



SEC (Size Exclusion Chromatography)
correlation viscosity= f (DP)

INDEX

01

Introduction

02

Bergerac Type Viscosity

03

SEC Analysis

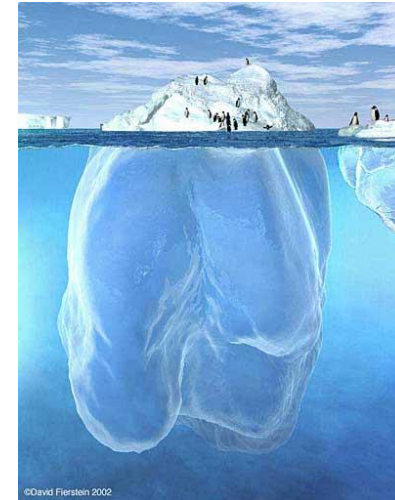
04

Correlation viscosity = f (DP from SEC)

Viscosity of Nitrocellulose is an important property for the processability of Nitrocellulose in propellant process

IT'S A CLASSIC SPECIFICATION

IT'S AN AVERAGE VALUE



What is the goal:

**To obtain distribution
curve rather than
average information**

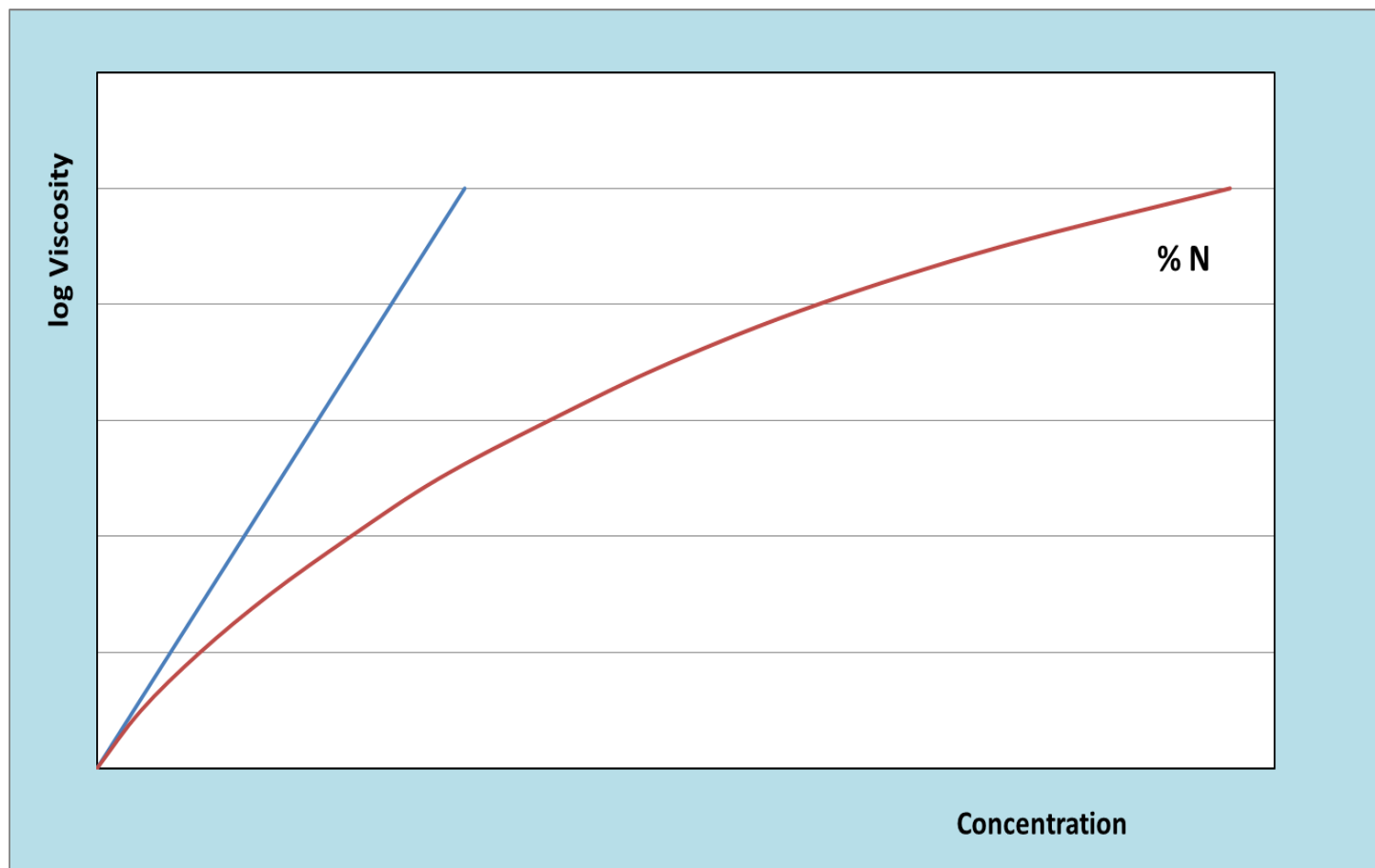
**To determine viscosity and
distribution of degree of
polymerization from the
same analysis**

