

The Bio21 NMR Cave

Safety Induction & Protocols for
the Use of NMR Spectrometers

- 2017

BRUKER
800 US²



bio21
institute



THE UNIVERSITY OF
MELBOURNE

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 ^1H & ^{13}C are required)
(Time on these instruments *CAN* be booked)



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).



400MHz “Walkup” System – Robot400

Not available for sample submission from ~10am till 12pm every Thursday
- liquid nitrogen fill time



500MHz Multinuclear System – Chem500

- ⇒ 2 hour max 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.



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

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



Biochemistry/Biomolecular Spectrometers



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

Solid-State Research Group Spectrometers

(Prof. Frances Separovic)

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



Solid-State Research Group Spectrometers

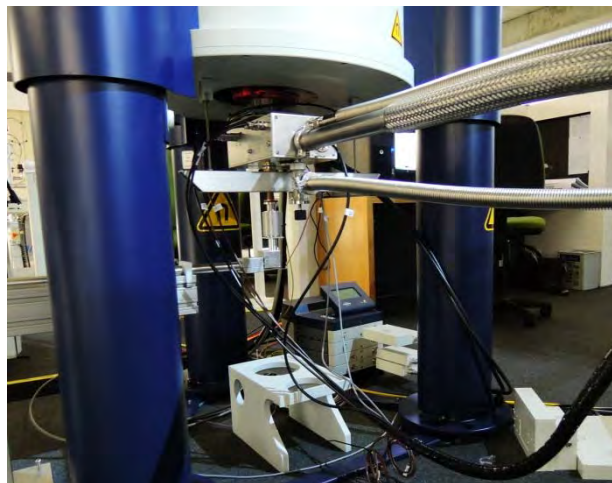
(Prof. Frances Separovic)

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



Solid-State Research Group Spectrometers

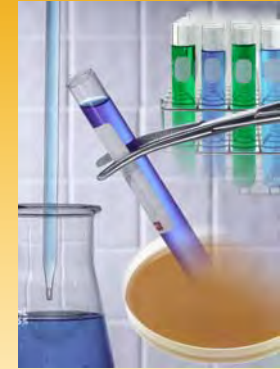
DNP is a very complex hardware combination including a wide-bore 400MHz spectrometer which can have the main field of the magnet varied during the experiment. A gyrotron powered by a cryogen-free supercon magnet generates an intense microwave field at 263GHz.



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



Safety

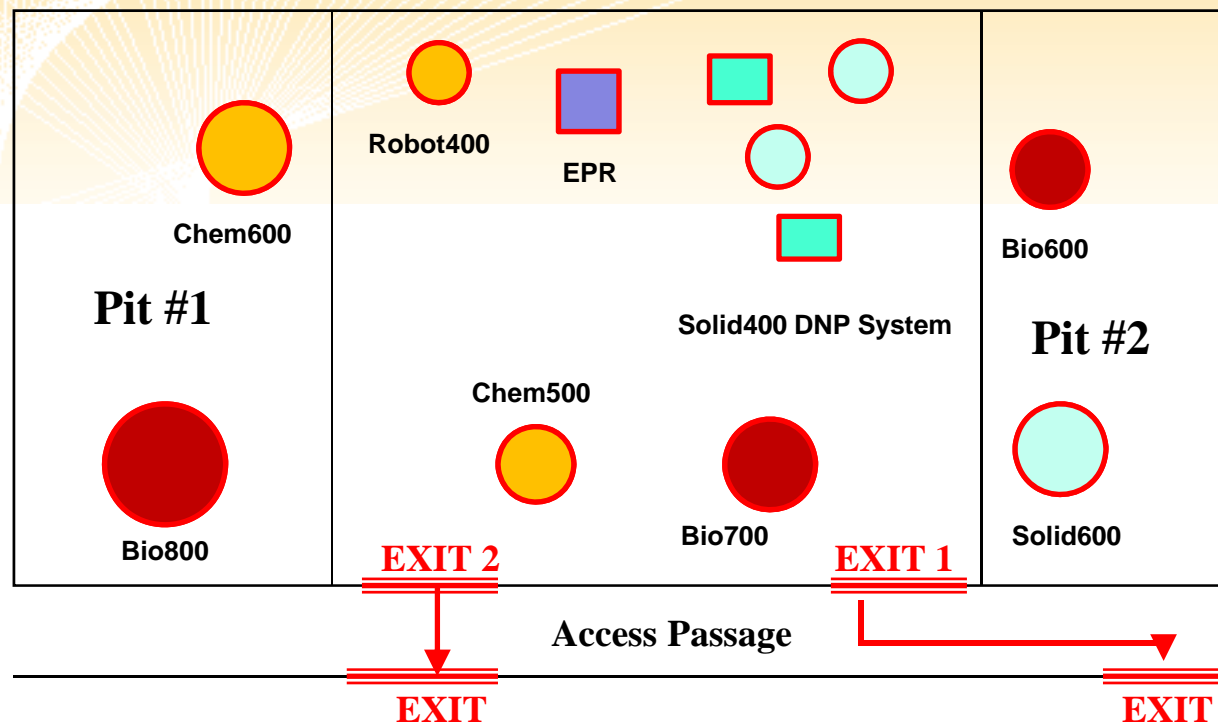


- **Safety is the #1 consideration in all activities undertaken at the University.**
- **It is the responsibility of all staff and students**

