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<b>INDUSTRIAL SECTOR</b>	Agrofood
<b>RESEARCHER</b>	María Luisa Marina Alegre Antonio Luis Crego Navazo Miguel Guijarro Diez María Castro Puyana
<b>DEPARTMENT</b>	Analytical Chemistry and Chemical Engineering
<b>CONTACT DETAILS</b>	91 885 49 35 91 885 63 90  @ antonio.crego@uah.es
<b>WEB SITE</b>	<a href="#">(Micro)- Separation Techniques.</a>

## DESCRIPTION OF THE RESEARCH RESULTS

### TITLE

Novel method for the detection of adulteration of saffron with gardenia

### ABSTRACT

Researchers from the (Micro)-Separation Techniques Research Group of the Department of Analytical Chemistry, Physical Chemistry and Engineering of the University of Alcalá has developed a procedure for the detection of adulterations of saffron with gardenia based on the detection of geniposide by Liquid Chromatography with High-Resolution Mass Spectrometry detection. The procedure allows to detect adulterations with gardenia in an unambiguous and sensitive way. A problem not solved at present by any other existing methods.



The group is looking for companies in the agro-food sector to sign technical cooperation agreements, commercial agreements with technical assistance or patent licensing agreements.

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## DESCRIPTION AND SPECIAL FEATURES

The object of the invention is the development of a process for the detection of adulterations of saffron with gardenia based on the detection of geniposide by Liquid Chromatography with High-Resolution Mass Spectrometry detection.

The procedure for the determination of geniposide is based on the use of a C18 column and a liquid chromatography equipment coupled to a quadrupole-time-of-flight (QTOF) Mass Spectrometry (MS) detector through an ionization source by orthogonal electrospray with Jet Stream thermal focus technology. The mass spectrometer is equipped with an Agilent Technologies Mass Hunter software for mass spectrometer control and the data acquisition and analysis.

The analytical characteristics evaluated by the procedure, object of the invention are:

- Linearity: it was evaluated as the range of working concentrations between 0.8 and 8  $\mu\text{g}/\text{mL}$  geniposide.
  - Detection limit: it was evaluated as the concentration of geniposide that gives a value of the signal-to-noise ratio of 3. The limit is 0.01  $\mu\text{g}/\text{mL}$ , which allows to detect about 42  $\mu\text{g}$  of geniposide per gram of analyzed sample that is up to 0.0042%, a value that demonstrates the high sensitivity of the method developed for the determination of geniposide in saffron.
  - Limit of quantification: it is evaluated as the concentration of geniposide that results in a value of the signal-to-noise ratio of 10, obtaining a limit of 0.03  $\mu\text{g}/\text{mL}$ .
  - Precision: it is evaluated as instrumental repeatability and intermediate precision. The instrumental repeatability is determined as the value of the relative standard deviation (RSD) in % for the peak areas obtained when 3 consecutive injections of a standard geniposide solution are performed at two concentration levels (0.8 and 8  $\mu\text{g}/\text{mL}$ ) and at a solution of an authentic adulterous saffron sample with percentages of 10 and 90 % of gardenia extract. The values obtained for RSD were lower than 1.5 %. Intermediate precision was determined from the RSD values of the peak areas obtained by injecting (replicate for two consecutive days) three replicates of a standard geniposide solution at two concentration levels (0.8 and 8  $\mu\text{g}/\text{mL}$ ) and at a solution of a sample of authentic saffron adulterated with percentages of 10 and 90 % of extract of gardenia. The values obtained for the RSD were inferior to 1.8 % for the standard solutions of geniposide and to 2.9 % for the samples of saffron.
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- Accuracy: it was evaluated as the recovery obtained for the geniposide when a sample of authentic saffron was enriched with standard geniposide at a concentration of 1 µg/mL. The average recovery values obtained were  $89 \pm 14$  %.

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## INNOVATIVE ASPECTS

The use of geniposide as a marker of adulterations of saffron with extracts of gardenia allows to perform a quality control of the saffron and to detect adulterations of saffron with gardenia in a sensible and unequivocal way.

- It allows to detect the adulteration of saffron with gardenia through the determination of geniposide.
- It allows to determine the content of geniposide in adulterated saffron.
- It allows to separate the geniposide peak of the rest of the components of the saffron in a time less than 2 minutes.
- It allows to detect up to 0.004 % of geniposide in saffron making it a very sensitive procedure.
- It allows to identify unequivocally the geniposide through its mass spectrum.

