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1. General information

1.1 Characterization

¹H NMR spectra were recorded on Bruker 400/600 MHz spectrometer and the chemical shifts were reported in parts per million (δ) relative to internal standard TMS (0 ppm) for CDCl₃. The peak patterns are indicated as follows: s, singlet; d, doublet; dd, doublet of doublet; t, triplet; m, multiplet; q, quartet. The coupling constants, *J*, are reported in Hertz (Hz). ¹³C NMR spectra were obtained at Bruker 100/150 MHz and referenced to the internal solvent signals (central peak is 77.0 ppm in CDCl₃, DMSO-*d*₆ 39.52 ppm). CDCl₃ was used as the NMR solvent. Flash column chromatography was performed over silica gel (200-300 mesh). All reagents were weighed and handled in air at room temperature. Unless otherwise noted, all reactions were performed under a nitrogen atmosphere. Other reagents were purchased from Alfa, Acros, Aldrich, and TCI and used without further purification. Deionized water (DIW) was used in all experiments.

1.2 Synthesis of CoFe-LDH nanoarrays on carbon cloth

The CoFe-LDH on carbon cloth (CoFe-LDH/CC) was synthesized via an electrosynthesis method. In a typical procedure, a piece of carbon cloth (3 cm × 3 cm) was pretreated with 2.0 M HCl solution for 15 min, to ensure a clean surface. The asobtained substrate was used as the working electrode and placed in an electrochemical cell which was assembled in a three-electrode configuration, by using platinum wire as the counter electrode and Ag/AgCl as the reference electrode. The electrolyte for electrosynthesis of CoFe-LDH/CC was obtained by dissolving Co(NO₃)₂·6H₂O (7.858 g) and FeSO₄·7H₂O (2.506 g) in 120 mL of distilled water. The potentiostatic deposition was carried out at a potential of -1.0 V vs. Ag/AgCl. The resulting CoFe-LDH/CC were withdrawn and rinsed with distilled water, and then dried at room temperature.

1.3 Material characterization

X-ray diffraction (XRD) patterns were recorded by a Rigaku XRD-6000 diffractometer, using Cu-K α radiation (0.15418 nm) at 40 kV, 30 mA. The morphology was investigated using a scanning electron microscope (SEM; Zeiss SUPRA 55) with an accelerating voltage of 20 kV, combined with energy dispersive X-ray spectroscopy (EDX). Transmission electron microscopy (TEM) images were recorded with Philips Tecnai 20 and JEOL JEM-2010 HR-TEM. X-ray photoelectron spectroscopy (XPS) measurements were performed using an ESCALAB 250 instrument (Thermo Electron) with Al K α radiation. Raman measurements were carried out with 633 nm of excitation by using a confocal Raman microspectrometer (Renishaw, inVia-Reflex, 633 nm).

1.4 Electrochemical measurements.

A CHI 660B electrochemical workstation (Shanghai ChenhuaInstrument Co., China) was utilized for electrochemical measurements. Cyclic voltammetry (CV) curves and galvanostatic charge-discharge tests were carried out using a classical threeelectrode cell with a saturated Ag/AgCl electrode as the reference, a platinum plate as the counter in MeCN/H₂O. The CoFe-LDH acted directly as the working electrode.

2. Experimental procedures

2.1 Procedures for selective electrooxidation reaction of sulfide 1

A mixture of sulfide **1** (0.5 mmol) and $nBu_4NH_2PO_4$ (0.5 mmol), MeCN/H₂O (2.0 mL, 1:1 v/v) were added to a 10 mL reaction tube. The mixture was stirred 2h at room temperature in an undivided cell using CoFe-LDH as the working electrode, Pt (10 mm \times 10 mm \times 0.3 mm) as the counter electrode and Ag/AgCl as the reference electrode. The resulting reaction solution was directly filtered through a pad of silica by ethyl acetate and concentrated *in vacuo*. NMR yields were determined by ¹H NMR using dibromomethane as an internal standard. Solvent was evaporated and the resulting residue was purified by flash column chromatography on silica gel with ethyl acetate/petroleum ether (1:3) as an eluent to afford the metathesis products **2** and **3**. The Faradaic efficiency was calculated through the following equation:

$$FE(\%) = 100\% \times \frac{mol \ of \ formed \ sulf oxides}{total \ passed \ charge/(2 \times F)}$$
(1)

Where F is the Faraday constant (96485 C mol⁻¹).

