Electronic Supplementary Material (ESI) for Dalton Transactions.
This journal is © The Royal Society of Chemistry 2023

Electronic Supplementary Information

Synthesis of Ethane-disulfonate Pillared Layered Cobalt Hydroxide towards Electrocatalytic Oxygen Evolution Reaction

Jianbo Liang*, Hang Zu, Huiling Si, Yanhong Ma and Mengyao Li
Department of Chemistry, Capital Normal University, Beijing, P.R. China
*Email:liangjianbo@cnu.edu.cn

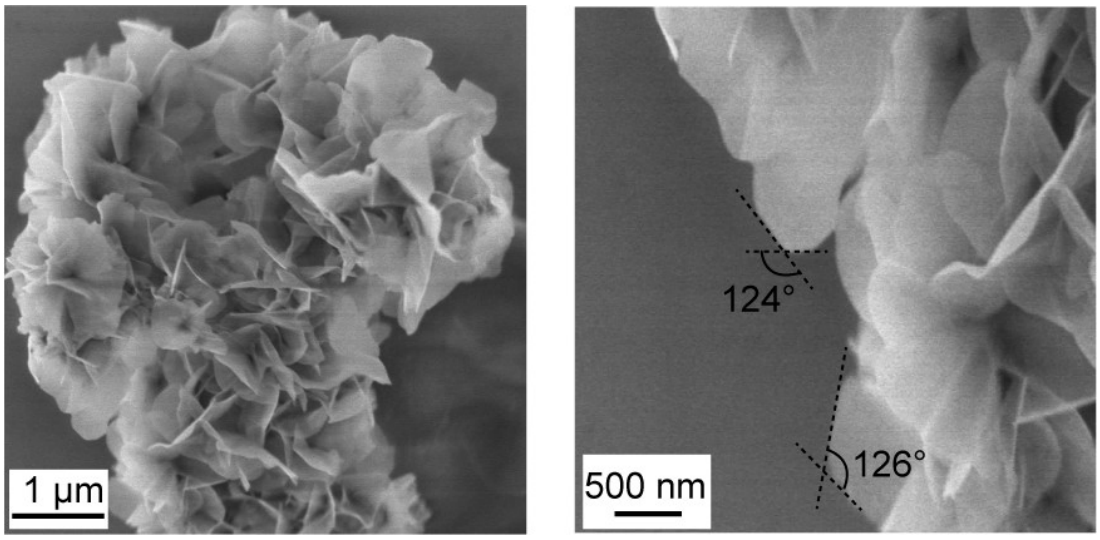

Figure S1. SEM images of the Co-C2 sample.

The Co-C2 sample comprises aggregates of nanosheets with a lateral size of 1~2 μm. The thickness of nanosheets is about tens of nanometers. Some nanosheets show well-developed edges. The angel between the neighboring edges was close to 120 degree.

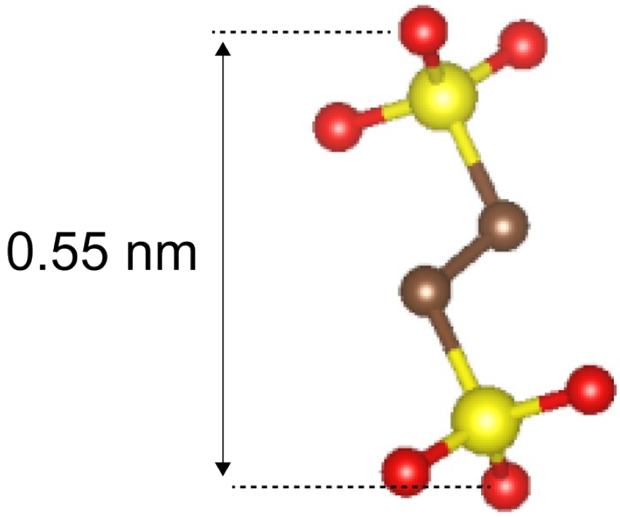

Figure S2. A geometry illustration of the ethane-disulfonate molecule (H atoms are omitted).

The axis length of the molecule is estimated by the distance of oxygen atoms at the two ends.