Emergency Evacuation

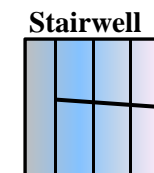
- Procedure in the event that the Building Evacuation Alarm is activated

Cave – main level



Underground
Carpark

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



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.



Emergency exit only – opposite center access doors to Cave.

Safety Equipment

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

eye-wash
station



emergency
shower

First-Aid
Cabinet

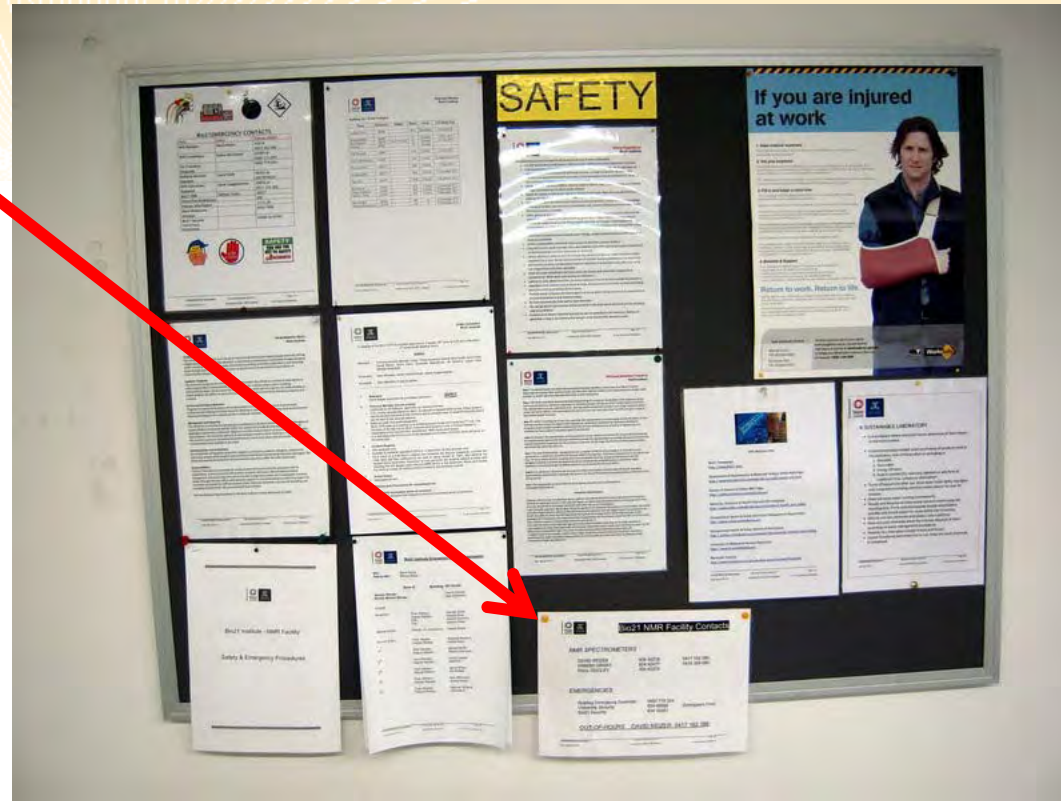
Non-Magnetic fire
extinguisher (CO₂)

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

Safety Issues



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

(superconducting magnets)

- hazards with ferromagnetic items
- pacemakers & ICDs
- some piercings
- [ATM cards]



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....

