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A METHOD FOR OBTAINING A COMPOSITION, (U)
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FOREIGN TECHNOLOGY DIVISION

A METHOD FOR OBTAINING A COMPOSITION

By

Ye.I. Minsker, N.V. Varlamova, et al

EDITED TRANSLATION

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A METHOD FOR OBTAINING A COMPOSITION

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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.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	$\sinh^{-1}$
cos	cos	ch	cosh	arc ch	$\cosh^{-1}$
tg	tan	th	tanh	arc th	$\tanh^{-1}$
ctg	cot	cth	coth	arc cth	$\coth^{-1}$
sec	sec	sch	sech	arc sch	sech^{-1}
cosec	csc	csch	csch	arc csch	csch^{-1}

Russian	English
rot	curl
lg	log

A METHOD FOR OBTAINING A COMPOSITION

Ye. I. Minsker, N. V. Varlamova,
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N. F. Orlov, and T. F. Altukhova

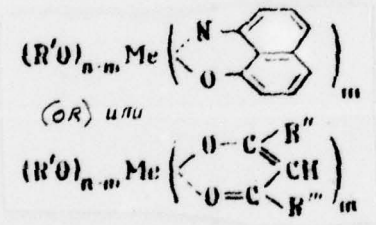
There is a known method for obtaining compositions by mixing the hydroxylpolydiorganosiloxanes, filler, and hardening catalyzers.

In order to impart to the composition higher adhesive properties to the various materials, in the method being proposed we use a mixture consisting of silicon-organic compounds with the general formula $R'H_{2n}CnSi(OR")_3$ as the hardening catalyzer, where n=1–5;

R'-H, $NH_2N(R"')_2$;

R"-alkyl, cycloalkyl, acetyl;

R"'-alkyl, cycloalkyl, and chelate compound of metals with the general formula



where n=3,4; m=1–3;

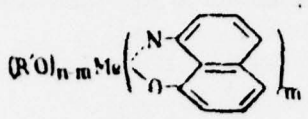
R' - Alkyl with the carbon atom number of up to 5;

R" - $CH_3(CH_2)_x$ (where x=0–4);

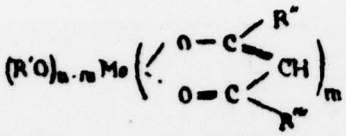
R'"- alkyl or alkoxyl; Me–Ti or Al.

The components of the hardener mixture should have the following ratios (in wt. by part):

$RH_{2n}C_nSi(OR')_3$ 0.1 ÷



(OR) или 0.1 - 40



It is better to use the chelate compounds of metals in a solution of silane with the general formula $(R''''O)_4Si$, where R'''' - alkyl with the carbon atom number from 1 to 5 with the ratio 2:1-1:5.

These compositions are self-adhesive, elastic, heat-resistant, and moisture-resistant organosilicon materials which do not require the use of primers. These properties of the compositions make it possible to use them as glues and sealers.

TABLE 1

(1) Исходные компоненты							
(4) Название		(2) содержание, вес. ч. (3) номер состава					
		1	2	3	4	5	6
(5) СКТ-Н		100	100	100	100	100	100
(6) Наполнитель	ZnO			150	150	150	200
	SiO_2	25	25				
(7) Катализатор отверждения	Ia	15	12,5	12,5	25	12,5	0,2
(8) Ускоритель отверждения	IIIб	—	—	—	—	5	—

KEY: 1) Initial components 2) Content, wt. by part 3) Composition No. 4) Components 5) SKT-N 6) Filler 7) Hardening catalyst 8) Accelerator

For cementing transparent materials one should use the compositions without a filler with the use of aluminum diisopropoxyacetylacetonate, which provides the necessary optical transparency of the bonded joint. High adhesion properties of these materials are retained both under the usual conditions and under the effect of a 98% humidity and temperature of up to 250°C.

Example 1. The compositions are prepared in a blade mixer out of α,ω-dihydroxypolydimethylsiloxane (SKT-N) with the viscosity at 14'42" according to VZ-1 (nozzle - 5.4 mm), fillers, and hardening catalyst.

