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Validated flow-injection method for rapid aluminium determination in anti-perspirants

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Abstract

A flow-injection (FI) method for the rapid determination of aluminium in anti-perspirants has been developed. The method is based on the spectrophotometric detection at 535 nm of the complex formed between Al ions and the chromogenic reagent eriochrome cyanine R. Both the batch and FI methods were validated by checking the parameters included in the ISO-3543-1 regulation. Variables involved in the FI method were optimized by using appropriate statistical tools. The method does not exhibit interference from other substances present in anti-perspirants and it shows a high precision with a R.S.D. value ($n = 6$) of 0.9%. Moreover, the accuracy of the method was evaluated by comparison with a back complexometric titration method, which is currently used for routine analysis in pharmaceutical laboratories. The Student's *t*-test showed that the results obtained by both methods were not significantly different for a significance level of 95%. A response time of 12 s and a sample analysis time, by performing triplicate injections, of 60 s were achieved. The analytical figures of merit make the method highly appropriate to substitute the time-consuming complexometric method for this kind of analysis.

Keywords: FIA; Anti-perspirants; Spectrophotometry; Aluminium; Eriochrome cyanine R; UV

1. Introduction

Anti-perspirants act against feeding of bacteria present in the skin, which give rise with sweat to the appearance of disagreeable odour. In order to do that, anti-perspirants obstruct cutaneous pores by astringency and by formation of proteic aggregates in the glands exit canals. Astringent agents used are aluminium compounds, mainly chlorhydrates. In spite of the controversy about anti-perspirant functioning, nowadays aluminium chlorhydrate is not included in the list of forbidden substances. Moreover, it is not subjected to restriction concerning its concentration and can be freely used, as well as other aluminium salts, in cosmetic products. Nevertheless, the industry devoted to the anti-perspirants fabrication is interested in knowing the aluminium concentration both in the raw material and the final product, for routine quality control.

Many methods can be found in the literature for the determination of aluminium. Methods applied for aluminium analysis in natural waters [1], and for the speciation of Al in environmental samples [2] have been reviewed. Methods using atomic absorption with electrothermic atomization have been used for Al analysis in soft drinks [3], sugar cane spirit [4], serum and urine [5,6], environmental samples [7], lubricant oils [8] or baby's food [9]. Electroanalytical techniques, mainly cathodic adsorption stripping voltammetry, have been also employed [10–13]. Many of the recently published works on Al determination are spectrofluorometric methods, for the analysis of waters [14–18], foodstuffs [15], plant nutrient solution [19], biological fluids [20], hemodialysis solutions [20,21] and pharmaceutical products [22]. Moreover, Al-Kindi et al. have reviewed fluorimetric methods for aluminium determination using sequential injection analysis (SIA) [23]. Also, spectrophotometric methods for Al determination in geochemical samples by reaction with xylenol orange [24], in dialysis concentrates with 3,5-diterbutylsalicyl fluorone and 1-butyl-3-trimethylsilylimidazolium [25], in post-hemodialysis fluid [26] or natural waters [27] using pyrocathecol violet, in water samples with chrome azurol S [28,29], in floc-

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ulants, tea, underground sewage and human hair with indigo carmine [30], or in pharmaceutical suspensions and granite with 2,2′,3,4-tetrahydroxy-3′-sulfo-5′-nitroazobenzene [31] have been reported. Concerning chromatographic methods, they have been applied to the determination of Al, and other metals in pharmaceutical and food-supplement formulations [32], wine [33], biological and environmental samples [34], waters [35] and drinks [36]. Other methods make use of techniques such as diffuse reflectance spectroscopy [37], γ-ray transmission techniques [38], ICP-AES [39,40] or catalytic spectrophotometric methods [41]. It is important to remark the very few works reported with application to the pharmaceutical industry, in which the quality control is critical.

The reaction between Al and the chromogenic reagent eriochrome cyanine R has been used previously for the spectrophotometric determination of Al in waters [42,43], soils [43,44] and plant tissues [45]. The complex exhibits a high molar absorptivity which implies a high sensitivity. It has been applied to the spectrophotometric determination of Al in hemodialysis solutions, using a flow preconcentration system [46], but, to the best of our knowledge, there is no any automated method based on this reaction applied to the Al analysis in pharmaceutical or cosmetic products.

