Homogeneity of a plasticised Nitrocellulose binder formulation

Dr E Stubbs and Dr S Hazelwood

Introduction

Plasticised nitrocellulose (NC) binder formulations are used at AWE to carry out laboratory scale thermal ageing tests which characterises the material both chemically and physically.

One method of chemically characterising NC binder formulation is gel permeation chromatography (GPC) which measures the molecular mass distribution (mmd) of the NC.

To observe any significant ageing effects on the NC, the analysis of the binder must exclude other sources of variation, the analytical procedure must therefore be accurate and robust.

Current methodologies require a relatively small sample (≈20 mg) to be analysed. Recent analysis, has shown that a large variation was observed between samples generated from the same NC lacquer, whereas a larger sampling size (≈2 g) produced a more representative and reproducible result (Figure 1 and 2, right).

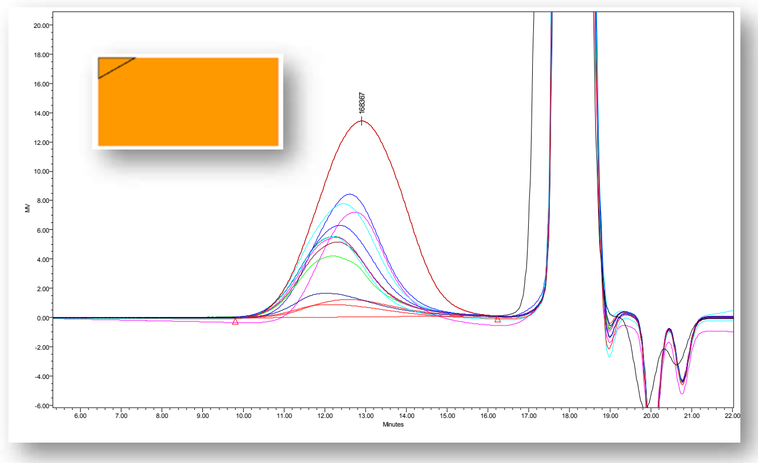

Figure 1: Small sampling size

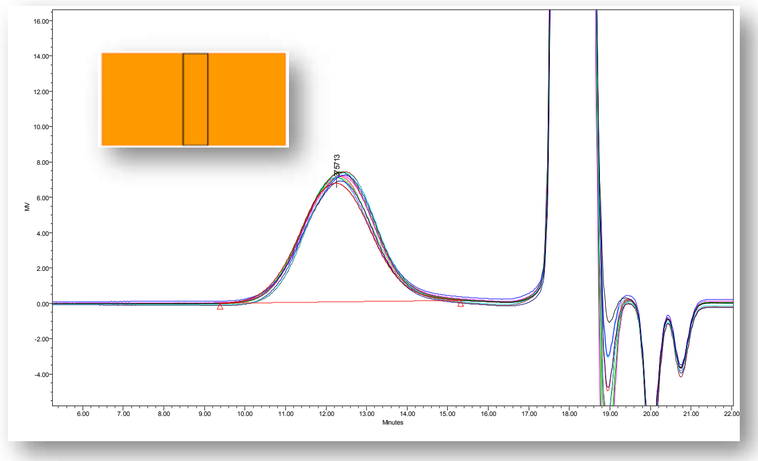

Figure 2: Large sampling size

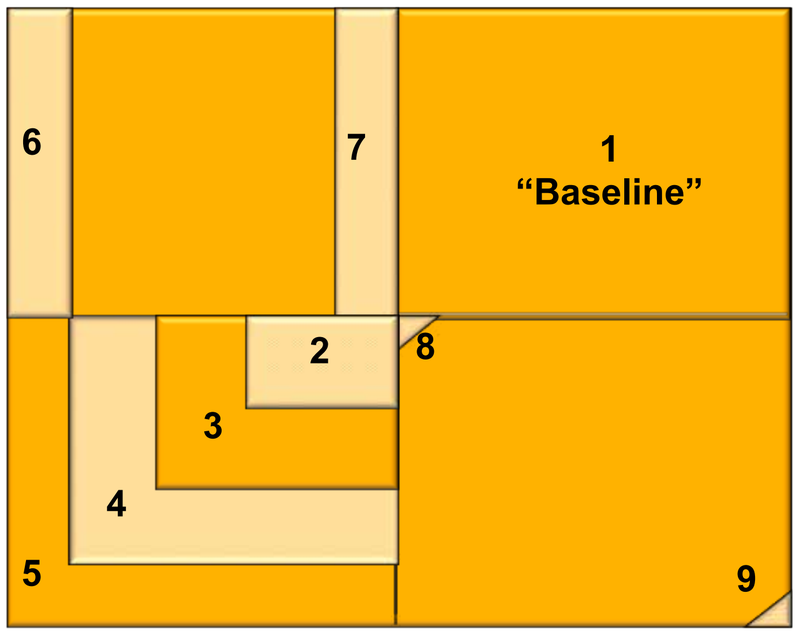

Figure 3: Binder sheet sampling site

This led to the conclusion that binder sheets are not entirely homogeneous, and sample preparation is pivotal to produce reliable results.

