

## Use of the BRUKER SampleJet Autosampler

### 1. Overview

The SampleJet autosampler is a high capacity, high throughput sample robot system, with 5 temperature-controlled racks of 96 samples (short 4-inch NMR tubes) and 99 samples (4- to 7-inch length) on the outer ring of the rotating tray. This handout describes the general use of the SampleJet however it is not a comprehensive description of all functions of the autosampler.

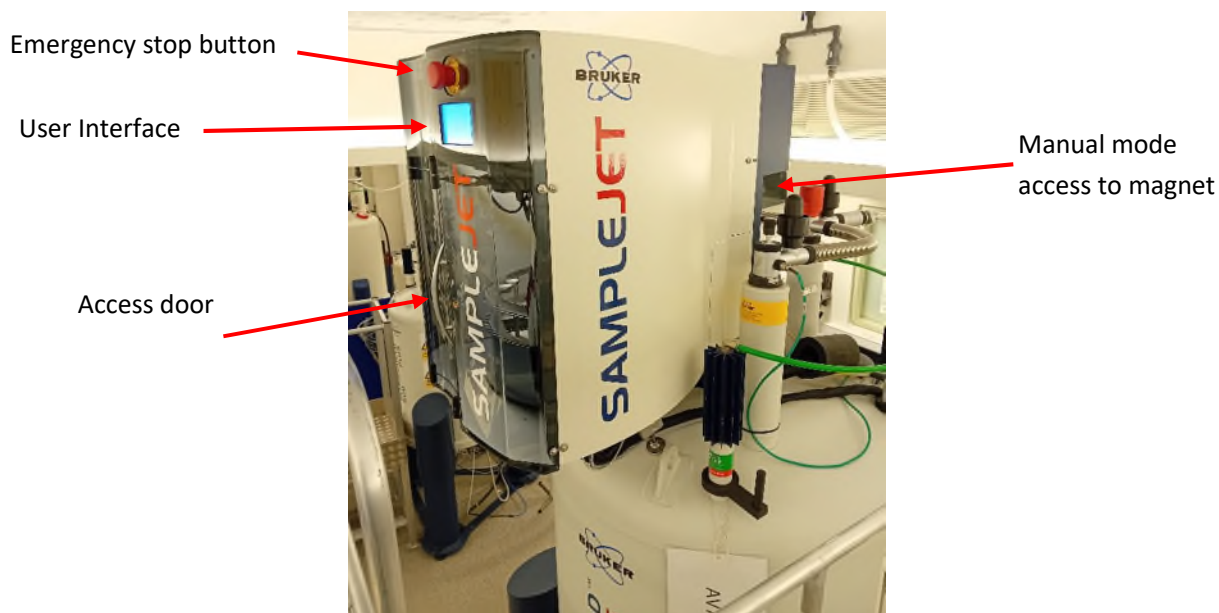







Figure 1: SampleJet side view

### **WARNING**

- **Do not use the SampleJet if you have not been trained by a member of the NMR staff.**
- A flashing light indicates the access door or the black plastic door covering the manual mode access to the magnet is not properly closed.
- Do not reach deep into the SampleJet. This can cause the SampleJet to error, and potentially injure yourself or damage the SampleJet.
- Never interrupt the 'busy' state of the SampleJet. Wait until the operation is finished and the state is idle before loading/unloading samples, and starting new actions from the display.
- Do not force the SampleJet
  - If a sample/rack feels stuck, do not apply force to remove it. This may lead to damage to the autosampler or sample destruction.
- Keep the doors closed by default
  - The SampleJet will be much faster due to safety checks and code readings that can be skipped.

