# JNM-ECA II Series
# JNM-ECX II Series
# JNM-ECS Series

# AUTO TUNE PROBE

INMECAXS II _V50-ATP-1
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JEOL RESONANCE

# JNM-ECAII Series
# JNM-ECXII Series
# JNM-ECS Series

## AUTO TUNE PROBE

The form of a probe differs depending on the model.

**Please be sure to read this instruction manual carefully, and fully understand its contents prior to the operation or maintenance for the proper use of the instrument.**

JEOL RESONANCE

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Note: For servicing and inquiries, please contact your service office.

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# SAFETY PRECAUTIONS

**Although this instrument is protected with safety device which prevents the occurrence of accident that could result in an injury, harm, and damage to the users or instrument itself, the safety feature may not work properly if you use the instrument for the purpose of use not intended or in an improper usage. For the proper use of the instrument, please be sure to read all of the instructions, descriptions, notices, and precautions contained in this manual carefully to understand them fully prior to the operation or maintenance. This section, "Safety Precautions," contains important information related to safety for using of the instrument.**

**The safety indications and their meanings are as follows:**

⚠ **DANGER:** An imminently hazardous situation which, if not avoided, will result in death or serious injury.

⚠ **WARNING:** A potentially hazardous situation which, if not avoided, could result in death or serious injury.

⚠ **CAUTION:** A potentially hazardous situation which, if not avoided, may result in minor or moderate injury, or a situation that could result in serious damage to facilities or acquired data.

**Labels bearing the following symbols are attached to dangerous locations on the instrument. Do not touch any of these locations with your hands or anything else.**


**Examples of symbols**

- Use the instrument properly within the scope of the purpose and usage described in its brochures and manuals.

- Never open/remove protective parts (exterior panels) and parts that can't be opened/removed without use of tool (including key), or disconnect/ connect the cables/connectors that are not described in this manual.

- Never attempt to do any works of disassembling/assembling the instrument other than those described in this manual.

- Never make modifications that include installing substitute parts and disabling safety devices or other safety features.

- Never disconnect the grounding wire or move it from the prescribed position. Failure to follow this instruction could result in electric shock.

- The AC power cord provided with this system is supplied for the particular device so that never use it for any other equipment.

- To avoid falling, do not climb onto the operation table and console during daily operation or during maintenance or inspection.

- When you dispose of the instrument or liquid or other waste, follow all applicable laws and regulations, and dispose of it in a proper manner without polluting the environment.

- Be sure to read the "Safety Precautions" section of the manuals for the accessories attached to or built into the instrument.

- If anything is unclear, please contact your JEOL RESONANCE service office.

# ⚠ 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.

- Ware protective glasses and leather gloves to protect yourself from any coolant splashes.
  If you do not, you run the risk of frostbite.

- Before replacing the probe, stop the measurement.
  If you replace the probe or change cable connections while RF power or FG power is being output, you might be burned or get an electric shock, and the power amplifier might be damaged.

- Before replacing the probe or changing the cable connections, confirm that the RF power and FG power are off.
  If you replace the probe or change cable connections while RF power or FG power is being output, you might be burned or get an electric shock, and the power amplifier might be damaged. Also, if you apply RF power to the probe with the probe not properly installed in the SCM, electromagnetic radiation exceeding the specified level might leak outside the instrument.

- Some kinds of samples heat up when irradiated with RF power or might boil when warmed. For some organic hydrocarbon samples or volatile samples, you should avoid measurement of the sample or should investigate the physical properties of the sample thoroughly, and should perform the measurement suitable for the physical properties in order to avoid danger.
  A sample might decompose when heated, or boiling of the sample might damage the probe.

- When replacing the probe, take care to support it firmly to avoid dropping the probe.
  If the probe falls accidentally, you might be injured.

- When replacing probes, take care of your head.
  Striking your head against the SCM might injure your head.

- Watch your step when you are around the SCM.
  You might stumble on the vibration-damping pier and cables around the SCM, and be injured.

- Do not touch the probe heater or the Dewar adaptor during a controlled-temperature experiment.
  If you touch the heater connector with your hand when controlling the temperature, you might receive an electric shock.

- Do not carry out a controlled-temperature experiment with the probe heater removed.
  The probe heater might heat up and even catch fire during a controlled-temperature experiment.

- Do not carry out a controlled-temperature experiment with air not flowing.
  The probe heater might heat up and even catch fire during a controlled-temperature experiment.

- When taking out the sample after a controlled-temperature experiment, wait until the sample returns to room temperature and wear thick gloves.

  If you do not, you might get burns or frostbite because the sample and the sample holder are hot or cold.

# PRECAUTIONS FOR USE

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

- **Since the probe is adjusted precisely at the factory before delivery, do not remove the cover of the probe.**
  If you remove the cover, readjustment of the probe is needed. Even when you need to carry out an operation other than those in the operation manual, contact the JEOL RESONANCE service office without touching the probe.
- **When setting the sample tube to the probe, make sure that**
  **・there is no sample tube in the probe, and**
  **・the air for floating the sample tube is flowing.**
  In order to confirm that the probe is empty, check that the EMPTY lamp on the Head amplifier chassis is lit. Also, in order to confirm that the floating air is flowing, check that the sample tube floats properly when the sample tube is placed in the inserting port of the sample tube of the SCM.
- **When inserting the sample tube, handle the spinner rotator properly.**
  Failure to do so may damage the sample tube.
- **Before removing the probe from the SCM, make sure the sample tube is removed from the probe.**
  If you pull down the probe without removing the sample tube, the sample tube may come into contact with the SCM and be broken.
- **When connecting the auto-tune probe without LCD to the flexible shafts, do not turn the probe dial manually.**
  If you force it, the probe will be damaged.
- **After connecting the flexible shafts to the auto-tune probe without LCD, be sure to create a dial file.**
  If you do not do so, the actual value of the dial will be different from that in the file, so that the auto-tuning unit will drive the dial to exceed its variable range, damaging the probe.
- **When you enter the value, check the actual value of the Autotune probe again.**
  If you enter a wrong value, the auto-tuning unit might drive the dial to exceed the variable range, damaging the auto-tune probe.
- **The order in which dials of the auto-tune probe without LCD are arranged may be different from that of the parameters on the entry screen. So, be careful to enter each value correctly.**
  If you enter a wrong value, the auto-tuning unit might drive the dial to exceed the variable range, damaging the auto-tune probe.
- **When taking out the sample after a temperature control experiment, wait until the sample returns to room temperature.**
  Failure to do so may damage the sample tube.
- **Be careful to handle the stator section at the top of the probe so that it does not come into contact with the SCM, etc.**
  Failure to do so may damage the stator and cause a rotation error.

