8.2 Telecommunication

Please refer to the AVANCE NMR layout in chapter *Floor Plan [▶ 75]*. The following ports/ connections are required:

- Telephone/data ports behind the workstation.
- Ethernet cable between the AVANCE cabinet and the workstation.

8.3 Compressed Gas

Some components of the AVANCE system and the vibration isolators, which are integrated into the magnet legs, operate with compressed gas.

8.3.1 General Requirements

Compressed gas line: The standard AVANCE system requires one compressed gas line with at least two regulated outputs.

Regulators: Each output should terminate with a regulator (including gage head) suited for a pressure range of 0 - 8.6 bar (0 - 125 psi). The output of the regulator should allow the quick connect for an 8 mm diameter gas tube..

- Compressed nitrogen gas needed for temperature control with VT experiments in order to achieve optimal NMR performance. For example, the BCU-I cooling unit requires a dew point of -51°C (-60°F) for the compressed gas.
- Compressed air or nitrogen gas for spinning.
- Compressed air or nitrogen gas for sample ejection, and for the magnet's vibration isolation units.
- Compressed air or nitrogen gas for the optional CryoProbe system.

8.3.2 Gas Supply

The gas supply used depends on the frequency of the system:

- 200-400 MHz: Compressed air
- 500 MHz: Nitrogen gas with >95% purity.
- 600-800 MHz: Nitrogen gas with >96% purity.

Notes:

- A nitrogen separator (offered by Bruker as an option) can be built into the AVANCE cabinet as an available solution. This will produce the nitrogen gas required for VT work. However, this is not suitable for larger flow rates required by MAS experiments.

  The nitrogen separator is suitable for use with the BCU-I cooling unit. However the nitrogen output from the separator is not pure enough and this unit should not be used with a N2 exchanger or BCU-II cooling unit for low temperature work.
- If a CryoCooling unit is to be installed, a secondary regulator, T-split from the supply line is recommended.
- The Emergency Sample Protection Device is used in conjunction with the CryoProbe system, and requires a cylinder of air or nitrogen gas.

System	Operating Pressure	Recommended Average Flow*
AVANCE + VT Unit	6-8 bar (80-120 psi)	43 l/min. (~1.52 cfm)
AVANCE + VT + Sample Changer	6-8 bar (80-120 psi)	55 l/min. (~1.95 cfm)**
AVANCE + MAS	6-8 bar (80-120 psi)	300 l/min. (~11 cfm)
* This is the actual consumption and minimum needed at the instrument input after the N2 supply (either a bulk tank, or a N2 separator). The sample lift to exchange a sample needs for about 1 minute 100 l/min. For a standard NMR system we expect 3...5 sample exchanges (which are included in recommended flow). ** A recommended flow of 100 liters/min. per sample for sample exchange (about 1 minute), average consumption will depend on the number of samples being used. Typical number of sample exchanges is up to 12 per hour (which is included in the recommended average flow). For non-MAS work, if an air-compressor and N2 separator are used, the flow requirements are 50% higher, i.e. 3 cfm. It is recommended to use a dual unit oil-less air-compressor rated at minimum double capacity of the specified requirement. Please refer to the next section on air compressors.		

Table 8.2: Pressure and Flow Requirements

8.3.3 Other Specifications

Oil Content:

Purity: ISO 8573-1 2010 [1:1:0] (oil free).

Water Content:

For the BCU-I cooling unit the compressed gas should have a dew point of -51°C (–60°F). For the BCU-II cooling unit, the dew point requirement is - 80°C (-112°F).

For room temperature work and higher: Dew point of < 4°C (39.2°F).

For low temperature work: The dew point must be at least 20°C (68°F) below the operating temperature.

If a cooling unit is used, then the dew point of the compressed nitrogen should be at least 10°C (50°F) below the temperature at the heat exchanger output.

Solid Impurities:

Use 5 micron filters for high resolution NMR. For MAS probes use 1 micron filters. The filters should retain a minimum of 99.99% of the specified particles.

VT Nitrogen Gas Quality

Proton Frequency (MHz)	Purity (volume percentage)
500	≧ 95
600-800	≧ 96
850-1000	≧ 97

Table 8.3: VT Nitrogen Gas Quality (500 MHz and above)

8.3.4 Compressed Air System

When designing a suitable compressed air system the following points must be taken into consideration:

- To prevent magnetic impurities from entering the magnet use only copper or stainless steel lines. Do not use iron or steel pipes. Plastic piping is unsuitable where very low dew points are required. Water vapor in the air will permeate plastic piping limiting minimum dew points to typically -25°C.
- To avoid surges in the air pressure (e.g. during sample lift) install a container of 10-20 liters in the air supply line to act as a buffer. Locate the buffer after the dryers in the supply line. **Buffer containers** must meet the appropriate safety requirements. They must have a working pressure of 16 bar and be proofed up to 30 bar. Use tanks which are internally coated with water and acid resistant material. This will prevent corrosion from impurities such as SO_2.

