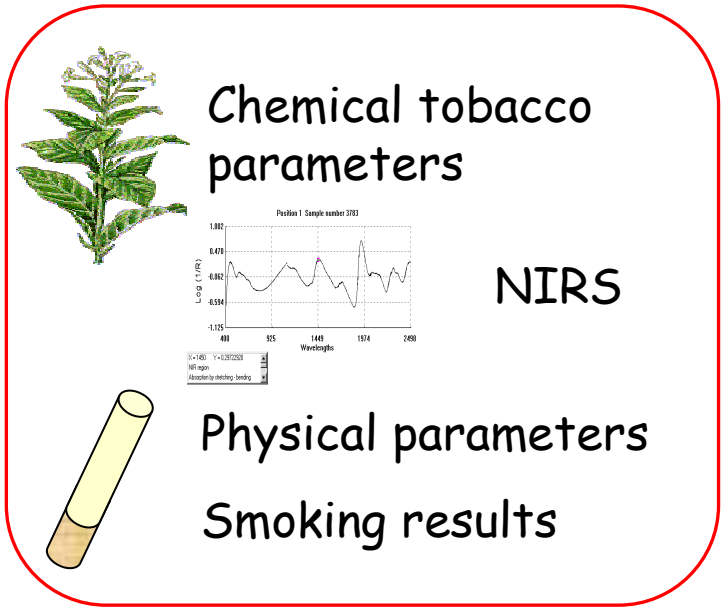
Prediction of a biological variable (Ames test on TPM) and of a chemical variable (TSNAs in smoke) according to the tobacco's chemical and physical characteristics

M. Bouzige, B. Vidal, JP. Biesse,
C. Jousserand, V. Marchand, B. Dumery

ALTADIS RESEARCH CENTER
Fleury les Aubrais, FRANCE

Introduction

- Chemical analyses of special analytes or biological tests on smoke are rather complex and time consuming
- Aim of this study :
  - Define predictive models based on fast analyses in order to give estimates to the blenders easily
  - Study the link between tobacco properties and special analytes contents or biological activity



Material

- 105 tobaccos of different types
  - Burley : 28 tobaccos, 9 countries
  - Flue Cured : 37 tobaccos, 8 countries
  - Sun Cured : 13 tobaccos, 5 countries
  - Dark : 27 tobaccos, 8 countries

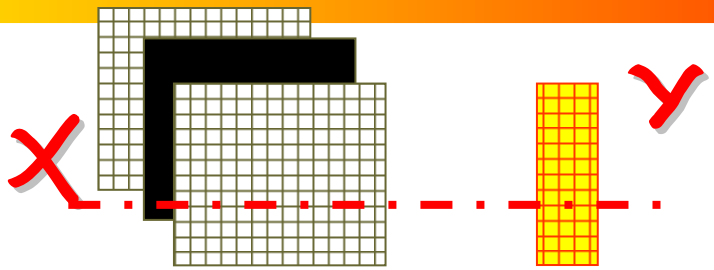
- Samples are representative of the tobacco market (origin, stalk position, maturity, quality)

- Production of cigarettes for each tobacco sample
  - All the cigarettes are made with the same NTM
  - The cigarettes have the same draw resistance

Procedure

105 Tobaccos
105 Cigarettes

Analyses



Choice based on NIRS results
Closest Neighbours method

Calibration Set
72 tobaccos

Validation Set
33 tobaccos

Linear,
Partial Least Square
Regressions

Adjustment quality
Cross validation
Error of estimation....

Error of
prediction

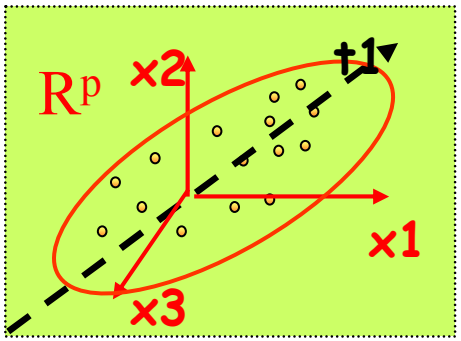
Models of prediction

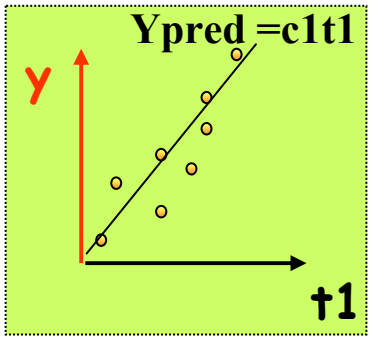
Altadis

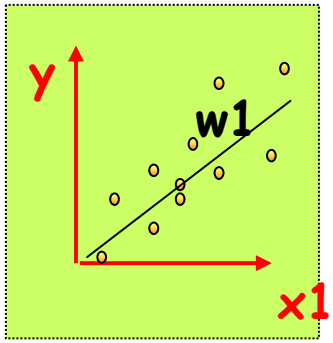
Partial Least Square Regression

- Link a block of explanatory variables to one or many variables to be explained

- Principle : find a subspace which gives at the same time the best description of the individuals according to X values and the best prediction of Y





