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CHARACTERISATION OF NITROCELLULOSE BY GEL PERMEATION CHROMATOGRAPHY

PART 1

TECHNIQUE AND CALIBRATION

by

D E Hillman and J I Paul

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CHARACTERISATION OF NITROCELLULOSE BY GEL PERMEATION CHROMATOGRAPHY

PART I
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E E Longhurst
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SUMMARY

The characterisation of nitrocellulose by gel permeation chromatography is reviewed in the light of experience in MQAD. Methods are compared for the calibration of molecular weights by several 'universal' calibration methods. The influence of the concentration effect is very large, and extrapolation of average molecular weights to zero concentration is considered to give the best repeatability. Problems arising from peak shape, solution stability and the influence of degree of nitration are discussed.

1. INTRODUCTION

This programme of work began as part of an investigation into the batch to batch variations in performance of cast double base propellant (nitrocellulose + nitroglycerine + additives). It was thought that characterisation of the nitrocellulose by gel permeation chromatography (GPC) might give useful information. However, a number of problems were encountered which were either not found with other polymers, or were not present to the same degree.

The principal problems were:

a. Chromatogram shape - a long tailing distribution on the low molecular weight end of the distribution is difficult to measure.

b. Presence of major impurity peaks at the lower molecular weight end of the molecular weight distribution.

c. Influence of sample concentration.

d. Stability of solutions.

e. Calibration of the GPC for nitrocellulose molecular weight.

f. Influence of degree of nitration.

This report discusses these problems in the light of our experience of the characterisation of propellant grade nitrocellulose. Many workers have examined nitrocellulose by GPC, but almost invariably only as a means of characterising cellulose. The latter is insoluble in GPC solvents and is, therefore, converted to nitrate under mild conditions to obtain a derivative which is soluble in GPC solvents. The problems encountered in this work have not, therefore, corresponded fully with those reported by other workers. This report is, therefore, partly an account of experimental work.

2. EXPERIMENTAL

Apparatus - Waters Associates Model 200 GPC

Column system - 4 × 4 ft columns packed with 'Styragel' of exclusion limits 10^6; 3×10^5; 3×10^4; 10^3Å

Solvent - Tetrahydrofuran (inhibited with 0.025% butylated hydroxy toluene or 0.1% hydroquinone)

Sample size - 2 ml

Span - X8

Flow rate - 1 ml/min

Syphon size - 5 ml

Injection time - 5 minutes

Temperatures: Detector - ca 35°C
Degasser - ca 50°C

Sample preparation - Samples were air-dried at ambient temperatures for at least 5 hours after removal of excess water by absorption on filter paper. The dried samples were dissolved in THF overnight to give a nominal ½% solution. This was diluted accurately 1:1 and both solutions examined by GPC. Average molecular weights were extrapolated to zero concentration.

3. RESULTS AND DISCUSSION

3.1 Chromatogram shape

Two principal types of nitrocellulose sample were examined:

a. the product of the mild laboratory nitration of cotton linters ('nitrated linters').

b. the final stabilised product of the industrial nitration process ('mech pyro').

Figure 1 shows a typical chromatogram for the mechanical pyro sample. This material gave a very wide molecular weight distribution with a long tail extending to very low molecular weights and which merged into the normal solvent impurity peaks. Small variations in the height of this tail, due to slight baseline instability, resulted in large variations in the number average molecular weight ($\bar{M}_n$). The repeatability of $\bar{M}_n$ is, therefore, generally poor. The nitrated linters sample gave a similar shaped chromatogram, although the average molecular weight was considerably higher. Segal[1] showed a chromatogram with some tailing which appeared to be resolved from the impurity peaks. However, the molecular weight range of the material which he examined appeared to be very much higher than those encountered in this project, and resolution would, therefore, be expected to be more complete.

3.2 Impurity peaks

Segal[1] showed that these impurity peaks at the end of the trace were due to dissolved air, moisture or other solvents.

Propellant grade nitrocellulose is normally stored damped with water and, therefore, some degree of drying is required to avoid an enormous negative impurity peak at the end of the GPC chromatogram with consequent further lack of resolution of the tailing peak. Segal[2] used vacuum oven drying for 1 hour at 50-55°C. Work at Woolwich has used air-drying of minimum sample sizes at ambient temperature for at least 5 hours after removal of excess water by absorption on filter paper. Removal of water is not complete, but the size of the impurity peak is not excessive.

