

H.J Brouwer, A. Kleindienst, J-P. Chervet, N.J. Reinhoud and M. Eysberg
Antec Leyden, Industrieweg 12, 2382 NV Zoeterwoude, The Netherlands

Introduction

Iodine is an essential trace element for humans. It is a component of the thyroid hormone thyroxin, which regulates the body's metabolic state and the growth and development in children. Iodine deficiency is an important health problem throughout much of the world. The dietary uptake of sufficient trace amounts of iodine is therefore necessary for mental and physical development.

HPLC in combination with DC or Pulsed electrochemical detection is commonly used for the analysis of iodide [2-4], using metal working electrodes such as platinum [3] and silver [2,4]. With the introduction of conductive diamond an interesting new electrode material became available in electrochemistry [7]. Diamond has several advantages over conventional electrode materials such as a wide potential window in aqueous solutions, excellent chemical inertness and stability. In this poster a sensitive and reliable LC-EC method is presented for the analysis of iodide based on DC amperometry using the Magic Diamond™ (MD) electrode.

Method

HPLC ALEXYS Iodide analyzer (p/n 180.0095A)
Column ALE-315 column, 150x3.0mm, 3µm C18 (p/n 250.1104)
Temperature 35 °C
Flow rate 400 µL/min



Fig. 1. Left : ALEXYS Iodide analyzer. Right: Flexcell™ with MD electrode disc.

Detection limit and Linearity

The linearity has been studied in the concentration range 6 µg/L – 1.3 mg/L. Within this range a linear detector response was observed with correlation coefficients better than 0.999. A concentration detection limit (C_{LOD}) of approximately 0.4 µg/L (3 nM) was found.

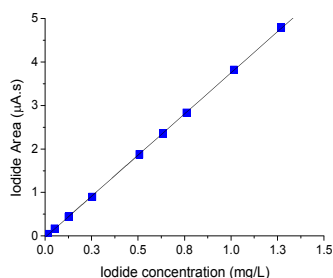


Fig. 2. Calibration plot for Iodide in water (injection volume 20 µL).
Concentration range 6 µg/L – 1.3 mg/L . $R = 0.9999$.

Repeatability

Table 1. Repeatability of 20 µL injections of Iodide in water (n=10).

Concentration Iodide(mg/L)	RSD (%)		
	$t_{Retention}$	Area	Height
0.13	0.1	0.9	1.0
1.26	0.1	0.2	0.3

The long-term repeatability was assessed using a solution with a high Iodide concentration of 13.7 mg/L in water. 225 repetitive injections were executed in a time period of 22 hours, see figure 3. The relative standard deviations (RSD%) for the peak area was 1.4%, demonstrating the good response stability of MD even at high iodide concentration.

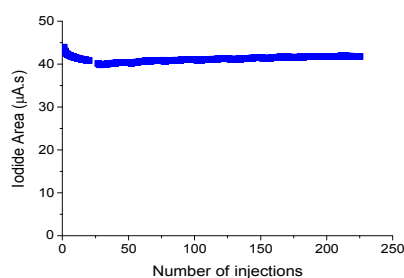


Fig. 3. Long-term repeatability of detection of Iodide on a MD electrode.
Sample concentration 13.7 mg/L (injection volume 20 µL).

Iodide in Milk Products

A low-fat (1.5%) milk was analysed. The following sample preparation procedure was performed prior to injection:

- 1 mL 3% acetic acid was mixed with 5 mL of sample and centrifuged.
- Supernatant passed over a RP SPE column (Alltech C18-fast column).
- The eluate (first 2 mL discarded) was collected and 20 µL injected in the LC system.

The concentration of iodide in the milk sample was estimated to be 45 µg/L.

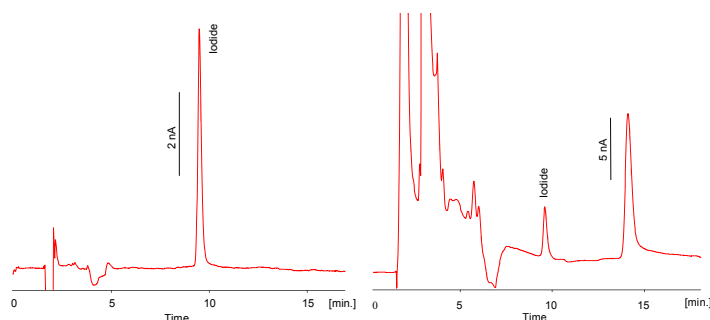


Fig. 4. Left: chromatogram of 50.8 µg/L (400 nM) of potassium iodide (KI) in water.
Right: chromatogram of Iodide in a low-fat (1.5%) milk sample (Injection volume 20 µL).

Conclusion

The ALEXYS[®] iodide analyzer provides a user-friendly and reliable solution for the determination of iodide by means of DC amperometry. With the new inert Magic Diamond™ electrode Iodide can be detected with excellent reproducibility and sensitivity.

References

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