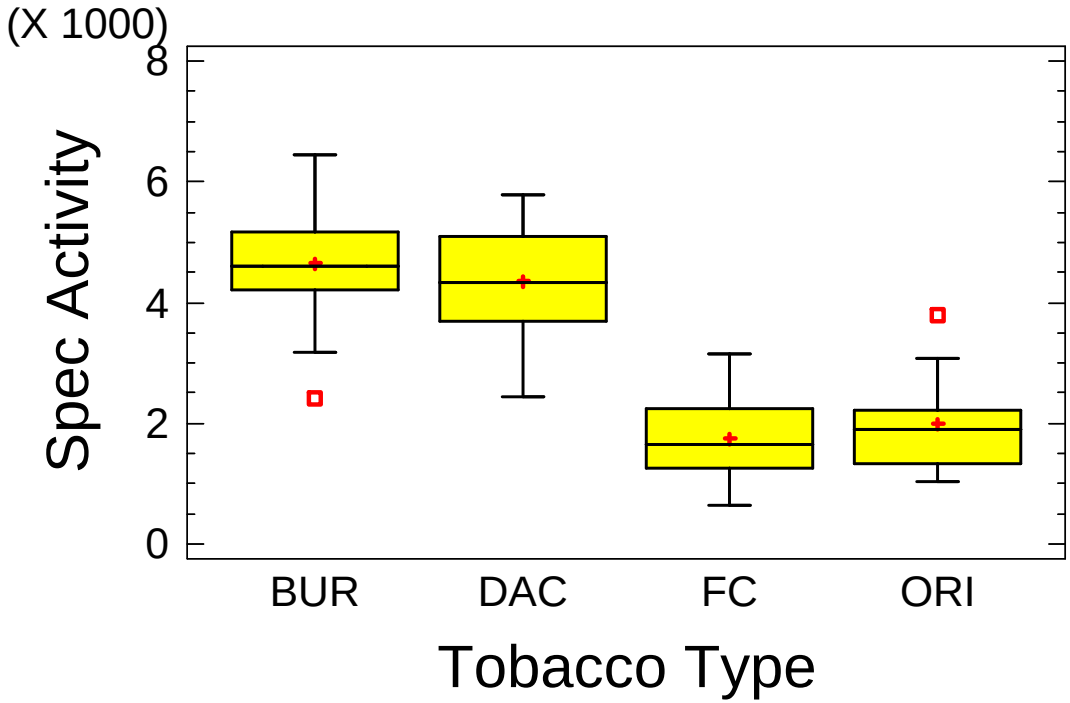


- Variables can be highly correlated and more numerous than the observations
- There can be missing values

Altaïs

Specific activity overview

Specific activity (rvts/mg TPM) vs tobacco type



Ames test
Strain TA98
S9 activation

Specific Activity	B	DAC	FC	Oriental
Min	2416	2444	652	1032
Max	6438	5792	3141	3802
Mean	4641	4362	1751	2006

