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**SPECIFICATION FOR**

**THE PROVISION OF NMR**

**SPECTROMETER**

**UK SBS PR19047**



## Specification Document – Supplies & Services

<b>Title of Request:</b>	<b>UK SBS PR19047 NMR SPECTROMETER</b>
<b>Required Delivery Date:</b>	6 <sup>th</sup> March 2020
<b>Required Commissioning Date:</b>	Two weeks from delivery– No later than end of March 2020

### 1. Introduction

- 1.1. The ISIS Deuteration Facility supports ISIS Users experiments by providing deuterated compounds for use within beamline experiments.
- 1.2. The group is responsible for the synthesis of simple and more often large complex compounds. Part of this, we have a requirement to analyse the samples produced before handing over to the users.
- 1.3. The type of samples that we produce range from simple perdeuterated fatty acids to complex surfactants where we require to determine where the deuterium has been incorporated.
- 1.4. With the group expanding and an increase in demand for more complex molecules, we require our own instrument to carry out this analysis. Most of the samples we produce are perdeuterated, and therefore we will require a dedicated  $^2\text{H}$  channel.
- 1.5. Furthermore, with the growth of the group, more complex mixtures are needed to be analysed particularly using  $^1\text{H}$ ,  $^2\text{H}$  and  $^{13}\text{C}$  spectroscopy. In particular the use of  $^{13}\text{C}$  with  $^1\text{H}$  and  $^2\text{H}$  decoupled allowing for simpler spectra for the users to interpret.
- 1.6. We are currently at around 7 users, running at least 5 samples each per day, with a minimum of 3 experiments per sample. This results in approximately 85 experiments per day. However this is fluctuating based on summer student projects and/or visiting researchers also requiring the use of the spectrometer, where during these times we expect the use to increase further.
- 1.7. This broad range of users will require the spectrometer to be run in several different modes of software interface i.e. users vs superusers.

### 2. Aims & Objectives

- 2.1. We require a dedicated NMR spectrometer from a company with proven technology.
- 2.2. The spectrometer should meet the brief and fulfil the minimal requirements set out below.
- 2.3. The spectrometer would be located within the Deuteration Facility, for the sole use by the Deuteration team.
- 2.4. It is required that the instrument is delivered, installed and commissioned by the 6th March 2020.

### 3. Background to the Requirement

- 3.1. The Deuteration Facility has recently undergone a refurbishment and as part of this the size of the facility has been expanded to include an annex for analytical equipment.
- 3.2. Refurbishment of this annex has taken into account the requirement to locate a NMR spectrometer in this room, however, prior to UKRI awarding a contract we would require a site visit to ensure that this annex is completely capable of housing the spectrometer. Floor plans for the annex and photos indicating the location for the spectrometer are to be included along with this document (***Annex floorplan.pdf, Spectrometer location 1.jpeg, Spectrometer location 2.jpeg***)
- 3.3. With team expanding, the background of the team is very different and understanding of NMR varies, from complex understanding of the fundamentals of NMR spectroscopy and being to fully operate the spectrometer manually all the way down to only understanding how to load a sample and interpret the spectra. It is therefore important that we can set the spectrometer to allow it be used to its full potential for some users (i.e. superusers) and also only allow a very limited amount of flexibility for others (i.e. fully automated users). We are also expecting the current number of users to rise. This will be due to undergraduate students who will undertake projects within the facility will also require access to the instrument, along with visiting researchers, who may require the ability to load pulse sequences and experiments, similar to those that they use at their own institutes to allow easy data comparison.
- 3.4. The spectrometer will be managed in house with no additional technical support staff. We therefore require spectrometer maintenance to take no longer than one hour per week.
- 3.5. Due to the nature of our work we normally synthesis complex molecules, with spectra that require long times to acquire and interpret. We require a technique that can simplify this process for us.  $^{13}\text{C}$  NMR spectra is usually acquired  $^1\text{H}$  decoupled resulting in simpler spectra for the user to interpret. However,  $^2\text{H}$  couplings are still observed and therefore require a longer acquisition time and usually result in complex spectra to interpret. The ability to run the same experiment with  $^2\text{H}$  decoupled at the same time is a requirement.
- 3.6. We will require a spectrometer that is can analyse a wide range of compounds from simple deuterated fatty carboxylic acids to small deuterated proteins, without having to manually change aspect of the spectrometer. This includes changing the probe or any other hardware manipulations.
- 3.7. The Facility also routinely batch produces several key intermediates. Quality control is vital during our deuteration reactions. We require each batch to reach a final deuterium incorporation level of a minimum of 95%. Currently we monitor this by the addition of an internal standard to a known concentration of our product and calculate the amount of deuterium by reduction in  $^1\text{H}$  resonances in a  $^1\text{H}$  NMR spectrum. We require the ability to determine the level of deuterium incorporation for within a compound as an automated process.

