MANCHESTER 1824
The University of Manchester

Optimising NMR efficiency and automation

Ralph W. Adams

UKMRM, University of Strathclyde, 2017

What is an NMR Lab for?

Routine analysis of samples

Non-routine analysis of samples

Methods development

Teaching

Somewhere to keep cool during the heat of the summer

What does an NMR Lab need?

NMR spectrometers

Users (customers)

Staff

What are their roles?

NMR spectrometers

- Run experiments

Users (customers)

- Come up with problems
- Solve problems

Staff

- Keep Users happy
- Keep NMR spectrometers happy
- Keep the accountants happy

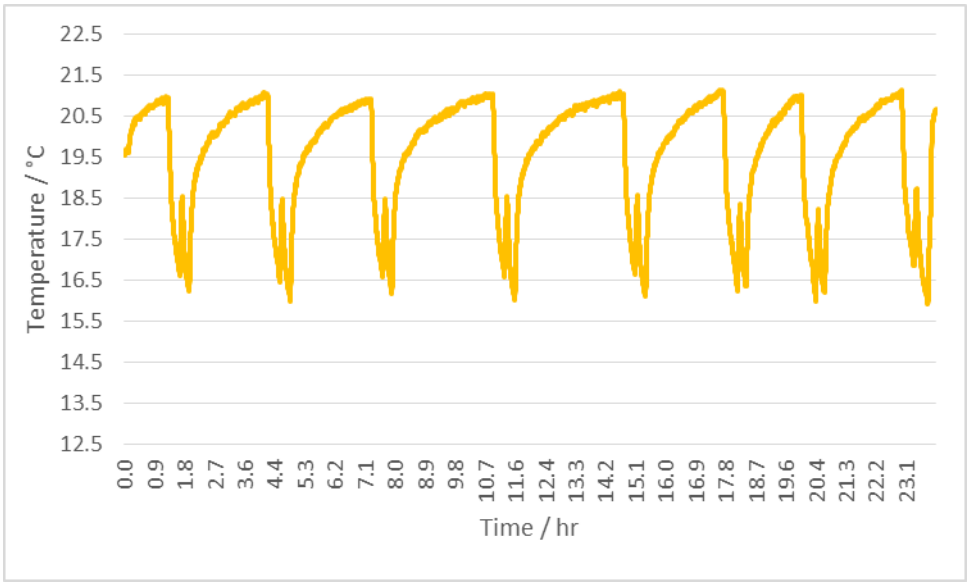
Keeping NMR spectrometers happy

- Magnet
  - Full of cryogens
  - Nicely vacuumed
  - Vibration-free with stable air supply pressure
  - Stable temperature
- Console
  - Cool
  - No dust (nicely vacuumed*)
  - Stable temperature and electrical supply
- (Cold) probe
  - Calibrated
  - (Nicely vacuumed)
  - (Cold)

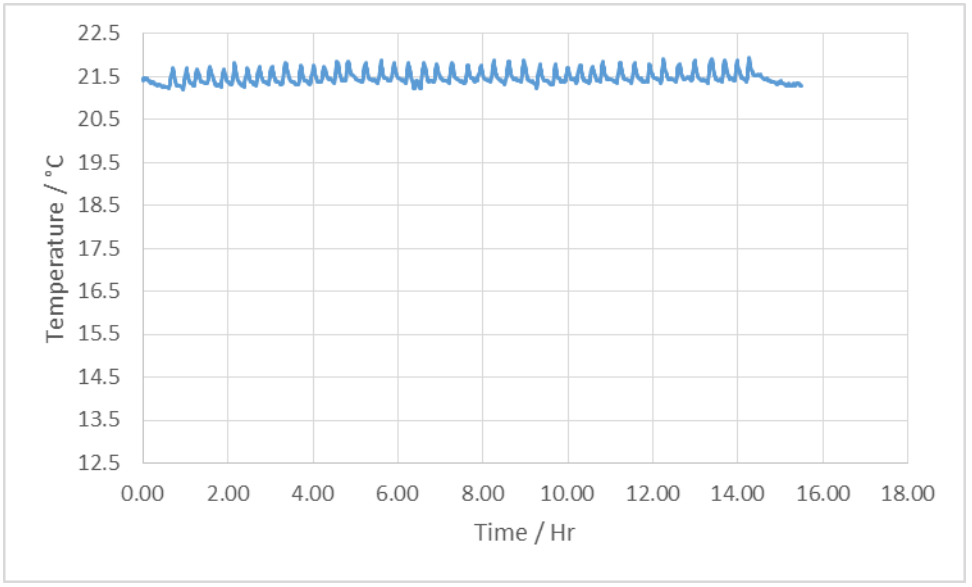
* Use compressed air to remove dust, not a vacuum cleaner

