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FOREIGN TECHNOLOGY DIVISION

METHOD OF PREPARING HETERO-ORGANIC PHOSPHONITRILE POLYMERS

by

S. M. Zhibukhin, V. V. Kireyev,
et al.

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Zhivukhin, S. M. ; Kireyev, V. V. ; Kolesnikov, G. S. and Raygorodskiy, I. M.

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13. ABSTRACT

This method of preparing hetero-organic phosphonitrile polymers by the interaction of alkoxy- or -chloroalkoxy-phosphazenes with hetero-organic compounds containing halogens is distinguished by the fact that hetero-organic compounds containing a halogen in the organic radical are used. This is done to raise the hydrolytic stability of polymers. The general formula for these compounds is given. [AA1016879]

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Chlorinated Organic Compound						
Organosilicon Compound						
Organogermanium Compound						
Organolead Compound						
Organotitanium Compound						
Organic Azine Compound						
Ethoxy Compound						

EDITED TRANSLATION

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METHOD OF PREPARING HETERO-ORGANIC PHOSPHONITRILE POLYMERS

By: S. M. Zhivukhin, V. V. Kireyev, et al.

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U. S. BOARD ON GEOGRAPHIC NAMES TRANSLITERATION SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А а	*А а*	A, a	Р р	*Р р*	R, r
Б б	*Б б*	B, b	С с	*С с*	S, s
В в	*В в*	V, v	Т т	*Т т*	T, t
Г г	*Г г*	G, g	У у	*У у*	U, u
Д д	*Д д*	D, d	Ф ф	*Ф ф*	F, f
Е е	*Е е*	Ye, ye; E, e*	Х х	*Х х*	Kh, kh
Ж ж	*Ж ж*	Zh, zh	Ц ц	*Ц ц*	Ts, ts
З з	*З з*	Z, z	Ч ч	*Ч ч*	Ch, ch
И и	*И и*	I, i	Ш ш	*Ш ш*	Sh, sh
Й й	*Й й*	Y, y	Щ щ	*Щ щ*	Shch, shch
К к	*К к*	K, k	Ъ ъ	*Ъ ъ*	"
Л л	*Л л*	L, l	Ы ы	*Ы ы*	Y, y
М м	*М м*	M, m	Ь ь	*Ь ь*	'
Н н	*Н н*	N, n	Э э	*Э э*	E, e
О о	*О о*	O, o	Ю ю	*Ю ю*	Yu, yu
П п	*П п*	P, p	Я я	*Я я*	Ya, ya

* ye initially, after vowels, and after ъ, ь; e elsewhere. When written as ё in Russian, transliterate as yё or ё. The use of diacritical marks is preferred, but such marks may be omitted when expediency dictates.

METHOD OF PREPARING HETERO-ORGANIC PHOSPHONITRILE POLYMERS

S.M. Zhivukhin, V. V. Kireyev, G. S. Kolesnikov and I. M. Raygorodskiy

(Applicant: Moscow Institute of Chemical Technology im. D. I. Mendeleyev)

A method exists for preparing polymer compounds containing phosphorous elements with bonds $-N=\underset{|}{P}-O-Э$, where Э — the organo-substituted atoms of silicon, tin, germanium, lead, titanium and others. Such polymers, containing in the basic chain the $P-O-Э$ bond, provide unsatisfactory hydrolytic stability, which reduces the possibility of their practical application.

In order to raise the hydrolytic stability of polymers, it is proposed that the hetero-organic compounds be used which contain a halogen in the organic radical. These compounds have the following general formula:

$$\left[(\mathrm{Hal}R_1)_\pi \overset{R_2}{\underset{R_3}{Si}}\right]_m Y_p$$

where R_1 - methyl, ethyl, propyl,

R_2, R_3 - alkyl- or aryl-radicals;

Y - alkyl, aryl, oxygen, methoxy-, ethoxy-,

n, m - 1, 2, 3, 4,

p - 0, 1, 2, 3, and

they interact with alkoxyphosphazenes (AFA) or alkoxylchlorophosphazenes (AKhFA).

The proposed method makes it possible to sharply increase the hydrolytic stability of polymers without losing their heat and fire resistance; a high process rate is provided. During the accomplishment of the method alkyl chloride is given off which, being inert under the conditions of the process, does not effect the course of the side-reactions and can be easily eliminated from the process.

Example 1. Heated in a four-necked flask (equipped with an agitator, a thermometer, a low-temperature trap and a bubbler for the nitrogen supply) at 160°C for 5 h are 5.24 g of hexabutoxycyclotriphosphazotriene and 2.13 g of $[ClCH_2-\overset{CH_3}{\underset{CH_3}{Si}}-]_3O$. In the trap are caught 1.40 g of butyl chloride (83% of the theoretical). After the reaction mixture is cooled, it is extracted with petroleum ether (fraction 40-70°C), and then is dried out. A solid transparent polymer (4.2 g of it) is received that is brown in color.

Found, %: P 16.3, N 7.40, C 33.41, H 7.80, Si 9.5.

Example 2. Hexabutoxycyclothiphosphazotriene (5.29 g) and tetrakis(chloromethyl)dimethyldisiloxane (2.62 g) are heated in the device described in Example 1 at 160°C for 4 h. The quantity of liberated butyl chloride is 1.5 g (89% of the theoretical). After cooling and extraction we received 4.7 g (75.5% of the

theoretical) of transparent light-yellow polymer, which is insoluable in organic solvents.

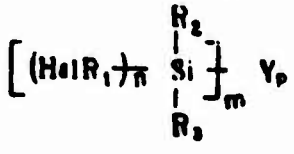
Found, %: C 30.6, H 6.93, Cl 6.92, N 8.64, P 18.90, Si 8.16.

Example 3. Hexabutoxytriphosphazene (4.3 g) and tetramethyltetrachloromethylcyclotetrasiloxane (2.48 g) are heated for 2 h at 160°C. The reaction mixture is vacuum evaporated at 160°C and dried out. Received are 5.38 g of light-yellow insoluble polymer.

Example 4. Hexabutoxycyclotriphosphazotriene (5.24 g), bis(chloromethyl)tetramethyldisiloxane (2.13 g), o-dichlorobenzene (20 ml) and quinoline (0.1 g) are heated at 160°C for 3 h. After the solvent is distilled in a vacuum, a solid transparent polymer (4.0 g), brown in color, is received.

Object of the Invention

This method of preparing hetero-organic phosphonitrile polymers by the interaction of alkoxy- or -chloroalkoxy-phosphazenes with hetero-organic compounds containing halogens *is distinguished* by the fact that hetero-organic compounds containing a halogen in the organic radical are used. This is done to raise the hydrolytic stability of polymers. The general formula for these compounds is



where R_1 - methyl, ethyl, propyl,

R_2, R_3 - alkyl or aryl,

Y - alkyl, aryl, oxygen, methoxy, ethoxy,

n, m - 1, 2, 3, 4,

p - 0, 1, 2, 3.