



# A New 2-Compartment Flow Through Cell for the Simultaneous Detection of Electrochemical Reaction Products by a Detection Electrode and Mass Spectroscopy



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## ABSTRACT

In this article we present a novel 2-compartment flow through cell (6-electrode cell). The 6-electrode cell allows the operation of two working electrodes independent from each other one of which serves as a detection electrode. A current at the detection electrode signifies the formation of soluble species at the generator electrode. This approach is reminiscent of an RRDE arrangement.

However, the 6-electrode cell allows combination of RRDE-analogue experiments with differential electrochemical mass spectroscopy (DEMS). Volatile products formed at the detection electrode, therefore, can be identified *via* mass spectroscopy, which is not possible with an RRDE-arrangements. At the same time it is possible to identify volatile products formed at the generator electrode.

We demonstrate the application of the 6-electrode cell for the oxygen reduction reaction in Li<sup>+</sup>-containing DMSO based electrolyte. We will show that soluble products that react at the detection electrode are due to the oxidation of superoxide.

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## 1. Introduction

The detection of electrochemical reaction products and intermediates is an ongoing challenge. A reasonable distribution of current and a continuous, well defined transport of reactants to the electrode as well as a short detection time, has to be ensured. To this end several cells have been designed to combine electrochemistry with mass spectroscopy [1–7].

Of these the dual thin layer cell has found ample applications in our group. It was introduced by Jusys *et al.* in 1999 [4] and was only slightly modified ever since. The cell was originally designed to combine electrochemical quartz crystal micro balance (eQCMB) measurements with differential electrochemical mass spectroscopy (DEMS) [4]. The dual thin layer cell is operated under continuous electrolyte flow, which causes a defined mass transport [8–12].

It is an interesting feature of the dual thin layer cell that the combination of eQCMB and DEMS allows both the detection of solid and volatile products of electrochemical reactions [4]. However, the most important benefit of the dual thin layer cell is that it allows to conduct DEMS measurements at smooth and

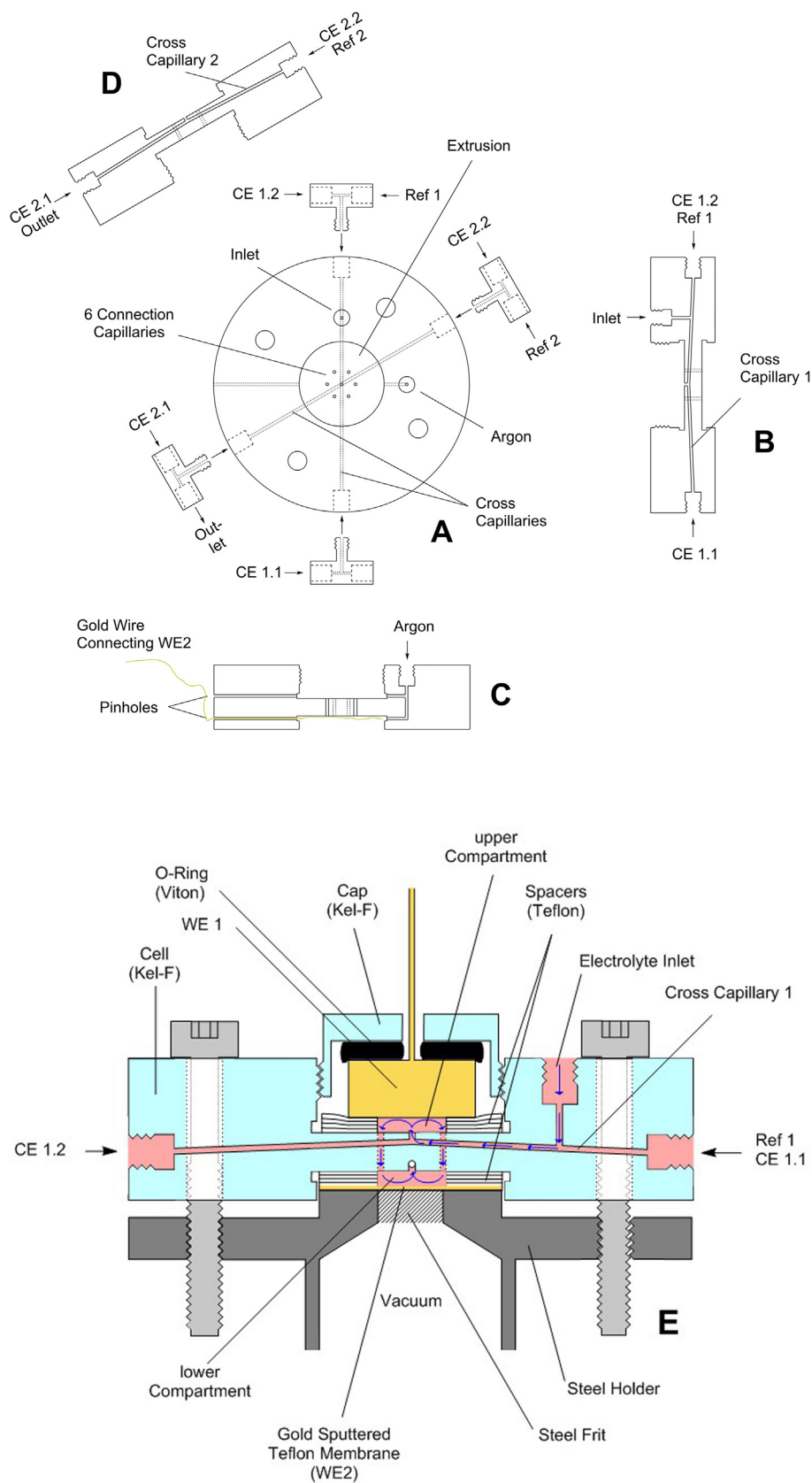
even single crystalline electrodes under defined convection conditions. The dual thin layer cell made it possible to conduct DEMS studies on the electrochemical oxidation of fuels such as methanol [13–16] and ethanol [17–19]. Others investigated the effect of the oxidation of small molecules at BDD-electrodes [20–22]. Using the dual thin layer cell, the effect of the surface state of single crystal electrodes on the CO-oxidation [23] or hydrogen evolution were investigated [24]. Recently we have employed the dual thin layer cell to investigate oxygen reduction from organic electrolytes [25,26].

Extensive studies have been conducted to characterise the convection conditions in the dual thin layer [8–12]. This allowed us to use the cell in combination with mass spectroscopy to determine the solubility of gases and their diffusion coefficient in various electrolytes [12].

A modification of the original dual thin layer cell allows the combination of ATR-FTIR spectroscopy with DEMS [27]. This arrangement was used to investigate the CO-oxidation at platinum [27]. Another modification of the dual thin layer cell allows RRDE-like applications [28]. In those arrangements the steel holder (with the steel frit covered by the porous Teflon membrane) in the lower compartment is replaced by a second working electrode that is used to detect incoming electrochemical active products of the working electrode in the upper compartment [28]. This cell was

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**Fig. 1.** Schematic drawing of the 6-electrode cell. A: Top view; B, C and D: cross section. E: cross section of the assembled cell.

used to study the oxygen reduction reaction at single crystal electrodes, which is hardly possible in an RRDE experiment [28]. However, the arrangement described by Jusys *et al.* cannot be

combined with DEMS. Wang *et al.* have introduced a double-band-electrode channel flow differential electrochemical mass spectrometry cell that allows RRDE-like applications in combination

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