Keeping NMR spectrometers happy

Somewhere to keep cool during the heat of the summer



One air conditioning unit



Two air conditioning units

Console cooling fan failure leads to spectrometer instability

Keeping users happy

Users want

… good, appropriate and sufficient equipment

… good staff

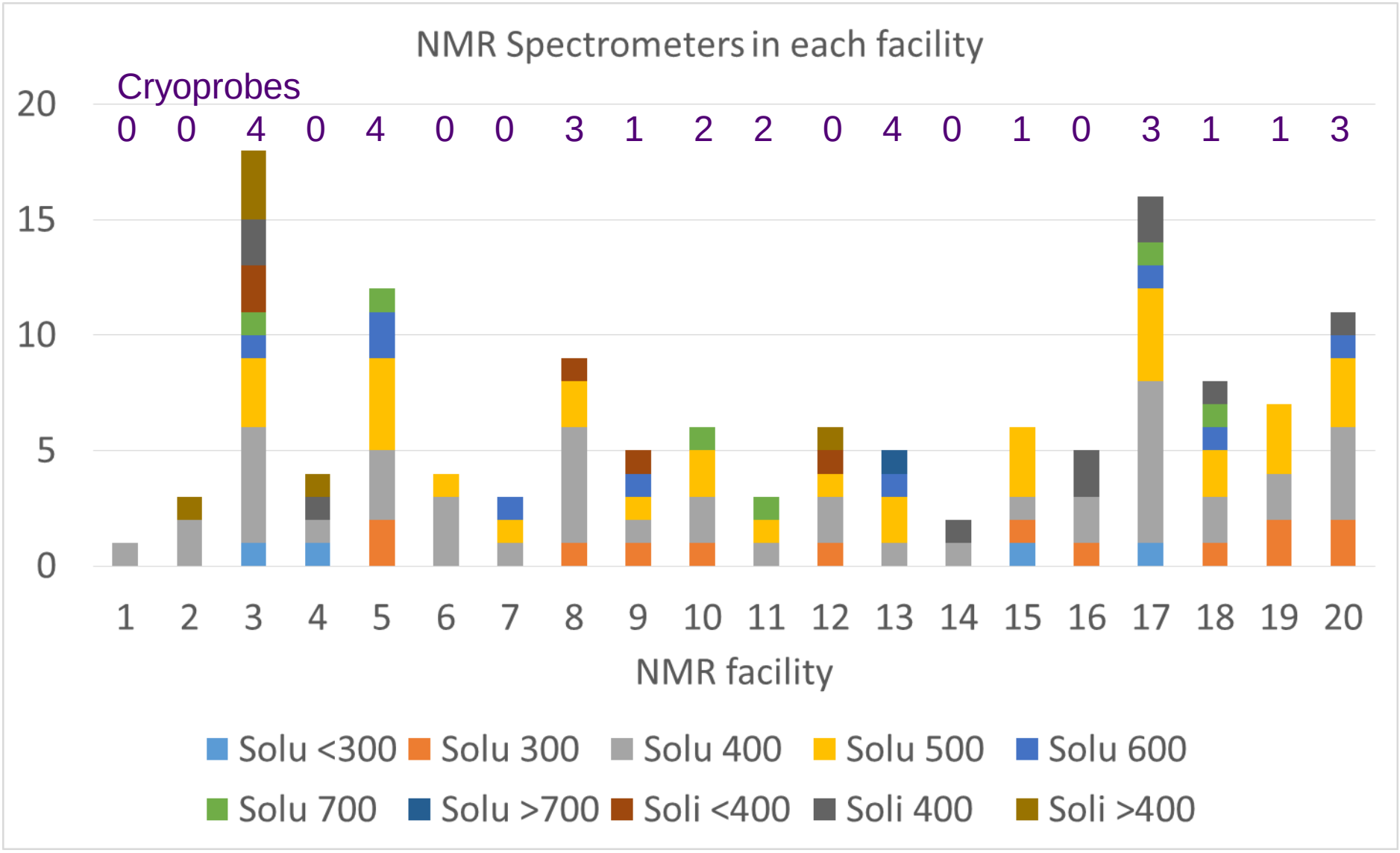
… adequate training

… efficient use of equipment

… to know what's going on

Survey Results – Number of Instruments

What equipment do you have in your facility?



Fills, servicing and maintenance

He fills have to take priority. Evaporation makes it costly to keep a dewar hanging around, and fill/delivery may only be available at set times. Can do fills early to avoid busy periods

N_2 fills can be scheduled around points of peak usage

Maintenance should be done asap.

Servicing can be scheduled for quiet times and done in house

Preventative maintenance?

Survey Results – Facility operation

N_2 fills

10 labs	All fills done on a single day
8 labs	distributed over a week
2 labs	distributed over 2 weeks

He fills

11 labs	In house
9 labs	outsourced

User logins

8 labs	One for each user (incl. NMR kiosk/NOMAD)
6 labs	One for each research group
6 labs	One login

