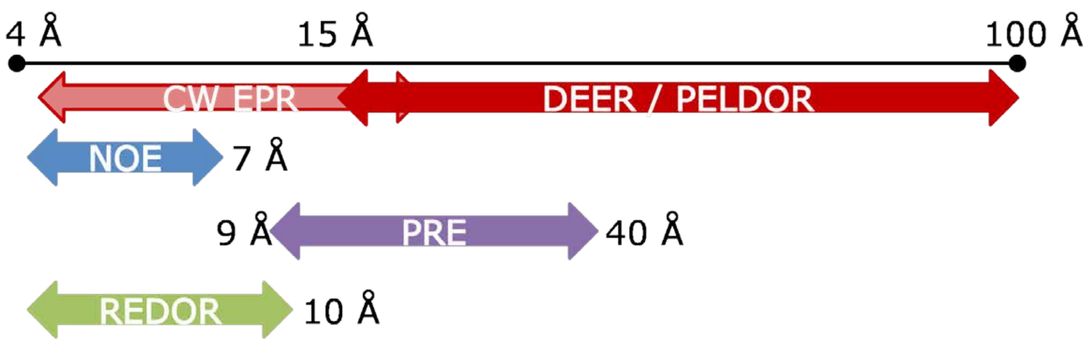
Rapid Distance Determination by EPR

Long Range EPR Distance Constraints

Pulse dipolar EPR spectroscopy is a powerful tool for the measurement of long distances (15-100 Å) between paramagnetic centers. Furthermore, information about the dynamics of the interacting spins is obtained from the distance distribution profiles. The most popular technique is the pulse Double Electron Electron Resonance (DEER / PELDOR).



Common Paramagnetic Centers

- Organic Radicals
  - Nitroxide spin labels, tyrosine, tryptophan, quinones, …
- Transition Metals
  - Cu, Mn, Fe, Mo Ni, Co, Lanthanides, …

DEER spectroscopy is a complementary technique to NMR methodologies (NOESY, HSQC, REDOR, etc.) which provides supplemental long distance constraints for structure determinations and dynamic studies of large biomolecules. DEER also has no restrictions on the size of the macromolecule that can be studied.

DEER at 34 GHz (Q-band)

While DEER/PELDOR measurements have been traditionally performed at 9 GHz (X-band), the recent availability of high power amplifiers at 34 GHz (Q-band) has revolutionized the field. The advantages of Q-band over X-band are both increased concentration and effect sensitivity.

The Bruker high power Q-band system incorporates a 150 W TWT microwave amplifier and the large volume Q-band resonator (QT-II) to enhance the performance available from the DEER experiment.

High Concentration Sensitivity

The new large volume Q-band resonator (QT-II) provides a two fold increase in sensitivity for pulse EPR measurements. This allows lower sample concentrations (down to 1 µM) to be measured and decreases the total measurement time (minutes instead of hours).

High Dipolar Modulation Sensitivity

An increase in available microwave power directly translates into an enhanced dipolar modulation depth (effect sensitivity). This increased sensitivity results in high precision distance determinations and much higher sample throughput.

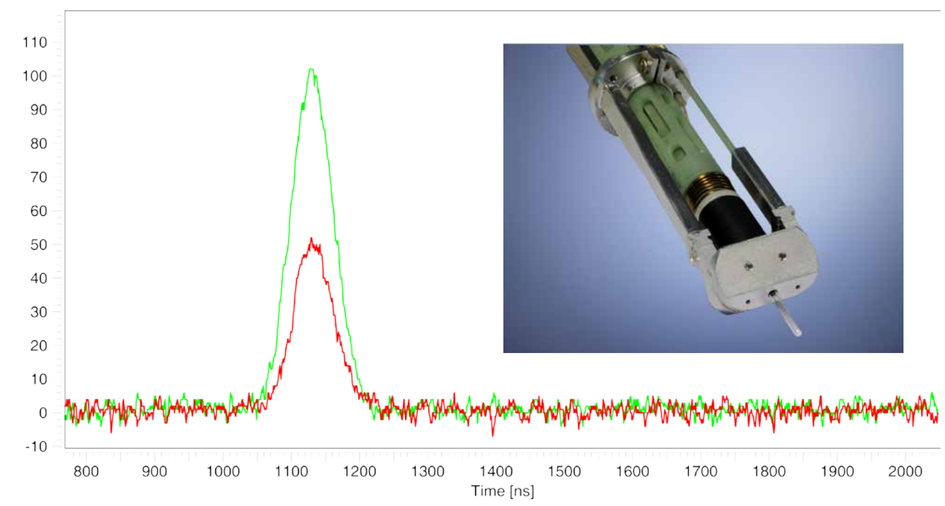


Fig. 1 The Q-band large volume resonator (QT-II) provides a factor of two increase in concentration sensitivity compared to previous resonators.

