

Analysis of Anions using IC with chemical suppression

Introduction

ICSpak is a suppressor technology for anion chromatography developed by JASCO. The packing material has a sulfonic acid group chemically bonded to a styrene-divinylbenzene porous polymer and packed in a PEEK column. The suppressor is used in anion chromatography to decrease the background signal from the eluent and to change the sample to an ion-pair with high equivalent conductivity. This suppressor can be re-cycled using regeneration and washing steps. The more sensitive detection of nitrate and nitrite ions is made using a UV detector.

This report describes the analysis of anions using inert system and ICSpak.

Keyword: Chemical suppressor, anion, Shodex IC SI-90 4E, ICSpak, inert

Experimental

[Equipment]

Pump: PU-4080i
 Pump option: DG unit
 Autosampler: AS-4050i
 Column oven: CO-4060
 Detector: UV-4075 (Inert Cell)
 Shodex CD-5

[Conditions]

Column: Shodex IC SI-90 4E (4.6mmI.D. x 250mmL)
 Suppressor: ICSpak (4.6mmI.D. x 100mmL)
 Eluent: 1.8mM Na₂CO₃ + 1.7mM NaHCO₃
 Flow rate: 1.0mL/min
 Column temp.: 25°C
 Wave length: 215nm
 Injection volume: 100 µL
 Standard Sample: Anion Mixture (Described below)

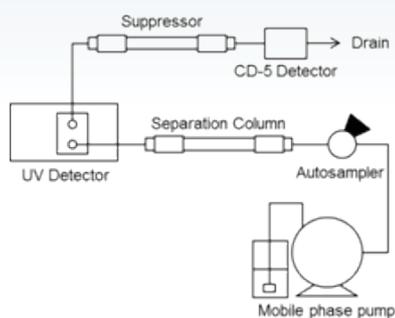


Fig.1 The flow path diagram

Table 1 The concentration of anion standard solution mixture

Anions	Conc. [mg/L]
F ⁻	0.2
Cl ⁻	0.3
NO ₂ ⁻	0.5
Br ⁻	1
NO ₃ ⁻	1
PO ₄ ³⁻	1.5
SO ₄ ²⁻	1.5

Results

Fig.2 Chromatograms of anion standard solution mixture. 7-component anions.

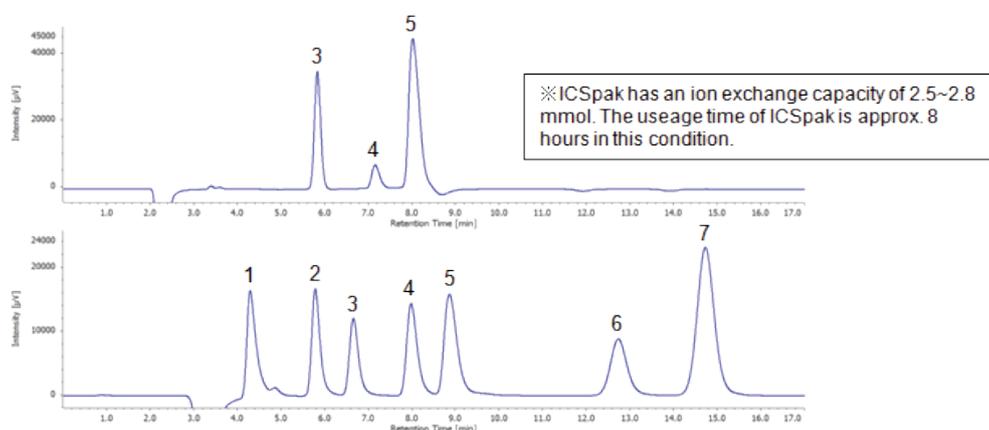


Figure 2. Chromatograms of anion standard solution mixture using Shodex IC SI-90 4E and chemical suppressor ICSpak Top: UV detector, Bottom: Conductivity detector 1: F⁻, 2: Cl⁻, 3: NO₂⁻, 4: Br⁻, 5: NO₃⁻, 6: PO₄³⁻, SO₄²⁻

Table 2 Detection limit for a range of anions.

Table 2 Detection limit values for anions

Anions	Concentration [mg/L]	Injection volume [ng]	UV S/N=3 [mg/L]	CD-5 S/N=3 [mg/L]
NO ₂ ⁻	0.5	50	0.89	1.46
Br ⁻	1	100	8.39	1.50
NO ₃ ⁻	1	100	1.33	1.34
F ⁻	0.2	20		0.32
Cl ⁻	0.3	30		0.40
PO ₄ ³⁻	1.5	150		3.67
SO ₄ ²⁻	1.5	150		1.45

The CO-4065 can be equipped with two optional valves. Analysis and regenerating/washing can be performed in parallel by using the CO-4065 with two 2-position/6-port switching valves with two ICSpak suppressor columns and a multi-solvent regenerating/washing pump (e.g., the PU-4180 fitted with a low-pressure gradient unit) as shown in the fig.3.