Altadis

Comparison of different models

- All types of tobaccos

Adjustment quality

Cross Validation Robustness

Error of estimation

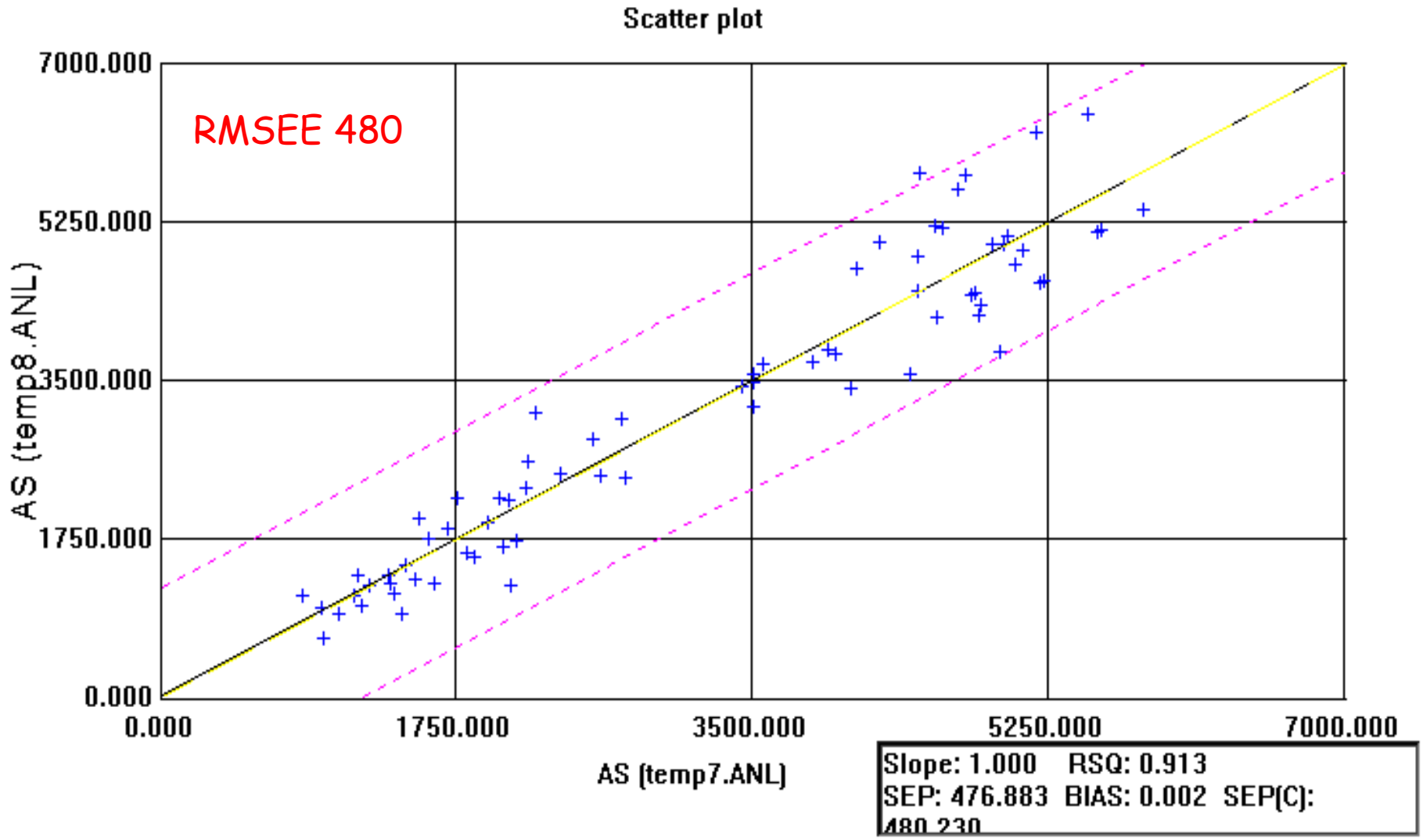
Error of prediction

X variables	R2Y	Q2	RMSEE	RMSEP
NIRS (MPLS)	0.91	0.83	480	430
NIRS	0.85	0.80	648	503
Chemical (1)	0.84	0.83	653	555
N (linear regression)	0.84	0.84	655	675
Chemical (2)	0.82	0.82	695	588
Chemical & Smoking	0.79	0.79	746	632
Physical	0.59	0.56	1058	907