**Establish correlation between viscosity and degree of
polymerization in order to obtain viscosity directly from SEC analysis**

**Bergerac type viscosity is existing and used since 1982
method developed by SNPE**

Used for viscosity determination of every batch of NCE even
if the final product is analyzed following customers viscosity

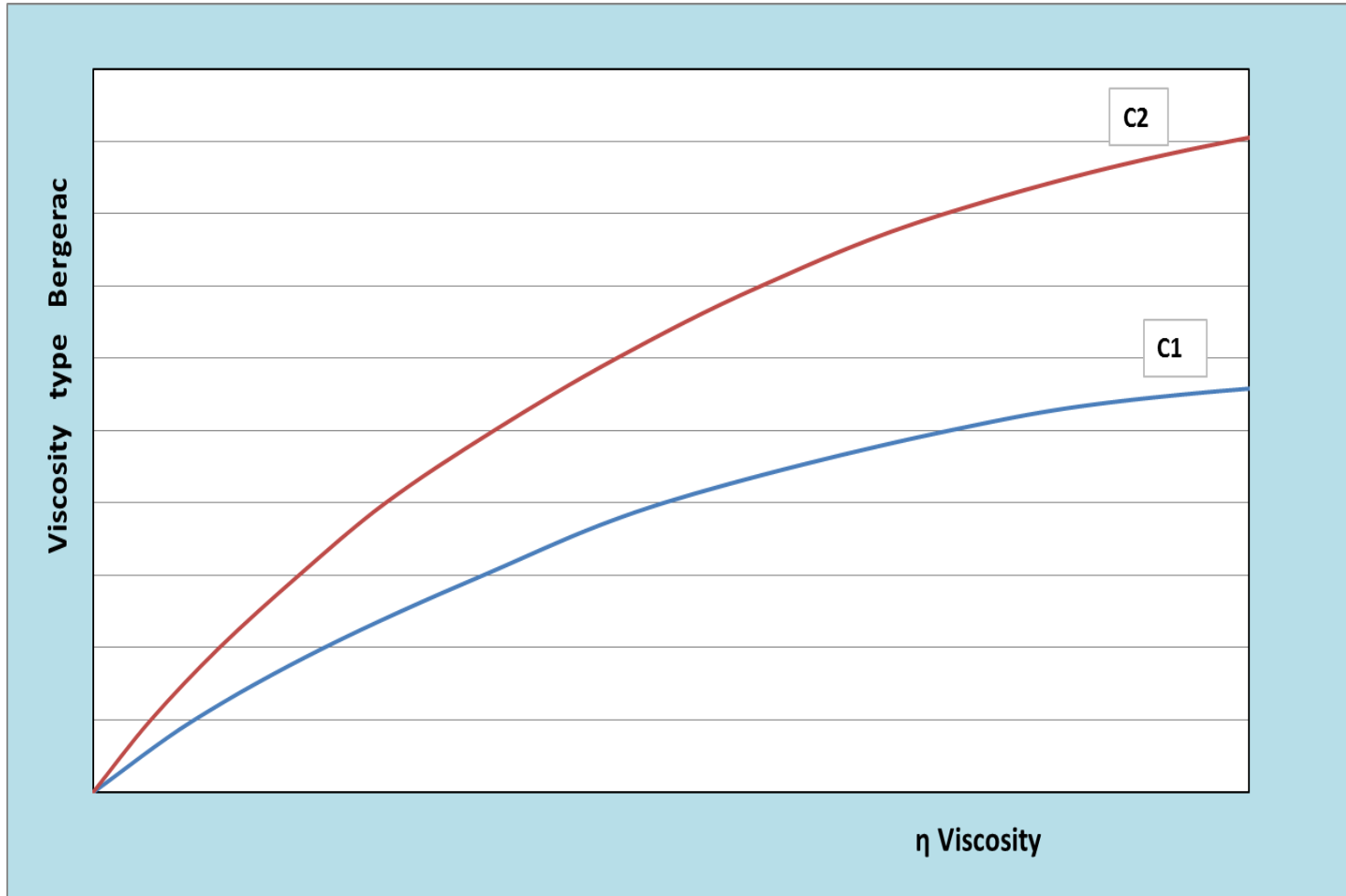
It's shorter than other analysis and [solution] is lower
Viscosity type: 8 h
other viscosity methods : up to 18 h



$$\text{Intrinsic Viscosity} = \lim_{c \rightarrow 0} \left(\frac{\text{specific viscosity}}{c} \right)$$

$$\text{Specific viscosity} = \frac{\text{solution viscosity} - \text{solvent viscosity}}{\text{solvent viscosity}}$$