## 2. SampleJet Terms and Hardware

	<p><b>Caps</b> Every tube transported using the SampleJet needs a special cap with a barcode. The SampleJet will hold the tube by this cap when transported. Green and purple caps are for 3mm and 5mm tubes, respectively.</p> <p>If an incorrect cap is used or the cap is covered, the SampleJet will not recognise the tube and will not transport it.</p>
	<p><b>Balls</b> Coloured balls are to close open caps and to mark caps. Once inserted, these cannot be removed.</p>
	<p><b>Shuttles</b> Almost all sample will be transferred into a shuttle, rather than a spinner, before being transferred to the magnet. A shuttle should never be removed from the SampleJet or moved to a different position.</p> <p>The AV700 is currently set up with two shuttles for 3mm (green) and 5mm (purple) tubes.</p>
	<p><b>Racks</b> The SampleJet is set up to hold 5 racks of 96 4-inch NMR tubes. These are placed in the centre of the carousel and are labelled 1 to 5.</p>
	<p><b>Tube Storage</b> OpenShop storage: These are sample tubes stored in the round, outer ring. There are 99 samples in total; 99 without spinners and 3 with spinners. See Figure 5. (In the centre, there are 5 slots for racks with 96 4-inch tubes each.)</p>
	<p><b>Operating Modes</b> The SampleJet is set up for two operating modes; 'automatic' and 'manual'. 'Automatic' mode uses the SampleJet to transport samples whilst 'manual' bypasses the SampleJet entirely.</p>
	<p><b>States</b> Idle: SampleJet has no current commands. Busy: Transporting a sample. Manual: SampleJet is in manual sample mode. Error: System is in an error mode and cannot be used.</p>

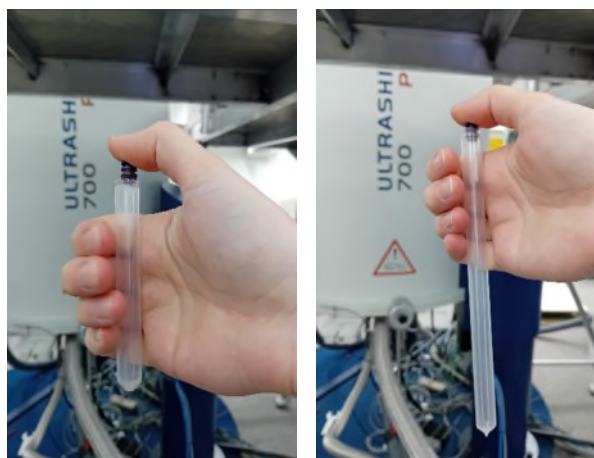
### 3. Sample Preparation

**WARNING:** For OpenShop samples, tubes must not be longer than 7 inches. For rack samples, tubes must not be longer than 4 inches.

**WARNING:** The caps need to hold properly onto the tube. The caps are also consumables and should only be used once. If caps are used over again, they might not sit tight enough on the tubes. If the cap does not hold onto the sample with sufficient force, the sample could be lost during transportation in the SampleJet.

#### 3.1. Fitting Bruker caps onto NMR Tubes

1. Place an unused cap without force onto the NMR tube and insert it into the appropriate safety tube as shown.
2. Press with the thumb on the cap until a sharp click is heard. The cap **must** click into place. If the cap does not click, it must not be used and has to be disposed of.



**Figure 2:** Procedure of fitting SampleJet NMR caps onto NMR tubes using the safety tube for a (left) 5mm 4-inch and (right) 5mm 7-inch tube.

#### 3.2. Cleaning of NMR tubes

If NMR tubes are not completely clean on the outside, they will contaminate the SampleJet hardware as well as the NMR system. Clean all tubes before loading them into the SampleJet and avoid touching the tube once cleaned.

#### 3.3. Writing on NMR tubes

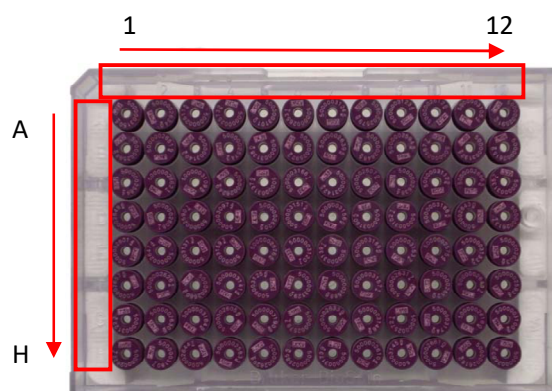
Labelling is only allowed on 7-inch OpenShop tubes. Writing should only be present in the top 6 mm -36 mm below the NMR cap. Labelling below this region will interfere with the shuttles ability to clamp the tube. Labelling should not be in the top 6 mm so the tube can hang freely on the OpenShop metal grate.

#### 4. Numbering of Sample Holders

If you are unsure the number of a sample position, open IconNMR and all sample positions will be detailed alongside their sample rack positions (Figure 3).

