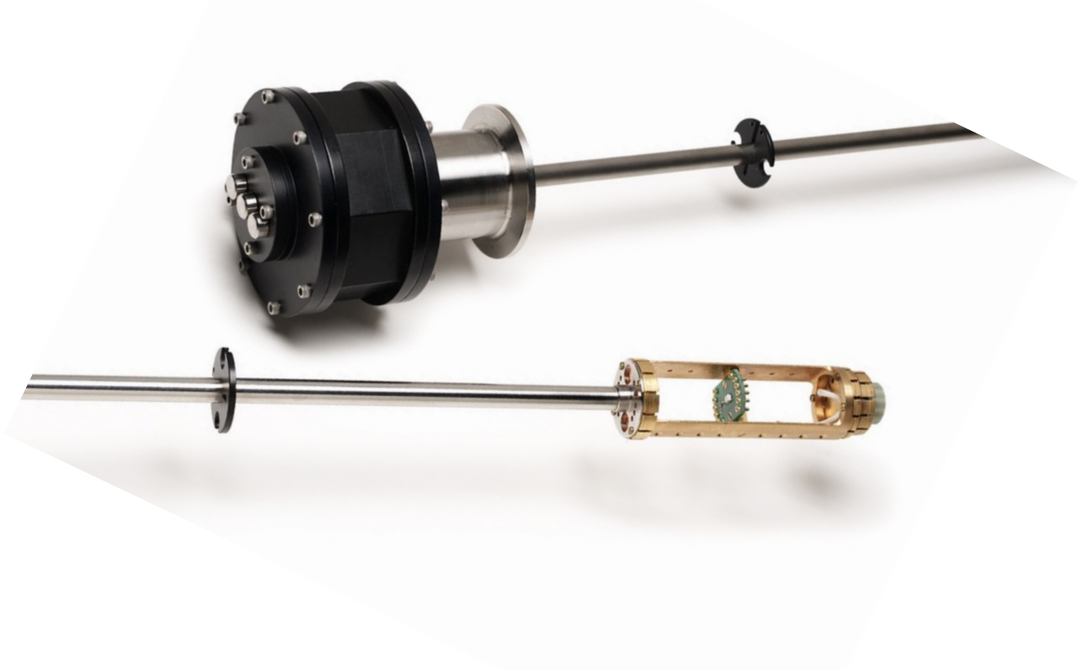
# SUPPLIES FOR CUSTOM EXPERIMENTS

- For semi-rigid coax above 1GHz: coax.co.jp
- For flexible coax above 1GHz: SHF or Totoku Electric
- For flexible coax below 1GHz: New England Wire Technologies
- For “standard” RF hermetic feedthroughs (good down to 10e-7 Torr) Pasternack or Fairview Microwave
- For UHV RF feedthroughs: Solid Sealing Technology or CeramTec
- For fiber-optic feedthroughs: Thor Labs or Solid Sealing Technology



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# MULTIFUNCTION PROBE
# EXAMPLE 1: PHOTOCONDUCTIVITY

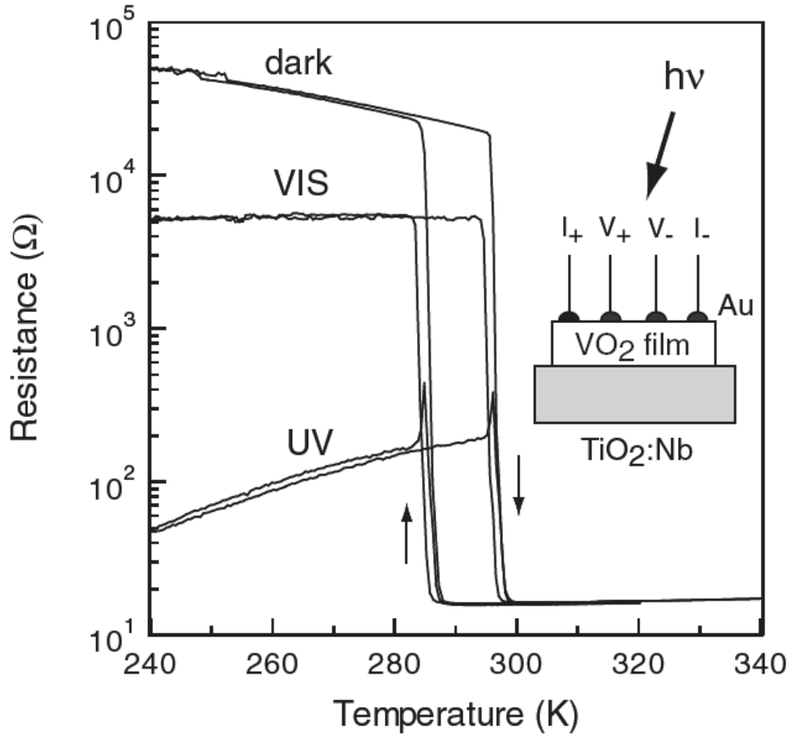


**Figure 1.** The temperature dependence of the in-plane resistance measured on cooling and heating, using a four-probe method in a Quantum Design physical property measurement system (PPMS). The sample is a 10 nm thick $VO_2$ film grown on an n-type $TiO_2$:Nb (nominally 0.05 wt%) substrate with 0.5 mm thickness. Gold electrodes were put on the $VO_2$ film to obtain an ohmic contact. A current flow was measured at a constant voltage of 0.2 mV. An UV light (wavelength: 300–400 nm; irradiance: 140 mW $cm^{-2}$) or a visible (VIS) light (wavelength: 400–700 nm; irradiance: 350 mW $cm^{-2}$) from a xenon lamp was guided via an optical fibre into the PPMS to irradiate the sample during the measurements.

Muraoka et al.J. Phys.: Condens. Matter **14** (2002) L757–L763


**BANANA BREAKOUT BOX (3084-100)**

**QDUSA.COM**
**QUANTUM DESIGN**
**PARTS & SPARES**
**WEBSITE**

**BNC BREAKOUT BOX (4087-457)**


**Fischer-to-LEMO Cable (3084-384)**
**Useful for Type-B Variant (and base system)**


**LCC Chip Carrier Test Station (4087-455)**

**Fisher-to-Fischer Cable (3084-383)**
**Useful for Type-G and Type-M Variants**

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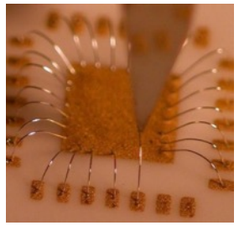
# SAMPLE PREPARATION

- ATTACHING LEADS:
  - Soldering (not possible for many materials)
  - Spot-welding (requires large sample/wire)
  - Conductive paint (good control; mechanically weak)
  - Conductive epoxy (harder to work with; mechanically strong after cure)
  - Deposit conductive material directly (req. deposition system)
  - Wire bonding (req. specialized equipment/training)

- CHOICE OF LEAD MATERIAL:
  - Gold/Silver/Copper wire (depends heavily on how leads will be attached)
  - Wires of various conducting alloys, etc.



Wire Bonder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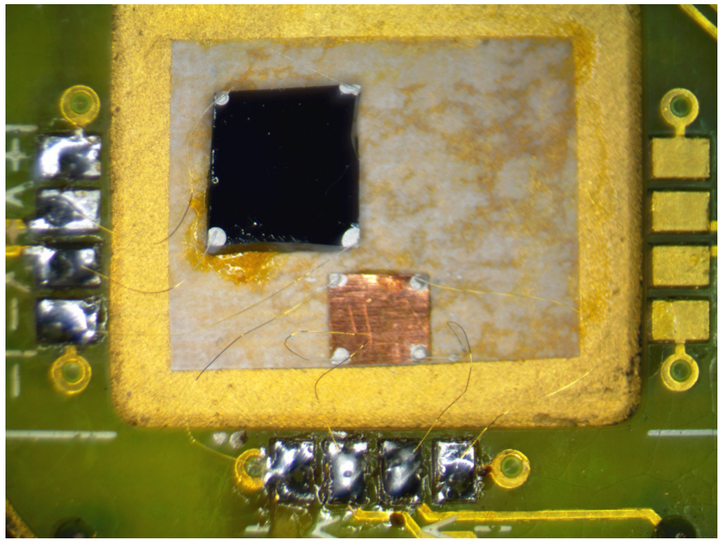
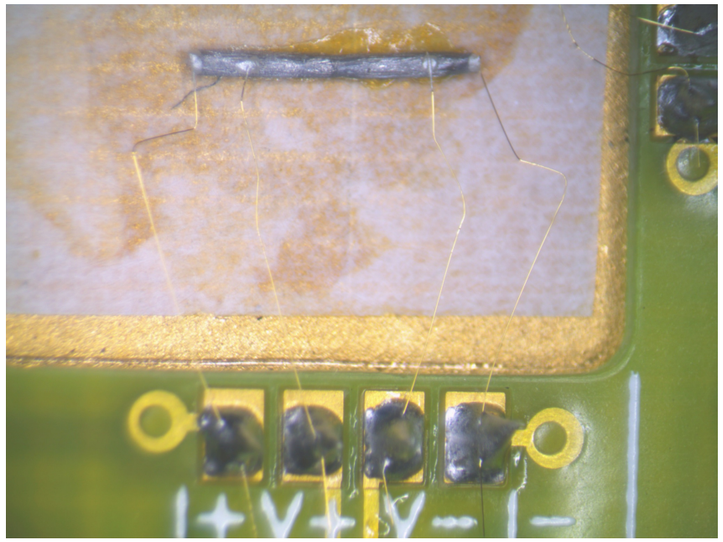
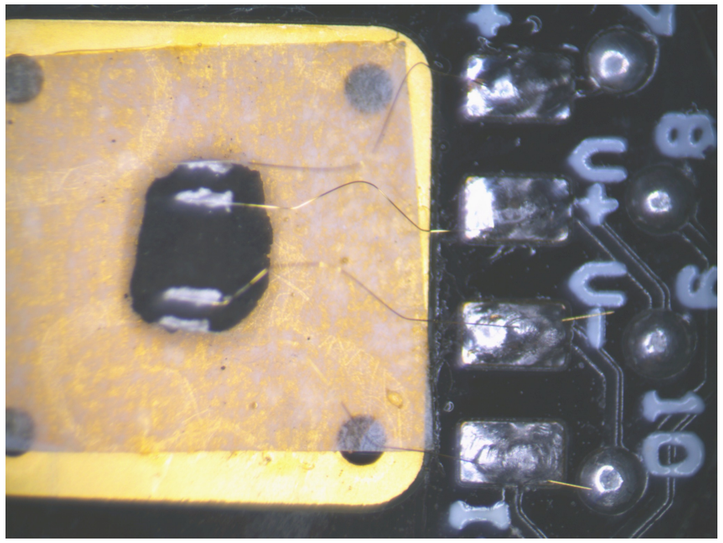
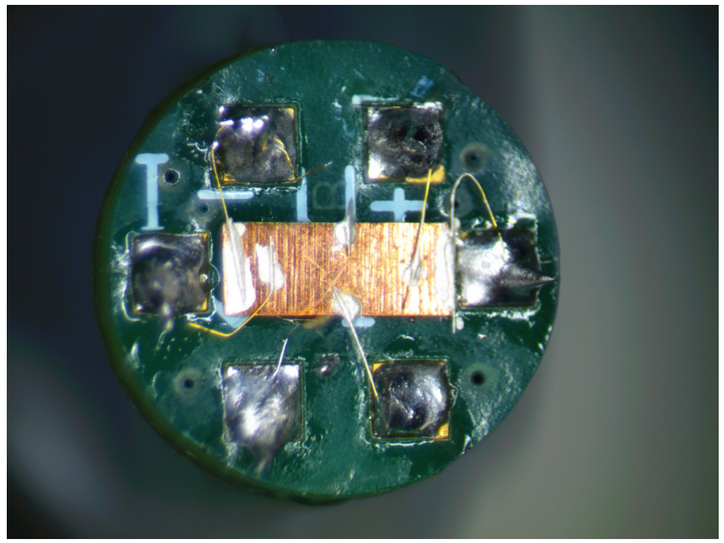
# SAMPLE PREPARATION





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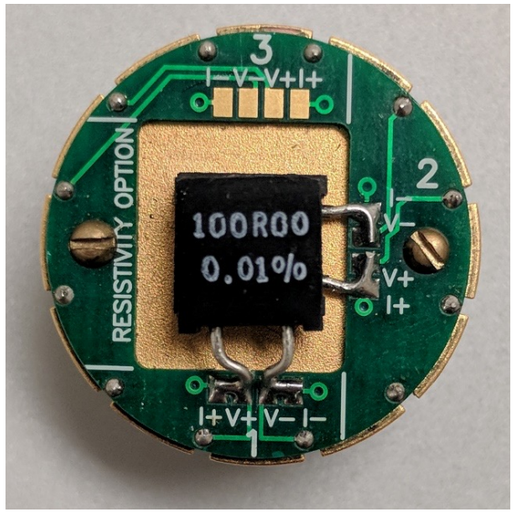
# CHECK SAMPLE CONTACTS AND CONNECTIONS

BAD CONNECTIONS ARE THE MOST COMMON
ISSUE WITH TRANSPORT MEASUREMENTS

Sanity Check Puck






AFTER SAMPLE MOUNTED ON PUCK OR ROTATOR BOARD
TEST CONTACTS WITH OHMMETER

IS THE CONTACT RESISTANCE TOO HIGH?  POLARITY?
REWORK CONTACTS (Ag Paint / Solder Blob?)