The three major components in a suitable compressed air supply line include the compressor, dryer and appropriate filters:

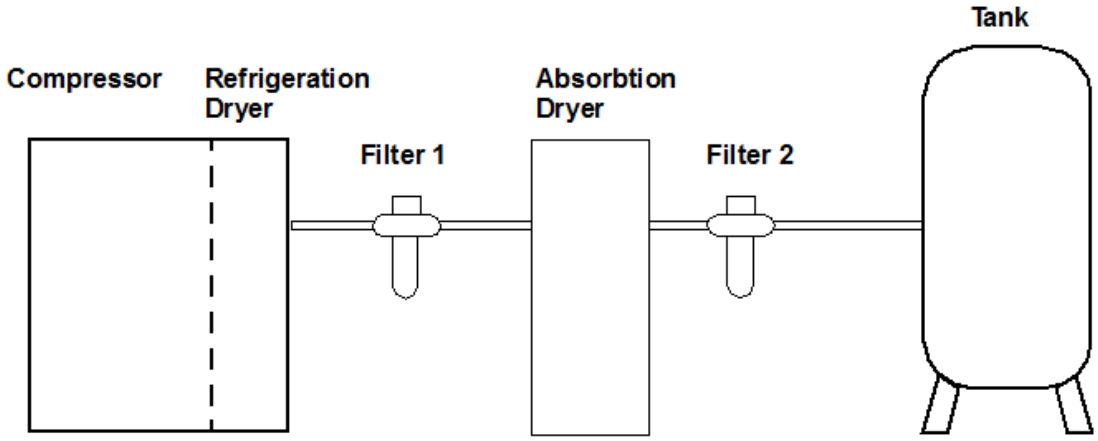

Figure 8.1: Example of a Typical Dryer/Filter System Setup

Filter 1:	General purpose liquid and dust removal filter (0.1 mg/m^3 - 0.1 ppm, 1 micron).
Filter 2:	High-efficiency dust, liquid and aeresol filter (0.1 mg/m^3 - 0.01 ppm, 1 micron).

When using a dryer/filter system setup, the following questions should be addressed:

- Pressure loss in piping?
- Efficiency loss in dryer?
- Pressure loss in filter?
- What is the required pressure?
- What is the required flow rate?

8.3.4.1 Air Compressors

When choosing an air compressor the following points should be considered:

- Ideally the compressor should be installed in a **dust free**, cool (use air conditioning as required) and dry place.
- The compressor must be **oil-free**. This can be achieved by using membrane or Teflon coated piston and scroll compressors. The compressor should be fitted with a fine dust inlet filter.
- The compressor must be capable of delivering the required flow rate and pressure suited to your particular system (see Compressed Gas Requirements). Generally the compressor should be large enough so it does not run continuously (e.g. > 50% of the time), which will cause overheating.
- The extra cost of choosing an oversized system may often be justified. The reduction in duty cycles will lower maintenance costs and extend the life of the system. A suitable compressor coupled to an adequate buffer will ensure a more **constant flow rate** leading to better performance. When spinning, the system uses a constant flow of air, but surges will occur during sample lift.
- Take into account the **pressure loss** along the line between the compressor and the final gate valve. The pressure drop depends on the pipe diameters. An internal diameter of 8 mm has been found to be suitable. The plastic tubing used to carry the supply from the final gate valve to the console has an outside diameter of 8 mm and is supplied by Bruker.
- Some types of **dryers**, e.g., absorption dryers can use up to 25% of the air flow to regenerate the drying material. If this type of dryer is used then the output capacity of the compressor must be sufficient to supply this requirement.
- Many compressors are fitted with dryer and a tray to collect **excess water**. Regular checking of the dryer and emptying of the water collector will ensure trouble free operation. This arrangement is quite satisfactory in environments with normal humidity (< 80%). However in areas of higher humidity (> 80%) a cooling coil with an automatic water drain must be fitted to the compressor outlet. This will ensure that filters do not become overloaded.
- Although not directly concerned with air quality, compressors are a **source of vibrations** which may interfere with NMR performance. You should consider using a compressor fitted with a vibration damping housing if it is to be situated close to the spectrometer. The output noise level should be < 75 dBA.

