Efficient design of multituned transmission line NMR probes: The electrical engineering approach

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A B S T R A C T
Transmission line-based multi-channel solid state NMR probes have many advantages regarding the cost of construction, number of RF-channels, and achievable RF-power levels. Nevertheless, these probes are only rarely employed in solid state-NMR-labs, mainly owing to the difficult experimental determination of the necessary RF-parameters. Here, the efficient design of multi-channel solid state MAS-NMR probes employing transmission line theory and modern techniques of electrical engineering is presented. As technical realization a five-channel (1H, 31P, 13C, 2H and 15N) probe for operation at 7 Tesla is described. This very cost efficient design goal is a multi port single coil transmission line probe based on the design developed by Schaefer and McKay. The electrical performance of the probe is determined by measuring of Scattering matrix parameters (S-parameters) in particular input/output ports. These parameters are compared to the calculated parameters of the design employing the S-matrix formalism. It is shown that the S-matrix formalism provides an excellent tool for examination of transmission line probes and thus the tool for a rational design of these probes. On the other hand, the resulting design provides excellent electrical performance. From a point of view of Nuclear Magnetic Resonance (NMR), calibration spectra of particular ports (channels) are of great importance. The estimation of the π/2 pulses length for all five NMR channels is presented.

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1. Introduction

Nuclear Magnetic Resonance (NMR) is one of the major spectroscopic techniques for the study of liquid or solid biological and chemical systems. Especially in the field of solid state NMR the last years have seen strong advances in both the design of new experimental schemes and in the experimental hardware, the latter in the emergence of high-field NMR spectrometers with magnetic field of 14–21 Tesla and more.

NMR techniques require both, to deliver the high radio frequency (RF) power via resonant circuit to a sample coil placed inside of an extremely strong, superconducting magnet, and to receive very weak RF signals generated by the same sample. The sample coil including the sample under examination (the sample coil) acts simultaneously like a receiver and a generator. An NMR probe has to be able to deliver an RF high power from a low noise RF spectrometer’s amplifier through the input/output connector of the NMR probe to the sample coil, where the power is to be entirely utilized, as well as to reveal the smallest possible energy losses of the signal generated by the sample afterwards. The detected low RF power is to be next delivered to the spectrometer’s receiver. [3] The high RF power mentioned above is typically in the range of 50–1200 W. In many advanced solid state NMR experiments the sample is irradiated simultaneously with several RF fields B1 at different frequencies. The emergence of these advanced sequences for solid state NMR and the ongoing increase of the available magnetic fields lead to an increasing demand on designing, modelling and developing new types of NMR probes.

The most common types of modern solid state NMR probes are two or three channel probes built on the base of lumped elements (mainly capacitors and inductivities) and equipped with a single
RF sample coil. Each input/output port is equipped with two variable capacitors, connected parallel and in series, playing the roles of a tuning and matching circuit for the RF-coil to the desired frequencies. In the language of electrical engineering such a probe is a two or three port lumped element common coil frequency multiplexer. Owing to the relatively limited space inside the magnet, conventional resonance circuit designs with lumped elements are limited to three to four simultaneous resonance frequencies even in so-called widebore magnets (approximately 89 mm bore diameter). Moreover, these probes are very difficult to construct, owing to the necessity to accommodate a large number of electrical tuning elements, mechanical elements for the MAS, and the sample tempering. As a result of these difficulties most triple or quadrupole resonance solid-state NMR probes are from commercial vendors with typical prices on the order of $50k and above, and often not available for all magnetic field strengths. Finally the change of NMR-nuclei usually employs the replacement of electrical plugin, and extensive retuning and rematching of the channels. Thus performing a set of experiment with different nuclei on the same sample in a row is often not feasible with conventional probes.

