## Supplementary Information

## Metal-deposited bismuth oxyiodide nanonetworks with tunable enzyme-like

## activity: sensing of mercury and lead ions

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**Table S1.** Elemental ratio (Bi/X; X = Cl, Br or I) analysis of BiOX nanostructures by XPS, ICP-OES or ICP-MS.

Nanostructures	XPS	Bi/X (ICP-OES or ICP-MS)
BiOCl	7.2	$4.9^{a}$
BiOBr	7.0	$5.5^{b}$
BiOI	3.4	$2.2^{b}$

<sup>a</sup>Determined by ICP-OES. <sup>b</sup>Determined by ICP-MS.

**Table S2.** Analytical results for the detection of  $Hg^{2+}$  and  $Pb^{2+}$ -spiked water samples by O<sub>2</sub>/AR–Au NPs/BiOI nanocomposites and  $H_2O_2/AR$ –NiO NPs/BiOI nanocomposites sensing systems, respectively.

Probe	Real sample	Added Hg <sup>2+</sup> (nM)	Found Hg <sup>2+</sup> (nM)	Recovery	RSD $(n = 3)$
O2/AR–Au NPs/BiOI nanocomposites	Tap water	20	20.7	104%	6.8%
		100	99.9	99.9%	6.7%
		500	539	108%	6.8%
		20	19.5	97.5%	5.9%
	River water	100	106	106%	5.2%
		500	529	106%	5.3%
	Lake water	20	20.4	102%	7.3%
		100	104	104%	5.1%
		500	559	112%	2.3%
		20	22.1	111%	8.0%
	Sea water	100	99.4	99.4%	6.9%
		500	555	111%	4.0%
Probe	Real sample	Added Pb <sup>2+</sup> (nM)	Found Pb <sup>2+</sup> (nM)	Recovery	RSD (n = 3)
Probe	Real sample	<b>Added Pb<sup>2+</sup> (nM)</b> 20	<b>Found Pb<sup>2+</sup> (nM)</b> 18.8	Recovery 94.0%	<b>RSD (n = 3)</b> 7.8%
Probe	Real sample Tap water	Added Pb <sup>2+</sup> (nM) 20 100	<b>Found Pb<sup>2+</sup> (nM)</b> 18.8 96.3	Recovery           94.0%           96.3%	<b>RSD (n = 3)</b> 7.8% 7.8%
Probe	Real sample Tap water	Added Pb <sup>2+</sup> (nM) 20 100 500	Found Pb <sup>2+</sup> (nM)           18.8           96.3           545	Recovery           94.0%           96.3%           109%	<b>RSD (n = 3)</b> 7.8% 7.8% 7.5%
Probe	Real sample Tap water	Added Pb <sup>2+</sup> (nM) 20 100 500 20	Found Pb <sup>2+</sup> (nM)         18.8       96.3         545       21.5	Recovery           94.0%           96.3%           109%           108%	RSD (n = 3) 7.8% 7.8% 7.5% 7.5%
Probe	Real sample Tap water River water	Added Pb <sup>2+</sup> (nM) 20 100 500 20 100	Found Pb <sup>2+</sup> (nM)         18.8       96.3         545       21.5         106       106	Recovery           94.0%           96.3%           109%           108%           106%	RSD (n = 3) 7.8% 7.8% 7.5% 7.5% 8.2%
Probe H2O2/AR-NiO	Real sample Tap water River water	Added Pb <sup>2+</sup> (nM) 20 100 500 20 100 500	Found Pb <sup>2+</sup> (nM)         18.8       96.3         545       21.5         106       533	Recovery           94.0%           96.3%           109%           108%           106%           107%	RSD (n = 3) 7.8% 7.8% 7.5% 7.5% 8.2% 6.0%
Probe H2O2/AR-NiO NPs/BiOI	Real sample Tap water River water	Added Pb <sup>2+</sup> (nM) 20 100 500 20 100 500 20	Found Pb <sup>2+</sup> (nM)         18.8       96.3         545       21.5         106       533         19.4       19.4	Recovery           94.0%           96.3%           109%           108%           106%           107%           97.0%	RSD (n = 3) 7.8% 7.8% 7.5% 7.5% 8.2% 6.0% 8.2%