A theoretical charge for the selective oxidation of diphenyl sulfide **1a** (0.5 mmol): (0.5 \times 10⁻³ mol) \times 2 \times (6.02 \times 1023 mol⁻¹) \times (1.6 \times 10⁻¹⁹ C) = ~96 C

2.2 Gram-scale syntheses of sulfoxides catalyzed by CoFe-LDH/CC.

A mixture of sulfide **1a** (10 mmol) and $nBu_4NH_2PO_4$ (0.25 M), MeCN/H₂O (20.0 mL, 1:1 v/v) were added to a 50 mL reaction tube. The mixture was stirred 7 h at room temperature in an undivided cell using CoFe-LDH as the working electrode, Pt (10 mm × 10 mm × 0.3 mm) as the counter electrode and Ag/AgCl as the reference electrode. The resulting reaction solution was directly filtered through a pad of silica by ethyl acetate and concentrated *in vacuo*. NMR yields were determined by ¹H NMR using dibromomethane as an internal standard. Solvent was evaporated and the resulting residue was purified by flash column chromatography on silica gel with ethyl acetate/petroleum ether (1:3) as an eluent to afford the metathesis products **2a** 1.6360g.

A mixture of sulfide **1m** (10 mmol) and $nBu_4NH_2PO_4$ (0.25 M), MeCN/H₂O (20.0 mL, 1:1 v/v) were added to a 50 mL reaction tube. The mixture was stirred 7 h at room temperature in an undivided cell using CoFe-LDH as the working electrode, Pt (10 mm × 10 mm × 0.3 mm) as the counter electrode and Ag/AgCl as the reference electrode. The resulting reaction solution was directly filtered through a pad of silica by ethyl acetate and concentrated *in vacuo*. NMR yields were determined by ¹H NMR using dibromomethane as an internal standard. Solvent was evaporated and the resulting residue was purified by flash column chromatography on silica gel with DCM/MeOH = 10/1 as an eluent to afford the metathesis products **2m** 1.7906g.

2.3 Electron paramagnetic resonance (EPR) experiment

In standard conditions, a mixture of diphenyl sulfide **1a** (0.5 mmol) and $nBu_4NH_2PO_4$ (0.5 mmol), MeCN/H₂O (2.0 mL, 1:1 v/v) were added to a 10 mL reaction tube. The reaction was stirred with an applied potential of 2.0 V under air at room temperature for 20 min, followed by the addition of DMPO (5,5-dimethyl-1-pyrroline N-oxide). After that, the solution was taken out for the EPR test. The blank test was carried out same as standard conditions without potential bias.

2.4 Procedures for the synthesis of pharmaceutical precursors with sulfides moieties

Methyl acetyl-D-methioninate (**1m**). Methionine (2 g, 13.5 mmol) was placed in a 100 mL flask, and dissolved in glacial acetic acid (30 mL). Acetic anhydride (1.90 g, 2.10 mL, 19 mmol) was added, and the mixture was refluxed for 2 h. The solvent was removed in vacuo. Diethylether was added in order to recrystallize the product to form a white solid.

acetyl-D-methionine (1.92 g, 10 mmol) was placed in a 100 mL flask. Methanol (35 mL) was added, followed by the addition of thionyl chloride (3.11 mL, 42.6 mmol). DMF was added (100 μ L) after which the solution was stirred overnight. The solvent was evaporated in vacuo. Purification using column chromatography (EtOAc) yielded the product as a white solid.



Methyl (5-(propylthio)-1H-benzo[d]imidazol-2-yl)carbamate (1n) was synthesized according to the previous procedure.⁶



(Z)-2-(5-fluoro-2-methyl-1-(4-(methylthio)benzylidene)-1H-inden-3-yl)acetic acid
(10) was synthesized according to the previous procedure.⁷



6-Methoxy-2-(((4-methoxy-3,5dimethylpyridin-2-yl)methyl)thio)-

1Hbenzo[d]imidazole (Omperazole) (1p) was synthesized according to the previous procedure.⁸



3. Supplementary figures and tables



Fig. S1 SEM images of as-prepared CoFe-LDH/CC electrode at low magnification (a) and high magnification (b). (c) Histogram of thickness distribution of CoFe-LDH nanosheets. Average thickness is $9.4 \text{ nm} \pm 1.2 \text{ nm}$.



Fig. S2 SEM images of 10th recycled CoFe-LDH/CC.



Fig. S3 (a, b) TEM images and (c) SAED of 1st recycled CoFe-LDH/CC.



Fig. S4 Fe XPS spectra of fresh CoFe-LDH/CC and 1st recycled CoFe-LDH/CC.



