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PROJECT REPORT No. 240

EFFECTS OF VARIETY, SITE AND WEATHER ON WHEAT STARCH GELATINISATION TEMPERATURE; VALUE IN PREDICTING FLOUR PERFORMANCE

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EFFECTS OF VARIETY, SITE AND WEATHER ON WHEAT STARCH GELATINISATION TEMPERATURE; VALUE IN PREDICTING FLOUR PERFORMANCE

by

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ABSTRACT

WHEAT STARCH GELATINISATION TEMPERATURE

The project investigated the effect of variety, growth site and weather on starch in wheat from the 1999 harvest year. Starch viscosity was measured in dilute suspension by the Rapid Visco Analyser (RVA) and in simulated dough systems using a rheometer.

High viscosity is desirable in thickening applications, and, in certain papers, high gelatinisation temperature is correlated with greater loaf and cake volume.

This project has shown that weather information can be used with wheat variety and Hagberg Falling Number and protein content, to predict starch gelatinisation in the RVA (temperature and viscosity at defined stages during and after gelatinisation). It is unclear from these empirical models which weather/time period experienced by the wheat is critical. The weather measurements that appear as significant predictors are not the same as those reported in the literature from greenhouse studies, i.e. average or maximum temperatures. Hagberg Falling Number has a strong effect on gelatinisation, and, as expected from the action of alpha-amylase, it is inversely correlated to indices like Peak Viscosity, Trough Viscosity and Final Viscosity of flour slurries. There are significant varietal differences in gelatinisation temperature in flour slurries, with Soissons having a low gelatinisation temperature and Eclipse a high gelatinisation temperature. In a single site comparison, late drilling is associated with higher paste viscosity.

The study of wheat flour doughs yielded poorer predictive models. Biscuit wheats gave doughs with lowest minimum stiffness during the test. The stiffness of the dough at 90°C increased with Hagberg Falling Number. Both models could be improved by the inclusion of one, or more weather variables. Varietal differences in gelatinisation temperature are less marked in dough. Soissons has a low gelatinisation temperature and Hereward the highest gelatinisation temperature.

A predictive model for Hagberg Falling Number was identified which used a restricted set of weather variables; the value of this should be assessed by experts.

The results show that there are factors other than Falling Number which affect starch gelatinisation, and the rheological properties of slurries and doughs at high temperature. Further studies are required to establish whether models can be derived that are robust to seasonal and harvest date fluctuations.

The benefits of this preliminary study are not easy to quantify, however, it may be possible in the future to advise growers on the variety to sow, date of sowing and aspects of crop husbandry to give specific starch pasting properties.

SUMMARY

Background

The project is a preliminary investigation of the effect of wheat variety, growth site and weather on the properties of starch in flour. It is limited to samples of wheat from the 1999 harvest year.

There is a considerable body of information on the role of the wheat proteins in the performance of wheat flour in baked product applications. The role of starch has however been less thoroughly investigated even though it represents more than 80% of the dry solid of white flour. There is an indication from carefully controlled studies that climatic conditions during growth affect starch gelatinisation properties. There is no published information on the variability of starch properties in common UK wheat varieties grown in the field and one objective of this work is to obtain this information.

Wheat flour is used in the manufacture of a very wide range of foods, not only bread and cake but also biscuits, pastries, crêpes and pancakes and local specialities like Yorkshire Puddings and dumplings. It is also an ingredient in batter mixes for coating fish, meat and vegetables. Starch extracted from wheat flour is used in various products where there is no benefit from the presence of wheat protein. All of these products flow or expand during baking or cooking. This change is fundamental to the final product size and shape and to its eating texture. The event that causes the shape of the product to become fixed in size and shape is the gelatinisation of starch in the flour. There are indications that the higher the gelatinisation temperature in bread and cake, the greater the expansion of the product before it sets.

Wheat flour is also used as a thickener in soups and sauces where is confers viscosity and opacity to the product. In such applications, the user is generally seeking the highest viscosity from a given weight of flour.

Materials and Methods

Wheat samples were drawn from 16 UK trial sites (1999 crop year) and covered 7 varieties. Weather data for the 9 weeks preceding harvest was gathered. The wheat was cleaned and milled on a Quadrumat mill. Starch properties were measured in dilute flour suspensions by the Rapid Visco Analyser (RVA) and in simulated dough systems using a rheometer. These instruments allow the viscosity or stiffness of the sample to be measured continuously as a function of sample temperature over a temperature cycle from 50°C to 95°C and back to 50°C. They thus mimic thickening and baking applications of flour. These measurements of starch properties are affected by the presence of alpha-amylase. Because the 1999 crop year showed large variations in alpha-amylase activity in flour, the RVA test was also carried out in the presence of silver nitrate to deactivate the enzyme.

A statistical analysis was carried out to seek predictive models for starch gelatinisation properties, using the input variables of variety, weather and grain quality parameters like protein content and Hagberg Falling Number.

Weather information (on the day of harvest and weekly averages back to anthesis) can be used with wheat variety and commonly available intake measurements (Hagberg Falling Number and Protein), to predict rheological properties related to starch gelatinisation in the RVA (temperature of gelatinisation, viscosity at defined stages during and after gelatinisation). It is unclear from these empirical models which weather/time period or periods experienced by the wheat are critical in affecting gelatinisation. The weather measurements that appear in the "best fit" models as significant predictors are, however, not the same as reported in controlled studies in the literature, i.e. average or maximum temperatures.

Results and Discussion

Hagberg Falling Number has a strong effect on the gelatinisation events, and as expected from understanding of the action of alpha-amylase, Hagberg Falling Number is inversely correlated to viscometric indices like Peak Viscosity, Trough Viscosity and Final Viscosity of flour slurries. Inactivation of alpha amylase by silver nitrate reveals an effect of variety on the gelatinisation process.

A comparison of varieties shows significant differences in gelatinisation temperature in flour slurries, with Soissons having a characteristically low gelatinisation temperature and Eclipse a high gelatinisation temperature. This holds both in the presence and absence of alpha-amylase activity.

On a single site comparison, late drilling is associated with higher paste viscosities in RVA tests both without and with silver nitrate.

The study of wheat flour doughs in the rheometer gave results which yielded poorer predictive models. The biscuit wheats gave doughs with the lowest minimum stiffness during the test. The stiffness of the dough at 90°C increased with Hagberg Falling Number. Both of these models could be improved by the inclusion of one, or more weather variables.

In simulated dough systems, the differences in gelatinisation temperature are less marked, but again Soissons has a low gelatinisation temperature with Hereward the variety with the highest gelatinisation temperature within the sample set.

There is no correlation between gelatinisation properties in dilute slurries and in simulated dough systems.

Although it was not a stated objective of this project, the dataset was analysed to seek a predictive model for Hagberg Falling Number. The model derived gives a useful prediction of Hagberg Falling Number when tested on the validation set. It is different from other models in that the weather terms it includes all relate to weeks 4,5 and 6 before harvest, i.e. the model is using weather data from a reasonably limited part of the growth phase. The relevance of this model, and its relationship to past work is not known, but should be reviewed.

Conclusions and Potential Benefits to the Industry

The results reported in the literature showing an effect of growth temperature on gelatinisation temperature in wheats cultivated in greenhouse conditions could not be observed on field grown wheat.

Our findings show that there are factors other than Falling Number which affect starch gelatinisation, and through this, the rheological properties of flour slurries and doughs at high temperature. There is a small amount of published information showing a relationship between starch gelatinisation and finished product quality, and this area might prove fruitful for future research.

Growers can control the variety sown, date of sowing and aspects of crop husbandry such as nitrogen application and pesticide treatment. Our preliminary results hint at varieties, which are likely to give specific starch pasting properties (e.g. Eclipse has a high gelatinisation temperature in aqueous slurries). On a single site comparison there is evidence of a strong sowing date or maturity effect. These results need to be confirmed in future crop years to allow confidence in them to be built and a realistic estimate of their value to be made.

Breeders require information on the climatic effects on starch pasting so that they can tailor their new varieties to express certain growth characteristics at specific times. The weather models given here for starch properties have not identified a specific period, however, if there is a predictable effect of weather at a particular growth stage, this should emerge if the work is extended to cover future harvests.

The economic benefit of a specific starch gelatinisation temperature or high starch viscosity is difficult to estimate. The identification of strong varietal influences on starch gelatinisation in this work allows targeted application tests on flours of different starch behaviour to be undertaken in the future. From this, a realistic assessment of their benefit to the manufacturing industry could be derived.

Recommendations for Further Work

A number of recommendations are made for further work including:

- Check validity and robustness of models (cf weather and site) over future harvests
- Establish specific weather variables for starch behaviour prediction
- Extend sample set to other varieties
- Investigate relationship between Hagberg Falling Number and starch behaviour for a sample set in which HFN has been controlled in specific ways to high or low values. Relate to HGCA Hagberg Falling Number prediction.
- Investigate effect of level of disease on starch gelatinisation parameters
- Investigate in detail effect of sowing date on starch properties
- Investigate effect of maturity at harvest on starch properties

TECHNICAL DETAILS

Introduction

Aims

There is a considerable body of information on the role of the wheat proteins in the performance of wheat flour in baked product applications. The role of starch has been less thoroughly investigated even though it represents more than 80% of the dry solid of white flour. There is no published information on the variability of starch properties in common UK wheat varieties, and one objective of this work is to obtain this information.

There is an indication from carefully controlled studies that climatic conditions during growth affect starch gelatinisation properties. It is not known to what extent this effect can be observed in wheat grown in field plots.

The overall remit of this project is thus to investigate the effect of variety, growth site and weather on the properties of starch in wheat flour (as measured by viscosity development). The study is limited to wheat from the 1999 harvest year. The project is a preliminary investigation, and will indicate the range of gelatinisation properties which can arise and identify whether there is a need for more detailed studies. Data gathered would be used to predict the end use suitability of specific varieties and to identify whether there may be opportunities to grow specific varieties in specific regions to meet the quality criteria for niche markets.

Background

Properties of Starch

Starch is the semi-crystalline carbohydrate polymer that wheat lays down as an energy reserve for the germinating plant. It takes the form of bodies, termed starch granules falling in 2 distinct size ranges; A starch, large lenticular granules 15µm to 30µm in diameter and B starch, small spherical granules 1µm to 10µm in diameter[1]. When viewed under crossed polarising filters in a light microscope, a characteristic "Maltese cross" pattern is seen, a consequence of the partially crystalline arrangement of molecules in the granule.

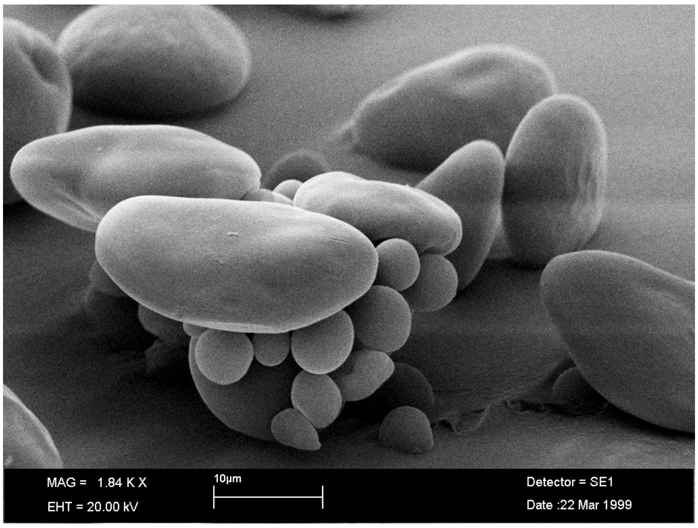

Figure 1 Electron micrograph of A and B starch granules

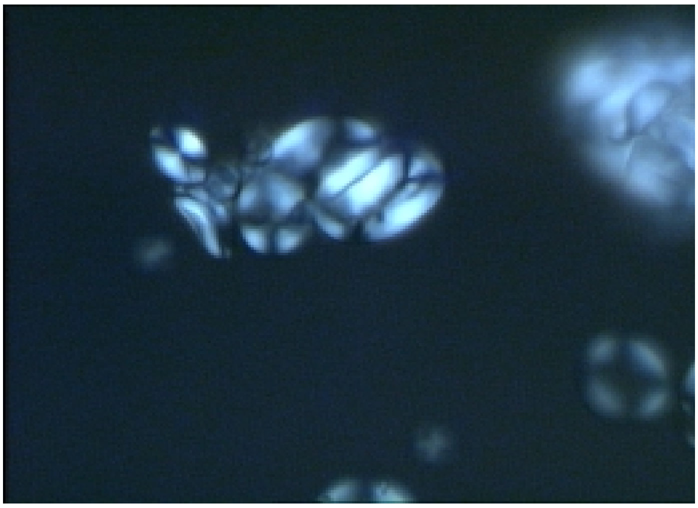

Figure 2 Wheat starch under crossed polarising filters showing Maltese cross

In the presence of cold water, the starch granules swell slightly. If the granules are heated in water, an irreversible loss of crystallinity occurs at a characteristic temperature, known as the gelatinisation temperature. This is followed by extensive swelling of the granules. There are many methods available to measure these changes, including optical, calorimetric and viscometric methods, and each records a slightly different aspect of the gelatinisation process. When trying to understand the performance of starch in a food application, the viscometric methods are preferred as these come closest to simulating the role of starch in the food product. The gelatinisation event depends upon the water content of the system. As water content falls below about 35%, gelatinisation temperature rises[2]. The extent of swelling also depends upon water availability. Maximum swelling is only achieved in very dilute starch preparations, a condition which is seldom seen in real food applications.

After gelatinisation, the starch continues to swell and the viscosity increases until some point at which swelling is at a maximum and further heating results in viscosity loss due to thermal effects. If gelatinised starch is cooled, the viscosity is seen to increase as molecular motion slows and polymer entanglements develop. If a significant amount of shear is applied to the gelatinised starch, this can cause a further level of molecular breakdown and viscosity loss.

The gelatinisation event is also influenced by the presence of soluble compounds (salt, sugar etc) in the food matrix[3], but these are not considered this report.

Effect of Growth Conditions and Variety on Starch Gelatinisation

There is evidence from studies in North America[4] that the temperature at which wheat starch gelatinises depends upon temperature of growth of the plant and upon the wheat variety in question.

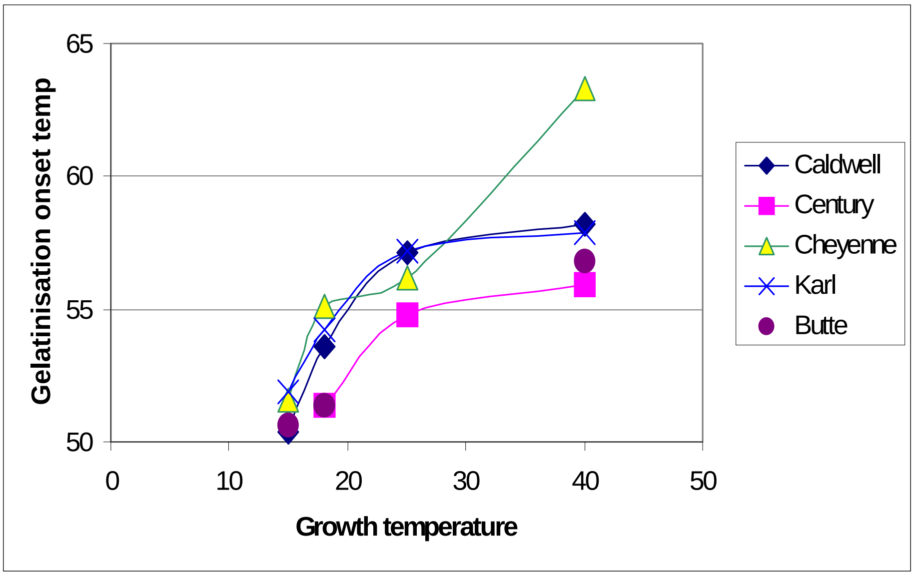

Figure 3 Effect of Growth Temperature on Starch Gelatinisation after Shi *et al*[4]

The effect of variety on gelatinisation temperature has also been investigated for Swedish wheats using differential scanning calorimetry (DSC)[5]. The authors concluded that the relations between end-use quality and DSC parameters could be expected to explain differences between varieties only in special cases.

The viscosity of wheat starch after gelatinisation is dependent upon alpha-amylase activity. The correlation between alpha-amylase activity and pasting viscosity is reported to be poor because it ignores the effect of variety and agronomy on starch properties[6].

There is no published information on starch gelatinisation temperature and viscosity for UK grown wheats.

The Role of Starch in Wheat Flour Performance

Wheat flour is used in the manufacture of a very wide range of foods, not only bread and cake but also biscuits, pastries, crepes and pancakes and local specialities like Yorkshire Puddings and dumplings. It is also an ingredient in batter mixes for coating fish, meat and vegetables. Starch extracted from wheat flour is used in various products where there is no benefit from the presence of wheat protein.

All of these products flow or expand during baking or cooking. This change is fundamental to the final product size and shape and to its eating texture. The event that causes the shape of the product to become fixed is the gelatinisation of starch in the flour[5, 7, 8, 9]. The higher the gelatinisation temperature, the greater the expansion of the product before it sets. This is reported in the literature for bread and cakes, but there is little scientific research on batter products and no discussion of the factors controlling structure development.

The effect of starch gelatinisation temperature on the specific volume of test baked bread has been investigated on a set wheat starch samples baked with a single batch of gluten[9] . Loaf specific volume increased with gelatinisation temperature.

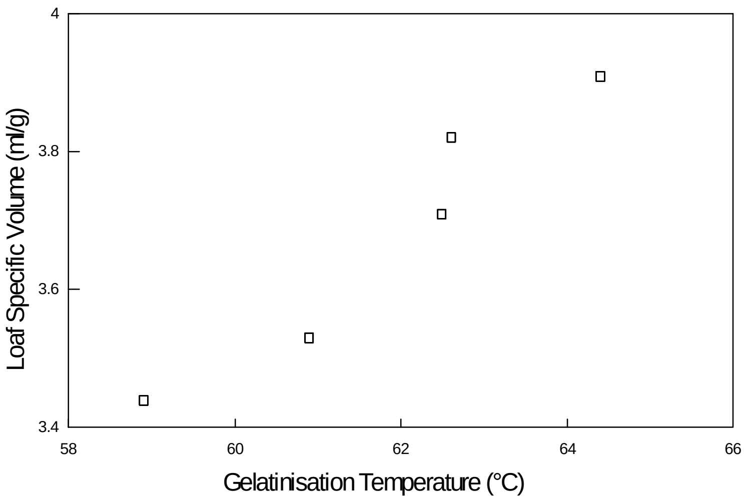

Figure 4 Effect of Gelatinisation Temperature on Loaf Volume after Soulaka and Morrison[9]

As loaf size increases, so the consumer's perception of value rises, but the baker must maintain control of size to ensure that sliced bread will fit into a bag of predefined dimensions.

Characteristics of the 1998/9 Trials Season

Wet weather during the autumn of 1998 delayed sowing considerably, especially in the north and west. The difficult conditions continues into spring and spray and fertiliser operations were impeded as well as the drilling of some spring sown trials. The quality of harvested grain was also seriously affected by heavy rainfall. Sprouting in the ear was observed at several sites and Hagberg Falling Number values depressed to levels at which they were unsuitable for bread making quality assessment. Samples harvested before this rainfall generally had high Hagberg Falling Numbers, but a delay in harvesting resulted, on certain sites, in a crop of very low Hagberg Falling Number.

Materials and Methods

Test milling

After hand cleaning, the grain was test milled on a Buhler Quadrumat unit (for details see Appendix A).

Measurements of starch gelatinisation

Traditionally, the Brabender Amylograph has been used in the laboratory to measure the development of viscosity during gelatinisation (pasting) of starch. This instrument heats a slurry of controlled solids content at a defined rate to 95°C while stirring. It then holds the pasted slurry at 95°C before cooling to 50°C. The output is a record of the force transmitted via the slurry to a recording “paddle” immersed in the slurry. The Newport Instruments Rapid Visco Analyser (RVA) carries out a similar test, but uses a smaller sample size and a shorter test cycle (see Appendix B for details). The RVA makes an electronic record of torque required to maintain a constant stirring speed, and this facilitates storage and handling of data from the test. For these reasons, we have chosen to use the RVA in our studies.

Several parameters can be extracted from the test as is shown in the figure and table below:

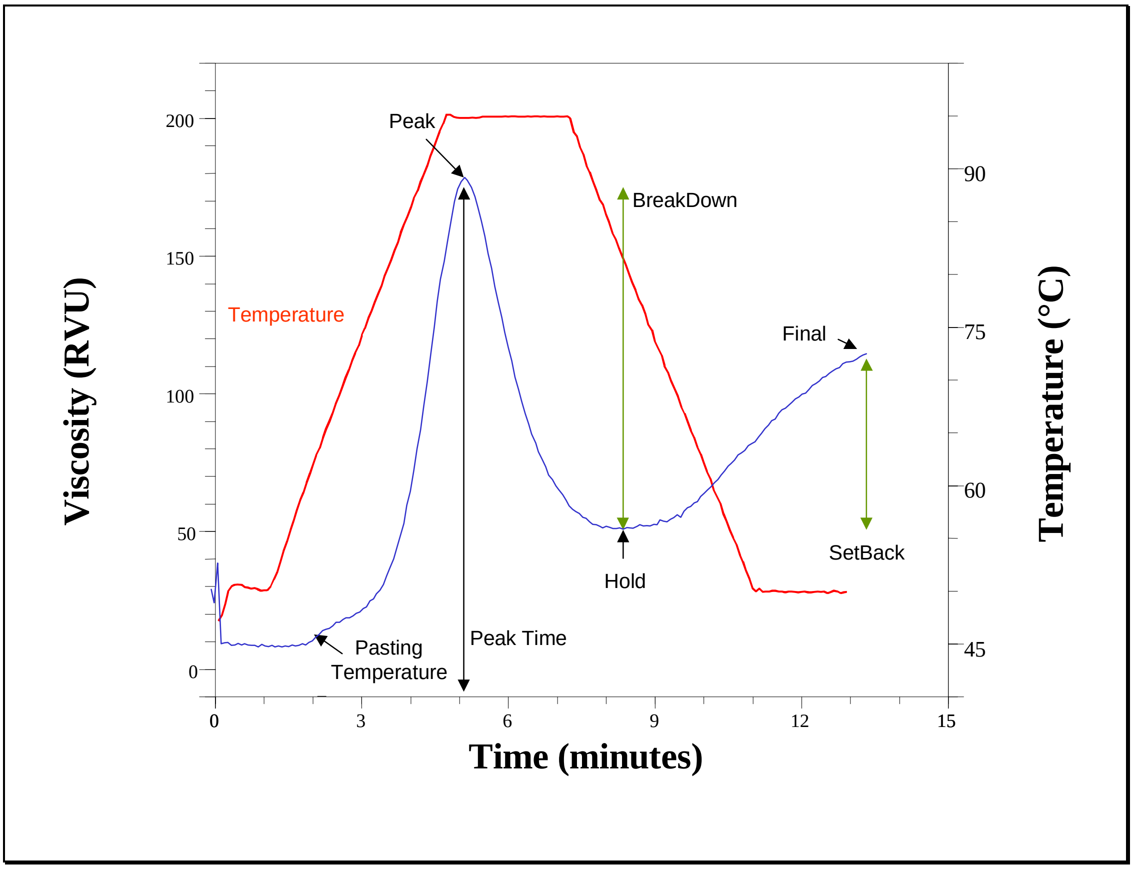

Figure 5 Typical RVA test and measurements from it

Parameter	Abbreviation	Abbreviation (with silver nitrate)
RVA Peak Viscosity	P_1	Ag_P_1
RVA Trough Viscosity	T_1	Ag_T_1
RVA Viscosity Breakdown	Break	Ag_Break
RVA Final Viscosity	Fin_V	Ag_Fin_V
RVA Viscosity Setback	Setba	Ag_Setba
RVA Time at Peak	Pk_T	Ag_Pk_T
RVA Pasting Temperature	PST	Ag_PST

The lowest water content that can be used in an RVA test is limited by the power of the instrument. It cannot measure materials like bread dough. Because gelatinisation depends upon water content, it is necessary to examine systems at dough moisture to get a fuller picture of starch performance in a realistic range of wheat flour applications. To measure dough systems, a research rheometer, such as the TA Rheometer AR 1000 has to be used. This instrument utilises a sample prepared in a separate mixer and measures the force required to maintain a defined oscillational motion applied to a thin layer of dough. The magnitude of the oscillation is chosen to avoid breaking any gel structure that might be formed during the heating and cooling cycle. This test thus applies minimal disturbance to the sample. Full details of the method are given in Appendix C. A typical rheometer measurement is shown in the figure below along with parameters extracted from it.