The aim of this work is to develop and validate a flow-injection (FI) method for the Al determination in anti-perspirants, which allows the substitution of the currently used procedure consisting on a complexometric back titration. The method makes use of UV–vis spectrophotometric detection of the Al–eriochrome cyanine R complex. Considering that an ideal analytical method for routine analysis and quality assurance should be automatic, simple, cost-effective, robust, precise and accurate, and have a high sampling rate, FIA is a well-established technique that fulfils the above-mentioned demands. Moreover, it has been extensively applied in pharmaceutical/cosmetic industry analysis due to the significant advantages that offers for the determination and monitoring of a single analyte in routine analysis.

2. Experimental

2.1. Apparatus

Spectrophotometric measurements were carried out using a Cary 50 spectrophotometer (Varian Inc., Walnut Creek, CA, USA) equipped with the Cary WinUV software. The intermediate precision of the method under batch conditions was also checked by using a Shimadzu (Izasa, Madrid, Spain) UV-1603 spectrophotometer. A Methrom 744 pH-meter (Gomensoro, Madrid, Spain) was employed for adjusting pH.

The FIA arrangement was composed of a Gilson Minipuls-3 peristaltic pump (Madrid, Spain), a Rheodyne (Jasco, Madrid, Spain) 50 injection valve and a Hellma 176.000-QS quartz flow cell with 1 cm optical path, which was located into the spectrophotometer measurement compartment. Gilson Tygon 0.6 cm^3/m tubes were used for pumping and the system employed Teflon 0.5 mm i.d. tubing.

2.2. Reagents and solutions

Sulfuric acid 95–98% (Panreac, Barcelona, Spain), acetic acid (Panreac, 100%), trihydrated sodium acetate (Panreac, 98%), eriochrome cyanine R (Fluka, Madrid, Spain).

A 1002 ± 2 mg/L Al(III) standard solution in 0.5 M nitric acid was provided by Merck (Madrid, Spain). More diluted standards of 5 and 50 mg/L were prepared by dilution with mQ water. 1:4, 3 and 0.01 mol/L sulfuric acid solutions were used. A pH 6 acetic acid/sodium acetate buffer solution was also employed. A 4.0 g/L eriochrome cyanine R solution in mQ water was prepared and, from this, the working solution was daily prepared by diluting 10-fold with mQ water. When the FIA method was used, an eriochrome cyanine R solution was daily prepared by solving 150 mg of the reagent in 250 mL of mQ water.

2.3. Samples

The samples, commercial anti-perspirants (milky viscous solution) as well as the excipients mixture and the raw materials were kindly provided by laboratories SUQUINSA (Madrid, Spain) and contain: synodor®, methyl- and phenyl-parahydroxybenzoate, phenoxyethanol, ethanol and aluminium chlorhydrate.

2.4. Procedures

2.4.1. Batch mode

One milliliter of 0.01 mol/L H_2SO_4, 10 mL of the buffer solution, the corresponding amount of a 5.0 mg/L Al^{3+} standard solution (or the appropriate amount of sample for the anti-perspirant analysis), and 5.0 mL of the eriochrome cyanine R working solution were added in this order to a 50 mL volumetric flask diluting to the mark with mQ water (Waters, Madrid, Spain). The reaction time was controlled from the addition of the complexing agent.

2.4.2. FIA mode

Standard solutions for the construction of the calibration graph were prepared in 10 mL volumetric flasks by diluting a 50 mg/L Al^{3+} standard solution with the acetic acid/sodium acetate buffer solution, which was also used as carrier solution. A flow rate of 2.2 mL/min was employed. The chromogenic reagent was pumped through a second channel at a flow rate of 1.0 mL/min and merged with the carrier solution as shown in Fig. 1. Spectrophotometric detection was performed at 535 nm.

2.4.3. Sample preparation

A commercial anti-perspirant spray purchased in a local drug-store containing approximately 2.7% (w/w) Al in the form of aluminium chlorhydrate was analyzed. About 0.25 g of the pasty liquid present in the spray container were accurately weighed into a 50 mL volumetric flask. Then, 1.0 mL of the 1:4 H_2SO_4 solution was added and let to react for approximately 30 min. After this, mQ water was added to the mark and the solution homogenized. A 50 μL aliquot of this solution was taken for the preparation of the measurement solution in the batch mode as

