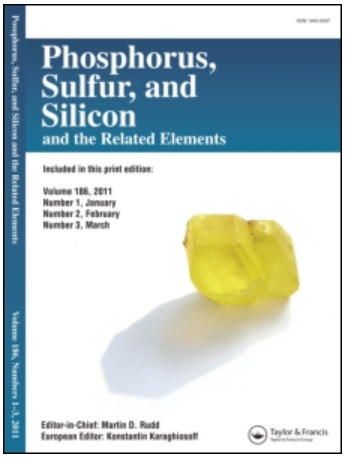

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Purity Determination of Phosphorus Compounds by Internat- Standard^{31} P Nmr Spectroscopy. an Experimental Study and Validation

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PURITY DETERMINATION OF PHOSPHORUS COMPOUNDS BY INTERNAL STANDARD ^{31}P NMR SPECTROSCOPY. AN EXPERIMENTAL STUDY AND VALIDATION.

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Due to the proportionality between the area of the NMR signal and the number of nuclei, internal standard ^{31}P NMR spectroscopy is in principle a rapid and simple method for determination of the purity of phosphorus compounds.
In principle the ratio of the integrals of the two resonances in a ^{31}P-NMR spectrum of a solution containing known amounts of a standard and a sample of known purity corresponds to the molecular ratio. Therefore, knowing the purity of the standard the purity of the sample can easily be determined.
In order to make a critical evaluation of the FT NMR measurements used in this method a number of new organic compounds containing two non-equivivalent phosphorus atoms have been synthesised. Since these compounds must yield ^{31}P-NMR spectra with two resonances with a 1:1 integral ratio, they provide an efficient tool in an analysis of how (mis-)adjustment of different aquisition and processing parameters will affect the purity analysis. Using these compounds for the evaluation of the method we found that during the aquisition and processing of the spectrum the following points must be followed strictly:

- The relaxation delay must be at least 5 times the longest T_1
- The carrier frequency must be positioned exactly between the two resonances
- Use Invers Gated decoupling to suppres nOe.
- Manual phasing of the spectrum
- Baseline correction before integration

A validation of the method has been carried out on samples of the insecticide Malathion in a xylene matrix with tributyl phosphate as internal standard. Five samples were prepared by mixing a Malathion standard of known purity with xylene. For each sample the recovery and the precision were determined. The results are summarized in the table below.

Conc.(calc)	20.5%	49.8%	74.4%	89.6%	99.5%
Conc.(meas)	20.2 ± 0.1%	49.4 ± 0.2%	74.1 ± 0.4%	89.0 ± 0.2%	99.3 ±0.5%
Recovery	98.5%	99.2%	99.6%	99.3%	99.8%
Precision	1.2%	1.0%	0.5%	0.3%	0.7%

As can be seen excellent recoveries are generally obtained. Furthermore, the precision of the method is excellent and significantly better than what is normally required of analytical methods for registration purposes.