* Solvent= butyl acetate



$$\text{Intrinsic Viscosity} = k [\text{DP}]^a$$

K, a depending of the polymer
and solvent

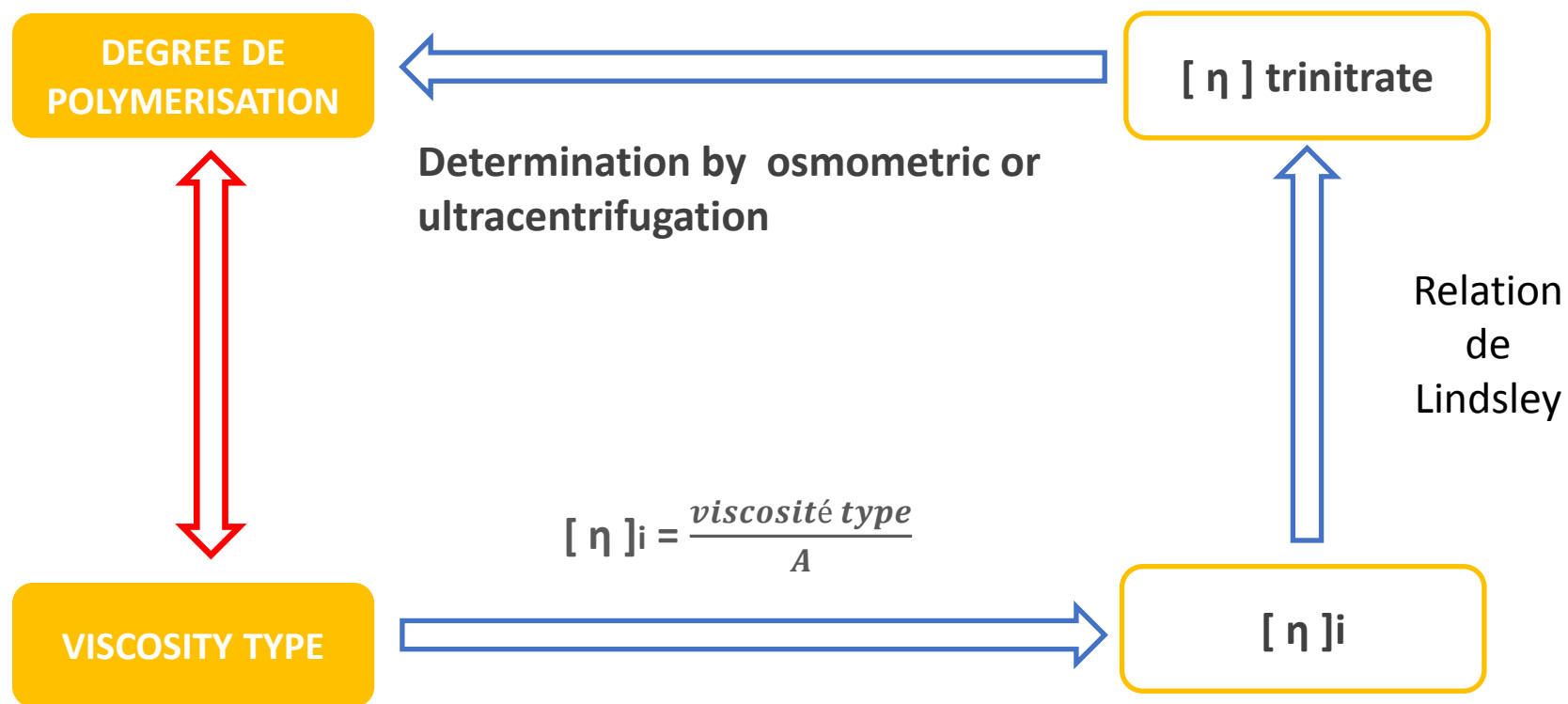
Empirical relation

$$\text{Intrinsic Viscosity} = 1/A \times \text{Type}$$

