Polyazamacrocycle ligands facilitate ^{89}Zr radiochemistry and yield ^{89}Zr complexes with remarkable stability.

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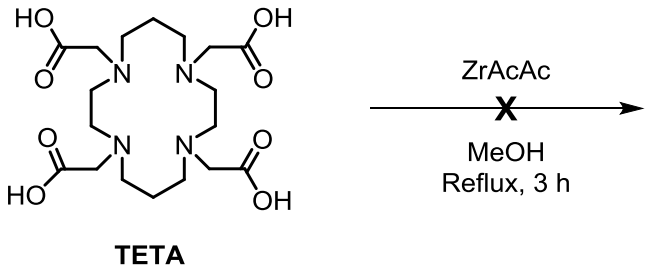
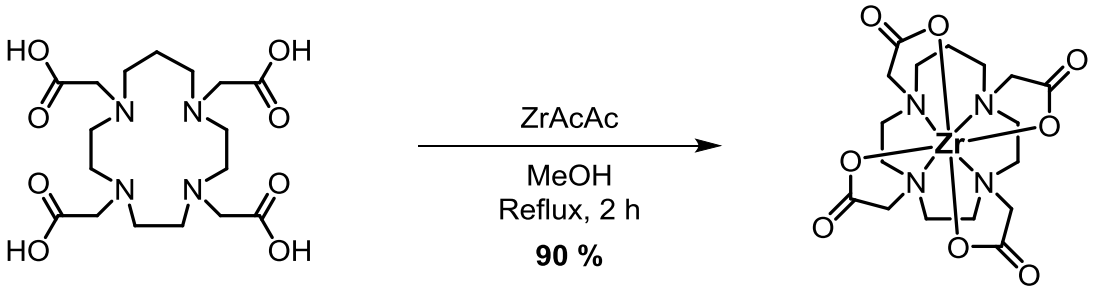


TETA

Scheme S1. Synthesis of zirconium complex of TETA



Scheme S2. Synthesis of zirconium complex of TRITA

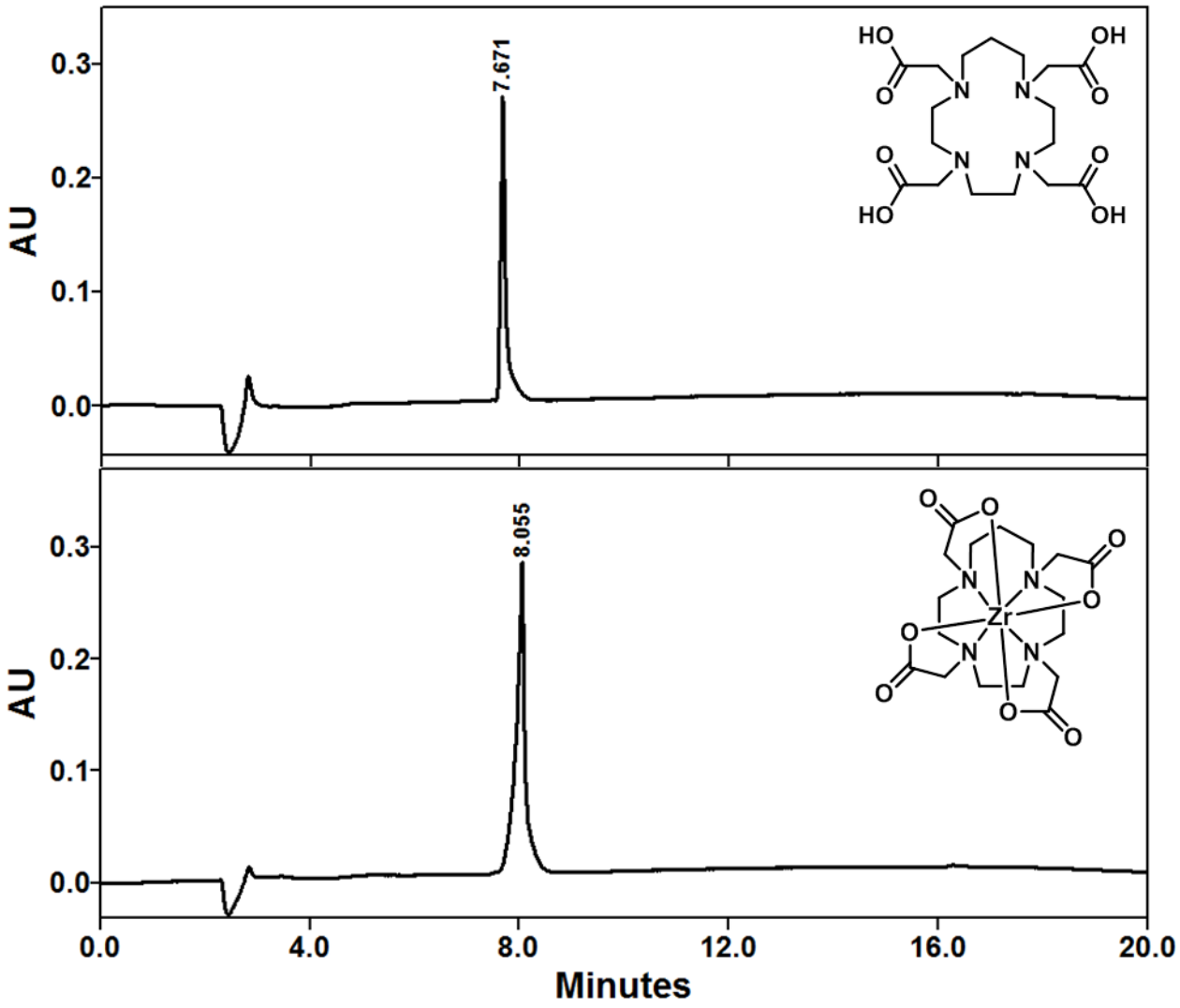


Figure S1. UV-HPLC chromatogram (201nm) of TRITA ligand (top) and nonradioactive ^{Nat}Zr-TRITA complex (bottom)

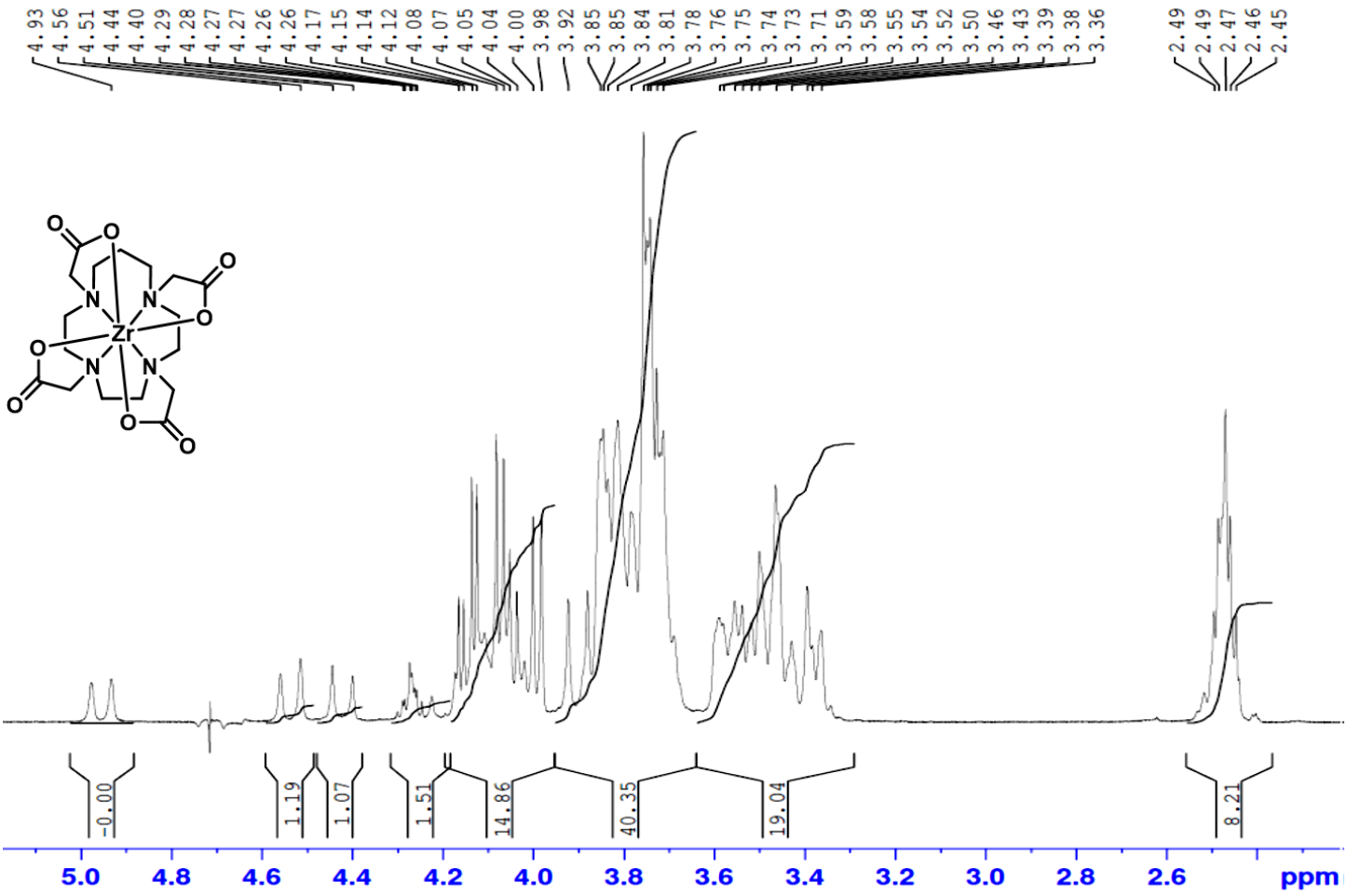


Figure S2. ^{1}H-NMR spectrum (D_2O-400 MHz) of the Zr-TRITA complex

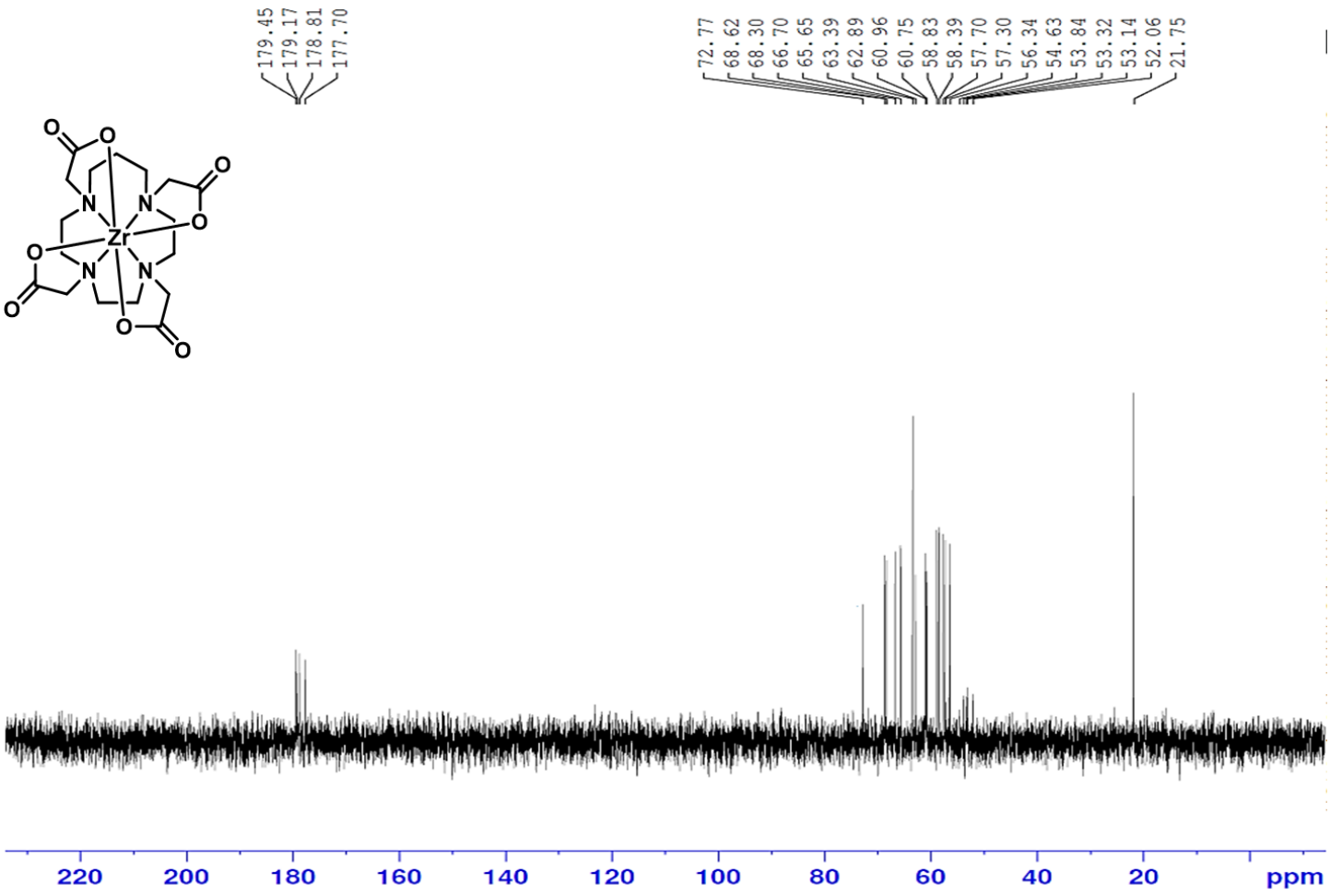


Figure S3. ^{13}C-NMR spectrum (D_2O-101 MHz) of the Zr-TRITA complex

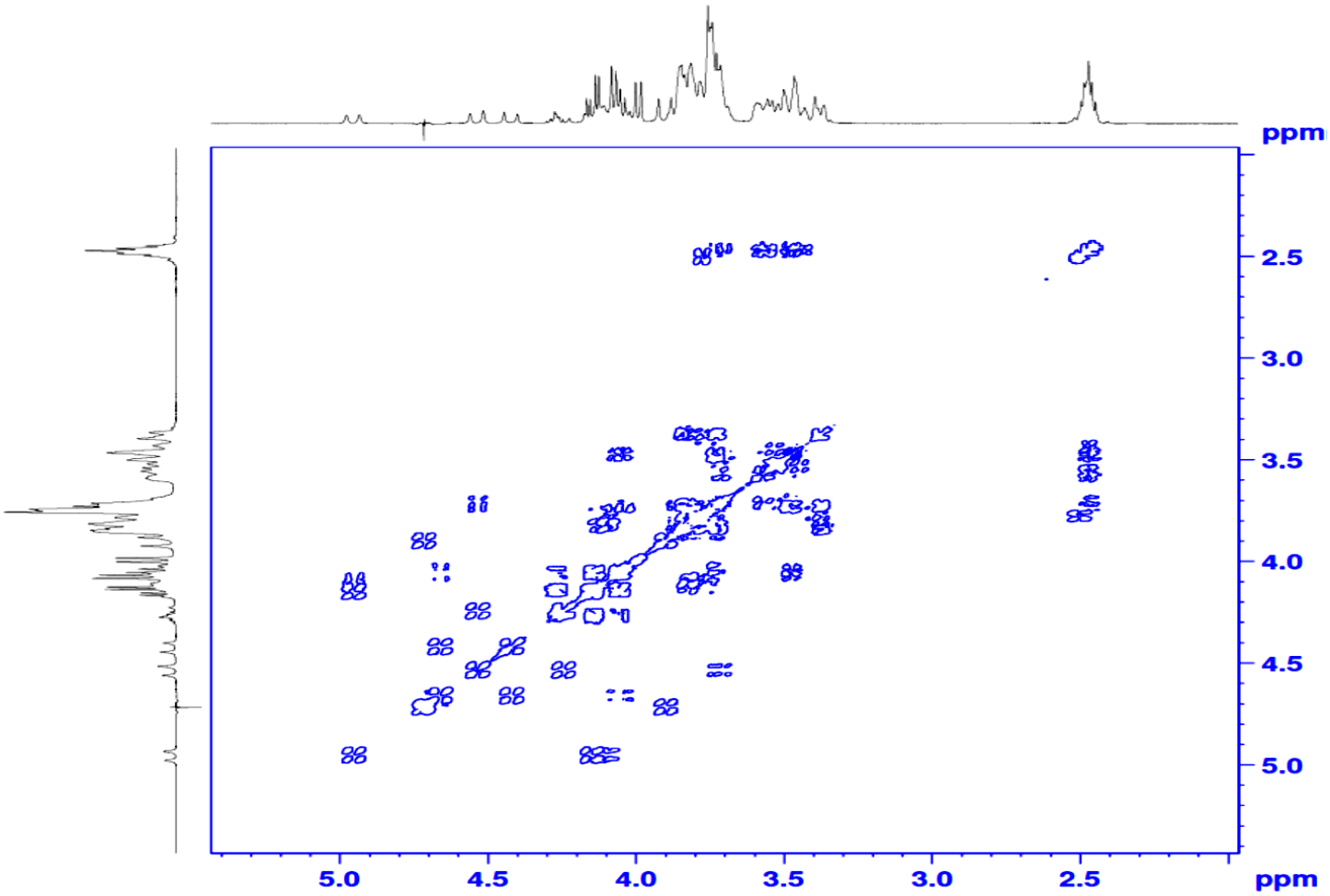


Figure S4. 2-D COSY NMR spectrum (D_2O-400 MHz) of the Zr-TRITA complex

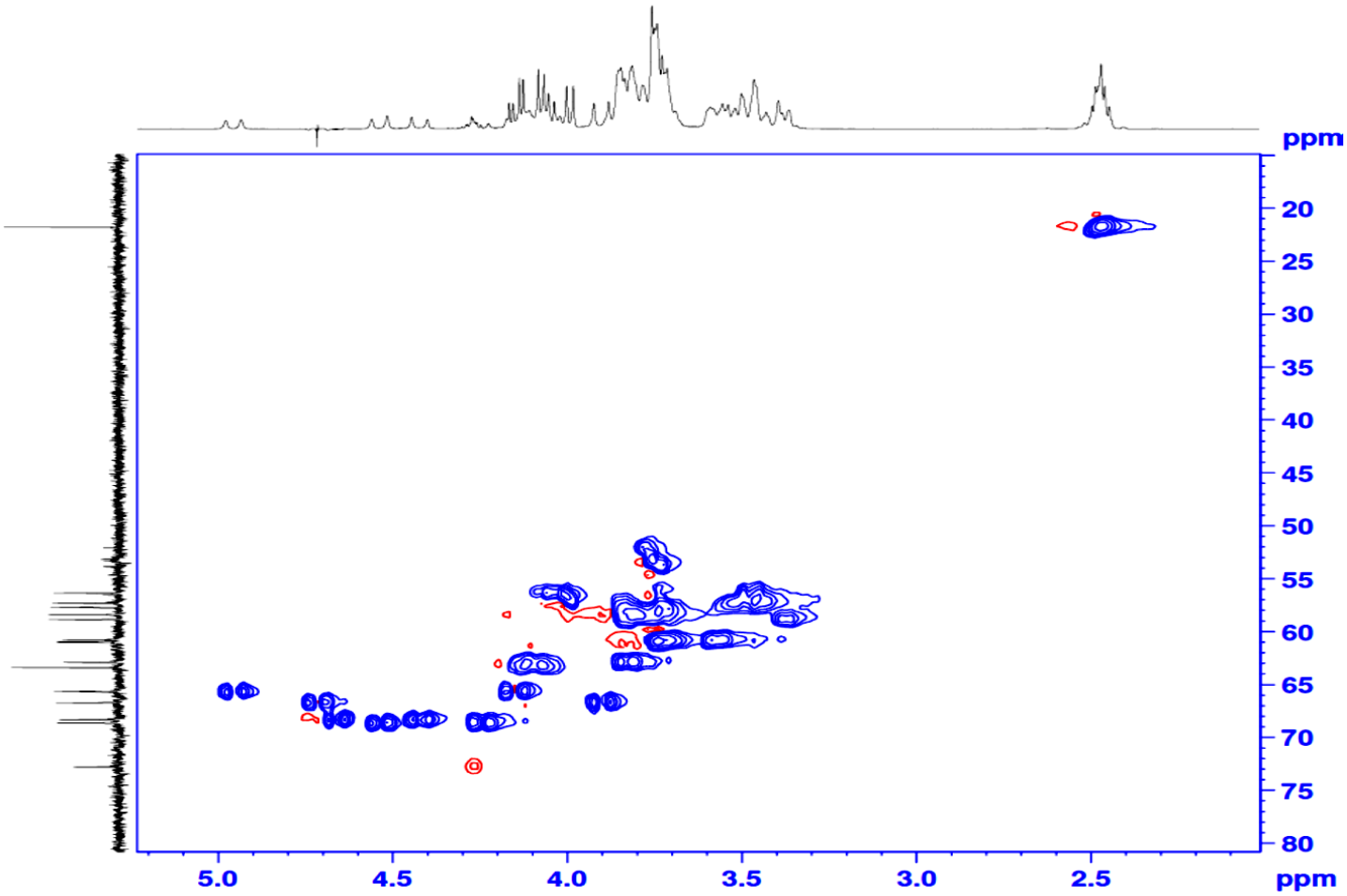


Figure S5. 2-D HSQC NMR spectrum (D_2O-400 MHz) of the Zr-TRITA complex

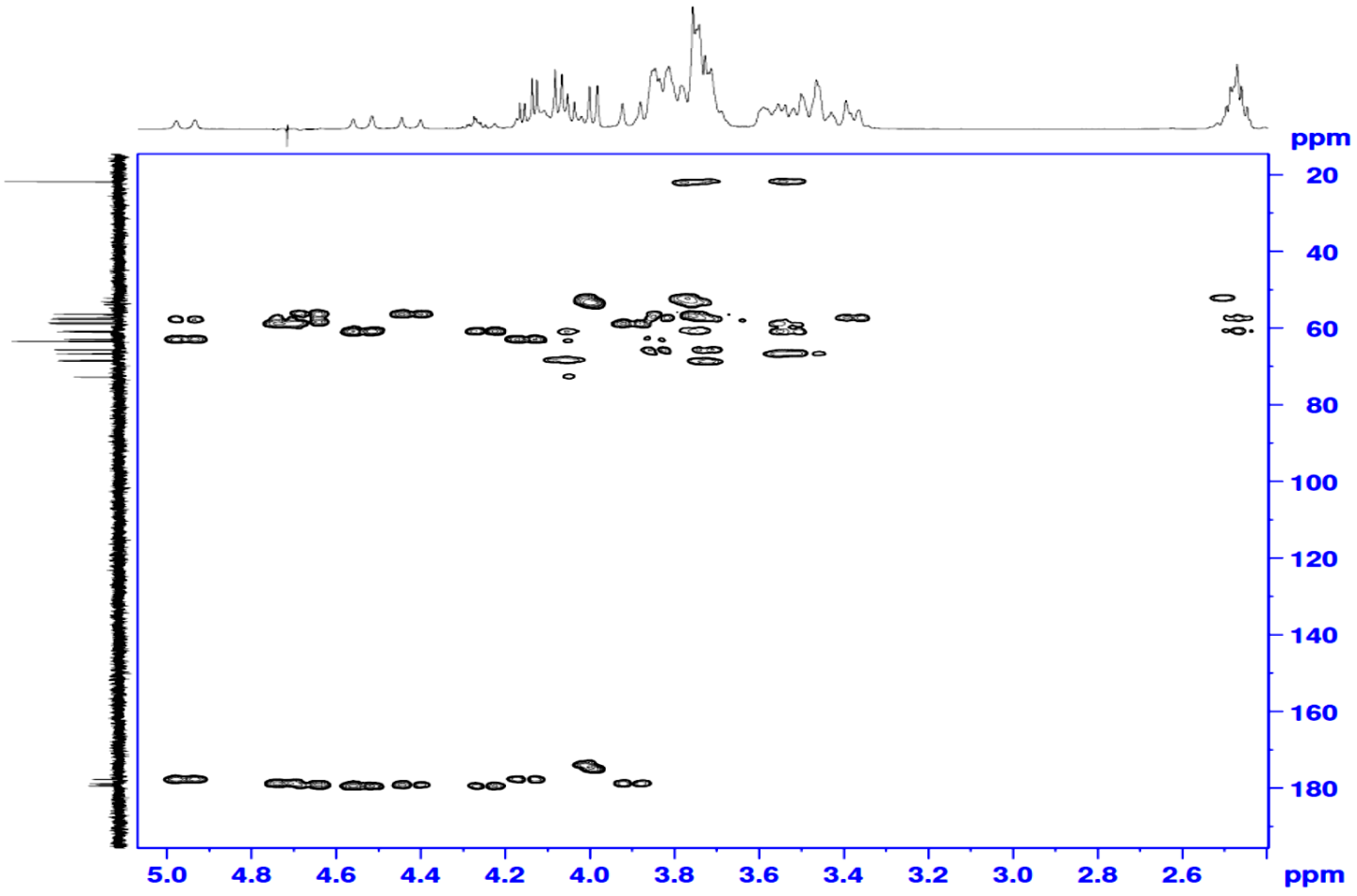


Figure S6. 2-D HMBC NMR spectrum (D_2O-400 MHz) of the Zr-TRITA complex

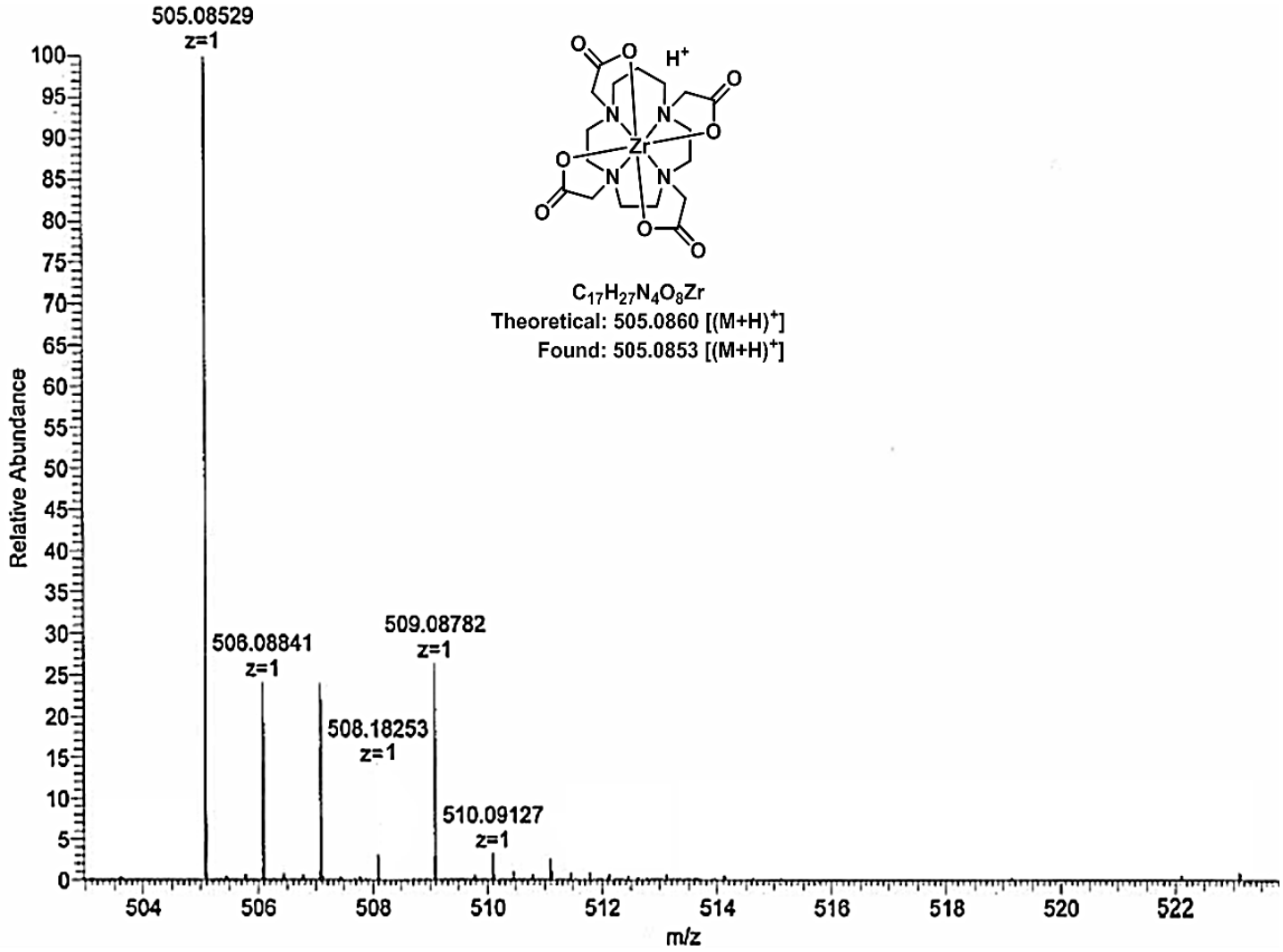
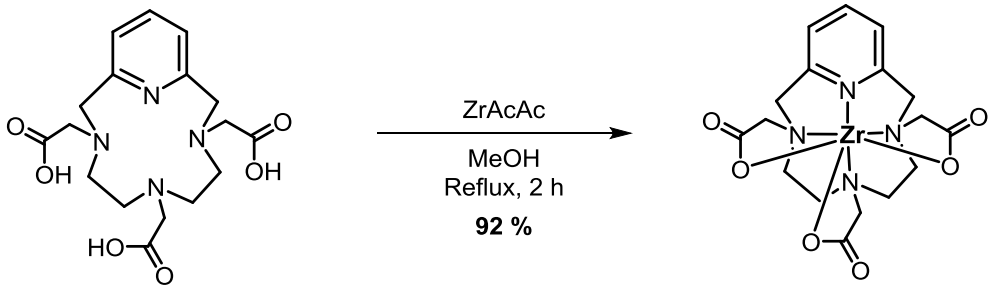


Figure S7. HR ESI FT-ICR MS (Positive mode) analysis of the Zr-TRITA complex



Scheme S3. Synthesis of zirconium complex of PCTA

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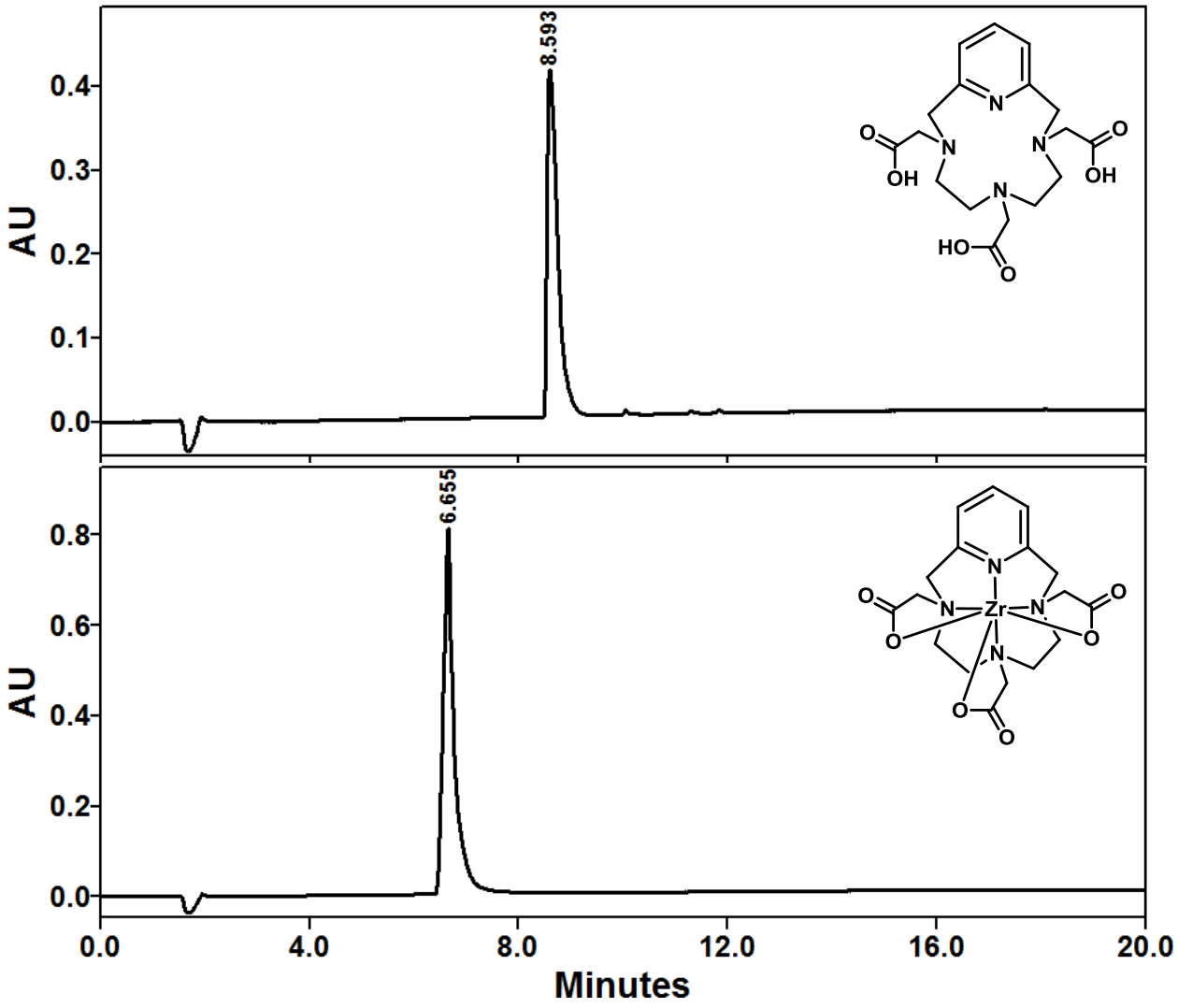

Figure S8. UV-HPLC chromatogram (201nm) of PCTA ligand (top) and nonradioactive ^{Nat}Zr-PCTA complex (bottom)

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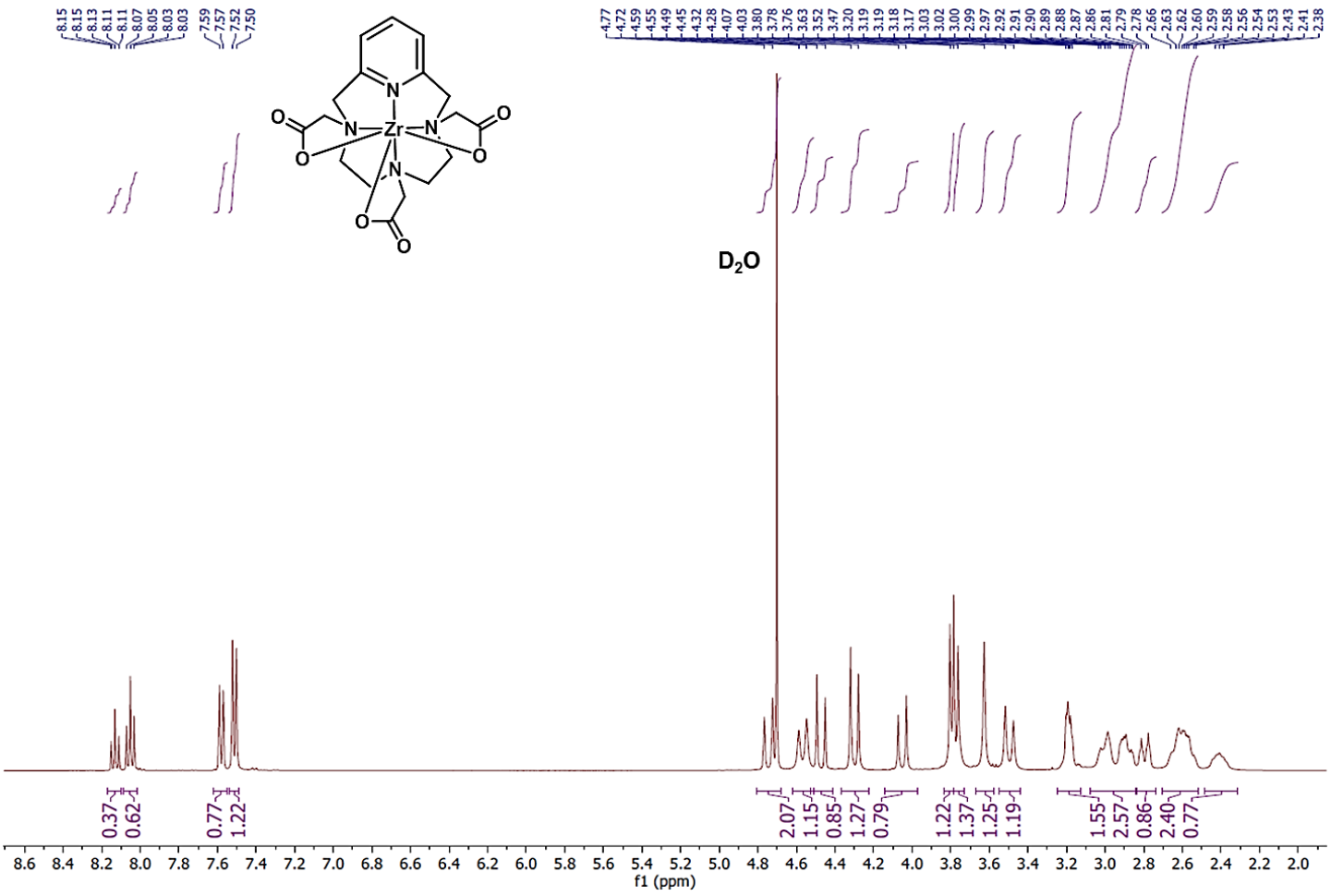


