SOP: filling liquid nitrogen on NMR magnets

This is only to be done by authorized personel

BRUKER
NMR Magnet System

UltraShield™ NMR Magnet Systems
Users Manual (english version)

Version 005

BRUKER Magnetics

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USERS MANUAL FOR ULTRASHIELD™ NMR MAGNET SYSTEMS

Refilling Procedure Nitrogen
Refilling Procedure Helium

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Safety

1 Safety during Refilling Procedure

During the refilling procedure, it is necessary to enter the marked danger area of the magnet system. In order to eliminate the danger associated with this you must adhere to the following safety precautions.

1.1 Protection from Magnetic Field

The magnet system generates a very strong magnetic field. It can influence electronic devices, magnetic media and ferromagnetic metals. Please observe the following warning notes for protection against the effects of the magnetic field.

Warning — **Danger of heart seizure for persons with pace-makers. Persons with pace-maker implants must not under any circumstances enter the marked danger zone, or carry out the refilling procedure.**

Caution — **Danger of injury due to flying metal parts. Do not use any magnetic tools or objects in the marked area. You could be hit by a piece of metal flying uncontrollably under the influence of the magnetic field.**

Note — Data on magnetic based storage media can be destroyed by the magnetic field. Do not carry any credit cards or similar objects with magnetic identification, when entering the marked area.

1.2 Protection against Ultra-Low Temperatures

Liquid nitrogen has a temperature of -196°C, and liquid helium has a temperature of -269°C. If the skin comes into contact with splashes of cryogenic fluids, this can lead to severe cold-burns.

Warning — **Danger of blindness if liquid nitrogen comes into contact with the eyes. Always wear protective goggles when carrying out the refilling procedure.**

Caution — **Danger of severe cold-burns if skin comes into contact with cryogenic fluids. There is also a danger of skin adhesion with super cooled metal parts. Always wear protective gloves and closed clothing when carrying out the refilling procedure.**

Note — The O-rings of the magnet system are also sensitive to low temperatures. Make sure that no liquid helium or liquid nitrogen comes into contact with the O-rings during the refilling procedure. The most endangered O-rings are located in the dewar flange, in the reduction flanges and in the closure flanges at the upper and lower end of the room temperature bore tube.

1.3 Protection from Gases

Evaporation of cryogenic fluids like helium and nitrogen can lead to suffocation, if the oxygen content in the immediate atmosphere, as required by the human body, is reduced.
Helium Gaseous is very light and rises up to the ceiling. Danger of suffocation due to helium is increased when working high up, for instance on a pedestal or ladder.
Nitrogen Gaseous is very heavy and sinks to the floor. Danger of suffocation increases when working near floor level or in pits.

Caution — **Danger of suffocation from excess gases caused by spilled cryogenic liquids, and from a quench. Make sure that the area is well ventilated and avoid working high up or low down (depending on the type of cryogenic fluid) after a quench.**

1.4 Protection against Fire and Explosion Risks

The extremely low temperatures associated with cryogenic fluids lead to condensation of the air's oxygen on the cold pipes. The condensed oxygen drips down and can combust spontaneously when coming into contact with oil or fat. Also contact with flames (e.g. lighters or lit matches) can result in explosive combustion.

Caution — **Danger of self-combustion or explosion. Respect the smoking ban during the refilling procedure, do not produce any flame of any sort and make sure that the immediate vicinity of the magnet system is clean (without oily cloths and similar things).**