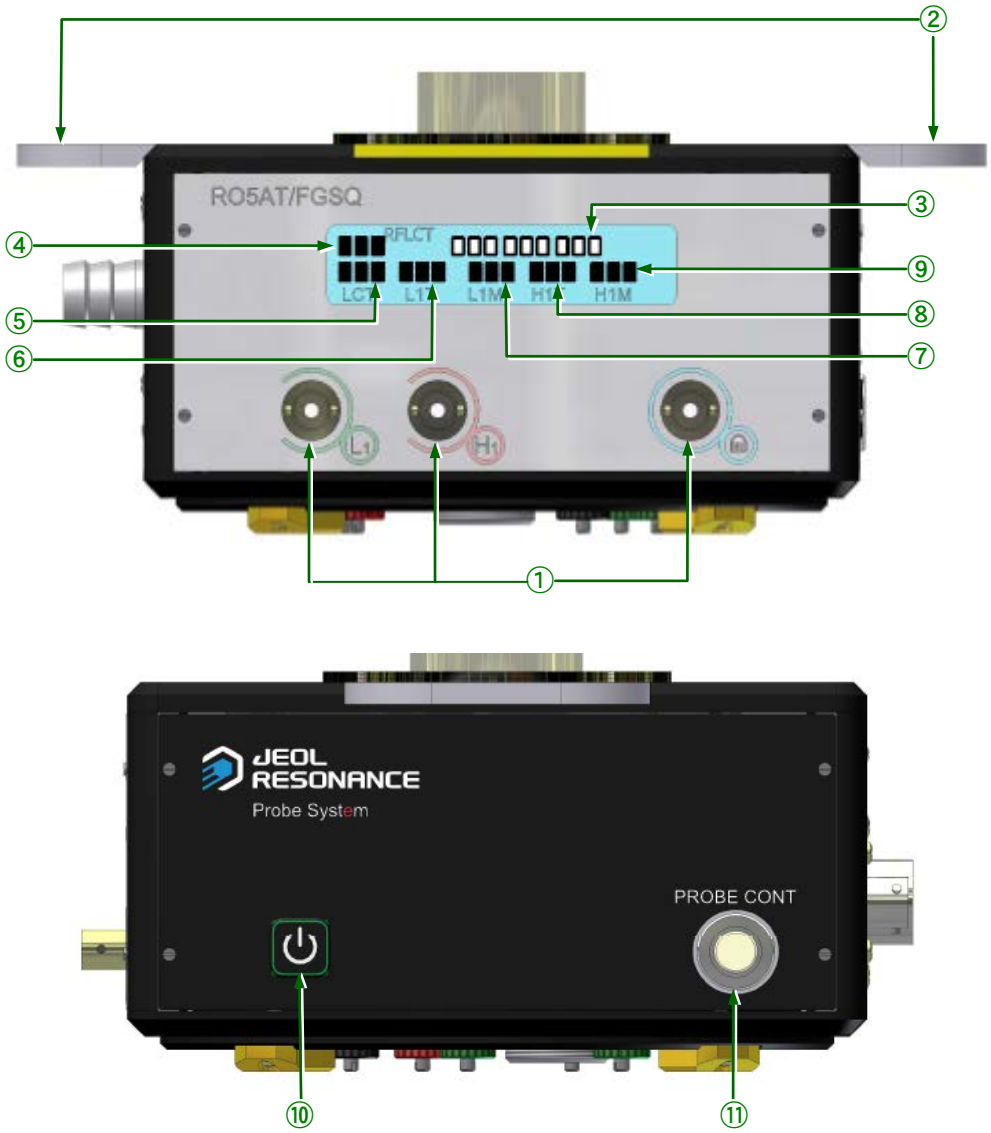
- **Be careful that dust, packing material, or filler material does not fall into the storage box. Also make sure that you put the cap on the top of probe when storing the probe.**
  If dust, etc. enters into the probe, the performance of the probe may be deteriorated seriously.
- **When attaching or removing the heater, do not apply excessive force to it.**
  Excessive force might damage the inside glass tube.
- **Be careful not to insert the cleaning stick into the inner area beyond the stator (more than 54 mm from the air bearing) with attention to the outer diameter of the stick.**
  The probe may be damaged and the performance may be deteriorated.

# 1 GENERAL

This instruction manual describes the usage method of the standard Auto tune probe. For details on the usage, including outer appearance, cable connections, and operations, since they depend on the model, refer to the instruction manual provided with each probe.
The standard probe of the ECA400II, ECA500II, ECA600II, ECX400II, and ECS400 FT NMR system is the 5mm FG/RO Digital Auto Tune Probe. The standard probe of the ECA300II and ECS300 FT NMR system is the Auto Tune 5mm FG/TH Tunable Probe.
By using the optional Auto Tuning Unit 2 (NM-01090AT), you can automatically tune the probe for the multiple nuclei.

