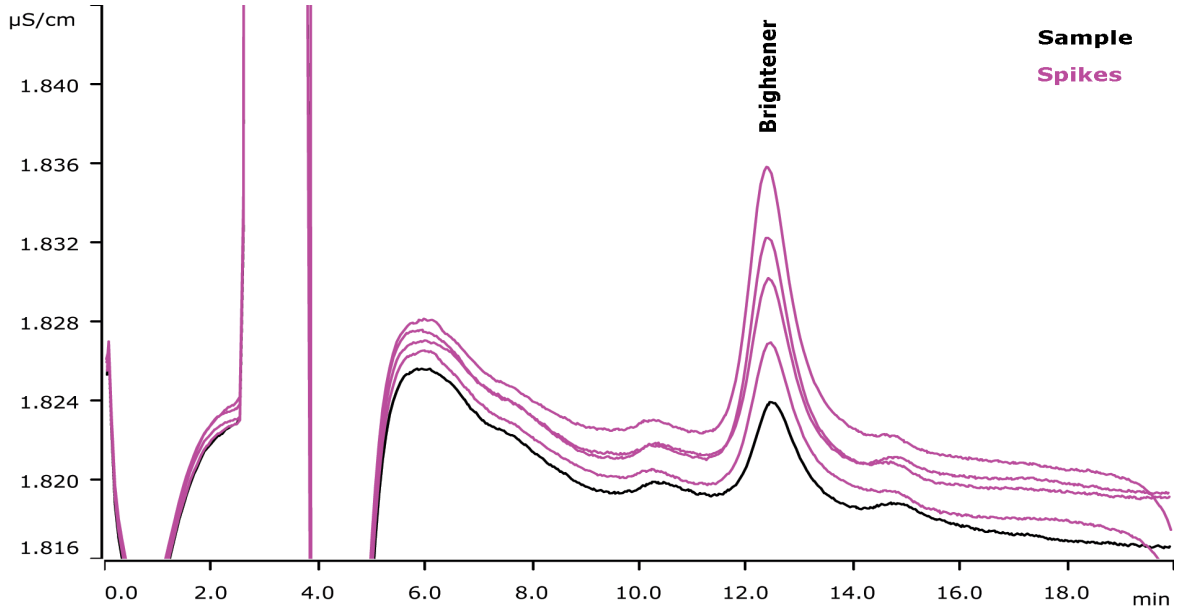
IC Application Note S–316

Brightener in aged copper plating bath by IC applying Inline Cation Removal and column switching



Brighteners are added to electroplating baths to improve the brightness of the final surface. The brightener concentration needs to be kept constant to maintain the plating quality. This Application Note describes the IC part of a simultaneous determination of a brightener by IC and CVS (cyclic voltammetric stripping). See AN-V-183 for similar CVS application.

Results

	Spike [µL/L]	Concentration [µL/L]	Recovery [%]
Brightener	-	538	-
1st spike	200	747	105
2nd spike	400	949	103
3rd spike	600	1185	108
4th spike	800	1418	110

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Sample

Aged copper plating bath

Sample preparation

Dilution 1:10 with ultrapure water, Inline Cation Removal

Columns

Metrosep A Supp 5 - 100/4.0 (1st dimension)	6.1006.510
Metrosep A Supp 4/5 Guard/4.0 (1st dimension)	6.1006.500
Metrosep A PCC VHC/4.0	6.1006.320
Hamilton PRP-1 (250/4.6, 5 µm) (2nd dimension)	82025-U*

* supplied by Sigma-Aldrich

Solutions

Eluent (both dimensions)	7.0 mmol/L sodium carbonate 3.0 mmol/L sodium hydrogen carbonate
SPM regenerant	1 mol/L sulfuric acid
Suppressor regenerant (both dimensions)	100 mmol/L sulfuric acid
Rinsing solutions	Ultrapure water

Analysis

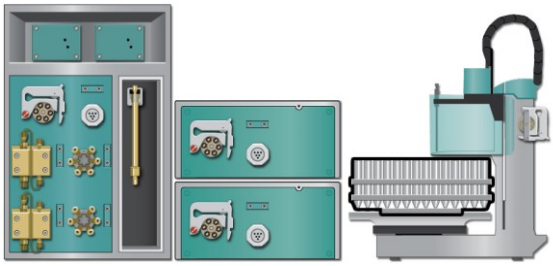
Separation applying two dimensional separation (cutting technique) First dimension: ion exchange Second dimension: reversed phase
Conductivity detection after sequential suppression

Parameters

Flow rate (system 1)	0.7 mL/min
Flow rate (system 2)	1.0 mL/min
Injection volume (system 1)	100 µL
Injection (system 2)	Metrosep A PCC VHC/4.0
P_{max} (system 1)	15 MPa
P_{max} (system 2)	15 MPa
Recording time (system 1)	35 min
Cutting window	20–30 min
Recording time (system 2)	20 min
Column temperature (both saytems)	30 °C

Instrumentation (IC)