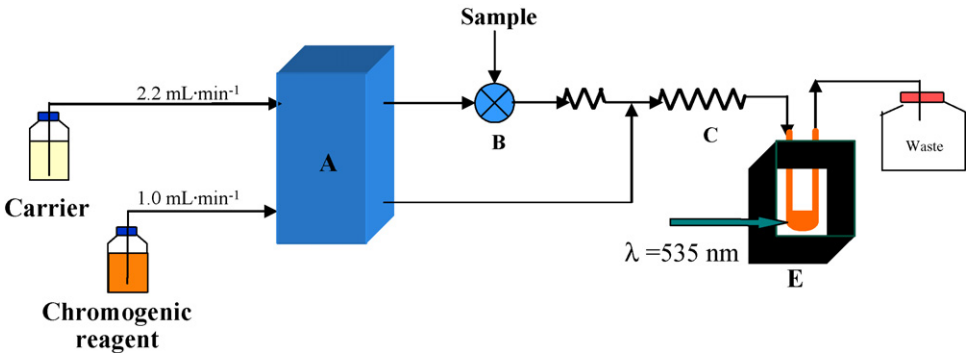

Fig. 1. Schematic display of the FI arrangement used: (A) peristaltic pump, (B) injection valve, (C) reaction coil (or reactor), and (E) spectrophotometer.

described above. Concerning FIA mode, 100 μL of the sample solution were diluted to 25 mL with acetic acid/sodium acetate buffer solution. An aliquot of 50 μL of this solution was injected into the injection valve.

2.4.4. Reference method

The method described is the one currently used for aluminium determination in anti-perspirants (provided by SUQUINSA). Approximately 1.7 g of the product contained into the spray were accurately weighed into a 250 mL Erlenmeyer flask, and 5 mL of the 1:4 H_2SO_4 solution, 25 mL mQ water and 25 mL of a 0.1 M EDTA ($Na_2H_2Y{\cdot}2H_2O$, Panreac, 99%) were added. The solution pH was adjusted to 4.5 with a 30% NaOH solution, and 10 mL of a 2.0 M acetic acid/ammonium acetate of pH 4.8 were then added. The solution was boiled for 3–4 min, and after cooling at room temperature, 60 mL ethanol (Panreac, absolute PA) and some drops of ditizone solution (0.5 g ditizone, Sigma ACS, in 1 L chloroform, Prolabo rectapur) were added. This solution was back titrated with a 0.100 mol/L $ZnSO_4$ (Panreac, 99–103%) until colour change of the indicator..

3. Results and discussion

The experimental conditions used were those of the 77059 (2002) UNE regulation for the determination of aluminium in waters, and are described in Section 2 for the batch mode. The absorption spectrum from a solution formed with 120 μg/L Al^{3+} and 5 mL of a 0.4 g/L eriochrome cyanine R solution showed a single absorption maximum at 535 nm, and then this wavelength was selected to carry out quantitative measurements.

Under the recommended experimental conditions, the complex formed herein reached the highest colour intensity within 5–10 min after its formation and it became unstable after 15 min. Nevertheless, experimental measurements within these periods of time showed a high variability, and then a study regarding the complex stability was performed. In order to do that, Al^{3+} standard solutions comprised between 60 and 180 μg/L were prepared and the absorbance was measured at different times after the addition of eriochrome cyanine R. Calibration plots for Al^{3+} were obtained at periods of time between 5 and 110 min. The corresponding slope values (Fig. 2) exhibit a maximum and stable value (R.S.D. lower than 2%, $n = 8$) for periods of time between 8 and 40 min, a slight decrease being observed from 50 min. Therefore, subsequent absorbance measurements were carried out between 10 and 40 min from the complex formation, thus allowing a considerable amount of samples to be processed.

3.1. Validation of the batch method

The reliability of the results provided by the batch method was evaluated by checking the parameters included in the ISO-3543-1 regulation: selectivity, range of linearity, precision, accuracy, detection limit and quantification limit. It was done in order to be applied to the analysis of aluminium in anti-perspirant samples.

Potential interferences in the analysis of anti-perspirants include excipients, degradation products and/or impurities. In order to check the method selectivity, the signal provided by a placebo, supplied by the manufacturer laboratory (SUQUINSA, Madrid), was measured. The proportional amount of matrix without analyte that would be in 0.2500 g of sample (0.2178 g according to the nominal composition of the analyzed anti-perspirant) was subjected to the same procedure described for the real samples. The absorbance measured was 0.0007 units, which means a 0.2% of the signal obtained in the analysis of a real sample. Therefore, it can be concluded that the sample matrix does not produce any significant interference in the Al quantification by the batch method.

