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NMR Spectroscopic and X-Ray Structural Studies of Two Diazadiphosphetidines

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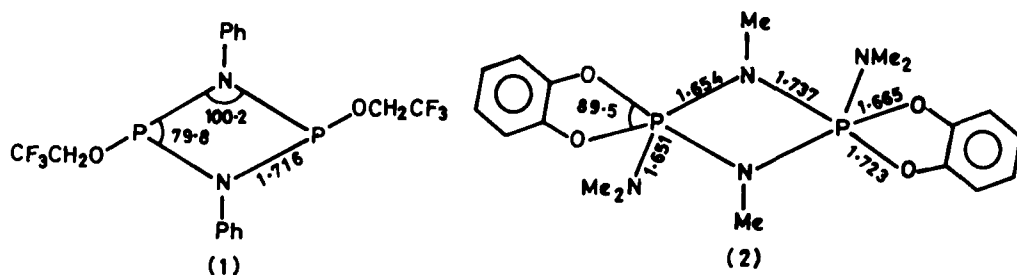
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The crystal structures of the two diazadiphosphetidines, $[\text{PhNP}(\text{OCH}_2\text{CF}_3)]_2$ (1) and $[\text{MeNP}(\text{NMe}_2)(\text{O}_2\text{C}_6\text{H}_4)]_2$ (2) have been determined. The trifluoroethoxy groups in (1) have a trans orientation. The phosphorus chemical shift for (1) is at 189.8 δ . On standing in solution, (1) transforms slowly (~ 10 days) and almost completely into its 'high-field' (cis) isomer (142.2 δ).



The $-\text{NMe}_2$ groups in (2) are trans to each other; the geometry around each phosphorus is slightly distorted trigonal bipyramidal. The ^1H NMR spectrum of (2) shows only one triplet (δ 2.64, $J_{\text{PH}} = 12$ Hz) for the ring 'NMe' protons at ambient temperatures; this signal does not split into two sets of doublets (as would be anticipated owing to coupling with two different phosphorus nuclei) even at -50°C . However, another triplet (δ 2.61, $J_{\text{PH}} = 12$ Hz) with a lower intensity appears for the ring 'NMe' protons after 24 hours at ambient temperature.

Similar results are observed for the alkoxy derivatives, $[\text{RNP}(\text{OR}')(\text{O}_2\text{C}_6\text{H}_4)]_2$ except that in these cases two sets of signals are observed for $\text{NR}(\text{R}=\text{Me})$ and OR' protons immediately upon dissolution of the compounds in CDCl_3 at ambient temperatures. Possible reasons for this behaviour are discussed.