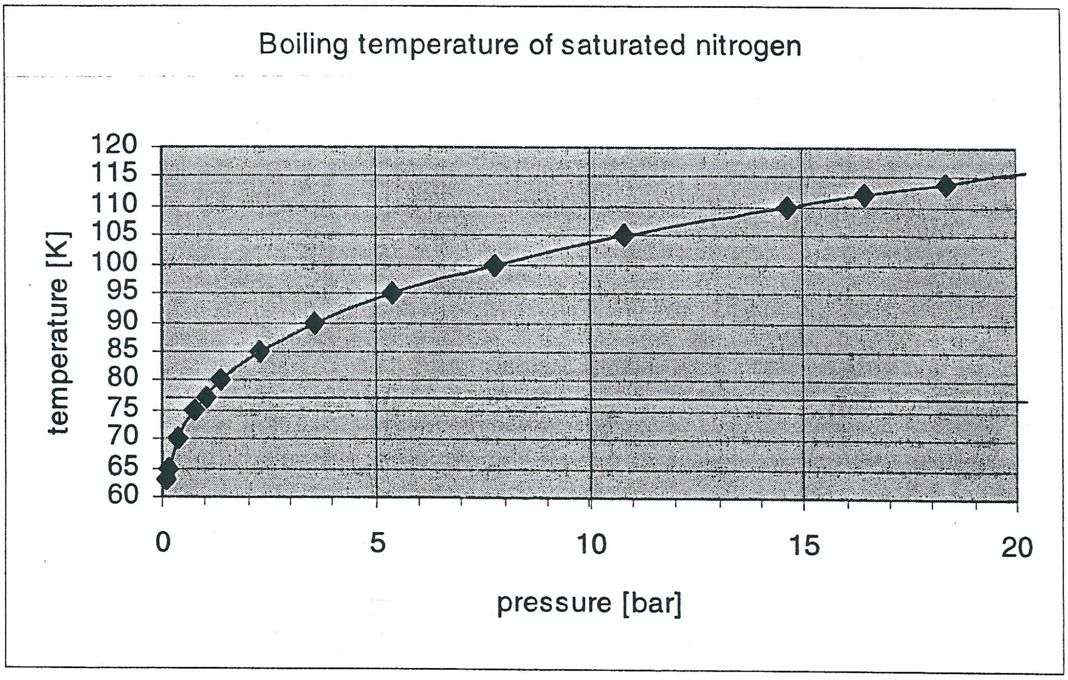
1.5 Protection against Explosion Risks due to High Pressure transport vessels

Cryogenic liquids, even when kept in insulated storage vessels (dewar vessels), remain at a constant temperature by their respective boiling points and will gradually evaporate.
The very large increase in volume accompanying the vaporization of the liquid into gas and the subsequent process of warming up is approximately 700:1 for helium and nitrogen and therefore:

Warning: — **Do not use cryogens that have been stored in high pressure containers for cryogenic liquids! If no other containers are available the pressure must be released completely before connecting the high pressure transport container to the cryostat. This would present an explosion hazard for the magnet system and could lead to severe damage!**

Temperature rise — The high pressure within high pressure transport containers leads to a large increase of the boiling temperature of the liquid gas. Transferring such overheated liquid gas into the low loss cryostat will result in very high boil off and strong oscillations until the liquid gas has cooled down to the boiling temperature at atmospheric pressure again!

Temperature diagram **The pressure dependence of the boiling temperature and thus of the temperature of liquid nitrogen stored at a given pressure within a transport vessel is given in the diagram below. As soon as the pressure is released the liquid will start to boil off strongly and will reduce its temperature back to 77 K at atmospheric pressure.**



Warning: **Containers for cryogenic liquids must not be closed completely as this would result in a large build up of pressure. This will present an explosion hazard and leads to large product losses!**

1.6 Physical Properties of Nitrogen

Liquid nitrogen is boiling at a temperature of -196°C. Nitrogen is colorless, odorless and not inflammable!

Identification:

22
1977

Heating leads to pressure increase and danger of bursting. Spilled fluid is extremely cold and evaporates very quickly. Fluid leads to severe cold burns and severe eye injuries. Gas causes suffocation without preceding symptoms. Nitrogen gas has a higher density than air, is sinking to the floor and is spreading along the floor. Together with moist air, production of fog is observed.

1.7 Physical Properties of Helium

Liquid Helium is boiling at a temperature of -296°. Helium is colorless, odorless and not inflammable!

Identification:

22
1963

Heating leads to pressure increase and danger of bursting. Spilled fluid is extremely cold and evaporates very fast. Fluid leads to cold burns and eye injuries. Gas causes suffocation without preceding symptoms. Helium gas has a lower density than air, is rising to the ceiling and is spreading along the ceiling. Together with moist air, production of fog is observed.

1.8 First Aid after Accidents with Cryogenic Fluids

Get injured people to safety. Injured people must lie comfortably. Remove tight clothes! Immediately remove wet clothes and thaw wound parts of the body with warm water. Do not rub frozen parts of the body. Cover them with sterilized sanitary pads.