For these reasons about a decade ago Schaefer and McKay developed and patented an ingenious alternative design, based on transmission lines, which they named a “Multi-tuned Single Coil Transmission Line Probe for Nuclear Magnetic Resonance Spectrometer”. With the exception of the RF-coil in the MAS spinning module, practically all circuit elements are outside the magnet bore. In such a design the lumped elements are replaced by distributed ones. Their particular probe was built for the resonance frequencies of the $^1$H, $^19$F, $^{31}$P, $^{13}$C, $^{15}$N and $^2$H nuclei in the 11.4 Tesla magnet of a 500 MHz spectrometer. [3,12] Their particular choice of nuclei was guided by the needs of typical organic or bioorganic samples. Following this patent the Schaefer group did construct several other probes for different field strengths. Since most elements of such a probe are available in a typical hardware store and the MAS-stator is the only expensive element, the construction of such a probe can be done for a price which is roughly about 20% of the price of a commercial probe. Despite this tremendous cost advantage these probes are employed by only a very small number of solid-state NMR labs. The main difficulty, which hampered the spreading of this design is the relatively complex construction of the transmission line elements, due to the narrow-bandedness of these elements. This narrow-bandedness requires that the design of transmission line probe has to be adapted at each magnetic field strength to the set of the characteristic frequencies of nuclei. This necessitates a tedious experimental optimization procedure of the different elements by experts in RF-technology or HAM radio. Detailed descriptions of the transmission line theory can be found in Refs. [2,4–11].

In the following we show that this optimization procedure can be strongly simplified by modern tools of electrical engineering. By numerical simulation of the electrical parameters of the probe employing commercially available software, the relevant parameters of the design can be calculated. The relevant RF-parameters are: the inductance and resistivity of the coil, determined by its material, number of turns, spacing, length, and diameter, the length of the particular transmission line segments, capacitor placement and capacity values. If a commercially available spinning-module is employed, the coil-parameters are fixed, and the lengths of the various transmission line segments are the primary optimizeable parameters. The starting point for design of the transmission line probe was a measurement of the sample coil impedance using a vector analyzer. For this the sample coil was connected to an approximately 1 m long transmission line.

The next step in the design is the approximate determination of the first- and subsequent impedance minima for the given NMR-frequencies. These minima act as junction points for additional transmission line stubs which serve as connectors to the input/output ports of the frequencies. According to the patent by Mc Kay and Schaeffer, [3] the length of these transmission line stubs cannot exceed the quarter wave length. The procedure of the impedance minimum estimation for the next frequency was performed by a simple impedance measurement at the minimum impedance point of the higher frequency, while the input/output port was already mounted and terminated with pure resistive impedance of 50 Ω. The searched minimum point was calculated on the base of transmission line theory supported by Smith charts. After the next input/output port was attached and terminated with a pure resistive load of 50 Ω, the impedance minimum point was checked again, and the whole procedure was repeated until the last input/output port was successfully installed.

To simplify the design and operation of the probe only capacitors were included as lumped input/output elements in the construction. To avoid too large capacity values of these input/output capacitors, which would be technically not achievable, their capacity was employed as an optimization constraint in the design. Both the final result of numerical simulation performed using a standard computer program and the result of measurements are presented in next chapters.

As a real world example of this approach the construction of a five-channel ($^1$H, $^{13}$P, $^{13}$C, $^2$H and $^{15}$N) transmission line probe, designed to operate in a field of 7 Tesla is described and the evaluation its electrical performance is given below. Finally, in order to illustrate the NMR performance of the built transmission line probe, calibration measurements of all channels are presented.

2. Scattering matrix parameters

From the point of view of electrical engineering, a multi-tuned transmission line probe is a multi port frequency multiplexer with a single sample coil. A successful modelling of a transmission line probe necessitates a mathematical framework, which is powerful enough to treat real-world circuit elements. In the microwave frequency region the most commonly used model to analyze circuits employs the scattering matrix parameters or more shortly the “S-parameters”. The stimulus signal is treated as a travelling RF wave incident upon the device, and the response signals as a second RF wave reflected from the device or transmitted through the device. A set of equations can be established relating these incident and “scattered” waves, which will completely characterize the device or network [1].