Fig. S5 XRD patterns of as-prepared CoFe-LDH/CC and its derived crystalline metal oxyhydroxide (*c*-CoFeOOH/CC) though anodization in 1 M KOH electrolyte.



Fig. S6 Wavelet transform of EXAFS spectra of fresh CoFe-LDH/CC, 1st recycled CoFe-LDH/CC, and *c*-CoFeOOH/CC reference.



Fig. S7. SCN⁻poisoning experiments



Fig. S8 Stability of the catalyst. CA measurement at 2.0 V versus Ag/AgCl. Standard reaction conditions: **1a** (0.25 M), $nBu_4NH_2PO_4$ (0.25 M), MeCN/H₂O (1:1 v/v), RT, 7 h, CoFe-LDH anode (working area: 1 cm²), Pt cathode, applied potential: 2.0 V vs. Ag/AgCl, under air.



Fig. S9 (a) The color of CoFe-LDH/NF as anode dependent on reaction time. (b), (c) XRD patterns and Co2p XPS spectra of fresh CoFe-LDH/CC and its structural evolution dependent on reaction time.



Fig. S10 (a) Conversions of 1a and yields of 2a over $Co(OH)_2/CC$, CoFe-LDH/CC, and $Fe(OH)_3/CC$ electrocatalysts. (b) $Co2p_{3/2}$ XPS spectra of used $Co(OH)_2/CC$ and CoFe-LDH/CC.

| Table S1. | ICP-AES | results of | of CoFe- | -LDH@CC. |
|-----------|----------------|------------|----------|----------|
| | | | | |

| | metal loading (mg/cm-2) |
|----|-------------------------|
| Со | 1.00 |
| Fe | 0.32 |

| S. 1a | CoFe-LDH (+) / Pt foil (-) Potential=2.0 V/ vs Ag/AgCl Undivided cell, RT 0.25 M <i>n</i> Bu ₄ NH ₂ PO ₄ , MeCN:H ₂ C | | 2a + | S 3a |
|----------|--|------------------------|----------------------|----------------------|
| Entry | Deviation from standard conditions | Conv. (%) ^b | Yield of $2a (\%)^b$ | Yield of $3a (\%)^b$ |
| 1 | PhMe as solvent ^d | <5 | trace | trace |
| 2 | THF as solvent ^c | <10 | trace | trace |
| 3 | DMSO as solvent ^c | <10 | trace | trace |
| 4 | ClCH ₂ CH ₂ Cl as solvent ^d | <5 | trace | trace |
| 5 | $MeCN/H_2O = 2:1$ as solvent | 81 | 75 | trace |
| 6 | $MeCN/H_2O = 1:2$ as solvent | 61 | 55 | trace |
| 7 | <i>n</i> Bu ₄ NClO ₄ as electrolyte | 45 | 30 | trace |
| 8 | <i>n</i> Bu ₄ NOH as electrolyte | 65 | 51 | 10 |
| 9 | <i>n</i> Bu ₄ NPF ₆ as electrolyte | 61 | 50 | 6 |
| 10 | no electrolyte | <5 | trace | trace |
| 11 | graphite instead of Pt as cathode | 21 | 15 | trace |
| 12 | GC instead of Pt as cathode | 45 | 40 | trace |

Table S2. Optimization of reaction conditions.^a

^{a,b} Conversion and yields were determined by 1H NMR using CH_2Br_2 as an internal standard. nd=not detected. ^cMeCN has higher conductivity than other polar solvents (THF and DMSO). ^dPhMe and ClCH₂CH₂Cl are nonpolar solvents.

Table S3 Comparison of reported catalytic systems and this work for sulfides oxidation.



| Entry | Cat. | Reaction conditions | t (h) | T (°C) | Yield 2a | Turnover Frequency | Ref. |
|-------|--|--|-------|--------|----------|--------------------|------------------------------------|
| | | | | | (%) | (h-1) | |
| 1 | Fe(acac) ₂ | O ₂ (2 MPa) | 2 | 100 | 91 | 31.6 | Green Chem. |
| | | PEG-1000 | | | | | 2012 , <i>14</i> , 130-135. |
| 2 | [(C ₂ N ₂ H ₈) ₄ (CH ₃ O) ₄ V | CH ₂ Cl ₂ , TBHP | 4 | 100 | Conv. = | 2.5 | J. Am. Chem. |
| | IV 4V V | | | | 100 | | Soc. 2019, |
| | 4O ₁₆]·4CH ₃ OH (V8- | | | | | | <i>141</i> , 19487- |
| | 1) (Heterogeneous | | | | | | 19497. |
| | Catalyst) | | | | | | |
| 3 | UO ₂ (OAc) ₂ ·2H ₂ O | O2 balloon, MeCN | 12 | RT | 68ª | 9.33 | Angew. |
| | | 18W blue LEDs*2 | | | | | Chem., Int. |
| | | | | | | | Ed. 2019, 58, |
| | | | | | | | 13499-13506. |
| 4 | CoFe-LDH | MeCN/HCl 1:1 v/v, | 2 | RT | 85 | 160.9 | This work |
| | | applied potential: 2.0 V | | | | | |

