

How to use Solid-State NMR Spectral Database (SSNMR_SD)

1. Home page
2. Search page
3. Display page of search results
4. Display page of spectral data
 - 4-1. NMR parameters
 - 4-2. Chemical shift references
 - 4-3. NMR spectra
 - 4-4. Pulse programs

1. Home page

SSNMR_SD consists of a home page, a search page, a display page of search results and display pages of spectral data. Although you can access SSNMR_SD freely, we ask you to agree the disclaimer each time when you use SSNMR_SD. When you click “I agree the disclaimer and use this database.” button at the bottom of the home page, you will move on to the search page which is an entrance of SSNMR_SD.

2. Search page

Users can search spectral data by using a substance name, an element and a nucleus (or a spin) as search items. Users do not need to input all the search items.

Click the “Search” button after inputting search items. Clicking the "Clear" button clears all the input values. Users can choose the number of hit results displayed per page by setting the pull down menu at the right side of the “Clear” button.

(a) Substance name

Users can use English and Japanese names. The wild card character (*) can be used.

(b) Element

Input an element. Use a blank or a comma (,) when you input more than one element.

When you input more than one element, substances containing all the input elements or none of the input elements are searched.

You can input the number of carbons contained. The lower limit and the upper limit are specified.

(c) Nucleus (spin)

Input the nucleus. Use a blank or a comma (,) when you input more than one nucleus.

You can select a nucleus by clicking the nucleus listed in the box. If you want to select more

than one nucleus from the box, click the nucleus during pressing Ctrl key for Windows and Linux or Command key for Mac.

3. Display page of search results

Search results are displayed in this page. The page presents information on the substances and nuclei of spectral data. When you click the nucleus, you move to Display page of spectral data.

4. Display page of spectral data

Display page of spectral data presents the nucleus, information on the substance, titles of NMR parameters and the titles of NMR spectra. Click one of the titles, and NMR parameters and NMR spectra are displayed.

The mark "Std" on the titles of NMR parameters and NMR spectra indicates that those are used in standard NMR measurements.

4-1. NMR parameters

NMR parameters such as chemical shifts (isotropic and anisotropic values), nuclear quadrupole interaction and so on are displayed. Assignment of each signal is presented when possible.

The mark "Std" indicates that the presented parameters are used in standard NMR measurements.

Several parameters displayed in NMR parameters are explained below:

(1) Assignment: Assignment of each signal is displayed. You can click a "diagram" button if present to see a structure schematic. When the schematic has numbers on atoms, for example, in the case of ^{13}C NMR spectra, C(3,5) means that signals of C(3) and C(5) are overlapped, whereas C(3/5) indicates that the signal is either C(3) or C(5). The mark * in the assignment like C(2*) means that the assignment is tentative.

(2) Peak position under MAS: Peak positions in MAS NMR spectra are displayed. Quadrupolar nucleus might show a peak position different from the chemical shift due to the second-order quadrupole shift. Peak positions are displayed when the chemical shift cannot be evaluated or when we want to display the peak position explicitly,

(3) Chemical shift (CS): Parameters of chemical shift interactions are displayed. Principal components of a chemical shift tensor δ_{11} , δ_{22} , δ_{33} are defined as to satisfy

$|\delta_{33} - \delta_{\text{iso}}| \geq |\delta_{11} - \delta_{\text{iso}}| \geq |\delta_{22} - \delta_{\text{iso}}|$. Isotropic shift δ_{iso} , Anisotropy $\Delta\delta_{\text{anis}}$ and asymmetry factor η_C are expressed as follows:

$$\delta_{\text{iso}} = \frac{1}{3}(\delta_{11} + \delta_{22} + \delta_{33})$$

$$\Delta\delta_{\text{anis}} = \delta_{33} - \frac{\delta_{11} + \delta_{22}}{2} = \frac{3}{2}(\delta_{33} - \delta_{\text{iso}})$$

$$\eta_c = \frac{\delta_{22} - \delta_{11}}{\delta_{33} - \delta_{\text{iso}}} \quad (0 \leq \eta_c \leq 1)$$

NMR signals are shifted by Knight shift when the substance is metallic. Knight shift has almost the same NMR property as the chemical shift. Consequently, this database does not differentiate Knight shift from the chemical shift.

(4) Quadrupole interaction (QI): Parameters of quadrupole interaction are displayed for quadrupolar nucleus ($I > 1/2$). Principal components of an electric field gradient tensor q_{11} , q_{22} , q_{33} are defined as to satisfy $|q_{33}| \geq |q_{11}| \geq |q_{22}|$. The principal components satisfy the relation $q_{11} + q_{22} + q_{33} = 0$. Quadrupole coupling constant QCC and asymmetry factor η_Q are expressed as follows:

$$QCC = \frac{e^2 Q q_{33}}{h}$$

$$\eta_Q = \frac{q_{22} - q_{11}}{q_{33}} \quad (0 \leq \eta_Q \leq 1)$$

(5) Euler angles between CS and QI: The axis system is transformed from chemical shift principal axis system (CS-PAS) to quadrupole interaction principal axis system (Q-PAS) with these angles.

