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EUROSensors 2012
Kraków Poland



Guidebook

**The 26th European Conference
on Solid-State Transducers**

Organized by **Wrocław University of Technology**
Faculty of Microsystem Electronics and Photonics

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D. Liang¹, J. Zhang³, L. Tanguy³, A. Ernst⁴, P. Koltay⁴, R. Zengerle²
¹HSG-IMIT -- Institut für Mikro- und Informationstechnik, Germany; ²HSG-IMIT. IMTEK. BIOSS - Centre for Biological Signalling Studies, University of Freiburg, Germany; ³IMTEK - Department of Microsystems Engineering, University of Freiburg, Germany; ⁴IMTEK - Department of Microsystems Engineering, University of Freiburg. BioFluidix GmbH, Germany
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¹Siemens AG, Germany; ²Siemens AG, TU Muenchen, Germany; ³TU Muenchen, Germany
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¹Department of Aeronautics and Astronautics, Massachusetts Institute of Technology, United States; ²IMDEA Materials Institute, Spain; ³Institute for Polymers and Composites/13N, University of Minho, Portugal
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¹Department of Electronics, AGH University of Science and Technology, Poland; ²INESC-MN and IN- Institute of Nanoscience and Nanotechnology, Portugal

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Chair: Joao Conde

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¹*BIOMARK/ISEP-IPP, Portugal;* ²*INESC TEC/FCUP, Portugal;* ³*REQUIMTE/FCT-UNL, Portugal*
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¹*BioMark/ISEP, Portugal;* ²*Universidade Federal de Pernambuco, Brazil;* ³*Universidade Nova de Lisboa, Portugal*
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¹*Gdansk University of Technology, Poland;* ²*Institute of Electron Technology, Poland;* ³*University of Gdansk, Poland;* ⁴*Wroclaw University of Technology, Poland*
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P. Freitas², L. Dias¹, A. Peres³, L. Castro², A. Veloso²
¹*CIMO, Escola Superior Agrária, Instituto Politécnico de Bragança, Portugal;* ²*Instituto Superior de Engenharia de Coimbra, Instituto Politécnico de Coimbra, Portugal;* ³*LSRE, Escola Superior Agrária, Instituto Politécnico de Bragança, Portugal*
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Determination of 2,4,6-trichloroanisole by cyclic voltammetry

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Summary

The electrochemical reduction of 2,4,6-trichloroanisole (TCA), which is a chlorinated arene with electron-donating substituents, was evaluated by cyclic voltammetry (CV). TCA is a major concern for the winery industry since it is related with “cork taint”, a wine defect. The results obtained in this work showed that CV could be used to detect and quantify TCA in preparative standard solutions. Linear relationships could be set between the current amplitude and TCA concentration ($R > 0.999$), being the detection (LOD) and quantification (LOQ) limits of 0.8 and 2.0 ppm, respectively. Although, these preliminary limits are higher than the human sensory threshold (around 5 ppt in wine), the simplicity and low-cost of the methodology confer this study a possible role in the development of more efficient, less expensive processes for TCA detection in the industry.

Motivation and results

Cork is used for wine stoppers although “cork taint”, a wine defect, is often attributed to the presence of TCA (Figure 1) in cork stoppers. TCA has a low sensory threshold and can migrate from spoiled corks to the wine, which makes its presence in cork a problem, being the economic loss estimated to be 10 billion US dollars worldwide [1]. TCA is detected by gas chromatography, which is usually beyond the economic possibilities of most cork producers [1]. Recently, other approaches make use of biosensors [2-4]. Nevertheless, a more simple and low-cost analytical procedure, which does not require a skilled technician, to quantify TCA is still a challenging task from both academic and industrial point of view.

