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Problem:

The classical wet chemistry based methods used to detect food constituents are time consuming and difficult to perform for routine analysis.

Objective:

To compare the performance of a flow injection analysis (FIA) system and Fourier transformed infrared spectroscopy (FTIR) in relation to conventional methods in terms of automatization, sample and reagent consumption for the detection and quantification of food constituents (i.e. NaCl).

Sampling:

▪ Samples of table and marine salt were collected from market places and from natural sea salt producers from Portugal.



Figure 1. Natural salt pans 'salinas' in Aveiro, Portugal.

▪ Salt is an ingredient extensively used in the food industry with important properties as bacteriostatic and flavouring agent.

Application to samples:

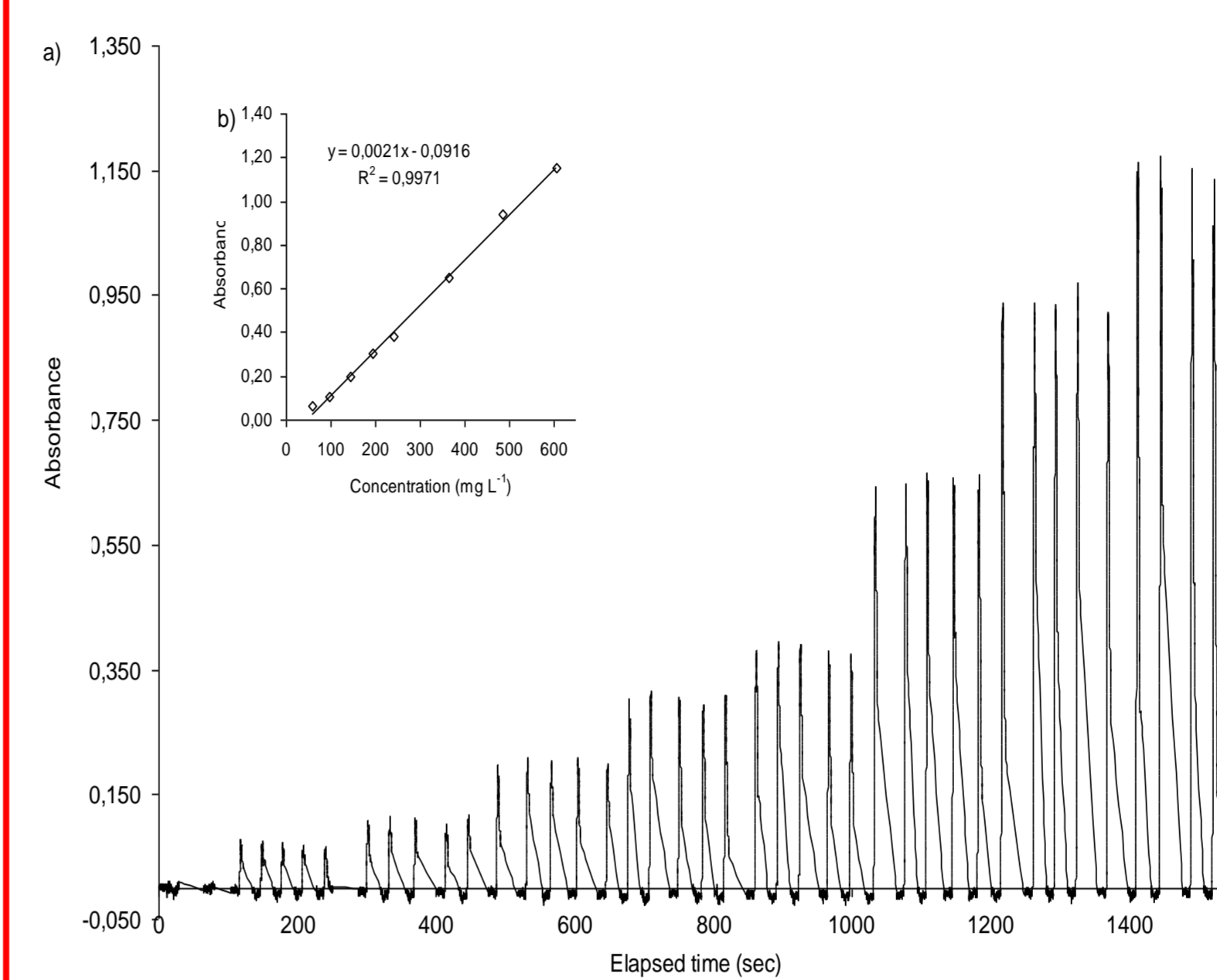


Figure 4. Flow injection peaks (a) and calibration curve (b) of chloride working standard solutions ranging from 0.05 to 0.6 g L⁻¹.

