

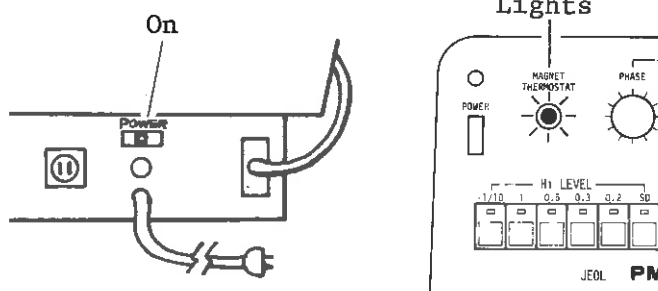
CHAPTER 3 OPERATION

3.1 START-UP

1. Magnet console (Rear)

POWER switch: On

Spectrometer MAGNET THERMOSTAT lamp lights up.



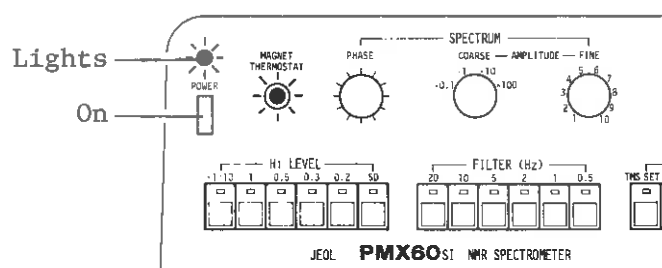
Notes (a): To carry out precise measurements, commence measurement after the POWER switch has been on for three days or more.

(b): Turn off the POWER switch for reasons of instrument break-down only. In all other cases leave it on.

2. Spectrometer

POWER switch: On

Red lamp lights up.



Notes (a): Start measurement after the POWER switch has been on for one hour.

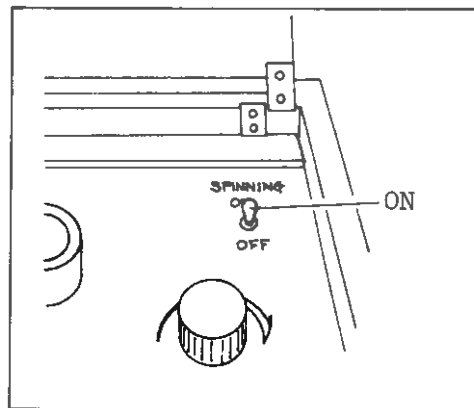
(b): Leave this switch on, except in cases of instrument break-down.

3.2 SAMPLE TUBE INSERTION

1. Magnet console (Top)

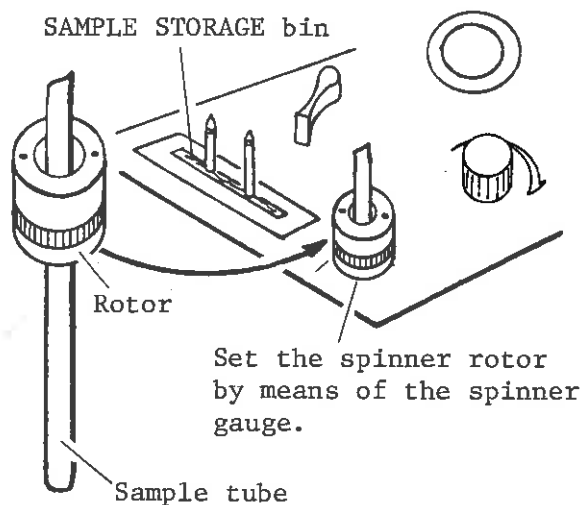
SPINNING switch: ON

Compressor starts.



2. Rotor set

- a. Preheat sample tubes in SAMPLE STORAGE bin for 5 minutes.
- b. Wipe the sample tube and rotor clean.
- c. Slide the rotor onto the sample tube as far as the mid-way point.
- d. Insert a sample tube into GAUGE, secure the rotor and gently push the tube down until it stops.



Set rotor and push down sample tube.

Precautions

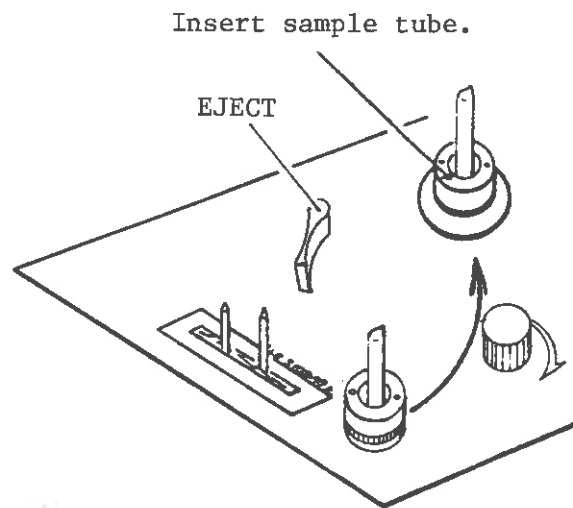
Since the use of a non-standard sample tube will result in tube rotation wobble, ejection problems, etc., always use a standard sample tube.

Sample tube specifications

Thickness difference: 0.05 mm
 Curvature degree: 0.025 mm/180 mm
 Inner diameter: 4.2 ± 0.03 mm
 Outer diameter: $5.0 \begin{smallmatrix} -0.02 \\ -0.1 \end{smallmatrix}$ mm

3. Sample tube insertion

- a. Sample tube lever: EJECT
- b. Remove sample tube from gauge and insert it into sample tube inlet.



Notes (a): Set the sample tube lever to EJECT to remove the sample tube.

(b): Do not insert the rotor or sample tube separately, as float problems will result.

(c): Do not attempt to insert a sample tube when one is already in the probe.

(d): Upon completion of measurement, leave the sample tube in the sample tube inlet.

4. Setting spinner

a. Sample tube lever: SET

The sample tube should make a sound when it reaches the bottom of the probe.

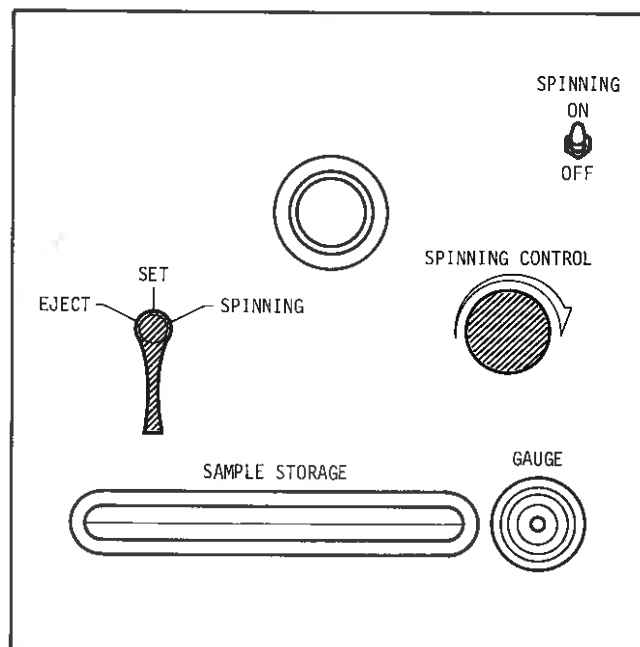
Note: The sample tube will float if it is rotated when not in contact with the bottom of the probe.

b. Sample tube lever: SPINNING

Sample tube starts to rotate.

c. SPINNING CONTROL knob:
Adjust for optimum rotating speed.