^a 68% yield was 2-(phenylsulfinyl)aniline.

 ${}_{b} Turnover \ Frequency \ (1/h) = \frac{mol \ of \ sulfide \ conversion}{mol \ of \ catalyst \ \times \ reaction \ time}$

| | CoFe-LDH (+) / Pt f Potential=2.0 V/ vs A Undivided cell, F 0.25 M <i>n</i> Bu ₄ NH ₂ PO ₄ , MeC | oil (-) g/AgCl RT SN:H ₂ O=1:1 | 0 2a + (1) | S Sa |
|-------|--|--|------------------------|--------------------|
| Entry | Radical scavenger (equiv) | Conv. (%) | Yield of 2a (%) | Yield of 3a |
| 1 | TEMPO (2.0) | <10 | 0 | 0 |
| 2 | BHT (2.0) | <10 | 0 | 0 |

 Table S4 Radical trapping experiments.

4. ¹H and ¹³C NMR spectra



Sulfinyldibenzene (2a): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.67-7.60 (m, 4H), 7.47-7.38 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 145.5, 130.9, 129.2, 124.6.



(Methylsulfinyl)benzene (2b): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.67-7.64 (m, 2H), 7.57-7.47 (m, 3H), 2.73 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 145.7, 131.0, 129.3, 123.5, 43.9.



1-Fluoro-4-(methylsulfinyl)benzene (2c): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a colorless oil. ¹H NMR (400 MHz, CDCl₃) δ 7.66-7.60 (m, 2H), 7.24-7.16 (m, 2H), 2.69 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 164.3 (d, J = 250 Hz), 141.20, 125.8 (d, J = 9.0 Hz), 116.7 (d, J = 23.0 Hz), 44.1.



1-Chloro-4-(methylsulfinyl)benzene (2d): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a colorless oil. ¹H NMR (600 MHz, CDCl₃) δ 7.62-7.58 (m, 2H), 7.53-7.50 (m, 2H), 2.72 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 144.2, 137.0, 129.5, 124.8, 43.9.



1-Bromo-4-(methylsulfinyl)benzene (2e): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.67 (d, J = 8.4 Hz, 1H), 7.53 (d, J = 8.4 Hz, 1H), 2.72 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 144.7, 132.4, 125.2, 125.0, 43.80.



1-Chloro-2-(methylsulfinyl)benzene (2f): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.91 (d, J = 7.6 Hz, 1H), 7.49 (t, J = 7.6 Hz, 1H), 7.41 (t, J = 7.6 Hz, 1H), 7.35 (d, J = 7.6 Hz, 1H), 2.78 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 143.4, 131.8, 129.6, 129.5, 128.0, 125.1, 41.5.



1-Chloro-3-(methylsulfinyl)benzene (2g): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.66-7.62 (m, 1H), 7.50-7.42 (m, 3H), 2.72 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 147.7, 135.5, 131.0, 130.4, 123.5, 121.5, 43.9.



1,3-Dichloro-5-(methylsulfinyl)benzene (**2h**): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.50 (d, J = 1.8 Hz, 2H), 7.44 (t, J = 1.8 Hz, 1H), 2.74 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 149.3, 136.1, 130.9, 121.8, 43.9.



(Cyclopropylsulfinyl)benzene (2i): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.66-7.61 (m, 2H), 7.52-7.45 (m, 3H), 2.29-2.18 (m, 1H), 1.25-1.15 (m, 1H), 1.05-0.83 (m, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 144.7, 130.7, 129.0, 123.8, 33.6, 3.2, 2.5.



(Benzylsulfinyl)benzene (2j): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 7.51-7.34 (m, 5H), 7.33-7.20 (m, 3H), 7.02-6.95 (m, 2H), 4.09 (d, J = 12.6 Hz, 1H),

4.00 (d, J = 12.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 142.6, 131.0, 130.2, 129.0 128.7, 128.3, 128.1 124.3, 63.4.



