Two-coil Ultra High Field No-E H/X/Y for XVT MAS and Wideline

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INTRO Obtaining useful NMR data on low-gamma quadrupolar nuclides in solid samples remains a major challenge that in many cases will only be addressed with the combination of higher B_0 fields, larger samples, lower temperatures, and efficient triple-resonance methods in standard-bore magnets. That need served as the primary motivation for this development effort. While single-sample-coil (single-coil) circuits have usually been used at fields above 600 MHz, such circuits are incompatible with high rf decoupling in large samples at high fields. Hence, a two-coil approach was chosen with an inner solenoid and an outer segmented transverse proton coil. Because of its half-turn capacitive segmenting, the High Frequency (HF) E field in the sample is much lower than that seen in most "E-Free" probes, and it vanishes in the center – hence, the name "No-E".

METHODS

- The inner X/Y solenoid is secured to a Si_3N_4 ceramic stator for minimal tuning change with temperature and with long-term aging.
- A novel method for tuning the outer resonator from ^1H to ^{19}F, suitable for all fields and sample sizes, was implemented and modeled.
- Accommodating the full range of nuclides, sample sizes, and polarizing fields with high efficiency and power handling requires lower stray capacitance and more flexibility in plug-in component options than appears possible with plug-in-circuit-board approaches.
- Individual plug-in tuning elements work better!
- Detailed rf circuit models for the solenoid X/Y circuit and the HF circuit were developed, based on an earlier similar structure, with tuning data from several sample sizes at fields from 7-14 T.
- The detailed models showed that with several changes in the probe structure and circuits, efficient fully multi-nuclear H/X/Y performance could be expected to at least 30 T with small or large samples.

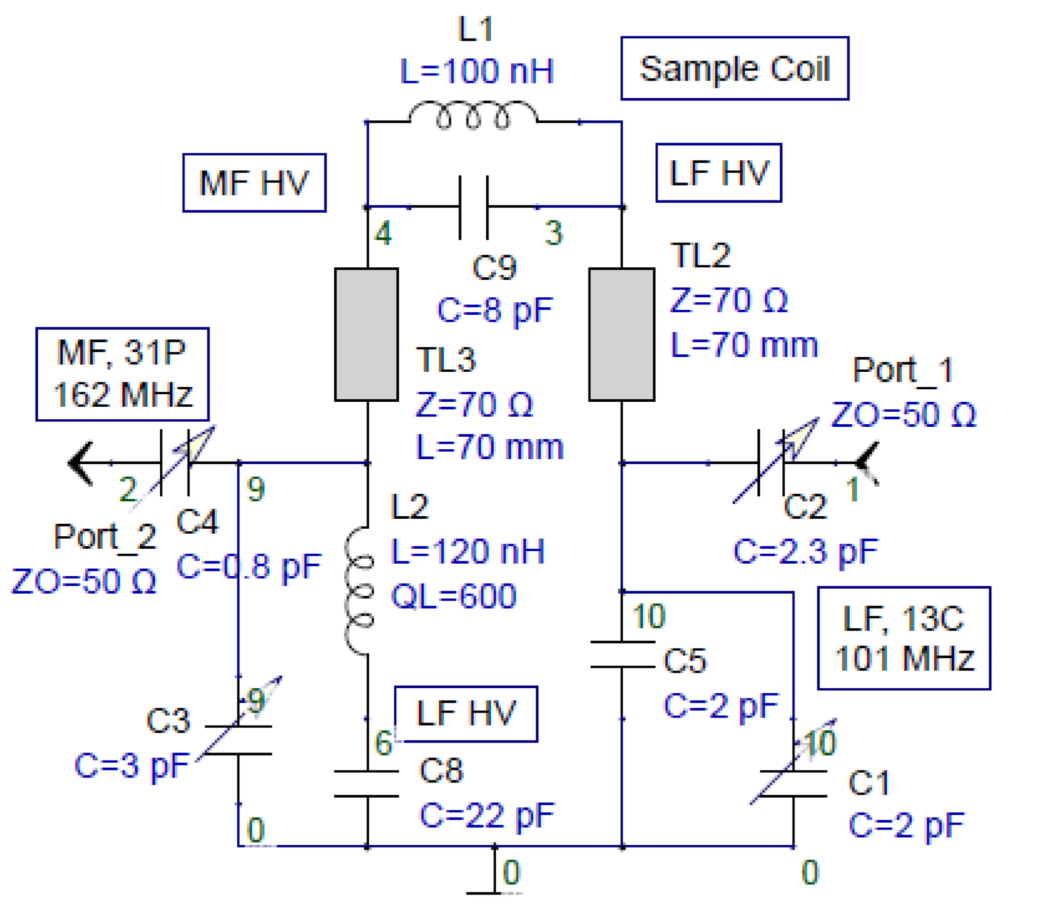


Fig. 1. Highly simplified low-field approximation of the basic X/Y solenoid rf circuit for conceptual purposes, with approximate values for the 5-mm 9.4-T $^{31}P/^{13}C$ case.