3.3 Sample concentration effect

Lack of repeatability of weight average molecular weights ($\bar{A}_w$) when using water damped samples without prior drying was traced to the influence of polymer concentration. Therefore, initially, samples were examined at approximately ½% concentration, diluted to exactly half concentration and re-run. The average molecular weights were then extrapolated to zero concentration. Repeatability of $\bar{A}_w$ was then good and correlation with viscosity measurements became acceptable.

Typical plots of $\bar{A}_w$ against concentration are shown in Figure 2. For nitrated linters the plot is approximately linear at low concentrations, but deviates markedly at above 0.3% concentration. For the mech pyro sample the change of molecular weight with concentration is much less, and is clearly linear up to 1%. Assuming the linear extrapolation at low concentrations to be correct, failure to correct to zero concentration would lead to errors as shown below:

	$\bar{A}_w$ Nitrated linters	$\bar{A}_w$ Mech pyro
½% concentration:	27,700	9,700
zero concentration:	36,000	10,400
% error at ½% concentration:	23	7

Whilst the assumption of a linear relationship between $\bar{A}_w$ and concentration may not be fully correct and the extrapolation based on two concentrations only may be criticised, there is clearly a minimisation of the errors due to the concentration effect.

The sample concentration effect is widely known for other polymers, but does not appear to have been investigated for nitrocellulose, although workers have generally used low concentrations eg ½% - ¼%. Brookshaw, Hillman and Paul[3] have shown that the effect is unusually large for ethyl cellulose, where it increases markedly with increase in polymer molecular weight. This is in accordance with the observations of other workers, eg Troth[4] who showed a linear relationship between concentration effect and intrinsic viscosity for polyethylene. Therefore, enormous errors would be predicted in the results of other workers, eg Wadsworth et al[5] who have investigated nitrocellulose at very much higher degrees of polymerisation, eg DP_w 2490 or higher compared with DP_w of the order of 1000 for our samples. Such errors may be of great importance in the study of calibration methods (Section 3.5).

3.4 Stability of solutions

The effect of ageing on nitrocellulose solutions has been considered by Segal[1], and his figures for DP_w after 10 days and 54 days standing showed little change.

TIME	$\overline{DP}_w$ (x10^{-2})
Initial	16.8
After 10 days	16.8
After 54 days	15.2

At the same time, Segal noted that it was well known that cellulose trinitrate dissolved in organic solvents underwent degradation as indicated by viscosity loss. Carignen and Turngren[6] found by light scattering experiments that nitrocellulose degraded in ethyl acetate from $\bar{M}_w = 20 \times 10^4$ to $\bar{M}_w = 14 \times 10^4$ in 14 days, although acetone solutions were stable over that period. Unfortunately, no light scattering data was obtainable using THF as solvent.

In the present work, weight average molecular weights were calculated for nitrated linters at different concentrations, both freshly prepared and after storage for 6 days. Figure 2 shows that there is a decrease in the value of $\bar{A}_w$ from 38,000 to 30,000 over 6 days after extrapolation to zero concentration. The influence of ageing at higher concentrations is largely obscured by the concentration effect. However, for the lower molecular weight mech pyro sample there is no apparent drop in molecular weight. Indeed, for both samples at ½% concentration only, there is no apparent change in molecular weight, which thus confirms Segal's observed 'slow' ageing effect.

3.5 Molecular weight calibration

Characterisation of nitrocellulose by GPC has been carried out by a number of workers in the past, and considerable difficulty has been encountered in establishing valid molecular weight figures. Ideally, GPC calibration should be carried out with well characterised narrow distribution fractions of the polymer under test. Where such fractions are not available, the instrument is calibrated with polystyrene standards, and conversion from polystyrene molecular weights to those of the test polymer is made by one of several 'universal' calibration methods. These are based on the use of extended chain length, hydrodynamic volume or unperturbed dimensions of the polymer and of polystyrene. The two types of nitrocellulose were characterised by a number of these methods, and the resultant average molecular weights are listed in Table I.