The other validation parameters are summarized in Table 1. The concentrations range tested for the linearity study was selected based on the expected Al content in the sample. Standard solutions of Al^{3+} were prepared by triplicate at five concentration levels comprised between 60 and 180 μg/L. The

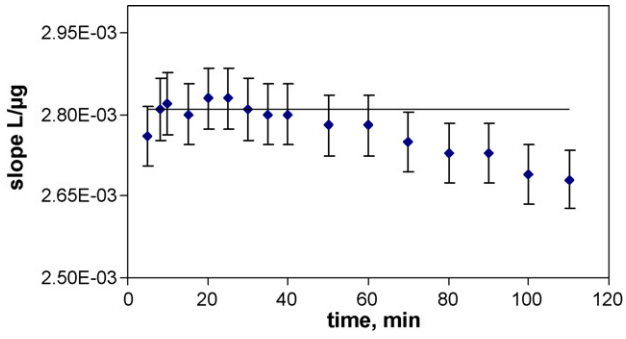

Fig. 2. Slope values of the Al^{3+} calibration graph between 60 and 180 μg/L obtained by the batch method using eriochrome cyanine R as a function of time.

Table 1
Validation parameters for the batch method of aluminium determination in anti-perspirants using eriochrome cyanine R

Standards linearity	$a \pm$ L.C.	-0.031 ± 0.016
	$b \pm$ L.C.	$(3.04 \pm 0.13) \times 10^{-3}$
	r	0.998
Instrumental precision	Mean $\pm$ L.C. ($\mu g\,L^{-1}$) (R.S.D.)	118.5 ± 0.8 (0.93%)
Method precision		
Intra-assay: day 1	Mean $\pm$ L.C. (%, w/w) (R.S.D.)	2.80 ± 0.04 (1.8%)
Intra-assay: day 2	Mean $\pm$ L.C. (%, w/w) (R.S.D.)	2.86 ± 0.03 (1.1%)
Inter-assay: day 1 + day 2	Mean $\pm$ L.C. (%, w/w) (R.S.D.)	2.83 ± 0.03 (1.8%)
Intermediate	Mean $\pm$ L.C. (%, w/w) (R.S.D.)	2.82 ± 0.03 (2.3%)
Sample accuracy	Recovery (%) (R.S.D.)	102 ± 4 (4.96%)
LOD	Concentration: solution	$16.6\,\mu g\,L^{-1}$
	Concentration: samples	0.33% (w/w)
LOQ	Concentration: solution	$32.0\,\mu g\,L^{-1}$
	Concentration: samples	0.64% (w/w)

a: intercept (absorbance units); b: slope ($L\,\mu g^{-1}$); L.C.: limits of confidence ($\alpha = 0.05$).

analysis of the slope value shows that it is significantly different from zero ($t_{exp} = 61.10 > t_{tab,.95,13} = 2.16$), thus indicating proportionality. However, the intercept value is also significantly different from zero ($t_{exp} = 4.11$) and does not include the zero value in its confidence interval, at the same significance level, either. This indicates the existence of bias, and, therefore, the signal provided by a sample should be interpolated at least between two standards. A random distribution of residuals was observed when they were plotted versus the concentration estimated value. Moreover, the analysis of the variance homogeneity for the response factors indicated that the concentration factor did not affect the variability of the results.

Concerning precision, the instrumental repeatability was tested by measuring 10 times the absorbance from a 120 μg/L standard solution. As it can be seen in Table 1, the R.S.D. value of the concentration calculated after interpolation into the calibration graph was lower than 1%. The overall precision of the method was calculated with eight replicates of the anti-perspirant sample (see Table 1). The results indicate a good intra-assay precision with R.S.D. values lower than 2% in all cases. The intermediate precision was checked to evaluate the method variability when some of the involved factors are modified. In this case, both the spectrophotometer and the day of the analysis were changed. An overall R.S.D. value lower than twice that obtained for the sample repeatability was achieved.

Accuracy was evaluated by performing recovery studies after adding to the above-mentioned placebo, aluminium chlorhydrate at five different concentration levels ranging between 50% and 150% of the nominal Al content in the anti-perspirant. Each addition was made in duplicate. As it is seen in Table 1, recoveries are close to 100%. The Cochran's test was applied to verify whether the concentration factor affected the variability of the results. The $G_{exp} = 0.3807$ was lower than $G_{tab} = 0.8412$ ($\alpha = 0.05$; $k = 5$; $n = 2$), indicating variance equality between the five sample groups.

