SAFETY - General

1) Are you allowed to bring food or beverages into the NMR facility?

   Circle one: **YES / NO**

2) Which of the following are OK to wear in the magnet rooms of the facility (circle them)?

   - Lab coat
   - Safety glasses
   - Gloves you wore at your lab bench
   - Fresh gloves from the NMR facility

3) What MUST you use for transporting NMR samples from your lab to the NMR room?

4) Is it OK to lay NMR samples flat on a desk or lean them against a computer monitor or keyboard?

   Circle one: **YES / NO**

5) BROKEN SAMPLE (on the floor): If you break a sample so that the broken tube is on the floor and the sample has spilled into the carpet, what do you do?

6) BROKEN SAMPLE (in an autosampler carousel): If you break the top off of a sample so the bottom part is still held in a spinner in the autosampler carousel, what do you do? What do you do with the sample and spinner?

7) In one short sentence, please describe what happens if broken glass gets into the magnet bore.

8) TRUE OR FALSE? You are required to report EVERY sample you break or see broken, even if you are not the person who broke it.

   Circle one: **TRUE / FALSE**

9) What is Josh’s cell phone number, which you should use to report broken samples and other safety problems, and anything that prevents data acquisition?
SAFETY – Specific to NMR

10) What is the significance of the “5 gauss radius” around an NMR magnet?  
(CIRCLE all that are TRUE)

a. It is a safety limit marked on the floor around each NMR magnet
b. It only applies to the radial distance from the magnet center, so it is completely safe for someone with a pacemaker to be underneath a magnet, or someone with a large steel wrench to use it on top of the magnet.
c. Inside the 5-gauss radius, the magnetic field is strong enough to attract tools and other magnetizable metal objects, risking a magnet quench that can destroy a magnet and cause injury or death.
d. Anyone with a magnetizable medical implant (like a pacemaker or cochlear implant) must stay outside the 5-gauss radius, or else risk injury or death.

11) Which metals are DANGEROUS to bring near an NMR magnet (circle them)?

aluminum    brass    surgical stainless steel    gold    silver
iron        copper    steel    titanium    nickel

12) TRUE OR FALSE? It is very important to keep the lab chairs OUTSIDE the 5-gauss lines.
Circle one:   TRUE / FALSE

13) Every NMR magnet in the facility contains hundreds of liters of two dangerous liquids. 
What are the liquids? _________________________ and _________________________
What TWO dangers do they pose (hint: one is related to the quantity of cryogens in the NMR lab)?
1) _________________________ 2) _________________________

NMR SAMPLES

14) What is the minimum volume of sample required to achieve sharp peaks from a sample in a standard 5mm-diameter tube?
Circle one:  0.2 mL  0.6 mL  1.0 mL

15) TRUE OR FALSE? If the liquid in your sample is “three fingers” tall, it should be enough for ordinary data quality.
Circle one:  TRUE / FALSE

16) What will your spectrum peaks look like if there are particles floating in your sample? Draw a picture if you want to.
17) What three precautions must you take if you dry NMR tubes in an oven?
   1. 
   2. 
   3.

18) Sometimes NMR tubes must be discarded. Name TWO problems with an NMR tube that would merit discarding it.

_____________________________ and _____________________________

19) How much does a new NMR tube cap cost? Can you buy them in large inexpensive bags in distinctive colors and shapes? Should scratched, torn, and bent caps always be discarded?

20) Which of the following are ACCEPTABLE ways of labeling an NMR tube? Circle all that apply.
   a. Sharpie marker on top part of tube
   b. Sharpie marker in the middle of the tube, where the spinner holds it
   c. On the cap
   d. I don’t need a label
   e. A flag of tape that extends off the side of the tube
   f. A short piece of label tape securely wrapped around the tube

21) What is the maximum total length allowed for an NMR tube when using one of our autosamplers?
   Circle one: 7 inches / 8 inches / 8¾ inches (222 mm) / 9 inches

22) TRUE OR FALSE? A new “600 MHz” grade tube made of precision glass will almost always give you better data than a cheap “high throughput” or “100 MHz” grade tube made of economy glass.
   Circle one: TRUE / FALSE

23) After removing other people’s samples from the carousel, where do you put them?
   Circle one: a. Top rack
   b. Bottom rack
   c. Take-me beaker

24) Are you ever allowed to remove someone else’s sample from one of the racks?
   Circle one: YES / NO
EXPERIMENT SELECTION & SETUP

25) PARAMETER NAMES: Write the corresponding two- or three-letter parameter names in the appropriate boxes in the diagram below (possible values = D1, AQ, O1P, SW, NS):

![Diagram of pulse sequence and spectrum with labeled parameters](image)

What is the meaning of the parameter “NS”, in three words?