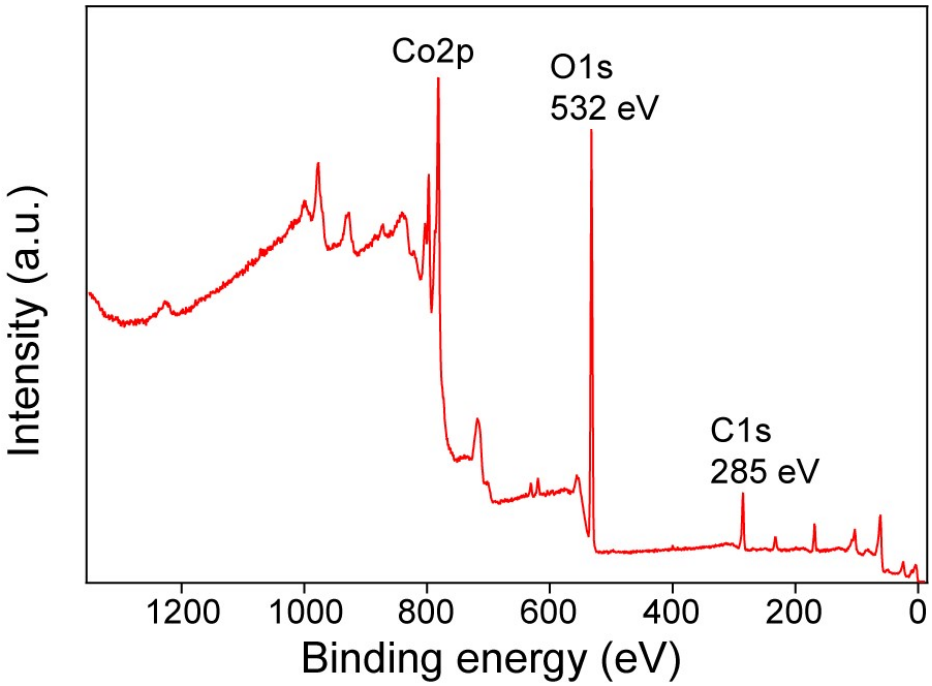

Figure S3. The full XPS spectrum of the Co-C2 sample.

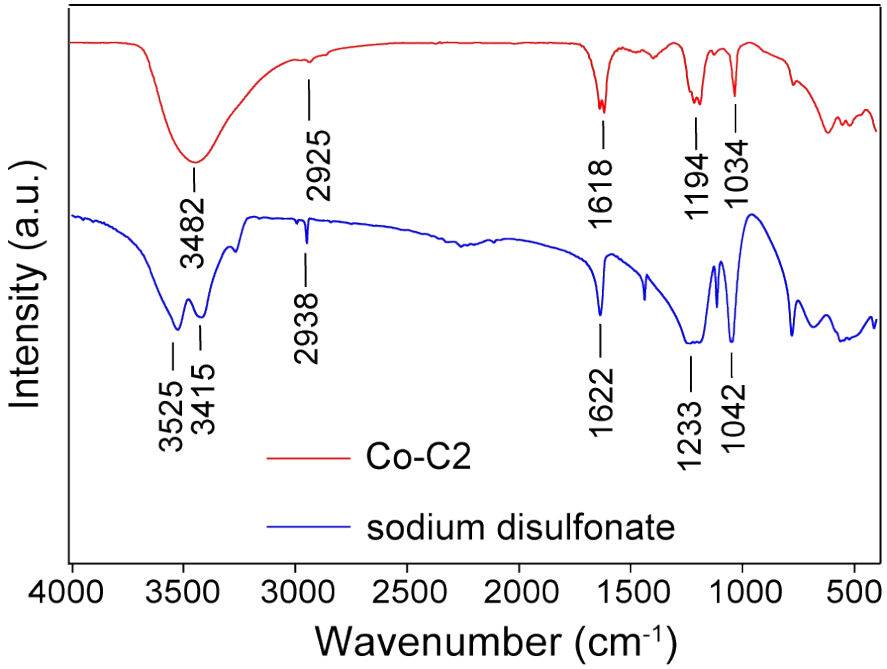

Figure S4. FT-IR spectra of the Co-C2 sample and sodium salt of ethane-disulfonate.

