Electronic Supplementary Information (ESI) for 1 2 Close band center and rapid adsorption kinetics facilitate selective 3 electrochemical sensing of heavy metal ions 4 5 Zong-Yin Song,^{a,b,§} Pei-Hua Li,^{a,b,§} Meng Yang,^{a,*} Shi-Hua Chen,^{a,b} Xiang-Yu Xiao,^{a,b} 6 Wanchun Duan,^b Chu-Hong Lin,^{a,*} Xing-Jiu Huang^{a,b,*} 7 8 ^a Key Laboratory of Environmental Optics and Technology, And Environmental 9 Materials and Pollution Control Laboratory, Institute of Solid State Physics, HFIPS, Chinese Academy of Sciences, Hefei 230031, China 10 11 ^b Department of Materials Science and Engineering, University of Science and 12 Technology of China, Hefei 230026, China 13 14 * Corresponding author 15 16 E-mail addresses: myang@iim.ac.cn;chlin@iim.ac.cn; xingjiuhuang@iim.ac.cn 17 Tel.: +86 551 6559 1167; Fax:+86 551 6559 2420. 18 § Z. Y. Song and P. H. Li contributed equally to this work.

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66 1. Experimental Section

1.1. DFT calculations. All calculations on HMIs/Ni(111) (HMIs include As(III), 67 Cu(II), Pb(II), and Hg(II), abbreviated as H₃AsO₃/Ni(111), Cu/Ni(111), Pb/Ni(111), 68 and Hg/Ni(111)) were developed by Generalized Gradient Approximation (GGA) with 69 the Perdew-Burke-Ernzerhof (PBE) exchange-correlation parameterization by using 70 CASTEP code. The ultrasoft pseudo potentials are subjected to reciprocal space. The 71 calculation accuracy is set to fine. The pseudopotential Schrödinger equation was self-72 consistently solved. The Ni $3d^84s^2$, Pb $5d^{10}6s^26p^2$, Hg $5d^{10}6s^2$, Cu $3d^{10}4s^1$, As $4s^24p^3$, 73 H 1s¹, O 2s²2p⁴ electrons were particularly treated as valence electrons. The wave 74 functions were expanded in plane-wave basis sets with a kinetic energy cutoff of 400 75 eV for all systems studied in this work.¹⁻² The adopted vacuum was 13 Å. We fixed two 76 bottom layers and allowed the two surface layers to relax fully. Brillouin-zone 77 integrations were performed using a Monkhorst-Pack grid of special k-point meshes, 78 and meanwhile a $(2 \times 2 \times 1)$ k-point grid was applied. The geometry optimization 79 parameters of total energy convergence, max atom force, max atom stress, max 80 displacement, was 5×10^{-6} eV, 2×10^{-5} eV Å⁻¹, 0.05 Gpa, 0.001 Å, respectively. The E_{ads} 81 is defined as $E_{ads} = E_{adsorbent+slab} - (E_{slab} + E_{adsorbent})$, where $E_{adsorbent+slab}$, E_{slab} and $E_{adsorbent}$ 82 are the energy of the HMIs absorbed on slab system, isolated slab surface, and isolated 83 HMIs, respectively. Under this definition, the more energetically stable adsorption 84 accompanied by the more negative value. The Mulliken charge equation was applied to 85 analyze the electronegativity of the center metal atom. The density of state (DOS) is 86 projected to the *d*-state, and the interaction with the state of the adsorbate can be 87 characterized by the DOS moment of the *d* orbital. The first moment is the *d*-band 88 89 center:

$$\varepsilon_d = \frac{\int_{-\infty}^{\infty} n_d(\varepsilon)\varepsilon d\varepsilon}{\int_{-\infty}^{\infty} n_d(\varepsilon)d\varepsilon}$$

91 ε_d is the center of the *d* band, ε is energy, $n_d(\varepsilon)$ is the density of electrons, corresponding 92 to X and Y in the DOS diagram, respectively. The center position of the band in the 93 following is calculated by this formula.