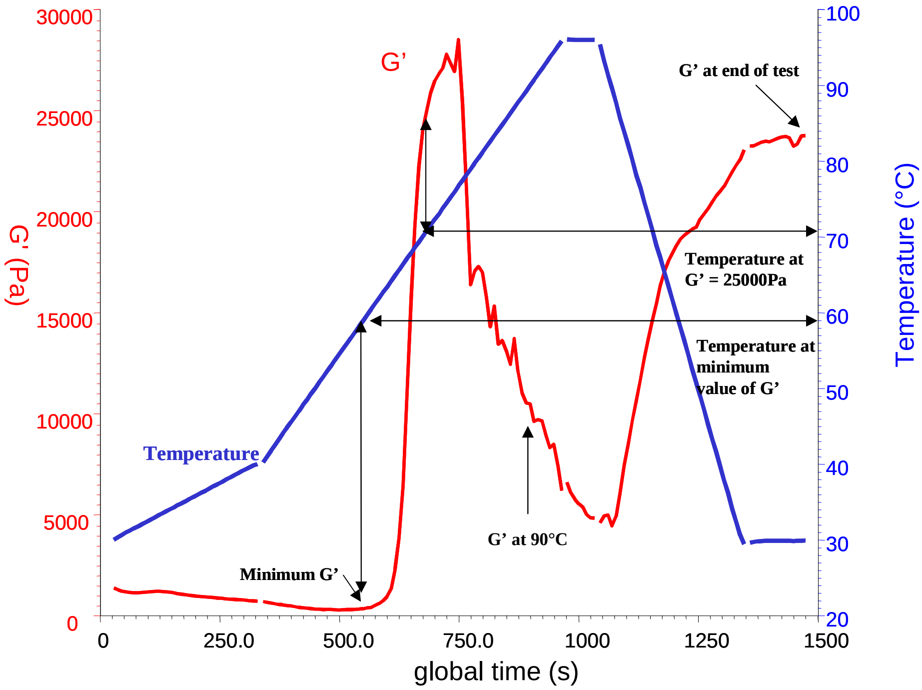

Figure 6 Rheometer Test

Parameter	Abbreviation
Temperature at which G'= 25,000Pa	T25000
Minimum value of G'	Min_G
Temperature at minimum value of G'	V_Min_G
G' at 90°C	G_at_90
G' at end of test	G_at_ET

The viscosity of a flour slurry or dough depends not only on the inherent properties of the starch, but also on the presence and activity of amylases, notably alpha-amylase. Alpha-amylase breaks the starch molecule in random positions. If alpha-amylase attacks the starch chain in a central position, molecular size is dramatically reduced and this causes a fall in slurry viscosity. The Hagberg Falling Number test is routinely used to estimate alpha-amylase activity of grain and of flour. A low Falling Number signifies a flour of high alpha-amylase activity. In the 1999 crop year, the harvest contained samples ranging from the minimum Falling Number (62s) to values typical of a normal UK wheat crop (>250s). The results and interpretation of our survey were expected to depend strongly on alpha-amylase activity, and so it was decided to run all RVA measurements in 2 ways:

1. Using the flour "as is" to include the effect of alpha-amylase
2. Using silver nitrate in the flour slurry to inactivate alpha-amylase

Silver nitrate inactivates alpha-amylase and allows a measurement of the viscosity potential of starch to be made, i.e. the viscosity that the starch would develop in the absence of alpha-amylase.

Other Wheat and Flour Testing Methods

Moisture content (CCFRA - Manual of Methods for Wheat and Flour Testing, Second Edition April 1997, Revised 1999, FTWG Method No. 0008), protein content (latest HGCA Guidelines) and Hagberg Falling Number (ISO/CD 3093) were determined on the flour by RHM Technology . Wheat analyses were provided by the sample providers (NIAB or New Farm Crops). NIAB used the same analytical methods as RHM Technology, but New Farm Crops used an NIR method calibrated against standard samples.

Interpretation of Measurements of Starch Gelatinisation

The measurements of flour viscosity in the Rapid Visco Analyser and of dough stiffness in the rheometer were chosen to simulate the use of flour in a high water environment and a reduced water environment respectively. As discussed earlier, there is limited published work on starch gelatinisation and its effect on final product size and shape. The parameters highlighted in the following paragraphs are based on a consideration of the role of starch in different types of product.

Wheat flour may be used as a thickener in soups and sauces where it provides viscosity along with a whitening effect. In comparison with refined starches, wheat flour provides a lower viscosity on an equal usage basis, but it adds opacity and whiteness, which are desirable in certain products. For application in "instant" products (dry products reconstituted with boiling water) the ideal thickener has a low gelatinisation temperature combined with a high viscosity. Flours sold for thickening applications in the UK[10] are sold on the basis of Brabender Amylograph viscosity, which is a measurement of pasting viscosity using a relatively large sample of flour slurry.

The RVA parameters of particular importance are likely to be:

RVA Parameter	Requirement for instant products
Pasting temperature	Low = early onset of thickening
Peak viscosity	High = high viscosity per gram used

Wheat flour has a more complex role to play in products prepared from batter. Products in this category include hot plate goods (pancakes, crumpets, crêpes, etc) and Yorkshire Puddings. In these products, the batter loses viscosity as it is heated from preparation temperature towards setting temperature, but the batter undergoes a rapid rise in viscosity as the starch gelatinises. This viscosity rise usually stops the flow and expansion of the batter. A high gelatinisation temperature will allow greater flow and expansion, which may be of benefit, but excess expansion can lead to a loss of control over the size and shape of the product. The manufacturer will probably have other components in the recipe that affect gelatinisation temperature, and so will seek a flour that is of consistent performance. After gelatinisation, the starch provides the mechanical strength that supports the product and gives it a characteristic bubble structure. Lack of viscosity at high temperature could result in insufficient strength and collapse of the product, either when hot, or as it cools.

RVA Parameter	Requirement for batter products
Pasting temperature	Controlled = reproducible flow and expansion
Peak viscosity	High = structure has strength at high temperature
Trough viscosity	High = structure has strength as it cools

The tests carried out on the rheometer were on a full recipe bread dough, including yeast and commercial improver. The temperature profile simulates baking and the early stages of cooling of bread. In practice, rate of heating during baking is generally slower with rolls having a baking time of 7 – 10 minutes, 800g bread 25 – 35 minutes and specialities like batch bread extended bakes of up to 2 hours. As the dough is heated, its stiffness falls, reaching a minimum around 55°C. Further heating leads to gelatinisation of starch and this signals the end of the expansion of the dough[7, 9]. We have chosen the temperature at which G' = 25,000Pa as a marker of the gelatinisation event. The stiffness of the dough after gelatinisation is mostly due to the strength of the starch phase, and it is the swollen starch that provides rigidity as the product is cooled.

Rheometer parameter	Requirement for dough based products
Temperature at G' = 25000Pa	High value preferred for maximum expansion
G' at 90°C	High value required for stability
G' at end of test	High value indicates resistance to collapse

Samples Investigated

A total of 104 samples were tested. These were chosen from a NIAB sample set and from New Farm Crops trial plots for which weather data were available from weather the Novartis weather system. One criterion for the selection of a site was that the trial plots yielded a reasonable crop. The samples can be categorised in various ways:

Variety	Earliness of Ripening	Hard/soft	NABIM Group	Rye chromosome
Soissons	9	Hard	Breadmaking (Group 2)	Negative
Savannah	6	Hard	Feed (Group 4)	Positive
Riband	7	Soft	Biscuit (Group 3)	Negative
Hereward	7	Hard	Breadmaking (Group 1)	Negative
Equinox	7	Hard	Feed (Group 4)	Positive
Eclipse	7	Soft	Biscuit (Group 3)	Negative
Charger	8	Hard	Breadmaking (Group 2)	Negative

The intention was to use sites where samples of each of the chosen varieties could be provided, and this was largely successful.

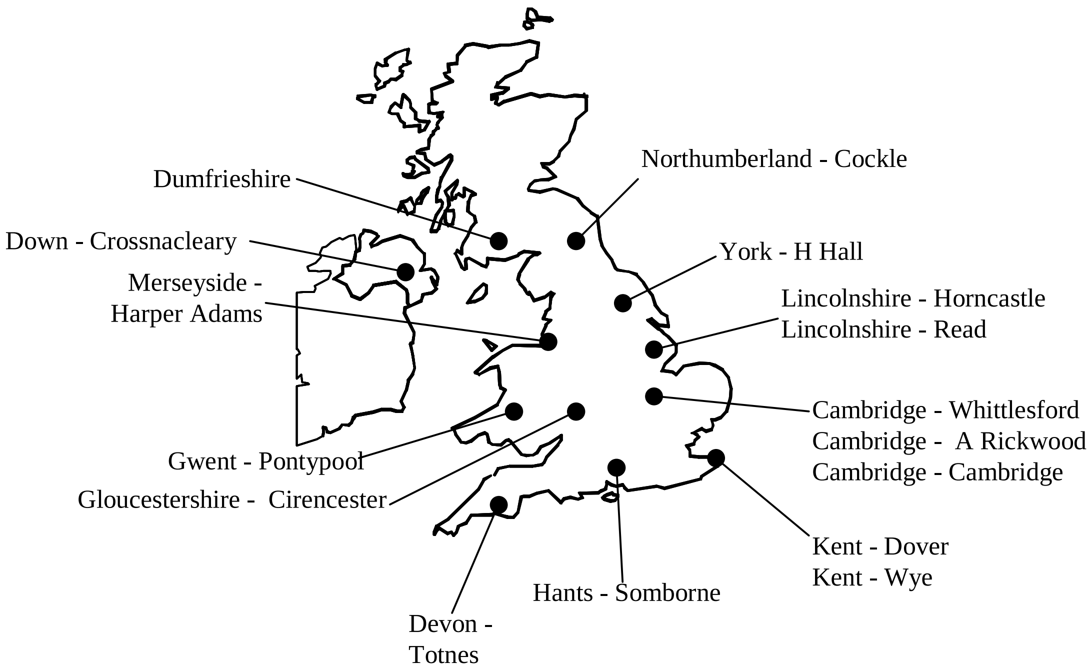

Figure 7 Trial Sites in the UK

Provider	District	Site	Drilling
NFC	Cambridge	Whittlesford	Spring
NFC	Kent	Dover	Winter
NFC	Lincolnshire	Read	Winter
NIAB	Cambridge	A Rickwood	Winter
NIAB	Cambridge	Cambridge	Winter
NIAB	Devon	Totnes	Winter
NIAB	Gloucestershire	Cirencester	Winter
NIAB	Gwent	Pontypool	Winter
NIAB	Hampshire	Somborne	Winter
NIAB	Kent	Wye	Winter
NIAB	Lincolnshire	Horncastle	Winter
NIAB	Merseyside	Harper Adams	Winter
NIAB	Northern Ireland	Crossnacleary	Winter
NIAB	Northumberland	Cockle	Winter
NIAB	Scotland	Dumfriesshire	Winter
NIAB	Yorkshire	H Hall	Winter

The following weather information was used:

Parameter	Abbreviation
Minimum temperature (in defined week)	Tmin_x (x = week number, see below for definition)
Maximum temperature (in defined week)	Tmax_x
Day degrees (in defined week)	Ddeg_x
Averaged weekly rainfall	Arain_x
Averaged weekly humidity	Hum_x
Averaged weekly wind speed	Wind_x

For the purposes of this report, week 0 is defined as day of harvest and week 1, the 1–7 days before harvest. Weather data up to 9 weeks before harvest was used in the data analysis to include the growth period post anthesis. In the event of rain being recorded on the day of harvest (week 0), it is not known whether this occurred before or after the crop was removed from the field.

There are only 15 separate sets of weather data, because the Cambridge (Cambridge) and Cambridge (Whittleford) sites are practically adjacent. The weather data were obtained from Novartis Internal Weather Profiling Software.

Appendix D contains the mean, standard deviation, maximum and minimum values for each parameter in the dataset.

Data Analysis Methods
Statistical analysis was carried out using MS Excel and Statistica version 5.5 – basic statistics, and general stepwise regression modules (Statsoft Inc. Tulsa, USA). For details see Appendix E.

Results and Discussion
The best-fit models for each RVA and rheometer parameter are given in Appendix F, and shown graphically in Appendix G.

The preliminary exploratory data analysis indicated not unexpectedly that;

(1) The whole grain Falling Number and protein measurements determined at NIAB and NFC correlates strongly with the white flour Falling Number and protein measurements made at RHM Technology. These results thus help to confirm the linkage between the samples and the test results. Whole grain Falling Number and protein measurements were used as explanatory variables in preference to the white flour measurements because these would be available at intake testing.

(2) Dew point is strongly correlated with minimum temperature, it was therefore decided to eliminate dew point as an explanatory variable.

(3) The RVA parameters (without addition of silver nitrate) are strongly correlated with each other. It is therefore unlikely that all of the parameters are required to characterise a flours starch gelatinisation temperature.

(4) The RVA parameters (without addition of silver nitrate) are strongly correlated with Falling Number. As a result Falling Number is nearly always included in the models predicting the RVA parameters. The addition of weather data, however, allows improvements over the simple linear regression models.

(5) Correlations between the RVA parameters (with addition of silver nitrate) are less pronounced with the exception of Final Viscosity with Trough and Setback. These are very strongly correlated ($r = 0.923$ and 0.933 respectively).

(6) Subtracting RVA parameters (with addition of silver nitrate) from RVA parameters (without addition of silver nitrate) resulted in derived measurements that resembled the original RVA parameters (without addition of silver nitrate) i.e. they are correlated with each other. It was therefore decided not to model these derived parameters as little, if any, addition information on starch gelatinisation temperature would be gained beyond that of modelling just the RVA parameters.

(7) Weekly cumulative figures for rainfall and the average weekly rainfall are perfectly correlated so average figures have been used.

(8) Some weekly average wind speeds are strongly correlated with each other.

(9) As a result of the numerous moderate and strong correlations in this data set it is possible to construct large numbers of similar models for each parameter (i.e., models which are equally good at predicting the gelatinisation temperature parameters but use different explanatory variables).

The table below summarises the best fit models for the starch performance parameters thought to be important for flour performance in instant thickening, batter and dough applications.

Application/property	Requirement	Predictive ability	Interpretation
		Instant thickener	
Pasting temperature	Low	Weak model derived using Tmax_9, Tmax_5, Wind_1 and Variety. Eclipse and Charger have high Pasting Temperatures relative to Soissons	There is some effect of weather and variety on Pasting Temperature, but much of the variability cannot be explained from the measured parameters.
Peak Viscosity	High	Modelled using Hagberg Falling Number, Arain_5, Arain_0, Hum_0, Wind –6 and Variety	As expected, high levels of alpha-amylase activity degrade starch and reduce peak viscosity. Weather does affect Peak Viscosity as does Variety
		Batter applications	
Pasting temp	Controlled	Weak model derived using Tmax_9, Tmax_5, Wind_1 and Variety	There is some effect of weather and variety on Pasting Temperature, but much of the variability cannot be explained from the measured parameters.
Peak Viscosity	High	Modelled using Hagberg Falling Number, Arain_5, Arain_0, Hum_0, Wind –6 and Variety	As expected, high levels of alpha-amylase activity degrade starch and reduce Peak Viscosity. Weather affects Peak Viscosity as does Variety.
Trough 1	High	Modelled using Hagberg Falling Number, Arain_2 and Wind_6	As expected, high levels of alpha-amylase activity degrade starch and reduce Trough Viscosity. Weather also affects Trough Viscosity.
		Dough applications (limited water)	
Temperature at G' = 25000Pa	High	While the dataset can be modelled using Hagberg Falling Number, Weather and Variety variables, the model has little predictive value.	
G' at 90 °C	High	A weak model was derived using Hagberg Falling Number, Weather and Variety	Alpha-amylase activity degrades starch and reduces modulus at 90°C
G' at end of test	High	No valid model	

It can be seen that Falling Number is a significant explanatory variable for many starch gelatinisation parameters. This is in line with current experience and can be understood in terms of the known effect of alpha-amylase on wheat starch. All the models are improved by the inclusion of at least one weather variable although no pattern emerges as to which week's weather is most important in determining the starch gelatinisation properties in the 1999 UK harvest. Harvest date does not appear as a significant variable in this set of best-fit models. The findings of Šebecic[6], that Amylograph maximum viscosity does not depend upon alpha-amylase activity alone, parallel our findings, although there are significant differences of detail between their methods and ours.

In general the rheometer models are poor predictors when compared with the RVA models. Most of the models, however, are again improved by the inclusion of at least one weather variable except for G' at end of test where there are no valid models. Again no pattern has emerged as to which week's weather is most important in determining the starch gelatinisation temperature in the 1999 UK harvest.

When silver nitrate is used to deactivate alpha-amylase, Falling Number is only a significant explanatory variable for RVA Viscosity Setback. The effect of Variety is more apparent in these tests. It is assumed that by minimising the effect of the enzyme on starch performance, the measurements give information on the inherent properties of the starch granules and of the molecules laid down by the plant in the granule.

The models of the RVA test with silver nitrate are, in general, also improved by the inclusion of at least one weather variable. Again no pattern has emerged as to which week's weather is most important in determining the starch gelatinisation temperature in the 1999 UK harvest.

A varietal effect on pasting temperature can be observed. In the flour RVA test, Eclipse and Charger have, on average, significantly higher pasting temperatures than the other varieties. When the RVA test is done in the presence of silver nitrate, Eclipse again has the highest pasting temperature. Hereward, Savannah, Equinox and Soissons have consistently low pasting temperatures with silver nitrate, but Riband and Charger are noticeably variable in pasting temperature in this test. Under simulated bread dough conditions, the spread of gelatinisation temperature, as measured by T25,000 is relatively narrow. Hereward has the highest T25,000 and Riband the lowest value of this measurement.

	Pasting Temperature				
	N	Mean	Std.Dev.	Lower confidence limit (95%)	Upper confidence limit (95%)
Eclipse	14	76.83	5.26	73.79	79.86
Riband	16	70.49	5.43	67.59	73.38
Charger	15	74.87	5.90	71.61	78.14
Hereward	16	69.63	6.15	66.36	72.91
Savannah	18	71.03	7.03	67.53	74.52
Equinox	15	71.05	7.15	67.09	75.01
Soissons	10	66.85	4.61	63.55	70.15

	Pasting temperature with silver nitrate				
	N	Means	Std.Dev.	Lower confidence limit (95%)	Upper confidence limit (95%)
Eclipse	14	82.39	4.75	79.65	85.13
Riband	16	73.40	8.23	69.01	77.78
Charger	15	69.57	8.23	65.01	74.13
Hereward	16	64.48	0.68	64.12	64.84
Savannah	18	66.79	6.55	63.53	70.04
Equinox	15	65.84	4.83	63.16	68.52
Soissons	10	65.01	1.10	64.22	65.79

	Temperature at G' = 25,000				
	N	Means	Std.Dev.	Lower confidence limit (95%)	Upper confidence limit (95%)
Eclipse	12	68.38	1.50	67.43	69.33
Riband	16	67.18	2.10	66.06	68.30
Charger	14	68.90	1.64	67.95	69.85
Hereward	16	69.23	1.20	68.59	69.87
Savannah	17	68.33	1.89	67.36	69.30
Equinox	15	68.51	1.41	67.73	69.30
Soissons	10	67.07	1.71	65.85	68.29

In the absence of silver nitrate, the difference between the highest average pasting temperature and the lowest average pasting temperature is approximately 10°C. Eclipse has a characteristically high pasting temperature in the dilute conditions of the RVA test. This is seen both in the presence of alpha-amylase activity and when enzyme activity has been removed by the addition of silver nitrate. It may reflect an inherent difference in starch properties between this variety and others. It would be interesting to compare the performance of Eclipse flour and one of the other flours grown at the same site in a batter or thickening application; the expectation is of different performance.

Subsets of the samples used in this study have been used to investigate effects, but due to the small number of samples involved, no statistical significance can be attributed to the results. Such studies serve to identify factors, which may merit further investigation.

The effect of site in a geographical location can be assessed by a comparison of wheat grown at Kent (Dover), a New Farm Crops site and Kent (Rye, RL/T) a NIAB site. There are no significant differences in any of the measured parameters (RVA, RVA + silver nitrate or rheometer) between sites, however, there is a wide spread of results on each site, and this will make it difficult to recognise local influences.

The effect of drilling date is confounded with site in almost all cases, but at Cambridge (NIAB) site, all varieties were sown on 15th October 1998 whereas at the nearby Whittlesford (NFC) site drilling was carried out on 5th February 1999. The harvest date at Whittlesford was 3 days earlier than harvest at Cambridge. The crop harvest at Cambridge was delayed and it is thought that the wheat was about 2 weeks beyond optimum maturity. Whittlesford wheat (late sowing) is associated with higher values of many RVA parameters (Peak Viscosity, Trough Viscosity, Viscosity Breakdown, Final Viscosity, Viscosity Set Back and Peak time) and of RVA parameters in the silver nitrate test (Peak Viscosity, Trough Viscosity, Viscosity Breakdown, Final Viscosity). Of the rheometer parameters, only G' at 90°C showed a significant effect of drilling date (late drilling gave higher value of G'). As the user is generally seeking high values of the viscosity parameters, there is a need to investigate whether this effect is generally valid, whether late sowing is a benefit for particular applications, or whether this effect is due to the Cambridge wheat being past optimum maturity. These observations are not correlated with the protein content of the samples (and by subtraction, the approximate starch content). The comment is offered that protein variability is less for the late sown crop and that early sowing may allow the protein yielding potential of the varieties to be exploited.

The wheat varieties chosen for this study cover all 4 NABIM groups. The results can be analysed to seek group characteristics. The biscuit (Group 3) wheats have the lowest value of G' at its minimum, and the breadmaking wheats (Groups 1 and 2) tend to have the highest value of this parameter. In other words, for a standard water addition, bread flour dough is stiffer than biscuit flour dough. This is consistent with their application. The biscuit manufacturer wishes to add the lowest possible amount of water in forming the dough for moulding, whereas the bread baker aims to include a high level of water in his dough. In the RVA tests, alpha-amylase is the dominant effect, but if this is removed by the use of silver nitrate, the group 4 wheats deliver the highest final viscosity.

Although it is not a stated objective of this project, the dataset has been analysed to seek a predictive model for Hagberg Falling Number. The model derived gives a useful prediction of Hagberg Falling Number when tested on the validation set. It is different from the other models including weather terms in that all relate to weeks 4,5 or 6 before harvest, i.e. the model is using weather in a reasonably limited part of the growth phase. The relevance of this model, and its relationship to past work is not known, but should be reviewed by those with greater knowledge of this property.

APPRAISAL OF BENEFITS TO THE INDUSTRY

This study was conceived as a preliminary investigation of the variability of starch pasting properties in UK wheats, and as a test of the hypothesis that they are influenced by weather. The findings show that starch properties are variable, that part of that variability can be explained by Hagberg Falling Number, but that there are specific weather and varietal influences to consider.

Growers can control the variety sown, date of sowing and aspects of crop husbandry such as nitrogen application and pesticide treatment. Our preliminary results hint at varieties, which are likely to give specific starch pasting properties (e.g. Eclipse has a high gelatinisation temperature in aqueous slurries). On a single site comparison there is evidence of a strong sowing date or maturity effect. These results need to be confirmed in future crop years to allow confidence in them to be built and a realistic estimate of their value to be made.

Breeders require information on the climatic effects on starch pasting so that they can tailor their new varieties to express certain growth characteristics at specific times. The weather models given here for starch properties have not identified a specific period that is most important, however that is not necessarily surprising as there are only 15 weather conditions to be analysed. If there is a predictable effect of weather at a particular growth stage, this should emerge if the work is extended to cover future harvests.

The value of the model for Hagberg Falling Number should be evaluated by experts in this property.

The gelatinisation behaviour of wheat flour in dilute systems (e.g. 12% as in RVA tests) cannot predict the gelatinisation behaviour observed in limited water systems like bread dough (55 - 60% water). It is practical to carry out a viscosity test on a small flour sample in the RVA (or an instrument designed to heat and measure a dilute flour suspension) and assess its potential in thickening or batter applications. It is much more difficult to mix small quantities of dough, however this is necessary if an assessment of starch behaviour under limited water conditions is needed.