$$\rightarrow \text{Type viscosity} = f(\text{DP})$$

Solvent= butyl acetate

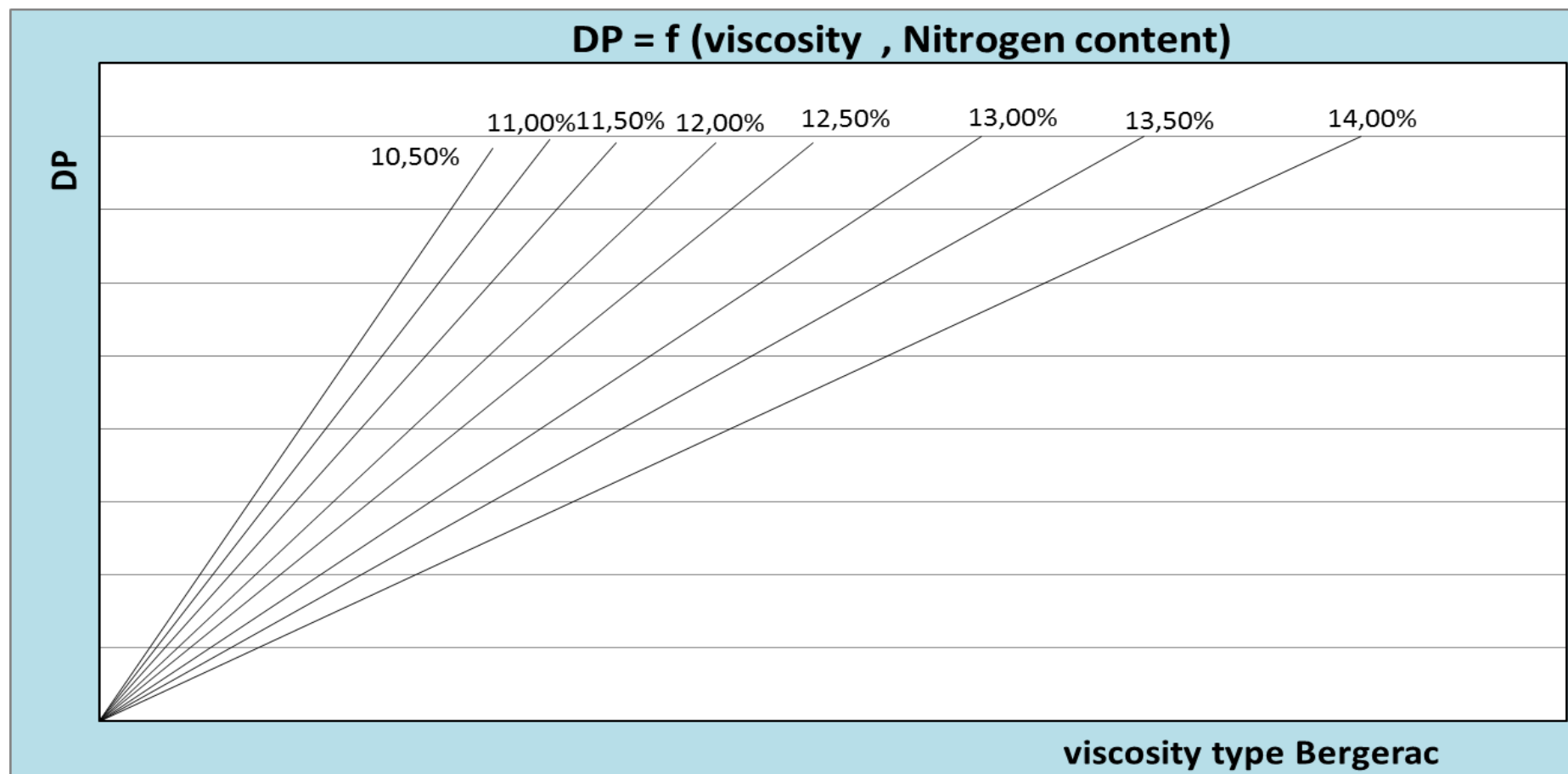
EMPIRICAL DETERMINATION OF CURVES (determined in the 80')



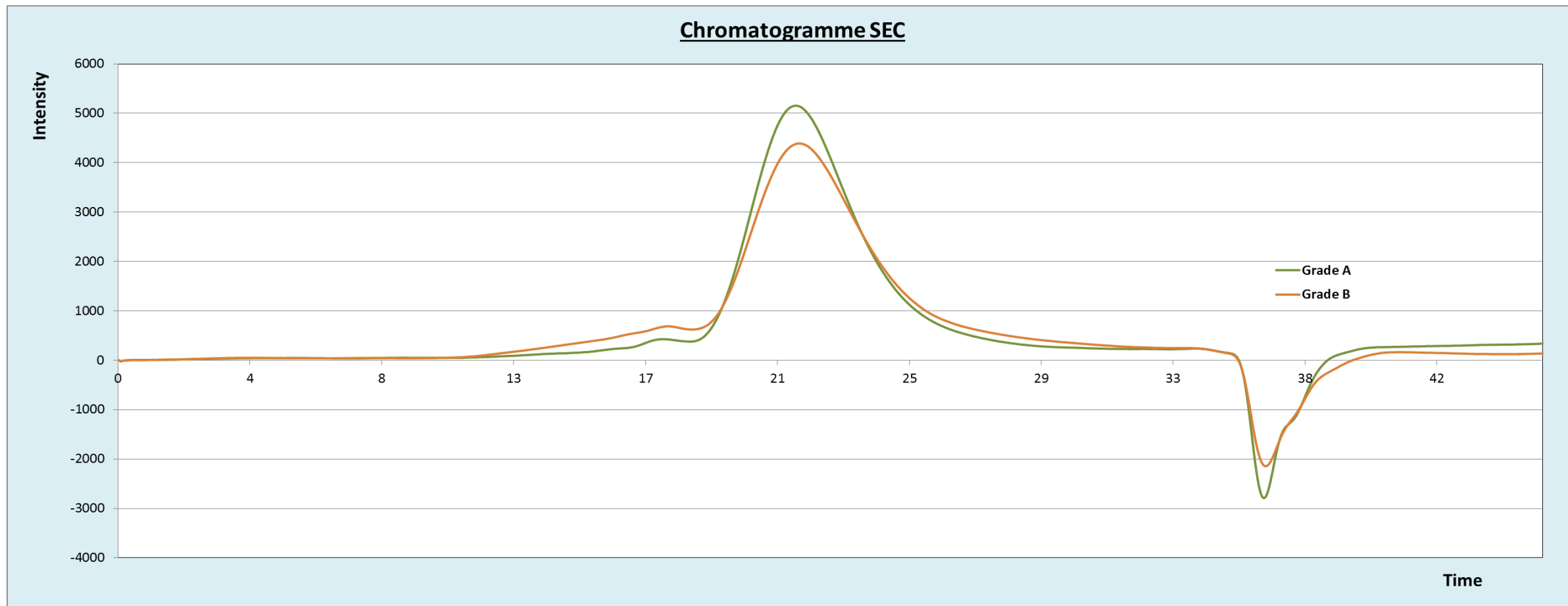
[η] : intrinsic viscosity of the same DP trinitrate