The detection (LOD) and quantification (LOQ) limits were calculated according the IUPAC criteria, $3s_b/m$ and $10s_b/m$, respectively, where s_b is the standard deviation ($n = 10$) of blank (placebo) measurements, and m is the slope of the calibration graph. The respective values are included in Table 1, expressed both as the Al concentration in solution and the corresponding Al percentage (w/w) in the anti-perspirant.

3.2. Optimization of the FIA method

Taking into account the need for controlling the reaction time, the possibility of developing a FI method based on the reaction of Al^{3+} with eriochrome cyanine R was evaluated. The used FI arrangement was that shown in Fig. 1, and the different variables that can affect the absorbance were optimized with the aim to obtain the highest sensitivity in the shortest possible time and at the lowest possible cost. Flow rates of the carrier solution (acetic acid/acetate buffer of pH 6) and of chromogenic reagent (at a 0.4 g/L concentration) were firstly optimized, by injecting 50 μL of a 600 μg/L Al^{3+} standard solution, and using a 150 cm reactor. A three-level factorial design was employed to check the effect of these two factors. The flow rates of both solutions were varied between 1.0 and 3.0 mL/min, and three Al injections were carried out in each experiment. The obtained experimental absorbance data were treated with Statgraphics Plus 5.1 (Statistical Graphics Corp.), and Table 2 shows the effect estimates for the variables as well as the interaction between them, using the p-value statistical parameter to confirm the significance of

Table 2
Effect estimates for the variables carrier flow and chromogenic reagent flow as well as for the interaction between them

Factor	Effect	S.E.	p-Value
CF(L)	0.038	0.010	0.0344[a]
EF(L)	−0.080	0.010	0.0044[a]
CF(Q)	0.036	0.018	0.1346
EF(Q)	−0.011	0.018	0.5785
CF × EF	0.026	0.013	0.1271

L: linear factor; Q: quadratic factor; CF: carrier flow rate; EF: eriochrome flow rate.
[a] Significant factors ($\alpha = 0.05$).

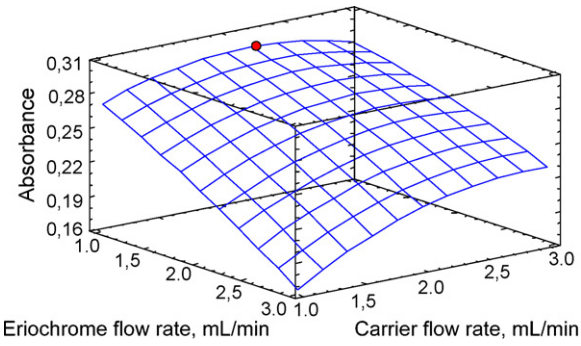

Fig. 3. Response surface showing the effect of eriochrome cyanine R and carrier flow rates on absorbance.

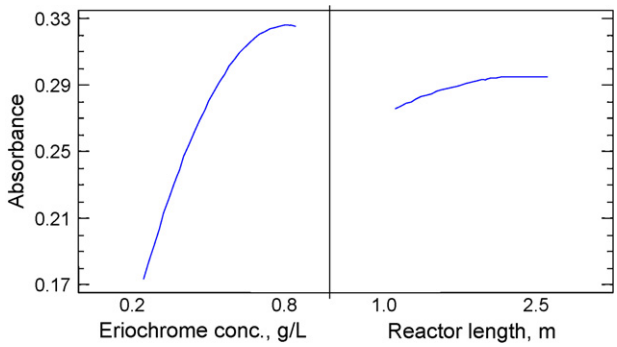

Fig. 4. Principal effect plot concerning the influence of eriochrome concentration and reactor length on absorbance.

the tested factors. As it can be seen, both independent variables had significant influence on absorbance ($\alpha = 0.05$), but not the interactions between them.

Using ANOVA, a second-order model describing the absorbance as a function of both variables checked was established. The following equation was obtained with a determination coefficient $R^2 = 0.9654$, explaining 96.54% of the variability in the response:

$$A = 0.2611 + 0.0651\,\mathrm{CF} - 0.0445\,\mathrm{EF} - 0.018\,\mathrm{CF}^2 + 0.0132\,\mathrm{CF}\,\mathrm{EF} - 0.0055\,\mathrm{EF}^2 \quad (1)$$

CF being carrier flow rate and EF eriochrome flow rate.