For the Co-C2 sample (the red curve), the broad band centered at 3482 cm^{-1} corresponds to the stretching vibration of O-H from the hydroxyl groups and water molecules. The band at 1618 cm^{-1} is the bending vibration of water. The peaks at 2925 cm^{-1} is assigned to the stretching vibration of C-H. The bands in range of 1000~1200 cm^{-1} are attributed to the vibrations of the SO_3 group. The bands at 1194 and 1304 cm^{-1} are attributed to the vibration of S=O from the sulfonate group. Compared to sodium salt of ethane-disulfonate (the blue curve), the Co-C2 sample shows a red-shifted of 39 cm^{-1} for the S=O vibration, which may be attributed from the chemical interactions between the sulfonate groups and Co_{Td} ions.

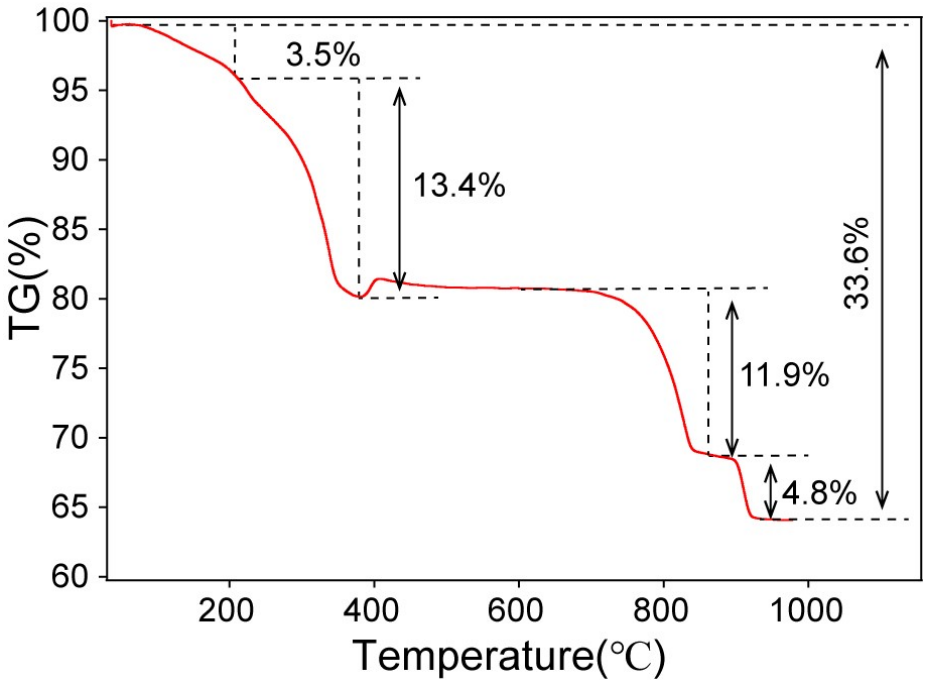

Figure S5. Thermogravimetric data of the Co-C2 sample.

In the measurement, the sample is heated from the room temperature to 1000°C at a rate of 5 degree per minute. The total weight loss of the sample is calculated as 33.6%. The final product is determined as CoO. The cobalt content in the solid is calculated as 47.5%, which is consistent with the value determined by chemical analysis.