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## TECHNOLOGY KEYWORDS

Saffron, adulteration, gardenia, geniposide, Liquid Chromatography, Mass Spectrometry.

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## SCIENTIFIC DOMAINS

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| <input type="checkbox"/> Information and Communication Technologies                  | <input type="checkbox"/> Biological Sciences                   |
| <input type="checkbox"/> Industrial Manufacture, Material and Transport technologies | <input type="checkbox"/> Agriculture and Marine Resources      |
| <input checked="" type="checkbox"/> Other Industrial Technologies                    | <input checked="" type="checkbox"/> Agrofood Industry          |
| <input type="checkbox"/> Energy  | <input checked="" type="checkbox"/> Measurements and standards |
| <input type="checkbox"/> Physical and Exact Sciences                                 | <input type="checkbox"/> Environment and risk prevention       |
|  | <input type="checkbox"/> Socioeconomics                        |



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### CURRENT STAGE OF DEVELOPMENT OF THE RESEARCH RESULT

- Development phase
- Developed, available for demonstration
- Already on the market

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### FUNDING RESEARCH

- European RTD project
- Regional project
- National project
- Private funding

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## COMMERCIAL ASPECTS

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### COMPETITIVE ADVANTAGES

- The presence of geniposide in extracts of gardenia and its absence in saffron allows to propose the geniposide molecule as marker of adulterations of saffron with gardenia.
- It is of interest to the food sector for its potential to control the quality of saffron and avoid economic fraud.
- Application with reasonable costs and possibility of distribution in the international market: USA, Europe and Japan

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### CURRENT STATE OF INDUSTRIAL AND INTELLECTUAL PROPERTY

- Patent applied
- Exclusive rights
- Patent granted
- Secret know how
- Software registered
- Copyright protected

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### COMMENTS

The patent has been applied to the Spanish Patent and Trademark Office on February 19, 2016 with application number P201600133.

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### TYPE OF COLLABORATION SOUGHT

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| <input checked="" type="checkbox"/> Technical cooperation | <input checked="" type="checkbox"/> Commercial agreement with technical assistance |
| <input type="checkbox"/> Joint venture agreement          | <input checked="" type="checkbox"/> License agreement                              |
| <input type="checkbox"/> Manufacturing agreement          |  |

### COMMENTS

The group is looking for companies in the agro-food sector to sign technical cooperation agreements, commercial agreements with technical assistance or patent licensing agreements.

The present invention has a great interest for both, the food industry and the administration, as it provides a sensitive analytical procedure which allows the quality control and safety control of saffron.

Some examples of target companies are: Carmencita, Azafranes Manchegos, and The saffron company.

### ADDITIONAL FORMATION (PICTURES)

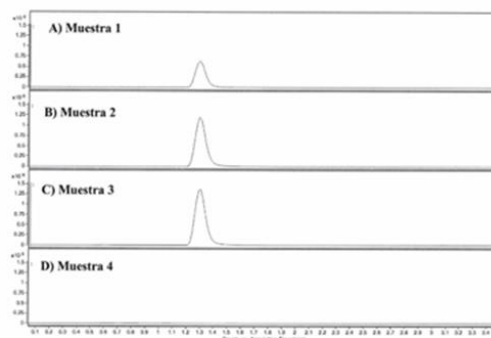


FIG. 1


Figure 1. Chromatograms of extracted ion at a ratio  $m/z$   $433.1384 \pm 50$  ppm obtained by liquid chromatography with quadrupole-time of flight mass spectrometry detection, for four samples of saffron suspected to have been adulterated. A), B), C) samples of saffron adulterated with gardenia. D) sample of unadulterated saffron with gardenia.




## OTRI CONTACT DETAILS

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### UNIVERSIDAD DE ALCALÁ-OFICINA DE TRANSFERENCIA DE RESULTADOS DE INVESTIGACIÓN (OTRI)

 Edificio Politécnico  
Campus Externo UAH  
28871 Alcalá de Henares (Madrid)

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 + 34 91 885 45 68 / 45 61

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 + 34 91 885 48 46

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 [otriuah@uah.es](mailto:otriuah@uah.es)

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