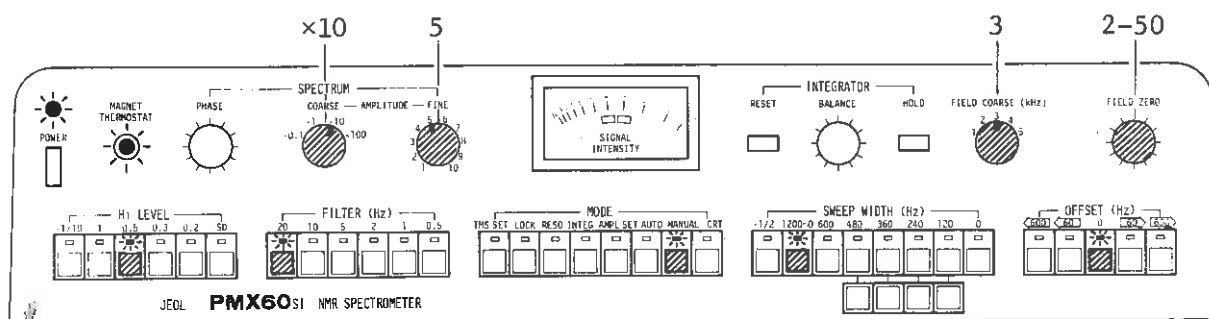
Note: An eddy will form in the sample tube in the event the rotating speed is too fast, resulting in a deterioration of resolution. But if it is too slow, it may give rise to a spinning side band.



3.3 INITIAL SETTINGS AND NMR SIGNAL OBSERVATION

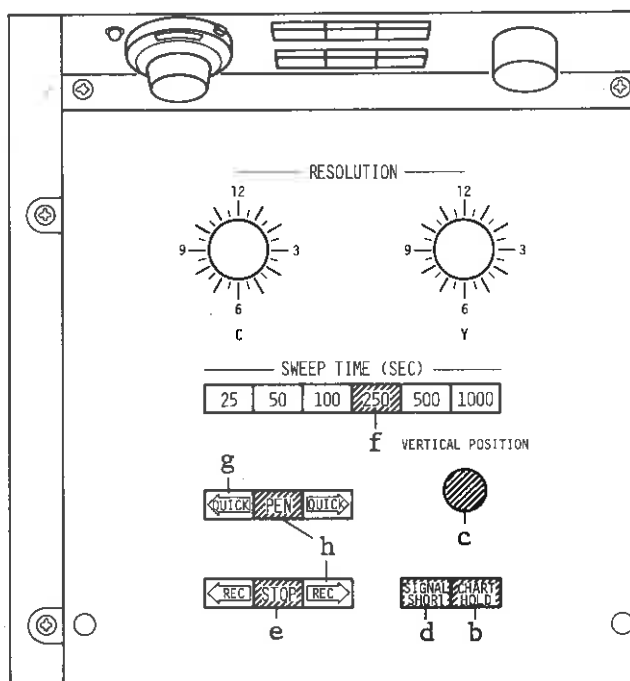
1. Initial spectrometer setting

AMPLITUDE-COARSE: $\times 10$
 FINE: 5
 FIELD COARSE : 3
 FIELD ZERO : 2-50
 H₁ LEVEL : 0.5
 FILTER : 20
 MODE : MANUAL
 SWEEP WIDTH : 1200 $\sim$ 0
 OFFSET : 0



2. Recording signal

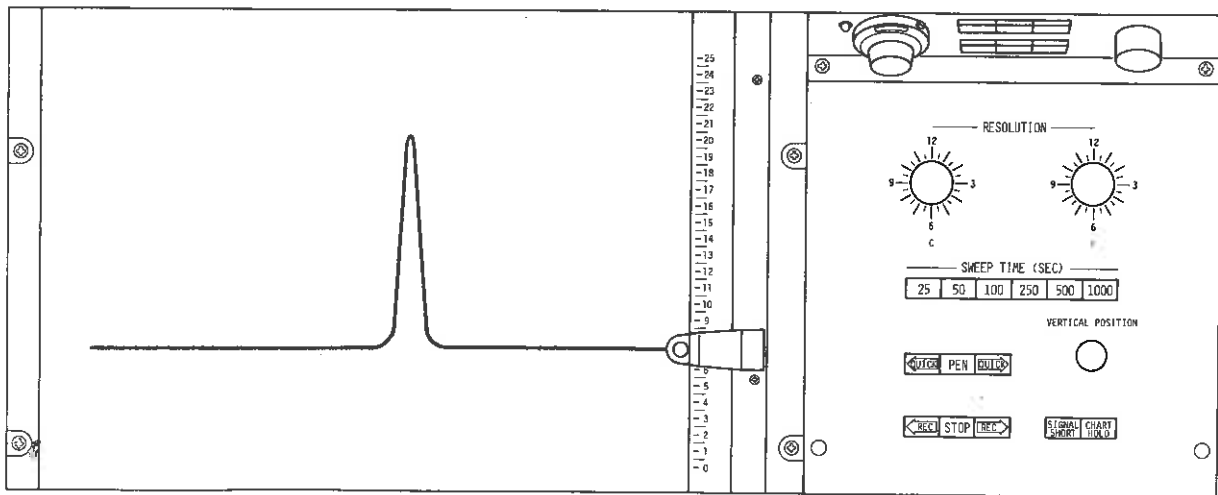
- a. Align the chart paper with the paper guide and set it on the recorder.
- b. CHART HOLD : Depress
- c. VERTICAL POSITION: Adjust so that pen settles in center of vertical axis.
- d. SIGNAL SHORT : Release (Up)
- e. STOP : Depress
- f. SWEEP TIME : Depress 250 button
- g. QUICK : Depress
- h. PEN and REC : Depress



Notes (a): When either  or  is pushed, sweep is performed in the direction of the arrow.

(b): Do not move the carriage arm when CHART HOLD is depressed. Release the CHART HOLD button when the recorder is not to be used.