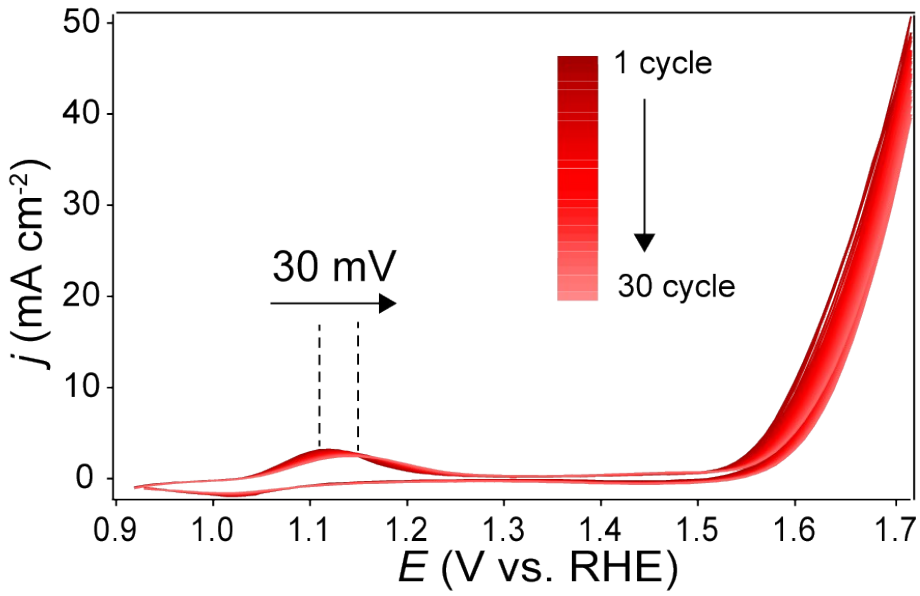

Figure S6. The CV curves of the Co-C2 sample in the activation.

The CV curves came to steady after 20 circles. LSV curves are acquired after scanned for 30 CV circles.

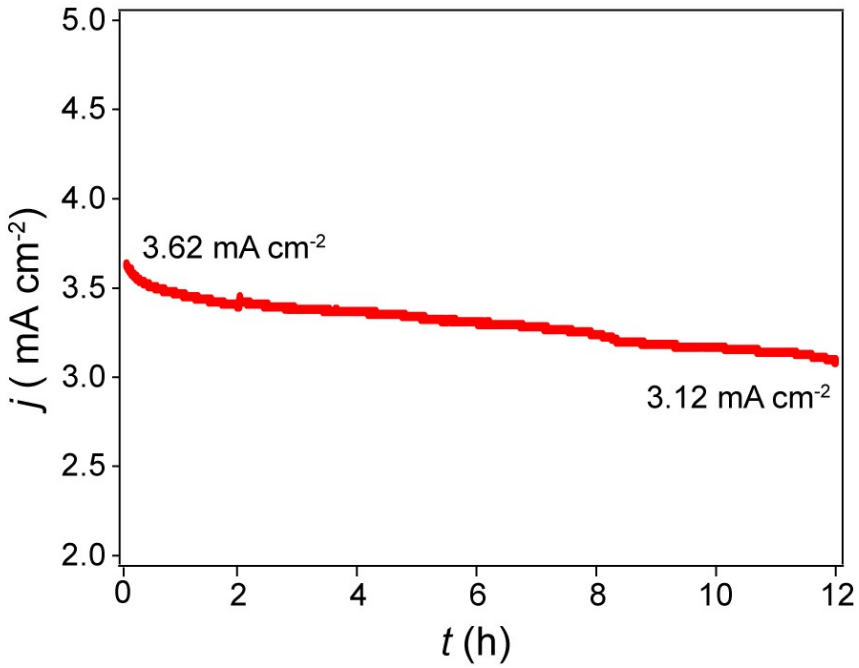

Figure S7. The chronopotentiometry measurement of the Co-C2 sample at the overpotential of 350 mV.

The initial current density was 3.62 mA/cm^2. The value dropped to 3.12 mA/cm^2 after working 12 hours.