# 2 NAMES AND DESCRIPTIONS OF PARTS

## ■ 5 mm FG/RO digital auto tune probe



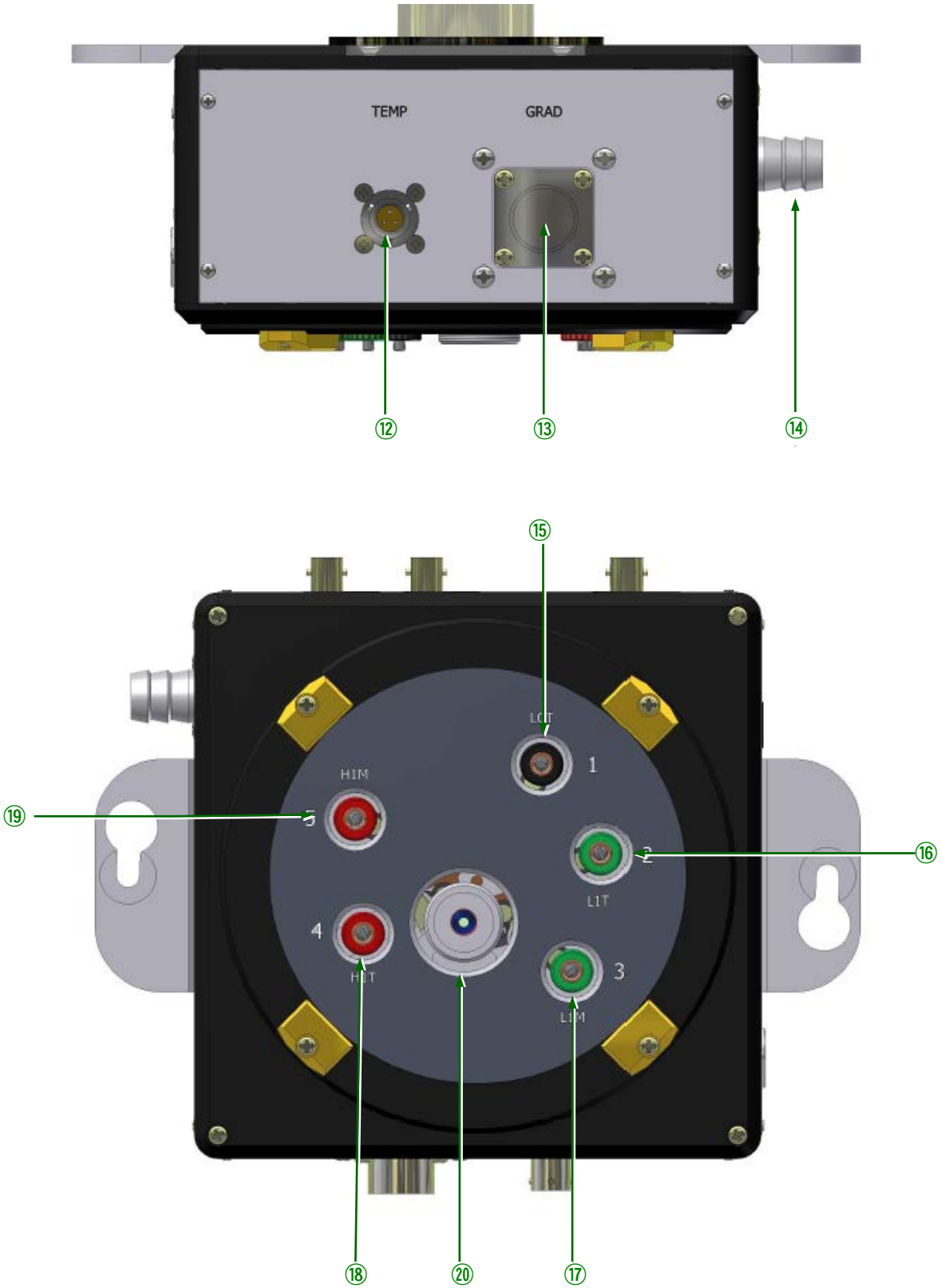



**Fig.1 5 mm FG/RO digital auto tune probe**

① L1, H1, connectors
Connector for the cable connecting to the head amplifier chassis
Connect L1 to LF1, H1 to HF1, and to the Lock port of the head amplifier chassis respectively.

② Probe fixing guide
Guide plate for attaching the probe to the SCM

③ Tuning level meter
This meter is used to tune and match the probe.

④ Display of the LCT (LF COARSE TUNE) range
This indicates the range (X, A, B C, and D) of the LF COARSE TUNE axis.

⑤ Display of the value of the LCT (LF COARSE TUNE) dial
This indicates the dial value (0 to 400) of the LF COARSE TUNE axis.

⑥ Display of the value of the L1T dial
This indicates the dial value of the LF1 TUNE axis.

⑦ Display of the value of the L1M dial
This indicates the dial value of the LF1 MATCH axis.

⑧ Display of the value of the H1T dial
This indicates the dial value of the HF1 TUNE axis.

⑨ Display of the value of the H1M dial
This indicates the dial value of the HF1 MATCH axis.

⑩ Switch for turning on/off the display of the LCD
This switch turns on/off the display of the tuning level meter and dial values on the LCD.

⑪ PROBE CONT connector
Connector for supplying power to the probe and exchanging information between the spectrometer and the probe

⑫ TEMP connector
Connector for detecting the sample temperature

⑬ GRAD connector
To perform measurement while applying a field gradient, connect this connector to the cable from the Field Gradient unit.

⑭ EXHAUST
Exhausts the air used in the probe.

⑮ Dial 1 (LCT knob)
Switches the frequency band (LF COARSE TUNE) of the LF channel

⑯ Dial 2 (L1T knob)
Adjusts the tuning of the LF channel

⑰ Dial 3 (L1M knob)
Adjusts the matching of the LF channel

⑱ Dial 4 (H1T knob)
Adjusts the matching of the HF channel

⑲ Dial 5 (H1M knob)
Adjusts the tuning of the HF channel

⑳ Adapter joint
This is connected to the room-temperature adapter when measurement is performed at room temperature and to the probe heater when measurement is performed at controlled temperature.

## ■ Auto tune 5 mm FG/TH tunable probe

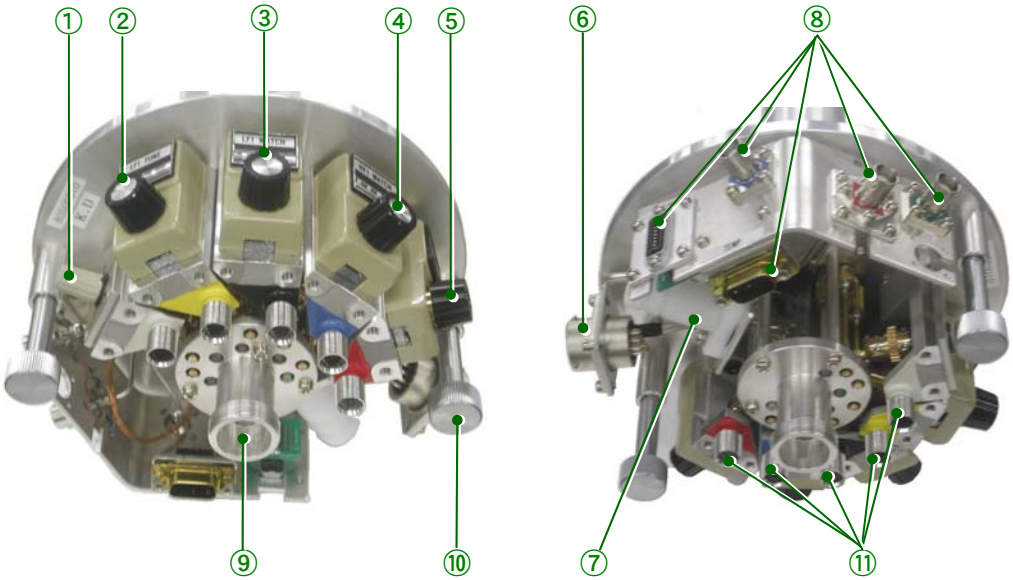



**Fig. 2 Auto tune 5 mm FG/TH tunable probe**

① LF1 COARSE knob
Switches the observation frequency range.

