Determination of the Polyoxyethylene (POE) Percentage in Poloxamers

Application Note – Pulsar 003

Background

Poloxamers are an important category of polymers with significant applications in industry, especially for pharmaceutical, cosmetic and personal care products.The most common uses are related to its surfactant properties. In the cosmetics and personal care products, Poloxamers are used for various formulations including mouthwashes, lens cleaning solutions, skin cleansers, shampoos and conditioners. In the pharmaceutical industry, Poloxamers are used as suspension and emulsifying agents, coating materials and importantly as components of drug delivery systems. The United States Pharmacopeia and The National Formulary (**USP–NF**) specifies 5 different types of poloxamers with different chain lengths of x and y (see Figure 1).

Poloxamers are non-ionic block copolymers comprising a central polyoxypropylene block flanked by two polyoxyethylene blocks (figure 1). The central block is hydrophobic while the flanking blocks are hydrophilic resulting in a polymer which is an amphiphile and suitable for use as a non-ionic surfactant. The amphiphilic character of the polymer is dependent on the chain lengths and can be tailored for specific applications.

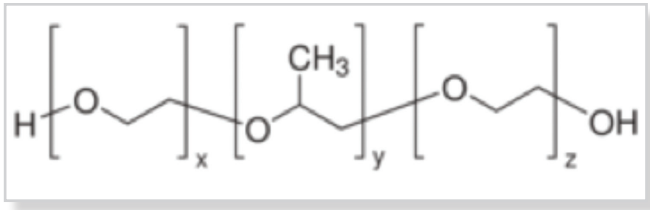

Figure 1: *Structure of generic poloxamer*

Poloxamers are identified by their poloxamer number which takes the form "P abc", where the number ab multiplied by 100 is the mass per mole of polyoxypropylene in the polymer and c multiplied by 10 is the percentage of polyoxyethylene. For example, poloxamer p188, contains 1800 g/mol polyoxypropylene and 80% polyoxyethylene.

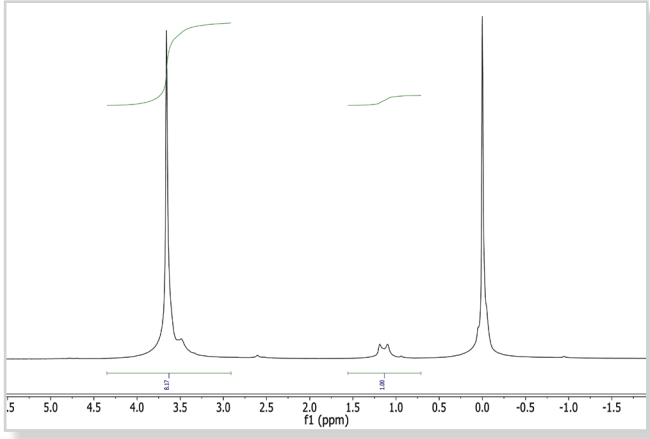

Figure 2: *^{1}H spectrum of poloxamer 188 in $CDCL_3$*

Analysis

The method for performing the NMR analysis involves collecting a 1-dimensional ^{1}H spectrum and integrating the region around 1.1 ppm (region 1) corresponding to the $-CH_3$ group on the polyoxypropylene block and the region around 3.7 ppm (region 2) corresponding to the $-CH_2-$ and -CH- groups on all three blocks.

The percentage of polyoxyethylene in the polymer can be evaluated from $3300\alpha / (33\alpha + 58)$ where $\alpha = (I2 / I1) - 1$ *and* $I1$ and $I2$ are the integrals of region one and two respectively.

The method relies on integrating regions of the spectrum containing multiple peaks, but does not require that these peaks are fully resolved, simply that region one and region two are well separated.

Figure 2 shows the ^{1}H spectrum of poloxamer 188 acquired at 1.4 T, ^{1}H resonance frequency of 60 MHz. The data is a single scan collected over a large spectral width, 5.2 KHz with 64K complex data points that are zero filled on acquisition. The spectrum shows that the two integral regions are sufficiently well separated to allow integration.

OXFORD INSTRUMENTS

The Business of Science®

The results of integrating the spectra of four commercially available poloxamers are given in table 1. The reproducibility of the NMR results was determined by collecting 16 spectra for each sample and calculating the % of polyoxyethylene from each data set. An average value and standard deviation were determined from these results. The estimate show excellent comparison with the values implied by the poloxamer.

Name	Poloxamer number	% POE (NMR)	% POE (p-number)
Kolliphor P188	P 188	82.1 ± 0.6	80
Kolliphor P407	P 407	72.4 ± 0.4	70
Pluronic F127	P 367	73.8 ± 0.6	70
Synperonic F108	P 308	83.4 ± 0.8	80

Table 1: *% of polyoxyethylene in a poloxamer determined by NMR at 60 MHz. The numbers are compared to the values associated with the poloxamer number.*

Oxford Instruments Industrial Analysis

For more information please email: **industrial@oxinst.com**

UK
Tubney Woods, Abingdon,
Oxfordshire, OX13 5QX, UK
Tel: +44 1865 393 200

China
Room 1/E, Building 1,
Xiangzhang Garden,
No. 248 Donglan Road,
Shanghai 201102, China
Tel: +86 21 6073 2925

India
11, Marwah's Complex,
Andheri East,
Mumbai, 400072, India
Tel.: +91 22 4253 5100

Japan
Haseman Bldg,
Tokyo, 135-0047, Japan
Tel: +81 3 5245 3251

Singapore
10 Ubi Crescent,
Singapore, 408564, Singapore
Tel: +65 6337 6848

USA
300 Baker Avenue, Suite 150,
Concord, Mass 01742, USA
Tel: +1 978 369 9933