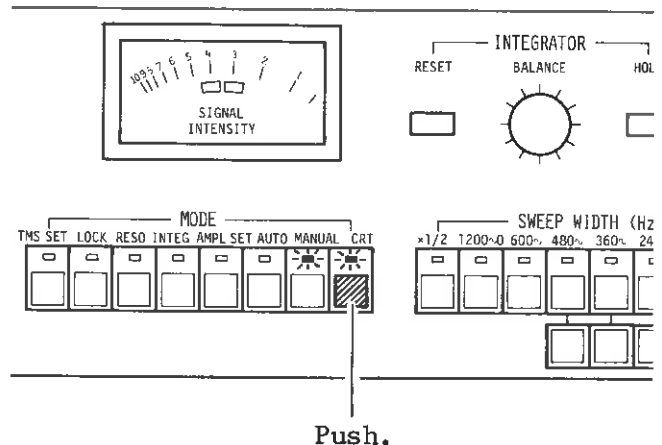
(c): Adjust the FIELD ZERO knob so that the signal can be observed in the center of the recorder whenever the signal cannot be recorded. If the signal still cannot be recorded, adjust the FIELD COARSE knob in steps until the signal can be recorded. Search for the position where the pen deflects.



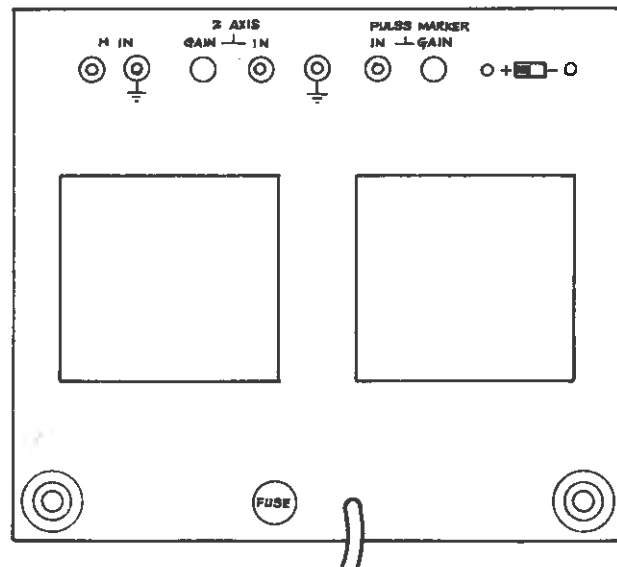
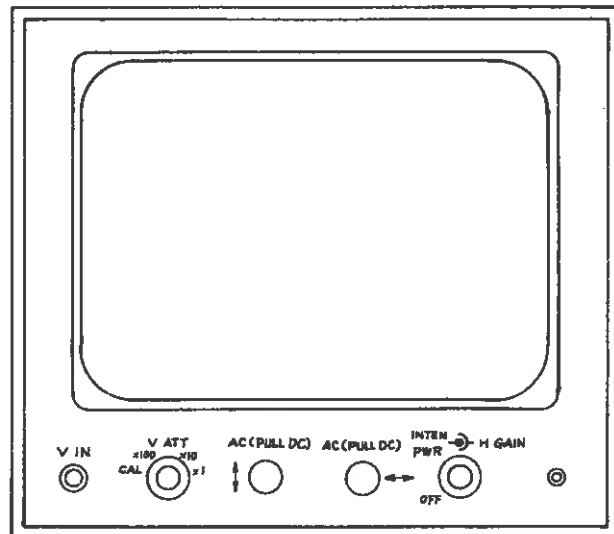
3. Oscilloscope observation

a. Spectrometer
MODE : Push CRT.

b. Oscilloscope
POW : ON



- c. AC (PULL DC): Pull.
- d. Disconnect cables connected to H IN (in rear of oscilloscope).
- e. Adjust $\leftrightarrow$ and $\updownarrow$ knobs until spot settles in middle of screen.
- f. Reconnect cables to H IN.
- g. Adjust H GAIN until brightness line covers full screen.
- h. Adjust INTEN so that waveform is of easy-to-see brightness.
- i. Adjust $\updownarrow$ knob so that the bright line is 1/5 from bottom of screen.
- j. V. ATT outer knob: $\times 10$
- k. V. ATT inner knob: Turn fully clockwise.
- l. Adjust FIELD ZERO and FIELD COARSE knobs until signal can be seen in center of oscilloscope.



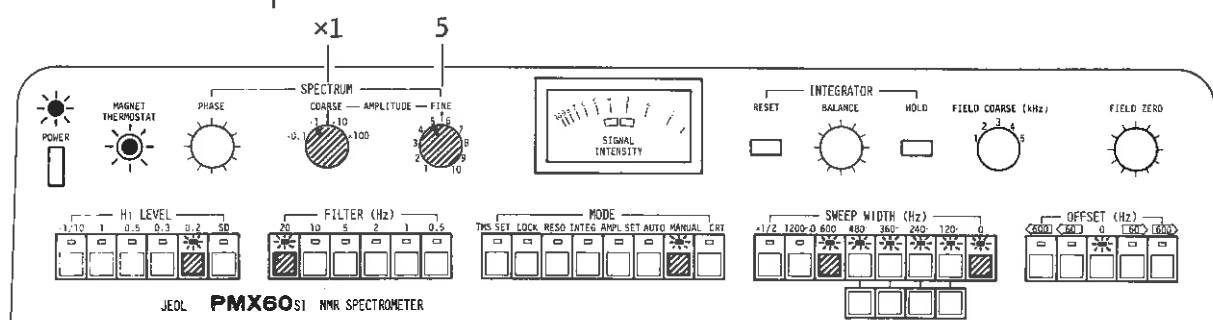
Note: Do not move the controls referred to in steps c. ~ k. once they have been set.

