

Analyses of samples from the production of Spolapret OS

Sample I - PCl_3

Sample II - $\text{HP(O)(OCH}_3)_2$

Sample III - SPOLAPRET OS

^1H , ^{13}C and ^{31}P NMR spectra were measured on a JEOL JNM-FX 100 spectrometer at 99.602, 25.047 and 40.324 MHz, respectively. The compounds I and II were measured as 1 : 1 (v/v) solutions in deuteriochloroform, the compound III as 4 : 1 (v/v) in deuteriumoxide at 300K. ^1H chemical shifts were referred to internal hexamethyldisiloxane (HMDSO; $\delta = 0.00$); ^{13}C chemical shifts to the signal of CDCl_3 ($\delta = 77.00$) and ^{31}P chemical shifts were referred to external neat H_3PO_4 (85%; $\delta = 0.0$). Positive values of chemical shifts denote downfield shifts. In addition, density (d) and refractive index (n) were measured with compounds I and II at 20°C .

Sample I - PCl_3

 $\delta(^{31}\text{P}) = 219.5$

$n = 1.513$

$d = 1.574 \text{ kg}\cdot\text{m}^{-3}$

Sample II - $\text{HP(O)(OCH}_3)_2$

 $\delta(\text{P-H}) = 6.76; \quad {}^1\text{J}(^{31}\text{P}, ^1\text{H}) = 697.8 \text{ Hz}$

$\delta(\text{OCH}_3) = 3.77; \quad {}^3\text{J}(^{31}\text{P}, ^1\text{H}) = 11.7 \text{ Hz}$

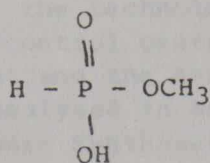
$\delta(\text{OCH}_3) = 50.32; \quad {}^2\text{J}(^{31}\text{P}, ^{13}\text{C}) = 5.9 \text{ Hz}$

$\delta(^{31}\text{P}) = 10.9$

$n = 1.403$

$d = 1.203 \text{ kg}\cdot\text{m}^{-3}$

The sample contains 2% of monoester



The above-mentioned two sets of data are in an excellent agreement with published values [D.G. Gorenstein, Progr. NMR Spectr. 16, 1 - 98 (1983) and CRC Handbook of Chemistry and Physics, 51st Edition. The Chemical Rubber Co., Cleveland, USA, (1970).]