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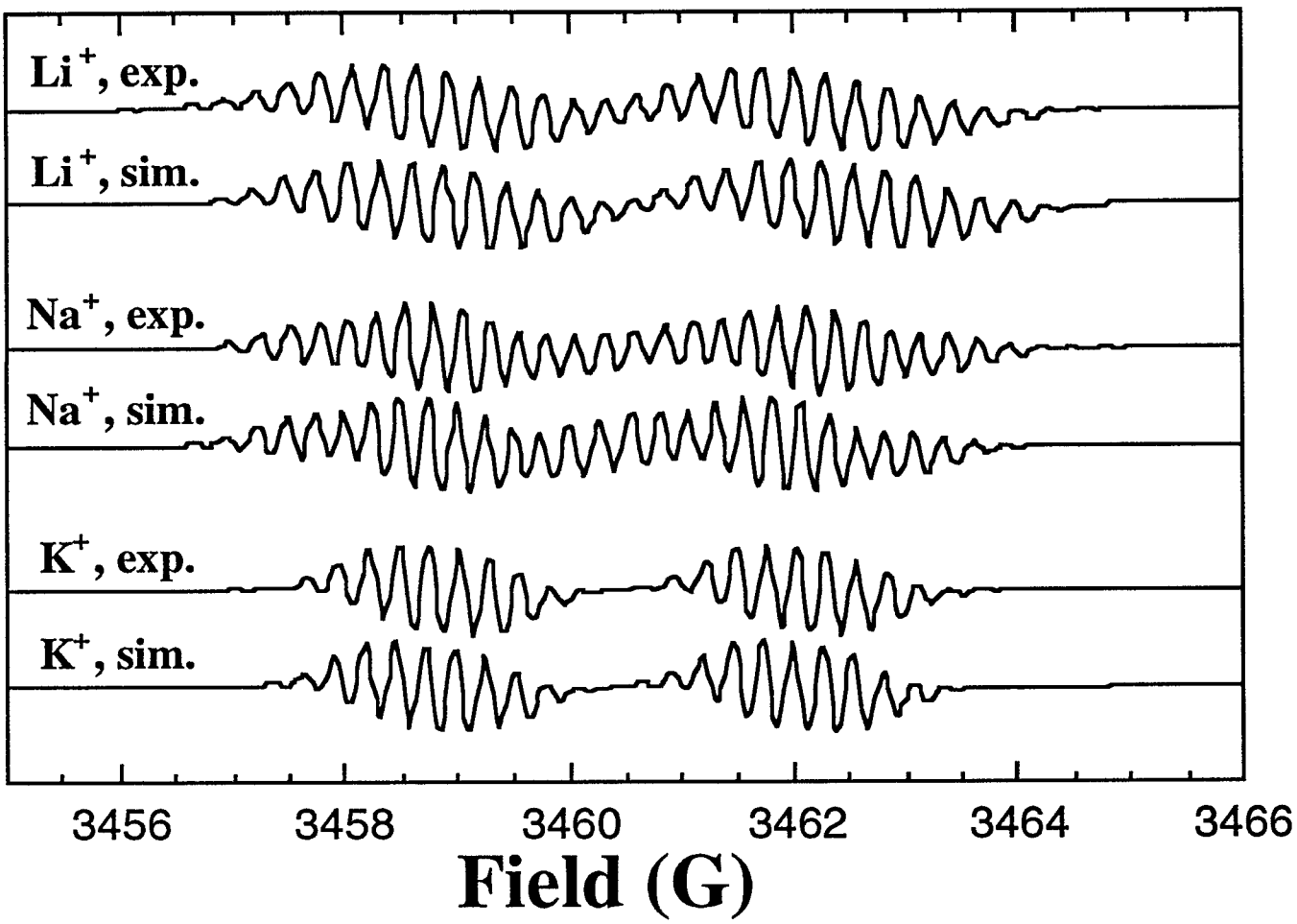
General Experimental

EPR sample preparation was carried out under a nitrogen atmosphere in a Vacuum Atmospheres glovebox. THF and toluene were distilled from sodium benzophenone-ketyl prior to use. Solvents were subjected to several freeze-pump-thaw cycles prior to use. X-band EPR spectra were recorded on an IBM-Brüker E200SRC spectrometer. A quartz finger dewar filled with liquid nitrogen was used for recording spectra at 77 K. Variable-temperature fluid solution EPR experiments were performed using an IBM Model ER4111VT variable-temperature unit. Chemicals were purchased from Aldrich Chemical Co. and used as received, except for the amines which were vacuum distilled from CaH_2 and subjected to several freeze-pump-thaw cycles prior to use. Lithium from Aldrich may contain 5% Na. See ref. 1 for preparation and details of the bulk electrolysis of **1**.

