

ARC-MMA&ISS User Guide during Phase 2A under CV-19 CHEM1 Access Update

Updated August 31, 2020

Context for Opening in Midst of SARS-2 Pandemic

The Analytical Resources CORE, ARC, is enabling a return to normal research service activities, consistent with best safe practices during the pandemic. The return to a normal routine will take place in stages. As we learn more about the SARS-2 / Covid-19 viral pandemic then this will guide our response. Your feedback is welcome.

COVID-19 / CV19 / SARS-2 are used interchangeably, imprecisely, in this document to refer to the virus or acute symptoms caused by the virus

Staging the Phasing in of Research Services

Please watch for, observe and pay attention to notices, signage etc. that will provide up to date information about the CIF Staging and your personal responsibilities, our expectations during that stage.

Phase 2 : Ramp UP

As of July 20, 2020, we have begun rolling out our Phase 2 operations. These are providing more of our traditional, self-service operations in a few key laboratory areas that can be carefully monitored and controlled so that we reduce the potential for spread of CV-19.

Phase 3 : Return to Full Operations

(e.g., tours allowed)

It is difficult to imagine at this time, August 31, 2020, returning to full operations any time soon.

General Safety Protocols – Use of PPE

We will be limiting the number of people in the lab so that we can practice safe social distancing. Watch for postings regarding limited hours of operation. We will follow the emerging and fluid guidelines provided by our administration that may include sentinel testing, skin temperature measurements, live virus exposure, antibody presence, etc. Sanitizer solutions will be available at every station.

We require that you wear a face cover in our CIF laboratories during this time, and until further notice for two reasons, this reminds everyone you come in contact with that the virus potential has not been depleted and helps reduce the viral load spread from the breath of individuals who are unwitting, or asymptomatic carriers of covid-19. Everyone is expected to practice these measures so that we can reduce exposure and viral loads.

Masks also remind us that a primary infection route is by picking up virus on your hands, then touching your face. The mask helps train you to stop this infection behavior.

Resist the Impulse, Do Not Touch Your Face.

Use of Disinfectants and Sanitizer

Use sanitizer solutions provided to disinfect hands prior to entering the lab and before using equipment.

Wipe down work stations (keyboard, mouse, other surfaces) prior to leaving the lab.

Do Not Ever Wear Gloves While Using the Keyboard or Mouse

Where provided, please remove and dispose of cellophane covers over, e.g., keyboards after your use and replace with new sheets from roll provided.

We are making an effort to provide personal use, 2 oz bottles of disinfectant solution to everyone that comes into the CIF, originally this was courtesy of Dr. Karen Dobos Group and the OVP. Keep with you at all times. If available, please take one. Look for refill stations.

Traffic Patterns in Laboratory

Pay very close attention to traffic control patterns as they will evolve over the course of our return to full service.

We will strictly limit the number of people in the laboratory so that there should be no more than one person per 50 square feet (1 meter included radius) of space around an instrument.

We will be posting maximum room occupancy values and ask that you follow those guidelines (Table X-X)

CHEM1 Traffic Flow in ARC Basement Laboratory

Please follow posted instructions related to walkways and flow patterns through the lab.

- ❖ *One enters through main double doors at end of B-wing access corridor, on east side into C01.*
- ❖ *C05 Mass Spec laboratory, enter through C01 and exit through C05 doors to the west.*
- ❖ *C04 can be accessed from C01 and exit flows west to the stairwell corridor, west side of CHEM1.*
- ❖ *C03, including C03 Main Lab, PPMS and CO3E 500 NMR Lab enter through C01 and exit when finished into C02 from double doors on east side of C03.*

Generally, follow traffic patterns and flow instructions such as floor marking, etc that are provided and follow the chained / roped off routeways where you encounter them.

Staff Office Hours

ARC staff continue to be available virtually to students, research personnel and faculty by appointment, Teams, Zoom, etc..

In person visits may also be possible by pre-arrangement with staff, following guidelines and directives for these activities.

Word on Training

Our normal training activities are suspended through the First and Second Phase activities. As a result, All training requests made prior to the CSU Research shutdown have been closed. Those of you impacted by this may request training again and we will evaluate each request for feasibility, work arounds during this period.