Probe H2O2/AR–NiO NPs/BiOI nanocomposites	Real sample Tap water River water Lake water	Added Pb <sup>2+</sup> (nM) 20 100 500 20 100 500 20 100	Found Pb <sup>2+</sup> (nM)         18.8       96.3         545       21.5         106       533         19.4       112	Recovery           94.0%           96.3%           109%           108%           106%           107%           97.0%           112%	RSD (n = 3)         7.8%         7.8%         7.5%         7.5%         8.2%         6.0%         8.2%         7.2%
Probe H <sub>2</sub> O <sub>2</sub> /AR–NiO NPs/BiOI nanocomposites	Real sample Tap water River water Lake water	Added Pb <sup>2+</sup> (nM) 20 100 500 20 100 500 20 100 500	Found Pb <sup>2+</sup> (nM)         18.8       96.3         96.3       545         21.5       106         533       19.4         112       548	Recovery           94.0%           96.3%           109%           108%           106%           107%           97.0%           112%           110%	RSD (n = 3)         7.8%         7.8%         7.5%         7.5%         8.2%         6.0%         8.2%         7.2%         4.7%
Probe H <sub>2</sub> O <sub>2</sub> /AR–NiO NPs/BiOI nanocomposites	Real sample Tap water River water Lake water	Added Pb <sup>2+</sup> (nM) 20 100 500 20 100 500 20 100 500 20 20 20 20 20	Found Pb <sup>2+</sup> (nM)         18.8       96.3         96.3       545         21.5       106         533       19.4         112       548         20.9       20.9	Recovery           94.0%           96.3%           109%           108%           106%           107%           97.0%           112%           110%           105%	RSD (n = 3)           7.8%           7.8%           7.5%           7.5%           8.2%           6.0%           8.2%           7.2%           4.7%           7.4%
Probe H <sub>2</sub> O <sub>2</sub> /AR–NiO NPs/BiOI nanocomposites	Real sample Tap water River water Lake water Sea water	Added Pb <sup>2+</sup> (nM) 20 100 500 20 100 500 20 100 500 20 100 500 100 500	Found Pb <sup>2+</sup> (nM)         18.8         96.3         545         21.5         106         533         19.4         112         548         20.9         107	Recovery           94.0%           96.3%           109%           108%           106%           107%           97.0%           112%           110%           105%           107%	RSD (n = 3)           7.8%           7.8%           7.5%           7.5%           8.2%           6.0%           8.2%           7.2%           4.7%           7.4%           6.2%



*Scheme S1.* Schematic representation of the evaluation of metal-deposited BiOI having (A) POXand OX- and (B) CAT-like activities.



*Figure S1.* SEM images of as-prepared (A) BiOCl nanosheets, (B) BiOBr nanosheets, and (C) BiOI nanonetworks.



*Figure S2.* XRD patterns of as-prepared (A) BiOCl nanosheets, (B) BiOBr nanosheets, and (C) BiOI nanonetworks.



*Figure S3.* Survey scan XPS spectra of as-prepared (A) BiOCl nanosheets, (B) BiOBr nanosheets, and (C) BiOI nanonetworks.



*Figure S4.* (A) TEM image, (B) HRTEM image, and (C) high-angle annular dark-field scanning TEM mapping images of NiO NPs/BiOI nanocomposites.



*Figure S5.* (A) TEM image, (B) HRTEM image, and (C) high-angle annular dark-field scanning TEM mapping images of ZnO NPs/BiOI nanocomposites.



*Figure S6.* (A) TEM image, (B) HRTEM image, and (C) high-angle annular dark-field scanning TEM mapping images of MnO<sub>2</sub> NPs/BiOI nanocomposites.