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1.2. Preparation of Ni nanowires. Dissolve 1.2 g NaOH in 35 mL ethylene glycol to 95 create a homogeneous solution.³ Then add 10 mL of N₂H₄·H₂O with a mass percentage 96 of 80% to NaOH, stir the solution to form solution A. Disperse 0.1 g of NiCl₂·6H₂O 97 (metal-based, Aladdin) in 5 mL of ethylene glycol to make solution B. The prepared 98 were heated at 80°C for 10 min, respectively. Poured solution B to A neatly without 99 stirring, and place the beaker in a magnet environment. The solution turned from blue 100 to black instantly, followed by the precipitation of black flocs. After 10 min, a layer of 101 fluffy black product was floating on the surface of the solution. The products were taken 102 out and centrifuged with isopropanol five times. Finally dried at 60°C until aried. 103

104

105 **1.3. Material characterization.** X-ray diffraction (XRD) was performed on an X'Pert 106 Pro MPD X-ray diffractometer, using Cu K_{α} (λ K_{$\alpha 1$} =1.5418 Å) as the radiation source 107 to realize the phase characterization. XPS data were measured by a VG ESCALAB 108 MKII spectrometer with an Mg K_{α} X-ray source (1253.6 eV, 120 W). The 109 morphological characterization of nanomaterials was achieved by field-emission 110 scanning electron microscopy (FESEM, Quanta 200 FEG, FEI Company, USA). The 111 energy dispersive spectrometer (EDS; JEM-2010 transmission electron microscope

operating at 200 kV; quantitative method: Cliff Lorimer thin ratio section) was used to 112 characterize the surface condition and the element distribution of the Ni nanowires (Ni 113 114 NWs). Transmission electron microscopy (TEM) and high-resolution transmission electron microscopy (HRTEM) was performed using a JEM-2010 transmission electron 115 microscope operating at 200 kV (quantitative method, Cliff Lorimer thin ratio section). 116 117 Electrochemical experiments were performed by a CHI660E computer-controlled potentiator (Chen-Hua Instruments Co., Shanghai, China). Electrochemical tests were 118 operated with GCE as the working electrode, Pt wire as the counter electrode, and 119 120 Ag/AgCl as the reference electrode. SWASV technology was adopted to detect the target analyte. 121

122 1.4. Fabrication of Modified Electrode. The structure modification GCE of the nickel nanowires was performed: the nickel nanowires (1 mg) were dispersed in 1 mL of ultra-123 high purity water, and ultrasonicated for 15 minutes. Before modification, the bare 124 glassy carbon electrode (GCE) was mechanically ground with alumina powder, then 125 cleaned with 1:1 HNO₃, ethanol, and ultra-high purity water, respectively, and then 126 dried with N₂ on the electrode surface. Finally, 5 µL of Ni nanowire suspension was 127 dropped into GCE, and the modified electrode was dried in a natural environment for 128 129 24 h.

130 1.5. EXAFS Analysis. The As K-edge EXAFS spectra were obtained in transmission 131 mode at the BL11B beamline of Shanghai Synchrotron Radiation Facility. Athena 132 software was used to subtract the background signal of the pre-edge and post-edge and 133 to normalize spectra intensity. IFEFFIT software was employed to calibrate the energy 134 scale and correct the background.

1.6. Electrochemical Experiments. Square wave anodic stripping voltammograms 135 (SWASV) and linear sweep voltammetry (LSV) were applied to study the 136 137 electrochemical behavior of HMIs at Ni NWs. The optimized SWASV parameters were 15 Hz frequency, 25 mV amplitude, and 4 mV step potential. For cumulative steps, 138 HMI were deposited to the working electrode at the potential of -0.8 V (vs. Ag/AgCl) 139 140 for 150 s in a 0.1 M HAc-NaAc buffer solution (pH 5.0). The voltammetry measurement was carried out immediately after the deposition. The anodic stripping 141 currents were all obtained at the potential range of -0.8 to 0.6 V. After each test, the 142 143 electrode was immersed in the same HAc-NaAc buffer solution at 0.8V for 100s regeneration under stirring. The stripping LSV was measured under the same condition, 144 and the scan rate of LSV was set as 0.1 V s⁻¹. The same GCE and the reference electrode 145 (Ag/AgCl) was applied for all the experiments of this work. Each experiment was 146 repeated three times to ensure reproducibility. Cyclic voltammetry (CV) and 147 electrochemical impedance spectroscopy (EIS) in a mixed solution of 0.1 M KCl and 148 5.0 mM K₃[Fe(CN)₆] solution were operated to characterize the modified GCEs. 149

150 1.7. Adsorption experiments

Adsorption experiments were carried out at ambient temperature with an adsorbent load of 1.0 mg mL⁻¹. In most case, the Ni nanowires (8 mg) were dispersed in 8 mL HAc-NaAc buffer solution, which was added 2 mL As(III), Hg(II), Cu(II), and Pb(II) solutions with concentrations 0.01 mol L⁻¹, respectively. Then incubate with shaking at 200 rpm for 24 h at 18°C to achieve equilibrium. Finally, a concrete sample was obtained by centrifuged, then washed twice with deionized water, and then freeze-dried for 12 h.