Experiment Table		
Holder	Type	Status
▷ 1 E12 - 193	U	Available
▷ 1 F12 - 194	U	Available
▷ 1 G12 - 195	U	Available
▷ 1 H12 - 196	U	Available
▷ 2 A1 - 201	U	Available
▷ 2 B1 - 202	U	Available

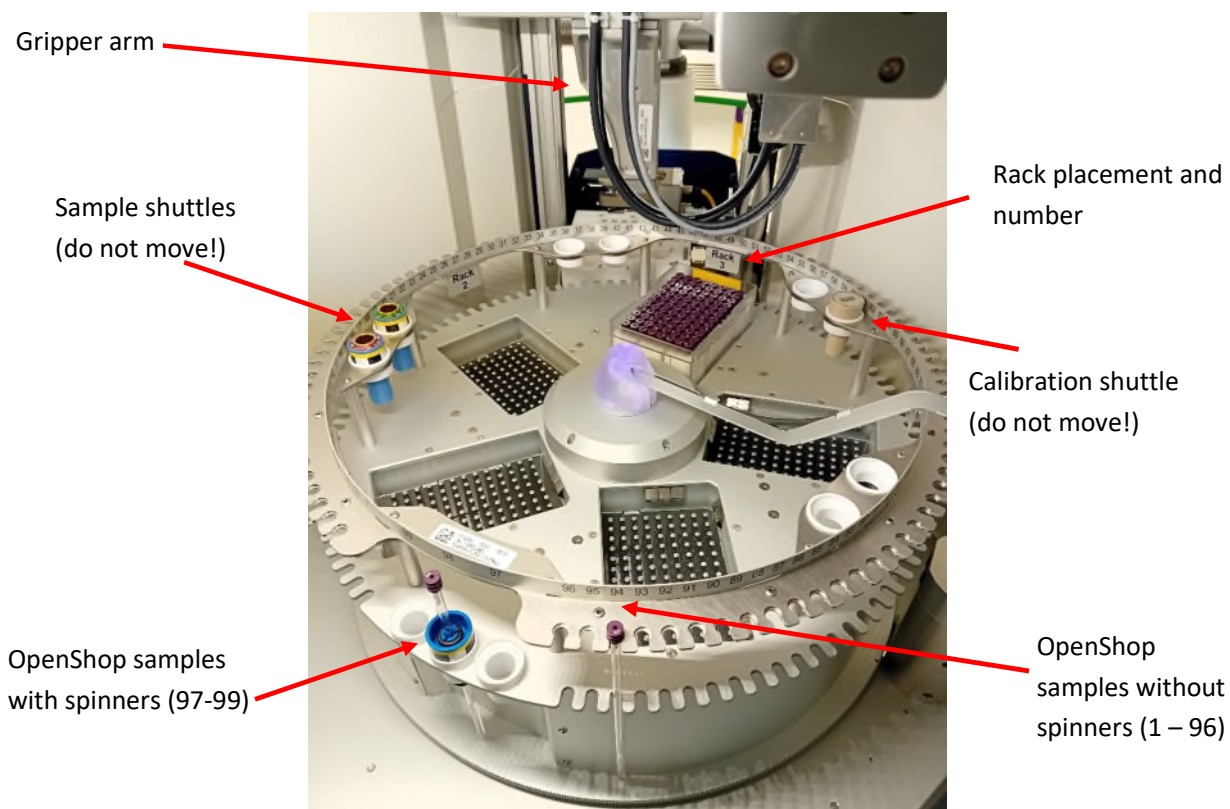
**Figure 3:** IconNMR set up for the SampleJet. For Holder '1 E12 – 193', '1' denotes the rack number, 'E12' the position within the rack, and '193' the corresponding sample number to be used within TopSpin or the SampleJet interface.