② LF1 TUNE dial
Used to tune the observation frequency channel.

③ LF1 MATCH dial
Used to match the observation frequency channel.

④ HF1 MATCH dial
Used to match the irradiation and $^{1}H$ observation frequency channel.

⑤ HF1 TUNE dial
Used to tune the irradiation and $^{1}H$ observation frequency channel.

⑥ GRAD connector
To perform measurement while applying a field gradient, connect this connector to the cable from the Field Gradient unit.

⑦ Exhaust port
Exhausts the air used in the probe.

⑧ LF1, HF1, LOCK, TEMP,STATUS connectors
These are connectors for connecting the cables to the head amplifier chassis or the spectrometer. When you replace the probe, reconnect them.

⑨ Adapter joint
This is connected to the room-temperature adapter when measurement is performed at room temperature and to the probe heater when measurement is performed at controlled temperature.

⑩ Probe retaining screw
Attaches the probe to the magnet.

⑪ Inlet Screws
To carry out automatic tuning, connect the flexible shaft from the optional automatic tuning unit to each dial through this port.

# 3 PREPARATION

## 3.1 Installing/Removing the Probe

This section describes how to attach/remove the probe to/from the SCM, and how to connect the cables and tubes to the probe.

**⚠ CAUTION**

- **When replacing the probe, take care to support it firmly to avoid dropping the probe.**
  If the probe falls accidentally, your hands or feet might be injured.

- **When replacing probes, take care to protect your head.**
  Striking your head against the Superconducting magnet might cause injury.

- **Before replacing the probe or changing the cable connections, confirm that the RF power and FG power are off.**
  If you replace the probe or change cable connections while RF power or FG power is being output, you might be burned or get an electric shock, and the power amplifier might be damaged. Also, if you apply RF power to the probe with the probe not properly installed in the SCM, electromagnetic radiation exceeding the specified level might leak outside the instrument.

### 3.1.1 Mounting the probe to the SCM

1. Remove the probe from the storage box and remove the cap.
2. Gently insert the probe into the superconducting magnet from the probe insertion port under the superconducting magnet.
3. Fix the probe to the SCM.

#### ● 5 mm FG/RO Digital Auto Tune Probe

As shown in Fig. 3, lift the probe while adjusting the large holes of the probe fixing guide to the position of the probe retaining screws on the SCM bottom, and turn the probe slowly along with its fixing guide (clockwise when viewed from the bottom). Confirm that the probe retaining screws are positioned at the ends of the holes of the probe fixing guide, and then fasten the nuts of the probe retaining screws.

When you turn the probe, you can find several locations at which the probe can been attached. However, if the position differs from the location at which the shim file was created, good resolution sometimes cannot be obtained. Mark the shim mount and probe when creating a shim file so that the probe can always be set up at the same position after it is changed.

For details on the shim file, refer to Section 3.7 of the instruction manual for the JNM-ECAII/ECXII/ECS Series, “Handling of Hardware” manual.