Figure S9. ^{1}H-NMR spectrum (D_2O-400 MHz) of the Zr-PCTA complex

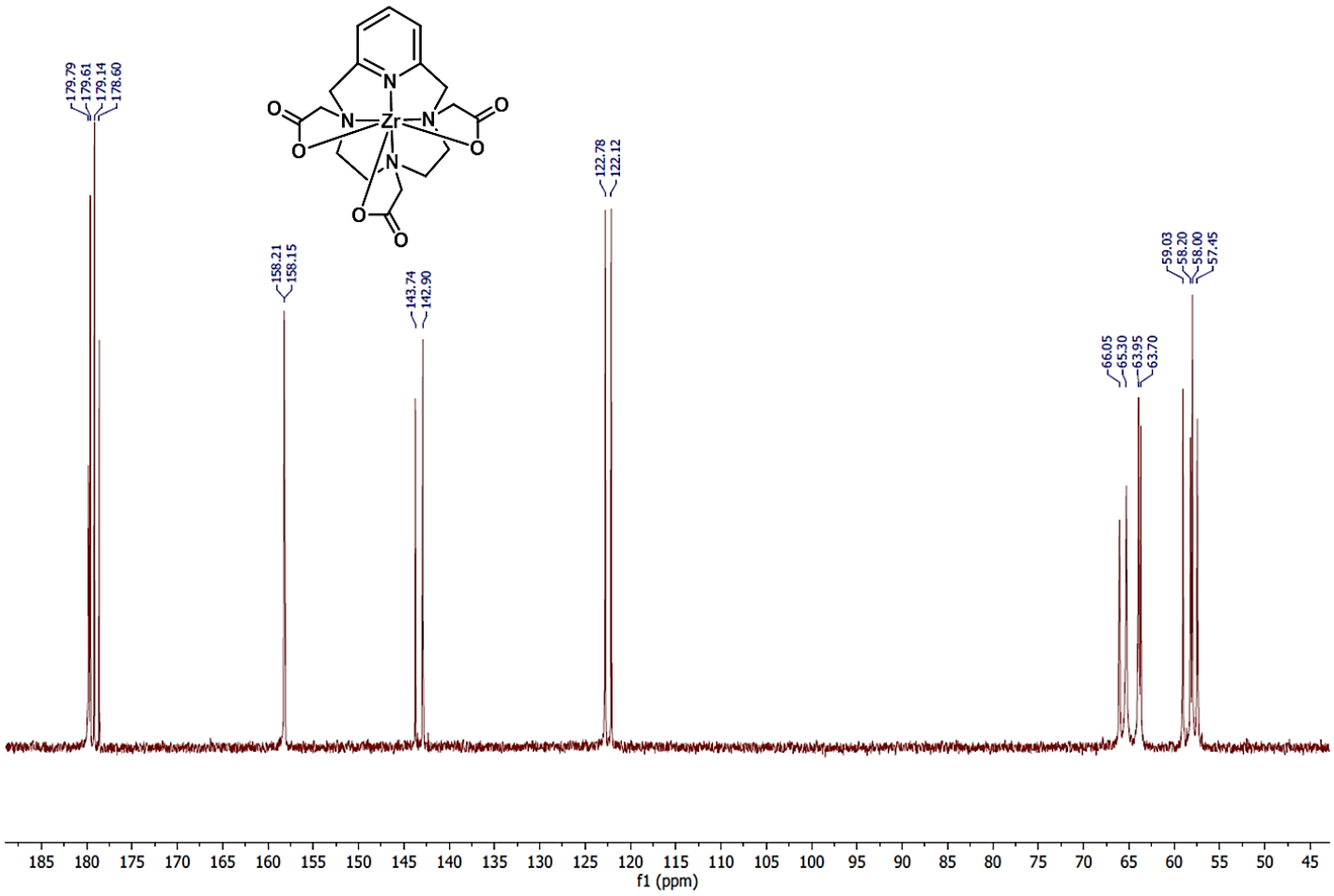


Figure S10. ^{13}C-NMR spectrum (D_2O-101 MHz) of the Zr-PCTA complex

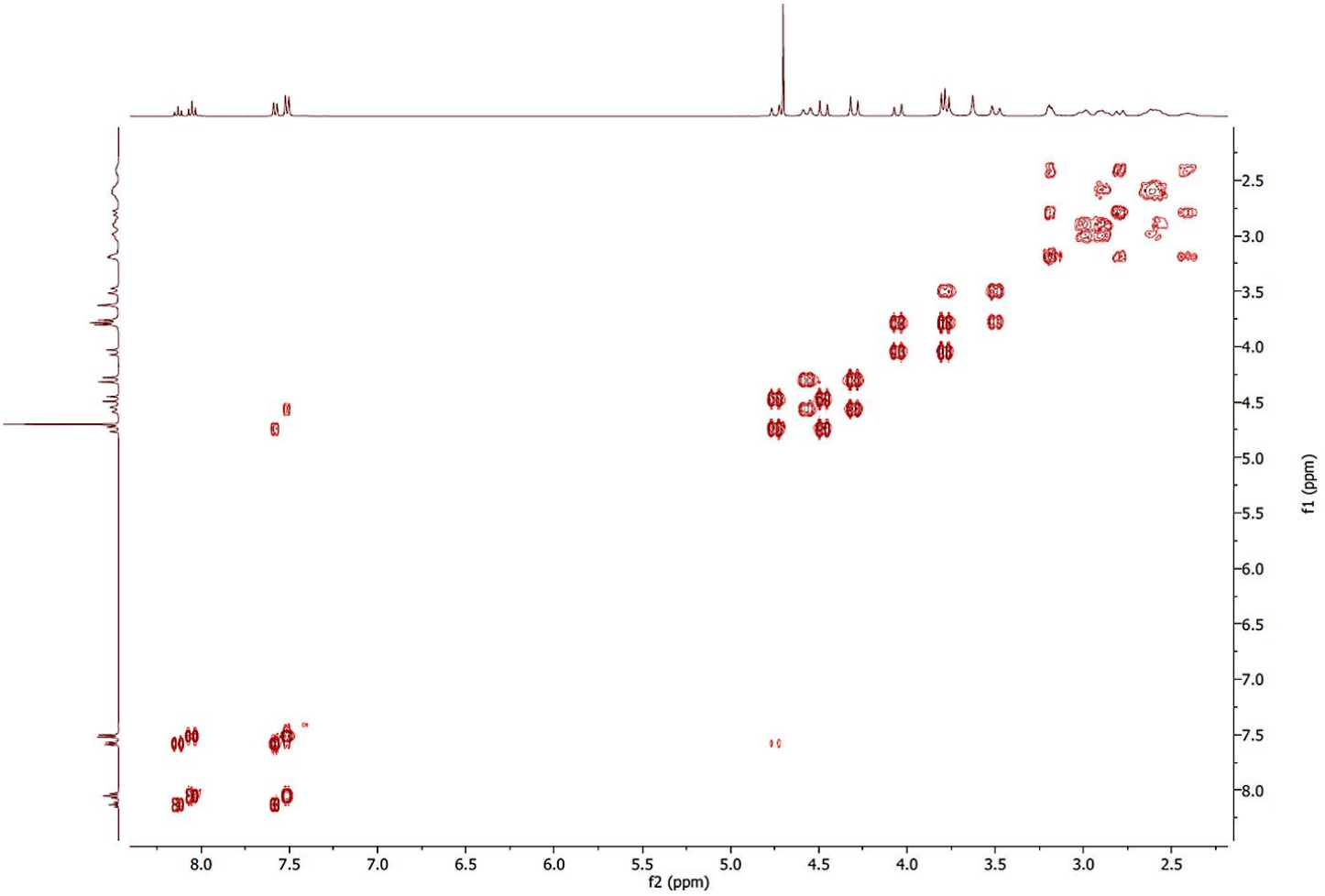


Figure S11. 2-D COSY NMR spectrum (D_2O-400 MHz) of the Zr-PCTA complex

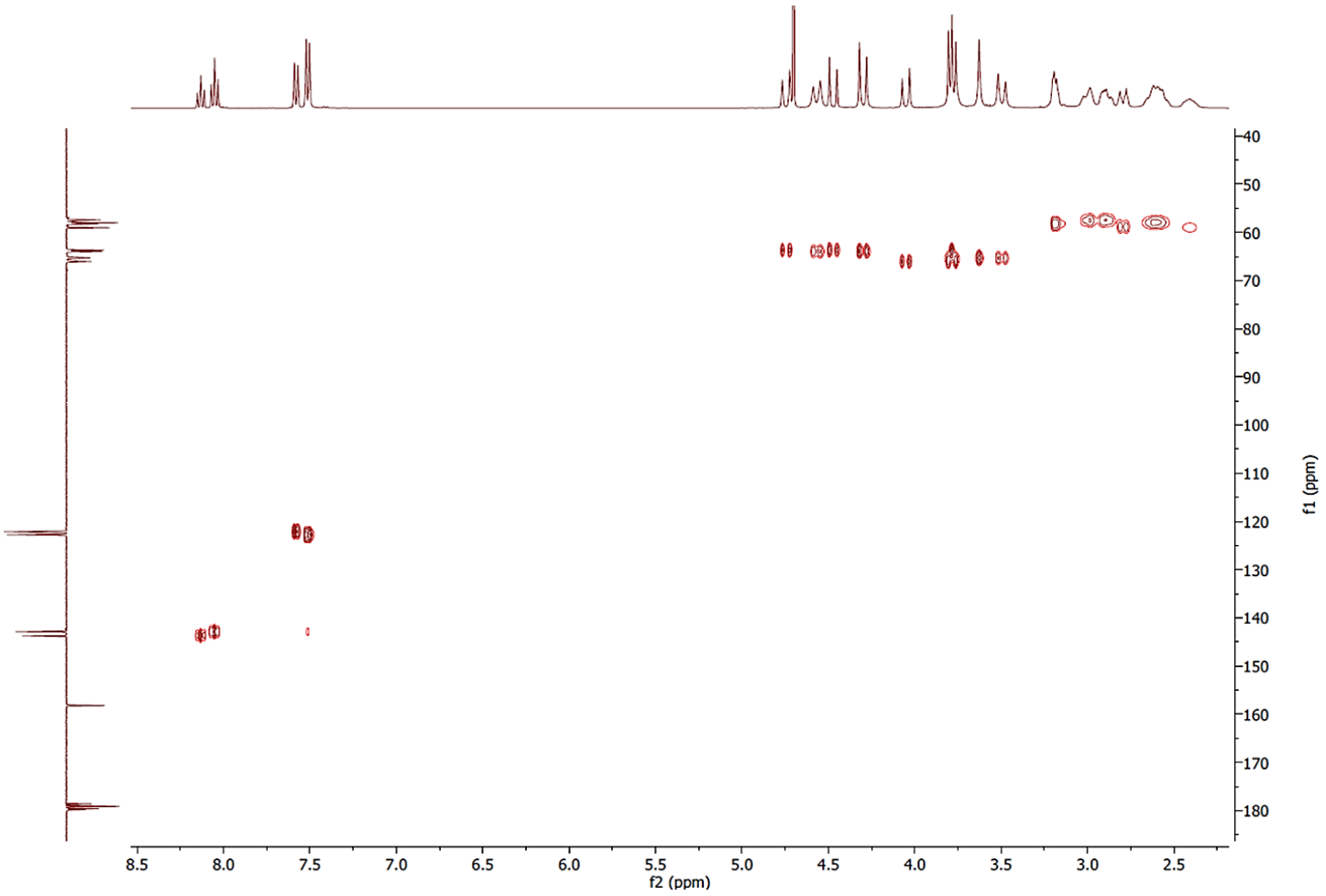


Figure S12. 2-D HSQC NMR spectrum (D_2O-400 MHz) of the Zr-PCTA complex

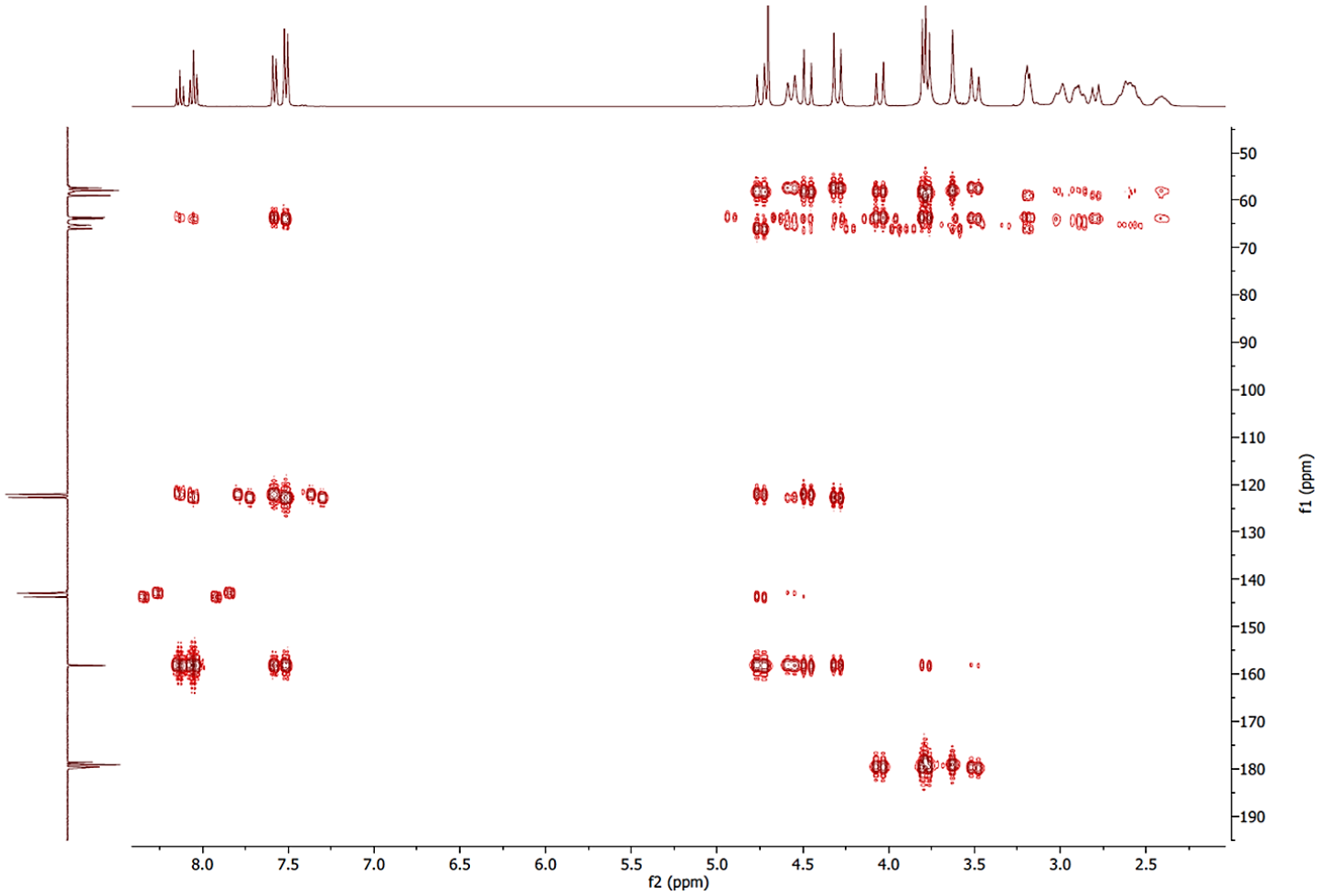


Figure S13. 2-D HMBC NMR spectrum (D_2O-400 MHz) of the Zr-PCTA complex

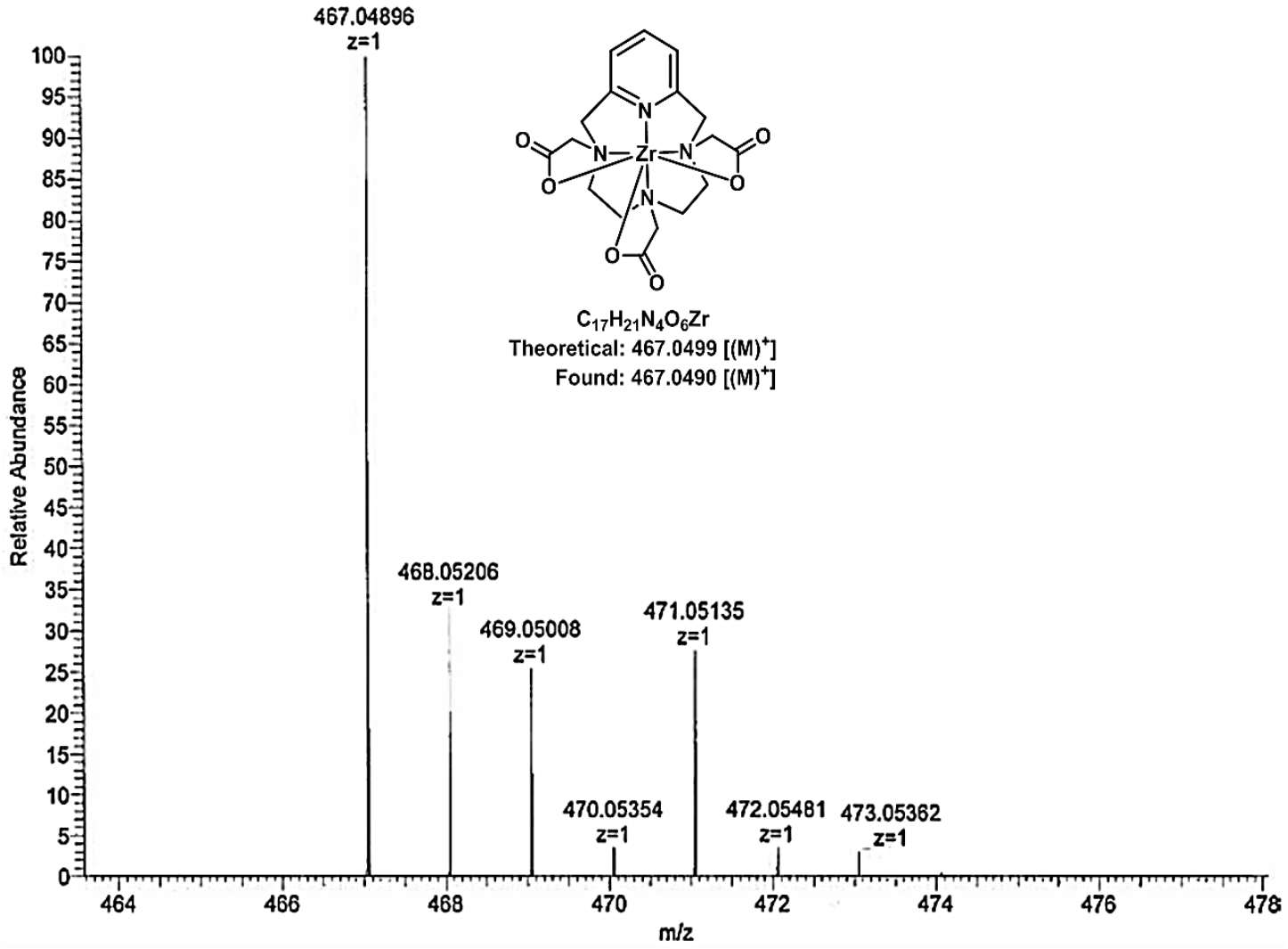
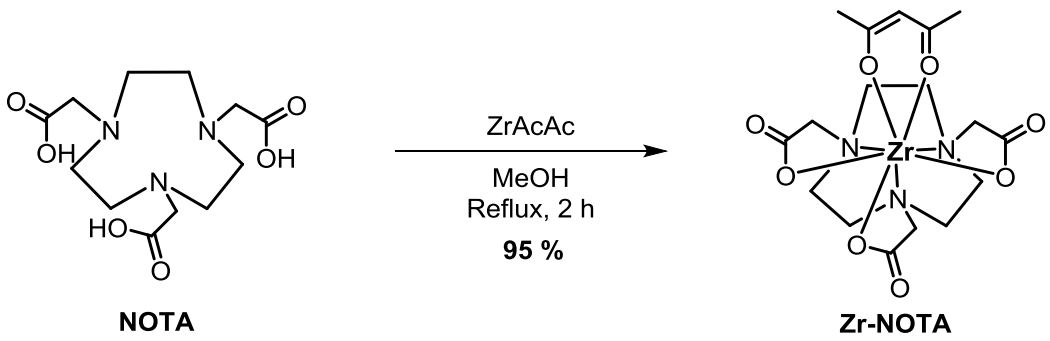


Figure S14. HR ESI FT-ICR MS (Positive mode) analysis of the Zr-PCTA complex



Scheme S4. Synthesis of zirconium complex of NOTA

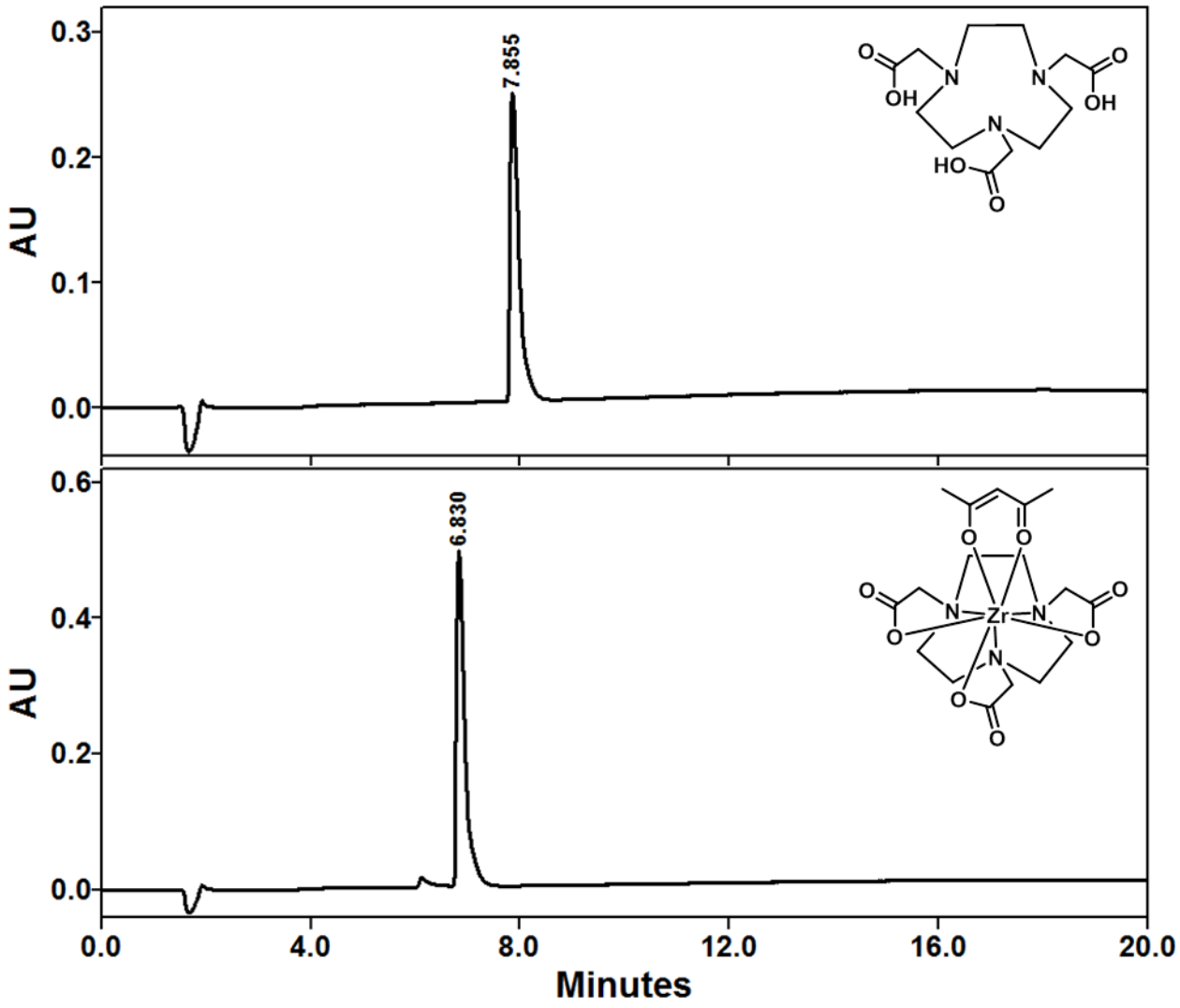


Figure S15. UV-HPLC chromatogram (201nm) of NOTA ligand (top) and nonradioactive ^{Nat}Zr-NOTA-AcAc complex (bottom)

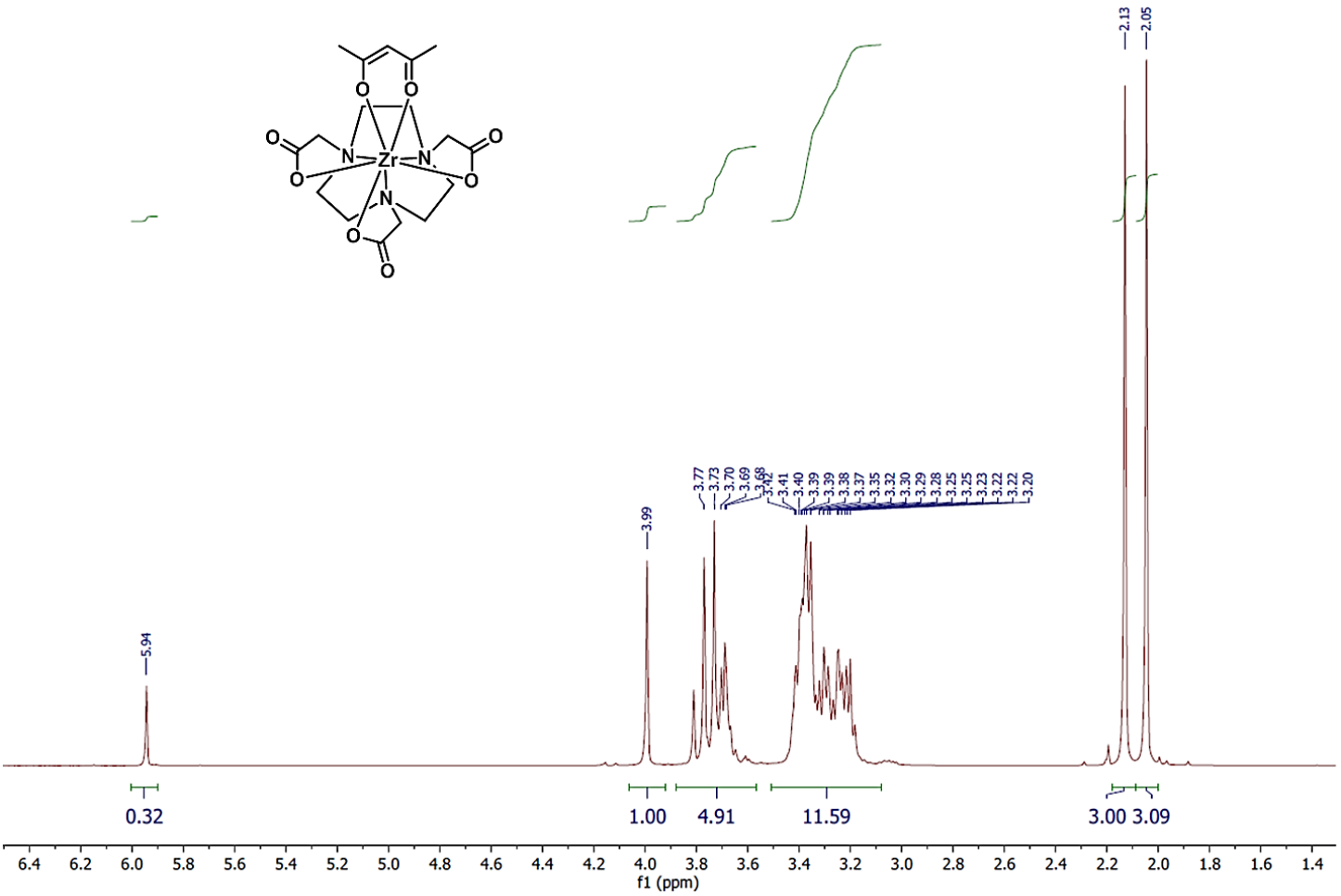


Figure S16. ^{1}H-NMR spectrum (D_2O-400 MHz) of the Zr-NOTA-AcAc complex

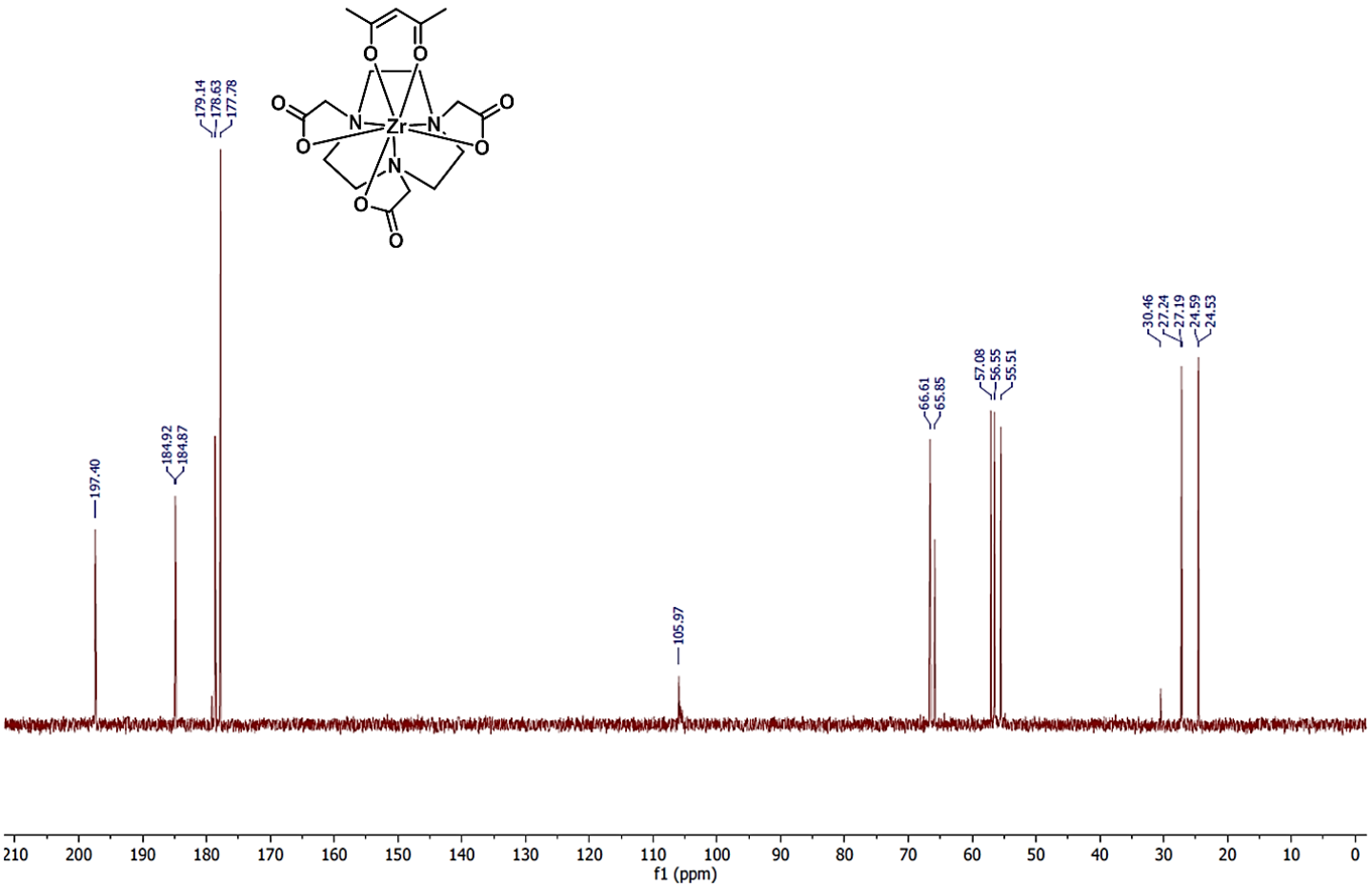


Figure S17. ^{13}C-NMR spectrum (D_2O-101 MHz) of the Zr-NOTA-AcAc complex

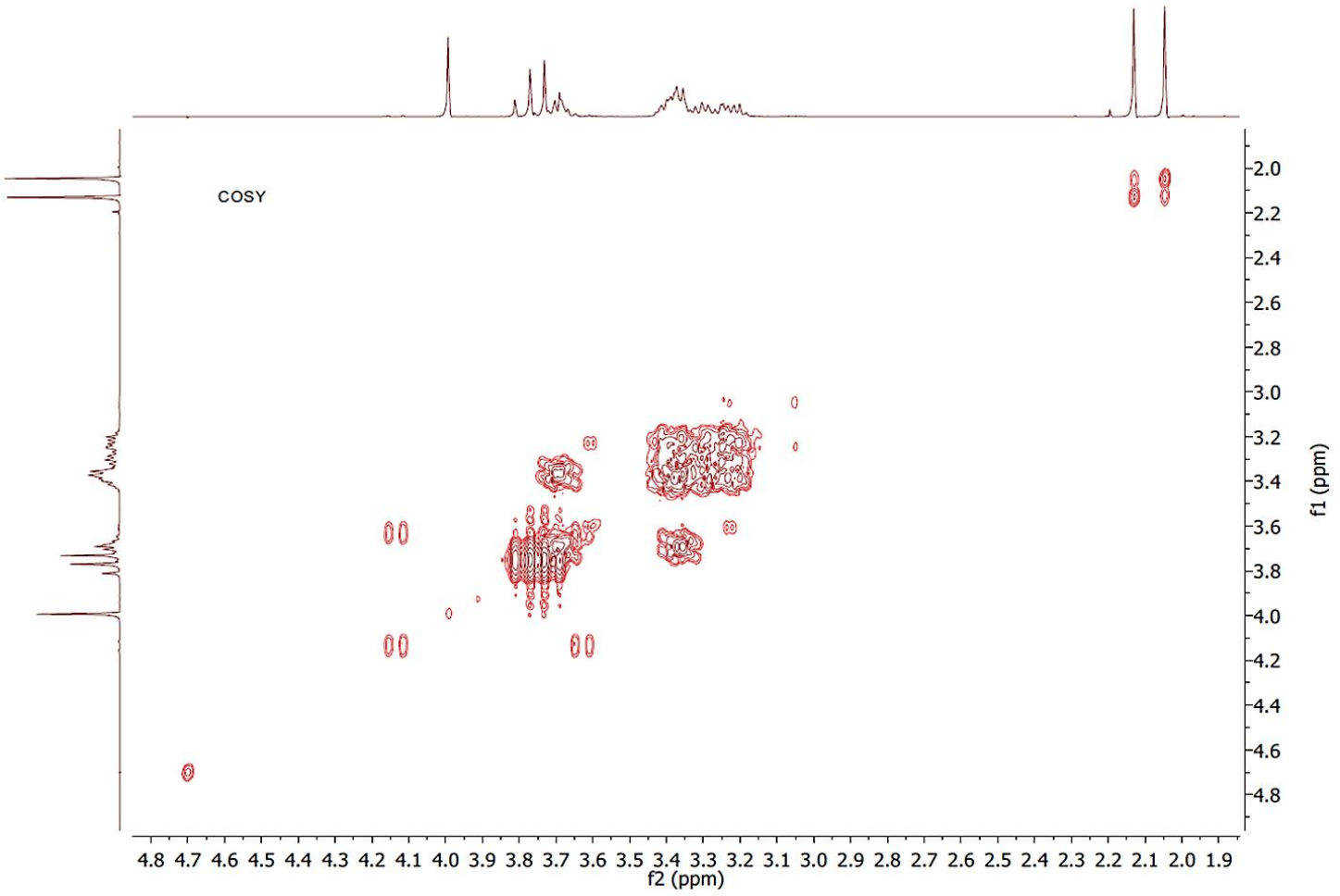


Figure S18. 2-D COSY NMR spectrum (D_2O-400 MHz) of the Zr-NOTA-AcAc complex

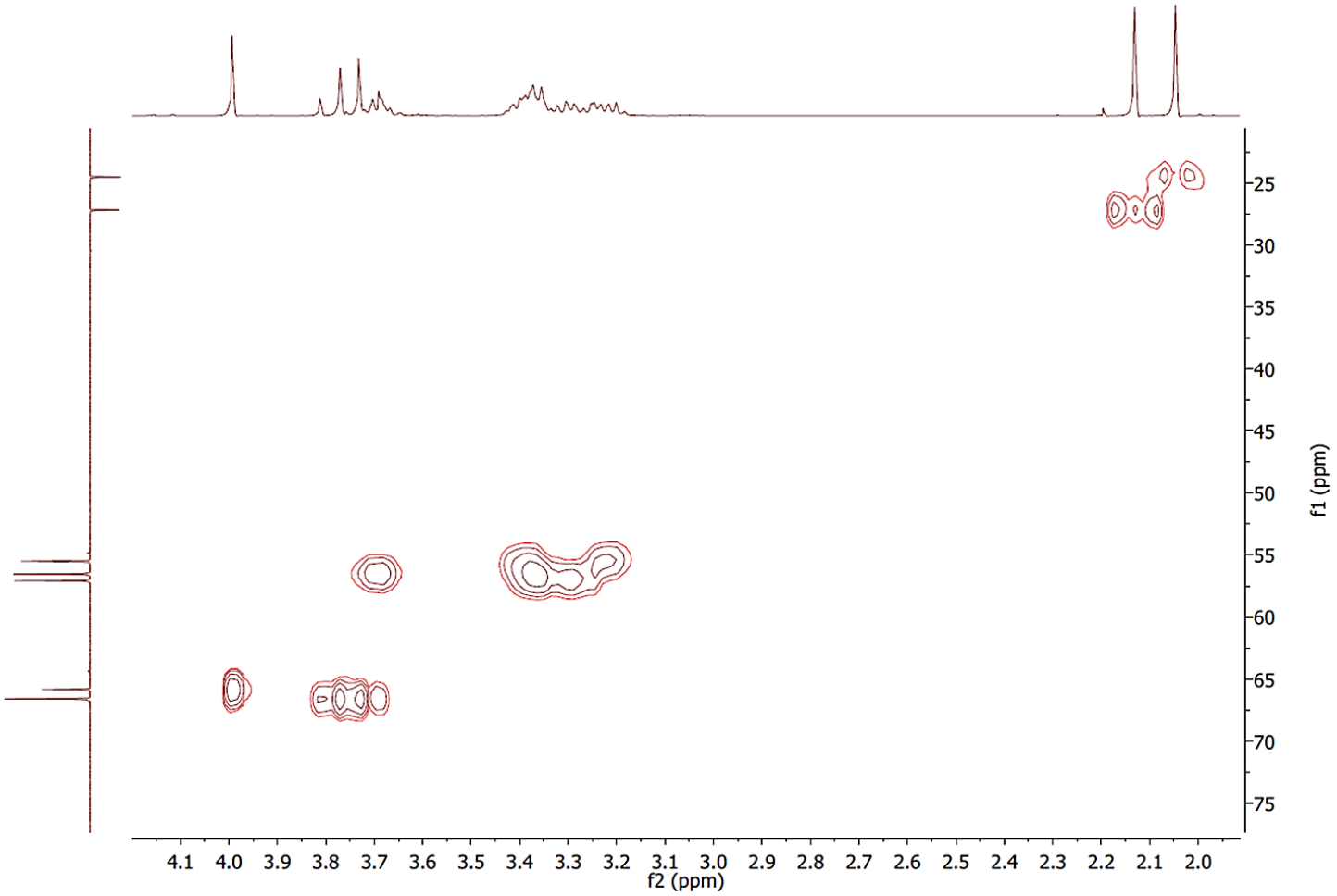


Figure S19. 2-D HMQC NMR spectrum (D_2O-400 MHz) of the Zr-NOTA-AcAc complex

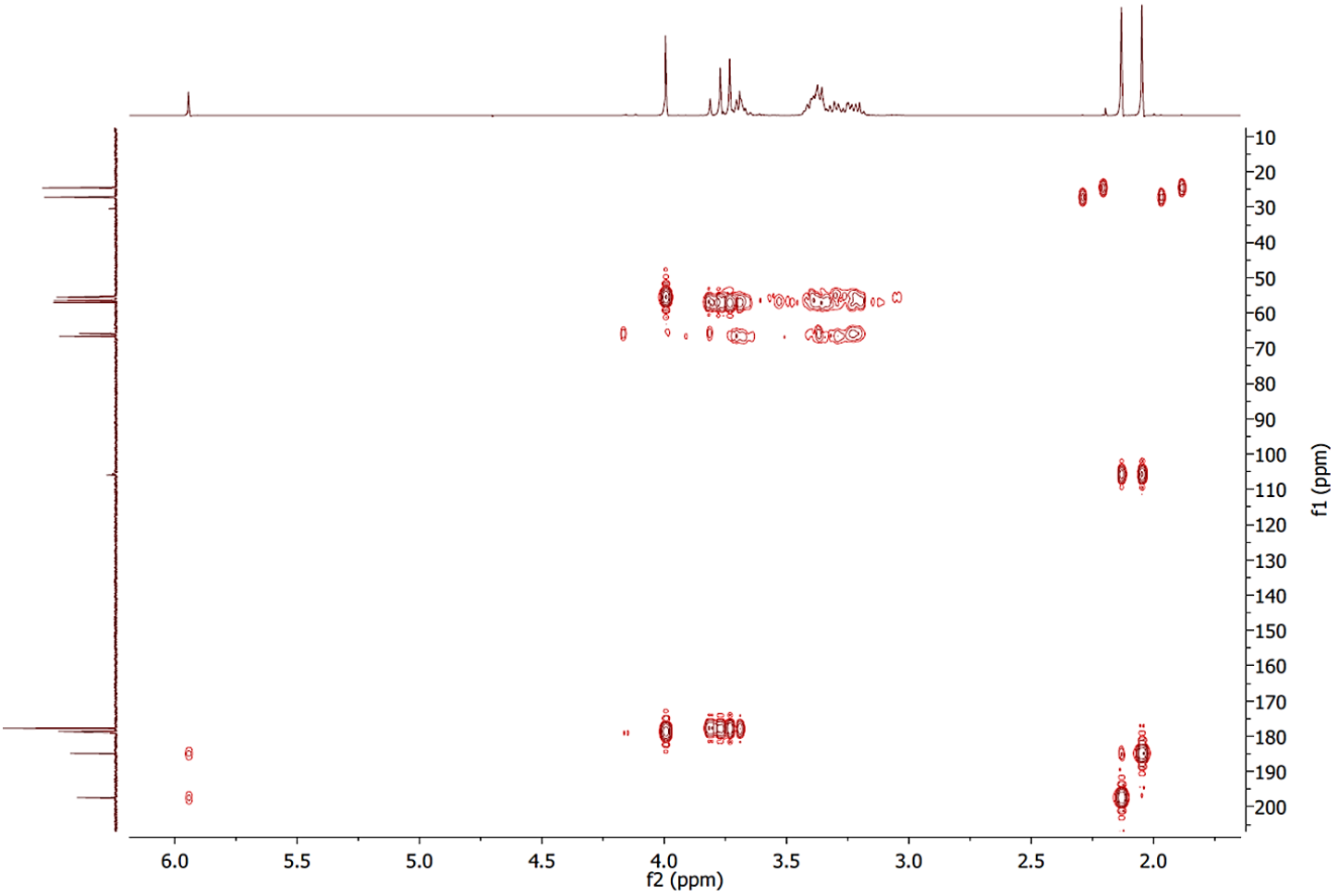


Figure S20. 2-D HMBC NMR spectrum (D_2O-400 MHz) of the Zr-NOTA-AcAc complex

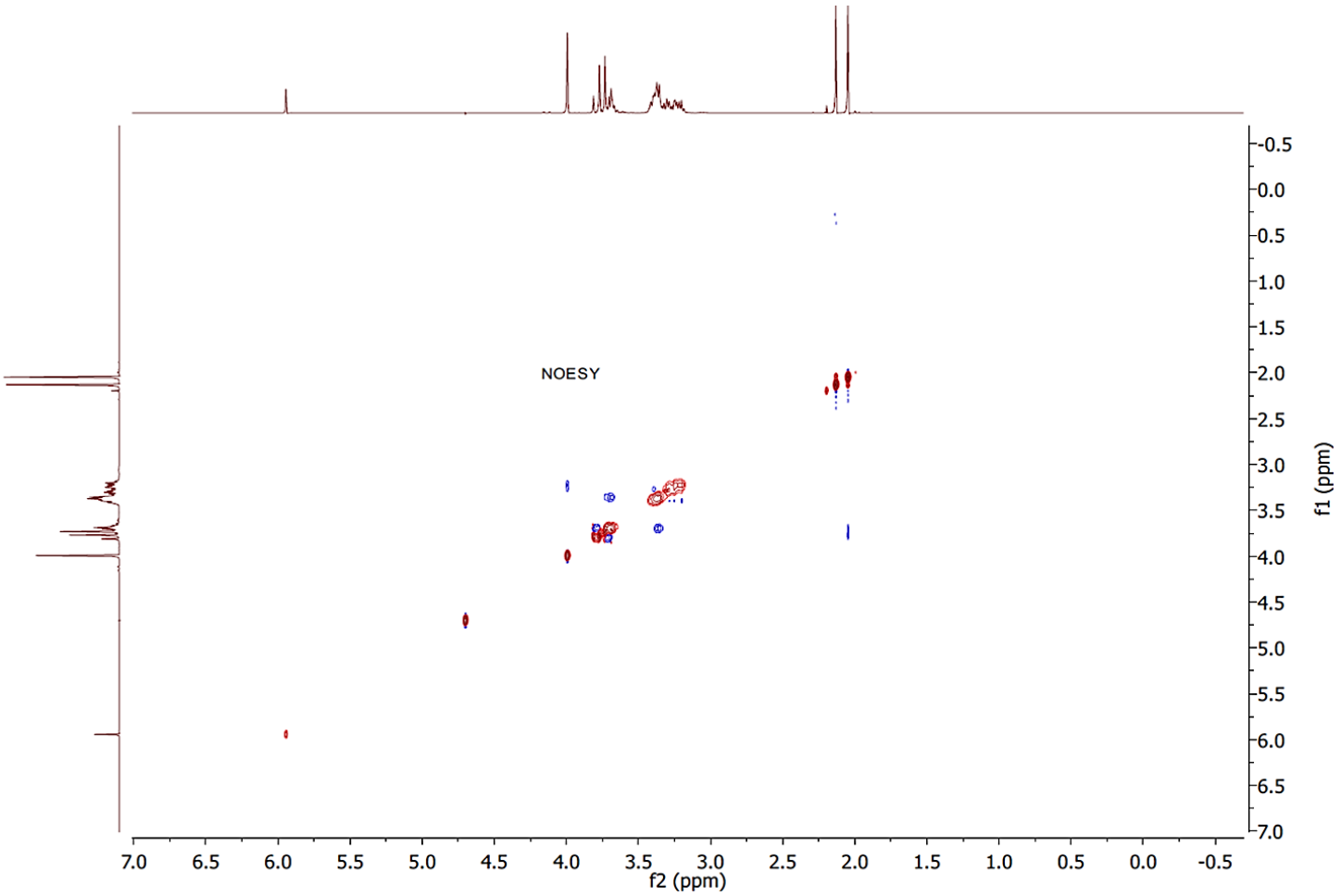


Figure S21. 2-D NOESY NMR spectrum (D_2O-400 MHz) of the Zr-NOTA-AcAc complex

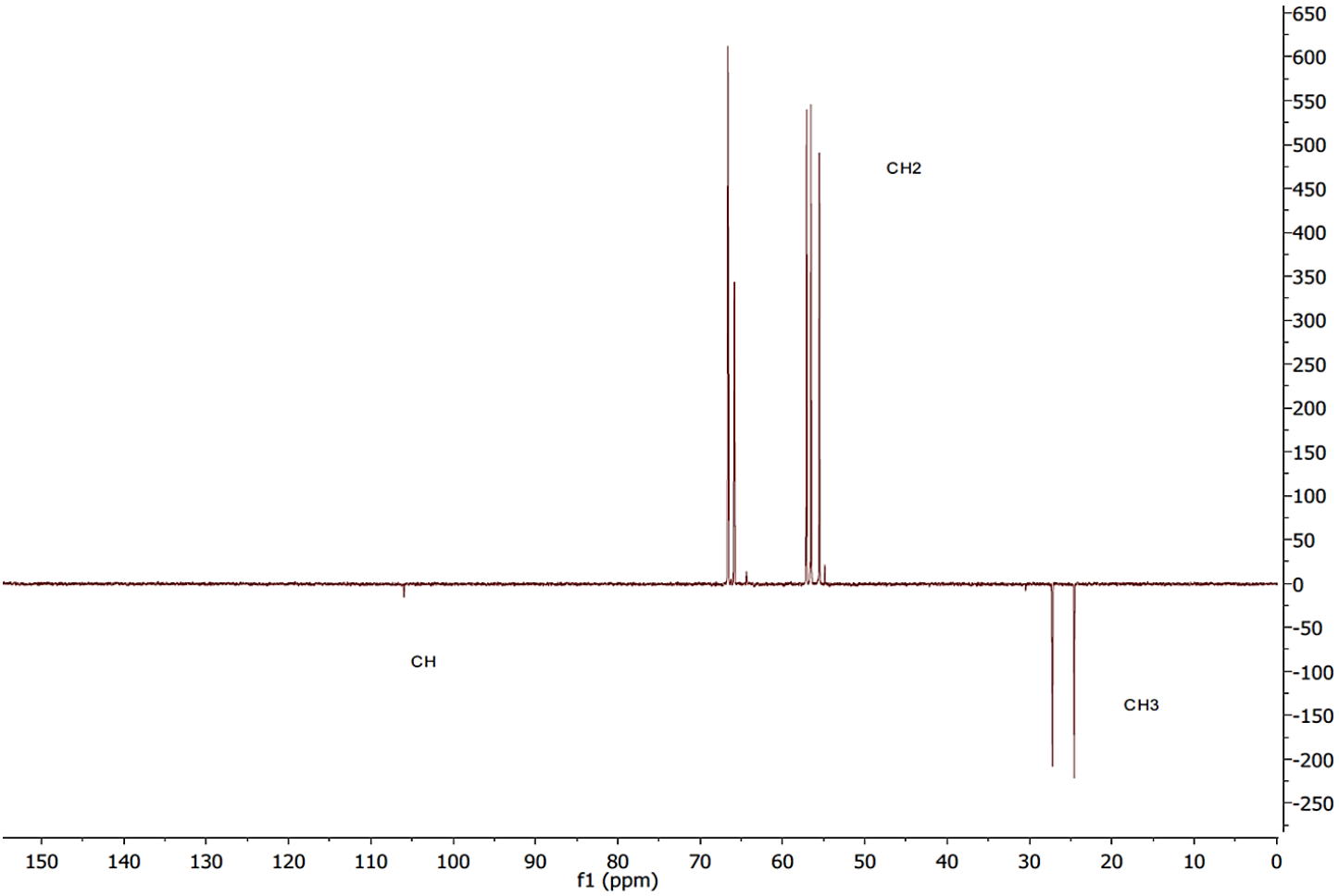


Figure S22. 2-D ^{13}C APT NMR spectrum (D_2O-101 MHz) of the Zr-NOTA-AcAc complex

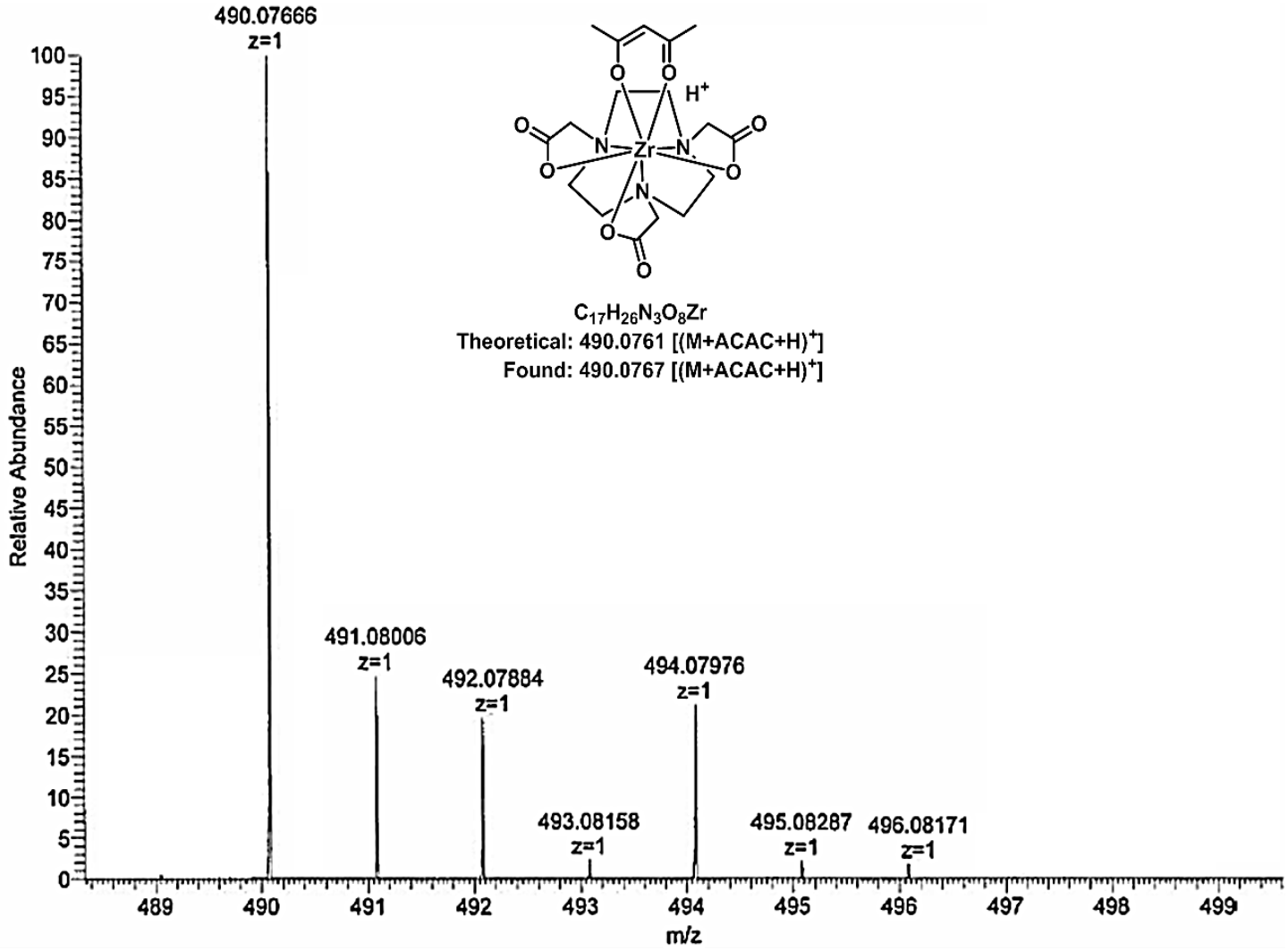


Figure S23. HR ESI FT-ICR MS (Positive mode) analysis of the Zr-NOTA-AcAc complex

Crystal Structure Analysis of Zr-TRITA, [Zr($C_{17}H_{26}N_4O_8$)] – 3.33 H_2O

Crystallographic Experimental Details:

Data Collection and Structure Solution. A clear colourless needle-like specimen of [Zr($C_{17}H_{26}N_4O_8$)] – 3.33 H_2O, approximate dimensions 0.010 mm x 0.040 mm x 0.470 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker APEX CCD system equipped with a graphite monochromator and a MoK_α sealed x-ray tube (λ = 0.71073 Å). X-rays were provided by a fine-focus sealed x-ray tube operated at 50kV and 30mA.

The total exposure time was 18.65 hours. The frames were integrated with the Bruker SAINT Software[1] package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 61502 reflections to a maximum θ angle of 30.14° (0.71 Å resolution), of which 19202 were independent (average redundancy 3.203, completeness = 99.4%, R_{int} = 5.90%, R_{sig} = 7.12%) and 16822 (87.61%) were greater than $2\sigma(F^2)$. The final cell constants of a = 19.2657 (17) Å, b = 6.9888 (6) Å, c = 49.787 (4) Å, β = 99.7028 (11)°, volume = 6607.6 (10) $Å^3$, are based upon the refinement of the XYZ-centroids of 9909 reflections above 20 σ(I) with 6.826° < 2θ < 55.22°. Data were corrected for absorption effects using the multi-scan method (SADABS)[2]. The ratio of minimum to maximum apparent transmission was 0.841. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.776 and 0.994.

The structure was solved and refined using the Bruker SHELXTL Software Package[3], using the space group Cc, with Z = 12 for the formula unit, [Zr($C_{17}H_{26}N_4O_8$)] - 3.33 H_2O (3 formula units/asymmetric unit). The hydrogen atoms of the “TRITA” ligand were included in the structural model as fixed atoms (using idealized sp^3-hybridized geometry and C-H bond lengths of 0.99Å) "riding" on their respective carbon atoms. The isotropic thermal parameters for all “TRITA” hydrogen atoms were fixed at values 1.2 times the equivalent isotropic thermal parameter of the carbon atom to which they are covalently bonded. The isotropic thermal parameters of the water hydrogen atoms were allowed to refine independently. The final anisotropic full-matrix least-squares refinement on F^2 with 981 variables converged at R_1 = 3.96%, for the observed data and wR_2 = 7.93% for all data. The goodness-of-fit was 0.996. The largest peak in the final difference electron density synthesis was 0.550 $e^-/Å^3$ and the largest hole was -0.618 $e^-/Å^3$ with an RMS deviation of 0.078 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.706 g/cm^3 and F(000), 3520 e^-.

Refinement Details. Crystal data, data collection and structure refinement details are summarized in tables below.

Computing details. Data collection: Bruker *APEX3* v2016.1-0; cell refinement: Bruker *APEX3* v2016.1-0; data reduction: Bruker *APEX2* v2014.11-0; program(s) used to solve structure: *SHELXS97* (Sheldrick 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2014); molecular graphics: Bruker *APEX2* v2014.11-0; software used to prepare material for publication: Bruker *APEX2* v2014.11-0[1-6].