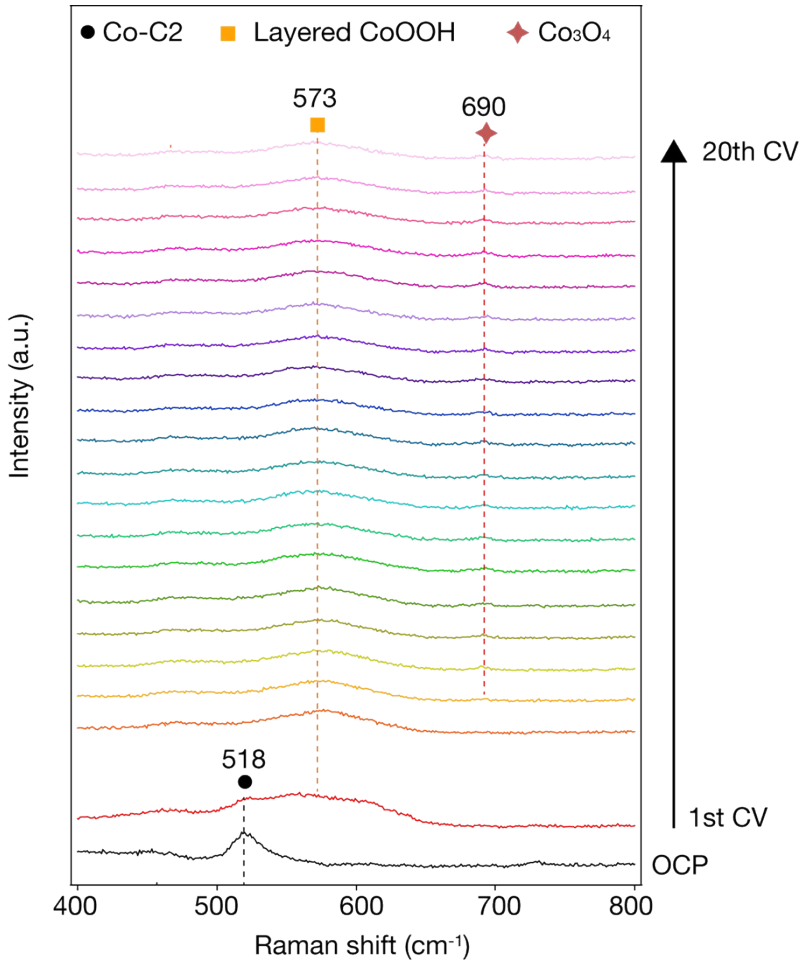

Figure S8. The Raman spectra of the sample collected after each round of CV scanning.

Two main Raman peaks at 518 cm^{-1} can be assigned to the vibrations of Co(II)-O in the Co-C2 sample. The peak appeared at 573 cm^{-1} after the second CV scan can be associated with the layered CoOOH. The weak peak at 690 cm^{-1} is assigned as Co_3O_4. Trace amount of Co_3O_4 was detected after the sample was cycled by twenty arounds of CV scanning.

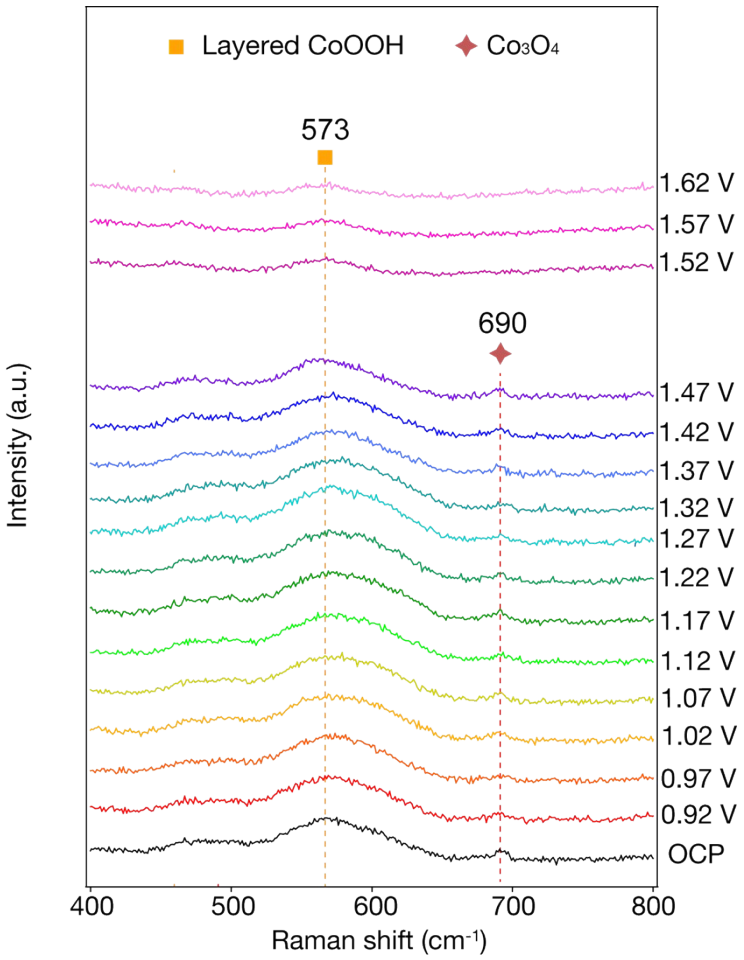

Figure S9. In-situ Raman spectra of the activated catalyst with respect to increasing potential. The peak at the 573 cm^{-1} is assigned as layered CoOOH. The peak at 490 cm^{-1} is assigned to the spinel Co_3O_4.

