LIGHT SENSITIVE SAMPLE TUBES

For containment of NMR samples that are sensitive to light. Constructed from Pyrex glass, the tubes are subjected to a high temperature ion exchange reaction to impart the light blocking properties to the glass surfaces.

In order to form stronger bonding, a special optical coating technique is used (as opposed to the usual amberization of other tubes found on the market). A maximum blocking per cent results for wavelengths <600nm.

Results may vary and depend on the nature of the sample.



Catalog Number	Usage MHz	Tube OD	Tube Length
NE-UP5-LS-7	Up to 700	5mm	7" (178mm)
NE-H10-LS-7	Up to 500	10mm	7" (178mm)





SCREW THREAD SAMPLE TUBES

The convenience of a screw cap - the security of a Teflon / glass seal. Each tube includes an open-top cap and a Teflon / Silicone septum. A 22 gauge, standard point needle is recommended for a clean puncture. To maintain spinning quality, choose the tube length that places the threads closest to the spinner. Optional lengths are available on special order.

Application Note:

These tubes are ideally suited for glove box preparation of air or moisture sensitive samples. The screw cap provides easy access for kinetic studies. Another proven application has been tracking the evolution of hydrolysis products within bioabsorbable materials.

Catalog Number	Usage MHz	Tube OD (mm)	Cap Size (mm)	Length (mm)
NE-HL5-ST-158	400-500	5	8	158
NE-UL5-ST-158	500-700	5	8	158
NE-HL5-ST-168	400-500	5	8	168
NE-HP5-ST-168	400-500	5	8	168
NE-UL5-ST-168	500-700	5	8	168
NE-UP5-ST-168	500-700	5	8	168
NE-H10-ST-178	400-500	10	15	178

Extra Caps and Septums

Additional screw caps and septums are conveniently packaged for use with the Screw Thread sample tubes. White silicone rubber lined solid caps resist most solvents; Teflon lined solid caps (see below) are used where solvent attack occurs with the white silicone rubber. The open-top cap and Teflon / Silicone septum combine to give the most inert closure with needle access.



Catalog Number	Description	Cap Size (mm)	Thread
3008	Cap, Open-top	8	8-425
3015	Cap, Open-top	15	15-425
3108	Cap, Solid Top, Silicone white rubber liner	8	8-425
3108T	Cap, Solid, Teflon liner	8	8-425
3115	Cap, Solid Top, Silicone white rubber liner	15	15-425
3208	Septum, Teflon / Silicone	8	-
3215	Septum, Teflon / Silicone	15	-

CONTROLLED ATMOSPHERE VALVE (CAV) SAMPLE TUBE

A linear valve system for performing controlled atmosphere experiments by NMR. The concentric valve mechanism consists of a sturdy, precision machined TFE plug with a Viton o-ring seal. A glass vacuum adapter is supplied with each system. It is secured to the top of the valve with a press fit sealed by an o-ring.

Application Note:

To use the CAV, the glass adapter is first secured to the vacuum line. With the valve attached and in the open position, vacuum transfers can be made to and from the sample tube. Once the proper sample environment is attained, the valve can be closed and the system can be removed from the vacuum line. The process is repeated to retrieve the sample.

This system is very useful in the study of organometallics or other moisture and air sensitive samples. Note: It is not recommended to use ketones, aldehydes, ethers or aliphatic esters with Viton rubber. Aegis o-rings are available for use with these compound types (see below) and must be ordered separately.

Note: To the catalog number, add the length "L" (XXX), in mm, that places the valve directly on top of the spinner. This is necessary to maintain good spinning quality. **Example: NE-CAV-170 indicates L=170mm**.

Catalog Number	Sample Tube	Wall (mm)	Usage MHz
NE-CAV5-XXX	NE-HP5	0.38	400-500
NE-CAV5-M-XXX	NE-HP5-M	0.77	300-400
NE-CAV5-H-XXX	NE-HP5-H	1.4	300-400
NE-CAV10-XXX	NE-H10	0.46	300-500
NE-CAV10-M-XXX	NE-H10-MW	1.00	300-400
NE-CAV10-H-XXX	NE-H10-HW	1.7	300-400
NE-CAV5-Q-XXX	NE-HQ5	0.50	300-500
NE-CAV10-Q-XXX	NE-HQ10	0.50	300-500

Spinner Position when set in depth gage

PRESSURE VALVE (PCAV) SAMPLE TUBE

Constructed as the CAV Sample Tube above; except, the PCAV Sample Tube allows for easy connection to a gas manifold with a nut and ferrule for 1/16" tubing. The extended plug tip gives finer control of flow. When the valve is completely closed, the gas line is easily disconnected from the top of the valve body. The system has been successfully tested to **20 atmospheres** with the 5mm **medium-wall** tube.