The preparation of $Na_2\mathbf{1}^{2-\bullet\bullet}$/PMDTA was carried out in a glovebox under a nitrogen atmosphere. A 1 mM solution (PMDTA: THF 1:9, 10 mL) of **1** was stirred for 90 min. over a freshly prepared sodium mirror. The initial light green solution darkened then became colorless. This solution was then filtered through glass wool into a second flask containing another 10 mL of the bis-orthoquinone solution. This resulted in an immediate color change to a dark green solution. This solution was used immediately for EPR spectroscopy. Precipitation of $Na_2\mathbf{1}^{2-\bullet\bullet}$ is observable within a few hours and gives a blue-green solid.

Sample Simulated EPR Spectra

0.83 mM M(3,5-DBSQ)/177 mM PMDTA/THF
EPR Spectra and Simulations

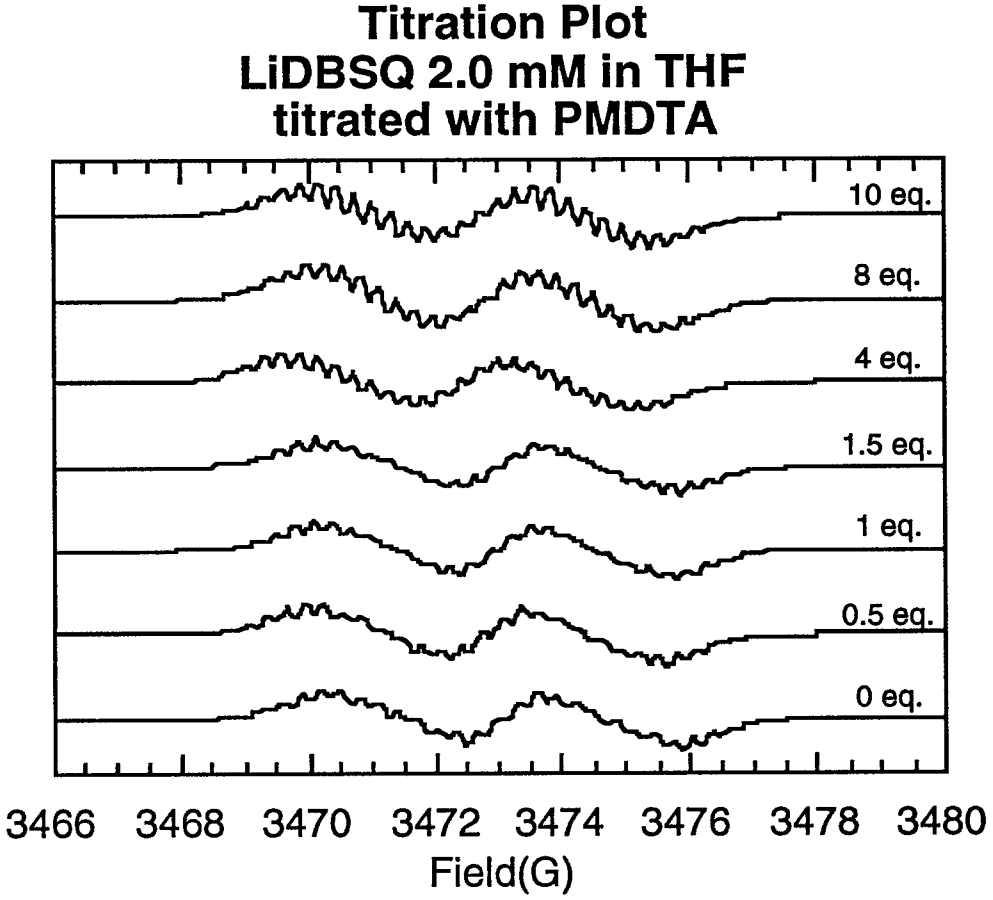


Tabulated Hyperfine Coupling Constants from Simulated EPR Spectra

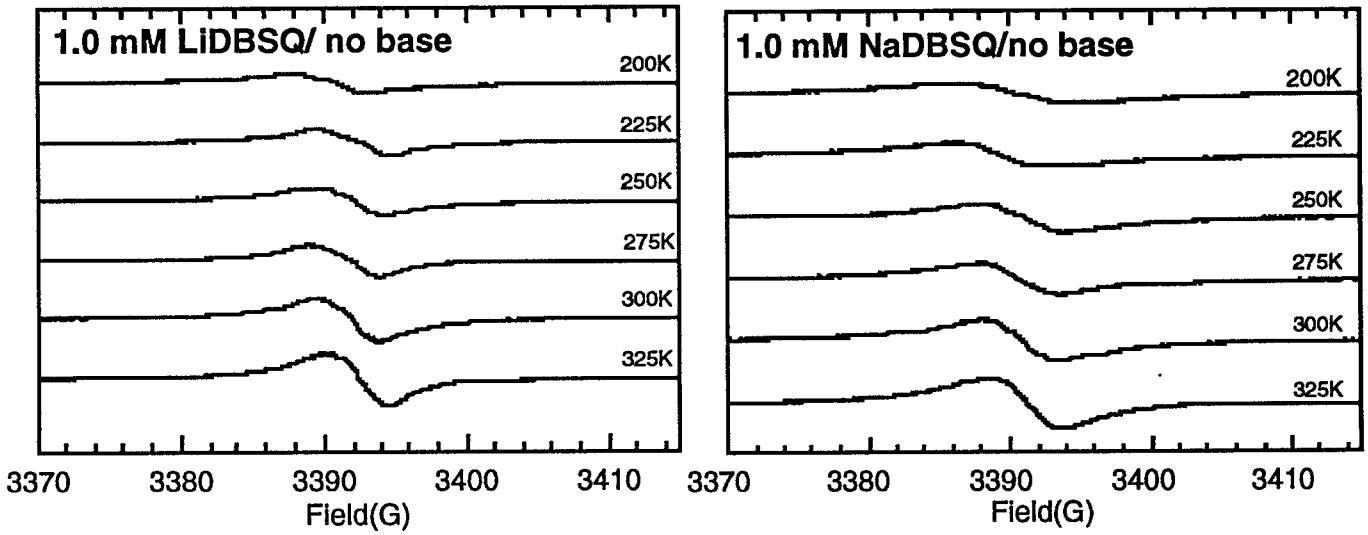
Solvent	Metal	Amine	a_H (G)	a_H (G)	a_H (G)	a_M (G)	LW[a] (G)	Coeff.[b]
THF	Li	None	3.42	0.91	0.29	0.50	0.10	0.976
		TEA	3.42	0.71	0.28	0.58	0.11	0.990
