

Supporting Information

***tert*-Butylzinc Hydroxide as Efficient Predesigned Precursor of ZnO Nanoparticles**

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Experimental Section

General Remarks

All manipulations were conducted under a nitrogen atmosphere by using standard Schlenk techniques. Di-*tert*-butylzinc was prepared according the literature procedure.¹ All other reagents were purchased from commercial vendors. Solvents were dried and distilled prior to use. Redistilled water was degassed carefully by six freeze-pump-thaw cycles before use. NMR spectra were acquired on Varian Mercury 400 Spectrometer. IR spectra were obtained using a Perkin-Elmer System 2000 FT-IR spectrophotometer. Elemental analyses were performed on a Vario EL apparatus (Elementar Analysensysteme GmbH). Samples for PXRD measurements were prepared under anaerobic conditions in the glove box LabStar MB-10 Compact of MBraun (Garching, Germany).

X-Ray Crystallography

Data were collected using the 'oil drop technique' to mount crystals on a Nonius Kappa-CCD equipped with an Oxford Cryostream low-temperature device. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

Powder X-ray diffraction

Powder XRD data were collected on a Siemens D5005 diffractometer (Bruker AXS). Measurements employed Ni-filtered Cu K α radiation of a copper sealed tube charged with 40kV voltage and 40mA current and Bragg-Brentano geometry with beam divergence of 1 deg. in the scattering plane. The sample was spread in a glove-box in argon atmosphere over surface of a porous glass plate fixed to the sample holder. *In situ* measurements were done in deoxygenated and dried He using a stainless-steel camera with a steel sample holder containing an electric heater and a steel cap with a X-ray window sealed by a Capton tape. Diffraction patterns were measured in the range of 5-90 degrees of scattering angle by step scanning with step of 0.02 degree. The heating rate between the acquisition steps of XRD patterns was 2K \cdot min⁻¹.

Transmission Electron Microscopy

Microscopic samples were prepared by droplet coating of acetone suspensions on Ni grids (Agar Scientific, holey carbon film, 300 mesh) and examined using a Cs corrected scanning transmission electron microscope (STEM HITACHI HD2700) at nominal magnifications between x1300000 and x5000000. The electron optical parameters were C S = 0.6 mm, C C = 1.2 mm, electron energy spread = 1.5 eV.

From these images, first indications of particle structure were obtained. Particle size distributions were calculated by counting the diameters of 100 particles in the lower magnification images, defining size intervals of 0.25 nm between $d_{\min} \leq d \leq d_{\max}$ and counting the number of particles falling into these intervals.

Thermogravimetry

TG experiments were performed in argon with a heating rate of 2°C min⁻¹ using a TA Instruments Q600 apparatus.

X-Ray Crystallography

Crystal data for **1**, $C_{24}H_{60}O_6Zn_6$: $M = 837.06$, crystal dimensions $0.42 \times 0.34 \times 0.10 \text{ mm}^3$, monoclinic, space group $P 2_1/c$ (no. 14), $a = 9.9031(3) \text{ \AA}$, $b = 10.5542(5) \text{ \AA}$, $c = 18.1019(7) \text{ \AA}$, $\beta = 108.814(2)^\circ$, $U = 1790.89(12) \text{ \AA}^3$, $Z = 2$, $F(000) = 864$, $D_c = 1.552 \text{ g m}^{-3}$, $T = 100(2) \text{ K}$, $\mu(\text{Mo-K}\alpha) = 3.983 \text{ mm}^{-1}$, Nonius Kappa-CCD diffractometer, $\theta_{\text{max}} = 22.46^\circ$, 2315 unique reflections. The structure was solved by direct methods using the SHELXS97^[2] program and was refined by full matrix least-squares on F^2 using the program SHELXL97.^[3] All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydrogen atoms were introduced at geometrically idealized coordinates with a fixed isotropic displacement parameter equal to 1.2. Refinement converged at $R1 = 0.1032$, $wR2 = 0.1414$ for all data and 172 parameters ($R1 = 0.0606$, $wR2 = 0.1292$ for 1618 reflections with $I_o > 2\sigma(I_o)$). The goodness-of-fit on F^2 was equal 1.121. A weighting scheme $w = [\sigma^2(F_o^2 + (0.0418P)^2 + 3.1964P)]^{-1}$ where $P = (F_o^2 + 2F_c^2)/3$ was used in the final stage of refinement. The residual electron density = $+0.71 / -0.85 \text{ e\AA}^{-3}$. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-812506. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

