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Construction of Ag/AgCl Reference Electrode from Used Felt-Tipped Pen Barrel for Undergraduate Laboratory

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Electroanalytical investigations are based on the control or observation of potential at the working electrode with respect to a suitable electrode of constant potential (1). The latter, a reference electrode (RE), has a fixed potential and is ideally non-polarizable; that is, its potential does not change as current flows through it. Among the recommended REs, Ag/AgCl is often the first choice of electrochemists due to simplicity, inexpensive design, and nontoxic components (2). In the electrochemical literature, it is regarded as an electrode of the second kind, whose active component is Ag coated with AgCl and that is in direct contact with Cl[−] solution of some salt in an appropriate solvent. The controlling redox process is (3)



and the potential, as per the Nernst equation, is calculated as

$$E_{\text{Ag/AgCl}} = E_{\text{Ag/AgCl}}^\circ - \frac{RT}{F} \ln a_{\text{Cl}^-} \quad (2)$$

that is, the potential value is a function of Cl[−] ions activity, which is related to the Cl[−] ions concentration.

A complete RE consists of an active component filling an appropriate body material, sealed to prevent evaporative loss and contamination, and connected via a suitable junction to the external electrolyte in an electrochemical cell. Various construction designs for Ag/AgCl RE have been reported (4–9) and many are commercially available. Here we describe a simple and inexpensive procedure for making a reliable, stable, and long-lasting Ag/AgCl RE.

Preparation of Ag/AgCl Reference Electrode

The procedure can be reproduced in any general chemistry laboratory. Requirements are a plastic barrel of a used felt-tipped pen, Ag wire, agar powder, and KCl. Construction is completed in three steps.

Step 1: Coating of Ag Wire with AgCl

Silver wire (99.9%) is immersed in 0.1 M HNO₃ for a few seconds to remove any oxide from the metal surface and then rinsed thoroughly with distilled water. The AgCl layer is applied by immersing the Ag wire in 0.05 M KCl solution and connecting it to the positive terminal of a 3-V battery. The circuit is completed by connecting the negative terminal to a Pt wire

immersed in the same solution. If Pt wire is not available then Ag wire can also be used. The voltage is applied for 10 minutes or until the wire is completely coated with a grayish-white layer of silver chloride.

Step 2: Construction of Main Body and Junction

The plastic body of a used felt-tipped pen with a fibrous tip is repeatedly washed with distilled water followed by 1-propanol to remove the colored ink. The pen is then filled to 1/3 volume with a hot solution of 0.6 g agar powder in 20 mL of 3 M KCl. The gel is carefully poured to avoid entrapment of air bubbles and allowed to cool slowly to form a thick gel.

Step 3: Assembling the Complete Electrode

Most of the remaining pen volume is filled with 3 M KCl solution and the Ag wire coated with AgCl from step 1 is fitted in the pen cap so that it is partially immersed in the filling solution (Figure 1). The cap is then sealed using a piece of adhesive tape to avoid the evaporative loss of the filling solution.

Hazards

Nitric acid is corrosive and can cause severe burns. Propanol is a flammable liquid and vapor and is harmful if swallowed, inhaled, or absorbed through skin. Agar powder may cause respiratory tract irritation if inhaled.

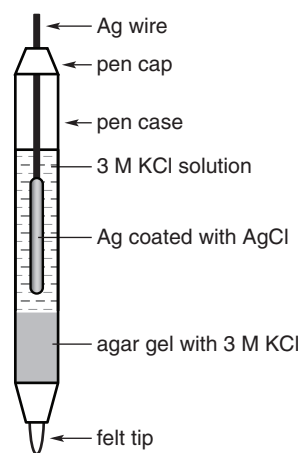


Figure 1. Sketch of Ag/AgCl reference electrode constructed out of used felt-tipped pen barrel.

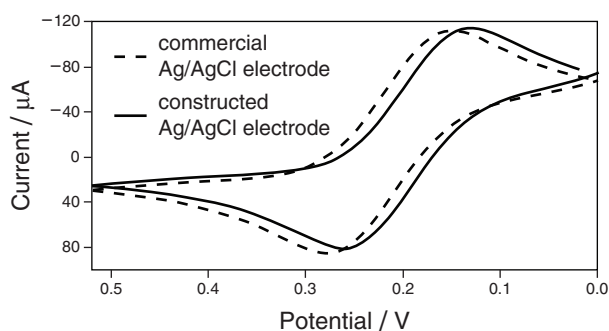


Figure 2. Cyclic voltammograms recorded on 10 mM $\text{K}_3\text{Fe}(\text{CN})_6$ and 100 mM KNO_3 as a supporting electrolyte. Pt disc electrode was used as a working electrode.

Calibration of the Electrode

Two methods were used to calibrate the electrode potential (2). In first method the constructed electrode and a commercially available RE (saturated calomel electrode or Ag/AgCl) were placed in a beaker containing 3 M KCl. The electrode potential, measured by a high impedance voltmeter, was 0.040–0.050 V versus the calomel electrode and 0.002 V versus the commercial Ag/AgCl electrode (5). In the second calibration method, the redox couple for $\text{Fe}^{3+}/\text{Fe}^{2+}$ was examined through cyclic voltammetry in a three-electrode setup. Cyclic voltammograms (CV) were recorded with a Metrohm Autolab 100 potentiostat–galvanostat in a 10 mM $\text{K}_3\text{Fe}(\text{CN})_6$ solution in 100 mM KNO_3 . A good correlation between the CVs with the constructed Ag/AgCl electrode and the commercially available Ag/AgCl reference electrode was observed (Figure 2). The redox potential for the $\text{Fe}^{3+}/\text{Fe}^{2+}$ couple from the CV with the constructed RE showed a shift of 20 mV towards negative compared to commercial Ag/AgCl, which is within the experimental error. The peak potential difference and peak

height remained constant for multiple numbers of cycles, which is an indication of the stability.

Electrode Life, Storage, and Refilling

The constructed electrode shows reproducible results for at least six months without refilling or changing the filling solution, provided it is properly sealed from the top. We recommend storing the electrode in 3 M KCl solution before and after use.

If required the electrode can be reconstructed by removing the old coating of AgCl and repeating steps 1–3. This procedure takes less than twenty minutes.

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