



INNOVATIVE SILICONE SPECIALTIES



## Siltech LLC

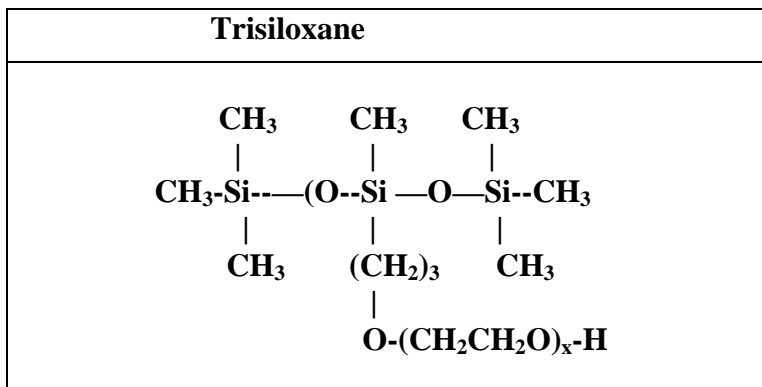
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### Topical Report: Hydrolytic Stability Dimethicone Copolyols

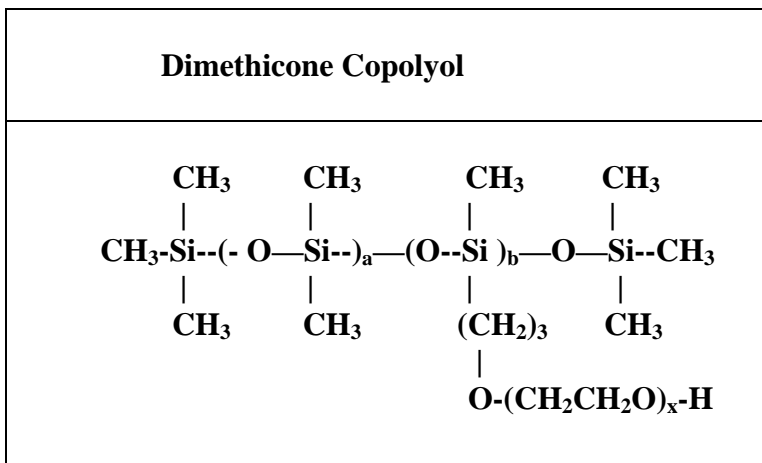
#### Background

Dimethicone Copolyol compounds (INCI PEG dimethicone) are an important class of materials used in the personal care market. There are two distinct classes of these materials. One class the trisiloxane has hydrolytic instability issues and consequently are not used in the personal care market.

Trisiloxanes have only three "Si" atoms. They conform to the following structure;



Dimethicone copolyol compounds have more than three silicone molecules and it is this structural difference that makes them more stable.





The desirable properties of both trisiloxanes and dimethicone copolyol compounds are shown below.

Silsurf®	Molecular Weight	Wetting 0.1% Sol	CMC mg/l	Surface Tension @ CMC	Super Wetting*
<b>A-008</b>	<b>633</b>	<b>7 sec</b>	<b>20</b>	<b>21</b>	<b>56</b>
<b>A-208</b>	<b>855</b>	<b>8 sec</b>	<b>20</b>	<b>22</b>	<b>5</b>
<b>B-208</b>	<b>1398</b>	<b>10 sec</b>	<b>20</b>	<b>23</b>	<b>2</b>
<b>D-212CG</b>	<b>2706</b>	<b>257 sec</b>	<b>23</b>	<b>26</b>	<b>2</b>

The term super wetting as applied here relates to the ability of a 1% solution of the test surfactant to spread on paraffin without mechanical means. The trisiloxane (Silsurf A-008) is the only product in the series that demonstrates these properties. However, it also is the material with the most limited hydrolytic stability. Blending this material with another silicone glycol neither overcomes the hydrolytic instability of the trisiloxane, nor does it improve the wetting of the non trisiloxane component.

While super wetting is very structure specific, Draves wetting, (the ability to sink a cotton skein in a surfactant solution) is quite different. There is a gradual increase in wetting time as a function of increased molecular weight. Eye irritation of the surfactant drops off as molecular weight increases. This means there are a number of compounds that have outstanding Draves Wetting, and at the same time low irritation.