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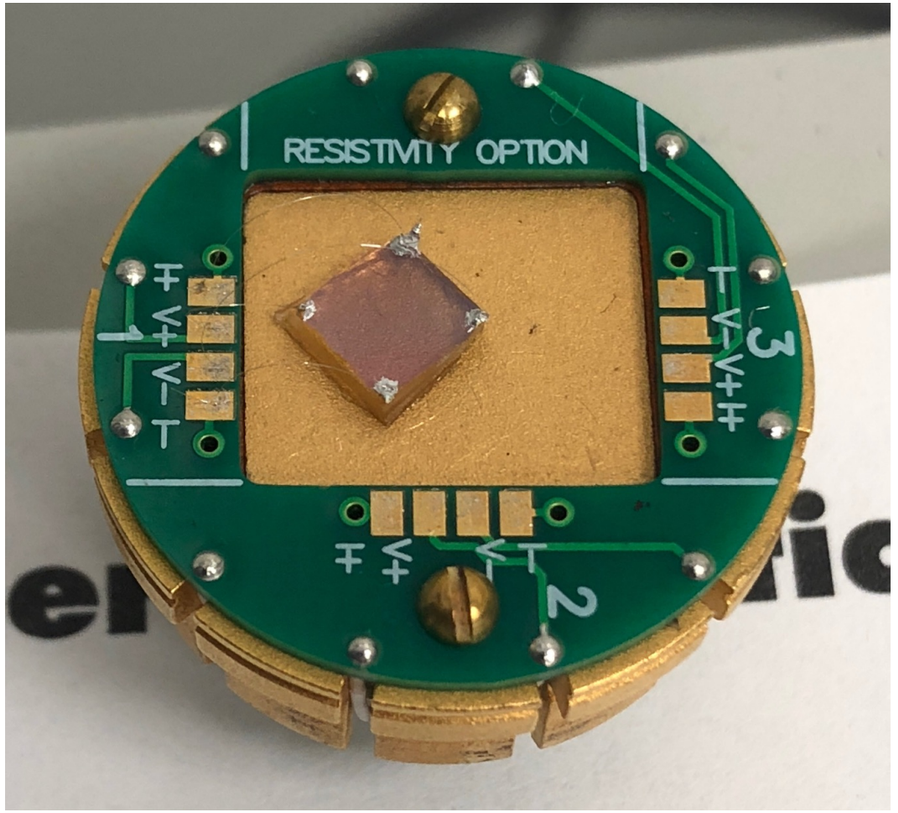
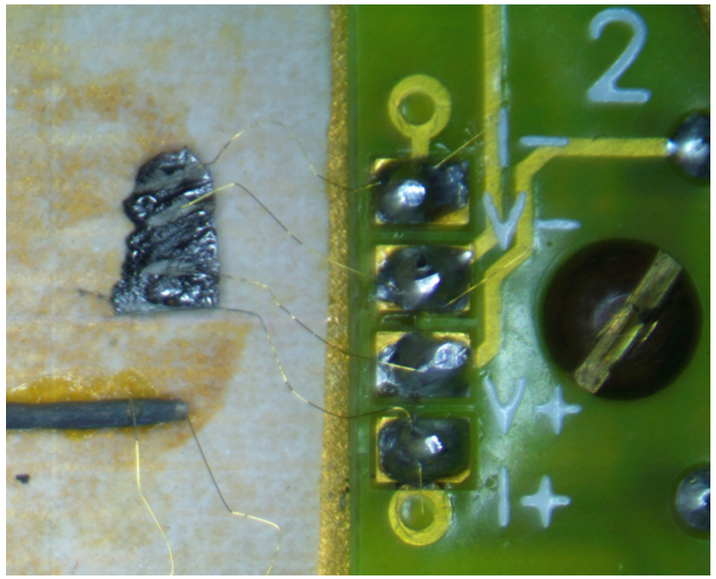
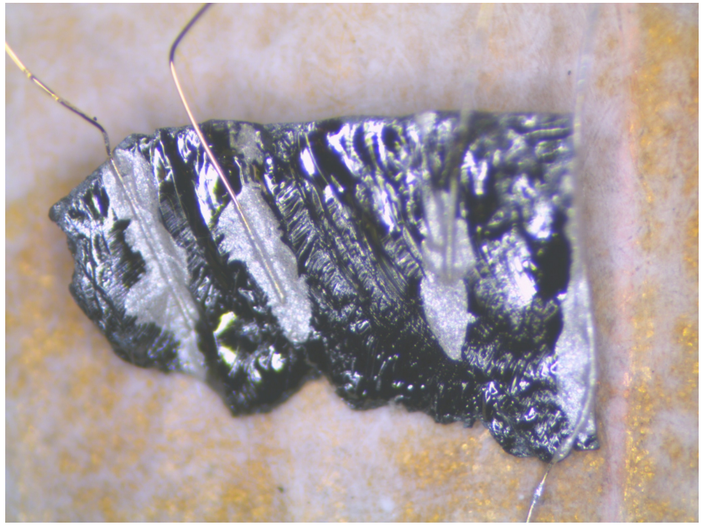
# ELECTRICAL TRANSPORT SAMPLE PREPARATION

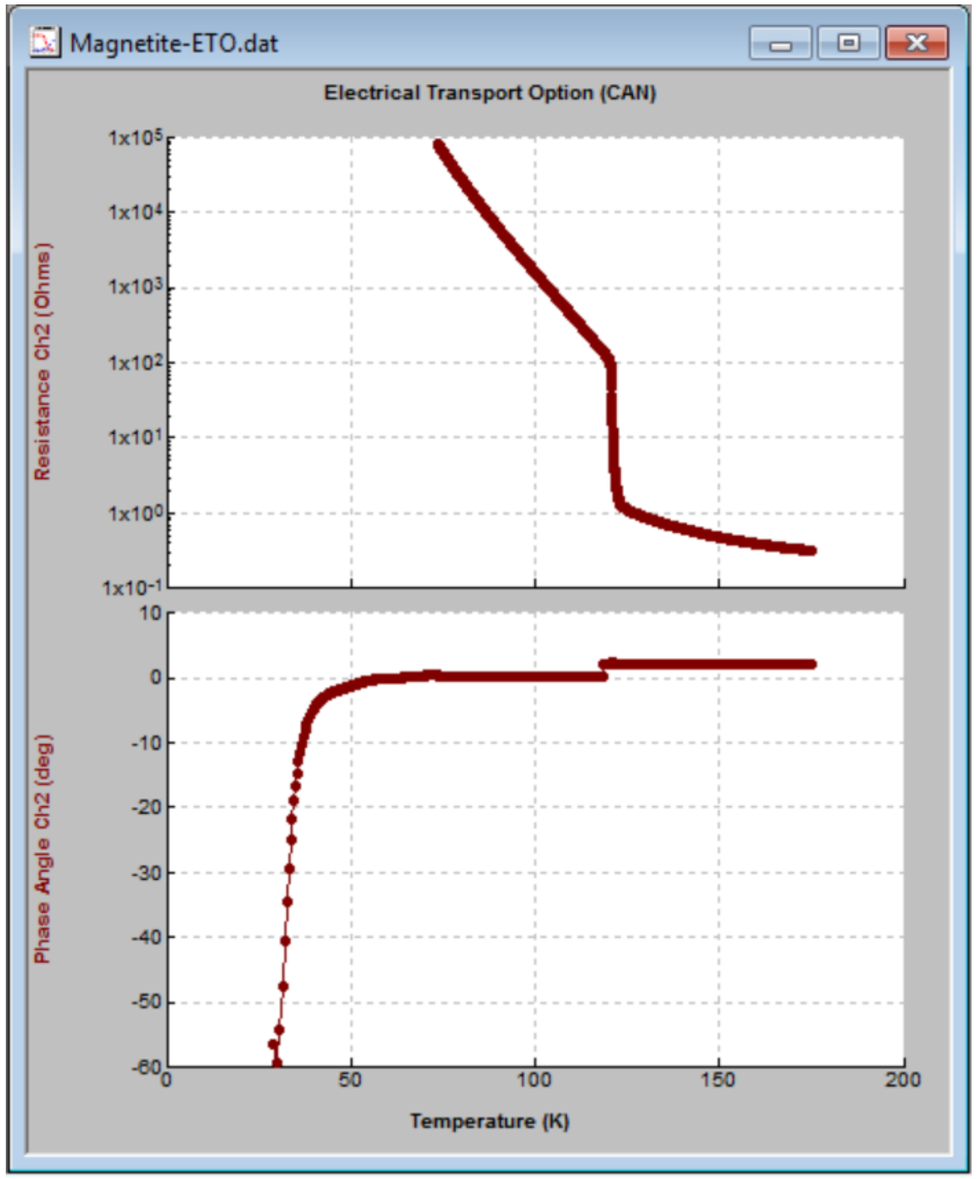
- Mount sample to the puck (Ch1/Ch2)
  - Electrically isolated
  - Thermally shorted
- Two-Probe Lead Configuration
  - High Resistance (1 MΩ – 5 GΩ)
  - AC voltage excitation
  - Current response measured via lock-in
  - Uses the +I/-V contacts
- Four-probe Lead Configuration
  - Lower Resistance (μΩ – 10 MΩ)
  - AC current excitation
  - Voltage response measured via lock-in



# ELECTRICAL TRANSPORT EXAMPLE



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# ELECTRICAL TRANSPORT: HALL EFFECT

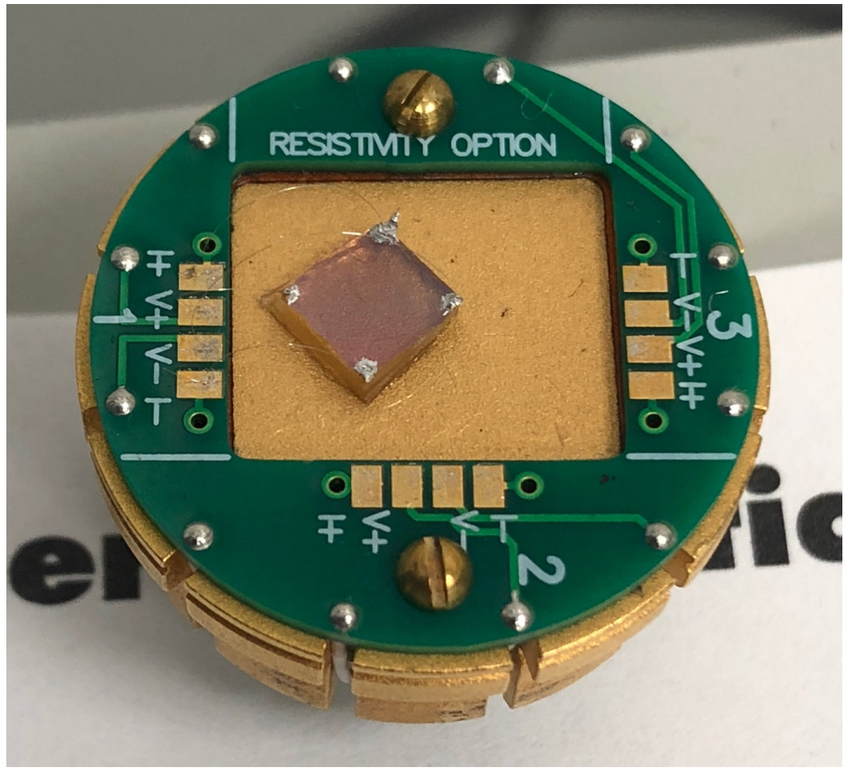
Voltage measured across the direction normal to that of the applied current

ETO reports the Hall Coefficient:

$$R_H = {}^{E_H}/_{(j\,B)} = \frac{A}{I} \times \frac{V_H}{l} \times \frac{1}{B}$$

Carrier density/type can then be extracted:

