The Bio21 NMR Cave

Safety Induction & Protocols for the Use of NMR Spectrometers

- 2017

bio21 institute

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How do I get to use the NMRs ?

- ***Protocol for 2016:***

Step #1: **Safety Induction & Cave Protocols *(This presentation)***

Step #2: **Complete Safety Questionnaire *at the conclusion of this presentation* (& pass !)**
(this will enable your swipe card to be activated for Cave access)

Step #3: **Introductory Training to use Chemistry 400MHz "walkup" auto-sampler system.**
(Time on this instrument cannot be booked)

Step #4: **As determined by your project – small group training on the Chem600 & Chem500**
(esp. if nuclei other than ^{1}H & ^{13}C are required)
(Time on these instruments CAN be booked)

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400MHz “Walkup” System – Robot400

- Max 30min slots during the day, 1 or more samples per 30 min. session – longer experiments submitted to overnight queue (6pm till 8am).

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400MHz "Walkup" System – Robot400

Not available for sample submission from ~10am till 12pm every Thursday

- liquid nitrogen fill time

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500MHz Multinuclear System – Chem500

⇨ <u>2 hour max</u> slots during the day, overnight runs (5pm to 9am)

-for dilute samples & long 2D experiments.

-used when other nuclei such as ^{31}P, ^{19}F & ^{77}Se are required.

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Routine 600MHz $^{1}H/^{13}C$ System – Chem600

- Min slot: 15min, max 2hr during the day, overnight runs (5pm to 9am) for dilute samples & long 2D experiments

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Biochemistry/Biomolecular Spectrometers

800MHz

600MHz

700MHz

Instrument operator training for Biochemistry students who will be using these instruments will usually be provided by Dr Shenggen Yao.

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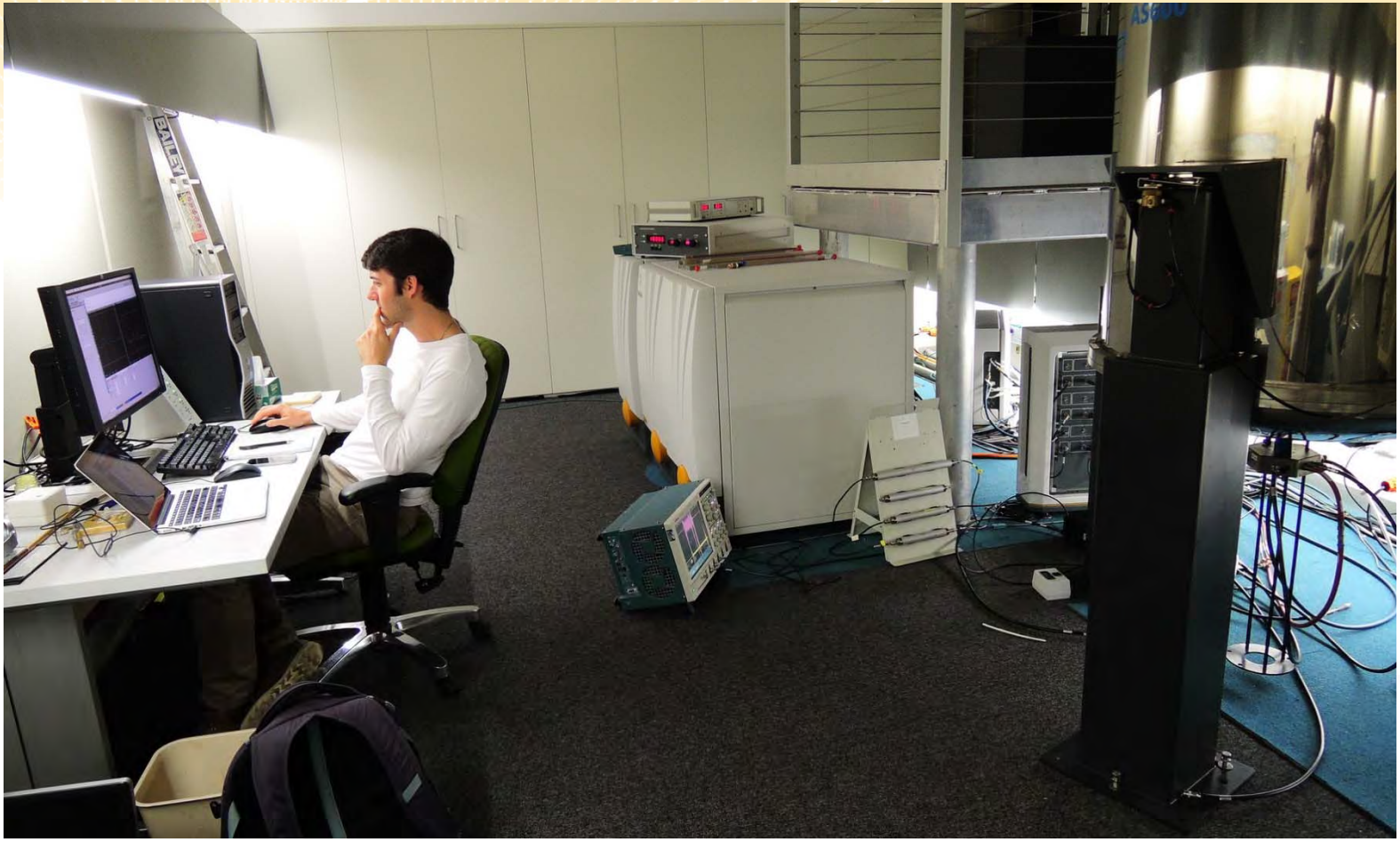
Solid-State Research Group Spectrometers

(Prof. Frances Separovic)

Varian VNMRS-600 (600MHz) system with 8 probes including high-spinning MAS probes optimised for biomolecules.



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Solid-State Research Group Spectrometers

(Prof. Frances Separovic)

Bruker AVIII-HD 400MHz Wide-Bore System with Dynamic Nuclear Polarisation (DNP) “accessory”.

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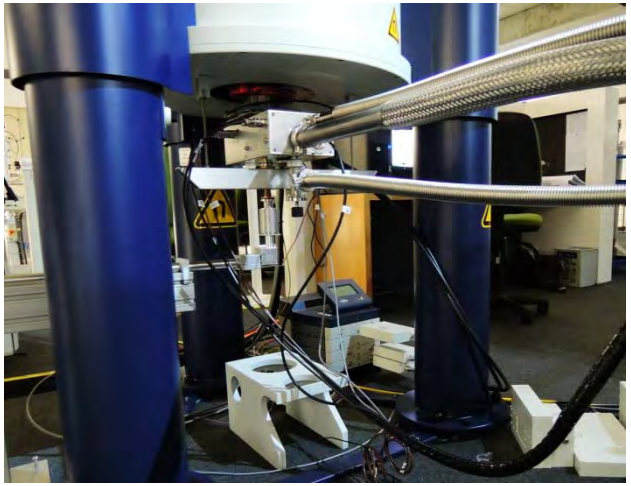
Solid-State Research Group Spectrometers

DNP is a very complex hardware combination including a wide-bore 400MHz spectrometer which can have the main field of the magnet varied during the experiment. A gyrotron powered by a cryogen-free supercon magnet generates an intense microwave field at 263GHz.



The polarization process yields a signal enhancement factor up to ***200*** for some solid-state samples.