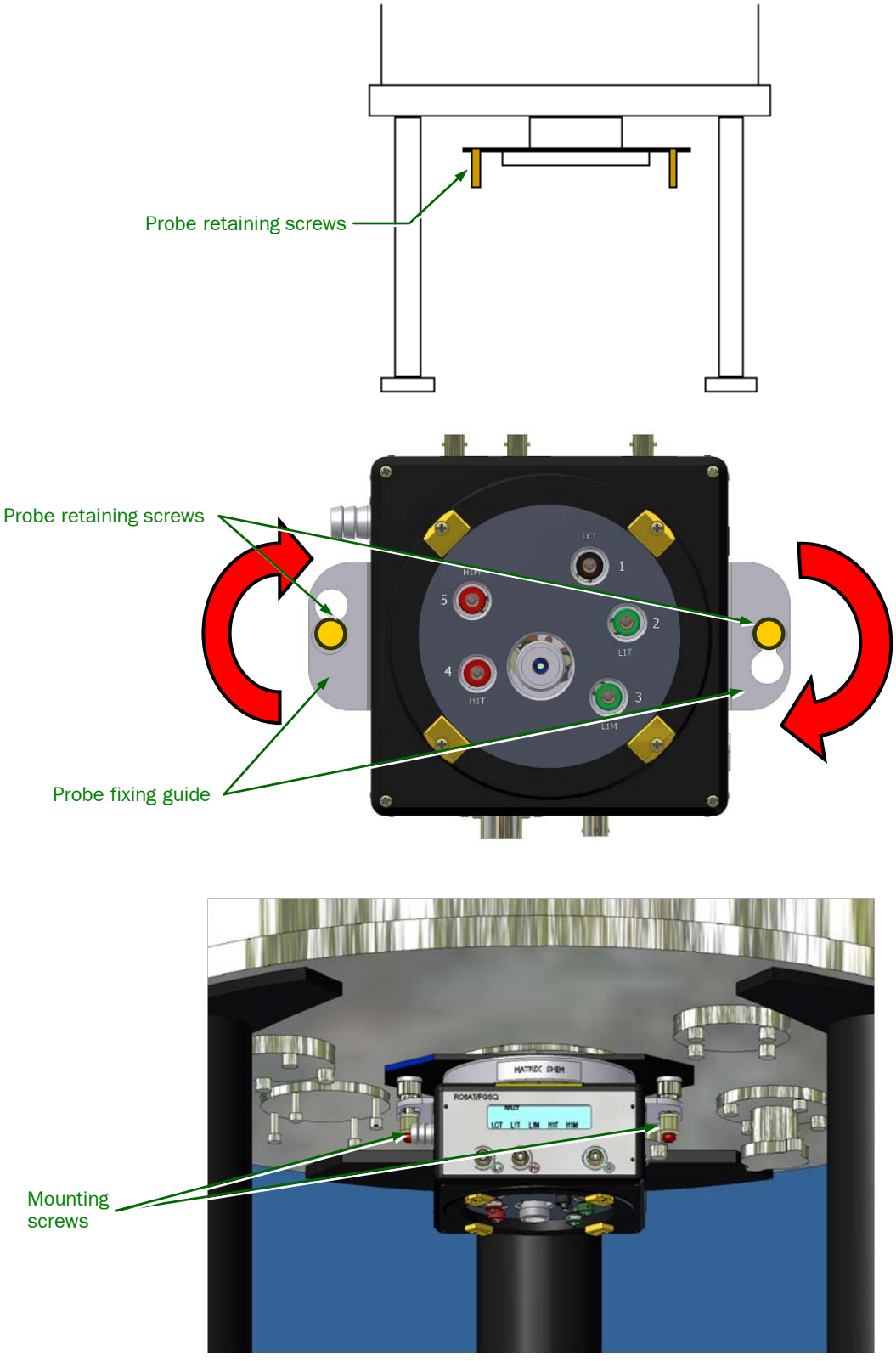



**Fig. 3 Installing the probe**

## Auto tune 5 mm FG/TH Tunable Probe

- Place the probe and the room temperature shim mount in close contact with each other and attach them with the two provided retaining screws as shown in Fig. 4.
- When you turn the probe, you can find several locations at which the probe can been attached. However, if the position differs from the location at which the shim file was created, good resolution sometimes cannot be obtained. Mark the shim mount and probe when creating a shim file so that the probe can always be set up at the same position after it is changed.

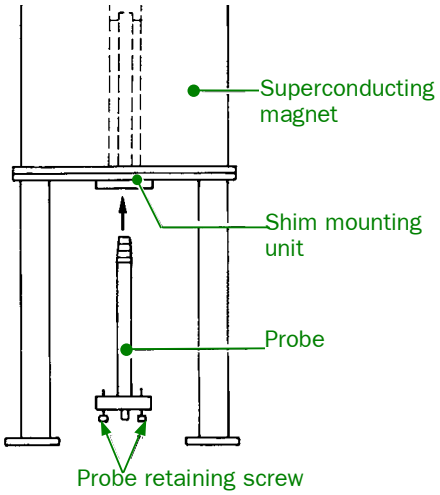



**Fig. 4 Installing the Auto tune 5 mm FG/TH Tunable Probe**

### 3.1.2 Connecting the cables and hoses

#### ● 5 mm FG/RO Digital Auto Tune Probe

Connect the cables and hoses between the head amplifier chassis (or the probe adapter) and the probe as shown in Fig. 5.

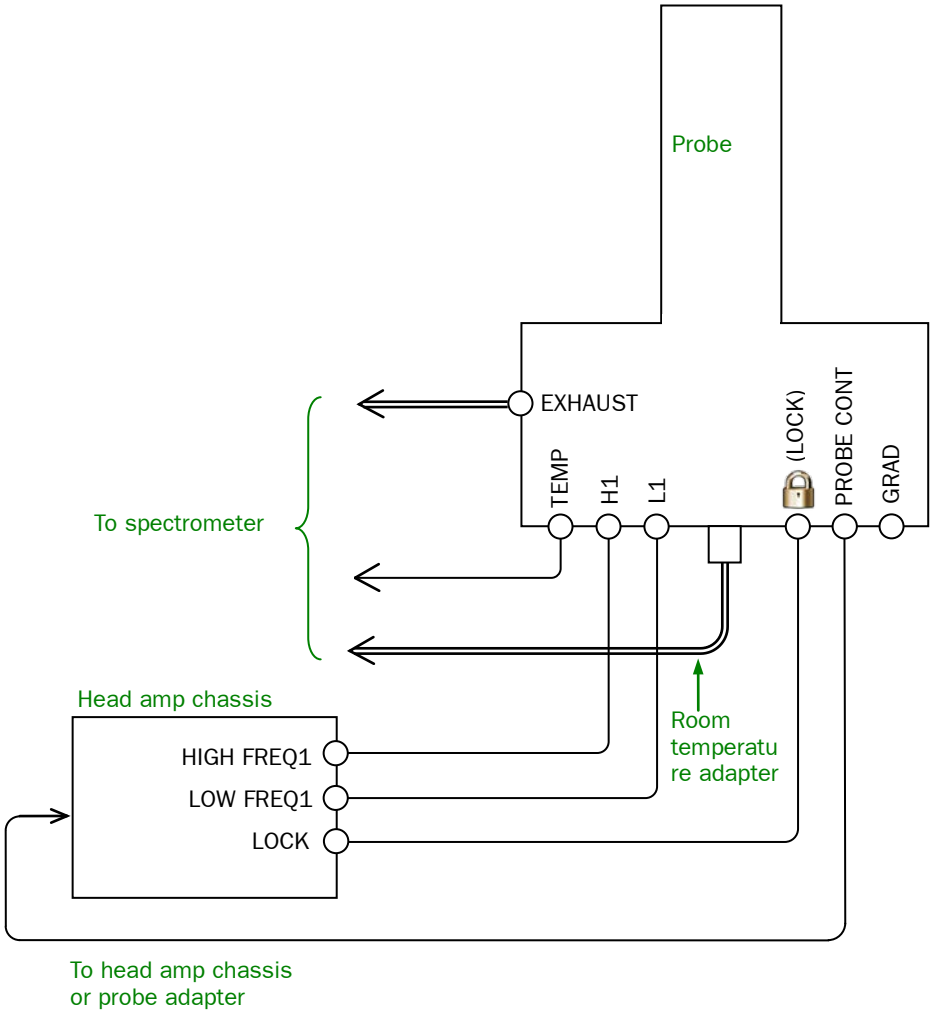



**Fig. 5 Connection diagram**

- When attaching the temperature-control unit, connect the probe heater in place of the room-temperature adapter and connect the thermocouple cable to the TEMP connector.
- For details on the methods of mounting/removing the probe heater, operating the heater, and controlling the temperature, refer to Chapter 4 in the “Handling of Hardware” manual of the JNM-ECAII/ECXII/ECS series FT NMR system.
- For information on the Probe adapter, refer to the instruction manual for the Probe Adapter.
- To perform measurement while applying a pulsed magnetic field gradient, connect the FG cable that comes from the Field Gradient unit to the GRAD connector.
- For connection when the Auto tuning unit 2 is configured, refer to the instruction manual for the Auto tuning unit 2.

## ● Auto tune 5 mm FG/TH tunable probe

Connect the cable and hose between the head amplifier chassis and the probe as shown in Fig. 6.