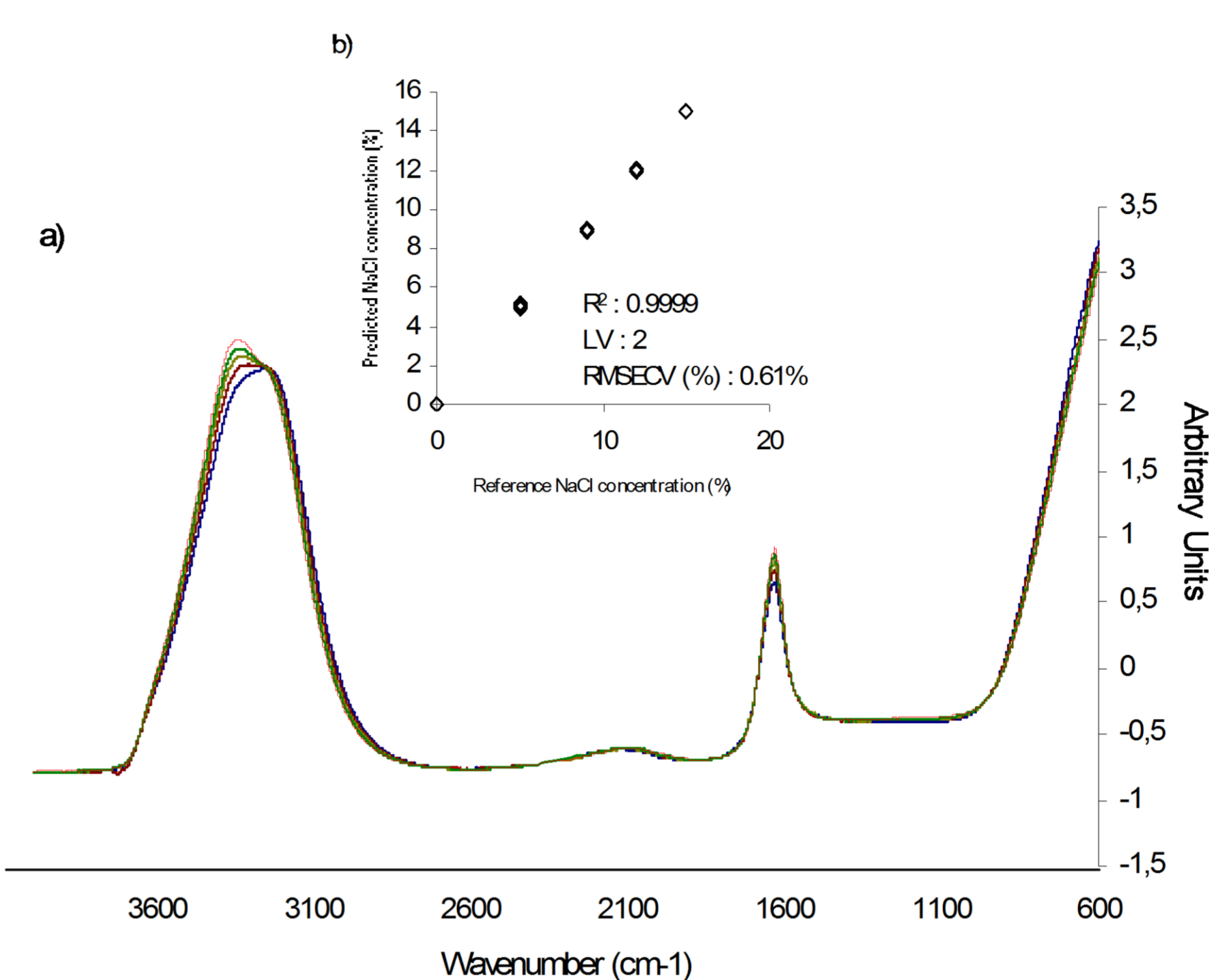


Figure 5. Mid-infrared spectrum (a) and calibration curve (b) of chloride working standard solutions ranging from 50 to 150 g L⁻¹.

Table 2. Determination of chloride in table and marine salt samples by FIA, FTIR-ATR and reference methods and corresponding relative deviations (RD).

Sample origin	Sample number	FIA ± sd	FTIR-ATR ± sd	Ref. Method ± sd	
		(% NaCl)	(% NaCl)	% NaCl	RD (%) ^a
Spain	1	97.5±0.2	95.9±0.05	97.4±0.4	0.03
Aveiro, Portugal	2	93.3±0.9	85.8±0.04	94.7±0.8	-1.5
Algarve, Portugal	3	96.1±0.9	91.3±0.05	96.3±0.2	-0.2
Aveiro, Portugal	4	94.7±0.05	86.9±0.05	94.7±1.9	0.1
France	5	95.2±1.1	92.8±0.04	96.9±0.5	-1.7
Algarve, Portugal	6	99.6±1.4	101.0±0.01	95.2±0.2	4.6
Aveiro, Portugal	7	89.10±0.2	70.6±0.07	87.7±0.10	1.6
Algarve, Portugal	8	98.1±0.1	88.5±0.03	96.3±0.1	1.9
Algarve, Portugal	9	96.5±0.9	87.9±0.02	96.2±0.06	0.4
Algarve, Portugal	10	93.8±0.3	86.4±0.05	96.3±0.02	-2.5

^a Relative deviations of the FIA method compared with the reference method

^b Relative deviations of the FTIR-ATR method compared with the reference method

Table 3. Analytical characteristics of the FIA and FTIR-ATR systems for the determination of sodium chloride.

	FIA	IR spectroscopy
LOD (g L ⁻¹)	0.03	1.6
LOQ (g L ⁻¹)	0.10	5.2
Working range (g L ⁻¹)	0.05 – 0.6	50 – 150
Determination rate / h	40	20
Reagent consumption		Not used
Silver Nitrate	0.24 g h ⁻¹	
Polyvinyl alcohol	0.14 g h ⁻¹	
Nitric acid	90 ml h ⁻¹	