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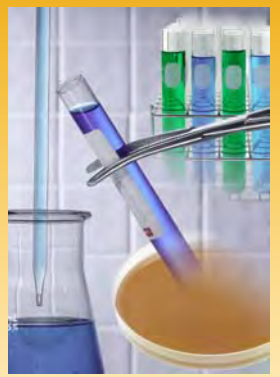
Safety

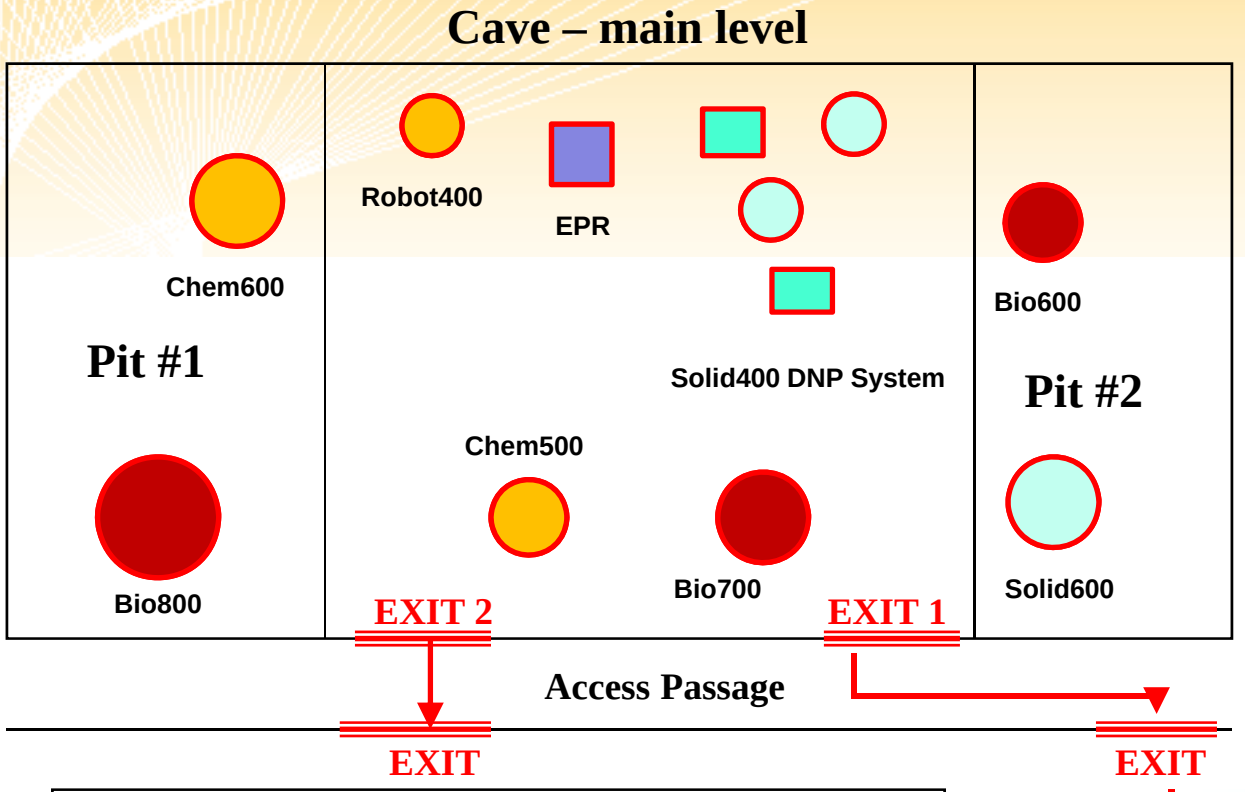


- **Safety is the #1 consideration in all activities undertaken at the University.**

- **It is the responsibility of all staff and students**

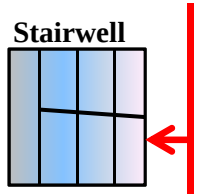
Emergency Evacuation

- **Procedure in the event that the Building Evacuation Alarm is activated**



Underground Carpark

- **Proceed up the fire escape stairs & proceed to the Atrium. Exit by the main entrance & await further instructions from wardens**



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Safety Procedures

- **Evacuation procedure in the event that the Building Evacuation Alarm is activated**

"Normal" Cave entry/exit – also used as emergency exit to basement.

<u>Emergency exit only</u> – opposite center access doors to Cave.

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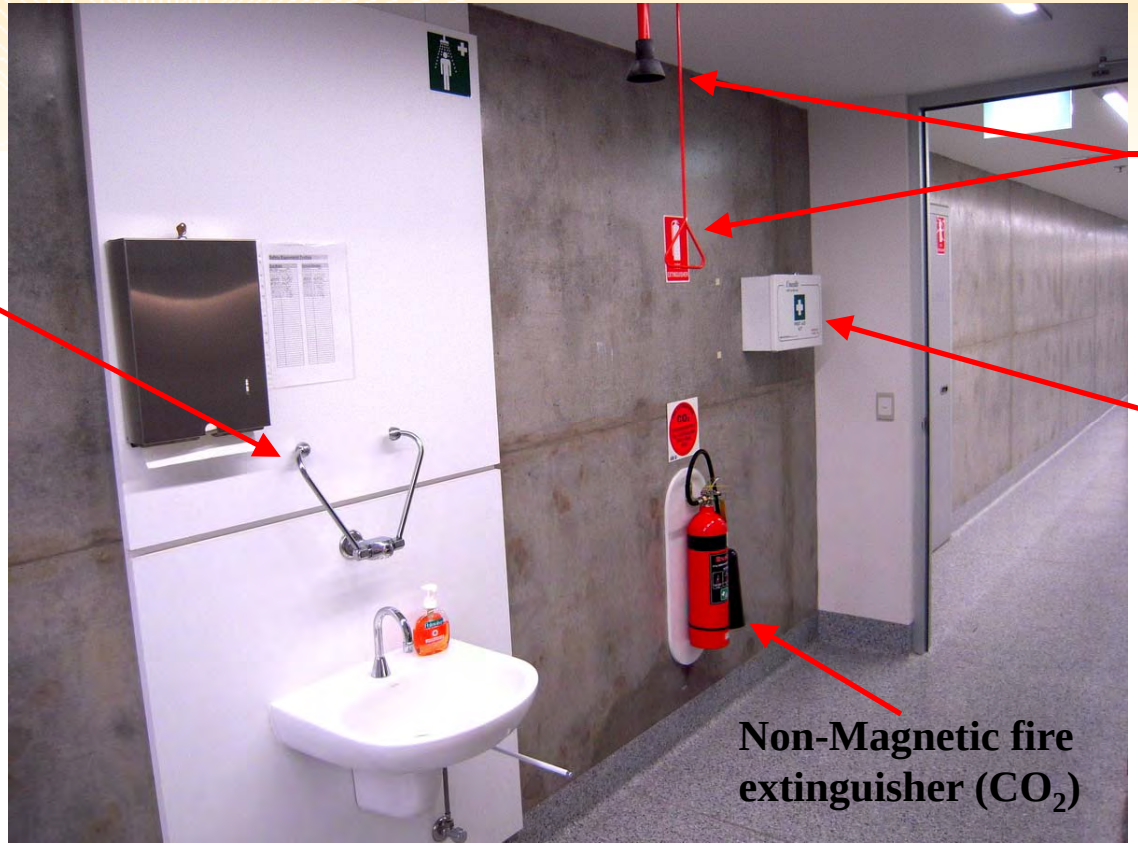
Safety Equipment

- **Personal protection facilities in the event of contamination from a chemical spill, injury or small fire**



Safety Features

❑ Safety Notice Board & Emergency Contact Numbers

☞**Bio21 Security is the first # to contact in case of an after-hours emergency**

Contact Tel #s

Bio21 Security – 24/7: ext 42481

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Safety Issues

- **Strong magnetic fields** ***(up to 19Tesla)***

  (superconducting magnets)

  - hazards with ferromagnetic items

  - pacemakers & ICDs

  - some piercings

  - [ATM cards]

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Safety Issues

- **Strong magnetic fields – ctd.**

Prohibited ferromagnetic items:

- **Tools (screwdriver, spanner, wrench)**
- **Knives, eating utensils**
- **Stapler**
- **Hole punch**
- **Paper-clips**
- **Metal containers**

Caution with….

