# Pemulen Revised: pHuck the pH Meter

*by Chris Stavroudis*

It is never fun to admit one was wrong. Particularly when one is the "expert," and one has been wrong for quite a long while about something. To see just how long I was wrong, I refer you to the *WAAC Newsletter* of May 1989. In that article, with some prescience, I said "You must check the pH yourself with pH papers or a meter; trust no one." Trust no one, including, it would seem, me.

It turns out that pH meters do not accurately measure the pH of a Pemulen or Carbopol gel, even when thinned extensively with distilled water. Richard Wolbers suspects that the polymer (Pemulen or Carbopol) interferes with the reference electrode incorporated into the pH meter.

To measure the pH of a Pemulen gel, remove a small amount of the gel (about 1 gram) and dilute with distilled or deionized water (about 10 mL) until the solution is a thinner, evenly dispersed liquid. At this point the pH can be measured with pH papers.

However, to measure the pH more precisely, and now also accurately, add approximately 1/2 gram salt (NaCl) to the well dispersed gel and stir – the viscosity should go way down and the solution will become a bit cloudy. A pH meter can then be used to measure the pH of the thinned gel. (If this all sounds vaguely familiar, you may have heard of this trick in another context. The news media reported widely that this is what "the kids" are doing with hand sanitizer these days – adding salt to collapse the gel -- so that they can drink the alcohol used therein.)

So, here follows corrected recipes for making stock Pemulen TR2 gels and a revised method of measuring the pH of those gels. Note that these recipes correct those that graced the pages of this very *Newsletter* just a few issues ago (September 2010).

As before, all of these recipes are for a 2% gel concentrate that is ultimately diluted to a 1% working solution. By making stock solutions at 2%, additional components, say from the MCP, can be added to modify the aqueous chemistry of the Pemulen phase before solvents are added.

pH 6.5: Suspend 4g Pemulen TR2 into 100mL distilled or deionized water, stir until uniform and well dispersed. Dissolve 6.7 grams (6.0 mL) triethanolamine into 95mL water. With vigorous stirring, mix the Pemulen suspension with the TEA solution. Check the pH and adjust to pH 6.5 with additional TEA or Pemulen. Bring final volume to 200mL.

pH 7.5: Suspend 4g Pemulen TR2 into 100mL distilled or deionized water, stir until uniform and well dispersed. Dissolve 8.5g (7.6mL) triethanolamine into 90mL water. With vigorous stirring, mix the Pemulen suspension with the TEA solution. Adjust to pH 7.5, and bring final volume to 200mL.

pH 8.0: Suspend 4g Pemulen TR2 into 100mL distilled or deionized water, stir until uniform and well dispersed. Dissolve 13g (11.5mL) triethanolamine into 85mL water. With vigorous stirring, mix the Pemulen suspension with the TEA solution. Adjust to pH 8.0, and bring final volume to 200mL.

pH 8.5: Suspend 4g Pemulen TR2 into 100mL distilled or deionized water, stir until uniform and well dispersed. Dissolve 9.0 grams (8.0 mL) triethanolamine and 12.5g (12.5mL) 10% sodium hydroxide solution into 85mL water. With vigorous stirring, mix the Pemulen suspension with the TEA/NaOH solution. Adjust to pH 8.5 with additional 10% NaOH solution; and bring final volume to 200mL.

[Note that the above recipe for a pH 8.5 Pemulen gel is neutralized with both TEA and sodium hydroxide solution. The reason for this is that TEA has a pKa of 7.76. At a pH of 8.5, the 85% of the TEA is present in its molecular form and only 15% is present as the triethanolammonium ion. Because only the triethanolammonium ions can react with the carboxylic acid sites, for a given number of moles of Pemulen, we would need to add 6 times as many moles of TEA, which would be quite a lot of TEA.]

These recipes, one hopes, will give you the pH promised. If the pH is low, add a bit more base to the stock gel, mix well, and repeat the testing process. If the pH is too high, add in a bit more of the Pemulen dispersed in distilled water.

When finished, rinse your pH electrode in dilute sodium hydroxide (~1%) to ensure that the Pemulen is dissolved away before rinsing the electrode in tap and then distilled water between measurements.

To prepare the 1% working solution dilute the stock Pemulen gel 1:1 with other aqueous preparations. If diluting the stock gel with a concentrated stock solution from the MCP, for each 5mL of stock Pemulen, add 2mL of concentrated MCP solution and 3mL water. If adding two MCP components, add 2mL of each MCP solution and 1mL water to the 5mL of stock Pemulen gel.

I find that a small, wide-mouthed "ointment" jar placed on a magnetic stirrer works wonders for dispersing the thick Pemulen stock gel in the aqueous solution – give it a few minutes. You may want to tape the jar to the stirrer since as the viscosity increases, the stirrer may start spinning the jar as well.

To use your Pemulen TR2 to make an emulsion:

To make the emulsion, take a small amount of the 1% working Pemulen gel and place in a jar. Add any non-water soluble solvent and shake. It should instantly form a stable emulsion. (Even better, stir on the above mentioned magnetic stirrer until a smooth, creamy emulsion has formed.) 2% benzyl alcohol in the Pemulen is an excellent starting point for testing the emulsion. Pemulen TR2 can make stable emulsions with as much as 30%, and sometimes up to 50%, added solvent by volume.