The apparatus is filled with SKT-N and with a given amount of filler, after which, the mixture is mixed again for 3–4 h. Into the pastes prepared in this manner under the conditions under which the material is prevented from coming into contact with the moisture of the air the hardening catalyst, la, is introduced (a solution of aluminum diisopropoxyacetylacetonate in tetraethoxysilane with the 1:2 ratio) with the accelerator lllb (diethylaminomethyltriethoxysilane) or without it. The components are used in the amounts shown in Table 1.

The prepared compositions, with the exception of the compositions 2 and 6, are dispensed into air-tight metal tubes from which it is applied to hard backings in the form of a layer which is not over 3 mm thick. The materials are cured in the air at room temperature. The tests in the initial state are carried out after 150 h after the material has been applied to the backing. The adhesion is determined by separating a brass grid (GOST 6613-53) from the backing with the applied and cured composition according to TU-18-1-61. Table 2 shows the results of the tests.

Example 2. The composition is prepared using the procedure in example 1 with the introduction of the hardening catalyst lb (a solution of titanium dibutoxybis-(acetylacetonate) in tetraethoxysilane with 1:1 ratio) into its composition and the accelerators (or without them) in the amounts shown in Table 3. The compositions are cured and prepared for testing using the procedure in example 1. All compositions, with the exception of composition 3, are placed into the hermetically sealed tubes. The test results

are given in Table 4.

TABLE 2

(3) Показатель	(1) Номер состава																				
	1				2				3				4				5				6
	(2) Подложка																				
	(4) сталь	(5) медь	(6) алюминий	(7) органическое стекло	(4) сталь	(5) медь	(6) алюминий	(7) органическое стекло	(4) сталь	(5) медь	(6) алюминий	(7) органическое стекло	(4) сталь	(5) медь	(6) алюминий	(7) органическое стекло	(4) сталь	(5) медь	(6) алюминий	(7) органическое стекло	(6) алюминий
(8) Адгезия, $кг/см^2$ в исходном состоянии	1,0	0,75	0,8	—	1,3	1,5	0,8		2,35	2,22	1,90	1,92	2,6	2,42	2,03	2,06	2,6	1,95	2,00	2,55	1,6
(9) после воздействия 98%-ной влажности в течение 170 *час*	0,9	0,67	0,72		—	—	—	—	1,65	1,45	0,90	1,30	1,75	1,63	1,23	1,47	1,95	1,10	1,04	1,40	1,0
(10) после воздействия температуры 250°C в течение 50 *час*	—	—	—	—	—	—	—	—	1,05	0,55	0,45			1,1	0,65	0,5		0,95	0,9	0,6	1,8
(11) Время образования поверхностной пленки, *час*		24				7				10				7				0,5			8

KEY: 1) Composition No. 2) Backing 3) Index 4) Steel 5) Copper 6) Aluminum 7) Organic glass 8) Adhesion, kg/cm^2, in the initial state 9) After the effect of a 98% humidity for 170 h 10) After the effect of temperature at 250°C for 50 h 11) - Time of surface film formation, h

Example 3. The composition is prepared according to the procedure used in example 1 with the addition of catalyst 1b and accelerator 111b in the amounts shown in Table 5 (composition 2) and is tested according to a known procedure using the cross method as per VTU and STU-36-13-61-62 for the KI-2 cement in section IV-12 after a preliminary holding of the samples in the air for 150 h. The results are presented in Table 6.

Example 4. The composition is prepared in the same way as in example 1 with the addition of catalyst 1a and accelerator 111b in the amounts shown in Table 5 (composition 1) and is tested by the procedure used in example 3. The results are presented in Table 6.

Example 5. The composition is prepared as in example 1 (compositions 3 and 4 in Table 7) with the addition, as an adhesive hardening catalyst, of the IIa compound, which is a solution of aluminum diisopropoxy-8-oxyquinolate in tetraethoxysilane (1:2) and the 111b accelerator (or without it) in the amounts shown in Table 7. The obtained compositions were tested as a bonding material according to the procedure used in examples 3 and 4. The results are presented in Table 7.