Individual results may vary and cannot be guaranteed.

The above "Notes" also apply here for o-ring chemical compatability and the "L" Dimension.

Catalog Number	Sample Tube	Wall (mm)	Usage MHz
NE-PCAV5-XXX	NE-HP5	0.38	400-500
NE-PCAV5-M-XXX	NE-HP5-M	0.77	300-400
NE-PCAV5-H-XXX	NE-HP5-H	1.4	300-400
NE-PCAV10-XXX	NE-H10	0.46	300-500
NE-PCAV10-M-XXX	NE-H10-MW	1.00	300-400
NE-PCAV10-H-XXX	NE-H10-HW	1.7	300-400

CAV / PCAV Spare Parts

Catalog Number	Description	Tube
NE-CAV5-G-XXX	Valve Body, only, Thin-Wall Tube	CAV, PCAV
NE-CAV5-M-G-XXX	Valve Body, only, Medium-Wall Tube	CAV, PCAV
NE-CAV5-H-G-XXX	Valve Body, only, Heavy-Wall Tube	CAV, PCAV
NE-CAV-P	Plug, TFE with knob and sealing O-ring	CAV
NE-PCAV-P	Plug, TFE with knob, o-rings and fittings	PCAV
NE-CAV-SO	Sealing O-ring, Viton	CAV, PCAV
NE-CAV-SO-A	Sealing O-ring, Aegis	CAV, PCAV
NE-CAV-RO	Retaining O-ring, Viton, to retain valve knob	CAV, PCAV
NE-CAV-RO-A	Retaining O-ring, Aegis, to retain valve knob	CAV, PCAV
NE-CAV-A	Adapter, only, glass	CAV
NE-CAV-JA	Adapter, with 24/40 outer joint, glass	CAV
NE-PCAV-FN	Ferrule/Nut Set, one ferrule and one nut	PCAV
References: Constrained Geometry (Chromium Catalysts for Olefin Polymerization: Y. Liang, G.P.A. Yap, A.L. Rheingold, K.H. The	opold.

Constrained Geometry Chromium Catalysts for Olefin Polymerization; Y. Liang, G.P.A. Yap, A.L. Rheingold, K.H. Theopold, Organometallics 1996, 15, 5284

[(Ph)₂ nacnac]MCl₂(THF)₂ (M=Ti, V, Cr)-- A New Class of Homogeneous Olefin Polymerization Catalysts Featuring β-Diiminate Ligands, W.K. Kim, M.J. Fevola, L.M. Liable-Sands, A.L. Rheingold, K.H. Theopold *Organometallics* 1998, 17, 4541







GEL NMR SAMPLE TUBES and ACCESSORIES

The Gel Apparatus and accessories are designed for RDC studies of uniformly aligned proteins and detergent micells by NMR. The precision Gel Chamber casts consistently uniform gels. Samples can then be uniformly stretched, with repeatability, and loaded into the active area of the sample tube, assuring maximum sample concentration in the coil. The precision of the apparatus will give repeatable sample conditions.

The accessories described here are designed for forming and stretching gel samples of 6mm, 5.4mm and 5mm diameter to the 4.2mm ID of the sample tube as it is being loaded into the tube. All hardware components are easily cleaned and are reusable.

GEL KITS

These kits are designed for the first-time user and provide all of the hardware items needed to cast and stretch the samples. For the science of RDC measurements as related to gels and proteins, one must search the many papers available, only a few of which are cited on **Page 18**. Additional and replacement components are all available separately.

Gel NMR Starter Kits

(includes a gel press assembly, all plugs and accessories, and a special gel NMR sample tube)

Add "-Br" suffix to Cat. Nos. for Bruker that requires a short end plug (8mm Length).