- **ATM cards (magnetic strip)**
- **valuable analog quartz watches**

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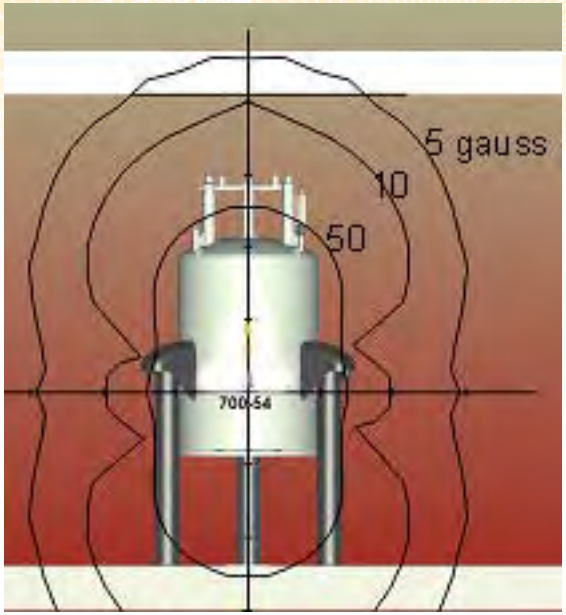
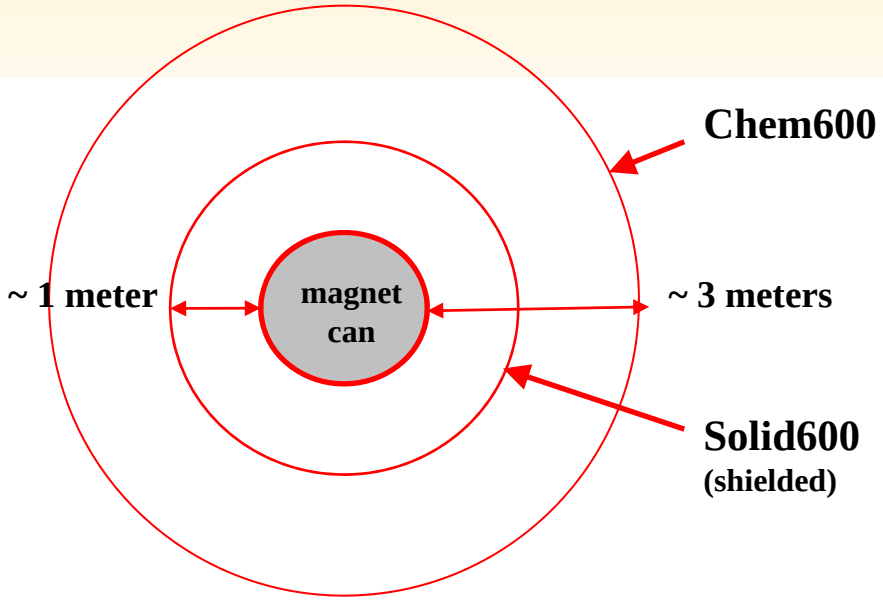
Safety Issues

Strong magnetic fields – *ctd.* : "5-Gauss Line"

= the distance from the magnet center line where the residual magnetic field has decreased to 5 Gauss





(Historically, the 5 Gauss line was the boundary where the risk of interference with magneticaly switched medical devices (cardiac pacemakers, ICDs) was negligible.)

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Safety Issues

- **Liquified gases (cryogens) used to cool magnet coil & maintain superconductivity**

  - liquid helium (LHe) – 4.2ºK – primary coolant bath for coil

  - liquid nitrogen (LN2) – 77ºK – cools LHe chamber

- **Contact with skin, eyes would cause serious "burn" injury**

- **Potential risk (very low at this site) of asphyxiation in the event of a magnet "quench"**

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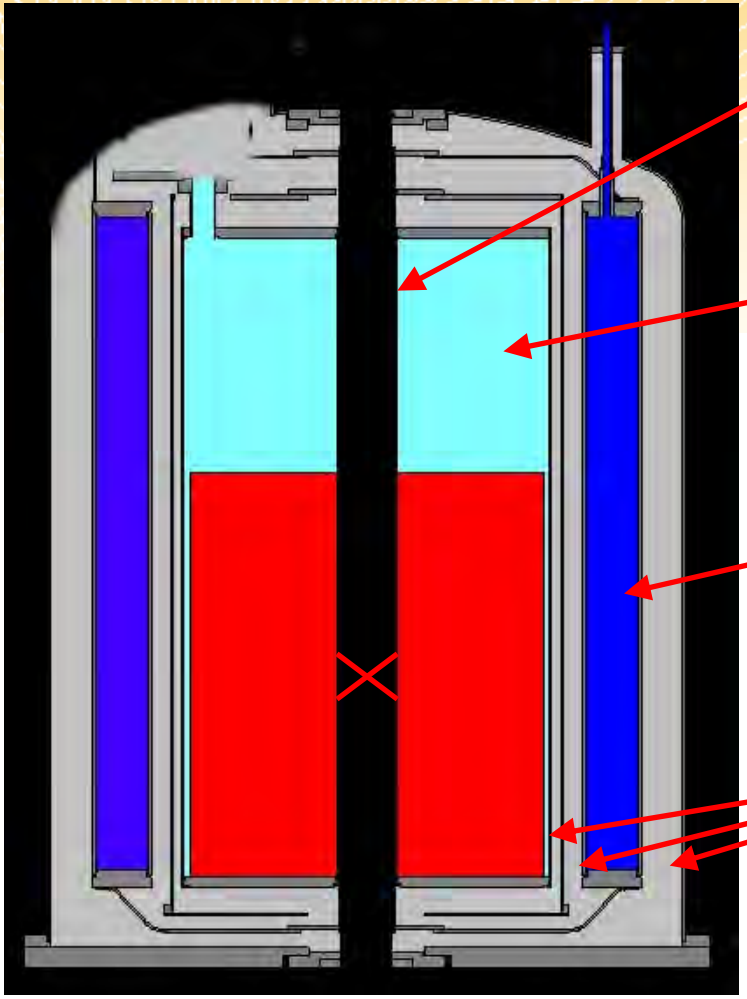
Supercon Magnet Cutaway



central bore tube for sample access

liquid helium chamber in direct contact with solenoid

annular liquid nitrogen chamber surrounding the LHe chamber

vacuum insulation chambers (3)

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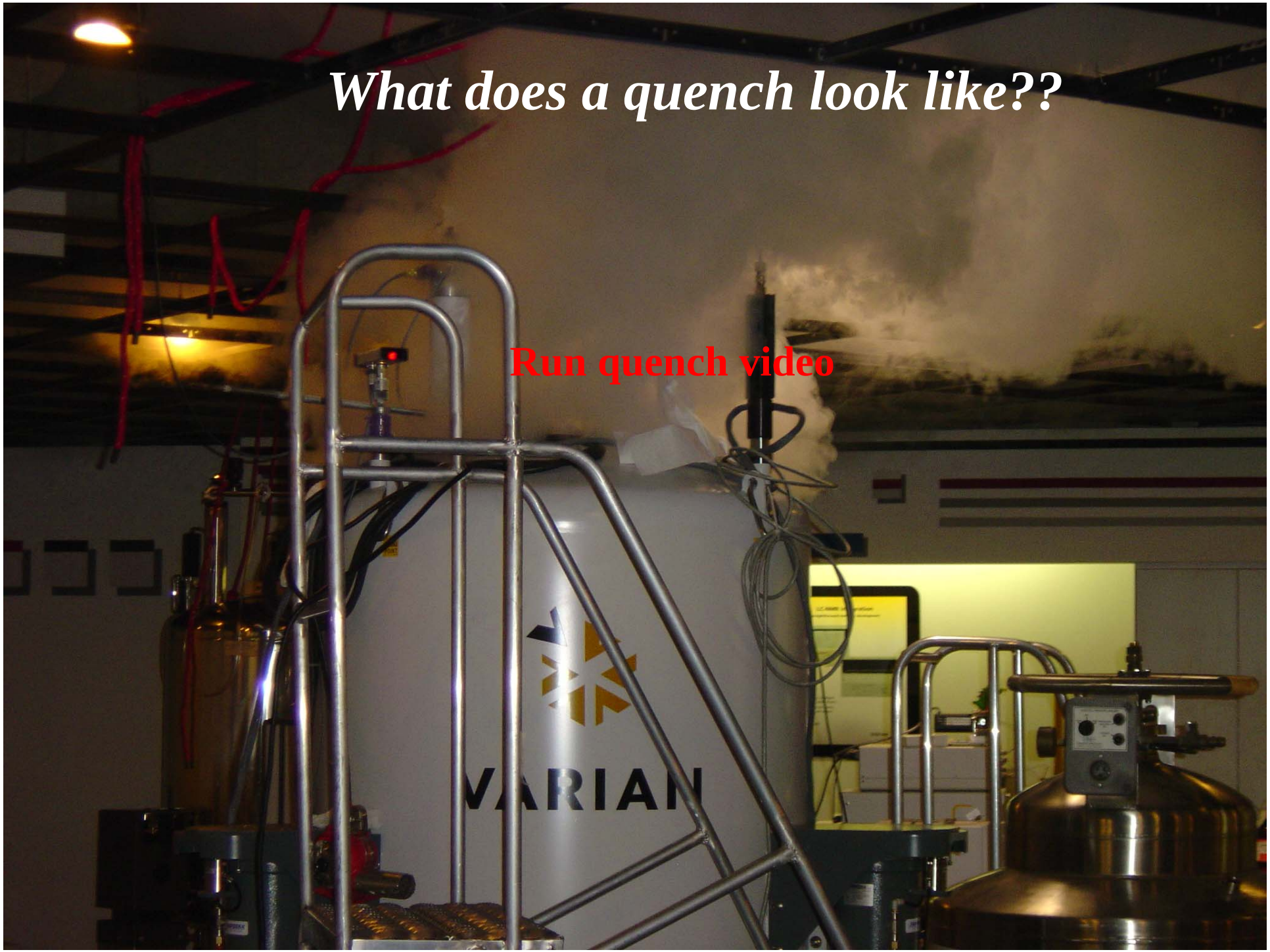
What does a quench look like??