Keeping users happy

Users want

… good, appropriate and sufficient equipment

… **good staff**

… adequate training

… efficient use of equipment

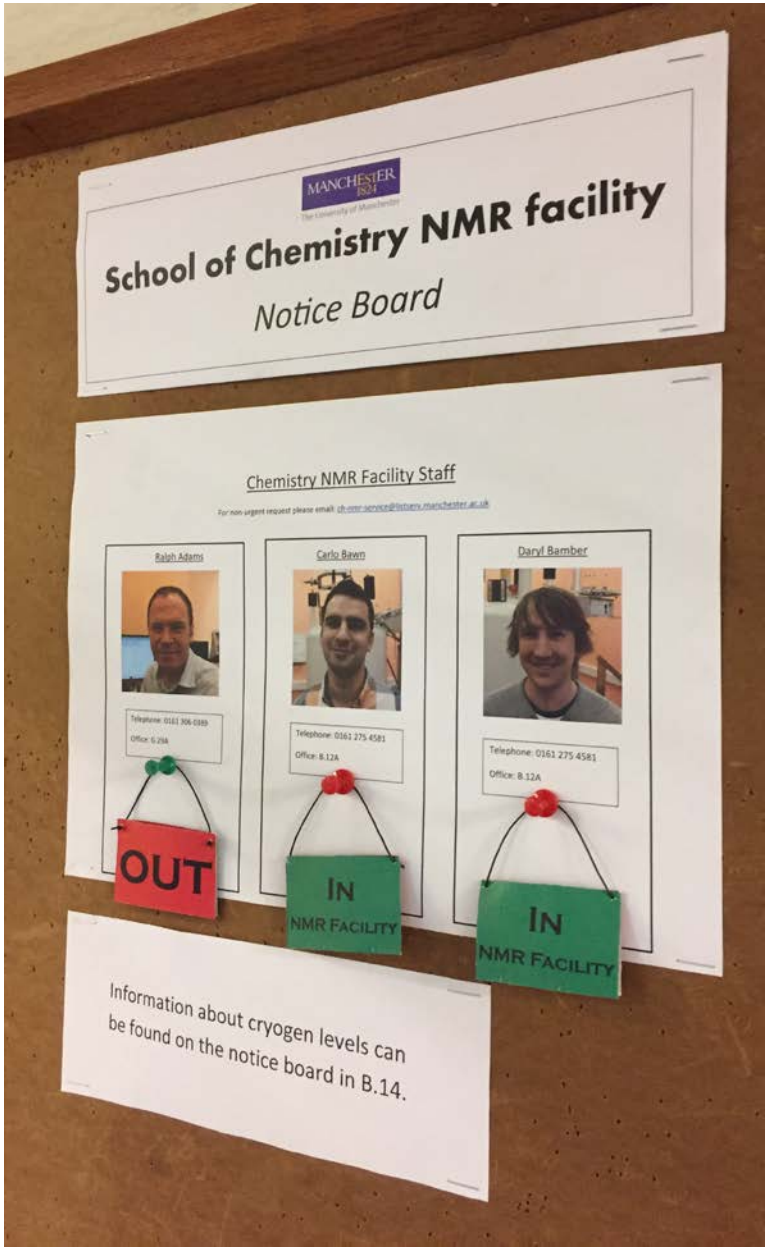
… to know what's going on

Keeping users happy

Staff attributes

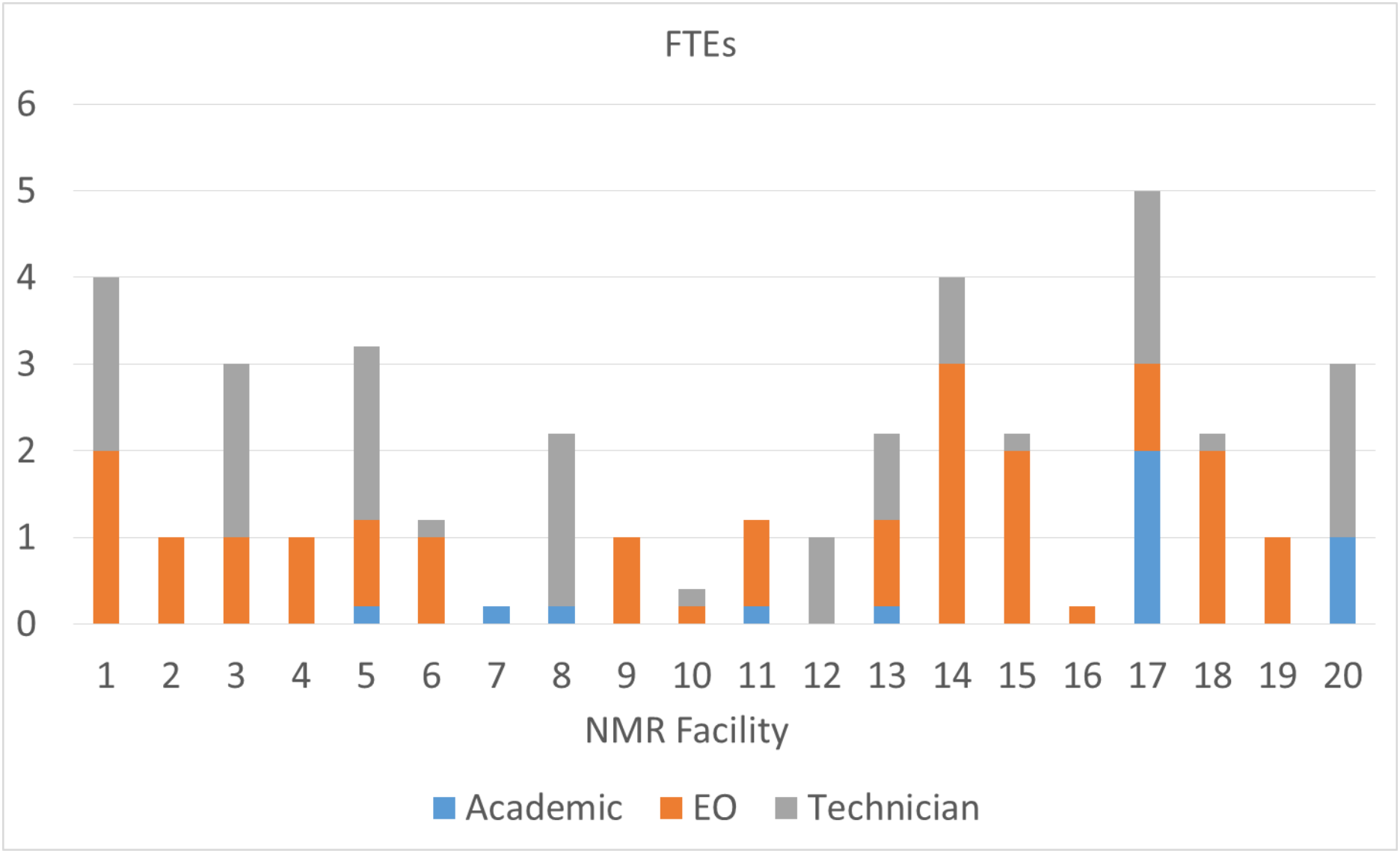
Approachable?
Friendly?
Helpful?
Available?
Responsive?
Skilful?
Knowledgeable?

Publications?



Survey Results – Number of Staff

How many people work in your facility?