**Figure 4:** Enumeration of the rack

**Table 1:** Sample holder numbers

Hardware	Holder Number Range
Magnet	0
Sample Storage: OpenShop	1 to 99 (97-99 with spinners only)
Sample Storage: Racks (5 96 well plates)	101 to 596 Rack 1: 101 – 196 (1A1-1H12) Rack 2: 201 – 296 (2A1-2H12) Rack 3: 301 – 396 (3A1-3H12) Rack 4: 401 – 496 (4A1-4H12) Rack 5: 501 – 596 (5A1-5H12)
Pre-heating unit	618 to 625



**Figure 5:** SampleJet sample organisation.

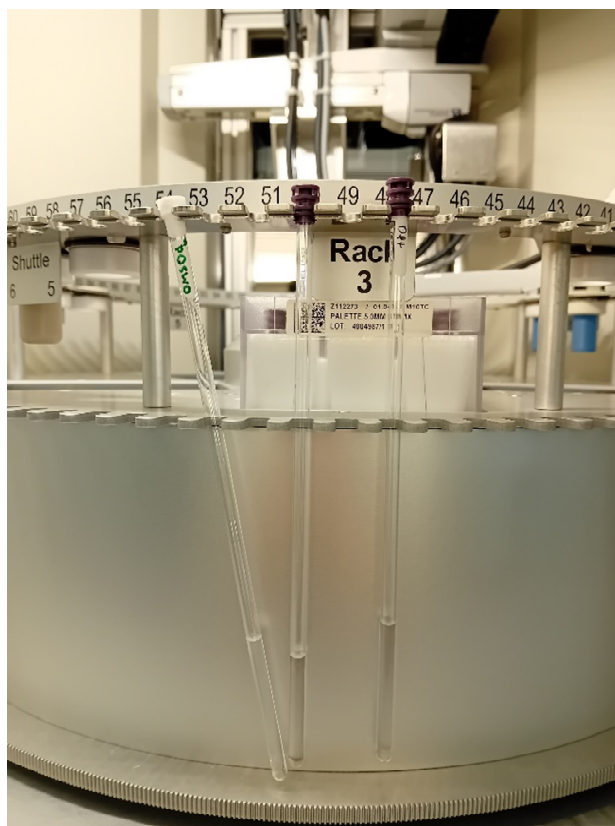
## 5. Placement of NMR samples – OpenShop

**WARNING:** It is crucial that samples are placed correctly in the SampleJet otherwise they may be damaged during their transportation.

Using the SampleJet interface, follow the below steps:

1. Access the main menu by pressing 'X'
2. Select 'Sample Access'
3. Select 'Access Sample'
4. Select sample number of interest. The system will state it is busy whilst moving to the position. Wait until the system is idle to place the sample. Ensure that it is placed in a straight fashion, and is being held by the sides of the cap, not the bottom of the cap.

Note for the samples with spinners in position 97-99, the height of the sample tube must not be more than 60mm above the spinner otherwise the SampleJet will break the sample tube.



**Figure 6:** Three OpenShop samples placed within the SampleJet.

(left) Incorrect cap and incorrect placement. Sample has not been placed in a straight fashion.

(middle) Correct cap and correct placement. Sample is being held by the sides of the cap.

(right) Correct cap but incorrect placement. Sample is being held by the bottom of the cap.

(below) Left photo zoomed in to highlight correct and incorrect cap position.



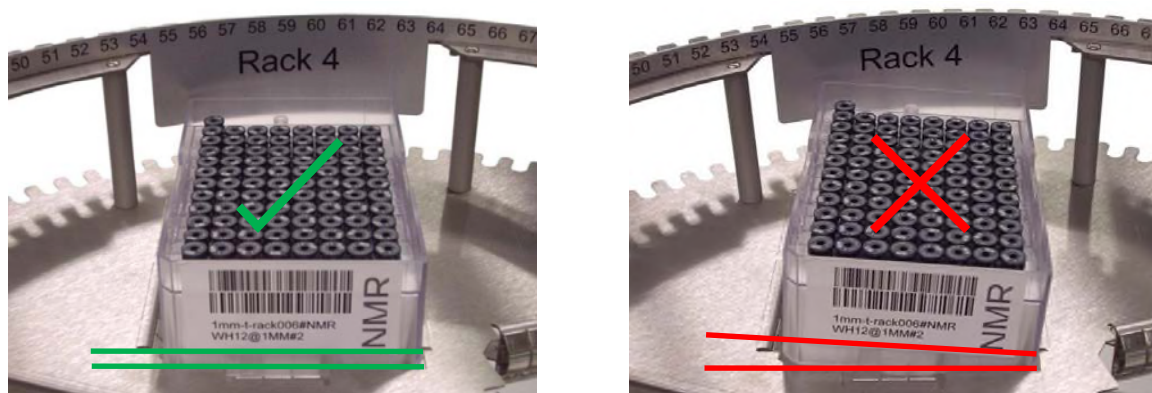
## 6. Placement of NMR samples – Rack Plates

**WARNING:** It is crucial that samples are placed correctly in the SampleJet otherwise they may be damaged during their transportation.