▶ **A quench is when the magnet coil *ceases to become superconducting* & the stored energy is converted to heat in a matter of 2-3 seconds. This boils off all the cryogens in the magnet Dewar (vacuum can) & vents them into the lab.**

❖ **Low-Oxygen alarm will sound if there is a hazard caused by venting inert gases – evacuate the lab immediately!**


What does a quench look like??
Run quench video
VARIAN

Primary Entry into the Cave

“Digital” lock combination provided after training

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General Protocol in the Cave

- **Only individuals who have completed the Safety Induction process are permitted to work in the NMR Cave.**
  >>>>> *(i.e. no visitors allowed)* !!!

- **You must have received operator training on the Chem500 & Chem600 to make web bookings & operate these instruments.**

- **Food & drinks must not be consumed in the Cave.**

- **Any person found to be using or have used any unauthorised program incl. games, personal web browsing and/or file downloads on the host PCs will have their access suspended immediately.**

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General Protocol in the Cave – Repeat .....

- **Only individuals who have attended <u>this Safety Induction process</u> – or – <u>receive an on-the-spot verbal induction</u> at the Cave entrance are permitted to enter the NMR Cave.**

⇨ **Absolutely no visitors/friends/non-NMR-user colleagues etc allowed – no exceptions!**

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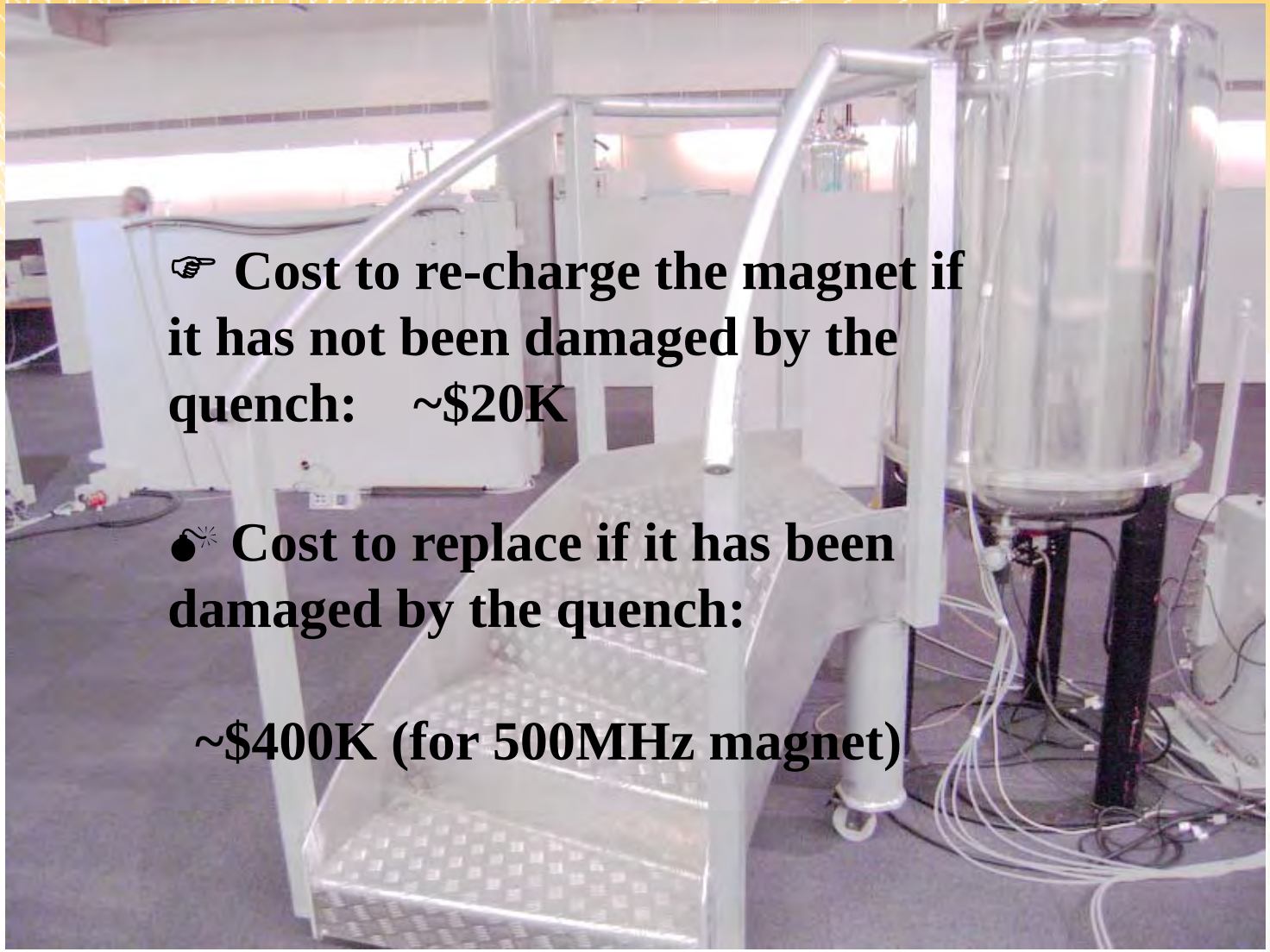
General Protocol in the Cave

If a supercon magnet is bumped or is hit by a flying metallic object, it could quench..... The Chem500 sample insertion tube is reached by platform/steps as shown below – *always tread slowly & carefully* to avoid moving these steps.

