Electronic Supplementary Information

A Low-Cost Strategy for ^{43}Ca Solid-State NMR

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^{43}Ca MACS Fabrications

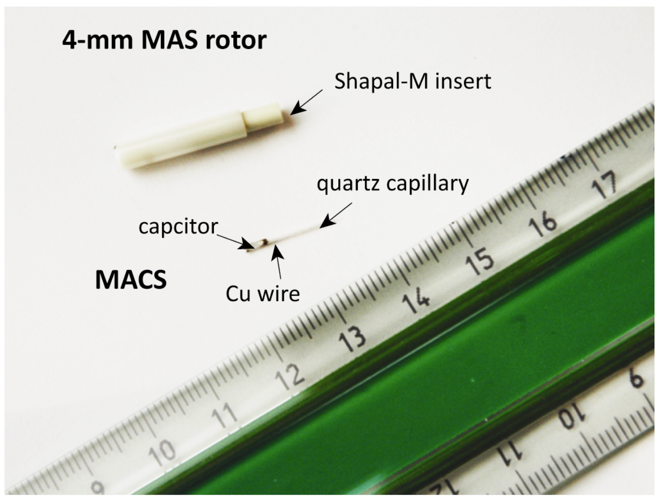

Figure 1. Photographic image of a typical inductive MACS solenoid, alongside a standard Bruker 4-mm MAS rotor for comparison.

^{43}Ca and ^{1}H MACS coils used here were constructed by manually winding a solenoid from 62 μm copper wire around a quartz capillary (Vitrocom Inc.), and soldered to a surface-mount capacitor (Murata manufacturing Co. Ltd) forming a resonant LC circuit. The resonance frequency ($f = 1/2\pi\cdot(LC)^{-1/2}$) (*i.e.* 33.6 MHz for ^{43}Ca or 499.1 MHz for ^{1}H) is dependent upon the total inductance, L (a factor of the solenoid dimensions) and the total capacitance, C. A small drop of cyanoacrylate glue was applied to affix the solenoid to the capillary. The MACS solenoid was tightly fitted inside a Shapal-M® (aluminum nitride) insert designed to fit snugly inside a Bruker 4-mm MAS rotor. The Shapal-M® insert centers the sample/coil and allows for efficient heat dissipation away from the sample during rapid spinning.[1] The MACS coils' designs and dimensions are summarized in Figure 1 and Table 1.

Table 1. A summary of the micro-coils' dimensions and resonance properties.

	^{43}Ca MACS	^{1}H MACS
Quartz capillary	1.0 mm o.d.	0.87 mm o.d.
Coil dimensions	62 μm Cu wire 23 turns 1.8 mm length	62 μm Cu wire 10 turns 2.4 mm length
Capacitor	100 pF	3.3 pF
Frequency	35.5 MHz	494.5
Quality factor	49	78

NMR Experimental Methods

^{43}Ca and ^{1}H MACS coils used here were constructed by manual winding of micro-solenoids. Detailed procedures and coils' properties are described above. 66% ^{43}Ca-enriched $Ca(OH)_2$ was used as a test sample for ^{43}Ca MACS experiments. ^{43}Ca and ^{1}H NMR were performed on an 11.75 T Bruker Avance II spectrometer operating at a frequency of 33.58 and 499.13 MHz, respectively, with a standard Bruker 4-mm triple-resonance HXY MAS probe. ^{43}Ca and ^{1}H NMR spectra were acquired under sample spinning rates of 4000 and 8000 ± 1 Hz, respectively. No ^{1}H-decoupling pulse was used in the experiment because the applied MAS frequeny is sufficient to suppresse the weak ^{1}H-^{43}Ca dipole.[2] The ^{43}Ca and ^{1}H chemical shifts were referenced to the centre-of-gravity of the observed signal.

^{43}Ca MQ-MACS

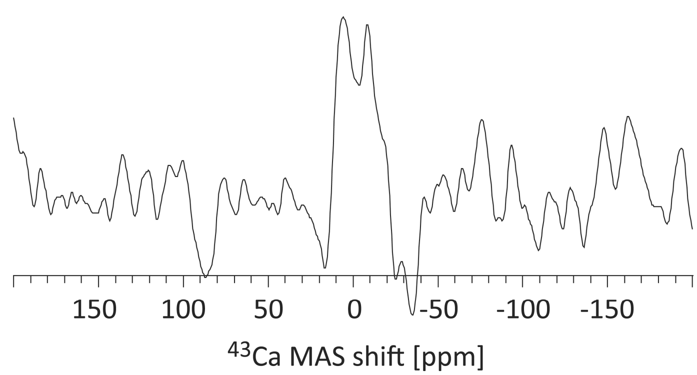

Figure 2. One-dimensional ^{43}Ca MAS spectrum of the first t_1 increment of a z-filtered 3Q-MAS experiment with a ^{43}Ca MACS coil. A standard 24-phase-cycle z-filtered 3Q-MAS experiment was performed: using excitation and conversion pulses of 6 μs and 3 μs, respectively, both with an input power of 2.5 W. A z-filter delay of 20 μs followed by a pulse of 4.5 μs (at 0.25 W power) was used. A total of 6000 scans with a recycle delay of 30 s was collected (50 hrs)

References

1. P. M. Aguiar, J. -F. Jacquinot, D. Sakellariou, *J. Magn. Reson.* 2009, **200**, 6.
2. A. Wong, A. P. Howes, R. Dupree, M. E. Smith, *Chem. Phys. Lett.* 2006, **427**, 201.