[η]_i : Viscosity of nitrate with Nitrogen content X

EMPIRICAL DETERMINATION OF CURVES (determined in the 80')

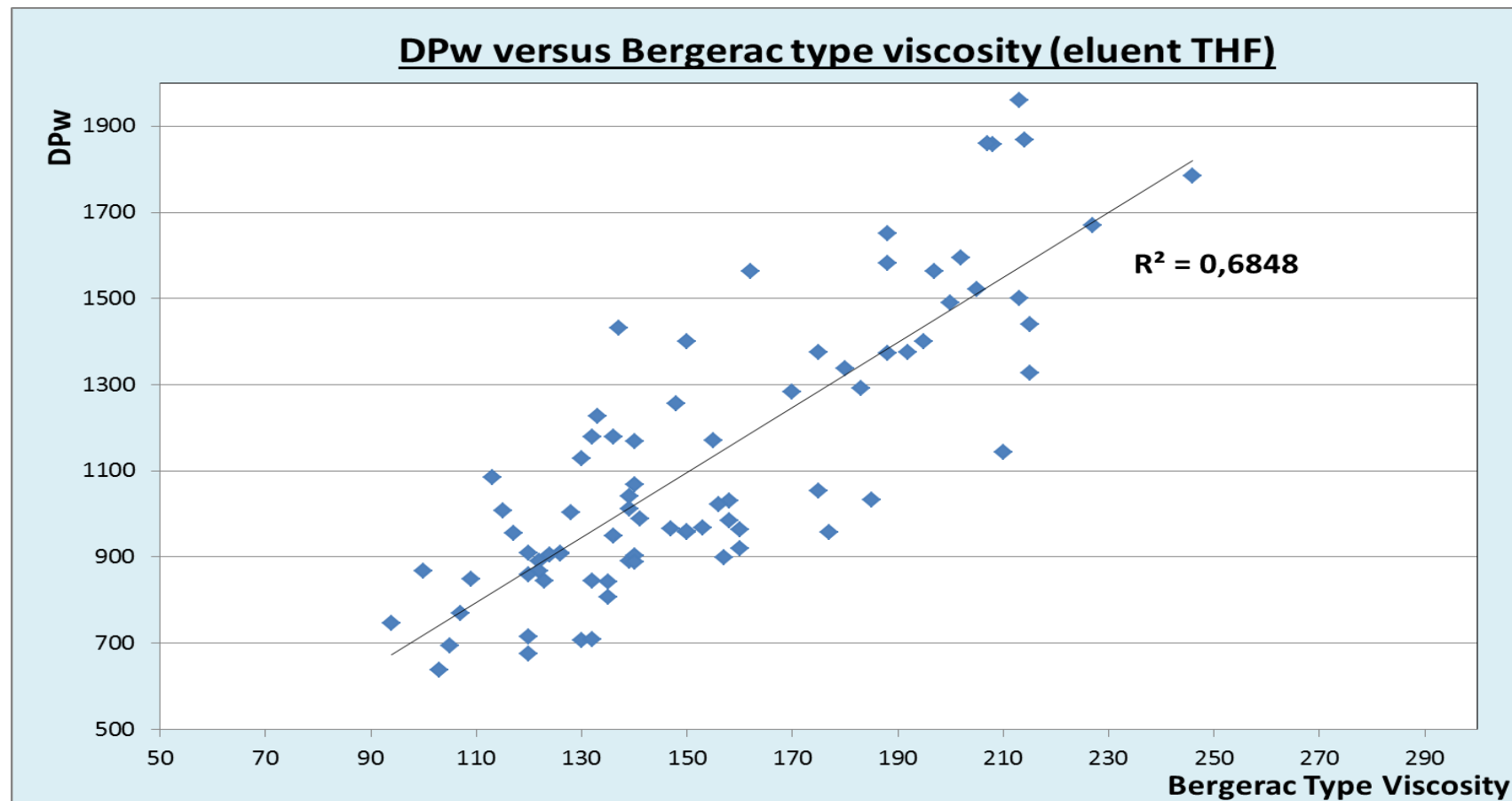


Condition: THF/ isocratic / 3 columns / [NCE]= 0,6 %
 Detector RID - External calibration with polystyrene standard