The economic benefit of a specific starch gelatinisation temperature or high starch viscosity is difficult to estimate. The identification of strong varietal influences on starch gelatinisation in this work allows targeted application tests on flours of different starch behaviour to be undertaken in the future. From this, a realistic assessment of their benefit to the manufacturing industry could be derived.

CONCLUSIONS

1 The investigation was undertaken to investigate whether the hypothesis that the weather experienced by wheat in the field between anthesis and harvest has an effect on the starch gelatinisation. Weather information (on the day of harvest and weekly averages back to anthesis) can be used with wheat variety and commonly available intake measurements (Hagberg Falling Number and Protein), to predict rheological properties related to the starch gelatinisation event. It is unclear from these empirical models which weather/time period or periods experienced by the wheat are critical in affecting gelatinisation. The weather measurements that appear as significant predictors are, however, not the same as reported in controlled studies in the literature[4], i.e. average or maximum temperatures.

2 Hagberg Falling Number has a strong effect on the gelatinisation events, and as expected from understanding of the action of alpha-amylase, Hagberg Falling Number is inversely correlated to viscometric indices like Peak Viscosity, Trough Viscosity and Final Viscosity of flour slurries. Inactivation of alpha-amylase by silver nitrate reveals an effect of variety on the gelatinisation process.

3 On a single site comparison, late drilling is associated with higher paste viscosities in RVA tests both without and with silver nitrate.

4 The study of wheat flour doughs in the rheometer gave results, which yielded poorer predictive models. The biscuit wheats gave doughs with the lowest minimum stiffness during the test. The stiffness of the dough at 90°C increased with Hagberg Falling Number. Both of these models could be improved by the inclusion of one, or more weather variables.

5 The results show that there are factors other than Falling Number which affect starch gelatinisation, and through this, the rheological properties of flour slurries and doughs at high temperature. There is a small amount of published information showing a relationship between starch gelatinisation and finished product quality, and this area might prove fruitful for future research.

RECOMMENDATIONS FOR FURTHER WORK

The validity of the models needs to be checked over at least one more harvest. With a more extensive data set, it may be possible to establish which weather variables and periods between anthesis and harvest are most important. Such information would be necessary before any recommendation could be made on targeting grain with a particular growth history to a particular application.

The sample set was limited to 7 varieties, however, this represents only some of the Recommended List wheats. A more comprehensive sample set would broaden the applicability of the findings. Further discussion with breeders might reveal new varieties with unusual starch properties which merit investigation.

The samples tested in this work were grown under trial conditions that ensured a very low level of disease in the crop (approximately 5% disease on average). These conditions are used to highlight varietal differences. Samples from untreated plots were unfortunately not available. No information on the effect of disease levels on starch properties has therefore been gathered.

This work has shown the importance of Hagberg Falling Number for viscosity in dilute flour slurries. HGCA sponsored research has shown 4 separate mechanisms influencing alpha-amylase activity in the crop. This study uses wheat from a single crop year harvested in August where, at certain sites, rain delayed the harvest. It is, therefore, not possible to establish if the relationship between starch paste viscosity and Hagberg Falling Number is due to the time chosen for harvesting. It may be that early harvesting in order to preserve a high Hagberg Falling Number will have a different effect on pasting viscosity than late harvesting at an identical level of Hagberg Falling Number . An investigation of sprouting and dormancy could also provide a fuller picture of factors controlling starch gelatinisation.

Because of the effect of Hagberg Falling Number on paste viscosity it may be possible to predict gelatinisation behaviour of a crop before harvest using the existing HGCA Hagberg Falling Number model. This could be investigated in a small, planned study.

It is known that disease can effect Hagberg Falling Number. Again the link with starch properties remains to be established.

In a single plot comparison, we have seen an apparent large effect of date of sowing and/or maturity at harvest on starch properties. This question should be probed rigorously to seek a specific mechanism whereby growers might influence starch properties. In this experiment, it would be important to harvest samples at the point when the crop reached maturity.

Further work topic	Planned study	Observational study
Check validity and robustness of models (cf weather and site) over future harvests		✔
Establish specific weather variables for starch behaviour prediction		✔
Extend sample set to other varieties	✔?	✔
Investigate relationship between Hagberg Falling Number and starch behaviour for a sample set in which HFN has been controlled in specific ways to high or low values. Relate to HGCA Hagberg Falling Number prediction.	✔	
Investigate effect of level of disease on starch gelatinisation parameters	✔	✔
Investigate in detail effect of sowing date on starch properties	✔	
Investigate effect of maturity at harvest on starch properties	✔	

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10. Spillers Milling Product Brochure "Steam Treated Functional Flours"

Appendix A QUADRUMAT MILLING METHOD

1. Upon arrival, the grain is given a unique sample code. This code is entered into a spreadsheet along with the information provided with the sample (variety, origin, supplier etc). The grain is then cleaned by hand to remove foreign grains and other impurities. The sample is then bagged for storage. Grain moisture content is determined in duplicate on 5g samples using the standard method. The moisture content is used to calculate the water addition requirement for conditioning.
2. All wheat samples were conditioned to 15.5% moisture by addition of water followed by vigorous manual tumbling for 2 minutes. Samples were left overnight to condition. Milling was carried out on the day following conditioning.
3. Conditioned wheat was milled on a Buhler Quadrumat test mill. The flour fractions were collected and weighed to measure flour yield.
4. The Quadrumat was dry-cleaned between samples to minimise cross contamination.
5. Because some wheat samples had a very low Hagberg Falling Number (high alpha-amylase activity), they were milled separately from wheats of higher Hagberg Falling Number to minimise alpha-amylase contamination. (Although the Quadrumat was cleaned between samples, it is impossible to avoid a small level of cross contamination.)

Appendix B RAPID VISCO ANALYSER METHOD

1. Scope

   Testing wheat flours

2. Reference

   The Rapid Visco Analyser (RVA) instruction manual.

3. Principle

   The RVA is a viscometer with variable temperature and shear control. It is commonly used to measure viscosity of starch or starch containing materials cooked, in water. The results given can determine the quality of the starch and or level of treatment the flour has had.

4. Apparatus

   Analytical Balance
   Measuring cylinder/dropper
   RVA
   Thermocline software for Windows/Lotus123 v2.1
   Aluminium containers
   Spatula

5. Reagents

   De-ionised Water
   Silver Nitrate ($AgNO_3$) [1g/l]

6. Procedure

   The test was carried out at a standard ratio of 3g dry flour solids to 25g water. From a knowledge of the flour moisture content, the amount of flour necessary to give 3g of dry flour solids was weighed to 0.002g accuracy and corresponding amount of water was added with a pipette which allowed an accuracy of 0.03g.

   To avoid lumps of flour in the container the samples were stirred with a spatula before the canisters were inserted into the RVA.

   The test was carried out in duplicate. The flours were tested both with pure de-ionised water and silver nitrate solution . The silver nitrate solution was used as an inhibitor of alpha-amylase to investigate how the starch cooks without the influence of enzymes.

The measured parameters are:

Pasting Temperature:	Temperature at which the rate of increase in viscosity is greater than 24 centipoise/sec.
Peak1:	Viscosity at peak
Peaktime:	Time at peak 1
Trough 1:	Viscosity at trough
Final Viscosity:	Viscosity at end
Breakdown:	Peak 1 – trough 1
Setback:	Final Viscosity – trough 1

Appendix C RHEOMETER DOUGHS METHOD

1. Principle

The rheometer is a controlled stress or controlled rate instrument. It measures deformation in the non-destructive region of elastic or visco-elastic deformation. The measuring system consists of the fixed heating/cooling plate, the stator, and the rotor, a motor driven geometry.

2. Apparatus

Desktop Computer
Rheology Navigator 2 plus software v2.0
Julabo FS18 Water Bath
AR 1000 Rheometer
Balance
Beaker
Pipette
Geometry (parallel plate: 40mm diameter stainless steel parallel plate)
Minorpin Mixer, mixing bowl
Spatula
Stopwatch
Razor blade

3. Reagents

Flour (50g)
Water (30ml)
Yeast (1.1g)
Commercial Flour improver for breadmaking (0.5g)
(The improver contains ascorbic acid, enzyme active soya flour, DATEM and an enzyme preparation. It is a commercial product whose precise composition is not disclosed by the supplier.)
Salt (0.9g)
Light paraffin oil

4. Procedure

Preparation of dough:

Just before the test commenced the dough was mixed. First the dry ingredients (flour, salt, flour improver) were weighed into the mixing-bowl, then yeast and water were added. The dough was mixed for 5min in a Minorpin mixer.

Rheometer test:

A piece of dough from the mixing-bowl was presented to the rheometer and the sample prepared for the test. The surplus of dough around the geometry was cut off with a razor blade. Finally the edge was covered with paraffin to avoid evaporation during the test. The time between mixing and start of the test is set to 5min.

During the test the sample was heated from 30°C to 40°C over 300s. It was then heated to 95°C over 630s held at 95°C for 60s, then cooled down to 30°C over 300sec and finally held for 60sec at 30°C. See figure below.

Data analysis:

The test is an oscillation heating cooling ramp test. The variable measured is G'. G' is the elasticity or the storage modulus. Four different values are taken from the received graph for statistical analysis.

- Minimum of G' (before dough setting temperature)
- Temperature at G' = 25000Pa
- G' at 90°C
- G' at end of test

The setting temperature is arbitrarily defined as the temperature at G' = 25000Pa.

Appendix D SUMMARY STATISTICS

Descriptive Statistics					
Variable	**Valid N**	**Mean**	**Minimum**	**Maximum**	**Std.Dev**
HFN_ALL:- Wholemeal HFN	93	197.29	62.000	329.0	68.308
LN_HFN_A: =log(HFN_ALL)	93	5.21	4.127	5.8	.410
PROT_ALL:- Wholemeal Protein Content	79	12.10	8.850	15.1	1.271
HFN_FL: RHM T Milling - Falling Number	104	247.80	62.000	558.0	97.742
LN_HFN_F: =log(HFN_FL)	104	5.42	4.127	6.3	.448
DRY_MC: Milling - Dry MC (%)	104	12.65	11.100	16.0	1.134
WATERADD: Milling - Water added (cm3)	104	16.75	0.000	26.0	6.416
RECOVERY: Milling - Recovery (%)	104	98.68	96.200	102.0	.802
FLOUR400: Milling - Flour from 400g of grain	104	255.96	231.00	280.0	12.22
MMOIS_FL: Milling - Flour Moisture	104	14.76	13.500	17.0	.564
CMOIS_FL: Chemistry - Flour Moisture	104	14.53	13.452	16.2	.499
PROT_FL: Chemistry - Flour Protein	104	9.01	6.286	12.0	1.147
P_1: RVA – Peak 1	104	1336.14	166.000	2967.0	734.103
T_1: RVA – Trough 1	104	471.61	-35.000	1602.0	434.235
BREAK: RVA - Break – down	104	864.54	184.000	1592.0	335.832
FIN_V: RVA - Final Visc	104	1048.77	-29.000	3009.0	856.391
SETBA: RVA – Setback	104	577.16	6.000	1547.0	427.278
PK_T: RVA – Peak Time	104	4.89	2.800	5.8	.740
PST: RVA – Pasting Temp	104	71.67	63.550	83.1	6.583
AG_P_1: RVA - Peak 1SN	104	3651.11	3125.000	4099.0	222.338
AG_T_1: RVA - Trough 1SN	104	2057.88	1618.000	2437.0	140.455
AG_BREAK: RVA - Break – downSN	104	1593.23	1216.000	1992.0	161.668
AG_FIN_V: RVA - Final ViscSN	104	3717.39	3011.000	4475.0	269.745
AG_SETBA: RVA – SetbackSN	104	1659.52	1308.000	2038.0	150.145
AG_PK_T: RVA - Peak TimeSN	104	5.95	5.670	6.1	.107
AG_PST: RVA - Pasting TempSN	104	69.64	62.700	85.6	8.148
T25000: Rheometer – temperature at G' = 25000 fr	100	68.28	60.700	73.6	.1.781
MIN_G: Rheometer – Minimum Value of G'	104	676.60	140.800	2942.0	.466.505
V_MIN_G: Rheometer –temperature at that point f	104	54.07	46.400	57.5	.2.283
G_AT_90: Rheometer - G' at 90C	104	10317.41	.288.0	20710.0	4301.567
G_AT_ET: Rheometer - G' at end of test	104	26882.37	9556.000	58590.0	8474.240
TMIN_9: Weather data – Tmin_9	104	10.67	7.182	12.9	1.650
TMIN_8: Weather data – Tmin_8	104	11.81	8.555	14.0	1.609
TMIN_7: Weather data – Tmin_7	104	12.58	8.286	15.9	2.058
TMIN_6: Weather data – Tmin_6	104	12.72	9.222	14.9	1.901
TMIN_5: Weather data – Tmin_5	104	12.91	8.571	15.5	2.195
TMIN_4: Weather data – Tmin_4	104	13.86	10.508	15.8	1.367
TMIN_3: Weather data – Tmin_3	104	13.04	7.818	17.0	2.573
TMIN_2: Weather data – Tmin_2	104	12.62	6.635	17.0	2.591
TMIN_1: Weather data – Tmin_1	104	12.03	7.429	14.8	2.216
TMIN_0: Weather data – Tmin_0	104	12.47	5.222	17.9	2.598
TMAX_9: Weather data – Tmax_9	104	19.43	15.867	23.1	1.775
TMAX_8: Weather data – Tmax_8	104	20.09	16.717	23.3	1.945
TMAX_7: Weather data – Tmax_7	104	21.30	15.717	23.8	2.192
TMAX_6: Weather data – Tmax_6	104	21.23	16.571	24.2	2.474
TMAX_5: Weather data – Tmax_5	104	22.12	17.795	25.0	1.812
TMAX_4: Weather data – Tmax_4	104	23.07	18.353	26.0	2.289
TMAX_3: Weather data – Tmax_3	104	22.48	15.623	27.6	3.766
TMAX_2: Weather data – Tmax_2	104	20.71	15.251	28.9	3.692
TMAX_1: Weather data – Tmax_1	104	20.23	16.117	25.4	2.276

Descriptive Statistics					
Variable	**Valid N**	**Mean**	**Minimum**	**Maximum**	**Std.Dev**
TMAX_0: Weather data – Tmax_0	104	20.72	16.522	26.6	2.443
DDEG_9: Day degrees = (Tmax(9)+Tmin(9))/2	104	15.05	12.667	17.5	1.431
DDEG_8: Day degrees = (Tmax(8)+Tmin(8))/2	104	15.95	12.936	18.6	1.549
DDEG_7: Day degrees = (Tmax(7)+Tmin(7))/2	104	16.94	13.917	19.6	1.895
DDEG_6: Day degrees = (Tmax(6)+Tmin(6))/2	104	16.97	14.175	19.5	1.897
DDEG_5: Day degrees = (Tmax(5)+Tmin(5))/2	104	17.51	14.214	20.1	1.630
DDEG_4: Day degrees = (Tmax(4)+Tmin(4))/2	104	18.46	15.857	20.3	1.369
DDEG_3: Day degrees = (Tmax(3)+Tmin(3))/2	104	17.76	12.798	21.5	2.961
DDEG_2: Day degrees = (Tmax(2)+Tmin(2))/2	104	16.67	11.302	22.0	2.992
DDEG_1: Day degrees = (Tmax(1)+Tmin(1))/2	104	16.13	12.357	20.0	2.045
DDEG_0: Day degrees = (Tmax(0)+Tmin(0))/2	104	16.59	11.806	22.3	2.166
ARAIN_9: Weather data – Arain_9	104	1.64	.029	5.4	1.593
ARAIN_8: Weather data – Arain_8	104	1.34	0.000	4.4	1.338
ARAIN_7: Weather data – Arain_7	104	1.27	0.000	4.7	1.353
ARAIN_6: Weather data – Arain_6	104	.75	0.000	1.8	.508
ARAIN_5: Weather data – Arain_5	104	.57	0.000	2.9	.800
ARAIN_4: Weather data – Arain_4	104	1.77	0.000	9.5	2.666
ARAIN_3: Weather data – Arain_3	104	2.21	0.000	7.3	2.241
ARAIN_2: Weather data – Arain_2	104	2.39	0.000	8.0	2.022
ARAIN_1: Weather data – Arain_1	104	3.15	0.000	16.3	3.945
ARAIN_0: Weather data – Arain_0	104	1.21	0.000	17.0	4.269
HUM_9: Weather data – Hum_9	104	54.83	42.572	75.7	7.456
HUM_8: Weather data – Hum_8	104	58.24	47.021	78.5	6.701
HUM_7: Weather data – Hum_7	104	56.16	48.429	73.7	6.216
HUM_6: Weather data – Hum_6	104	56.35	42.906	72.3	8.013
HUM_5: Weather data – Hum_5	104	53.53	40.516	70.5	8.096
HUM_4: Weather data – Hum_4	104	52.84	34.859	68.7	9.130
HUM_3: Weather data – Hum_3	104	53.06	38.434	68.8	8.520
HUM_2: Weather data – Hum_2	104	57.42	40.401	68.1	6.411
HUM_1: Weather data – Hum_1	104	57.02	48.013	76.0	6.509
HUM_0: Weather data – Hum_0	104	56.59	41.883	79.3	9.794
WIND_9: Weather data – Wind_9	104	7.45	4.843	11.1	1.985
WIND_8: Weather data – Wind_8	104	6.88	4.657	11.0	1.626
WIND_7: Weather data – Wind_7	104	7.91	4.400	15.1	2.625
WIND_6: Weather data – Wind_6	104	8.18	6.300	10.8	1.221
WIND_5: Weather data – Wind_5	104	7.95	4.943	13.5	2.222
WIND_4: Weather data – Wind_4	104	8.39	5.624	15.4	2.260
WIND_3: Weather data – Wind_3	104	7.54	3.329	13.6	2.451
WIND_2: Weather data – Wind_2	104	7.54	4.443	11.6	2.012
WIND_1: Weather data – Wind_1	104	7.40	5.071	10.6	1.515
WIND_0: Weather data – Wind_0	104	7.65	4.000	19.6	3.645

Appendix E DATA ANALYSIS METHODS

Statistical analysis was carried out using MS Excel V2000 and Statistica version 5.5 – basic statistics, and general stepwise regression modules (Statsoft Inc. Tulsa, USA).

A general stepwise regression approach was used (General forward stepwise covariance analysis) as it can handle both categorical data such as wheat variety and continuous variables such as the weather data. The various rheological properties relating to starch gelatinisation (the response variables) were individually modelled using wheat variety, whole grain Falling Number and protein values and weather data as the explanatory variables.

Approximately two thirds of the sample data was selected at random and used to construct the calibration models. The remaining one third (validation set) was used to test the prediction ability of each model. The validation set covered all sites, but not all varieties.

For both calibration and validation various diagnostic statistics have been produced to assess the goodness of fit, these include residual plots, normal probability plots and 95% confidence and prediction intervals.

In interpreting the models the following points should be noted:

1. A good model is not the same as establishing cause and effect. All the models are empirical in nature and therefore significant variables included in a model cannot be interpreted as **causing** an effect in the response. For example, in the a model for RVA Trough Viscosity, R^2_{adj}=0.66
   RVA Trough viscosity = – 40.1772 + 4.6069 * Hagberg Falling Number + 42.6493 * Arain_2 – 53.9819 * Wind _6

   The average rain for week 2 preceding harvest cannot be said to be causing an increase in RVA Trough Viscosity. It is, however, a useful predictor of RVA Trough Viscosity.

2. In multiple regression models the regression coefficients are not independent of each other and should therefore be interpreted carefully. Using the example above, we can say that for each unit increase in Hagberg Falling Number at a given **constant** Arain_2 value the RVA Trough Viscosity is estimated to increase by 4.6069. Also, assuming a constant Falling Number, each unit increase in Arain_2 results in an estimated increase in RVA Trough Viscosity by 42.6493.

3. The order, size or sign of the coefficient cannot be used as indicators of the importance of the variables in the model.

4. For many, if not all models in this study there are a number of alternatives which could have been produced i.e., they will have different combination of explanatory variables and regression coefficients but give similar calibration and prediction results.

5. In the models that include the categorical variable, Variety, only 6 of the 7 varieties (Savannah, Riband, Hereward, Equinox, Eclipse, Charger) appear in the parameter estimate table. If we are interested in predicting a response for one of these varieties it takes the value 1 otherwise it is zero. Since there can only be one variety for each sample the result is seven equations, six of which have an additional constant (the wheat varieties regression coefficient/parameter). This is a correction to the response variable relative to the behaviour if the wheat variety is Soissons. For example, in a model for Viscosity Breakdown:

STAT. VISUAL GSR	**Parameter Estimates (hgca_sitepairs.sta) Sigma-restricted parameterization**		
Effect	**Level of Effect**	**BREAK Parameter**	**BREAK Std.Err**
Intercept		1381.48	148.6172
HFN_ALL		1.289	0.3731
Tmin_0		32.993	11.4828
Arain_4		-18.965	8.6776
Arain_3		38.403	11.3556
Arain_2		-111.388	21.5135
Arain_0		55.895	9.8961
Hum_3		-13.300	2.5960
Wind_9		-43.913	11.4454
VARIETY	Eclipse	244.828	47.0110
VARIETY	Riband	-100.508	35.4234
VARIETY	Charger	-28.938	33.9920
VARIETY	Hereward	-5.825	29.0651
VARIETY	Savannah	-87.082	31.4487
VARIETY	Equinox	-128.432	30.6127

Soissons

Viscosity breakdown = 1381.480 + 1.289 * Hagberg Falling Number + 32.993 * Tmin_0 – 18.965 * Arain_4 + 38.403 * Arain_3 – 111.388 * Arain_2 + 55.895 * A rain _ 0 – 13.300 * Hum3 – 43.913 * Wind_9

Savannah

Viscosity breakdown = 1381.480 – 87.082 + 1.289 * Hagberg Falling Number + 32.993 * Tmin_0 – 18.965 * Arain_4 + 38.403 * Arain_3 – 111.388 * Arain_2 + 55.895 * A rain _ 0 – 13.300 * Hum3 – 43.913 * Wind_9

Riband

Viscosity breakdown = 1381.480 –100.508 + 1.289 * Hagberg Falling Number + 32.993 * Tmin_0 – 18.965 * Arain_4 + 38.403 * Arain_3 – 111.388 * Arain_2 + 55.895 * A rain _ 0 – 13.300 * Hum3 – 43.913 * Wind_9

Hereward

Viscosity breakdown = 1381.480 –5.825 + 1.289 * Hagberg Falling Number + 32.993 * Tmin_0 – 18.965 * Arain_4 + 38.403 * Arain_3 – 111.388 * Arain_2 + 55.895 * A rain _ 0 – 13.300 * Hum3 – 43.913 * Wind_9

Equinox

Viscosity breakdown = 1381.480 –128.432 + 1.289 * Hagberg Falling Number + 32.993 * Tmin_0 – 18.965 * Arain_4 + 38.403 * Arain_3 – 111.388 * Arain_2 + 55.895 * A rain _ 0 – 13.300 * Hum3 – 43.913 * Wind_9

Eclipse

Viscosity breakdown = 1381.480 +244.828 + 1.289 * Hagberg Falling Number + 32.993 * Tmin_0 – 18.965 * Arain_4 + 38.403 * Arain_3 – 111.388 * Arain_2 + 55.895 * A rain _ 0 – 13.300 * Hum3 – 43.913 * Wind_9

Charger

Viscosity breakdown = 1381.480 –28.938 + 1.289 * Hagberg Falling Number + 32.993 * Tmin_0 – 18.965 * Arain_4 + 38.403 * Arain_3 – 111.388 * Arain_2 + 55.895 * A rain _ 0 – 13.300 * Hum3 – 43.913 * Wind_9

These models are therefore only valid for the pure wheat varieties listed above.

(*Note: The choice of Soissons for the base equation is purely arbitrary - it is the last unique wheat variety encountered in the data table as the statistics package reads down the table.*)

Appendix F BEST-FIT MODELS

RVA regression models (no silver nitrate):
The viscosity parameters can be modelled quite well. In all cases, the inclusion of one or more weather terms improves the prediction over that obtained using Hagberg Falling Number alone. The models for Time at Peak and for Pasting Temperature are less robust, particularly the Pasting Temperature model which has little predictive capacity.

