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Journal Name:	International Research Journal of Pure and Applied Chemistry
Manuscript Number:	2013_IRJPAC_7257
Title of the Manuscript:	New Methylphenylsiloxane Resins and High Temperature Resisting Enamels on Their Basis
Type of the Article	

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This journal's peer review policy states that **NO** manuscript should be rejected only on the basis of '**lack of Novelty**', provided the manuscript is scientifically robust and technically sound.

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PART 1: Review Comments

	Reviewer's comment	Author's comment (if agreed with reviewer, correct the manuscript and highlight that part in the manuscript. It is mandatory that authors should write his/her feedback here)
Compulsory REVISION comments	1. The first two lines of Experimental for the synthesis of the materials need to be written with details, because the cited references are either patents written in Russian or a conference proceeding. The description should include what are the starting materials, chlorosilanes or alkoxysilane, and the reaction procedures. Example of a paper: - D. R. Bujalski et al., <i>Macromolecules</i>, 2003, <i>36</i>, 180-197. 2. In the Experimental section for ^{29}Si NMR, the authors need to write the condition for quantitative analysis. 3. In the description in the first 6 lines in page 5, the exact compositions of the resins should be described, namely, exact values of a, b, c, d, , x, and y for materials 1-6. 4. In the above item 3, the authors also need to provide how they have determined the SiOH and Si(OEt) contents. The ^{29}Si NMR spectrum cannot differentiate between SiOH and Si(OEt), while the Si(OEt) content could be theoretically obtained from ^1H NMR. 5. For the TGA studies in Table 1 and the sentences below Table 1, the term "destruction" should be "decomposition". 6. Then, the authors need to describe the definition of "Decomposition" point in Table 1. Is it the temperature at which 5% weight decrease is	1. 200 g of toluene, 32.8 g of oligophenylsiloxane (OphS), 53.4g of MTEOS (methyltriethoxysilane), catalytical amounts of sulfuric acid are fed to the reactor with temperature control heating, a stirrer, a backflow condenser The reaction mixture under continuous stirring is heated to the temperature of 60-70°C, is held for an hour, then acetic acid is added dropwise. The reaction mass is held for 1-4 hours at a temperature of 75-85 °C depending on required resin properties. Then ethylacetate reaction by product is distilled and the reaction mass is brought to neutral reaction of the environment by the conventional methods. The solvent is next distilled to volatiles content not more than 3 wt %. . Element analysis, % C=47.20, %H=4.63 %Si=23.18 2. ^{29}Si NMR spectra were measured with the employment of pulse program known as "Inverse Gated Heteronuclear Decoupling". In order to reduce relaxation time chrome tris(acetylacetonate) was added to the solutions. Delay between the pulses was 10 s. As a result ^{29}Si nuclei signal of various structural fragments are well separated. 3. This comment will be accepted in a new amended version of Table 1. 4. Low content of both hydroxyl and ethoxy