- Analytical error of measurement : ~ 400 rvts/mg of NFDPM

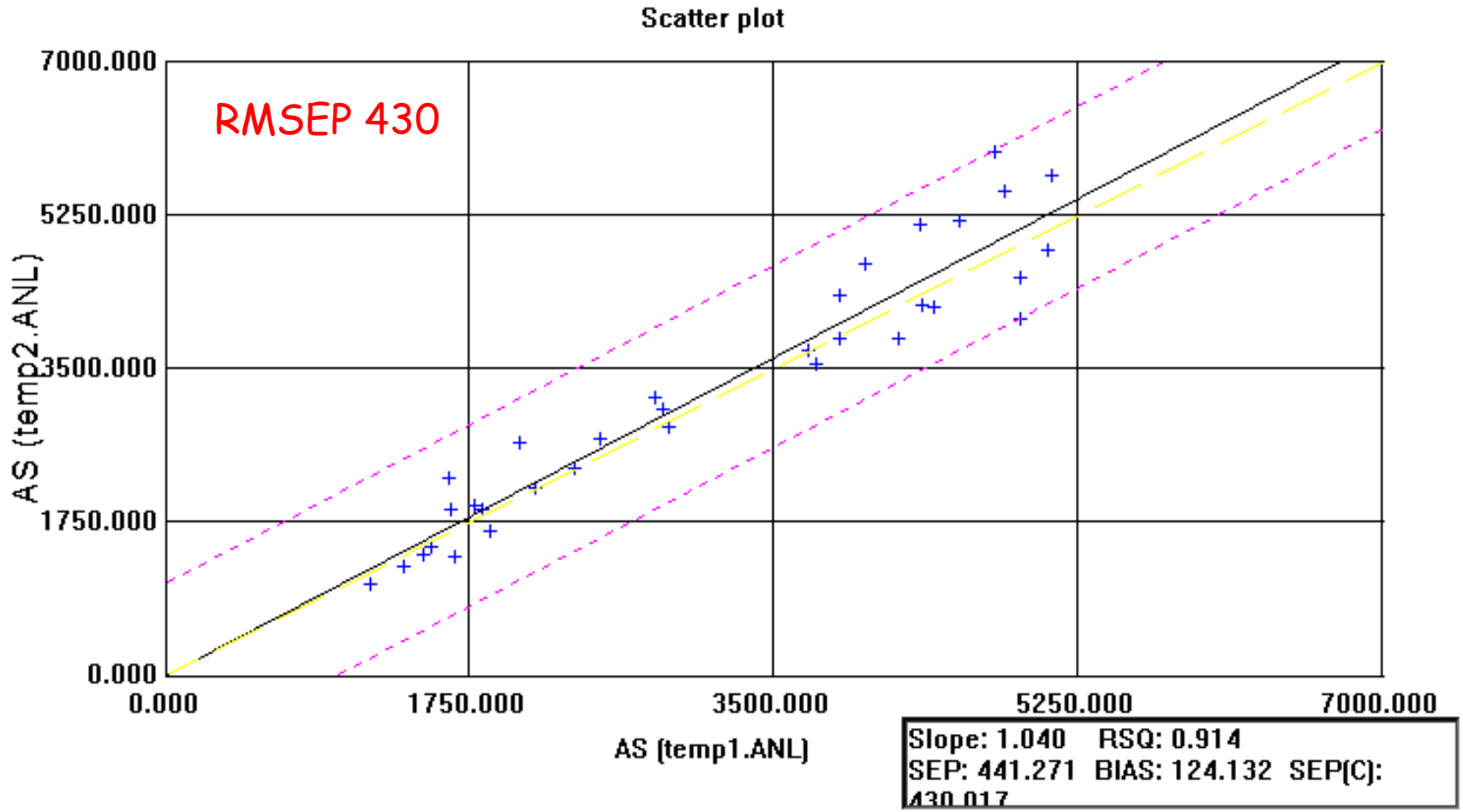
Altadis

Prediction of Specific Activity with NIRS : Calibration Set



Altadis

Prediction of Specific Activity with NIRS : Validation Set



NIRS is an easy and quick method to forecast the specific activity of a tobacco

Altadis

Specific Activity : Link with Chemistry

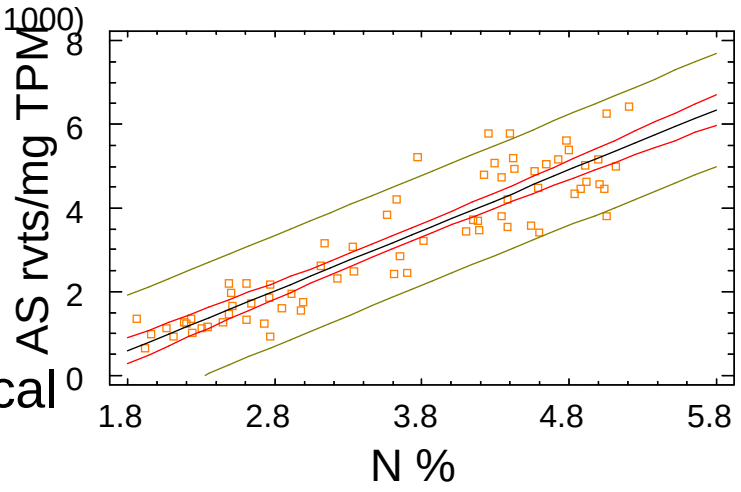
- All types of tobaccos

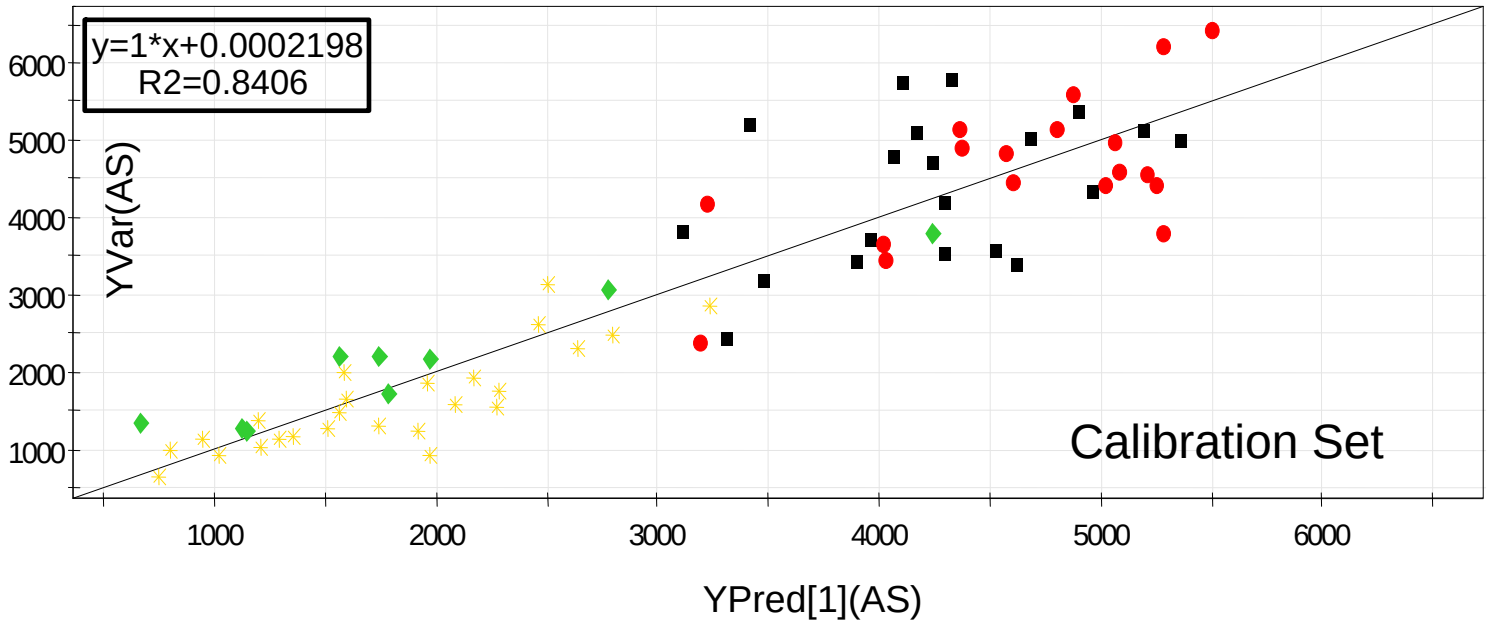