TABLE 3

(3). Исходные компоненты	(1) Содержание компонентов, вес. ч. (2) номер состава					
	1	2	3	4	5	6
(4) СКТ-Н	100	100	100	100	100	100
(5) Наполнители ZnO	—	—	—	150	150	150
(5) Наполнители SiO_2	25	25	25	—	—	—
(6) Катализатор 16	15	12,5	12,5	12,5	25	12,5
(7) Ускоритель 111а	—	—	0,33	—	—	—
(7) Ускоритель 111б	—	—	—	—	—	5

KEY: 1) Content of components, parts by wt. 2) Composition No.
3) Initial components 4) SKT-N 5) Fillers 6) Catalyst 1b
7) Accelerator - 111a, 111b

Example 6. The composition is prepared the same way as in example 1 (see Table 7, composition 5) with the addition of catalyst 11b, which is a solution of titanium dibutoxybis-(8-oxyquinolate) in tetraethoxysilane with 1:1 ratio, into the composition. The composition was tested as a bonding material according to the procedure used in examples 3 and 4. The results are given in Table 8.

TABLE 4

(3) Показатель	(1) Номер состава																							
	1				2				3				4				5				6			
	(2) Подложка																							
	(4) сталь	(5) медь	(6) алюминий	(7) органическое стекло	сталь	медь	алюминий	органическое стекло	сталь	медь	алюминий	органическое стекло	сталь	медь	алюминий	органическое стекло	сталь	медь	алюминий	органическое стекло	сталь	медь	алюминий	органическое стекло
(8) Адгезия, $кг/см^2$ в исходном состоянии	1,37	1,25	1,57	—	1,30	1,15	1,55	1,15	2,35	2,20	1,95	—	2,60	2,37	2,03	—	2,7	2,48	2,20	1,92	3,50	3,30	2,90	2,50
(9) после воздействия 98%-ной влажности в течение 170 час	1,30	1,25	1,50	—	1,17	1,12	1,47	1,12	1,47	—	—	—	0,6	0,5	—	—	0,72	0,6	0,50	—	1,90	1,50	1,20	1,0
(10) после воздействия температуры 250°С в течение 50 час	—	—	—	—	—	—	—	—	—	—	—	—	1,15	1,05	0,8	—	1,30	1,20	0,90	—	2,55	2,35	1,65	—
(11) Время образования поверхностной пленки, час	24				8				10				10				7				0,5			

KEY: 1) Composition No. 2) Backings 3) Indices 4) steel 5) copper 6) aluminum 7) organic glass 8) Adhesion, kg/cm^2 in the initial state 9) after the effect of a 98% humidity for 170 h 10) after the effect of temperature at 250°C for 50 h 11) Time it takes to form a surface film, h

TABLE 5

(2) Исходные компоненты		(1) Номер состава 1	(1) Номер состава 2
(3) СКТ-Н		100 150	100 150
(4) Катализатор	1а 1б	12,5 —	— 12,5
(5) Ускоритель	111б	5	5

KEY: 1) Composition No. 2) Initial components 3) SKT-N 4) Catalyst - 1a, 1b 5) Accelerator 111b

TABLE 6

(3) Показатель	(1) Номер состава															
	1									2						
	(2) Склеиваемые материалы															
	(4) стекло—стекло	(5) сталь—сталь	(6) сталь—стекло	(7) алюминий—алюминий	(8) алюминий—стекло	(9) органическое стекло—органическое стекло	(10) пластмасса—пластмасса	(11) дерево—дерево	(4) стекло—стекло	(5) сталь—сталь	(6) сталь—стекло	(7) алюминий—алюминий	(8) алюминий—стекло	(9) органическое стекло—органическое стекло	(10) пластмасса—пластмасса	(11) дерево—дерево
(12) Адгезия, кг/см² в исходном состоянии	14,5	20,5	9,0	15,6	14,6	11,2	18,3	18,0	19,0	22,2	10,8	18,9	19,3	11,2	11,2	10,0
(13) после воздействия 98%-ной влажности в течение 72 час	11,5	17,8	5,6	14,0	13,2	8,9	11,2	9,2	11,6	19,0	10,5	11,6	16,7	9,5	9,3	4,5