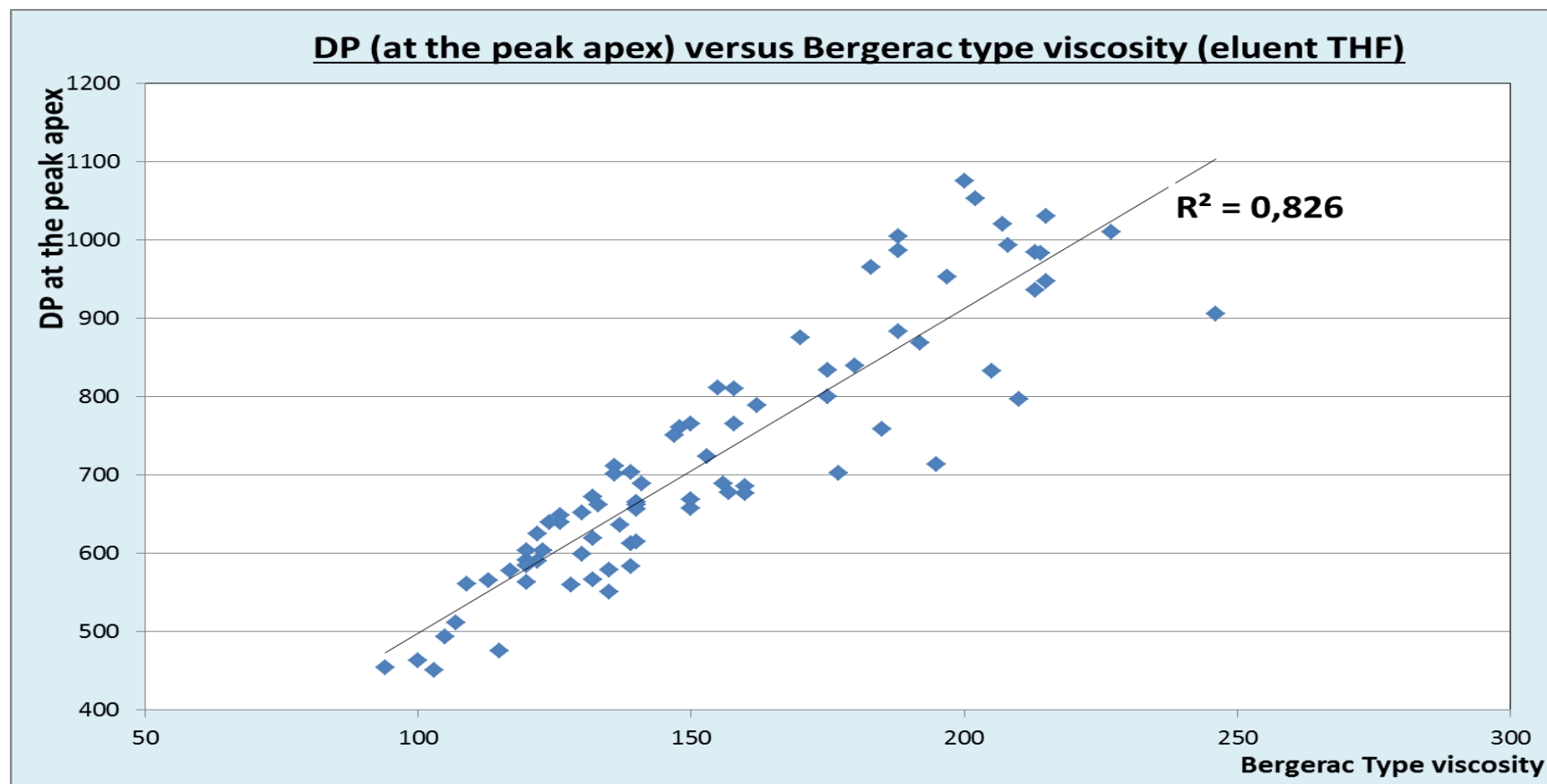
Condition: THF / isocratic / 3 columns / [NCE]= 0,6 %