**WARNING:** For the 96 well plate NMR racks, if a tube is touching the plate bottom, the cap is incorrectly placed or the tube length is too long.

Using the SampleJet interface, follow the below steps:

1. Access the main menu by pressing 'X'
2. Select 'Sample Access'
3. Select 'Access Rack'
4. Select rack number of interest. The system will state it is busy whilst moving to the rack position. Wait until the system is idle to place the rack.
5. Place the rack gently into the rack position **without the cover**, ensuring it is not placed at an angle.



**Figure 7:** How to insert a rack correctly in the SampleJet.

## 7. Operating Modes

When the SampleJet is in charge, the 'Automatic' mode is active and barcoded caps must be used. This mode will ensure that the correct shuttle is used for the tubes in the storage rack. This is the default mode for the SampleJet. For samples that must be manually inserted into the magnet, the SampleJet can be changed from 'Automatic' to 'Manual' mode. In 'Manual' mode, the SampleJet is not in use and no transports can be triggered.

Below describes the steps to change between each mode. If unsure what mode the system is in, the screen SampleJet display will state either 'Manual' or 'Idle' ('Automatic').

### 7.1. 'Manual' to 'Automatic' mode

**WARNING:** Prior to changing mode, you must eject any sample present in the magnet. If you are unsure whether a sample is in the magnet, run through the manual eject procedure in Section 8.

1. On the SampleJet interface, select 'X' to return to the menu
2. Select 'Sample Transport'
3. Select 'Leave Manual Mode'

The system will confirm no sample is present within the magnet (BST). Wait until this process is finished. Once the system states it is idle (bottom left hand corner), the SampleJet is ready to use.

If a sample was present in the magnet, it will attempt to eject it however the black slider will be closed. The black slider will then open so the sample can be retrieved. The SampleJet is now in an error state and the gas flow will not turn itself off. This cannot be turned off using 'ej'/'ij' commands. The Emergency Stop button above the screen must be pressed and the SampleJet restarted. Refer to Section 12.

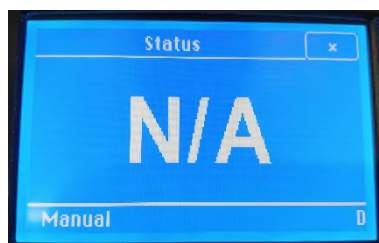


**Figure 8:** SampleJet interface when in automation mode and is idle.

## 7.2. 'Automatic' to 'Manual' mode

Before changing mode, all samples must be returned to storage.

1. On the SampleJet interface, select 'X' to return to the menu.
2. Select 'Sample Transport'
3. Select 'Return All Samples to Storage'. This will move any samples from either the magnet or the heater back to their original position in the autosampler. Wait until all samples have been returned to the OpenShop or the sample racks. If no sample were present, select 'Acknowledge'.
4. Under 'Sample Transport', select 'Switch to Manual Mode'.



**Figure 9:** SampleJet interface when in manual mode.

## 8. Manual injection/ejection of NMR samples in spinners (manual mode)

In manual mode, samples can be directly injected/ejected from the magnet bore, bypassing the SampleJet altogether. If manual mode is used, samples must be placed in a normal NMR spinner. The sample does not require the use of a barcoded cap.