3.4 RESOLUTION ADJUST

3.4.1 RECORDER METHOD

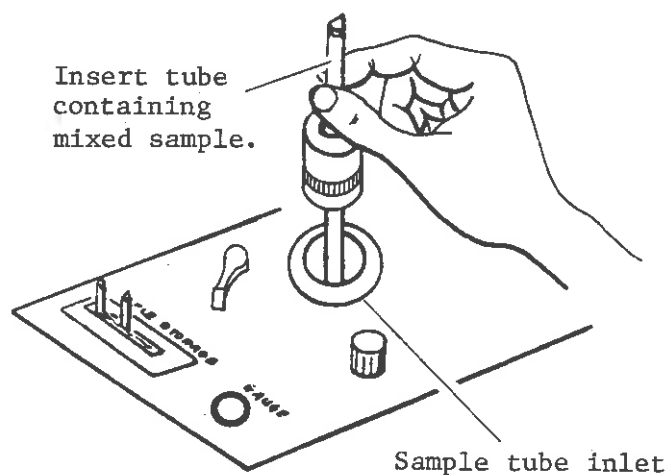
1. Spectrometer

AMPLITUDE-COARSE: $\times 1$
 FINE: 5
 H₁ LEVEL : 0.2
 FILTER : 20
 MODE : MANUAL
 SWEEP WIDTH : 600 $\sim$ 0



2. Sample tube insertion

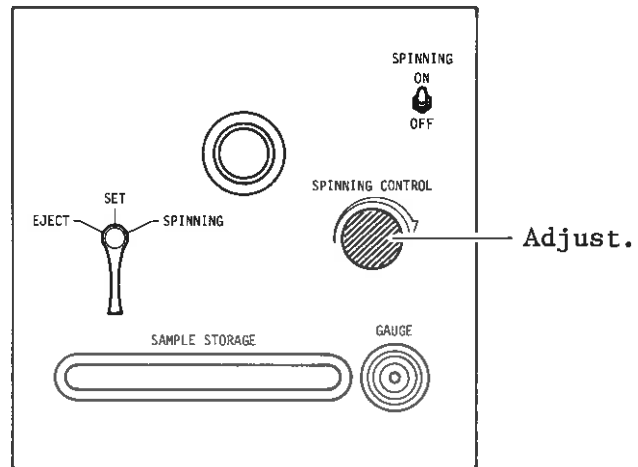
- a. Insert the sample tube and set the sample tube lever to SET.
- b. Sample tube lever: SPINNING



Note: Before inserting a sample tube, make sure that there is no sample tube already in the inlet.

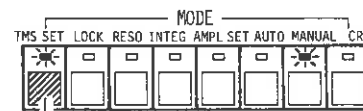
3. Spinning adjustment

- a. Rotate the SPINNING CONTROL knob fully counterclockwise.
- b. Turn the SPINNING CONTROL knob clockwise until a spinning rate of 30 Hz is realized. (Refer to Sect. 3.5.)

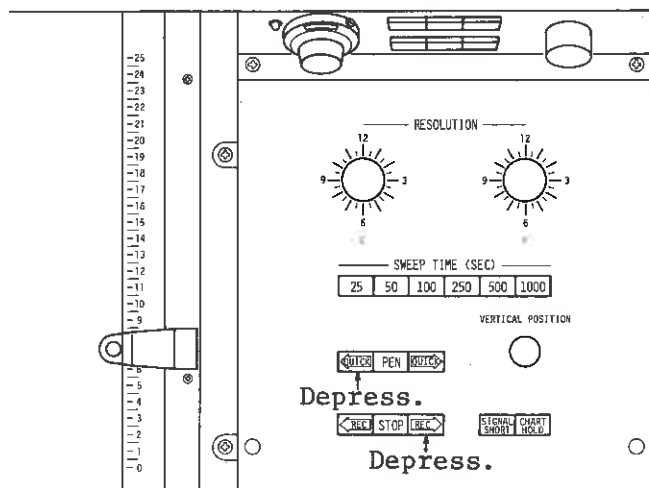
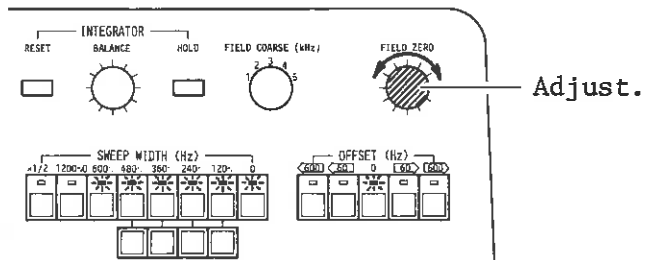


4. Adjust the FIELD ZERO knob so that a mixed sample signal can be recorded on the recorder chart paper.

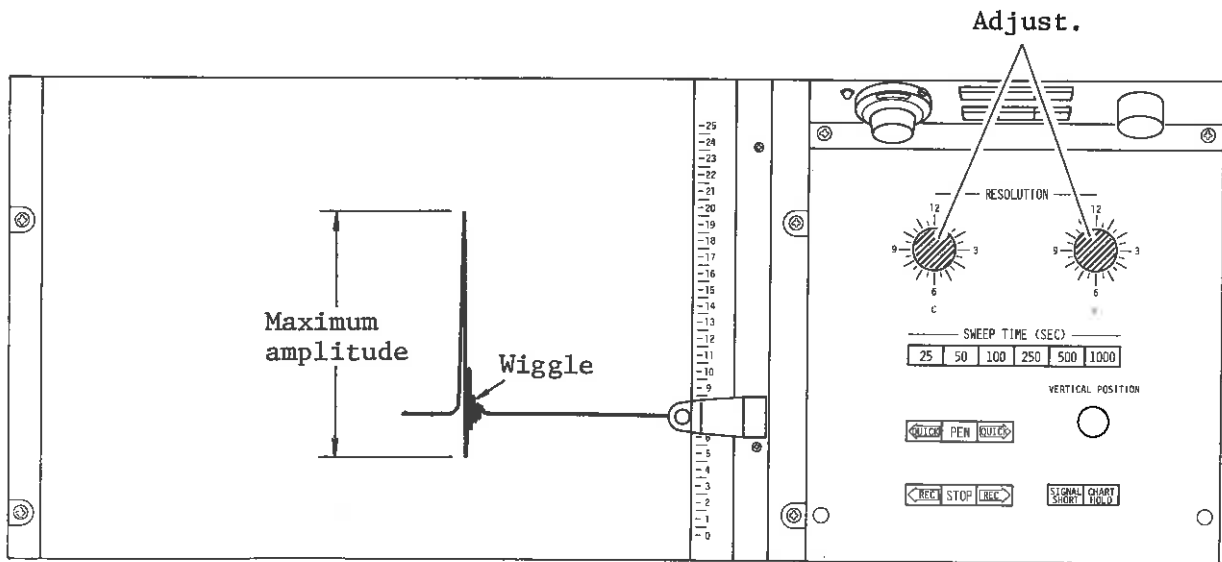
- a. MODE : TMS SET
- b. FIELD ZERO: Rotate fully counterclockwise.
- c. Gradually rotate FIELD ZERO clockwise until the recorder pen initially moves up and down. (The signal observed at this time will be the TMS signal.)
- d. MODE : Release TMS SET.
- e. Depress **QUICK** to set the carriage arm to the far left.
- f. Depress the **REC** and PEN buttons to record a mixed sample signal.



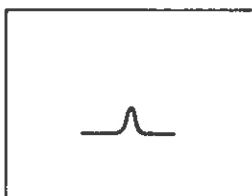
Push.



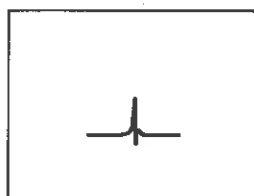
5. Alternately adjust the RESOLUTION Y and C knobs for maximum TMS signal amplitude and wiggle.



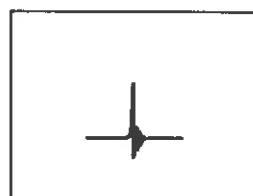
Stages of TMS signal resolution are shown below.



