Our tubes:
JEOL Cat. # NM-05410ST32 (Zirconia, ZrO2).

NM-02153ST4

NM-05420ST4

NM-02622ST32

NM-05410ST32

NM-02730SHT25

SOLID-STATE NMR SAMPLE TUBE SET

For the proper use of the instrument, be sure to read this instruction manual. Even after you read it, please keep the manual on hand so that you can consult it whenever necessary.

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JEOL

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The following precautions are important which, if not followed, may result in damage to the instrument itself.

PRECAUTIONS FOR USE

■ Precautions for the sample tube overall

- **Do not use anything except water or ethanol when cleaning the sample tube.**
  Use of organic solvents may distort or deteriorate the sample tube.
- **Do not operate out of the temperature range defined in the specification.**
  If operated out of the temperature range, the cap may come off during operation. This may cause spinning malfunction and damage to the probe.
- **If there is any damage, contamination, distortion or abrasion of the sleeve, spinning cap or bottom cap, do not use it.**
  This may cause spinning malfunction, explosion of a sample tube, and damage to the probe.
  Confirm that there is no sleeve scratch (☞Sect. 7.1).
  The abrasion of the turbine flutes cause lower spinning efficiency and disable the acceleration of spinning.
- **Do not use the sample tube if the sleeve and cap are fit loosely.**
  If the fitting part of the cap wears, the cap may fit to the sleeve loosely. This may cause explosion of the sample tube and damage to the probe when spinning within the probe.
- **Do not rub the marking of the bottom cap for spinning detection with a cloth or gauze.**
  If the marking is scraped off, it may cause spinning malfunction; the sample tube may explode, or the probe may be damaged.

■ Precautions for sampling

- **Do not use a liquid or semisolid sample in a sample tube.**
  The sample tube is only for solid-state samples and does not have the highly airtight structure required for liquid or semisolid samples. If you use liquid or semisolid samples, the sample may leak out of the sample tube, causing spinning error and damage to the probe.
- **Do not use anything with a hard tip, such as a pair of tweezers or needle.**
  It will damage the sample tube and may cause spinning malfunction.

■ Precautions for spinning the sample tube

- **When spinning the sample tube, first confirm the marking on the bottom cap.**
  If the marking has been worn off, a spinning detection error may result; the sample tube may explode, or the probe may be damaged.
  For the 4 mm and 3 mm bottom caps, repaint it as necessary, referring to sect. 5.2.1.
  For the 2.5 mm bottom cap, replace the bottom cap with one having a clear marking.
- **Before spinning the sample tube, confirm that there is no sleeve scratch.**
  Using a sample tube with sleeve scratch may explode the sample tube, causing probe damage.
- **Do not spin a sample tube at speeds exceeding the maximum spinning speed.**
  It may cause explosion of the sample tube and damage to the probe.
- **Do not use the sample tube if there is a gap between the sleeve and cap.**
  If there is a gap between the sleeve and cap, the cap may drop off when spinning. This may damage the sample tube and the probe.

■ Precautions for applying the spinning detection marker

- **Make sure to use a black permanent ink pen.**
  Using other types of markers may cause detection error, the sample tube may explode, or the probe may be damaged.
- **When spinning the sample tube at high-speed after remarking, check to see that the marking does not come off at the desired spin rate.**
  The marking may come off during high-speed spinning, depending on the type of black permanent ink pen.
- **Before using this sample tube, confirm that the marker is dry completely.**
  It may cause malfunction of spinning detection. In addition, it will contaminate the inside of the spinner causing malfunction of spinning. Then, explosion of the sample tube and damage of the probe may occur.

■ Precaution for determining the sleeve scratch

- **Make sure to use your nail to perform sleeve scratch inspection.**
  If a blade or tool is used, the sample tube may be damaged.

1 GENERAL

This product is a sample tube set used for measuring solid-state nuclear magnetic resonance (NMR).