General Protocol in the Cave



☞ **Cost to re-charge the magnet if it has not been damaged by the quench: ~$20K**

💣 **Cost to replace if it has been damaged by the quench:**

~$400K (for 500MHz magnet)

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The NMR White Board:

- Always check this before entering the cave (advises instrument problems)
- Add comments of problems/failures that you have experienced (+ your name)
- Training sessions booking forms posted here (after swipe card access enabled)

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Sample Protocol

* **The following types of samples must NOT be run on any spectrometers:**

  - **bio-hazardous materials**
  - **radioactive materials**
  - **volatile organo-metallics**
  - **volatile sulphur containing cpds. e.g. thiols, thiophenols**
  - **volatile selenium containing cpds.**
  - **trifluoracetic acid as solvent or major co-solvent**
  - ***if in doubt, ask!***

* **If you need to run samples that fit into these categories, you must consult David Keizer or Hamish Grant. In such cases, you will be required to use thick-walled NMR tubes to minimise the rick of breakage & contamination.**

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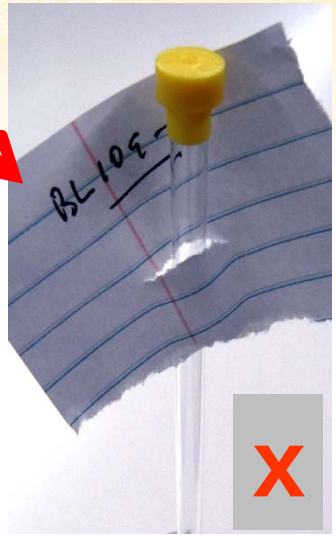
Sample Tube Protocol

★ **Sample Tube I.D. – mandatory, e.g. :**

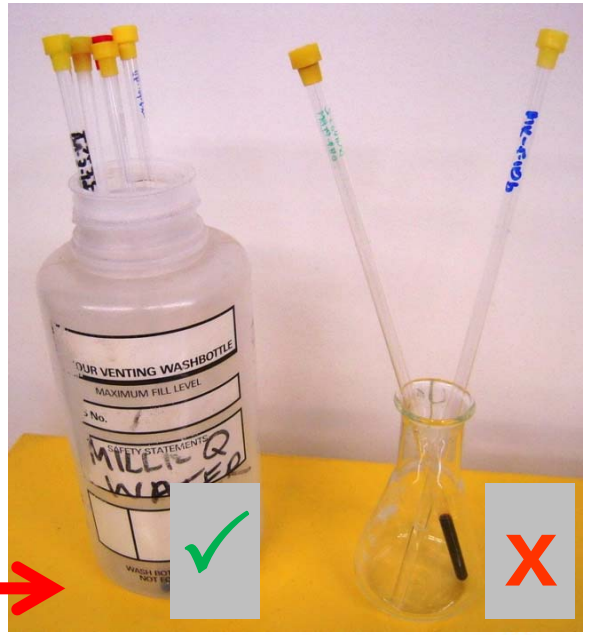
- annotate tube using marker pen with your initials
- attach mylar film-type film

- **Loose paper "poke through" tags are *not* acceptable**



★ **Tube Transportation: (from your lab & back)**

No shirt pockets, glass flasks, esp. no metal containers….



Sample Tube Protocol (ctd)

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Home :: NMR Consumables :: Tubes

Products Catalogue

Catering (28)
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Cryogenic (6)
Domestic-> (55)
Labware-> (673)
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|_ Deuterates (11)
|_ Misc (2)
|_ Tubes (6)
Radiation (0)
Safety-> (92)
Services-> (18)
Stationery (241)

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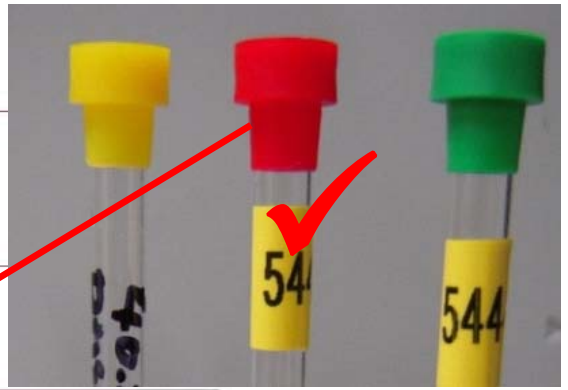
Tubes

Displaying **1** to **6** (of **6** products)

Product Image	Qty.-	Item Name	Price
	0	**NMR Tube 5mm 7" (507-PP-8) box of 5 - ON REQUEST** Properties: MHz Rating: 300, Length (inch): 8, OD...	Login for price
	0	**NMR Tube 5mm 7" (535-PP-7) box of 5 - ON REQUEST** Properties: MHz Rating: 600, Length (inch): 7, OD...	Login for price
	0	**NMR Tube 5mm 7" (541-PP-7) box of 5 - ON REQUEST** Properties: MHz Rating: 800, Length (inch): 7, OD...	
	390	**NMR Tube 5mm 7" Standard - NO cap** Standard tube for robots and Chemistry use - NO...	
	1089	**NMR Tube Caps, SampleJet (Bio700 ONLY)** REQUIRED for all samples inserted via SampleJet...	
	250	**NMR Tubes Caps, Standard (Red)**	Login for price



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Sample Tube Breakages

❑ **Sample tube breakages are uncommon but could be very rare indeed if every precaution is taken when carrying the sample to & from the Cave – and – great care is taken when.....**

▶ **Inserting the tube into the spinner (slowly!)**

▶ **Inserting & retrieving the spinner & tube from the top of the magnet during insert/eject cycles (on manual-insert spectrometers – Chem500 & 600)**

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Sample Tube Breakages Protocol:

- **If it is *your* sample, clean up the glass & sample residue using the disposable gloves & paper towel provided.**

- **Place glass fragments in the yellow plastic container on the table near the Cave entry.**

- **If it is the *standard $CDCl_3$ sample*, report breakage immediately to Hamish Grant (leave a message on the white board if he is not in the office). Clean up the broken glass & retrieve the special white teflon tube cap (if applicable).**

- **If HG is not available to immediately replace the broken standard tube, leave a note on the keyboard advising the next user that this sample has been broken & is not in the magnet.**

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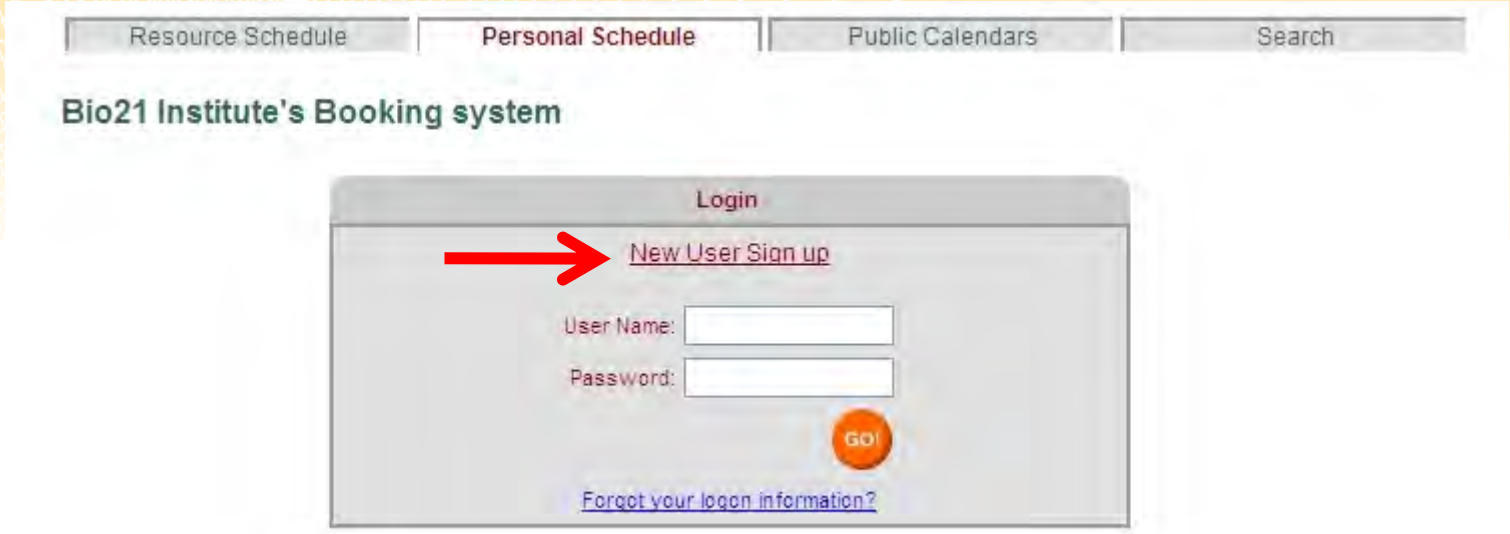
Booking Time on the Chem500 & 600