Training is one of the most important aspects of our campus research laboratories and we will make this more available as soon as possible and likely as we move through our Phase 2 operations and gain best-practice experience in the face of the CV-19.

The CIF was developing online training videos before, and we are accelerating that activity now in the ARC. Some of these are likely to become available and they will reduce the in-person training time while still providing proficiency testing. Please stay tuned for more information and links.

Instrument Scheduling – Note that not all instruments online

- ❖ *Check iLab for Up To Moment Access.*
- ❖ *If available, access through iLab.*

Requests for instrument time will have to be done in iLab. This includes time on workstations or computers for data collection or processing.

Conditions for use and access that may be imposed, include:

- ❖ *One Request per PI – staff may limit iLab requests for instrument time to one request per PI for some high demand, or complex instruments.*
- ❖ *Remote Operations may be required where access is provided to load sample(s) and then the investigator returns to their remote location*
- ❖ *Linked iLab Calendars for instruments that are physically proximate (PPMS and MPMS, XPS and XRD, TGA/DSC and DLS) – this automatically disables calendar scheduling on an instrument that is near another that has been reserved.*
- ❖ *Limited access provided to small group of investigators. Staff may arrange for appropriate time on an instrument in consultation with a research group to allow a limited number of dedicated investigators access. This will limit traffic in the ARC spaces.*
- ❖ *Rotations and Shift work of operators on different days etc.*
- ❖ *Where possible, First-come-first-serve remains the default mode of prioritization for scheduling instrument time/services.*

Exceptions Include:

- ❖ *Critical Research / Priority Use evaluated by Department Heads / Research Associate Deans / ARC Head may Override default priorities (e.g. critical research may have to be prioritized).*
- ❖ *Triage - The ARC Head and Center Directors will limit scheduling time in the event instrument demand exceeds capacity.*

CHEM1 Basement Lab Safety Protocols

- ❖ *Social Distancing: We will be limiting the number of people in the lab so that we can practice safe social distancing.*
- ❖ *Do Not Congregate around instruments – conduct your business and leave.*
- ❖ *Sanitizer solutions will be available at every station. Use sanitizer solutions provided to disinfect hands prior to entering the lab and before using equipment.*
- ❖ *Masks are Required: We require that you wear a face cover in our CIF laboratories at all times*

Masks also remind us that a primary infection route is by picking up virus on your hands, then touching your face. The mask helps train you to stop this infection behavior.

Resist the Impulse, Do Not Touch Your Face.

- ❖ *Do Not Ever Wear Gloves While Using the Keyboard or Mouse*

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Generally, follow traffic patterns and flow instructions such as floor marking, etc that are provided and follow the chained / roped off routeways where you encounter them.

Instrument Scheduling – Note that not all instruments online

Both 400 MHz self-service NMRs, plus the 500 and 600 NMRs in CHEM1 are available at this time for self-service operations, by appointment under the above protocols

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CHEM1 NMR General CV-19 Access Rules, Phase 2A:

- Pandemic PPE Must be worn at all times. Do not congregate anywhere. Practice social distancing. Protective Eyewear is always required. Remove your gloves when you are not handling samples, before touching the keyboard, or non-lab building surfaces, doorknobs.
- We are limiting self-service access to a small group of investigators that are not well served yet by CRB109 NMR resources. The CHEM1 lab is NOT available yet to all NMR operators. Requests for access should be made through iLab and will be reviewed by staff. This is CV-19 social distancing policy guidance.
- Time spent doing NMR in CHEM1 is considered part of your lab shift time as per guidelines.
(Exception: if you have an approved, Special NMR Project in the CHEM1 lab – AND if you are explicitly told so by Staff then this is NOT considered part of your lab shift)
- Schedule your time using the iLab appropriate instrument calendar in advance of your need.
- You are required to use the **SLACK #mma-CHEM1-general** channel to virtually queue up, signal that you are in or done in the lab, retrieving samples, communicate generally with colleagues. **Staff do not follow this channel**, so **use the #mma-nmr-general** channel for communications with Staff
- Observe all Block Out Schedules on iLab and Slack (if any) including Staff set-aside times. Staff continue to provide full-service operations in CHEM1
- **1:1:Oh** ONE lab scientist may be on ONE console at a time. And, Oh Yea, The On-Deck circle is located in YOUR laboratory – do not congregate in the hallway outside the C03.
- As many as **four NMR investigators**, one at each NMR console may be present at one time. That is two in C03 at each 400, one in C03E at the 500, one in B02 on the 600. Check, observe and obey occupancy rules
 - **DO NOT CONGREGATE IN LOBBY!**
- Large sample volume needs that outstrip limited capacity, and severely limits others' use should continue to go to CHEM1 – just as you would do before the CV-19 pandemic.
- Slack Open Slots – NEO 12 O, or 400MR 10 O – when you leave.
- Follow Staff instructions and If asked to leave when Staff are present then please do so immediately.
- **W:W:W** Wash your hands before using the keyboard and mouse. Wipe down the area with provided sanitizer and paper towels gently when you leave (or use plastic wrap). Wash hands when you finish.