Gel Diameter		500 MHz	700 MHz	900+ MHz
6mm to 4.2mm	Cat. No.	NE-373-A-6/4.2	NE-373-B-6/4.2	NE-373-C-6/4.2
5.4mm to 4.2mm	Cat. No.	NE-373-A-5.4/4.2	NE-373-B-5.4/4.2	NE-373-C-5.4/4.2
5mm to 4.2mm	Cat. No.	NE-373-A-5/4.2	NE-373-B-5/4.2	NE-373-C-5/4.2

Additional / Replacement Parts

Sample Tubes

	500 MHz	700 MHz	900+ MHz
Cat. No.	NE-HP5-GT-7	NE-UP5-GT-7	NE-SP5-GT-7

Plugs and Accessories (for all Starter Kits)

Catalog Number	Description
NE-370-A-5	Gel End Plug, Ultem
NE-370-A-5-S	Gel End Plug, Ultem, Short
NE-370-A-5-O	Gel End Plug Spare O-Ring, Buna-N
NE-370-B-5	Gel Top Plug, Ultem
NE-371	Extraction Rod, for end plug
NE-341-5	Support Rod, Kel-F, for top plug
NE-312-5-GT	Support Cap, PTFE





GEL PRESSES and ADDITIONAL / REPLACEMENT COMPONENTS

Some operational suggestions to help getting started

The gel press should be assembled as shown in the sketch. Please note that the arrow on the body of the Gel Chamber must point downward towards the thread of the Funnel. This will provide a smooth transition through which the gel will pass.

It is also suggested that several "gel only" samples be tried first to get the feel of how the press will operate.

It is very important that the Gel sample tube be oriented in the correct way. Since both ends of this tube are opened, the "bottom end" with the special finish is opposite to the catalog mark on the tube. This is the end that must be inserted into the bottom of the Funnel. The special end finish is necessary to allow the End Plug to seal the tube without breaking it.

Gel Presses

(includes piston driver, cap, piston, gel chamber and funnel)

Size	6mm to 4.2mm	5.4mm to 4.2mm	5mm to 4.2mm
Cat. No.	NE-372-6/4.2	NE-372-5.4/4.2	NE-372-5/4.2





Additional / Replacement Parts

Pistons, PTFE, w/Buna O-Ring

Size	6mm to 4.2mm	5.4mm to 4.2mm	5mm to 4.2mm
Cat. No.	NE-372-C-6	NE-372-C-5.4	NE-372-C-5

Piston Spare O-Rings, Buna-N				
Cat. No.	NE-372-C-6-O	NE-372-C-5.4-O	NE-372-C-5-O	

Gel Chambers, PTFE

Size	Size 6mm to 4.2mm		5mm to 4.2mm	
Cat. No. NE-372-D-6		NE-372-D-5.4	NE-372-D-5	

Funnels, PTFE

Size	6mm to 4.2mm	5.4mm to 4.2mm	5mm to 4.2mm	
Cat. No.	NE-372-E-6	-	-	
Cat. No		NE-372-E-5.4	NE-372-E-5	

Piston Driver and Cap

(for all Gel Presses)

Cat. No.	Description
NE-372-A	Piston Driver, Brass
NE-372-B	Cap, Brass
NE-372-B	Cap, Brass

Some Reflection on Gel NMR Sample Preparation

The information presented below is intended to be used as a guide in developing your procedure and not as a set of specific instructions. A technique needs to be developed by the user that works best, and the first attempt will be a learning experience.

What stretch ratio can give the optimal size of RDC?

Version 6 to 4.2mm: works well with proteins like protein G. Version 5.4 to 4.2mm: generally good for strong aligning proteins. Version 5 to 4.2mm: generally good for proteins that are both large and elongated.

Experimentation with different stretch ratios may be necessary to determine which one is best for the samples at hand.

Allow 2-3 days for complete sample preparation.

Some gel solutions may be basic and/or contain unreacted components which could affect the stability of the protein if it were added to the initial solution.

Approximately 250µl of gel solution would make a sample column height of about 20mm.

Assuming protein has not been added, seal the bottom of the Gel Chamber (parafilm) and fill with gel solution. Allow to polymerize for at least 1-2 hours. The seal (parafilm) can then be taken off the chamber.