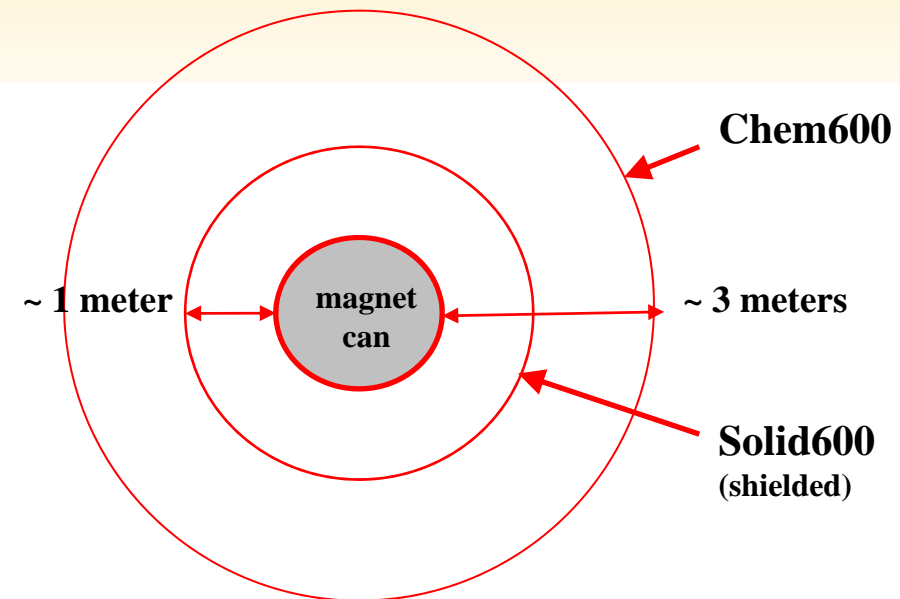
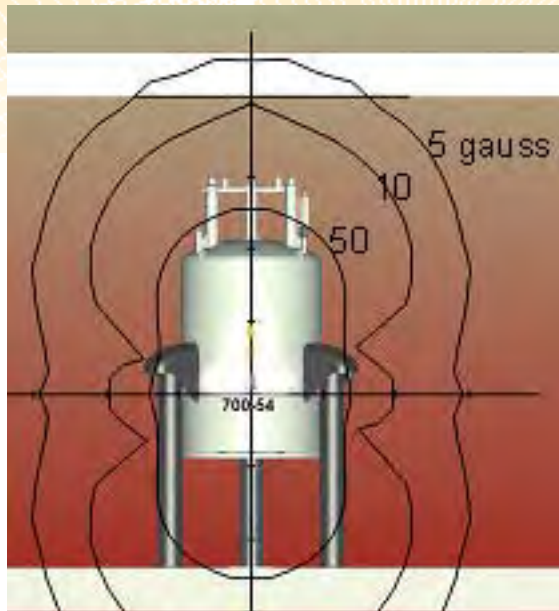
- ATM cards (magnetic strip)
- valuable analog quartz watches



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



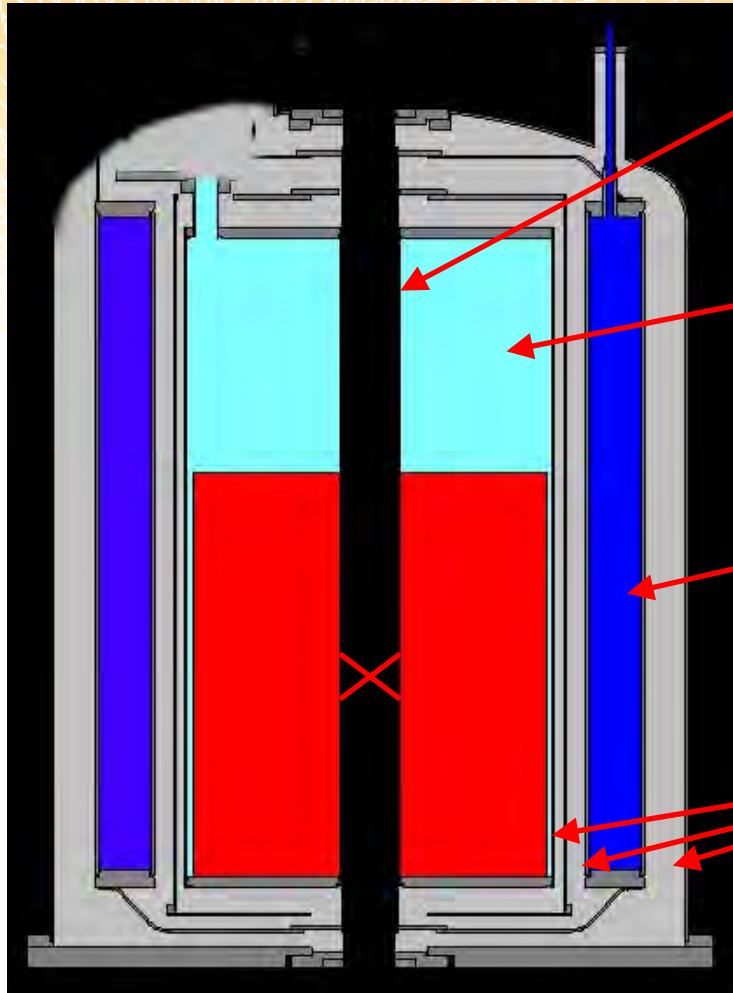
(Historically, the 5 Gauss line was the boundary where the risk of interference with magnetically switched medical devices (cardiac pacemakers, ICDs) was negligible.)

Safety Issues



- **Liquified gases (cryogenes) used to cool magnet coil & maintain superconductivity**
 - liquid helium (LHe) – 4.2°K – primary coolant bath for coil
 - liquid nitrogen (LN2) – 77°K – 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”**

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)



What does a quench look like??