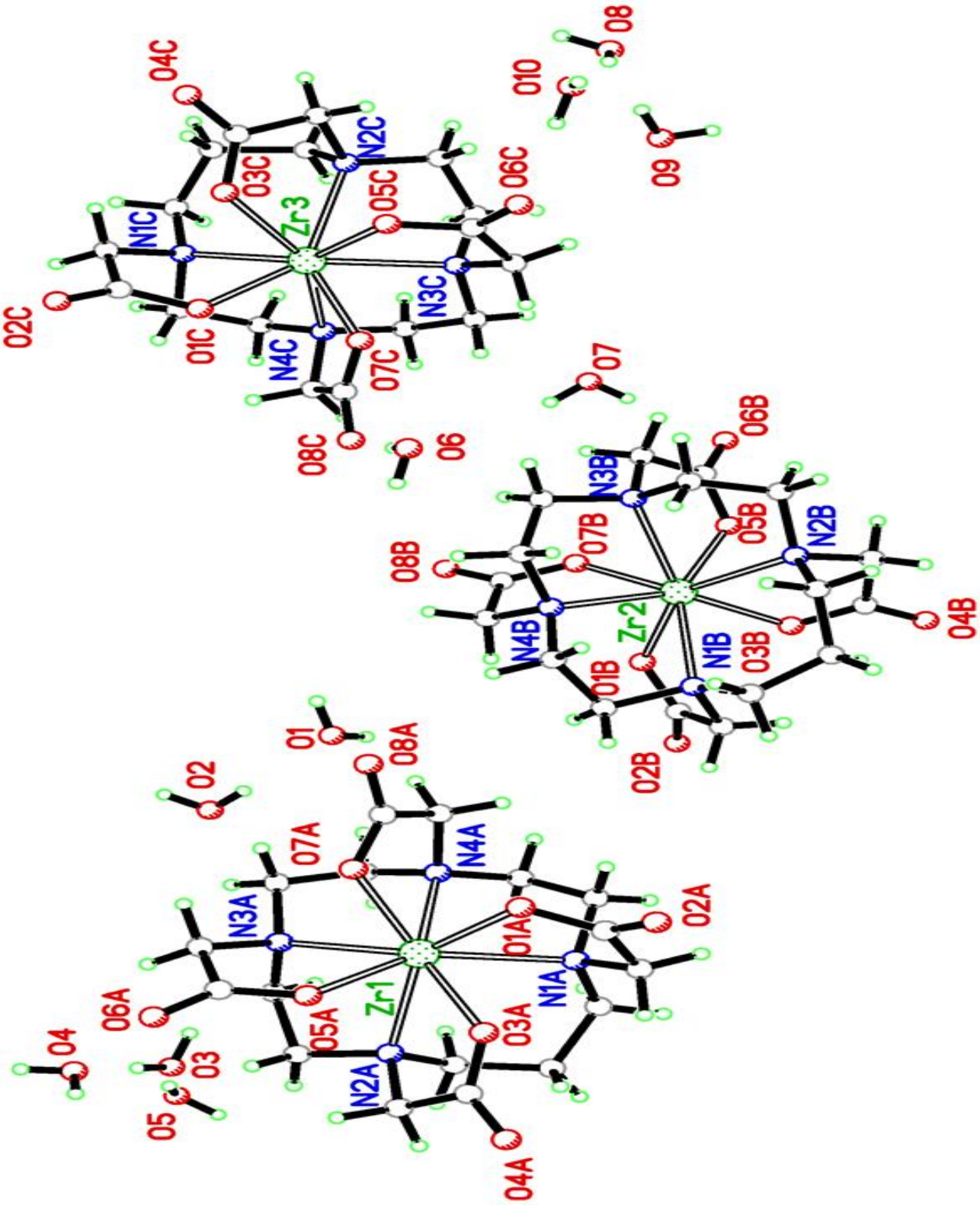


Figure S24. Plot for [Zr($C_{17}H_{26}N_4O_8$)] - 3.33 H_2O showing the contents of the asymmetric unit. All atoms are represented by arbitrary-sized spheres and only selected atoms are labeled.

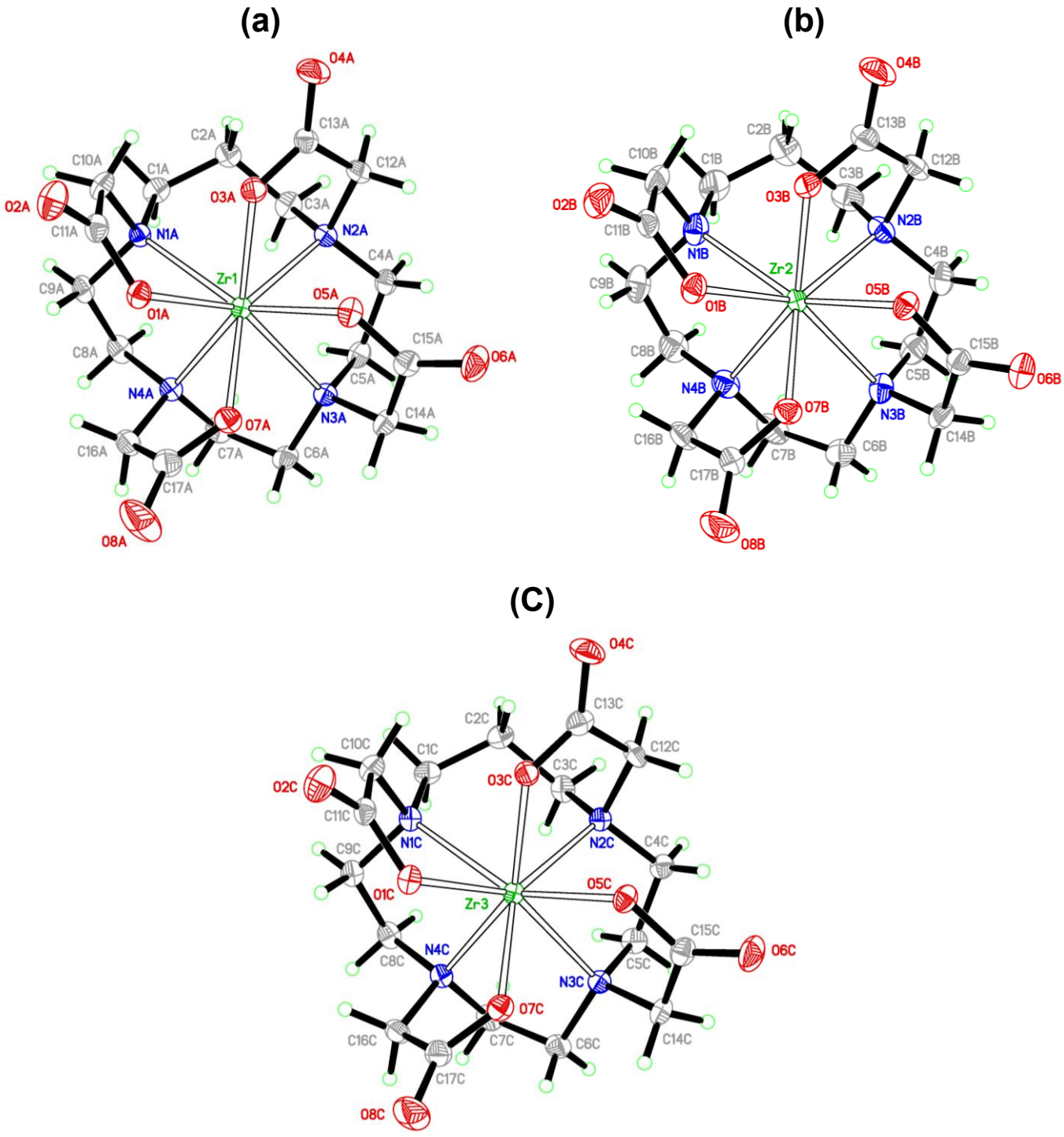


Figure S25. 50% probability plots for the 3 crystallographically-independent [Zr($C_{17}H_{26}N_4O_8$)] molecules in the solid-state structure of [Zr($C_{17}H_{26}N_4O_8$)] – 3.33 H_2O with hydrogen atoms represented by arbitrary-sized spheres.

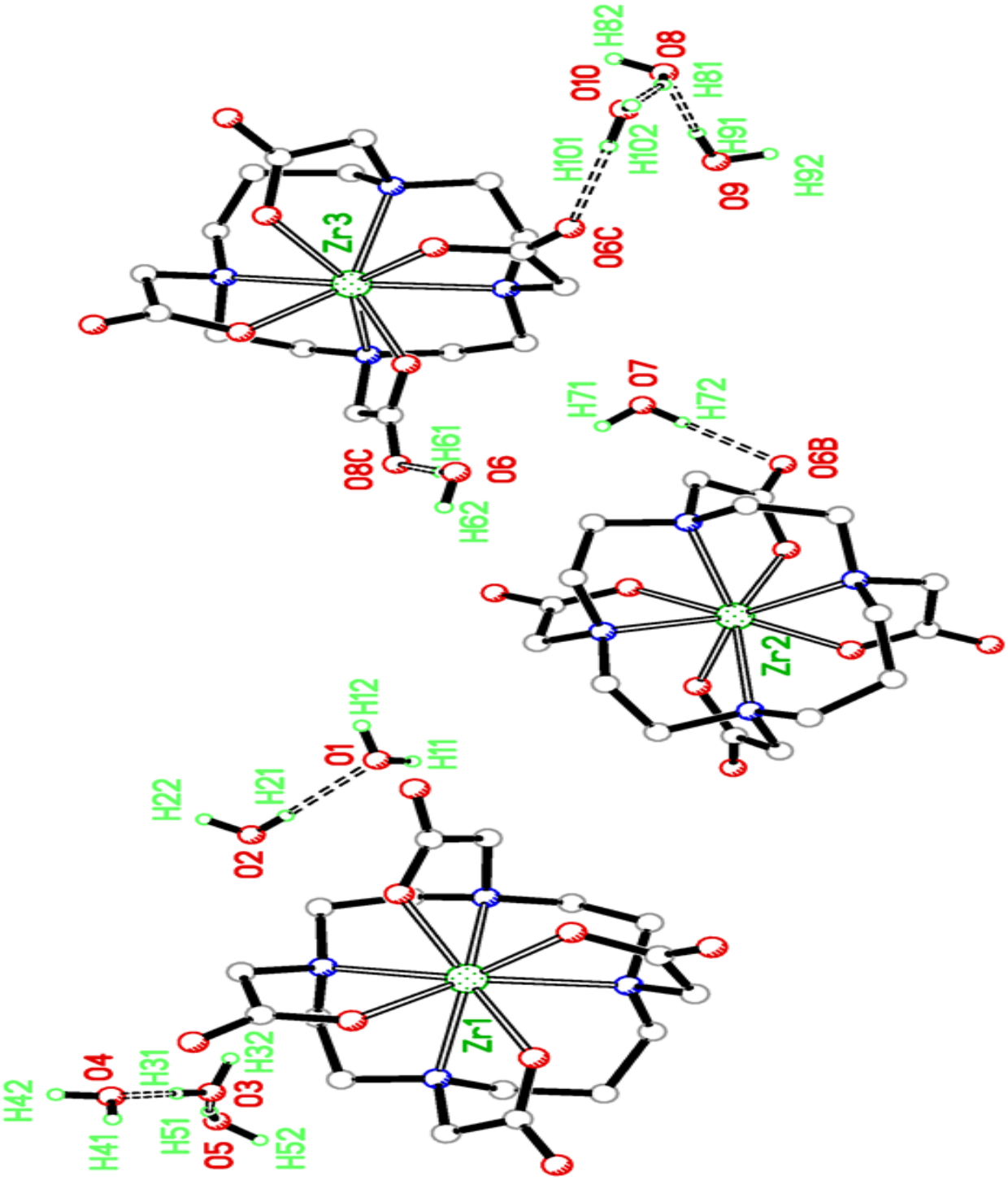


Figure S26. Plot showing the contents of the asymmetric unit for [Zr($C_{17}H_{26}N_4O_8$)] - 3.33 H_2O with all atoms represented by arbitrary-sized spheres. Ligand hydrogen atoms have been omitted and hydrogen-bonding interactions involving the lattice water molecules with the 3 independent [Zr($C_{17}H_{26}N_4O_8$)] molecules are represented by dashed-open bonds.

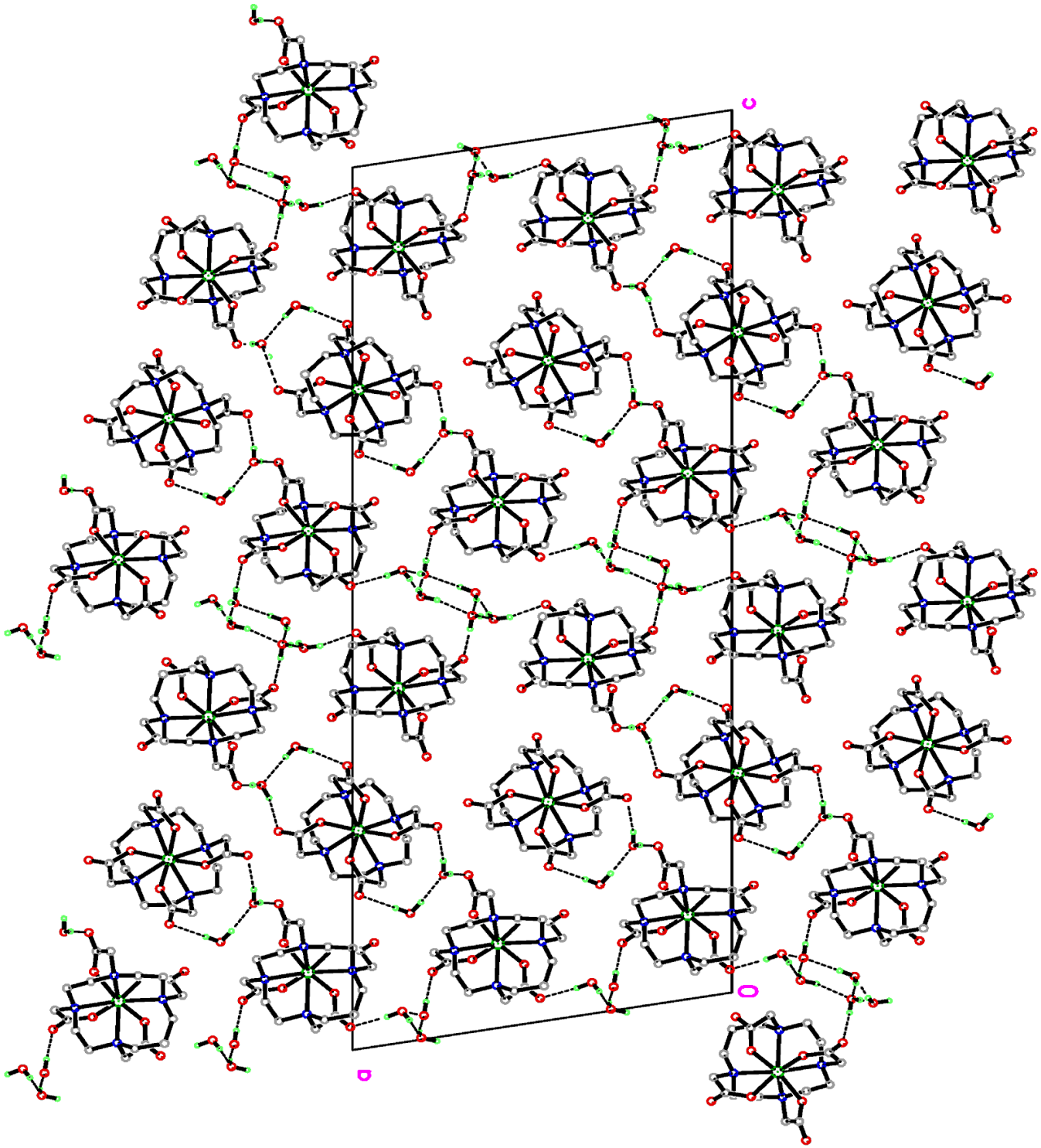


Figure S27. Packing plot of the solid-state structure of [Zr($C_{17}H_{26}N_4O_8$)] – 3.33 H_2O viewed in projection down the $\vec{b}$ axis of the unit cell. All atoms are represented by dummy-sized spheres and ligand hydrogen atoms have been omitted for clarity. Hydrogen-bonding interactions involving the lattice water molecules are represented by dashed-solid bonds.

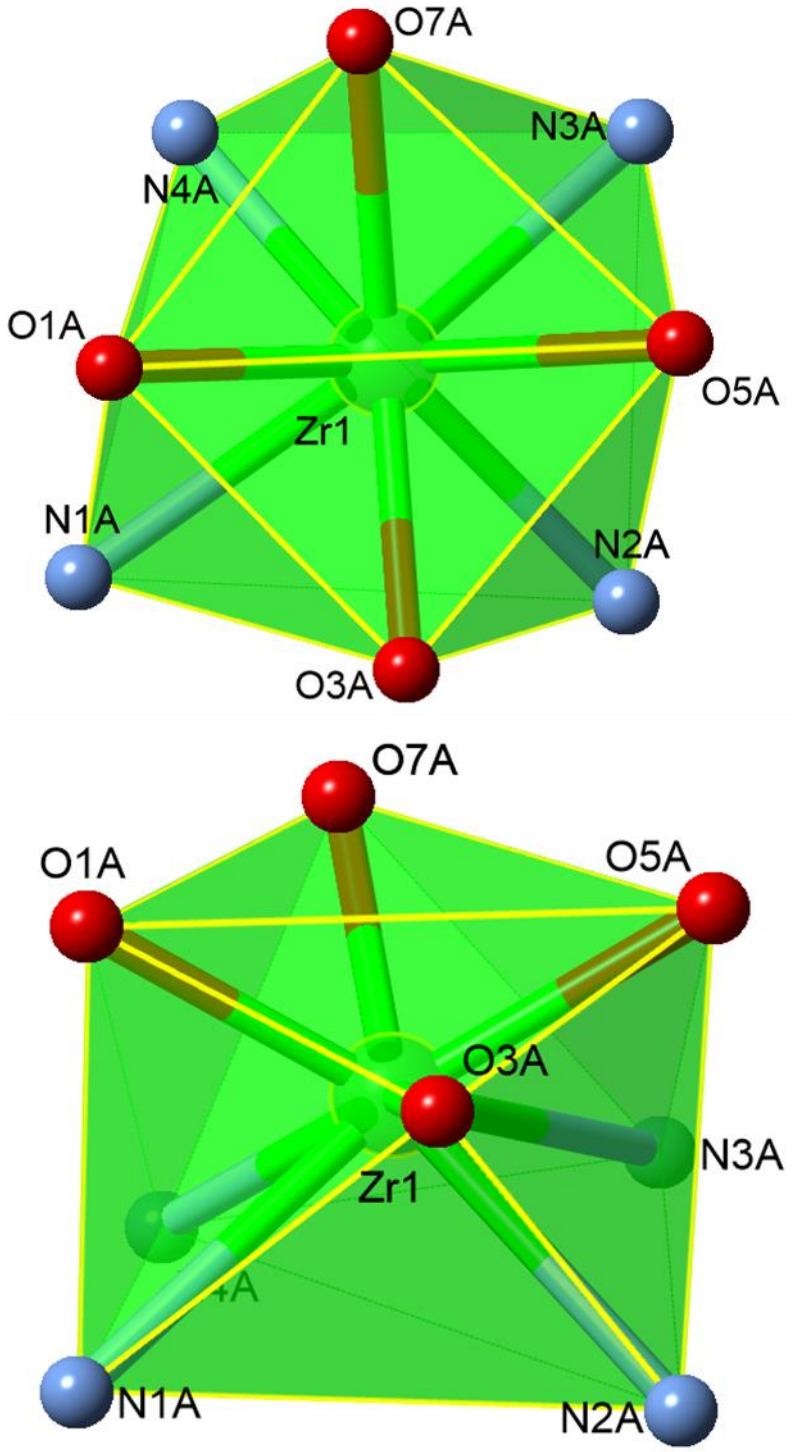


Figure S28. Molecule 1, top and side views showing the "distorted" square antiprism arrangement about Zr1.

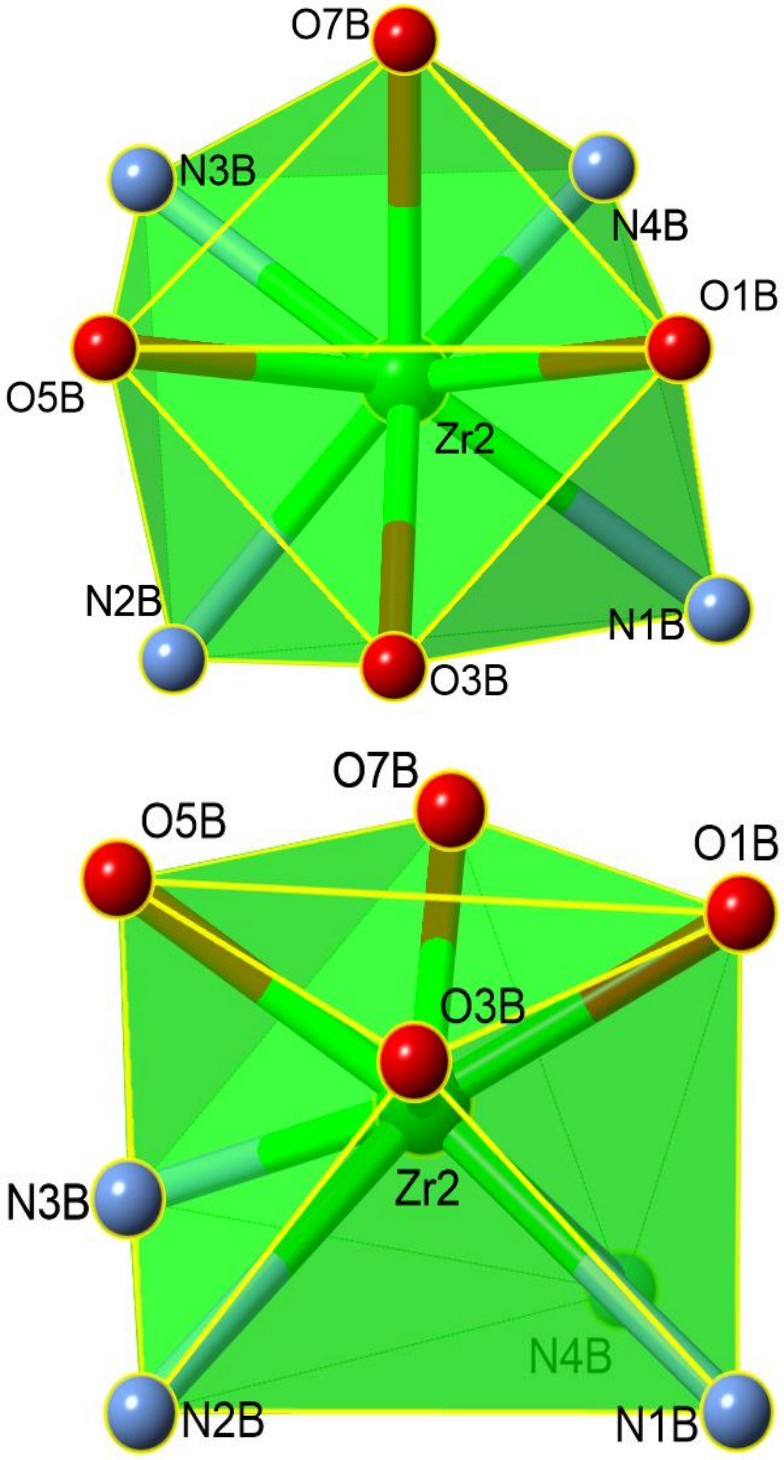


Figure S29. Molecule 2, top and side views showing the "distorted" square antiprism arrangement about Zr2.

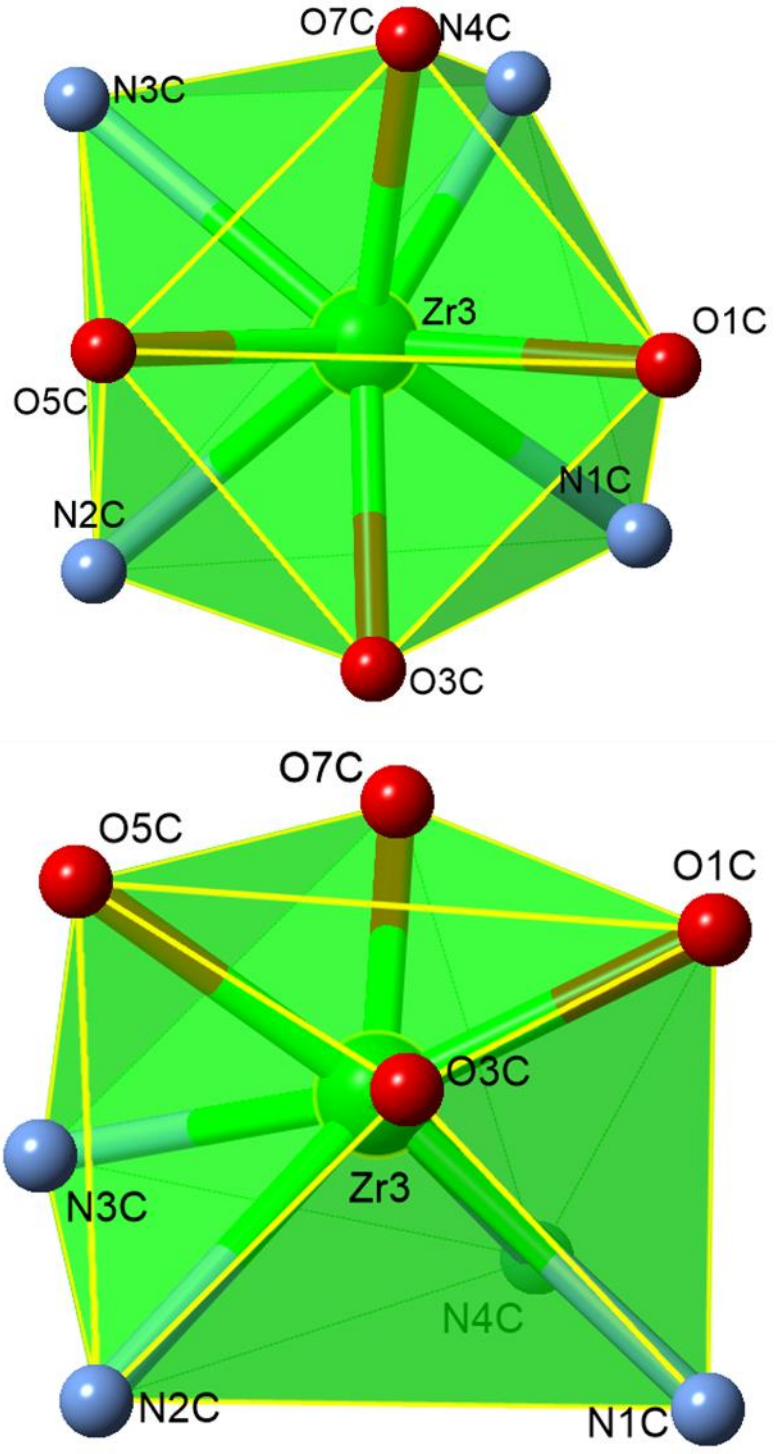


Figure S30. Molecule 3, top and side views showing the "distorted" square antiprism arrangement about Zr3.

Table S1. Crystal structure data collection parameters

Crystal data

$C_{17}H_{32.67}N_4O_{11.33}Zr$	$F(000) = 3520$
$M_r = 565.69$	$D_x = 1.706$ Mg m^{-3}
Monoclinic, *Cc*	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 19.2657$ (17) Å	Cell parameters from 9909 reflections
$b = 6.9888$ (6) Å	$\theta = 3.4–27.6°$
$c = 49.787$ (4) Å	$\mu = 0.57$ mm^{-1}
$\beta = 99.7028$ (11)°	$T = 193$ K
$V = 6607.6$ (10) Å^3	Needle, colourless
$Z = 12$	0.47 × 0.04 × 0.01 mm

Data collection

Bruker APEX CCD diffractometer	19202 independent reflections
Radiation source: sealed x-ray tube	16822 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.059$
ϕ and ω scans	$\theta_{max} = 30.1°$, $\theta_{min} = 3.4°$
Absorption correction: multi-scan Data were corrected for scaling and absorption effects using the multi-scan technique (*SADABS*). The ratio of minimum to maximum apparent transmission was 0.841. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.776 and 0.994.	$h = -27\rightarrow27$
$T_{min} = 0.627$, $T_{max} = 0.746$	$k = -9\rightarrow9$
61502 measured reflections	$l = -69\rightarrow69$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.079$	$w = 1/[\sigma^2(F_o^2) + (0.0304P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.003$
19202 reflections	$\Delta\rangle_{max} = 0.55$ e Å^{-3}
981 parameters	$\Delta\rangle_{min} = -0.62$ e Å^{-3}
32 restraints	Absolute structure: Flack x determined using 6966 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259)
Primary atom site location: structure-invariant direct methods	Absolute structure parameter: 0.016 (16)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table S2. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zr1	0.87976 (2)	1.48693 (5)	0.90232 (2)	0.01309 (8)
O1A	0.94954 (16)	1.6045 (4)	0.87864 (6)	0.0184 (6)
O2A	1.05440 (18)	1.7422 (4)	0.88036 (7)	0.0288 (8)
O3A	0.94959 (16)	1.6617 (4)	0.93078 (6)	0.0193 (6)
O4A	0.99029 (18)	1.7775 (5)	0.97191 (7)	0.0293 (8)
O5A	0.81246 (16)	1.6897 (4)	0.91621 (6)	0.0195 (6)
O6A	0.70833 (18)	1.7928 (5)	0.92409 (7)	0.0298 (8)
O7A	0.81553 (16)	1.5623 (4)	0.86471 (6)	0.0198 (6)
O8A	0.8047 (3)	1.5478 (6)	0.81969 (8)	0.0492 (12)
N1A	0.99584 (19)	1.3074 (5)	0.90816 (7)	0.0167 (7)
N2A	0.87506 (19)	1.3835 (5)	0.94934 (7)	0.0164 (7)
N3A	0.76393 (19)	1.3369 (5)	0.90257 (7)	0.0169 (7)
N4A	0.86712 (19)	1.2041 (5)	0.87279 (7)	0.0169 (7)
C1A	1.0215 (2)	1.1799 (6)	0.93188 (9)	0.0218 (9)
H1A	1.0736	1.1759	0.9346	0.026*
H1B	1.0042	1.0486	0.9273	0.026*
C2A	0.9994 (2)	1.2385 (6)	0.95851 (9)	0.0217 (9)
H2A	1.0262	1.1613	0.9734	0.026*
H2B	1.0122	1.3743	0.9622	0.026*
C3A	0.9217 (2)	1.2142 (6)	0.95891 (9)	0.0231 (10)
H3A	0.9157	1.1832	0.9778	0.028*
H3B	0.9048	1.1028	0.9474	0.028*
C4A	0.8013 (2)	1.3337 (6)	0.95242 (9)	0.0206 (9)
H4A	0.7747	1.4524	0.9545	0.025*
H4B	0.8021	1.2556	0.9691	0.025*
C5A	0.7651 (2)	1.2244 (7)	0.92816 (9)	0.0215 (9)
H5A	0.7900	1.1018	0.9268	0.026*

H5B	0.7163	1.1948	0.9305	0.026*
C6A	0.7427 (2)	1.2061 (7)	0.87889 (9)	0.0220 (9)
H6A	0.7030	1.1243	0.8821	0.026*
H6B	0.7273	1.2818	0.8621	0.026*
C7A	0.8044 (3)	1.0849 (6)	0.87558 (10)	0.0230 (10)
H7A	0.7919	1.0035	0.8592	0.028*
H7B	0.8161	0.9998	0.8916	0.028*
C8A	0.9300 (3)	1.0753 (6)	0.87712 (9)	0.0219 (10)
H8A	0.9275	0.9886	0.8926	0.026*
H8B	0.9304	0.9960	0.8607	0.026*
C9A	0.9959 (2)	1.1913 (6)	0.88279 (9)	0.0205 (9)
H9A	0.9987	1.2774	0.8672	0.025*
H9B	1.0374	1.1057	0.8852	0.025*
C10A	1.0485 (2)	1.4633 (6)	0.90717 (10)	0.0206 (10)
H10A	1.0912	1.4099	0.9013	0.025*
H10B	1.0624	1.5194	0.9256	0.025*
C11A	1.0172 (2)	1.6172 (6)	0.88738 (9)	0.0200 (9)
C12A	0.8985 (3)	1.5472 (7)	0.96777 (9)	0.0209 (10)
H12A	0.9202	1.4976	0.9859	0.025*
H12B	0.8570	1.6249	0.9702	0.025*
C13A	0.9508 (2)	1.6722 (6)	0.95666 (8)	0.0182 (8)
C14A	0.7121 (3)	1.4947 (6)	0.90166 (10)	0.0209 (10)
H14A	0.6729	1.4531	0.9108	0.025*
H14B	0.6923	1.5237	0.8824	0.025*
C15A	0.7445 (2)	1.6723 (6)	0.91518 (9)	0.0193 (9)
C16A	0.8585 (3)	1.2848 (7)	0.84484 (9)	0.0229 (10)
H16A	0.8299	1.1954	0.8321	0.028*
H16B	0.9054	1.2956	0.8393	0.028*
C17A	0.8234 (3)	1.4800 (7)	0.84240 (10)	0.0253 (11)

Zr2	0.98417 (2)	0.64557 (5)	0.74773 (2)	0.01392 (9)
O1B	0.96739 (17)	0.4937 (4)	0.78253 (6)	0.0221 (7)
O2B	1.01413 (19)	0.3776 (5)	0.82316 (7)	0.0298 (8)
O3B	1.08407 (16)	0.5300 (4)	0.76364 (6)	0.0207 (7)
O4B	1.1952 (2)	0.4663 (6)	0.76049 (8)	0.0435 (10)
O5B	1.00764 (17)	0.4699 (4)	0.71588 (6)	0.0200 (6)
O6B	0.98271 (18)	0.3468 (5)	0.67429 (6)	0.0275 (8)
O7B	0.88833 (16)	0.4978 (4)	0.73509 (6)	0.0208 (6)
O8B	0.7769 (2)	0.4490 (6)	0.73879 (9)	0.0455 (11)
N1B	1.0211 (2)	0.8346 (6)	0.79070 (8)	0.0263 (9)
N2B	1.0737 (2)	0.8271 (6)	0.72934 (8)	0.0243 (8)
N3B	0.9286 (2)	0.7841 (5)	0.70354 (7)	0.0235 (8)
N4B	0.8879 (2)	0.8596 (6)	0.75476 (8)	0.0260 (9)
C1B	1.0698 (3)	1.0021 (7)	0.79180 (10)	0.0340 (12)
H1C	1.0938	1.0188	0.8109	0.041*
H1D	1.0413	1.1184	0.7867	0.041*
C2B	1.1250 (3)	0.9882 (8)	0.77382 (12)	0.0341 (13)
H2C	1.1606	1.0891	0.7794	0.041*
H2D	1.1490	0.8632	0.7772	0.041*
C3B	1.0991 (3)	1.0069 (7)	0.74395 (11)	0.0317 (11)
H3C	1.0602	1.1010	0.7412	0.038*
H3D	1.1377	1.0598	0.7354	0.038*
C4B	1.0474 (3)	0.8777 (7)	0.69983 (9)	0.0291 (11)
H4C	1.0768	0.9819	0.6942	0.035*
H4D	1.0521	0.7649	0.6882	0.035*
C5B	0.9737 (3)	0.9388 (7)	0.69571 (10)	0.0303 (11)
H5C	0.9580	0.9726	0.6763	0.036*
H5D	0.9691	1.0540	0.7068	0.036*
C6B	0.8564 (3)	0.8633 (7)	0.70449 (10)	0.0295 (11)

H6C	0.8416	0.9473	0.6885	0.035*
H6D	0.8221	0.7569	0.7036	0.035*
C7B	0.8566 (3)	0.9730 (7)	0.72973 (12)	0.0315 (12)
H7C	0.8077	1.0097	0.7311	0.038*
H7D	0.8841	1.0919	0.7290	0.038*
C8B	0.9057 (3)	0.9970 (7)	0.77784 (10)	0.0322 (11)
H8C	0.9282	1.1123	0.7716	0.039*
H8D	0.8622	1.0372	0.7844	0.039*
C9B	0.9547 (3)	0.9048 (7)	0.80055 (10)	0.0320 (12)
H9C	0.9308	0.7954	0.8077	0.038*
H9D	0.9677	0.9981	0.8155	0.038*
C10B	1.0516 (3)	0.6865 (7)	0.81084 (9)	0.0276 (11)
H10C	1.0510	0.7337	0.8296	0.033*
H10D	1.1011	0.6615	0.8090	0.033*
C11B	1.0094 (3)	0.5024 (7)	0.80616 (9)	0.0205 (9)
C12B	1.1355 (2)	0.6962 (7)	0.73046 (10)	0.0263 (10)
H12C	1.1317	0.6266	0.7130	0.032*
H12D	1.1792	0.7734	0.7328	0.032*
C13B	1.1408 (3)	0.5543 (7)	0.75321 (10)	0.0245 (10)
C14B	0.9220 (3)	0.6292 (7)	0.68324 (10)	0.0245 (10)
H14C	0.9286	0.6831	0.6655	0.029*
H14D	0.8738	0.5756	0.6810	0.029*
C15B	0.9744 (3)	0.4704 (6)	0.69093 (9)	0.0201 (9)
C16B	0.8336 (3)	0.7192 (7)	0.76119 (10)	0.0270 (10)
H16C	0.8449	0.6829	0.7807	0.032*
H16D	0.7867	0.7812	0.7581	0.032*
C17B	0.8309 (3)	0.5423 (7)	0.74395 (10)	0.0242 (10)
Zr3	0.61650 (2)	0.93584 (5)	0.59569 (2)	0.01259 (8)
O1C	0.54787 (16)	1.0550 (4)	0.61947 (6)	0.0167 (6)

O2C	0.44296 (18)	1.1945 (5)	0.61812 (7)	0.0270 (7)
O3C	0.54762 (15)	1.1125 (4)	0.56761 (6)	0.0169 (6)
O4C	0.50589 (18)	1.2283 (5)	0.52644 (6)	0.0286 (8)
O5C	0.68470 (15)	1.1367 (4)	0.58198 (6)	0.0179 (6)
O6C	0.78919 (17)	1.2398 (5)	0.57495 (7)	0.0288 (8)
O7C	0.68136 (16)	1.0100 (4)	0.63314 (6)	0.0184 (6)
O8C	0.6949 (2)	0.9930 (5)	0.67855 (7)	0.0348 (9)
N1C	0.50009 (19)	0.7586 (5)	0.59010 (7)	0.0157 (7)
N2C	0.62043 (19)	0.8311 (5)	0.54863 (7)	0.0168 (7)
N3C	0.73157 (19)	0.7827 (5)	0.59534 (7)	0.0161 (7)
N4C	0.62806 (18)	0.6534 (5)	0.62530 (7)	0.0160 (7)
C1C	0.4736 (2)	0.6320 (6)	0.56630 (9)	0.0205 (9)
H1E	0.4215	0.6293	0.5637	0.025*
H1F	0.4906	0.5002	0.5708	0.025*
C2C	0.4955 (2)	0.6900 (6)	0.53974 (9)	0.0202 (9)
H2E	0.4833	0.8264	0.5362	0.024*
H2F	0.4681	0.6140	0.5248	0.024*
C3C	0.5738 (2)	0.6626 (6)	0.53914 (9)	0.0209 (9)
H3E	0.5906	0.5510	0.5507	0.025*
H3F	0.5795	0.6313	0.5202	0.025*
C4C	0.6939 (2)	0.7806 (6)	0.54540 (9)	0.0197 (9)
H4E	0.7207	0.8990	0.5434	0.024*
H4F	0.6928	0.7030	0.5287	0.024*
C5C	0.7298 (2)	0.6699 (6)	0.56963 (9)	0.0206 (9)
H5E	0.7044	0.5481	0.5710	0.025*
H5F	0.7785	0.6389	0.5672	0.025*
C6C	0.7524 (3)	0.6525 (7)	0.61911 (9)	0.0217 (10)
H6E	0.7682	0.7286	0.6358	0.026*
H6F	0.7919	0.5695	0.6159	0.026*

C7C	0.6900 (3)	0.5321 (6)	0.62260 (10)	0.0219 (10)
H7E	0.6779	0.4464	0.6067	0.026*
H7F	0.7022	0.4513	0.6390	0.026*
C8C	0.5651 (3)	0.5270 (6)	0.62123 (10)	0.0204 (9)
H8E	0.5645	0.4493	0.6378	0.024*
H8F	0.5673	0.4386	0.6058	0.024*
C9C	0.4995 (2)	0.6441 (6)	0.61536 (9)	0.0182 (9)
H9E	0.4578	0.5592	0.6129	0.022*
H9F	0.4967	0.7308	0.6309	0.022*
C10C	0.4482 (2)	0.9165 (7)	0.59114 (9)	0.0198 (9)
H10E	0.4052	0.8642	0.5968	0.024*
H10F	0.4349	0.9731	0.5728	0.024*
C11C	0.4794 (2)	1.0698 (6)	0.61101 (9)	0.0177 (9)
C12C	0.5976 (3)	0.9977 (7)	0.53042 (9)	0.0204 (10)
H12E	0.5759	0.9502	0.5122	0.024*
H12F	0.6394	1.0749	0.5282	0.024*
C13C	0.5455 (2)	1.1226 (6)	0.54166 (9)	0.0199 (9)
C14C	0.7844 (2)	0.9378 (6)	0.59640 (10)	0.0193 (9)
H14E	0.8230	0.8951	0.5870	0.023*
H14F	0.8048	0.9644	0.6156	0.023*
C15C	0.7525 (2)	1.1189 (6)	0.58320 (9)	0.0194 (9)
C16C	0.6378 (3)	0.7361 (7)	0.65354 (9)	0.0203 (9)
H16E	0.6655	0.6452	0.6664	0.024*
H16F	0.5910	0.7508	0.6591	0.024*
C17C	0.6746 (3)	0.9275 (7)	0.65582 (10)	0.0210 (10)
O1	0.7400 (3)	0.9006 (6)	0.81582 (8)	0.0454 (10)
H11	0.764 (2)	0.803 (5)	0.8175 (9)	0.024 (14)*
H12	0.720 (3)	0.897 (8)	0.8000 (6)	0.06 (2)*
O2	0.6500 (3)	0.7903 (8)	0.85387 (10)	0.0560 (12)

H21	0.676 (2)	0.834 (9)	0.8438 (11)	0.06 (2)*
H22	0.6105 (13)	0.825 (10)	0.8464 (11)	0.06 (2)*
O3	0.6848 (2)	0.9531 (6)	0.97189 (8)	0.0348 (9)
H31	0.663 (4)	1.054 (6)	0.9696 (11)	0.07 (3)*
H32	0.690 (3)	0.912 (6)	0.9571 (5)	0.022 (13)*
O4	0.6282 (2)	1.3038 (6)	0.96380 (9)	0.0393 (9)
H41	0.641 (3)	1.408 (6)	0.9704 (17)	0.10 (3)*
H42	0.5855 (9)	1.304 (8)	0.9636 (12)	0.039 (18)*
O5	0.6766 (2)	0.6024 (6)	0.99721 (10)	0.0462 (11)
H51	0.674 (2)	0.705 (4)	0.9893 (8)	0.016 (12)*
H52	0.7162 (17)	0.597 (9)	1.0061 (13)	0.07 (3)*
O6	0.7598 (2)	1.3433 (6)	0.68332 (8)	0.0446 (10)
H61	0.743 (3)	1.236 (4)	0.6829 (11)	0.05 (2)*
H62	0.760 (4)	1.383 (8)	0.6987 (6)	0.06 (2)*
O7	0.8479 (3)	0.2243 (8)	0.64502 (10)	0.0533 (12)
H71	0.820 (2)	0.235 (12)	0.6557 (11)	0.08 (3)*
H72	0.8863 (15)	0.258 (9)	0.6535 (10)	0.049 (19)*
O8	0.8191 (3)	1.0465 (6)	0.50202 (10)	0.0473 (11)
H81	0.825 (3)	1.149 (5)	0.5097 (12)	0.06 (2)*
H82	0.7775 (12)	1.038 (8)	0.4955 (13)	0.05 (2)*
O9	0.8689 (2)	0.7518 (6)	0.53532 (9)	0.0382 (9)
H91	0.850 (2)	0.832 (6)	0.5242 (8)	0.027 (15)*
H92	0.9090 (14)	0.747 (9)	0.5317 (12)	0.06 (2)*
O10	0.8118 (2)	1.3998 (5)	0.52682 (7)	0.0332 (8)
H101	0.806 (3)	1.365 (6)	0.5419 (5)	0.025 (14)*
H102	0.821 (4)	1.514 (3)	0.5279 (11)	0.06 (2)*

Table S3. Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zr1	0.01429 (19)	0.01012 (17)	0.01510 (17)	0.00042 (16)	0.00320 (13)	0.00088 (14)
O1A	0.0184 (16)	0.0182 (15)	0.0195 (15)	0.0010 (13)	0.0056 (12)	0.0039 (11)
O2A	0.0273 (19)	0.0212 (17)	0.041 (2)	-0.0034 (14)	0.0142 (15)	0.0066 (14)
O3A	0.0186 (16)	0.0187 (15)	0.0209 (15)	-0.0042 (12)	0.0043 (12)	-0.0034 (11)
O4A	0.0305 (19)	0.0296 (18)	0.0268 (17)	-0.0100 (15)	0.0017 (14)	-0.0098 (14)
O5A	0.0201 (16)	0.0140 (15)	0.0256 (16)	-0.0004 (12)	0.0074 (13)	0.0004 (11)
O6A	0.0232 (18)	0.0229 (17)	0.044 (2)	0.0053 (14)	0.0084 (15)	-0.0059 (15)
O7A	0.0219 (17)	0.0189 (16)	0.0180 (15)	0.0038 (13)	0.0017 (12)	0.0029 (11)
O8A	0.076 (3)	0.048 (3)	0.022 (2)	0.032 (2)	0.006 (2)	0.0100 (16)
N1A	0.0172 (19)	0.0159 (17)	0.0170 (17)	0.0038 (15)	0.0032 (14)	0.0009 (13)
N2A	0.0190 (19)	0.0139 (17)	0.0162 (17)	-0.0009 (14)	0.0029 (14)	0.0002 (13)
N3A	0.0177 (19)	0.0131 (17)	0.0196 (18)	-0.0029 (14)	0.0026 (14)	-0.0021 (13)
N4A	0.0192 (19)	0.0139 (16)	0.0176 (17)	0.0006 (14)	0.0032 (14)	0.0007 (13)
C1A	0.022 (2)	0.017 (2)	0.026 (2)	0.0047 (18)	0.0008 (18)	0.0058 (17)
C2A	0.025 (2)	0.020 (2)	0.018 (2)	0.0041 (18)	0.0014 (17)	0.0052 (16)
C3A	0.026 (3)	0.016 (2)	0.026 (2)	0.0032 (19)	0.0037 (19)	0.0064 (17)
C4A	0.020 (2)	0.022 (2)	0.021 (2)	-0.0014 (18)	0.0077 (17)	0.0021 (16)
C5A	0.019 (2)	0.020 (2)	0.028 (2)	-0.0045 (18)	0.0080 (18)	0.0032 (17)
C6A	0.018 (2)	0.021 (2)	0.027 (2)	-0.0059 (19)	0.0025 (18)	-0.0034 (18)
C7A	0.028 (3)	0.014 (2)	0.028 (2)	-0.0034 (18)	0.0045 (19)	-0.0065 (17)
C8A	0.032 (3)	0.0102 (19)	0.024 (2)	0.0071 (18)	0.0064 (19)	-0.0013 (16)
C9A	0.025 (2)	0.017 (2)	0.020 (2)	0.0084 (18)	0.0070 (18)	-0.0007 (16)
C10A	0.014 (2)	0.020 (2)	0.028 (3)	0.0024 (18)	0.0050 (18)	0.0034 (17)
C11A	0.021 (2)	0.018 (2)	0.023 (2)	0.0012 (18)	0.0077 (17)	-0.0022 (16)
C12A	0.022 (3)	0.021 (2)	0.019 (2)	-0.0022 (19)	0.0024 (18)	-0.0051 (16)
C13A	0.020 (2)	0.015 (2)	0.019 (2)	0.0024 (17)	0.0028 (16)	-0.0021 (15)
C14A	0.016 (2)	0.018 (2)	0.029 (3)	0.0010 (18)	0.0033 (19)	0.0004 (17)