Keeping users happy

Users want

… good, appropriate and sufficient equipment

… good staff

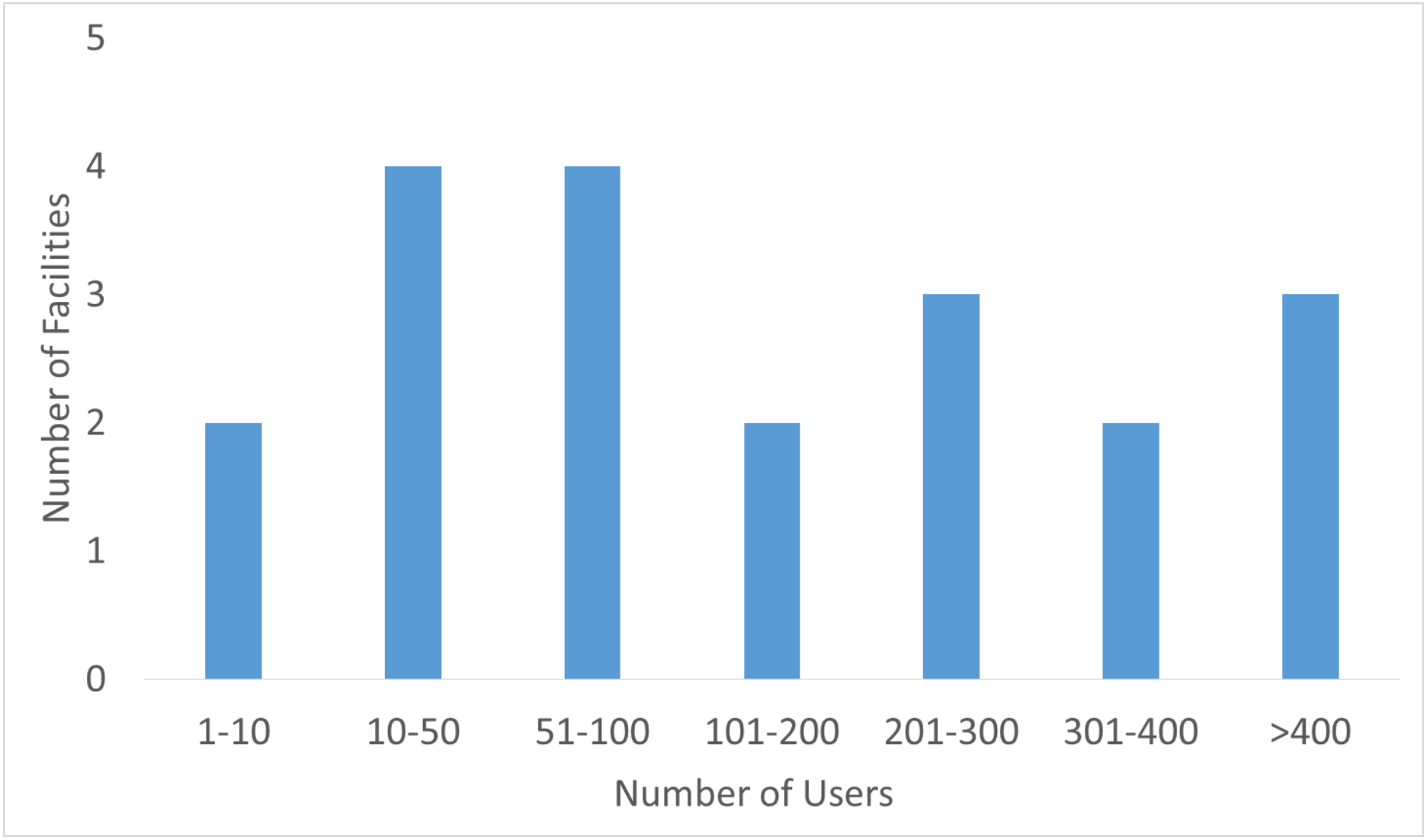
… **adequate training**

… efficient use of equipment

… to know what's going on

Survey Results – Number of Users

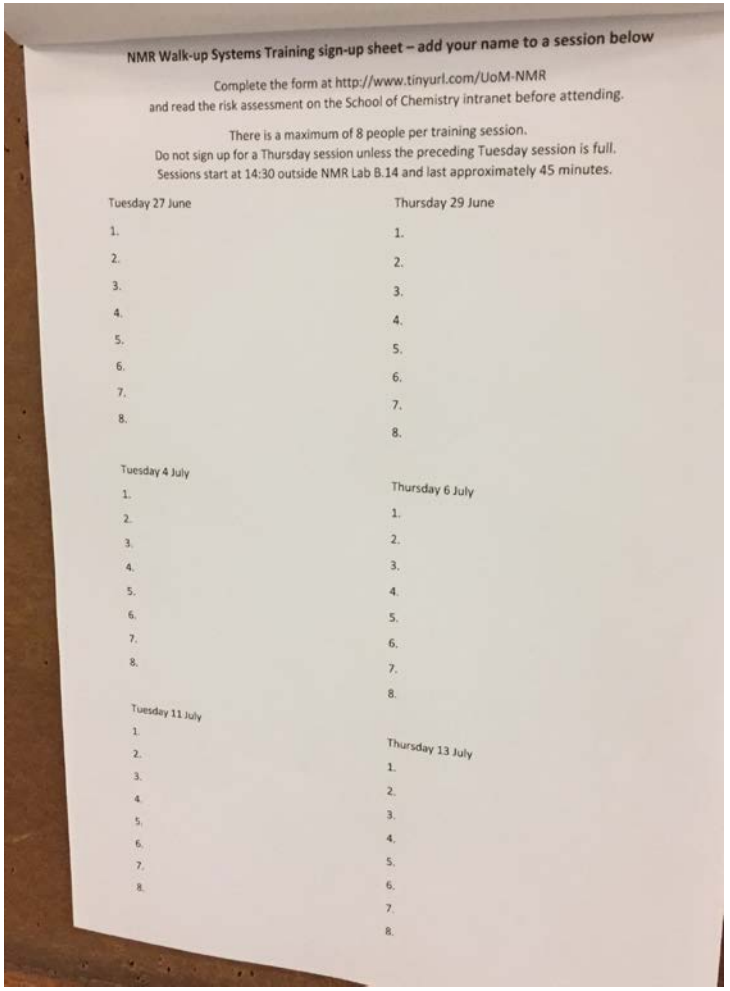
How many people use your NMR facility?



