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Supplementary information

Structure and catalytic activity of ultrasmall Rh, Pd and (Rh + Pd)

nanoparticles obtained by mediated electrosynthesis

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Fig. S1. CV curves of the { $[RhCl_6]^{3-}$ (1.5 mM) – MV²⁺ (3 mM) – CTAC (10 mM)} system with the potential scan in negative and positive directions. GC, H₂O/0.1 M KCl, v = 100 mV/s.



Fig. S2. CV curves of the $\{[PdCl_4]^{2-}(1.5 \text{ mM}) - CTAC (10 \text{ mM})\}\)$ (a) and $\{[PdCl_4]^{2-}(1.5 \text{ mM}) - MV^{2+}(3 \text{ mM}) - CTAC (10 \text{ mM})\}\)$ (b) systems with the potential scan in negative and positive directions. GC, H₂O/0.1 M KCl, v = 100 mV/s.



Fig. S3. CV curves of the { $[PdCl_4]^{2-}$ (1.5 mM) – CTAC (10mM)} system after microelectrolysis at E = -0.25 V for various periods. GC, H₂O/0.1 M KCl, v = 100 mV/s.



Fig. S4. Photos of solutions in the electrolysis cell after passing various amounts of electricity (F number with respect to metal ions) during electrosynthesis of MNPs in the presence of PVP (a) and CTAC (b-d): Rh (a,b), (Rh/Pd) (b), Pd, (Pd/Rh) (c), (Pd-Rh) (d). The F axis is not scaled, is given for clarity.



Fig. S5. Energy-dispersion spectra of MNPs in a stabilizer shell: RhNPs – PVP (a), RhNPs – CTAC (b), PdNPs – CTAC (c), (Rh/Pd)NPs – CTAC (d), (Pd/Rh)NPs – CTAC (e), (Pd-Rh)NPs – CTAC (f) obtained by SEM (Al and Ti peaks are from the support).