8.3.4.2 Dryers

Refrigeration Dryers

This type of dryer removes moisture from gas by cooling to within a few degrees of the freezing point of water. The condensed moisture is removed in a separator and drain trap mechanism located immediately downstream of the dryer. This drain should be valve switched automatically.

Advantages

- None of the compressed gas is wasted in regeneration which is more suitable if the capacity of the compressor is marginal.
- Maintenance free.
- Not as susceptible to oil mist contamination as adsorption dryers, thus do not have the same need for pre-filters.

Disadvantage

- These type of dryers are limited because of their inability to produce very low dew points. The recommended dew point for room temperature work of 4°C is only just achievable. Therefore if low temperature NMR is to be carried out, this type of dryer is unsuitable.

Absorbtion Dryers

The air is passed through cartridges of synthetic zeolite known as Molecular Sieves. The sieves are hygroscopic and retain water molecules when air is passed through them. Two sieves are normally used alternatively. A portion of the dry air output of sieve A is fed into sieve B to regenerate it. The amount used in regeneration is typically 15% but up to 25% may be required for very low dew points. The process is automatically reversed at regular intervals with the output of sieve B used to regenerate sieve A.

Advantages

- Much lower dew points are achievable compared to refrigeration dryers.
- Automatic Regeneration: Normally the sieves will last for many years if they do not become contaminated with oil, e.g. from mist in the air.
- The drying agent may be easily replaced.

Disadvantages

- Up to 25% of throughput is used to achieve the automatic regeneration.
- Requires the use of more dust filters.
- Filters at the input (oil < 0.01 mg/m^3) are required due to the susceptibility to oil contamination from mist in the air.
- The use of absorption dryers may lead to the generation of dust and so the dried air output must be fed through an appropriate filter (1 micron).
- These dryers require more maintenance than refrigeration dryers.
- They can be noisy when switching between the two cartridges.
- Due to the different absorption rates of nitrogen and oxygen the **N2/O2 composition** may change. To prevent this, an absorption dryer should be placed between the compressor and buffer tank.

8.3.4.3 Filters

Micro-filters must be fitted as the last element in the supply line. For specification see the section *Other Specifications [▶ 62]*.

Absorption dryers are prone to oil contamination and as such the input must be fitted with a oil filter (oil < 0.01 mg/m^3 99.9% removal efficiency). To protect the dryers, regardless of type, you are advised to install a water filter and an oil filter between the compressor and the dryer. Adsorption dryers may generate dust and may need extra dust filters at the output.

The output of refrigeration dryers must be fed through a carbon activated filter.

Water filters must be fitted with automatic water drains as opposed to manual drains. The use of valve switched drains is strongly recommended. Floater switched drains have a tendency to become jammed and hence require regular maintenance.

If you are particularly concerned about oil contamination in the air supply then you must consider using a submicron filter followed by an activated charcoal filter as this combination is particularly effective in removing oil.

8.4 Liquid Nitrogen Requirements

Purity of Liquid Nitrogen

Bruker has no specification for the quality of the liquid nitrogen. Inhouse we are using a purity of >99.9 %. There are no field gradients known because of the para-magnetism of oxygen. Nitrogen and oxygen are fully mixable over the whole concentration range.

Liquid nitrogen has a lower boiling point at −196 °C (77 K) than oxygen's −183 °C (90 K), and vessels containing liquid nitrogen can condense oxygen from air. When most of the nitrogen has evaporated from such a vessel, there is a risk that liquid oxygen remaining can react violently with organic material. Conversely, liquid nitrogen or liquid air can be oxygen-enriched by letting it stand in open air; atmospheric oxygen dissolves in it, while nitrogen evaporates preferentially.

A few percent of oxygen in the liquid nitrogen vessel will only give a minor temperature increase of the boiling point and will thus not increase the helium loss. With the higher boiling point the amount of oxygen will increase over the time.

Small nitrogen liquefiers will achieve a nitrogen content of >98 %. When using such a quality of nitrogen it is recommended to check the content of oxygen in the nitrogen vessel regularly. To limit the oxygen content it is advisable to refill when the nitrogen vessel is at least half empty. To get rid of the oxygen completely, wait until the liquid has evaporated before refilling. Be aware when only the helium has to cool the magnet coil the boil off will be about 40 times higher.

Refer to the chapter *Safe Handling of Cryogenic Substances [▶ 15]* for important information on the safe handling of liquid nitrogen.