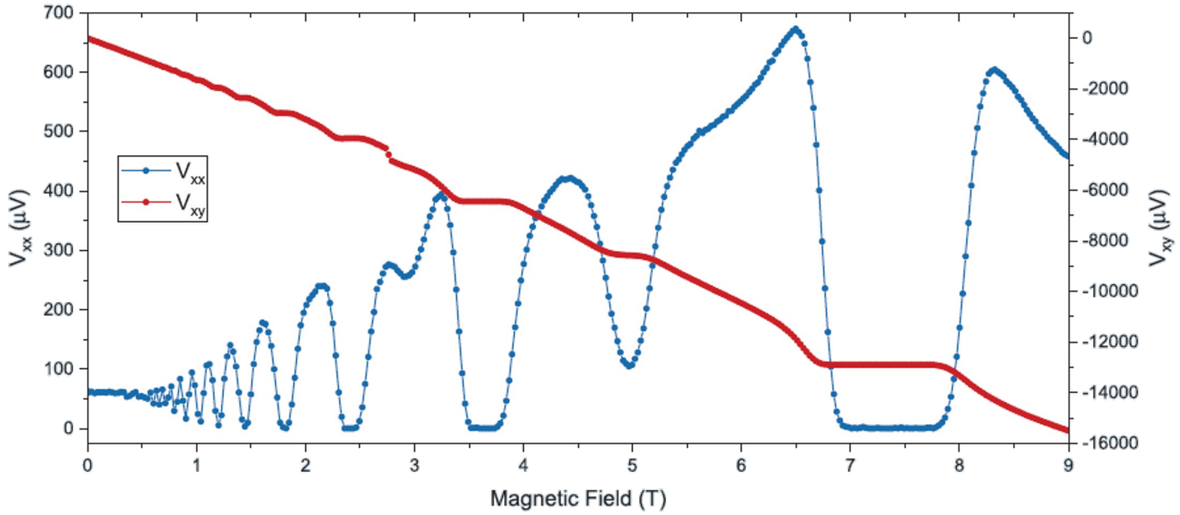
$$R_H = {}^{1}/_{(n\,q)}$$

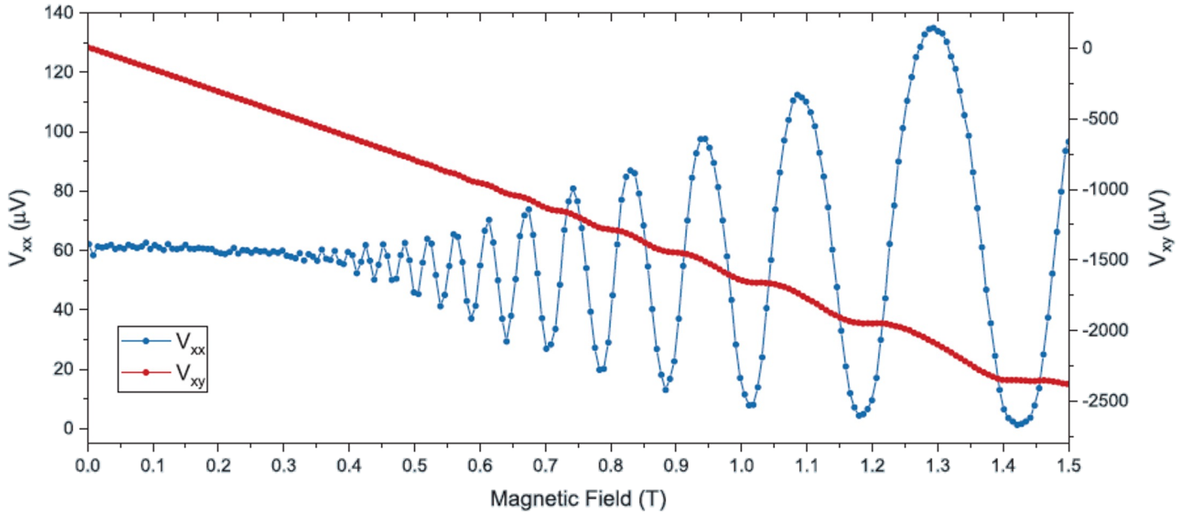


# D542 van der Pauw—Hall Transport

**Key Features:**

- Option software fully integrated to MultiVu, enabling sequence commands to configure a measurement of the van der Pauw resistivity or Hall coefficient
- IV-Curve utility enables a quick determination of sample contact quality at the beginning of a measurement
- Switching wiring permutations is handled automatically by MultiVu for common measurements (vdP, Hall), while user-defined switching profiles can be configured using custom sequences
- Three multiplexed measurement channels accommodated on a single puck
- Compatible with Helium-3 and Dilution Refrigerator options (two channels)


**GaAs 2D Electron Gas @ 1.7K**

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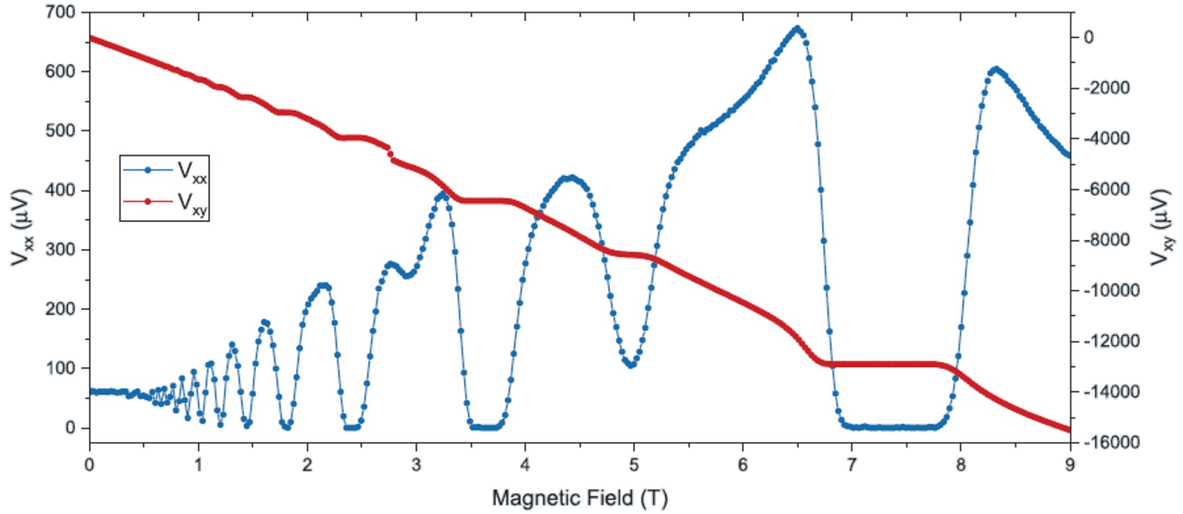
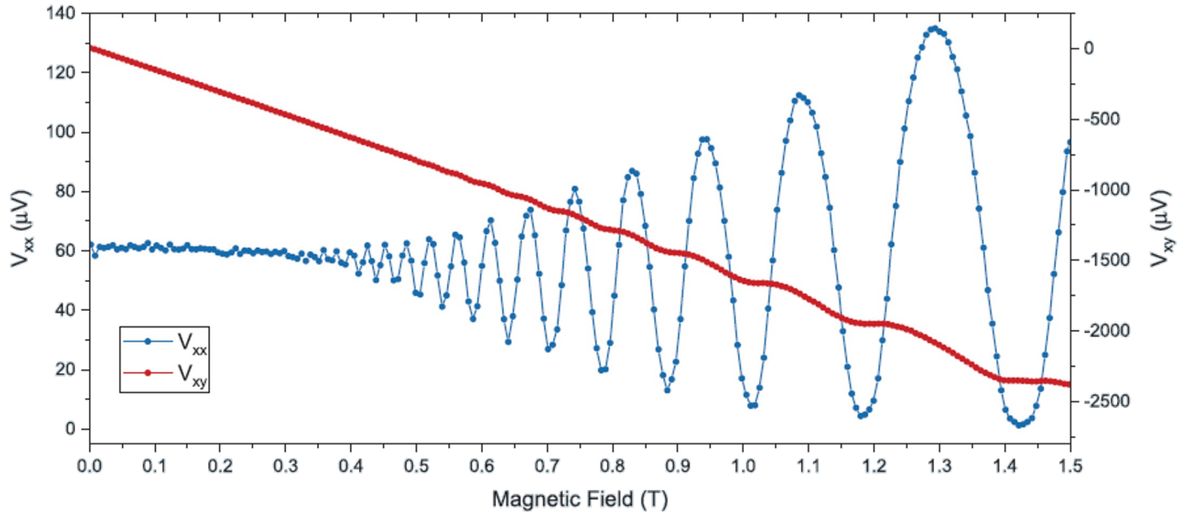
# M91 FastHall™ Measurement Controller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Lake Shore CRYOTRONICS
ADVANCING SCIENCE

Quantum Design

*Sample provided by Dr. M. Pendharkar, Chris Palmstrøm Group, UCSB*

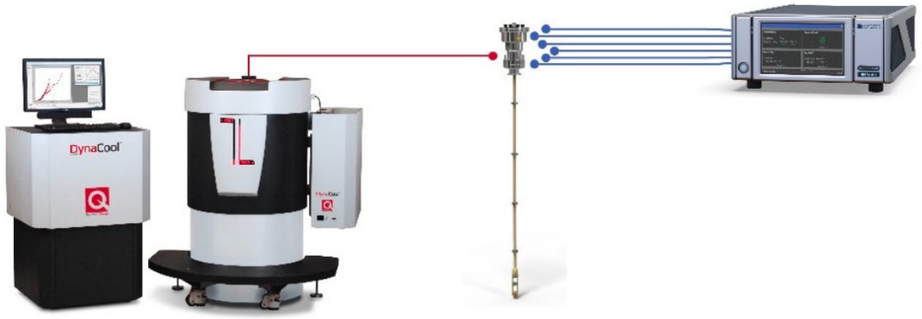
# M91 FastHall™ Measurement Controller

Adapter box connects M91 to PPMS

**M91-PPMS-KIT1**


Fully guarded signal path with insert from M91-HR to PPMS

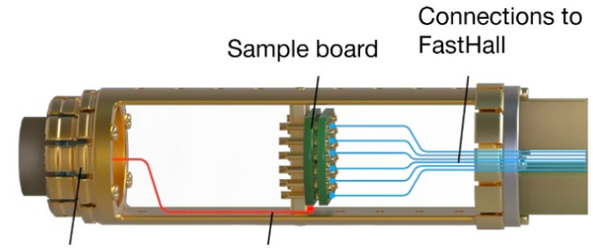
**M91-PPMS-KIT2**




**PPMS-INSERT-HALL**

Insert only is also available


Consumable Lake Shore sample board. Low cost, van der Pauw and Hall bar samples can easily be mounted to these boards.




Zoom in view of Hall insert. Only connection to PPMS is the sensor. Sample board plugs in here.

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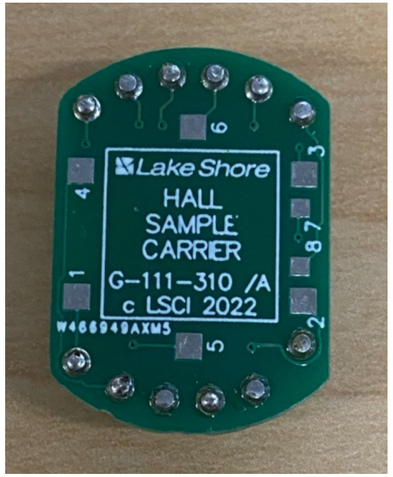
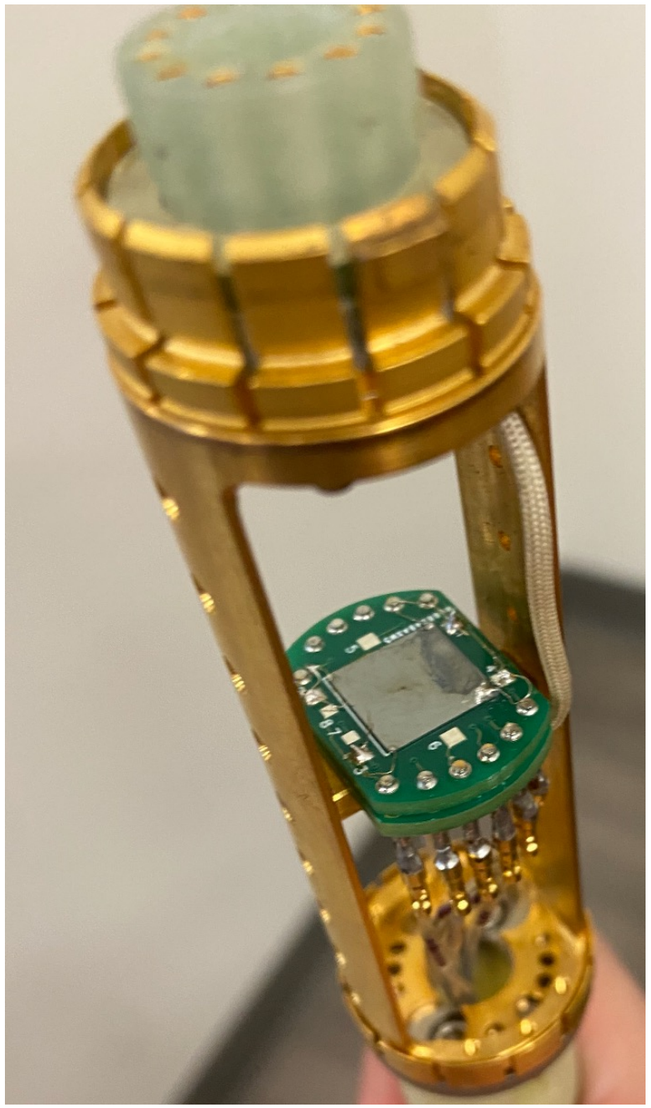
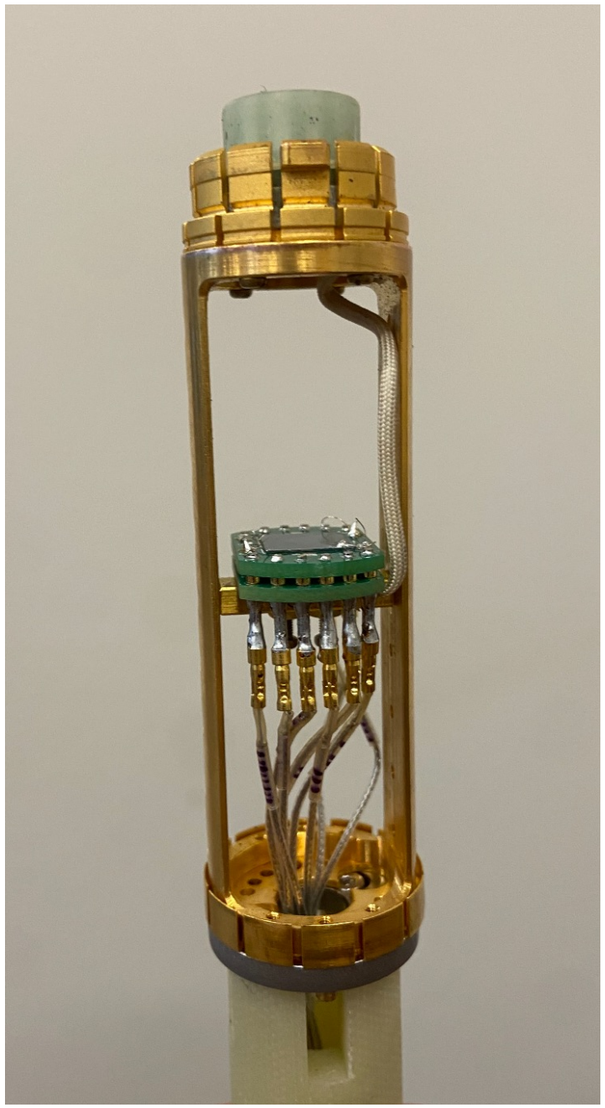
# LAKESHORE TRIAX GUARDED PPMS MEASUREMENT INSERT