2-(Methylsulfinyl)pyridine (2k): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 8.56 (d, J = 4.4 Hz, 1H), 7.96 (d, J = 8.0 Hz, 1H), 7.89 (td, J = 7.6, 1.6 Hz, 1H), 7.36-7.30 (m, 1H), 2.79 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 165.8, 149.4, 138.0, 124.5, 119.1, 41.1.



Tetrahydro-2H-thiopyran 1-oxide (2l): Isolated by flash column chromatography (ethyl acetate/petroleum ether = 1:2, $R_f = 0.6$) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 2.96-2.84 (m, 2H), 2.83-2.72 (m, 2H), 2.34-2.17 (m, 2H), 1.75-1.56 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ 48.7, 24.5, 19.0.



Methyl (2R)-2-acetamido-4-(methylsulfinyl)butanoate (2m): Isolated by flash column chromatography (DCM/MeOH = 10/1, R_f = 0.6) as a white solid. ¹H NMR (400 MHz, CDCl₃) δ 4.71-4.62 (m, 1H), 3.76 (s, 3H), 2.92-2.72 (m, 2H), 2.62 (d, *J* = 1.8 Hz, 3H), 2.40-2.29 (m, 1H), 2.26-2.11 (m, 1H), 2.04 (d, *J* = 2.5 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 171.5 (d, *J* = 4.2 Hz), 170.5-169.7 (m), 52.2 (d, *J* = 3.0 Hz), 50.9 (d, *J* = 19.6 Hz), 49.9 (d, *J* = 22.8 Hz), 38.1 (d, *J* = 2.4 Hz), 25.1 (d, *J* = 42.0 Hz), 22.51.



Methyl (5-(propylsulfinyl)-1H-benzo[d]imidazol-2-yl)carbamate (2n): Isolated by flash column chromatography (DCM/MeOH = 10/1, R_f = 0.6) as a white solid. ¹H NMR (600 MHz, DMSO-*d*₆) δ 11.94 (s, 2H), 7.74 (s, 1H), 7.58 (d, *J* = 8.0 Hz, 1H), 7.35 (dd, *J* = 8.0, 1.2 Hz, 1H), 3.81 (s, 3H), 2.88-2.73 (m, 2H), 1.69-1.57 (m, 1H), 1.54-1.43 (m, 1H), 0.96 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (150 MHz, DMSO-*d*₆) δ 154.3, 148.6, 136.2, 116.8, 114.2, 109.9, 58.2, 52.6, 15.3, 12.9.



(Z)-2-(5-fluoro-2-methyl-1-(4-(methylsulfinyl)benzylidene)-1H-inden-3-yl)acetic acid (2o): Isolated by flash column chromatography (DCM/MeOH = 10/1, $R_f = 0.6$) as a yellow solid. ¹H NMR (600 MHz, CDCl₃) δ 7.71 (d, J = 8.0 Hz, 2H), 7.63 (d, J = 8.0Hz, 2H), 7.16-7.09 (m, 2H), 6.89 (dd, J = 8.8, 1.8 Hz, 1H), 6.57-6.51 (m, 1H), 3.58 (s, 2H), 2.83 (s, 3H), 2.20 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 174.7, 163.3 (d, J = 246Hz), 146.6 (d, J = 9.0 Hz), 144.9, 141.6, 139.8, 138.4, 131.5 (d, J = 2.4 Hz), 130.3, 129.5 (d, J = 2.4 Hz), 128.2, 123.9, 123.7 (d, J = 9.0 Hz), 110.7 (d, J = 24 Hz), 106.2 (d, J = 24 Hz), 43.6, 31.5, 10.5. ¹⁹F NMR (376 MHz, CDCl₃) δ -112.8.



6-Methoxy-2-(((4-methoxy-3,5-dimethylpyridin-2-yl)methyl)sulfinyl)-1H-

benzo[d]imidazole (2p): Isolated by flash column chromatography (DCM/MeOH = 10/1, R_f = 0.6) as a white solid. ¹H NMR (600 MHz, DMSO- d_6) δ 13.46 (s, 1H), 8.19 (s, 1H), 7.56 (s, 1H), 7.10 (s, 1H), 6.93 (d, J = 8.2 Hz, 1H), 4.76 (d, J = 13.6 Hz, 1H), 4.66 (d, J = 13.6 Hz, 1H), 3.81 (s, 3H), 3.68 (s, 3H), 2.20 (s, 3H), 2.17 (s, 3H).



































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