LOD: limit of detection

LOQ: limit of quantification

Manifolds and Operation Conditions

1. FIA System

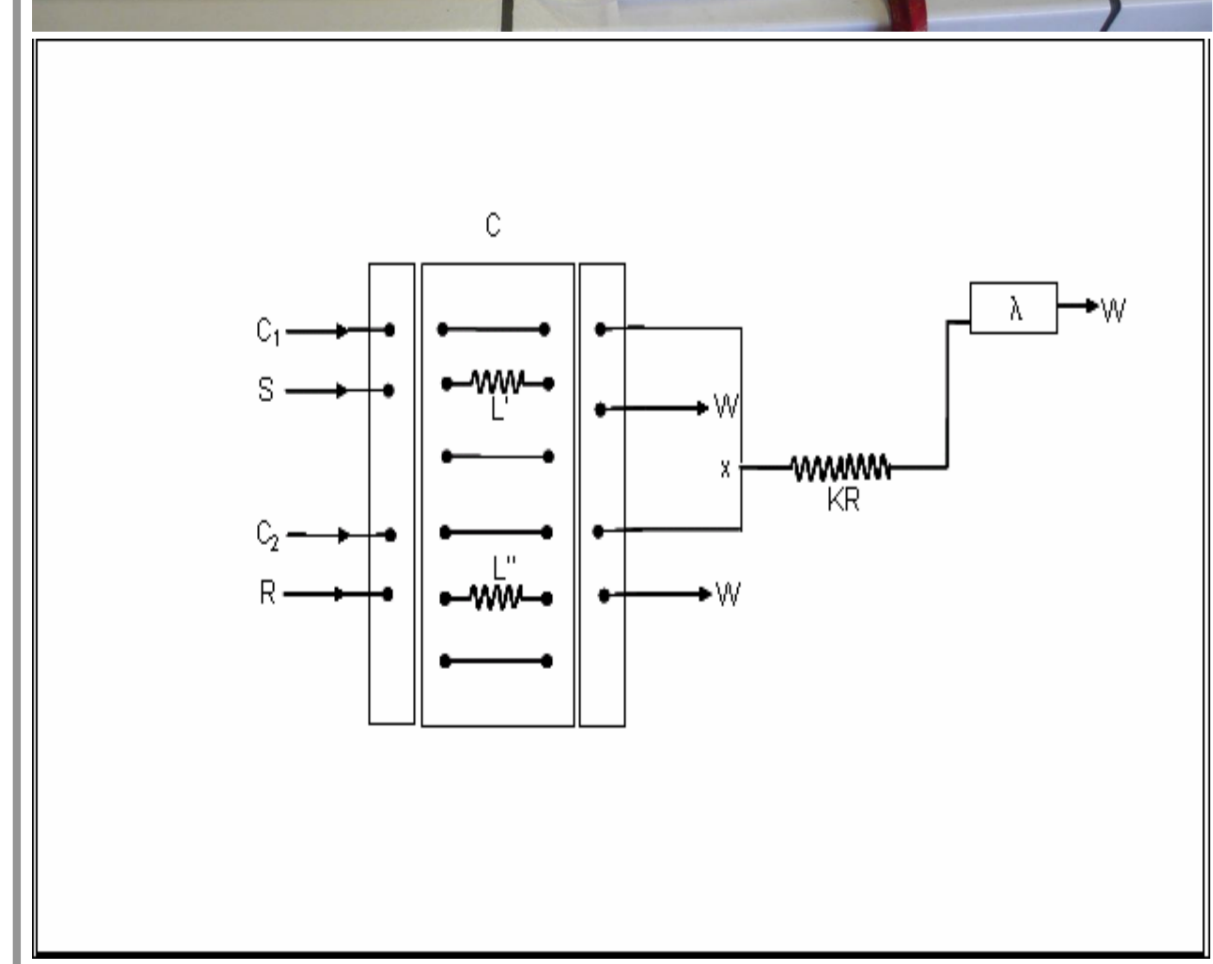
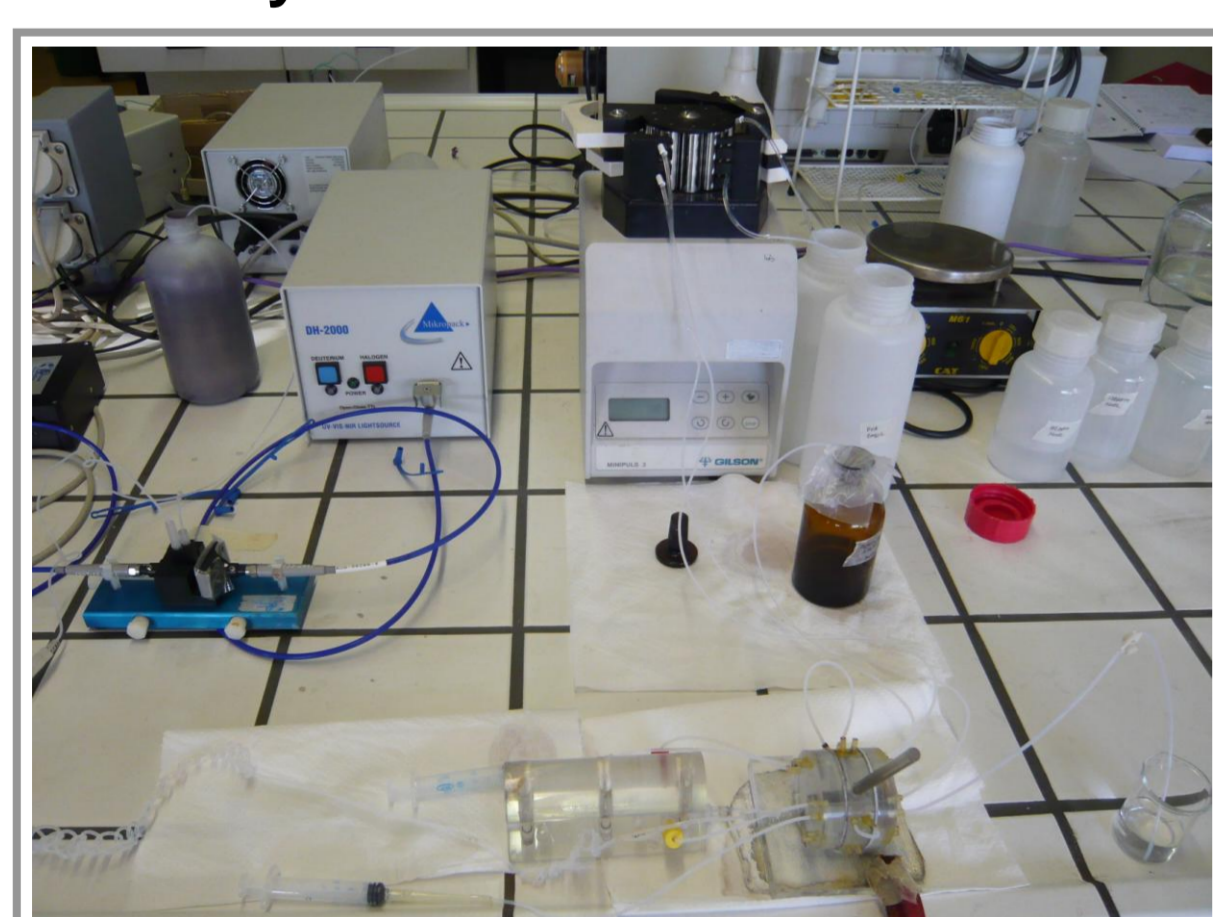