2 SPECIFICATIONS

Items		NM-02153ST4 NM-05420ST4	NM-02622ST32 NM-05410ST32	NM-02730SHT25
Sample tube size	Outer diameter	4 mm	3.2 mm	2.5 mm
	Inner diameter	2.6 mm	2.2 mm	1.7 mm
	Sleeve length	18 mm	18 mm	12.4 mm
Sample volume	When using spacers	69 µL	49 µL	17 µL
	When not using spacers	37 µL	27 µL	9.5 µL
Materials	Sleeve	Zirconia (ZrO_2)		
	Spinning cap	Vespel		
	Bottom cap	PEEK		Vespel
	Spacer	PCTFE[*1]		
Maximum spinning speed[*2]	At room temperature	18 000 Hz	22 000 Hz	32 000 Hz
	At high temperature VT	16 000 Hz	19 000 Hz	28 000 Hz
	At low temperature VT	14 000 Hz	17 000 Hz	25 000 Hz
Guaranteed spinning speed (at room temperature)		17 000 Hz		
Temperature range[*3]	When using spacers	–60 to +80 °C		
	When not using spacers	–60 to +150 °C		

This product is consumable. To maintain performance and safety, be sure to perform inspection and parts replacement before use following the instructions of this manual (☞ Chap. 6).
However, when a failure or a damage occurs within 90 days after installation, if it is clearly caused by defects in material or workmanship, JEOL RESONANCE will repair or exchange without charge.

⚠ CAUTION

Be sure to read this manual carefully and follow the instructions. Also, be sure to perform inspection and maintenance when any scratch or damage found on this product (☞ Chap. 6).
If you don't follow the instructions of this manual, you might be injured and the instrument might be damaged.

[*1] PCTFE : Poly Chloro Tri Fluoro Ethylene
[*2] Comparing the specification value of the probe, the slower value is applied.
[*3] Comparing the specification value of the probe, the lower value is applied as an upper limit temperature and the higher value is applied as a lower limit temperature.

3 CONFIGURATIONS

	NM-02153ST4 NM-02622ST32 NM-02730SHT25	NM-05420ST4 NM-05410ST32
Sample tube set	5 sets	1 set
Sleeve	1	
Spinning cap	1	
Bottom cap	1	
Spacer	2	
Spare parts	2 sets	–
Spinning cap	1	–
Bottom cap	1	–
Instruction manual	1	

4 DESCRIPTION OF PARTS

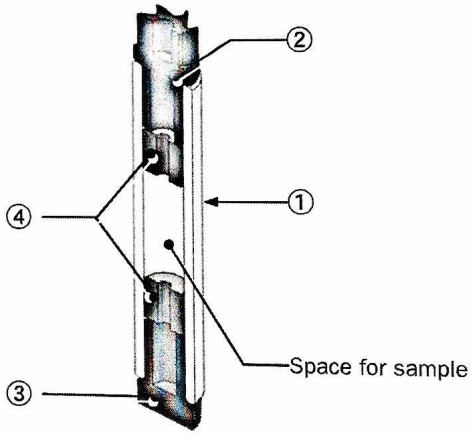

Fig. 1 Components of a sample tube (cross-sectional view)

① Sleeve

The sleeve is the outer wall of the sample tube. It will be filled with sample. It is made of an extremely hard ceramic material called Zirconia (ZrO_2).

② Spinning cap

The spinning cap is used to plug the top of the sample tube, and is inserted into sleeve by press fitting. The spinning cap has turbine flutes that are precisely machined so that the sample tube can be spun at a high speed by blowing an air-jet into the flutes. The spinning cap is made of Vespel (polyimide resin).

③ Bottom cap

The bottom cap is used to plug the bottom of the sample tube, and is inserted into sleeve by press fitting.

The topside surface of the bottom cap has a spinning detection marking on half of the side surface.

- 4 mm and 3.2 mm bottom caps
  4 mm and 3.2 mm bottom caps are made of PEEK.