- For best performance, the high-current X/Y plug-ins must be just below the sample region. They provide voltage/impedance reduction to the long leads going to the LF/MF variables farther down.
- The HF fine-tune variables are in the region below the main LF/MF plug-ins. Long leads to the HF resonator aren't a problem, as lead VSWR is kept sufficiently low for low lead losses.
- The LF/MF tune/match variable (quartz) capacitors are in the lowest rf zone.
- With ten plug-in capacitors and coils, detailed circuit simulations show the novel X/Y circuit achieves optimal performance for H/X and H/X/Y combinations from ^{31}P to below ^{107}Ag.

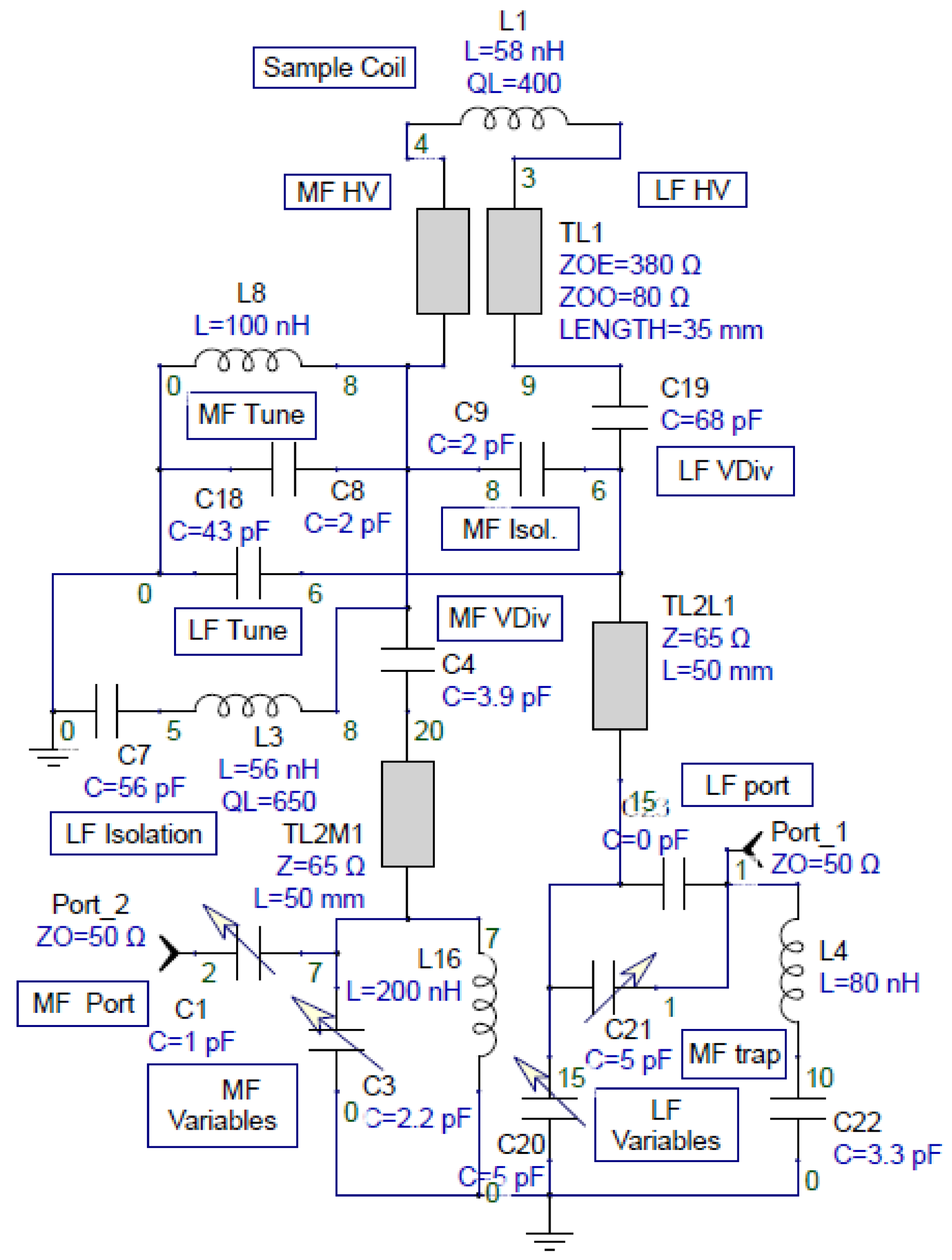


Fig. 2. The substantially complete X/Y circuit model (except stray capacitances, lead inductances, and a few ^1H traps are not shown) with approximate values for the 5-mm 28-T $^{13}C/^{14}N$ case. All the capacitors shown (except the variables) and the coils (except L1 and L4) are individual plug-ins.

- The various parasitics (stray capacitances, lead inductances, and resistances) in the X/Y and the HF detailed models were refined by comparing simulation results to bench and NMR experimental results from several earlier lower-field probes with similar structures and circuits.
- Subsequent simulations showed the X/Y circuit could be triple-tuned to $^{19}F/X/Y$, thereby permitting quad-resonance $^1H/^{19}F/X/Y$ operation.