158 1.8. Kinetic Simulations.

159 The simulation model contains the diffusion of HMI and the electrochemical reaction160 at the electrode. The electrochemical adsorption and desorption reactions of HMIs are161 simply modeled:

$$162 \qquad M^{n^+}(sol) + e^- \xrightarrow{k_{ads}} M^0(ads)$$

163 M^{n+} is the HMI, where n is 2 for Cu, Hg, Pb and 3 for As. k_{ads} (unit: m s⁻¹) and k_{des} (unit: 164 s⁻¹) are the apparent adsorption and desorption rate constants. The reaction equations 165 are written as:

$$\frac{\partial \Gamma_{\mathrm{M}^{0}}}{\partial t} = D\nabla c_{\mathrm{M}^{n^{+}}} = k_{\mathrm{ads}} \exp\left(-\frac{0.5F}{RT}(E-E_{\mathrm{f}})\right) c_{\mathrm{M}^{n^{+}}} - k_{\mathrm{des}} \exp\left(\frac{0.5F}{RT}(E-E_{\mathrm{f}})\right) \Gamma_{\mathrm{M}^{0}}$$

167 Γ is the surface coverage (unit: mol m⁻²) of the deposited heavy metal, *t* (unit: s) is the 168 reaction time, *c* (unit: mol m⁻³) is the concentration of HMI, *F* is the Faraday constant 169 (96485 C mol⁻¹), *R* is the gas constant (8.314 J mol⁻¹ K⁻¹), *T* is the room temperature 170 (298.15 K), *E* (unit: V) is the applied potential of the working electrode, E_f (unit: V) is 171 the formal potential of Mⁿ⁺/M⁰, *D* (unit: m² s⁻¹) is the diffusion coefficient of HMI. The 172 diffusion coefficient for all HMIs in this work is assumed to be 5×10⁻¹⁰ m² s⁻¹. The 173 diffusion of HMI in solution is described by Fick's second law:

$$\frac{\partial c_{M^{n^+}}}{\partial t} = D\nabla^2 c_{M^{n^+}}$$

175 The current is calculated via:

$$I = AFn \frac{\partial \Gamma_{M^0}}{\partial t}$$

177 where A is the surface area of the electrode.

To obtain the surface coverage of deposited heavy metal, a series of linear-sweeping 178 stripping voltammetry (LSV) of HMIs are measured under the same conditions as the 179 SWASV experiments, where the LSV peak charge (surface coverage = LSV peak 180 charge/(AFn)) can be calculated via integrating the peak current. For simulation, the 181 peak current is divided by the square-wave scanning frequency f, the electrode area 182 $\pi r_{\rm ele}^2$ and the surface coverage of deposited HMIs $\Gamma_{\rm HMI}$, where $\Gamma_{\rm HMI}$ is calculated from 183 the linear-sweeping voltammograms. Therefore, by combining the experimental 184 SWASV with the simulation model, the adsorption and desorption rate constants of 185 186 Hg(II) can be determined. Similarly, the adsorption and desorption rate constants are 187 also measured for Cu(II), Pb(II), and As(III), as listed in Table S7. To test the producibility of the kinetics simulation, the same SWASV and LSV experiments are 188 repeated at another Ni NWs modified electrode, where the simulated kinetic parameters 189 for the 1st and the 2nd test are both shown in Table S7. The kinetic parameters measured 190 at the two electrodes are very close, proving the reliability of both simulation and 191 experiment. 192

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Fig. S1. The top view of (a) As(III); (b) Cu(II), (c) Pb(II), and (d) Hg(II) and side view
of (e) As(III), (f) Cu(II), (g) Pb(II), and (h) Hg(II) adsorption configuration on Ni(111).
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Fig. S2. The side-view of charge density difference of (a) As(III), (b) Cu(II), (c) Pb(II), and (d) Hg(II), and the isovalue is $0.02 \ e^{-3}$.