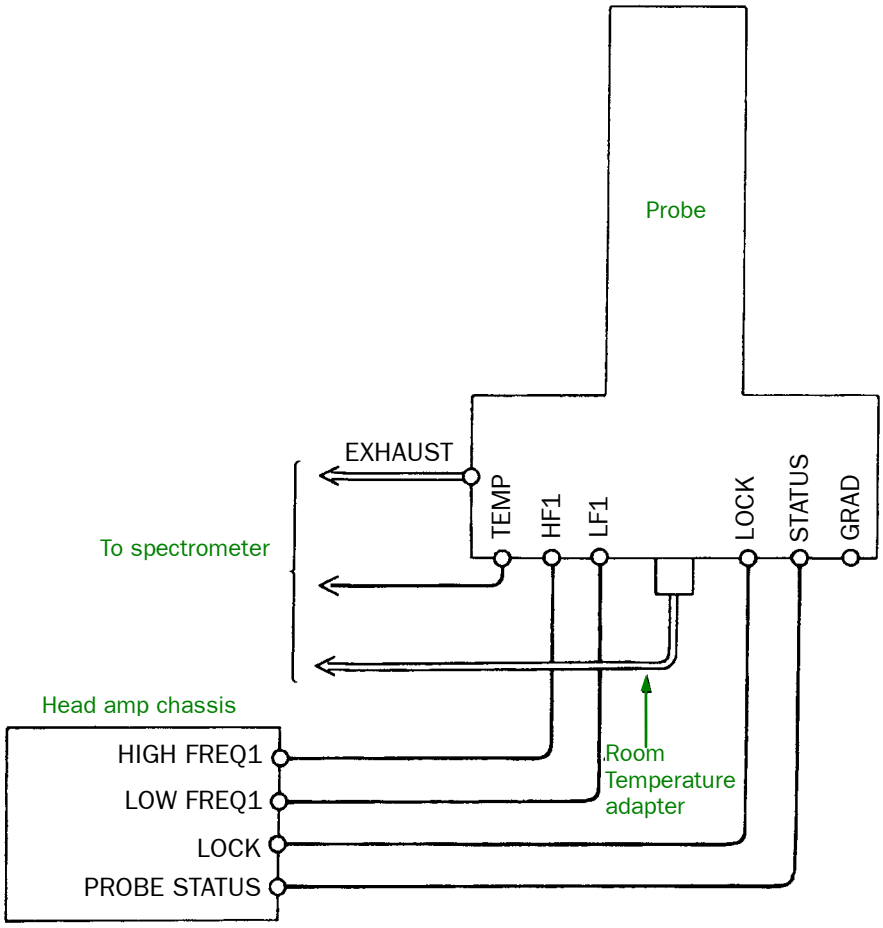



**Fig. 6 Connection diagram of the Auto tune 5 mm FG/TH Tunable Probe**

- When attaching the temperature-control unit, connect the probe heater in place of the room-temperature adapter and connect the thermocouple cable to the TEMP connector.
- When the probe is changed, the probe status is automatically loaded.
- To perform measurement while applying a pulsed magnetic field gradient, connect the FG cable that comes from the Field Gradient unit to the GRAD connector.
- To carry out automatic tuning, connect the flexible shaft from the optional automatic tuning unit 2 to each dial through the insertion port of the probe.
  - ☞ For details, refer to the instruction manual for the Auto tuning unit 2.

### 3.1.3 Removing the probe from the SCM

1. Stop the present measurement and take out the sample.
2. Remove the cables and hoses that are connected to the probe.
3. Remove the temperature-control unit if it has been attached.
4. Remove the probe from the SCM.

## ● 5 mm FG/RO Digital Auto Tune Probe

After loosening the probe retaining screws, turn the probe slowly counterclockwise when viewed from the bottom so that the large holes of the probe fixing guides are positioned at the probe retaining screws. Confirm that the holes of the probe fixing guides are free from the probe retaining screws, and slowly pull down the probe.

## ● Auto tune 5 mm FG/TH Tunable Probe

As shown in Fig. 7, loosen the retaining screws of the probe and pull the probe straight down. Then, take out the probe when the probe top has come out of the superconducting magnet.

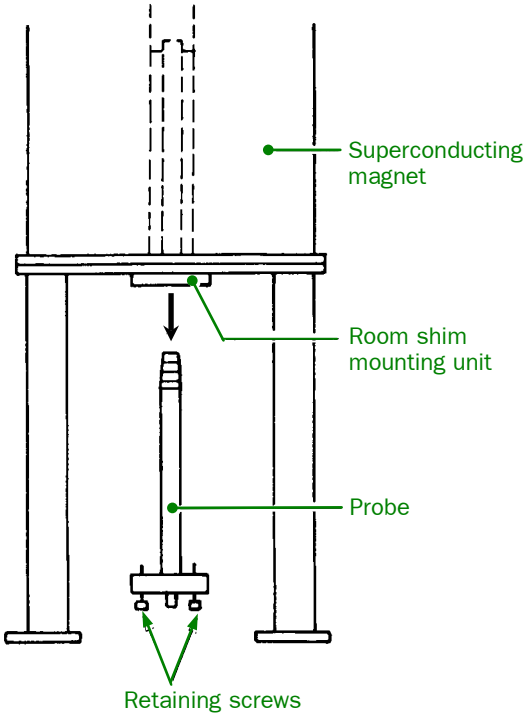



**Fig. 7 Removing the Auto tune 5 mm FG/TH Tunable Probe**

**5.** Put the cap on the probe and store it in the storage box.

**⚠ CAUTION**

**When taking out the sample after a controlled-temperature experiment, wait until the sample returns to room temperature and wear thick gloves.**

If you do not, you might get burns or frostbite because the sample and the sample holder are hot or cold.

**—CAUTION—**

- **When taking out the sample after a temperature control experiment, wait until the sample returns to room temperature.**
  Failure to do so may damage the sample tube.

## 3.2 Manual Probe Tuning

Tuning of the probe is necessary in order to efficiently apply RF pulses to the sample. If the tuning shifts, experiment conditions change, and the signal-to-noise ratio of the spectrum decreases.
When changing the sample, the shift of tuning may occur. Especially when the dielectric constant of a new sample or the material of the sample tube differs from that of the previous one, the tuning deviates greatly. In that case, tune the probe so that the experiment will be performed under the best conditions.
You can use both methods, manual tuning and automatic tuning, to tune the probe.

✍ When you tune the probe, tune an LF nucleus first, then tune the HF nucleus. The following is the procedure.