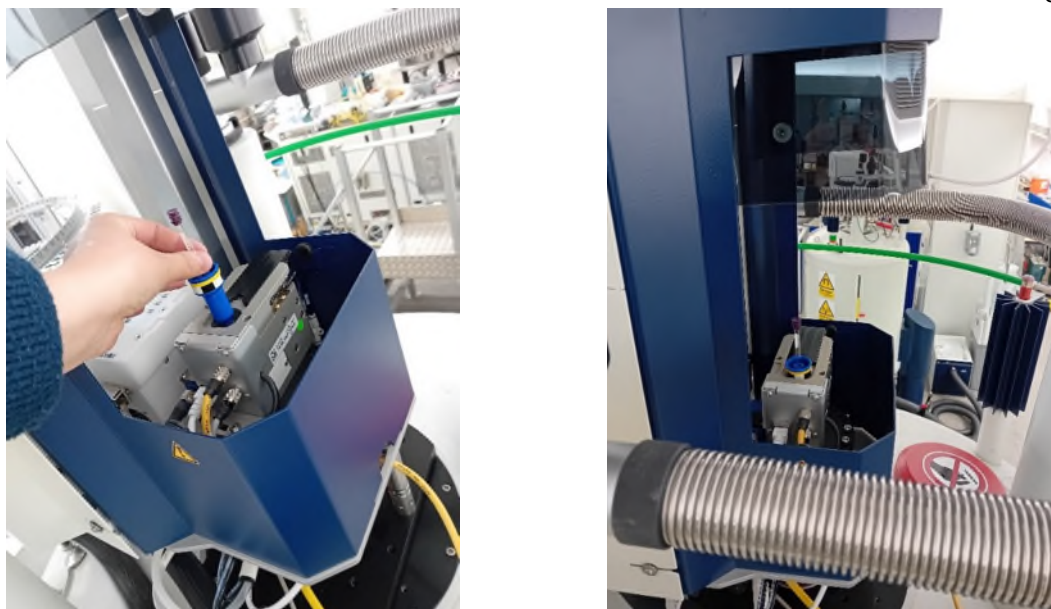
**WARNING:** During manual injection/ejection, be careful of the hardware around the entry point of the magnet. This includes the SampleJet itself as well as the N<sub>2</sub> and He ports and tubing.

At the back of the SampleJet, lift the black plastic cover to gain access to the entry point of the magnet. When the plastic cover is at the top, tilt the cover towards the SampleJet to lock it in place (Figure 10).



**Figure 10:** (left) Black plastic cover in its default position and (right) raised and secured in place to gain access to the magnet bore.





**Figure 11:** (left) Placing a 5mm NMR tube with spinner into the entry point of the magnet with the lift gas on. (right) Sample placed into the entry point of the magnet, ready for the ejection gas to be turned off.

## 9. How to run samples outside of IconNMR

**WARNING:** Ensure the SampleJet is in 'Automation Mode' (Section 7.1).

**WARNING:** Ensure the samples tubes and caps are set up correctly (Section 3).

Both Openshop and rack samples can be directly added to the magnet using TopSpin, or using the SampleJet interface. If using TopSpin, use the 'sx' command followed by a number to empty the magnet and insert a sample with a given holder number. To remove a sample, use the 'sx ej' command.

To add samples to the magnet via the SampleJet interface, the following steps.

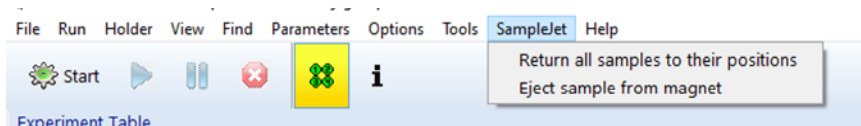
1. Confirm the system is in 'Automation Mode'. If not, refer to Section 7.1.
2. Select 'X' to return to the menu.
3. Select 'Sample Transport'
4. Select 'Storage to Magnet'
5. Type in the sample position and click OK
6. If a sample is already present in the magnet, it will return that sample to its original position, then transfer the desired sample. Once transferred, the interface will state the sample position and sample ID (barcode on cap).



**Figure 12:** SampleJet interface in automation mode and when sample 1 is present in the magnet

Samples can be ejected from the magnet using the 'sx ej' command in TopSpin, the SampleJet interface and via the IconNMR window. To eject samples from the magnet via the SampleJet, take the following steps.

1. Confirm the system is in 'Automation Mode'. If not, refer to Section 7.1.
2. Select 'X' to return to the menu.
3. Select 'Sample Transport'
4. Select 'From Magnet to Storage' to move the sample in the magnet back to its original position. To move all samples back to their original position, select 'Return all samples to storage'.



**Figure 13:** IconNMR interface highlighting the options to move samples back to their original position in the autosampler.