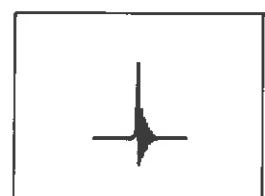
Poor



Slightly improved



Good



Optimum

3.4.2 OSCILLOSCOPE METHOD

1. Repeat steps 1 ~ 3 of Sect. 3.4.1, Recorder Method

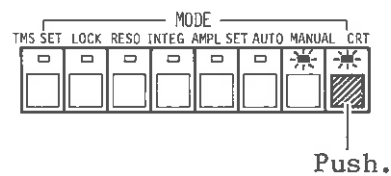
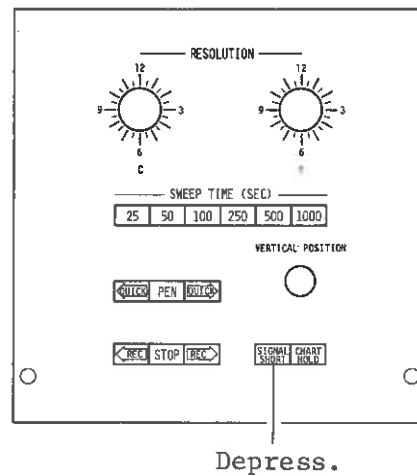
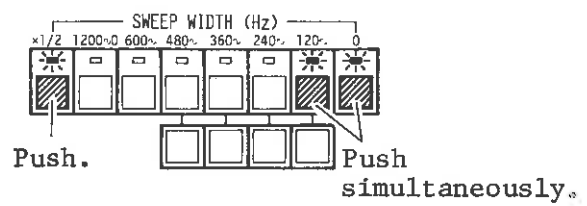
2. Signal Observation by Oscilloscope.

- a. Repeat steps a, b, and c of Item 4, Sect. 3.4.1.

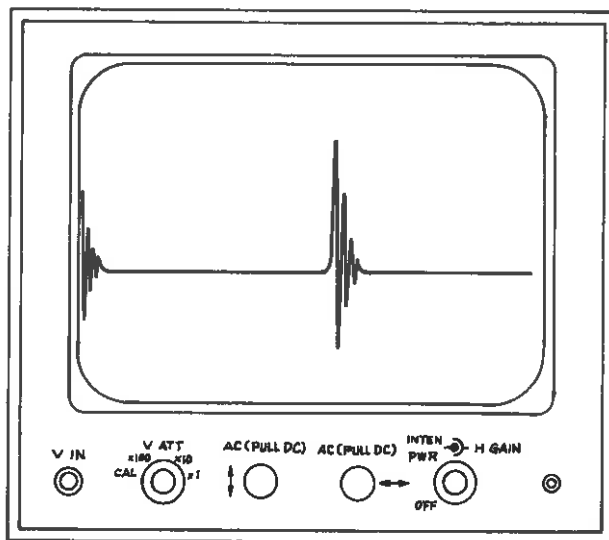
- b. SWEEP WIDTH : 120 ~ 0
× 1/2

- c. SIGNAL SHORT: Push

- d. MODE : CRT

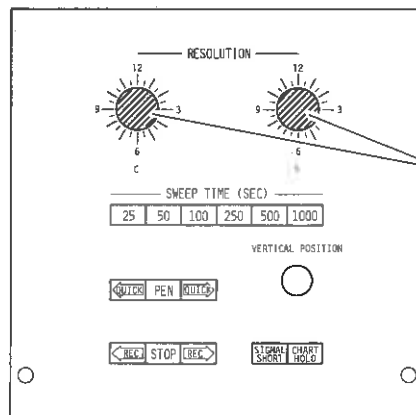


Signal observed on oscilloscope

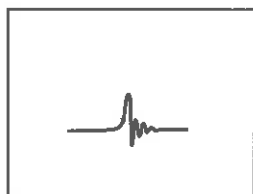


3. Adjust the RESOLUTION Y and C knobs so that the TMS signal wiggle hold time is maximum.

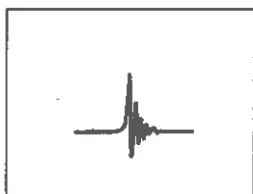
The initial peak of the wiggle should be about 90% of the TMS main peak.



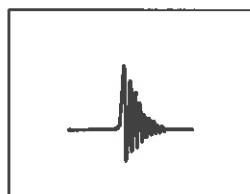
Stages of TMS signal resolution are shown below.



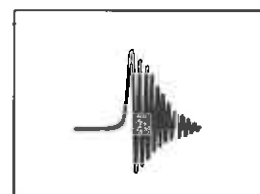
Poor



Slightly improved



Good

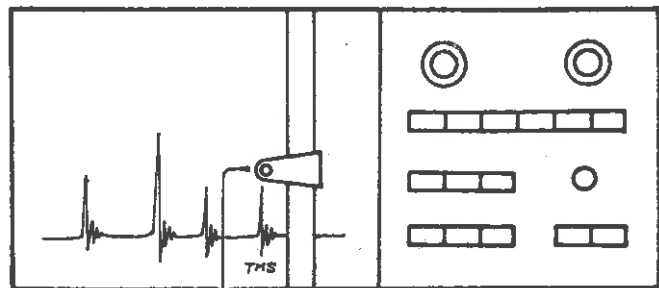


Optimum

3.4.3 SIGNAL INTENSITY METER METHOD

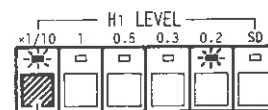
This method is used to obtain a resolution that is even higher than that previously obtained.

1. Lower the recorder pen on the lock signal peak, e.g., TMS.



Set pen to TMS peak.

2. H₁ LEVEL : $\times 1/10$



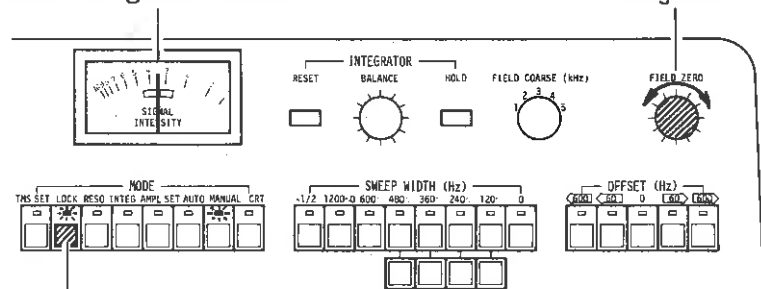
Push.

3. MODE : LOCK

Depress the HOLD key. After several seconds have passed, the SIGNAL INTENSITY meter needle will settle in the green area. Move the FIELD ZERO knob left and right. If the meter also moves left and right, lock is normal.

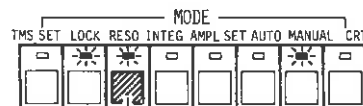
Set in green area.

Adjust.



Push.