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# M91 FastHall™ Measurement Controller

| | M91-PPMS-KIT1<br>Add M91 measurement benefits to a PPMS. | M91-PPMS-KIT2<br>Add M91 measurements benefits AND improved signal quality to a PPMS. Main benefit of insert is the signal path is fully guarded from the instrument to the sample. |
|---|---|---|
| | 10 mΩ | 10 mΩ |
| | 10 MΩ | 200 GΩ |
| | 10 mV to 10 V | 10 mV to 10 V |
| | 10 nA to 100 mA | 10 nA to 100 mA |
| | No field reversal required for FastHall method (van der Pauw samples) | No field reversal required for FastHall method (van der Pauw samples) |
| | PPMS sample puck | Lake Shore sample board |
| | Semi-guarded<br>Standard twisted pair wiring. Guarding stops at feedthrough box | Fully guarded<br>(Internal coaxial) from instrument to sample |
| | M91 standard resistance | M91-HR high resistance |
| | MeasureLink | MeasureLink |
| | ▶ Analysis kit datasheet<br>▶ Hall analysis on PPMS | ▶ Analysis kit datasheet<br>▶ One-page flyer<br>▶ Hall analysis on PPMS |

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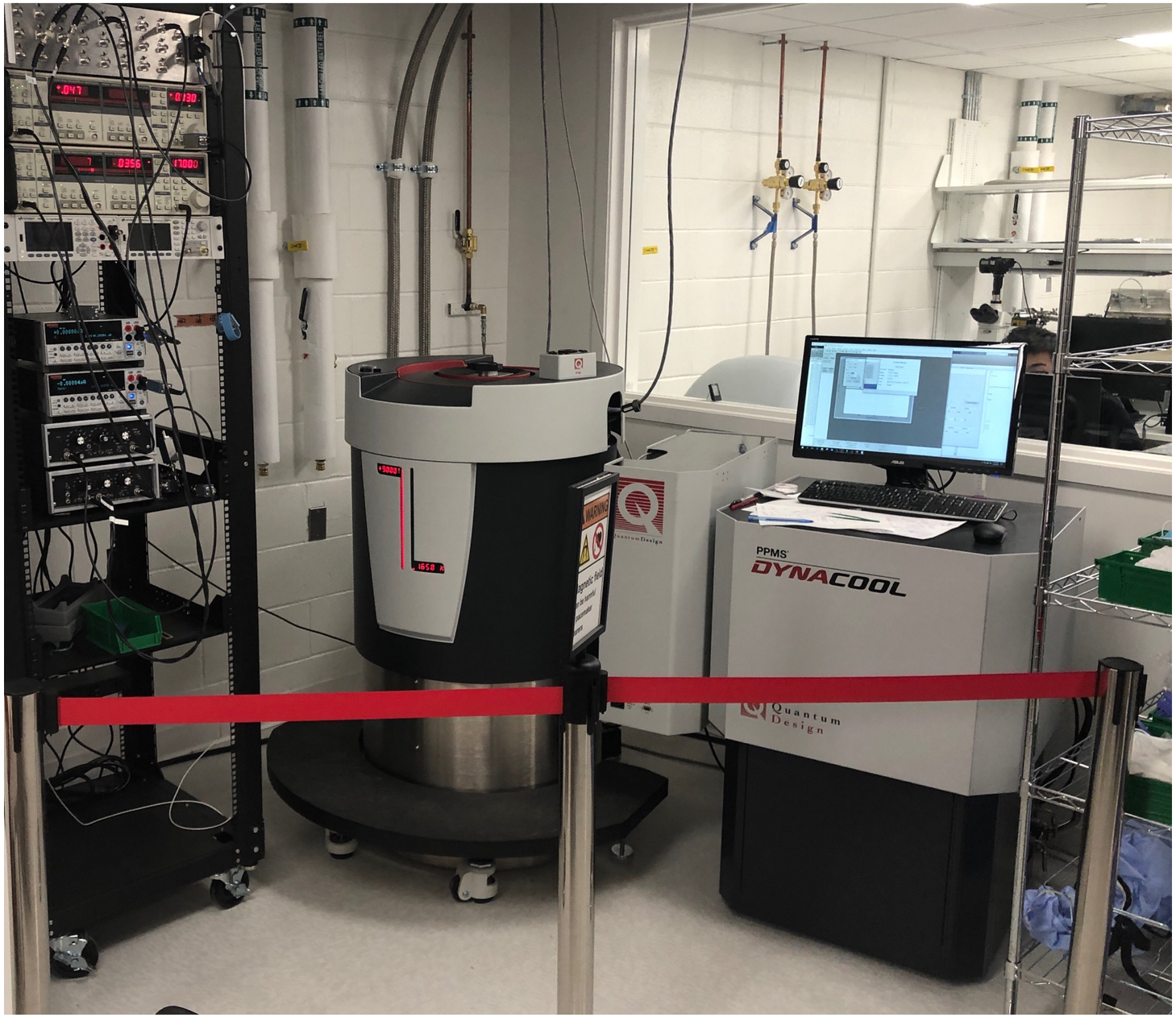


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# M81 SYNCHRONOUS SOURCE MEASURE SYSTEM - DC TO 100kHZ


| Instrument model | Maximum channel capacity | Number of source channels | Number of measure channels |
|---|---|---|---|
| M81-SSM-6 | 6 | 3 | 3 |

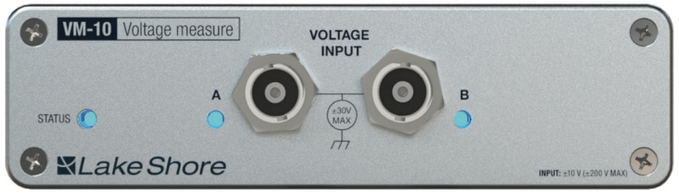
M81-SSM MODULES

## VS-10 voltage source module

2X


## VM-10 voltage measure module

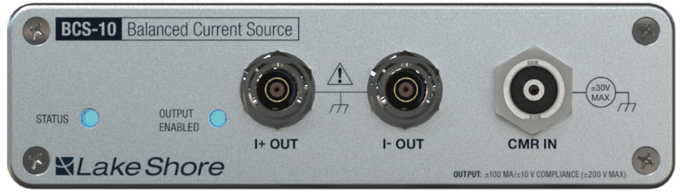
2X




M81-SSM MODULES

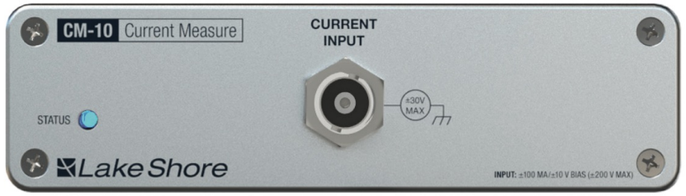
## BCS-10 balanced current source module

1X



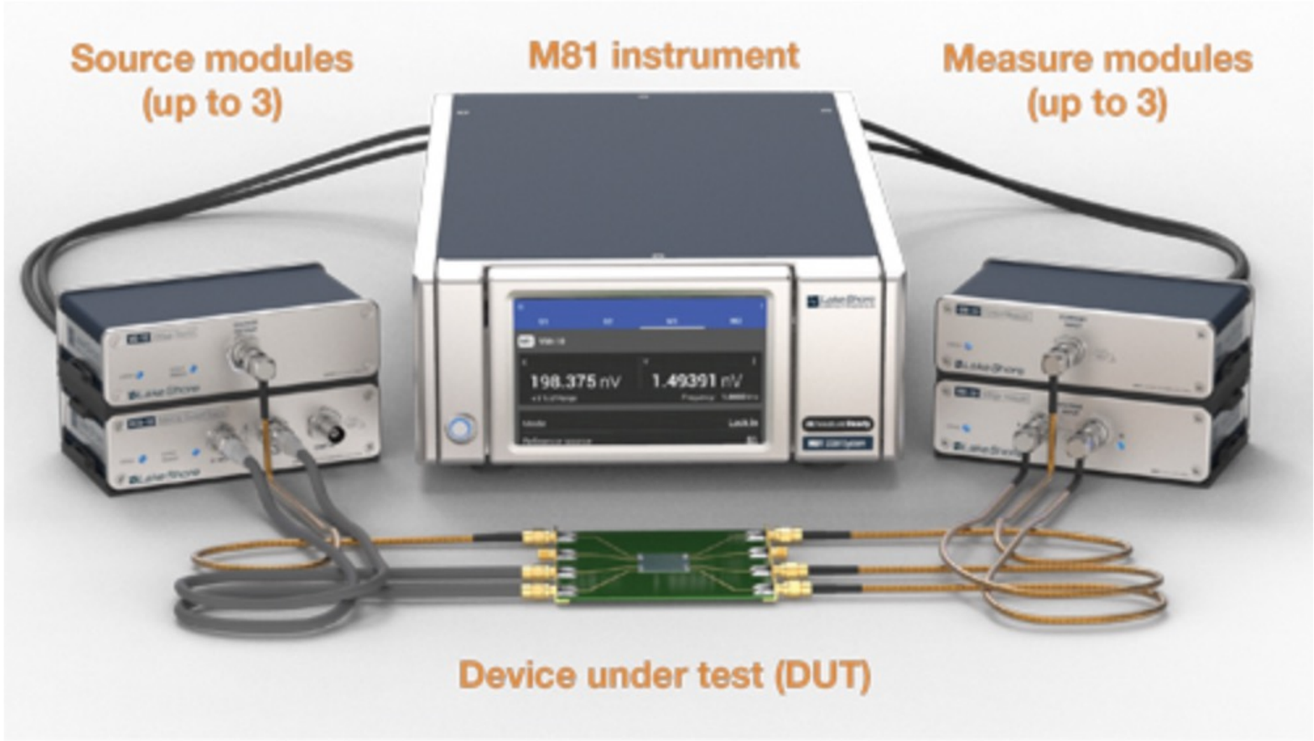

## CM-10 current measure module

1X




Quantum D

# M81 SYNCHRONOUS SOURCE MEASURE SYSTEM - DC TO 100kHZ




- Connect up to three source modules and up to three measure modules
- Exchange modules and adapt the configuration for each measurement
- All modules are capable of measuring with DC and AC to 100 kHz
- All modules are optimized for the highest precision with common amplitude and frequency references

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# M81 MODULES FOR A MULTI-GATED HALL-BAR DEVICE