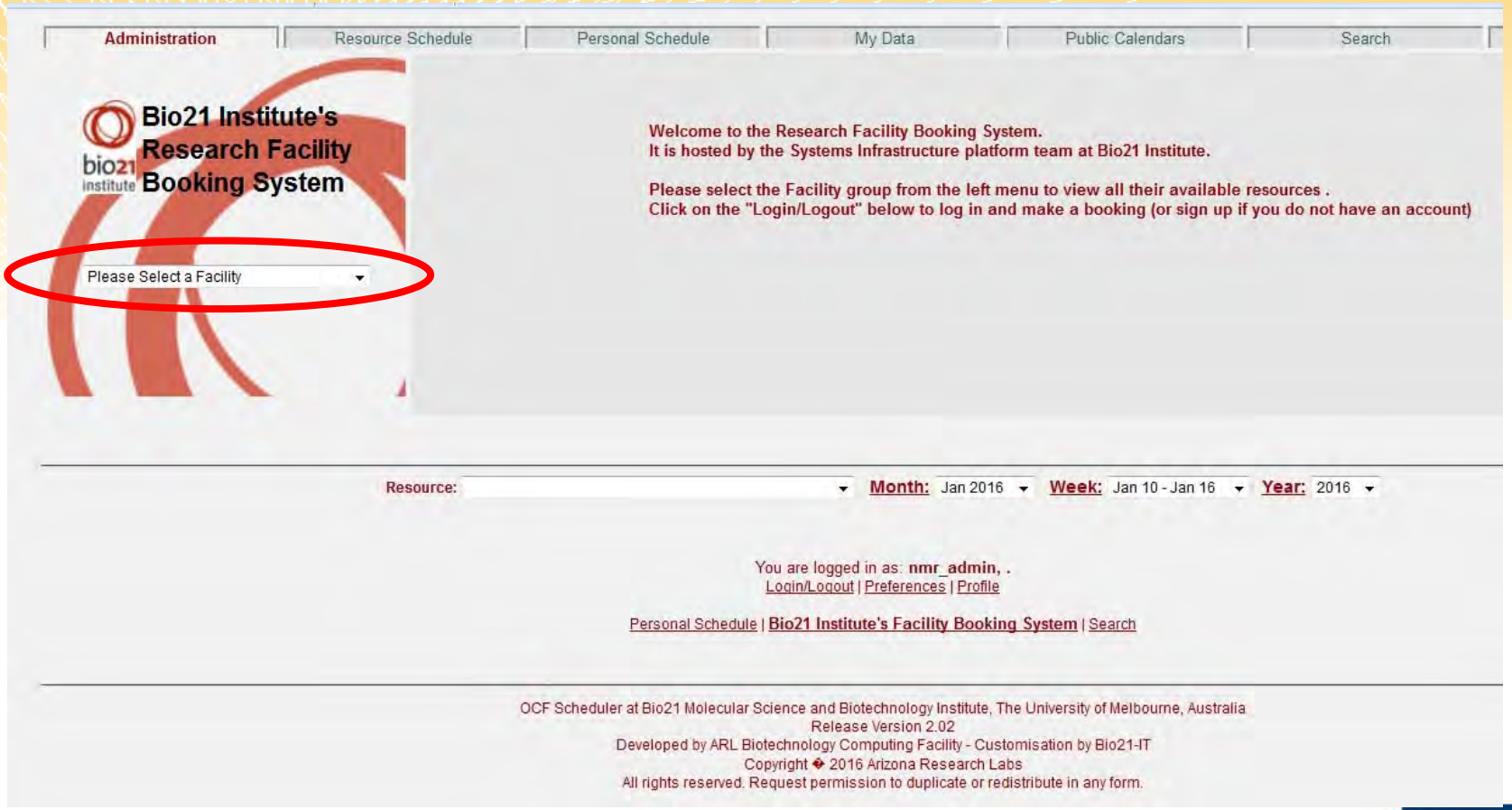
Website: https://www.bio21.org/booking/login



Click on "New User Sign up" to register on first access.