3.5.1 Q-factor method

This method relies on calibration in terms of polystyrene extended chain length (Å). The polymer is characterised in terms of extended chain length which is multiplied by the Q-factor to obtain molecular weight. The Q-factor is defined as molecular weight per unit Angstrom length.

Segal[1,7] used a Q-factor of 58 for cellulose trinitrate. This was derived from X-ray measurements[1]. Lower values were calculated for more normal degrees of nitration (eg $Q = 55.9$ at 13.5% N). Segal's results [1,8] were unexpectedly high and Meyerhoff[9] cautioned against this approach. Meyerhoff himself used cellulose nitrate fractions [9,10,11], but their history has been regarded as ill-defined[12]. Table I lists the results for the two samples used in this work, and, as expected, this method gives very much higher molecular weights than any other.

Normally nowadays the Q-factor method is regarded as satisfactory where there is a similar degree of solvent-polymer interaction which is shown by the 'a' exponent in the Mark Houwink equation. In this case, however, 'a' for polystyrene is approx 0.72, whereas that for nitrocellulose may be as high as 1.14.

3.5.2 Effective density factor ($\bar{Q}$)

Change[13] has suggested the use of this modified Q-factor approach. $\bar{Q}$ is obtained by dividing the number average molecular weight (M_n), obtained by osmometry, by the peak extended chain length, obtained by GPC of polystyrene standards. By using a range of nitrocellulose samples, a factor of $\bar{Q} = 10.5$ was obtained. This factor is, therefore, based on properties in solution rather than in the solid state.

This approach gave much lower molecular weights than the Q-factor method, and the results were in broad agreement with those obtained by other methods (Table I). However, the assumption that peak extended chain length corresponds with $\bar{M}_n$ is an approximation which may invalidate the precision of this otherwise logical approach.

3.5.3 Hydrodynamic volume

The 'universal' calibration based on hydrodynamic volume was originally proposed by Benoit[14]. A plot of elution volume against log $\{\eta\}M$ gives a calibration which is valid for a very wide range of polymers. $\{\eta\}M$ is called the 'coil size', which is a measure of the hydrodynamic volume, $\{\eta\}$ is the intrinsic viscosity and M the molecular weight. Polystyrene is used to calibrate the GPC in terms of $\{\eta\}M$, and a number of approaches may be used to evaluate the nitrocellulose molecular weight.

Segal[2] applied this concept by measurement of the intrinsic viscosities of nitrocellulose fractions eluted from the GPC. Unfortunately, the concentrations in most of the fractions were too low for accurate evaluation of $\{\eta\}$. However, it was clearly shown that this method gave figures 75% lower than those obtained by the Q-factor method.

By combining the Benoit hydrodynamic volume relationship with the Mark Houwink equation, the following formula is obtained for a given elution volume:

$$\text{Log } M_P = \frac{1}{1 + a_P} \log \left(\frac{K_{PS}}{K_P}\right) + \frac{(1 + a_{PS})}{(1 + a_P)} \log M_{PS}$$

where M = molecular weight

a, K = Mark Houwink constants

PS, P = subscripts corresponding to polystyrene and polymer under test respectively

Therefore, if values of 'a' and 'K' are known, a polystyrene calibration can easily be transposed for use with another polymer. However, the values of these constants quoted in the literature vary very greatly, and are said to be dependent on the degree of polymerisation.

Molecular weight averages for the two nitrocellulose samples are given in Table I using different literature values of 'a' and 'K'. In general, they show reasonable agreement, although the values of Jenkins[15] are clearly low, whilst those of French[19] are high. The relatively low values of Dp with our samples may favour consistent results by these different calibration methods since this avoids the alleged discontinuity at about $D_p = 1000$ in the relationship for 'a' and 'K' against D_p.

Wadsworth, Segal and Timpa[5] used the above method expressed in terms of

$$M = \left(\frac{\text{coil size}}{K}\right)^{\frac{1}{(1 + a)}}$$

where coil size = $\{\eta\}M$

Calibration in terms of coil size against elution volume is carried out using standard polystyrene fractions.