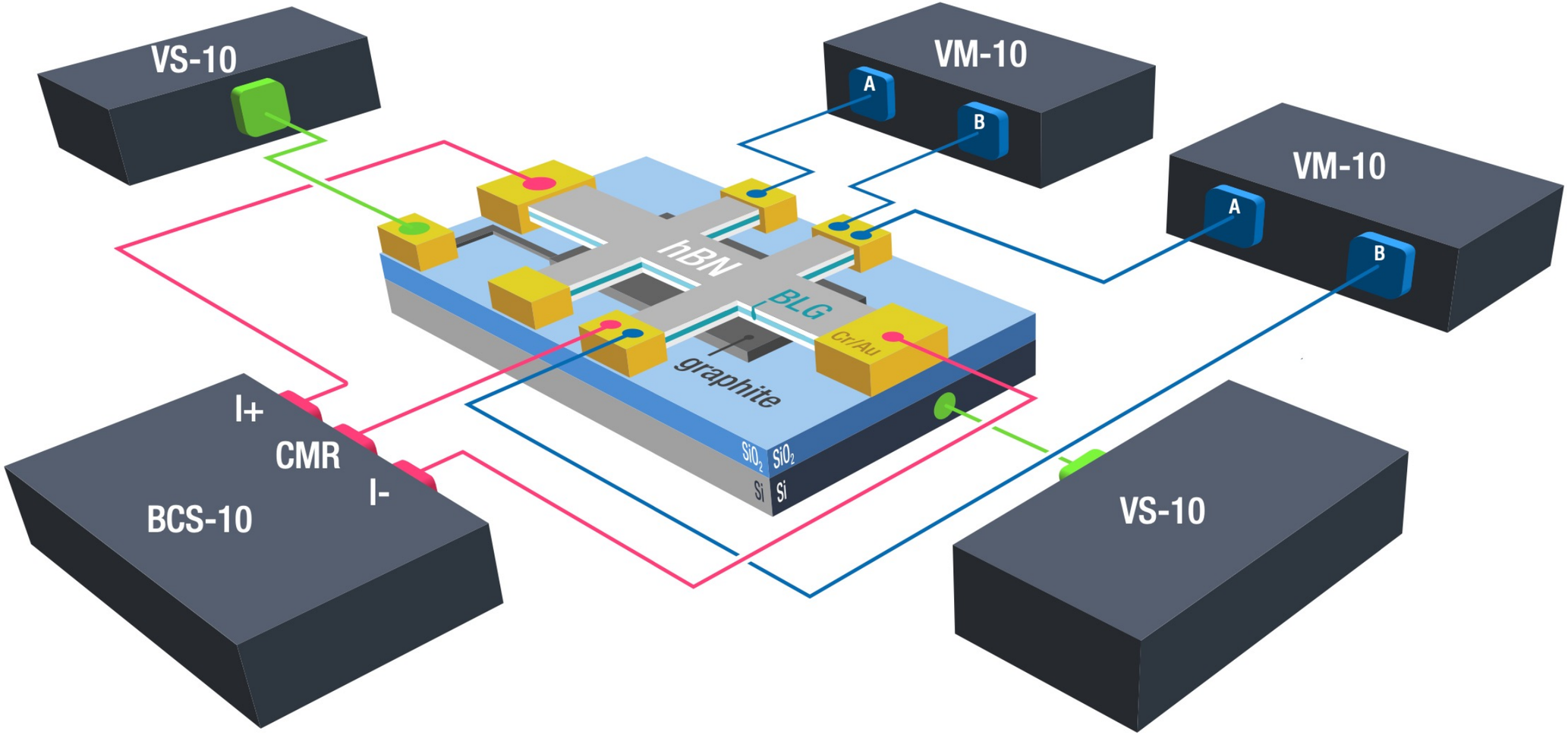



***Figure 1.*** *M81 wiring diagram to a multi-gated Hall-bar device.*

# TOP MATERIAL RESEARCH APPLICATIONS AND M81 MODULES USED IN THE MEASUREMENT

**DC transport**

### I-V curves, 4-wire

(VS module + CM module, primarily)
**Ideal for:** 2D materials, nanowires, organic semiconductors
**M81-SSM advantages:** Low-voltage source noise, low-current measure noise

**AC transport**

### AC resistance, sheet resistance, and AC current Hall

(BCS module + VM module)
**Ideal for:** Metal-insulator transitions, 2D materials, superconducting materials
**M81-SSM advantages:** AC current Hall: synchronous measurement of resistance and Hall voltages; and simultaneous measurement of up to three devices in a cryostat at different frequencies

### Photodiodes and phototransistors

(CM module + occasionally VS module)
**Ideal for:** IR sensitive materials, solar-blind materials, 2D materials
**M81-SSM advantages:** Programmable offset voltage source

**AC transport**

### Spin transport

(DC/AC: BCS module + VM module)
**Ideal for:** Spin orbit torque (SOT), non-local resistance, spin valves
**M81-SSM advantages:** SOT: synchronous measurement of resistance, Hall voltages, and harmonic Hall voltages

### Differential conductance

(VS module + CM module)
**Ideal for:** MIS junctions, Josephson junctions, defect characterization in transistors
**M81-SSM advantages:** Junctions: dual DAC AC and DC sourcing (source at appropriate range)

### Thermal transport

(AC, BCS module + VM module)
**Ideal for:** Thermoelectric materials, 1D materials
**M81-SSM advantages:** Phase-correlated current sources, synchronous harmonic detection

# MPMS3 SQUID MAGETOMETER


## MPMS 3 Features

- Cryogen Free with EverCool
- SQUID Sensitivity
- Multiple Measurement Modes
  (Including Traditional MPMS DC Scan)
- Temperature Range: 1.8 – 400 K
- 7 Tesla Magnet

## MPMS 3 Options

- SQUID AC Susceptibility Measurement:
  0.1 Hz to 1 KHz, sensitivity: 5 x $10^{-8}$ emu at 0 T
- Ultra-Low Field Capability: ±0.05 G with 7 T magnet
- VSM with Oven up to 1000 K
- Magneto-Optic Measurement (FOSH, Light Source)
- Horizontal Rotator
- High Pressure Cell for Magnetometry
- Helium-3 Insert

# Electrical Transport Option (for MPMS3)

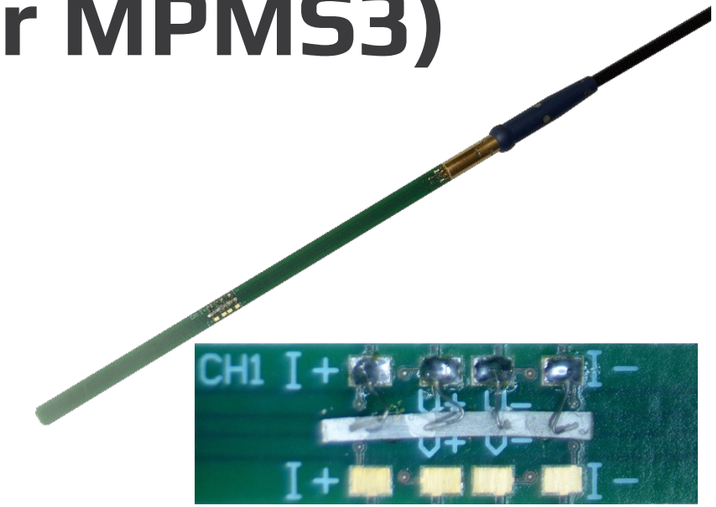
**Measurements:**
- AC resistance
- Hall effect
- Differential resistance/conductance

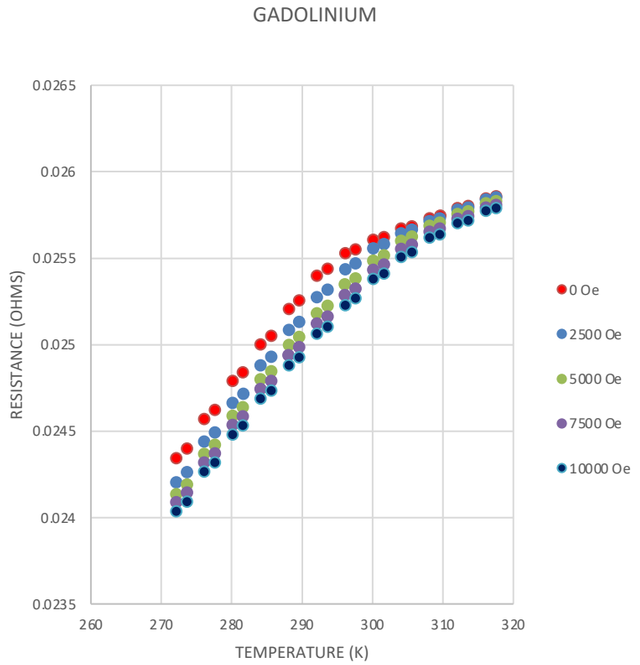
**Resistance Range:**
- <1 μΩ – 10 MΩ (4-probe)
- 2 MΩ – 5 GΩ (2-probe)

