# WINE AUTHENTICITY AND QUALITY DETERMINATION BY NMR: WINE-PROFILING™

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## INTRODUCTION

Nowadays, NMR spectroscopy is a powerful analytical technique in the field of food traceability and authenticity. Along the last 20 years, the application of NMR in food analysis have risen exponentially,[1] mainly due the huge information that is collected from a NMR spectrum with a simple and quick preparation of the sample.

A basic research work by some of the authors in the past[2] showed the applicability of Nuclear Magnetic Resonance (Bruker Avance I 400 MHz spectrometer) as tool for the study and control of the wine production process. The alcoholic and malolactic fermentations were evaluated by NMR comparing other "classic" analytic techniques (**Figure1**); metabolic transformations of $^{13}C$ labeled amino acids in wine were monitored by NMR (**Figure 2**); and the more promising study of Rioja terroir by $^{1}H$ NMR metabolomics. Through this kind of NMR data analysis, it was possible to classify different wines of Rioja depending on the vintage, year, areas of origin and even the winery, using chemometric methods as IECVA. It was also possible to find biomarkers (**Figure 3**).
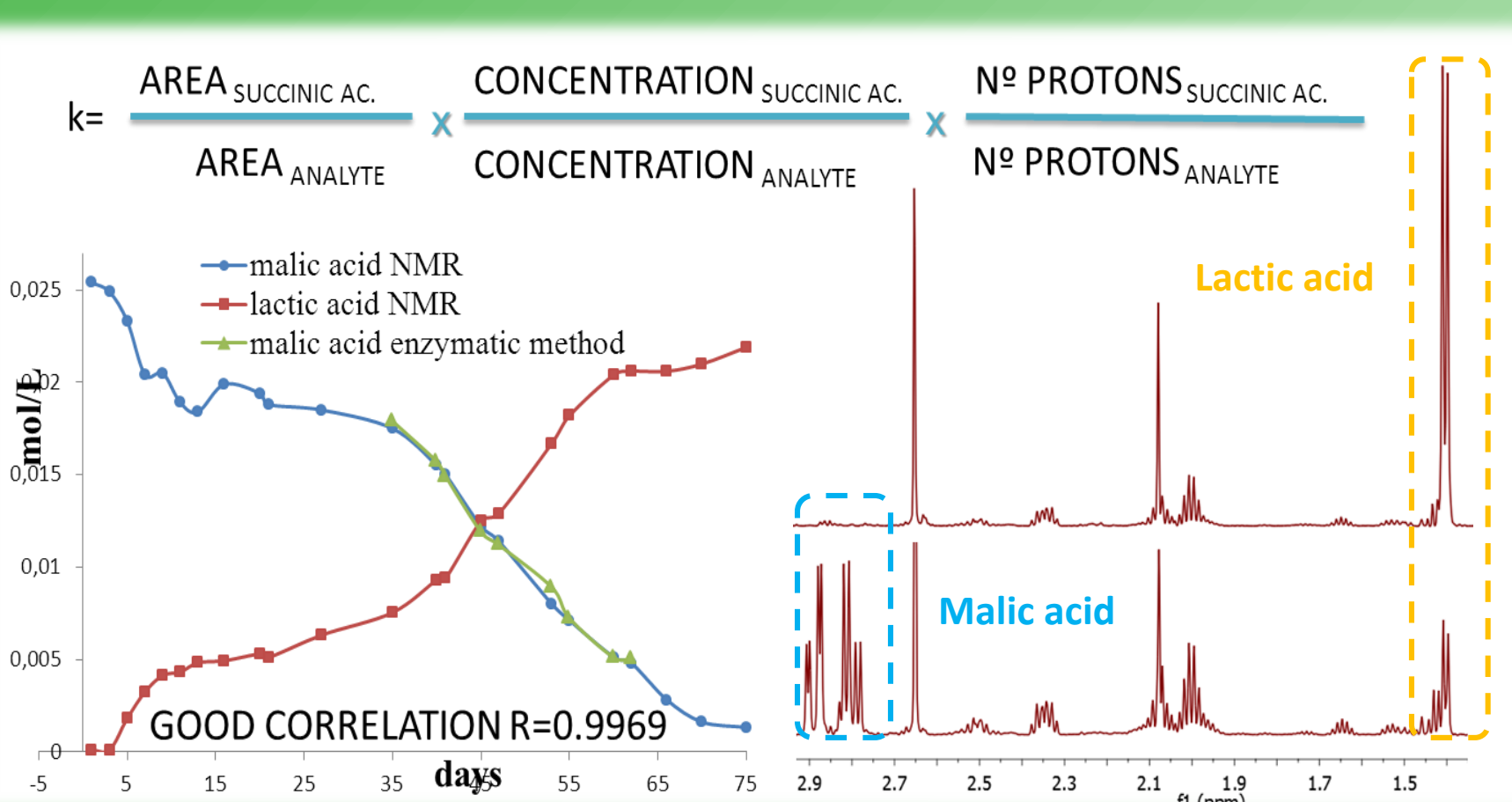


