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CHARACTERISATION OF PHOSPHORUS COMPOUNDS BY ^{31}P , ^nX -SHIFT CORRELATED NMR TECHNIQUES IN ONE AND TWO DIMENSIONS

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Examples for 2-D shift correlations between ^{31}P and another heteronucleus via ^nX -detected 2D-INEPT or ^{31}P -detected HMQC experiments are presented. The former technique is best suited for ^{31}P , ^{15}N correlations and permits the determination of intramolecular connectivities as well as of relative signs of couplings.¹ The "inverse" HMQC experiment allows easy recording of the ^{77}Se NMR spectrum of an equilibrium mixture of **2** and **3**, disregarding the severe exchange broadening of the ^{77}Se -resonances (Figure 1).

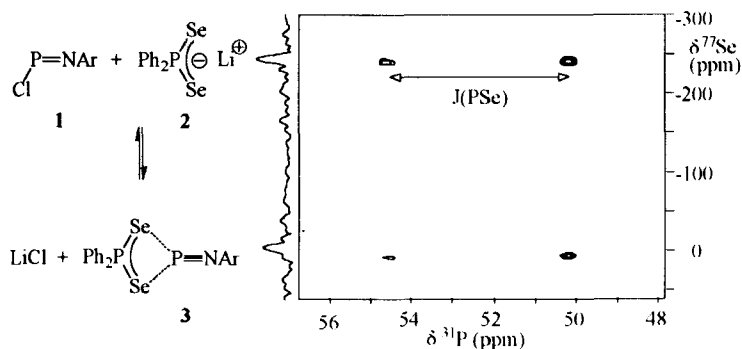


Figure 1:
 121.5 MHz
 ^{31}P , ^{77}Se HMQC
 spectrum (-30°C)
 of a 1:2 mixture of
1 and **2**.

In addition, schemes for ^{31}P , ^nX correlations with selective 1D-experiments are suggested. These are based on a combination of tailored excitation of selected ^{31}P nuclei and non selective ^{31}P , ^nX magnetisation transfer. The tailored excitation is achieved by application of shaped pulses on the ^{31}P channel, or alternatively by selective ^1H , ^{31}P cross polarisation.² With the latter method, the sensitivity gain is in favourable cases similar to ^1H , ^nX INEPT experiments. The new techniques are employed for determination of the stereochemistry in phosphorus-nitrogen ring systems¹, and signal assignment in product mixtures².

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