		TMEDA	3.41	0.71	0.28	0.58	0.10	0.994
		PMDTA	3.38	0.63	0.27	0.56	0.11	0.995
	Na	None	3.33	0.70	0.27	0.49	0.09	0.997
		TEA	3.32	0.69	0.27	0.49	0.09	0.995
		TMEDA	3.32	0.69	0.27	0.49	0.09	0.996
		PMDTA	3.33	0.71	0.27	0.50	0.08	0.999
	K	None	3.28	0.80	0.27	---[c]	0.11	0.998
		TEA	3.28	0.79	0.27	---[c]	0.11	0.998
		TMEDA	3.28	0.79	0.27	---[c]	0.11	0.998
		PMDTA	3.28	0.79	0.27	---[c]	0.10	0.997
Toluene	Li	None	---[d]	---[d]	---[d]	---[d]	---[d]	---[d]
		TEA	---[d]	---[d]	---[d]	---[d]	---[d]	---[d]
		TMEDA	3.41	0.71	0.33	0.55	0.20	0.973
		PMDTA	3.37	0.61	0.27	0.57	0.12	0.998
	Na	None	---[d]	---[d]	---[d]	---[d]	---[d]	---[d]
		TEA	---[d]	---[d]	---[d]	---[d]	---[d]	---[d]
		TMEDA	3.33	0.64	0.27	0.59	0.10	0.983
		PMDTA	3.32	0.62	0.78	0.57	0.09	0.997
	K	None	---[d]	---[d]	---[d]	---[d]	---[d]	---[d]
		TEA	---[d]	---[d]	---[d]	---[d]	---[d]	---[d]
		TMEDA	---[d]	---[d]	---[d]	---[d]	---[d]	---[d]
		PMDTA	3.26	0.70	0.27	---[c]	0.14	0.995

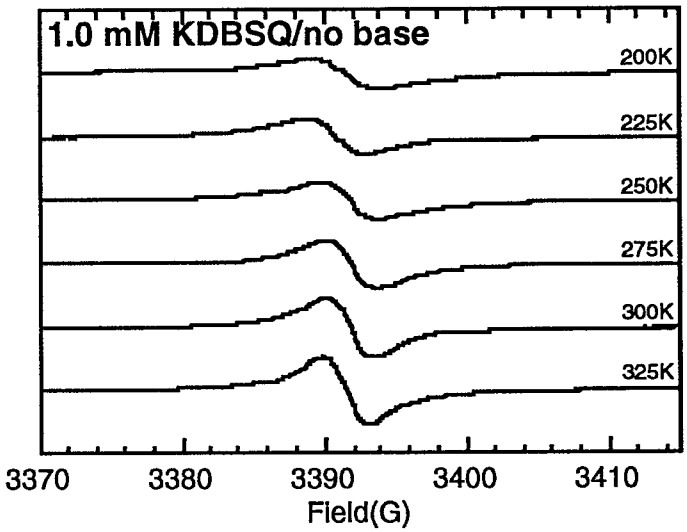
[a]Line width. [b]Correlation coefficient. [c]Hfcc not observed. [d]Lines too broad for meaningful simulation.