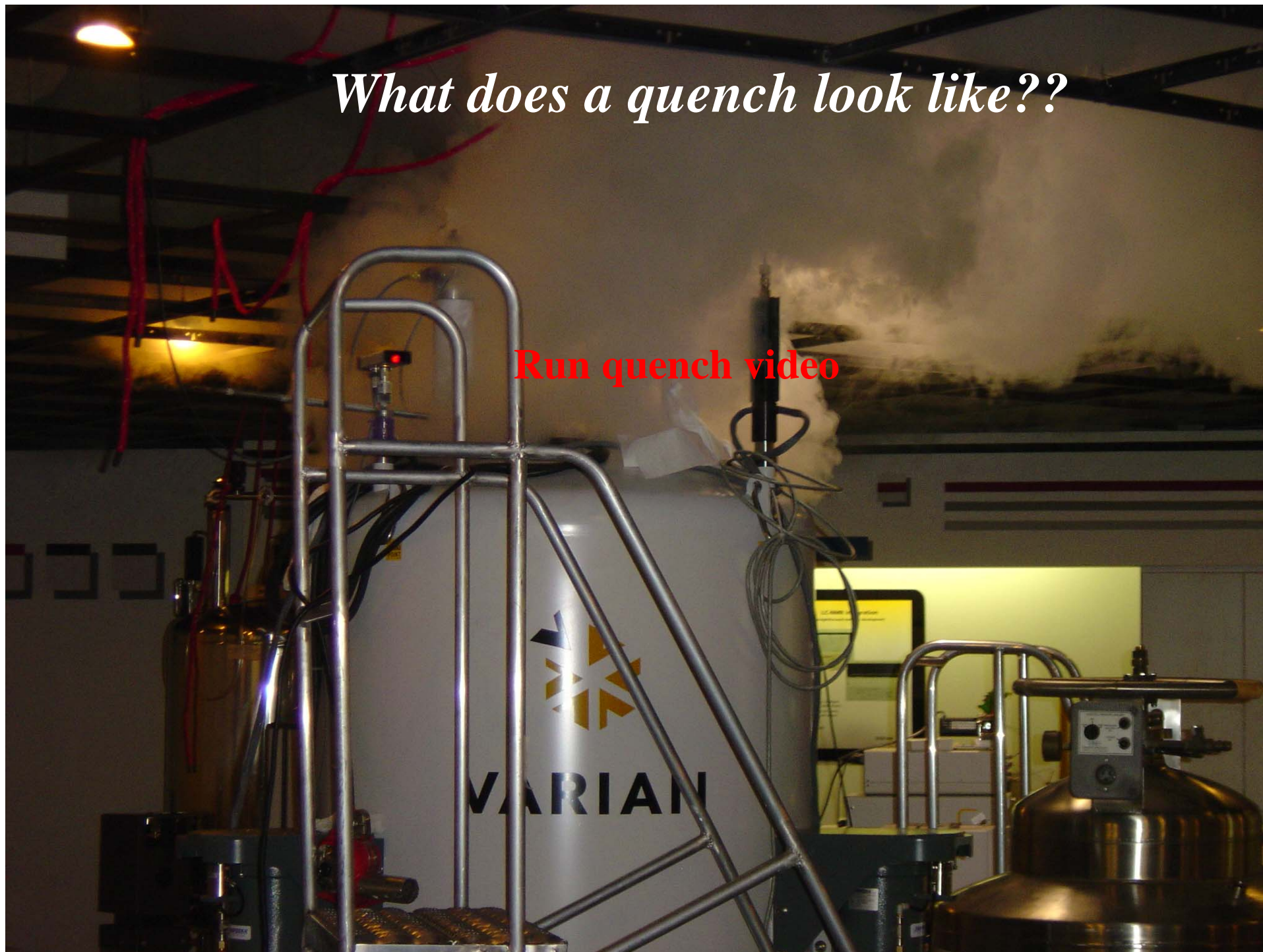
► A quench is when the magnet coil ceases to become superconducting & the stored energy is converted to heat in a matter of 2-3 seconds. This boils off all the cryogenics in the magnet Dewar (vacuum can) & vents them into the lab.



❖ 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



Primary Entry into the Cave



“Digital” lock combination provided after training

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.

General Protocol in the Cave – Repeat

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

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

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.