The five port device is part of a transmission line network, where the transmission lines are terminated with their characteristic impedances. Referring to the Fig. 1, $U_{1(1)}$, $U_{1(2)}$, $U_{1(2)}$, $U_{1(2)}$, $U_{1(2)}$, ..., $U_{1(2)}$, $U_{1(2)}$, are the exciting and reflected voltages, which are exciting the network or are reflected from ports 1, 2, ..., 5, respectively. From these measurable quantities, a set of network equations can be written and presented in a matrix form, as follows [1]:

$$
\begin{bmatrix}
U_{1(1)} \\
U_{1(2)} \\
U_{1(3)} \\
U_{1(4)} \\
U_{1(5)}
\end{bmatrix} =
\begin{bmatrix}
S_{11} & S_{12} & S_{13} & S_{14} & S_{15} \\
S_{21} & S_{22} & S_{23} & S_{24} & S_{25} \\
S_{31} & S_{32} & S_{33} & S_{34} & S_{35} \\
S_{41} & S_{42} & S_{43} & S_{44} & S_{45} \\
S_{51} & S_{52} & S_{53} & S_{54} & S_{55}
\end{bmatrix}
\begin{bmatrix}
U_{1(1)} \\
U_{1(2)} \\
U_{1(3)} \\
U_{1(4)} \\
U_{1(5)}
\end{bmatrix},
$$

(1)
where \([S]\) is the scattering- or S-parameter matrix. Dividing both sides of this equation by the square root of the characteristic impedance converts the voltages to their equivalent power levels [1]:

\[
\begin{bmatrix}
  b_{(1)} \\
  b_{(2)} \\
  b_{(3)} \\
  b_{(4)} \\
  b_{(5)}
\end{bmatrix} =
\frac{1}{\sqrt{Z_0}}
\begin{bmatrix}
  S_{11} & S_{12} & S_{13} & S_{14} & S_{15} \\
  S_{21} & S_{22} & S_{23} & S_{24} & S_{25} \\
  S_{31} & S_{32} & S_{33} & S_{34} & S_{35} \\
  S_{41} & S_{42} & S_{43} & S_{44} & S_{45} \\
  S_{51} & S_{52} & S_{53} & S_{54} & S_{55}
\end{bmatrix}
\begin{bmatrix}
  a_{(1)} \\
  a_{(2)} \\
  a_{(3)} \\
  a_{(4)} \\
  a_{(5)}
\end{bmatrix},
\]

(2)

where:

- \(a_{(1)}, a_{(2)}, \ldots, a_{(5)}\) are the power waves incident upon the port \(1, 2, \ldots, 5\);
- \(b_{(1)}, b_{(2)}, \ldots, b_{(5)}\) are the power waves reflected from the port \(1, 2, \ldots, 5\), respectively.

The elements \(S_{ij}\) of the column \(j\) of the Scattering matrix can be determined experimentally by setting all input powers except \(a_j\) to zero [2]:

\[
S_{ij} = \frac{b_i}{a_j} \quad \text{for} \quad k = 1, 2, \ldots, 5, \quad k \neq j.
\]

(3)

and

\[
S_{ij} = \frac{a_k}{a_j} \quad \text{for} \quad k = 1, 2, \ldots, 5, \quad k \neq j.
\]

(4)

so the calculated power ratio is

\[
|S_{ij}|^2 = \frac{|b_i|^2}{|a_j|^2}, \quad i, j = 1, 2, \ldots, 5.
\]

(5)

For the case of the typical standard impedance of \(Z_0 = 50 \, \Omega\), the condition \(a_k = 0\) is achieved by terminating the ‘K’- input/output port with a 50 \, \Omega load (reference impedance).

Although the S-matrix looks rather abstract there is a direct physical interpretation of the S-Parameters:

- \(S_{ii}\) is a measure for the mismatch between required standard impedance of \(Z_0 = 50 \, \Omega\) and the tuning and matching conditions achieved in the multiplexer. The ideal value of this parameter would be 0 (no reflection at all). For practical purposes a value of \(-19 \, \text{dB}\) or less is sufficient, which corresponds to roughly 1\% power mismatch.
- The squares of the off-diagonal elements of the scattering matrix of a multi-channel NMR probes \((S_{ij}, i \neq j)\) measure the mutual isolation of the ports. This isolation is important for the practical application of the probe. The ideal value is again 0, however in practice, a slightly worse performance compared to the diagonal elements is often tolerable because additional band pass filters may be applied to improve the isolation condition.