Detector RID - External calibration with polystyrene standard



Condition: THF / isocratic / 3 columns / [NCE]= 0,6 %

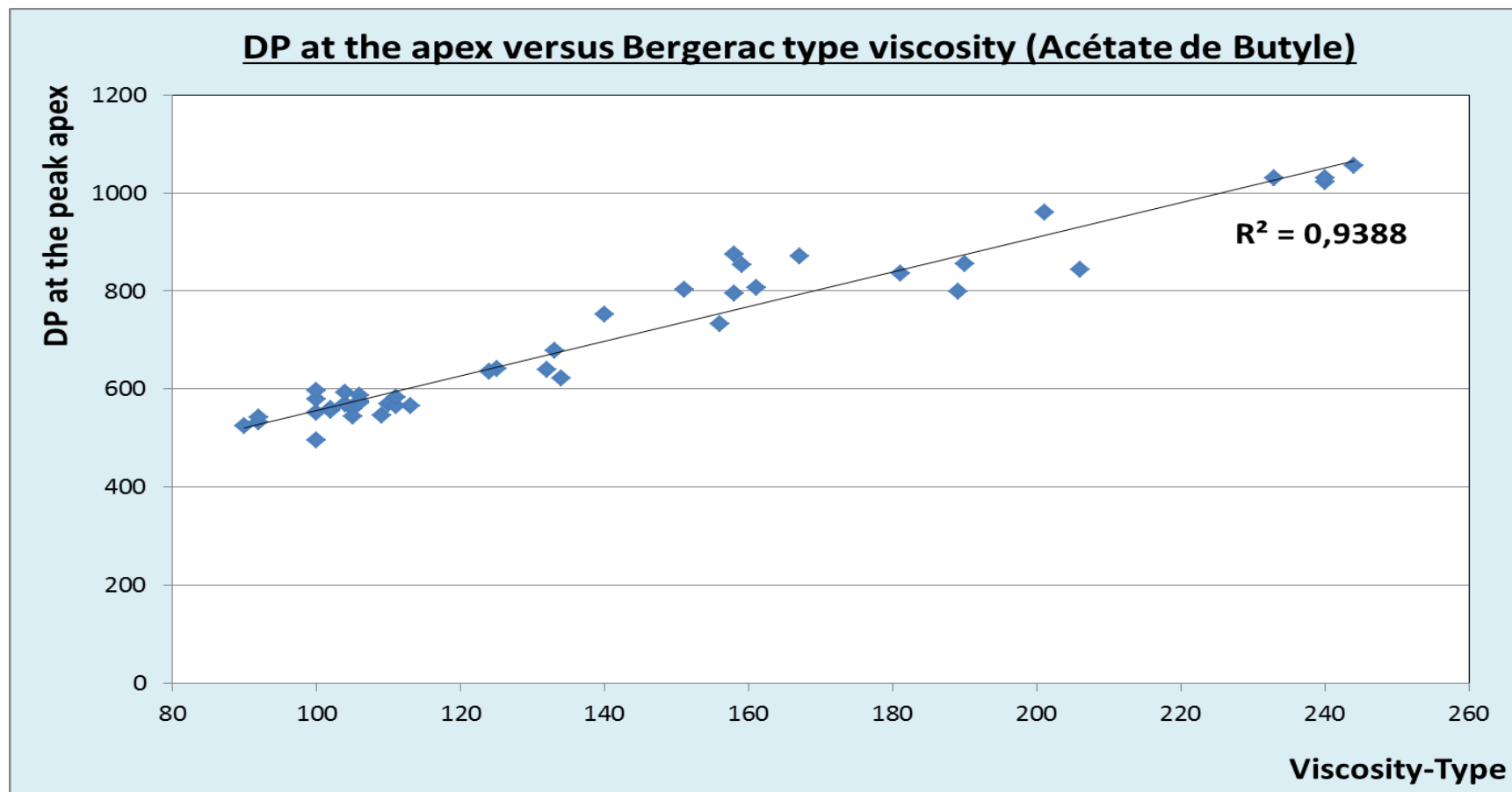
Detector RID - External calibration with polystyrene standard



Grade B

Condition: **butyl acetate** / isocratic / 3 columns / [NCE]= 0,6 %

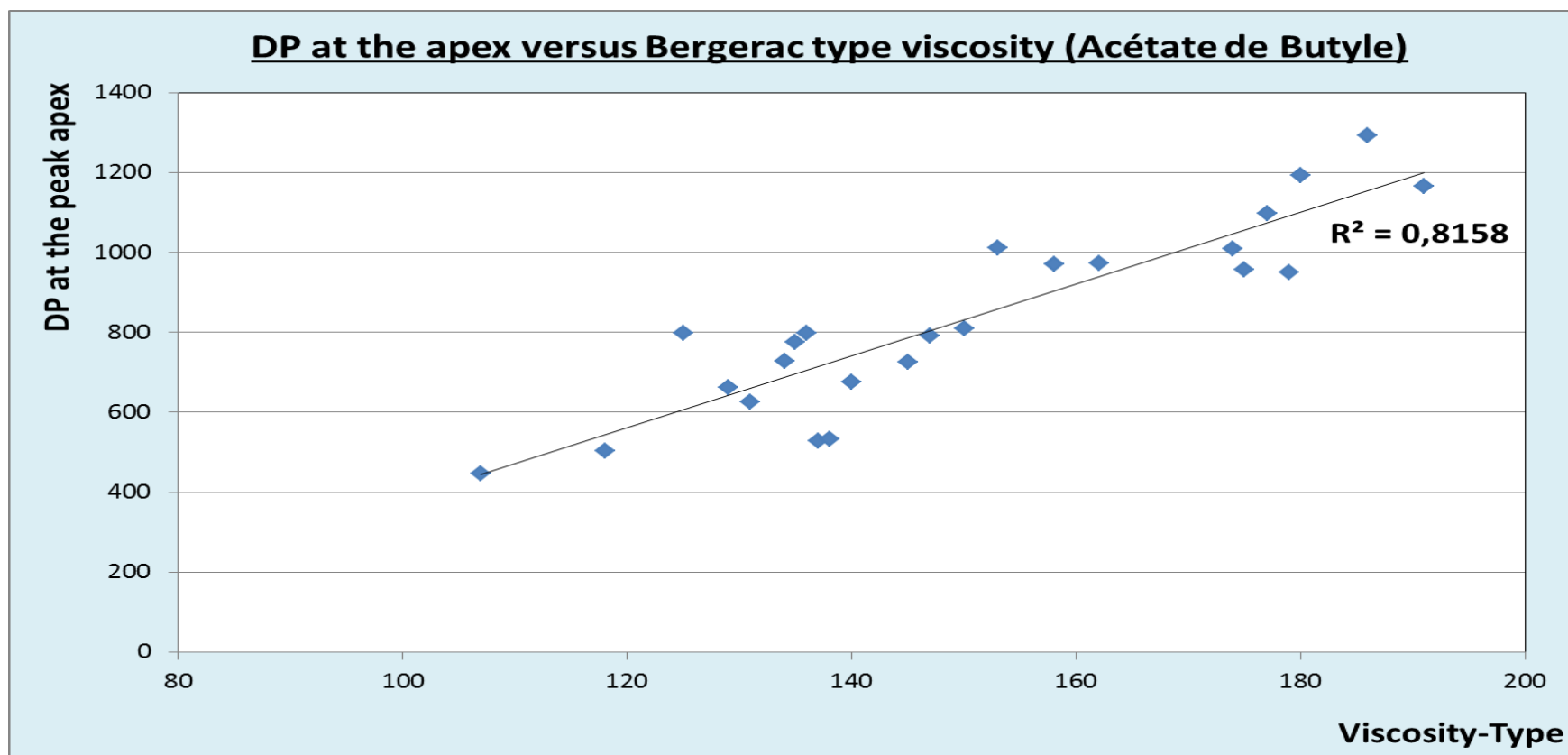
Detector RID - External calibration with polystyrene standard