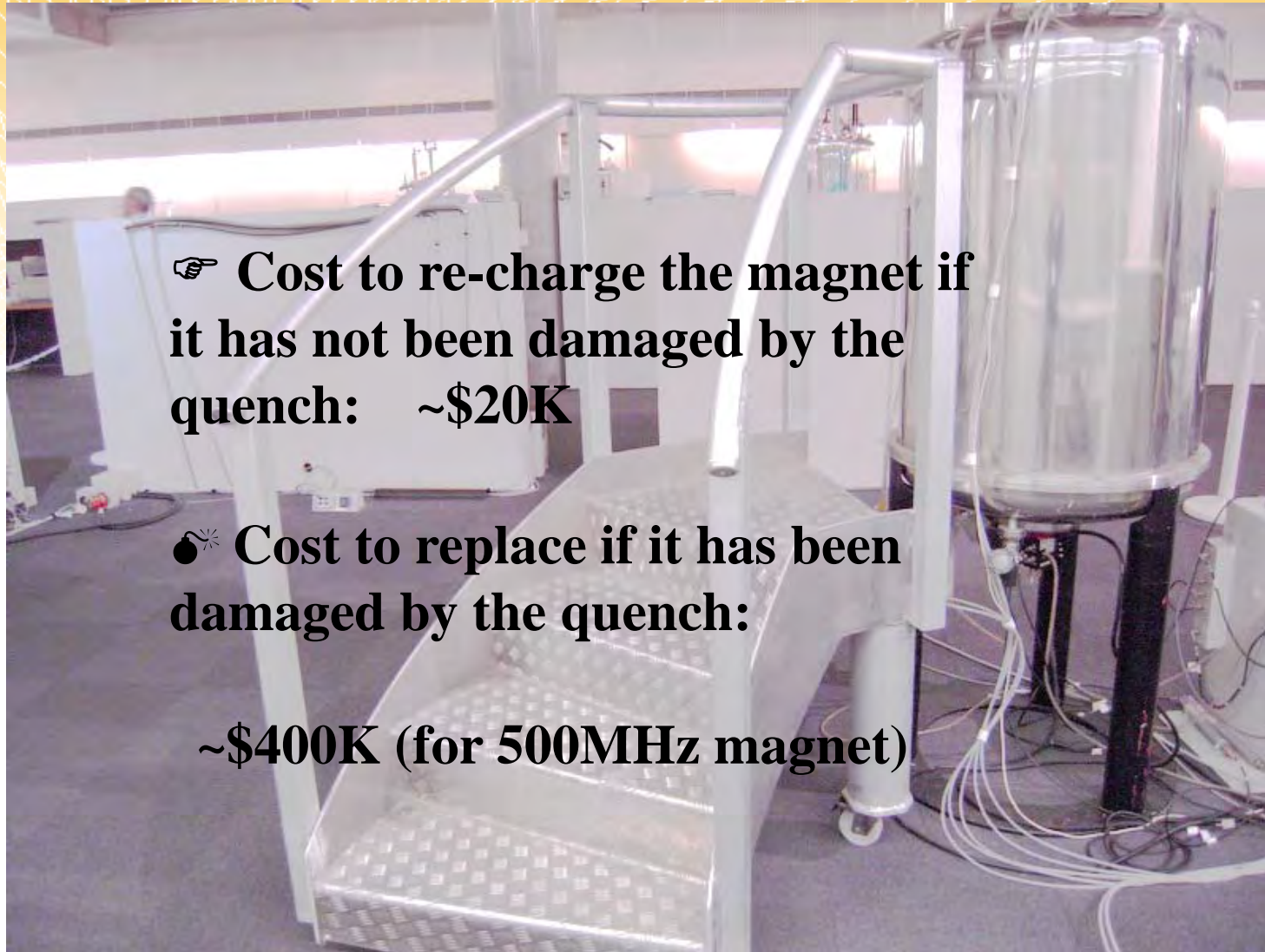


General Protocol in the Cave

☞ 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)



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)



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.
- trifluoroacetic 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 risk of breakage & contamination.**

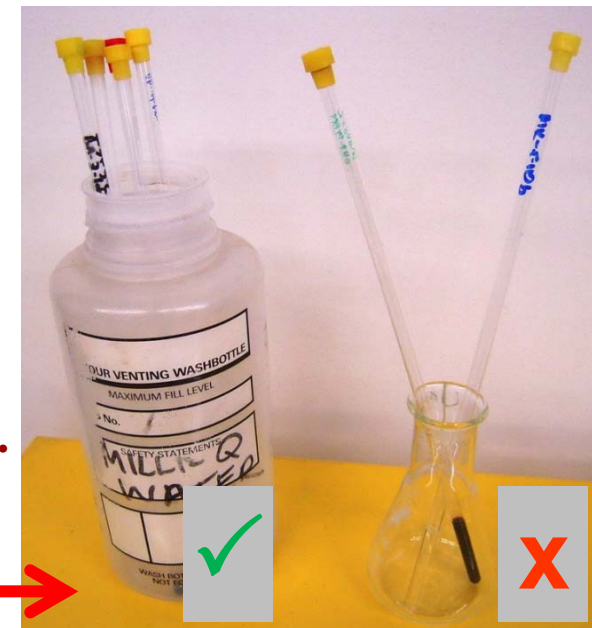


Sample Tube Protocol


- ★ 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: No shirt pockets, glass flasks, (from your lab & back) esp. no metal containers....