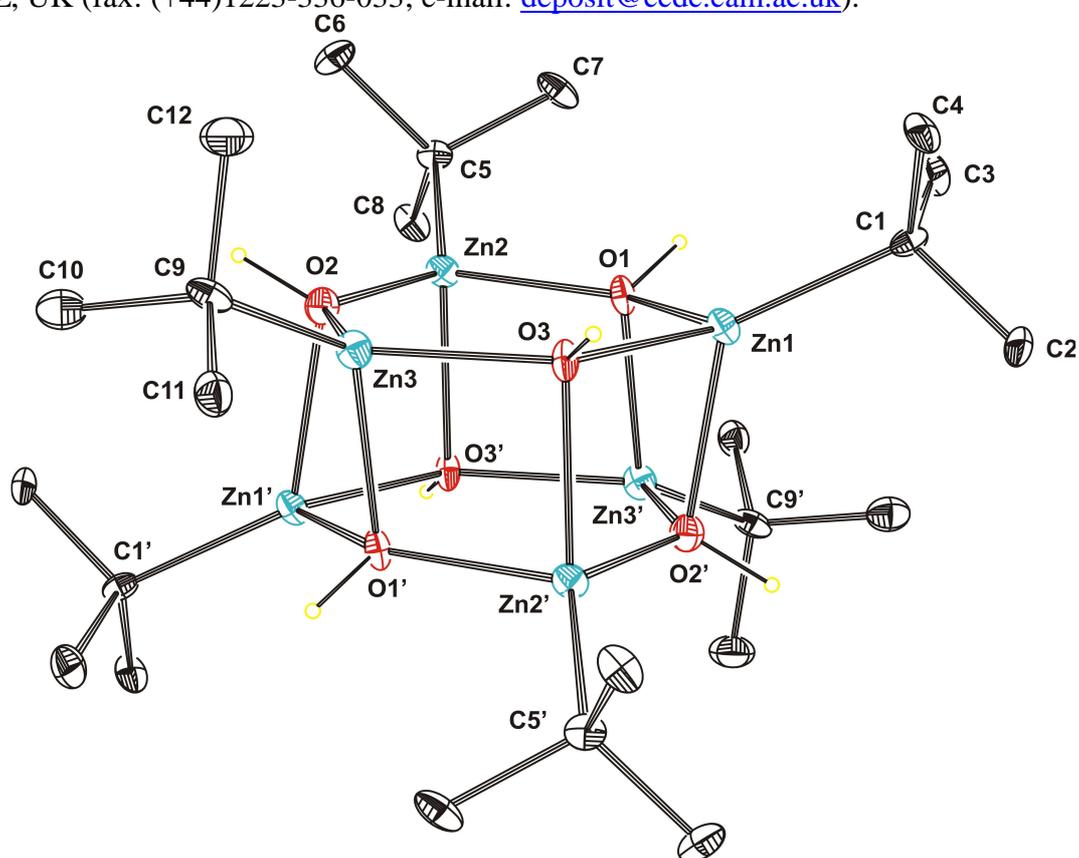


Figure S1. ORTEP diagram of molecular structure of **1** with thermal ellipsoids set at 40% probability; hydrogen atoms of *tert*-butyl groups have been omitted for clarity.

Table S1. Selected Bond Lengths (Å) and Angles (deg) for Compound 1.

Zn1 – C1	2.009(11)
Zn1 – O1	2.044(7)
Zn1 – O3	2.024(7)
Zn1 – O2'	2.183(8)
Zn2 – C5	2.004(11)
Zn2 – O2	2.019(8)
Zn2 – O3'	2.147(8)
Zn3 – C9	1.971(12)
Zn3 – O2	2.045(7)
Zn3 – O3	2.044(7)
Zn3 – O1'	2.113(8)
C1 – Zn1 – O1	117.8(4)
C1 – Zn1 – O2'	124.9(4)
C1 – Zn1 – O3	133.6(4)
O1 – Zn1 – O3	100.9(3)
O1 – Zn1 – O2'	82.5(3)
O3 – Zn1 – O2'	82.5(3)
C5 – Zn2 – O1	114.3(4)
C5 – Zn2 – O2	132.9(4)
C5 – Zn2 – O3'	126.2(4)
O1 – Zn2 – O2	103.9(3)
O1 – Zn2 – O3'	83.0(3)
O2 – Zn2 – O3'	83.5(3)
C9 – Zn3 – O2	119.9(4)
C9 – Zn3 – O3	126.1(4)
C9 – Zn3 – O1'	123.1(4)
O2 – Zn3 – O3	107.5(3)
O2 – Zn3 – O1'	84.2(3)
O3 – Zn3 – O1'	83.7(3)

Thermogravimetric analysis.