Carefully remove the gel with the use of the Piston and an appropriate tool for pushing it through the chamber. Be careful not to scratch the inner surface of the chamber.

Dialyze the gel against deionized water overnight to remove unreacted chemicals.

While the gel is still soft, it may be cut to some appropriate length. Allow for the length of the Piston and for some air space between it and the surface of the gel.

As a guide line, the gel length should be about $\frac{1}{3}$ the length of the Gel Chamber allowing room for an air space and the Piston. The air space will allow more of the gel to be expelled.

Dry overnight at room temperature or for some hours at an elevated temperature (40-45°C). The gel will become somewhat rigid and smaller in diameter.

Place the "dried" gel back into the Gel Chamber, seal the bottom (parafilm) and add protein until the gel swells to the full diameter of the chamber. Let it sit for 24 hours or longer to allow the protein to diffuse in.

Remove any excess protein that has not been diffused into the gel.

Assemble the Gel Press and load the gel into the sample tube.

The Piston will only go as far as the top of the actual funnel area which may still contain some gel. With the Gel Chamber removed from the Funnel, the action of an auto-pipet may be used to apply enough air pressure to expel the remaining gel.

Seal the bottom of the sample tube by gently pressing the Gel End Plug in to place, in such a manner as to avoid trapped air bubbles (the Extraction Rod is used only to remove the Gel End Plug from the sample tube).

Insert the Gel Top Plug on top of the gel column with the Support Rod.

Ideally, the sample column should be centered in the receiver coil area. It may take a few trials to find the optimum sample position for this application in the probe.

Some references: Bax, A. Tjandra, N. (1997) J. Biomol. NMR, **10**, 289-292 Chou, J.J., Li, S., Klee, C.B. and Bax, A. (2001) Nat. Struct. Biol., **8**, 990-997 Chou, J.J., Gaemers, S., Howder, B., Louis, J.M. and Bax, A. (2001) J. Biomol. NMR, **21**, 377-382 Tycko, R., Blanco, F.J. and Ishii, Y. (2000) J. Am. Chem. Soc., **122**, 9340-9341

TIP-OFF SEALING SYSTEMS APPLICATION NOTES

FLAME-SEALING NMR SAMPLE TUBES:

There are two basic ways to prepare flame-sealed samples. One method, suitable for both Pyrex and non-Pyrex tubes, is to use a "vacuum adapter" to attach the tube to the vacuum line via a hose or other mechanical means. Sample tubes are held in place by an o-ring compressed finger tight by a threaded bushing.

The second way, for Pyrex tubes only, starts with "flame-sealing the sample tube" to the port on the vacuum line. Although more difficult to attain a good seal, this method ensures the integrity of the sample system and allows for vacuum transfers under very controlled conditions. Many researchers, adept at glassblowing, prefer this method.

FLAME-SEALING TECHNIQUES:

Hand-held micro torches (available from major lab supply dealers) provide a small, yet very hot, flame to easily seal tubes. A very uniform, concentric seal is important to maintain good spinning quality. The way to accomplish this is by heating the glass evenly around the tube. The best seals are made with pre-constricted tubes. Increased glass thickness in the seal area reduces the chance of the hot glass imploding due to the vacuum in the tube. It is important that only the seal area be heated. Finally, the key to making good seals is practice.

FREEZE / THAW DEGASSING:

This method is often used to remove Oxygen from samples. However, with aqueous samples, it can be difficult to complete the process without breaking the tube. One effective technique is as follows: Once the sample is loaded and the tube is secured under vacuum, immerse the tube into the freezing medium (liquid N_2 , dry ice/alcohol) very slowly so that the sample freezes from the bottom, up. Likewise, in the thaw cycle, remove the tube very slowly so that the sample melts from the top, down. The use of a small cylindrical lab dewar (see **Page 20**) and a lab jack greatly aid in this process. Some practice is recommended before valuable samples are used.



TIP-OFF SAMPLE TUBES

Both Pyrex and non-Pyrex tubes are offered. Pyrex tubes are necessary if you need to reopen the tube and do additional glassblowing. The non-Pyrex tubes are ideal for samples that will be archived. In both cases, a 50mm section of 5mm OD tubing is sealed to a 7-inch sample tube. The extra tubing allows the tube to be secured in the Tip-Off Adapter (see **Page 20**) with ample length for safe flame sealing. A uniform constriction, with a 2mm ID, is placed just above the 7-inch length.

