Electronic supplementary materials *Mendeleev Commun.*, 2021, **31**, 638–640

The first example of anodic corrosion of Pd in aqueous ethylenediamine with formation of colloidal palladium

Marina D. Vedenyapina, Vladimir V. Kuznetsov, Stanislav A. Kulaishin, Nina N. Makhova and Marina M. Kazakova

Experimental part

Ethylenediamine (EDA) and K_2CO_3 were purchased from "Acros" and used without further purification. The working solutions were prepared on bidistilled water. The electrolysis using a Pd anode was investigated by cyclic voltammetry in a 0.1 M solution of EDA in a 0.05 M aqueous solution of K_2CO_3 (supporting electrolyte, pH 11.1). Cyclic voltammograms (CVs) were registrated on computerized IPC-Compact potentiostat in three-electrode cell. Pd wire (99.99%) with a diameter and 0.3 mm in length 0.3 cm soldered in glass was used as the working electrode. Pt wire of the same sizes soldered in glass served as the auxiliary electrode and a silver chloride electrode (Ag/AgCl/3 M KCl) was used as the reference electrode.

The anodic corrosion was carried out with use of two-electrode cell without separation of anode and cathode compartments. Pd and steel wires with a diameter of 0.3 mm poured in solution of electrolyte on 15 mm were served as anode and cathode, accordingly. The electrolysis was performed in galvanostatic conditions. Concentration of EDA in 0.05 M solution of K_2CO_3 was 1.0 mol L^{-1}; volume of working solution was 20 cm^3. The electrolysis was proceeded for 40 h. After the end of electrolysis, the Pd and steel electrodes were weighed on an ABJ220-4NM electronic analytical balance (Kern, USA) (d = 0.0001 g).

The microstructure of both electrodes after electrolysis was studied by field emission scanning electron microscopy (FE-SEM) using a Hitachi SU8000 electron microscope. The images were taken in the secondary electrons registration mode at an accelerating voltage of 2–30 kV and a working distance of 8.4–11.0 mm. The study of the steel cathode after the end of electrolysis was carried out by energy-dispersive X-ray spectroscopy (EDX) using an Oxford Instruments X-max energy-dispersive spectrometer (United Kingdom). The samples of both electrodes were placed on the surface of an aluminum table 25 mm in diameter and fixed with two screws.

The morphology of colloidal Pd particles obtained after electrolysis of Pd anode was investigated by transmission electron microscopy (TEM) on a Hitachi HT7700 electron microscope (Japan). The images were taken in the transmitted electrons registration mode (bright field mode) at an accelerating voltage 100 kV. The working solution was applied to a thin carbon film fixed on a copper mesh with a diameter of 3 mm, fixed in a special holder. The sample was applied in liquid form, followed by vacuum drying. The optimization of analytical measurements was carried out within the framework of the previously described approach.[S1]

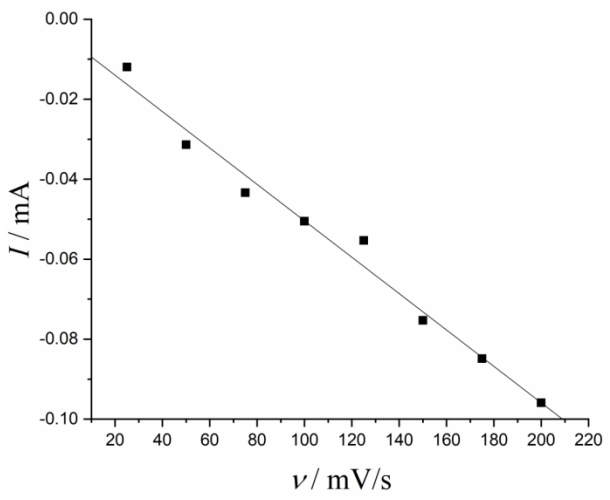

Figure S1 Dependence of the cathodic maximum current peaks ($I_{p,c.}$) in the CV on the potential scan rate [see Figure 1(*b*) in the main text].

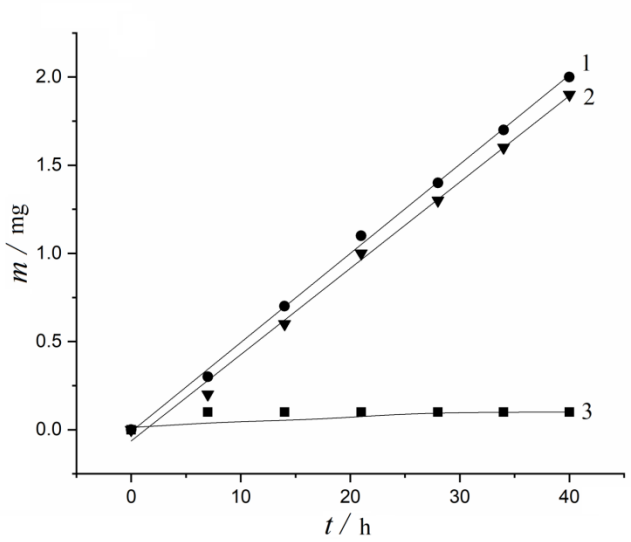

Figure S2 The time dependences of Pd anode dissolution and cathode mass increase in the 0.1 M EDA solution at a current of 5 mA: (*1*) mass loss of the Pd anode (m_{corr}), (*2*) calculated mass of Pd in solution (m_{sol}) and (*3*) mass of the cathode deposit of Pd (m_{dep}).

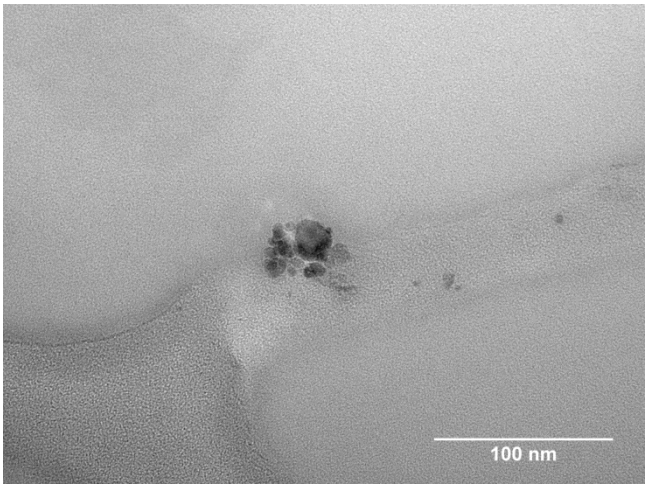

Figure S3 Transmission electron microscope image of the electrolyte after anodic corrosion of Pd in a EDA solution at a current of 5 mA.

Reference

S1 V. V. Kachala, L. L. Khemchyan, A. S. Kashin, N. V. Orlov, A. A. Grachev, S. S. Zalesskiy and V. P. Ananikov, *Russ. Chem. Rev.*, 2013, **82**, 648.