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RS-EXPL-PROTECT for any NMR / EPR System up to 500 Bar in the case of Varian / Agilent / JEOL / Magritek or in excess of 500 Bar if a Bruker or Oxford Instruments Pulsar system is to be used.

Preamble.

In recent times the interest in high pressure NMR / EPR has been increasing necessitating the development of an explosion protection chamber to protect the user and his or her colleagues. If the system being used contains no gas and is 100% liquid based as in the case of the Extreme Pressure NMR systems currently running up to 3 kbar, no explosion protection is required as the system cannot explode as per basic physics. However, if a **gas is present the risk of explosion is real** and is not to be taken lightly.

Initial adjustment: If your laboratory only has one NMR system or only one is to be used for high pressure NMR set the explosion protection chamber up such that the legs are in contact with the top of the magnet and the bottom of the protection chamber just touches the top of the upper barrel protruding from the magnet. A pneumatic seal is not required so a slight gap of 0.5mm or less is acceptable.



Fig. 2, Varian / Agilent system adjustment

If you have an older style Bruker system where the optic sensor is not integrated into the upper barrel (see Fig. 1) make sure the inner tube is slightly deeper than the outer tube so no gap is visible as illustrated here. As the support is provided by O rings simply push on the inner tube to move it a few mm relative to the outer tube.



Fig. 1, Non-integrated optic sensor

In the case of the older Varian systems without any sample changer upper barrel additional hardware the protection tube can even be placed even lower so it covers the input to the upper barrel completely, see Fig. 2.

There is no bottom to the Protection chamber by intention. The rationale is if there were one and the tube were to explode, the remains of the tube would exit the protection chamber at a high velocity. With an open bottom the force would go in both directions and not just upwards thereby eliminating the risk of a missile in the laboratory. The force downwards would be taken up either by the bench or the floor of the laboratory.

Installation:

- I. **For Varian / Agilent / JEOL / Magritek NMR Systems:** As the OD of the sample holder mechanism or spinner turbine is larger than the inner bore of the inner tube this tube is to be removed by pressing down on it whilst holding the outer tube. A fair amount of force is required to move the inner tube relative to the outer tube but once it moves it can be fairly easily removed and kept to one side in case a Bruker spectrometer is to be used one day. If so this inner tube is re-installed.



Fig. 3, Parts from left to right: Screws, white support ring, rods, pressure tube inserted in tube holder and spinner turbine, tube assembly.

- II. **For Bruker and or Oxford Instruments Pulsar NMR Systems:** The spinner turbines for Bruker NMR Instruments are a nigh on perfect fit inside the inner tube so you can use the explosion protection chamber as a double walled device. In the case of Oxford Instruments there are no spinner turbines so the tube can be supported by the supplied tube holder

and attached strings.

- III. Pre-adjust the explosion protection chamber to the chosen magnet by inserting the tube assembly into the central hole of the white large ring to the required height. Roll the black O-ring up to the white large ring to prevent the tube assembly from slipping through. Insert the red rods into the three outer of three holes of the white large ring, rotate them such that the feet do not contact with any object on the magnet surface. Should any particular item get in the way, rotate a foot through any angle to see if this outer hole pattern is acceptable for your magnet or magnets. This will give the chamber the widest footprint and consequently the highest stability.
- IV. Insert the sample tube to be used for the experiment into the tube support ring containing two non-magnetic cords for support in the chamber when on the bench and for later insertion and extraction from the NMR magnet.
- V. Insert the sample tube into the appropriate spinner turbine for the system in use and set the sample depth in the normal way. (69mm below the spinner turbine Max for Varian / Agilent systems and 79mm below the spinner turbine Max for Bruker systems)
- VI. Insert the assembly into the explosion protection chamber which has been pre-adjusted for your particular magnet and is now standing on the bench in preparation for sample filling and pressure connection to the pressurizing gas source. Use the two cord support rings which fit over two of the three leg adjustment screws ensuring the cord fits into the screw slot or simply wind enough cord around the thumb screw thread to make sure the cord will not unravel. The second cord is simply a backup for the first cord but we recommend both be used as illustrated.
- VII. Once the sample tube has been pressurized to whatever pressure is to be used, it can then be disconnected from the pressure source. In the case of tethered pressure experiments the pressurizing tubing can be left connected to the sample tube assuming it's flexible and long enough to be inserted into the magnet all the way to the NMR measurement position.
- VIII. **It would be wise** to take the tube to the maximum planned measurement pressure **outside of the magnet** to make sure it can indeed take the planned pressure even if that particular tube should be able to take more pressure than planned. If not it simply explodes but with little to no material damage and definitely no user damage. The explosion protection chamber is dramatically cheaper than the NMR probe so better to damage this item than the NMR probe. No doubt the NMR instrument owner will agree wholeheartedly.
- IX. Hold the entire assembly in your hand gripping both cords and release the two ring connections to the two adjustment screws. The tube depth inside the protection chamber is now set by just how much of the cord is allowed to enter the top of the protection tube. Place the entire assembly on the magnet and carefully lower the tube assembly until it reaches the measurement position in the NMR magnet. The table tennis ball prevents the cords from falling into the magnet and can be draped around any suitable device in the vicinity, say the LN2 fill ports or similar.

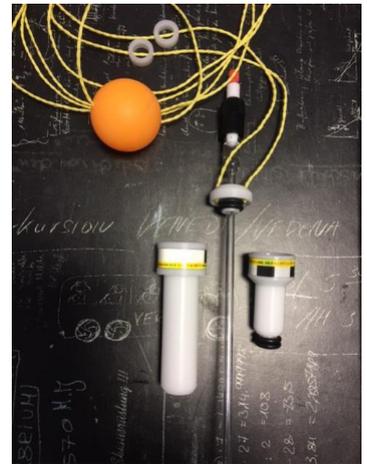


Fig. 4, sample tube inserted in tube connection, with separate spinner turbines



Fig. 5, completed standard setup. Note the cords tied to the screws

Should you have any comments about this or any of our other products, be they good or bad, we would greatly appreciate hearing from you!