## 4. Scope

- 4.1. We require a fully automated 3-channel system 400 MHz NMR spectrometer with a dedicated  $^2\text{H}$  amplifier
- 4.2. The spectrometer will be housed in the Deuteration facility for sole use by the Deuteration team to analyse samples produced for our Users.
- 4.3. We require the spectrometer to be delivered with all the required equipment to operate such as a computer suitable for running the spectrometer, spinners for holding samples and an autosampler for fully automation of the spectrometer
- 4.4. The NMR Spectrometer must be delivered by Friday 6<sup>th</sup> March 2020 and installation and commissioning to be completed within 2 weeks of delivery
- 4.5. We require training to be provided on 2 levels, i.e. Superuser training for those responsible for maintaining and training further users and User training for current group meetings detailing how to run simple experiments

## 5. Requirement

- 5.1. Fully automated 400 MHz NMR spectrometer with a 3-channel system with one of these to include a dedicated  $^2\text{H}$  amplifier to allow for the running of  $^{13}\text{C}$  experiments with both  $^1\text{H}$  and  $^2\text{H}$  decoupling.
- 5.2. The system must be capable of observing the following nuclei (but not limited to) with sensitivities greater or equal to those listed:
  - 5.2.1.  $^1\text{H}$  ( $\geq 550:1$ ),  $^2\text{H}$ ,  $^{10}\text{B}$ ,  $^{13}\text{C}$  ( $\geq 210:1$  and  $\geq 230:1$  ( $\{^1\text{H}\}$ )),  $^{19}\text{F}$  ( $\geq 500:1$ ),  $^{31}\text{P}$  ( $\geq 200:1$ ),  $^{77}\text{Se}$ ,  $^{125}\text{Te}$ .
- 5.3. The following experiments will be able to run as standard:  
 $^1\text{H}$ ,  $^2\text{H}$ ,  $^{13}\text{C}\{^1\text{H}\}$ ,  $^{13}\text{C}\{^2\text{H}\}$ ,  $^{13}\text{C}\{^1\text{H},^2\text{H}\}$ ,  $^{31}\text{P}$  (plus various decoupled experiments), DEPT-45, DEPT-90, DEPT-135 COSY, HMBC, HSQC, NOESY, solvent suppression experiments. These experiments should involve no more than the user loading a sample onto the spectrometer and selecting the desired experiment from a dropdown list.
- 5.4. An auto sampler should be included to allow for full automation.
  - 5.4.1. We require a fully automated autosampler, loading from a waist height with an capability of queuing a minimum of 12 samples and a maximum of 24. The auto sampler must also be capable the ability to load Young's taps tubes.
- 5.5. We require the ability to carry variable temperature NMR.

5.5.1. Temperature range from -40 °C up to 120 °C, ideally without the need to use different spinners.

5.5.2. We will also require information on how cooling and heating is achieved and how long it requires for the sample and spectrometer to stabilise at each temperature.

5.6. Suppliers should also give information on average liquid helium and liquid nitrogen consumption per year.

### 5.7. Test Samples

Four test samples will be issued, the samples will all be supplied from the same batch and will be screened before being sent. Request for test samples must be made to UKRI as soon as possible following Bidder registration on Emptoris. Requests must be submitted via the Emptoris e-Sourcing messaging system and clearly state your full organisation details, address, contact name etc.

A cut off date to request samples is 09/08/2019. Data should be compiled and returned as part of any Bid. The Contracting Authority expects to receive processed spectra for all experiments run along with the raw data files.

The Contracting Authority will require the following spectra to be run on these samples to determine the suitability of the spectrometer:

$^1\text{H}$ ,  $^2\text{H}$ ,  $^{13}\text{C}\{^1\text{H}\}$ ,  $^{13}\text{C}\{^2\text{H}\}$ ,  $^{13}\text{C}\{^1\text{H}, ^2\text{H}\}$  and any other spectrum that the supplier deems necessary

The samples will be detailed below and the results that we require from the test outlined:

#### 5.7.1. Sample 1

Partially deuterated Dodecanoic acid produced by Procedure 1 (Appendix 1)

Data that we require from this is:

Identify any impurities in the sample along with purity calculation for the target compound  
Calculation of deuterium incorporation levels (%D)

During each of these measurements, we will require the whole process from making up the sample through to data collection and deuterium incorporation calculations to be filmed in real time. All of these recordings will include showing the PC screen to allow determination ease of software use.