26) TRUE OR FALSE? To get accurate integral values, you must ensure that the sum of D1+AQ must be greater than or equal to 4X the longest T1 relaxation time of the signals of interest.

_Circle one:_ TRUE / FALSE

27) Is the $^1$H T1 measurement experiment available on the fully automatic instruments?

_Circle one:_ YES / NO

28) For a typical $^1$H 1D spectrum of a typical organic compound sample, is ONE scans (NS=1) enough to get an acceptable signal strength?

_Circle one:_ YES / NO

29) In the ICON NMR interface, what do buttons A, B, and C do? What are they for?

_A:_

_B:_

_C:_
30) What information do you get from a 2D HSQC spectrum? HSQC crosspeaks represent what kind of correlation?

31) Which 2D experiment gives crosspeaks between $^{13}\text{C}$ atoms and $^1\text{H}$ atoms **THREE BONDS** away?

*Circle one:* TOCSY / HMBC / COSY

32) What information do you get from a 2D COSY spectrum? COSY crosspeaks represent what kind of correlation?

**DATA EVALUATION**

33) We often assess spectrum quality using the term “linewidth”.
   
   a. In the box, sketch a singlet peak and indicate where you would measure its linewidth.
   
   b. In a spectrum of yours, which peak(s) do you choose for measuring linewidth when evaluating overall spectrum quality?
   
   c. In a good spectrum, what is the maximum linewidth, in Hz, of those selected peak(s)?
   
   d. **BONUS:** Name two reasons why you might have broad sample peaks even if your solvent peak is sharp.

34) Situation: You took a 1-scan spectrum (NS=1) with a poor signal-to-noise ratio (S/N), and now you want to increase your S/N by a factor of 8. How many scans do you need?

*Circle one:* 2 4 8 16 32 64 128 256
DATA PROCESSING

35) Briefly explain the difference between “time domain” data and “frequency domain” data. Which is raw data and which is processed? What transformation is used to make one into the other? Use diagrams if you find it helpful.

36) Which processing parameter is adjusted to make the spectrum on the top look like either the middle or the bottom spectrum? Identify either the two-letter parameter name or one of the names of the technique. (Hint: what is happening to the sharp peaks in the top spectrum to make them look like the broader peaks at the bottom?)

37) PHASING: Which statement best describes the spectrum at the right?
Circle one:

a. It looks OK
b. It needs zero-order phase correction
c. It needs first-order phase correction
38) DIGITAL RESOLUTION: Look carefully at the number of points used to draw each peak.

What is the name of the processing technique used to make these spectra appear different? (Hint: it refers to altering the time-domain data)

What the 2-letter name of the processing parameter that controls this appearance?

(Hint: It refers to the “Size of the real spectrum”)
IDENTIFICATION OF SPECTROMETER COMPONENTS

39) SPECTROMETER COMPONENTS: In the image below, identify the following

a. Magnet
b. Console
c. Probe
d. Autosampler
e. Computer
ORIENTATION - SEARLE

40) SAFETY & MISC: In the image below, identify the following
   a. First aid kit
   b. Dustpan & broom
   c. Emergency contacts
   d. Fire extinguisher
   e. Support lab with fume hood
   f. Craft table

41) SPECTROMETERS: In the image below, identify the following
   a. 400-1
   b. 500-1
   c. 500-2
   d. 600-1
ORIENTATION - GCIS

42) GENERAL & Safety: In the image below, identify the following:
   a. Safety cart with gloves and cleaners
   b. Safety contact information
   c. First aid kit
   d. Dustpan and broom
   e. Spectrometer 400-2
   f. Instrument usage rules

43) In the image below, please identify
   a. What is the name of this spectrometer? ________________
   b. The console
   c. The spectrometer computer