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Two-Dimensional Pulsed TRIPLE at 95 GHz

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Abstract

The one-dimensional (1D) pulsed TRIPLE resonance experiment, introduced by Mehring et al. (M. Mehring, P. Höfer, and A. Grupp, Ber. Bunseges. Phys. Chem. 91, 1132-1137 (1987)) is a modification of the standard Davies ENDOR experiment where an additional RF π-pulse is applied during the mixing time. While the first RF pulse is set to one of the ENDOR transitions, the frequency of the second RF pulse is scanned to generate the TRIPLE spectrum. The difference between this spectrum and the ENDOR spectrum yields the difference TRIPLE spectrum, which exhibits only ENDOR lines that belong to the same Ms manifold as the one selected by the first RF pulse. We have extended this experiment in two dimensions (2D) by sweeping the frequencies of both RF pulses. This experiment is particularly useful when the spectrum is congested and consists of signals originating from different paramagnetic centers. The connectivities between the peaks in the 2D spectrum enable a straightforward assignment of the signals to their respective centers and Ms manifolds, thus providing the relative signs of hyperfine couplings. Carrying out the experiment at high fields has the additional advantage that nuclei with different nuclear gyromagnetic ratios are well separated. This is particularly true for protons which appear at significantly higher frequencies than other nuclei. The feasibility and effectiveness of the experiment is demonstrated at W-band (94.9 GHz) on a crystal of Cu2+-doped L-histidine. Homonuclear 1H-1H, 14N/35Cl-14N/35Cl and heteronuclear 1H-14N/35Cl 2D TRIPLE spectra were measured and from the various connectivities in the 2D map the 1H, 14N, and 35Cl signals that belong to two different Cu2+ centers were identified and grouped according to their Ms manifolds. © 2000 Academic Press.

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Keywords

ENDOR, High field pulsed EPR, TRIPLE