RVA Peak Viscosity = 1346.343 + 4.347* Hagberg Falling Number + 118.672 * Arain_5 + 17.638 * Arain_0 + 26.143 * Hum_0 – 292.121 * Wind _6 + 260.814 * Eclipse – 305.829 * Riband – 135.103 * Charger + 11.292 * Hereward + 37.958 * Savannah – 159.640 * Equinox

$r^2_{adj}= 0.86$

(This model shows evidence of overfitting, although all parameters stated are significant.)

RVA Trough viscosity = – 40.1772 + 4.6069 * Hagberg Falling Number + 42.6493 * Arain_2 – 53.9819 * Wind _6

$r^2_{adj}= 0.73$

Viscosity breakdown = 1381.480 + 1.289 * Hagberg Falling Number + 32.993 * Tmin_0 – 18.965 * Arain_4 + 38.403 * Arain_3 – 111.388 * Arain_2 + 55.895 * A rain – 0 13.300 * Hum3 – 43.913 * Wind_9 +244.828 * Eclipse – 100.508 * Riband – 28.938103 * Charger - 5.825 * Hereward - 87.082 * Savannah – 128.432 * Equinox

$r^2_{adj}= 0.88$

(There are 3 outliers in the validation set using this equation, suggesting that it is not adequate for all cases.)

RVA Final Viscosity = 185.070 + 9.247* Hagberg Falling Number + 66.630 * Arain_2 – 122.760 * Wind_6

$r^2_{adj}= 0.76$

RVA Viscosity Setback = 378.1603 + 4.3496 * Hagberg Falling Number + 12.6317 * Arain_0 – 77.2099 * Wind_6

r^2_{adj}= 0.78

RVA Time at Peak = 5.53257 + 0.007618 * Hagberg Falling Number – 0.095379 * Ddeg_8 – 0.076588 * Wind_9

r^2_{adj}= 0.73

(A small number of data points with a low value of time at peak (about 3 minutes) have a strong influence on this model. While these are fitted well in the validation set, it may be more appropriate to restrict the model to more typical peak times (4 to 6 minutes).)

RVA Pasting Temperature = 45.74185 – 1.74364 * Tmax_9 + 2.29236 * Tmax_5 + 1.30232 * Wind_1 + 8.44798 * Eclipse – 3.20180 * Riband + 6.03682 * Charger – 2.67543 * Hereward – 0.83445 * Savannah – 1.43662 * Equinox

r^2_{adj}= 0.46

RVA (with added of silver nitrate)

Falling Number is now only a significant explanatory variable for Viscosity Setback. All the models are again improved by the inclusion of at least one weather variable except for pasting temperature which is unable to give useful predictions (see previous notes). Again no pattern has emerged as to which week's weather is most important in determining the starch gelatinisation temperature in the 1999 UK harvest. By inactivating alpha-amylase, variety is used more often as a predictor of behaviour.

The regression models are:

RVA Peak Viscosity with silver nitrate = 5402.452 – 43.507 * Protein + 187.574 * Tmin_1 – 168.407 * Ddeg_1 – 30.403 * Arain_1 – 13.932 * Hum_5 + 31.058 * Eclipse + 44.481 * Riband + 17.192 * Charger + 45.033 * Hereward + 69.729 * Savannah + 15.598 * Equinox

r^2_{adj}= 0.84

RVA Trough Viscosity with silver nitrate = 2691.634 – 28.300 * Ddeg_8 – 64.318 * Arain_5 – 21.714 * Wind_9 – 29.793 * Eclipse – 10.473 * Riband + 36.882 * Charger – 8.735 * Hereward + 149.974 * Savannah + 82.350 * Equinox

r^2_{adj}= 0.74

RVA Viscosity Breakdown with silver nitrate = 991.6659 + 54.7178 * Tmin_8 – 14.9210 * Tmin_8 – 94.8206 * Arain_8 + 47.0422 * Arain_7 – 43.1647 * Arain_3 + 11.7372 * Hum_1 – 35.7996 * Wind_7 + 75.9613 * Eclipse + 99.2136 * Riband – 20.8348 * Charger + 8.7548 * Hereward – 62.9204 * Savannah – 40.0889 * Equinox

r^2_{adj}= 0.87

RVA Viscosity Breakdown with silver nitrate = 4171.163 – 27.537 * Tmin_7 – 208.061 * Arain_6 – 156.701 * Eclipse + 11.823 * Riband + 26.092 * Charger – 94.242 * Hereward + 383.732 * Savannah + 181.595 * Equinox

r^2_{adj}= 0.79

RVA Viscosity Setback with silver nitrate = 1980.976 + 0.294 * Hagberg Falling Number – 24.087 * Ddeg_9 – 76.722 * Arain_6 – 131.837 * Eclipse + 44.253 * Riband – 19.020 * Charger _ 69.048 * Hereward + 235.048 * Savannah + 93.980 * Equinox

r^2_{adj}= 0.80

RVA Time at Peak with silver nitrate = 4.981247 + 0.016124 * Tmax_6 + 0.017436 * Tmax_5 – 0.007883 * Arain_3 + 0.004526 * Hum_1 + 0.091827 * Eclipse – 0.040394 * Riband + 0.021590 * Charger + 0.0345853 * Hereward – 0.021309 * Savannah + 0.067401 * Equinox

$r^2_{adj} = 0.58$

(Note: Time at Peak has a small number of discrete values over a narrow range due to the method of collecting the data.)

RVA Pasting Temperature with silver nitrate = 69.80577 + 14.31923 * Eclipse + 1.83173 * Riband – 0.42452 * Charger – 5.33911 * Hereward – 1.03077 * Savannah – 4.90077 * Equinox

$r^2_{adj} = 0.43$

(Note: The results fall into 2 distinct groups (group at about 65°C and group at about 83°C), so the data does not lend itself to robust modelling.)

Rheometer

In general these models are poor predictors when compared with the RVA models. Most of the models, however, are again improved by the inclusion of at least one weather variable except for G' at end of test where there are no valid models. Again no pattern has emerged as to which week's weather is most important in determining the starch gelatinisation temperature in the 1999 UK harvest.

The regression models are:

Temperature at which G' = 25,000Pa = 52.20894 – 0.00747 * Hagberg Falling Number + 0.65413 * Ddeg_5 + 0.42726 * Ddeg_1 – 1.49787 * Arain_6 + 0.52431 * Eclipse – 0.90431 * Riband +0.58399 * Charger + 0.97718 * Hereward + 0.10386 * Savannah + 0.17789 * Equinox

$r^2_{adj} = 0.64$

(Note that while this equation is a reasonable fit to the calibration set, it does not predict the validation set effectively.)

Minimum Value of G' = 1041.996 – 35.642 * Wind_7 – 543.350 * Eclipse – 391.276 * Riband + 14.696 * Charger + 347 .432 * Hereward – 282.849 * Savannah – 168.218 * Equinox

r^2_{adj}= 0.57

Temperature at Minimum Value of G' = 55.43634 – 1.90077 * Arain_6

r^2_{adj}= 0.18

G'at 90°C = 26018.18 + 20.62 * Hagberg Falling Number + 450.34 * Tmax_6 – 1325.01 * Ddeg8 – 1052.16 * Wind_5 – 1769.06 * Eclipse – 1559.97 * Riband – 444.75 * Charger + 222.54 * Hereward + 197.87 * Savannah – 1993.81 * Equinox

r^2_{adj}= 0.64

No significant model could be derived for G' at end of test.

Hagberg Falling Number Model

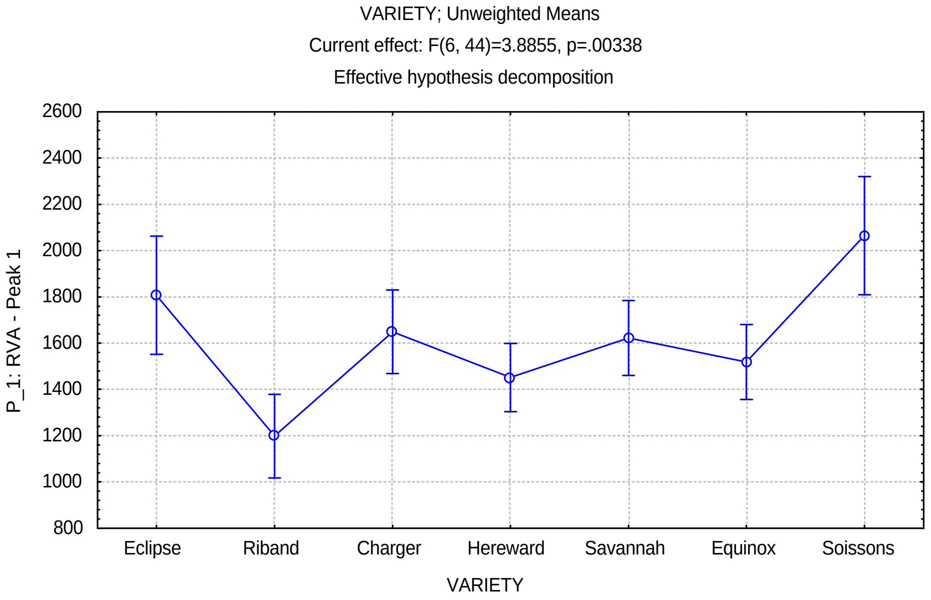
ln (Hagberg Falling Number) = 12.11504 + 0.10253*Tmin_4 – 0.16865*Tmax_4 – 0.05112*Ddeg_5 – 0.06010*Arain_4 – 0.01729*Hum_6 – 0.27528*Wind_6 – 0.02085* Eclpise – 0.27894* Riband – 0.07434* Charger – 0.01897* Hereward + 0.01765* Savannah + 0.06302* Equinox

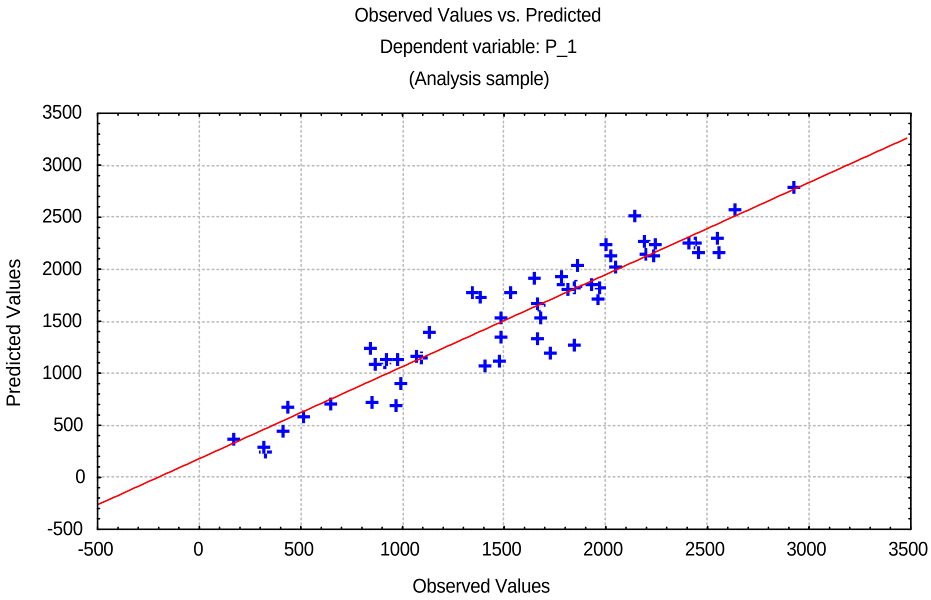
r^2_{adj}= 0.72

(ln = $\log_e$ = Natural log)

Appendix G PLOTS OF BEST-FIT MODELS

Figure G1 (a to c) RVA Peak Viscosity





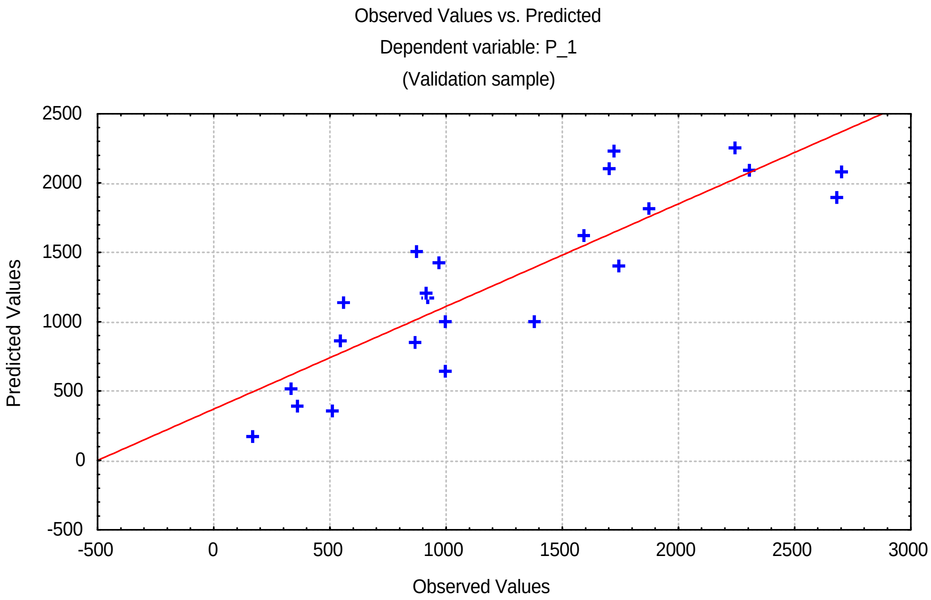
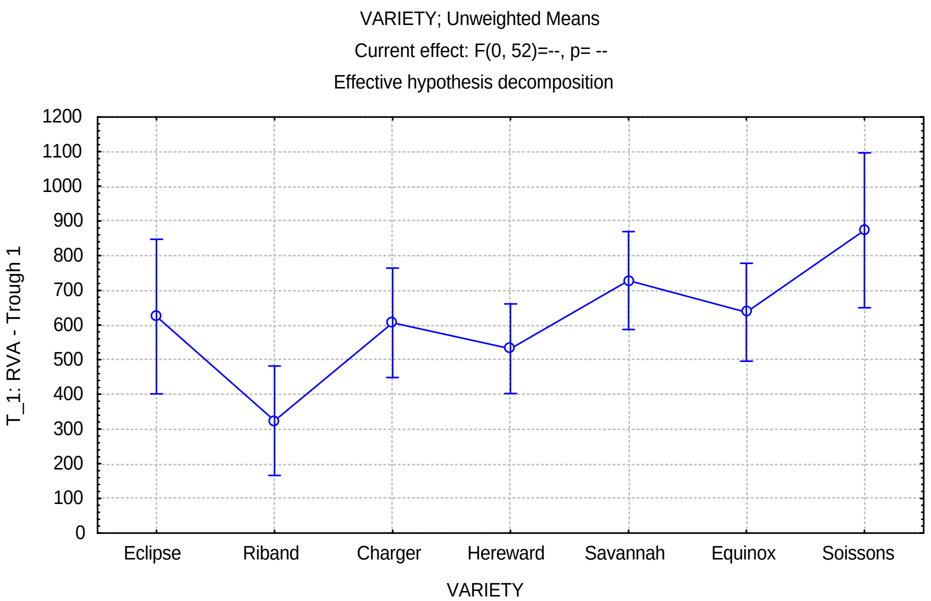
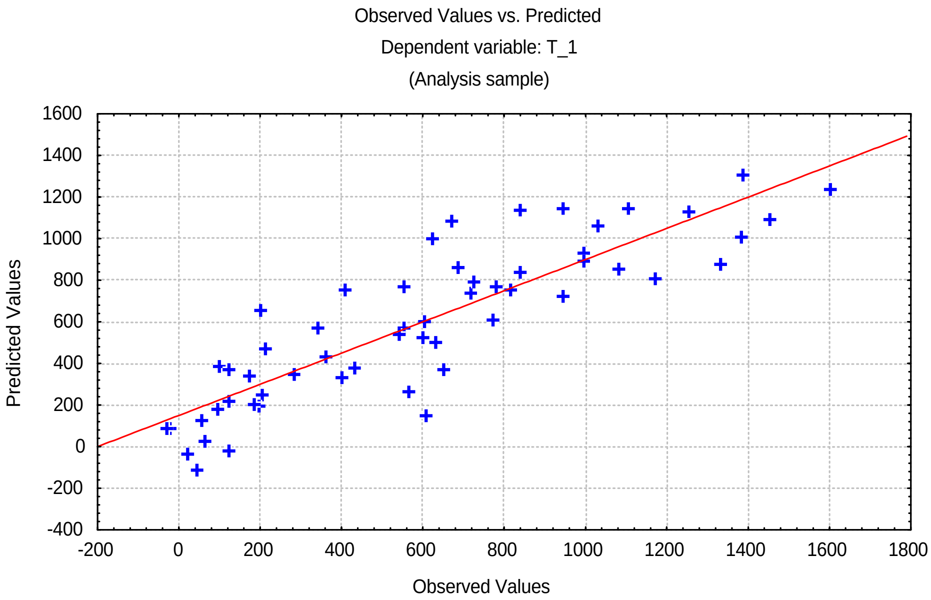


Figure G2 (a to c) **RVA Trough viscosity**




Observed Values vs. Predicted
Dependent variable: T_1
(Analysis sample)
Predicted Values
Observed Values
1600
1400
1200
1000
800
600
400
200
0
-200
-400
-200
0
200
400
600
800
1000
1200
1400
1600
1800

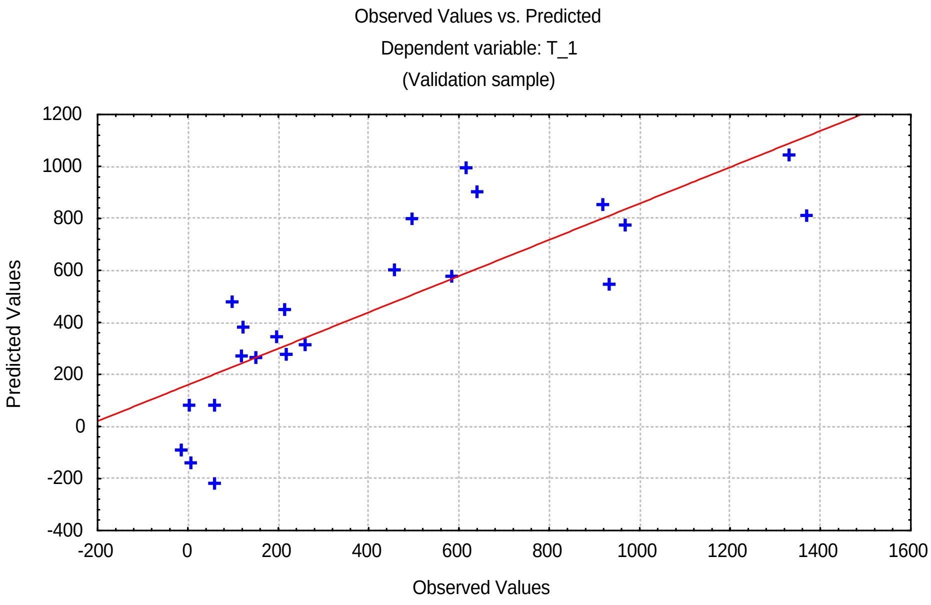
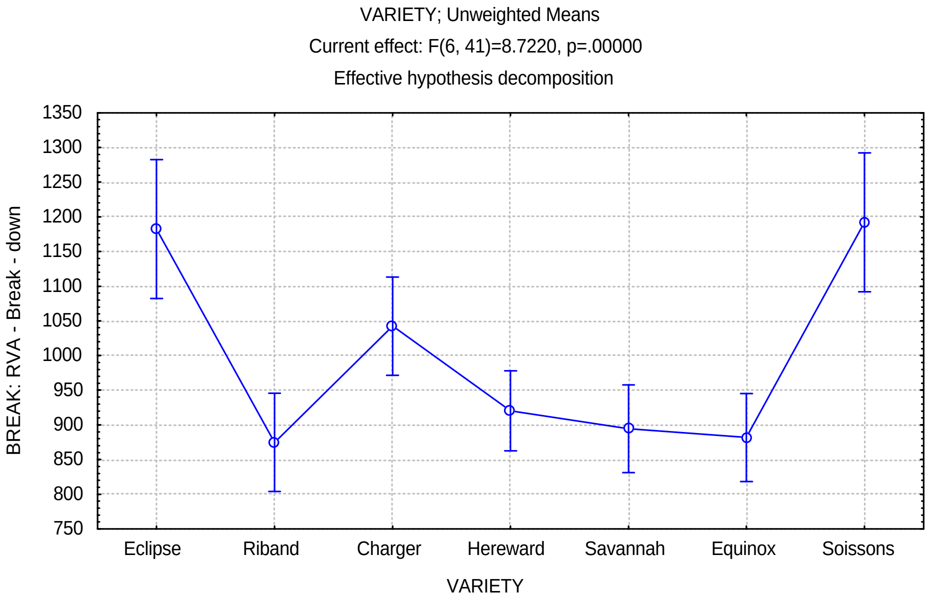
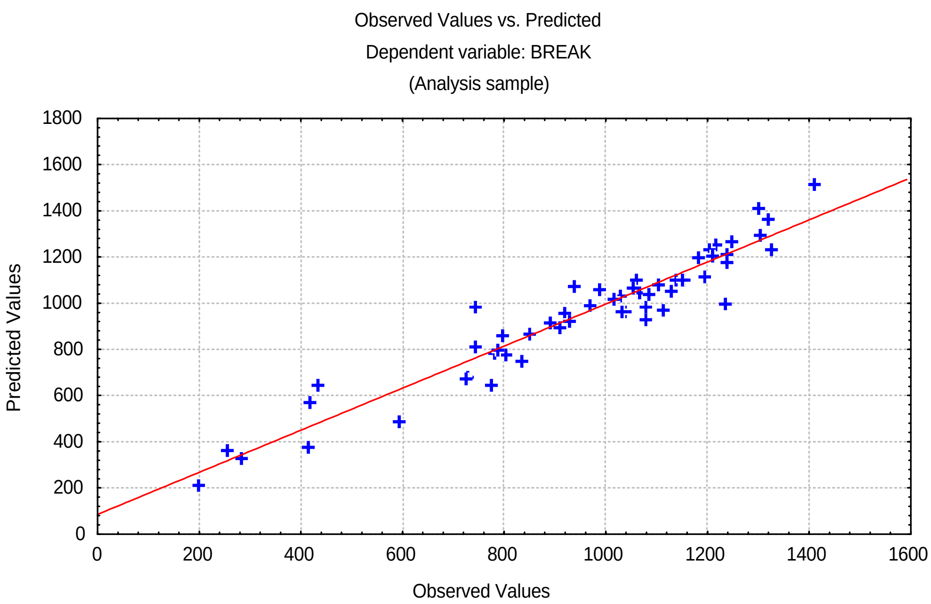

Observed Values vs. Predicted
Dependent variable: T_1
(Validation sample)
Predicted Values
Observed Values
1200
1000
800
600
400
200
0
-200
-400
-200
0
200
400
600
800
1000
1200
1400
1600