The model was used to generate a response surface, which is displayed in Fig. 3. As can be observed, an increase in the carrier flow rate produced an increase in absorbance between 1.0 mL/min and approximately 2.5 mL/min, but higher flow rates gave rise to lower responses. Moreover, an increase in the eriochrome cyanine R flow rate led to a decrease of absorbance, which can be attributed to a decrease in the reaction time and therefore to the amount of the complex formed. The model predicted the highest absorbance at a carrier flow rate of 2.16 mL/min and at an eriochrome cyanine R flow rate of 1.0 mL/min. As working values, 2.2 and 1.0 mL/min were taken for the carrier and the chromogenic reagent flow rates, respectively. It should be noted that flow rates of eriochrome cyanine R lower than 1.0 mL/min produced a decrease in the absorbance.

Following a similar methodology, the reactor length and the concentration of erichrome cyanine R were also optimized as they are variables that may be interrelated. The eriochrome concentration (EC) was varied between 0.2 and 0.8 g/L and the reactor length (RL) between 100 and 250 cm. As can be seen in the principal effects plot (Fig. 4), the factor affecting more to the absorbance is the chromogenic reagent concentration, which, as expected, produced an increase in the response with higher concentrations. Concerning the reactor length, its effect is much less important and there is not a significant increase in the signal when that length increases. The fitted model provides the corresponding quadratic equation, with a $R^2 = 0.986$, the optimum parameters being 235 cm for the reactor length and 0.77 g/L for the eriochrome cyanine R concentration. However, data analysis permits to conclude that, for a given reactor length, only a slight increase in the absorbance (around 6%) was produced by increasing the concentration of eriochrome cyanine R from 0.6 to 0.8 g/L. Considering that the more concentrated solution implied a higher reagent consumption and showed some solubility problems, a 0.6 g/L concentration was decided to be used as the loss in sensitivity was not important.

Regarding the reactor length, although it has not an important effect on the signal, its increase gave rise to longer time of analysis, larger peak width and a decrease in the reproducibility. So, 10 successive injections of a 600 μg/L Al^{3+} solution, using 0.6 g/L eriochrome cyanine R, yielded R.S.D. values lower than 2.0% for reactor lengths of 2.0 m or shorter while the R.S.D. was of 3.13% for a 3.0 m reactor. Consequently, the 2.0 m reactor was selected for further work.

3.3. Validation of the FIA method

Once the FIA method was optimized, its validation was accomplished. A calibration graph for Al^{3+} was constructed with analyte standard solutions ranging from 200 and 1200 μg/L and by measuring the absorbance in triplicates. Linearity was lost for concentrations higher than 1000 μg/L. The signal outputs corresponding to Al^{3+} concentrations comprised between 150 and 900 μg/L are displayed in Fig. 5, and the parameters of this linear portion are summarized in Table 3. The slope value is significantly different from zero ($t_{exp} = 99.56 > t_{tab} = 2.12$; $\alpha = 0.05$) and it does not include the zero value in its confidence interval ($\alpha = 0.05$). Similarly to the batch method, the intercept is also significantly different from zero ($t_{exp} = 8.48$). Consequently, the method has a bias and interpolation between at least two points of the calibration graph will be needed for sample analysis. In addition, the residuals distribution exhibited a random behaviour.

Both the instrumental and method precision were calculated. In order to do that six replicates of the same anti-perspirant sample were analyzed. Concerning instrumental precision, the same analytical solution was injected in the FIA system for 10 times. The corresponding absorbance values were interpolated into the calibration graph, and the R.S.D. value of the Al concentration was of 1.32% indicating a very good precision. Regarding the method precision, the analysis of the six sample replicates yielded a mean Al concentration in the anti-perspirant of 2.67 ± 0.03%, with a R.S.D. value of 0.9%. The values of the

Table 3
Validation parameters for the FIA method of aluminium determination in anti-perspirants using eriochrome cyanine R