Good relationship with Nitrogen

R2 = 0.84

RMSEE = 655 rvts/mg TPM

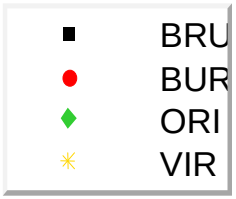
No significant improvement with extra chemical compounds (RMSEP 555 rvts/mg TPM)





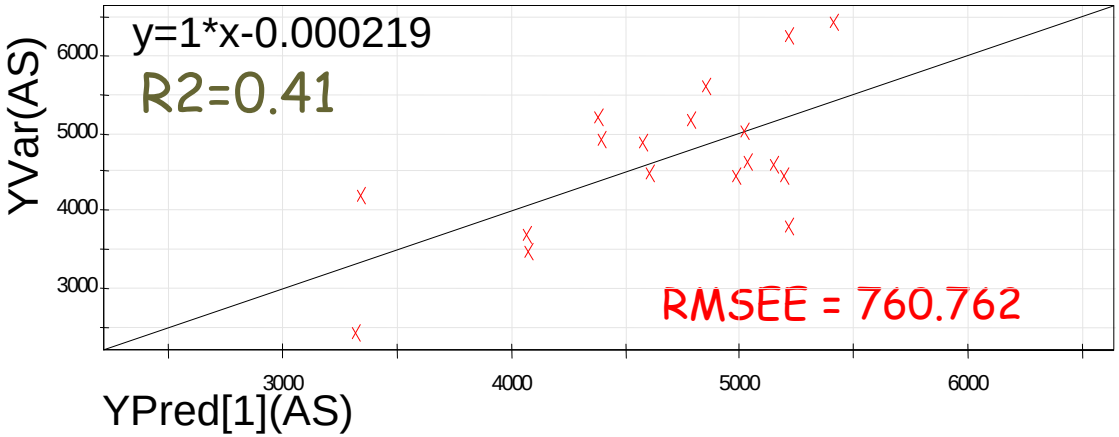
Validation Set

RMSEP = 675



Altadis

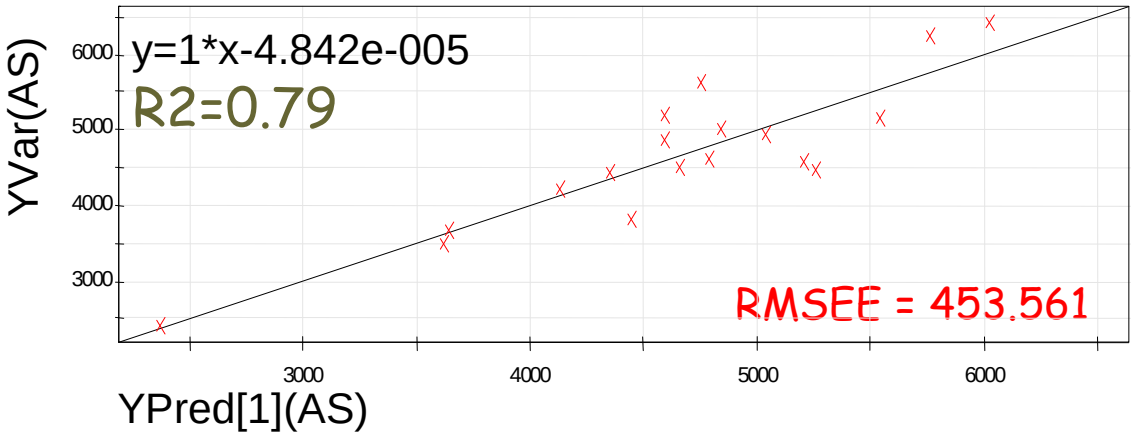
Specific Activity : Link with Chemistry on Burley Tobaccos

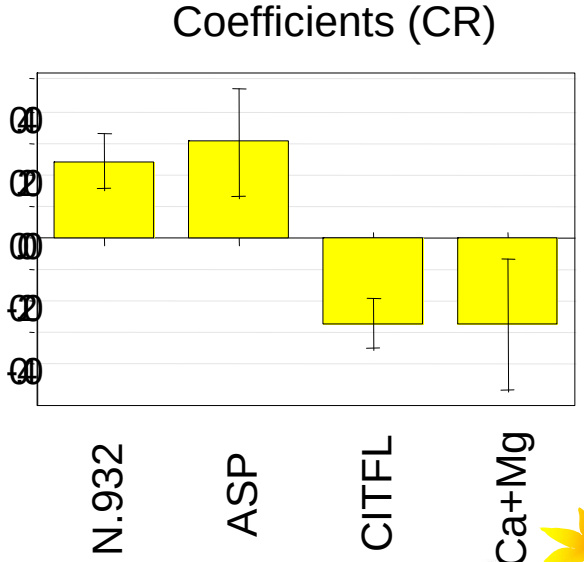


Linear regression :