Figure G3 (a to c) Viscosity breakdown





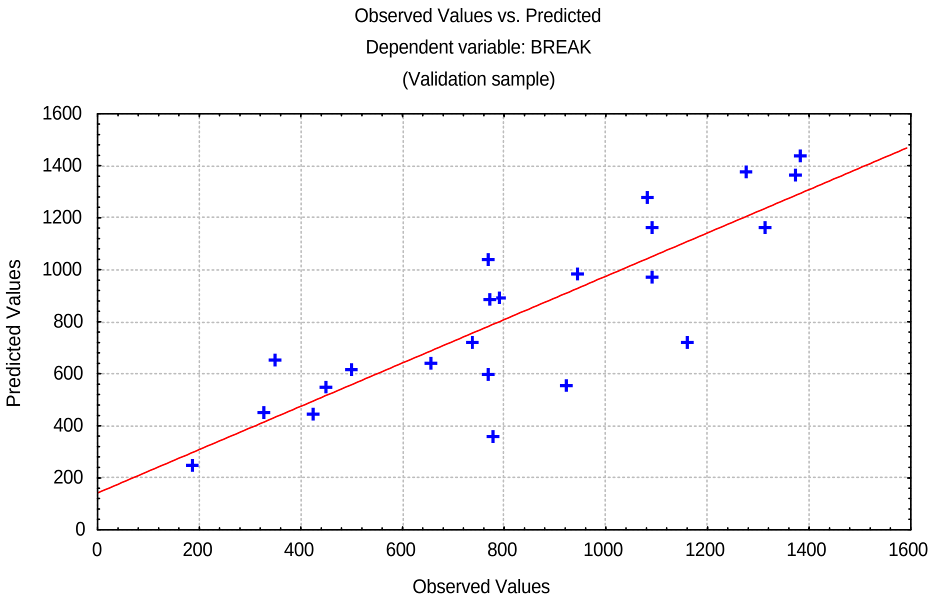
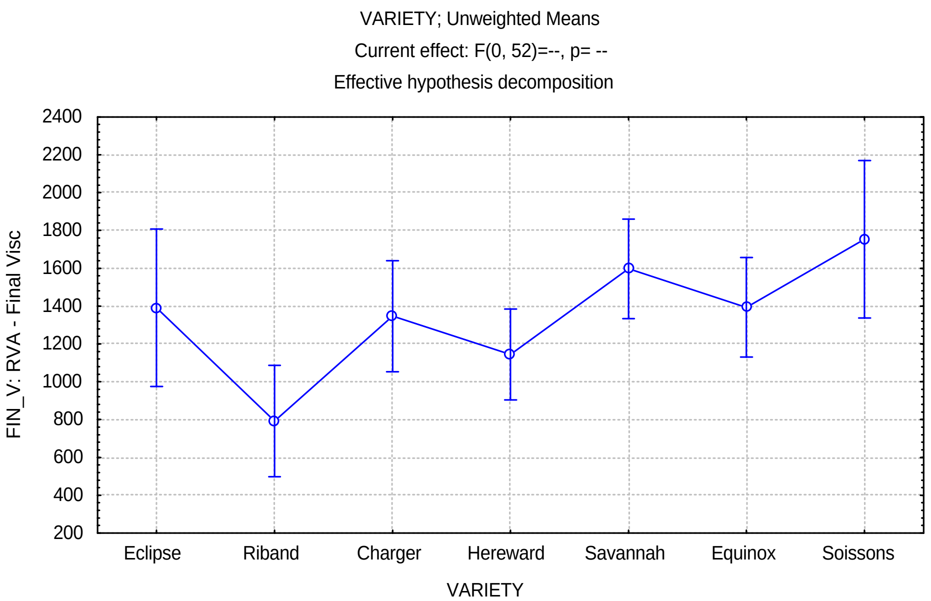
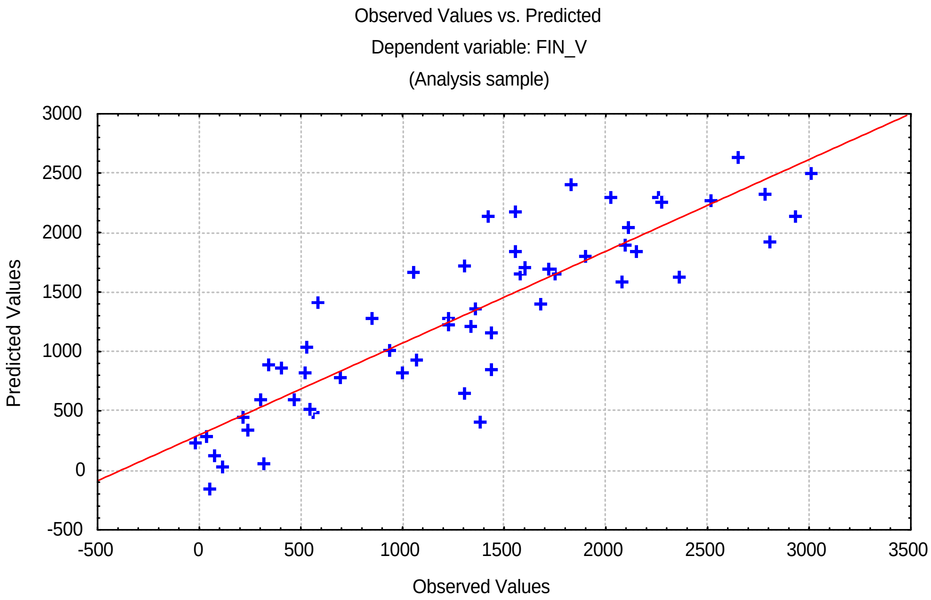


Figure G4 (a to c) RVA Final Viscosity




Observed Values vs. Predicted
Dependent variable: FIN_V
(Analysis sample)
Predicted Values
Observed Values

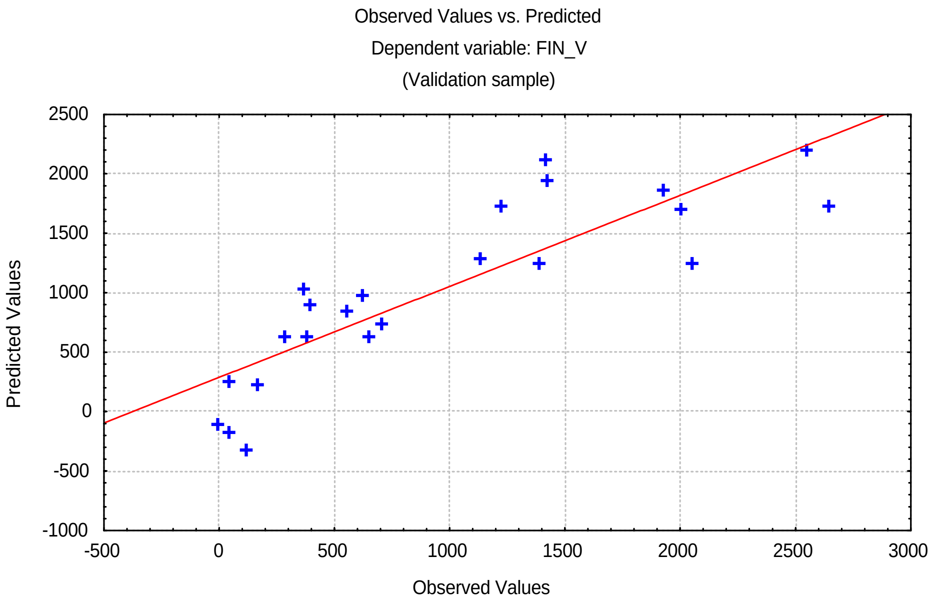
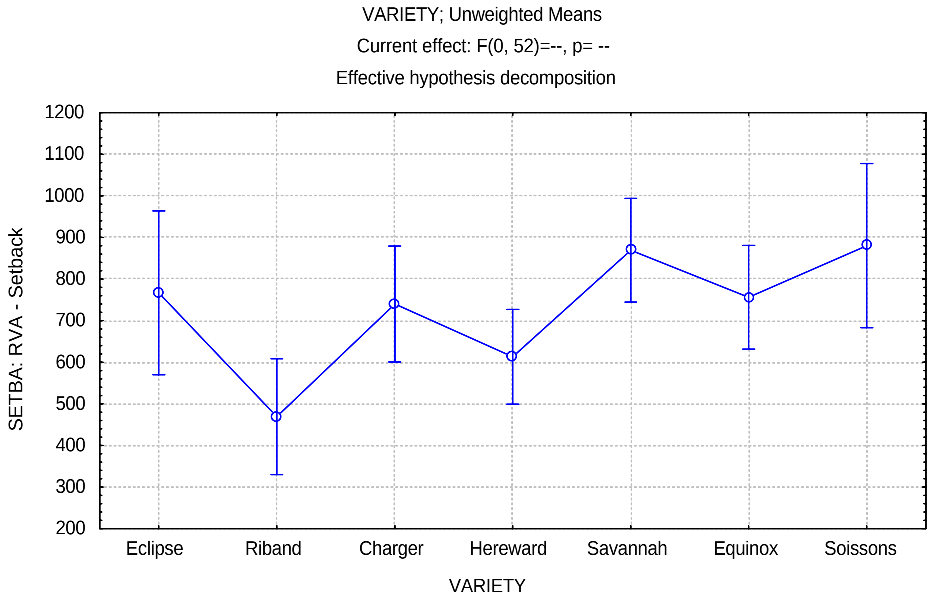
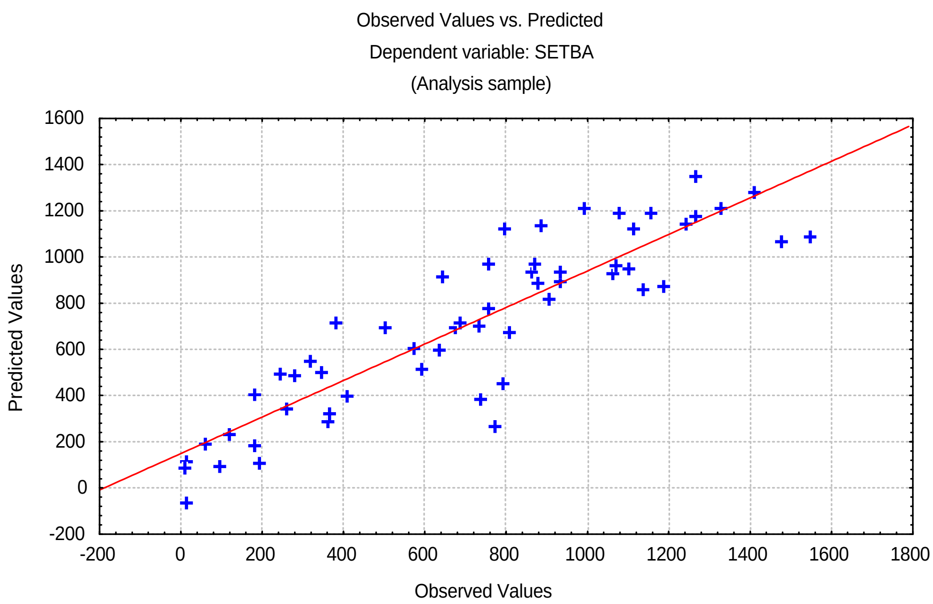

Observed Values vs. Predicted
Dependent variable: FIN_V
(Validation sample)
Predicted Values
Observed Values

Figure G5 (a to c)   RVA Viscosity Setback





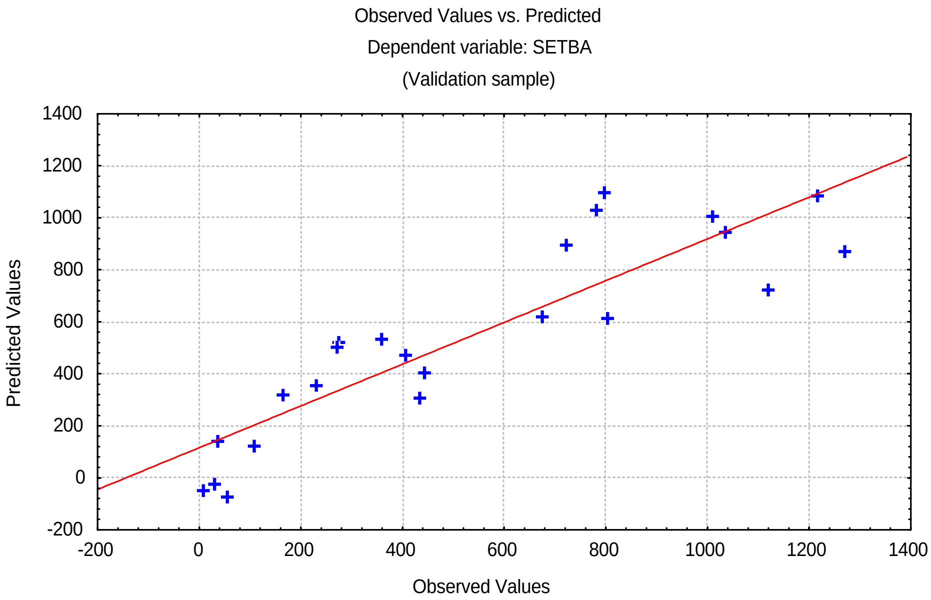
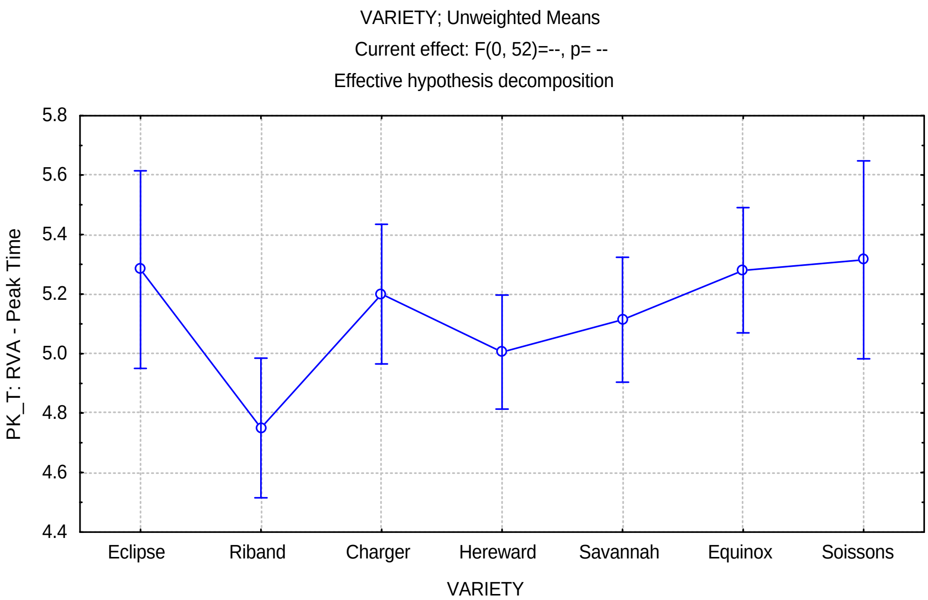
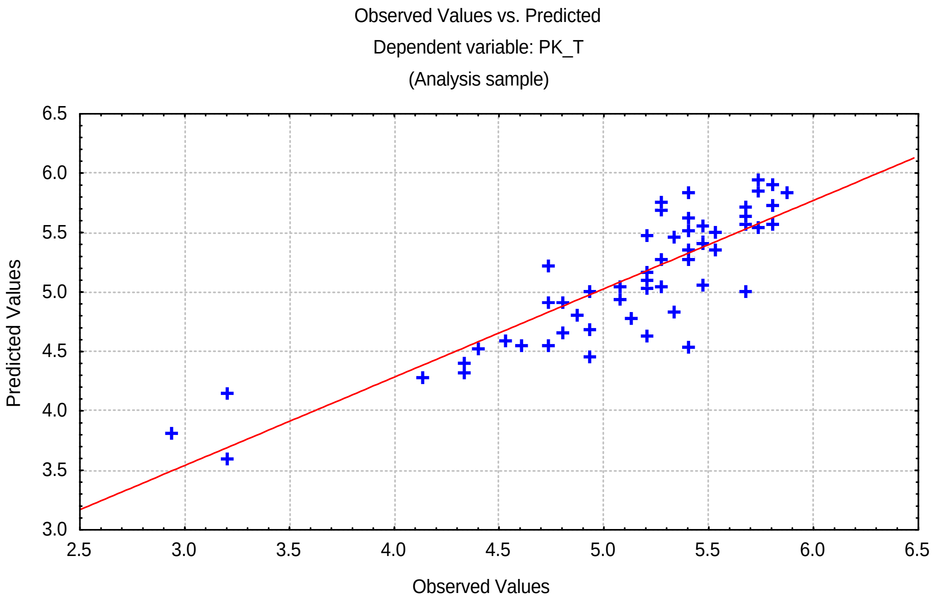


Figure G6 (a to c) **RVA Time at Peak**




Observed Values vs. Predicted
Dependent variable: PK_T
(Analysis sample)
Predicted Values
Observed Values
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5

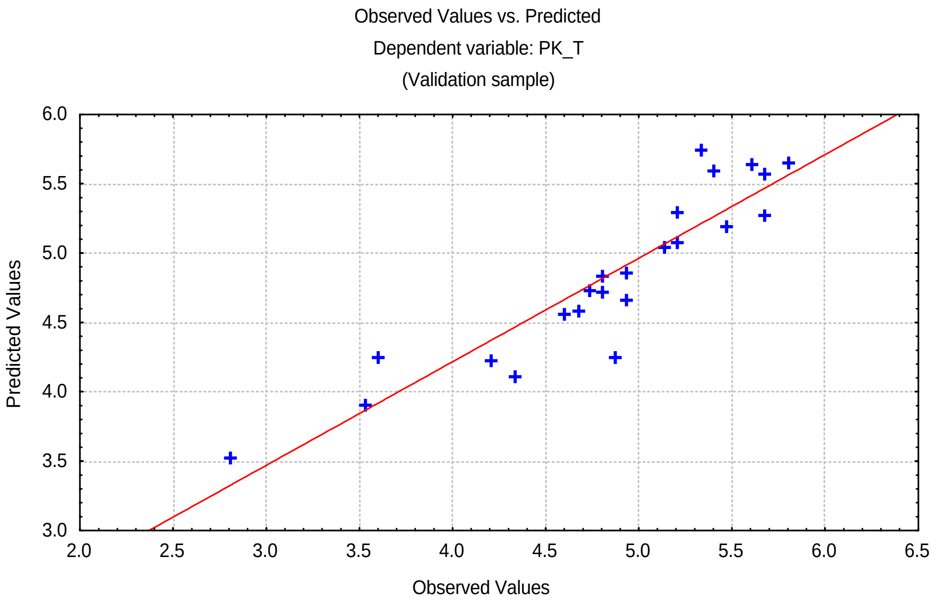
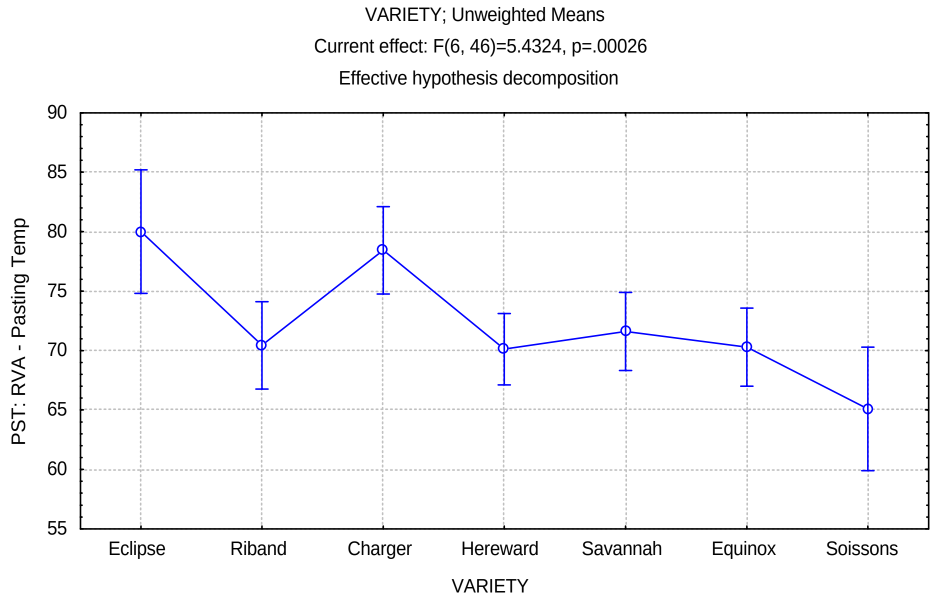
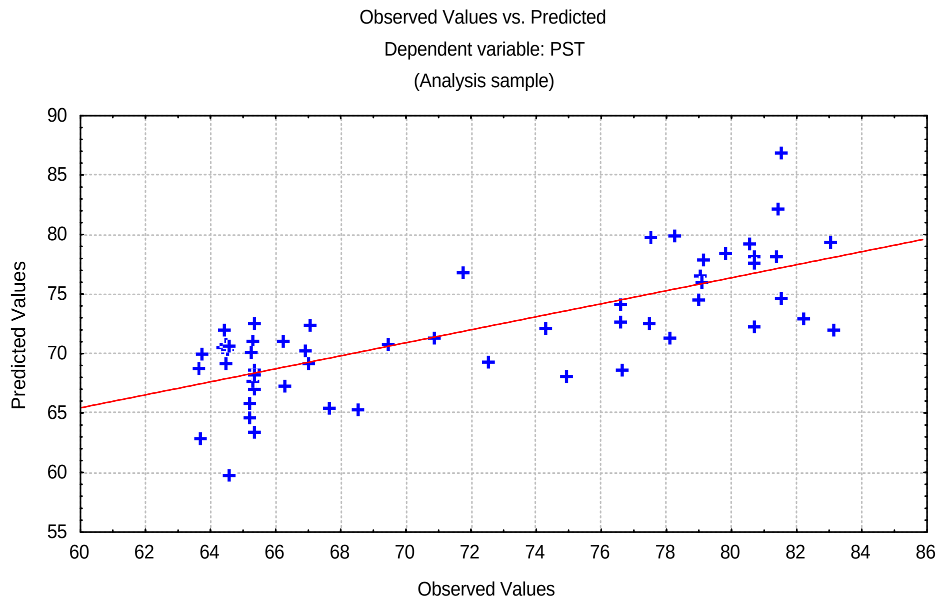

Observed Values vs. Predicted
Dependent variable: PK_T
(Validation sample)
Predicted Values
Observed Values
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5

Figure G7 (a to c) RVA Pasting Temperature





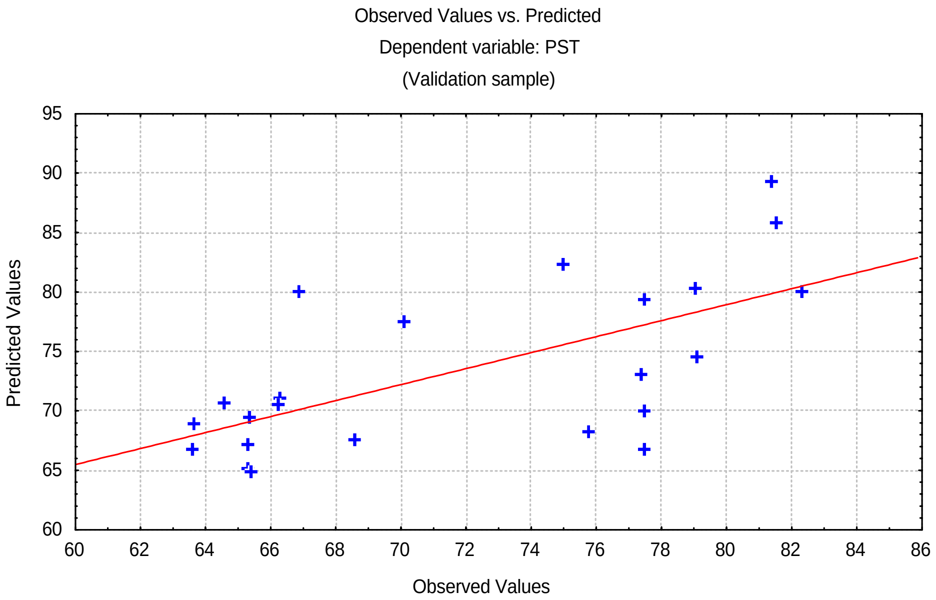
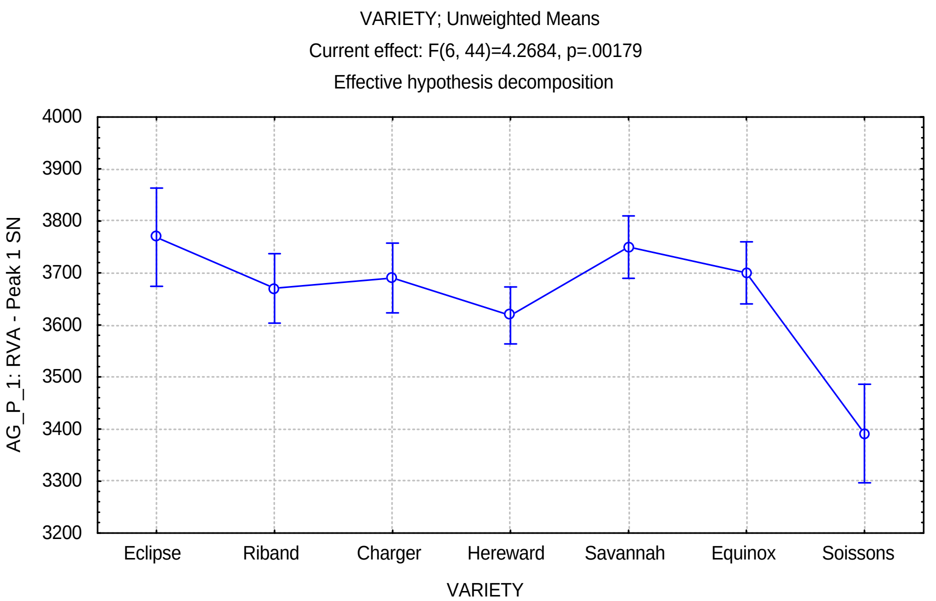
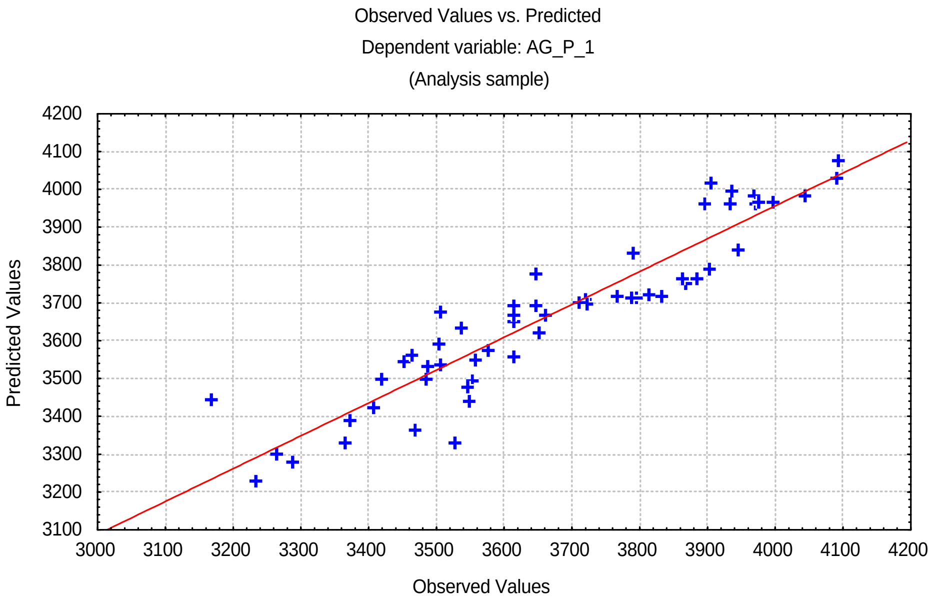