Standards linearity	$a \pm$ L.C.	-0.030 ± 0.007
	$b \pm$ L.C.	$(6.03 \pm 0.13) \times 10^{-4}$
	r	0.9992
Precision		
Instrumental precision	Mean ± L.C. ($\mu g\,L^{-1}$) (R.S.D.)	568 ± 5 (1.32%)
Method precision	Mean ± L.C. (%, w/w) (R.S.D.)	2.67 ± 0.03 (0.9%)
LOD	Concentration: solution	16.1 $\mu g\,L^{-1}$
	Concentration: samples	0.080% (w/w)
LOQ	Concentration: solution	29.0 $\mu g\,L^{-1}$
	Concentration: samples	0.14% (w/w)

a: intercept (absorbance units); b: slope (L μg^{-1}); L.C.: limits of confidence ($\alpha = 0.05$).

detection and quantification limits shown in Table 3 were calculated according to the same criteria commented for the batch method. Although lower limits should be expected for batch measurements, as reaction reaches completeness, and therefore a higher signal, the opposite results were obtained. They can be justified with a higher standard deviation in the blank sample measurement (0.0067 a.u.) in the batch as compared with the FIA method (0.000857 a.u.). This is probably due to much lower time dispersion for automatic measurements.

The accuracy of the method was evaluated by comparison of the results with those provided by a reference method, which is the one currently used for the routine analysis of Al in the anti-perspirant samples. The reference method, described in Section 2, consisted of a complexometric back titration. Six replicates were prepared from the same sample and were independently analyzed with each method. The obtained results are summarized in Table 4. Statistical analysis of the values showed that $F_{exp} = 1.36$ was lower than $F_{tab}(\alpha = 0.05; 6; 6) = 5.05$, indicating homogeneity of variances between both methods. The mean values comparison test gave a $t_{exp} = 2.15$, lower than $t_{tab,\,.95,\,10} = 2.23$, and therefore it can be concluded that the results obtained by both methods are not significantly different for a confidence level of 95%, thus confirming the good accuracy of the proposed method.

Table 4
Comparison of the results obtained for the analysis of aluminium in anti-perspirant samples using a FIA method with eriochrome cyanine R and a complexometric back titration

	Reference method (volumetric)	FIA method
Al concentration (%, w/w)	2.69	2.63
	2.71	2.69
	2.72	2.66
	2.72	2.66
	2.67	2.68
	2.68	2.70
Mean ± L.C (%, w/w)	2.70 ± 0.02	2.67 ± 0.03

L.C.: limits of confidence ($\alpha = 0.05$).

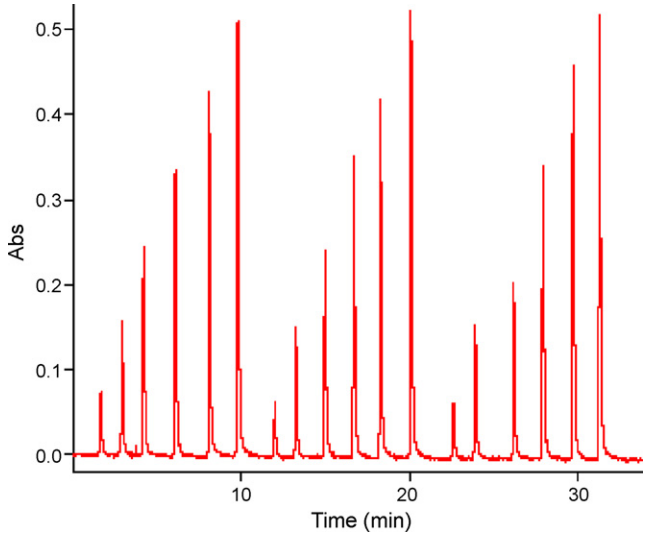

Fig. 5. Output signal corresponding to Al^{3+} concentrations between 150 and 900 µg/L obtained by the FI method with eriochrome cyanine R.

Moreover, under the optimized conditions, the response time (time needed to reach the peak maximum after the injection) was of 12 s, with peak mean width of 7 s, which allows a sample analysis, by triplicate injection, to be carried out in 1 min.

A proper assay for robustness testing was not performed, but this method has been working in our laboratory for real samples batch liberation during around 2 years with good results.

4. Conclusion

A FIA method with spectrophotometric detection for the determination of aluminium in anti-perspirants has been developed by detecting the complex formed by Al ions and eriochrome cyanine R. Validation of the method exhibits analytical figures of merit which makes it highly appropriate to substitute the tedious complexometric method currently used in pharmaceutical/cosmetic laboratories to carry out this type of analysis. The FIA method developed is rapid, precise, accurate and of low cost and therefore we think that it will be of high interest for laboratories of quality control dealing with this kind of samples.

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