1.9 Protection against Mechanical Danger

To isolate the magnet system as well as possible from mechanical disturbances from the environment, inflatable rubber buffers or other shock absorbing components are often used. They protect the magnet system from vibration, but the system is still sensitive to lateral shocks.

Caution — **Danger of overturning when moving or mounting the magnet system. When moving the magnetic system, follow the corresponding instructions in the magnet manual. It is strictly forbidden to climb the magnet system.**

Nitrogen Refilling Procedure

2 Transport Vessel for Liquid Nitrogen

There are various forms of transport vessels for liquid nitrogen (chemical formula N_2). Described here are the characteristics which are valid for all vessel implementations and which you should be aware of for a safe execution of the refilling procedure.

2.1 Danger source - Ultra-low Temperature

The transport vessel contains liquid nitrogen with a temperature of -196°C. Please observe the warning notes in chapter 1 "Safety during Refilling Procedure", page 3.

2.2 Requirements for Nitrogen Transport Vessel

The transport vessel must fulfil the following requirements.

- It must not be ferromagnetic. That means it must not be made up of any material, which is susceptible to magnetic fields.

Warning	**Danger of injury: Magnetic transport vessels could be pulled uncontrollably towards the magnet system and could trap or crush people.**

Caution	**Danger of destruction of the magnet system. Magnetic transport vessels could be pulled uncontrollably towards the magnet system and lead to destruction.**

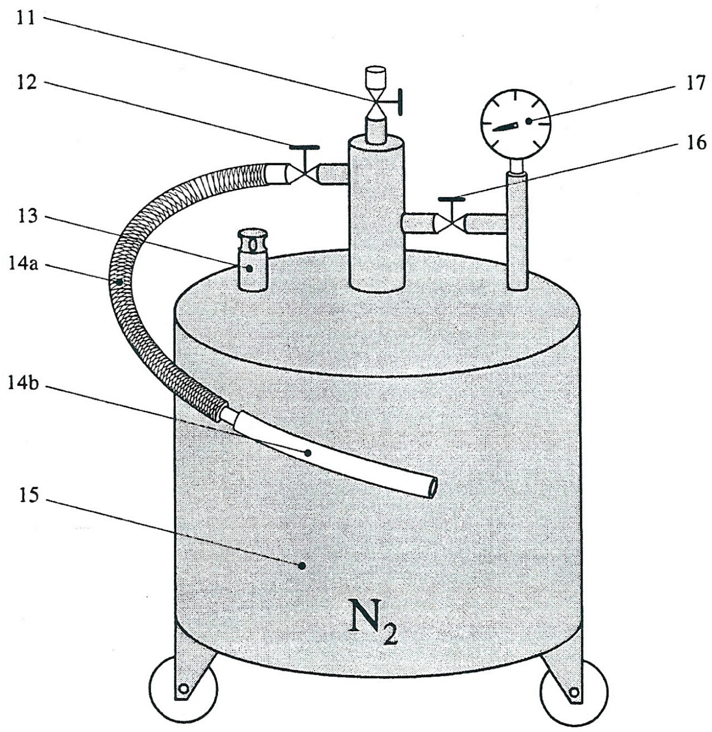
- It must possess a pressure release valve, which releases evaporating nitrogen.
- For fluid extraction, a transfer hose must be used which either has a metallic sleeve or is made of Teflon/Latex.

Warning	**Danger of injury due to plastic hoses splintering under extremely low temperature conditions. Use only specified hoses made of Teflon or Latex or a surrogated PFA hose.**

Note	A complete **Nitrogen Refilling Set**, consisting of surrogated PFA hose and the necessary adaptors, is available from Bruker AG as Part Number Z53144

2.3 Main components

A transport vessel for liquid nitrogen consists of the following main components:



Transport vessel for liquid nitrogen

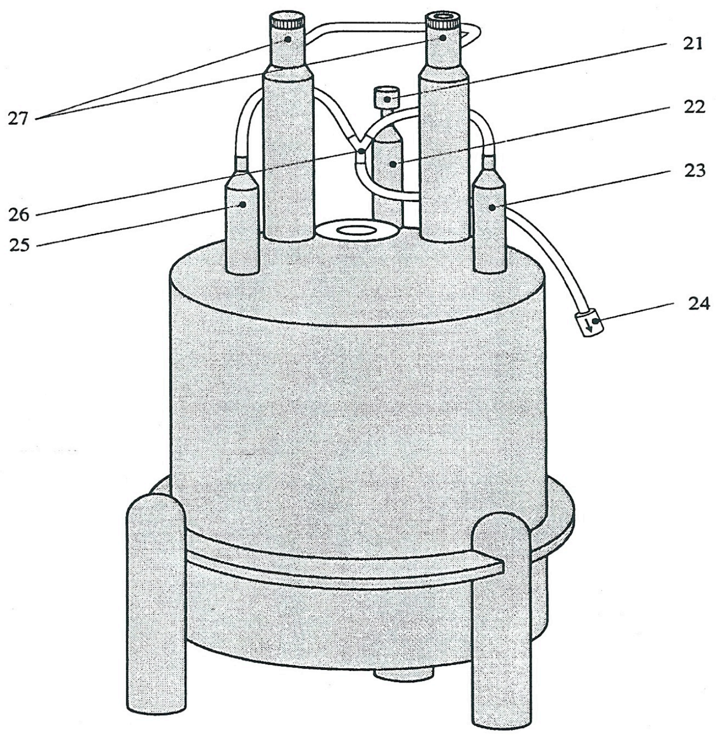
Legend

11	Gas release valve
12	Liquid nitrogen extraction tap
13	Excess-pressure release valve
14a	Transfer hose with meshed metal sleeving
14b	Teflon or Latex transfer hose or surrogated PFA hose
15	Transport vessel
16	Pressure generation armature
17	Manometer

3 Magnet System with N_2 Flow System

Described here after are the individual elements common to all magnet systems with N_2 flow systems, which you should know about and observe to ensure safe execution of the nitrogen refilling procedure.

Caution — **The magnet system contains liquid nitrogen and a magnet that produces a very strong magnetic field. Please observe the safety measures as described in chapter 1 "Safety during Refilling Procedure", page 3.**



Magnet System with N_2 Flow System

Legend

21	Safety valve	25	Left-hand nitrogen turret
22	Rear nitrogen turret	26	N_2 flow system
23	Right-hand nitrogen turret	27	Helium turrets
24	Check valve		

4 Measurement of Fluid Level

The fluid level measurement indicates how much nitrogen needs to be added. Depending on the required quantity, the filling process will take between 5 and 15 minutes.

4.1 Tools for Fluid Level Measurement

The easiest way to measure the fluid level is by means of a dip-stick. This is a long tube with a round end-piece, available as Part Number Z27451 from Bruker. If you do not have a dip-stick available you can also carry out the fluid level measuring process with the help of an epoxy rod.

4.2 Measuring the Fluid Level with a Dip-stick

Follow the procedure outlined below:

1. Slowly insert the dip-stick into the right-hand nitrogen turret (23) and observe the upper end-piece.

Caution — **Danger of eye injury by spraying nitrogen. Wear protective goggles during this process.**

2. Stop inserting as soon as nitrogen starts to spray out of the end-piece of the dip-stick.

   The nitrogen starts to spray out when the warm end of the dip-stick dips into the cold nitrogen fluid. The boiling and evaporating nitrogen is hurling nitrogen splashes through the hollow centre of the dip-stick.

3. Take note of the position of the dip-stick, by taking hold of it directly at the end of the nitrogen turret and pull it out of the vessel.
4. Determine the fluid level, by holding the dip stick next to the magnet system: The lower end indicates the current level of liquid nitrogen.

4.3 Measuring the Fluid Level with the Epoxy rod

Follow the procedure outlined below:

1. Slowly insert the epoxy rod into the right hand nitrogen turret (23) until it touches the floor of the vessel.
2. Let the epoxy rod cool off on this position and take note of its position by taking hold of it directly at the entrance of the turret.
3. Pull the rod out of the vessel and swing it about in the air.

   Note — The rod is now covered with a layer of ice along the length of the stick which was submerged in the liquid nitrogen.

4. Determine the fluid level by holding the rod next to the magnet system: The position where the layer of ice starts indicates the current liquid nitrogen level.

5 Transfer Preparation

5.1 Preparing the Transport Vessel

Follow the procedure outlined below:

1. Reduce the pressure to a **maximum of 0.35 bar**, by opening the gas release valve (11).
2. Close all other valves and taps.