(6) Indirect spin coupling (J): Parameters of indirect spin coupling are displayed.

(7) Fraction: Fractions of signals are displayed when two or more than two signals are present.

4-2. Chemical shift references

Chemical shift values in SSNMR_SD are defined with respect to the standard substances in Table 1. In other words, the chemical shifts of the standard substances are defined as 0 ppm.

Table 1. Standard substances for the chemical shift

Nucleus	Substance	Chemical formula	State
1H	tetramethylsilane	Si(CH ₃) ₄	neat
2H	deuterated water	D ₂ O	neat
3He	helium-3 gas	He	100 kPa
6Li	lithium chloride	LiCl	1.0 M aqueous solution
7Li	lithium chloride	LiCl	1.0 M aqueous solution
11B	borontrifluoride-ethylether complex	(C ₂ H ₅) ₂ O·BF ₃	neat
13C	tetramethylsilane	Si(CH ₃) ₄	neat
15N	nitromethane	CH ₃ NO ₂	neat
17O	water	H ₂ O	neat
19F	hexafluorobenzene	C ₆ F ₆	neat

23Na	sodium chloride	NaCl	1.0 M aqueous solution
27Al	aluminum nitrate	Al(NO ₃) ₃	1.0 M aqueous solution
29Si	tetramethylsilane	Si(CH ₃) ₄	neat
31P	phosphoric acid	H ₃ PO ₄	85% aqueous solution
33S	cesium sulfate	Cs ₂ SO ₄	1.0 M aqueous solution
35Cl	potassium chloride	KCl	solid
39K	potassium chloride	KCl	1.0 M aqueous solution
51V	vanadium(V) trichloride oxide	VOCl ₃	neat
59Co	potassium hexacyanocobaltate(III)	K ₃ Co(CN) ₆	saturated aqueous solution
63Cu	cuprous chloride	CuCl	solid
65Cu	cuprous chloride	CuCl	solid
77Se	dimethylselenium	Se(CH ₃) ₂	neat
79Br	potassium bromide	KBr	solid
87Rb	rubidium chloride	RbCl	1.0 M aqueous solution
93Nb	hexafluoronioobium ion	[NbF ₆] ⁻	48% HF aqueous solution
119Sn	tetramethyltin	Sn(CH ₃) ₄	neat
125Te	dimethyltellurium	Te(CH ₃) ₂	neat
127I	potassium iodide	KI	solid
129Xe	xenon gas	Xe	zero pressure
133Cs	cesium chloride	CsCl	1.0 M aqueous solution
195Pt	¹ H (tetramethylsilane) x 0.214	Si(CH ₃) ₄	neat
207Pb	tetramethyllead	Pb(CH ₃) ₄	neat

4-3. NMR spectra

An NMR spectrum is displayed as well as a list of peak positions. NMR measurement parameters are displayed when one clicks the "Measurement Parameters" button. The pulse program diagram is displayed when one clicks the pulse program name in the Measurement Parameters window.

The mark "Std" indicates that the presented spectrum is used as a reference in NMR measurements.

Several parameters displayed in "Measurement Parameters" are explained below:

- (1) Temperature: The temperature indicated by the temperature controller is displayed usually. "Temperature (calibrated)" is used when the temperature is calibrated. "Temperature (nominal)" is used when the uncalibrated temperature is displayed as well.
- (2) MAS/static: Sample spinning mode is displayed. "MAS" is magic angle spinning, "VAS" is variable angle spinning, "off-MAS" is off-magic angle spinning and "static" is a static sample. The angle between the spinning axis and the magnetic field is displayed when the sample is spinning at the angle other than the magic angle.

- (3) Spinning rate: The spinning rate of the sample is displayed. When the sample is static, this parameter is not displayed or is 0 kHz.
- (4) Probehead: “MAS” is MAS probehead, “BB” is a broadband probehead with a solenoid coil and “HR” is a high resolution probehead for solution.
- (5) Observed frequency: The frequency corresponding to the 0-ppm shift position is displayed. This value is used when the unit is converted between Hz and ppm.
- (6) Decoupling frequency: The pulse frequency of the decoupling pulse is displayed.
- (7) The pulse widths in Instrument: They are determined for a substance with $QCC=0$ in the case of quadrupolar nucleus.

4-4. Pulse programs

Pulse programs used to acquire the spectral data are listed in Table 2.

Table 2. Pulse programs

Abbreviation	Full name
CP	cross polarization
CPTPPM	cross polarization with TPPM decoupling
HD	single pulse with high power decoupling
INEPT-DEC	INEPT with decoupling
INEPT-NON	INEPT without decoupling
QE	solid echo/quadrupole echo
SE	spin echo
SP	single pulse
VACP	cross polarization with variable pulse amplitude for observed spin