In this work, the performance of a CV device (PG580 Potentiostat-Galvanostat, Uniscan) with a silver working electrode (M295Ag, Radiometer), a platinum counter electrode (M241Pt, Radiometer) and a Ag/AgCl double-junction reference electrode (M90-02, Orion), was evaluated. Voltamograms for standard solutions of TCA (0-100 ppm) in 0.1 M of tetrabutylammonium perchlorate acetonitrile/water (30:20 v/v) were recorded between -2.0 and 1.6 V, at a scan rate of 100 mV s⁻¹. Increasing concentrations of TCA had an increasing effect on the cathodic and anodic profiles (Figure 2). Based on these results, two linear calibration curves ($R > 0.999$) were found relating the normalized amplitude of the current intensity (which was obtained by subtracting the solvent effect) and TCA concentrations (for 4-16 ppm and 16-100 ppm). Theoretical LOD and LOQ were found to be 0.8 and 2 ppm, respectively, which are still higher than the sensory threshold (1-10 ppt). Even so, these preliminary results show the feasibility of the CV system for TCA analysis, although further work is required to reduce those analytical limits.

References

- [1] R. Juanola, L. Guerrero, D. Subirà, V. Salvadó, S. Insa, J.A. Garcia Regueiro and E. Anticó, *Relationship between sensory and instrumental analysis of 2,4,6-trichloroanisole in wine and cork stoppers*, *Analytica Chimica Acta*, 513 (2004) 291–297
- [2] E. Moore, M. Pravda and G.G. Guilbault, *Development of a biosensor for the quantitative detection of 2,4,6-trichloroanisole using screen printed electrodes*, *Analytica Chimica Acta* 484 (2003) 15–24
- [3] M.V. Duarte, P. Lozano-Sanchez and I. Katakis, *Copper UPD as non-specific adsorption barrier in electrochemical displacement immunosensors*, *Biosensors and Bioelectronics* 24 (2009) 2205–2210
- [4] V. Varelasa, N. Sanvicens, M. Pilar-Marco and S. Kintzios, *Development of a cellular biosensor for the detection of 2,4,6-trichloroanisole (TCA)*, *Talanta* 84 (2011) 936–940

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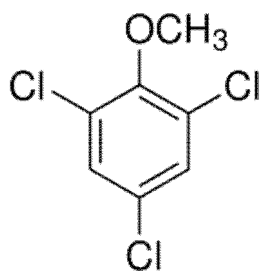


Fig. 1: Structure molecular formula of 2,4,6-trichloroanisole.

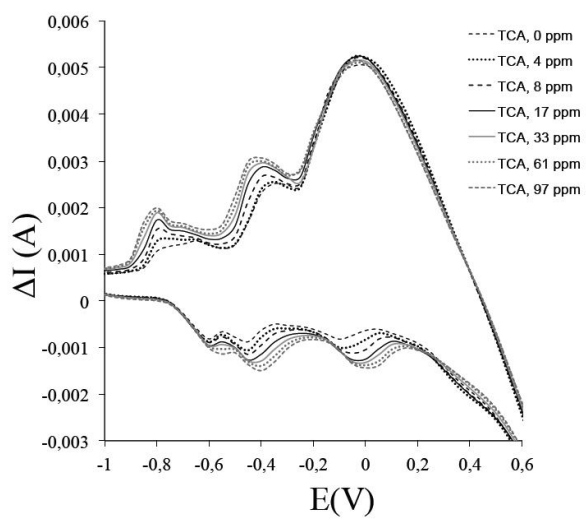


Fig 2: Insert concise explanation of figure here.

Determination of 2,4,6-trichloroanisole by cyclic voltammetry

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INTRODUCTION

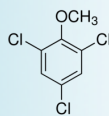
Cork is used for wine stoppers



Use of cork stoppers may be responsible for "cork taint" (a wine defect) attributed to 2,4,6-trichloroanisole (TCA)



TCA, produced by natural fungal, can migrate from spoilt cork stoppers to the wine



TCA Human sensory threshold ≈ 5 ppt in wine

ANALYTICAL TECHNIQUES

Standard methods

→ chromatographic techniques coupled (or not) to solid phase microextraction
→ beyond the economic possibilities of most cork producers.