5.2 Preparing the Magnet System

Follow the procedure outlined below:

3. If the magnet system is equipped with vibration dampers: Let the air escape out of the dampers by turning the switch on the base plate to the "Down" position.
4. Pull out the hose of the N_2 flow system (26) from the front nitrogen turret, and if there is a heat exchanger present, remove it.

Note — Not all magnet systems possess a heat exchanger. Heat exchangers are fastened onto the nitrogen turrets and inhibit icing up of the outlets.

5. Check that the outlets at both of the front nitrogen turrets are free (23, 25).

   You can check whether the outlets are free by observing whether evaporating nitrogen is escaping from them. Another possibility is to carefully insert a thin rod through the nitrogen turrets into the tank.

Caution — **Never try to remove ice from the nitrogen turrets without prior contact with your local Bruker Service Department.**

6. Insert a Teflon hose onto the left-hand turret (25) and fasten the end so that it is pointing away from the magnet system.

Note — For this it is easiest to use the corresponding hose from the N_2 flow system.

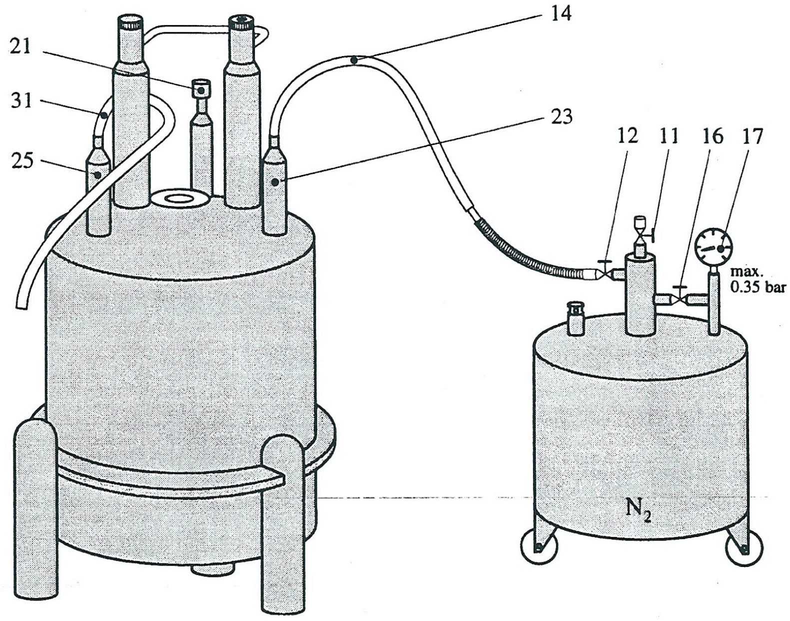
7. Check the current fluid level in the nitrogen tank in the right-hand turret (see section 4 "Measurement of Fluid Level", page 10).

Caution — **Danger of the magnet system bursting due to excess pressure. Always leave the safety valve (21) in its correct position.**

6 Filling procedure

Connect the transfer hose from the transport vessel to the magnet system. With a slight pressure differential (maximum 0.35 bar), the nitrogen will be drawn from the transport vessel into the magnet system. The end of the filling process can be recognised in that nitrogen will start spraying out of the left hand turret.

The connection between the transport vessel and the magnet system is made by the transfer hose.



System prepared for N_2 filling procedure

Legend

11	Gas release valve
12	Liquid nitrogen extraction tap
14	Transfer hose
16	Pressure generation armature
17	Manometer
21	Safety valve
23	Right-hand nitrogen turret
25	Left-hand nitrogen turret
31	Teflon hose

6.1 Establishing Connection and Nitrogen Transfer.

1. Insert the free end of the transfer hose (14) onto the right-hand nitrogen turret (23).
   It suffices to make sure that it is firmly placed. You do not require any additional fastening means.

Note: Make sure that the transfer hose is not creased or kinked, otherwise problems may occur with the filling process.

2. Supervise the entire filling procedure so that you can immediately intervene should the pressure exceed 0.35 bar or when the filling procedure is ended.
3. Make sure that the pressure in the transport vessel is not higher than 0.35 bar. You can regulate the pressure by opening the gas release valve (11) (for pressure decrease) or by pressing the armature (16) (for pressure increase).