Figure G8 (a to c) **RVA Peak Viscosity with silver nitrate**




Observed Values vs. Predicted
Dependent variable: AG_P_1
(Analysis sample)
Predicted Values
4200
4100
4000
3900
3800
3700
3600
3500
3400
3300
3200
3100
3000
3100
3200
3300
3400
3500
3600
3700
3800
3900
4000
4100
4200
Observed Values

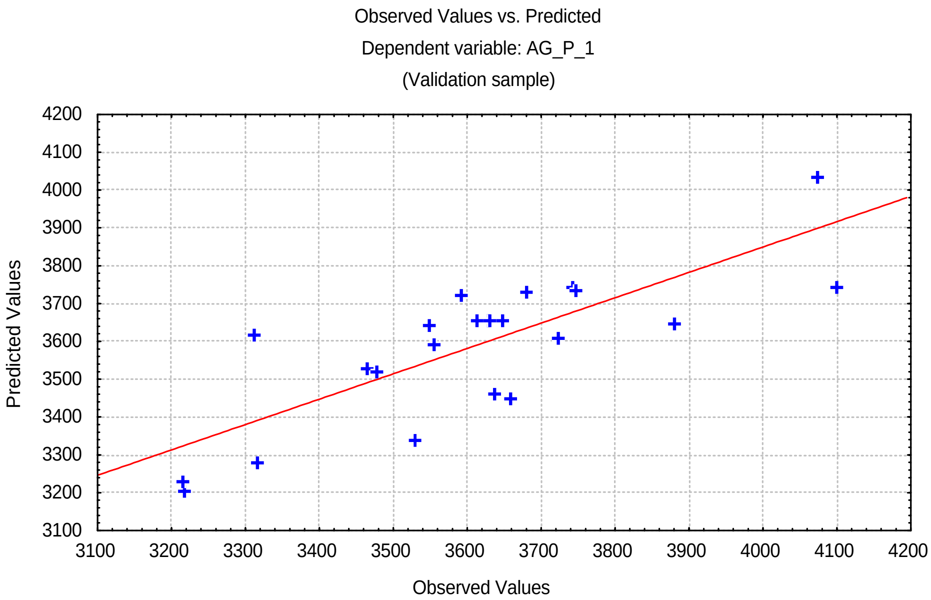
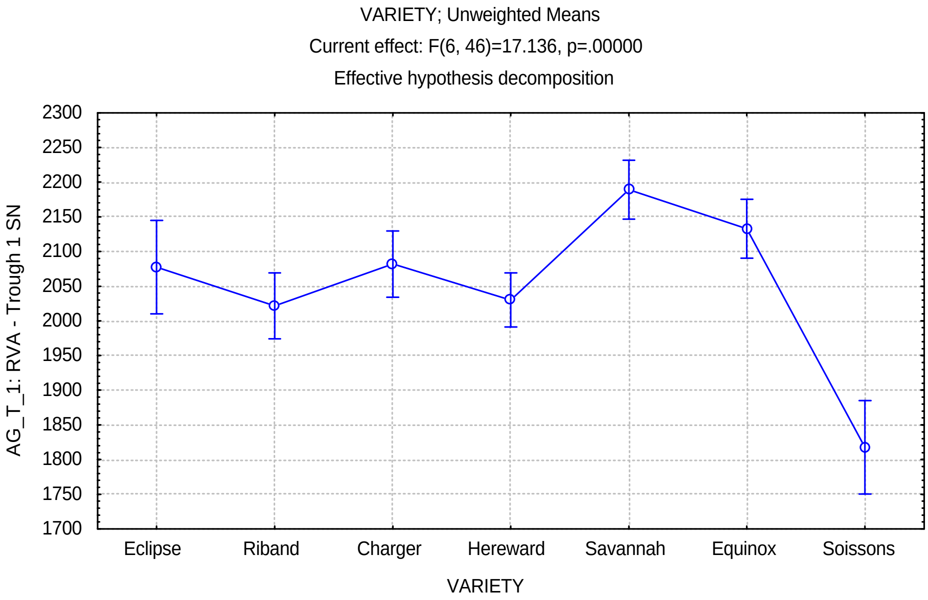
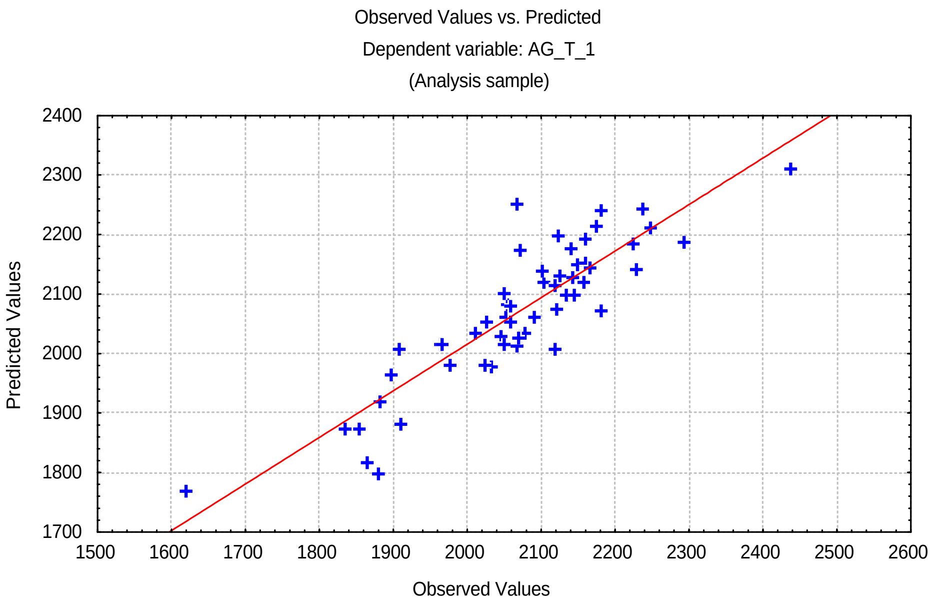

Observed Values vs. Predicted
Dependent variable: AG_P_1
(Validation sample)
Predicted Values
4200
4100
4000
3900
3800
3700
3600
3500
3400
3300
3200
3100
3100
3200
3300
3400
3500
3600
3700
3800
3900
4000
4100
4200
Observed Values

Figure G9 (a to c) **RVA Trough Viscosity with silver nitrate**





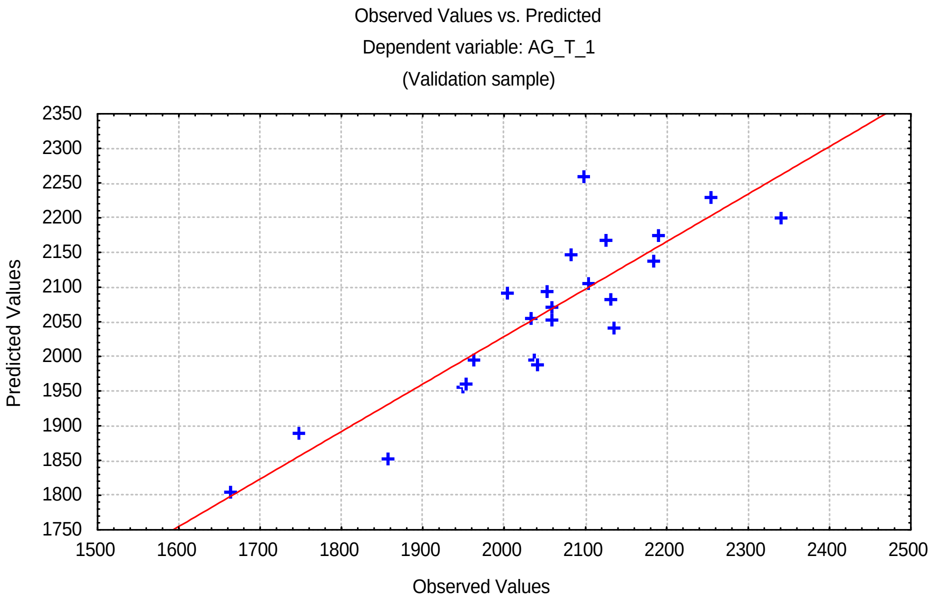
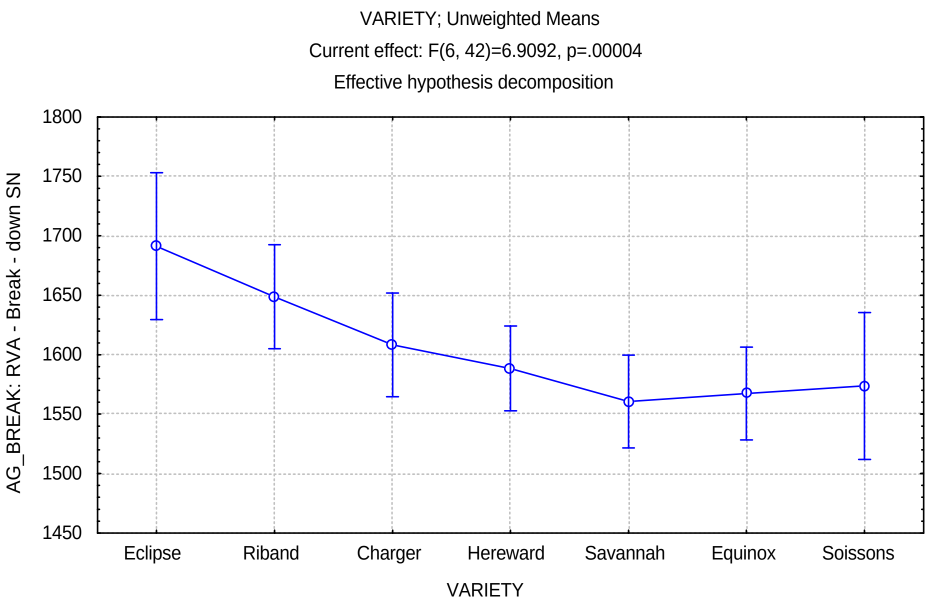
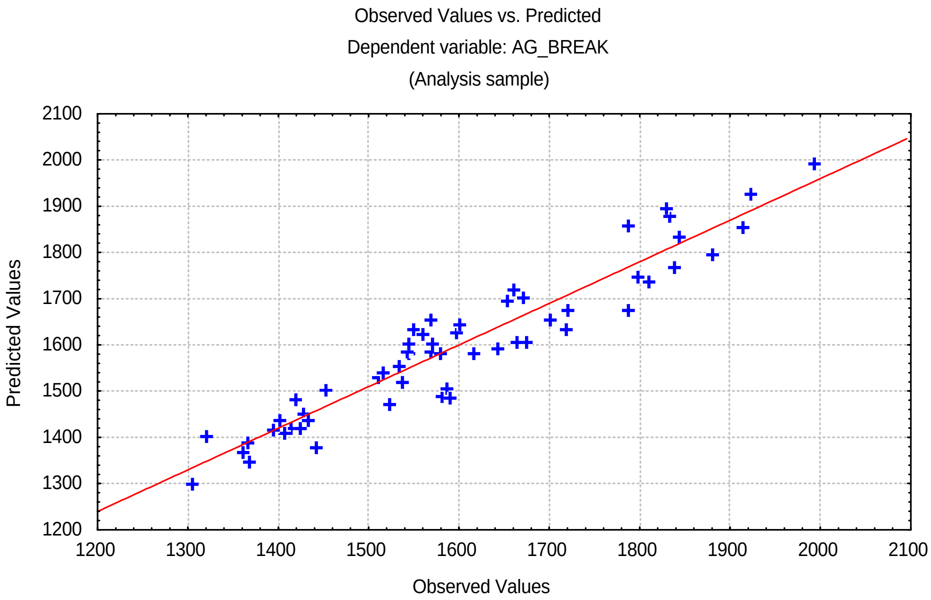


Figure G10 (a to c) **RVA Viscosity Breakdown with silver nitrate**




Observed Values vs. Predicted
Dependent variable: AG_BREAK
(Analysis sample)
Predicted Values
Observed Values
1200
1300
1400
1500
1600
1700
1800
1900
2000
2100

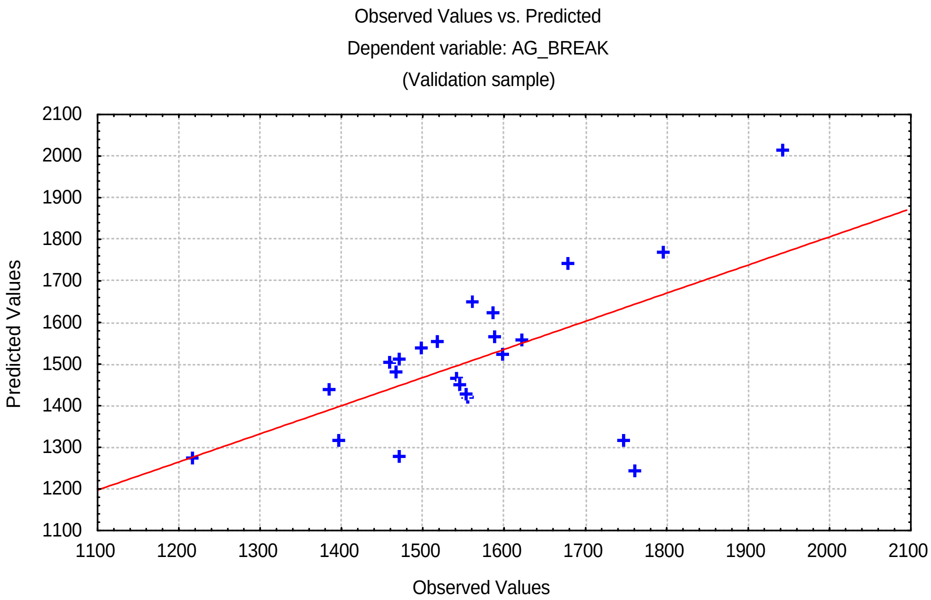
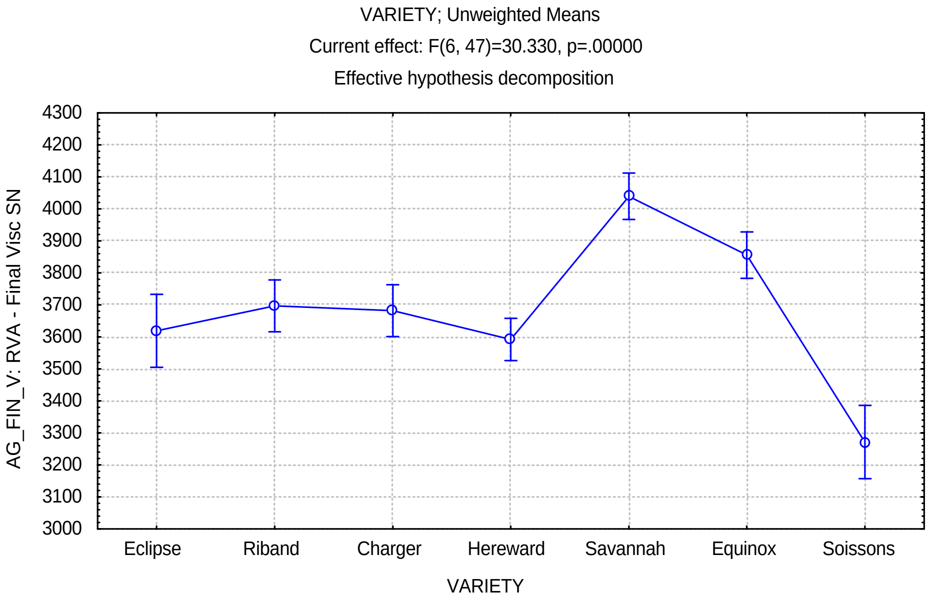
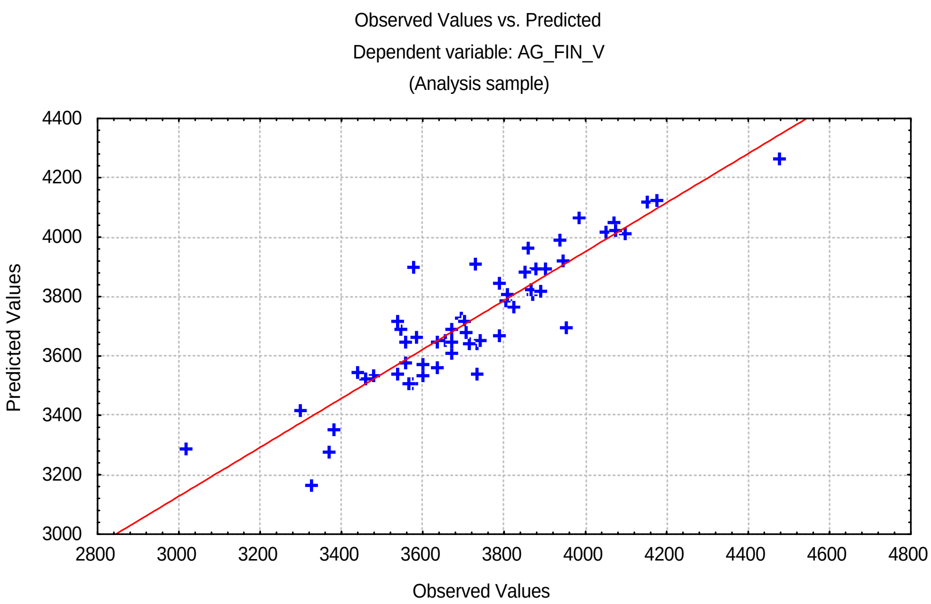

Observed Values vs. Predicted
Dependent variable: AG_BREAK
(Validation sample)
Predicted Values
Observed Values
1100
1200
1300
1400
1500
1600
1700
1800
1900
2000
2100

Figure G11 (a to c)   RVA Viscosity Breakdown with silver nitrate





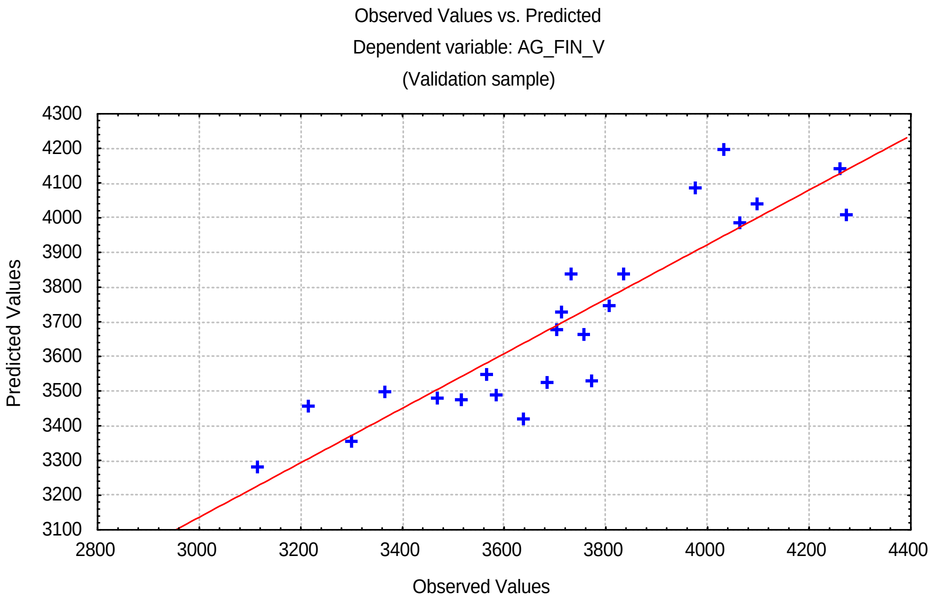
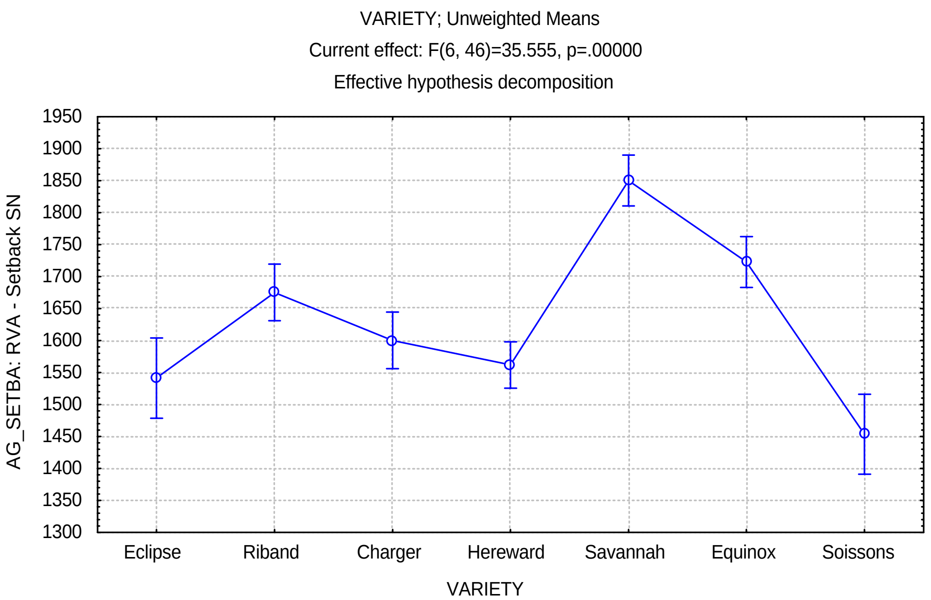
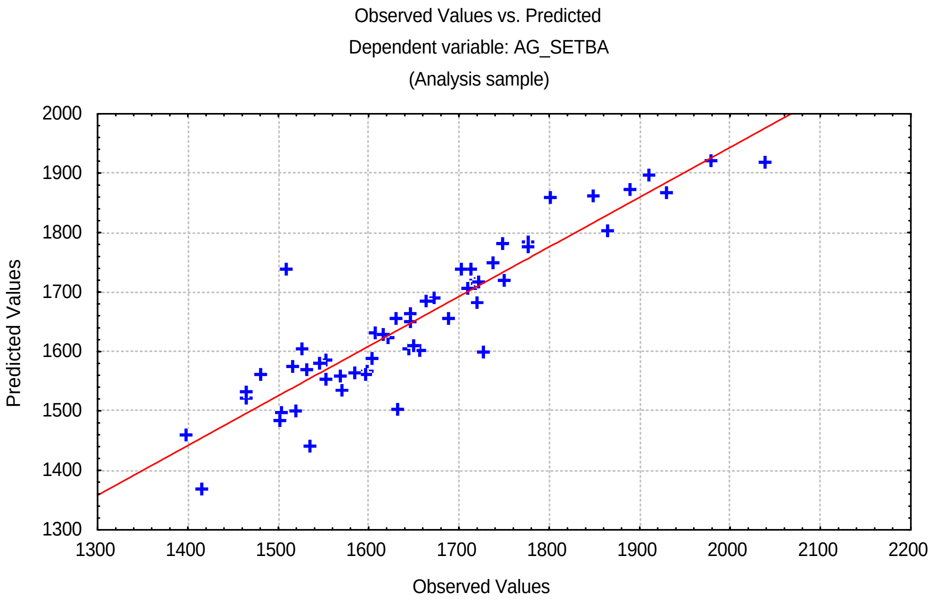


