

Quantum Design PPMS VSM user training seminar

part 2: experiment design

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outline of seminar

- choosing which VSM coil set to use
- sample preparation
- sample mounting rules
- low-T sample holders (1.9 – 400 K)
- oven sample holder (300 – 1000 K)
- installing sample in cryostat
- measurement sequence examples



choosing the coilset

- standard (6 mm bore)
 - $7e-7$ emu noise floor (1 sec. avg.)
 - 4 mm+ samples can rub the coilset
 - heating at low T
 - background signal and higher noise in field
- large bore coilset (12 mm bore)
 - $1e-6$ emu noise floor
 - larger samples (<1cm), less risk of rubbing the coilset
 - larger uniform detection region
 - more “parasitic” background signal in field



VSM sample preparation

- beware of introducing magnetic impurities
 - no cutting/handling with ferrous tools
 - clean with solvents: dust is ~5% Fe !
- width should be narrower than holder
- length should be <4mm for best accuracy
 - see table in [VSM user manual](#)
 - [VSMcoilcalc.exe](#) program on Pharos
 - best thermal uniformity for oven samples
- in oven: need thin (<1mm thick) samples
 - high thermal gradient between stick and Cu shield



VSM sample mounting rules

- need low background signal
 - *uniform* and *nonmagnetic* sample holder
- durable attachment to survive large accelerations and extreme temperatures
 - default $A=2\text{mm}$, $f=40\text{ Hz}$
 - $\text{accel.} = A \cdot (2\pi f)^2 = 126\text{ m/sec}^2 > 12\text{x "g"}$
- see VSM app note [1096-306](#)



VSM sample holders (1.9 – 400 K)



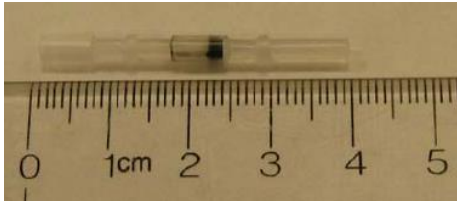
- 4mm wide quartz paddle (half-rod) 4096-392
 - best for vertical plate-like samples
 - use Duco, GE 7031 varnish, clean tape, etc...



- 3.5mm wide brass trough (half-tube) 4096-391
 - round samples (3 – 4 mm diam.)
 - horizontal flat samples on 3.5mm diam. quartz rods 4096-399
 - powder samples in capsules (4096-388, next page)
 - spring force holds samples, adhesives not needed
 - 5.5mm version used for large bore (12mm) coilset



VSM sample holders (1.9 – 400 K)



- powder sample capsules
 - each part has male + female side, 2 needed for usage
 - injection molded polypropylene
 - background depends on separation of the two halves
 - can look diamagnetic or paramagnetic (MvH linear)