### 10. How to set the temperature control of the 5 96 well plate sample racks

The temperature of the 5 racks are individually controlled and can be set between 277 - 317 K. This is used to ensure the sample temperature controlled during sample storage before and after sample analysis. It is recommended to either not use temperature control or to set the temperature to 298.00 K. Below follows the procedure to change the temperature of rack 1 to 298.00 K.

1. Select 'X' to return to the menu.
2. Select 'Sample Conditioning'
3. Storage Temperature. The dotted button is the one selected i.e., is the on button is spotted, the sample temperature control is on.
4. Select 'set rack temperature'. Here will detail the temperatures of each rack to 2 decimal places and will continuously update with the current temperature.
5. Select the desired rack (rack 1). Here, it will specify the target temperature and the current temperature.
6. Select 'set target temperature' and type the desired temperature to 2 decimal places.



**Figure 14:** SampleJet interface when adjusting the storage temperature. (left) Overview of the storage temperature for all racks, (middle) rack 1 temperature and (right) setting of the rack 1 temperature.

## 11. Running with sample heating prior to insertion

There are three options regarding the use of the sample heating prior to sample injection into the magnet. By default, the sample heater is used in a dry setting and each sample tube used in automation is placed in the heater for a minimum of 30 seconds before injection into the magnet. Its time within the heater may be longer than the minimum time if it is waiting for the previous sample to be ejected from the magnet. This duration is set within the configuration of IconNMR and cannot be changed by general users. If you require this time to be altered for your analysis, you must consult a member of the NMR staff prior to starting your analysis.

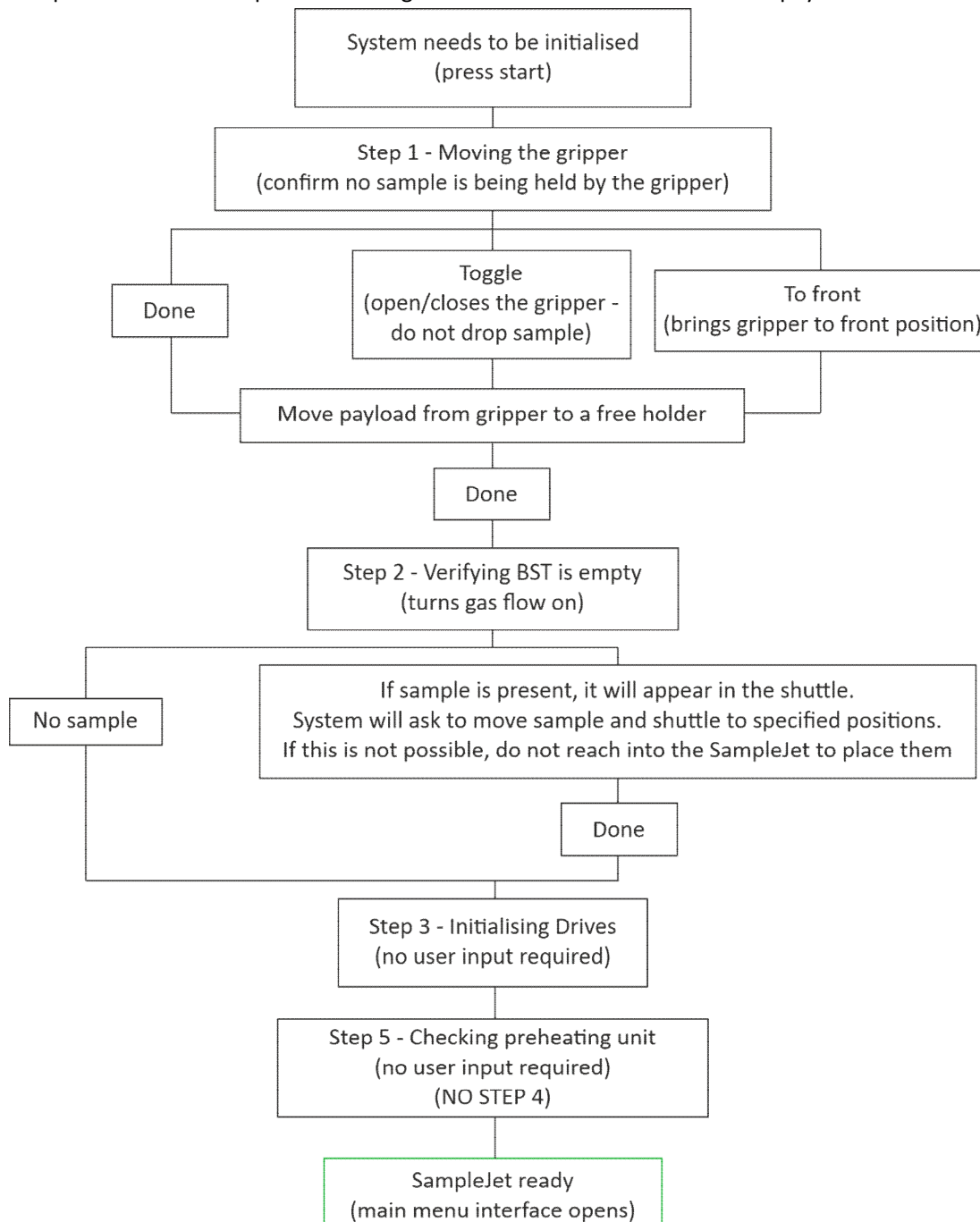
**Table 2:** Three modes when using the sample heating system