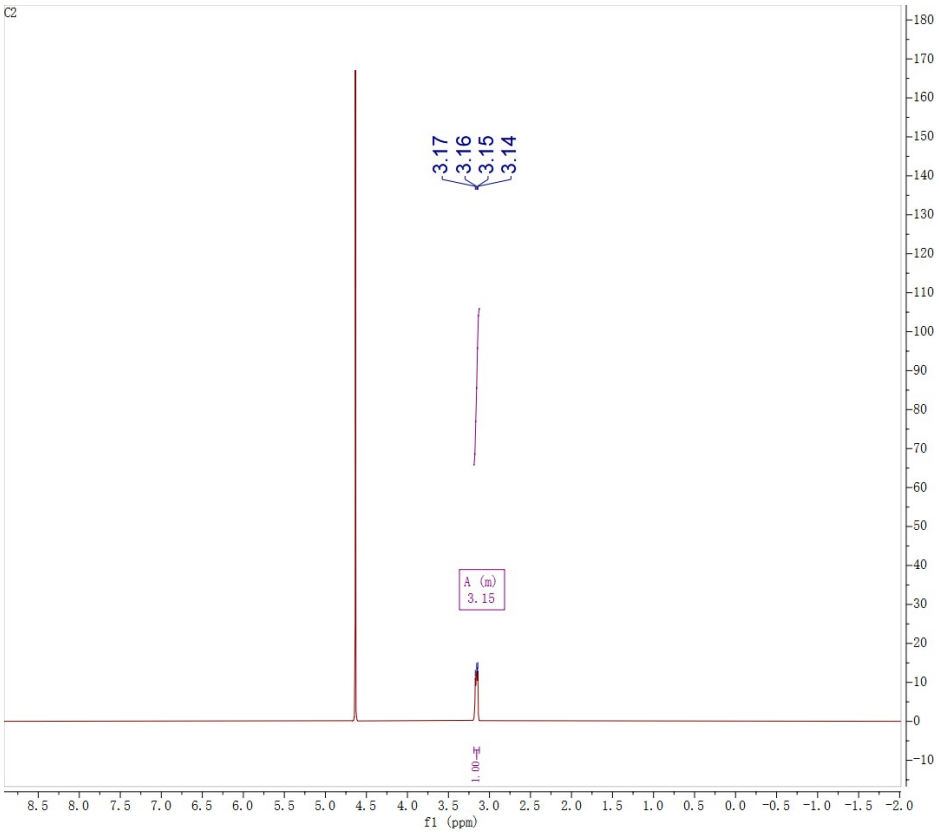

Figure S10. The H^1 nuclear magnetic resonance (H-NMR) spectrum of the sodium salt of ethane- disulfonate.

The peaks at around 3 ppm are attributed to the H in the sodium ethane-disulfonate. This spectrum confirms the purity of the sample.

Table S1. Refined structure parameters of the Co-C2 sample.

atom	X	Y	Z	B	Occupation
Co1	0.5000	0.5000	0.0000	0.628	1.0
Co2	0.1505	0.1717	-0.0293	1.098	1.0
Co3	0.6671	0.1471	0.0105	0.000	1.0
Co4	0.0419	0.5092	0.1245	9.251	1.0
O1	0.6937	0.3924	-0.0454	0.000	1.0
O2	-0.4261	0.7084	0.1382	0.001	1.0
O3	0.8656	0.0220	-0.0973	0.087	1.0
O4	0.4496	0.2670	0.0449	0.000	1.0
O5	0.3800	0.0532	-0.1878	0.000	1.0
O6	0.9783	0.2672	0.1839	0.006	1.0
C1	0.406	0.0753	-0.5217	0.000	1.0
S1	0.1803	0.1591	-0.563	0.000	1.0
O7	0.7109	0.3798	-0.4501	0.000	1.0
O8	-0.0134	0.3043	-0.5075	2.235	1.0
O9	0.1801	0.1912	-0.4309	0.000	1.0

Space group: P-1;

Cell Parameters: a = 6.37682 Å, b = 8.42837 Å, c = 9.79597 Å, α = 97.099°, β = 98.881°, γ = 99.604°;

R_p = 0.078, R_{wp} = 0.103, R_b = 0.069, R_f = 0.063

See Ref 34 in the main text for the meaning of the factors of R_p, R_{wp}, R_b and R_f in refining XRD data.

1 μ m