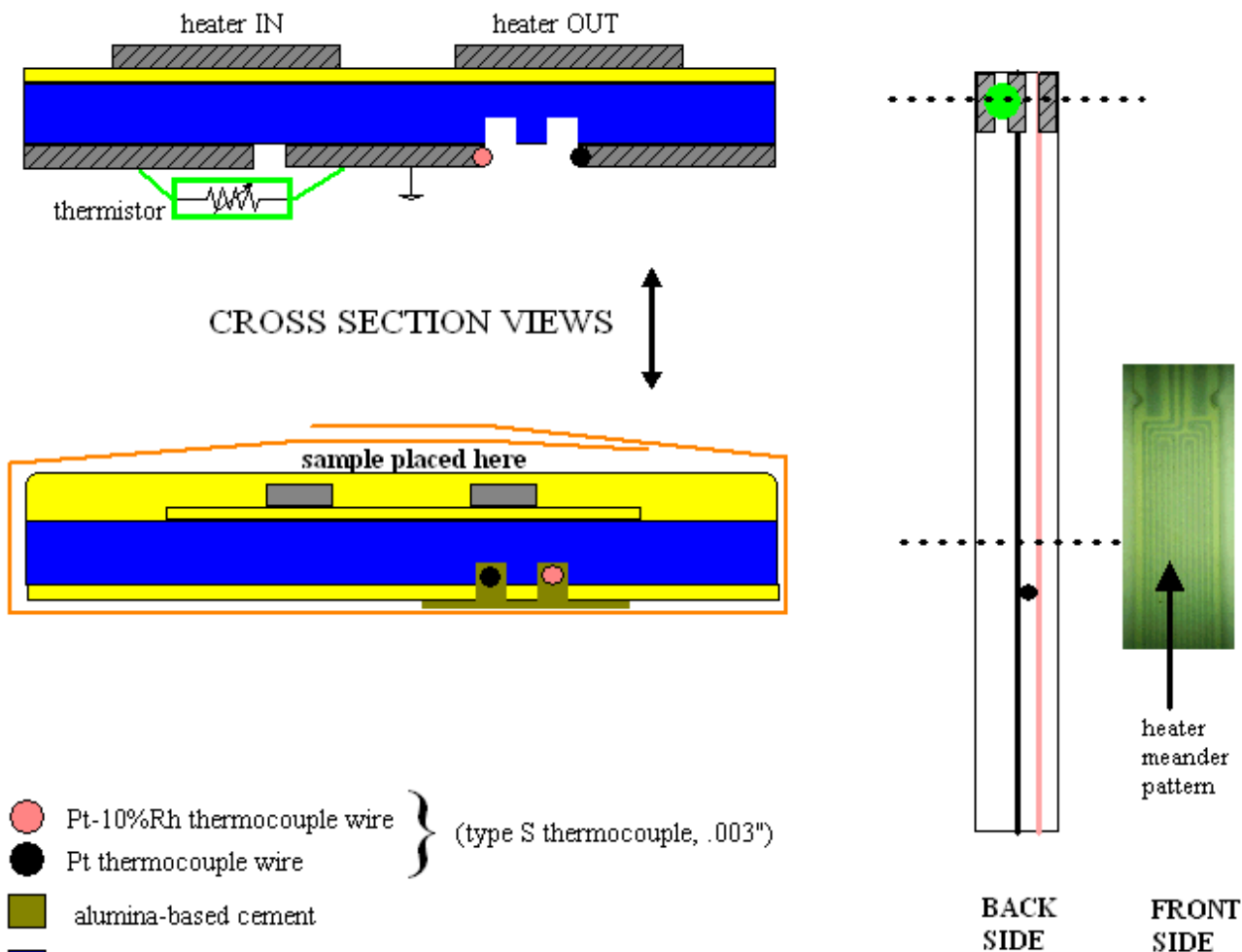


VSM sample holders (1.9 – 400 K)

- drinking straws (6mm): usable on large bore only!
 - See QD for straws (AGC2-BOX) and new straw adapter (4084-815 is box of 3)
 - Films perpendicular: 4.1x4.1mm
 - Films parallel: 6mm wide
- liquid samples
 - No official QD holder designed yet
 - Must have no air bubbles above liquid!



VSM oven sample holder (300 – 1000 K)



- Pt-10%Rh thermocouple wire
 - Pt thermocouple wire
 - alumina-based cement
 - YSZ substrate (0.020" thick)
 - heater element: platinum conductor
 - ▨ platinum contact pads
 - dielectric over- and underlayer
 - low emissivity copper foil shield (0.002")
- } (type S thermocouple, .003")



VSM oven sample holder (300 -1000 K)

- heater region: 3.5 mm wide x 25 mm long
 - mount sample on heater, NOT back side
(thermocouple wires in grooves can be damaged)
 - Temp. uniformity best over 4mm at center
- sample mounting (training videos on Pharos)
 - DRY (no cement):
 - Sandwich sample between heater and a thin ceramic plate
 - WET (Zircar cement):
 - Should cover the sample; Dry with heat gun



VSM oven sample holder (300 – 1000 K)

- wrap with provided 50 μm thick copper foil
 - needs to be clean, shiny (low emissivity)
- max. temperature can be raised to 1100 K
 - Fe $T_c = 1043$ K (standard sample available)
 - contact QD



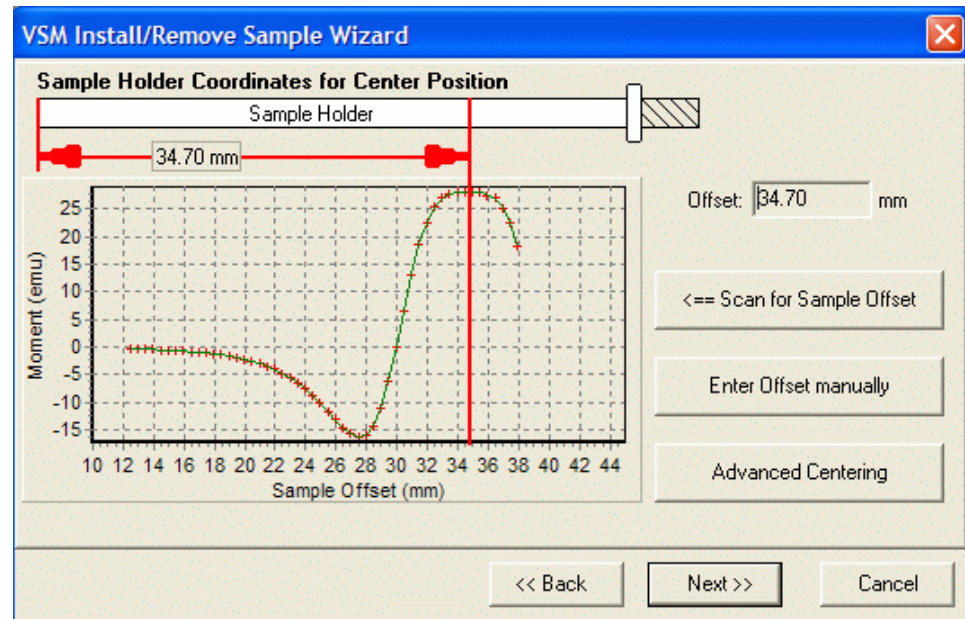
installing sample in cryostat

- attach holder firmly to sample rod
- inspect for straightness along whole length
 - small deviations can lead to rubbing in coil set
- inspect for breaks in glue joints or glass parts
- use extended purge if going to low T