#### 5.7.2. Sample 2

Partially deuterated Methyl glycoside

Data that we require from this is:

Identify any impurities in the sample along with purity calculation for the target compound  
Calculation of deuterium incorporation levels (%D)

### 5.7.3. Sample 3

Alkyl chain deuterated Dodecyl triethylene glycol ether (dC12hE3)

Data that we require from this is:

Identify any impurities in the sample along with purity calculation for the target compound  
Calculation of deuterium incorporation levels (%D)

Any spectra that you feel we help with structural determination/purity/physical properties of the compounds supplied should also be included.

### 5.8. Training

Onsite training to be carried out for a minimum of two superusers within 2 weeks of commissioning the instrument.

A minimum of 2 places on UK based training course for new operators is required to cover the following areas:

- training for standard high-resolution NMR spectrometers,
- instrument operation for typical 1D and 2D NMR experiments,
- automation operation and
- spectrometer maintenance

We require full support for designing, uploading and processing of data for complex experiments such as 3D or 4D experiments should we require it.

Further training can be included along with prices in a separate document in the Pricing Schedule.

### 5.9. Computer(IT)

5.9.1. It is essential that the IT hardware offered to Contracting Authority by the tendering organisation shall incorporate a workstation grade Computer Hardware System capable of achieving Full Operational Functionality of the proposed instrument. The expected specification should meet or exceed the following example as a minimum:

Intel Xeon or AMD Ryzen or equivalent workstation grade processor (minimum 8-core);  
AMD FirePro or Nvidia Quadro or equivalent workstation grade graphics card (minimum 4GB DDR5 RAM);  
2TB SSD Storage (in RAID configuration if multiple drives);  
32GB DDR4 RAM;  
30" 3840x2160 (4K) LED-LCD monitor;  
1 GBit Ethernet.

5.9.2. It is essential that the proposed IT hardware will have sufficient processing speed, system memory & data storage capacity to allow the proposed system to operate at full functionality for a minimum of 2 (two) Years without either data acquisition or data processing being constrained in any way.

5.9.3. The proposed system shall be supplied with a comprehensive software package facilitating full operational functionality. It is expected that this software shall, as far as possible, be intuitive to the user for basic operation and therefore not require extensive training to be provided. The software shall come equipped with an extensive help menu explaining the function of all aspects of the software.

5.9.4. There must be provision for a minimum of 10 licenses for processing of data away from the attached PC and site network.

5.9.5. Software updates, both major and minor versions, should be included for free.

## **5.10. Support**

5.10.1 Must include at least 2 years warranty, including any preventative maintenance services required.

5.10.2 To avoid premature obsolescence, replacement and spare parts for components used in the proposed system shall remain available for a minimum period of 2 (two) Years from the date of their last manufacture.

5.10.3 The Contracting Authority shall expect a Maximum On-Site Attendance Time of 2 (two) working days from first notification of a problem with the proposed instrument.

5.10.4 The Contracting Authority shall expect a Maximum On-site Fix Time of 5 (five) working Days from first diagnosis of a problem with the proposed instrument.

5.10.5 The Contracting Authority shall expect that during the Warranty Period, should it be necessary for one, or more, major Components of the proposed System to be repaired away from the Contracting Authority's premises that Appropriate Replacement Equipment and/or a Demo System will be provided on Free Loan to Contracting Authority for the period of repair.

## 6. Timetable

### 6.1. Delivery, Installation and Commissioning:

Delivery of the instrument must be made by Friday 6<sup>th</sup> March 2020

Delivery of the instrument will be made to:

**Dr James Tellam**  
**R55, Cryogenic Extention, 2<sup>nd</sup> Floor,**  
**STFC Rutherford Appleton Laboratory**  
**Harwell Campus, Didcot, OX11 0QX**

### 6.2. Payment terms are defined as follows:

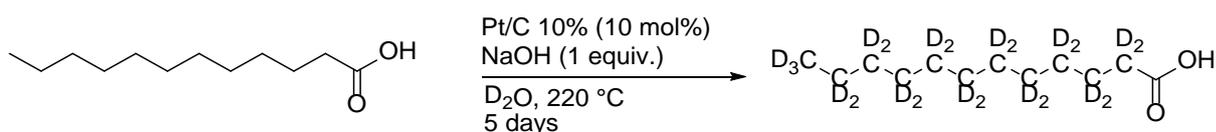
95% upon delivery

5% on commissioning and final user acceptance.

### 6.3. Installation and commissioning plan to be proposed and must be completed within 2 weeks from delivery.

## 7. Appendix

### 7.1. Procedure 1



All materials are combined and heated in the 600ml Parr reactor for 5 days. The reactor was cooled to RT. The solution was acidified with HCl, filtered and extracted with diethyl ether. The organic phase was dried over MgSO<sub>4</sub>, filtered and concentrated in vacuo to afford a white solid.