RMSEE = 761 rvts/mg TPM
RMSEP = 735 rvts/mg TPM

With extra chemical indicators, improvement.
RMSEE = 454, RMSEP = 571



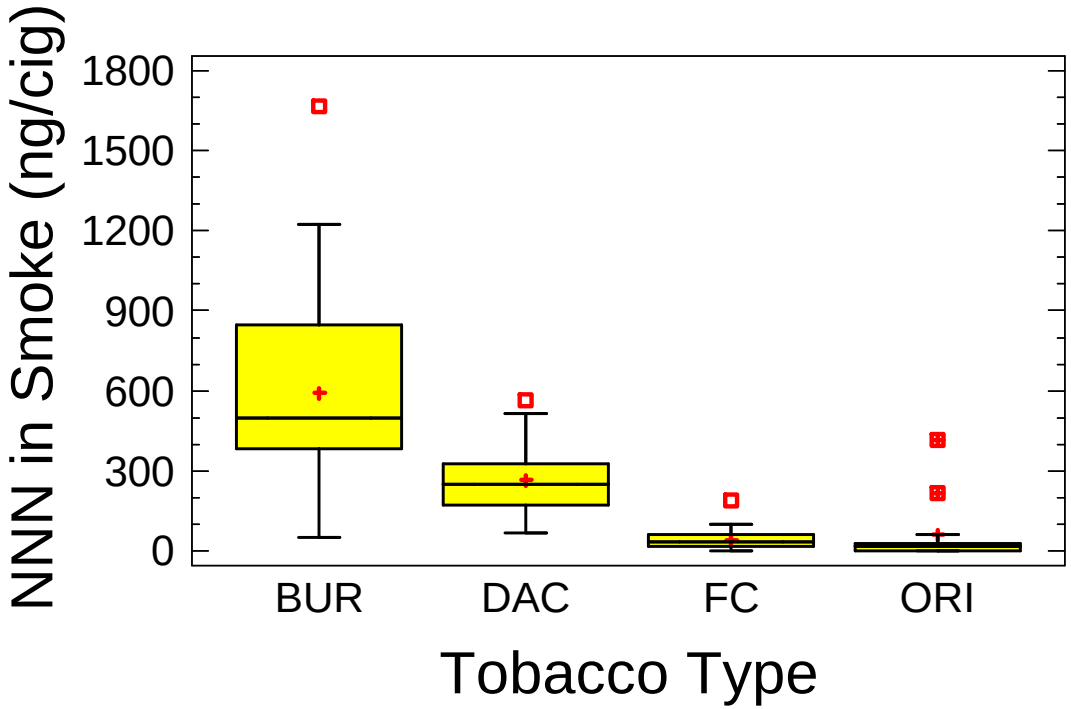


Optimisation of Burley choice according to chemical properties

Altadis

NitrosoNorNicotine in smoke overview

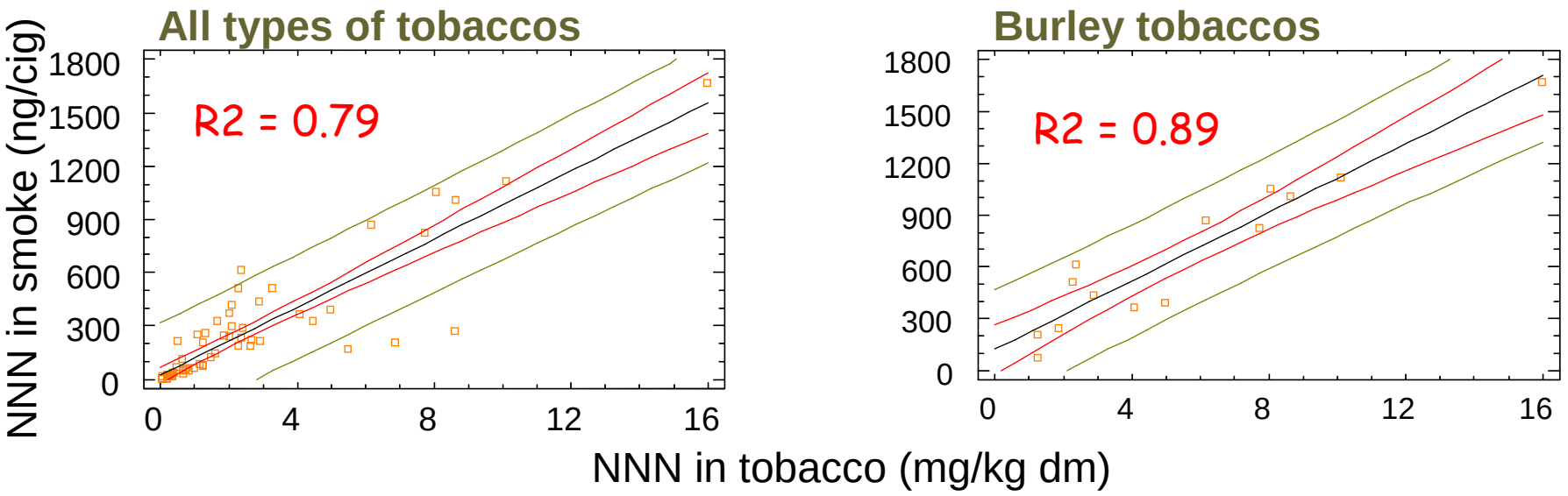
NNN in Smoke content according to tobacco type