3.5.4 Method of unperturbed dimensions (Dawkins)[16]

A method of calibration which has been widely used in this laboratory for other polymers is based on conversion from a polystyrene and the test polymer. However,

values of $\left(\frac{\bar{L}_o^2}{M}\right)^{\frac{1}{2}}$ where $\left(\bar{L}_o^2\right)^{\frac{1}{2}}$ is the unperturbed root mean

square chain displacement[5], were calculated by Krigbaum[17] and shown to increase with M for nitrocellulose. He calculated values of 115×10^{-10} to 205×10^{-10} over a molecular weight range of 40,000 to 2½ million. This approach was not, therefore, considered in this programme of work.

Meyerhoff[10] proposed the use of $\{\eta\}^{\frac{1}{2}}M^{\frac{1}{2}}$ in place of $\{\eta\}M$ in Benoit's method for nitrocellulose. He later preferred the use of acetone as GPC solvent. This cannot be used with 'Styragel' columns, because of shrinkage of the column packing, unless the column is packed in an acetone slurry. This, therefore, precludes the use of acetone in a laboratory where a wide range of polymers is examined.

The report by Cerignen and Turngren[6] mentions further attempts to characterise nitrocellulose fractions and the intention to use preparative GPC to produce narrow molecular weight distribution fractions in the future. The use of well characterised narrow molecular weight distribution samples is clearly the only unambiguous method of calibration, eg as attempted by Alexander and Muller[18].

Because of the confused state of calibration methods for nitrocellulose, much work has been reported from this laboratory in terms of extended chain length (Å). This allows easy comparison of molecular weight distributions, the actual molecular weights being approximately 10 times as large.

Summarising, Table I lists the results obtained by using seven of these calibration methods to characterise the samples of nitrated linters and mech pyro.

Methods 3-7 are based on the use of hydrodynamic volume and show considerable variation according to the literature values of 'a' and 'K' chosen for nitrocellulose and polystyrene. The results by methods 2, 3, 4 and 6 are closely comparable, suggesting that these should be regarded as preferred methods for nitrocellulose of propellant grade or for comparison of cotton linters used in propellant manufacture. The use of the Q-factor of 10.5 suggested by Chang[13] appears to be the simplest way of obtaining actual molecular weights. Future work involving measurement of average molecular weights by light scattering will attempt to verify this approach. These 'recommended' methods, however, may not be equally applicable to nitrocellulose of higher average molecular weight, eg such as examined by Segal and others.

3.6 Influence of degree of nitration

Segal et al[7] have investigated the effect of degree of nitration of cellulose on molecular weight distribution data. Complete esterification to give the theoretical trinitrate (14.14%N) is difficult in practice, and Segal prepared 4 nitrocelluloses with nitrogen contents ranging from 13.51% to 13.81%. He noted that the GPC chromatograms were not significantly different, and concluded that the degree of nitration had little effect on GPC behaviour in this limited range of nitrogen content. In the present work, however, the degree of nitration for nitrated linters was approx. 13.5%N, whilst the mech pyro generally contains approx. 12.5% nitrogen. Thus, Segal's conclusions do not necessarily apply to the work reported here.

Carignen and Turngren[6] have speculated that fractionation of military grade nitrocellulose (N = 13.15%) into molecular weight fractions, ranging from less than 5000 to greater than 200,000, would have corresponding nitrogen contents of less than 12% up to greater than 13.5%. French et al[19] examined 11 nitrocellulose samples, with nitrogen contents ranging from 11.68% to 13.13%, and they used Lindsley and Franks[20] findings that, at given polymer chain lengths, intrinsic viscosity at one nitrocellulose nitrogen content could be converted to the corresponding intrinsic viscosity of cellulose trinitrate by multiplying it by a factor.

The work of Lindsley and Franks[20] gives an empirical equation for the calculation of intrinsic viscosity in acetone for any nitrocellulose of known nitrogen content. Figure 3 shows a plot of log $\{\eta\}M$ against nitrogen content, assuming that this equation is valid for THF solutions. This shows that considerable errors would be introduced by failing to take account of variation in nitrogen content. For example, in the comparison of nitrated linters (13.5%N) and industrial nitrocellulose (12.5%N), the former would be approximately 40% higher in $\{\eta\}M$ value due to the effect of nitrogen content only.

The range investigated by Segal[7] above (ie 13.5 - 13.8%N) would, however, give only 10% change, which would agree with Segal's findings that there was no significant change over this limited range of nitrogen contents.

