A DOUBLE-LINE SEQUENTIAL INJECTION SYSTEM USING A LONG PATHLENGTH LIQUID WAVEGUIDE CAPILLARY FLOW CELL FOR THE SPECTROPHOTOMETRIC IRON DETERMINATION IN WATERS

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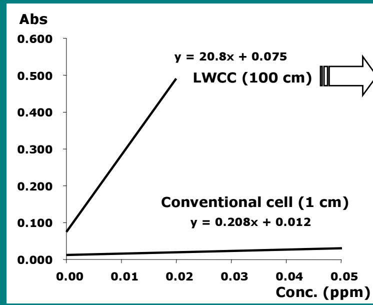
Introduction

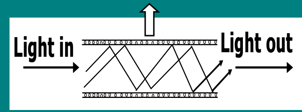
Iron is present at abundance in earth crust, although in waters it appears in very low concentrations. The objective of this work is to determine trace levels of iron using spectrophotometric detection, based on the reaction with ferrozine.

A long liquid waveguide capillary flow cell with 100 cm of optical path was applied to increase the sensitivity of the iron determination.

A double-line sequential injection analysis (SIA) system was developed to automate this determination.



i.d.=550 µm; i.v.= 250 µL



Total internal reflection by the wall of the flow cell

Pathlength can be increased

Great improvement of sensitivity
(without deteriorating other analytical caracteristics)

Manifold

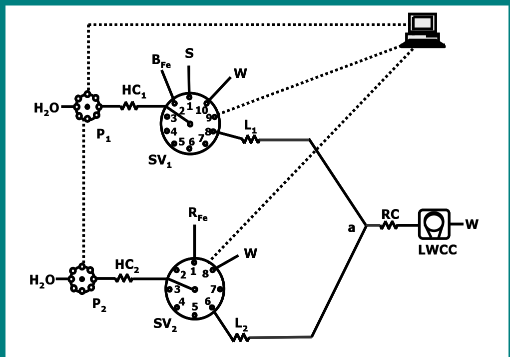


Fig. 1. Double line sequential injection manifold for the determination of iron in waters. SV_1, SV_2: selection valves; P_1, P_2: peristaltic pumps; HC_1, HC_2: holding coils (2 m); RC: reaction coil (85 cm); L_1, L_2: reactors (25, 14 cm); a: confluence; LWCC: liquid-core waveguide capillary flow cell (100 cm of optical path); W: waste; S: sample or standard; BFe: acetate buffer solution; RFe : color reagent (ferrozine).

Protocol sequence

Step	Selection valves positions		Operation time (s)	Flow rate (mL/min)		Description
	Selection valve 1	Selection valve 2		Pump 1	Pump 2	
1	1	1	9.1	1.66	0.56	Aspirate sample and ferrozine reagent
2	2	1	3.0	0.77	0.28	Aspirate buffer and ferrozine reagent
3	8	6	50	3.81	1.16	Propel towards detector and signal registration

Figures of merit

Detection limit (ppb)	0.15 ± 0.01
Quantification limit (ppb)	0.49 ± 0.04
Working range (ppb)	0.15 - 20
Determinatin rate/h	41
Reagent consumption (mmol/assay)	
Ferrozine	0.00025
Ammonium acetate	0.080
Acetic acid	0.080
Ascorbic acid	0.0045
Waste produced (mL/assay)	4.14

Interference studies

Added species	Tested concentration* (ppb)	Relative deviation (%)
Zinc	1000	5.00
Aluminium	1000	3.73
Cadmium	1000	20.9
Manganous	1000	11.7
Copper	10	7.56

* Using a standard solution of 5 ppb of iron

Application to water samples

Concentration of iron added (ppb)	Recovery (%)			
	Sample 1	Sample 2	Sample 3	Sample 4
2	99 ± 9	91 ± 3	93 ± 2	99 ± 4
4	99 ± 5	98 ± 2	92 ± 5	94 ± 2
10	105 ± 2	103 ± 2	95 ± 1	97 ± 2
20	104 ± 5	102 ± 2	93 ± 1	97 ± 2

Sample 1- Groundwater; Sample 2- Tap water; Sample 3 and 4- Seawater; n=6

Acknowledgements

Ricardo Páscoa and Ildikó Tóth acknowledge financial support from FCT and FSE (III Quadro Comunitário) through the grants SFRH/BD/30621/2006 and SFRH/BPD/5631/2001, respectively.