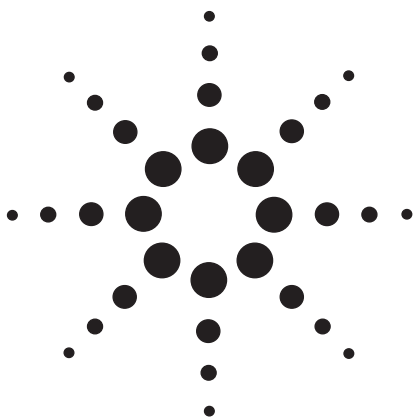




Analysis of Selected Anions with HPLC and Electrochemical Detection

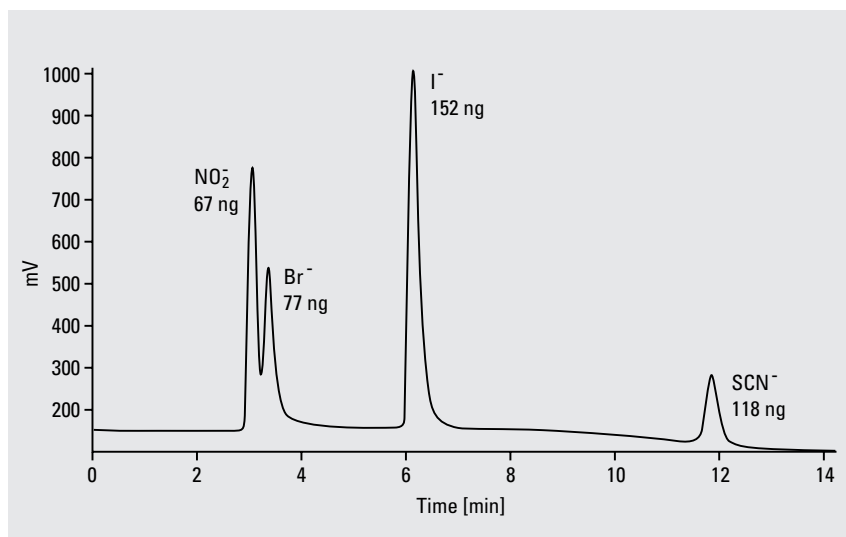
Application Note

Food

Angelika Gratzfeld-Huesgen
and Rainer Schuster

Traditionally, conductivity is the standard detection method for ion analysis. During the last 10 years, however, there has been increasing interest in electrochemical detection (ECD) for the analysis of anions. This is due to the high sensitivity and selectivity of this type of detection.

Figure 1 shows how certain anions can be selectively detected in the presence of several other anions. In total, 16 anions were injected. Four that can be oxidized: iodide, nitrite, bromide, thiocyanide, and twelve that cannot be oxidized: fluoride, chloride, azide, nitrate, phosphite, sulfate, molybdate, hydrogencarbonate, perchlorite, phosphate, selenate and perchlorate.



Chromatographic conditions

Column:	200 x 4 mm, Spherisorb ODS2, 5 μm
Mobile phase:	water with 5.2 g/l K_2HPO_4 + 3.6 g/l KH_2PO_4 + 3 g/l TBAHSO_4^* /ACN=85:15
Flow rate:	1 ml/min
Inj. volume:	0.1 μl
Potential:	1 V
Mode:	amperometry
Electrode:	glassy carbon

* Tetrabutylammoniumdihydrogensulphate

Figure 1
Analysis of standard



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In order to demonstrate the selectivity and sensitivity, iodide in table salt was analyzed. There is currently a great deal of interest in the determination of iodide in various foodstuffs, because some data indicates that the consumption of iodide has increased, leading to a potential rise in thyroid disorders. Table salt is almost 100% sodium chloride and

with conductivity detection a large chloride peak may overlap with the iodide. Figure 2 shows the selective detection of iodide in a large excess of chloride.

Detection potential is of great importance for selectivity and sensitivity and must be determined for each set of analysis parameters.

The Agilent 1049A ECD in combination with an auto-sampler provides a time-saving way to find the optimum potential for several compounds through its auto-increment mode. The potential is automatically increased within a series of runs and the best detection potential can be selected from the chromatogram plots.