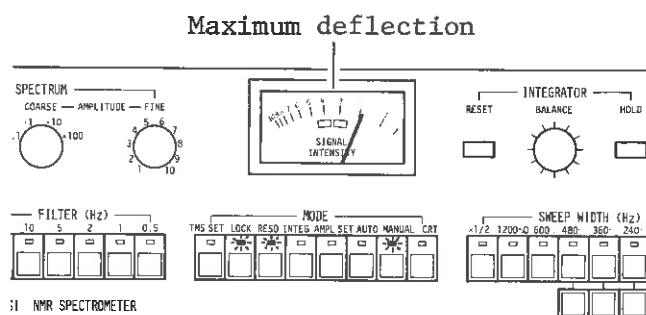
4. MODE : RESO



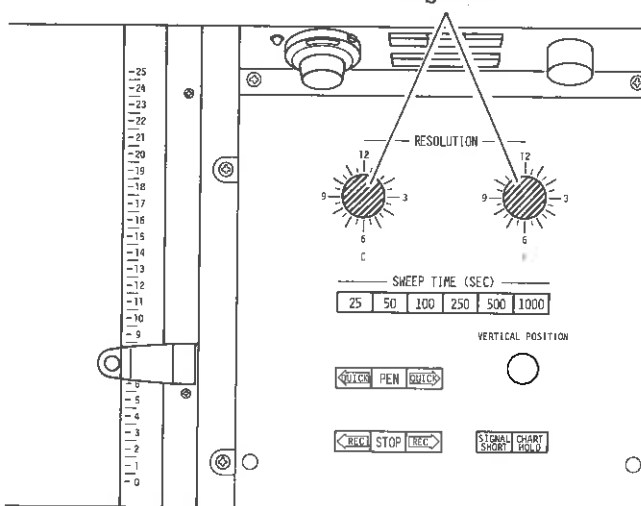
Push.

SIGNAL INTENSITY meter will deflect.

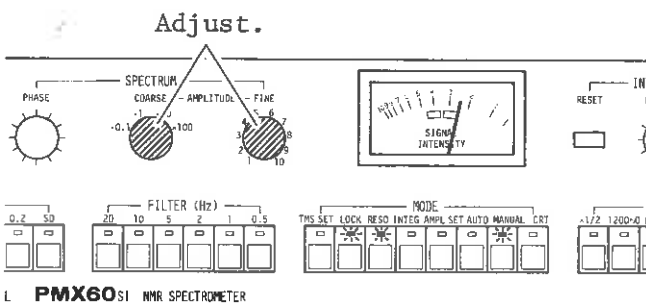
5. Adjust the RESOLUTION Y and C knobs for maximum meter deflection.



Adjust.



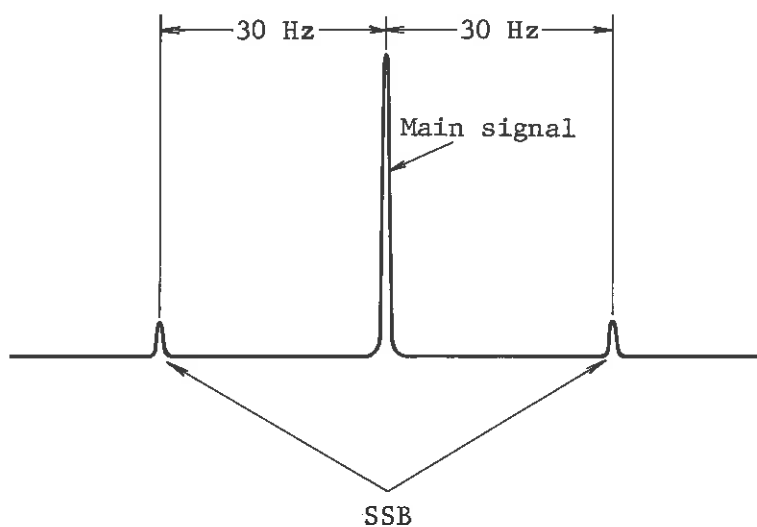
6. Adjust the AMPLITUDE knobs in the event of meter over-scale.



Note: In the event of insufficient magnet warm-up and a lack of thermal equilibrium, the signal will drift so that RESO will not function.

3.5 SPINNING ADJUSTMENT

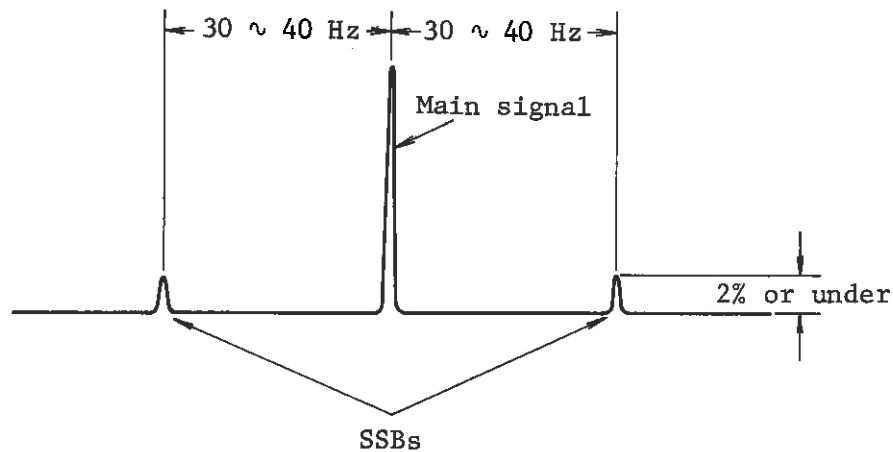
The ideal spinning rate is 30 ~ 40 revolutions per second. This rate can be confirmed by recording the spinning side bands (SSB). For example, in case the SSB is recorded at a point 30 Hz distant from the main signal, the rotation rate becomes 30 Hz (see diagram).



Note: When a good TMS waveform (high resolution waveform) cannot be obtained even after spinning adjustment, either consult an experienced NMR operator or repeat resolution adjustment from the beginning. Resolution will normally improve in 5 min. Never attempt to adjust the F control when adjusting resolution.

3.6 SPINNING SIDE BANDS (SSB)

When large SSBs are observed, set the spinner rotation rate to 30 ~ 40 rotations per sec. and adjust the X, Z, and YZ knobs to decrease SSB.



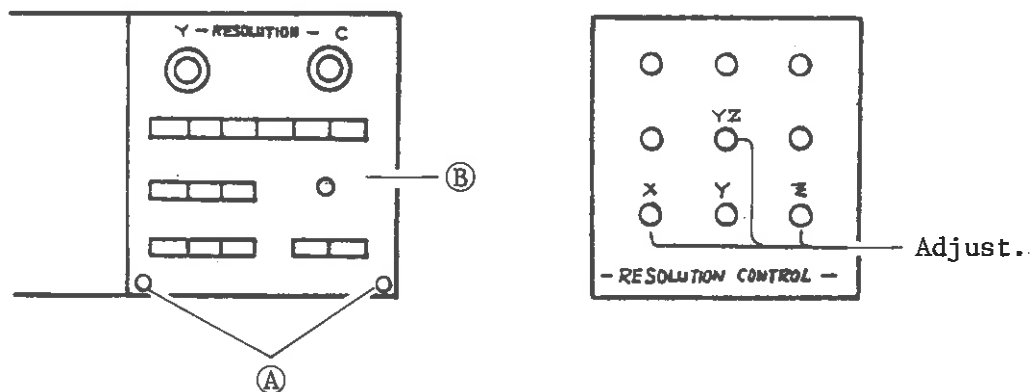
Notes (a): An SSB amplitude that is 2% of the main signal amplitude or less is a normal amplitude.