The estimated and measured values of \(S_{ii}\) of the NMR probe are compared in this paper.

3. Results and discussion

3.1. NMR probe construction

In the following we describe the construction and electrical performance of a new five frequency transmission line NMR probe of the Schaefer and McKay-type with a single sample coil. This NMR probe is designed to operate at a field of 7 Tesla, which corresponds to operating frequencies in the range of approximately 30–300 MHz.

Its circuit diagram is shown in Fig. 2. At the magnetic field of 7 T the Larmor frequencies for the NMR active nuclei: \(^1\text{H}, ^{31}\text{P}, ^{13}\text{C}, ^{2}\text{H}, ^{15}\text{N}\) are \(f_1 = 299.99 \, \text{MHz}, f_2 = 121.44 \, \text{MHz}, f_3 = 75.44 \, \text{MHz}, f_4 = 46.01 \, \text{MHz}\) and \(f_5 = 30.40 \, \text{MHz}\), respectively. These five frequencies are the operating frequencies of the NMR probe. The coaxial transmission lines are built as concentric copper conductors, which are created employing standard copper tubings of 6.6 cm inner diameter for the outer and 1.8 cm outer diameter for the inner copper tube. Copper is used as a constructing material not only because of its very low resistivity, but also because of its nonmagnetic properties [12] and its easy availability as prefabricated copper tubes. The multiplexer is built by connecting these coaxial transmission lines at the proper impedance conditions of the individual frequencies (see below). The inner tubes are connected by soldering, the outer tubes are connected mechanically via T-shaped joints. The whole circuit is terminated by the impedance of the sample coil. Its value can be taken from the Smith chart 3. The sample coil is part of a standard Varian/Chemagnetics 4 mm spinning module (sample spinning speed up to 16 kHz). The characteristic impedance \(Z_{\text{TL}}\) of this type of transmission lines amounts to 78.1 \, \Omega. Two adjustable capacitors in each input/output port for the frequencies \(f_1\)–\(f_5\) act as tuning and matching circuits, and match the impedance of the

![Fig. 1. A five port device terminated in Z1–Z5 impedance.](image-url)

![Fig. 2. Schematic diagram of our Schaefer and McKay type single coil transmission-line frequency multiplexer NMR-probe.](image-url)
system to the required 50 Ω impedance standard of the RF-channels of the NMR spectrometer in order to minimize the energy losses.

The most critical point of this frequency multiplexer design and modelling is the selection of the proper length of the coaxial line segments. As explained by Schaefer and McKay in detail, these segments have to be chosen in such a way that the various RF-channels are electrically separated from each other as good as possible. From transmission line theory it is known, that the impedance values repeat periodically every one half wave length along the line [6–11], similar to a standing wave. The simplest possible example of an NMR transmission line probe is a frequency multiplexer which is reduced to only one port for one particular frequency [13]; in practice a piece of a coaxial transmission line of a correct length depending on the frequency \( f_i \), terminated with a sample coil on one side, and equipped with two variable capacitors in the input/output port on another side. The two variable capacitors must be placed at a point, where the input impedance of the transmission line is inductive. The location of such a point can be easily done by standard impedance measurement techniques. The junction point for the next multiplexer segment (for \( f_2 \) port) must have an impedance minimum at the already existing port \( f_1 \). The same is true for the \( f_3 \) port, which must have an impedance minimum at the \( f_2 \) port and so on. By attaching the \( n \)-th port at an impedance minimum, it is electrically isolated from ports \( 1, \ldots, n-1 \). This isolation minimizes the power going from the \( 1, \ldots, n-1 \) ports to the \( n \)-th port and makes the tuning of the \( 1, \ldots, n-1 \) ports independent on the \( n \)-th port. To avoid an extensive trial and error scheme in the construction, a theoretical simulation of the real transmission line is advisable. Such a simulation is only possible if the full complex impedance values of the load (sample coil) are known, since the sample coil impedance is not purely inductive. Thus, for a good simulation it is indispensable to measure the real coil impedance characteristic as a function of the RF frequency. The measured impedance of our sample coil is presented in Fig. 3. The design goal is to transform this impedance and finally match it at particular frequencies in all \( f_1-f_5 \) ports with the desired system impedance (\( Z_0 = 50 \Omega \)) [12]. Following the impedance values of the sample “coil” and proceeding in the way described above, “step by step” all minimum impedance points (junction points) for the concerned frequency multiplexer are determined. At the end, following the chart (see Fig. 2), the final optimizations of tuning and matching capacities can be performed numerically. The values of all capacitors were constrained in the interval of 1–50 pF, since the technical construction of the capacitors does not allow them to achieve higher values.