KEY: 1) Composition No. 2) Bonded materials 3) Index 4) glass-glass 5) steel-steel 6) steel-glass 7) aluminum-aluminum 8) aluminum-glass 9) organic glass-organic glass 10) plastic-plastic 11) wood-wood 12) Adhesion, kg/cm^2 in the initial state 13) after the effect of a 98% humidity for 72 h

TABLE 7

(2) Исходные компоненты	(1) Номер состава		
	3	4	5
(3) СКТ-Н	100 150	100 150	100 150
(4) Катализатор отверждения IIа	25	37	—
Катализатор отверждения IIб	—	—	12,5
(5) Ускоритель отверждения IIIб	5	—	5

KEY: 1) Composition No. 2) Initial components 3) SKT-N 4) Hardening catalyst - lla, llb 5) Accelerator lllb

Object of the invention

1. A method used for obtaining a composition by mixing the hydroxylpolydiorganosiloxanes, mineral filler, and hardener is *distinguished* by the fact that, in order to impart higher adhesion properties to the composition for various materials, we used a mixture consisting of siliconorganic compounds as the hardener with

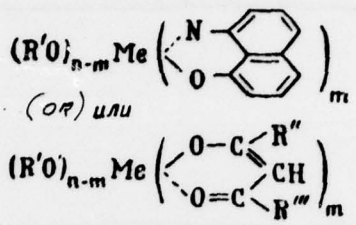
the general formula

$$R'H_{2n}C_nSi(OR'')_3,$$

where n=1–5; R'–H, NH_2, $N(R''')_2$;

R" - alkyl, cycloalkyl, and acetyl;

R'" - alkyl, cycloalkyl, and chelate compound of metals with the general formula



where n=3,4; m=1–3;

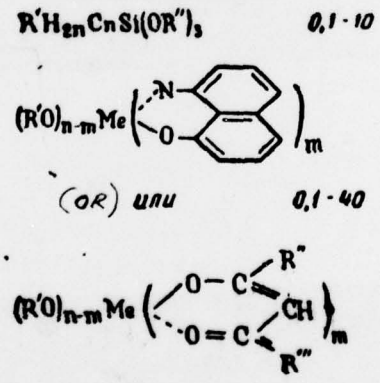
R' - alkyl with carbon atom number up to 5;

R" - $CH_3(CH_2)_x$ (where x=0–4);

R'"- alkyl or alkoxyl;

Me - Ti or Al.

2. The method in section 1 is *distinguished* by the fact that the components of the hardening mixture are used in the following ratios (in parts by wt.):

Siloxane	100
	0,1 - 10
(OR) или	0,1 - 40

3. The method in section 1 is *distinguished* by the fact that the chelate compounds of metals are used in a solution of silane with the general formula $(R''''O)_4Si$, where R"" - alkyl with the carbon atom number from 1 to 5, in the ratio of 2:1–1:5.

TABLE 8

(3) Показатель	(1) Номер состава																	
	3						4						5					
	(2) Подложка																	
	(4) сталь	(5) органическое стекло	(6) алюминий	(4) сталь	(7) пластмасса	(8) дерево	(9) стекло	(5) органическое стекло	(6) алюминий	(4) сталь	(7) пластмасса	(8) дерево	(9) стекло	(5) органическое стекло	(6) алюминий	(4) сталь	(7) пластмасса	(8) дерево
(10) Адгезия, кг/см² в исходном состоянии	11,5	8	13	5	9	12	11	5	10	5	9	12	16	11,6	8,7	7,6	15,6	19,2

KEY: 1) Composition No. 2) Backing 3) Index 4) steel 5) organic glass 6) aluminum 7) plastic 8) wood 9) glass 10) Adhesion, kg/cm^2 in the initial state

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C619	MIA REDSTONE	1
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