

Molybdenum Magnetic Shielding and Quadrupolar Tensors for a Series of Molybdate Salts: A Solid-State ^{95}Mo NMR Study

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Experimental Details for Acquiring Spectra:

Figure 1: The ^{95}Mo NMR spectra of the central transition of MAS samples of (a-d) Li_2MoO_4 , Rb_2MoO_4 , K_2MoO_4 , and Cs_2MoO_4 were acquired using a one-pulse experiment at 11.75 T. The spectra are the sum of (a-d) 6471, 2665, 2051, 4930 scans, respectively, and were acquired at spinning rates of (a,c) $\nu_{\text{rot}} = 5.0$ kHz, (b) 6.0 kHz and (d) $\nu_{\text{rot}} = 3.0$ kHz and using pulse delays of 10 s. Gaussian linebroadening functions of (a-d) 10 Hz, 10 Hz, 0 Hz and 20 Hz, respectively, were applied to the FIDs prior to Fourier transformation, FT.

Figure 2: Simulated (top, red) and experimental (bottom, black) ^{95}Mo NMR spectra of the central transition of MAS samples of (a-e) BaMoO_4 , SrMoO_4 , CdMoO_4 , CaMoO_4 and PbMoO_4 , respectively. The spectra shown in (a-c) were acquired using a one-pulse experiment at $B_0 = 11.75$ T and $\nu_{\text{rot}} = 6$ kHz and are the sum of (a-c) 7724, 2319, and 2067 scans, respectively. Pulse delays of (a-c) 20 s, 10 s, and 30s were used and Gaussian functions of 50 Hz, 20 Hz, and 50 Hz, respectively, were applied to the FIDs prior to FT. The spin-echo experiment was used to acquire the spectra shown in (d, e); a total of 924 and 256 scans were collected using a pulse delay of 30 s and the FIDs were processed after applying Gaussian functions of 20 Hz.

Figure 3: Experimental (a) and simulated (b, c) solid-state ^{95}Mo NMR spectra of the central transition of stationary samples of (i) K_2MoO_4 and (ii) Rb_2MoO_4 acquired at 11.75 T (left) and 17.63 T (right). The ^{95}Mo NMR spectrum of a stationary sample of K_2MoO_4 acquired at 11.75 T was obtained using a one-pulse experiment with a 10 s pulse delay and is the sum of 2199 scans; a Gaussian linebroadening function of 100 Hz was applied to the FID prior to FT. The spectrum acquired at 17.63 T was obtained using a QCPMG experiment with $M = 72$, $\nu_{\text{QCPMG}} = 200$ Hz, a 10 s pulse delay and is the sum of 800 scans; a Gaussian linebroadening function of 2 Hz was applied to the FID prior to FT. (ii) The ^{95}Mo NMR spectrum of a stationary sample of Rb_2MoO_4 acquired at 11.75 T was obtained using a one-pulse experiment with a 10 s pulse delay and is the sum of 5622 scans; a Gaussian linebroadening function of 50 Hz was applied to the FID prior to FT. The spectrum acquired at 17.63 T was obtained using a QCPMG experiment with $M = 32$, $\nu_{\text{QCPMG}} = 250$ Hz, a 10 s pulse delay and is the sum of 1024 scans; a Gaussian linebroadening function of 2 Hz was applied to the FID prior to FT.

Figure 4: Experimental (a) and simulated (b, c) ^{95}Mo NMR spectra of the central transition of stationary samples of Cs_2MoO_4 acquired using a one-pulse experiment at 11.75 T (left) and a spin-echo experiment at 21.14 T (right). The spectrum acquired at 11.75 T (21.14 T) is the sum of 2414 (432) scans and was obtained using a 10 s (30 s) pulse delay; the FID was processed after applying a Gaussian linebroadening function of 30 Hz (50 Hz).

Figure 5: Experimental (a) and simulated (b, c) ^{95}Mo NMR spectra of the central transition of stationary samples of Li_2MoO_4 acquired using a one-pulse experiment at 11.75 T (left) and a spin-echo experiment at 21.14 T (right). The spectrum acquired at 11.75 T (21.14 T) is the sum of 3072 (1896) scans and was obtained using a 10 s (30 s) pulse delay; the FID was processed after applying a Gaussian linebroadening function of 120 Hz (50 Hz).

Figure 6: Simulated (top, red) and experimental (bottom, black) solid-state ^{95}Mo NMR spectra of the central transition of stationary samples of (a-e) CdMoO_4 , PbMoO_4 , CaMoO_4 , BaMoO_4 , and SrMoO_4 acquired at the indicated magnetic fields. (a) **CdMoO₄**: The spectrum acquired at 11.75 T (a, left) was obtained using a one-pulse experiment with a 10 s pulse delay and is the sum of 7416 scans; a Gaussian linebroadening function of 100 Hz was applied to the FID prior to Fourier transformation. The spectrum acquired at 21.14 T (a, right) was obtained using a spin-echo experiment with a 30 s pulse delay and is the sum of 1364 scans; a Gaussian linebroadening function of 50 Hz was applied to the FID prior to Fourier transformation. (b) **PbMoO₄**: The spectrum acquired at 11.75 T (b, left) was obtained using a one-pulse experiment with a 20 s pulse delay and is the sum of 4336 scans; a Gaussian linebroadening function of 50 Hz was applied to the FID prior to Fourier transformation. The spectrum acquired at 21.14 T (b, right) was obtained using a spin-echo experiment with a 30 s pulse delay and is the sum of 512 scans; a Gaussian linebroadening function of 50 Hz was applied to the FID prior to Fourier transformation. (c) **CaMoO₄**: The spectrum acquired at 11.75 T (c, left) was obtained using a one-pulse experiment with a 15 s pulse delay and is the sum of 5304 scans; a Gaussian linebroadening function of 50 Hz was applied to the FID prior to Fourier transformation. The spectrum acquired at 21.14 T (c, right) was obtained using a spin-echo experiment with a 30 s

pulse delay and is the sum of 1344 scans; a Gaussian linebroadening function of 100 Hz was applied to the FID prior to Fourier transformation. (d) **BaMoO₄**: The spectrum acquired at 11.75 T (d, left) was obtained using a one-pulse experiment with a 15 s pulse delay and is the sum of 3476 scans; a Gaussian linebroadening function of 50 Hz was applied to the FID prior to Fourier transformation. The spectrum acquired at 17.63 T (d, right) was obtained using a QCPMG experiment with $M = 72$, $\nu_{\text{QCPMG}} = 200$ Hz, a 15 s pulse delay and is the sum of 448 scans; a Gaussian linebroadening function of 2 Hz was applied to the FID prior to FT. (e) **SrMoO₄**: The spectrum acquired at 11.75 T (e, left) was obtained using a one-pulse experiment with a 20 s pulse delay and is the sum of 4031 scans; a Gaussian linebroadening function of 30 Hz was applied to the FID prior to FT. The experimental spectrum acquired at 17.63 T (e, bottom, right) was obtained using a QCPMG experiment; 8 scans were collected using $M = 64$, $\nu_{\text{QCPMG}} = 250$ Hz, and a pulse delay of 20 s. The spectrum (e, top, right) was acquired using a spin-echo experiment and is the sum of 544 scans; a 20 s pulse delay was used and a Gaussian linebroadening function of 20 Hz was applied to the FID prior to FT.