Figure G12 (a to c) RVA Viscosity Setback with silver nitrate




Observed Values vs. Predicted
Dependent variable: AG_SETBA
(Analysis sample)
Predicted Values
Observed Values
2000
1900
1800
1700
1600
1500
1400
1300
1300
1400
1500
1600
1700
1800
1900
2000
2100
2200

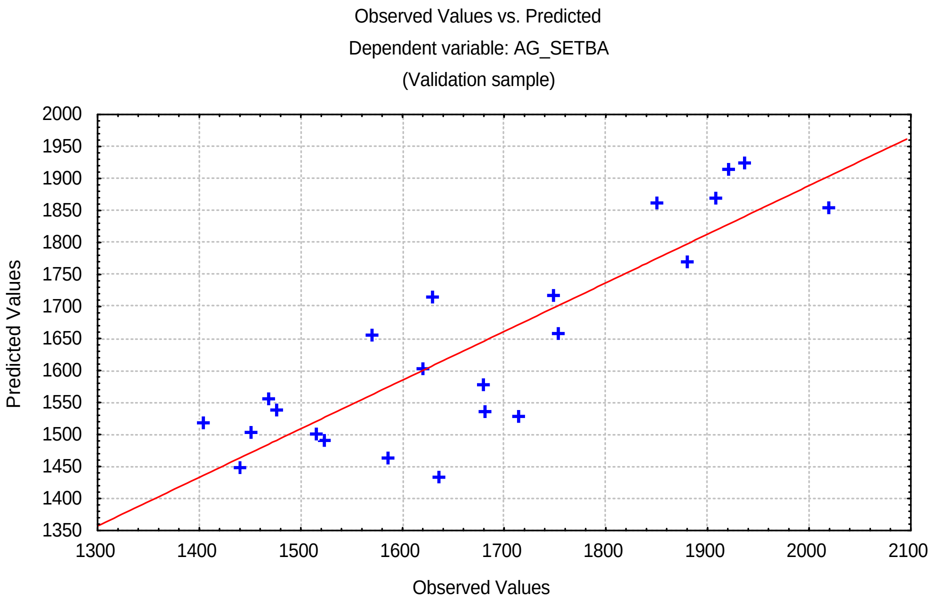
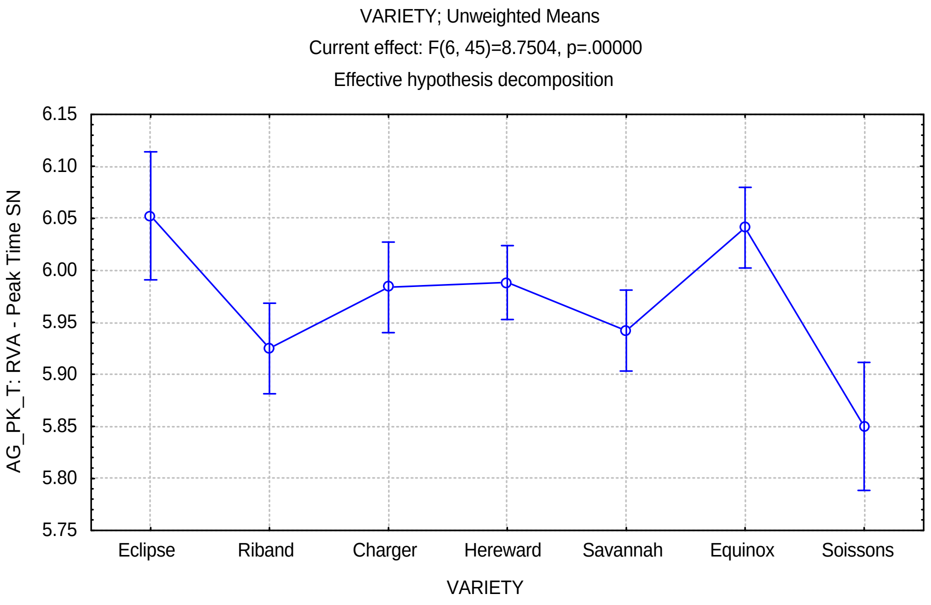
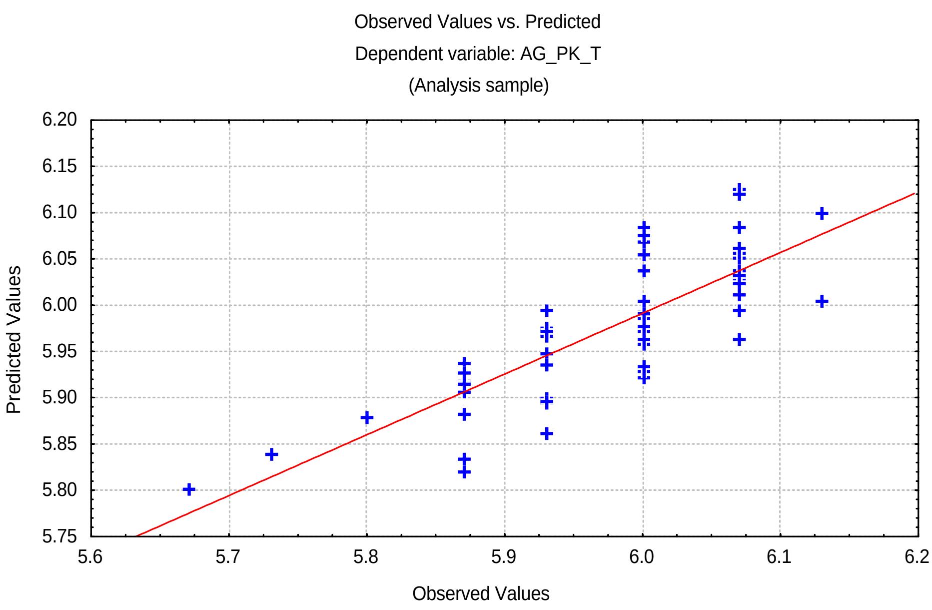

Observed Values vs. Predicted
Dependent variable: AG_SETBA
(Validation sample)
Predicted Values
Observed Values
2000
1950
1900
1850
1800
1750
1700
1650
1600
1550
1500
1450
1400
1350
1300
1400
1500
1600
1700
1800
1900
2000
2100

Figure G13 (a to c) RVA Time at Peak with silver nitrate





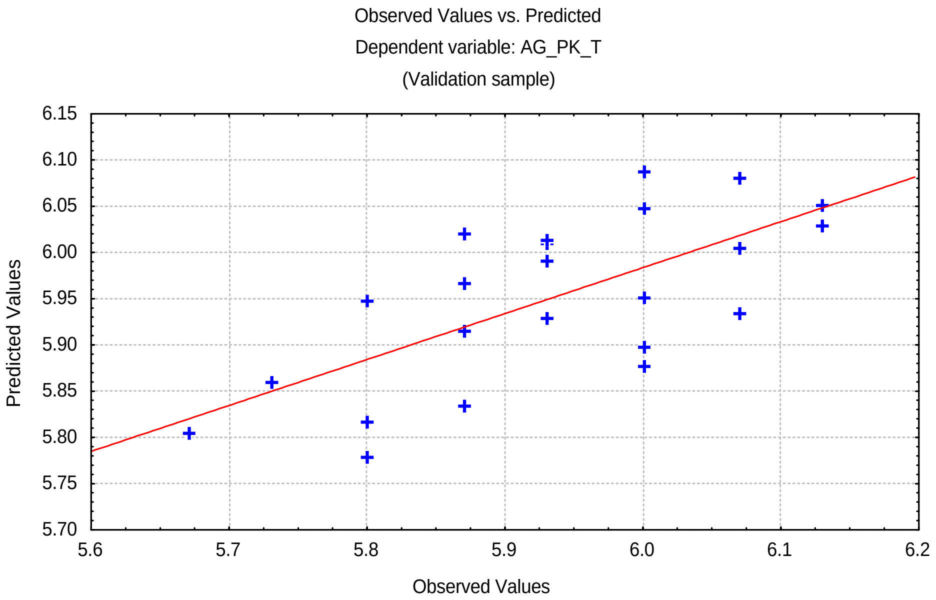
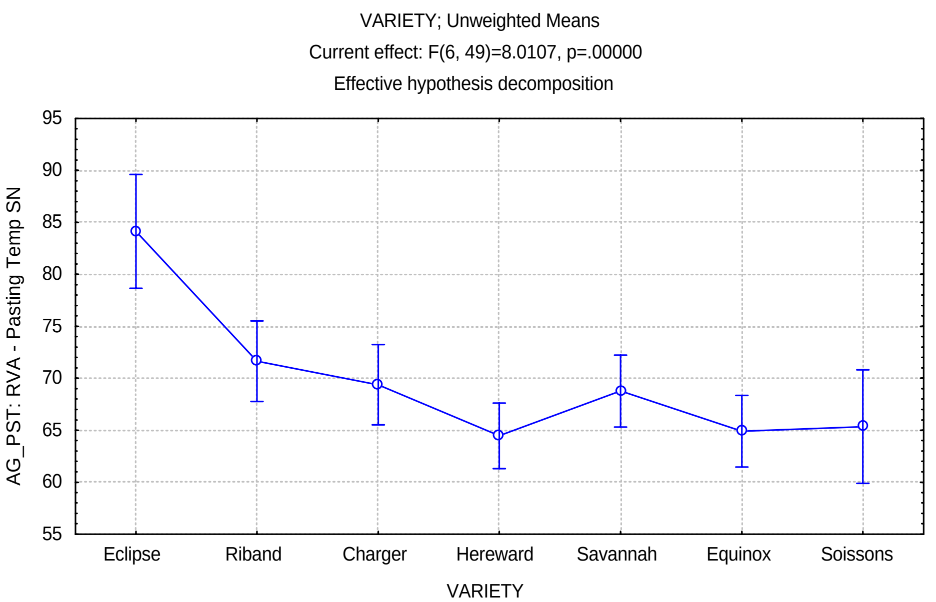
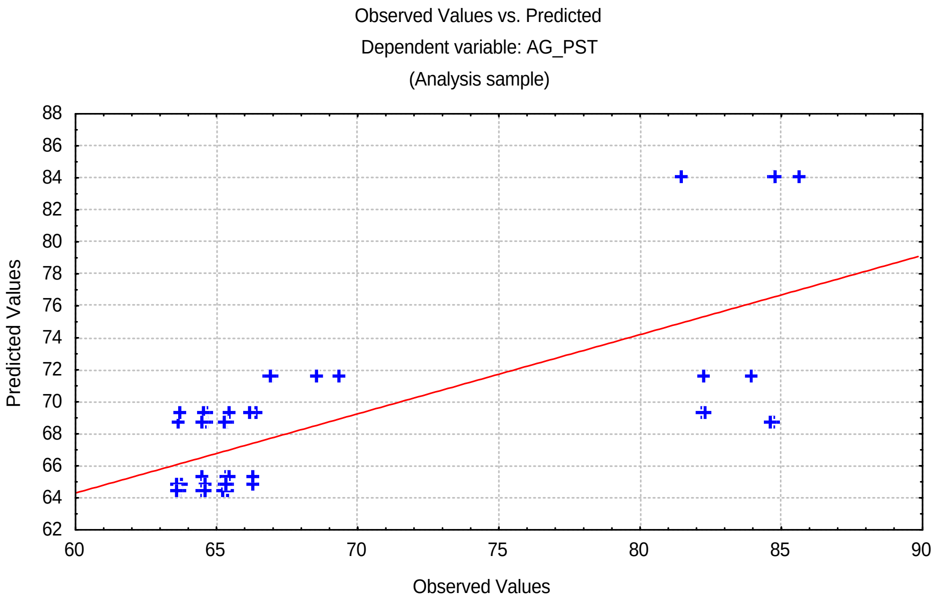


Figure G14 (a to c) **RVA Pasting Temperature with silver nitrate**




Observed Values vs. Predicted
Dependent variable: AG_PST
(Analysis sample)
Predicted Values
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
65
70
75
80
85
90
Observed Values

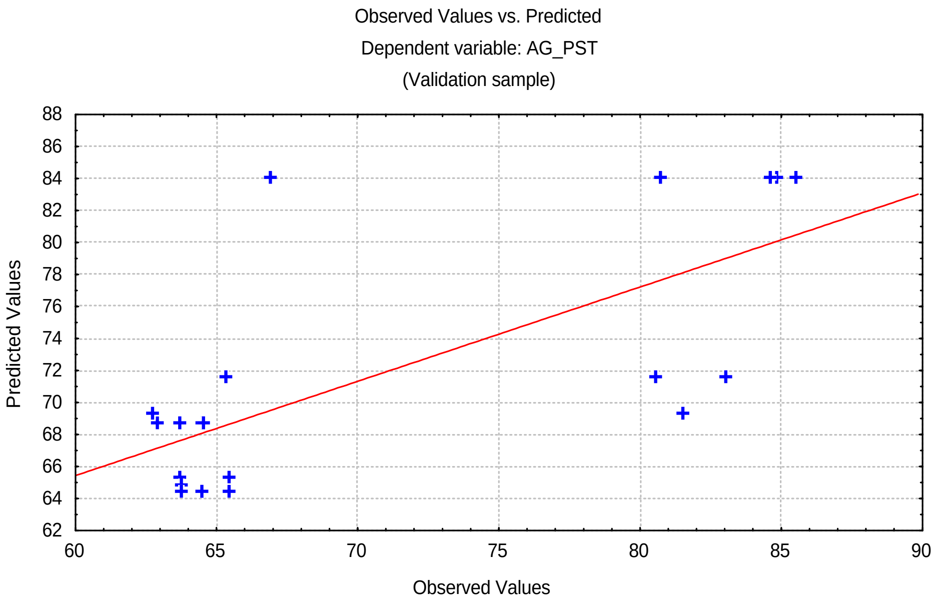
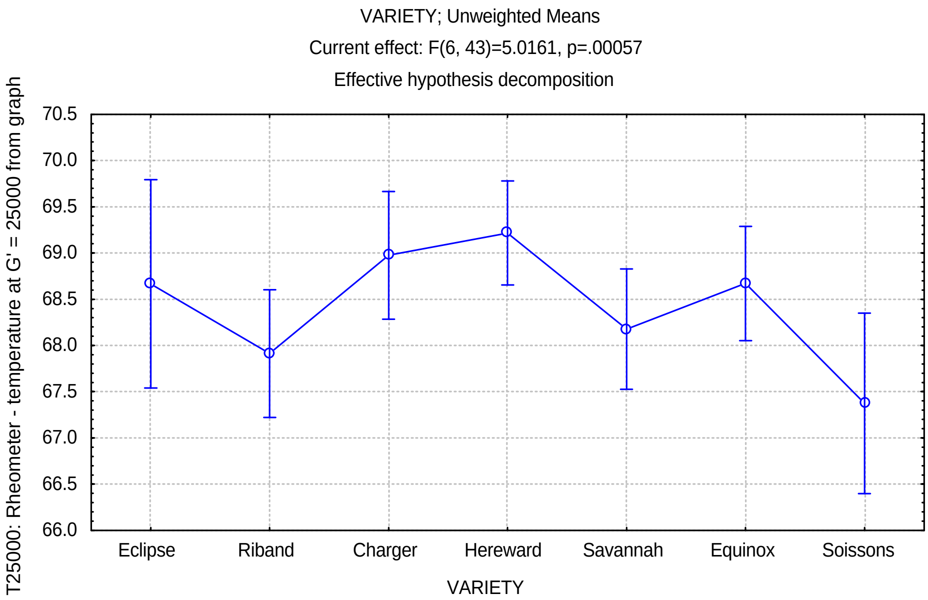
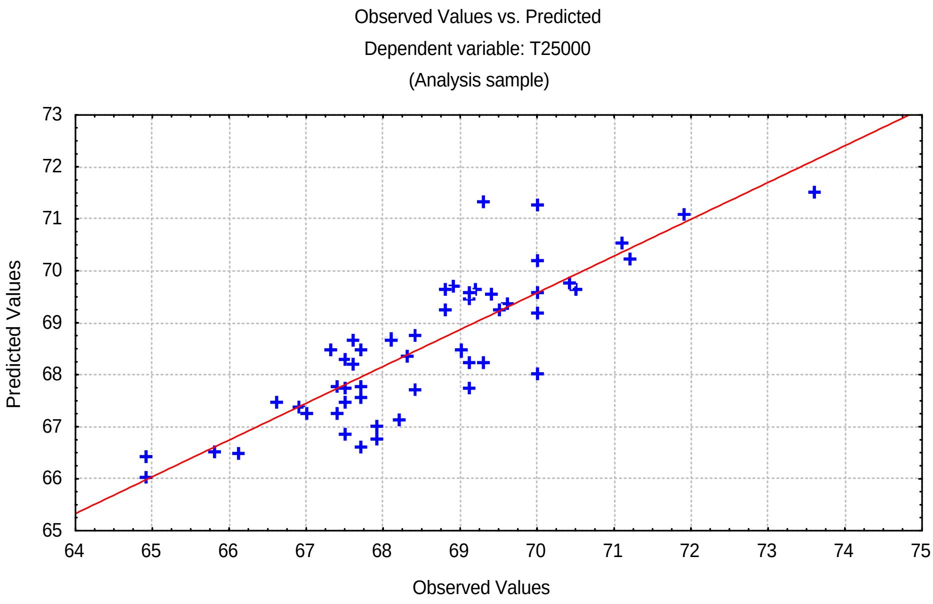

Observed Values vs. Predicted
Dependent variable: AG_PST
(Validation sample)
Predicted Values
88
86
84
82
80
78
76
74
72
70
68
66
64
62
60
65
70
75
80
85
90
Observed Values

Figure G15 (a to c) Temperature at which G'





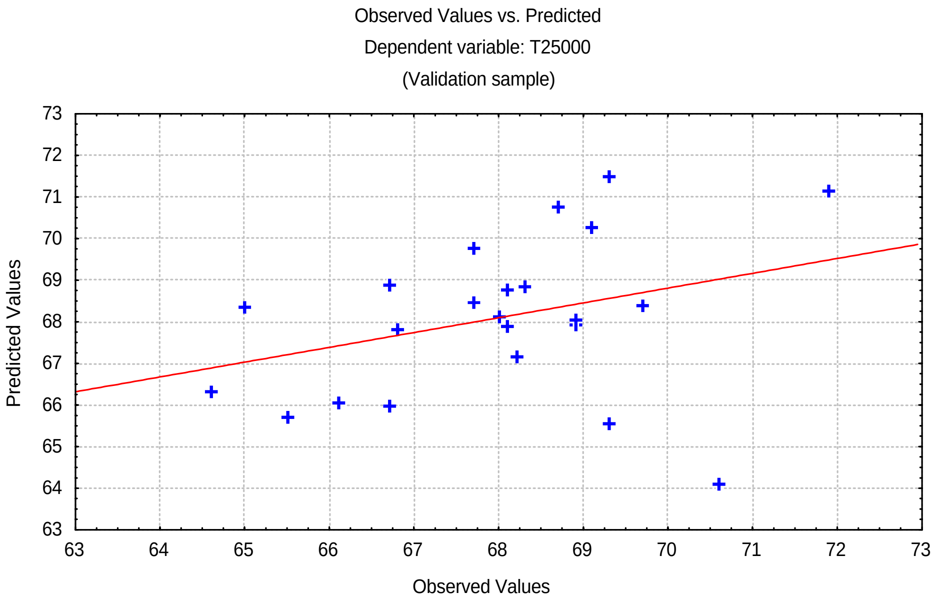
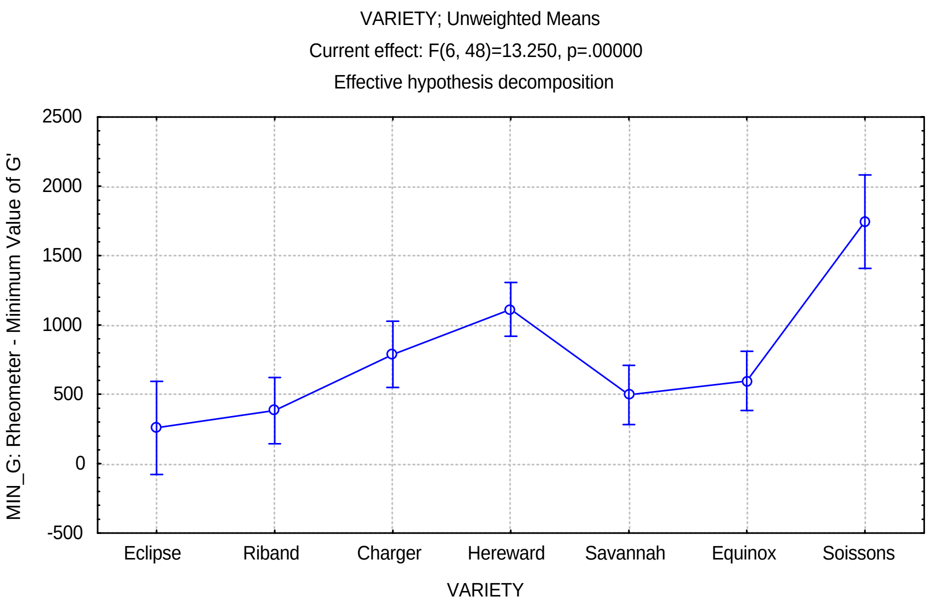
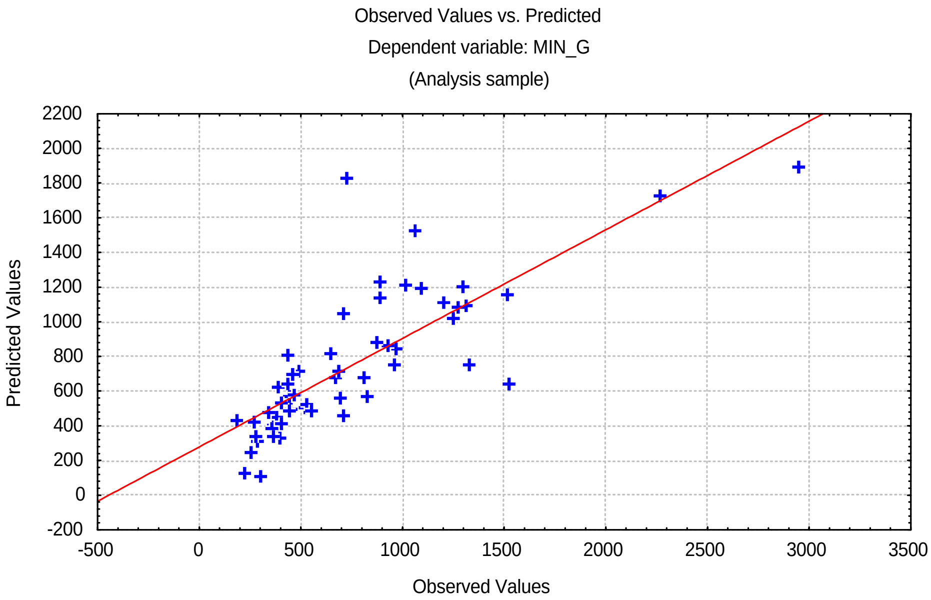


Figure G16 (a to c) **Minimum Value of G'**




Observed Values vs. Predicted
Dependent variable: MIN_G
(Analysis sample)
Predicted Values
Observed Values
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
-500
0
500
1000
1500
2000
2500
3000
3500