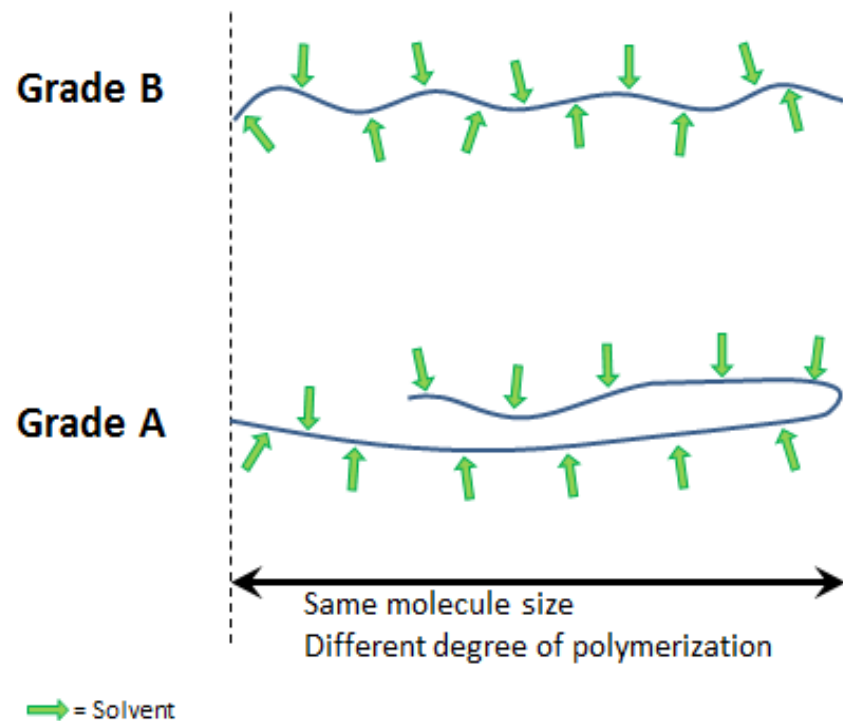
Grade B

Condition: butyl acetate / isocratic / 3 columns / [NCE]= 0,6 %

Detector RID - External calibration with polystyrene standard



Grade A



Better correlation with Grade B than Grade A

- Grade B ($N > 13,35 \%$) structure is more “rigid”
no influence of N content
- Grade A : impacted by N content

Hydrodynamic volume more affected by OH bond

- The solvent used for SEC change the DP obtained : there is no unique DP
- DP calculate from type viscosity Bergerac sounds better

Wood pulp cellulose used : DP=1000

Bergerac type viscosity	DP from SEC	DP calculated (from Bergerac type viscosity)
233	1031	653
190	855	548
181	837	509
169	797	483
156	732	447
133	678	383
124	636	352
111	583	327
102	559	289

Is there any interest to know the real DP ?

CONCLUSIONS

With other viscosity methods, the correlation is worst because of the higher concentration compare to Type viscosity.

We have not seen a better correlation when we select the type of cellulose used for Grade B.

-Grade B (N > 13,35 %) better correlation than grade A

- Grade B structure is more rigid (not impacted by N content)
- % N influence the hydrodynamic volume of the molecule and specially for Grade A
- More hydroxyl interaction between solvent and molecule which modify the “aspect” of the molecule
- Distribution of N in grade A could be affected by amorphous and crystalline region, internal/external fiber

CONCLUSIONS



Good correlation between Bergerac Type Viscosity and DP for grade B

-Good repeatability of the analysis

Additional studies have to be done for grade A:
Correlation with different nitrogen content

At the same time, from SEC Analysis we can determine:

- Distribution of molecular weight (more relevant information)
- **BERGERAC TYPE VISCOSITY** (and DP)

Quick analysis: 1 hour, instead of more than 8 hours

MANY THANKS FOR YOUR ATTENTION

Claude GUILLAUME
MANUCO
Technical Manager

c.guillaume@manuco-nc.com
+33 553636118
+33 673994542