Experimental

[Equipment]

Pump: PU-4180
 Pump option: DG unit
 LPG unit
 R-Pump unit: R-PU-4080i
 Autosampler: AS-4050i
 Column oven: CO-4065
 Oven option: 2 way 6 port x 2
 Detector: UV-4075 (Inert Cell)
 Shodex CD-5

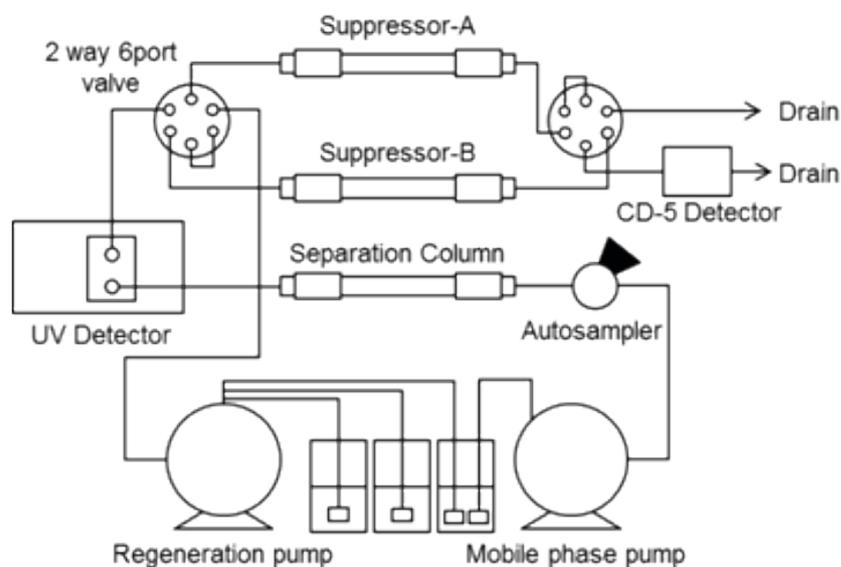


Figure 3 Flow path of a system for parallel analysis and regenerating/washing

System on page 1

Part No.	Model	Description
7002-J011A	PU-4180i	Inert HPLC Pump
7006-H003A	DG-4000-04	4-channel Degasser Unit, for Analytical
7018-J002A	AS-4050i	Inert Autosampler
7021-J002A	CO-4060	Column Oven
7026-J002A	UV-4075	UV-Visible Detector
7025-H136A		UV-4000 Inert flow cell unit
0507-1334	*CD-200	Conductivity Detector
7058-J011A	BS-4000-1	Bottle Stand
0507-2330	F6995244	Shodex IC SI-90 4E
0509-0457A		ICSpak Suppressor
6688-H564W	LC-Net CG	LC-Net Cable CG (x 3)
	ChromNAV V2	Chromatography Data System
7001-H402A		HPLC PEEK Start Up Kit for LC-4000
7001-H405A		Maintenance Tool Kit

CD-200 was used to detect electrical conductivity because CD-5 is not available currently.

System on page 2

Part No.	Model	Description
7002-J004A	PU-4180	RHPLC Pump (Base Unit)
7006-H003A	DG-4000-04	4-channel Degasser Unit, for Analytical
7007-H002A	LG-4080	Low Pressure Gradient Unit, w/o Tera mixer
7012-J002A	R-PU-4080i	Expand pump unit
7018-J002A	AS-4050i	Inert Autosampler
7023-J002A	CO-4065	Column Oven, Valve mountable model
9999		2 way 6 port x 2
7026-J002A	UV-4075	UV/VIS detector
7025-H136A		UV-4000 Inert flow cell unit
0507-1334	*CD-200	Conductivity Detector
7058-J011A	BS-4000-1	Bottle Stand
0507-2330	F6995244	Shodex IC SI-90 4E
0509-H169A		ICSpak supressor
6688-H564W	LC-NetCG	LC-Net cable CG (3 pieces)
ChromNAV V2		Chromatography Data System
7001-H402A		HPLC PEEK Start Up Kit for LC-4000
7001-H405A		Maintenance Tool Kit

*A CD-200 was used to detect electrical conductivity because the newer CD-5 was not available.

JASCO INC.

28600 Mary's Court, Easton, MD 21601 USA
 Tel: (800) 333-5272, Fax: (410) 822-7526
 Application Library: <http://www.jascoinc.com/applications>



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