✍ The marking of the bottom cap made of PEEK is painted with a black permanent ink pen. If the black-marking has scraped off, repaint it by yourself (☞ Sect. 5.2.1).

- 2.5 mm bottom cap

2.5 mm bottom cap is made of Vespel, the same as the spinning cap.

✍ The marking of the bottom cap made of Vespel is sputtered with gold. If the gold-marking has scraped off, replace the bottom cap with one having a clear marking.

—CAUTION—

- **Do not rub the marking of the bottom cap for spinning detection with a cloth or gauze.**
  If the marking is scraped off, it may cause spinning malfunction; the sample tube may explode, or the probe may be damaged.
- **When spinning the sample tube, first confirm the marking on the bottom cap.**
  If the marking has been worn off, a spinning detection error may result; the sample tube may explode, or the probe may be damaged.
  For the 4 mm and 3 mm bottom caps, repaint it as necessary, referring to sect. 5.2.1.
  For the 2.5 mm bottom cap, replace the bottom cap with one having a clear marking.

④ Spacer

The spacer is used to adjust the sample position relative to the sample coils, which detect NMR signals. It is made of fluorination resin called PCTFE (Poly Chloro Tri Fuluoro Ethylene). The spacer has a hole in the center that is used for gas-venting and removing the spacer from the sleeve.

5 OPERATION

☞ Be sure to refer to Chap. 6 "■Inspection before use" and execute inspection.

—CAUTION—

- **Do not use anything except water or ethanol when cleaning the sample tube.**
  Use of organic solvents may distort or deteriorate the sample tube.
- **Do not operate out of the temperature range defined in the specification.**
  If operated out of the temperature range, the cap may come off during operation. This may cause spinning malfunction and damage to the probe.
- **If there is any damage, contamination, distortion or abrasion of the sleeve, spinning cap or bottom cap, do not use it.**
  This may cause spinning malfunction, explosion of a sample tube, and damage to the probe. Confirm that there is no sleeve scratch (☞ Sect. 7.1).
  The abrasion of the turbine flutes cause lower spinning efficiency and disable the acceleration of spinning.
- **Do not use the sample tube if the sleeve and cap are fit loosely.**
  If the fitting part of the cap wears, the cap may fit to the sleeve loosely. This may cause explosion of the sample tube and damage to the probe when spinning within the probe.

5.1 Sampling

⚠ CAUTION

- **Do not pack a sample in the sample tube that might ignite or explode by temperature, mechanical shock, or pressure.**
  The sample tube might break and explode, and you might be injured by the broken sample-tube pieces.

- **Make sure to use the corresponding Solid Sampling Set and follow the procedures in the instruction manual.**
  Installing or removing the cap using liquid nitrogen instead of the Solid Sampling Set may cause the cap to jump out due to expansion of inner air, and may cause injury. Also, it may cause explosion of a sample tube.

—CAUTION—

- **Do not use a liquid or semisolid sample in a sample tube.**
  The sample tube is only for solid-state samples and does not have the highly airtight structure required for liquid or semisolid samples. If you use liquid or semisolid samples, the sample may leak out of the sample tube, causing spinning error and damage to the probe.
- **Do not use anything with a hard tip, such as a pair of tweezers or needle.**
  It will damage the sample tube and may cause spinning malfunction.

☞ For the setup procedure of sample tube, refer to the instruction manual of the probe which are using.

5.2 Spinning the Sample Tube

☞ For MAS operation, refer to the instruction manual for the probe which are using, MAS Controller or MAS Valve Unit.