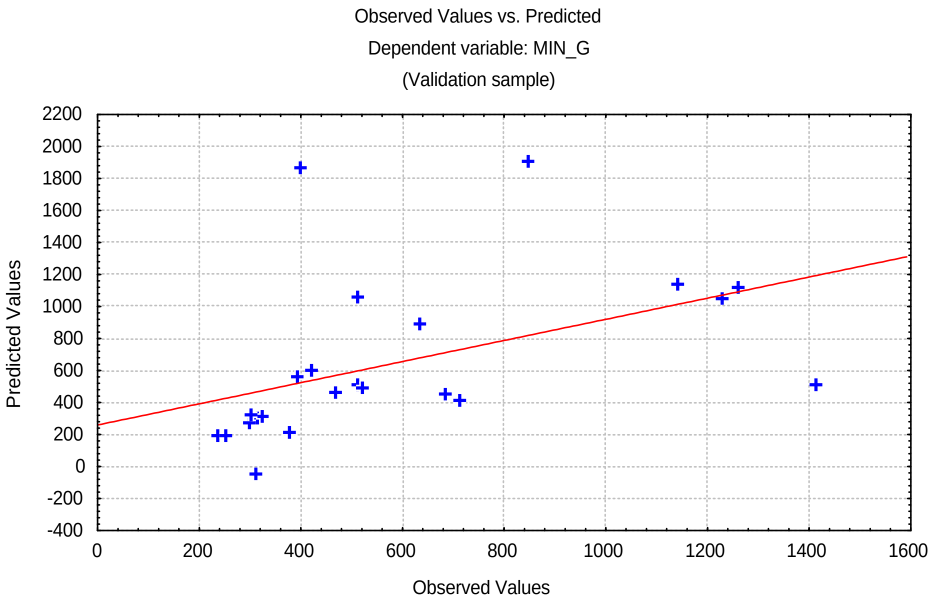
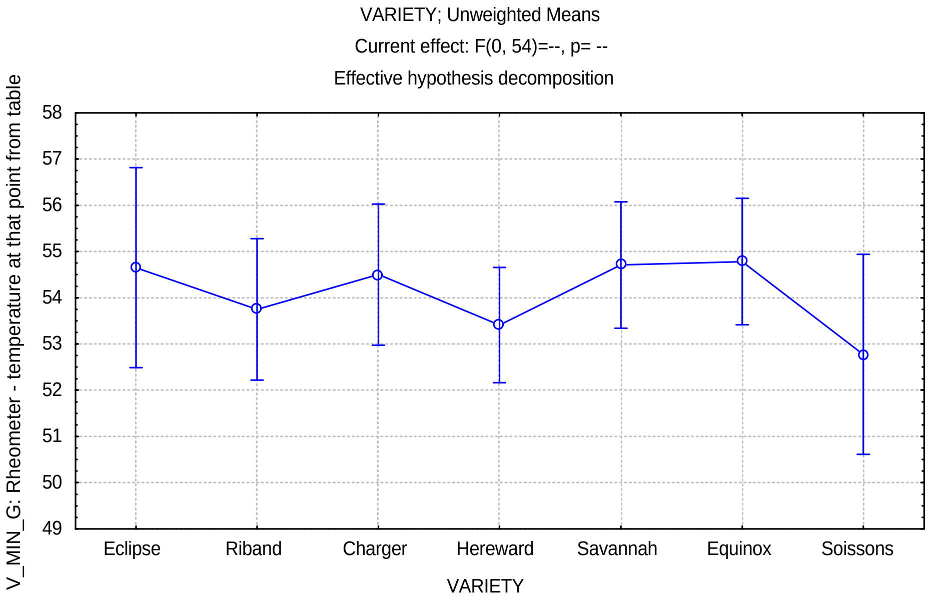
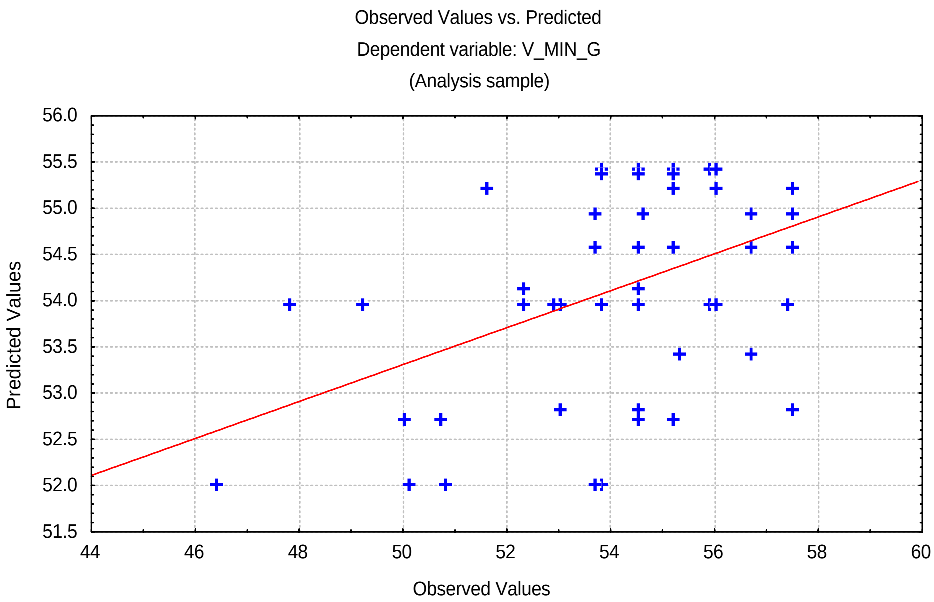

Observed Values vs. Predicted
Dependent variable: MIN_G
(Validation sample)
Predicted Values
Observed Values
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
-400
0
200
400
600
800
1000
1200
1400
1600

Figure G17 (a to c) Temperature at Minimum Value of G'





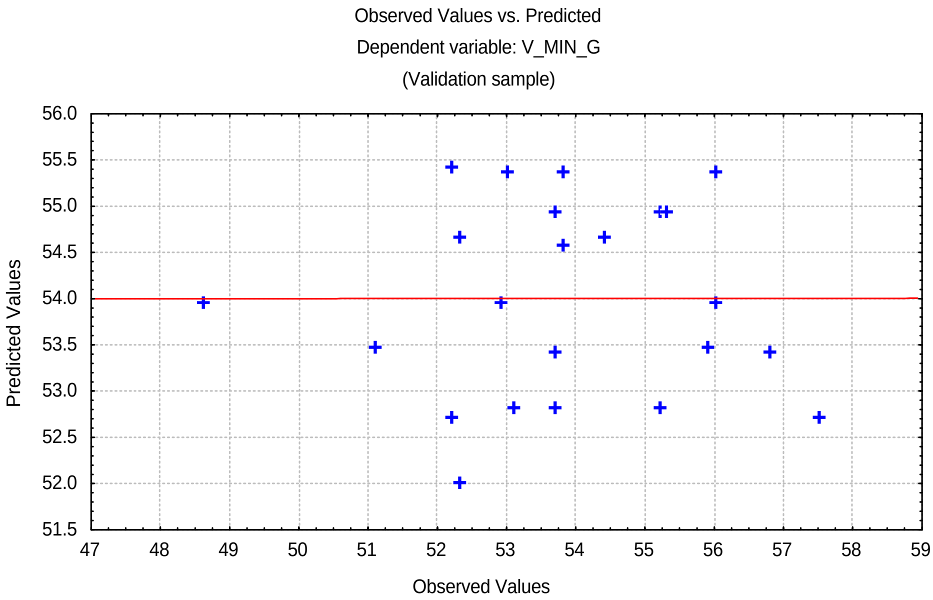
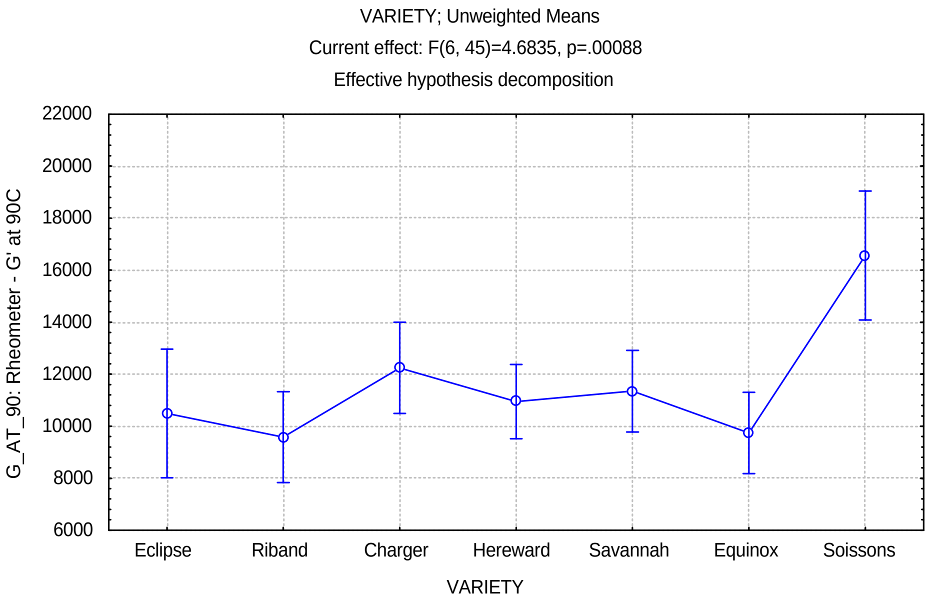
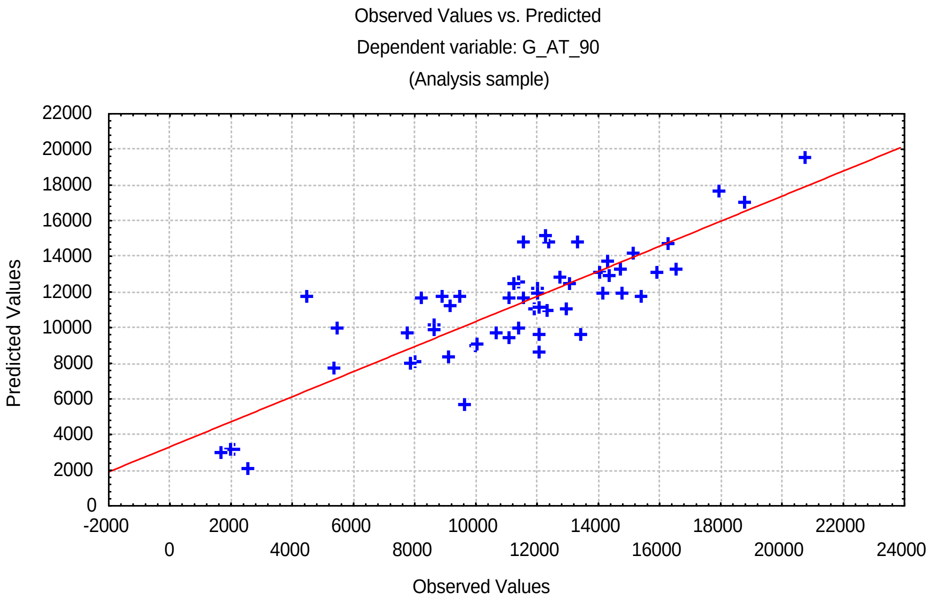
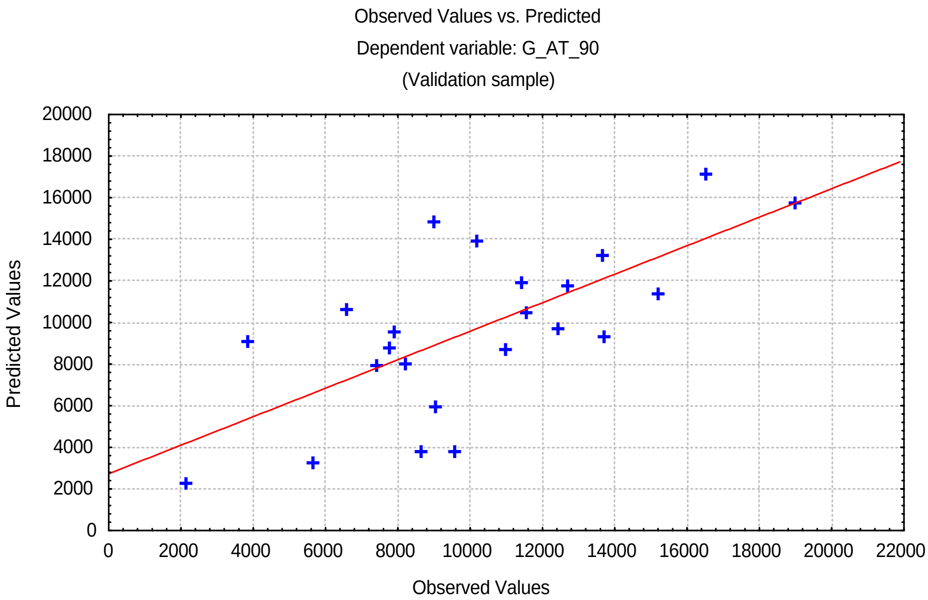


Figure G18 (a to c) **G'at 90°C**



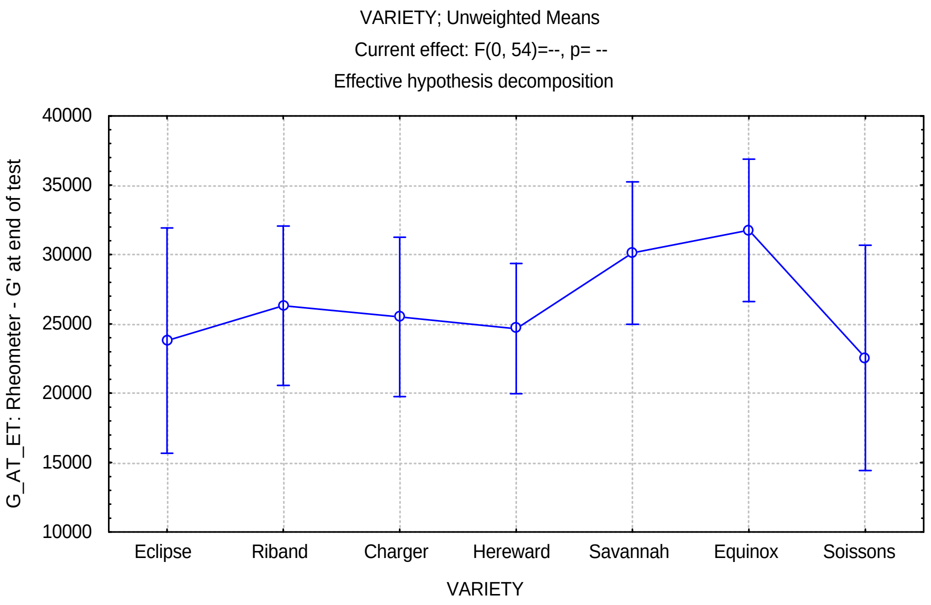


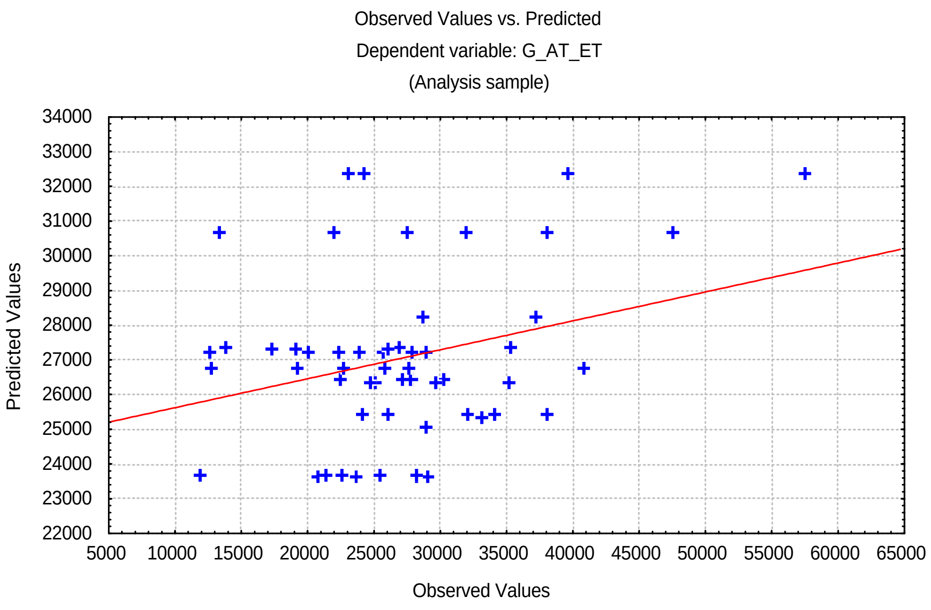
Observed Values vs. Predicted
Dependent variable: G_AT_90
(Analysis sample)
Predicted Values
Observed Values



Observed Values vs. Predicted
Dependent variable: G_AT_90
(Validation sample)
Predicted Values
Observed Values

Figure G19 (a to c) G' at end of test





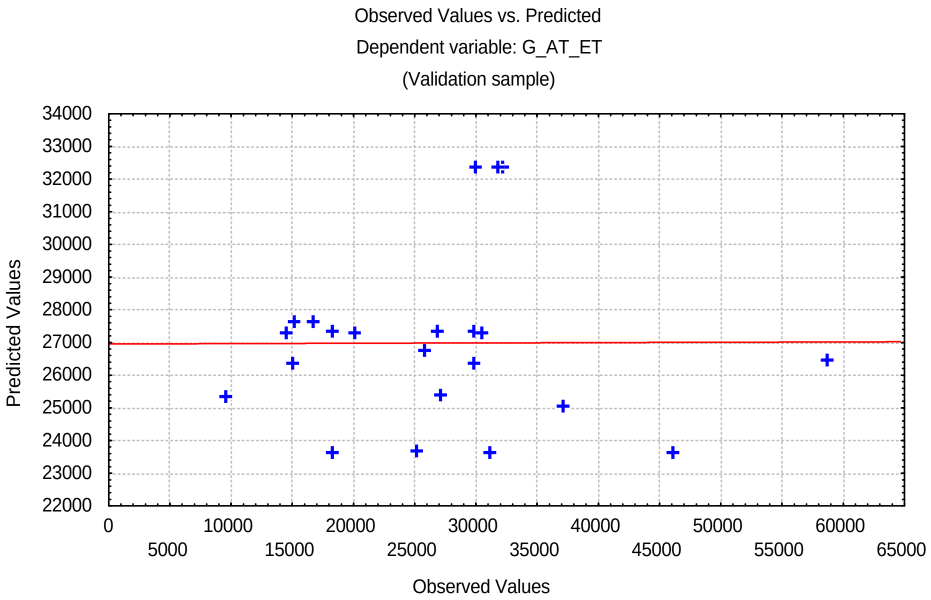
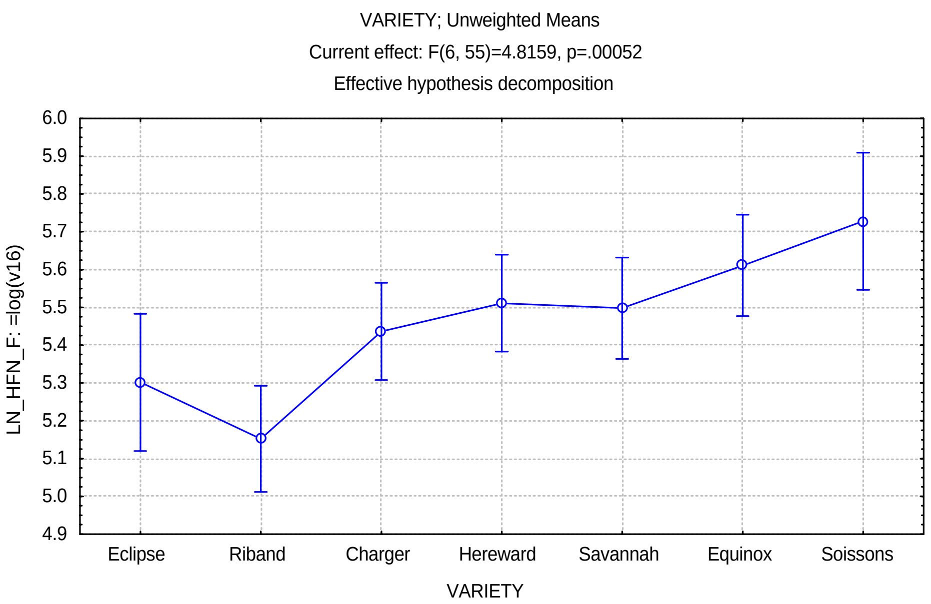
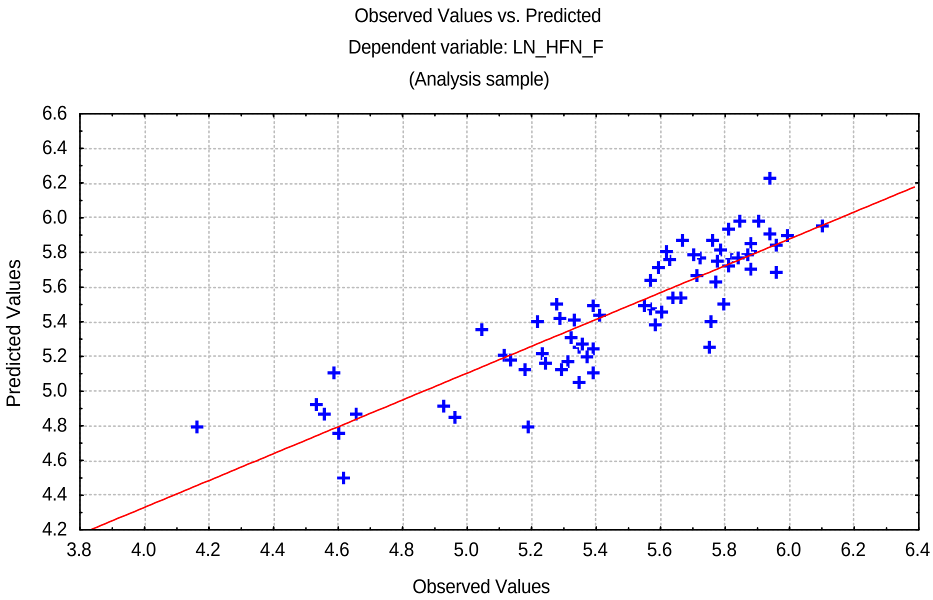
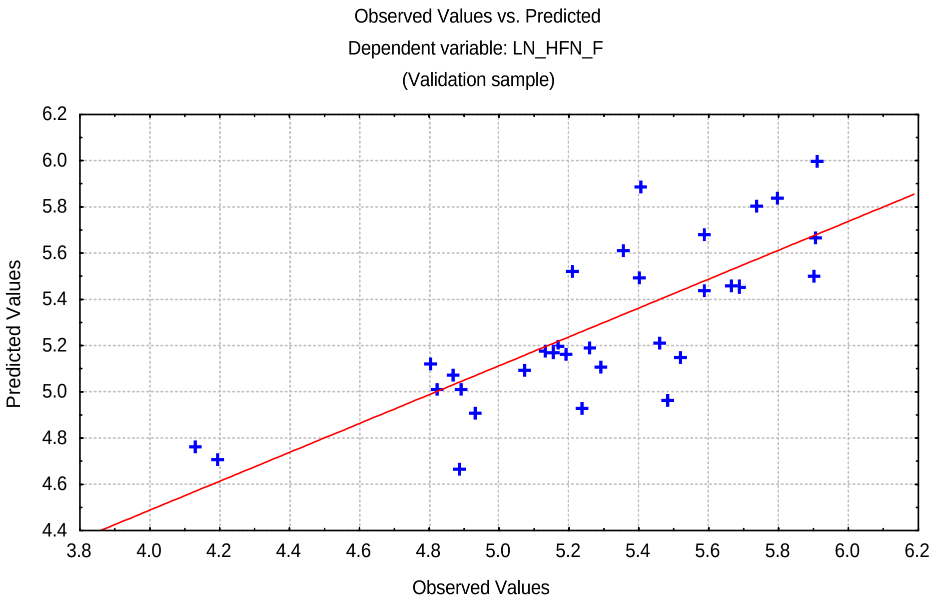


Figure G20 (a to c) **Ln (Hagberg Falling Number)**




Observed Values vs. Predicted
Dependent variable: LN_HFN_F
(Analysis sample)
Predicted Values
Observed Values


Observed Values vs. Predicted
Dependent variable: LN_HFN_F
(Validation sample)
Predicted Values
Observed Values