### 3.2.1 $^{1}$H tuning in normal measurement

#### ● 5 mm FG/RO Digital Auto Tune Probe

1. Perform the 1H tuning with reference to the “Handling of Hardware” manual.
2. Push the display switch of the LCD to turn on the display.

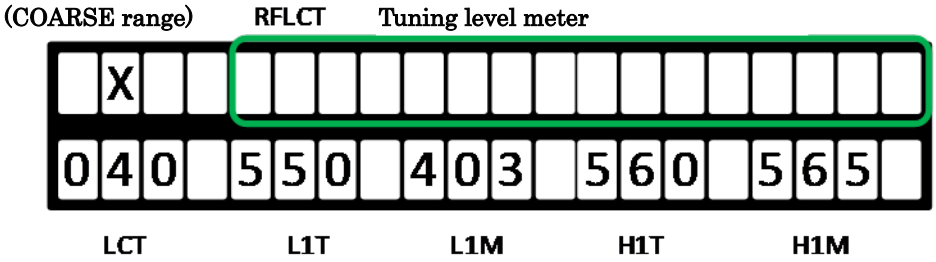



**Fig. 8 Example of the display of the LCD**

3. First, adjust the dial 4 (H1T knob) to minimize the reading on the tuning level meter of the LCD attached to the probe or on the tuning level meter of the Head amplifier chassis. Next, adjust the dial 5 (H1M knob). Finally, adjust the dial 4 (H1T knob) once more.

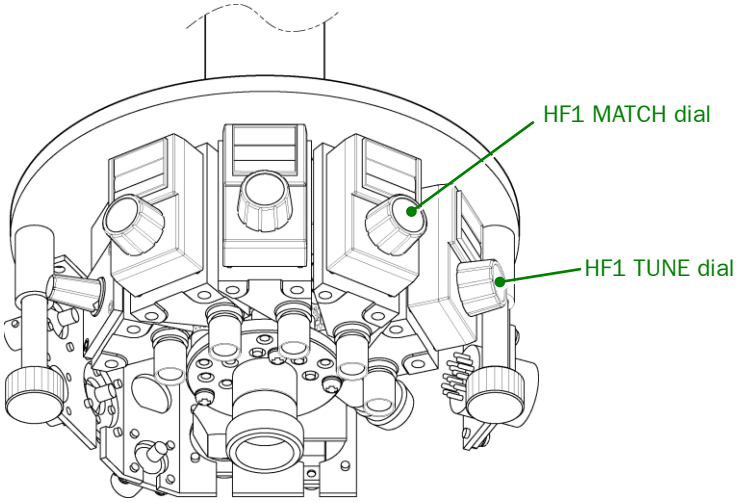
✍ If the display of the LCD disappears, push the switch again to display it.

✍ If the knob is not turned for ten seconds, the display of the LCD is automatically turned off. Also, if the measurement is started before the display of the LCD is turned off, be aware that spurious signals may appear in the spectrum.

✍ Pushing the switch while the LCD display is turned on turns off the LCD display.

### ● Auto tune 5 mm FG/TH Tunable Probe

1. Perform the 1H tuning with reference to the “Handling of Hardware” manual.
2. First, adjust the HF1 TUNE dial of the probe so as to minimize the reflection of LEVEL METER (for the JNM-ECS series, numerical value on the display panel) on the head amplifier chassis, and next, adjust the HF1 MATCH dial. Finally, adjust the HF1 TUNE dial once again.




## 3.2.2 $^{13}$C Tuning in normal measurement

### ● 5 mm FG/RO Digital Auto Tune Probe

1. Perform the 13C tuning with reference to the “Handling of Hardware” manual.
2. Push the display switch of the LCD to turn on the display.

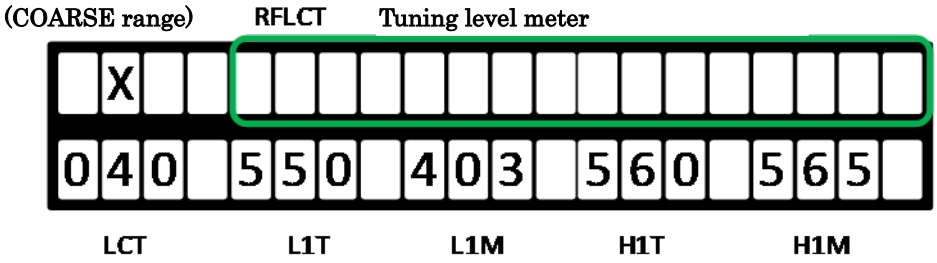
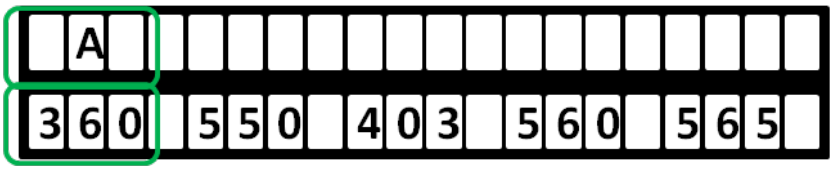



**Fig. 9 Example of the display of the LCD**

3. Set the frequency band of the LF channel to the 13C frequency range.
   Turn the LCT (LF COARSE TUNE) knob to set the position required for the measurement nucleus of the LF channel as follows.
   ✍ For $^{13}$C tuning, Set the LCT to A.
   (Example) When setting the LCT to A,
   Turn the LCT knob so as to display 360 on the LCT dial value display.
   ✍ At that time, confirm that the LCT range display indicates A.

LCD range display



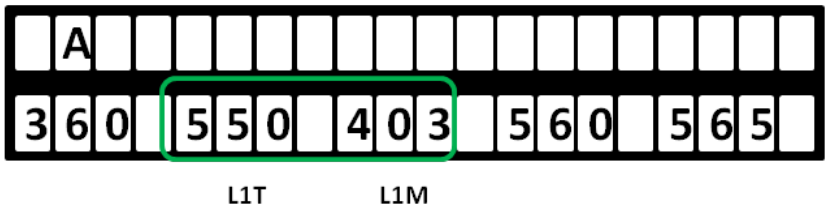

LCD dial value display

The range of the LCT corresponds to the LCT dial value as follows.

| LCT range display | LCT dial value display |
|---|---|
| X | 40 |
| A | 360 |
| B | 280 |
| C | 200 |
| D | 120 |

For the position of the LF1 COARSE TUNE, refer to the dial tables that belong to the probe.