—CAUTION—

- **When spinning the sample tube, first confirm the marking of the bottom cap.**
  If the marking has been worn off, a spinning detection error may result; the sample tube may explode, or the probe may be damaged.
  For the 4 mm and 3 mm bottom caps, repaint it as necessary, referring to sect. 5.2.1.
  For the 2.5 mm bottom cap, replace the bottom cap with one having a clear marking.
- **Before spinning the sample tube, confirm that there is no sleeve scratch.**
  Using a sample tube with sleeve scratch may explode the sample tube, causing probe damage.
- **Do not spin a sample tube at speeds exceeding the maximum spinning speed.**
  It may cause explosion of the sample tube and damage to the probe.
- **Do not use the sample tube if there is a gap between the sleeve and cap.**
  If there is a gap between the sleeve and cap, the cap may drop off when spinning. This may damage the sample tube and the probe.

5.2.1 Applying the spinning detection marker on the 4 mm and 3.2 mm bottom cap

- If the marking on the bottom cap, 4 mm or 3.2 mm, for detecting the spinning has scraped off, repaint it by yourself.
- Use a commercially available black permanent ink pen for the marker. The suitable markers for a marking are shown in the website below.
  http://www.j-resonance.com/en/topics/topic20141121/
- The marker will not scrape off by normal spinning. However, it may scrape off if it is rubbed with cloth or gauze. Make sure to check the marker before spinning and reapply it if necessary.

◆ Apply marker on the half of the side surface of the bottom cap as uniformly as possible (Fig. 2).

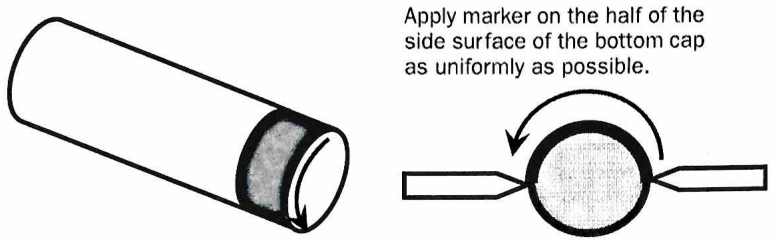

Fig. 2 Applying marker on the bottom cap

—CAUTION—

- **Make sure to use a black permanent ink pen.**
  Using other types of markers may cause detection error, the sample tube may explode, or the probe may be damaged.
- **When spinning the sample tube at high-speed after remarking, check to see that the marking does not come off at the desired spin rate.**
  The marking may come off during high-speed spinning, depending on the type of black permanent ink pen.
- **Before using this sample tube, confirm that the marker is dry completely.**
  It may cause malfunction of spinning detection. In addition, it will contaminate the inside of the spinner causing malfunction of spinning. Then, explosion of the sample tube and damage of the probe may occur.

6 MAINTENANCE

■ Inspection before use

The sample tube is very precisely machined. If it has a scratch or contamination, it will cause spinning error and damage of the instrument.

✍ Make sure to execute the following inspection before using a sample tube.

Period	Inspection items	Refer to
Before use	Scratch on the sleeve	☞ Sect. 7.1
	Scratch, contamination, distortion of spinning cap / bottom cap.	☞ Chap. 5, Sect. 5.2
	Spinning detection marking on the bottom cap	☞ Sect. 5.2.1

■ Parts replacement

To maintain the performance and safety of the instrument, periodical replacement of the parts is recommended.

Table 1 shows the parts to be periodically replaced. Replace it when any of "Guideline for replacement" is applicable.

To order the consumable parts, contact your JEOL RESONANCE sales or service office.

Table 1 Guideline of the parts replacement of the sample tube

Part name	Guideline for replacement
Sleeve	• When streaky damage is visible on the surface
Spinning cap	• When distortion of the turbine flutes is visible • When damage or cracks is visible on the fitting • When the fitting of the sleeve and the cap become loosed
Bottom cap	• When damage is visible on the bottom surface • When the spinning detection marking is worn off (only for 2.5 mm bottom cap) • When damage or cracks is visible on the fitting • When the fitting of the sleeve and the cap become loosed

7 REMARKS

7.1 Determining the Sleeve Scratch

—CAUTION—

Before spinning the sample tube, confirm that there is no sleeve scratch.
Using a sample tube with sleeve scratch may explode the sample tube, causing probe damage.