PLEASE FOLLOW THESE STEPS for ACCESSING the CHEM1 NMRs DURING YOUR WORKING SHIFT.

You Must also follow the CHEM1 NMR General CV-19 Access Rules, Phase 2A with highlights below.

DO NOT CONGREGATE

**1-1-Oh
W-W-W**

STEP 1

PPE including face mask and safety glasses Must be worn at all times. Do not congregate. Practice social distancing.

STEP 2

Schedule time in advance on the iLab Calendar. Also, If you haven't done this already then, request NMR service through iLab "Request Service". You only need to do this once per month.

STEP 3

Use "Slack" **#mma-CHEM1-general** to virtually queue your walkup access.

!You Must Do this to Retrieve Samples, Too!

Please state in Slack whether NEO400 (NEO), 400MR (V400) with number, #, of samples, e.g. if eight samples then, "NEO 8", or Retrieval (RET).

Join here: <https://join.slack.com/t/csu-arc/signup>. For more info: Slack User Guide. Join other relevant lab and instrument Slack channels to stay informed about general lab or instrument specific updates.

Wait for access in your laboratory, the on-deck, until your instrument opens.

STEP 4

The NMR site is empty and so you may go in and setup your sample tray, or do your advanced NMR experiment. Practice good hygiene, social distancing in the CHEM1 lab, and clean up after you are finished. *SLACK number of Open Slots, e.g., V400 10 0*

STEP 5

Use the **"Completed" check mark emoji** on your queue position in Slack to acknowledge to the CHEM1 community that you are finished, next batter is up. Also, use Completed check mark if you are delayed in the lab as a courtesy to others. Just queue back up when you are ready.

FREQUENTLY ASKED QUESTIONS REGARDING STEPS TO ACCESSING THE NMRs in CHEM1 DURING YOUR WORKING SHIFT.

1. *“Every time I get in to use the US400 (US4) there isn’t enough room left for my samples.”*

Bad Luck? Everyone – be sure to SLACK numbers of samples you submit in your Slack note: e.g., US4 6. Is there time available in CHEM1 NMRs?

2. *“Do I need to review the online notes about self-service access, submit my test as acknowledgement, since I already know how to do this stuff safely?”*

Yes.

3. *“Would it be possible to arrange or schedule even for, e.g., an hour or two to do some longer experiments?”*

Yup. If you are unsure then discuss your needs with staff.

4. *“Someone keeps tying up the NEO400 with all their samples so why doesn’t the ARC-MMA ban that activity!”*

Ummm. Yea, might be bad form/etiquette? But we don’t “ban” otherwise legitimate use of resources, and we aren’t the etiquette police?! Suggestions: form an investigator user group and discuss this, apply gentle peer pressure, use other resources in CHEM1 or CRB109.

We could turn on more restrictive iLab calendar features to control access (!No, Please no!)