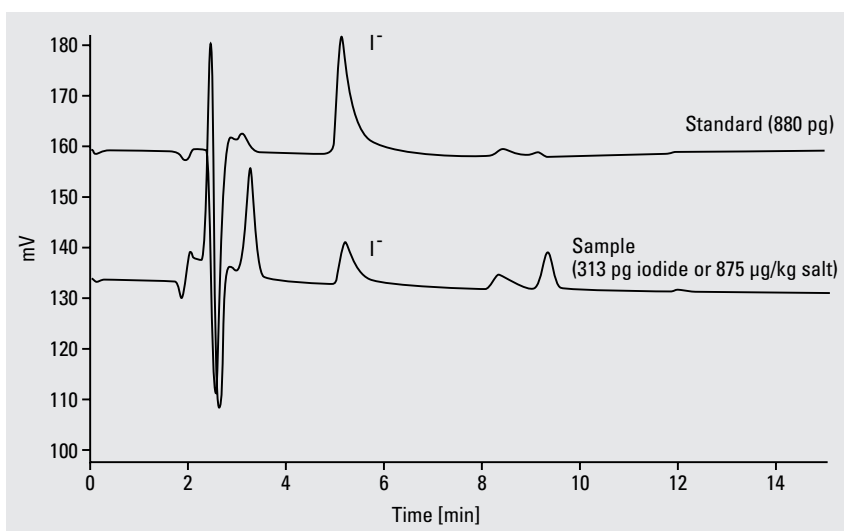


Figure 2
Analysis of iodide in table salt, dissolved in water

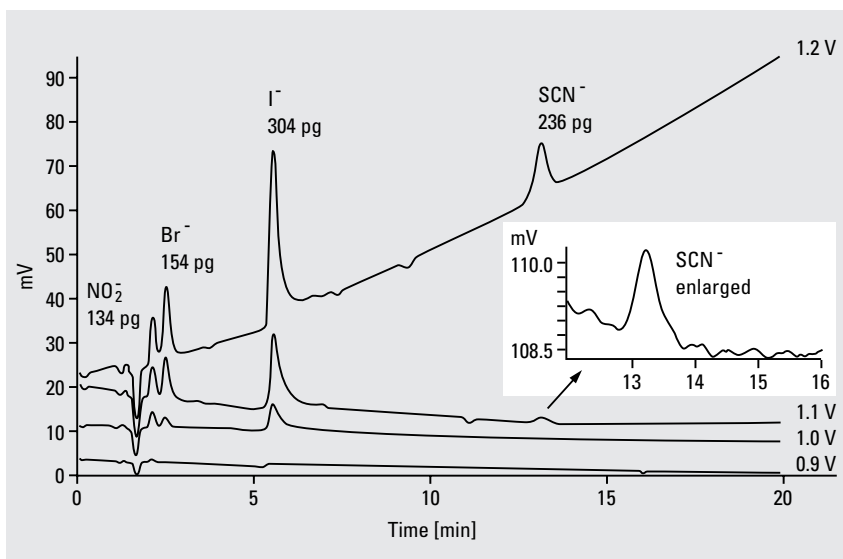


Figure 3
Optimizing detection potential using the 'auto-increment' mode

Chromatographic conditions

Column:	200 x 4 mm, Spherisorb ODS2, 5 µm
Mobile phase:	water with 5.2 g/l K_2HPO_4 + 3.6 g/l KH_2PO_4 + 3 g/l $TBAHSO_4^*$ /ACN=85:15
Flow rate:	1 ml/min
Inj. volume:	0.1 µl
Potential:	1 V
Mode:	amperometry
Electrode:	glassy carbon

* Tetrabutylammoniumdihydrogensulphate

An example is given in figure 3. The optimum potential was 1.1 V. At lower potentials the response was insufficient, at higher potentials the detector drift was unacceptable.

The minimum detectable level for iodide was about 40 µg iodide per kilogram table salt. The relative standard deviation for peak heights over 75 runs was 3% for 300 pg of iodide and an injection volume of 10 µl.

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