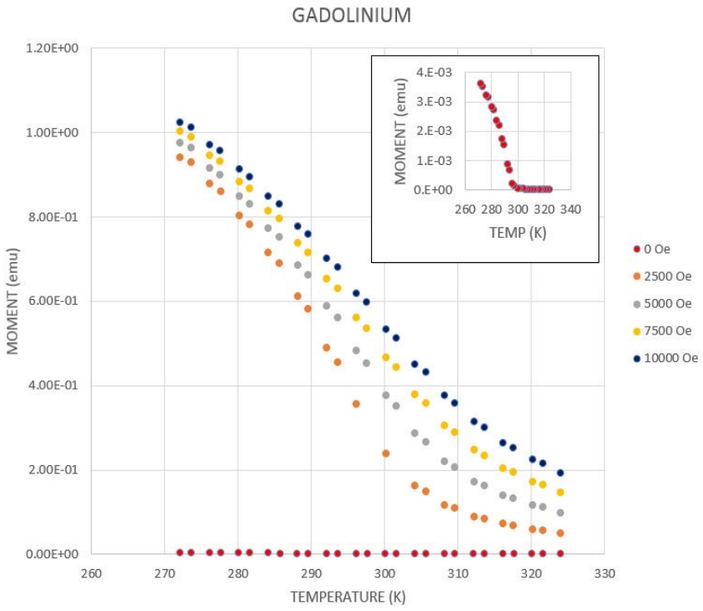
**AC Current Range:**
- 10 nA – 100 mA

**Experiment Leads:**
- 8 (2x 4-probe res. channels; parallel)
- 6 (1x 4-probe + bias; perpendicular)

**Orientations:**
- Plane parallel H-field
- Plane perpendicular H-field

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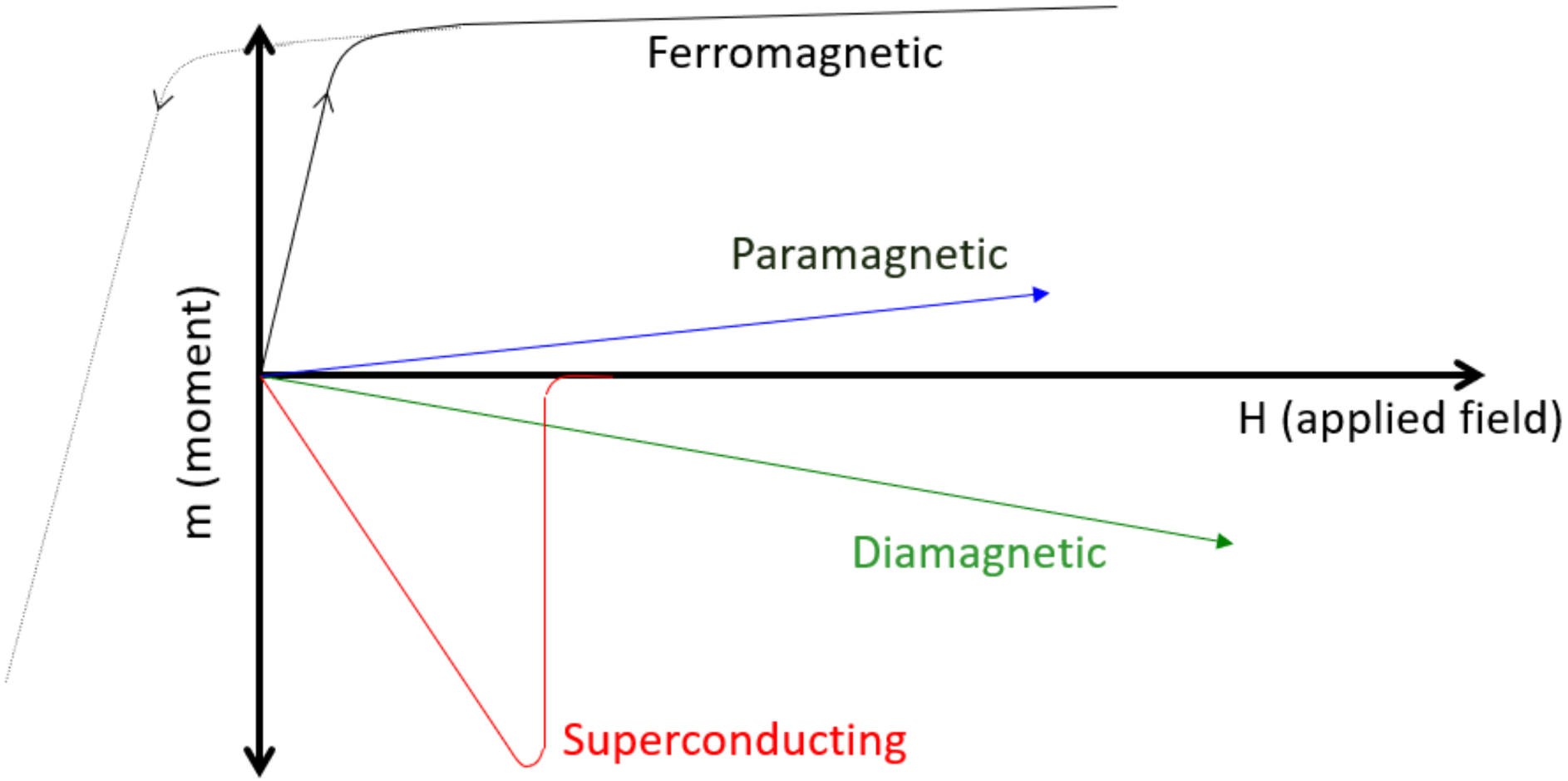
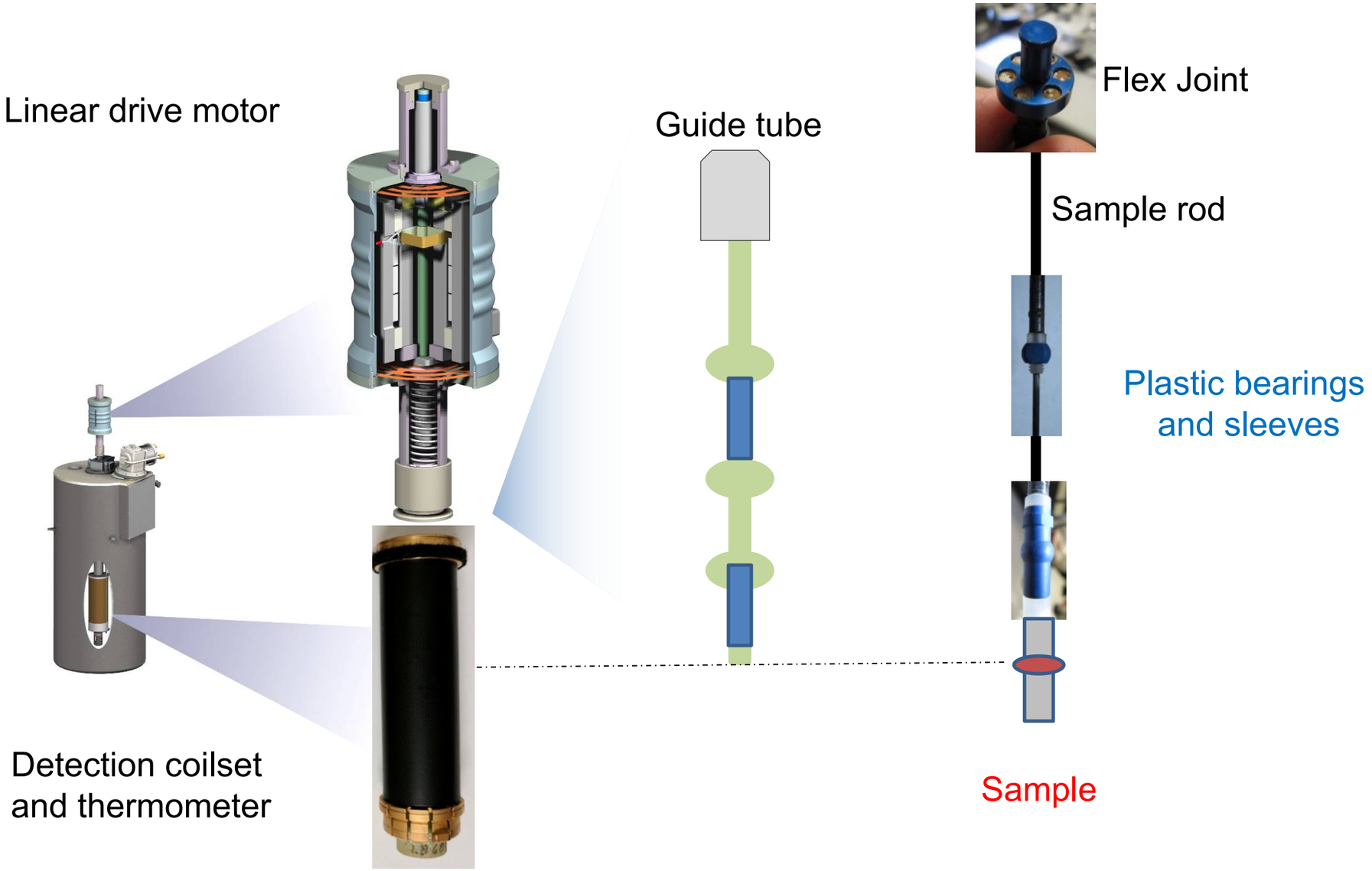

Ferromagnetic
Paramagnetic
m (moment)
H (applied field)
Diamagnetic
Superconducting

# VSM: Hardware

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# SUMMARY OF MAGNETOMETRY METHODS

## VSM (1.8-400 K or 300-1000 K with oven)

Standard coilset sensitivity: $7 \times 10^{-7}$ emu

Large bore coilset sensitivity: $1 \times 10^{-6}$ emu

## ACMSII (1.8-400 K)

AC Drive amplitude: 0.005-15 Oe (peak-to-peak)

AC Frequency range: 10 Hz-10 kHz

AC Sensitivity: $1 \times 10^{-8}$ emu

(order of magnitude decrease in sensitivity for every order of magnitude in drive frequency)

Phase resolution: 0.5°

**DC Sensitivity: $5 \times 10^{-6}$ emu**

# DYNACOOL VSM SAMPLE HOLDERS

***Quartz Holder***

Magnetically clean but a little fragile → small moment + high-sensitivity

***Brass Holder***

Robust but ~$10^{-6}$ emu background → large moments (do not use for AC)

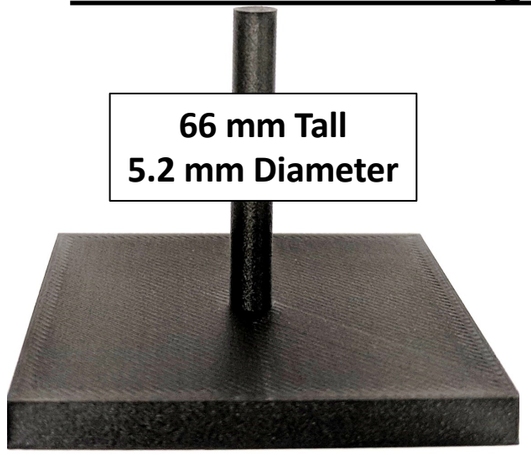
***Straw Holder***

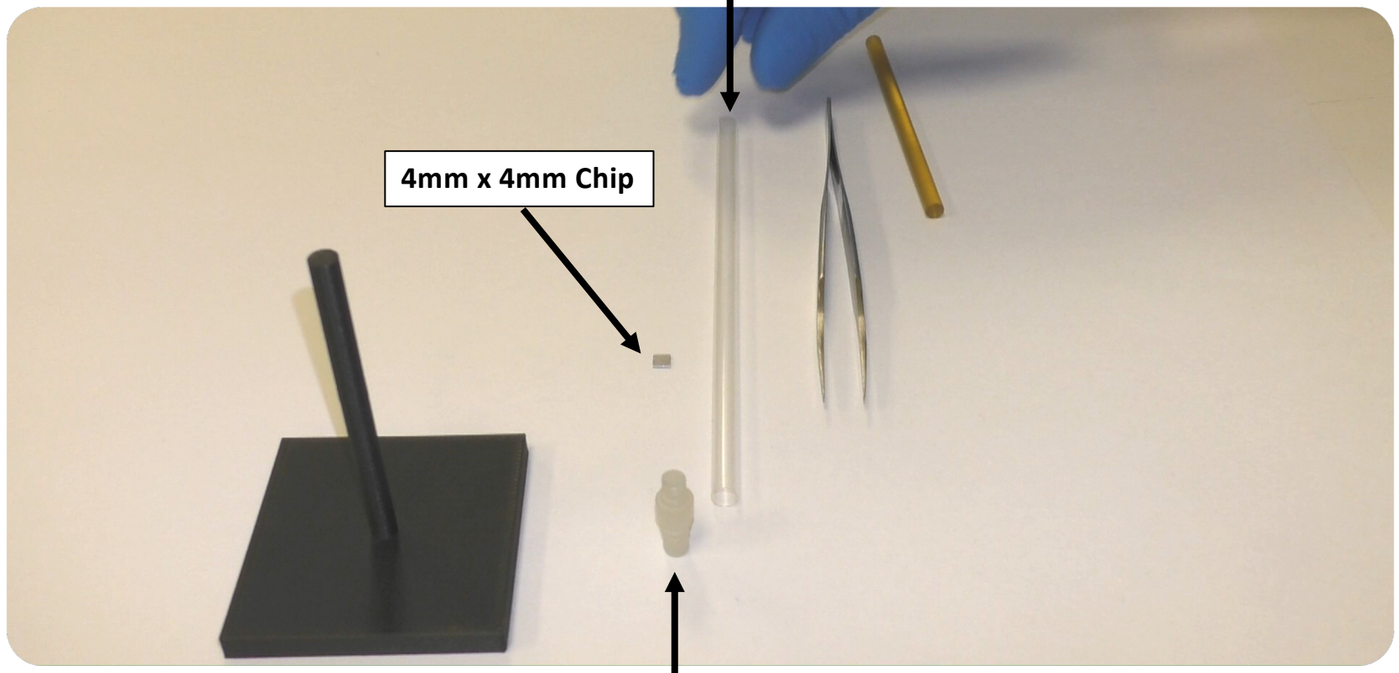
Magnetically clean ideal for DC scan mode

# DYNACOOL VSM STRAW SAMPLE HOLDER

- Drinking straws:
  - See QD for straws (AGC2-BOX) and straw adapter (4084-815 or 4500-614)
  - Perpendicular field: Cut wafer into 4 mm x 4 mm chip

## Straw Mounting Jig

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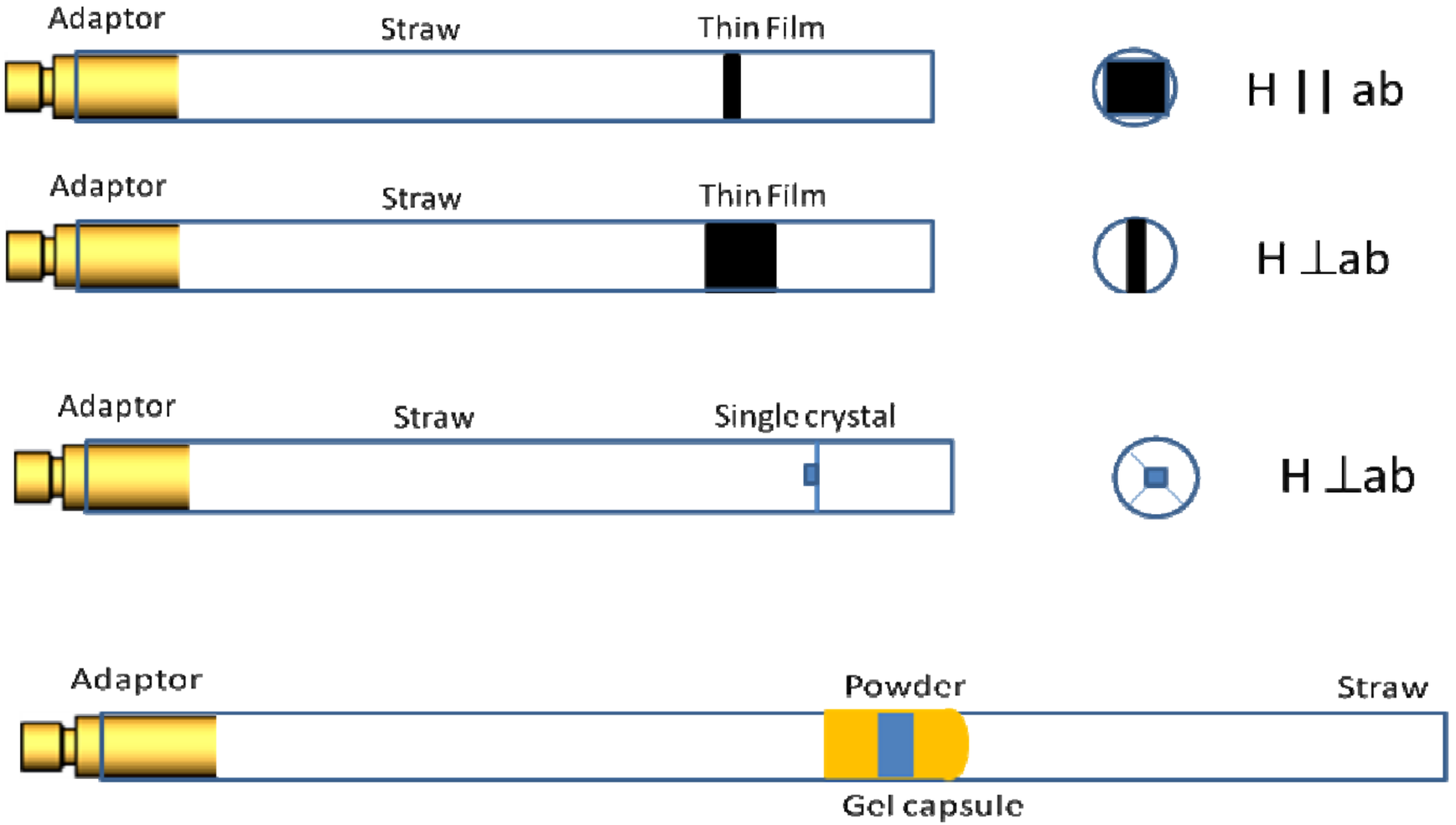
# DYNACOOL VSM STRAW SAMPLE HOLDER