Caution: **Danger of explosion due to excess pressure. Respect the allowed maximum pressure of 0.35 bar at all times.**

4. Open the liquid nitrogen extraction tap(12) and check whether nitrogen is being drawn through the transfer hose.

Note: If you are using a Teflon transfer hose it is easy to visually observe whether the filling procedure is running correctly.

5. Check whether gas is escaping from the Teflon transfer hose (31) at the left-hand nitrogen turret. That indicates that the turret is free and not iced up.

Caution: **Danger of eye injury, due to splashes of ultra-cold liquid nitrogen. Make sure that nobody is positioned near the areas where nitrogen is escaping from the hose.**

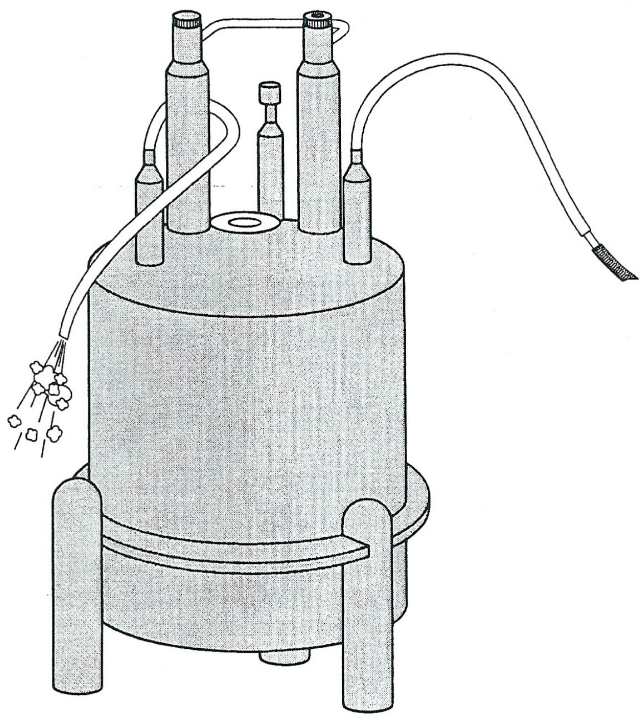
6. By observing the manometer (17), ensure that the pressure does not exceed 0.35 bar during the entire filling procedure and that the left-hand turret is always free (gas escaping at the Teflon hose).
7. Check whether the transport vessel contains enough fluid.

Note: The pressure indicated on the manometer (17) starts to rapidly sink as soon as the transport vessel becomes empty.

Note: The end of the filling procedure is recognised when liquid nitrogen starts spraying out of the left-hand nitrogen turret.

6.2 Terminating the Filling Procedure

The filling procedure is completed when nitrogen starts spraying out of the Teflon hose on the left-hand nitrogen turret.



End of the filling procedure

1. Stop the filling procedure by closing the liquid nitrogen extraction tap (12) and/or
2. releasing the pressure in the transport vessel by means of the pressure release valve (11).
3. Close the pressure exertion armature (16).

6.3 Return to Standard Operation after the Filling Procedure

After the filling procedure has been completed, follow the procedure outlined below.

1. Let the transfer hose (14) thaw out by waiting for about 10 minutes and/or
2. carefully warm the end of the transfer hose with a fan-heater.
3. Remove the transfer hose (14) from the right-hand nitrogen turret (23) of the magnet system.
4. Remove the Teflon hose (31) from the left-hand nitrogen turret (25).
5. Check that the neck tubes on both turrets are free (visible escaping of gas, or insertion of dip-stick).

Caution	**Never try to remove ice from the nitrogen turrets without prior contact with your local Bruker Service Department.**

6. Connect the N_2 flow system (26)

Note: Make sure that the check valve (24) is correctly mounted (arrow in direction of nitrogen flow) to allow the nitrogen gas to escape from the nitrogen tank.

7. If the magnet system is standing on vibration dampers: Activate the dampers by turning on the switch on the base plate to the "up" position.

6.4 Recording the Filling Procedure

The filling procedure replaces the evaporated nitrogen. If the filling procedure is accurately recorded, the average consumption can be estimated. Significant changes in nitrogen consumption is an early warning signal that the magnet system is not in order.

1. Record the date of the filling procedure and the quantity of nitrogen used.