EPR Spectrum as a Function of Equivalents of Amine



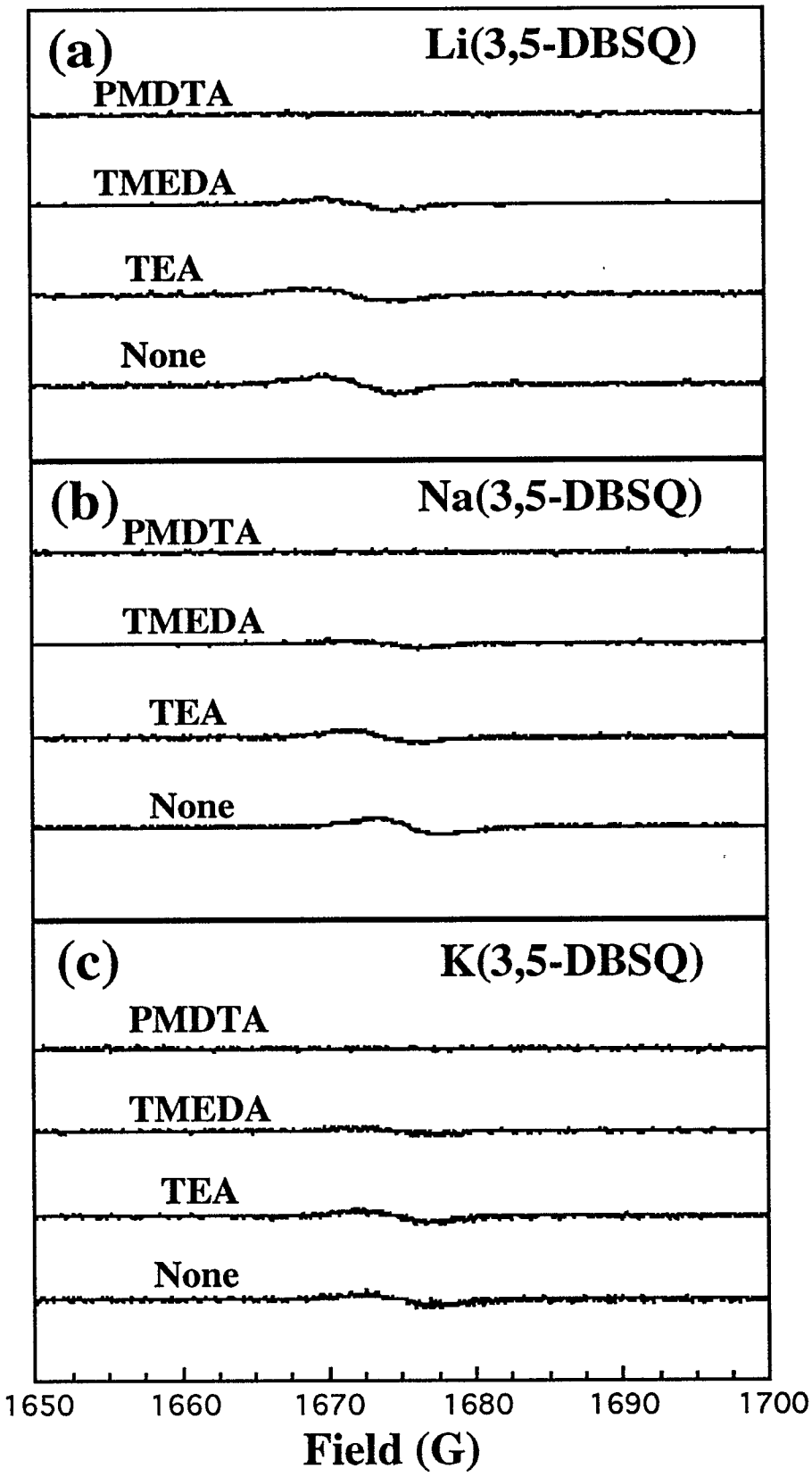
Variable-temperature EPR Spectra in Toluene





$\Delta m_s = 2$ Transitions

M(3,5-DBSQ) in THF at 77 K



M(3,5-DBSQ) in Toluene at 77 K