**Figure 1.** Quantification method using as external standard succinic acid
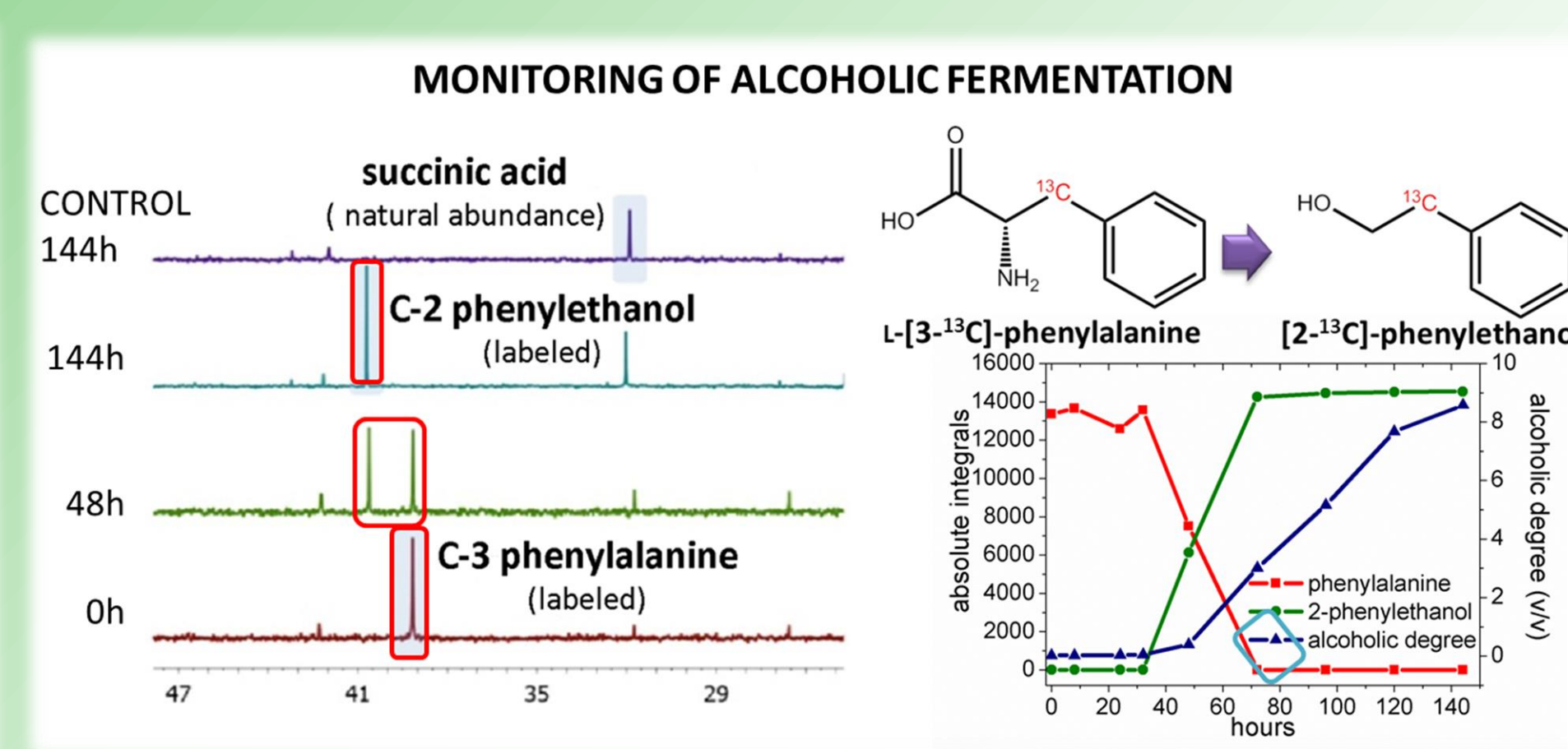
**Figure 2.** metabolic transformations of $^{13}C$ labeled amino acids in wine: **A)** Alcoholic Fermentation ; **B)** Malolactic Fermentation
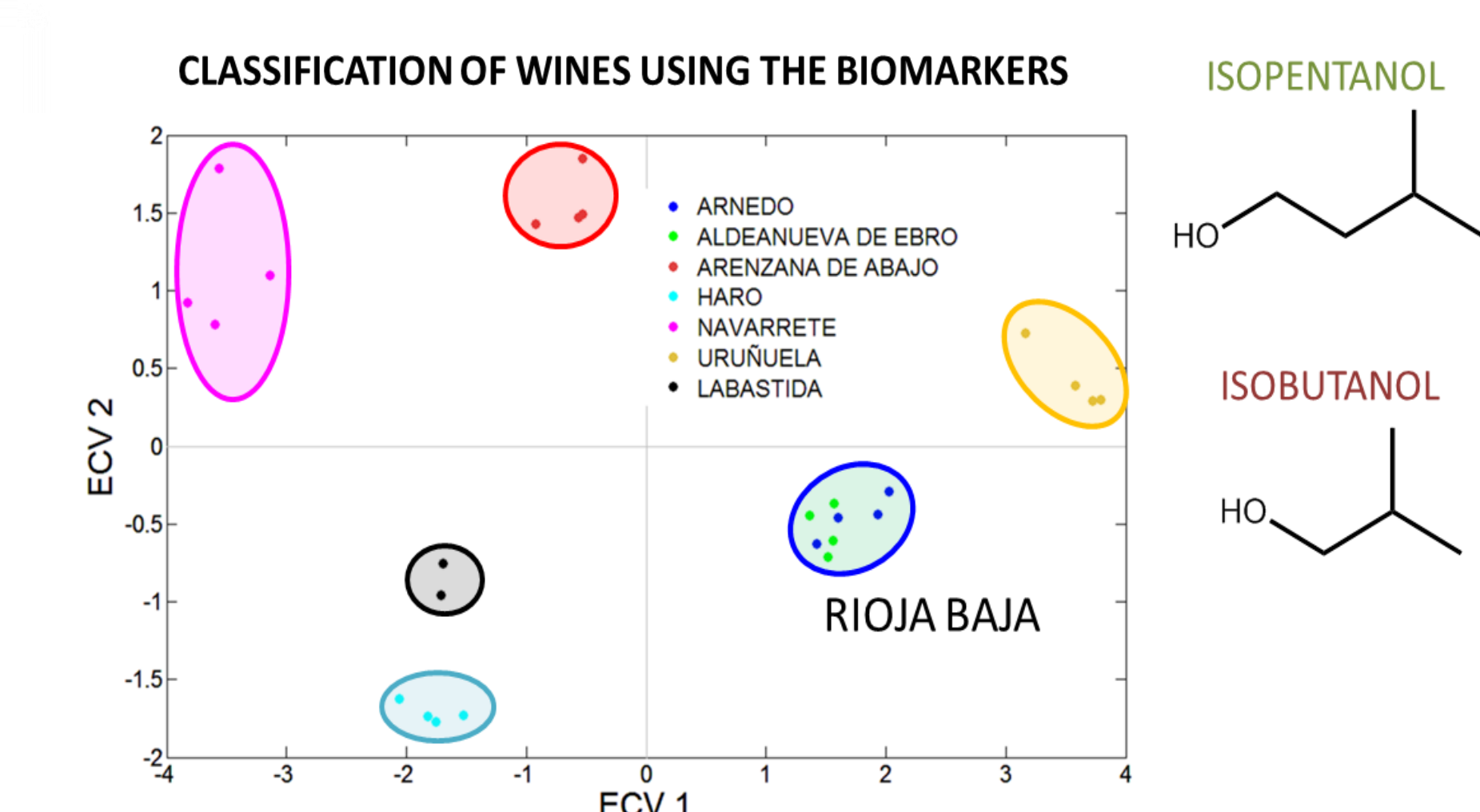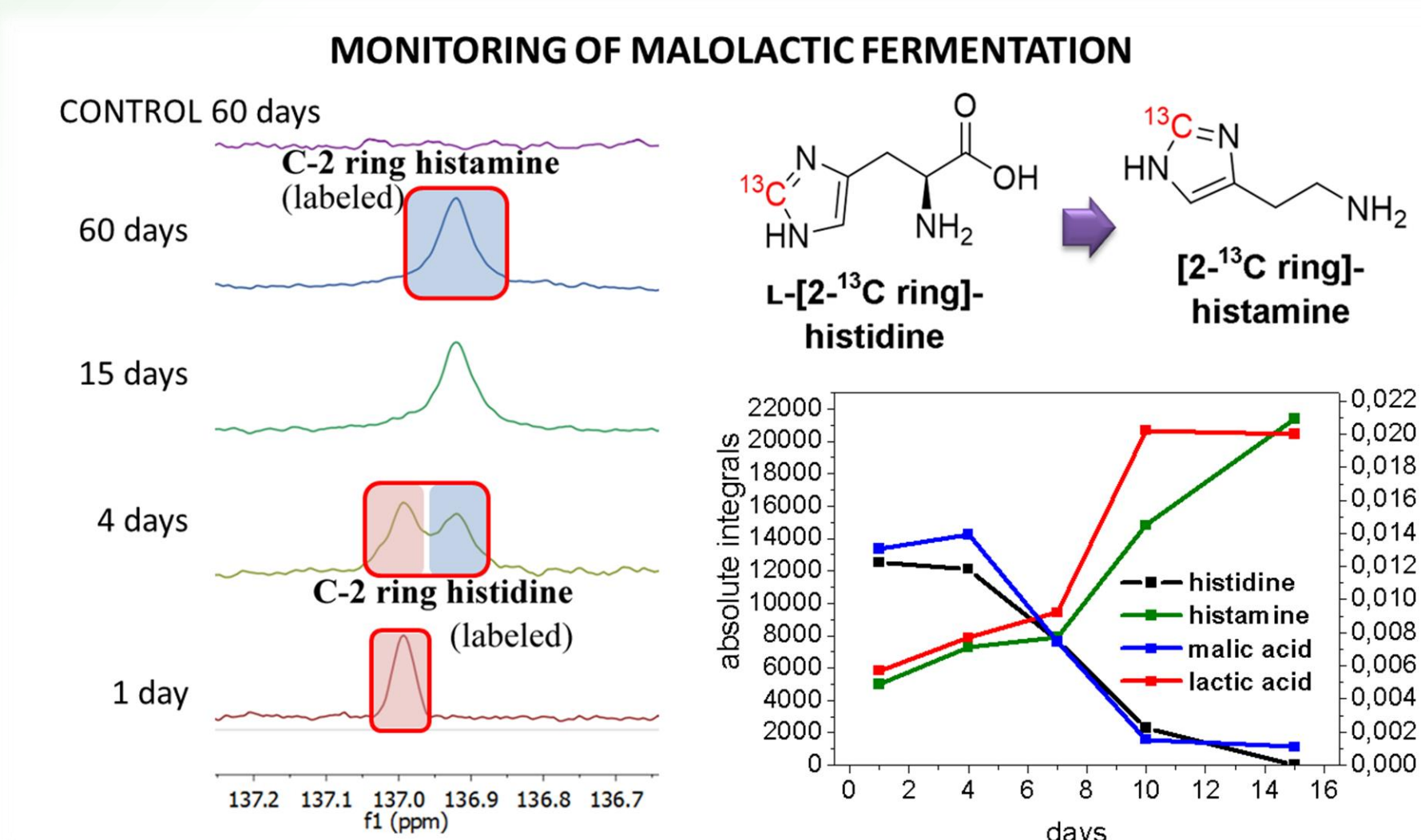
**Figure 3.** Two biomarkers of Rioja wines were found: isopentanol and isobutanol for the classification of wine according to the wineries