5. *“How do I get access to CHEM1 NMRs?”*

Access to the CHEM1 400 NMRs is currently prioritized to those that have been most acutely affected by loss of NMR services, or need additional NMR resources. Send us an eMail and we will review your request

6. *“I am in CHEM1 on the NEO400. May I use the 400MR instead if no one else is using it?”*

Sure! Schedule this request on iLab Calendar to be sure you don’t interfere with someone else, Slack your intention, please, e.g., NEO Complete check, V400 #, etc. and go ahead and use the other NMR

CRB109 NMR Access Quiz One:

1. The phrase 1:1:1 refers to one person in the CRB109 lab, one person on deck, and one person in the lounge chairs. True False
2. You may run as many samples in CRB as you want because this is the most efficient and fast way to get your NMR data back. True False
3. The Slack Channel, #mma-crb109-general is used by investigators to communicate efficiently with colleagues their CRB NMR status and is not monitored by Staff. True False
4. CSU guidelines require that you wear the CV-19 appropriate PPE in CRB-109 even when you are alone. True False
5. The maximum occupancy of the CRB109 lab is two, plus one person retrieving their samples and one person on deck. True False
6. I can drop my samples off at the table in front of CRB109 and Staff will pick them up and deliver them back to me. True False
7. The Complete check mark should always be used on the Slack channel when I complete my NMR or sample retrieval and leave the lab. True False
8. The three functional positions for CRB109 are the WUP (ASC400 Walkup), US4 # (US400 SampleCase) and RET (Sample retrieval).

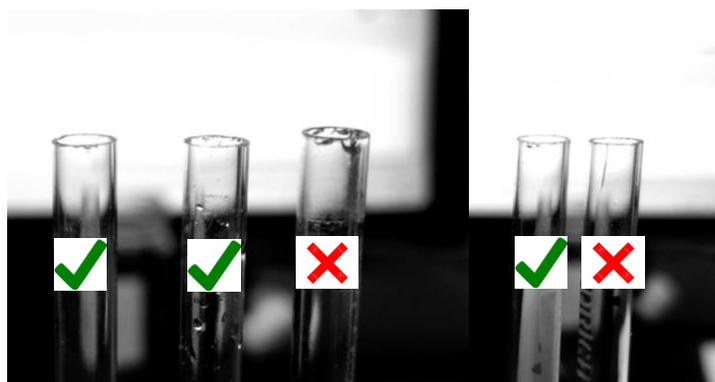
CRB109 NMR Access Quiz Two:

1. CSU guidelines require that you wear the CV-19 appropriate PPE in CRB-109 even when you are alone. True False
2. The maximum occupancy of the CRB109 lab is two, plus one person retrieving their samples and one person on deck outside. True False
3. I can drop my samples off at the table in front of CRB109 and Staff will pick them up and deliver them back to me. True False
4. The Complete check mark should always be used on the Slack channel when I complete my NMR (US4 or WUP) or sample retrieval (RET) and leave the lab. True False
5. The three functional positions for CRB109 are the WUP (ASC400 Walkup), US4 (US400 SampleCase) and RET (Sample retrieval).
6. The Slack code, "US4 6" means that it is my intention to put six samples on the US4 sample changer.
7. It is a great thing to put my large number of samples on the US400.
8. One should Slack out the number of open slots on the US400, "US4 6 O", when finished.

CHEM1 NMR Access Quiz One:

1. The phrase **1:1:Oh** refers to one person per NMR and one person waiting on deck in the hallway.
True False
2. The Slack Channel, #mma-CHEM1-general is used by investigators to communicate efficiently with colleagues about their CHEM1 NMR status and is not monitored by Staff. True False
3. CSU and ARC guidelines require that you wear the CV-19 appropriate PPE in CHEM1 C03 even when you are alone. True False
4. The CHEM1 NMRs are available by checking Slack to see if they are busy, noting your intent there and then walking into the laboratory. True False
5. The maximum occupancy of the CHEM1 C03 lab is four, plus one person retrieving their samples and one person on deck. True False
6. I can drop my samples off at the table in front of CHEM1 by arrangement. True False
7. The Complete check mark should always be used on the Slack channel when I complete my NMR or sample retrieval and leave the lab. True False
8. Functional positions for CHEM1 include the NEO # (NEO400 Prodigy SampleCase), V400 # and RET (Sample retrieval).