Adequate training

Sample preparation
Use of walk-up systems
Hands-on use of system
Health and safety
Rules and guidelines
Data processing
Interpretation

Training sessions
Standard operating procedures
Website
Operating manual
Lectures and Seminars
Books



NMR Walk-up Systems Training sign-up sheet – add your name to a session below

Complete the form at http://www.tinyurl.com/UoM-NMR
and read the risk assessment on the School of Chemistry intranet before attending.

There is a maximum of 8 people per training session.
Do not sign up for a Thursday session unless the preceding Tuesday session is full.
Sessions start at 14:30 outside NMR Lab B.14 and last approximately 45 minutes.

Tuesday 27 June
1.
2.
3.
4.
5.
6.
7.
8.

Thursday 29 June
1.
2.
3.
4.
5.
6.
7.
8.

Tuesday 4 July
1.
2.
3.
4.
5.
6.
7.
8.

Thursday 6 July
1.
2.
3.
4.
5.
6.
7.
8.

Tuesday 11 July
1.
2.
3.
4.
5.
6.
7.
8.

Thursday 13 July
1.
2.
3.
4.
5.
6.
7.
8.

Survey Results – What training is available to users?

Majority of labs:

Regular small group training for walk-up (45 mins – 1 hr)
1:1 hands on training (2 hr – 1 day)

Some labs:

Standard Operating Procedures
Competency exercises
NMR lecture course (2 day course every 2 years or 4 hours a week)

Performed by EO/technicians?

Risk assessments?

Survey Results – Facility operation

Third-party training

User courses

Online demos, webinars and walk-throughs

Processing Software

	ACD/Labs	Topspin	VnmrJ	Delta	Mestrenova	DOSY Toolbox
Individual	4	4	4	4	7	4
Facility		5			1	
Site	4	5			6	
Installed on Clusters		3			1	

Keeping users happy

Users want

… good, appropriate and sufficient equipment

… good staff

… adequate training

… **efficient use of equipment**

… to know what's going on

Efficient use of spectrometer time

Distribute usage across all instruments

Use NUS (COSY, HSQC, HMBC), 25% or 50%?

Multiplicity edited HSQC vs DEPT135+HSQC

How quantitative does it really need to be?

Remove some experiments from low sensitivity instruments (e.g. $^{13}C\{^{1}H\}$ from those that do not have cold probes)

Reduce overheads

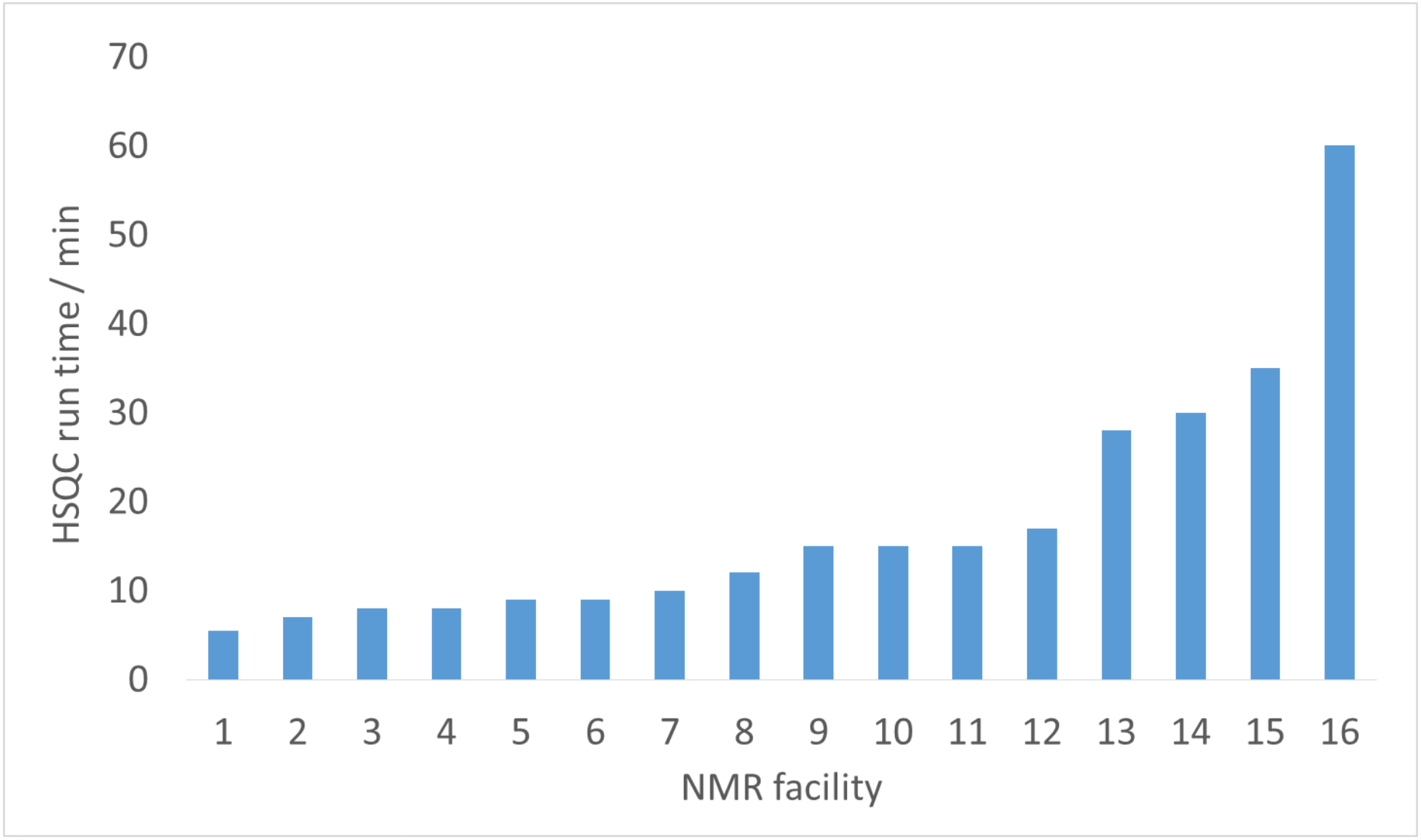
Survey Results – ^{1}H experiment time

How long does it take to run a single scan ^{1}H spectrum (full cycle from loading the sample to loading the next sample - including load, lock, tune, experiment, and shim) on a $CDCl_3$ sample on your automated instruments?