Catalog Number	Usage MHz	Tube OD (mm)	Wall (mm)	Glass Type
NE-HL5-TTO	400-500	5	0.38	non-Pyrex
NE-UL5-TTO	500-700	5	0.38	non-Pyrex
NE-HP5-TTO	400-500	5	0.38	Pyrex
NE-HP5-M-TTO	300-400	5	0.77	Pyrex
NE-UP5-TTO	500-700	5	0.38	Pyrex
NE-SP5-TTO	700-900+	5	0.38	Pyrex
NE-H10-TTO	400-500	10	0.46	Pyrex

ACCESSORIES FOR TIP-OFF SAMPLE TUBES

TIP-OFF ADAPTER, basic

The Tip-Off Adapter consists of an internal glass thread with a mating Nylon bushing fitted with a stainless steel heat shield. The shank of the adapter ($\frac{1}{2}$ " OD x 4" long) is secured to the vacuum manifold. Sample tubes are held in place by an extra thick FETFE o-ring compressed by the threaded bushing, finger tight. Tested to 10^{-7} Torr.

Catalog Number	Description
NE-250	Adapter, complete with one bushing and o-ring
NE-250-B	Bushing only, with heat shield and two o-rings
NE-250-O	O-Ring, FETFE, extra thick



TIP-OFF ADAPTER / VALVE

The Tip-Off Adapter / Valve features a high vacuum valve with a sturdy Teflon plug and two Teflon-clad o-rings.

The bulbous plug tip makes an inert and positive seal to isolate the vacuum line. The valve has a 0-4mm orifice.

The Tip-Off Adapter / Valve can be mounted easily with a clamp to a lab rack.

Catalog Number		Description
	NE-253-5	Tip-Off Adapter / Valve, complete
	NE-253-B	Bushing only, Nylon with heat shield and two o-rings
	NE-253-O	O-Ring, FETFE, extra thick
	NE-253-G	Glass Body, only
	NE-253-P	Teflon Plug / Cap, only

TIP-OFF DEWAR and CAP

A sturdy Pyrex dewar, fully silvered, with an aluminum base. Measuring $48 \text{mm ID } \times 150 \text{mm}$ internal depth, this dewar has a full capacity of 250ml. It is designed for use in the freeze/thaw method of degassing NMR samples. The large ID allows for easy inspection of the process so that it may be controlled more exactly.

An optional styrofoam dewar cap, sold separately, greatly reduces boil-off.

Catalog Number	Description
NE-8025	Dewar, 48mm ID x 150mm depth, 190mm OAL
NE-8348	Cap, styrofoam, with vent slot





NON-GLASS SAMPLE CELLS

A precision, non-glass sample cell for the study of Boron, Fluorine, Silicon and other types of samples that may be deemed "hazardous". Available in 5mm and 10mm versions, these precision machined cells fit smoothly into the respective standard thin wall sample tubes. A cap, made of the same material, screws into the top of the cell, providing a positive closure and the means for removing the cell with a threaded rod.

Boron and Silicon samples can be studied by supporting the cell in a glass sleeve so that only the cell is in the receiver coil area. With the cap secured to the top of the cell, a threaded rod is screwed into the top of the cap. This unit is slipped through a glass sleeve and is held in place by the support rod with a modified tube cap. Depending on the positioning of the poly cell in the RF coil area, it may be desirable to cut off excess length of the support rod.

Tests with Chloroform-d/TMS samples have shown line widths comparable to using the sample tube alone. Some additional shimming may be necessary. Individual results may vary and are dependent upon how well the instrument is tuned. We can offer no guarantee on performance and safety, especially with "hazardous" samples.