A single (recently prepared) binder sheet was taken and sampled across various sites (Figure 3, left). Consistent concentration was maintained by varying solvent volume added to sample.

A percentage recovery of NC was calculated from the various sampling sites relative to the baseline sample (Figure 4, left).

Sample	Mass of Sample (g)	Conc. (mg/mL)	Relative Recovery	Mw	IV
1 "Baseline"	19.8714	1.9991	100	435,822	4.9716
2	1.1050	2.2100	100	440,364	5.0517
3	3.0192	1.9995	113	485,682	5.5537
4	4.1572	1.9987	118	511,337	5.8117
5	6.6451	2.0015	97	420,772	4.8458
6	4.4131	1.9969	94	383,968	4.5514
7	4.4713	1.9961	105	453,324	5.1511
8	0.1240	2.0667	122	521,075	5.9621
9	0.1194	1.9900	88	352,265	4.3006

Figure 4

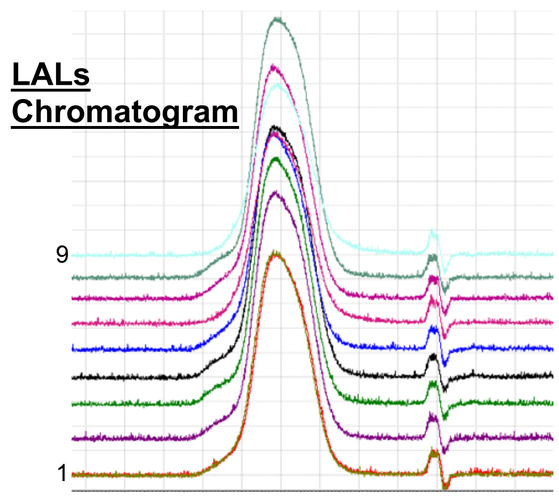

Figure 5

Analysis conditions

Instrument: Malvern TDA

Software: OmniSEC 5.2

Detectors: RI, RALs/LALs, Viscometer

Columns: x2 Viscotek T6000M General Mixed Org. 300 x 8.0 mm

Standards: Narrow PS105K calibration and Broad PS245K check standard

Reagents: THF stabilised with 100ppm BHT (3,5-bis-(tertiarybutyl)-4-hydroxytouene)

Flow Rate: 1 ml/min

Inj. volume: 100 µl

Run Length: 45 mins

Injections: Triplicate

Processing: Integration limits and baselines were set manually, known sample concentration rather than dn/dc