Figure 2. Flow injection analysis (FIA) manifold for the turbidimetric determination of chloride: S, sample or standard; C1, water; C2, nitric acid (2 mol L⁻¹); R, silver nitrate (1 × 10⁻² mol L⁻¹); L1, loop sample (15 μL); L2, loop silver nitrate (60 μL); C, commutator; KR, 200 cm knitted reactor; λ, UV/VIS spectrophotometer; W, waste.

2. FTIR-ATR System

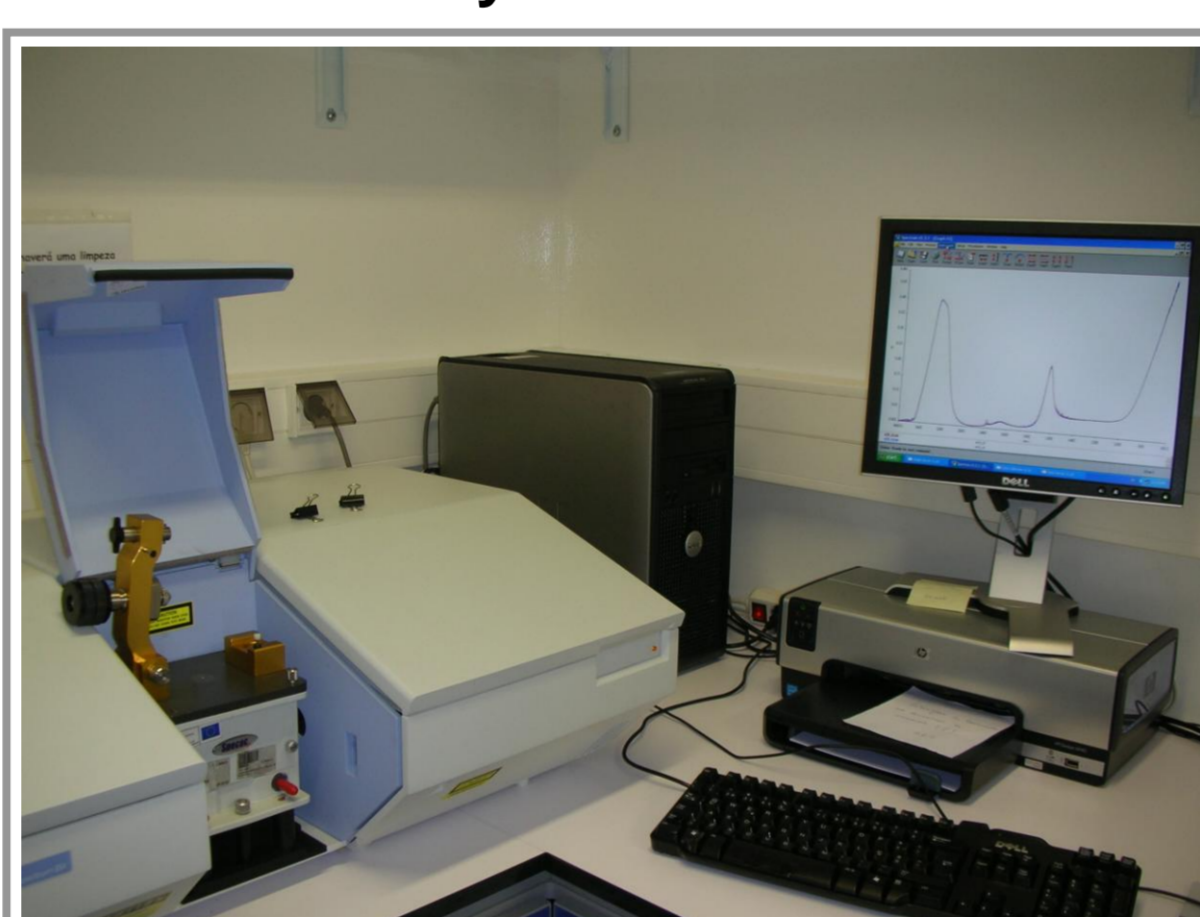


Figure 3. FTIR-ATR System.

Table 1. Characteristics of the FIA and IR-Spectroscopy detection systems

	FIA	IR spectroscopy
Mode	Turbidimetric	Transmittance
Detection system	Spectrophotometer (CCD-array detector, Ocean Optics)	Fourier Infrared – Spectrometer (Spectrum BX, Perkin–Elmer)
Spectral range	9090 – 50000 cm ⁻¹ (λ=425 nm)	600 – 4000 cm ⁻¹
Accessories	Peristaltic pump (Gilson, Manipuls 3) Cuvette holder (Ocean Optics) Optical fibers (i.d: 300μm) Tungsten/halogen light source (DH-200, Mikropack)	ATR Golden Gate (Specac)
Software	SpectraSuite	Spectrum v.5.3.1 and CATS program ^[1]

Reference

[1] Barros, A. 1999. Contribution à la sélection et la comparaison de variables caractéristiques. Ph. D. Thesis. Institut National Agronomique Paris-Grignon Paris. France.

Acknowledgements

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Conclusions:

- The developed FIA and FTIR methodologies allow determination of sodium chloride over a wide concentration range.
- The FIA methodology offers several advantages: high sampling rate, low cost of implementation and simple interpretation of the results.
- The FTIR-ATR methodology does not make use of reagents and allows the detection, if necessary, of other constituents in the matrix by the analysis of a single spectrum.
- The selection of any of these methodologies for food analysis will depend on the necessities, person's skill capacities and price that the food industry is willing to pay for the automatization of its analytical determinations.