(b): Do not adjust any knob, other than X, Z and YZ.

(c): Although resolution drops when YZ is adjusted, it will return to its original value upon re-adjusting Y.

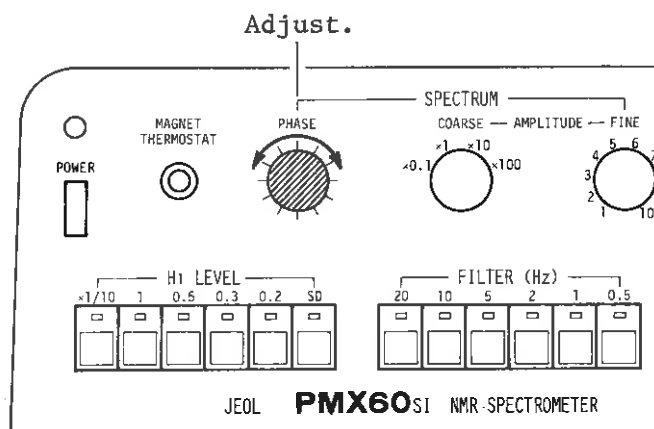
Adjusting X, Z and YZ knobs

Pull out knobs ① (see drawing). Then lift panel ② to gain access to the X, Z and YZ controls. After adjusting these controls, return panel ② to its former position.

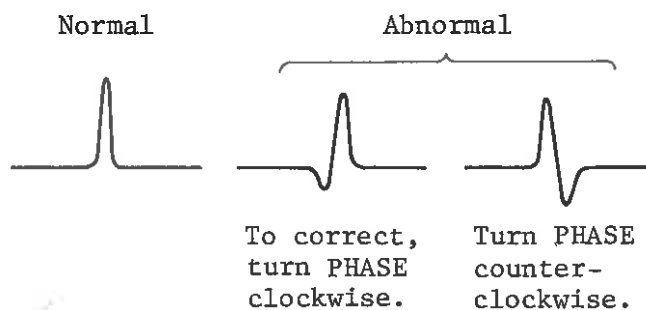


3.7 WAVEFORM ADJUSTMENT

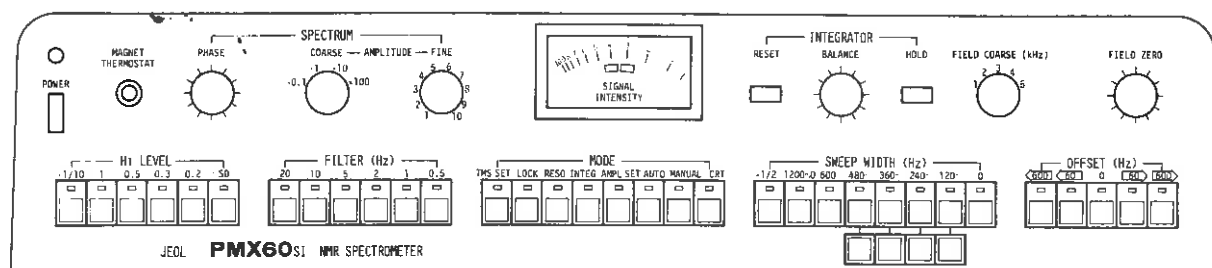
Adjust PHASE to obtain normal waveform.



Adjust the PHASE knob so that a normal waveform can be observed. In a normal waveform the base line level on either side of the peak is the same.



3.8 SIGNAL EXPANSION AND OFFSET



Use SWEEP WIDTH controls to expand signals.

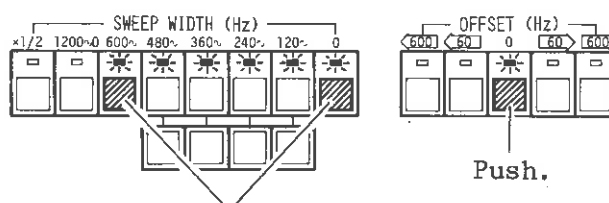
Use to offset signals.

3.8.1 SIGNAL EXPANSION

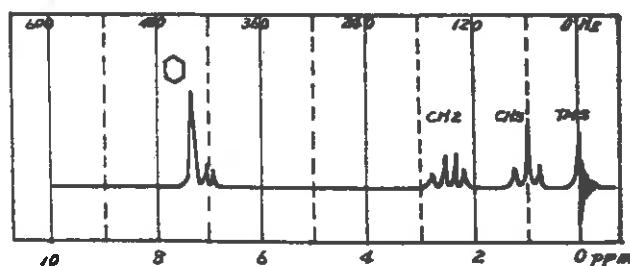
1. SWEEP WIDTH: Push 600~ and 0 keys simultaneously.

OFFSET : 0

Under these conditions record a signal of 90% ethylbenzene. (No. 427000467 is optional.)



Push simultaneously. (Lamps 600~, 480~, 360~, 240~, 120~ and 0 will go out.)

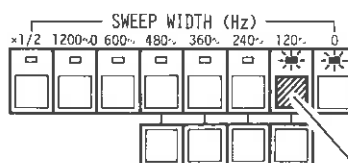


Signal of 90% ethylbenzene

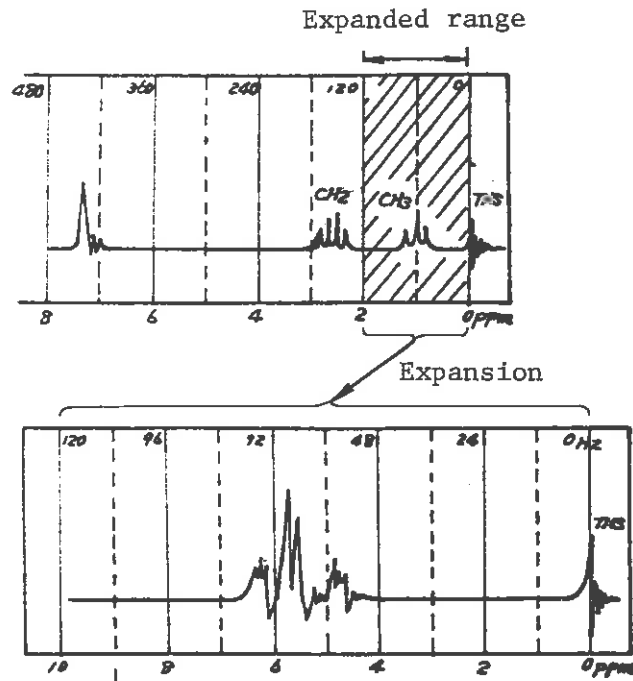
2. Expansion of 120 Hz width

- a. In case a signal in the 120 ~ 0 Hz range is expanded and recorded:

SWEEP WIDTH: Push 120~.