Mode	Description
Off	No drying or heating is applied.
Dry	Default mode Heater blows room temperature gaseous nitrogen over the NMR tube to remove any moisture that builds up during its time in the cooled rack.
Dry+Heat	Heater blows room temperature gaseous nitrogen over the NMR tube and heats the tube to a desired temperature for a specified time.

1. Select 'Sample Conditioning'
2. Select 'Heater Settings'
3. Select desired setting. If the heater was off prior to the change, it will take some time to turn on and equilibrate.
4. For 'Dry+Heat' only – Select 'set target temperature' and specify desired temperature. It is recommended to wait at least 5 minutes prior to submitting the first sample to ensure the heater temperature is stable prior to analysis.

## 12. Emergency Stop

The red emergency button on top of the SampleJet is used to immediately stop any movement of the carousel and the gripper. The interface will display 'ERROR – Emergency stop'. Releasing the emergency button (rotating clockwise) will restart the firmware and the SampleJet will start a recovery after the system has started up again. It is recommended to keep the access door closed during this startup (excluding during the actions taken by the user that the SampleJet states are to be completed) as it will significantly reduce the time it will take to complete. Note the sample tube throughout the restart is described as the 'payload'.



**Figure 15:** Typical for a typical SampleJet restart after the emergency stop button is pressed.

### 13. Other options

This is not a comprehensive manual highlighting all the capabilities of the SampleJet. It is to be used only as a guide. For a full description, it is recommended to use the Bruker SampleJet user manual available on the Bruker website.

### 14. SampleJet Consumables

There are a large variety of SampleJet consumables that can be purchased directly from Bruker. The most common consumables, a **BRIEF** description and their part numbers are listed below. Note that some of these can be purchased from other vendors. If you are unsure what consumables you need to purchase, please ask the NMR staff. This information was correct from April 2024.

Bruker Consumables: <https://store.bruker.com/en-GB/>

GPE Consumables: <https://www.gpescientific.co.uk/>

**Table 3:** SampleJet Consumables

<b>Bruker Consumables</b>		
<b>Bruker Part</b>	<b>Description</b>	<b>Part Number</b>
3mm Tubes with Caps	Diameter 3mm Tube length appr. 4"	Z112272 (96 pcs. in a rack)
3mm Tubes and Caps	Diameter 3mm Tube length 7"	Z117559 (100 pcs. each)
3mm Caps	Used for Standard 7" tubes	Z107163 (100 pcs.)
5mm Tubes with Caps	Diameter 5mm Tube length appr. 4"	Z112273 (96 pcs. in a rack)
5mm Tubes and Caps	Diameter 5mm Tube length 7"	Z107373 (100 pcs. each)
5mm Caps	Used for Standard 7" tubes	Z106779 (100 pcs.)
Cap sealing balls	Colour: white	Z72497 (100 pcs.)
Rack with 96 sample tubes	5 mm	Z112273
Rack with 96 sample tubes	3 mm	Z112272
<b>GPE Consumables</b>		
<b>GPE Part</b>	<b>Description</b>	<b>Part Number</b>
Sample Vault™ Racks	Rack with 96 sample tubes Coded (required)	SVOP-103.5-CC-96PK-5-96PK (plus many more)