Fig. S3. The TDOS of As(III), Cu(II), Pb(II), and Hg(II) before and after adsorption.



209 Energy(eV) Energy(eV) 210 **Fig. S4**. PDOS of HMIs (a) Cu(II), (b) Pb(II), (c) Hg(II), and (d) As(III) before and 211 after adsorption.



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Fig. S5. (a) SEM, (b) XRD, (c) EDX, and (d) Normalized XANES spectra comparison of Ni K-edge.

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Scanning electron microscopic (SEM) image of Ni NWs (Fig. S5a) clearly shown that the whole exterior of every nanowires is evenly wrapped by the spot burrs. The quantitative SEM energy dispersive X-ray spectrum (SEM-EDX) of Ni NWs (Fig. S5c) divulged the Ni/O atomic ratio of 81.48/4.78. In X-ray absorption near-edge structure (XANES) spectra (Fig. S5d), after the adsorption of As(III), the white line peak increases and the oxidation state of Ni increases, which means a loss of electrons.



226 Fig. S6. (a) CV and (b) Nyquist diagram of EIS at Ni NWs and bare GCE.



229 Fig. S7. CV at Ni NWs for 50 cycles.



233 Fig. S8. SWASV curves of the coexistence of 1 μ M As(III), Cu(II), Pb(II), and Hg(II). 234



236 Fig. S9. SWASV response current of (a) Pb(II) and (b) Hg(II) with Ni NWs and the

- 237 upper right corner is the calibration curve of peak current versus concentrations,
- 238 respectively. (All data has been deducted from the baseline current).



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Fig. S10. XPS spectra of (a) Ni 2p region of Ni NWs before adsorption and after adsorption of As(\mathbb{II}), Cu(II), Pb(II), and Hg(II), before and after adsorption of (b) As(\mathbb{II}), (c) Cu(II), (d) Pb(II), and (e) Hg(II).

For Ni 2*p* peaks of all adsorption configurations, in addition to the previous peaks of Ni(0) and Ni(II) at 852.5, 855.5, 860.6, 870.1, 872.9, and 879.2 eV, a new peak appears at 853.7eV after adsorption, which was found to be the characteristic peak of Ni(I). Moreover, the ratio of Ni(I) in the four sets of data are 7.51%, 24.64%, 20.03%, and 2.80%, corresponding As(III), Cu(II), Pb(II), and Hg(II), respectively. It can be discovered that the ratio of Ni(I) peaks recorded in Table S4 is positively correlated with the trend of adsorption energy and electrochemical sensitivity, which is further consistent with the Mulliken charge. The relative shift of the ions in the four sets of adsorption configurations is also shown in Fig. S10b-e, and the related data were also presented in Fig. S10. As(III) 3d at 44.6 eV in Ni NWs was a little higher than that in NaAsO2 (44.3 eV), which embodied a weak interplay between Ni NWs and As(III). While Cu(II) 3d XPS peak at 932.7 eV after adsorption was much lower than that in Cu(NO3)2 (933.5eV), it is worthy of note that peaks of Cu 3d, the binding energy of Pb 4f and Hg 4f were moved obviously than corresponding standard material.

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262 Fig. S11. Structure and Mulliken charge of Ni and HMIs in the different (a) As(III),

263 (b) Cu(II), (c) Pb(II), and (d) Hg(II) configurations after adsorption.

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Fig. S12. The charge density difference image of (a) As(III), (b) Cu(II), (c) Pb(II), and (d) Hg(II) on Ni(111). The blue region indicates the outflow of electrons, and the red region indicates the inflow of electrons.



Fig. S13. LSV response curves of (a) As(III), (b) Cu(II), (c) Pb(II), and (d) Hg(II) with
Ni NWs.



solutions, (b) the dependence of k_{ads} on E_{ads} calculated from DFT calculations. 276

278 3. Tables

Table S1. The bond length and total energy, single ion or molecular energy, adsorption
energy of As(III), Cu(II), Pb(II), Hg(II) on Ni(111) facet, respectively.