Push. (Lamps 120~ and 0 will go out.)

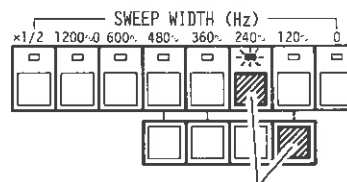


Signal of 0 ~ 120 Hz is expanded to full scale and recorded.

- b. In case a signal in the 240 Hz ~ 120 Hz range is expanded and recorded:

SWEEP WIDTH:

Push upper 240~
and lower 120~.

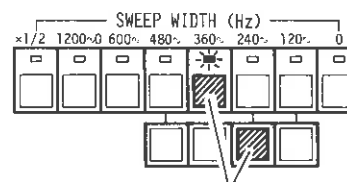


Push simultaneously
(240~ lamp will light.)

- c. In case a signal in the 360 Hz ~ 240 Hz range is expanded and recorded:

SWEEP WIDTH:

Push upper 360~
and lower 240~.



Push simultaneously
(360~ lamp will light.)

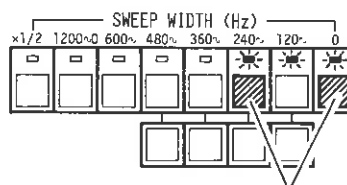
- d. Likewise, the signals of the respective ranges are expanded when the upper 480~ key and lower 360~, and upper 600~ and lower 480~, are pushed.

Note: In case the setting sequence is not continuous, depress the 600~ and 0 keys simultaneously. All lamps between these two keys will light up. Then set the desired width.

3. Expansion of 240 Hz width

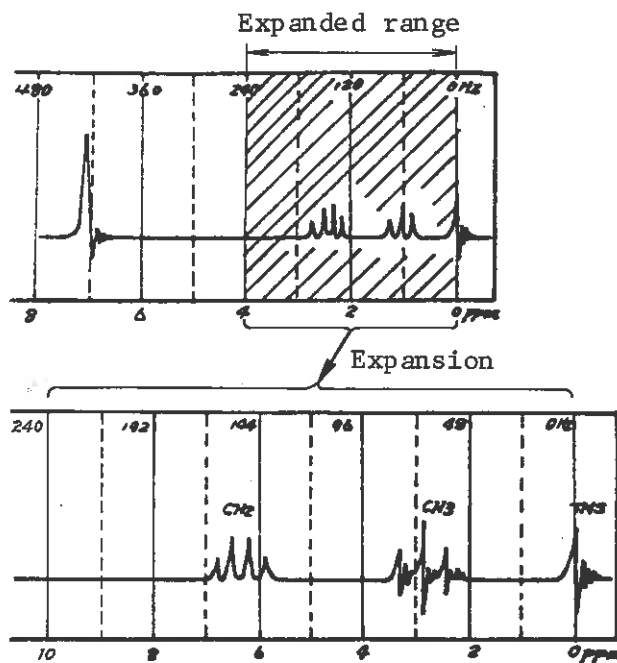
- a. In case a signal in the 240 ~ 0 Hz range is expanded and recorded:

SWEEP WIDTH: Push upper 240~ and 0 keys simultaneously.



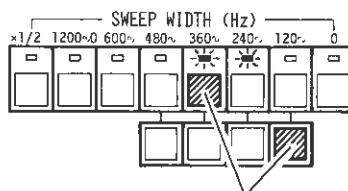
Push simultaneously. (240~, 120~ and 0 lamps will light.)

Signal in 240 ~ 0 Hz range is expanded to full scale.



- b. In case a signal in the 360 Hz ~ 120 Hz range is expanded and recorded:

SWEEP WIDTH: Push upper 360~ key and lower 120~.

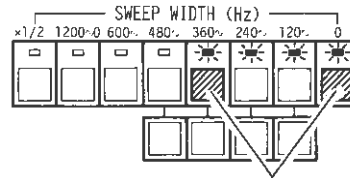


Push. (360~ and 240~ lamps will light.)

4. Expansion of 360 Hz width

In case a signal in the 360 Hz ~ 0 Hz range is expanded and recorded:

SWEEP WIDTH: Push upper 360~ and 0 keys simultaneously.

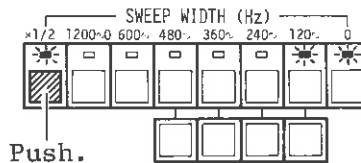


Push simultaneously. (360~, 240~, 120~ and 0 lamps will light.)

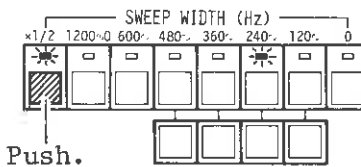
5. Using $\times 1/2$

SWEEP WIDTH: By pushing the $1/2$ key, all values of keys to the right are cut by $1/2$.

Example 1 As indicated below, full scale becomes 60 ~ 0 Hz.



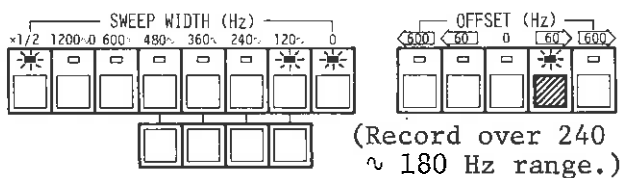
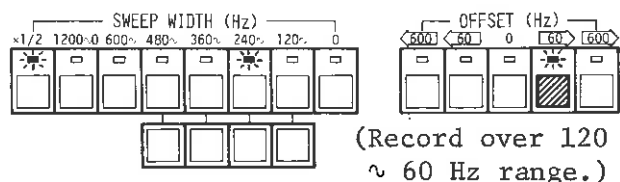
Example 2 As indicated below, full scale becomes 180 ~ 120 Hz.



Note: To release the $1/2$ rating, push the $1/2$ key once again.

3.8.2 SIGNAL OFFSET

When the $1/2$ and 120 ~ 0 keys are pushed, blanks appear between the 120 ~ 60 Hz values. To record over this range, set the OFFSET $\boxed{60}$ key. Likewise, to record over the 240 ~ 180 Hz range, push the 240~ and $1/2$ keys and set the OFFSET $\boxed{60}$ key. An example of a sweep range based on a combination of the sweep width and offset is given in the figure.



Observation range based on combined sweep width and offset
(Spectra within the arrow ($\longleftrightarrow$) range are recorded over full recorder scale.)

Example: Spectrum of mixed sample.