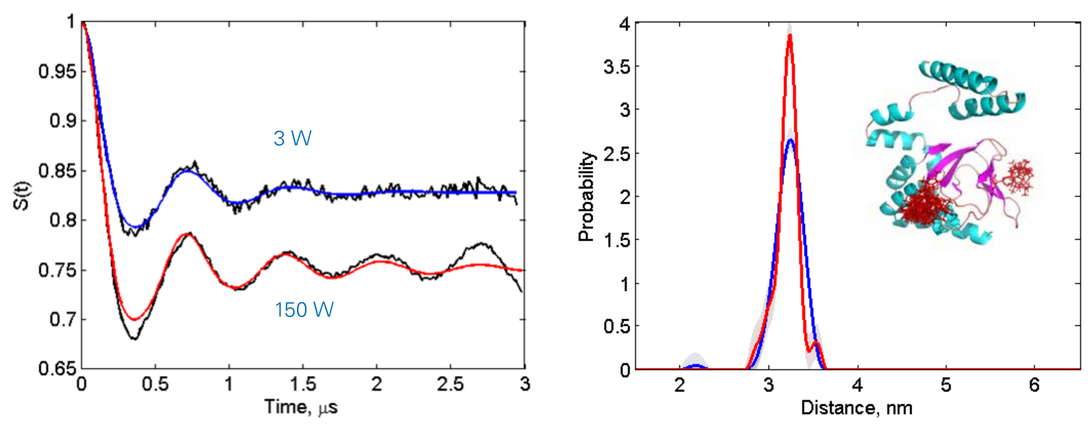


Fig. 2 DEER spectra acquired at 34 GHz (Q-band) of a doubly spin labeled protein (HCN2I-563R1/608R1). The power available from the Q-band TWT (150 W) results in an increase in the dipolar modulation depth compared to a solid state amplifier (3 W).
Data courtesy of Dr. S. Stoll, University of Washington.

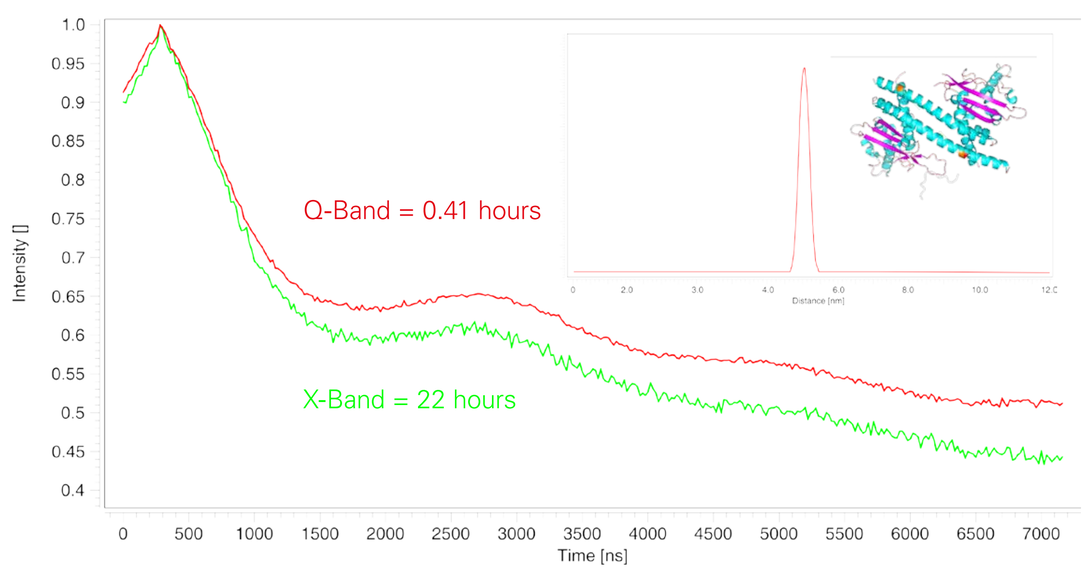


Fig. 3 DEER spectra acquired at 34 GHz (Q-band) and 9 GHz (X-band) of a doubly spin labeled protein (Vps75 A19 K20RX + E23A L16A mutation).
Data courtesy of mm-wave and HFESR group at the University of St. Andrews.

Summary

- High sensitivity
- High precision distances
- Distances from 15 to 100 Å
- Complementary to NOEs
- Dedicated resonator for high power operation
- High power Q-band TWT (150 W at 34 GHz)