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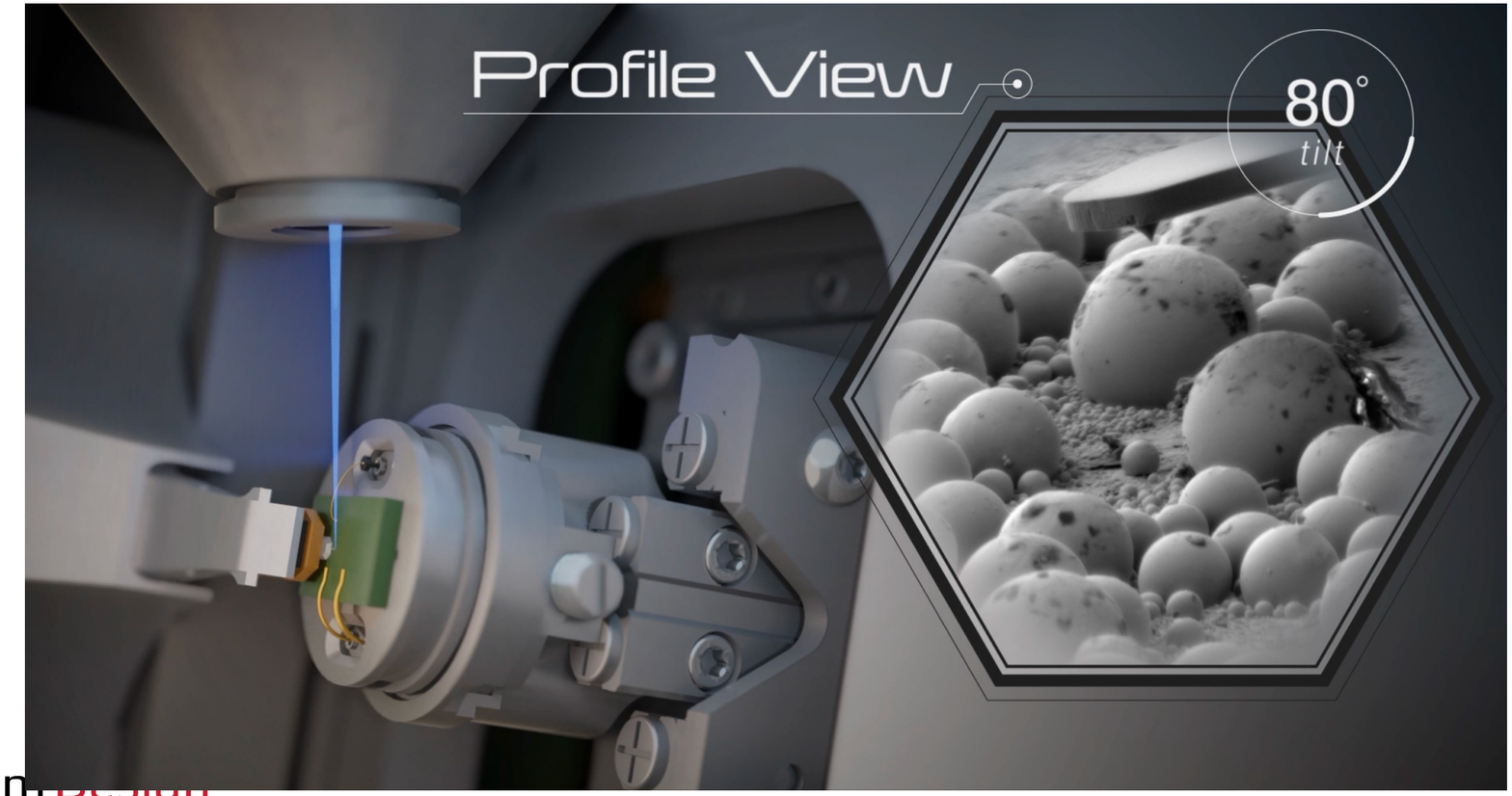
# FUSIONSCOPE SEM & AFM MICROSCOPE

**Seamless Combination of AFM and SEM Techniques**
Allows for measurement of an exact spot on the sample.
Automatically aligns both AFM and SEM operations for measurements and sample positioning.
**Dynamic View**
interactively overlay AFM imaging data onto SEM images while operating the microscope **in real time**
**Joint Coordinate System**
Allows locating an area of interest with the SEM and then automatically find that same area with the AFM.




fusion scope™ by Quantum Design

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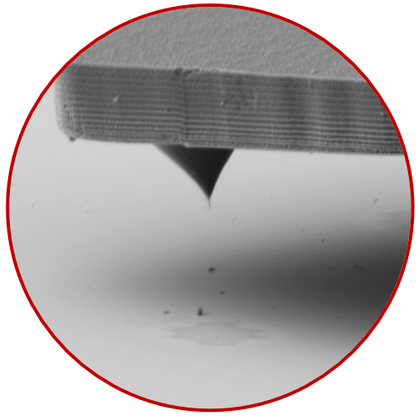
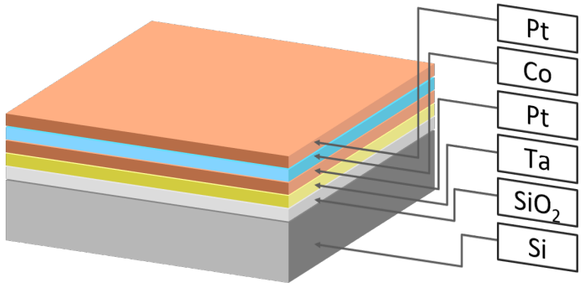
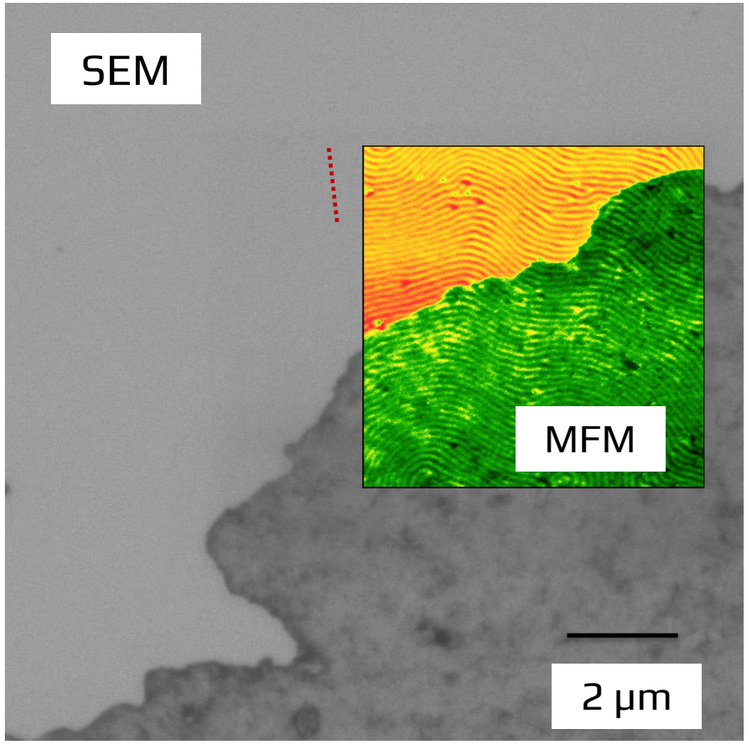
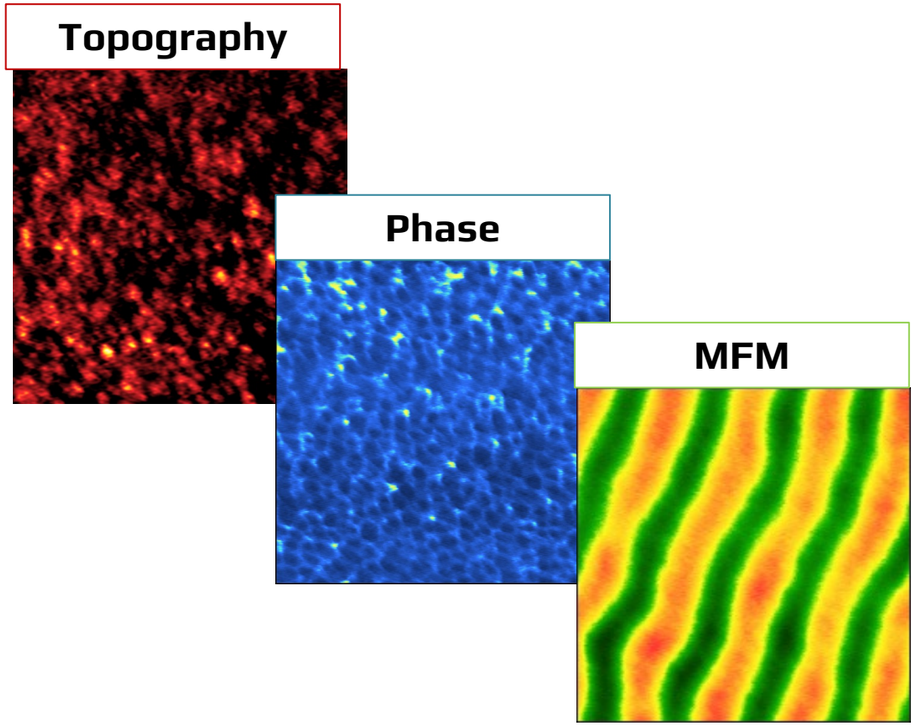
# Application Examples – MFM of Multilayer Co/Pt/Ta sample

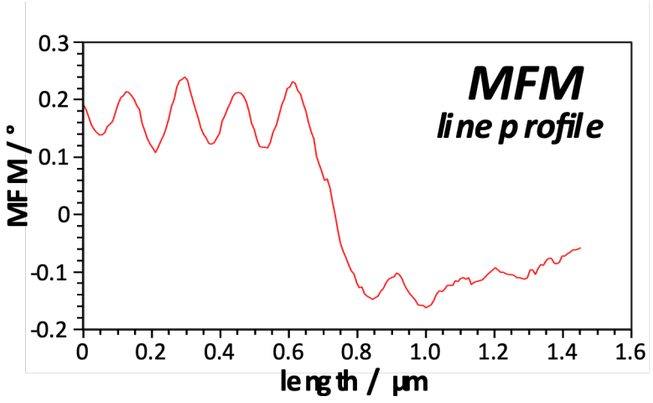


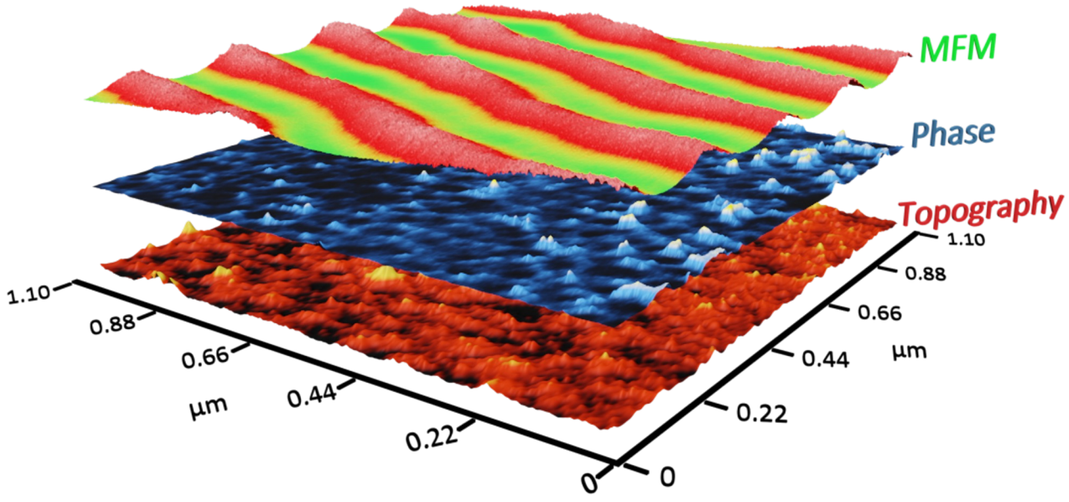



Multilayer Co/Pt/Ta sample
(Sample courtesy Prof. O. Hellwig &
Dr. Hlawacek, HZDR Dresden)