SPECIFICATIONS	
Sample Cell, 5mm version:	3.3mm ID x 4.1mm OD x 90mm long, 80mm maximum sample height
Sample Cell, 10mm version:	7.5mm ID x 8.9mm OD x 90mm long, 80mm maximum sample height
Cap, 5mm version:	20mm long with 4-40 internal thread
Cap, 10mm version:	20mm long with 1/4-20 internal thread



SAMPLE CELL with CAP

Catalog Number	Tube OD	Material	Useful Temp Range	Volume @ 60mm Sample Height
NE-362-T-5	5	PTFE	-73 to +204°C	304 µl
NE-362-T-10	10	PTFE	-73 to +204°C	2.6 ml
NE-362-K-5	5	Kel-F	-240 to +204°C	304 µl
NE-362-K-10	10	Kel-F	-240 to +204°C	2.6 ml
NE-362-P-5	5	P/P	0 to +80°C	304 µl
NE-362-P-10	10	P/P	0 to +80°C	2.6 ml

GLASS SLEEVE and SUPPORT ROD

Catalog Number	Description		
NE-205-5	Glass Sleeve, precision, 5mm OD x 120mm long		
NE-205-10	Glass Sleeve, precision, 10mm OD x 120mm long		
NE-341-5	Support Rod, Kel-F, " x 8", 4-40 thread		
NE-341-10	Support Rod, PVC, ¼" x 8", ¼-20 thread		



EXTRA SAMPLE CELLS and CELL CAPS

Matarial	Version	Sample Cell	Сар
Wateria	version	Cat.No.	Cat.No
PTFE	5mm	NE-360-T-5	NE-361-T-5
PTFE	10mm	NE-360-T-10	NE-361-T-5
Kel-F	5mm	NE-360-K-5	NE-361-K-5
Kel-F	10mm	NE-360-K-10	NE-361-K-10
P/P	5mm	NE-360-P-5	NE-361-P-5
P/P	10mm	NE-360-P-10	NE-361-P-10



COAXIAL INNER CELLS

A precision inner cell for use with 5mm and 10mm thin-wall NMR tubes. Ideal for "external" reference solvents or as a "micro tube". For use with 7" outer tubes. Both Bruker and Varian versions are available.

Catalog Number	Tube / Probe	OD	Stem Length	Capacity	Sample Capacity
NE-5-CIC	5mm / Bruker	2mm	50mm	60 µl	530 µl
NE-5-CIC-V	5mm / Varian	2mm	60mm	72 µl	636 μl
NE-10-CIC	10mm / Bruker	3mm	50mm	225 μl	2.8ml
NE-10-CIC-V	10mm / Varian	3mm	60mm	270 μl	3.36ml

TEFLON LINERS

An inexpensive way to handle corrosive samples that may react with the glass sample tube. The thin wall, round bottom liners are supplied with a Teflon plug closure. For use in standard thin wall 5mm and 10mm sample tubes. Results may vary and some higher order shimming may be necessary. For a precision alternative, see our **Non-Glass Sample Cells** on **Page 21**.

Catalog Number	Tube	Description		
NE-350-5	5mm x 7"	Round bottom, 8" long with solid Teflon plug; Volume @ 50mm height is @ 0.35ml		
NE-350-10	10mm x 7"	Round bottom, 8" long with solid Teflon plug; Volume @ 50mm height is @2.0ml		



HAZARDOUS NMR SAMPLE TUBE SYSTEM

An effective way to run 5mm NMR samples deemed "hazardous" in some respect. This system was designed to significantly enhance the high field spectral quality, while maintaining a measure of safety in running these samples. The results have shown a marked improvement over the use of traditional Teflon liners.

A specially designed Teflon plug seals the heavy wall (0.75mm) glass inner tube which then fits completely into our NE-HP5-7, 5mm sample tube. The inner tube has a sample volume of 125μ I for a 30mm sample height. A longer version of the 5mm tube cap seals the system.

Once the instrument is shimmed for the sample tube system, additional samples can be shimmed easily. At 500 MHz, spectral quality can be comparable to that of routine samples. Actual performance will vary and is dependent upon the condition of the instrument. The improved quality of spectra is a viable trade-off for the careful handling required for such samples. However, there is no expressed or implied warranty against breakage. An all-Pyrex system, not recommended for Fluorides.

Catalog Number	OD(mm)	Usage MHz	Description
NE-HP5-HSTS	5	Up to 500	Complete system, inner tube with plug, outer tube with cap
NE-HSTS-I	4	-	Inner tube with Teflon plug