Agilent 7620	2 min
Zymark	3 min
SampleXpress	3 – 5 min*
BACS-60	4 – 7 min
SampleXpress Lite	4 min
SampleCase	4 min
Jeol	6 – 10 min
SampleXpress on Fourier300	6 – 10 min

Survey Results – HSQC experiment time

How long does it take to run the default ^{1}H-^{13}C HSQC on your automated instruments?



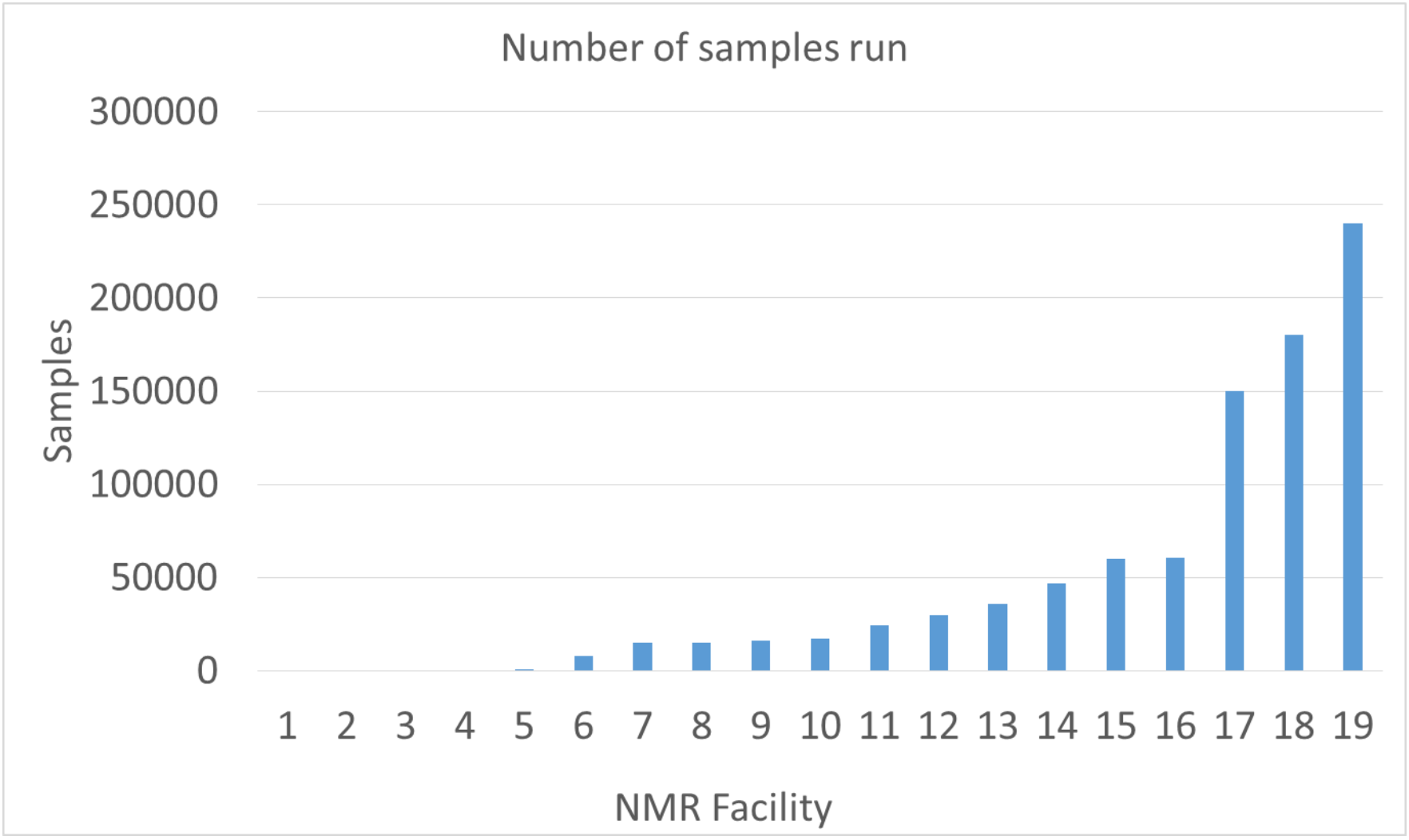
Survey Results – Facility operation

NUS

11 labs	No
5 labs	Occasionally
4 labs	Yes, in all/most places it can be used

Survey Results – Facility information

How many samples are run in your facility?



Data Distribution

9 labs	IT services data server
14 labs	Local data server
8 labs	Email copies of spectra
4 labs	Email raw data
4 labs	Paper copies
1 lab	USB

Survey Results – Facility operation

Variable temperature experiments

8 labs VT done in automation where possible

All labs allow manual VT

Night Queues

Start times range range from 16:30 to 22:00

End times range from 08:00 to 09:30

Keeping users happy

Users want

… good, appropriate and sufficient equipment

… good staff

… adequate training

… efficient use of equipment

… to know what's going on

Signage

… for health and safety compliance

… to be completely ignored?