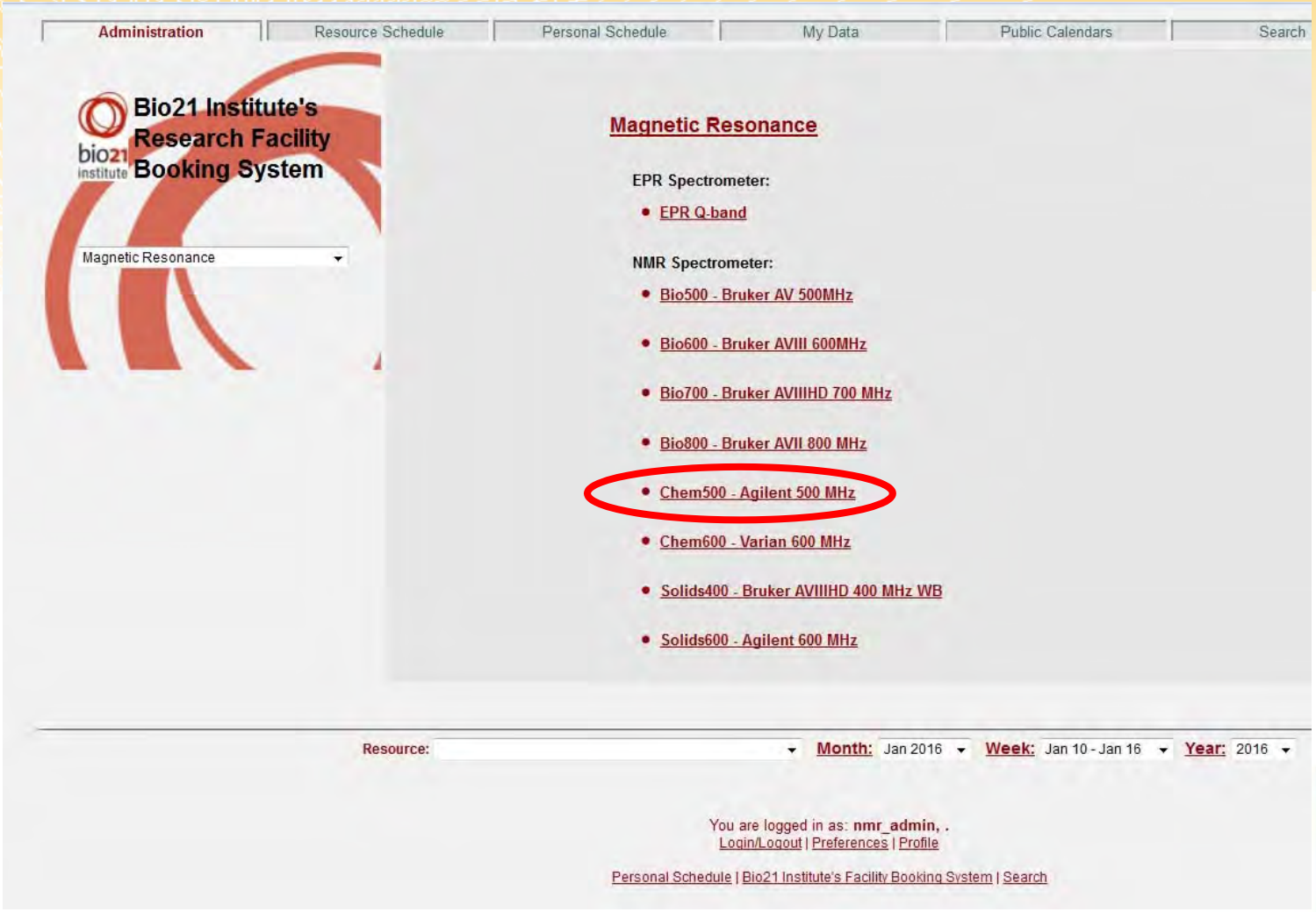
Booking Time on the Chem500 & 600

Website: https://www.bio21.org/booking/login



Booking Time on the Chem500 & 600

Website: https://www.bio21.org/booking/login



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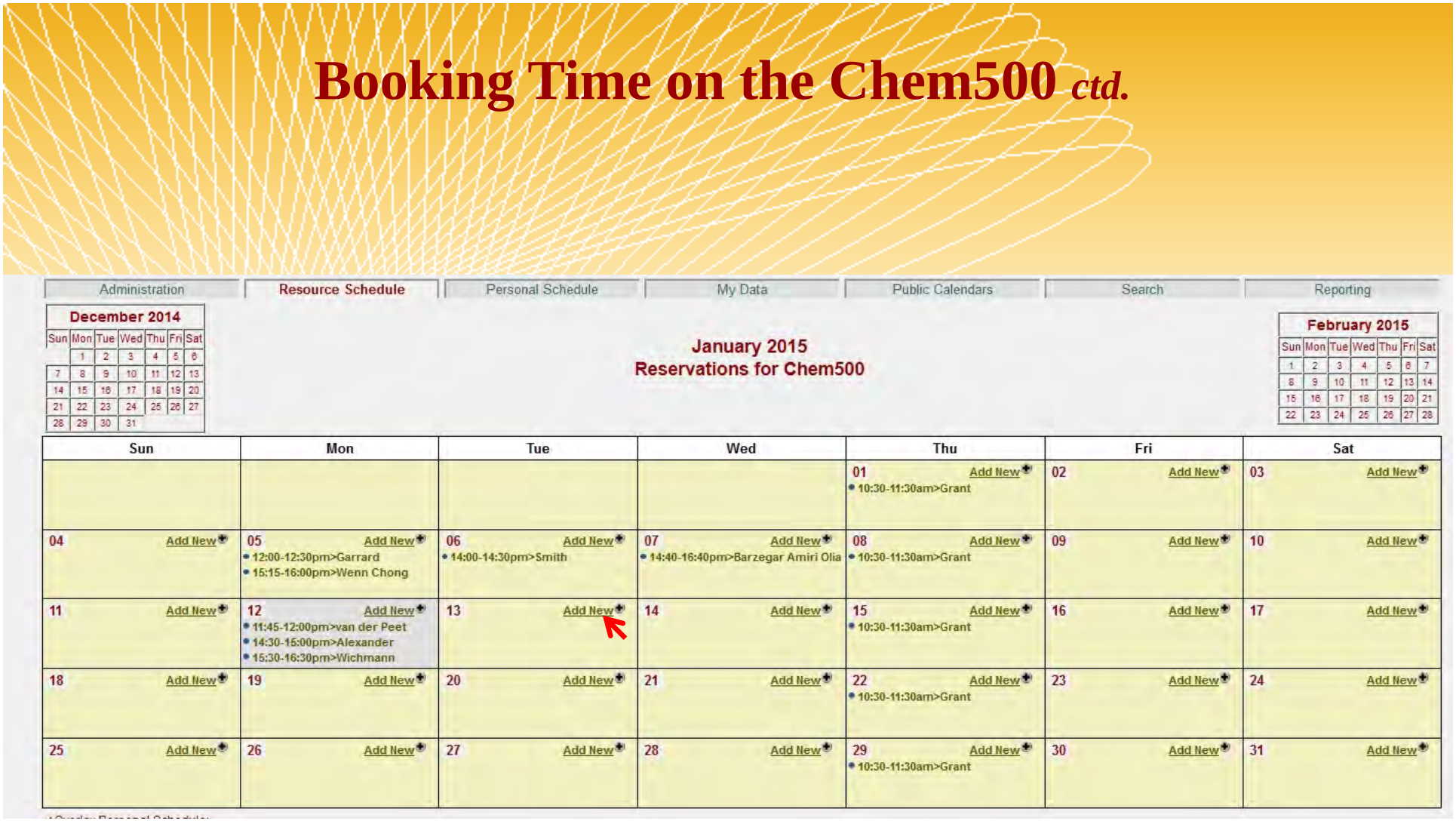
Booking Time on the Chem500 *ctd.*



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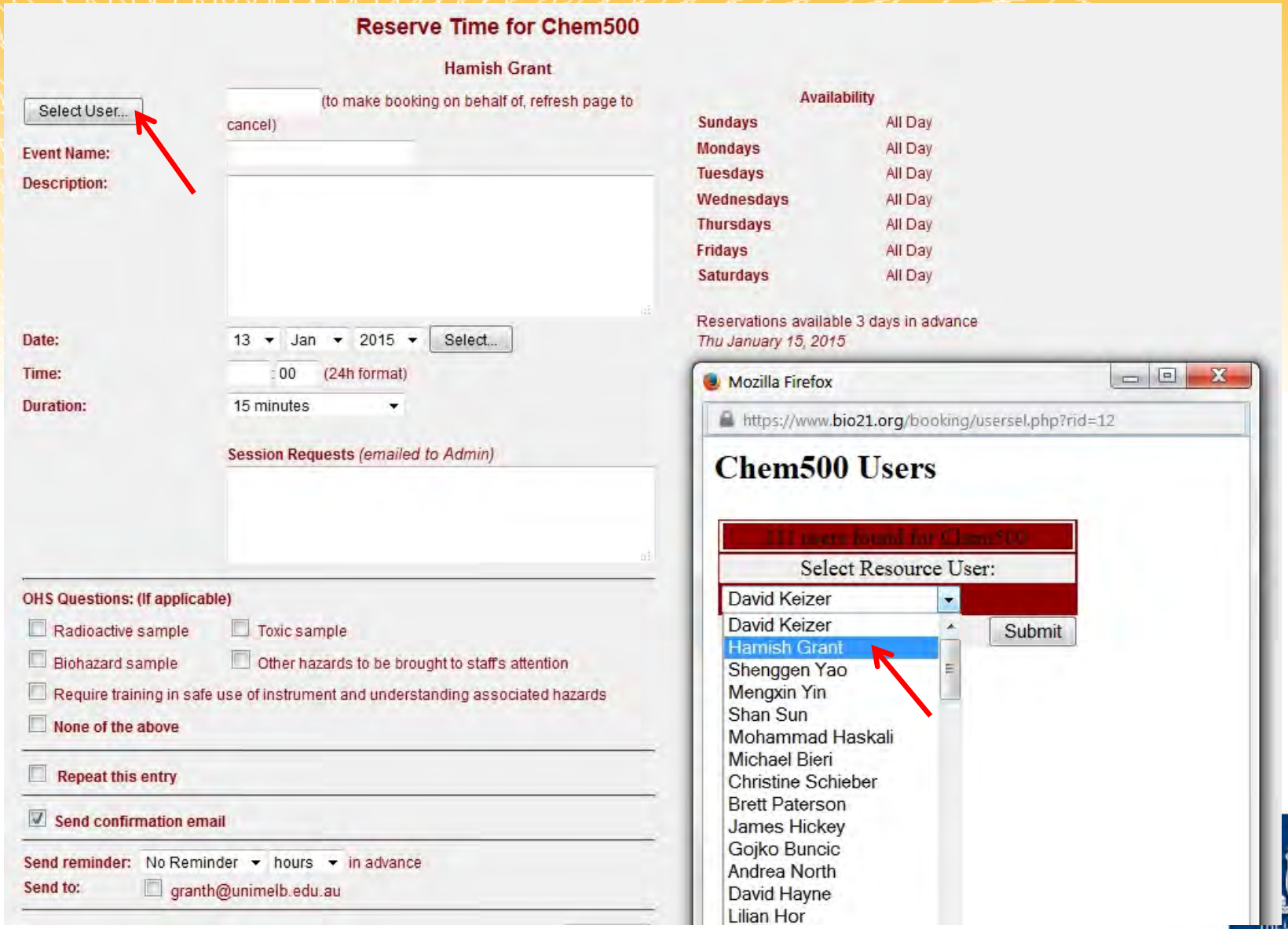
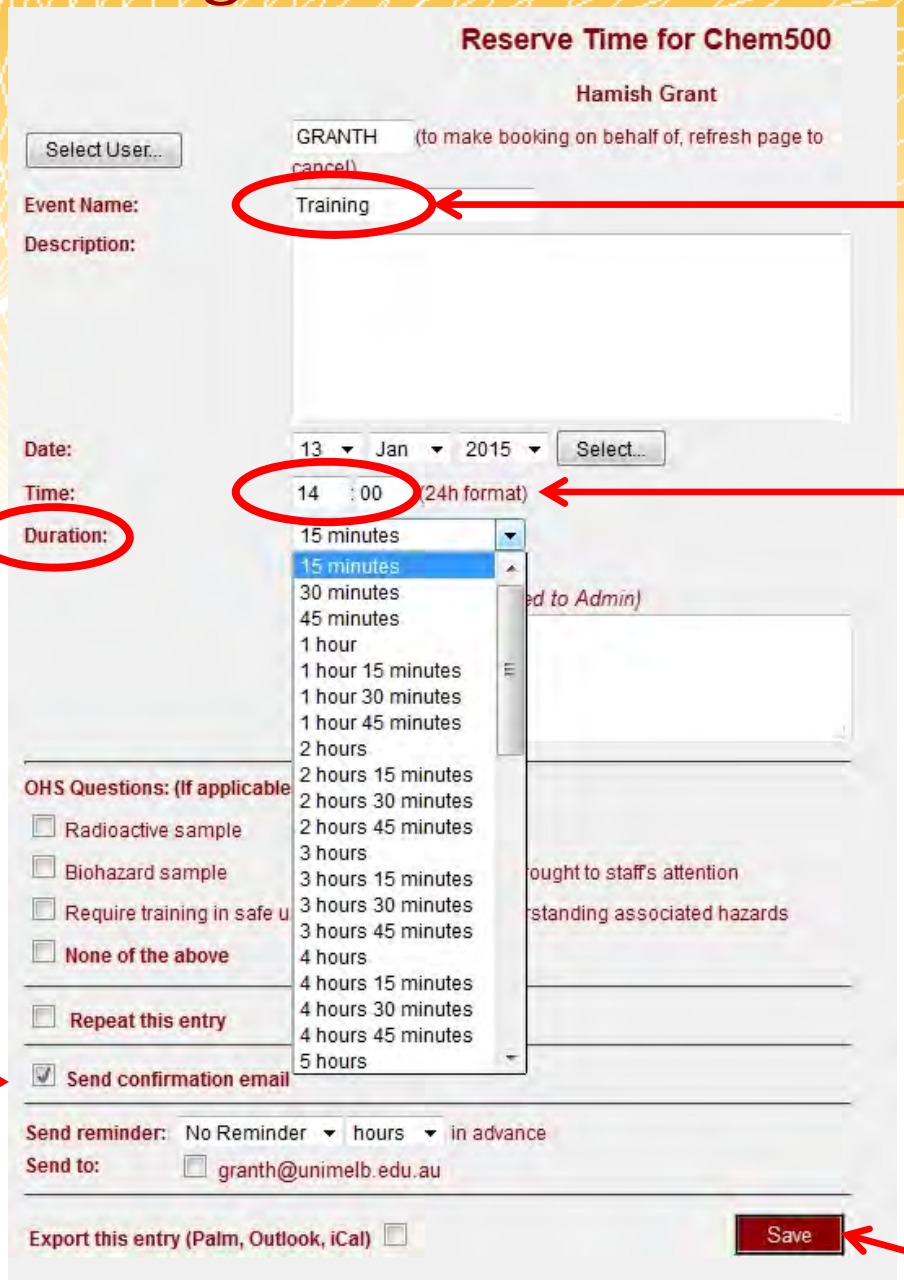
Booking Time on the Chem500 *ctd.*