C15A	0.020 (2)	0.018 (2)	0.019 (2)	0.0019 (18)	0.0027 (17)	0.0045 (16)
C16A	0.025 (3)	0.026 (2)	0.018 (2)	0.005 (2)	0.0038 (18)	-0.0001 (18)
C17A	0.025 (3)	0.029 (3)	0.021 (2)	0.006 (2)	0.002 (2)	0.0043 (18)
Zr2	0.0160 (2)	0.01155 (18)	0.01501 (16)	0.00001 (16)	0.00493 (15)	0.00014 (14)
O1B	0.0272 (18)	0.0213 (16)	0.0184 (15)	-0.0036 (13)	0.0052 (13)	0.0018 (12)
O2B	0.035 (2)	0.0315 (19)	0.0236 (18)	0.0058 (15)	0.0056 (15)	0.0091 (14)
O3B	0.0205 (17)	0.0223 (16)	0.0186 (15)	0.0022 (13)	0.0012 (12)	0.0021 (11)
O4B	0.024 (2)	0.066 (3)	0.042 (2)	0.0227 (19)	0.0099 (17)	0.0174 (19)
O5B	0.0247 (17)	0.0168 (15)	0.0178 (15)	0.0022 (13)	0.0020 (12)	-0.0018 (11)
O6B	0.036 (2)	0.0272 (18)	0.0201 (16)	-0.0007 (15)	0.0070 (14)	-0.0042 (13)
O7B	0.0190 (17)	0.0202 (16)	0.0228 (16)	-0.0024 (13)	0.0028 (13)	-0.0024 (12)
O8B	0.028 (2)	0.057 (3)	0.054 (3)	-0.0225 (19)	0.0146 (18)	-0.021 (2)
N1B	0.035 (2)	0.022 (2)	0.0226 (19)	-0.0064 (17)	0.0075 (17)	-0.0053 (15)
N2B	0.021 (2)	0.024 (2)	0.030 (2)	0.0006 (17)	0.0091 (16)	0.0043 (16)
N3B	0.031 (2)	0.0205 (19)	0.0193 (18)	0.0073 (17)	0.0052 (16)	0.0034 (14)
N4B	0.025 (2)	0.023 (2)	0.031 (2)	0.0005 (17)	0.0082 (17)	-0.0064 (16)
C1B	0.038 (3)	0.031 (3)	0.033 (3)	-0.012 (2)	0.005 (2)	-0.011 (2)
C2B	0.032 (3)	0.028 (3)	0.044 (3)	-0.011 (2)	0.011 (2)	-0.011 (2)
C3B	0.026 (3)	0.029 (3)	0.041 (3)	-0.006 (2)	0.006 (2)	0.004 (2)
C4B	0.039 (3)	0.028 (3)	0.023 (2)	-0.004 (2)	0.013 (2)	0.0086 (18)
C5B	0.038 (3)	0.029 (3)	0.024 (2)	-0.001 (2)	0.004 (2)	0.0089 (19)
C6B	0.027 (3)	0.032 (3)	0.028 (2)	0.009 (2)	0.0004 (19)	0.005 (2)
C7B	0.027 (3)	0.023 (3)	0.047 (3)	0.012 (2)	0.012 (2)	0.008 (2)
C8B	0.036 (3)	0.027 (3)	0.034 (3)	0.006 (2)	0.009 (2)	-0.006 (2)
C9B	0.049 (3)	0.024 (3)	0.025 (2)	0.006 (2)	0.014 (2)	-0.0063 (19)
C10B	0.030 (3)	0.037 (3)	0.015 (2)	-0.006 (2)	0.0018 (19)	-0.0058 (19)
C11B	0.022 (2)	0.023 (2)	0.018 (2)	0.0060 (19)	0.0082 (17)	0.0008 (17)
C12B	0.016 (2)	0.035 (3)	0.030 (2)	0.001 (2)	0.0092 (18)	0.001 (2)
C13B	0.020 (2)	0.027 (3)	0.027 (3)	0.004 (2)	0.0050 (19)	-0.0016 (18)

C14B	0.026 (3)	0.029 (3)	0.017 (2)	0.002 (2)	0.0001 (18)	0.0006 (17)
C15B	0.022 (2)	0.021 (2)	0.019 (2)	-0.0043 (19)	0.0053 (17)	0.0004 (16)
C16B	0.020 (2)	0.031 (3)	0.033 (3)	0.001 (2)	0.011 (2)	-0.004 (2)
C17B	0.021 (3)	0.031 (3)	0.022 (2)	-0.002 (2)	0.0067 (18)	0.0016 (18)
Zr3	0.01361 (19)	0.00982 (17)	0.01459 (17)	-0.00049 (15)	0.00310 (13)	-0.00080 (14)
O1C	0.0178 (16)	0.0135 (14)	0.0201 (15)	-0.0019 (12)	0.0064 (12)	-0.0034 (11)
O2C	0.0244 (18)	0.0219 (17)	0.0366 (19)	0.0032 (14)	0.0102 (14)	-0.0043 (14)
O3C	0.0174 (15)	0.0164 (15)	0.0173 (14)	0.0037 (12)	0.0047 (11)	0.0023 (11)
O4C	0.034 (2)	0.0274 (18)	0.0229 (17)	0.0105 (15)	0.0014 (14)	0.0090 (13)
O5C	0.0160 (15)	0.0143 (15)	0.0239 (16)	-0.0011 (12)	0.0049 (12)	0.0008 (11)
O6C	0.0209 (18)	0.0246 (18)	0.042 (2)	-0.0061 (14)	0.0087 (15)	0.0075 (14)
O7C	0.0178 (16)	0.0170 (15)	0.0200 (16)	-0.0024 (12)	0.0023 (12)	-0.0026 (11)
O8C	0.042 (2)	0.039 (2)	0.0212 (18)	-0.0130 (18)	0.0016 (16)	-0.0080 (14)
N1C	0.0179 (18)	0.0118 (16)	0.0180 (17)	-0.0015 (14)	0.0046 (13)	-0.0017 (12)
N2C	0.0171 (18)	0.0167 (18)	0.0168 (17)	-0.0004 (15)	0.0038 (14)	-0.0004 (13)
N3C	0.0165 (18)	0.0133 (17)	0.0186 (17)	0.0014 (14)	0.0027 (14)	0.0013 (13)
N4C	0.0188 (18)	0.0138 (16)	0.0154 (17)	-0.0018 (14)	0.0032 (13)	-0.0023 (12)
C1C	0.021 (2)	0.019 (2)	0.021 (2)	-0.0037 (18)	-0.0009 (17)	-0.0037 (16)
C2C	0.020 (2)	0.018 (2)	0.021 (2)	-0.0039 (18)	-0.0003 (17)	-0.0050 (16)
C3C	0.026 (2)	0.019 (2)	0.019 (2)	-0.0039 (19)	0.0058 (17)	-0.0069 (16)
C4C	0.018 (2)	0.021 (2)	0.021 (2)	0.0043 (18)	0.0058 (17)	-0.0033 (16)
C5C	0.024 (2)	0.018 (2)	0.020 (2)	0.0035 (18)	0.0045 (17)	-0.0058 (16)
C6C	0.022 (2)	0.023 (2)	0.021 (2)	0.0076 (19)	0.0030 (18)	0.0050 (17)
C7C	0.027 (3)	0.014 (2)	0.025 (2)	0.0066 (18)	0.0056 (19)	0.0055 (16)
C8C	0.026 (3)	0.014 (2)	0.022 (2)	-0.0047 (18)	0.0062 (19)	0.0007 (15)
C9C	0.021 (2)	0.015 (2)	0.019 (2)	-0.0042 (18)	0.0052 (17)	-0.0032 (15)
C10C	0.017 (2)	0.020 (2)	0.023 (2)	-0.0007 (18)	0.0037 (18)	-0.0027 (17)
C11C	0.018 (2)	0.016 (2)	0.020 (2)	-0.0003 (17)	0.0074 (17)	0.0030 (16)
C12C	0.028 (3)	0.019 (2)	0.015 (2)	0.0026 (19)	0.0078 (18)	0.0042 (16)

C13C	0.017 (2)	0.015 (2)	0.027 (2)	-0.0019 (17)	0.0011 (17)	0.0018 (16)
C14C	0.014 (2)	0.020 (2)	0.025 (2)	-0.0020 (17)	0.0055 (18)	-0.0003 (17)
C15C	0.019 (2)	0.018 (2)	0.022 (2)	-0.0033 (18)	0.0051 (17)	-0.0038 (16)
C16C	0.023 (2)	0.023 (2)	0.015 (2)	0.0005 (18)	0.0025 (17)	0.0020 (15)
C17C	0.020 (3)	0.024 (2)	0.018 (2)	0.0034 (19)	0.0034 (18)	-0.0015 (17)
O1	0.060 (3)	0.040 (2)	0.032 (2)	0.012 (2)	-0.003 (2)	-0.0060 (17)
O2	0.037 (3)	0.079 (3)	0.050 (3)	0.007 (3)	0.003 (2)	0.023 (2)
O3	0.047 (2)	0.029 (2)	0.028 (2)	0.0050 (19)	0.0079 (17)	0.0040 (15)
O4	0.036 (2)	0.032 (2)	0.052 (3)	0.0051 (18)	0.0124 (19)	-0.0029 (18)
O5	0.047 (3)	0.032 (2)	0.056 (3)	-0.013 (2)	-0.004 (2)	0.012 (2)
O6	0.060 (3)	0.040 (2)	0.034 (2)	-0.022 (2)	0.008 (2)	-0.0014 (18)
O7	0.037 (3)	0.070 (3)	0.050 (3)	-0.008 (2)	-0.001 (2)	-0.024 (2)
O8	0.045 (3)	0.034 (2)	0.059 (3)	0.012 (2)	-0.004 (2)	-0.013 (2)
O9	0.032 (2)	0.034 (2)	0.050 (3)	-0.0019 (18)	0.0085 (19)	0.0064 (18)
O10	0.044 (2)	0.026 (2)	0.030 (2)	-0.0060 (18)	0.0075 (17)	-0.0025 (15)

Table S4. Geometric parameters (Å, º)

Zr1—O1A	2.099 (3)	C6B—H6C	0.9900
Zr1—O5A	2.114 (3)	C6B—H6D	0.9900
Zr1—O7A	2.129 (3)	C7B—H7C	0.9900
Zr1—O3A	2.160 (3)	C7B—H7D	0.9900
Zr1—N4A	2.451 (4)	C8B—C9B	1.491 (7)
Zr1—N2A	2.466 (3)	C8B—H8C	0.9900
Zr1—N3A	2.468 (4)	C8B—H8D	0.9900
Zr1—N1A	2.538 (4)	C9B—H9C	0.9900
O1A—C11A	1.305 (6)	C9B—H9D	0.9900
O2A—C11A	1.218 (5)	C10B—C11B	1.519 (7)
O3A—C13A	1.287 (5)	C10B—H10C	0.9900

O4A—C13A	1.226 (5)	C10B—H10D	0.9900
O5A—C15A	1.307 (5)	C12B—C13B	1.496 (7)
O6A—C15A	1.223 (5)	C12B—H12C	0.9900
O7A—C17A	1.282 (6)	C12B—H12D	0.9900
O8A—C17A	1.223 (6)	C14B—C15B	1.506 (7)
N1A—C1A	1.496 (5)	C14B—H14C	0.9900
N1A—C10A	1.496 (6)	C14B—H14D	0.9900
N1A—C9A	1.501 (5)	C16B—C17B	1.501 (7)
N2A—C12A	1.488 (5)	C16B—H16C	0.9900
N2A—C4A	1.496 (6)	C16B—H16D	0.9900
N2A—C3A	1.514 (5)	Zr3—O1C	2.091 (3)
N3A—C14A	1.484 (6)	Zr3—O5C	2.113 (3)
N3A—C6A	1.493 (6)	Zr3—O7C	2.126 (3)
N3A—C5A	1.494 (5)	Zr3—O3C	2.148 (3)
N4A—C16A	1.485 (5)	Zr3—N4C	2.451 (3)
N4A—C7A	1.494 (6)	Zr3—N3C	2.465 (4)
N4A—C8A	1.496 (6)	Zr3—N2C	2.468 (3)
C1A—C2A	1.516 (6)	Zr3—N1C	2.536 (4)
C1A—H1A	0.9900	O1C—C11C	1.319 (5)
C1A—H1B	0.9900	O2C—C11C	1.209 (5)
C2A—C3A	1.510 (7)	O3C—C13C	1.288 (5)
C2A—H2A	0.9900	O4C—C13C	1.228 (5)
C2A—H2B	0.9900	O5C—C15C	1.303 (5)
C3A—H3A	0.9900	O6C—C15C	1.216 (5)
C3A—H3B	0.9900	O7C—C17C	1.294 (6)
C4A—C5A	1.498 (6)	O8C—C17C	1.222 (6)
C4A—H4A	0.9900	N1C—C9C	1.492 (5)
C4A—H4B	0.9900	N1C—C10C	1.495 (6)
C5A—H5A	0.9900	N1C—C1C	1.498 (5)

C5A—H5B	0.9900	N2C—C4C	1.493 (5)
C6A—C7A	1.491 (7)	N2C—C12C	1.496 (5)
C6A—H6A	0.9900	N2C—C3C	1.508 (5)
C6A—H6B	0.9900	N3C—C14C	1.482 (6)
C7A—H7A	0.9900	N3C—C6C	1.493 (5)
C7A—H7B	0.9900	N3C—C5C	1.499 (5)
C8A—C9A	1.492 (7)	N4C—C8C	1.486 (6)
C8A—H8A	0.9900	N4C—C7C	1.488 (6)
C8A—H8B	0.9900	N4C—C16C	1.503 (5)
C9A—H9A	0.9900	C1C—C2C	1.509 (6)
C9A—H9B	0.9900	C1C—H1E	0.9900
C10A—C11A	1.514 (6)	C1C—H1F	0.9900
C10A—H10A	0.9900	C2C—C3C	1.527 (6)
C10A—H10B	0.9900	C2C—H2E	0.9900
C12A—C13A	1.508 (6)	C2C—H2F	0.9900
C12A—H12A	0.9900	C3C—H3E	0.9900
C12A—H12B	0.9900	C3C—H3F	0.9900
C14A—C15A	1.497 (6)	C4C—C5C	1.501 (6)
C14A—H14A	0.9900	C4C—H4E	0.9900
C14A—H14B	0.9900	C4C—H4F	0.9900
C16A—C17A	1.518 (6)	C5C—H5E	0.9900
C16A—H16A	0.9900	C5C—H5F	0.9900
C16A—H16B	0.9900	C6C—C7C	1.502 (7)
Zr2—O1B	2.103 (3)	C6C—H6E	0.9900
Zr2—O5B	2.114 (3)	C6C—H6F	0.9900
Zr2—O3B	2.114 (3)	C7C—H7E	0.9900
Zr2—O7B	2.117 (3)	C7C—H7F	0.9900
Zr2—N2B	2.440 (4)	C8C—C9C	1.493 (6)
Zr2—N4B	2.453 (4)	C8C—H8E	0.9900

Zr2—N3B	2.476 (4)	C8C—H8F	0.9900
Zr2—N1B	2.513 (4)	C9C—H9E	0.9900
O1B—C11B	1.312 (6)	C9C—H9F	0.9900
O2B—C11B	1.208 (5)	C10C—C11C	1.512 (6)
O3B—C13B	1.297 (6)	C10C—H10E	0.9900
O4B—C13B	1.217 (6)	C10C—H10F	0.9900
O5B—C15B	1.298 (5)	C12C—C13C	1.507 (6)
O6B—C15B	1.226 (5)	C12C—H12E	0.9900
O7B—C17B	1.296 (6)	C12C—H12F	0.9900
O8B—C17B	1.218 (6)	C14C—C15C	1.508 (6)
N1B—C10B	1.491 (6)	C14C—H14E	0.9900
N1B—C1B	1.496 (6)	C14C—H14F	0.9900
N1B—C9B	1.527 (6)	C16C—C17C	1.510 (7)
N2B—C3B	1.492 (6)	C16C—H16E	0.9900
N2B—C12B	1.496 (6)	C16C—H16F	0.9900
N2B—C4B	1.513 (6)	O1—H11	0.818 (14)
N3B—C14B	1.471 (6)	O1—H12	0.818 (14)
N3B—C5B	1.479 (6)	O2—H21	0.825 (15)
N3B—C6B	1.506 (6)	O2—H22	0.824 (15)
N4B—C8B	1.492 (6)	O3—H31	0.814 (15)
N4B—C16B	1.507 (6)	O3—H32	0.814 (14)
N4B—C7B	1.514 (7)	O4—H41	0.822 (15)
C1B—C2B	1.503 (8)	O4—H42	0.820 (15)
C1B—H1C	0.9900	O5—H51	0.813 (14)
C1B—H1D	0.9900	O5—H52	0.817 (14)
C2B—C3B	1.492 (8)	O6—H61	0.816 (15)
C2B—H2C	0.9900	O6—H62	0.814 (14)
C2B—H2D	0.9900	O7—H71	0.826 (14)
C3B—H3C	0.9900	O7—H72	0.823 (15)

C3B—H3D	0.9900	O8—H81	0.810 (15)
C4B—C5B	1.465 (7)	O8—H82	0.816 (14)
C4B—H4C	0.9900	O9—H91	0.826 (14)
C4B—H4D	0.9900	O9—H92	0.822 (14)
C5B—H5C	0.9900	O10—H101	0.814 (14)
C5B—H5D	0.9900	O10—H102	0.815 (15)
C6B—C7B	1.471 (7)	N2B—C4B—H4D	109.4
O1A—Zr1—O5A	114.05 (12)	H4C—C4B—H4D	108.0
O1A—Zr1—O7A	75.02 (12)	C4B—C5B—N3B	110.4 (4)
O5A—Zr1—O7A	79.88 (12)	C4B—C5B—H5C	109.6
O1A—Zr1—O3A	76.12 (11)	N3B—C5B—H5C	109.6
O5A—Zr1—O3A	75.81 (12)	C4B—C5B—H5D	109.6
O7A—Zr1—O3A	129.97 (12)	N3B—C5B—H5D	109.6
O1A—Zr1—N4A	89.25 (12)	H5C—C5B—H5D	108.1
O5A—Zr1—N4A	137.09 (12)	C7B—C6B—N3B	110.4 (4)
O7A—Zr1—N4A	71.80 (12)	C7B—C6B—H6C	109.6
O3A—Zr1—N4A	146.86 (12)	N3B—C6B—H6C	109.6
O1A—Zr1—N2A	141.02 (12)	C7B—C6B—H6D	109.6
O5A—Zr1—N2A	76.30 (12)	N3B—C6B—H6D	109.6
O7A—Zr1—N2A	142.97 (12)	H6C—C6B—H6D	108.1
O3A—Zr1—N2A	70.22 (11)	C6B—C7B—N4B	111.9 (4)
N4A—Zr1—N2A	108.49 (12)	C6B—C7B—H7C	109.2
O1A—Zr1—N3A	146.55 (12)	N4B—C7B—H7C	109.2
O5A—Zr1—N3A	71.23 (12)	C6B—C7B—H7D	109.2
O7A—Zr1—N3A	73.59 (12)	N4B—C7B—H7D	109.2
O3A—Zr1—N3A	134.49 (11)	H7C—C7B—H7D	107.9
N4A—Zr1—N3A	70.23 (12)	C9B—C8B—N4B	109.9 (4)
N2A—Zr1—N3A	72.11 (12)	C9B—C8B—H8C	109.7
O1A—Zr1—N1A	67.82 (12)	N4B—C8B—H8C	109.7

O5A—Zr1—N1A	150.57 (12)	C9B—C8B—H8D	109.7
O7A—Zr1—N1A	126.40 (11)	N4B—C8B—H8D	109.7
O3A—Zr1—N1A	76.41 (12)	H8C—C8B—H8D	108.2
N4A—Zr1—N1A	70.53 (12)	C8B—C9B—N1B	110.6 (4)
N2A—Zr1—N1A	85.35 (12)	C8B—C9B—H9C	109.5
N3A—Zr1—N1A	124.72 (12)	N1B—C9B—H9C	109.5
C11A—O1A—Zr1	122.5 (3)	C8B—C9B—H9D	109.5
C13A—O3A—Zr1	126.4 (3)	N1B—C9B—H9D	109.5
C15A—O5A—Zr1	126.2 (3)	H9C—C9B—H9D	108.1
C17A—O7A—Zr1	121.0 (3)	N1B—C10B—C11B	110.2 (4)
C1A—N1A—C10A	108.9 (4)	N1B—C10B—H10C	109.6
C1A—N1A—C9A	107.2 (3)	C11B—C10B—H10C	109.6
C10A—N1A—C9A	105.6 (3)	N1B—C10B—H10D	109.6
C1A—N1A—Zr1	123.6 (3)	C11B—C10B—H10D	109.6
C10A—N1A—Zr1	103.1 (2)	H10C—C10B—H10D	108.1
C9A—N1A—Zr1	107.2 (2)	O2B—C11B—O1B	123.9 (5)
C12A—N2A—C4A	108.1 (3)	O2B—C11B—C10B	121.9 (4)
C12A—N2A—C3A	107.7 (3)	O1B—C11B—C10B	114.2 (4)
C4A—N2A—C3A	108.0 (3)	C13B—C12B—N2B	112.6 (4)
C12A—N2A—Zr1	107.7 (3)	C13B—C12B—H12C	109.1
C4A—N2A—Zr1	110.7 (2)	N2B—C12B—H12C	109.1
C3A—N2A—Zr1	114.4 (3)	C13B—C12B—H12D	109.1
C14A—N3A—C6A	109.7 (3)	N2B—C12B—H12D	109.1
C14A—N3A—C5A	109.3 (3)	H12C—C12B—H12D	107.8
C6A—N3A—C5A	108.3 (3)	O4B—C13B—O3B	123.7 (5)
C14A—N3A—Zr1	106.8 (3)	O4B—C13B—C12B	120.4 (5)
C6A—N3A—Zr1	112.6 (3)	O3B—C13B—C12B	115.9 (4)
C5A—N3A—Zr1	110.1 (3)	N3B—C14B—C15B	113.0 (4)
C16A—N4A—C7A	109.5 (4)	N3B—C14B—H14C	109.0

C16A—N4A—C8A	108.9 (3)	C15B—C14B—H14C	109.0
C7A—N4A—C8A	107.4 (3)	N3B—C14B—H14D	109.0
C16A—N4A—Zr1	103.9 (2)	C15B—C14B—H14D	109.0
C7A—N4A—Zr1	113.2 (2)	H14C—C14B—H14D	107.8
C8A—N4A—Zr1	113.8 (3)	O6B—C15B—O5B	123.0 (4)
N1A—C1A—C2A	115.2 (4)	O6B—C15B—C14B	120.4 (4)
N1A—C1A—H1A	108.5	O5B—C15B—C14B	116.5 (4)
C2A—C1A—H1A	108.5	C17B—C16B—N4B	111.9 (4)
N1A—C1A—H1B	108.5	C17B—C16B—H16C	109.2
C2A—C1A—H1B	108.5	N4B—C16B—H16C	109.2
H1A—C1A—H1B	107.5	C17B—C16B—H16D	109.2
C3A—C2A—C1A	114.0 (4)	N4B—C16B—H16D	109.2
C3A—C2A—H2A	108.8	H16C—C16B—H16D	107.9
C1A—C2A—H2A	108.8	O8B—C17B—O7B	123.2 (5)
C3A—C2A—H2B	108.8	O8B—C17B—C16B	120.5 (5)
C1A—C2A—H2B	108.8	O7B—C17B—C16B	116.3 (4)
H2A—C2A—H2B	107.6	O1C—Zr3—O5C	113.92 (12)
C2A—C3A—N2A	116.5 (4)	O1C—Zr3—O7C	74.97 (12)
C2A—C3A—H3A	108.2	O5C—Zr3—O7C	79.40 (12)
N2A—C3A—H3A	108.2	O1C—Zr3—O3C	76.07 (11)
C2A—C3A—H3B	108.2	O5C—Zr3—O3C	75.97 (11)
N2A—C3A—H3B	108.2	O7C—Zr3—O3C	129.64 (12)
H3A—C3A—H3B	107.3	O1C—Zr3—N4C	88.99 (11)
N2A—C4A—C5A	110.9 (4)	O5C—Zr3—N4C	136.95 (12)
N2A—C4A—H4A	109.5	O7C—Zr3—N4C	71.85 (11)
C5A—C4A—H4A	109.5	O3C—Zr3—N4C	146.90 (12)
N2A—C4A—H4B	109.5	O1C—Zr3—N3C	146.35 (12)
C5A—C4A—H4B	109.5	O5C—Zr3—N3C	71.17 (11)
H4A—C4A—H4B	108.0	O7C—Zr3—N3C	73.48 (12)

N3A—C5A—C4A	110.9 (4)	O3C—Zr3—N3C	134.66 (11)
N3A—C5A—H5A	109.5	N4C—Zr3—N3C	70.35 (12)
C4A—C5A—H5A	109.5	O1C—Zr3—N2C	141.19 (12)
N3A—C5A—H5B	109.5	O5C—Zr3—N2C	76.71 (12)
C4A—C5A—H5B	109.5	O7C—Zr3—N2C	142.87 (12)
H5A—C5A—H5B	108.0	O3C—Zr3—N2C	70.55 (11)
C7A—C6A—N3A	108.5 (4)	N4C—Zr3—N2C	108.52 (11)
C7A—C6A—H6A	110.0	N3C—Zr3—N2C	72.11 (12)
N3A—C6A—H6A	110.0	O1C—Zr3—N1C	67.96 (11)
C7A—C6A—H6B	110.0	O5C—Zr3—N1C	151.11 (11)
N3A—C6A—H6B	110.0	O7C—Zr3—N1C	126.40 (11)
H6A—C6A—H6B	108.4	O3C—Zr3—N1C	76.78 (11)
C6A—C7A—N4A	111.5 (4)	N4C—Zr3—N1C	70.22 (11)
C6A—C7A—H7A	109.3	N3C—Zr3—N1C	124.54 (11)
N4A—C7A—H7A	109.3	N2C—Zr3—N1C	85.35 (11)
C6A—C7A—H7B	109.3	C11C—O1C—Zr3	123.0 (3)
N4A—C7A—H7B	109.3	C13C—O3C—Zr3	126.6 (3)
H7A—C7A—H7B	108.0	C15C—O5C—Zr3	126.5 (3)
C9A—C8A—N4A	110.0 (3)	C17C—O7C—Zr3	121.8 (3)
C9A—C8A—H8A	109.7	C9C—N1C—C10C	105.5 (3)
N4A—C8A—H8A	109.7	C9C—N1C—C1C	107.5 (3)
C9A—C8A—H8B	109.7	C10C—N1C—C1C	108.9 (3)
N4A—C8A—H8B	109.7	C9C—N1C—Zr3	107.5 (2)
H8A—C8A—H8B	108.2	C10C—N1C—Zr3	102.7 (3)
C8A—C9A—N1A	109.4 (4)	C1C—N1C—Zr3	123.5 (3)
C8A—C9A—H9A	109.8	C4C—N2C—C12C	107.8 (3)
N1A—C9A—H9A	109.8	C4C—N2C—C3C	107.9 (3)
C8A—C9A—H9B	109.8	C12C—N2C—C3C	108.6 (3)
N1A—C9A—H9B	109.8	C4C—N2C—Zr3	110.7 (2)

H9A—C9A—H9B	108.2	C12C—N2C—Zr3	106.9 (2)
N1A—C10A—C11A	109.9 (4)	C3C—N2C—Zr3	114.7 (2)
N1A—C10A—H10A	109.7	C14C—N3C—C6C	109.0 (3)
C11A—C10A—H10A	109.7	C14C—N3C—C5C	109.4 (3)
N1A—C10A—H10B	109.7	C6C—N3C—C5C	108.7 (3)
C11A—C10A—H10B	109.7	C14C—N3C—Zr3	107.2 (3)
H10A—C10A—H10B	108.2	C6C—N3C—Zr3	112.4 (3)
O2A—C11A—O1A	123.8 (4)	C5C—N3C—Zr3	110.1 (2)
O2A—C11A—C10A	120.4 (4)	C8C—N4C—C7C	107.3 (3)
O1A—C11A—C10A	115.8 (4)	C8C—N4C—C16C	109.0 (3)
N2A—C12A—C13A	111.8 (4)	C7C—N4C—C16C	109.2 (3)
N2A—C12A—H12A	109.3	C8C—N4C—Zr3	114.0 (3)
C13A—C12A—H12A	109.3	C7C—N4C—Zr3	113.4 (2)
N2A—C12A—H12B	109.3	C16C—N4C—Zr3	103.7 (2)
C13A—C12A—H12B	109.3	N1C—C1C—C2C	115.2 (4)
H12A—C12A—H12B	107.9	N1C—C1C—H1E	108.5
O4A—C13A—O3A	124.0 (4)	C2C—C1C—H1E	108.5
O4A—C13A—C12A	120.3 (4)	N1C—C1C—H1F	108.5
O3A—C13A—C12A	115.7 (4)	C2C—C1C—H1F	108.5
N3A—C14A—C15A	112.0 (4)	H1E—C1C—H1F	107.5
N3A—C14A—H14A	109.2	C1C—C2C—C3C	114.0 (4)
C15A—C14A—H14A	109.2	C1C—C2C—H2E	108.8
N3A—C14A—H14B	109.2	C3C—C2C—H2E	108.8
C15A—C14A—H14B	109.2	C1C—C2C—H2F	108.8
H14A—C14A—H14B	107.9	C3C—C2C—H2F	108.8
O6A—C15A—O5A	123.4 (4)	H2E—C2C—H2F	107.7
O6A—C15A—C14A	120.9 (4)	N2C—C3C—C2C	115.8 (3)
O5A—C15A—C14A	115.7 (4)	N2C—C3C—H3E	108.3
N4A—C16A—C17A	113.2 (4)	C2C—C3C—H3E	108.3

N4A—C16A—H16A	108.9	N2C—C3C—H3F	108.3
C17A—C16A—H16A	108.9	C2C—C3C—H3F	108.3
N4A—C16A—H16B	108.9	H3E—C3C—H3F	107.4
C17A—C16A—H16B	108.9	N2C—C4C—C5C	110.6 (3)
H16A—C16A—H16B	107.7	N2C—C4C—H4E	109.5
O8A—C17A—O7A	124.6 (5)	C5C—C4C—H4E	109.5
O8A—C17A—C16A	118.7 (5)	N2C—C4C—H4F	109.5
O7A—C17A—C16A	116.7 (4)	C5C—C4C—H4F	109.5
O1B—Zr2—O5B	114.01 (11)	H4E—C4C—H4F	108.1
O1B—Zr2—O3B	75.83 (12)	N3C—C5C—C4C	110.9 (3)
O5B—Zr2—O3B	76.27 (12)	N3C—C5C—H5E	109.5
O1B—Zr2—O7B	75.59 (12)	C4C—C5C—H5E	109.5
O5B—Zr2—O7B	77.28 (12)	N3C—C5C—H5F	109.5
O3B—Zr2—O7B	128.19 (11)	C4C—C5C—H5F	109.5
O1B—Zr2—N2B	142.00 (14)	H5E—C5C—H5F	108.1
O5B—Zr2—N2B	76.50 (13)	N3C—C6C—C7C	108.6 (4)
O3B—Zr2—N2B	71.51 (13)	N3C—C6C—H6E	110.0
O7B—Zr2—N2B	141.12 (13)	C7C—C6C—H6E	110.0
O1B—Zr2—N4B	88.01 (13)	N3C—C6C—H6F	110.0
O5B—Zr2—N4B	136.35 (13)	C7C—C6C—H6F	110.0
O3B—Zr2—N4B	147.34 (12)	H6E—C6C—H6F	108.4
O7B—Zr2—N4B	72.38 (13)	N4C—C7C—C6C	111.2 (3)
N2B—Zr2—N4B	109.61 (14)	N4C—C7C—H7E	109.4
O1B—Zr2—N3B	145.70 (13)	C6C—C7C—H7E	109.4
O5B—Zr2—N3B	71.07 (12)	N4C—C7C—H7F	109.4
O3B—Zr2—N3B	135.41 (12)	C6C—C7C—H7F	109.4
O7B—Zr2—N3B	72.65 (13)	H7E—C7C—H7F	108.0
N2B—Zr2—N3B	71.96 (13)	N4C—C8C—C9C	110.2 (3)
N4B—Zr2—N3B	70.39 (13)	N4C—C8C—H8E	109.6

O1B—Zr2—N1B	67.97 (13)	C9C—C8C—H8E	109.6
O5B—Zr2—N1B	151.43 (14)	N4C—C8C—H8F	109.6
O3B—Zr2—N1B	76.86 (13)	C9C—C8C—H8F	109.6
O7B—Zr2—N1B	128.13 (13)	H8E—C8C—H8F	108.1
N2B—Zr2—N1B	85.93 (13)	N1C—C9C—C8C	109.4 (3)
N4B—Zr2—N1B	70.76 (13)	N1C—C9C—H9E	109.8
N3B—Zr2—N1B	124.67 (13)	C8C—C9C—H9E	109.8
C11B—O1B—Zr2	123.9 (3)	N1C—C9C—H9F	109.8
C13B—O3B—Zr2	125.1 (3)	C8C—C9C—H9F	109.8
C15B—O5B—Zr2	125.6 (3)	H9E—C9C—H9F	108.2
C17B—O7B—Zr2	122.2 (3)	N1C—C10C—C11C	110.6 (4)
C10B—N1B—C1B	110.2 (4)	N1C—C10C—H10E	109.5
C10B—N1B—C9B	105.3 (4)	C11C—C10C—H10E	109.5
C1B—N1B—C9B	107.0 (4)	N1C—C10C—H10F	109.5
C10B—N1B—Zr2	103.3 (3)	C11C—C10C—H10F	109.5
C1B—N1B—Zr2	121.9 (3)	H10E—C10C—H10F	108.1
C9B—N1B—Zr2	108.1 (3)	O2C—C11C—O1C	124.2 (4)
C3B—N2B—C12B	107.6 (4)	O2C—C11C—C10C	121.1 (4)
C3B—N2B—C4B	107.8 (4)	O1C—C11C—C10C	114.8 (4)
C12B—N2B—C4B	108.2 (4)	N2C—C12C—C13C	111.8 (4)
C3B—N2B—Zr2	116.7 (3)	N2C—C12C—H12E	109.3
C12B—N2B—Zr2	106.1 (3)	C13C—C12C—H12E	109.3
C4B—N2B—Zr2	110.1 (3)	N2C—C12C—H12F	109.3
C14B—N3B—C5B	110.1 (4)	C13C—C12C—H12F	109.3
C14B—N3B—C6B	108.6 (4)	H12E—C12C—H12F	107.9
C5B—N3B—C6B	109.0 (4)	O4C—C13C—O3C	124.3 (4)
C14B—N3B—Zr2	107.3 (3)	O4C—C13C—C12C	120.2 (4)
C5B—N3B—Zr2	109.6 (3)	O3C—C13C—C12C	115.5 (4)
C6B—N3B—Zr2	112.3 (3)	N3C—C14C—C15C	111.8 (4)

C8B—N4B—C16B	109.5 (4)	N3C—C14C—H14E	109.3
C8B—N4B—C7B	107.8 (4)	C15C—C14C—H14E	109.3
C16B—N4B—C7B	108.7 (4)	N3C—C14C—H14F	109.3
C8B—N4B—Zr2	114.9 (3)	C15C—C14C—H14F	109.3
C16B—N4B—Zr2	101.6 (3)	H14E—C14C—H14F	107.9
C7B—N4B—Zr2	114.1 (3)	O6C—C15C—O5C	123.5 (4)
N1B—C1B—C2B	115.4 (4)	O6C—C15C—C14C	120.8 (4)
N1B—C1B—H1C	108.4	O5C—C15C—C14C	115.6 (4)
C2B—C1B—H1C	108.4	N4C—C16C—C17C	113.2 (4)
N1B—C1B—H1D	108.4	N4C—C16C—H16E	108.9
C2B—C1B—H1D	108.4	C17C—C16C—H16E	108.9
H1C—C1B—H1D	107.5	N4C—C16C—H16F	108.9
C3B—C2B—C1B	115.9 (5)	C17C—C16C—H16F	108.9
C3B—C2B—H2C	108.3	H16E—C16C—H16F	107.8
C1B—C2B—H2C	108.3	O8C—C17C—O7C	125.3 (5)
C3B—C2B—H2D	108.3	O8C—C17C—C16C	118.3 (4)
C1B—C2B—H2D	108.3	O7C—C17C—C16C	116.4 (4)
H2C—C2B—H2D	107.4	H11—O1—H12	104 (3)
N2B—C3B—C2B	116.0 (4)	H21—O2—H22	103 (3)
N2B—C3B—H3C	108.3	H31—O3—H32	108 (3)
C2B—C3B—H3C	108.3	H41—O4—H42	104 (3)
N2B—C3B—H3D	108.3	H51—O5—H52	107 (4)
C2B—C3B—H3D	108.3	H61—O6—H62	107 (3)
H3C—C3B—H3D	107.4	H71—O7—H72	106 (3)
C5B—C4B—N2B	111.2 (4)	H81—O8—H82	107 (4)
C5B—C4B—H4C	109.4	H91—O9—H92	102 (3)
N2B—C4B—H4C	109.4	H101—O10—H102	107 (3)
C5B—C4B—H4D	109.4	C1B—N1B—C10B—C11B	-169.4 (4)
C10A—N1A—C1A—C2A	89.1 (5)	C9B—N1B—C10B—C11B	75.6 (4)

C9A—N1A—C1A—C2A	-157.1 (4)	Zr2—N1B—C10B—C11B	-37.7 (4)
Zr1—N1A—C1A—C2A	-31.8 (5)	Zr2—O1B—C11B—O2B	-155.5 (4)
N1A—C1A—C2A—C3A	69.3 (5)	Zr2—O1B—C11B—C10B	25.6 (5)
C1A—C2A—C3A—N2A	-89.6 (5)	N1B—C10B—C11B—O2B	-163.7 (4)
C12A—N2A—C3A—C2A	-61.1 (5)	N1B—C10B—C11B—O1B	15.3 (6)
C4A—N2A—C3A—C2A	-177.6 (4)	C3B—N2B—C12B—C13B	96.4 (5)
Zr1—N2A—C3A—C2A	58.6 (4)	C4B—N2B—C12B—C13B	-147.4 (4)
C12A—N2A—C4A—C5A	159.1 (4)	Zr2—N2B—C12B—C13B	-29.3 (5)
C3A—N2A—C4A—C5A	-84.6 (4)	Zr2—O3B—C13B—O4B	-169.8 (4)
Zr1—N2A—C4A—C5A	41.4 (4)	Zr2—O3B—C13B—C12B	8.4 (6)
C14A—N3A—C5A—C4A	-73.3 (4)	N2B—C12B—C13B—O4B	-164.2 (5)
C6A—N3A—C5A—C4A	167.2 (4)	N2B—C12B—C13B—O3B	17.5 (6)
Zr1—N3A—C5A—C4A	43.7 (4)	C5B—N3B—C14B—C15B	94.9 (5)
N2A—C4A—C5A—N3A	-58.2 (5)	C6B—N3B—C14B—C15B	-145.8 (4)
C14A—N3A—C6A—C7A	163.7 (4)	Zr2—N3B—C14B—C15B	-24.3 (5)
C5A—N3A—C6A—C7A	-77.0 (4)	Zr2—O5B—C15B—O6B	-168.4 (3)
Zr1—N3A—C6A—C7A	45.0 (4)	Zr2—O5B—C15B—C14B	9.6 (6)
N3A—C6A—C7A—N4A	-55.3 (5)	N3B—C14B—C15B—O6B	-168.7 (4)
C16A—N4A—C7A—C6A	-76.8 (4)	N3B—C14B—C15B—O5B	13.3 (6)
C8A—N4A—C7A—C6A	165.1 (4)	C8B—N4B—C16B—C17B	-161.0 (4)
Zr1—N4A—C7A—C6A	38.6 (4)	C7B—N4B—C16B—C17B	81.5 (5)
C16A—N4A—C8A—C9A	78.2 (4)	Zr2—N4B—C16B—C17B	-39.1 (4)
C7A—N4A—C8A—C9A	-163.3 (3)	Zr2—O7B—C17B—O8B	-173.4 (4)
Zr1—N4A—C8A—C9A	-37.2 (4)	Zr2—O7B—C17B—C16B	6.9 (6)
N4A—C8A—C9A—N1A	60.7 (4)	N4B—C16B—C17B—O8B	-153.1 (5)
C1A—N1A—C9A—C8A	82.7 (4)	N4B—C16B—C17B—O7B	26.6 (6)
C10A—N1A—C9A—C8A	-161.3 (3)	C9C—N1C—C1C—C2C	-157.5 (4)
Zr1—N1A—C9A—C8A	-51.9 (4)	C10C—N1C—C1C—C2C	88.7 (5)
C1A—N1A—C10A—C11A	-167.9 (3)	Zr3—N1C—C1C—C2C	-31.6 (5)

C9A—N1A—C10A—C11A	77.3 (4)	N1C—C1C—C2C—C3C	69.5 (5)
Zr1—N1A—C10A—C11A	-35.0 (4)	C4C—N2C—C3C—C2C	-177.5 (4)
Zr1—O1A—C11A—O2A	-149.9 (4)	C12C—N2C—C3C—C2C	-60.9 (5)
Zr1—O1A—C11A—C10A	30.5 (5)	Zr3—N2C—C3C—C2C	58.6 (4)
N1A—C10A—C11A—O2A	-168.7 (4)	C1C—C2C—C3C—N2C	-89.9 (5)
N1A—C10A—C11A—O1A	10.8 (5)	C12C—N2C—C4C—C5C	158.6 (4)
C4A—N2A—C12A—C13A	-149.8 (4)	C3C—N2C—C4C—C5C	-84.3 (4)
C3A—N2A—C12A—C13A	93.7 (4)	Zr3—N2C—C4C—C5C	42.0 (4)
Zr1—N2A—C12A—C13A	-30.2 (4)	C14C—N3C—C5C—C4C	-73.8 (4)
Zr1—O3A—C13A—O4A	-178.0 (3)	C6C—N3C—C5C—C4C	167.3 (4)
Zr1—O3A—C13A—C12A	0.8 (5)	Zr3—N3C—C5C—C4C	43.7 (4)
N2A—C12A—C13A—O4A	-158.9 (4)	N2C—C4C—C5C—N3C	-58.6 (5)
N2A—C12A—C13A—O3A	22.2 (6)	C14C—N3C—C6C—C7C	163.9 (4)
C6A—N3A—C14A—C15A	-152.5 (4)	C5C—N3C—C6C—C7C	-77.0 (4)
C5A—N3A—C14A—C15A	88.9 (4)	Zr3—N3C—C6C—C7C	45.1 (4)
Zr1—N3A—C14A—C15A	-30.2 (4)	C8C—N4C—C7C—C6C	165.2 (4)
Zr1—O5A—C15A—O6A	178.4 (3)	C16C—N4C—C7C—C6C	-76.7 (4)
Zr1—O5A—C15A—C14A	-2.9 (5)	Zr3—N4C—C7C—C6C	38.4 (4)
N3A—C14A—C15A—O6A	-156.6 (4)	N3C—C6C—C7C—N4C	-55.3 (5)
N3A—C14A—C15A—O5A	24.6 (5)	C7C—N4C—C8C—C9C	-163.1 (4)
C7A—N4A—C16A—C17A	91.3 (5)	C16C—N4C—C8C—C9C	78.8 (4)
C8A—N4A—C16A—C17A	-151.4 (4)	Zr3—N4C—C8C—C9C	-36.6 (4)
Zr1—N4A—C16A—C17A	-29.9 (4)	C10C—N1C—C9C—C8C	-160.7 (4)
Zr1—O7A—C17A—O8A	-157.8 (5)	C1C—N1C—C9C—C8C	83.3 (4)
Zr1—O7A—C17A—C16A	22.2 (6)	Zr3—N1C—C9C—C8C	-51.7 (4)
N4A—C16A—C17A—O8A	-169.9 (5)	N4C—C8C—C9C—N1C	60.2 (4)
N4A—C16A—C17A—O7A	10.1 (6)	C9C—N1C—C10C—C11C	76.8 (4)
C10B—N1B—C1B—C2B	87.9 (5)	C1C—N1C—C10C—C11C	-168.2 (3)
C9B—N1B—C1B—C2B	-158.2 (5)	Zr3—N1C—C10C—C11C	-35.8 (4)

Zr2—N1B—C1B—C2B	-33.3 (6)	Zr3—O1C—C11C—O2C	-150.0 (4)
N1B—C1B—C2B—C3B	71.5 (6)	Zr3—O1C—C11C—C10C	29.8 (5)
C12B—N2B—C3B—C2B	-65.6 (5)	N1C—C10C—C11C—O2C	-168.3 (4)
C4B—N2B—C3B—C2B	177.9 (4)	N1C—C10C—C11C—O1C	11.9 (5)
Zr2—N2B—C3B—C2B	53.4 (5)	C4C—N2C—C12C—C13C	-150.0 (4)
C1B—C2B—C3B—N2B	-86.2 (6)	C3C—N2C—C12C—C13C	93.3 (4)
C3B—N2B—C4B—C5B	-86.3 (5)	Zr3—N2C—C12C—C13C	-30.9 (4)
C12B—N2B—C4B—C5B	157.5 (4)	Zr3—O3C—C13C—O4C	-178.8 (3)
Zr2—N2B—C4B—C5B	42.0 (5)	Zr3—O3C—C13C—C12C	-0.5 (5)
N2B—C4B—C5B—N3B	-60.0 (5)	N2C—C12C—C13C—O4C	-158.0 (4)
C14B—N3B—C5B—C4B	-72.3 (5)	N2C—C12C—C13C—O3C	23.7 (6)
C6B—N3B—C5B—C4B	168.7 (4)	C6C—N3C—C14C—C15C	-151.4 (4)
Zr2—N3B—C5B—C4B	45.5 (4)	C5C—N3C—C14C—C15C	89.8 (4)
C14B—N3B—C6B—C7B	162.2 (4)	Zr3—N3C—C14C—C15C	-29.5 (4)
C5B—N3B—C6B—C7B	-77.8 (5)	Zr3—O5C—C15C—O6C	-180.0 (3)
Zr2—N3B—C6B—C7B	43.8 (5)	Zr3—O5C—C15C—C14C	-1.5 (5)
N3B—C6B—C7B—N4B	-51.7 (6)	N3C—C14C—C15C—O6C	-158.3 (4)
C8B—N4B—C7B—C6B	163.3 (4)	N3C—C14C—C15C—O5C	23.2 (5)
C16B—N4B—C7B—C6B	-78.2 (5)	C8C—N4C—C16C—C17C	-152.5 (4)
Zr2—N4B—C7B—C6B	34.4 (5)	C7C—N4C—C16C—C17C	90.5 (4)
C16B—N4B—C8B—C9B	78.1 (5)	Zr3—N4C—C16C—C17C	-30.7 (4)
C7B—N4B—C8B—C9B	-163.8 (4)	Zr3—O7C—C17C—O8C	-159.1 (4)
Zr2—N4B—C8B—C9B	-35.4 (5)	Zr3—O7C—C17C—C16C	20.2 (5)
N4B—C8B—C9B—N1B	57.2 (6)	N4C—C16C—C17C—O8C	-168.7 (4)
C10B—N1B—C9B—C8B	-159.2 (4)	N4C—C16C—C17C—O7C	12.0 (6)
C1B—N1B—C9B—C8B	83.6 (5)		
Zr2—N1B—C9B—C8B	-49.3 (5)		

Table S5. Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H11···O8*A*^{i}	0.82 (1)	1.95 (2)	2.755 (6)	170 (5)
O1—H12···O4*B*^{ii}	0.82 (1)	2.00 (3)	2.782 (6)	159 (7)
O2—H21···O1	0.83 (2)	2.06 (2)	2.881 (7)	171 (7)
O2—H22···O2*B*^{ii}	0.82 (2)	2.05 (2)	2.865 (6)	171 (7)
O3—H31···O4	0.81 (2)	1.88 (2)	2.685 (6)	170 (8)
O3—H32···O6*A*^{i}	0.81 (1)	1.93 (2)	2.736 (5)	174 (5)
O4—H41···O5^{iii}	0.82 (2)	1.94 (3)	2.733 (6)	160 (9)
O4—H42···O4*A*^{iv}	0.82 (2)	1.96 (2)	2.760 (5)	166 (6)
O5—H51···O3	0.81 (1)	1.97 (2)	2.772 (6)	170 (4)
O5—H52···O10^{v}	0.82 (1)	1.95 (2)	2.769 (6)	177 (6)
O6—H61···O8*C*	0.82 (2)	1.93 (2)	2.740 (5)	173 (7)
O6—H62···O8*B*^{iii}	0.81 (1)	2.02 (2)	2.824 (6)	169 (7)
O7—H71···O6^{i}	0.83 (1)	2.08 (3)	2.881 (7)	163 (8)
O7—H72···O6*B*	0.82 (2)	2.06 (2)	2.886 (6)	179 (7)
O8—H81···O10	0.81 (2)	1.98 (2)	2.775 (6)	165 (6)
O8—H82···O3^{vi}	0.82 (1)	1.97 (2)	2.764 (6)	166 (7)
O9—H91···O8	0.83 (1)	1.90 (2)	2.715 (6)	170 (5)
O9—H92···O4*C*^{vii}	0.82 (1)	1.93 (2)	2.754 (6)	175 (6)
O10—H101···O6*C*	0.81 (1)	1.94 (2)	2.745 (5)	170 (5)
O10—H102···O9^{iii}	0.82 (2)	1.91 (3)	2.699 (6)	163 (8)

Symmetry codes: (i) *x*, *y*-1, *z*; (ii) *x*-1/2, *y*+1/2, *z*; (iii) *x*, *y*+1, *z*; (iv) *x*-1/2, *y*-1/2, *z*; (v) *x*, -*y*+2, *z*+1/2; (vi) *x*, -*y*+2, *z*-1/2; (vii) *x*+1/2, *y*-1/2, *z*.

Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)

0.9419 (0.0224) x + 6.7776 (0.0020) y - 12.1401 (0.0530) z = 0.9781 (0.0496)

* 0.1240 (0.0015) O1A

* -0.1211 (0.0015) O3A

* 0.1163 (0.0014) O5A

* -0.1192 (0.0015) O7A

-1.0259 (0.0015) Zr1

-2.2041 (0.0046) N1A

-2.3020 (0.0044) N2A

-2.1551 (0.0045) N3A

-2.5963 (0.0042) N4A

Rms deviation of fitted atoms = 0.1202

- 1.1167 (0.0222) x - 6.6504 (0.0027) y + 15.3003 (0.0578) z = 4.2197 (0.0581)

Angle to previous plane (with approximate esd) = 3.785 (0.087)

* -0.1315 (0.0016) N1A

* 0.1273 (0.0015) N2A

* -0.1540 (0.0018) N3A

* 0.1582 (0.0019) N4A

-1.2851 (0.0017) Zr1

-2.5070 (0.0039) O1A

-2.0899 (0.0043) O3A

-2.3459 (0.0039) O5A

-2.2898 (0.0040) O7A

Rms deviation of fitted atoms = 0.1434

- 0.7072 (0.0240) x + 6.9685 (0.0009) y - 2.9665 (0.0656) z = 0.5485 (0.0507)

Angle to previous plane (with approximate esd) = 14.797 (0.070)

* -0.1138 (0.0014) O1B

* 0.1126 (0.0014) O3B

* -0.1103 (0.0014) O5B

* 0.1116 (0.0014) O7B

1.0360 (0.0014) Zr2

2.2000 (0.0051) N1B

2.2925 (0.0049) N2B

2.1717 (0.0049) N3B

2.5748 (0.0048) N4B

Rms deviation of fitted atoms = 0.1121

0.4842 (0.0279) x + 6.9620 (0.0010) y - 4.3223 (0.0623) z = 3.0083 (0.0498)

Angle to previous plane (with approximate esd) = 3.676 (0.030)

* -0.1207 (0.0018) N1B

* 0.1176 (0.0018) N2B

* -0.1407 (0.0021) N3B

* 0.1438 (0.0022) N4B

-1.2693 (0.0019) Zr2

-2.4853 (0.0042) O1B

-2.0945 (0.0046) O3B

-2.3433 (0.0041) O5B

-2.2898 (0.0043) O7B

Rms deviation of fitted atoms = 0.1312

- 0.7622 (0.0214) x + 6.7766 (0.0019) y + 12.1758 (0.0519) z = 14.1519 (0.0288)

Angle to previous plane (with approximate esd) = 19.143 (0.049)

* 0.1226 (0.0015) O1C

* -0.1195 (0.0015) O3C

* 0.1149 (0.0014) O5C

* -0.1180 (0.0015) O7C

-1.0270 (0.0015) Zr3

-2.2074 (0.0044) N1C

-2.3131 (0.0044) N2C

-2.1569 (0.0045) N3C

-2.5896 (0.0041) N4C

Rms deviation of fitted atoms = 0.1188

- 0.9408 (0.0219) x + 6.6587 (0.0026) y + 15.1199 (0.0565) z = 13.3718 (0.0336)

Angle to previous plane (with approximate esd) = 3.524 (0.085)

* 0.1312 (0.0016) N1C

* -0.1265 (0.0015) N2C

* 0.1530 (0.0018) N3C

* -0.1577 (0.0019) N4C

1.2864 (0.0017) Zr3

2.5042 (0.0037) O1C

2.1027 (0.0042) O3C

2.3521 (0.0040) O5C

2.2854 (0.0039) O7C

Rms deviation of fitted atoms = 0.1428

Crystal Structure Analysis of Zr-PCTA, $[Zr(C_{17}H_{21}N_4O_6)]_2O$ - 8 H_2O

Crystallographic Experimental Details:

Data Collection and Structure Solution. A clear colourless plate-like specimen of $[Zr(C_{17}H_{21}N_4O_6)]_2O$ - 8 H_2O, approximate dimensions 0.030 mm x 0.098 mm x 0.460 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker APEX CCD system equipped with a graphite monochromator and a Mo Kα sealed x-ray tube (λ = 0.71073 Å). X-rays were provided by a fine-focus sealed x-ray tube operated at 50kV and 30mA.

The total exposure time was 18.65 hours. The frames were integrated with the Bruker SAINT Software[1] package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 39,418 reflections to a maximum θ angle of 30.08° (0.71 Å resolution), of which 6124 were independent (average redundancy 6.437, completeness = 99.2%, R_{int} = 4.67%, R_{sig} = 2.93%) and 5433 (88.72%) were greater than 2σ(F^2). The final cell constants of a = 26.632 (4) Å, b = 10.6634 (17) Å, c = 15.884 (3) Å, β = 111.098 (2)°, volume = 4208.5 (12) $Å^3$, are based upon the refinement of the XYZ-centroids of 9951 reflections above 20 σ(I) with 6.877° < 2θ < 60.06°. Data were corrected for absorption effects using the multi-scan method (SADABS)[2]. The ratio of minimum to maximum apparent transmission was 0.895. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.7730 and 0.9830.

The structure was solved and refined using the Bruker SHELXTL Software Package[3], using the space group C2/c, with Z = 4 for the formula unit, $C_{34}H_{58}N_8O_{21}Zr_2$. The final model includes 4 “full” occupancy water molecules per asymmetric unit. The final structural model incorporated isotropic thermal parameters for all included hydrogen atoms. The hydrogen atoms of the PCTA ligand were included in the structural model as fixed atoms (using idealized sp^2- or sp^3-hybridized geometry and C-H bond lengths of 0.95 and 0.99 Å, respectively) "riding" on their respective carbon atoms. The isotropic thermal parameters for all PCTA hydrogen atoms were fixed at values 1.2 times the equivalent isotropic thermal parameter of the carbon atom to which they are covalently bonded. The isotropic thermal parameters of the water hydrogen atoms were allowed to refine independently. The final anisotropic full-matrix least-squares refinement on F^2 with 326 variables converged at R_1 = 2.33%, for the observed data and wR_2 = 6.33% for all data. The goodness-of-fit was 1.057. The largest peak in the final difference electron density synthesis was 0.529 $e^-/Å^3$ and the largest hole was -0.309 $e^-/Å^3$ with an RMS deviation

of 0.074 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.732 g/cm^3 and F(000), 2264 e^-.

Refinement Details. Crystal data, data collection and structure refinement details are summarized in tables below.

Computing details. Data collection: Bruker *APEX3* v2016.1-0; cell refinement: Bruker *APEX3* v2016.1-0; data reduction: Bruker *APEX2* v2014.11-0; program(s) used to solve structure: *SHELXS97* (Sheldrick 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2014); molecular graphics: Bruker *APEX2* v2014.11-0; software used to prepare material for publication: Bruker *APEX2* v2014.11-0[1-6].

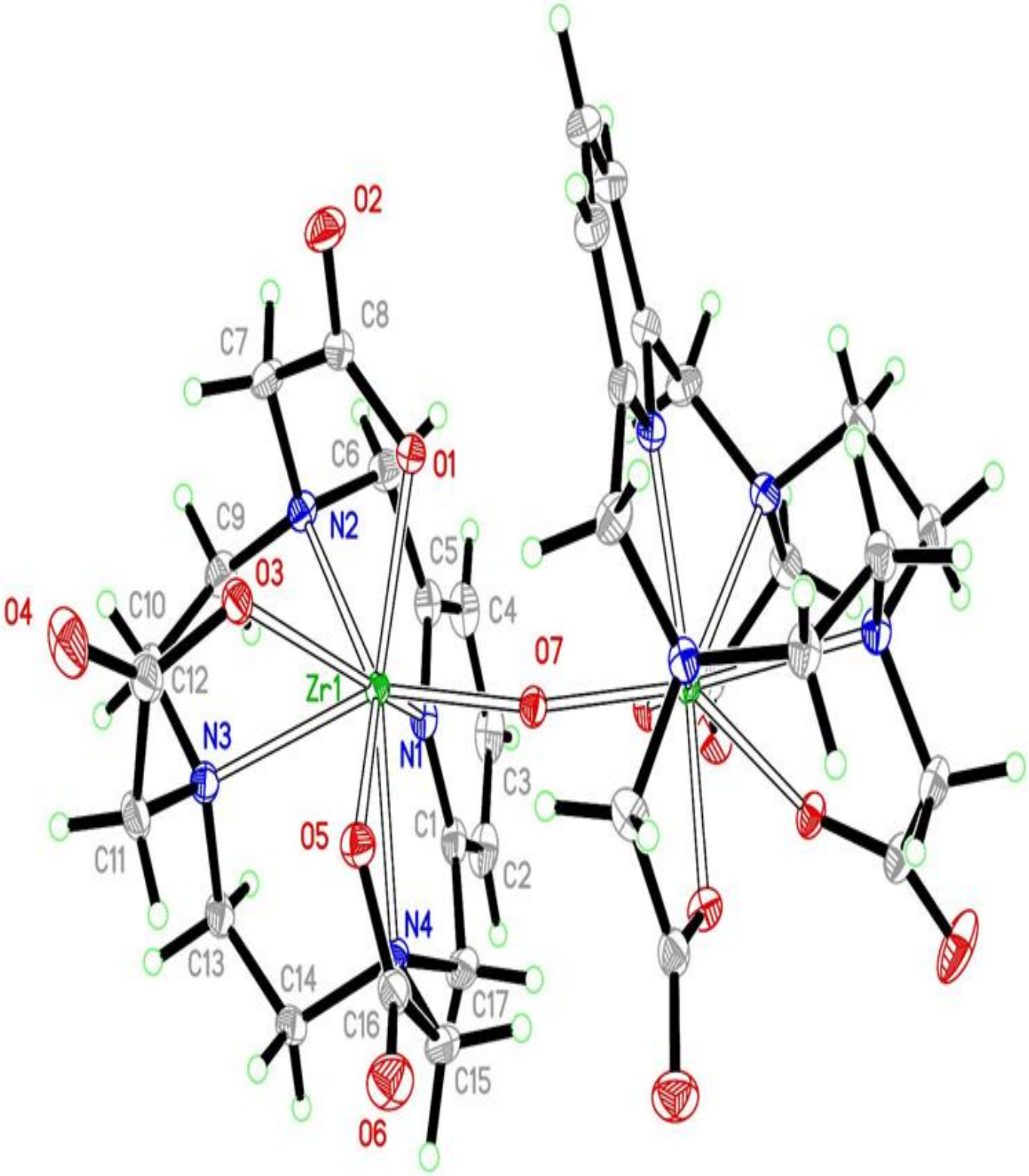


Figure S31. 50% probability plot for the dimeric unit of $[Zr(C_{17}H_{21}N_4O_6)]_2O$ - 8 H_2O with the water molecules omitted for clarity. The bridging oxygen atom, O_7, lies on the crystallographic 2-fold axis at ½, y, ¼ in the unit cell and only the non-hydrogen atoms of the asymmetric unit are labeled.

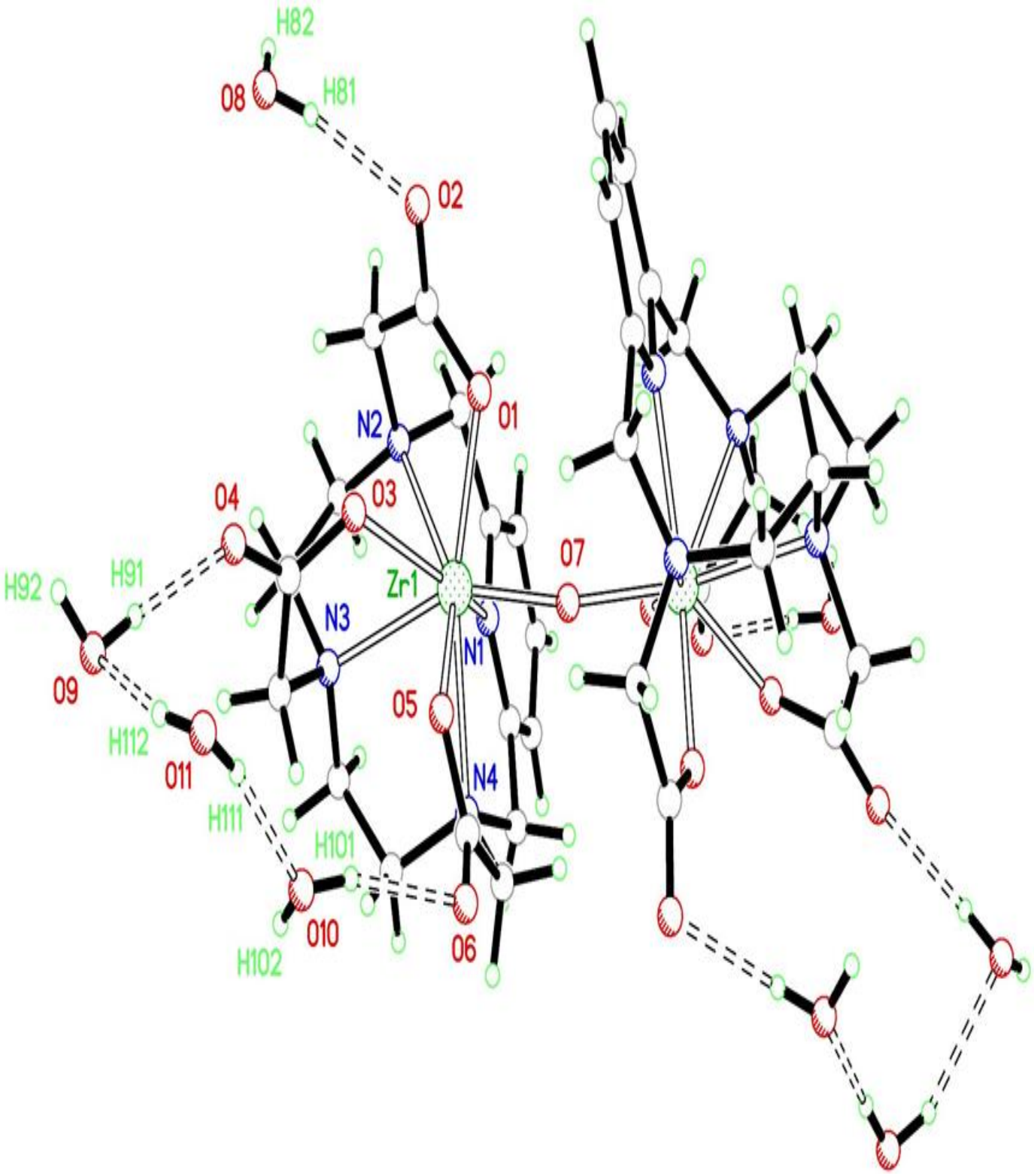


Figure S32. Plot of $[Zr(C_{17}H_{21}N_4O_6)]_2O$ - 8 H_2O with all atoms represented by dummy-sized spheres and hydrogen-bonding interactions represented by dashed-open bonds.

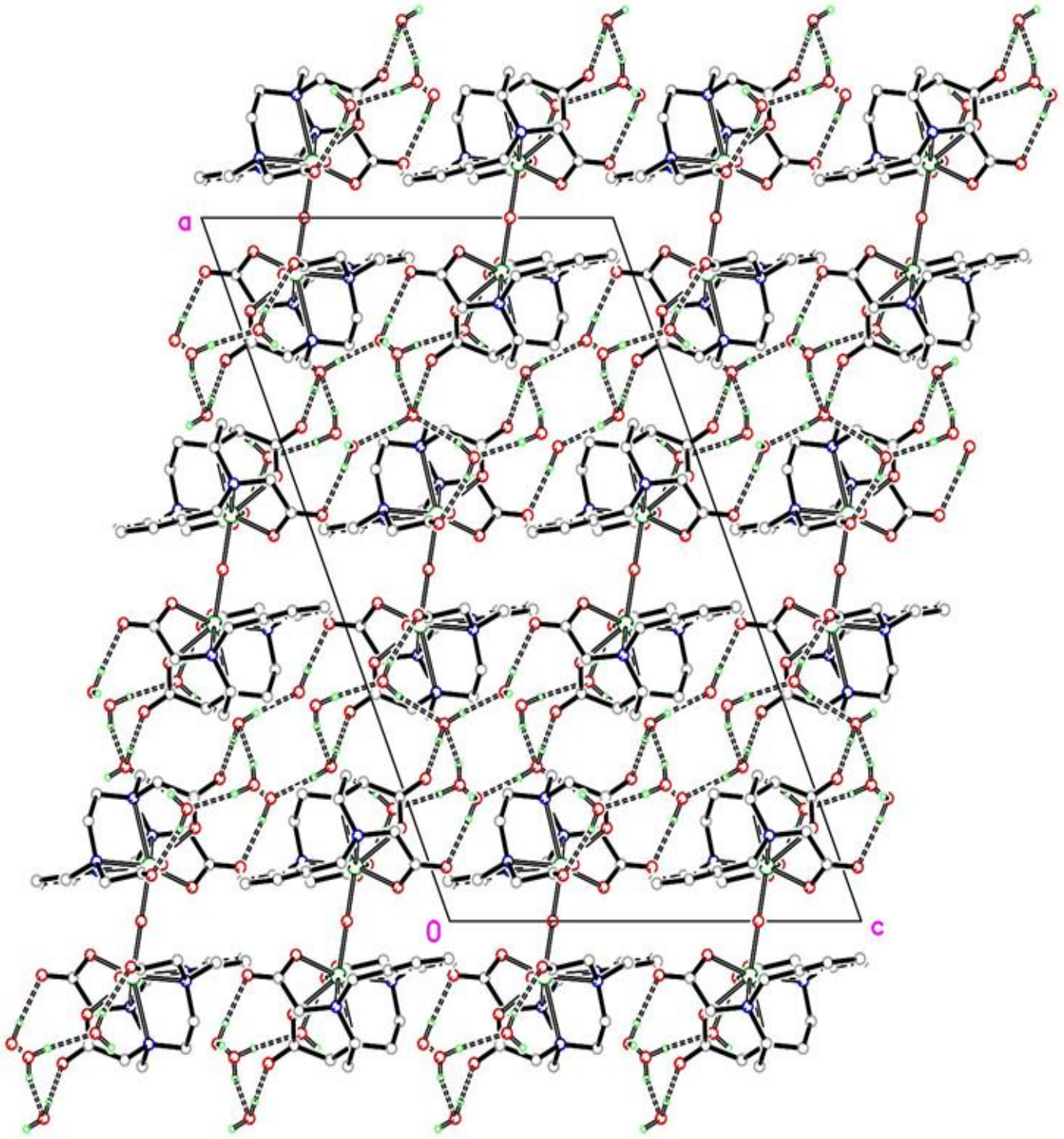


Figure S33. Packing plot of the solid-state structure of $[Zr(C_{17}H_{21}N_4O_6)]_2O$ - 8 H_2O viewed in projection down the b axis of the unit cell. All atoms are represented by dummy-sized spheres and non-water hydrogen atoms have been omitted for clarity. Hydrogen-bonding interactions represented by dashed-solid bonds.

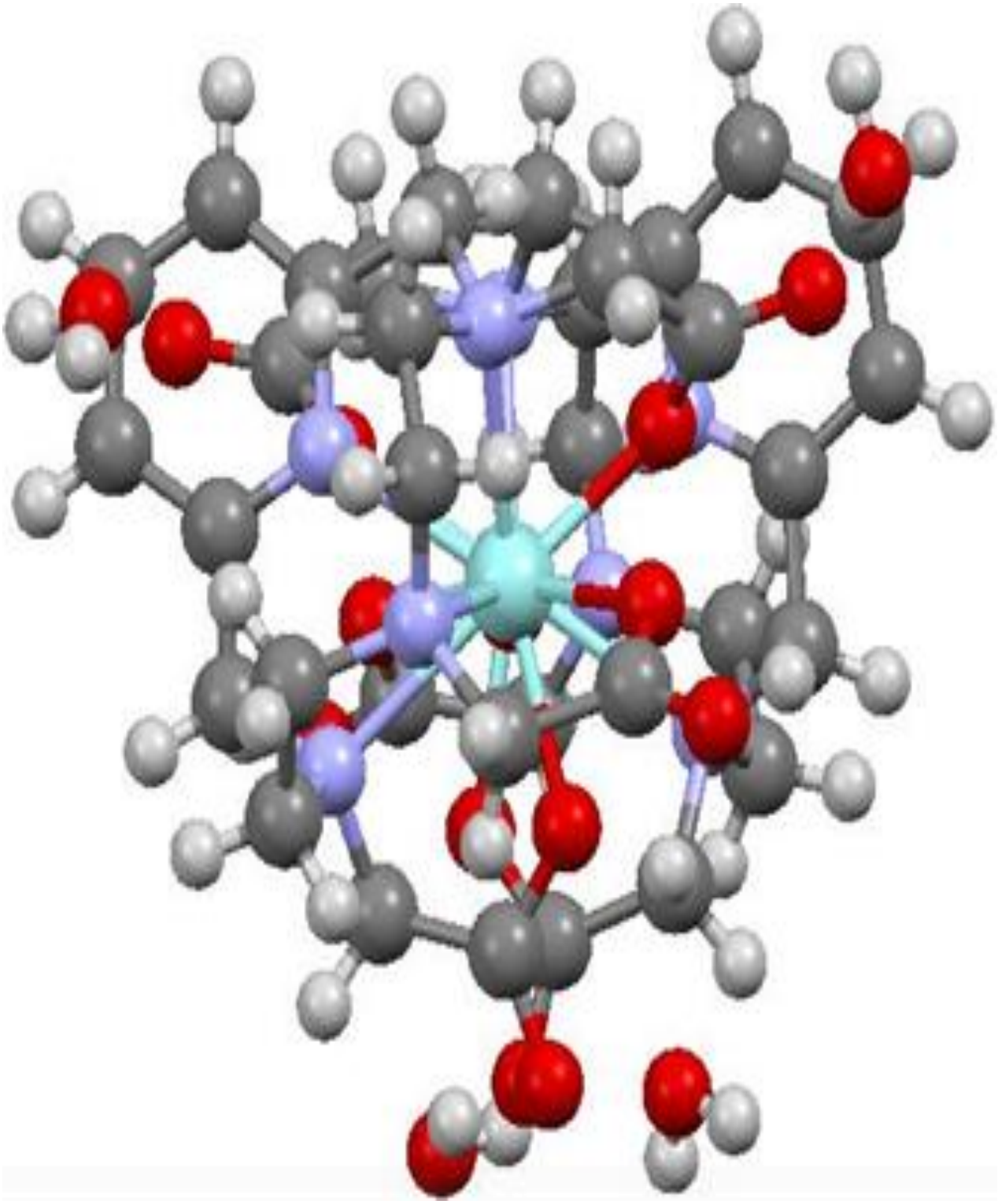


Figure S34. Plot of $[Zr(C_{17}H_{21}N_4O_6)]_2O$ - 8 H_2O, viewed down the Zr---Zr vector with all atoms represented by dummy-sized spheres.

Table S6. Crystal structure data collection parameters

Crystal data

$C_{34}H_{58}N_8O_{21}Zr_2$	$F(000) = 2264$
$M_r = 1097.32$	$D_x = 1.732$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 26.632$ (4) Å	Cell parameters from 9951 reflections
$b = 10.6634$ (17) Å	$\theta = 3.4–30.0°$
$c = 15.884$ (3) Å	$\mu = 0.59$ mm^{-1}
$\beta = 111.098$ (2)°	$T = 193$ K
$V = 4208.6$ (12) Å^3	Plate, colourless
$Z = 4$	0.46 × 0.10 × 0.03 mm

Data collection

Bruker APEX CCD diffractometer	6124 independent reflections
Radiation source: sealed tube	5433 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.047$
ϕ and ω scans	$\theta_{max} = 30.1°$, $\theta_{min} = 3.4°$
Absorption correction: multi-scan Data were corrected for scaling and absorption effects using the multi-scan technique (*SADABS*). The ratio of minimum to maximum apparent transmission was 0.895. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.773 and 0.983.	$h = -37 \rightarrow 37$
$T_{min} = 0.667$, $T_{max} = 0.746$	$k = -14 \rightarrow 14$
39418 measured reflections	$l = -22 \rightarrow 22$

Refinement

Refinement on F^2	12 restraints
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.023$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.063$	$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2 + 2.5169P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.002$
6124 reflections	$\Delta\rangle_{max} = 0.53$ e Å^{-3}
326 parameters	$\Delta\rangle_{min} = -0.31$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table S7. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zr1	0.42185 (2)	0.39753 (2)	0.18239 (2)	0.00952 (4)
O1	0.45174 (4)	0.28434 (8)	0.09702 (6)	0.01517 (17)
O2	0.42315 (4)	0.18849 (9)	-0.03754 (7)	0.0218 (2)
O3	0.36737 (4)	0.42299 (8)	0.03892 (6)	0.01564 (18)
O4	0.29752 (5)	0.51715 (12)	-0.06308 (7)	0.0342 (3)
O5	0.41950 (4)	0.59413 (8)	0.15041 (7)	0.01590 (18)
O6	0.43478 (5)	0.79600 (9)	0.18580 (7)	0.0258 (2)
O7	0.5000	0.41805 (11)	0.2500	0.0129 (2)
N1	0.42802 (4)	0.27938 (10)	0.31193 (7)	0.0141 (2)
N2	0.37848 (4)	0.19029 (10)	0.15047 (7)	0.0146 (2)
N3	0.32502 (4)	0.41969 (10)	0.16303 (7)	0.0147 (2)
N4	0.41500 (4)	0.53215 (10)	0.30742 (7)	0.0138 (2)
C1	0.43550 (5)	0.33262 (12)	0.39228 (9)	0.0162 (2)
C2	0.44272 (6)	0.26188 (14)	0.46930 (9)	0.0215 (3)
H2	0.4449	0.3014	0.5241	0.026*
C3	0.44668 (6)	0.13323 (15)	0.46466 (10)	0.0241 (3)
H3	0.4533	0.0833	0.5171	0.029*
C4	0.44087 (6)	0.07749 (13)	0.38275 (10)	0.0221 (3)
H4	0.4444	-0.0107	0.3785	0.027*
C5	0.42986 (5)	0.15307 (12)	0.30748 (9)	0.0166 (2)
C6	0.41690 (6)	0.10220 (11)	0.21398 (10)	0.0187 (3)
H6A	0.4501	0.0954	0.1998	0.022*
H6B	0.4006	0.0178	0.2090	0.022*
C7	0.37287 (6)	0.15697 (12)	0.05720 (9)	0.0176 (2)
H7A	0.3383	0.1894	0.0144	0.021*
H7B	0.3729	0.0646	0.0509	0.021*
C8	0.41929 (5)	0.21323 (11)	0.03532 (9)	0.0156 (2)

C9	0.32621 (5)	0.18499 (12)	0.16450 (9)	0.0179 (2)
H9A	0.3329	0.1771	0.2297	0.021*
H9B	0.3057	0.1104	0.1335	0.021*
C10	0.29368 (5)	0.30206 (12)	0.12784 (9)	0.0182 (2)
H10A	0.2816	0.3015	0.0612	0.022*
H10B	0.2612	0.3016	0.1444	0.022*
C11	0.30270 (5)	0.51515 (12)	0.09072 (9)	0.0182 (2)
H11A	0.3145	0.6001	0.1149	0.022*
H11B	0.2629	0.5126	0.0676	0.022*
C12	0.32331 (6)	0.48514 (12)	0.01498 (9)	0.0182 (2)
C13	0.31933 (5)	0.45841 (12)	0.24909 (9)	0.0169 (2)
H13A	0.3272	0.3862	0.2910	0.020*
H13B	0.2819	0.4856	0.2372	0.020*
C14	0.35762 (5)	0.56506 (12)	0.29194 (9)	0.0169 (2)
H14A	0.3472	0.6396	0.2523	0.020*
H14B	0.3544	0.5871	0.3504	0.020*
C15	0.44408 (6)	0.64965 (12)	0.30442 (9)	0.0177 (2)
H15A	0.4833	0.6369	0.3350	0.021*
H15B	0.4327	0.7173	0.3363	0.021*
C16	0.43185 (5)	0.68670 (11)	0.20747 (9)	0.0163 (2)
C17	0.44095 (6)	0.47195 (12)	0.39679 (8)	0.0184 (3)
H17A	0.4244	0.5050	0.4390	0.022*
H17B	0.4796	0.4943	0.4209	0.022*
O8	0.32558 (5)	0.09800 (12)	-0.17010 (9)	0.0361 (3)
H81	0.3553 (5)	0.1162 (18)	-0.1327 (12)	0.043 (6)*
H82	0.3235 (10)	0.0224 (10)	-0.1609 (17)	0.064 (8)*
O9	0.21960 (5)	0.68121 (11)	-0.15600 (8)	0.0306 (2)
H91	0.2423 (7)	0.6294 (17)	-0.1299 (12)	0.045 (6)*
H92	0.2044 (7)	0.6564 (18)	-0.2071 (7)	0.034 (5)*

O10	0.34067 (6)	0.84749 (13)	0.04255 (9)	0.0370 (3)
H101	0.3696 (5)	0.824 (2)	0.0776 (13)	0.054 (7)*
H102	0.3198 (7)	0.836 (2)	0.0690 (15)	0.057 (7)*
O11	0.30641 (5)	0.84782 (12)	-0.14118 (9)	0.0330 (3)
H111	0.3190 (8)	0.840 (2)	-0.0863 (6)	0.043 (6)*
H112	0.2771 (5)	0.814 (2)	-0.1565 (14)	0.049 (7)*

Table S8. Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zr1	0.00958 (6)	0.01080 (6)	0.00801 (6)	0.00027 (4)	0.00296 (4)	-0.00017 (3)
O1	0.0130 (4)	0.0175 (4)	0.0151 (4)	0.0003 (3)	0.0051 (3)	-0.0040 (3)
O2	0.0247 (5)	0.0266 (5)	0.0160 (5)	-0.0040 (4)	0.0096 (4)	-0.0071 (4)
O3	0.0152 (5)	0.0196 (4)	0.0118 (4)	0.0037 (3)	0.0044 (4)	0.0007 (3)
O4	0.0333 (6)	0.0523 (7)	0.0144 (5)	0.0219 (5)	0.0053 (5)	0.0083 (5)
O5	0.0187 (5)	0.0141 (4)	0.0147 (5)	0.0014 (3)	0.0057 (4)	0.0009 (3)
O6	0.0315 (6)	0.0151 (4)	0.0296 (6)	-0.0015 (4)	0.0093 (5)	0.0012 (4)
O7	0.0120 (6)	0.0141 (5)	0.0120 (6)	0.000	0.0037 (5)	0.000
N1	0.0128 (5)	0.0171 (5)	0.0127 (5)	-0.0004 (4)	0.0049 (4)	0.0021 (4)
N2	0.0154 (5)	0.0155 (5)	0.0133 (5)	-0.0019 (4)	0.0055 (4)	-0.0013 (4)
N3	0.0134 (5)	0.0184 (5)	0.0127 (5)	0.0006 (4)	0.0052 (4)	-0.0010 (4)
N4	0.0135 (5)	0.0162 (5)	0.0118 (5)	-0.0009 (4)	0.0047 (4)	-0.0015 (4)
C1	0.0124 (6)	0.0227 (6)	0.0130 (6)	0.0003 (4)	0.0041 (5)	0.0017 (4)
C2	0.0185 (7)	0.0334 (7)	0.0122 (6)	-0.0025 (5)	0.0050 (5)	0.0041 (5)
C3	0.0200 (7)	0.0324 (7)	0.0176 (7)	-0.0025 (5)	0.0038 (5)	0.0113 (5)
C4	0.0187 (7)	0.0220 (6)	0.0244 (7)	-0.0004 (5)	0.0064 (6)	0.0086 (5)
C5	0.0144 (6)	0.0176 (6)	0.0173 (6)	-0.0005 (4)	0.0052 (5)	0.0034 (4)
C6	0.0239 (7)	0.0129 (5)	0.0192 (7)	0.0020 (4)	0.0075 (5)	0.0014 (4)
C7	0.0197 (6)	0.0181 (6)	0.0152 (6)	-0.0038 (5)	0.0064 (5)	-0.0053 (4)
C8	0.0156 (6)	0.0156 (5)	0.0148 (6)	0.0016 (4)	0.0047 (5)	-0.0017 (4)

C9	0.0169 (6)	0.0200 (6)	0.0181 (6)	-0.0055 (5)	0.0082 (5)	-0.0018 (5)
C10	0.0124 (6)	0.0241 (6)	0.0174 (6)	-0.0033 (5)	0.0045 (5)	-0.0033 (5)
C11	0.0158 (6)	0.0229 (6)	0.0148 (6)	0.0065 (5)	0.0044 (5)	0.0015 (5)
C12	0.0179 (6)	0.0207 (6)	0.0146 (6)	0.0037 (5)	0.0044 (5)	0.0004 (4)
C13	0.0139 (6)	0.0239 (6)	0.0148 (6)	0.0008 (4)	0.0074 (5)	-0.0010 (4)
C14	0.0159 (6)	0.0205 (6)	0.0159 (6)	0.0025 (5)	0.0077 (5)	-0.0028 (5)
C15	0.0204 (6)	0.0158 (5)	0.0184 (6)	-0.0034 (5)	0.0088 (5)	-0.0054 (4)
C16	0.0139 (6)	0.0145 (5)	0.0206 (6)	0.0012 (4)	0.0064 (5)	-0.0003 (4)
C17	0.0217 (7)	0.0222 (6)	0.0095 (6)	0.0006 (5)	0.0035 (5)	-0.0016 (4)
O8	0.0294 (7)	0.0374 (7)	0.0299 (7)	-0.0040 (5)	-0.0034 (5)	0.0022 (5)
O9	0.0272 (6)	0.0316 (6)	0.0269 (6)	0.0093 (5)	0.0024 (5)	0.0008 (5)
O10	0.0359 (7)	0.0451 (7)	0.0289 (7)	0.0038 (6)	0.0103 (6)	0.0094 (5)
O11	0.0315 (7)	0.0420 (7)	0.0271 (6)	-0.0052 (5)	0.0125 (5)	0.0011 (5)

Table S9. Geometric parameters (Å, º)

Zr1—O7	1.9809 (3)	C4—H4	0.9500
Zr1—O5	2.1527 (9)	C5—C6	1.5000 (19)
Zr1—O1	2.1695 (9)	C6—H6A	0.9900
Zr1—O3	2.2343 (10)	C6—H6B	0.9900
Zr1—N1	2.3674 (11)	C7—C8	1.5228 (18)
Zr1—N2	2.4603 (11)	C7—H7A	0.9900
Zr1—N3	2.4959 (12)	C7—H7B	0.9900
Zr1—N4	2.5097 (11)	C9—C10	1.5115 (19)
O1—C8	1.2932 (15)	C9—H9A	0.9900
O2—C8	1.2269 (16)	C9—H9B	0.9900
O3—C12	1.2805 (16)	C10—H10A	0.9900
O4—C12	1.2298 (17)	C10—H10B	0.9900
O5—C16	1.2998 (15)	C11—C12	1.5253 (18)

O6—C16	1.2257 (16)	C11—H11A	0.9900
O7—Zr1^{i}	1.9810 (3)	C11—H11B	0.9900
N1—C1	1.3445 (17)	C13—C14	1.5154 (19)
N1—C5	1.3506 (16)	C13—H13A	0.9900
N2—C7	1.4779 (16)	C13—H13B	0.9900
N2—C6	1.4845 (17)	C14—H14A	0.9900
N2—C9	1.4879 (16)	C14—H14B	0.9900
N3—C13	1.4870 (16)	C15—C16	1.5076 (19)
N3—C11	1.4890 (17)	C15—H15A	0.9900
N3—C10	1.4988 (17)	C15—H15B	0.9900
N4—C15	1.4827 (16)	C17—H17A	0.9900
N4—C17	1.4829 (17)	C17—H17B	0.9900
N4—C14	1.4995 (17)	O8—H81	0.823 (9)
C1—C2	1.3905 (18)	O8—H82	0.824 (9)
C1—C17	1.4920 (19)	O9—H91	0.813 (9)
C2—C3	1.380 (2)	O9—H92	0.811 (9)
C2—H2	0.9500	O10—H101	0.811 (9)
C3—C4	1.388 (2)	O10—H102	0.816 (9)
C3—H3	0.9500	O11—H111	0.818 (9)
C4—C5	1.3830 (18)	O11—H112	0.813 (9)
O7—Zr1—O5	87.48 (4)	N2—C6—C5	107.50 (10)
O7—Zr1—O1	81.37 (3)	N2—C6—H6A	110.2
O5—Zr1—O1	112.35 (4)	C5—C6—H6A	110.2
O7—Zr1—O3	135.18 (3)	N2—C6—H6B	110.2
O5—Zr1—O3	71.81 (4)	C5—C6—H6B	110.2
O1—Zr1—O3	71.32 (4)	H6A—C6—H6B	108.5
O7—Zr1—N1	81.77 (3)	N2—C7—C8	109.67 (10)
O5—Zr1—N1	135.27 (4)	N2—C7—H7A	109.7
O1—Zr1—N1	108.72 (4)	C8—C7—H7A	109.7

O3—Zr1—N1	140.14 (4)	N2—C7—H7B	109.7
O7—Zr1—N2	122.32 (4)	C8—C7—H7B	109.7
O5—Zr1—N2	148.70 (4)	H7A—C7—H7B	108.2
O1—Zr1—N2	67.80 (4)	O2—C8—O1	125.52 (12)
O3—Zr1—N2	79.32 (4)	O2—C8—C7	118.76 (11)
N1—Zr1—N2	65.27 (4)	O1—C8—C7	115.72 (11)
O7—Zr1—N3	153.34 (3)	N2—C9—C10	110.38 (10)
O5—Zr1—N3	86.43 (4)	N2—C9—H9A	109.6
O1—Zr1—N3	124.82 (3)	C10—C9—H9A	109.6
O3—Zr1—N3	66.43 (4)	N2—C9—H9B	109.6
N1—Zr1—N3	84.43 (4)	C10—C9—H9B	109.6
N2—Zr1—N3	70.86 (4)	H9A—C9—H9B	108.1
O7—Zr1—N4	82.49 (3)	N3—C10—C9	112.50 (11)
O5—Zr1—N4	68.01 (4)	N3—C10—H10A	109.1
O1—Zr1—N4	163.82 (4)	C9—C10—H10A	109.1
O3—Zr1—N4	122.15 (4)	N3—C10—H10B	109.1
N1—Zr1—N4	67.59 (4)	C9—C10—H10B	109.1
N2—Zr1—N4	120.96 (4)	H10A—C10—H10B	107.8
N3—Zr1—N4	71.17 (4)	N3—C11—C12	107.94 (10)
C8—O1—Zr1	120.30 (8)	N3—C11—H11A	110.1
C12—O3—Zr1	123.71 (8)	C12—C11—H11A	110.1
C16—O5—Zr1	126.62 (8)	N3—C11—H11B	110.1
Zr1—O7—Zr1^{i}	167.32 (7)	C12—C11—H11B	110.1
C1—N1—C5	118.36 (11)	H11A—C11—H11B	108.4
C1—N1—Zr1	122.72 (8)	O4—C12—O3	124.23 (13)
C5—N1—Zr1	118.53 (8)	O4—C12—C11	120.54 (12)
C7—N2—C6	108.98 (10)	O3—C12—C11	115.20 (11)
C7—N2—C9	112.25 (10)	N3—C13—C14	110.28 (10)
C6—N2—C9	109.10 (10)	N3—C13—H13A	109.6

C7—N2—Zr1	107.05 (7)	C14—C13—H13A	109.6
C6—N2—Zr1	105.70 (7)	N3—C13—H13B	109.6
C9—N2—Zr1	113.48 (7)	C14—C13—H13B	109.6
C13—N3—C11	112.01 (10)	H13A—C13—H13B	108.1
C13—N3—C10	110.36 (10)	N4—C14—C13	112.29 (10)
C11—N3—C10	105.69 (10)	N4—C14—H14A	109.1
C13—N3—Zr1	110.83 (8)	C13—C14—H14A	109.1
C11—N3—Zr1	105.52 (8)	N4—C14—H14B	109.1
C10—N3—Zr1	112.24 (8)	C13—C14—H14B	109.1
C15—N4—C17	109.13 (10)	H14A—C14—H14B	107.9
C15—N4—C14	108.08 (10)	N4—C15—C16	109.33 (10)
C17—N4—C14	110.76 (10)	N4—C15—H15A	109.8
C15—N4—Zr1	105.69 (7)	C16—C15—H15A	109.8
C17—N4—Zr1	111.62 (7)	N4—C15—H15B	109.8
C14—N4—Zr1	111.36 (7)	C16—C15—H15B	109.8
N1—C1—C2	122.17 (12)	H15A—C15—H15B	108.3
N1—C1—C17	116.40 (11)	O6—C16—O5	123.51 (13)
C2—C1—C17	121.18 (12)	O6—C16—C15	121.59 (12)
C3—C2—C1	118.77 (13)	O5—C16—C15	114.88 (11)
C3—C2—H2	120.6	N4—C17—C1	112.42 (10)
C1—C2—H2	120.6	N4—C17—H17A	109.1
C2—C3—C4	119.41 (13)	C1—C17—H17A	109.1
C2—C3—H3	120.3	N4—C17—H17B	109.1
C4—C3—H3	120.3	C1—C17—H17B	109.1
C5—C4—C3	118.58 (13)	H17A—C17—H17B	107.9
C5—C4—H4	120.7	H81—O8—H82	102.1 (17)
C3—C4—H4	120.7	H91—O9—H92	107.4 (17)
N1—C5—C4	122.43 (13)	H101—O10—H102	104.9 (18)
N1—C5—C6	114.38 (11)	H111—O11—H112	104.7 (17)

C4—C5—C6	123.15 (12)	C11—N3—C10—C9	150.29 (11)
C5—N1—C1—C2	-2.78 (19)	Zr1—N3—C10—C9	35.76 (13)
Zr1—N1—C1—C2	-175.53 (10)	N2—C9—C10—N3	-51.90 (14)
C5—N1—C1—C17	171.55 (11)	C13—N3—C11—C12	165.15 (11)
Zr1—N1—C1—C17	-1.20 (16)	C10—N3—C11—C12	-74.62 (13)
N1—C1—C2—C3	5.4 (2)	Zr1—N3—C11—C12	44.47 (11)
C17—C1—C2—C3	-168.69 (13)	Zr1—O3—C12—O4	167.64 (12)
C1—C2—C3—C4	-3.0 (2)	Zr1—O3—C12—C11	-14.25 (16)
C2—C3—C4—C5	-1.6 (2)	N3—C11—C12—O4	153.59 (13)
C1—N1—C5—C4	-2.18 (19)	N3—C11—C12—O3	-24.60 (16)
Zr1—N1—C5—C4	170.88 (10)	C11—N3—C13—C14	-71.27 (13)
C1—N1—C5—C6	175.56 (12)	C10—N3—C13—C14	171.26 (10)
Zr1—N1—C5—C6	-11.38 (15)	Zr1—N3—C13—C14	46.29 (12)
C3—C4—C5—N1	4.4 (2)	C15—N4—C14—C13	152.02 (11)
C3—C4—C5—C6	-173.17 (13)	C17—N4—C14—C13	-88.48 (13)
C7—N2—C6—C5	169.20 (10)	Zr1—N4—C14—C13	36.36 (12)
C9—N2—C6—C5	-67.92 (13)	N3—C13—C14—N4	-56.36 (14)
Zr1—N2—C6—C5	54.44 (11)	C17—N4—C15—C16	160.02 (10)
N1—C5—C6—N2	-30.48 (16)	C14—N4—C15—C16	-79.45 (12)
C4—C5—C6—N2	147.25 (13)	Zr1—N4—C15—C16	39.86 (11)
C6—N2—C7—C8	-82.07 (12)	Zr1—O5—C16—O6	172.59 (10)
C9—N2—C7—C8	156.95 (10)	Zr1—O5—C16—C15	-5.77 (16)
Zr1—N2—C7—C8	31.81 (11)	N4—C15—C16—O6	154.58 (12)
Zr1—O1—C8—O2	147.14 (11)	N4—C15—C16—O5	-27.02 (15)
Zr1—O1—C8—C7	-32.89 (14)	C15—N4—C17—C1	-150.19 (11)
N2—C7—C8—O2	175.40 (11)	C14—N4—C17—C1	90.95 (12)
N2—C7—C8—O1	-4.57 (15)	Zr1—N4—C17—C1	-33.74 (13)
C7—N2—C9—C10	-79.60 (13)	N1—C1—C17—N4	24.35 (16)
C6—N2—C9—C10	159.49 (10)	C2—C1—C17—N4	-161.26 (12)

Zr1—N2—C9—C10	41.93 (12)		
C13—N3—C10—C9	-88.40 (13)		

Symmetry code: (i) -*x*+1, *y*, -*z*+1/2.

Table S10. Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O5^{ii}	0.95	2.59	3.5066 (18)	163
C4—H4···O2^{iii}	0.95	2.50	3.2094 (17)	132
C6—H6*B*···O6^{iv}	0.99	2.61	3.3529 (16)	132
C7—H7*B*···O10^{iv}	0.99	2.46	3.396 (2)	158
C9—H9*A*···O11^{ii}	0.99	2.41	3.3316 (19)	155
C14—H14*B*···O4^{ii}	0.99	2.63	3.3596 (18)	131
C15—H15*A*···O5^{i}	0.99	2.56	3.4912 (18)	158
C15—H15*B*···O2^{ii}	0.99	2.34	3.2556 (17)	154
O8—H81···O2	0.82 (1)	2.04 (1)	2.8598 (16)	171 (2)
O8—H82···O11^{iv}	0.82 (1)	1.97 (1)	2.7852 (18)	171 (2)
O9—H91···O4	0.81 (1)	1.90 (1)	2.7093 (16)	176 (2)
O9—H92···O8^{v}	0.81 (1)	1.93 (1)	2.7335 (18)	175 (2)
O10—H101···O6	0.81 (1)	1.98 (1)	2.7669 (18)	164 (2)
O10—H102···O9^{vi}	0.82 (1)	2.02 (1)	2.8285 (19)	169 (2)
O11—H111···O10	0.82 (1)	1.92 (1)	2.7262 (18)	170 (2)
O11—H112···O9	0.81 (1)	2.09 (1)	2.8574 (18)	158 (2)

Symmetry codes: (i) -*x*+1, *y*, -*z*+1/2; (ii) *x*, -*y*+1, *z*+1/2; (iii) *x*, -*y*, *z*+1/2; (iv) *x*, *y*-1, *z*; (v) -*x*+1/2, *y*+1/2, -*z*-1/2; (vi) -*x*+1/2, -*y*+3/2, -*z*.

Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane)

21.3519 (0.0087) x + 3.2726 (0.0041) y - 12.1847 (0.0048) z = 6.5766 (0.0038)

* -0.3241 (0.0006) N1

* 0.2939 (0.0006) N2

* -0.2499 (0.0005) N3

* 0.2802 (0.0005) N4

1.5091 (0.0006) Zr1

2.8172 (0.0013) O1

2.1775 (0.0015) O3

2.4921 (0.0014) O5

2.4212 (0.0011) O7

Rms deviation of fitted atoms = 0.2883

19.8900 (0.0061) x + 4.1453 (0.0038) y - 12.2662 (0.0037) z = 8.7824 (0.0028)

Angle to previous plane (with approximate esd) = 5.852 (0.053)

* 0.1912 (0.0005) O1

* -0.1994 (0.0005) O3

* 0.1793 (0.0004) O5

* -0.1711 (0.0004) O7

-0.9813 (0.0004) Zr1

-2.9373 (0.0012) N1

-2.3114 (0.0014) N2

-2.5779 (0.0014) N3

-2.0929 (0.0014) N4

Rms deviation of fitted atoms = 0.1855

Crystal Structure Analysis of Zr-NOTA, $[Zr(C_{12}H_{18}N_3O_6)(OH)]_2$ - 6 H_2O

Crystallographic Experimental Details:

Data Collection and Structure Solution. A clear colorless plate-like specimen of $[Zr(C_{12}H_{18}N_3O_6)(OH)]_2$ - 6 H_2O, approximate dimensions 0.080 mm x 0.300 mm x 0.460 mm, was used for the X-ray crystallographic analysis. The X-ray intensity data were measured on a Bruker APEX CCD system equipped with a graphite monochromator and a Mo K_α sealed x-ray tube (λ = 0.71073 Å). X-rays were provided by a fine-focus sealed x-ray tube operated at 50kV and 30mA.

The total exposure time was 18.73 hours. The frames were integrated with the Bruker SAINT Software[1] package using a narrow-frame algorithm. The integration of the data using a monoclinic unit cell yielded a total of 31714 reflections to a maximum θ angle of 30.50° (0.70 Å resolution), of which 5078 were independent (average redundancy 6.245, completeness = 99.8%, R_{int} = 3.13%, R_{sig} = 1.83%) and 4778 (94.09%) were greater than $2\sigma(F^2)$. The final cell constants of a = 11.7960 (7) Å, b = 10.9411 (6) Å, c = 14.1815 (8) Å, β = 114.2100 (10)°, volume = 1669.31 (16) $Å^3$, are based upon the refinement of the XYZ-centroids of 9847 reflections above 20 σ(I) with 7.32° < 2θ < 62.98°. Data were corrected for absorption effects using the multi-scan method (SADABS)[2]. The ratio of minimum to maximum apparent transmission was 0.909. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.7330 and 0.9450.

