

NEW METHOD FOR PREPARING ORGANOFUNCTIONAL IMIDO SILANES

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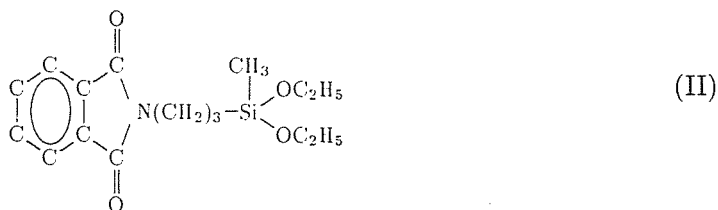
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Abstract

A study has been carried out on the preparation of some organofunctional imido silanes. This has been accomplished by means of addition of methylhydor-diethoxysilane to N-allylphthalimide by hydrosilylation reaction. The product can be hydrolyzed to the corresponding hydroxyl compounds.

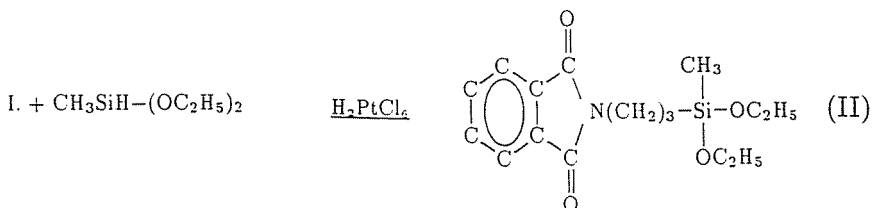
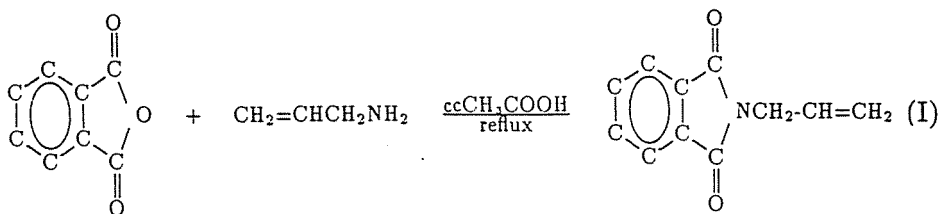
Keywords: poly(imidisiloxanes), siloxane, adhesion promoters, polyimides.

The aim of the present work is to reveal a new method for preparing novel organofunctional imidosilanes having the formula:



Experimental

The synthesis of these compounds was carried out according to the following reactions:



The second reaction was carried out in a dry clean three-necked flask equipped with condenser, dropping funnel and CaCl_2 tube.

A mixture of 100 mmol, 18.7 g N-allylphthalimide (ELDERFIELD et al. 1955, SAHA et al. 1952) in 55 ml hot dry benzene was stirred. The whole mixture was heated under reflux temperature, and 50 μl of H_2PtCl_6 (0.5 % concentr.) solution in ethyleneglycoldimethylether and hexanol (product of Wacker Chemie GmbH) containing 11 mmol, 14.72 g $\text{CH}_3\text{SiH}(\text{OC}_2\text{H}_5)_2$ (SPEIER et al., 1957, SHOSTAKOVSKII et al., 1957) was added to the reaction mixture dropwise in a time period of 3 hours, after addition the heating was continued for further 45 hours at 110 °C on oil bath.

Following the distillation of the solvent from the reaction mixture under atmospheric pressure, the residual oil was distilled under vacuum to give compound II, b.p. 144° /0.2 .1,333 hekto Pa, with a yield of 80 % ($n_D^{20} = 1.5090$). The average molecular weight was about 305 g, it was determined by using ebulliometric method in CHCl_3 . Chemical analysis was:

	Calc.	Found
C	59.78 %	60.00 %
H	7.21 %	7.72 %
N	4.36 %	4.54 %

Hydrolysis was carried out with a 2:1 ratio of water and the initial compound by use of *p*-toluene sulfonic acid as catalyst and after refluxing the mixture for 6 hours we obtained a solid colourless powder as product (m.p. 204–205 °C). The completion of the reaction was proved by ¹H-NMR spectra (until no signals appear assigned to the OC₂H₅ groups). Yield was 86 %.

Infrared spectral data (cm⁻¹) of compound II.

$\nu_{\text{C=O}}$	1700–1750(s)	$\nu_{\text{Si-O}}$	1020
$\nu_{\text{Si-CH}_3}$	1280	$\nu_{\text{Si-CH}_2}$	1400-1420
ν_{CH_2}	2885–2900	$\nu_{\text{Si-OC}_2\text{H}_5}$ as Si-O-C	1100 doublet

¹H-NMR data of compound (II) in CDCl₃, δ TMS = 0 ppm
 0.03 (3H, s, Si-CH₃)
 0.4–0.7 (2H, q, Si-CH₂-C)
 1.0–1.2 (4H, t, N-(CH₂)₂-C)
 1.4–1.9 (4H, m, 2 OCH₂)
 3.4–3.82 (6H, q, C-CH₃)
 7.47–7.8 (4H, m, aromatic)
 s = singlet, t = triplet, q = quartet, m = multiplet

Discussion

The advantage of this new method is that the mentioned imidosilane derivative can be prepared by an easier and simpler method than reported in (GIULIANA et al., 1987). It was declared derivative that the addition of silane to the unsaturated C=C fragment in terminal position undergoes according to anti Markovnikov rule. This was proved for the investigated reaction too from ¹H-NMR data, since no signal appeared from C-CH₃ group.

The industrial use of this silane derivative in addition to adhesion promoters (GIULIANA et al., 1987) is highly promising specially for preparing different types of polymers with other organosilicon compounds, e.g. (CH₃)₂Si(OEt)₂.

Our interest is also directed to this point, the results of work in this field will be published later.

Acknowledgement

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