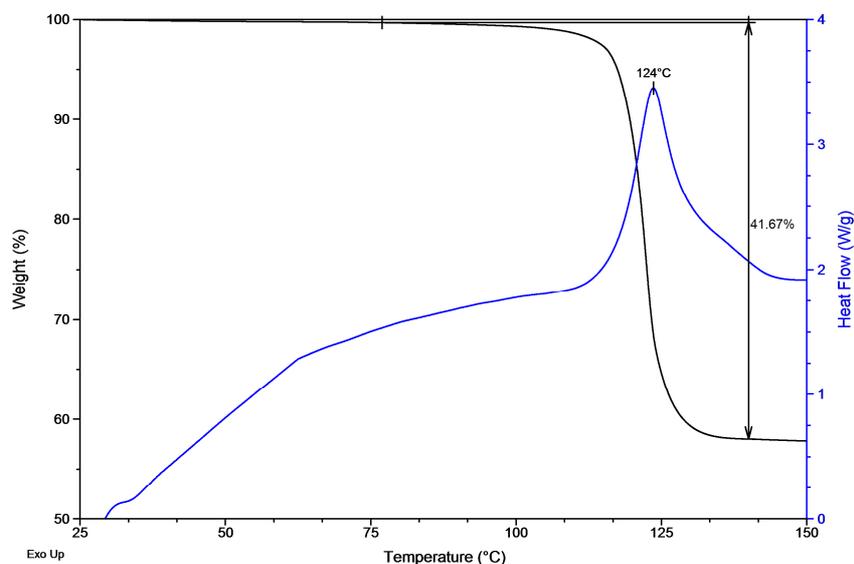


Figure S2. TGA and DSC profiles of the grinded sample of compound **1**.

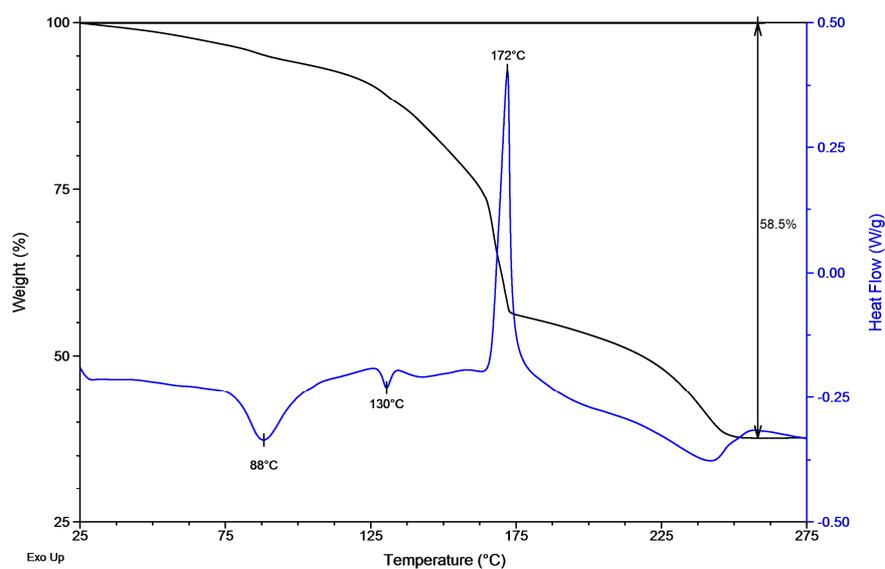


Figure S3. TGA and DSC profiles of the grinded sample of compound **2**.

[1] Lehmkuhl, H.; Olbrychs, O. *Liebigs Ann. Chem.* **1975**, 1162-1175.

[2] SHELXS-97, Program for Structure Solution": Sheldrick, G. M. *Acta Crystallogr. Sect. A* **1990**, 46, 467.

[3] Sheldrick, G. M. SHELXL-97, Program for Structure Refinement, Universität Göttingen, 1997.