The structure was solved and refined using the Bruker SHELXTL Software Package[3], using the space group $P2_1/n$, with Z = 2 for the formula unit, $C_{24}H_{50}N_6O_{20}Zr_2$. The final model includes 6 full-occupancy water molecules per dimeric unit. The arrangement of three CH2 groups about nitrogen atom N3 shows a slight rotational disorder (93/7%) about the Zr-N3 bond. The carbon atoms for the major orientation are designated by carbons C8, C10 and C11 and their associated hydrogen atoms. The carbon atoms for the minor orientation are designated by carbons C8', C10' and C11' and their associated hydrogens. The final structural model incorporated isotropic thermal parameters for the carbon atoms of the minor orientation and for all included hydrogen atoms. The hydrogen atoms of the “NOTA” ligand were included in the structural model as fixed atoms (using idealized sp^3-hybridized geometry and C-H bond lengths of 0.99Å) "riding" on their respective carbon atoms. The isotropic thermal parameters for all “NOTA” hydrogen atoms were fixed at values 1.2 times the equivalent isotropic thermal parameter of the carbon atom to which they are covalently bonded. The isotropic thermal parameters of the

water hydrogen atoms were allowed to refine independently. The final anisotropic full-matrix least-squares refinement on F^2 with 276 variables converged at R_1 = 1.92%, for the observed data and wR_2 = 5.18% for all data. The goodness-of-fit was 1.030. The largest peak in the final difference electron density synthesis was 0.521 $e^-/Å^3$ and the largest hole was -0.240 $e^-/Å^3$ with an RMS deviation of 0.060 $e^-/Å^3$. On the basis of the final model, the calculated density was 1.841 g/cm^3 and F(000), 952 e^-.

Refinement Details. Crystal data, data collection and structure refinement details are summarized in tables below.

Computing details. Data collection: Bruker *APEX3* v2016.1-0; cell refinement: Bruker *APEX3* v2016.1-0; data reduction: Bruker *APEX2* v2014.11-0; program(s) used to solve structure: *SHELXS97* (Sheldrick 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2014); molecular graphics: Bruker *APEX2* v2014.11-0; software used to prepare material for publication: Bruker *APEX2* v2014.11-0[1-6].

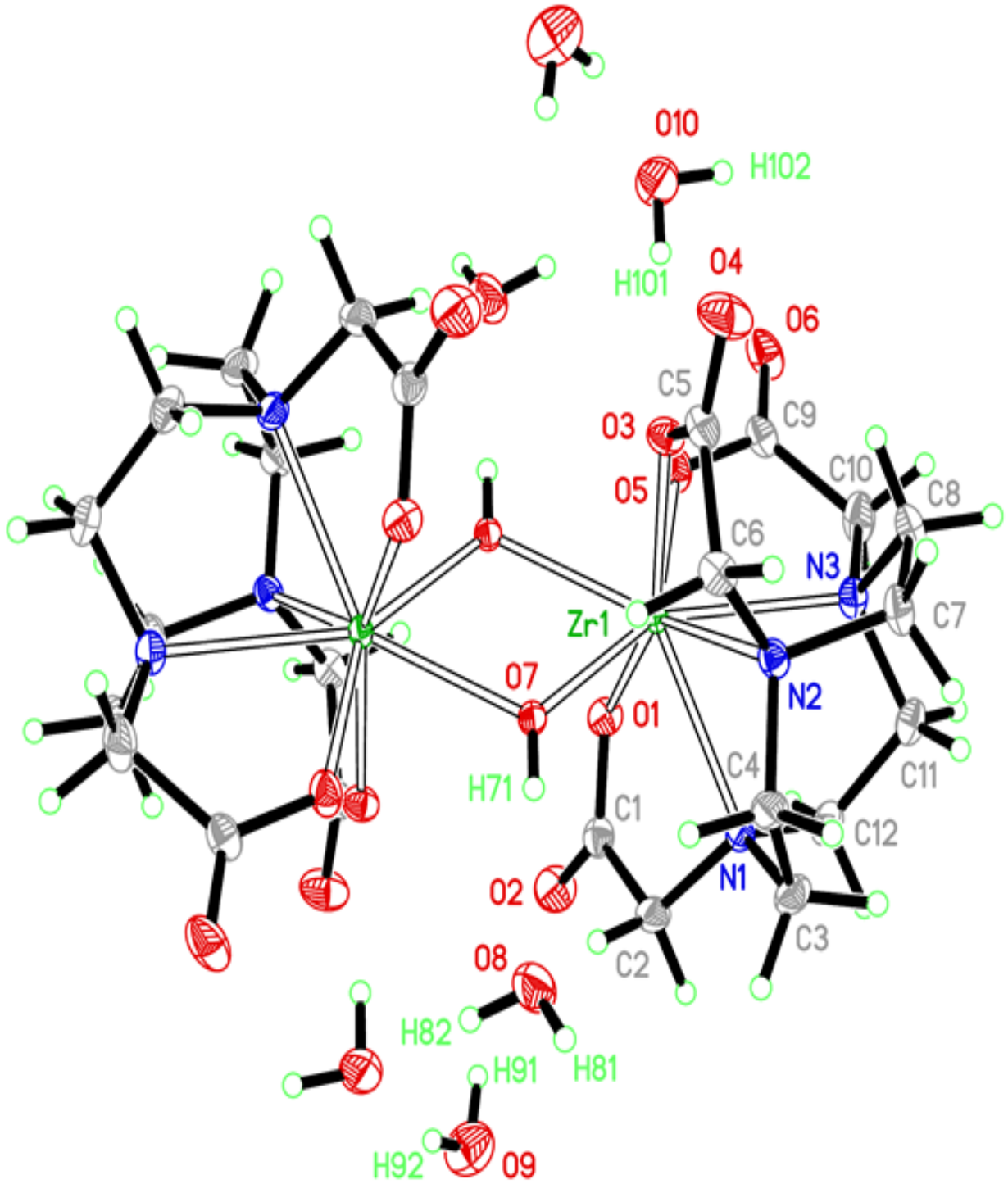


Figure S35. 50% probability plot for the dimeric unit of $[Zr(C_{12}H_{18}N_3O_6)(OH)]_2$ - 6 H_2O with disordered atoms C8', C10' and C11' omitted for clarity. The two halves of the dimer are related by the crystallographic inversion center at ½, ½, 0 in the unit cell. Only the non-hydrogen atoms of the asymmetric unit are labeled.

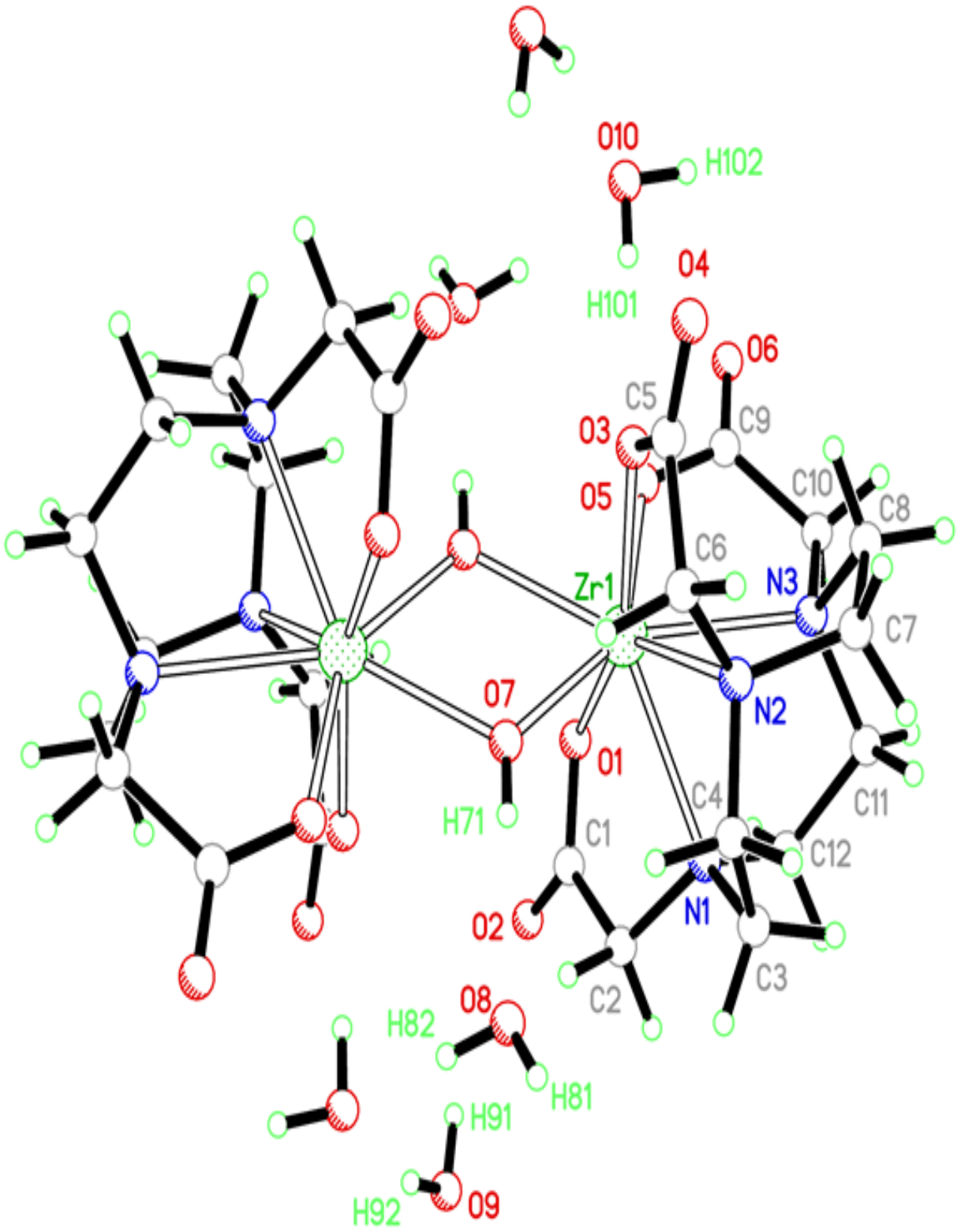


Figure S36. Plot of $[Zr(C_{12}H_{18}N_3O_6)(OH)]_2$ - 6 H_2O with disordered atoms C8', C10' and C11' omitted for clarity. All atoms represented by arbitrary-sized spheres.

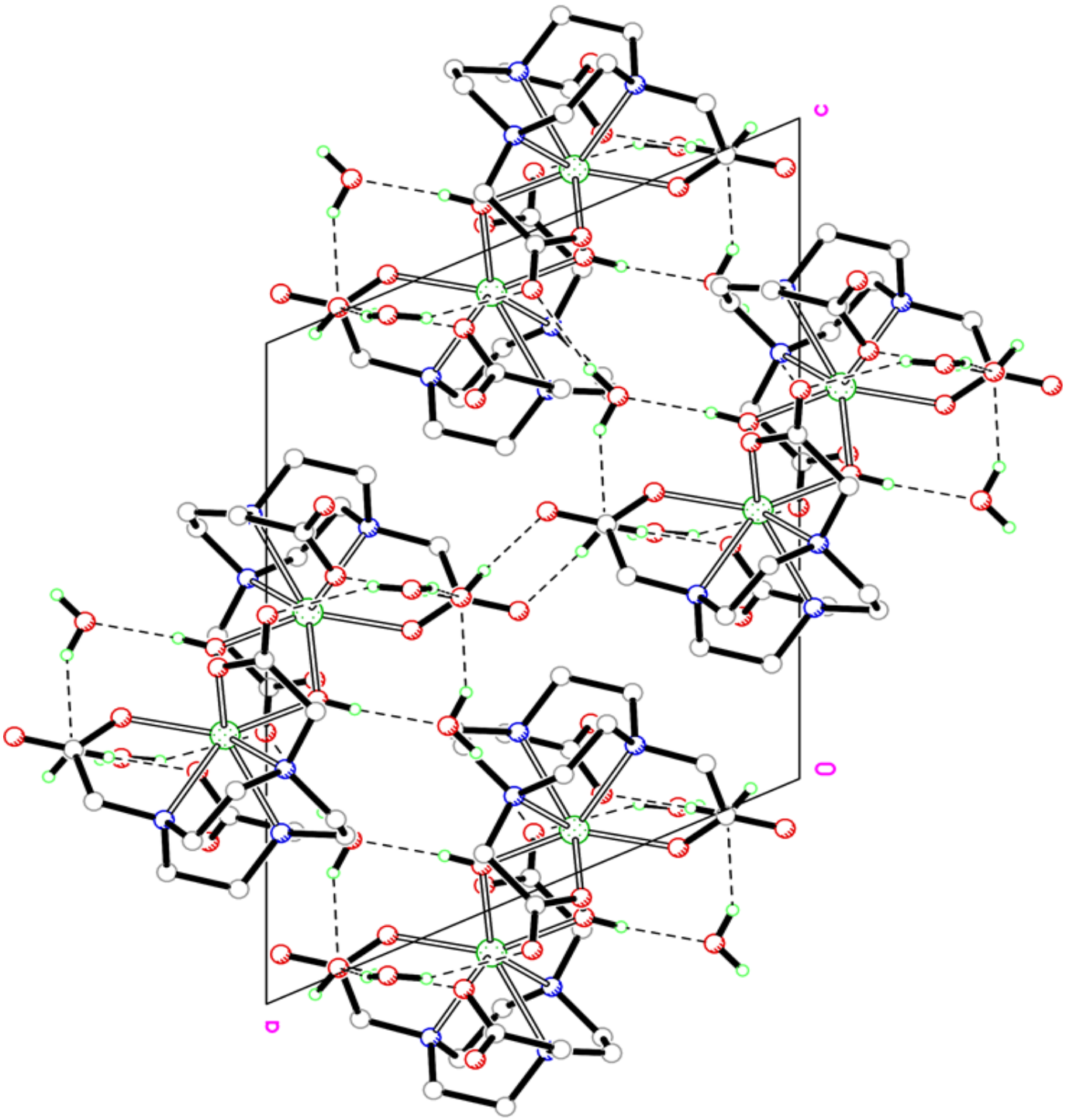


Figure S37. Packing plot of the solid-state structure of $[Zr(C_{12}H_{18}N_3O_6)(OH)]_2$ - 6 H_2O viewed in projection down the *b* axis of the unit cell showing the major orientation (93%). All atoms are represented by dummy-sized spheres and ligand hydrogen atoms have been omitted for clarity. Hydrogen-bonding interactions are represented by dashed-solid bonds.

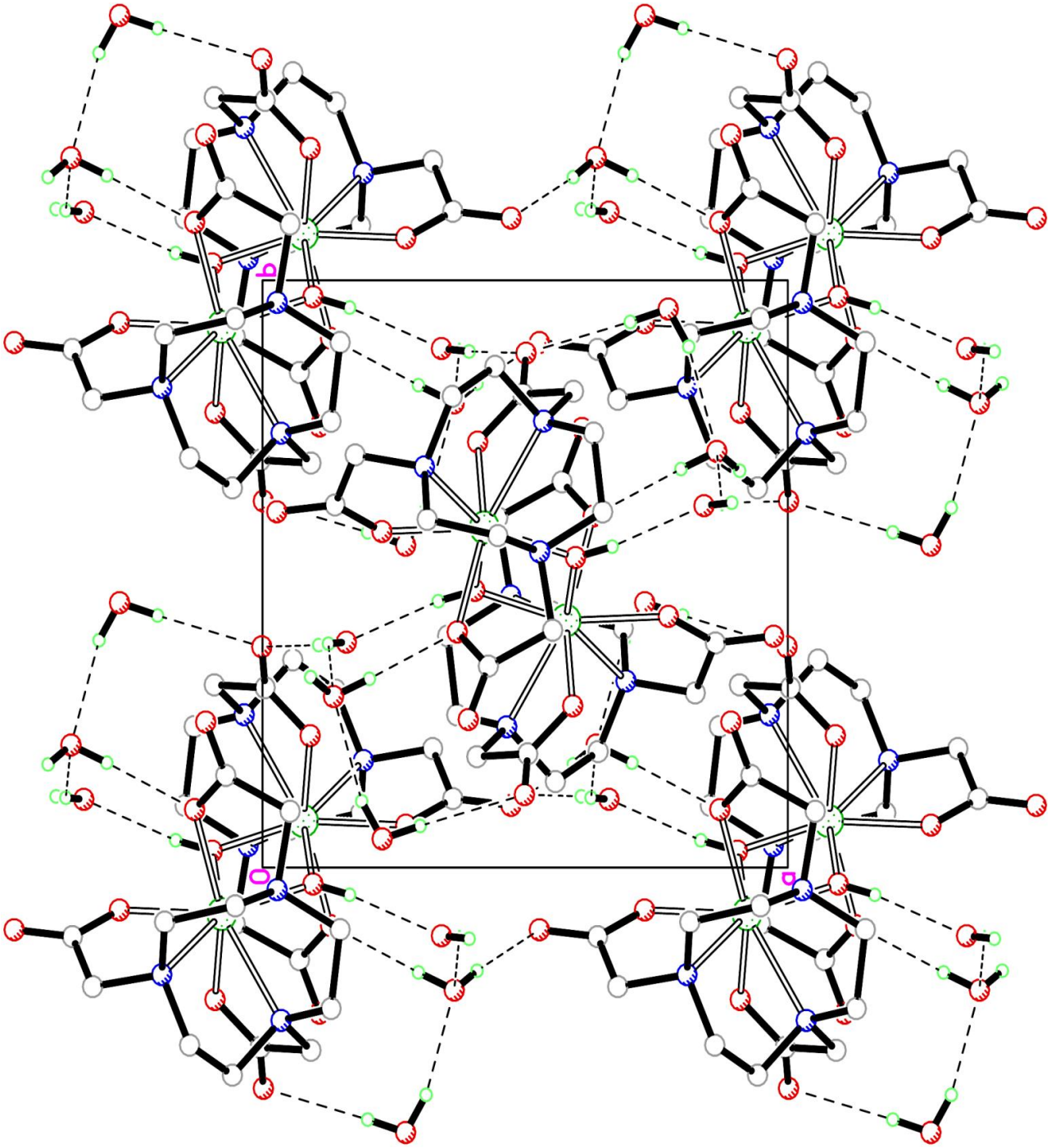


Figure S38. Packing plot of the solid-state structure of $[Zr(C_{12}H_{18}N_3O_6)(OH)]_2$ - 6 H_2O viewed in projection down the *c* axis of the unit cell showing the major orientation (93%). All atoms are represented by dummy-sized spheres and ligand hydrogen atoms have been omitted for clarity. Hydrogen-bonding interactions are represented by dashed-solid bonds.

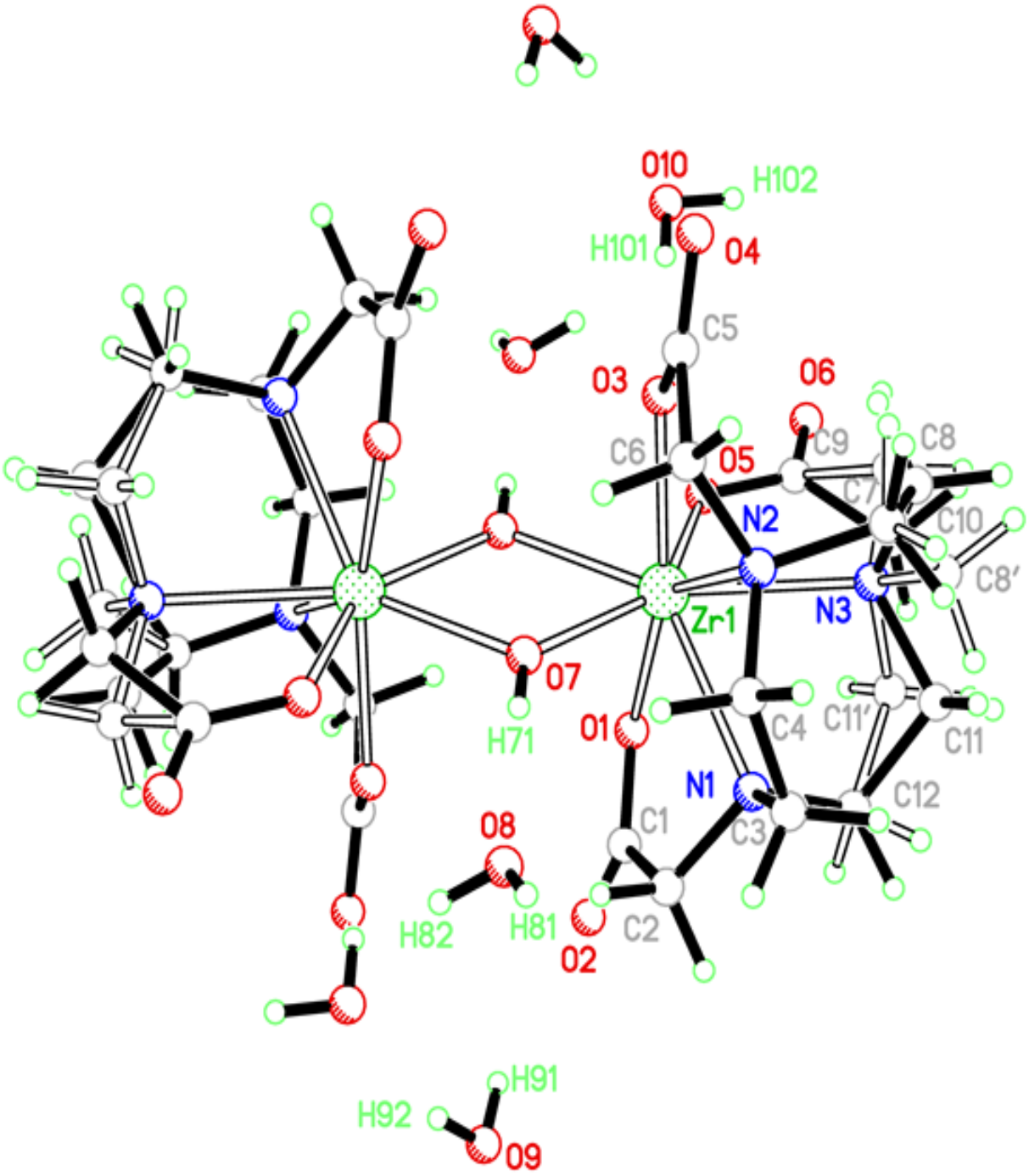


Figure S39. Plot of $[Zr(C_{12}H_{18}N_3O_6)(OH)]_2$ - 6 H_2O showing the disordered arrangement of carbon atoms about the Zr1-N3 bond with the major (93%) orientation designated by carbon atoms C8, C10 and C11 and the minor (7%) orientation designated by carbon atoms C8', C10' and C11'. All atoms are represented by arbitrary-sized spheres.

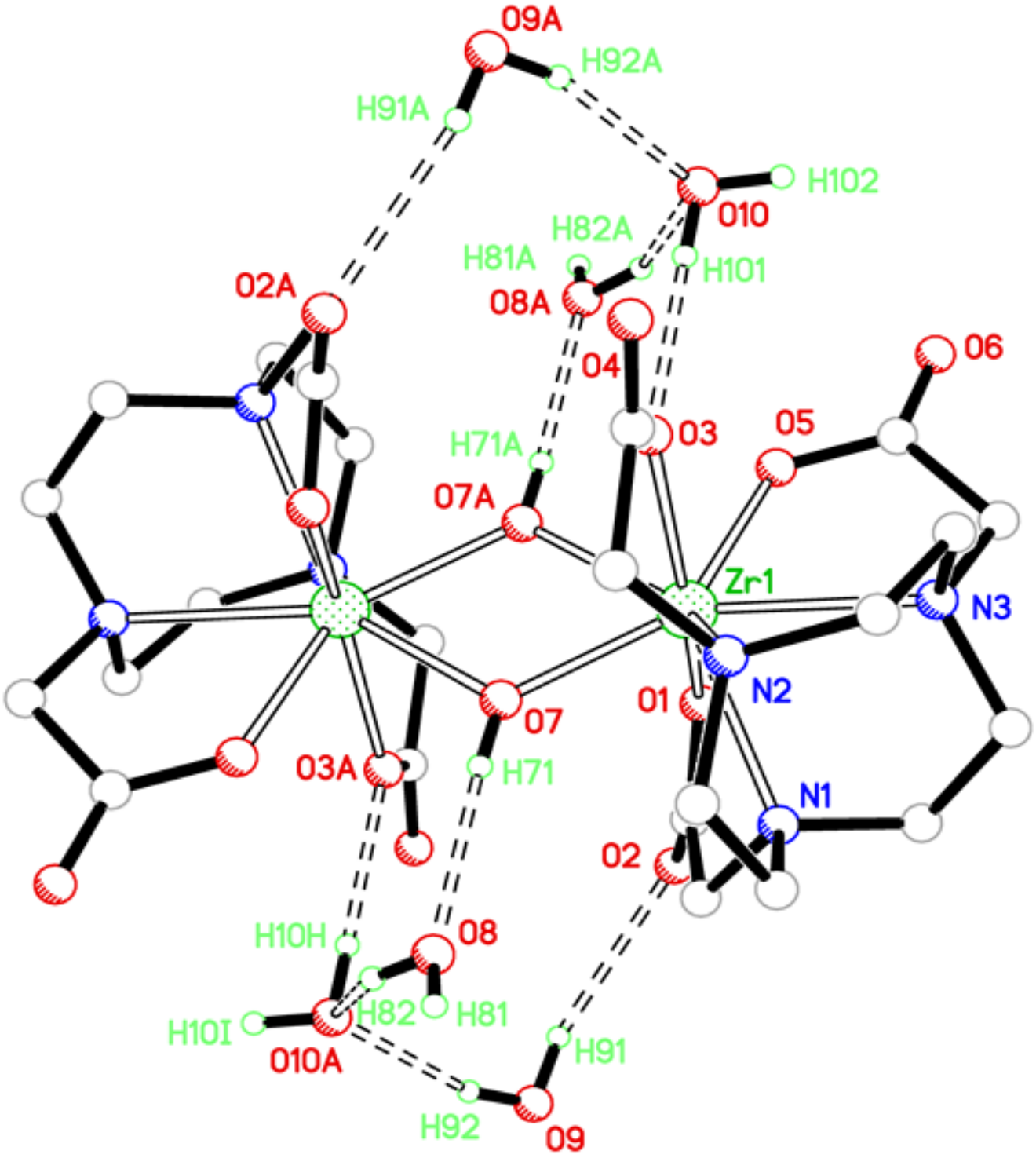


Figure S40. Plot of $[Zr(C_{12}H_{18}N_3O_6)(OH)]_2$ - 6 H_2O with disordered atoms C8', C10' and C11' omitted for clarity. All atoms represented by arbitrary-sized spheres. Ligand hydrogen atoms have been omitted and hydrogen-bonding interactions involving the bridging OH groups and lattice water molecules are represented by dashed-open bonds.

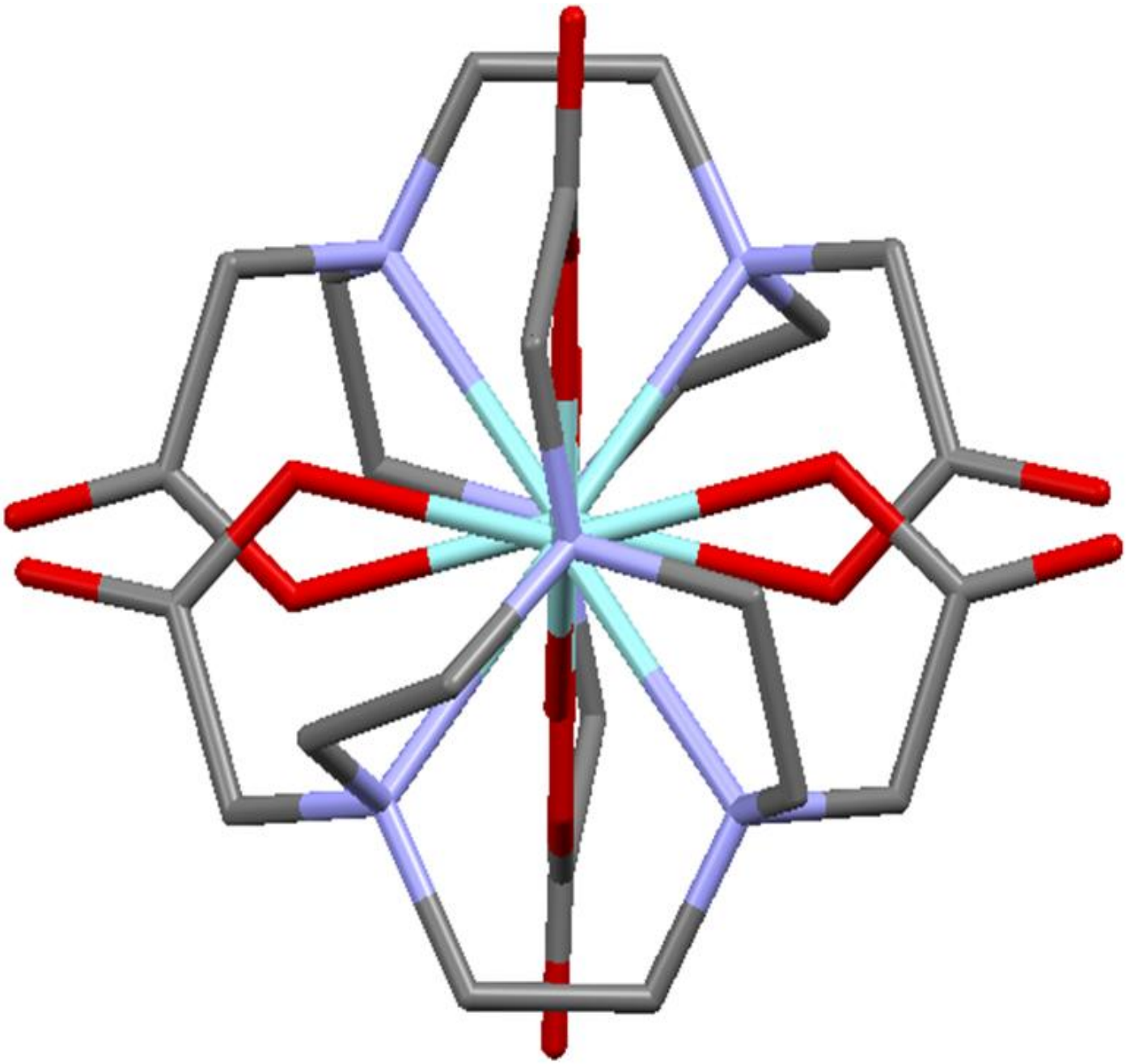


Figure S41. Plot of $[Zr(C_{12}H_{18}N_3O_6)(OH)]_2$ - 6 H_2O, viewed down the Zr···Zr vector, with disordered atoms C8', C10' and C11'. All hydrogen atoms and water molecules have been omitted for clarity.

Table 11. Crystal structure data collection parameters

Crystal data

$C_{24}H_{50}N_6O_{20}Zr_2$	F(000) = 952
M_r = 925.14	D_x = 1.841 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo Kα radiation, λ = 0.71073 Å
a = 11.7960 (7) Å	Cell parameters from 9847 reflections
b = 10.9411 (6) Å	θ = 3.7–31.5°
c = 14.1815 (8) Å	μ = 0.72 mm^{-1}
β = 114.210 (1)°	T = 193 K
V = 1669.31 (16) Å3	Irregular plate, colorless
Z = 2	0.46 × 0.30 × 0.08 mm

Data collection

Bruker APEX CCD diffractometer	5078 independent reflections
Radiation source: sealed x-ray tube	4778 reflections with $I > 2\sigma(I)$
Graphite monochromator	R_{int} = 0.031
ϕ and ω scans	θ_{max} = 30.5°, θ_{min} = 3.5°
Absorption correction: multi-scan Data were corrected for scaling and absorption effects using the multi-scan technique (*SADABS*). The ratio of minimum to maximum apparent transmission was 0.909. The calculated minimum and maximum transmission coefficients (based on crystal size) are 0.733 and 0.945.	h = -16→16
T_{min} = 0.678, T_{max} = 0.746	k = -15→15
31714 measured reflections	l = -20→20

Refinement

Refinement on F^2	10 restraints
Least-squares matrix: full	Hydrogen site location: mixed
$R[F^2 > 2\sigma(F^2)] = 0.019$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.052$	$w = 1/[\sigma^2(F_o^2) + (0.0269P)^2 + 0.7232P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.002$
5078 reflections	$\Delta\rangle_{max} = 0.52$ e Å^{-3}
276 parameters	$\Delta\rangle_{min} = -0.24$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table S12. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Zr1	0.42226 (2)	0.57931 (2)	0.06624 (2)	0.00986 (4)	
O1	0.40948 (7)	0.72464 (7)	-0.04018 (6)	0.01716 (15)	
O2	0.50149 (9)	0.87590 (9)	-0.08801 (7)	0.02681 (19)	
O3	0.37057 (7)	0.40118 (7)	0.10355 (6)	0.01641 (15)	
O4	0.39167 (9)	0.25215 (9)	0.21731 (7)	0.0285 (2)	
O5	0.22734 (7)	0.57222 (8)	-0.02677 (6)	0.01836 (16)	
O6	0.02701 (8)	0.60589 (10)	-0.06612 (7)	0.02521 (19)	
O7	0.59586 (7)	0.52797 (7)	0.07190 (6)	0.01279 (14)	
H71	0.6656 (10)	0.5464 (17)	0.1103 (12)	0.032 (5)*	
N1	0.53517 (8)	0.76027 (8)	0.15638 (7)	0.01482 (16)	
N2	0.52750 (8)	0.53771 (9)	0.25042 (7)	0.01404 (16)	
N3	0.30800 (8)	0.68228 (9)	0.15594 (7)	0.01593 (17)	
C1	0.49519 (11)	0.80627 (10)	-0.02259 (9)	0.0173 (2)	
C2	0.59196 (10)	0.81112 (10)	0.08854 (8)	0.0179 (2)	
H2A	0.6659	0.7625	0.0958	0.021*	
H2B	0.6185	0.8966	0.1084	0.021*	
C3	0.63025 (11)	0.74006 (11)	0.26511 (8)	0.0187 (2)	
H3A	0.6042	0.7846	0.3136	0.022*	
H3B	0.7109	0.7744	0.2719	0.022*	
C4	0.64753 (10)	0.60560 (11)	0.29505 (8)	0.0170 (2)	
H4A	0.7068	0.5685	0.2702	0.020*	
H4B	0.6834	0.5984	0.3713	0.020*	
C5	0.42989 (10)	0.34316 (10)	0.19085 (8)	0.01681 (19)	
C6	0.55222 (10)	0.40409 (10)	0.25705 (9)	0.0167 (2)	
H6A	0.5821	0.3759	0.3296	0.020*	
H6B	0.6161	0.3844	0.2309	0.020*	
C7	0.44795 (11)	0.56521 (11)	0.30816 (9)	0.0189 (2)	

H7A	0.4834	0.6362	0.3541	0.023*	0.932 (3)
H7B	0.4502	0.4943	0.3522	0.023*	0.932 (3)
H7C	0.3961	0.4927	0.3042	0.023*	0.068 (3)
H7D	0.5031	0.5785	0.3819	0.023*	0.068 (3)
C8	0.31342 (12)	0.59285 (12)	0.23689 (10)	0.0189 (3)	0.932 (3)
H8A	0.2708	0.5164	0.2037	0.023*	0.932 (3)
H8B	0.2699	0.6268	0.2777	0.023*	0.932 (3)
C9	0.13783 (10)	0.61751 (11)	-0.00904 (8)	0.0174 (2)	
C10	0.17627 (12)	0.70261 (13)	0.08255 (10)	0.0231 (3)	0.932 (3)
H10A	0.1659	0.7883	0.0579	0.028*	0.932 (3)
H10B	0.1215	0.6895	0.1190	0.028*	0.932 (3)
C11	0.36107 (12)	0.80236 (12)	0.20561 (10)	0.0207 (3)	0.932 (3)
H11A	0.2928	0.8603	0.1963	0.025*	0.932 (3)
H11B	0.4102	0.7906	0.2807	0.025*	0.932 (3)
C12	0.44248 (11)	0.85349 (10)	0.15727 (9)	0.0198 (2)	
H12A	0.4868	0.9263	0.1965	0.024*	0.932 (3)
H12B	0.3904	0.8794	0.0855	0.024*	0.932 (3)
H12C	0.4398	0.9206	0.1096	0.024*	0.068 (3)
H12D	0.4719	0.8886	0.2276	0.024*	0.068 (3)
C8'	0.3707 (13)	0.6658 (13)	0.2725 (11)	0.011 (3)*	0.068 (3)
H8'A	0.4201	0.7401	0.3029	0.013*	0.068 (3)
H8'B	0.3052	0.6602	0.2990	0.013*	0.068 (3)
C10'	0.1814 (13)	0.6236 (13)	0.1139 (11)	0.011 (3)*	0.068 (3)
H10C	0.1225	0.6730	0.1315	0.013*	0.068 (3)
H10D	0.1860	0.5405	0.1428	0.013*	0.068 (3)
C11'	0.3084 (15)	0.8044 (15)	0.1259 (12)	0.017 (3)*	0.068 (3)
H11C	0.2652	0.8555	0.1585	0.020*	0.068 (3)
H11D	0.2621	0.8108	0.0500	0.020*	0.068 (3)
O8	0.84053 (9)	0.61618 (12)	0.19501 (8)	0.0332 (2)	

H81	0.8933 (16)	0.621 (2)	0.2538 (8)	0.058 (7)*	
H82	0.8746 (17)	0.619 (2)	0.1560 (12)	0.048 (6)*	
O9	0.77230 (11)	0.95110 (11)	-0.03463 (9)	0.0365 (2)	
H91	0.7000 (10)	0.9291 (19)	-0.0639 (19)	0.067 (8)*	
H92	0.8114 (15)	0.8871 (12)	-0.0234 (16)	0.041 (5)*	
O10	0.13509 (9)	0.29129 (10)	-0.00865 (8)	0.0288 (2)	
H101	0.2034 (10)	0.3214 (17)	0.0250 (14)	0.042 (5)*	
H102	0.0916 (15)	0.3243 (18)	0.0166 (15)	0.047 (6)*	

Table S13. Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zr1	0.00987 (5)	0.01125 (5)	0.00919 (5)	0.00046 (3)	0.00465 (4)	-0.00013 (3)
O1	0.0198 (4)	0.0154 (3)	0.0149 (3)	-0.0001 (3)	0.0057 (3)	0.0023 (3)
O2	0.0367 (5)	0.0235 (4)	0.0218 (4)	-0.0045 (4)	0.0135 (4)	0.0053 (3)
O3	0.0162 (3)	0.0172 (3)	0.0151 (3)	-0.0020 (3)	0.0058 (3)	0.0023 (3)
O4	0.0302 (4)	0.0279 (5)	0.0271 (4)	-0.0075 (4)	0.0114 (4)	0.0090 (4)
O5	0.0126 (3)	0.0262 (4)	0.0155 (4)	0.0029 (3)	0.0050 (3)	-0.0031 (3)
O6	0.0128 (4)	0.0415 (5)	0.0204 (4)	0.0033 (3)	0.0059 (3)	0.0035 (4)
O7	0.0102 (3)	0.0162 (3)	0.0117 (3)	-0.0009 (3)	0.0042 (2)	-0.0029 (3)
N1	0.0172 (4)	0.0140 (4)	0.0136 (4)	-0.0012 (3)	0.0067 (3)	-0.0013 (3)
N2	0.0135 (4)	0.0170 (4)	0.0120 (4)	0.0004 (3)	0.0057 (3)	0.0011 (3)
N3	0.0149 (4)	0.0194 (4)	0.0139 (4)	0.0025 (3)	0.0064 (3)	-0.0012 (3)
C1	0.0218 (5)	0.0145 (5)	0.0175 (5)	0.0012 (4)	0.0101 (4)	0.0007 (4)
C2	0.0202 (5)	0.0166 (5)	0.0180 (5)	-0.0041 (4)	0.0089 (4)	-0.0001 (4)
C3	0.0194 (5)	0.0200 (5)	0.0138 (4)	-0.0035 (4)	0.0038 (4)	-0.0035 (4)
C4	0.0140 (4)	0.0223 (5)	0.0123 (4)	-0.0022 (4)	0.0029 (4)	-0.0004 (4)
C5	0.0174 (5)	0.0181 (5)	0.0173 (5)	0.0009 (4)	0.0096 (4)	0.0022 (4)
C6	0.0161 (4)	0.0169 (5)	0.0163 (5)	0.0026 (4)	0.0058 (4)	0.0043 (4)

C7	0.0195 (5)	0.0264 (5)	0.0135 (4)	0.0035 (4)	0.0094 (4)	0.0016 (4)
C8	0.0184 (5)	0.0242 (6)	0.0184 (5)	0.0025 (4)	0.0118 (4)	0.0016 (4)
C9	0.0148 (4)	0.0225 (5)	0.0155 (5)	0.0031 (4)	0.0069 (4)	0.0037 (4)
C10	0.0171 (5)	0.0313 (7)	0.0202 (6)	0.0089 (5)	0.0068 (4)	-0.0022 (5)
C11	0.0247 (6)	0.0187 (6)	0.0206 (6)	0.0027 (4)	0.0112 (5)	-0.0050 (4)
C12	0.0259 (5)	0.0135 (5)	0.0213 (5)	0.0023 (4)	0.0111 (4)	-0.0024 (4)
O8	0.0223 (4)	0.0549 (7)	0.0243 (5)	-0.0115 (4)	0.0114 (4)	-0.0111 (5)
O9	0.0382 (6)	0.0298 (5)	0.0380 (6)	0.0007 (5)	0.0120 (5)	-0.0027 (5)
O10	0.0179 (4)	0.0323 (5)	0.0346 (5)	-0.0014 (4)	0.0092 (4)	-0.0087 (4)

Table S14. Geometric parameters (Å, º)

Zr1—O7	2.0930 (7)	C4—H4B	0.9900
Zr1—O5	2.1320 (8)	C5—C6	1.5156 (15)
Zr1—O1	2.1546 (8)	C6—H6A	0.9900
Zr1—O3	2.1714 (8)	C6—H6B	0.9900
Zr1—O7^{i}	2.2176 (7)	C7—C8'	1.385 (14)
Zr1—N2	2.4313 (9)	C7—C8	1.5218 (17)
Zr1—N1	2.4355 (9)	C7—H7A	0.9900
Zr1—N3	2.4719 (9)	C7—H7B	0.9900
Zr1—Zr1^{i}	3.5680 (2)	C7—H7C	0.9900
O1—C1	1.2944 (14)	C7—H7D	0.9900
O2—C1	1.2260 (14)	C8—H8A	0.9900
O3—C5	1.3095 (13)	C8—H8B	0.9900
O4—C5	1.2142 (14)	C9—C10	1.5089 (17)
O5—C9	1.2811 (14)	C9—C10'	1.604 (14)
O6—C9	1.2298 (14)	C10—H10A	0.9900
O7—Zr1^{i}	2.2175 (7)	C10—H10B	0.9900
O7—H71	0.804 (9)	C11—C12	1.4983 (18)

N1—C2	1.4874 (14)	C11—H11A	0.9900
N1—C12	1.4991 (14)	C11—H11B	0.9900
N1—C3	1.5053 (14)	C12—C11'	1.553 (16)
N2—C6	1.4861 (14)	C12—H12A	0.9900
N2—C4	1.4906 (14)	C12—H12B	0.9900
N2—C7	1.5073 (14)	C12—H12C	0.9900
N3—C11'	1.403 (16)	C12—H12D	0.9900
N3—C8	1.4897 (15)	C8'—H8'A	0.9900
N3—C10	1.4903 (15)	C8'—H8'B	0.9900
N3—C11	1.5008 (16)	C10'—H10C	0.9900
N3—C10'	1.506 (14)	C10'—H10D	0.9900
N3—C8'	1.520 (14)	C11'—H11C	0.9900
C1—C2	1.5212 (15)	C11'—H11D	0.9900
C2—H2A	0.9900	O8—H81	0.813 (9)
C2—H2B	0.9900	O8—H82	0.807 (9)
C3—C4	1.5216 (16)	O9—H91	0.817 (9)
C3—H3A	0.9900	O9—H92	0.818 (9)
C3—H3B	0.9900	O10—H101	0.818 (9)
C4—H4A	0.9900	O10—H102	0.820 (9)
O7—Zr1—O5	143.51 (3)	N2—C4—H4A	109.3
O7—Zr1—O1	90.64 (3)	C3—C4—H4A	109.3
O5—Zr1—O1	81.11 (3)	N2—C4—H4B	109.3
O7—Zr1—O3	96.77 (3)	C3—C4—H4B	109.3
O5—Zr1—O3	77.21 (3)	H4A—C4—H4B	108.0
O1—Zr1—O3	152.51 (3)	O4—C5—O3	124.58 (11)
O7—Zr1—O7^{i}	68.30 (3)	O4—C5—C6	123.31 (10)
O5—Zr1—O7^{i}	75.25 (3)	O3—C5—C6	112.10 (9)
O1—Zr1—O7^{i}	79.52 (3)	N2—C6—C5	106.34 (9)
O3—Zr1—O7^{i}	78.77 (3)	N2—C6—H6A	110.5

O7—Zr1—N2	81.26 (3)	C5—C6—H6A	110.5
O5—Zr1—N2	127.15 (3)	N2—C6—H6B	110.5
O1—Zr1—N2	138.50 (3)	C5—C6—H6B	110.5
O3—Zr1—N2	68.95 (3)	H6A—C6—H6B	108.7
O7^{i}—Zr1—N2	132.20 (3)	C8'—C7—N2	115.0 (6)
O7—Zr1—N1	83.23 (3)	N2—C7—C8	113.00 (9)
O5—Zr1—N1	124.98 (3)	N2—C7—H7A	109.0
O1—Zr1—N1	68.52 (3)	C8—C7—H7A	109.0
O3—Zr1—N1	138.55 (3)	N2—C7—H7B	109.0
O7^{i}—Zr1—N1	136.71 (3)	C8—C7—H7B	109.0
N2—Zr1—N1	70.12 (3)	H7A—C7—H7B	107.8
O7—Zr1—N3	146.51 (3)	C8'—C7—H7C	108.5
O5—Zr1—N3	69.98 (3)	N2—C7—H7C	108.5
O1—Zr1—N3	97.34 (3)	C8'—C7—H7D	108.5
O3—Zr1—N3	90.96 (3)	N2—C7—H7D	108.5
O7^{i}—Zr1—N3	145.13 (3)	H7C—C7—H7D	107.5
N2—Zr1—N3	71.24 (3)	N3—C8—C7	110.23 (10)
N1—Zr1—N3	69.86 (3)	N3—C8—H8A	109.6
O7—Zr1—Zr1^{i}	35.27 (2)	C7—C8—H8A	109.6
O5—Zr1—Zr1^{i}	108.26 (2)	N3—C8—H8B	109.6
O1—Zr1—Zr1^{i}	83.89 (2)	C7—C8—H8B	109.6
O3—Zr1—Zr1^{i}	87.02 (2)	H8A—C8—H8B	108.1
O7^{i}—Zr1—Zr1^{i}	33.025 (19)	O6—C9—O5	124.74 (11)
N2—Zr1—Zr1^{i}	109.17 (2)	O6—C9—C10	119.57 (11)
N1—Zr1—Zr1^{i}	112.54 (2)	O5—C9—C10	115.26 (10)
N3—Zr1—Zr1^{i}	177.60 (2)	O6—C9—C10'	120.0 (5)
C1—O1—Zr1	123.33 (7)	O5—C9—C10'	107.9 (5)
C5—O3—Zr1	124.81 (7)	N3—C10—C9	111.61 (10)
C9—O5—Zr1	129.13 (7)	N3—C10—H10A	109.3

Zr1—O7—Zr1^{i}	111.70 (3)	C9—C10—H10A	109.3
Zr1—O7—H71	132.2 (13)	N3—C10—H10B	109.3
Zr1^{i}—O7—H71	115.6 (13)	C9—C10—H10B	109.3
C2—N1—C12	105.93 (9)	H10A—C10—H10B	108.0
C2—N1—C3	111.93 (8)	C12—C11—N3	109.54 (10)
C12—N1—C3	109.33 (8)	C12—C11—H11A	109.8
C2—N1—Zr1	105.01 (6)	N3—C11—H11A	109.8
C12—N1—Zr1	108.29 (6)	C12—C11—H11B	109.8
C3—N1—Zr1	115.83 (7)	N3—C11—H11B	109.8
C6—N2—C4	109.62 (8)	H11A—C11—H11B	108.2
C6—N2—C7	108.16 (9)	C11—C12—N1	110.88 (9)
C4—N2—C7	111.36 (9)	N1—C12—C11'	115.0 (6)
C6—N2—Zr1	104.63 (6)	C11—C12—H12A	109.5
C4—N2—Zr1	109.91 (6)	N1—C12—H12A	109.5
C7—N2—Zr1	112.89 (6)	C11—C12—H12B	109.5
C8—N3—C10	109.83 (10)	N1—C12—H12B	109.5
C8—N3—C11	109.60 (9)	H12A—C12—H12B	108.1
C10—N3—C11	108.17 (10)	N1—C12—H12C	108.5
C11'—N3—C10'	114.4 (9)	C11'—C12—H12C	108.5
C11'—N3—C8'	112.5 (8)	N1—C12—H12D	108.5
C10'—N3—C8'	110.0 (8)	C11'—C12—H12D	108.5
C11'—N3—Zr1	101.9 (7)	H12C—C12—H12D	107.5
C8—N3—Zr1	103.72 (7)	C7—C8'—N3	116.4 (10)
C10—N3—Zr1	109.94 (7)	C7—C8'—H8'A	108.2
C11—N3—Zr1	115.46 (7)	N3—C8'—H8'A	108.2
C10'—N3—Zr1	105.3 (5)	C7—C8'—H8'B	108.2
C8'—N3—Zr1	112.4 (5)	N3—C8'—H8'B	108.2
O2—C1—O1	124.60 (11)	H8'A—C8'—H8'B	107.3
O2—C1—C2	120.65 (10)	N3—C10'—C9	105.8 (8)

O1—C1—C2	114.76 (9)	N3—C10'—H10C	110.6
N1—C2—C1	108.15 (9)	C9—C10'—H10C	110.6
N1—C2—H2A	110.1	N3—C10'—H10D	110.6
C1—C2—H2A	110.1	C9—C10'—H10D	110.6
N1—C2—H2B	110.1	H10C—C10'—H10D	108.7
C1—C2—H2B	110.1	N3—C11'—C12	111.9 (10)
H2A—C2—H2B	108.4	N3—C11'—H11C	109.2
N1—C3—C4	112.71 (9)	C12—C11'—H11C	109.2
N1—C3—H3A	109.0	N3—C11'—H11D	109.2
C4—C3—H3A	109.0	C12—C11'—H11D	109.2
N1—C3—H3B	109.0	H11C—C11'—H11D	107.9
C4—C3—H3B	109.0	H81—O8—H82	108.3 (17)
H3A—C3—H3B	107.8	H91—O9—H92	103.6 (17)
N2—C4—C3	111.60 (9)	H101—O10—H102	102.1 (16)
Zr1—O1—C1—O2	166.26 (9)	Zr1—O5—C9—O6	-177.79 (9)
Zr1—O1—C1—C2	-13.66 (13)	Zr1—O5—C9—C10	9.74 (15)
C12—N1—C2—C1	-71.30 (10)	Zr1—O5—C9—C10'	-28.1 (6)
C3—N1—C2—C1	169.63 (9)	C8—N3—C10—C9	-92.35 (12)
Zr1—N1—C2—C1	43.18 (9)	C11—N3—C10—C9	148.06 (11)
O2—C1—C2—N1	155.90 (11)	Zr1—N3—C10—C9	21.16 (13)
O1—C1—C2—N1	-24.18 (13)	O6—C9—C10—N3	166.12 (11)
C2—N1—C3—C4	-114.42 (10)	O5—C9—C10—N3	-20.99 (16)
C12—N1—C3—C4	128.54 (10)	C8—N3—C11—C12	137.31 (10)
Zr1—N1—C3—C4	5.92 (12)	C10—N3—C11—C12	-102.96 (11)
C6—N2—C4—C3	164.89 (9)	Zr1—N3—C11—C12	20.67 (12)
C7—N2—C4—C3	-75.45 (11)	N3—C11—C12—N1	-51.06 (13)
Zr1—N2—C4—C3	50.41 (10)	C2—N1—C12—C11	168.66 (9)
N1—C3—C4—N2	-37.23 (13)	C3—N1—C12—C11	-70.57 (11)
Zr1—O3—C5—O4	169.35 (9)	Zr1—N1—C12—C11	56.45 (10)

Zr1—O3—C5—C6	-9.66 (13)	C2—N1—C12—C11'	125.3 (7)
C4—N2—C6—C5	-168.28 (9)	C3—N1—C12—C11'	-113.9 (7)
C7—N2—C6—C5	70.12 (10)	Zr1—N1—C12—C11'	13.1 (7)
Zr1—N2—C6—C5	-50.45 (9)	N2—C7—C8'—N3	40.0 (11)
O4—C5—C6—N2	-136.71 (12)	C11'—N3—C8'—C7	-137.8 (11)
O3—C5—C6—N2	42.32 (12)	C10'—N3—C8'—C7	93.4 (11)
C6—N2—C7—C8'	-151.3 (6)	Zr1—N3—C8'—C7	-23.4 (11)
C4—N2—C7—C8'	88.2 (6)	C11'—N3—C10'—C9	65.5 (11)
Zr1—N2—C7—C8'	-36.0 (6)	C8'—N3—C10'—C9	-166.8 (8)
C6—N2—C7—C8	-105.91 (11)	Zr1—N3—C10'—C9	-45.5 (8)
C4—N2—C7—C8	133.57 (10)	O6—C9—C10'—N3	-159.9 (5)
Zr1—N2—C7—C8	9.37 (12)	O5—C9—C10'—N3	48.8 (8)
C10—N3—C8—C7	176.36 (10)	C10'—N3—C11'—C12	-171.2 (9)
C11—N3—C8—C7	-64.94 (12)	C8'—N3—C11'—C12	62.4 (12)
Zr1—N3—C8—C7	58.89 (10)	Zr1—N3—C11'—C12	-58.2 (10)
N2—C7—C8—N3	-47.53 (13)	N1—C12—C11'—N3	32.5 (12)

Symmetry code: (i) -*x*+1, -*y*+1, -*z*.