References:
1. FD Doty, J Kulkarni, C Turner, G Entzminger, A Bielecki, "Using a cross-coil to reduce RF heating by an order of magnitude in triple-resonance multinuclear MAS at high fields", JMR., 182, 239-253, 2006.
2. F. David Doty, "Guide to simulating complex NMR probe circuits", Concepts in Magn. Reson. Part A, 2018;e21463. 1-20. DOI: 10.1002/cmr.a.21463.

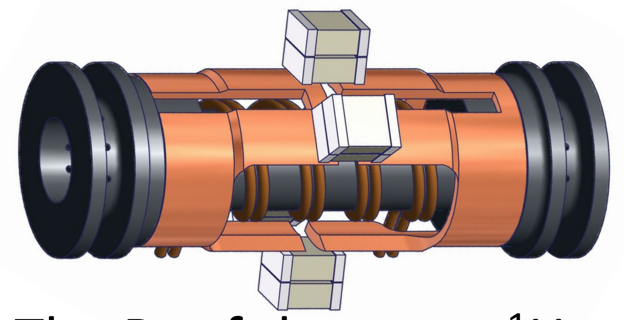
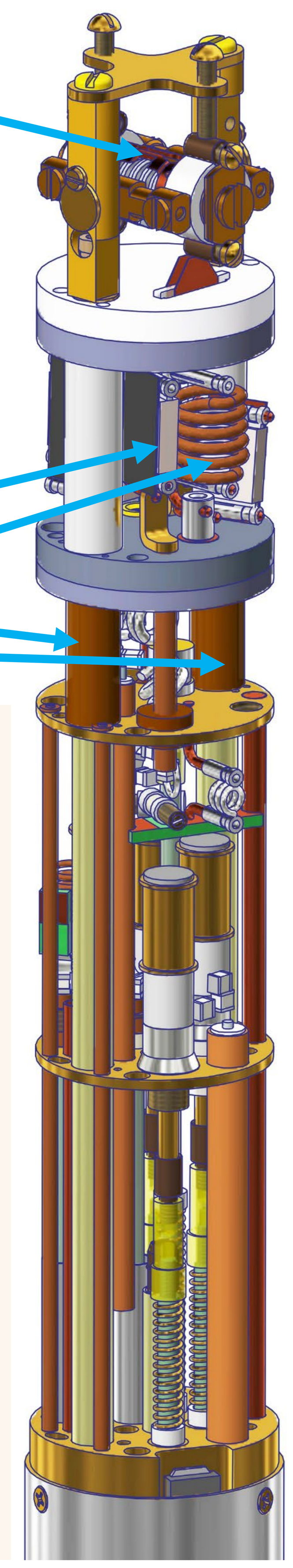


Fig. 3. The B_1 of the outer ^1H resonator, depicted above, is transverse, through the windows on the sides. The solenoid is mounted on the stator, and its leads come out between the bands on the bottom side of the ^1H resonator.



- An external thin-walled dewared shield-can surrounds all the zones seen here.
- The bearing and drive gases for MAS are supplied through separate dewared lines to the spinner.
- RT N_2 may be supplied into the X/Y tuning zone, moderating the temperatures in the regions below the sample zone.
- The gases all exhaust downward through a dewared line (not visible here) and out the base of the probe.
- The probe is designed for MAS operation from -160 °C to +180 °C.

Fig. 4. 5 mm H-F/X/Y wideline probe for use at 1.2 GHz, with a No-E ^1H coil.

RESULTS Following are some results predicted by the simulations for the 5-mm 28-T WL probe, for select nuclei.

Table 1. RF performance when 5 mm coil is tuned to $^1H/X$ or $^{19}F/X$.

Nuclides	Tuning Range, MHz	Primary freq. MHz	π/2 μs	Max P, W
^1H	1100-1230	1200	8.0	190
^{19}F	1100-1230	1129	9.5	200
^{71}Ga	356-377	366	5.1	94
^{61}Ni, ^{91}Zr, ^{85}Rb	105.5-119	112	5.9	379
^{67}Zn	73.5-77	75	5.6	692
^{109}Ag, ^{39}K	55.5-56.9	56	5.4	1127
^{73}Ge	41.5-42.2	41.9	5.4	2000

In the above (H/X mode) the X channel is balanced.

Table 2. RF performance when 5 mm coil is tuned to $^1H/X/Y$.

TT combos, ^1H/MF/LF	Freqs, MHz	MF π/2, μs	MF P, W	LF π/2, μs	LF P, W
$^1H/^{13}C/^{14}N$	302/122	9.0	110	16	300
$^1H/^{27}Al/^{17}O$	313/164	8.5	128	10	150

Status and Future Work Fabrication is currently underway on the above probe. Preliminary NMR results are expected at 11.7 T in a few months and at 28 T about a month later.

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