There appears to be no corresponding information in the literature on variation of intrinsic viscosity with nitrogen content in THF solution. Therefore, no conclusions can be drawn from the present work, except that the comparison of nitrocellulose samples of different nitrogen contents may require care in interpretation.

The examination of several series of samples of cellulose nitrated to different levels of nitrogen content suggests that complete solubility in THF requires an average nitrogen content of approximately 11%, ie a degree of substitution of at least dinitrate.

4. CONCLUSIONS

It has been shown that nitrocellulose can more repeatably be characterised by GPC by examination at two low concentrations with extrapolation of results to zero concentration.

Calibration methods have been reviewed and shown to be dependent on very variable literature data. Since no characterised narrow range nitrocellulose standards are available, initial calibration with polystyrene standards is necessary, followed by conversion to nitrocellulose figures by use of 'universal' calibration methods. The preferred methods have been suggested following the use of a number of calibration procedures to characterise two samples of nitrocellulose. The method of using a $\bar{Q}$-factor of 10.5 is simplest. For many purposes, the use of the equivalent polystyrene chain length provides a valid basis for comparison of samples.

Considerable care is needed in comparing samples nitrated to different nitrogen contents.

ACKNOWLEDGEMENTS

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TABLE 1 - NITROCELLULOSE: SUMMARY OF MOL. WTS. BY GPC

No		Method	Data	Nitrated Linters Sample			Mech Pyro Sample			Reference
				$\bar{M}_w (\times 10^{-4})$	$\bar{M}_n (\times 10^{-4})$	$\bar{M}_w/\bar{M}_n$	$\bar{M}_w (\times 10^{-4})$	$\bar{M}_n (\times 10^{-4})$	$\bar{M}_w/\bar{M}_n$	
1		'Q' factor	For nitrated linters Q = 55.9 For Mech Pyro: Q = 54.1	235	91.6	2.57	54.6	10.3	5.32	1,7
2		'$\bar{Q}$' factor of Chang	$\bar{Q}$ = 10.5	44.2	17.2	2.57	10.6	1.99	5.32	13
3	Hydrodynamic Value	'a' & 'K' values of Segal for D.P. $>$ & $\leq$ 1000 (with Shear)	a) For Nitrocellulose: i) D.P. $>$ 1000; K = 82.6×10^{-5}) a = 0.76) ii) D.P. $\leq$ 1000; K = 0.7×10^{-5}) a = 1.14) b) For Polystyrene: As in 4	41.7	21.2	1.97	11.7	4.03	2.92	21
4	Hydrodynamic Value	'a' & 'K' values of Segal for D.P.60-6000 (zero Shear)	a) For Nitrocellulose: K = 32.1×10^{-5}) a = 0.83) b) For Polystyrene: K = 13.4×10^{-5}) a = 0.71)	40.3	18.2	2.29	10.8	2.63	4.11	21
5	Hydrodynamic Value	a' & 'K' values of Ouano for Polystyrene and Jenkins for Nitrocellulose	a) For Polystyrene: K = 28.9×10^{-5}) a = 0.65) b) For Nitrocellulose: K = 6.06×10^{-5}) a = 1.014)	27.3	14.9	1.82	8.28	2.79	2.96	15(a) 23(b)

TABLE 1 (contd)

No		Method	Data	Nitrated Linters Sample			Mech Pyro Sample			Reference
				$\bar{M}_w (\times 10^{-4})$	$\bar{M}_n (\times 10^{-4})$	$\bar{M}_w/\bar{M}_n$	$\bar{M}_v (\times 10^{-4})$	$\bar{M}_n (\times 10^{-4})$	$\bar{M}_w/\bar{M}_n$	
6	Hydrodynamic Value	'a' & 'K' values of Chang	a) For Nitrocellulose: $K = 21.9 \times 10^{-5}$) $a = 0.89$) b) For Polystyrene: $K = 12.6 \times 10^{-5}$) $a = 0.72$)	35.9	16.5	2.17	9.15	2.46	3.71	13
7	Hydrodynamic Value	'a' & 'K' values of French et al for Nitrocellulose and Spatorico & Coulters for Polystyrene	a) For Nitrocellulose: $K = 18 \times 10^{-5}$) $a = 0.84$) b) For Polystyrene: $K = 11.1 \times 10^{-5}$) $a = 0.725$)	53.7	23.9	2.24	14.2	3.33	4.27	19(a) 22(b)