Wetting is a critical factor in almost every application for silicone surfactants. Silicone surfactants used for conditioning are high molecular weight materials. They are also poor wetting agents. Therefore the ability to spread uniformly on substrates is difficult, resulting in non-uniform films. It is therefore recommended that a lower molecular weight silicone surfactant be added to wet out the higher molecular weight silicone surfactant. This is true for textile fabrics, plastics and hair. Since wetting occurs at or below the CMC, one should add enough to have the product present at that concentration available after the formulation has been cut to use concentration.



### Hydrolytic Stability

**Method:** All the surface tension measurements are done with 0.1% solution at room temperature. Sigma 70 with Surface Tension software version 2.52 is used for the experiment. 0.1% solution is made by mixing 0.6 gram of silicone polyether with 600 ml of de-ionized water. The solution is then divided into three parts in 4 oz bottles. The first part is used “as is” without any pH adjustment. The second part is adjusted to pH value of 10 with ammonium hydroxide. And the third part is adjusted to pH value of 4 with acetic acid. The pH of the solution is re-adjusted to the desired value after the first week, if needed..

**Table 1**  
Surface tension measurements for Silsurf Products

Sample	Surface Tension vs. Time								
	Day 1	Day 2	Day 3	Day 4	Day 5	Day 7	Day 14	Day 21	Day 28
<b>Trisiloxane</b>									
Silsurf A008	21.0	20.8	20.8	20.8	20.9	20.9	20.7	20.8	20.7
Silsurf A008-pH10	20.8	21.0	21.5	21.6	22.4	21.7	21.6	21.6	24.0
Silsurf A008-pH4	20.8	22.3	25.3	28.9	<b>43.5</b>	<b>51.3</b>	<b>55.8</b>	<b>57.5</b>	<b>55.2</b>
Silsurf A008-pH8	20.9	21.0	20.8	20.9	20.9	20.9	20.6	21.3	21.3
<b>Dimethicone</b>									
<b>Copoyol</b>									
Silsurf A208	21.9	21.8	22.0	21.8	21.8	21.9	21.7	21.9	21.8
Silsurf A208-pH10	22.1	21.9	22.1	22.0	21.9	22.0	21.7	21.6	22.6
Silsurf A208-pH4	22.1	21.7	22.1	22.3	21.8	22.5	22.2	22.6	22.3
Silsurf B208	23.1	22.4	22.9	23.1	22.9	22.9	23.2	22.7	22.5
Silsurf B208-pH10	23.3	23.0	23.5	23.1	23.1	23.7	22.9	22.7	23.4
Silsurf B208-pH4	23.4	23.2	23.4	23.4	22.9	23.2	23.3	22.9	23.8
Silsurf D212-CG	26.2	26.6	25.8	25.7	25.5	26.2	26.1	25.9	25.9
Silsurf D212-pH10	25.9	26.2	26.5	25.3	25.2	25.6	25.9	26.9	26.0
Silsurf D212-pH4	25.8	26.9	26.2	25.2	25.4	26.8	25.8	26.2	27.0



**Table 2**  
**Surface tension measurements for Silsurf Products**

<b>Days</b>	<b>28.0</b>	<b>58.0</b>
	20.7	20.8
<b>Trisiloxane</b>		
Silsurf A008-pH7		
Silsurf A008-pH10	24.0	41.0
Silsurf A008-pH4	55.2	60.9
Silsurf A008-pH8	21.3	31.8
<b>Dimethicone Copolyol</b>		
<b>Days</b>	<b>28.0</b>	<b>58.0</b>
Silsurf A208-pH7	21.8	21.9
Silsurf A208-pH10	22.6	23.0
Silsurf A208-pH4	22.3	24.0
<b>Days</b>	<b>28.0</b>	<b>58.0</b>
Silsurf B208-pH7	22.5	23.4
Silsurf B208-pH10	23.4	24.3
Silsurf B208-pH4	23.8	25.4
<b>Days</b>	<b>28.0</b>	<b>58.0</b>
Silsurf D208-pH7	25.9	26.5
Silsurf D208-pH10	26.0	27.4
Silsurf D208-pH4	27.0	27.3



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## Conclusion

1. Silsurf A008 (the trisiloxane) at pH 4 is found to be unstable after 4 days. At pH 10 it took 58 days to show instability.
2. The dimethicone copolyols did not show the same instability. Over the range of pH values encountered by the majority of the personal care applications (pH 5-9) dimethicone copolyol compounds are expected to be stable.

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