- Do not use chipped, cracked, scratched or warped NMR tubes
 - There is an NMR tube gauge outside of the ARC Main Lab, Chemistry Basement (CHEM-1) that can be used to check for warped tubes
 - Visual inspection indicates when tubes are damaged (e.g. visible scratches, cracks, and when a tube is noticeably short, the cap will be removed to check for chips
 - Some NMR tubes have unpolished ends, these are fine for submission, if you aren't sure, just ask. In the image below the "x" on the left has obvious chips, so has become dangerous and the "x" on the right has a crack which is especially dangerous to the NMRs

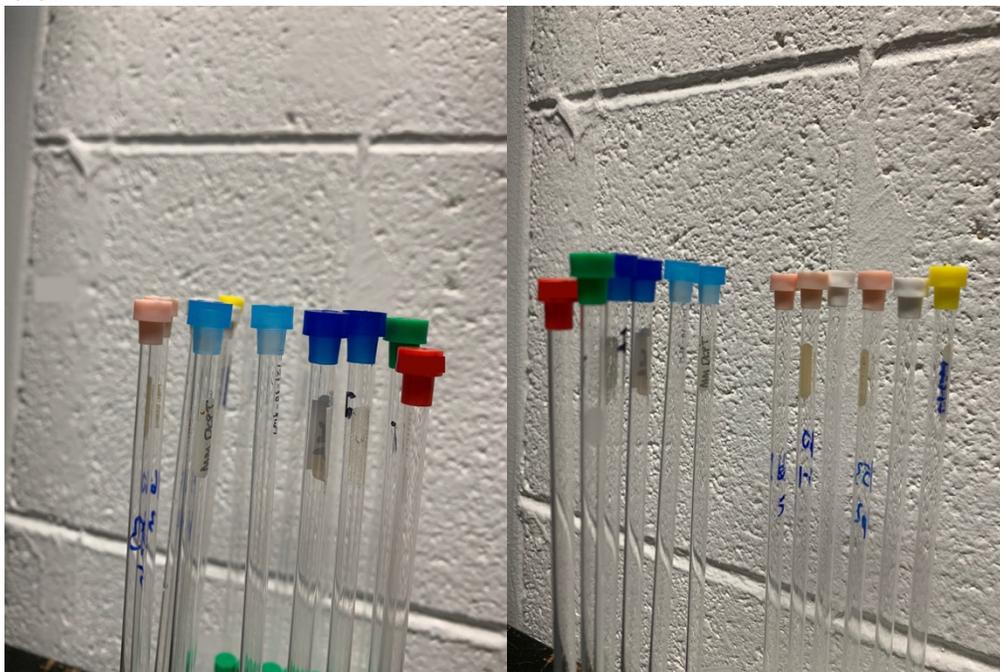


- Samples that are discovered in a damaged tube will be stored by facility staff and the user will be contacted to recover their sample, the damaged tube will be disposed of by facility staff
- Flags or loose tape are not allowed as NMR tube labels
- The maximum length tube to use in the Bruker systems with automation (CHEM-1: Bruker NEO 400, CRB-109: Bruker US400) is 22 cm and the Varian system with automation (CHEM-1: 400MR) is 24 cm; samples that exceed max length will be returned to the user to be transferred to a tube with acceptable length
- Do not close software, ICON or TopSpin on Bruker systems, when your sample acquisition is complete
- Pickup samples after data have been acquired
 - Samples that are not picked up will be moved to a '1-week' beaker in the fume hood located in CHEM1; samples that have not been picked up after 1 week will be moved to the 'free' beaker so that other users can access unwanted tubes

NMR Tube Selection / Care and Feeding

One of the tubes in the pictures below should be inspected and rejected – discarded in the sharps waste and replaced by a new tube. Which one, what color is the cap? Why are you basing this on?

One of the tubes is apparently a bit different than the rest but intact? Which one? What do you think is going on here?



1 - visually inspect intact samples (caps on) by “stacking” them quickly, comparing with other tubes that are similar in size (7, 8, 9 inch). Further review tubes that are obvious “mis-fits” or your spidey sense tingles. You can also spot these after they go on the sample changer and one tube is obviously much shorter than others considering its group, manufacturer or style. 2 - some samples are “air-sensitive” meaning the sample will begin to degrade if the cap is removed so avoid doing that (unless criteria 1.). 3 - take a picture of the tube. 4 - open a private Slack channel with the submitter and attach the picture.