	Ni-M (Å)	$E_{\text{total}} \left(\text{eV} \right)$	$E_{\rm M}~({\rm eV})$	$E_{\rm ads}({\rm eV})$
Pure Ni(111)	\	-108334.11	/	\
Hg/Ni(111)	2.74	-109466.53	-1130.69	-1.73
Cu/ Ni(111)	2.39	-109810.02	-1473.06	-2.85
Pb/ Ni(111)	2.66	-109992.73	-1656.05	-2.57
H ₃ AsO ₃ /Ni(111)	3.10	-109865.46	-1530.14	-1.21

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Table S2. The fitting parameters of ex-situ As K-edge EXFAS curves for the H₃AsO₃ adsorbed on Ni NWs (coordination number (CN), interatomic distance (R), Debye-Waller factor (σ^2), and inner potential shift (ΔE_0)).

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Sample	Path	CN	R(Å)	$\sigma^2(10^{-3} \text{ Å})$	$\Delta E_0 (eV)$	R-factor
H ₃ AsO ₃ /	As-O	2.84	1.86±0.02	2.01	-12.02±1.68	0.027
Ni NWs	As-Ni	1.40	3.05±0.01	4.20	-15.07±1.23	0.027

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Table S3. Mulliken charge with the content of Ni(I) of pure Ni(111) facet and Ni atom in different adsorption configurations.

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	Mulliken charge (e)	Ni(I) %
Pure Ni NWs	-0.14	0
As-Ni	-0.15	7.51
Cu-Ni	-0.28	24.64
Pb-Ni	-0.25	20.03
Hg-Ni	-0.16	2.80

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	Bond	Population
Hg/Ni(111)	Ni 50 -Hg	0.57
Cu/Ni(111)	Ni 50 - Cu	0.27
	Ni 73 - Cu	0.27
	Ni 83 - Cu	0.27
	Ni 80 - Cu	0.27
Pb/Ni(111)	Ni 50 - Pb	0.34
	Ni 53 - Pb	0.34
	Ni 43 - Pb	0.34
	Ni 20 - Pb	0.34
H ₃ AsO ₃ /Ni(111)	Ni 50 - As	0.75

Table S4. Bonding mode and population in different adsorption configurations.

302 **Table S5.** Mulliken charge (*e*) of first-layer, second-layer, ion, and bonding Ni in 303 different configurations.

303 u 304

	First-layer	Second-layer	Ion	Bonding-Ni
Pure Ni(111)	0	0.18	١	\
Hg/Ni(111)	-0.28	0.21	0.18	-0.3
Cu/Ni(111)	-0.32	0.22	0.15	-0.1
Pb/Ni(111)	-0.37	0.33	0.21	-0.11
H ₃ AsO ₃ /Ni(111)	-0.28	0.24	0.18	-0.46

306 **Table S6.** The band center (ϵ) and band shift (Δ d) of Ni and heavy metal ions before 307 and after adsorption. The distance of the band centers between Ni and the HMI 308 adsorbent(d_{Ni-M}) were also caluclated. 309

	Element	ε (Å)	$d_{\text{Ni-M}}(\text{\AA})$
Before adsorption	Ni-bulk	-1.44	
	Cu	-0.08	
	As	1.89	\
	Pb	0.03	
	Hg	3.56	
After adsorption	Cu-Ni	-1.10	
	As-Ni	-1.12	١
	Pb-Ni	-1.14	1
	Hg-Ni	-1.32	
	Cu	-1.80	0.70
	As	0.49	1.60
	Pb	-0.28	0.86
	Hg	0.17	1.49

310

311 **Table S7.** Simulated k_{ads} and k_{des} according to Fig. 5 (SWASV) and Fig. S8 (LSV). a/a'

312 are the simulation results corresponding to two independent tests.

313

HMI	$lg(k_{ads} (m s^{-1}))$	$\lg(k_{\rm des}({\rm s}^{-1}))$
As(III)	-9.38/-9.53	-2.10/-2.06
Cu(II)	-5.45/-4.48	1.90/0.50
Pb(II)	-5.95/-5.38	2.40/2.20
Hg(II)	-7.45/-7.23	4.40/2.80

Plane	Sites	E _{total} (eV)	E _{ads} (eV)
(001)	Тор	-109850.06	-1.96
	Hollow	-109850.87	-1.77
	Bridge	-109849.24	-1.14
(110)	Тор	-109834.39	0.64
	Hollow	-109834.07	0.96
	Bridge	-109834.15	0.88
(111)	Тор	-109856.61	-2.13
	Hollow	-109856.64	-2.85
	Bridge	-109856.56	-2.08

316	Table S8. The free energy of Ni(111), Ni(001), and Ni(110). The adsorption energy
317	of Cu(II) at different sites in different planes.

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