NNN in smoke (ng/cig)	B	DAC	FC	Oriental
Min	52	66	2	2
Max	1666	565	189	415
Mean	592	266	41	60

Comparison of different models

- No model obtained with NIRS : no quick & easy prediction
- Good linear relationship between NNN in tobacco and NNN in smoke



RMSEE = 143
RMSEP = 139

RMSEE = 133
RMSEP = 159

Other influent parameters

- No improvement with physical parameters : physical variables are not significant in the models
- Addition of extra chemical parameters :

X variables	R2Y	Q2	RMSEE	RMSEP
All tobaccos				
NNNT	0.79	0.79	146	139
NNNT, NOR, C/K	0.90	0.90	101	109
Burley				
NNT	0.89	0.89	143	159
NNNT, K	0.92	0.92	129	211

No significant improvement of the prediction quality
NNN in tobacco is the main factor which explains NNN in smoke

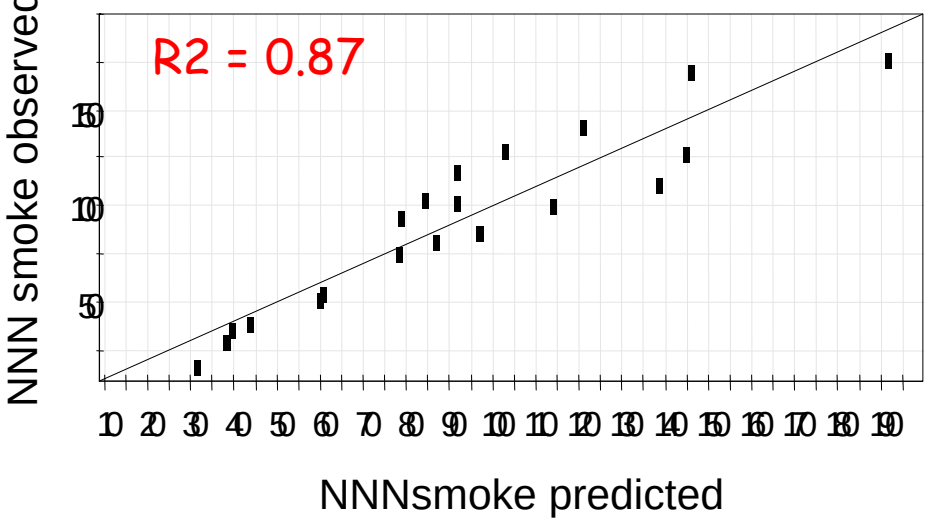
Altadis

Prediction for blends

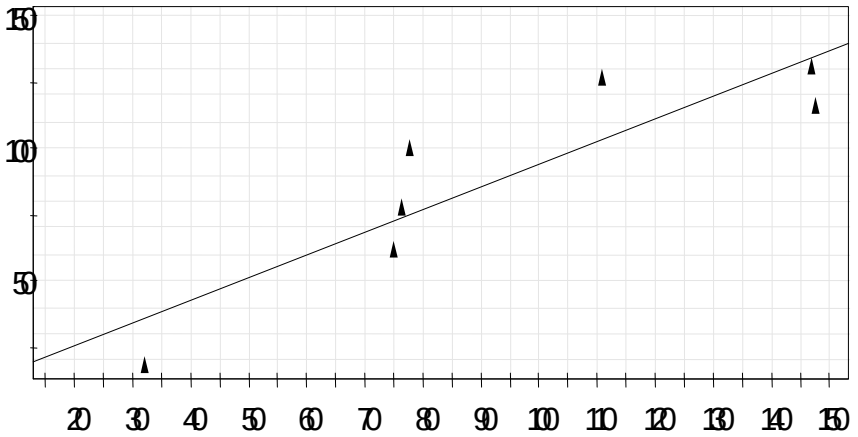
- 27 commercial cigarettes, tar between 9 and 12 mg
- Different NTM combinations (Filter ventilation from 0 to 29 %)
- Measurement of NNN in the blend and in smoke
- Same procedure as for As is tobaccos

Good results with linear regression NNN in smoke/NNN in tobacco

Calibration, RMSEE = 17 ng/cig



Validation, RMSEP = 18 ng/cig



For a limited range of tar, NNN is the main factor explaining NNN in smoke despite different cigarette NTM combinations

