

Saccharin in electrolytic nickel sulfate baths

Keywords

Nickel brightener, bright nickel sulfate bath, ion chromatography, IC, Dionex ICS-5000+, Dionex OmniPac PAX-500

Introduction

Saccharin decreases the tensile strength of electrolytic nickel deposits and can, with increasing concentrations, cause compressive stress. Sulfur can be introduced into the plate from the saccharin when class II nickel brighteners are not present, thus producing deposits with higher tensile strength, higher hardness, and relatively lower ductility. Saccharin, without other nickel brighteners present, produces a cloudy bright plate with relatively low luster.

Determination of saccharin by ion chromatography is faster and has fewer interferences than classical titrimetric methods of analysis. Labor requirements, thus operating costs, are significantly lower with ion chromatography than with classical titrimetric methods. Thermo Scientific™ Dionex™ ion chromatography systems are constructed from inert polymeric materials in the eluent flow path so that an eluent containing 30 mM sulfuric acid can be used and highly corrosive nickel bath samples can be injected directly into the system without corroding system components.

Application areas for saccharin as a class I nickel brightener include:

- Watts nickel bath
- Nickel chloride bath
- Nickel chloride-acetate bath
- Nickel fluoroborate bath
- Hard nickel bath
- Nickel chloride-sulfate bath
- Nickel cobalt bath
- Nickel sulfamate bath

Experimental

Recommended equipment

- Thermo Scientific™ Dionex™ DX 500 Ion Chromatography system* consisting of:
 - GP40 Gradient Pump with vacuum degassing option
 - AD20 Absorbance Detector
 - Autosampler
- Thermo Scientific™ Dionex™ PeakNet™ Chromatography Workstation

* Equivalent or improved results can be achieved using the Thermo Scientific™ Dionex™ ICS-5000+ HPIC™ system with a Thermo Scientific™ Dionex™ ICS-Series VWD Variable Wavelength Detector.

Sample preparation

The sample was diluted to 1/500 the original concentration with deionized water before direct injection.

Solutions and reagents

Eluent 1: 30 mM sulfuric acid/5% acetonitrile

Place approximately 500 mL of deionized water in a 1 L volumetric flask. Add 50 mL of HPLC-grade acetonitrile and mix. Add 1.7 mL (3.1 g) concentrated sulfuric acid and mix. Dilute to 1 L with deionized water.

Eluent 2: 30 mM sulfuric acid/25% acetonitrile

Place approximately 500 mL of deionized water in a 1 L volumetric flask. Add 250 mL of HPLC-grade acetonitrile and mix. Add 1.7 mL (3.1 g) concentrated sulfuric acid and mix. Dilute to 1 L with deionized water.

Standard: 1000 mg/L saccharin

Dissolve 1.126 g of sodium saccharin in deionized water. Dilute to 1 L.

Conditions

Column:	Thermo Scientific™ OmniPac™ PAX-500
Eluents:	A. 30 mM Sulfuric acid; 5% acetonitrile
	B. 30 mM Sulfuric acid; 25% acetonitrile
Flow Rate:	1 mL/min
Inj. Volume:	25 µL
Detection:	UV, 225 nm
Gradient Program:	
<i>Time</i>	<i>%A</i> <i>%B</i>
0.0	100 0
20.0	0 100
30.0	0 100

Results and discussion

Saccharin, a class I brightener in electrolytic nickel plating baths, was determined in a bright nickel sulfate bath using ion chromatography. The method was linear for injected samples with saccharin in the concentration range between 0.25 mg/L and 250 mg/L. Saccharin was well resolved from other UV-absorbing species present in older working baths. It is recommended that the column be reequilibrated with eluent A for 10 minutes prior to the next run.

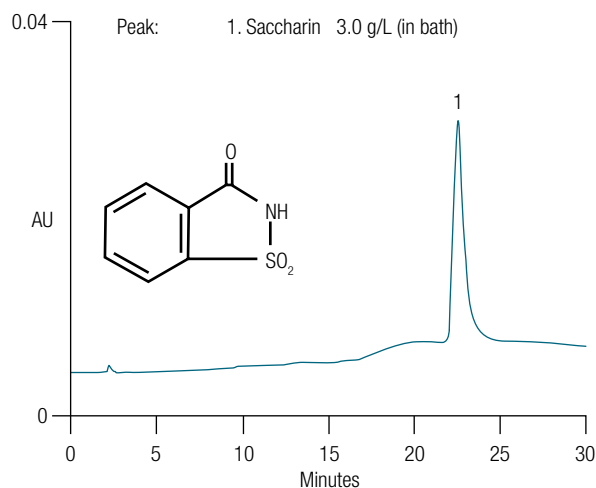


Figure 1. Saccharin in a new, fresh electrolytic nickel sulfate bath.

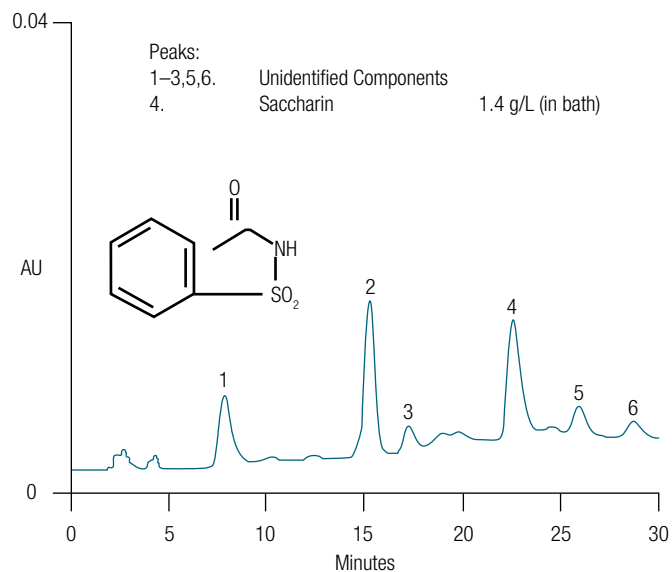


Figure 2. Saccharin in a working electrolytic nickel sulfate bath.

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