940 Professional IC Vario TWO/SeS/PP	2.940.2500
2 × IC Conductivity Detector	2.850.9010
2 × Extension Module Vario SeS/PP	2.942.0500
858 Professional Sample Processor	2.858.0020
2 × MSM-HC Rotor A	6.2842.000
SPM Rotor A	6.2835.000



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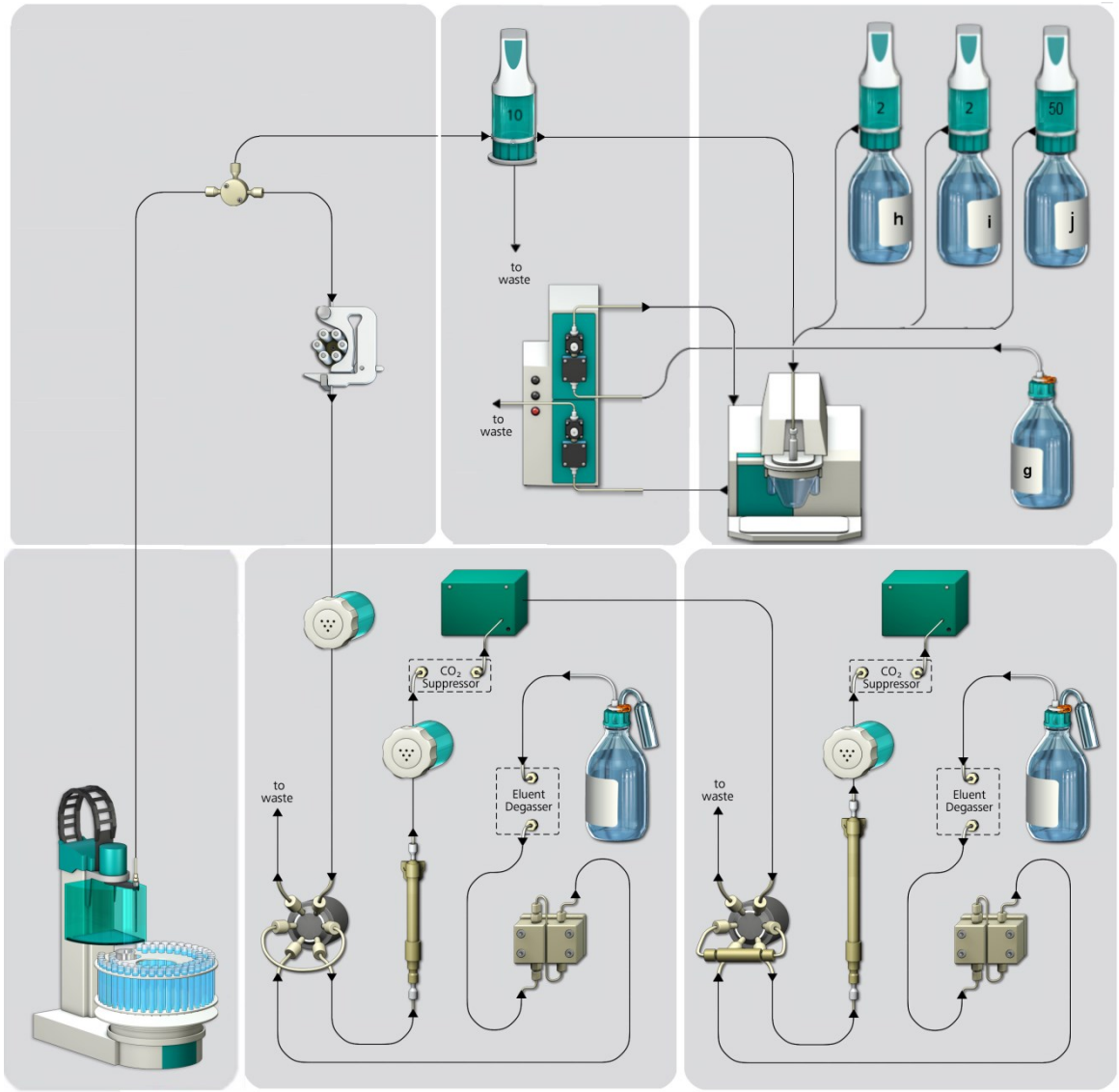
Flow chart: two-dimensional brightener determination (including CVS part)

Function: the 858 Professional Sample Processor keeps the samples for IC and CVS determinations. For CVS, an 800 Dosino delivers the sample solution to the CVS instrumentation (see AN-VA-183). For IC analysis, a peristaltic pump fills the sample loop through the SPM module. The SPM module removes the copper in the sample solution. On the first column, the matrix (sulfate) is preseparated. The brightener elutes at a similar retention time. To allow preconcentration of the brightener, the eluent undergoes sequential suppression. The first conductivity detector allows to set the correct cutting times.

After preconcentration of the respective volume, the preconcentration column is switched to the second IC system which now separates the hydrophilic anions (i.e., sulfate) from the more lipophilic brightener. After sequential suppression, the final chromatogram is recorded by the second conductivity detector.



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