## MATERIAL AND METHODS

The relevance achieved by these NMR research results (in a small region where wine is the first industry) helped to institutions of La Rioja to gain awareness of the NMR potential. The Comunidad Autónoma de La Rioja acquired the newly developed NMR equipment by Bruker: Wine-Profiling™, an innovative solution for the analysis of wine using NMR spectroscopy.[3] The main objective of the action was to establish Estación Enológica de Haro, with the help of new NMR facility, as a reference lab for wine authenticity and quality determination in Spain, as partner of the Bruker NMR database network.
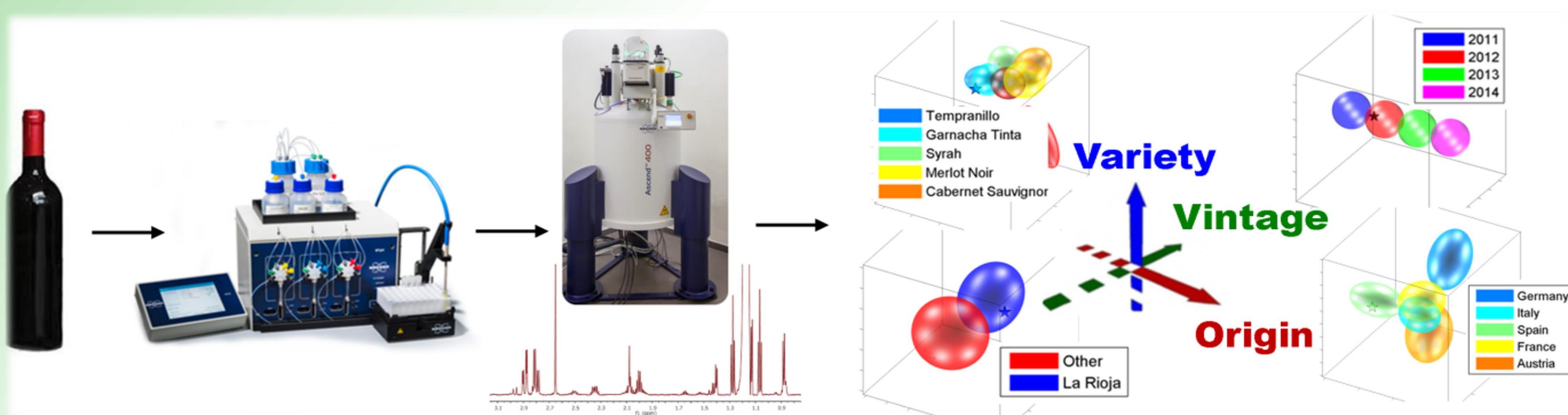


**Figure 4.** Wine-Profiling™ process.

Just 900 µL of wine is needed for the analysis by Wine-Profiling™ and a report is obtained with full information about the wine: certificate of authenticity (origin, variety and vintage) and quality with the quantification of 52 wine compounds in only 20 minutes (**Figure 4**). 215 wines were analyzed by Wine-Profiling™ in order to evaluate the running of the current models, for classification and quantification. The origin of these wines was Rioja.