Booking Time on the Chem500 *ctd.*



Entry in this field is mandatory

Note 24hr format required

Note: Max 2hrs contiguous booking time on Chem500/600 during 9am – 5pm

Optional

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Contacts

Instrument Training – Chem500, Chem600, Bio600 (Chemistry users) & Robot400:

- Dr Hamish Grant, Deputy Facility Manager –

xt 42477
granth@unimelb.....

- Initially, training sessions for the Robot400 will be bookable in groups of 5 from a sign-up form to be posted on the white board outside the NMR office

- Subsequent training on the Chem600 & Chem500 will also be bookable via the same system on an as-needed basis.

✶ Instrument problems & software issues – Dr Grant should be notified immediately (in person, NMR white board message or email for non-urgent matters)

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Contacts – ctd.

Instrument Training for Biomolecule Users –

Bruker Spectrometers: Bio800, 700, 600:

- Dr Shenggen Yao

xt 42203
shyao@unimelb.....

These spectrometers are prioritised to biomolecular projects & are normally reserved for Biochemistry post-graduate users & research students and staff from external academic & government institutions.

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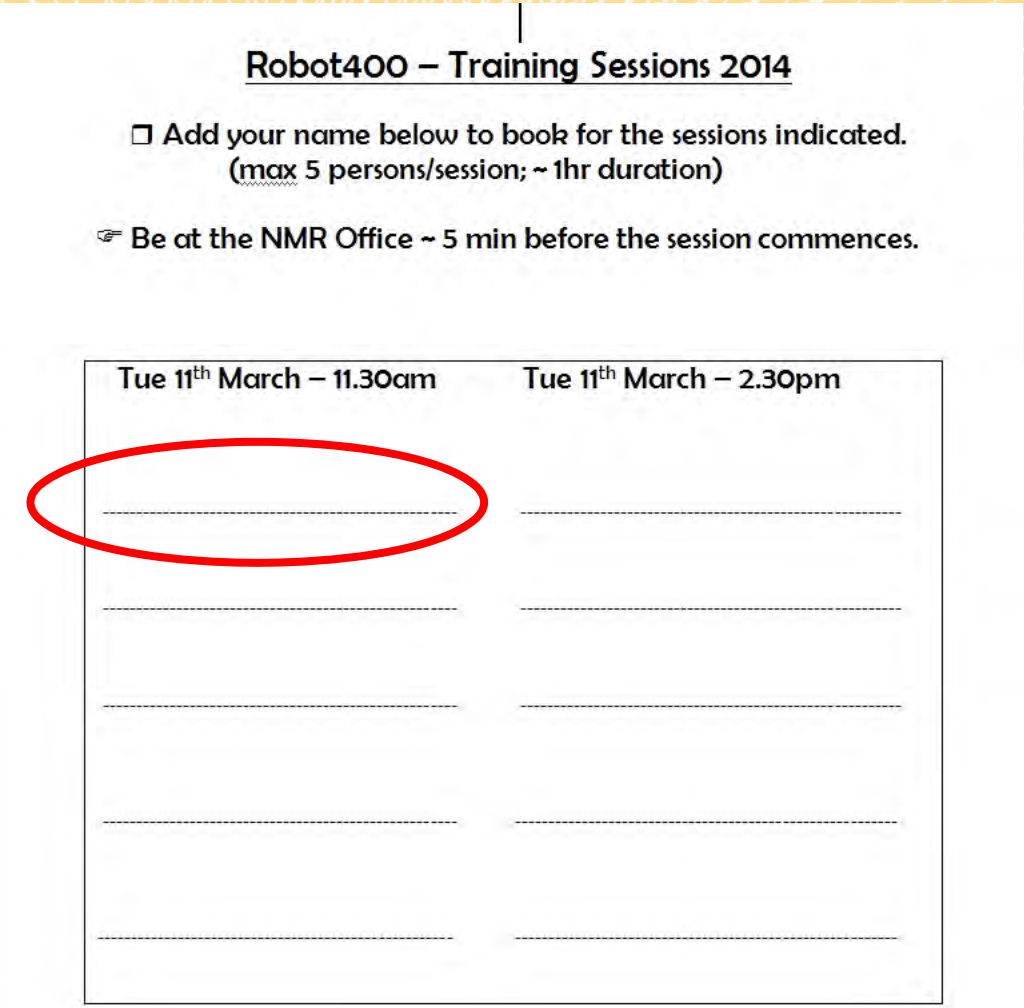
Contacts – *ctd.*

⋆ **Facility Manager:**

- Dr David Keizer –

xt 42218
dkeizer@unimelb.....

Training Sessions – Booking Sheets posted on Cave Whiteboard outside my office



Robot400 – Training Sessions 2014

❒ Add your name below to book for the sessions indicated.
(max 5 persons/session; ~ 1hr duration)

☞ Be at the NMR Office ~ 5 min before the session commences.

Tue 11th March – 11.30am	Tue 11th March – 2.30pm

You may need to have a colleague swipe you into the Cave to add your name to the list – your swipe card will not be active for 3-4 days after this Induction.

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Please complete the Safety & NMR Protocol Questionaire *now* & hand to me before leaving.

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