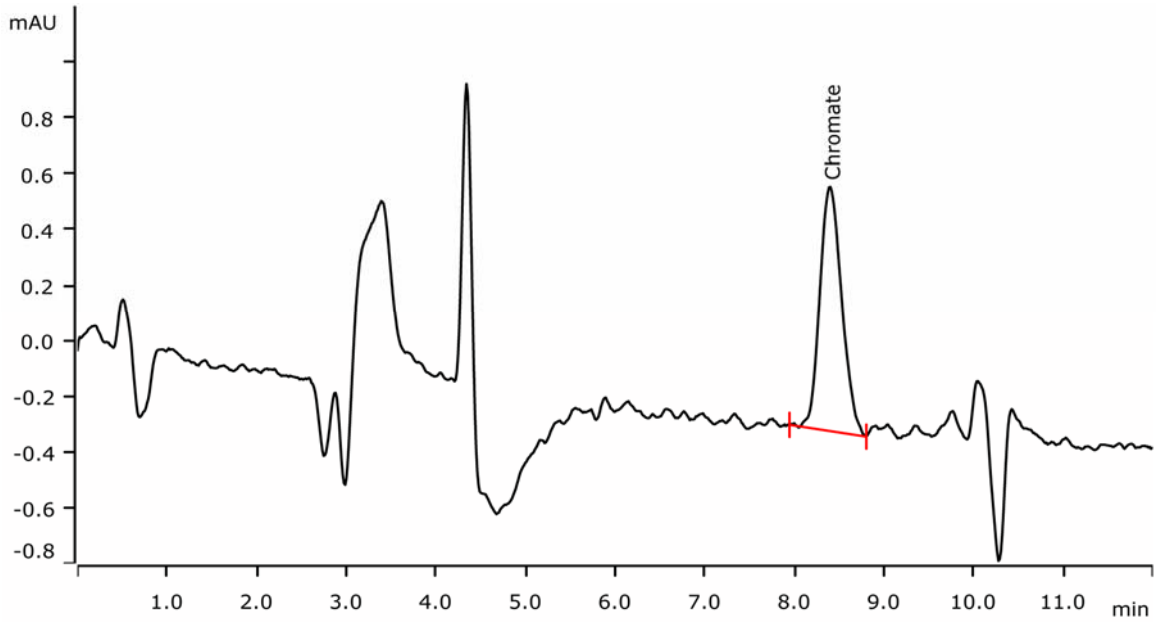
IC Application Note U–58

Chromate in dye samples using post-column reaction and subsequent UV/VIS detection



Dye samples are analyzed for trace chromate. Chromate (Cr(VI)) is considered toxic and potentially carcinogenic for which reason its concentrations should be as low as possible. This sample is prepared with C18 cartridges and injected applying Metrohm intelligent Preconcentration Technique (MiPCT). After each injection, the preconcentration column requires additional rinsing to eliminate matrix effects. For this purpose, no other instrument than an 800 Dosino is required. The system is optimized for sample volumes between 20 and 2000 µL. For most samples, additional rinsing of the preconcentration column is not required.

Results

	Concentration	Recovery
Chromate	0.20 µg/L	
Chromate spiked (2 µg/L)	2.20 µg/L	100%
Chromate in dye	0.8 mg/kg	

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Sample

Water-soluble dye

Sample preparation

Dissolution in ultrapure water, washing over C18 cartridge (6.1012.050) before preconcentration

Columns

Metrosep A Supp 5 - 250/4.0	6.1006.530
Metrosep A Supp 4/5 Guard/4.0	6.1006.500

Solutions

Eluent (inline eluent preparation)	12.8 mmol/L sodium carbonate 4.0 mmol/L sodium hydrogen carbonate 2.5 g/L ammonium sulfate
PCR reagent	2.0 mmol/L 1,5-diphenyl-carbazide
Adjustment buffer	33 g/L ammonium sulfate 65 mL/L ammonium hydroxide (25%)
Cleaning solution	50% acetone (v/v)

Parameters

Flow rate	0.8 mL/min
Flow rate PCR	0.2 mL/min
Injection volume	1500 µL
P_{max}	15 MPa
Recording time	12 min
Column temperature	45 °C
PCR temperature	45 °C
Light source (VIS)	Tungsten halogen lamp
Wavelength	538 ± 21 nm
Reference	650 ± 21 nm

Instrumentation

881 Compact IC pro – Cation	2.881.0010
858 Professional Sample Processor – Pump	2.858.0020
886 Professional Reactor	2.886.0110
887 Professional UV/VIS Detector	2.887.0010
2 x 800 Dosino	2.800.0020

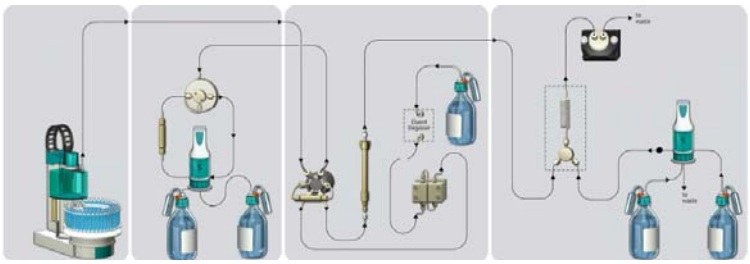
Analysis

Visible detection after PCR

Calibration MiPCT-ME

Calibration range	Factor of 100
Standard solution:	
Chromate	2.00 µg/L
1. Level	20 µL = 0.02 µg/L
2. Level	40 µL = 0.04 µg/L
3. Level	100 µL = 0.10 µg/L
4. Level	200 µL = 0.20 µg/L
5. Level	400 µL = 0.40 µg/L
6. Level	1000 µL = 1.00 µg/L
7. Level	2000 µL = 2.00 µg/L

Flow chart



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