8:20

Fit and Polish - some tube manufacturers do not “polish” their tube tops where the cap fits. This is not a defect. You wouldn’t know that anyway, because you didn’t take the cap off, because the tube was a normal, regular height.

Cleaning and Drying It is widely thought that tubes baking in a drying oven can cause bending or other warping. See this article <https://s3.wp.wsu.edu/uploads/sites/33/2013/10/NMR-Tube-Cleaning.pdf> regarding how to clean tubes, suggesting that if they absolutely need to be baked to be cleaned, it should be only for 30 min, < 125C. Some say they should be laid flat, I believe they should stand vertically. Do not put caps in the oven as they will melt!

Historical Treatise on NMR Tubes, Glass, etc.

by Mike Olsen, Scientific Glass Blowing, Colorado State University

So, you're noticing there are patterns to glass failure? Don't feel bad - it took me decades to figure any of them out. It certainly did help having a couple of half-day seminars on glass fracture back in the 80s with Dr Josef Francel, the Mgr of New Product Development at Owens-Illinois (Kimble glass) who became a dear friend. I hate to say it, but you have to let the glass 'talk' to you, and an individual piece will tell you its story. There can be many issues involved in glass fracture, and you REALLY have to use your imagination to understand what REALLY happened to *that particular piece*, or what are now pieces...

So, an NMR tube has a history including its manufacture, storage and handling, usage, and cleaning. Then there are the applied forces from capping and spinning (and dropping), as well as any heating such as my attaching a valve to a tube. Then there's anything intrinsic to any potential meso-scale structure that glass might have at its surface or within its bulk, and how such structure might influence crack propagation. This last point is speculative, and where Roy and I left-off on a little bit of independent work we did about a year ago.

Now, to address your many questions, back in the early 80's I met Victor Plumbo, who with his brother invented the 'vacuum redraw process' used to fabricate precision ID tubing of many shapes and dimensions, including NMR tubes and multi-bore tubing. Using the Plumbo brother's technique of sucking a hot, slightly over-sized tube down onto a (I preferred W) mandrel coated with a release agent (typically colloidal graphite) I was making precision tapered ~1mm ID square-bored capillary segments for particle size spectrometry when working for <https://www.pmeasuring.com/> in Boulder. Knowing I had done this, someone at Wilmad Glass later contacted me inquiring if I would be interested in fabricating NMR tubes for them at home in my spare time for \$0.90/ea (they were about \$10/ea retail at that time), and I declined.

NMR tubes are fabricated from a variety of glasses including fused quartz, but most typically of 'borosilicate' glass which is often described as 'Pyrex'. Borosilicate glass was invented in the late 19th century by Otto Schott in Jena Germany, and crafted into lenses by his good friend Carl Zeiss. That glass is now called 'Duran'. In the early 20th century the Corning Glass engineers (some would say reverse-) engineered several useful borosilicate glasses, so Corning's 'Pyrex' brand name includes several slightly different glasses (for example, the home cookware formula is different, and is now no longer even a borosilicate!), each identified by a 4-digit identifier. Standard lab Pyrex is 7740 (all my Pyrex is 7740), and the more expensive Pyrex NMR tubes are 7740. There are however cheap NMR tubes that are made from a chemically different 'Pyrex', and I'm not sure what it is, but some groups keeps buying them and asking me to attach 7740 J Young valves to them, and they won't stay attached due to a significant difference in the coefficient of expansion, resulting in bright stress 'fringes' when viewed in a polariscope. So, we've just touched on a first cause for NMR tube fracture: mis-matching of glass types when attaching a 7740 valve. I have attached a photo of two such tubes viewed in a polariscope, the right one showing colorful fringes at the base of the valve from a glass mis-match. But that's not really what you were asking about.