The optimization of all capacities was performed by use of the computer program ADS 2004 provided by Agilent Technologies. Optimization results, excluding capacity values which are beyond the scope of this paper, are discussed in next chapters. The final design-scheme can be obtained by the authors on demand.

3.2. Electrical performance

A comparison of the optimization results and the measured values of the reflection coefficients \( S_{11}, S_{22}, S_{33}, S_{44}, S_{55} \) in particular ports of Schaefer and McKay-type multiplexer is shown in the following. The first pair of drawings in Fig. 4 illustrates \( S_{ii} \) values [dB] versus frequency [MHz] in the full NMR frequency band width at a magnetic field of 7 T without additional RF-filters. Fig. 5 displays the \( S_{ii} \) dependence in the narrow band close to the frequency of interest. These experimental curves are measured with the help of additional RF-filters. These filters cause small changes of these curves as compared to the non-filtered curves in Fig. 4 and influence the tuning and matching conditions of the multiplexer. The best possible performance (the best tuning and matching sets) of the NMR probe are presented in Fig. 5; the pass band of applied filters is accordingly commented. In the first port no filter for 300 MHz is applied. The simulated and measured \( S_{ii} \) values remain in a good agreement. The measured \( S_{ii} \) values are frequently even better than the simulated ones, and the level of 30–40 dB is absolutely acceptable.

The fraction of the applied power which reaches the sample coil is essential for the performance of the NMR probe. This fraction can be estimated with the help of the ADS program by calculating the off-diagonal transmission \( S_{ij} \)-parameters (where \( i \neq j \)). They are measured for the possible RF power losses. Without additional filters, both theoretical and measured \( S_{ii}, i \neq j \), values are in good agreement. All combinations of transmission
Fig. 4. Simulated (on the left) and measured (on the right side) reflection S-parameters ($|S_{ii}|$ in dB versus frequency [MHz], $|S_{11}|$ on the top, $|S_{55}|$ on the bottom) of the frequency multiplexer. No filters applied.
S-parameters for the real NMR probe are presented in Fig. 6 and Table 1. The reason for attaching the filters during S-matrix parameter measurements and simulations was to monitor the probe, which is a real electric system, under normal work condition in an NMR experiment. The real energy dissipation, when filters are attached, can be concluded indirectly from the ratio of power levels and 90° pulse lengths resulting from nutation experiments performed for particular NMR nuclei. The result of nutation experiments are presented and discussed in the next section.

In the optimization with the ADS program no additional band pass filters were employed. The experimental performance of the
NMR probe is improved with reference to the simulated one. The spectral analysis shows that, with the exception of the \( S_{23} \) and \( S_{25} \) port, all ports are mutually well-separated on the level of \(-18\, \text{dB}\) or better. The values of \( S_{23} \) and \( S_{25} \) could be improved by a more selective, narrower band filter applied to the port \( f_2 \) at 121.44 MHz.