Communication

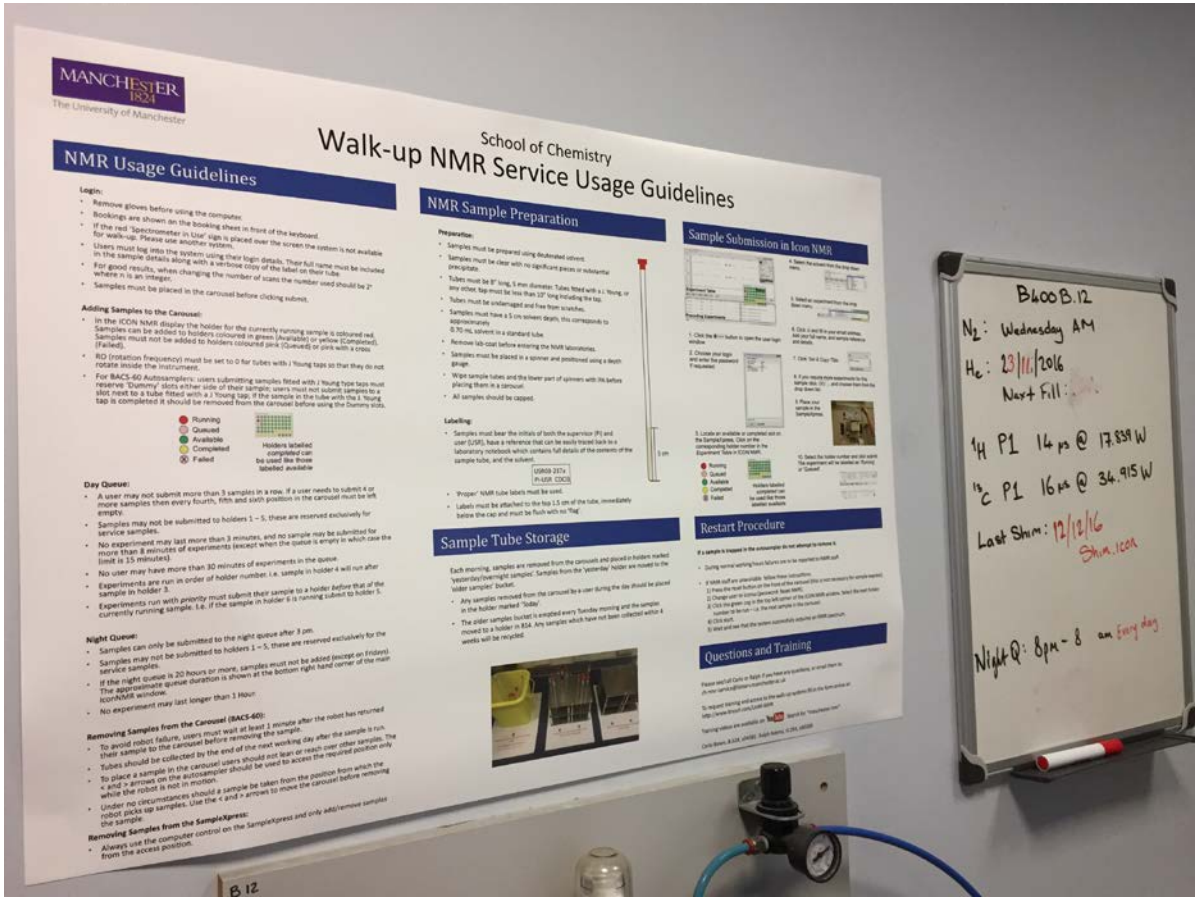
Posters

… to provide useful information

… to explain usage rules

… to make the facility less drab

… to be completely ignored?

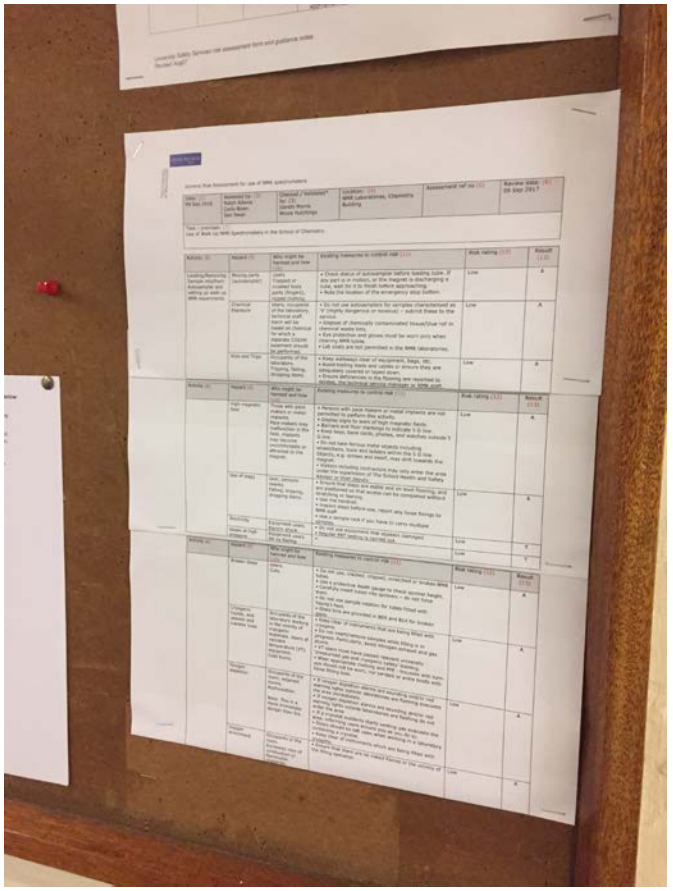


Notice board

… to provide useful information (in real time)

… to display risk assessments and SOPs for emergencies

… to be somewhat ignored?