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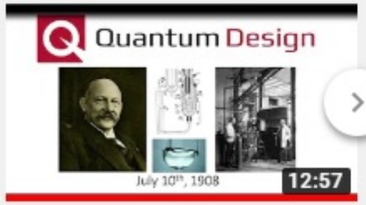


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# Application Notes

- Most Recently Added
- Base Systems
- Custom Experiments
- Electrical Transport
- Magnetometry
- Thermal Measurements
- Sub-Kelvin
- Spectroscopy

## Most Recently Added

- 1500-026 – Calibrating the Auto-Tracking capabilities for a custom sample holder using the MPMS 3 (MPMS 3)
- 1500-023 – Background subtraction using the MPMS 3 (MPMS 3)
- 1500-022 – MPMS 3 .rw.dat file format (MPMS 3)
- 1085-157 – 3D Heat Capacity Puck for PPMS DynaCool (DynaCool) – 02/19
- 1091-217 – Assigning an Absolute Scale to Susceptibility Measurements with the ACDR (PPMS, DynaCool) – 02/19

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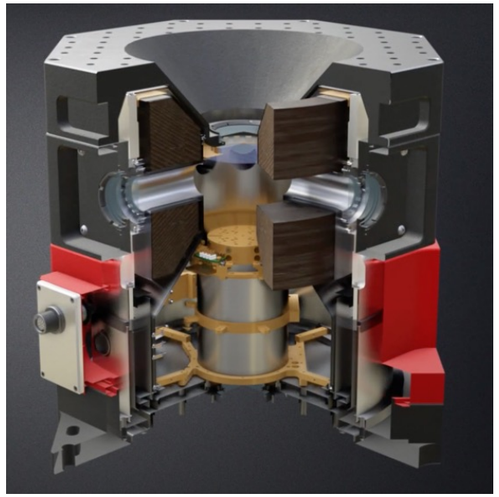
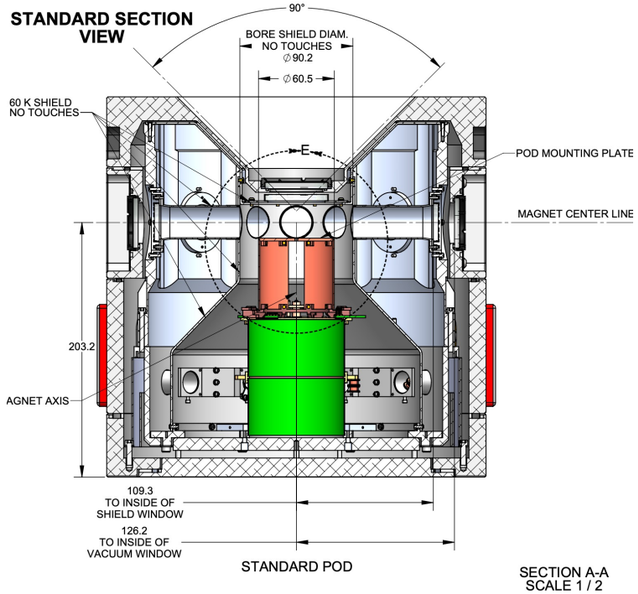
| MPMS3 |
| OptiCool |
| PPMS family (PPMS, Versalab, DynaCool) |
| QD Education |
| QD partner products |
| SHGMS magnetic separator |
| stand-alone SQUIDs |
| Windows 10 OEM Key |

Quantun

# OPTICOOL USER MANUAL
# CHAPTER 5 EXPERIMENTS IN OPTICOOL




| | |
|---|---|
| Chapter 1 | Getting Started |
| Chapter 2 | Hardware |
| Chapter 3 | Software |
| Chapter 4 | Theory of Operation |
| Chapter 5 | Experiments in OptiCool |
| Chapter 6 | Maintenance and Servicing |
| Chapter 7 | Pin-out and Interconnect Diagrams |
| Chapter 8 | System Setup |
| Appendix A | Options, Accessories, and Spare Parts |

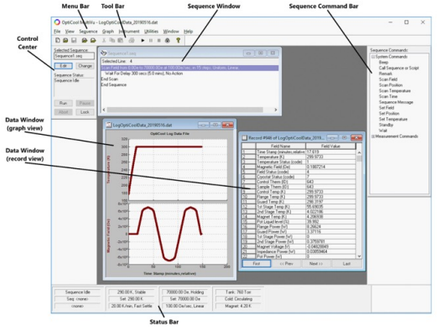
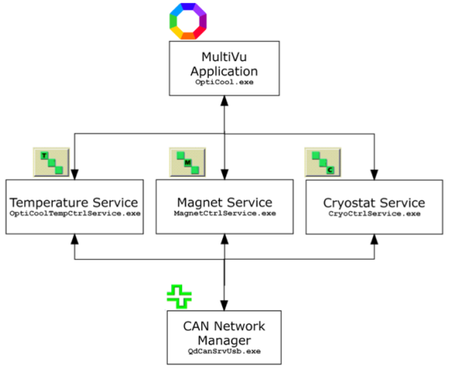




Figure 3.2: MultiVu Main Window.



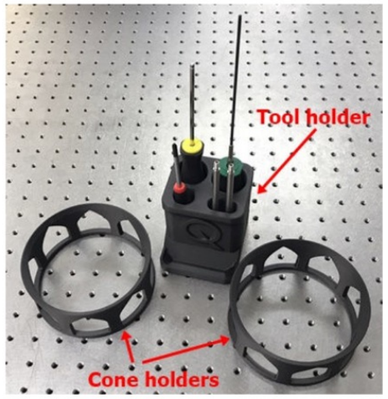
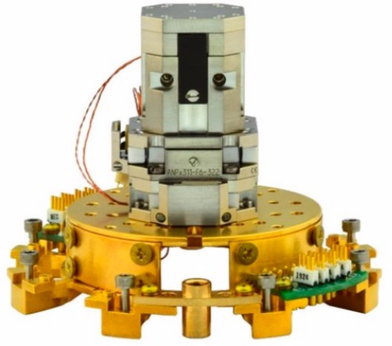




Figure 1.7: Cone holders and tool holder.



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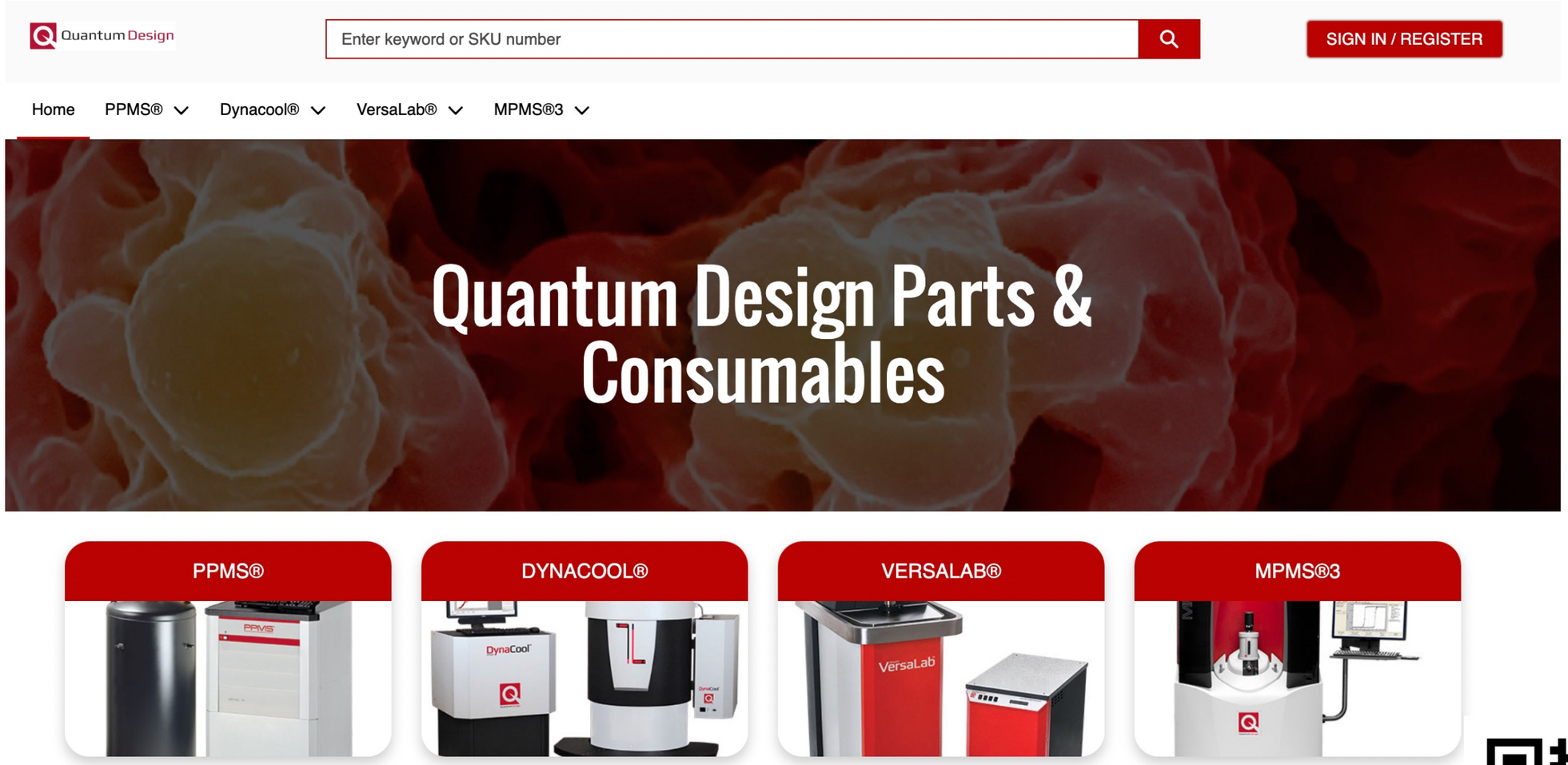


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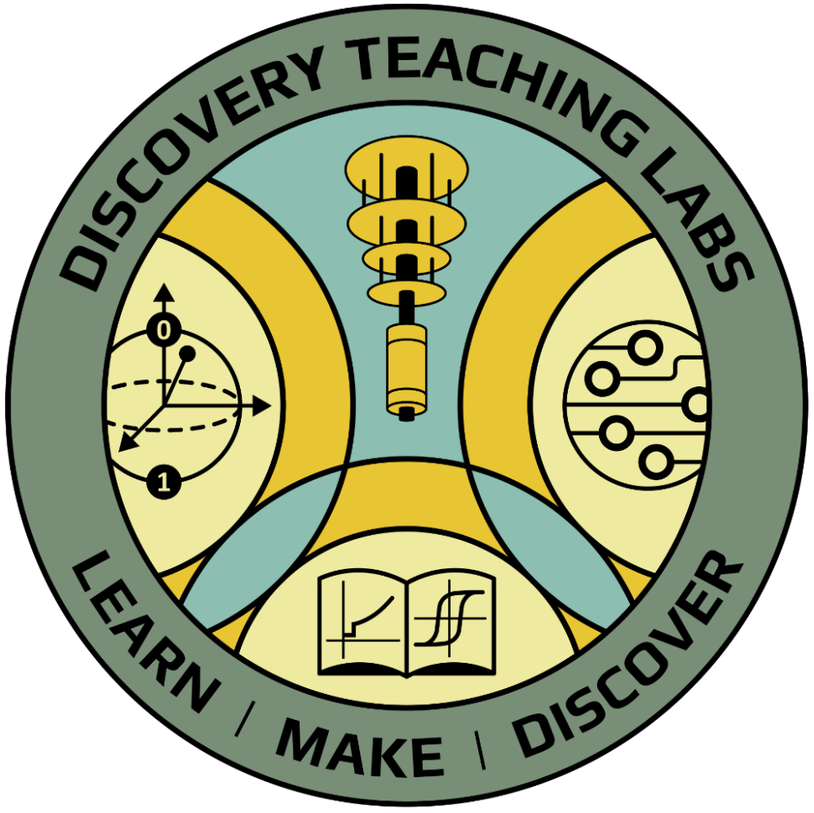

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