Sample Tube Protocol (ctd)



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Home :: NMR Consumables :: Tubes

Products Catalogue

Catering (28)

Chemicals-> (450)

Cryogenic (6)

Domestic-> (55)

Labware-> (673)

NMR Consumables-> (19)

_ Deuterates (11)

_ Misc (2)

_ Tubes (6)

Radiation (0)







Safety-> (92)

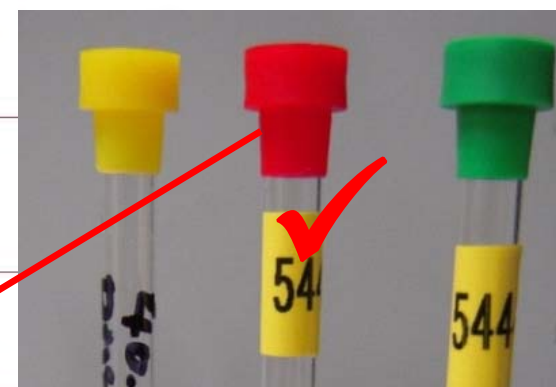
Services-> (18)

Stationery (241)

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



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!)

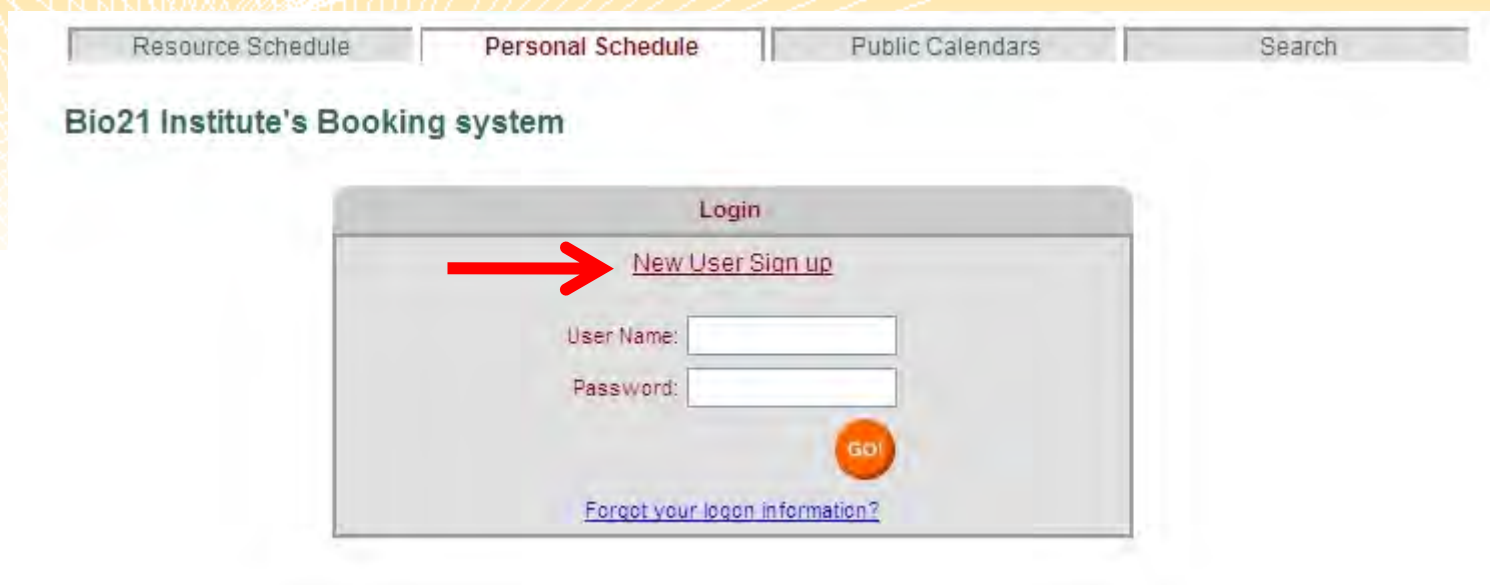
► Inserting & retrieving the spinner & tube from the top of the magnet during insert/eject cycles (on manual-insert spectrometers – Chem500 & 600)

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.