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	observed? 7. For the TGA study, the authors describe that thermal stability decreases with increasing phenyl content. However, the thermal decomposition mechanism may be simply silicon-carbon bond scission. If so, the difference in the weight loss could be simply the weight difference of CH₃ and C₆H₅. Can we call that difference in stability? The extent of decomposition may be similar. 8. Line 6 from the bottom on page 6, the full name of GPC is gel <u>permeation</u> chromatography. 9. Starting in the middle of page 7, the authors describe the performance of enamels. They should provide the type and amount of the fillers or pigments as well as the solvent used and substrates. 10. The quality index 1, 3, 5 in row 6-9 in Table 3 needs to be clarified. These performances may not be able to be expressed quantitatively, but the authors should at least indicate what 1, 3, and 5 means for each row. In the discussion for Figure 2, the authors discussed thermal stability in terms of weight loss. Is the heat resistance of the enamel also discussed by weight loss?	groups in the produced MPhSR is not sufficient for quantitative measurement of integrated intensity of both -<u>Si</u>-O- H silicon fragments signals in ²⁹Si NMR spectra (fig 1b) and -Si-O- CH₂-<u>CH</u>₃ methyl groups fragments in PMR spectra (fig. 1 a). Therefore we determined their content by chemical analysis. The data are presented in Table 2. 5. The term has been replaced. 6 Thermal decomposition proceeds in a few stages. The Table presents thermal decomposition start up temperature of oligomer main chain (stage 3). 7. In the selected range of [PhSi]_a/ [MeSi]_b ratios at MPhSR production the combination of two parameters responsible for thermal stability increase was observed, namely: phenyl radicals content and Mw increase, occurring at MTEOS content increase (see Table 3) Table 3 demonstrates that as compared with starting OPhS whose inorganic residue yield amounts to 45 wt % , for synthesized MPhSR inorganic residue yield increases within the range of 62-80 wt %. 8. The term has been replaced. 9.Heat resistant coating composition is as follows: Methylphenylsiloxane resin 3.5-13.0 Pentaphthalic varnish 3.0-14.5 Butyl methacrylate polymer 5.0-7.0 Heatproof pigment 10.5-15.0 Filler 8.0-13.0 Thickener 1-2 Rheological additive 1-2
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		Organic solvent – the rest 10. The tests were conducted in conformity with Russian government technical requirement GOST - 9.401-91
<u>Minor</u> REVISION comments	1. The references are Russian journals, published patents written only in Russian and patents not published yet. This is not sufficient for an academic publication in English. For example, the authors could cite the following articles for the introduction for references 1 and 3: - L. H. Brown, "Treatise on Coatings: Silicones in Protective Coatings", R. Myers, J. S. Long Ed; Marcel Dekker (1972) Vol. 1, Part 3, Chapter 13, pp. 513-563. - R. H. Baney, et al., <i>Chem. Rev.</i>, 1995, 95, 1409-1430. 2. The increase in molecular weight with increasing MeSi/PhSi ratio discussed in page 6-7 is in line with the tendency of faster reaction rate and gelation for MeSi(OR) and PhSi(OR). The authors could refer to the following references: - N. Shimizu et al., <i>Chem. Lett.</i>, 1992, 1263-1266. - D. A. Loy et al., <i>Chem. Mater.</i>, 2000, 12, 3624-3632.	Thank you for the comments. We'll make the amendments.
<u>Optional/General</u> comments		



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Table 1 Starting Reagents concentrations and their ratios

№ п.п	*Methylphenylsiloxane resin (MPhSR) composition	Starting Reagents concentrations and their ratios					
		Starting Reagents concentrations, mole			Ratio		
		Varnish KO-075	MTEOS	AA	AA/ MTEOS	* [PhSi] _a / [MeSi] _b	**** According to ²⁹ Si spectrum
1	$[(\text{PhSiO}_{1,5})_c(\text{PhSiO}(\text{OH}))_d]_{0,21}/$ $((\text{MeSiO}_{1,5})_x(\text{MeSiO}(\text{OH}))_y)_{0,42}$	0.21	0.42	2.0	4.76	0.50	0.47
2	$[(\text{PhSiO}_{1,5})_{0,94}(\text{PhSiO}(\text{OH}))_{0,06}]_{0,2}/$ $[(\text{MeSiO}_{1,5})_{0,69}(\text{MeSiO}(\text{OH}))_{0,31}]_{0,158}$	0.2	0.158	1.58	10.00	1.26 ()	1.17
3	$[(\text{PhSiO}_{1,5})_{0,92}(\text{PhSiO}(\text{OH}))_{0,08}]_{0,22}/$ $[(\text{MeSiO}_{1,5})_{0,78}(\text{MeSiO}(\text{OH}))_{0,22}]_{0,22}$	0.22	0.22	2.0	9.09	1.00	1.34
4	$[(\text{PhSiO}_{1,5})_{0,95}(\text{PhSiO}(\text{OH}))_{0,05}]_{0,25}/$ $[(\text{MeSiO}_{1,5})_{0,76}(\text{MeSiO}(\text{OH}))_{0,24}]_{0,158}$	0.25	0.158	1.88	11.8	1.58	1.87
5	$[(\text{PhSiO}_{1,5})_c(\text{PhSiO}(\text{OH}))_d]_{0,42}/$ $[(\text{MeSiO}_{1,5})_x(\text{MeSiO}(\text{OH}))_y]_{0,21}$	0.42	0.21	2.0	9.52	2.00 ()	2.03
6	$[(\text{PhSiO}_{1,5})_c(\text{PhSiO}(\text{OH}))_d]_{0,26}/$ $[(\text{MeSiO}_{1,5})_x(\text{MeSiO}(\text{OH}))_y]_{0,045}$	0.26	0.045	3.3	73.3	5.77 (-)	5.77