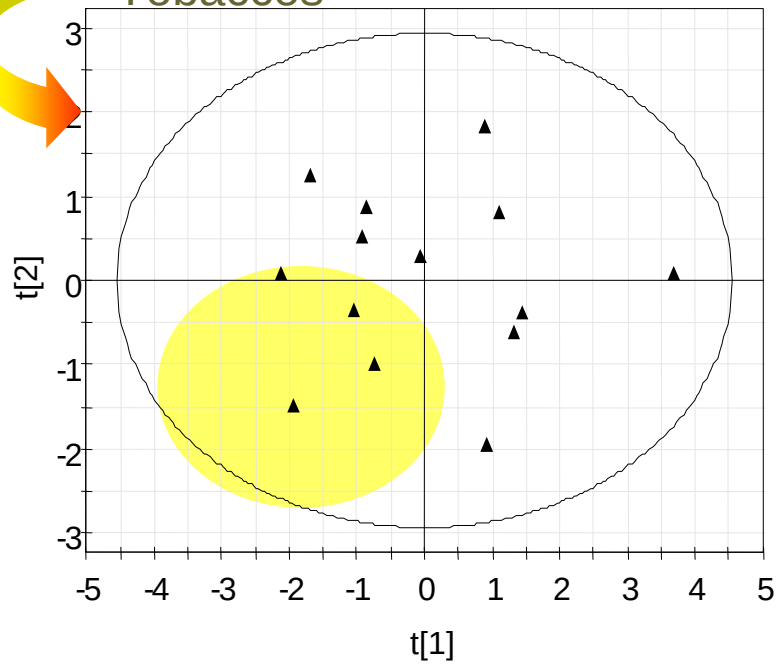
Altadis

Factors of prediction of NNN in Burley tobaccos

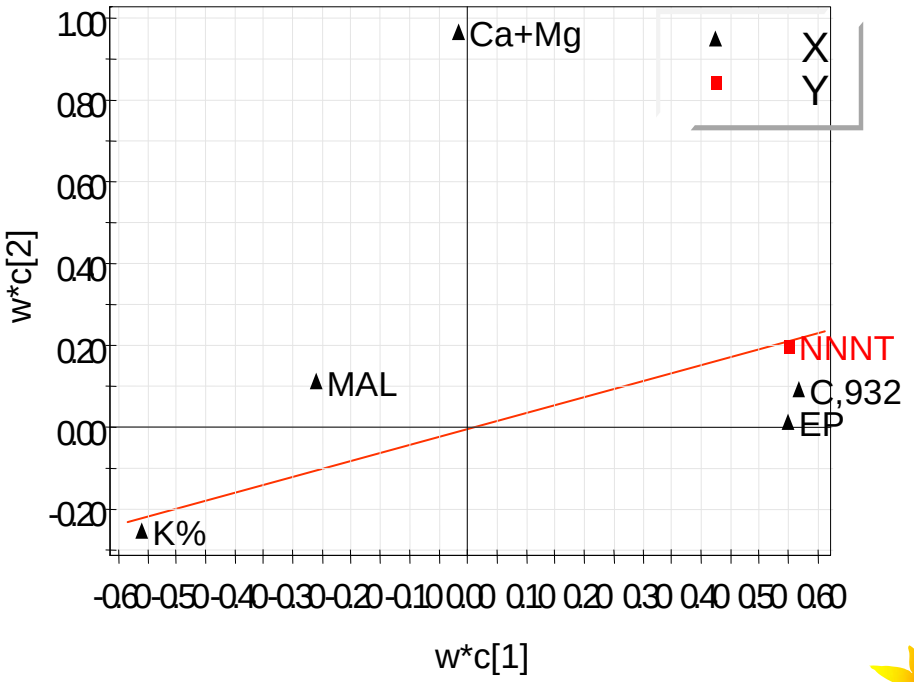
- Comparison of different models of prediction of NNN in Burley tobaccos

X variables	R2Y	Q2	RMSEE	RMSEP
NOR	0.77	0.75	2.1	1.53
AT, EP, NOR, K, C	0.86	0.80	1.73	1.68
MAL, EP, CaMg, K, C	0.84	0.76	1.81	2.23

Tobaccos



Variables



- NNN is not directly linked to tobacco blenders expertise

Altadis

Conclusion & Perspectives

Specific Activity

- NIRS is a quicker method to forecast results of Ames Test (strain TA98 S9 activation) for as is tobaccos
- Chemical models can be implemented with chemical parameters linked to tobacco properties

➢Keep up the models
➢Extend them to blends
➢Define responsible compounds & precursors

NNN in smoke

- No fast prediction of NNN in smoke based on NIRS results
- Transfer seems to be the main factor of explanation of NNN in smoke
  - For as is tobaccos
  - For cigarettes in a limited range of tar
- NNN in tobacco can be predicted from nornicotine content or from other chemical parameters
- NNN is not directly related to tobacco blenders expertise

➢Keep up the models
➢Validate the transfer hypothesis
➢Put under control factors of formation of NNN in tobacco

Altadis