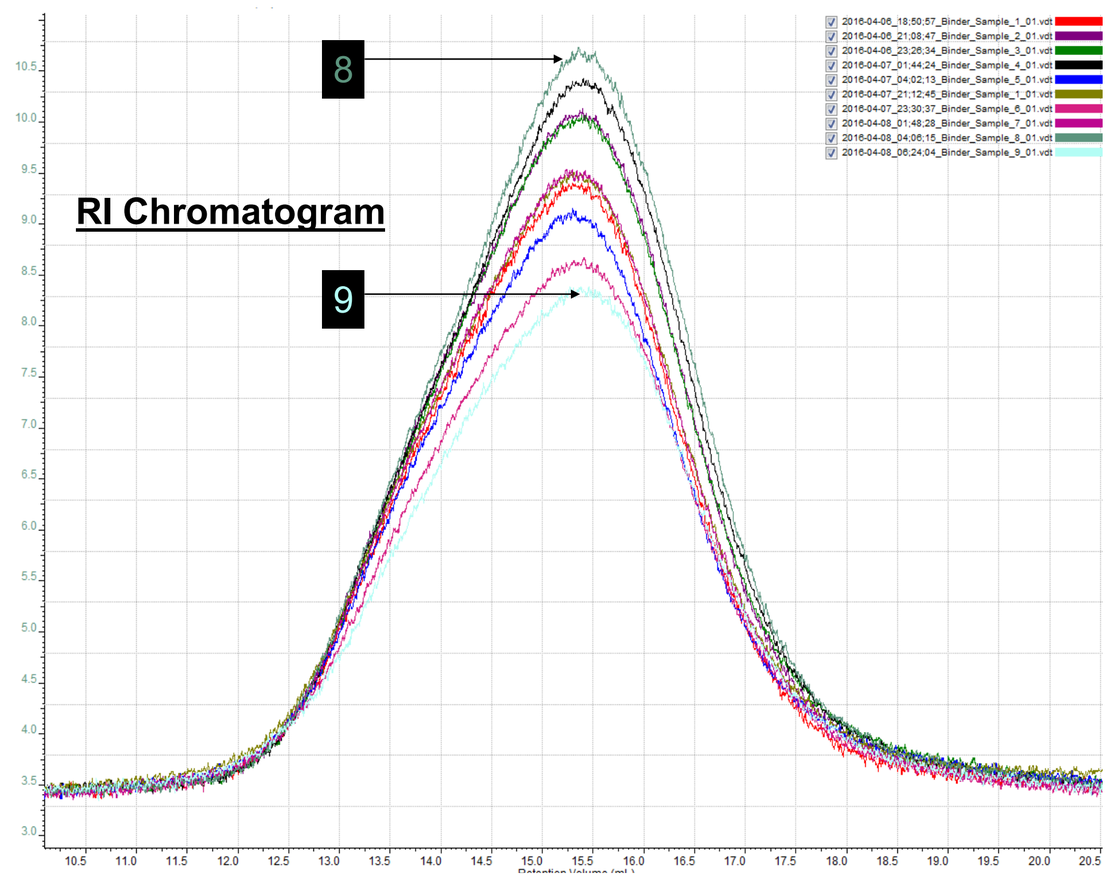

Figure 4

The results show clear variation between different sampling positions across the binder sheet.

Where NC recovery was low the Mw and IV were lower than expected, and where recovery was high, Mw and IV were also higher than expected. As well as variation across the light scattering and refractive index traces (shown Figure 5 and 6).

This data adds further weight to the theory that the binder sheet is not homogeneous throughout, and the variation is made worse by taking a small sample size.

The inhomogeneity appears to be caused during formation of the sheet, and potentially accelerated by the thermal ageing process

Conclusion

The importance of sample prep for GPC analysis has been demonstrated if accurate and reliable data is to be generated. The analysis has also shown that GPC is capable of providing deeper understanding into Nitrocellulose behaviour in various materials.

Dr E Stubbs and Dr S Hazelwood
AWE, Aldermaston, Reading, Berkshire, United Kingdom.

© British Crown Owned Copyright 2016/AWE

MG/*****/PME