Communication

Single NMR facility /service email address

… so that there is always someone available to answer straight away

Biannual users' meetings

… so that there is a forum to raise issues and make changes to the operating rules

… with a representative from each research group

Communication

Websites

… for general information
… for instrument booking
… to make service requests and submit queries
… to get real-time status information about the walk-up facility

email list

… for formal communication with entire user-base
… to warn and update users about instrument maintenance and failures

Twitter

… to add status information to the display screen

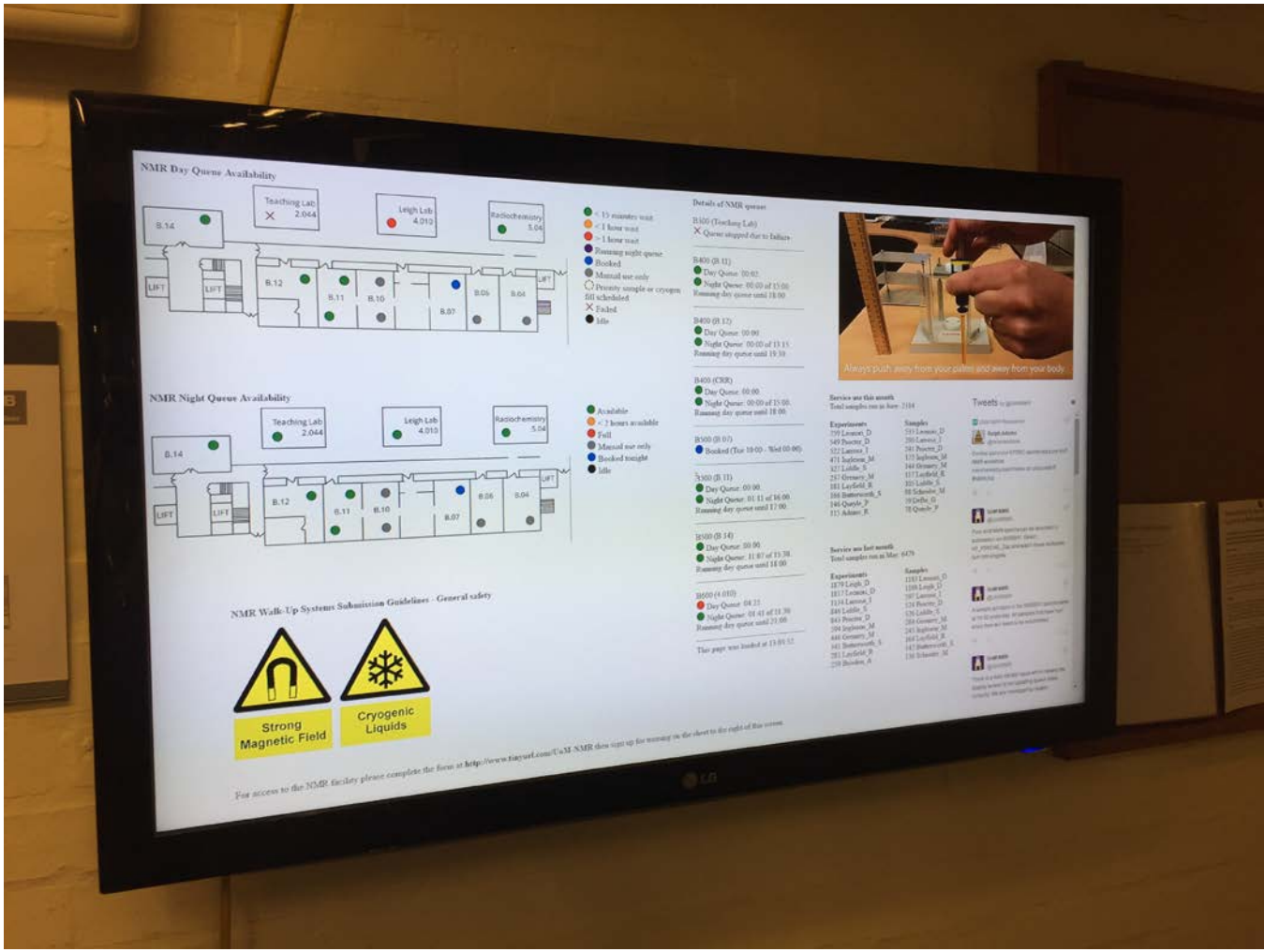
Communication

Display screen

… to provide useful information (in real time)

… to show usage statistic

… to be rarely ignored



Survey Results – Facility operation

Left over tubes each year

4 labs	< 10
6 labs	11 - 100
3 labs	101 - 300
3 labs	301 - 1000
2 labs	> 1000

Survey Results – Facility operation

Rules

13 labs	Rigorous enforcement
7 labs	Not strongly enforced
3 labs	Not enforced

Typical sanctions

Talk to/email supervisor
1 week ban for minor infractions
Delete peoples experiments/remove samples
Additional charges for repair costs / lost time
Talk to them / tell them off
1 – 3 month ban for serious cases

”Unfortunately not allowed to maim the users, but access may be restricted to working hours or in serious cases banned for 1-3 months.”

Workshop on Pure Shift NMR

12th September, 2017 • School of Chemistry, University of Manchester

Preliminary Programme

Introduction and historical background - *Gareth Morris*

Interferogram and real-time acquisition - *Peter Kiraly*

Zangger-Sterk and band-selective methods - *Laura Castañar*

---------- **Lunch and poster session** ----------

PSYCHE - *Mohammadali Foroozandeh*

Related methods - *Ralph Adams*

Practical implementation – *Mathias Nilsson*

Question and Answer session

MANCHESTER 1824
The University of Manchester

Please register using the online form at
http://nmr.chemistry.manchester.ac.uk/pureshift by **Friday 18th August**

EPSRC
Pioneering research and skills

MANCHESTER 1824
The University of Manchester

Optimising NMR efficiency and automation

Ralph W. Adams

UKMRM, University of Strathclyde, 2017