Table S15. Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O7—H71···O8	0.80 (1)	2.06 (1)	2.8594 (12)	172 (2)
O8—H81···O2^{ii}	0.81 (1)	2.08 (1)	2.8792 (14)	169 (2)
O8—H82···O10^{i}	0.81 (1)	2.27 (1)	2.9522 (16)	143 (2)
O9—H91···O2	0.82 (1)	2.30 (1)	3.0776 (16)	160 (3)
O9—H92···O10^{i}	0.82 (1)	2.04 (1)	2.8378 (16)	164 (2)
O10—H101···O3	0.82 (1)	2.02 (1)	2.8404 (12)	178 (2)
O10—H102···O6^{iii}	0.82 (1)	1.96 (1)	2.7766 (14)	174 (2)

Symmetry codes: (i) -*x*+1, -*y*+1, -*z*; (ii) *x*+1/2, -*y*+3/2, *z*+1/2; (iii) -*x*, -*y*+1, -*z*.

Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane).

- 1.8012 (0.0053) x + 5.1785 (0.0044) y + 12.1093 (0.0034) z = 4.8668 (0.0034)

* 0.0000 (0.0000) N1

* 0.0000 (0.0000) N2

* 0.0000 (0.0000) N3

-1.8253 (0.0005) Zr1

-2.3383 (0.0013) O1

-2.2028 (0.0014) O3

-2.6372 (0.0013) O5

-2.3353 (0.0013) O7

Rms deviation of fitted atoms = 0.0000

- 3.9828 (0.0061) x + 5.8162 (0.0026) y + 12.0108 (0.0022) z = 2.1012 (0.0025)

Angle to previous plane (with approximate esd) = 12.290 (0.049)

* 0.0000 (0.0000) O1

* 0.0000 (0.0000) O3

* 0.0000 (0.0000) O5

0.3820 (0.0007) Zr1

-0.5400 (0.0018) O7

2.0675 (0.0016) N1

1.9331 (0.0017) N2

2.5134 (0.0011) N3

Rms deviation of fitted atoms = 0.0000

- 2.4457 (0.0031) x + 5.6205 (0.0026) y + 11.9740 (0.0022) z = 2.4723 (0.0020)

Angle to previous plane (with approximate esd) = 8.192 (0.039)

* 0.1179 (0.0004) O1

* 0.1161 (0.0004) O3

* -0.1327 (0.0005) O5

* -0.1013 (0.0003) O7

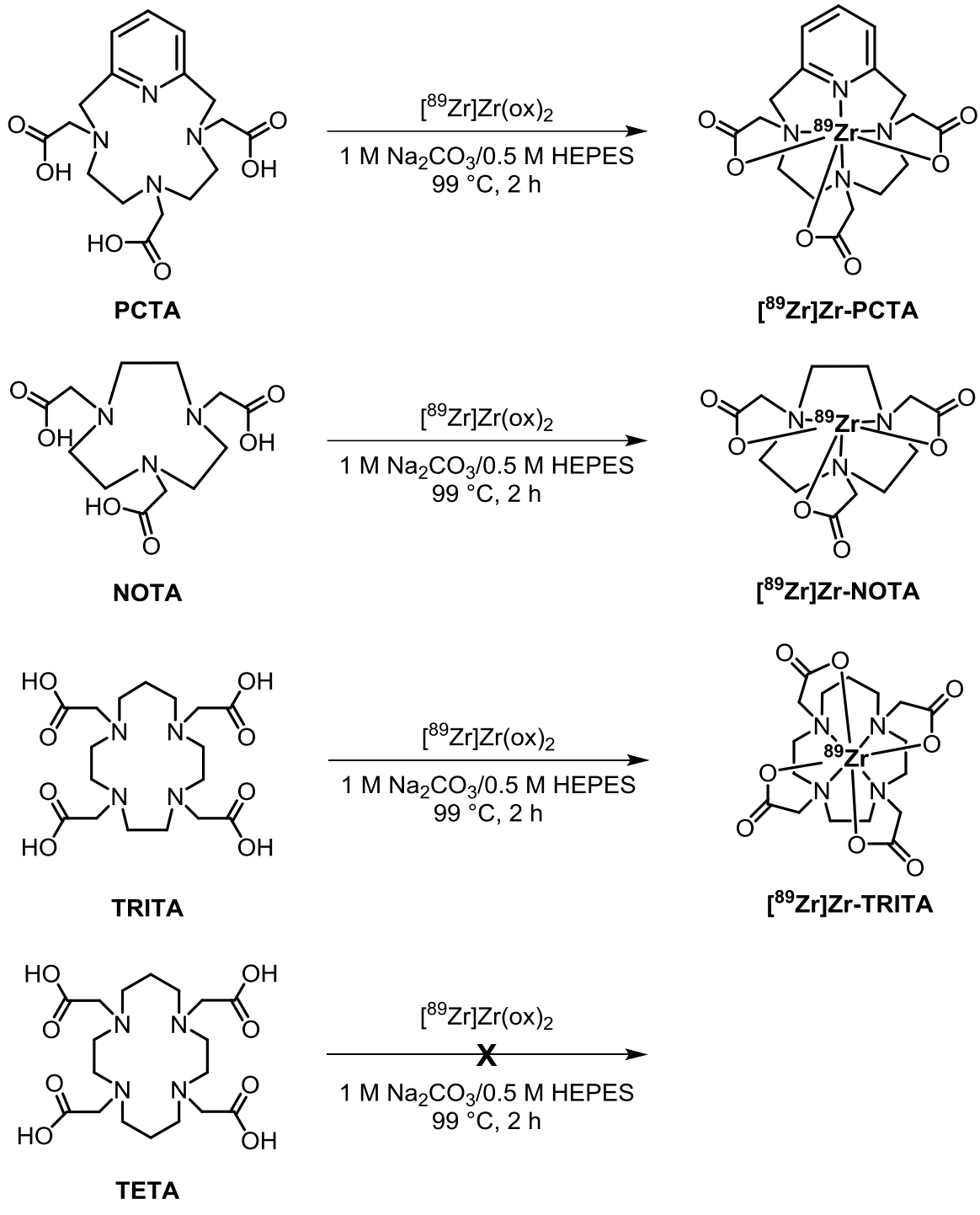
0.5441 (0.0004) Zr1

2.3644 (0.0011) N1

2.2583 (0.0011) N2

2.4764 (0.0011) N3

Rms deviation of fitted atoms = 0.1175



Scheme S5. Radiochemical Synthesis of [^{89}Zr]Zr-PCTA, [^{89}Zr]Zr-NOTA, [^{89}Zr]Zr-TRITA and [^{89}Zr]Zr-TETA using [^{89}Zr]Zr(ox)$_2$

Table S16. Summary of optimized radiochemistry conditions to prepare ^{89}Zr-complexes with [^{89}Zr]Zr(ox)$_2$

Radiochemistry conditions	Ligands (n = 10)			
	PCTA	**NOTA**	**TRITA**	**TETA**
Quantity (μg)	10-50	10-50	10-50	10-50
[^{89}Zr]Zr(ox)$_2$ added (MBq)	16.5-20.5	16.5-20.5	16.5-20.5	16.5-20.5
Reaction Buffer	0.5 M HEPES	0.5 M HEPES	0.5 M HEPES	0.5 M HEPES
Final Reaction pH	6.9-7.3	6.9-7.3	6.9-7.3	6.9-7.3
Reaction Temperature (°C)	99	99	99	99
Reaction time (min)	120	120	120	120
Radiochemical yield (%)	35 ± 9.3	15 ± 5.7	3 ± 1.3	0

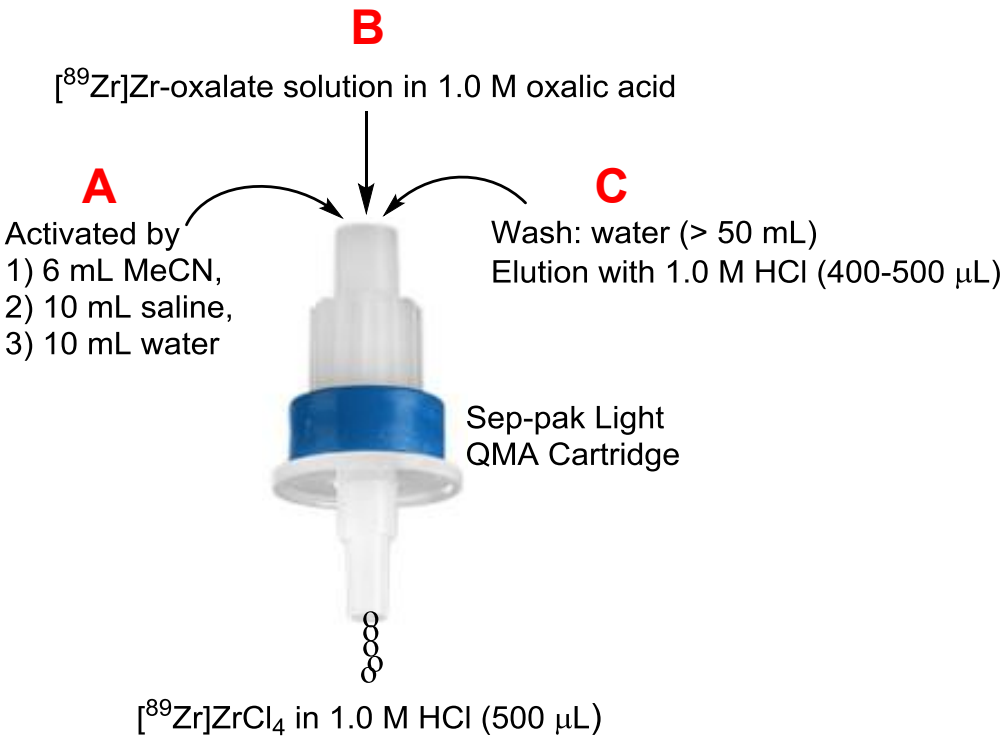
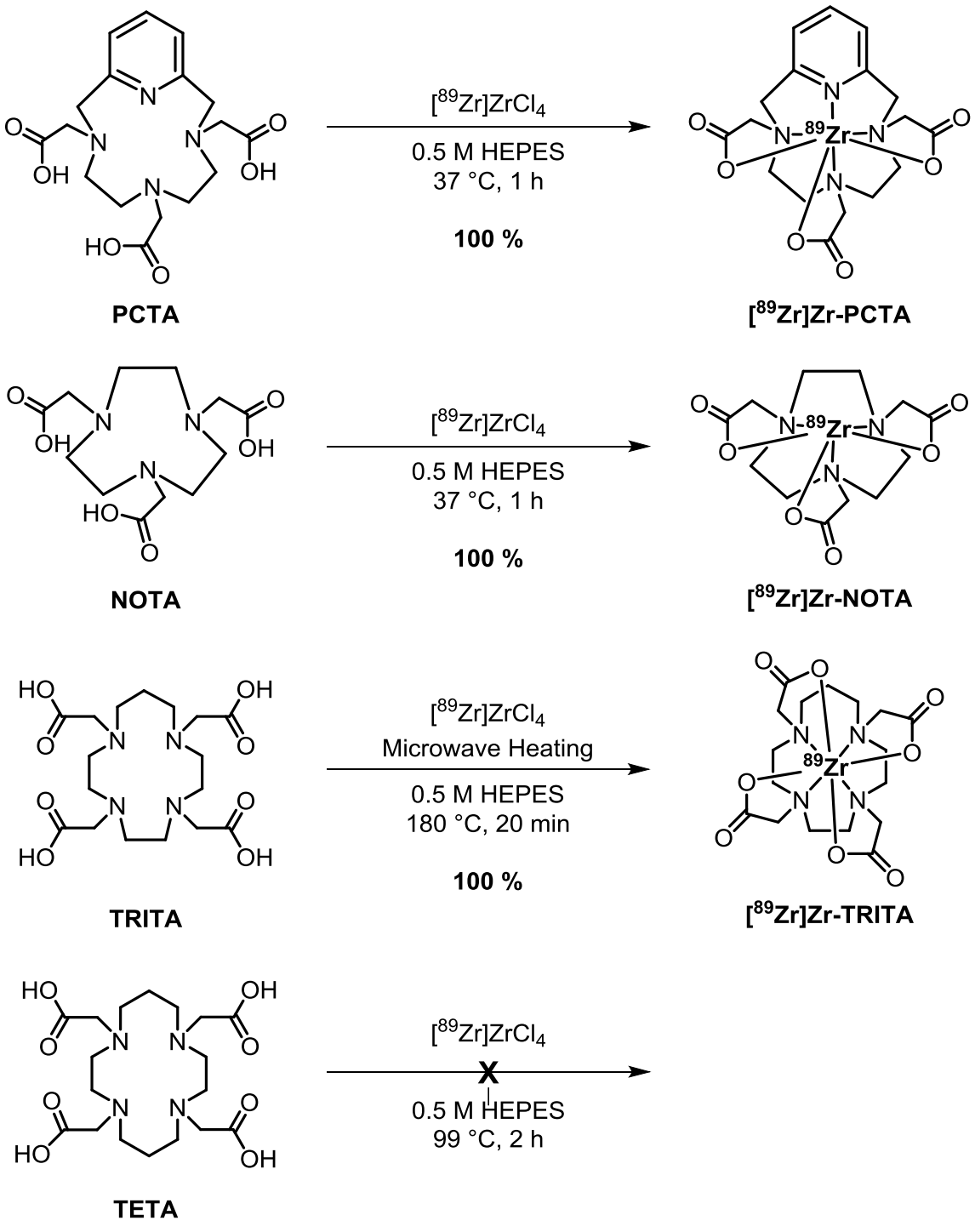


Figure S42. Schematic diagram for the production of [^{89}Zr]$ZrCl_4$ from [^{89}Zr]$Zr(ox)_2$



Scheme S6. Radiochemical Synthesis of [^{89}Zr]Zr-PCTA, [^{89}Zr]Zr-NOTA, [^{89}Zr]Zr-TRITA and [^{89}Zr]Zr-TETA using [^{89}Zr]ZrCl$_4$

Radiochemical Synthesis of [^{89}Zr]Zr-PCTA with [^{89}Zr]$ZrCl_4$:

Table S17. Summary of optimized radiochemistry conditions and [^{89}Zr]$ZrCl_4$ activity used to prepare [^{89}Zr]Zr-PCTA[a] complex (n = 4 for each buffer)

Quantity of Ligand	[^{89}Zr]$ZrCl_4$ added (MBq)	Reaction Buffer (pH 6.8 - 7.2)	Reaction Temperature (°C)	Reaction Time (min)	Radiochemical Yield by Radio-ITLC (%)
PCTA (7-10 µg)	18.5	1 M NH_4OAc	37	60	77.7 ± 0.9
	19.1	1 M NaOAc	37	60	59.1 ± 1.1
	18.3	1 M TRIS	37	60	27.2 ± 1.3
	18.4	1 M TMAA	37	60	15.9 ± 0.8
	19.2	0.5 M MES	37	60	93.1 ± 0.7
	18.8	**0.5 M HEPES**	**37**	**60**	**99.9 ± 0.1**
	18.5	0.5 M HEPES	24	60	70.8 ± 0.5
	18.4	0.5 M HEPES	24	120	92.3 ± 0.6

[a]PCTA ligand (7-10 µg) was labeled with [^{89}Zr]$ZrCl_4$ (18.3 – 19.2 MBq) using buffers (200 µL, pH 6.8 - 7.2) at 24°C or 37 °C for 60-120 min.

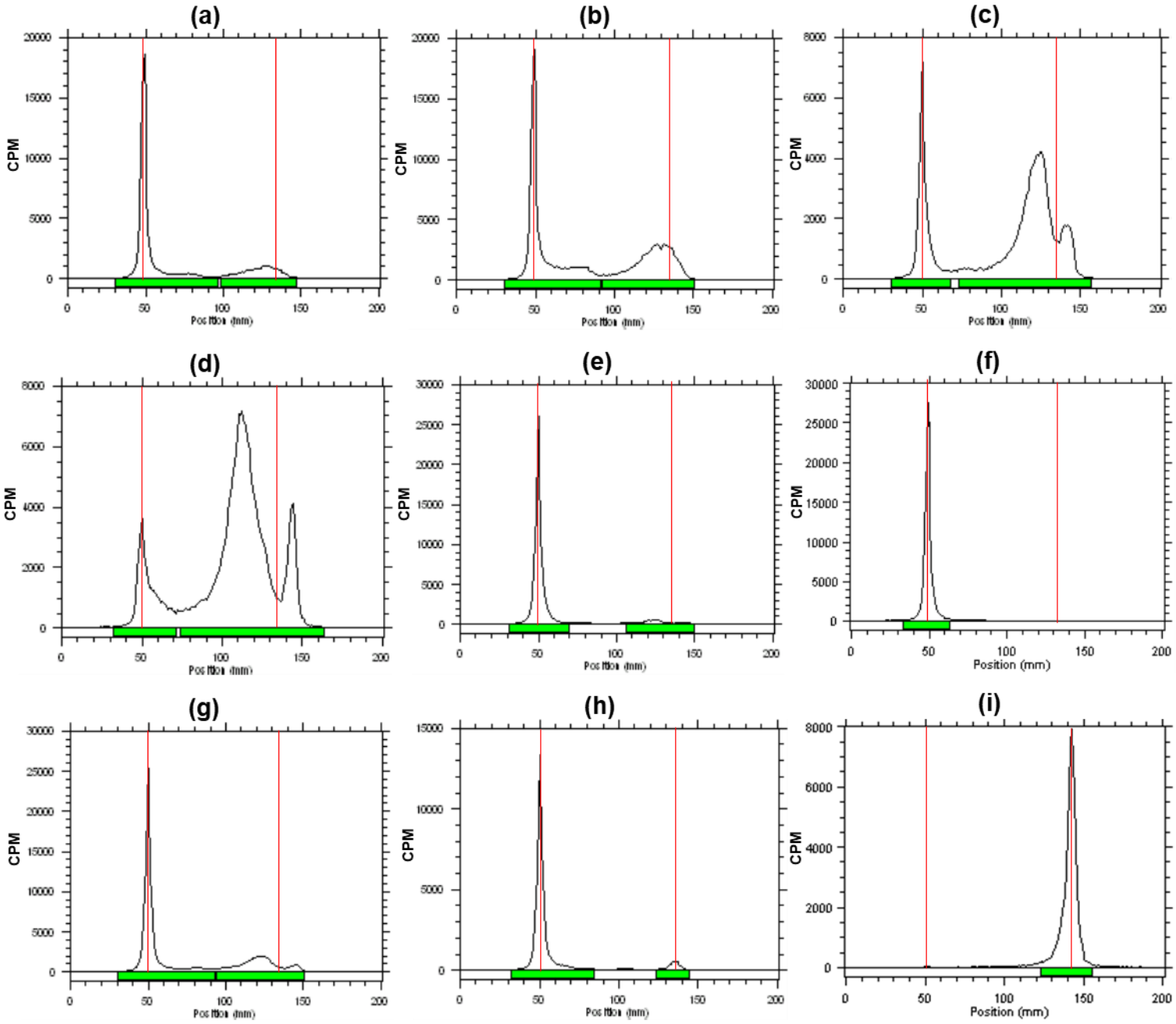


Figure S43. Radio-TLC analysis of [^{89}Zr]Zr-PCTA prepared by using different buffers at 24 °C or 37 °C for 60-120 min. (a) 1 M NH_4OAc, 37 °C, 60 min; (b) 1 M NaOAc, 37 °C, 60 min; (c) 1 M TRIS, 37 °C, 60 min; (d) 1 M TMAA, 37 °C, 60 min; (e) 0.5 M MES, 37 °C, 60 min; (f) 0.5 M HEPES, 37 °C, 60 min; (g) 0.5 M HEPES, 24 °C, 60 min; (h) 0.5 M HEPES, 24 °C, 120 min; (i) [^{89}Zr]$ZrCl_4$. In this ITLC-SA system, free ^{89}Zr formed a complex with EDTA and eluted with the solvent front (R_f ~ 1), while [^{89}Zr]Zr-PCTA complex remained at the origin (R_f ~ 0).

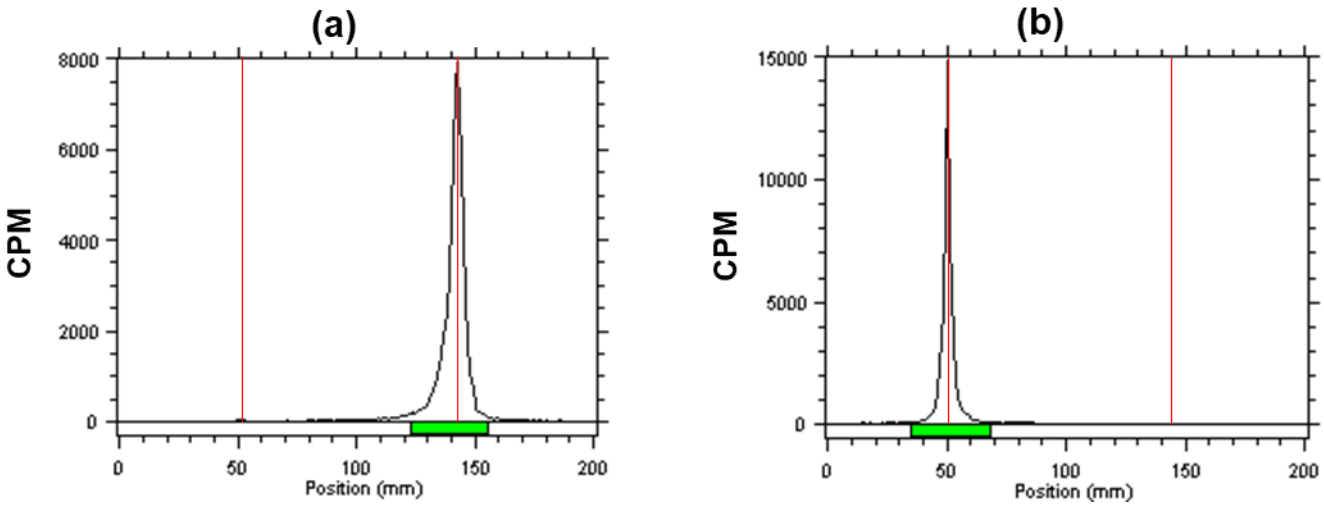


Figure S44. Quality control of [^{89}Zr]Zr-PCTA by radio-TLC. ITLC-SA of [^{89}Zr]$ZrCl_4$ (a), [^{89}Zr]Zr-PCTA (b)

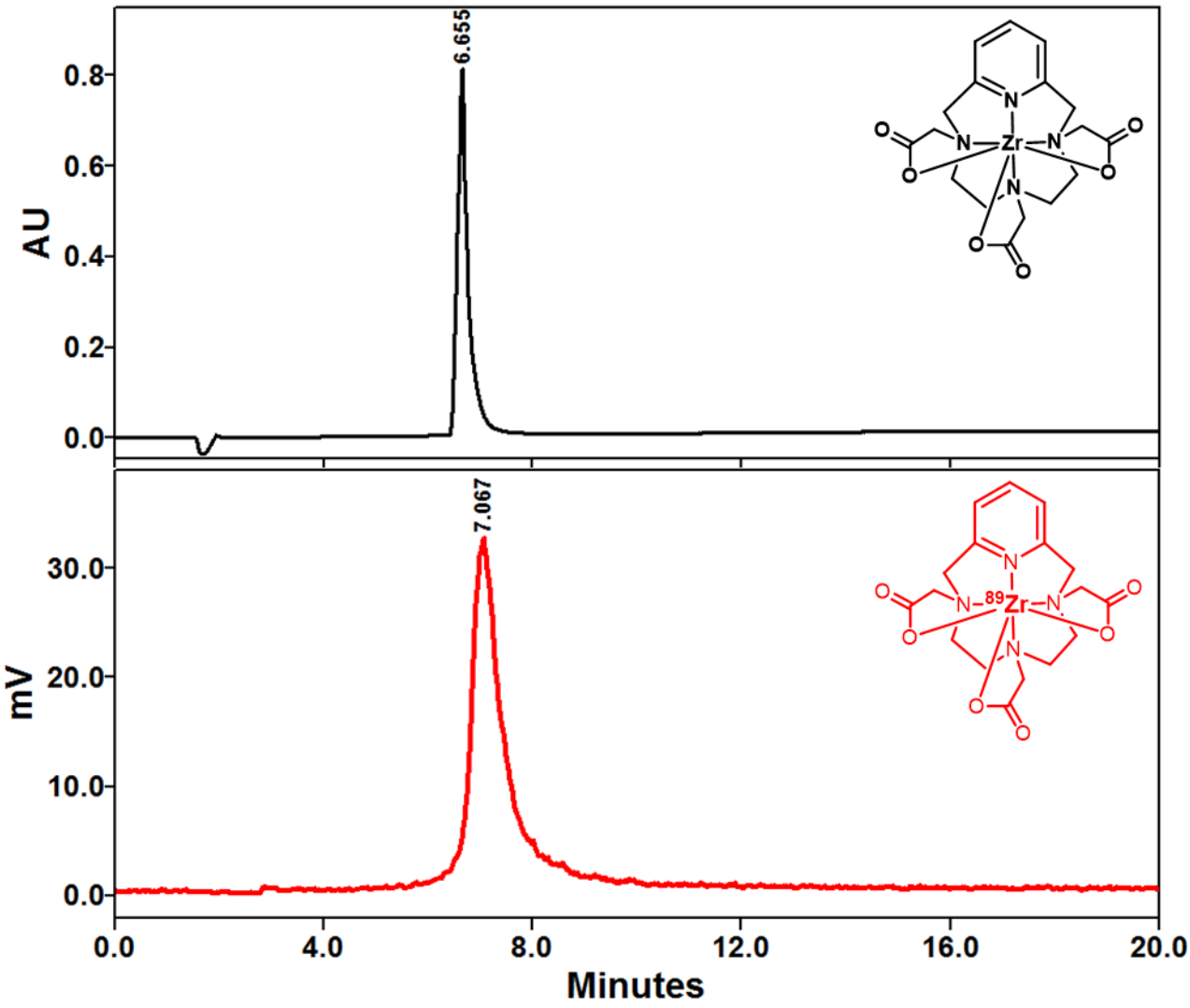


Figure S45. Quality control of [^{89}Zr]Zr-PCTA by radio-HPLC. UV-HPLC chromatogram (201 nm) of nonradioactive ^{Nat}Zr-PCTA complex (top) compared with radio-HPLC chromatogram of [^{89}Zr]Zr-PCTA (bottom)

Radiochemical Synthesis of [^{89}Zr]Zr-NOTA with [^{89}Zr]$ZrCl_4$:

Table S18. Summary of optimized radiochemistry conditions and [^{89}Zr]$ZrCl_4$ activity used to prepare [^{89}Zr]Zr-NOTA[a] complex (n = 4 for each buffer)

Quantity of Ligand	[^{89}Zr]$ZrCl_4$ added (MBq)	Reaction Buffer (pH 6.8 – 7.2)	Reaction Temperature (°C)	Reaction Time (min)	Radiochemical Yield by Radio-ITLC (%)
NOTA (7-10 µg)	18.1	1 M NH_4OAc	37	60	62.7 ± 1.2
	18.4	1 M NaOAc	37	60	30.2 ± 1.8
	17.9	1 M TRIS	37	60	20.5 ± 1.5
	18.0	1 M TMAA	37	60	2.2 ± 0.6
	18.5	0.5 M MES	37	60	74.5 ± 0.7
	19.1	**0.5 M HEPES**	**37**	**60**	**99.8 ± 0.2**
	18.7	0.5 M HEPES	24	60	66.7 ± 1.1
	18.9	0.5 M HEPES	24	120	85.4 ± 0.3

[a]NOTA ligand (7-10 µg) was labeled with [^{89}Zr]$ZrCl_4$ (17.9 – 19.1 MBq) using buffers (200 µL, pH 6.8 – 7.2) at 24°C or 37 °C for 60-120 min.

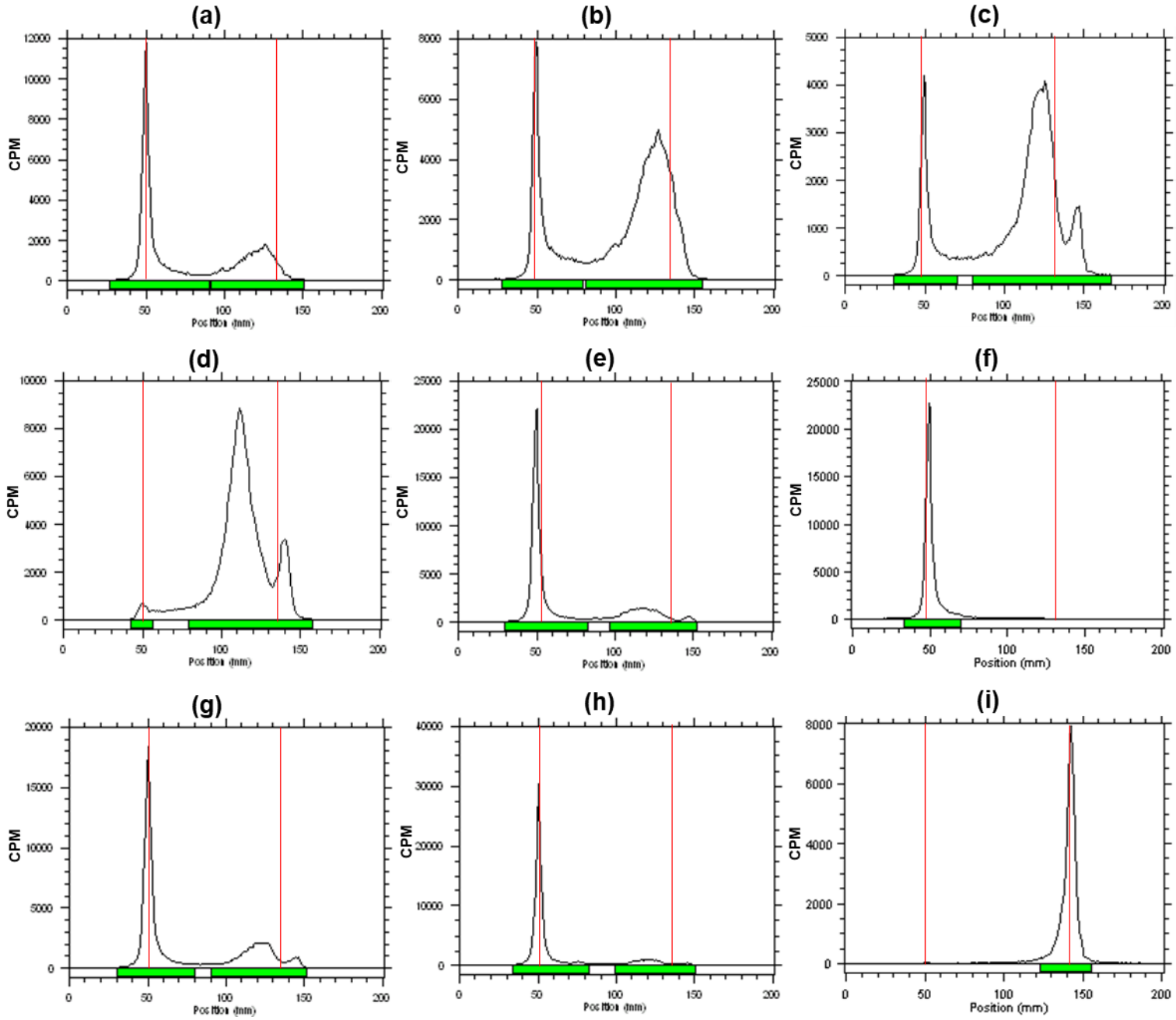


Figure S46. Radio-TLC analysis of [^{89}Zr]Zr-NOTA prepared by using different buffers at 24 °C or 37 °C for 60-120 min. (a) 1 M NH_4OAc, 37 °C, 60 min; (b) 1 M NaOAc, 37 °C, 60 min; (c) 1 M TRIS, 37 °C, 60 min; (d) 1 M TMAA, 37 °C, 60 min; (e) 0.5 M MES, 37 °C, 60 min; (f) 0.5 M HEPES, 37 °C, 60 min; (g) 0.5 M HEPES, 24 °C, 60 min; (h) 0.5 M HEPES, 24 °C, 120 min; (i) [^{89}Zr]$ZrCl_4$. In this ITLC-SA system, free ^{89}Zr formed a complex with EDTA and eluted with the solvent front (R_f ~ 1), while [^{89}Zr]Zr-NOTA complex remained at the origin (R_f ~ 0).

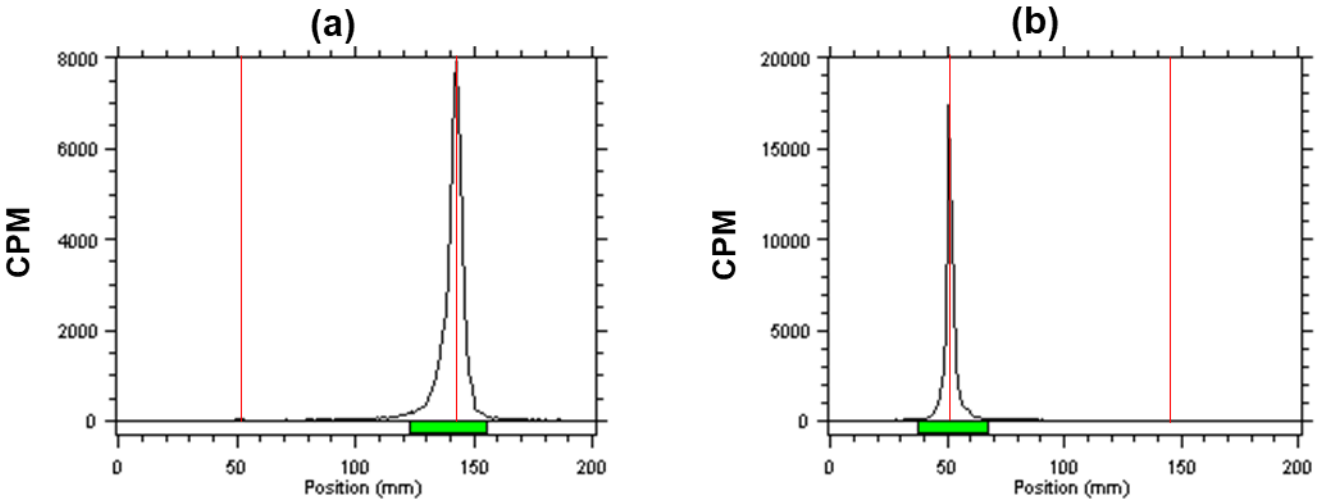


Figure S47. Quality control of [^{89}Zr]Zr-NOTA by radio-TLC. ITLC-SA of [^{89}Zr]$ZrCl_4$ (a), [^{89}Zr]Zr-NOTA (b)

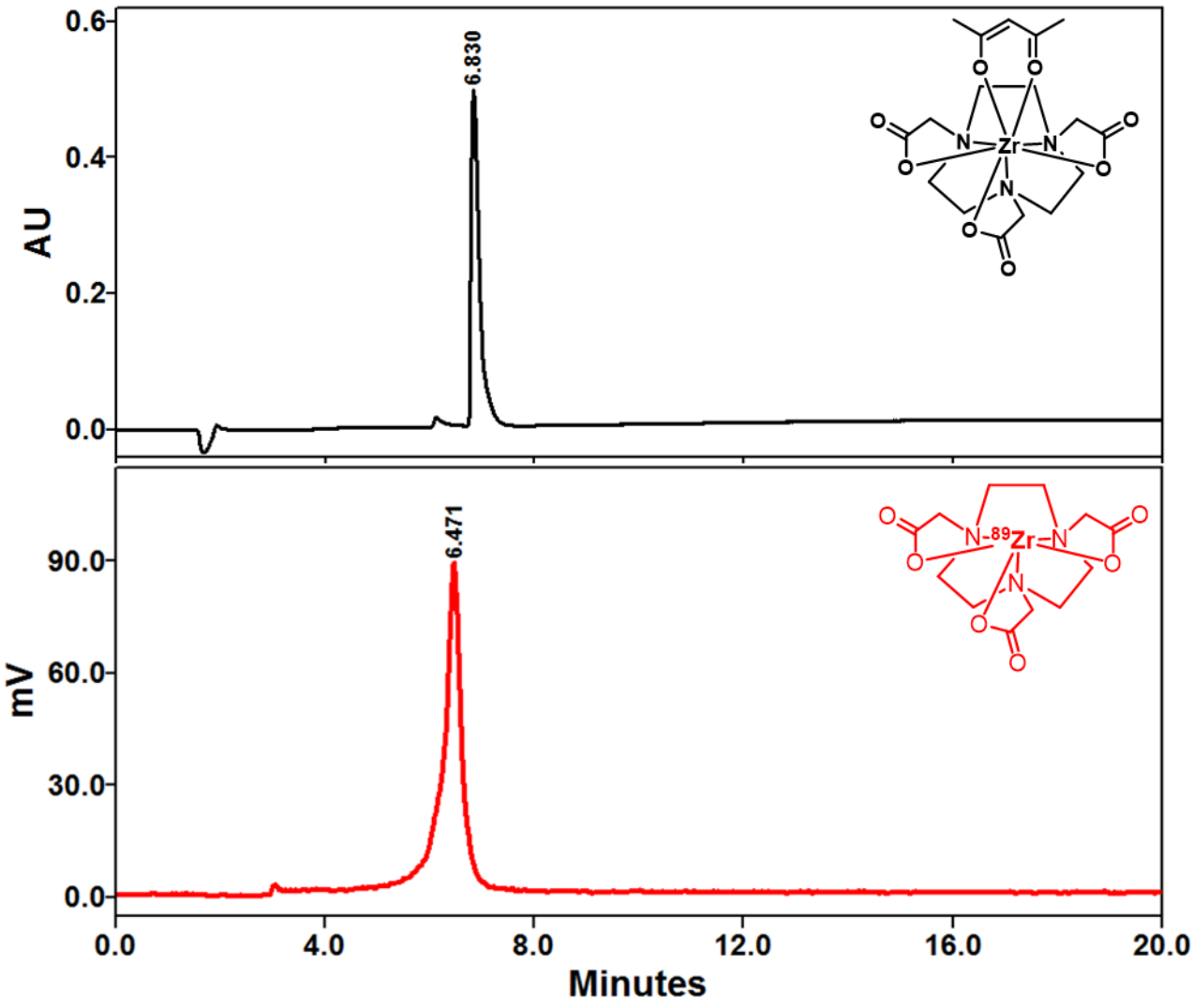


Figure S48. Quality control of [^{89}Zr]Zr-NOTA by radio-HPLC. UV-HPLC chromatogram (201 nm) of nonradioactive ^{Nat}Zr-NOTA complex (top) compared with radio-HPLC chromatogram of [^{89}Zr]Zr-NOTA (bottom)

Radiochemical Synthesis of [^{89}Zr]Zr-TRITA with [^{89}Zr]$ZrCl_4$:

Table S19. Conventional Heating: Summary of optimized radiochemistry conditions and [^{89}Zr]$ZrCl_4$ activity used to prepare [^{89}Zr]Zr-TRITA[a] complex (n = 4 for each buffer)

Quantity of Ligand	[^{89}Zr]$ZrCl_4$ added (MBq)	Reaction Buffer (pH 6.8 – 7.2)	Reaction Temperature (°C)	Reaction Time (min)	Radiochemical Yield by Radio-ITLC (%)
TRITA (10-15 µg)	17.3	1 M NH_4OAc	99	60	56.7 ± 2.1
	17.9	1 M NaOAc	99	60	43.2 ± 1.3
	17.5	1 M TRIS	99	60	22.2 ± 2.3
	17.1	1 M TMAA	99	60	16.8 ± 1.4
	17.4	0.5 M MES	99	60	62.9 ± 0.9
	18.0	0.5 M MES	99	120	78.7 ± 1.5
	17.7	0.5 M HEPES	99	60	66.8 ± 1.1
	18.1	**0.5 M HEPES**	**99**	**120**	**80.1 ± 1.2**

[a]TRITA ligand (10-15 µg) was labeled with [^{89}Zr]$ZrCl_4$ (17.1 – 18.1 MBq) using buffers (200 µL, pH 6.8 – 7.2) at 99 °C for 60–120 min.

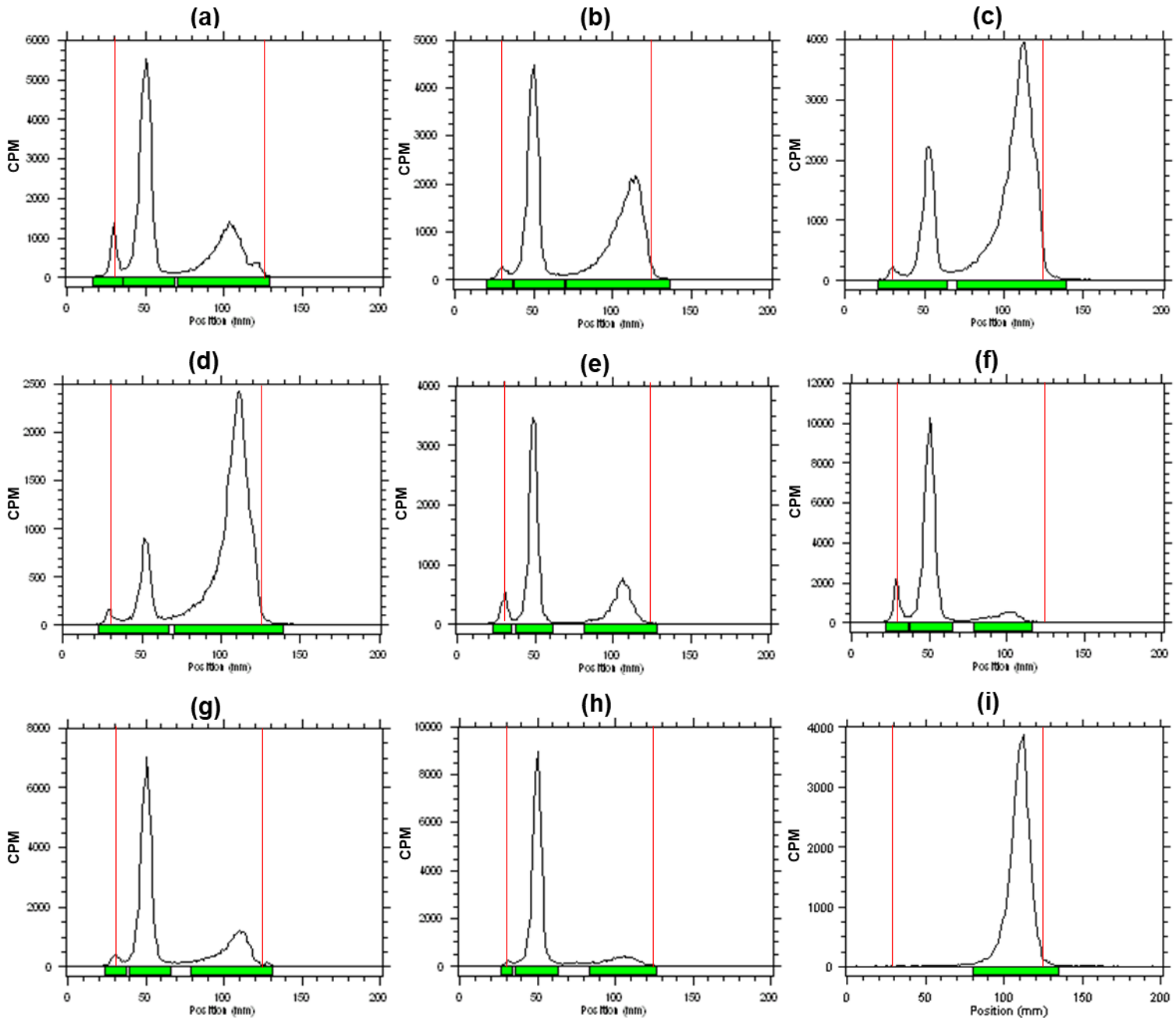


Figure S49. Conventional Heating: Radio-TLC analysis of [^{89}Zr]Zr-TRITA prepared by using different buffers at 99 °C for 60–120 min. (a) 1 M NH_4OAc, 99 °C, 60 min; (b) 1 M NaOAc, 99 °C, 60 min; (c) 1 M TRIS, 99 °C, 60 min; (d) 1 M TMAA, 99 °C, 60 min; (e) 0.5 M MES, 99 °C, 60 min; (f) 0.5 M MES, 99 °C, 120 min; (g) 0.5 M HEPES, 99 °C, 60 min; (h) 0.5 M HEPES, 99 °C, 120 min; (i) [^{89}Zr]$ZrCl_4$. In this ITLC-SA system, free ^{89}Zr formed a complex with EDTA and eluted with the solvent front (R_f ~ 1), [^{89}Zr]Zr-TRITA complex moved from origin (R_f = 0.20-0.25).