Proposed Method → Cyclic Voltammetry

Electrodes:

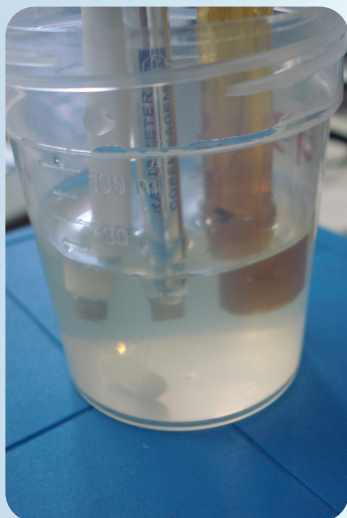
Working - Silver (M295Ag, Radiometer)
Counter - Platinum (M241Pt, Radiometer)
Reference - Ag/AgCl (M90-02, Orion)

Equipment:

Potentiostat-Galvanostat device (PG580, Uniscan)

Experimental conditions:

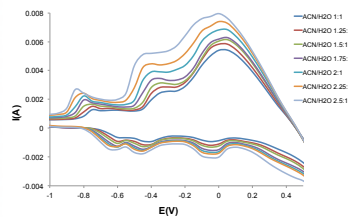
Room temperature
Voltammograms between -2.0 and 1.6 V
Potential scan rate of 100 mV/s
Solution analysis with two scans
Time analysis lower than 2 minutes
Calibration with standard addition method



RESULTS

1st Assay:

Cyclic voltammograms for different ACN/Water proportions - from 1:1 up to 2.5:1 v/v (TCA at ppm level)



Cyclic voltammetric signal profiles:

→ the relative amount of acetonitrile in the solvent mixture affects the intensity of the signals

→ tendency of increasing the signal

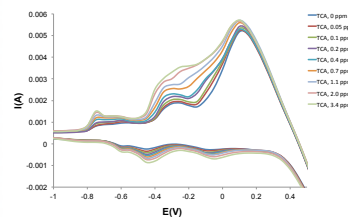
Better and more precise signal → High ACN proportions
Low-cost procedure → Low ACN proportions



SELECTED: ACN/water mixture 1.5:1 (v:v)

2nd Assay:

Increasing concentrations of TCA at ppm level

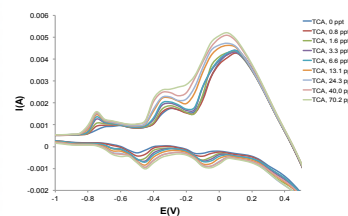


The profiles recorded show an increasing voltammetric signal with increasing TCA concentrations, for both cathodic and anodic regions.

3rd Assay:

Increasing concentrations of TCA at ppt level

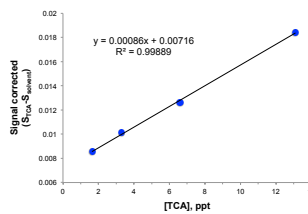
(covering the whole range of the human sensory threshold – 1.4 to 10 ppt)



Calibration curve → 0.8 to 70 ppt of TCA

→ Two linear zones

NEEDED → lower dynamic range



→ 1.6 to 13 ppt of TCA

→ Signal_{corrected} = 0.00086 × [TCA] + 0.0072

→ R = 0.9994

→ Limit of detection (LOD) = 0.4 ppt

→ Limit of quantification (LOQ) = 1.2 ppt

CONCLUSIONS:

The proposed cyclic voltammetric procedure offers new perspectives for simple, fast, sensitive and low-cost monitoring of TCA presence within all range of Human detection threshold for wines.

FUTURE WORK

→ Study responses against TCA in real aqueous samples from cork industry

→ Study the responses against structurally related and/or co-occurring compounds, namely:

- 2,4,6 – trichlorophenol
- 2,4,6 – tribromoanisole
- 2,6 – dichloroanisole
- 2,3,4,5 – tetrachloroanisole