Depending on the usage of the sleeve, there may be a big scratch or black streak, or a small scratch that cannot be recognized visually. If there is a scratch on the sleeve, it does not matter how big or small the scratch is, there is a possibility that the sample tube will explode during the spinning.
It may be difficult to distinguish a scratch from a black streak. Determine by following the procedure below.

✍ Whenever possible, this inspection should be performed after removing the bottom cap and the spinning cap from the sleeve.

1. Prepare the tip of the fingernail as sharp as possible.

—CAUTION—

Make sure to use your nail to perform sleeve scratch inspection.
If a blade or tool is used, the sample tube may be damaged.

2. Trace the whole circumference parallel to the sleeve axis (Fig. 3).
After tracing one line, rotate the sleeve a little and repeat the tracing all around the lateral side.

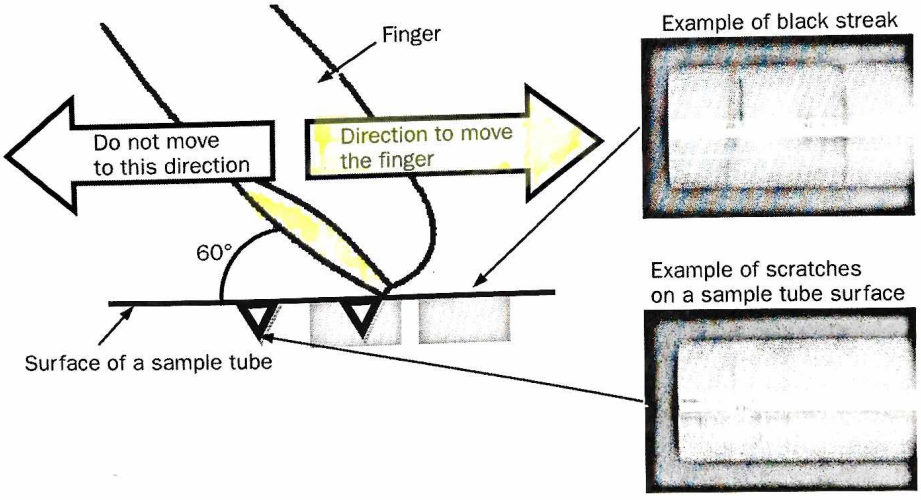

Fig. 3 Direction to trace with nail

3. Refer to "■Criteria for scratch" and distinguish scratches from streaks.

■ Criteria for scratch

● When the fingernail gets caught, or the surface feels uneven

There may be a scratch on the sleeve surface. Stop using it and exchange it for a new sleeve.

● When there is a black streak, but the fingernail does not get caught and the surface feels smooth

This sleeve still can be used. However, if the symptoms below are seen, exchange it with a new sleeve.

- There is an abnormal noise at the beginning and during spinning.
- The spin rate does not increase.
- The spinning is unstable.

✍ The black streak is regarded as the contamination which adhered to the surface of the sample tube. It is adhered between fine particles; it does not come off even when it is wiped.

✍ The black streak may appear during the spinning check before the sample tube delivery. Even if there is a black streak at the time of delivery, it does not affect the performance.

✍ If a new black streak appears after delivery of the sample tube, determine whether it is a scratch according to the criterion.

7.2 Background Signal of the Material

Table 2 shows the material, color and the main background signals observed by NMR experiment of each component. Due to the characteristic of the material, depending on the nuclei, the background signals in the table below may be observed.

Table 2 Characteristics of each component parts of the sample tube

Component Part Name	Materials	Color	Main background signals
Sleeve	Zirconia (ZrO_2)	White	^{17}O, ^{91}Zr
Spinning cap	Vespel	Brown	^{1}H, ^{13}C
Bottom cap	Vespel	Brown	
	PEEK	Light brown	
Spacer	PCTFE	Translucent white	^{13}C, ^{19}F

✍ It may differ slightly depending on the manufacturer of the resin and the ceramics.