Synthesis, Characterization and X-ray Attenuation Properties of Ultrasmall BiOI Nanoparticles: Towards Renal Clearable Particulate CT Contrast Agents

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Summary of structure determination for BiOI by X-ray powder diffraction

Table S1 Crystal data

| Crystal Data | Value |
|--------------|-------|
| BiOI         |       |
| $F_{000}$    | 288   |
| $M_r$        | 351.88|
| $D_x$        | 8.002 Mg m$^{-3}$ |
| Tetragonal, $P4/nmm$ |       |
| Melting point | ? K  |
| Hall symbol: -P 4a 2a |       |
| $K\alpha_1$, $K\alpha_2$ radiation, $\lambda = 1.540600, 1.544400$ Å |       |
| $a = 3.99399(4)$ Å |       |
| $b = 3.99399(4)$ Å |       |
| $c = 9.15486(8)$ Å |       |
| $V = 146.038 (3)$ Å$^3$ |       |
| $Z = 2$ |       |

Data collection

| D8 Advance powder diffractometer | $T = 295$ K |
| Radiation source: sealed tube | $2\theta_{min} = 7.00^\circ$ |
Monochromator: Ni beta-filter  \( 2\theta_{\text{max}} = 109.07^\circ \)
Specimen mounted in reflection mode  Increment in \( 2\theta = 0.02^\circ \)
Background-less sample holder  Scan method: step

**Refinement**

Least-squares matrix: full  54 parameters
\( R_p = 0.027 \)  2 constraints
\( R_{wp} = 0.036 \)  \( S = 1.39 \)
\( R_{exp} = 0.026 \)  \( (\Delta/\sigma)_{\text{max}} = 0.04 \)
\( R_{F2} = 0.016 \)

Profile function: CW Profile function number 3 with 19 terms, Pseudovoigt profile coefficients as parameterized in P. Thompson, D.E. Cox & J.B. Hastings (1987). J. Appl. Cryst., 20, 79-83. Asymmetry correction of L.W. Finger, D.E. Cox & A.P. Jephcoat (1994). J. Appl. Cryst., 27, 892-900.

\#1(GU) = 119.081 \#2(GV) = -5.084 \#3(GW) = 11.207
\#4(GP) = 25.131 \#5(LX) = 1.863 \#6(LY) = 26.859
\#7(S/L) = 0.0103 \#8(H/L) = 0.0103 \#9(trns) = 2.18
\#10(shift) = -8.9199 \#11(stec) = -9.60 \#12(tec) = 14.58
\#13(fsec) = 0.00 \#14(L11) = -0.469 \#15(L22) = -1.237
\#16(L33) = 0.031 \#17(L12) = 0.358 \#18(L13) = 0.126
\#19(L23) = 0.292

Peak tails are ignored where the intensity is below 0.005 times the peak Aniso. broadening axis 0 0 1.

Preferred orientation correction: March-Dollase, Ratio = 0.64143, h= 0, k= 0, l= 1; Preferred orientation correction range: Min= 0.4097, Max= 5.3361

### Table S2 Fractional atomic coordinates and isotropic displacement parameters (Å²)

|    | x     | y    | z             | \( U_{iso} \) |
|----|-------|------|---------------|---------------|
| Bi1| 0.25  | 0.25 | 0.13358 (3)   | 0.01169 (7)   |
| O1 | 0.25  | 0.75 | 0.0           | 0.01169 (7)   |
| I1 | 0.25  | 0.25 | 0.66586 (5)   | 0.01169 (7)   |
Table S3 Geometric parameters (Å, °)

| Bond                      | Distance (Å) | Bond Angle (°) |
|---------------------------|--------------|----------------|
| Bi1—O1i                   | 2.3417(2)    | O1—Bi1         | 2.3417(2) |
| Bi1—O1                    | 2.3417(2)    | O1—Bi1viii     | 2.3417(2) |
| Bi1—O1ii                  | 2.3417(2)    | O1—Bi1ix       | 2.3417(2) |
| Bi1—O1iii                 | 2.3417(2)    | O1—Bi1x        | 2.3417(2) |
| Bi1—I1iv                  | 3.3686(3)    | I1—Bi1iv       | 3.3686(3) |
| Bi1—I1v                   | 3.3686(3)    | I1—Bi1v        | 3.3686(3) |
| Bi1—I1vi                  | 3.3686(3)    | I1—Bi1vi       | 3.3686(3) |
| Bi1—I1vii                 | 3.3686(3)    | I1—Bi1vii      | 3.3686(3) |
| O1i—Bi1—O1                | 117.036(13)  | O1iii—Bi1—I1vi | 77.237(5) |
| O1i—Bi1—O1i               | 74.174(6)    | O1iii—Bi1—I1vi | 77.237(5) |
| O1i—Bi1—O1ii              | 74.174(6)    | I1iv—Bi1—I1v   | 72.716(7) |
| O1i—Bi1—I1iv              | 77.237(5)    | I1iv—Bi1—I1vi  | 72.716(7) |
| O1i—Bi1—I1v               | 142.2063(14) | I1iv—Bi1—I1vii | 113.940(15) |
| O1i—Bi1—I1vi              | 77.237(5)    | I1v—Bi1—I1vi   | 113.940(15) |
| O1i—Bi1—I1vii             | 142.2063(14) | I1v—Bi1—I1vii  | 72.716(7) |
| O1—Bi1—O1i                | 74.174(6)    | I1vi—Bi1—I1vii | 72.716(7) |
| O1—Bi1—O1ii               | 74.174(6)    | Bi1—O1—Bi1viii | 117.036(13) |
| O1—Bi1—I1iv               | 142.2063(14) | Bi1—O1—Bi1ix   | 105.826(6) |
| O1—Bi1—I1v                | 77.237(5)    | Bi1—O1—Bi1x    | 105.826(6) |
| O1—Bi1—I1vi               | 142.2063(14) | Bi1viii—O1—Bi1ix | 105.826(6) |
| O1—Bi1—I1vii              | 77.237(5)    | Bi1viii—O1—Bi1x | 105.826(6) |
| O1ii—Bi1—O1iii            | 117.036(13)  | Bi1ix—O1—Bi1x  | 117.036(13) |
| O1ii—Bi1—I1iv             | 77.237(5)    | Bi1iv—I1—Bi1v  | 72.716(7)  |
Symmetry codes: (i) x, y−1, z; (ii) 1/2−y, x, z; (iii) 3/2−y, x, z; (iv) −x, −y, 1−z; (v) −x, 1−y, 1−z; (vi) 1−x, −y, 1−z; (vii) 1−x, 1−y, 1−z; (viii) x, y+1, z; (ix) −x, −y, −z; (x) 1−x, 1−y, −z.

Data collection: XRD commander; cell refinement: GSAS; program(s) used to refine structure: GSAS; software used to prepare material for publication: Platon, publCIF.

Figure S1 Rietveld refinement plot of BiOI with the difference between observed and calculated patterns shown at the bottom and the reflection positions shown as the vertical lines.
Other spectroscopic characterization data

**Figure S2** EDX spectrum of a typical PVP-coated BiOI nanoparticle

**Figure S3** X-ray powder diffraction patterns of the BiOI NPs
Figure S4 Particle size distribution of PVP-coated BiOI NPs in water dispersion

Figure S5 The FT-IR spectrum of PVP-coated BiOI NPs
Figure S6 The TGA curve of PVP-coated BiOI NP

Figure S7 Calibration curve of absorbance vs. I\textsuperscript{-} concentration
**Figure S8** Fluorescence emission spectrum of dye-conjugated BiOI NPs.