VSM sample centering: *sample offset*

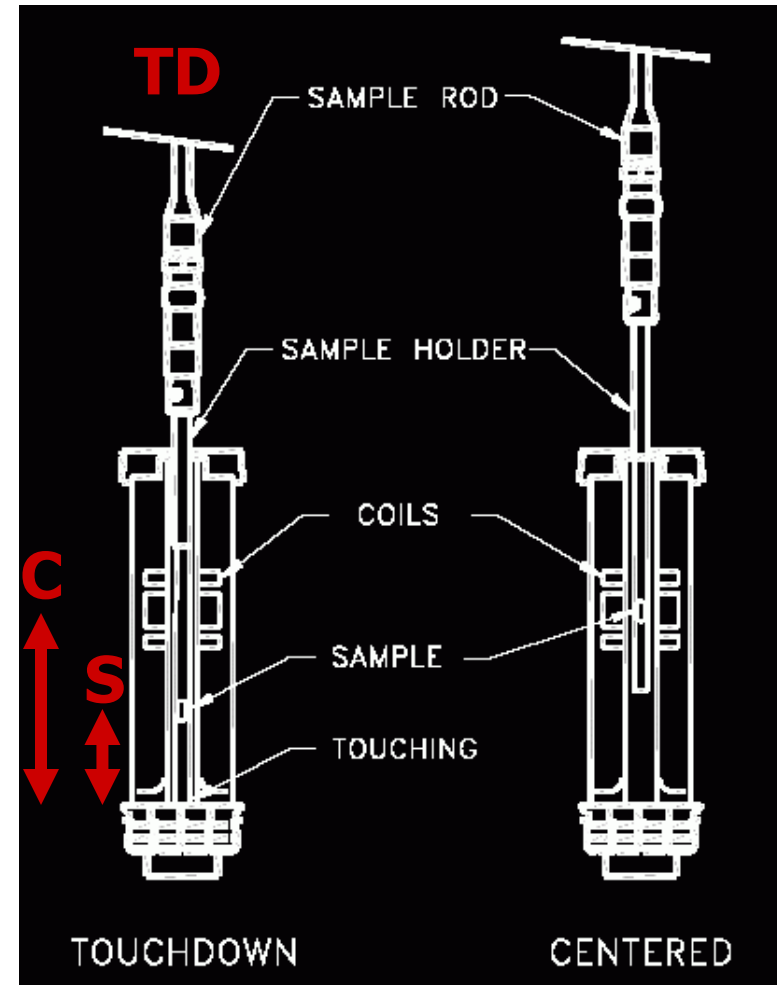
- user specifies the *sample offset* : distance from bottom end of holder to sample
 - either measured by user or scanned to find signal max
- easy to measure on bench



VSM sample offset

- coil height **C** above puck is known
- user supplies sample offset **S** from end of sample holder
- then motor operates at a location:

$$\begin{aligned} z &= \text{“Center Position”} \\ &= \mathbf{TD} + (\mathbf{C} - \mathbf{S}) \end{aligned}$$



sequence examples

- **M(H) 5-quadrant SWEEPING.seq**
 - “5-quad”: initial magnetization curve, then +H –H +H
 - continuous measuring while sweeping field, fastest measurement mode
 - no touchdowns: assumes stable at Temp. for > 1 hr
 - *sticky* autorange: change to *fixed* in cases of strong FM with steep M(H)
- **M(H) 5-quadrant STEPPING.seq**
 - uniform field spacing, magnet static and driven at each field
 - no touchdowns
 - *sticky* autorange: always OK for stepping
- **M(T) 300 to 20 to 300 K SWEEPING.seq**
 - down and up in temp., 1 K/min
 - touchdowns ON!
 - continuous measurement, 10 sec. average (why not? still 1681 points)
 - you will need to add a Set Field command (note magnet remanence)



sequence examples, part 2

- **M(T) 300 to 20 to 300 K STEPPING.seq**
 - stable Temp. at each point, get there fast (5 K/min)
 - Advanced setting: wait 60 sec. at each Temp. before measuring
 - Uniform T spacing of points (T^2 , \sqrt{T} , $1/T$, $\log T$ are possible)
- **remanent moment relaxation.seq**
 - measures $M(H)$ down from saturation (e.g., 3 tesla)
 - goes to “real zero field” (here, +18 Oe) which corrects for magnet remanence (different in each magnet! See app note 1070-207)
 - stays in driven mode to minimize field drift from flux creep
 - measures $M(\text{time})$ using “VSM Adv. Measure” command
 - stops, sets persistent magnet after 1 hr
 - motor amplitude drifts: can be corrected by knowing moment(amplitude) for your sample in situ
 - **moment vs amplitude scan around 2mm.seq**