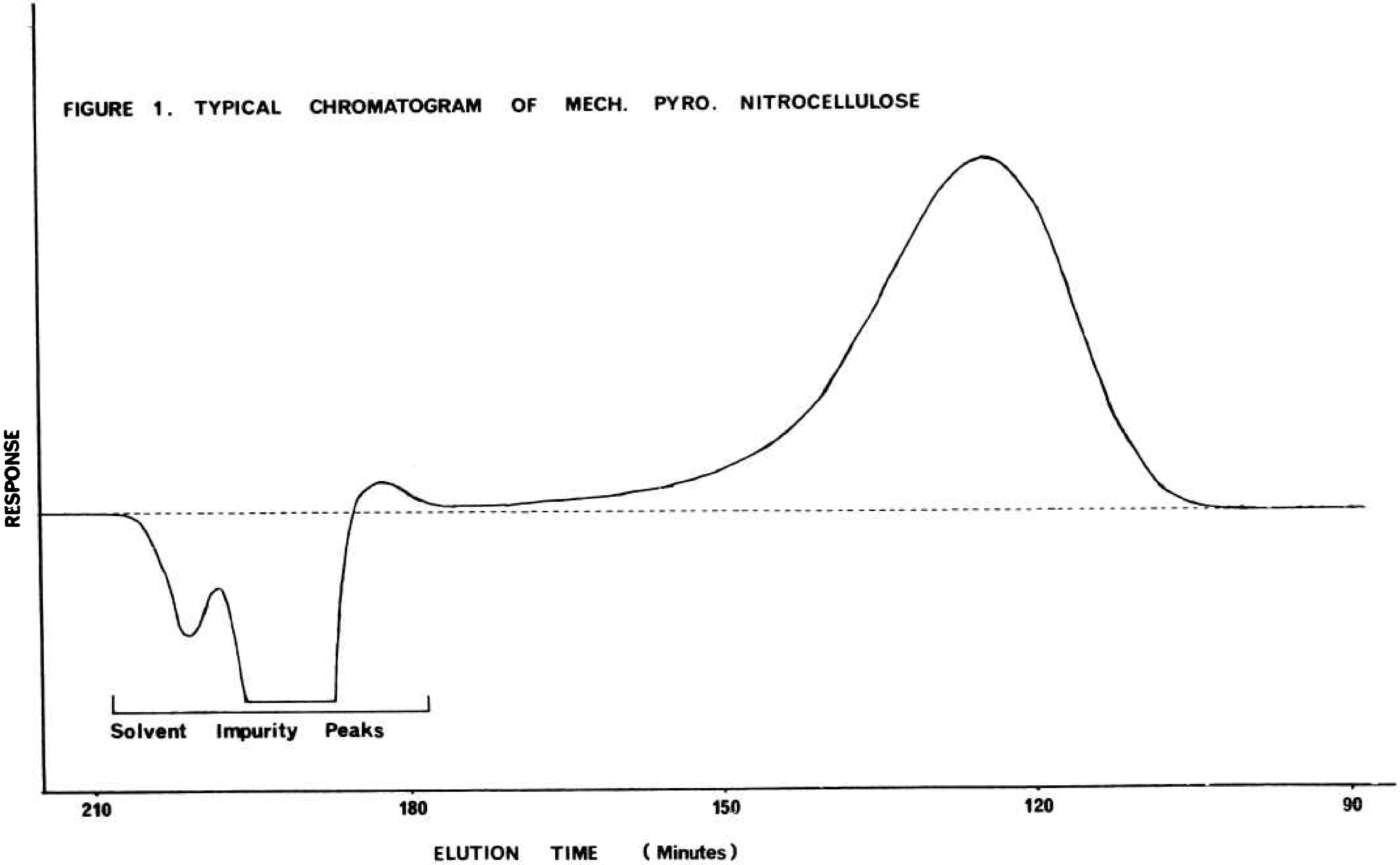


FIGURE 1. TYPICAL CHROMATOGRAM OF MECH. PYRO. NITROCELLULOSE

FIGURE 2. NITROCELLULOSE; CONCENTRATION AND AGEING EFFECTS

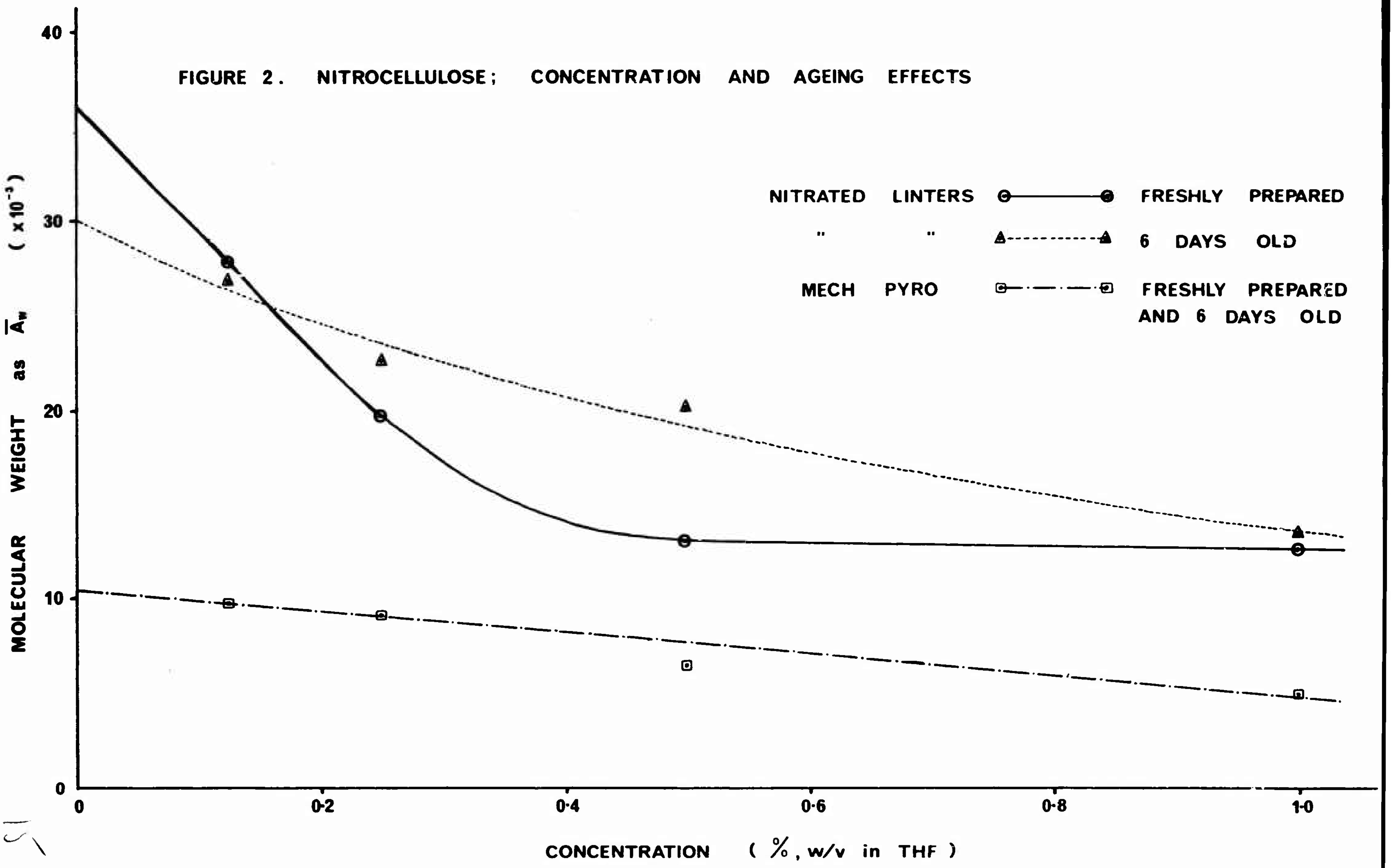
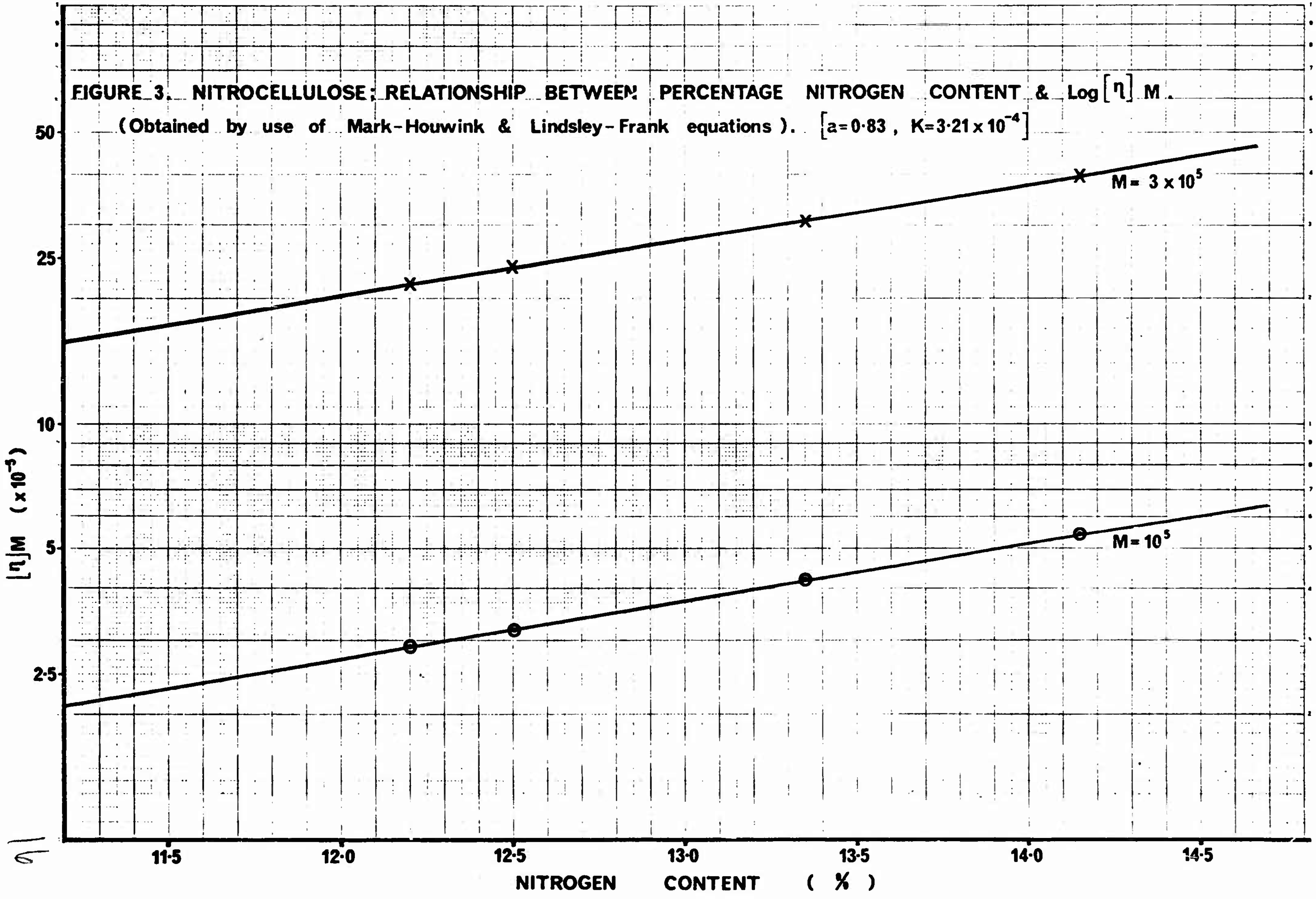


FIGURE 3. NITROCELLULOSE; RELATIONSHIP BETWEEN PERCENTAGE NITROGEN CONTENT & Log $[\eta]$ M.
(Obtained by use of Mark-Houwink & Lindsley-Frank equations). $[a = 0{\cdot}83,\ K = 3{\cdot}21 \times 10^{-4}]$



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Abstract

The characterisation of nitrocellulose by gel permeation chromatography is reviewed in the light of experience in MQAD. Methods are compared for the calibration of molecular weights by several 'universal' calibration methods. The influence of the concentration effect is very large, and extrapolation of average molecular weights to zero concentration is considered to give the best repeatability. Problems arising from peak shape, solution stability and the influence of degree of nitration are discussed.

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