First, a word about annealing: my understanding is that NMR glass is so thin and low-mass that the redrawing process itself (where the closed-end of the evacuated tube is uppermost, and heat is applied with a multi-ported ring-burner, beginning at that closed end and proceeding downward) is intrinsically self-annealing, and after removal from the mandrel, the excess glass (which was attached to the vacuum source) is diamond saw-cut to length. Further, unless supported with a mandrel, any subsequent annealing (to 565 C for 15 min for 7740) will cause an NMR tube to sag or even noodle in the oven.

Dr Francel had a mantra that glass needs two things for a fracture to initiate and propagate: tension, and a surface defect. If you have one but not the other, it will not fracture. For example, putting a surface scratch under compression is OK. The saw-cut open end of an NMR tube is a gigantic surface defect which will fracture if put under enough tension, like if the cap isn't put on right, or if cleaned with a pipe cleaner, or a q-tip is jammed in, etc. Then, there are NMR tubes that have been rattling around in a drawer, or tubes tinkling against each other and rolling around in a beaker. This is the history part I

alluded to, as you get longitudinal scratching of the outside of the tube by scuffing in a drawer (the point of contact of a cylinder on a flat surface is a line the full length of the cylinder), and NMR tubes in a beaker will accumulate circumferential external scratches from contact with the beaker's rim. An NMR tube that fractures longitudinally was probably STORED differently than one that snaps in half.

Unless an NMR tube has been flame-worked, like attaching a valve (which I will briefly and crudely flame-anneal but will NOT oven anneal), there's really nothing that will show up in a polariscope. Close visual inspection of a tube at ~10X will indicate a tube that should be retired due to scratching, as scratches can't be repaired or polished. NMR tubes can easily be trimmed to eliminate jagged open ends, and I can also seal shut a broken bottom.

My final point regarding any potential meso-scale structure in the glass enabling or directing fracture is purely speculative, and based upon TEM images of quartz glass (containing 0.7 PPM of Na) showing a) a dramatically smooth surface on flame-polished and conchoidally-fractured surfaces, and b) a very rough surface on ultrasonically 'crushed' samples, with the appearance of ~5 nm dia clusters arranged in twisted ropy columns. This last is something we have not seen previously discussed in the literature, and Prof Szamel also found intriguing. I can't imagine that anything post-manufacturing can affect internal glass structure, however what we observed over rather extensive regions all displayed a similar ropy axis of orientation that might presumably have different tensile strengths in different directions (presumably due to forces while still hot and plastic during manufacturing). Again, this anisotropy is tentative and speculative. I have attached a composite photo illustrating this: The red-boxed region in the lower-right image, is a very thin, conchoidally fractured surface (produced in a vortex mixer with isopropanol) which is enlarged as the upper-right image A. An FFT of A is the upper-left image, which exhibits a vague hexagonal pattern of bright spots. Two pairs of such spots were chosen (green boxes) and their inverse FFT produced the lower-left image labeled B (a sort-of dramatized topographic map). Combining images, A and B enhances what I describe as a uniform ropy orientation. A third photo vaguely shows the ropy chains of 5 nm 'cotton balls' in a separate, ultrasonically produced fragment.

I->n a similar vein, about 10 years ago I was wondering why some organic students would bring me lots of round-bottom flasks with star cracks, and other students from the same lab would not. The answer came to me with a thought experiment: Take two identical, empty 1-liter round bottom flasks. Hold them by their necks about an inch apart in front of you, and let them gently swing and tap each other - it's no big deal, but if you do it hard enough they might scuff. Now, fill each with a kilogram of water and repeat the experiment, and you'll probably wash the floor! Now, take those same flasks, plus several others of assorted sizes, submerge them in a tub of Nochromix, jiggle them around every day for a week or so, then drain, rinse and wash them in a tub with Alconox, similarly jiggling them around against each other in the suds. Just because they're submerged in liquid, doesn't mean they don't have exactly the same mass and velocity as when you held them in front of you in the open air. Thus, I propose that star-cracks originate from a sloppy dish washing technique. And, of those who bring me many flasks to repair, I have noticed that there are not just one or two, but sometimes seven or eight barely noticeable star-cracks in some individual flasks - that was the give-away that it was an individual's technique of something that was causing them.

Keep the fire in your eyes!

- Michael