**4.** Refer to the dial tables that belong to the probe, and turn the dial 2 (L1T knob) and the dial 3 (L1M knob) to set them to the values specified in the dial tables.




If the display of the LCD disappears, push the switch again to display it.

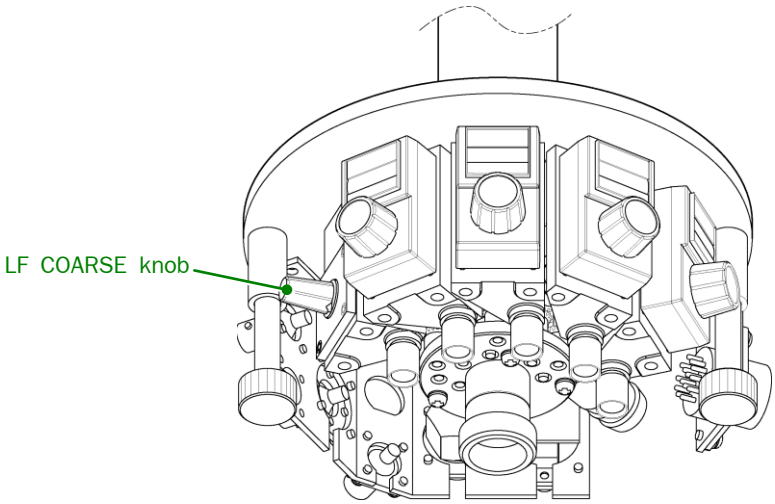
If the knob is not turned for ten seconds, the display of the LCD is automatically turned off. Also, if the measurement is started before the display of the LCD is turned off, be aware that spurious signals may appear in the spectrum.

Pushing the switch while the LCD display is turned on turns off the LCD display.

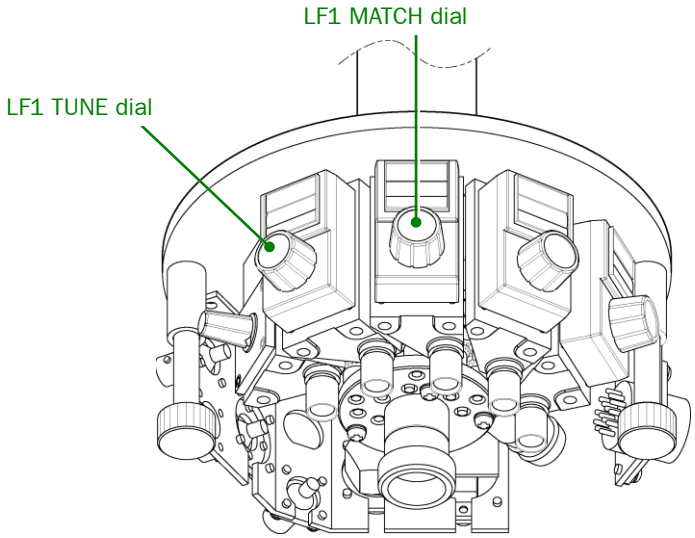
**5.** First, adjust the dial 2 (L1T knob) to minimize the reading on the tuning level meter of the LCD attached to the probe or on the tuning level meter of the Head amplifier chassis. Next, adjust the dial 3 (L1M knob). Adjust the dial 2 (L1T knob) and dial 3 (L1M knob) several times so as to minimize the reading on the tuning level meter.

## ● Auto tune 5 mm FG/TH tunable probe

1. Perform the 13C tuning with reference to the “Handling of Hardware” manual.
2. Set the LF COARSE knob of the probe to the 13C frequency range.
   For the tuning and matching dial values for the nucleus to be measured, refer to the values of the dial table supplied with the auto-tune probe.




3. First, adjust the LF1 TUNE dial of the probe so as to minimize the reflection of LEVEL METER (for the JNM-ECS series, numerical value on the display panel) on the head amplifier chassis.
4. Adjust the LF1 MATCH dial.
   Adjust the LF1 TUNE dial and LF1 MATCH dial several times so as to minimize the reflection (numerical value).



### 3.2.3 Precise $^{13}C$ tuning

#### ● 5 mm FG/RO Digital Auto Tune Probe

1. Adjust the dial 3 (L1M knob) to minimize the reading on the tuning level meter of the LCD attached to the probe or on the tuning level meter of the Head amplifier chassis.
2. Adjust the dial 2 (L1T knob) to minimize the reading on the tuning level meter of the LCD attached to the probe or on the tuning level meter of the Head amplifier chassis.
3. Turn the dial 3 (L1M knob) by +5 graduations, and adjust the dial 2 (L1T knob) to minimize the reading (F1) on the tuning level meter of the LCD attached to the probe or on the tuning level meter of the Head amplifier chassis.
   Memorize the reflection (F1) of LEVEL METER at that time.
4. Turn the dial 3 (L1M knob) by –5 graduations, and adjust the dial 2 (L1T knob) to minimize the reading (F2) on the tuning level meter of the LCD attached to the probe or on the tuning level meter of the Head amplifier chassis.

   Compare the reflection (F2) of LEVEL METER at this time with F1, and turn the dial 3 (L1M knob) a little to the direction that gave the smaller reading, and turn the dial 2 (L1T knob) to the direction that will give the smaller reading.

   ✍ This operation is repeated until the reflection of LEVEL METER becomes the minimum.

#### ● Auto tune 5 mm FG/TH tunable probe

1. Turn the LF1 MATCH dial to minimize the reflection of LEVEL METER on the head amplifier chassis.
2. Turn LF1 TUNE dial to minimize the reflection of LEVEL METER on the head amplifier chassis.
3. Turn the LF1 MATCH dial by +10 graduations and turn the LF1 TUNE dial to minimize the reflection (F1) of LEVEL METER on the head amplifier chassis.
   Memorize the reflection (F1) of LEVEL METER at that time.
4. Turn the LF1 MATCH dial by –10 graduations and turn the LF1 TUNE dial to minimize the reflection (F2) of LEVEL METER.

   Compare the reflection (F2) of LEVEL METER at this time with F1. Turn the LF1 MATCH dial a little to the direction that gave the smaller reading, and then turn the LF1 TUNE dial to the direction that will give the smaller reading.

   ✍ This operation is repeated until the reflection of LEVEL METER becomes the minimum.

## 3.3 Automatic Probe Tuning

☞ For information on the automatic tuning, refer to the instruction manual for the tuning unit 2.