3.3. NMR performance

The tuning and matching of probes for NMR and the determination of the electrical performance is only the first step in the development of a new probe. The results of this procedure, as stated above, can be successfully characterized by S-parameters.
The decisive step is the experimental test of the NMR-performance of the probe. Since the transmission and receiving properties of the probe are complementary, the NMR-performance of a probe in terms of sensitivity and S/N-ratio can be estimated by finding the proper values of pulse lengths and power levels related to a particular NMR active nucleus. Here shorter pulse lengths correspond to a higher sensitivity of the probe.

The NMR performance of the five channels of the probe is tested by studying simple model systems. The calibration of the $^1$H, $^{31}$P, $^{13}$C, $^2$H and $^{15}$N 90° pulse width via nutation experiments are presented in the supporting information. Such an array of experiments results in 13.7 μs and the corresponding $B_1$-field amounts to 18 kHz. The measured NMR line width is 63 Hz.

All resulting values of the $B_1$-fields are additionally collected in Table 2.

### Table 1

S$_9$ parameter values, according to Fig. 6.

<table>
<thead>
<tr>
<th>$S_9$</th>
<th>Meas. (dB)</th>
<th>Frequency (MHz)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$t_i$</td>
<td>Pass band</td>
<td>$t_j$</td>
</tr>
<tr>
<td>S$_{12}$</td>
<td>−20.6</td>
<td>299.99</td>
</tr>
<tr>
<td>S$_{13}$</td>
<td>−19.9</td>
<td>299.59</td>
</tr>
<tr>
<td>S$_{14}$</td>
<td>−18.3</td>
<td>299.99</td>
</tr>
<tr>
<td>S$_{15}$</td>
<td>−25.7</td>
<td>299.99</td>
</tr>
<tr>
<td>S$_{23}$</td>
<td>−6.2</td>
<td>121.44</td>
</tr>
<tr>
<td>S$_{24}$</td>
<td>−18.2</td>
<td>121.44</td>
</tr>
<tr>
<td>S$_{25}$</td>
<td>−15.2</td>
<td>121.44</td>
</tr>
<tr>
<td>S$_{26}$</td>
<td>−69.4</td>
<td>75.44</td>
</tr>
<tr>
<td>S$_{27}$</td>
<td>−57.7</td>
<td>75.44</td>
</tr>
<tr>
<td>S$_{28}$</td>
<td>−17.6</td>
<td>30.4</td>
</tr>
</tbody>
</table>

### Table 2

90°-pulse width, $B_1$-field strength at the particular power for all five nuclei.

<table>
<thead>
<tr>
<th>Channel</th>
<th>$90^\circ$-pulse (μs)</th>
<th>$B_1$-field strength (kHz)</th>
<th>Incident power (W)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$^1$H</td>
<td>1.5</td>
<td>166</td>
<td>250</td>
</tr>
<tr>
<td>$^{31}$P</td>
<td>4.8</td>
<td>52</td>
<td>115</td>
</tr>
<tr>
<td>$^{13}$C</td>
<td>9.4</td>
<td>26.6</td>
<td>410</td>
</tr>
<tr>
<td>$^2$H</td>
<td>5.6</td>
<td>44.6</td>
<td>490</td>
</tr>
<tr>
<td>$^{15}$N</td>
<td>13.7</td>
<td>18.3</td>
<td>399</td>
</tr>
</tbody>
</table>

### 4. Conclusion

The design and the performance of a new five channel transmission line-based NMR probes employing commercial electrical engineering software is presented in this work. The design bases on a single coil multi port frequency multiplexer of the Schaefer and McKay type. Such a kind of probe enables tuning and matching in all input/output ports independently yet in relatively narrow frequency bands. Employing modern electrical engineering calculators supported by a standard impedance measurements methods a rational probe is designed. Since this technique is reliable for all frequencies of interest in NMR, the simulation for all NMR fields can be performed successfully.

All features of the NMR probes and construction are presented here from the point of view both of electronic engineering and NMR test results. For example NMR line width, performance, NMR pulse length calibration are given. The practical application of this new probe for the study of heteronuclear dipolar interactions will be presented in detail in a following paper.

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### Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.ssnmr.2011.01.001

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