Booking Time on the Chem500 & 600

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



Resource Schedule Personal Schedule Public Calendars Search

Bio21 Institute's Booking system

Login

[New User Sign up](#)

User Name:

Password:

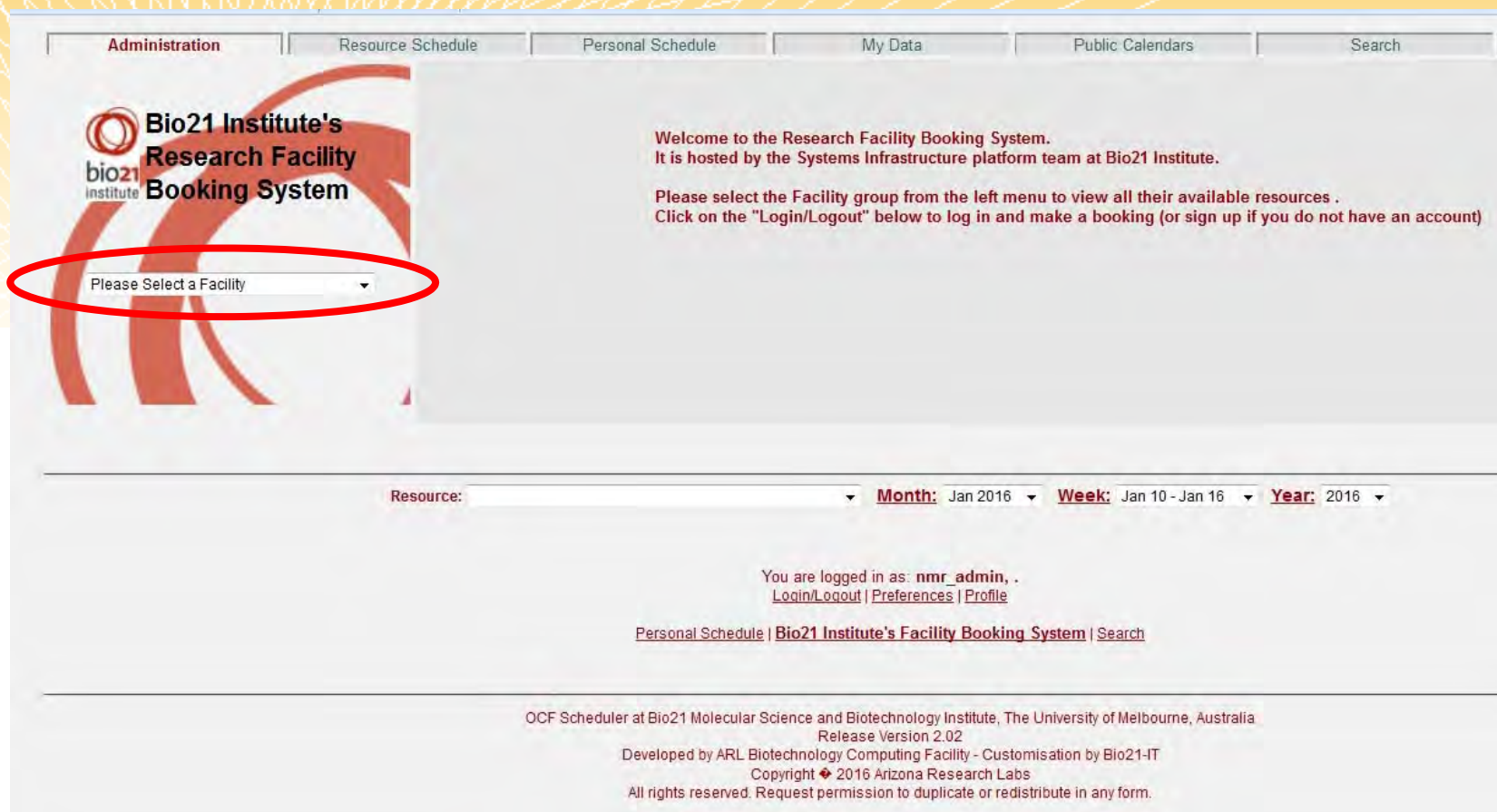
[Forgot your login information?](#)

Click on “**New User Sign up**” to register on first access.



Booking Time on the Chem500 & 600

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



Bio21 Institute's Research Facility Booking System

Welcome to the Research Facility Booking System.
It is hosted by the Systems Infrastructure platform team at Bio21 Institute.

Please select the Facility group from the left menu to view all their available resources .
Click on the "Login/Logout" below to log in and make a booking (or sign up if you do not have an account)

Please Select a Facility

Resource: Month: Jan 2016 Week: Jan 10 - Jan 16 Year: 2016

You are logged in as: **nmr_admin, .**
[Login/Logout](#) | [Preferences](#) | [Profile](#)

[Personal Schedule](#) | [Bio21 Institute's Facility Booking System](#) | [Search](#)

OCF Scheduler at Bio21 Molecular Science and Biotechnology Institute, The University of Melbourne, Australia
Release Version 2.02
Developed by ARL Biotechnology Computing Facility - Customisation by Bio21-IT
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Booking Time on the Chem500 & 600

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

Bio21 Institute's Research Facility Booking System

Magnetic Resonance

Magnetic Resonance

EPR Spectrometer:

- [EPR Q-band](#)

NMR Spectrometer:

- [Bio500 - Bruker AV 500MHz](#)
- [Bio600 - Bruker AVIII 600MHz](#)
- [Bio700 - Bruker AVIIIHD 700 MHz](#)
- [Bio800 - Bruker AVII 800 MHz](#)
- [Chem500 - Agilent 500 MHz](#)
- [Chem600 - Varian 600 MHz](#)
- [Solids400 - Bruker AVIIIHD 400 MHz WB](#)
- [Solids600 - Agilent 600 MHz](#)

Resource: **Month:** Jan 2016 **Week:** Jan 10 - Jan 16 **Year:** 2016

You are logged in as: **nmr_admin**, .
[Login/Logout](#) | [Preferences](#) | [Profile](#)

[Personal Schedule](#) | [Bio21 Institute's Facility Booking System](#) | [Search](#)



Booking Time on the Chem500 *ctd.*

Administration	Resource Schedule	Personal Schedule	My Data	Public Calendars	Search	Reporting																																																																													
December 2014 <table border="1"> <tr><td>Sun</td><td>Mon</td><td>Tue</td><td>Wed</td><td>Thu</td><td>Fri</td><td>Sat</td></tr> <tr><td></td><td>1</td><td>2</td><td>3</td><td>4</td><td>5</td><td>6</td></tr> <tr><td>7</td><td>8</td><td>9</td><td>10</td><td>11</td><td>12</td><td>13</td></tr> <tr><td>14</td><td>15</td><td>16</td><td>17</td><td>18</td><td>19</td><td>20</td></tr> <tr><td>21</td><td>22</td><td>23</td><td>24</td><td>25</td><td>26</td><td>27</td></tr> <tr><td>28</td><td>29</td><td>30</td><td>31</td><td></td><td></td><td></td></tr> </table>	Sun	Mon	Tue	Wed	Thu	Fri	Sat		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31				January 2015 Reservations for Chem500					February 2015 <table border="1"> <tr><td>Sun</td><td>Mon</td><td>Tue</td><td>Wed</td><td>Thu</td><td>Fri</td><td>Sat</td></tr> <tr><td>1</td><td>2</td><td>3</td><td>4</td><td>5</td><td>6</td><td>7</td></tr> <tr><td>8</td><td>9</td><td>10</td><td>11</td><td>12</td><td>13</td><td>14</td></tr> <tr><td>15</td><td>16</td><td>17</td><td>18</td><td>19</td><td>20</td><td>21</td></tr> <tr><td>22</td><td>23</td><td>24</td><td>25</td><td>26</td><td>27</td><td>28</td></tr> </table>	Sun	Mon	Tue	Wed	Thu	Fri	Sat	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
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Booking Time on the Chem500 *ctd.*

Reserve Time for Chem500

Hamish Grant

Select User... (to make booking on behalf of, refresh page to cancel)

Event Name:

Description:

Date: 13 Jan 2015 Select...

Time: :00 (24h format)

Duration: 15 minutes

Session Requests (emailed to Admin)

OHS Questions: (If applicable)

☐ Radioactive sample ☐ Toxic sample

☐ Biohazard sample ☐ Other hazards to be brought to staff's attention

☐ Require training in safe use of instrument and understanding associated hazards

☐ None of the above

☐ Repeat this entry

☒ Send confirmation email

Send reminder: No Reminder hours in advance

Send to: ☐ granth@unimelb.edu.au

Availability

Sundays	All Day
Mondays	All Day
Tuesdays	All Day
Wednesdays	All Day
Thursdays	All Day
Fridays	All Day
Saturdays	All Day

Reservations available 3 days in advance
Thu January 15, 2015

Chem500 Users

111 users found for Chem500

Select Resource User:

David Keizer	Submit
David Keizer	
Hamish Grant	
Shenggen Yao	
Mengxin Yin	
Shan Sun	
Mohammad Haskali	
Michael Bieri	
Christine Schieber	
Brett Paterson	
James Hickey	
Gojko Buncic	
Andrea North	
David Hayne	
Lilian Hor	

Booking Time on the Chem500 *ctd.*

Reserve Time for Chem500

Hamish Grant

Select User... GRANTH (to make booking on behalf of, refresh page to cancel)

Event Name: Training

Description:

Date: 13 Jan 2015 Select...

Time: 14 : 00 (24h format)

Duration: 15 minutes

OHS Questions: (If applicable)

☐ Radioactive sample

☐ Biohazard sample

☐ Require training in safe use

☐ None of the above

☐ Repeat this entry

☒ Send confirmation email

Send reminder: No Reminder hours in advance

Send to: ☐ granth@unimelb.edu.au

Export this entry (Palm, Outlook, iCal) ☐

Save

Entry in this field is mandatory

Note 24hr format required

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

Optional



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)**



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.



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 11 th March – 11.30am	Tue 11 th 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.





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



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