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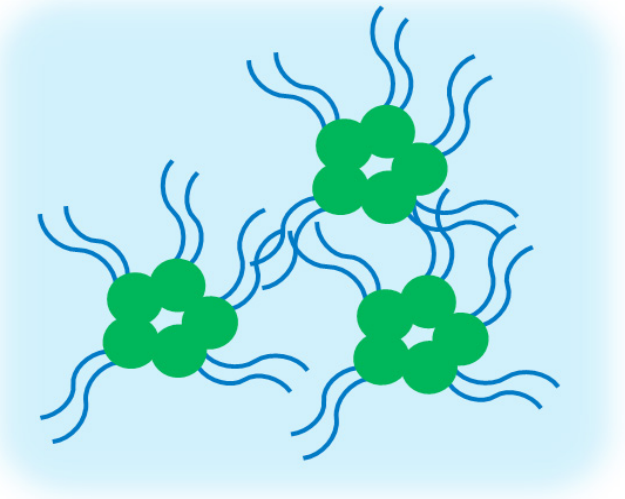
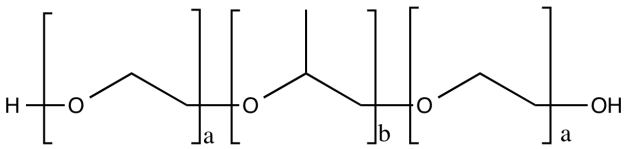
Determining weight percent oxyethylene in poloxamers

Introduction

A poloxamer is a synthetic block copolymer made up of ethylene oxide and propylene oxide. Poloxamers are known under the Commercial name of Synperonics, Pluronic (F127, F68,..), Kolliphor,.. and are commonly used as non-ionic surfactants. Here we demonstrate the NMR method published by the United States Pharmacopeia for determining weight percent oxyethlene of poloxamers using a Sympatens PAP 188.



USP Method



The poloxamer is made up of three domains, one hydrophobic polypropylene oxide block in the center straddled by hydrophilic polyethylene oxide blocks at both ends. The size and ratio of these blocks will change the chemical and physical properties of the polymer as a whole. The spectrum of the poloxamer is shown below. The doublet at about 1 ppm is the propylene $-CH_3$ peak, while the peak at about 3.6 ppm is the CH_2O resonance of both the oxyethylene and oxypropylene backbones and also the CHO peak from oxypropylene.

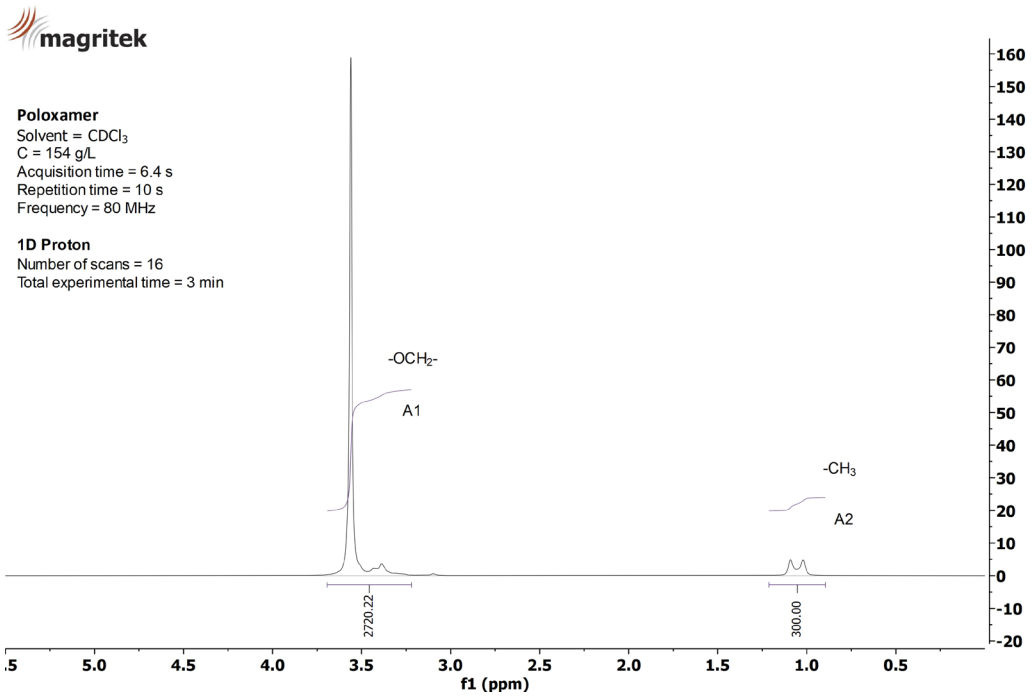
The integrals of these peaks are used to calculate the weight percentage oxyethylene from the equation:

$$\% \text{ oxyethylene} = 3300 \times \frac{\alpha}{33 \times \alpha + 58}$$

where $\alpha = A_1 / A_2 - 1$

and A_1 is the integral of the composite peak from 3.2 to 3.8 ppm and A_2 is the integral of the doublet at 1.08 ppm.

The poloxamer was dissolved in deuterated chloroform at a concentration of about 154 g/L and measured on a Spinsolve 80 Carbon benchtop NMR spectrometer. The measured weight percent oxyethylene was 82%.



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