The wines were analyzed by NMR and by traditional methods in the laboratory of Estación Enológica de Haro, laboratory accredited in ISO 17025.

### References

[1] A. Spyros, P. Dais *NMR Spectroscopy in Food Analysis*, RSC Publishing, **2012**.
[2] Eva López-Rituerto. Tesis Doctoral: *La Resonancia Magnética Nuclear como herramienta en el estudio y control del proceso de vinificación*, Universidad de La Rioja, **2012**.
[3] R. Godelmann, F. Fang, E. Humpfer, B. Schütz, M. Bansbach, H. Schäfer, M. Spraul *J. Agric. Food Chem.* **2013**, 61, 5610–5619.

## RESULTS

The classification results were very good. More than 90% of the wines were perfectly classified according to their variables: vintage, variety and region (Rioja and Spain). In particular, vintage and country (Spain) were perfectly classified (100% of the wines). The statistic model of Rioja works very well too, the 97% of the samples were classified as Rioja. The model of the variety classified a bit worse the wines than the other models, but the variety of the 94.2% of the samples was perfectly identified. This result is because of Rioja wines aren't usually monovarietals, but this model was built with this kind of wines, so this variety will be improved in next model.
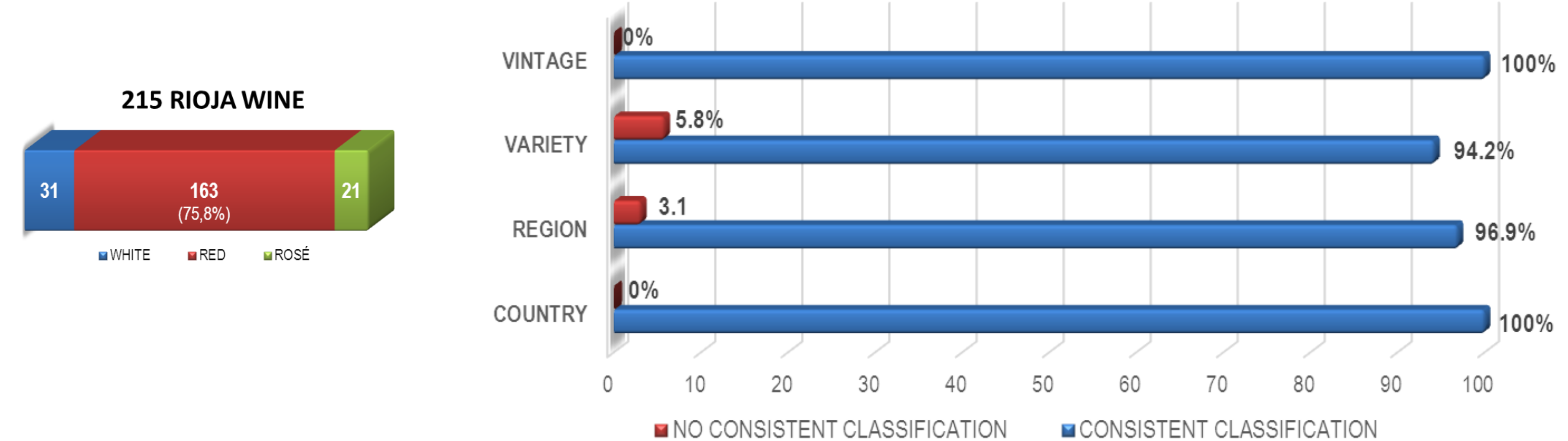


**Figure 5.** Results of classification of wine using Wine-Profiling™.

The NMR quantification were compared with traditional methods in order to evaluate the running of Wine-Profiling™. High correlation between these methods were found in most of the parameter that were studied. As example, the correlations of alcoholic degree, acetic, citric and malic acids are presented (**Figure 5**).
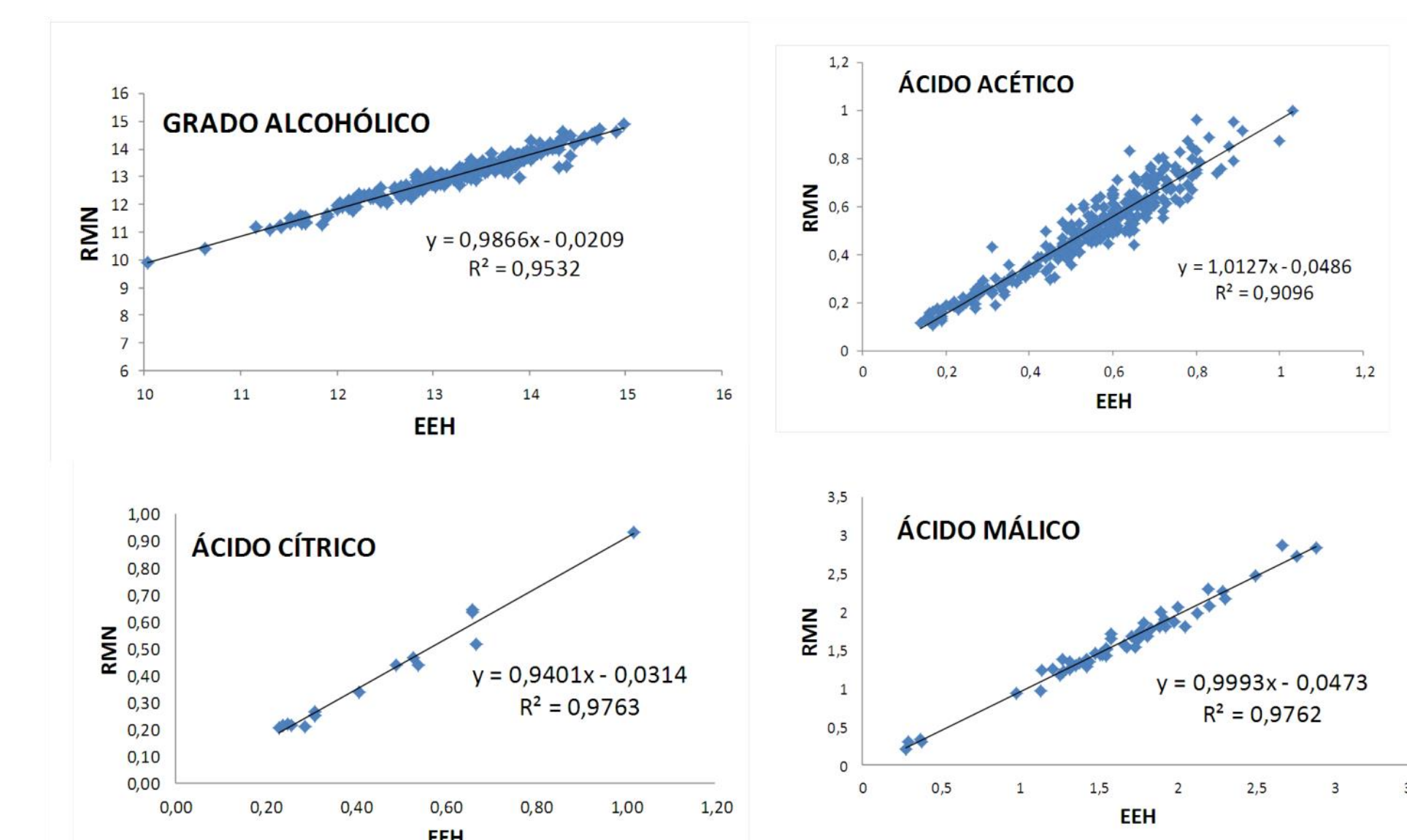


**Figure 6.** Quantification: correlation between traditional methods and Wine-Profiling.

## CONCLUSION

With this work we have demonstrated the enormous potential of $^{1}H$ NMR spectroscopy by Wine-Profiling™ in order to quantify and certificate the authenticity of origin, variety and vintage.