Table S20. Microwave Heating: Summary of optimized radiochemistry conditions and [^{89}Zr]$ZrCl_4$ activity used to prepare [^{89}Zr]Zr-TRITA[a] complex (n = 4 for each buffer)

Quantity of Ligand	[^{89}Zr]$ZrCl_4$ added (MBq)	Final Reaction pH	Reaction Temperature (°C)	Reaction Time (min)	Radiochemical Yield by Radio-ITLC (%)
TRITA (10-12 µg)	15.4	6.8-7.2	150	30	53.3 ± 1.7
	15.2	5.8-6.1	150	30	75.5 ± 0.9
	15.3	4.9-5.1	150	30	89.5 ± 0.8
	15.1	3.8-4.2	150	30	93.1 ± 0.6
	15.6	2.9-3.2	150	30	85.5 ± 1.3
	15.7	3.8-4.1	170	30	98.5 ± 0.3
	15.5	**3.9-4.2**	**180**	**20**	**99.9 ± 0.1**

[a]TRITA ligand (10-12 µg) was labeled with [^{89}Zr]$ZrCl_4$ (15.1 – 15.7 MBq) using 0.5 M HEPES buffer (50-70 µL, pH 7.1 -7.3) in Biotage Initiator microwave synthesizer at 150-180 °C for 20-30 min.

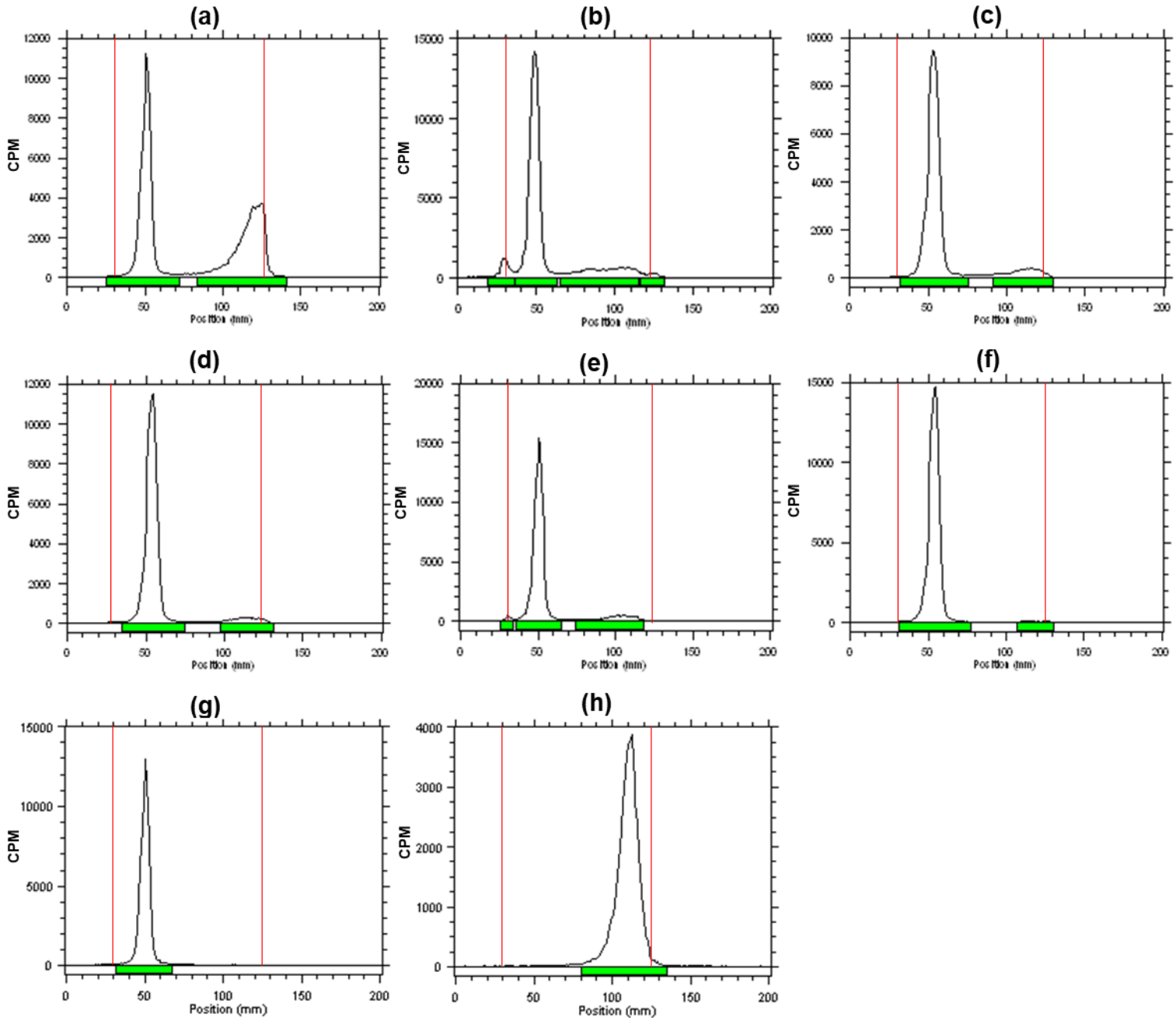


Figure S50. Microwave Heating: Radio-TLC analysis of [^{89}Zr]Zr-TRITA prepared with different reaction pH (7.2-2.9) at 150-180 °C for 20-30 min. (a) pH 6.8-7.2, 150 °C, 30 min; (b) pH 5.8-6.1, 150 °C, 30 min; (c) pH 4.9-5.1, 150 °C, 30 min; (d) pH 3.8-4.2, 150 °C, 30 min; (e) pH 2.9-3.2, 150 °C, 30 min; (f) pH 3.8-4.1, 170 °C, 30 min; (g) pH 3.9-4.2, 180 °C, 30 min; (h) [^{89}Zr]$ZrCl_4$. In this ITLC-SA system, free ^{89}Zr formed a complex with EDTA and eluted with the solvent front (R_f ~ 1), [^{89}Zr]Zr-TRITA complex moved from origin (R_f = 0.20-0.25).

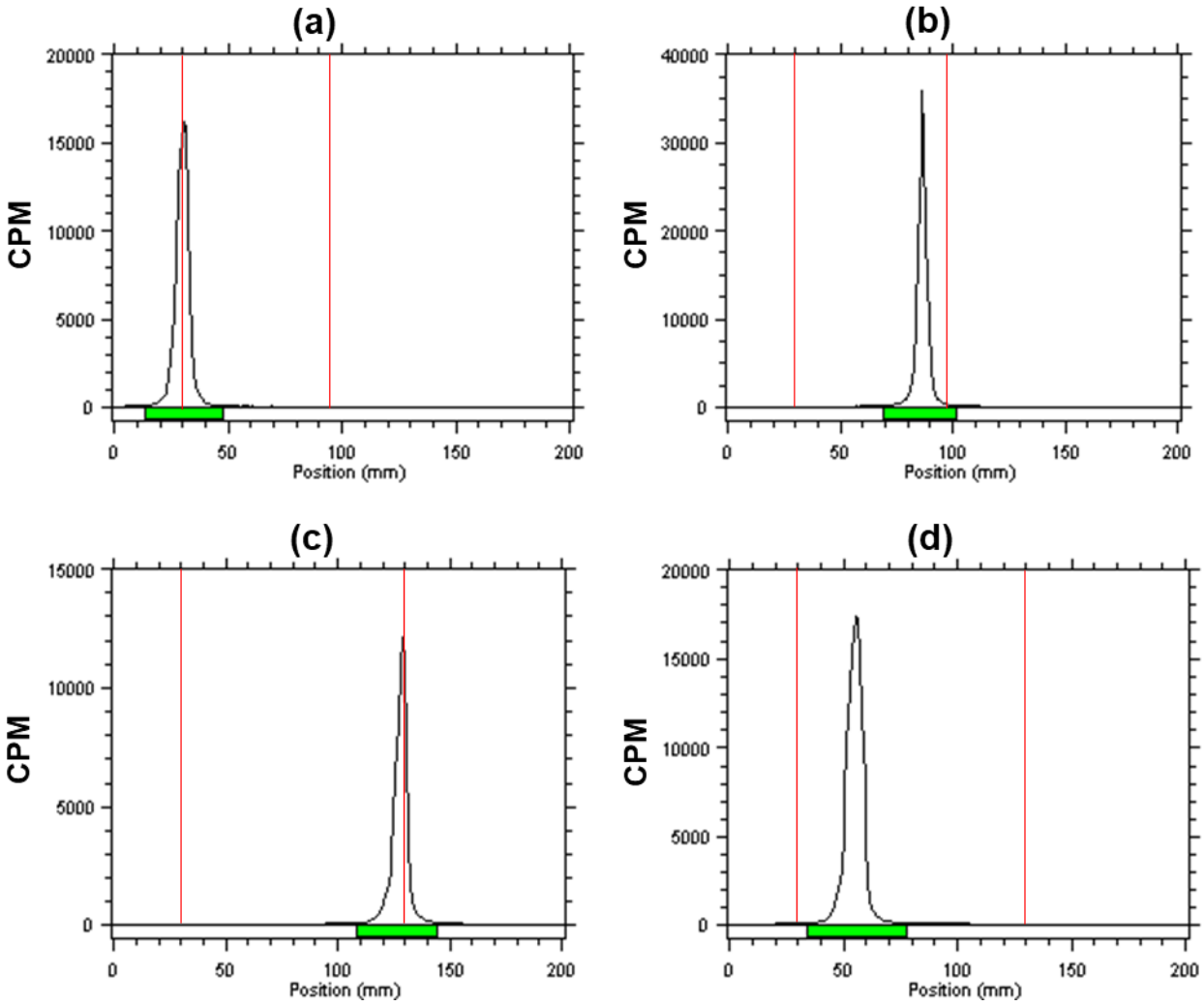


Figure S51. Quality control of [^{89}Zr]Zr-TRITA by radio-TLC. C-18-TLC of [^{89}Zr]$ZrCl_4$ (a), [^{89}Zr]Zr-TRITA (b), and ITLC-SA of [^{89}Zr]$ZrCl_4$ (c), [^{89}Zr]Zr-TRITA (d)

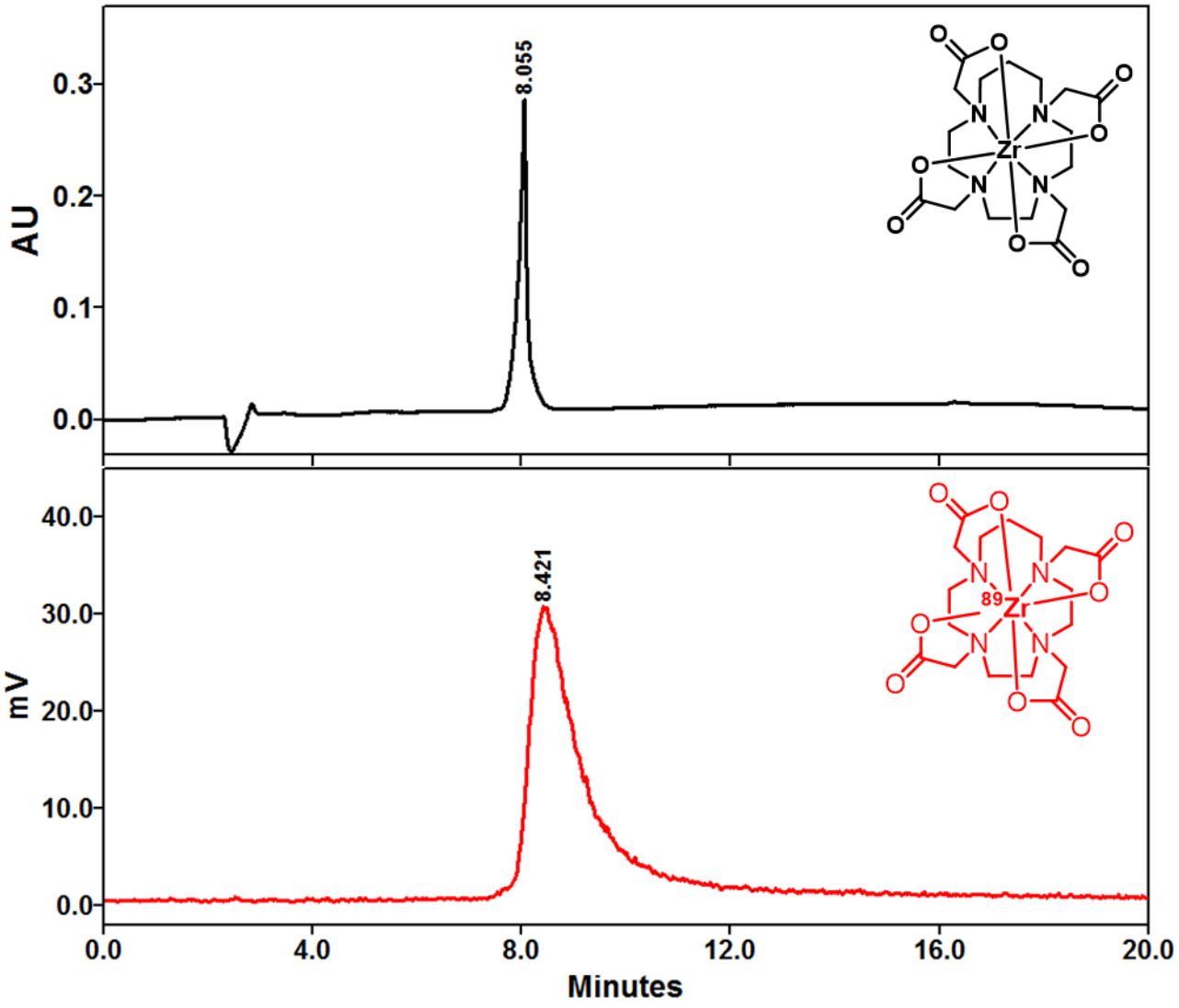


Figure S52. Quality control of [^{89}Zr]Zr-TRITA by radio-HPLC. UV-HPLC chromatogram (201 nm) of nonradioactive ^{Nat}Zr-TRITA complex (top) compared with radio-HPLC chromatogram of [^{89}Zr]Zr-TRITA (bottom)

Radiochemical Synthesis of [^{89}Zr]Zr-TETA with [^{89}Zr]$ZrCl_4$:

Table S21. Summary of optimized radiochemistry conditions and [^{89}Zr]$ZrCl_4$ activity used to prepare [^{89}Zr]Zr-TETA[a] complex (n = 4 for each buffer)

Quantity of Ligand	[^{89}Zr]$ZrCl_4$ added (MBq)	Reaction Buffer (pH 6.8 - 7.2)	Reaction Temperature (°C)	Reaction Time (min)	Radiochemical Yield by Radio-ITLC (%)
TETA (10-50 µg)	16.6	1 M NH_4OAc	99	120	0
	16.8	1 M NaOAc	99	120	0
	17.5	1 M TRIS	99	120	0
	19.2	0.5 M MES	99	120	0
	18.7	0.5 M HEPES	99	120	0.5 ± 0.1
	20.5	0.5 M HEPES	180[b]	30	1.5 ± 0.3

[a]TETA ligand (10-50 µg) was labeled with [^{89}Zr]$ZrCl_4$ (16.6 – 20.5 MBq) using buffers (200 µL, pH 6.8 - 7.2) at 99 °C for 120 min.
[b]TETA ligand (10-50 µg) was labeled with [^{89}Zr]$ZrCl_4$ (20.5 MBq) using 0.5 M HEPES buffer (50-70 µL, pH 7.1 - 7.3) in Biotage Initiator microwave synthesizer at 180 °C for 30 min.

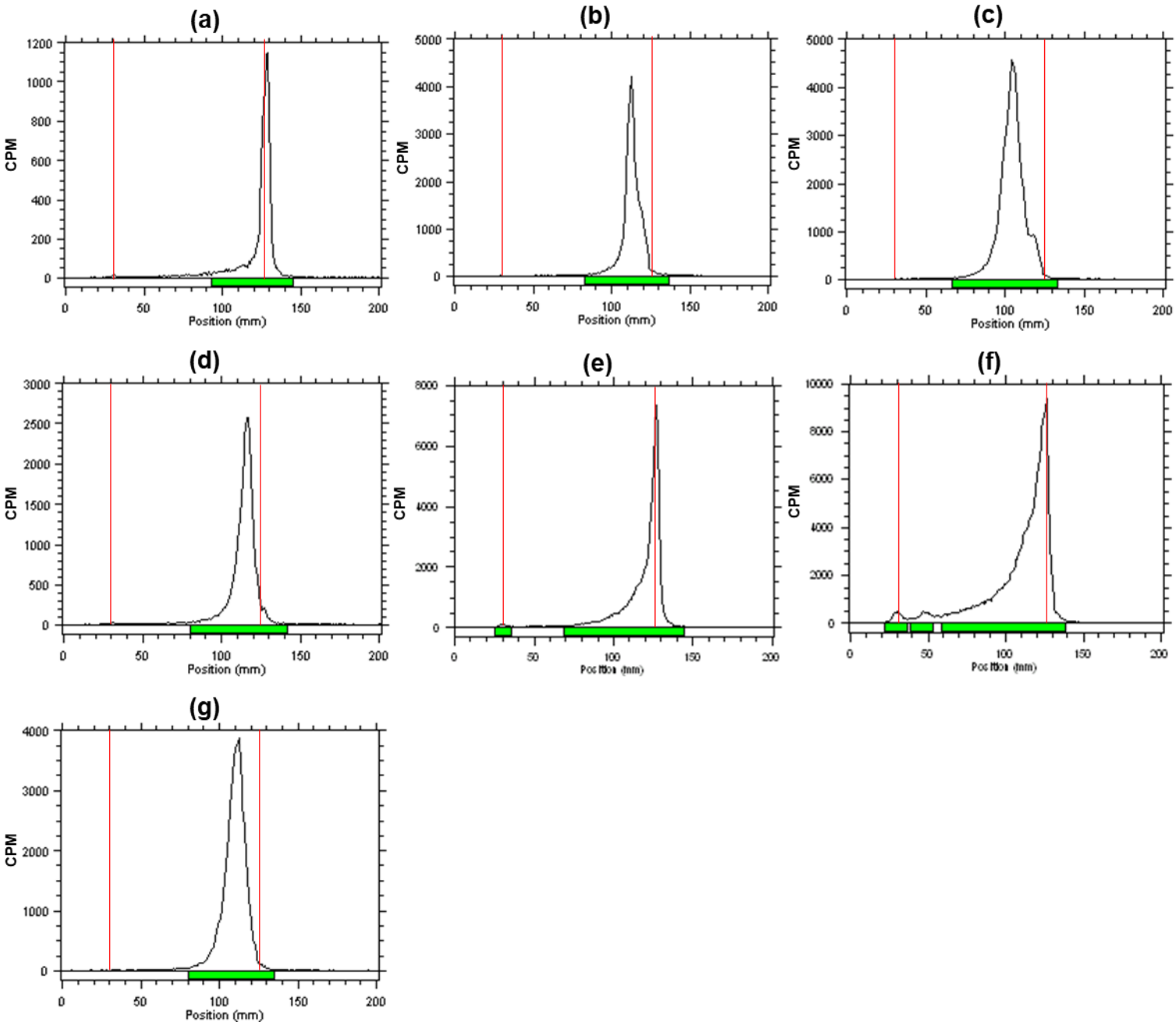


Figure S53. Radio-TLC analysis of [^{89}Zr]Zr-TETA prepared by using different buffers at 99 °C for 120 min. (a) 1 M NH_4OAc; (b) 1 M NaOAc; (c) 1 M TRIS; (d) 0.5 M MES; (e) 0.5 M HEPS (Conventional heating); (f) 0.5 M HEPES (Microwave heating); (g) [^{89}Zr]$ZrCl_4$. In this ITLC-SA system, free ^{89}Zr formed a complex with EDTA and eluted with the solvent front (R_f ~ 1), while [^{89}Zr]Zr-TETA complex remained at the origin (R_f ~ 0)

Table S22. Log P[7] values for all [^{89}Zr]Zr-complexes

Complex	Log P (n=5)
[^{89}Zr]Zr-PCTA	-3.09 ± 0.03
[^{89}Zr]Zr-NOTA	-2.51 ± 0.01
[^{89}Zr]Zr-TRITA	-3.13 ± 0.02

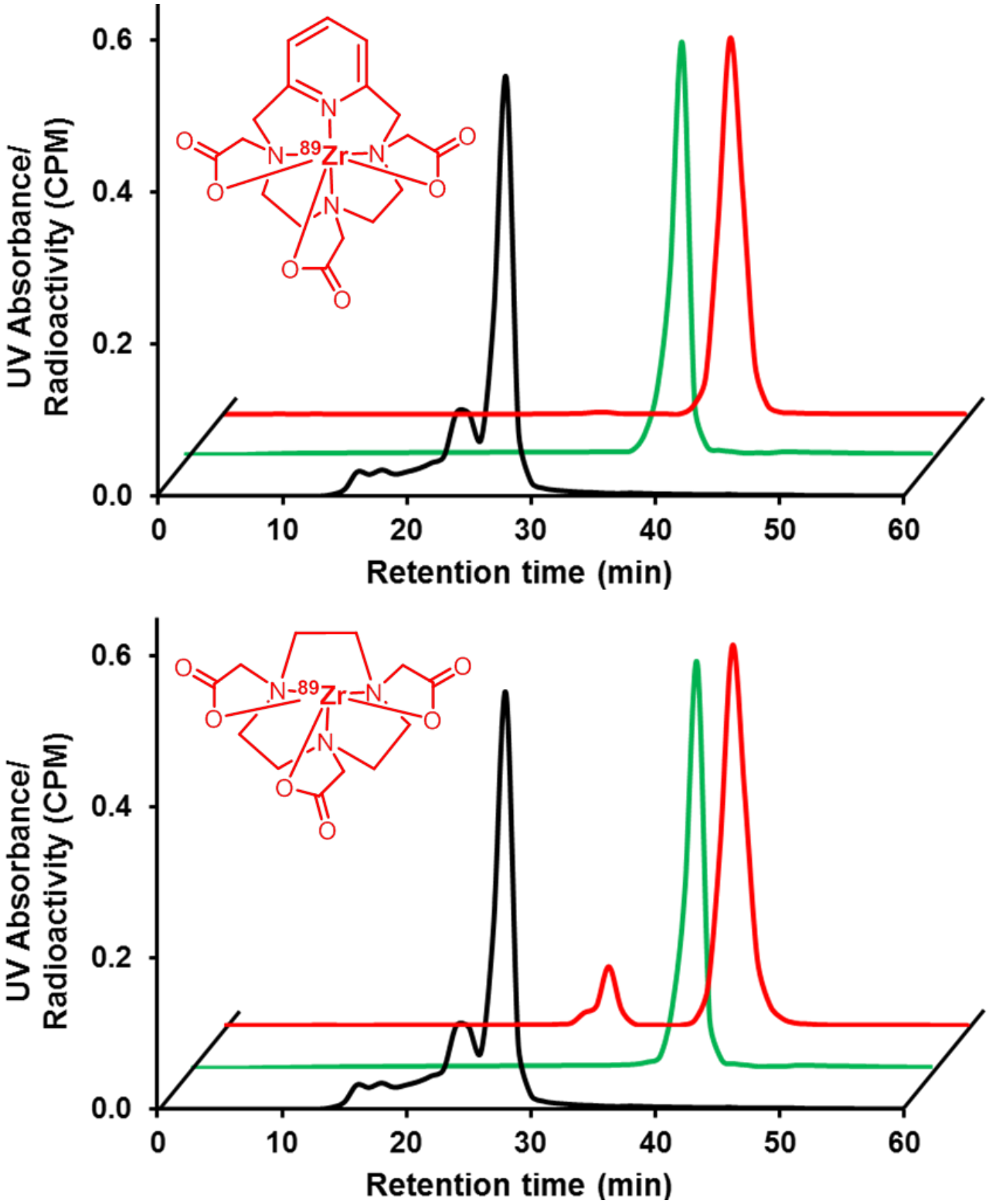


Figure S54. *In vitro* serum stability by SE-HPLC[8]. UV-SE-HPLC (220 nm, black, and green) and radio-SE-HPLC chromatogram (red) of [^{89}Zr]Zr-PCTA (top), and [^{89}Zr]Zr-NOTA (bottom) in serum after 7 days. Black lines are the UV absorbance due to the human serum components; green lines are the UV absorbance associated with ^{Nat}Zr-complexes and red lines are the radiotracer associated with [^{89}Zr]Zr-complexes.

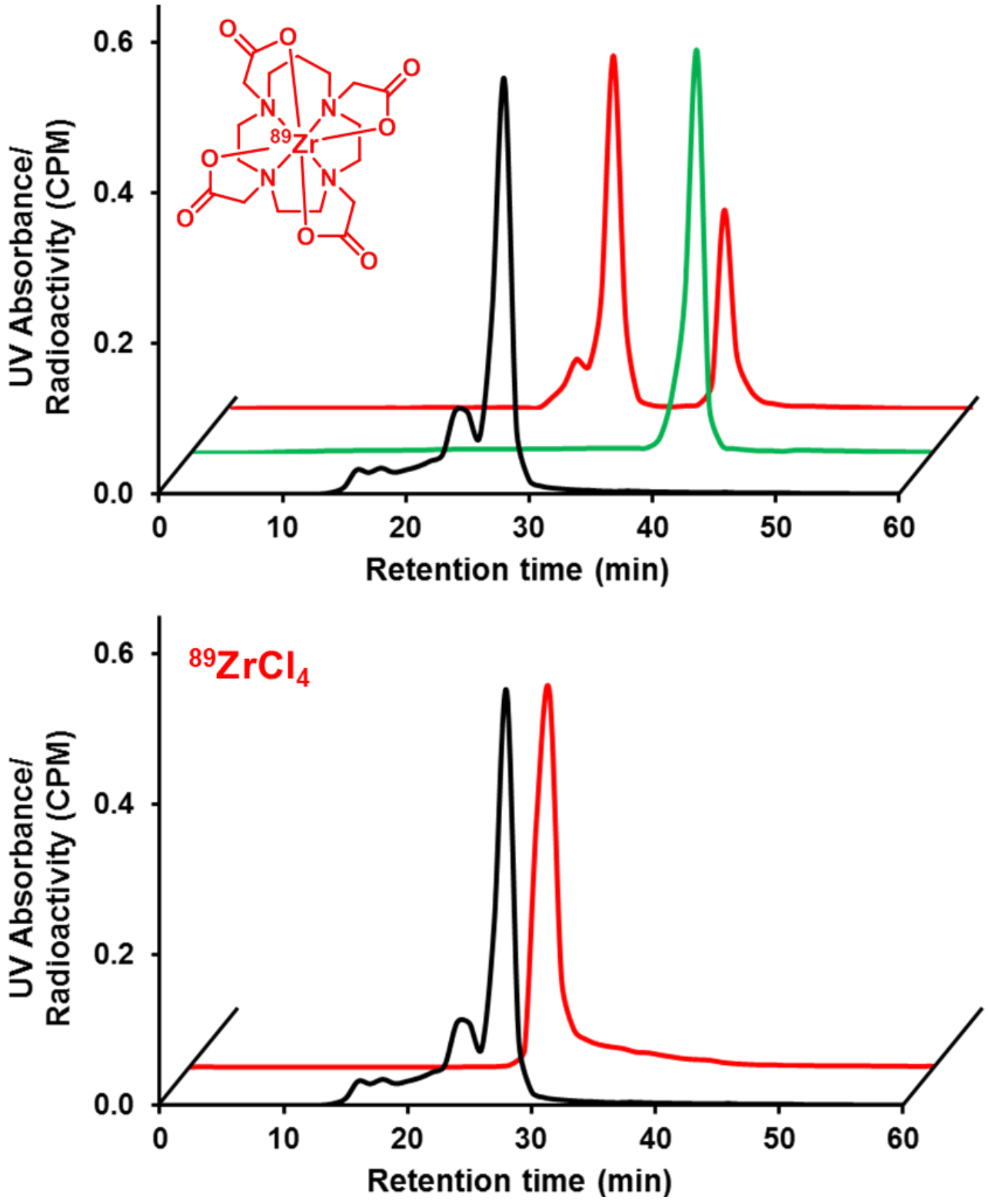


Figure S55. *In vitro* serum stability by SE-HPLC[8]. UV-SE-HPLC (220 nm, black, and green) and radio-SE-HPLC chromatogram (red) of [^{89}Zr]Zr-TRITA (top), and [^{89}Zr]$ZrCl_4$ (bottom) in serum after 7 days. Black lines are the UV absorbance due to the human serum components; green lines are the UV absorbance associated with ^{Nat}Zr-complexes and red lines are the radiotracer associated with ^{89}Zr-complexes.

Table S23. Biodistribution (%ID/g)[9-10] of [^{89}Zr]Zr-PCTA in selected organs at 2, 4, 24, 48, and 72 h p.i.

Tissue/Organ	2 h	4 h	24 h	48 h	72 h
Blood	0.013 ± 0.003	0.008 ± 0.002	0.002 ± 0.001	0.000 ± 0.001	0.000 ± 0.001
Heart	0.024 ± 0.003	0.020 ± 0.004	0.010 ± 0.003	0.008 ± 0.002	0.007 ± 0.002
Lung	0.061 ± 0.006	0.052 ± 0.005	0.026 ± 0.003	0.017 ± 0.002	0.014 ± 0.003
Liver	0.131 ± 0.005	0.104 ± 0.010	0.056 ± 0.003	0.037 ± 0.004	0.030 ± 0.003
Small intestine	0.122 ± 0.023	0.058 ± 0.016	0.012 ± 0.001	0.006 ± 0.001	0.004 ± 0.000
Large intestine	0.193 ± 0.060	0.413 ± 0.061	0.037 ± 0.008	0.011 ± 0.003	0.006 ± 0.001
Kidney	0.722 ± 0.068	0.571 ± 0.050	0.416 ± 0.016	0.272 ± 0.031	0.151 ± 0.010
Spleen	0.047 ± 0.007	0.041 ± 0.010	0.035 ± 0.004	0.026 ± 0.004	0.024 ± 0.003
Pancreas	0.016 ± 0.003	0.015 ± 0.003	0.009 ± 0.002	0.007 ± 0.002	0.003 ± 0.002
Stomach	0.027 ± 0.006	0.026 ± 0.005	0.009 ± 0.002	0.004 ± 0.001	0.003 ± 0.001
Muscle	0.013 ± 0.004	0.007 ± 0.002	0.006 ± 0.002	0.002 ± 0.001	0.001 ± 0.001
Fat	0.012 ± 0.001	0.008 ± 0.002	0.007 ± 0.002	0.006 ± 0.002	0.002 ± 0.002
Bone	0.104 ± 0.015	0.066 ± 0.010	0.113 ± 0.009	0.105 ± 0.007	0.094 ± 0.006

Table S24. Biodistribution (%ID/g)[9-10] of [^{89}Zr]Zr-NOTA in selected organs at 2, 4, 24, 48, and 72 h p.i.

Tissue/Organ	2 h	4 h	24 h	48 h	72 h
Blood	0.029 ± 0.002	0.014 ± 0.002	0.003 ± 0.001	0.001 ± 0.000	0.001 ± 0.000
Heart	0.037 ± 0.002	0.027 ± 0.003	0.015 ± 0.001	0.011 ± 0.002	0.007 ± 0.002
Lung	0.109 ± 0.008	0.068 ± 0.004	0.034 ± 0.003	0.017 ± 0.002	0.016 ± 0.001
Liver	0.591 ± 0.011	0.450 ± 0.025	0.158 ± 0.010	0.086 ± 0.007	0.069 ± 0.007
Small intestine	0.304 ± 0.025	0.174 ± 0.021	0.016 ± 0.001	0.008 ± 0.001	0.006 ± 0.001
Large intestine	0.857 ± 0.061	1.053 ± 0.092	0.052 ± 0.012	0.018 ± 0.003	0.013 ± 0.003
Kidney	1.052 ± 0.012	0.884 ± 0.020	0.466 ± 0.023	0.343 ± 0.045	0.301 ± 0.023
Spleen	0.057 ± 0.007	0.035 ± 0.004	0.028 ± 0.002	0.024 ± 0.005	0.021 ± 0.001
Pancreas	0.016 ± 0.002	0.016 ± 0.001	0.008 ± 0.002	0.006 ± 0.002	0.005 ± 0.001
Stomach	0.061 ± 0.014	0.053 ± 0.014	0.009 ± 0.003	0.005 ± 0.001	0.004 ± 0.001
Muscle	0.017 ± 0.004	0.011 ± 0.002	0.006 ± 0.003	0.004 ± 0.002	0.003 ± 0.002
Fat	0.014 ± 0.004	0.012 ± 0.002	0.009 ± 0.001	0.004 ± 0.002	0.004 ± 0.001
Bone	0.092 ± 0.009	0.069 ± 0.009	0.080 ± 0.008	0.051 ± 0.010	0.046 ± 0.006

Table S25. Biodistribution (%ID/g)[9-10] of [^{89}Zr]Zr-TRITA in selected organs at 2, 4, 24, 48, and 72 h p.i.

Tissue/Organ	2 h	4 h	24 h	48 h	72 h
Blood	1.249 ± 0.358	1.142 ± 0.349	0.042 ± 0.010	0.010 ± 0.003	0.004 ± 0.008
Heart	0.378 ± 0.065	0.314 ± 0.084	0.058 ± 0.008	0.066 ± 0.012	0.061 ± 0.009
Lung	0.725 ± 0.128	0.583 ± 0.187	0.144 ± 0.052	0.141 ± 0.043	0.154 ± 0.045
Liver	0.744 ± 0.145	0.543 ± 0.118	0.316 ± 0.047	0.380 ± 0.045	0.304 ± 0.022
Small intestine	0.457 ± 0.136	0.345 ± 0.062	0.062 ± 0.013	0.054 ± 0.007	0.030 ± 0.005
Large intestine	0.553 ± 0.141	0.804 ± 0.176	0.073 ± 0.016	0.051 ± 0.007	0.034 ± 0.005
Kidney	1.271 ± 0.218	1.228 ± 0.213	0.681 ± 0.015	0.744 ± 0.038	0.588 ± 0.036
Spleen	0.552 ± 0.194	0.287 ± 0.084	0.100 ± 0.012	0.161 ± 0.030	0.128 ± 0.016
Pancreas	0.364 ± 0.065	0.274 ± 0.037	0.119 ± 0.022	0.136 ± 0.026	0.103 ± 0.018
Stomach	0.198 ± 0.038	0.166 ± 0.039	0.040 ± 0.014	0.042 ± 0.010	0.020 ± 0.004
Muscle	0.224 ± 0.044	0.173 ± 0.026	0.063 ± 0.017	0.061 ± 0.035	0.037 ± 0.008
Fat	0.215 ± 0.049	0.153 ± 0.023	0.069 ± 0.014	0.091 ± 0.014	0.064 ± 0.024
Bone	2.058 ± 0.459	3.329 ± 0.220	6.043 ± 0.730	10.805 ± 0.803	8.041 ± 1.470

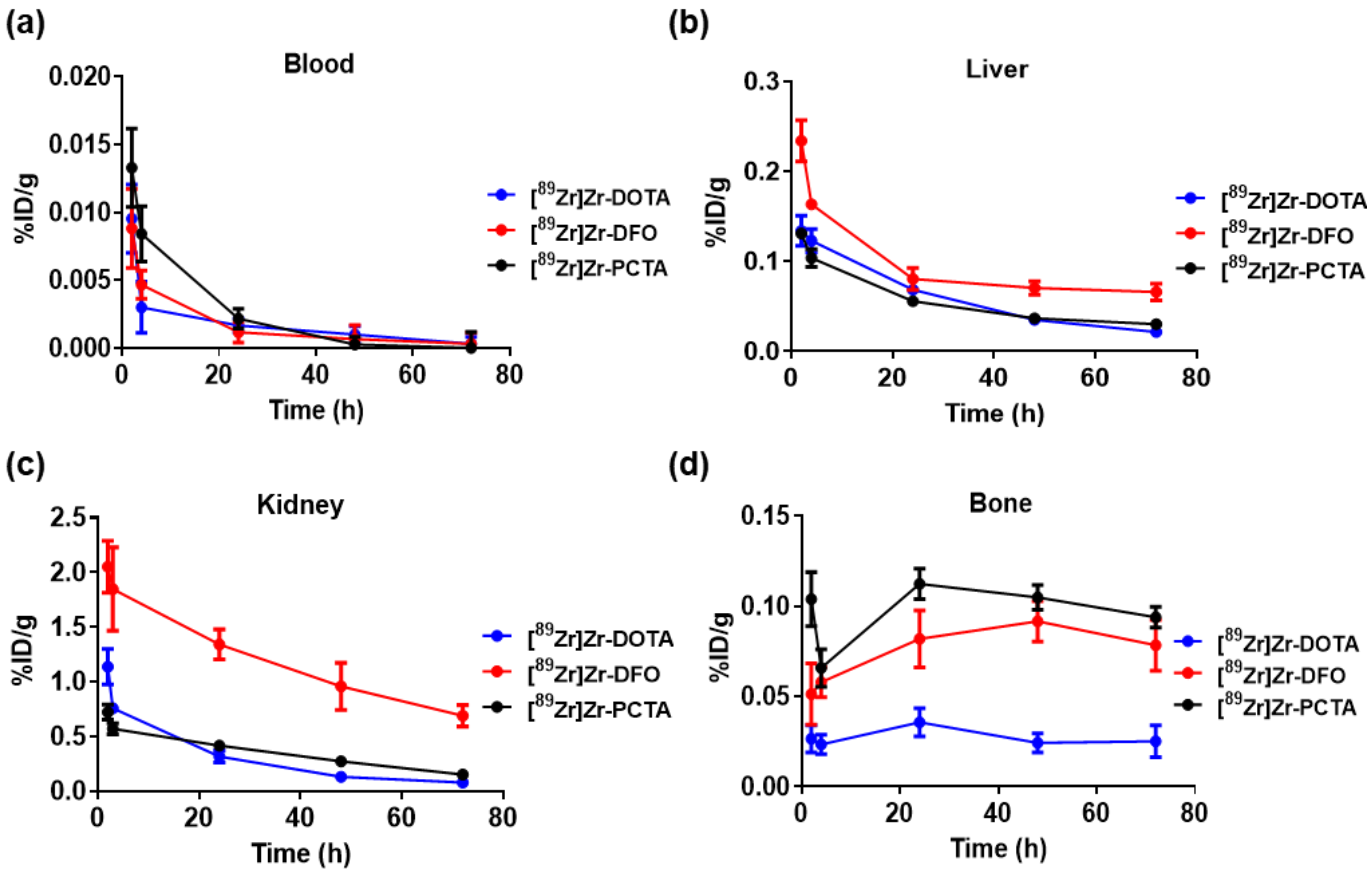


Figure S56. Biodistribution data summary of [^{89}Zr]Zr-DOTA[8], [^{89}Zr]Zr-DFO[9], and [^{89}Zr]Zr-PCTA in selected tissues: (a) blood, (b) liver, (c) kidney, and (d) bone.

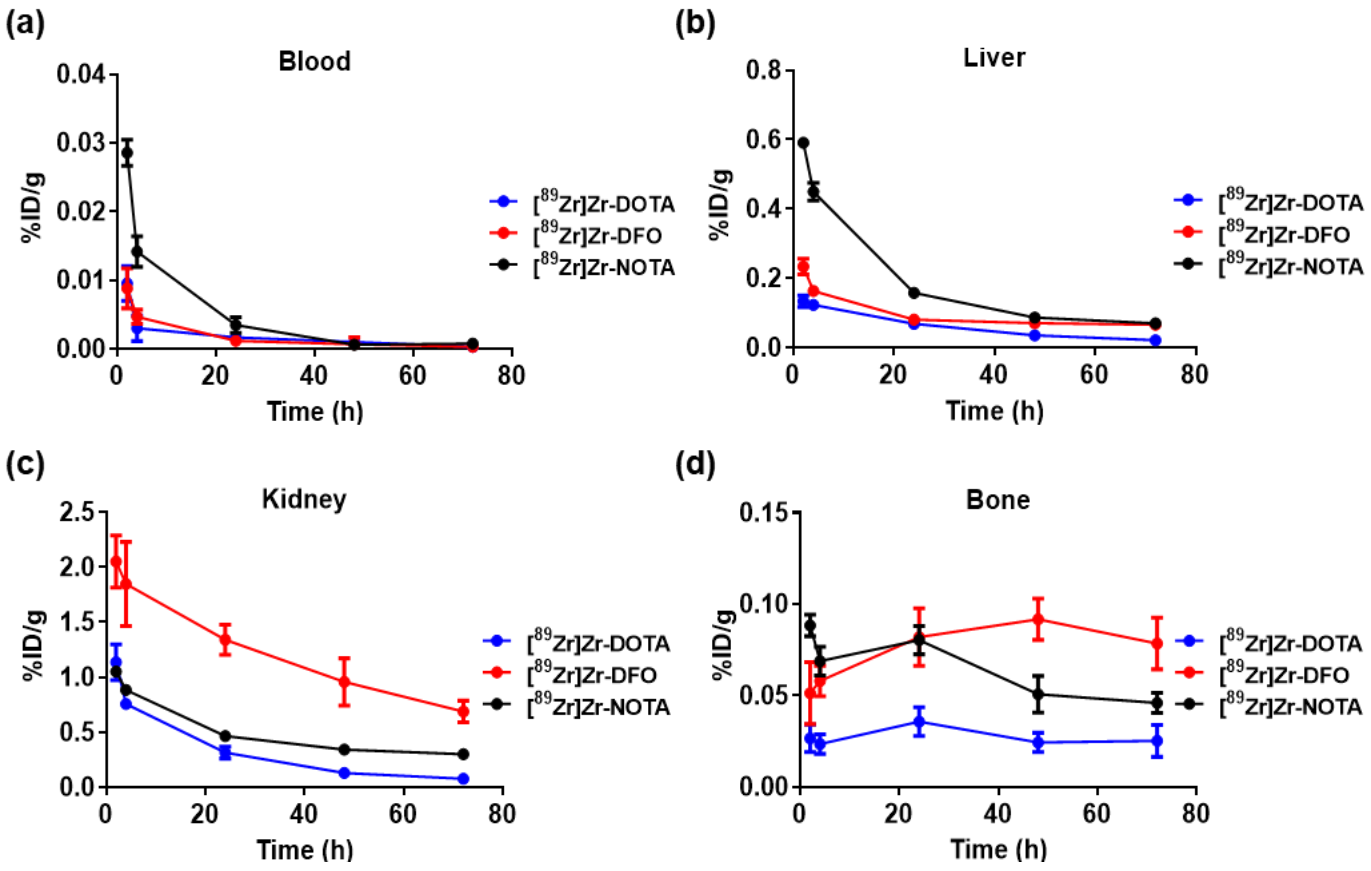


Figure S57. Biodistribution data summary of [^{89}Zr]Zr-DOTA[8], [^{89}Zr]Zr-DFO[9], and [^{89}Zr]Zr-NOTA in selected tissues: (a) blood, (b) liver, (c) kidney, and (d) bone.

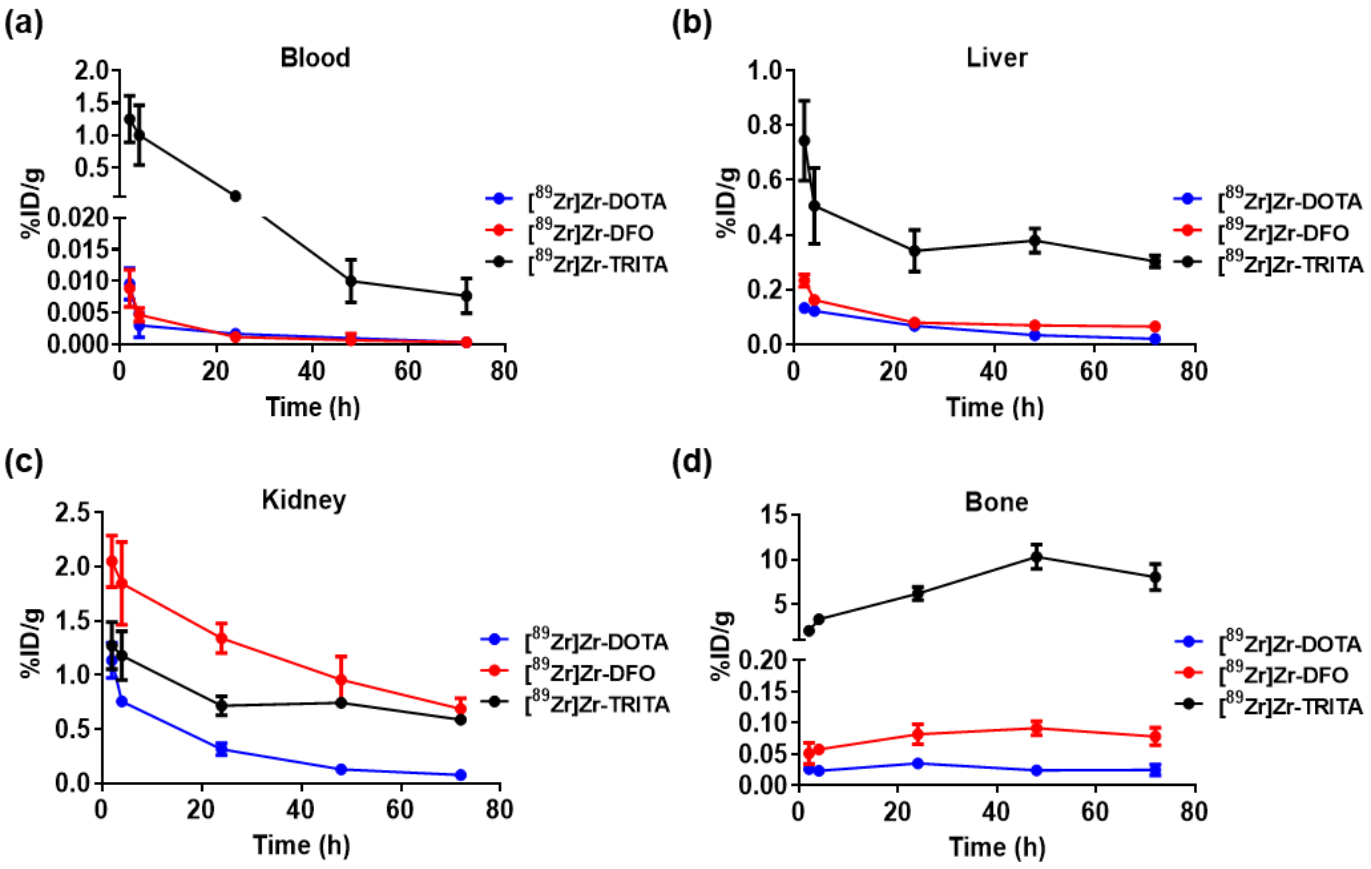


Figure S58. Biodistribution data summary of [^{89}Zr]Zr-DOTA[8], [^{89}Zr]Zr-DFO[9], and [^{89}Zr]Zr-TRITA in selected tissues: (a) blood, (b) liver, (c) kidney, and (d) bone.

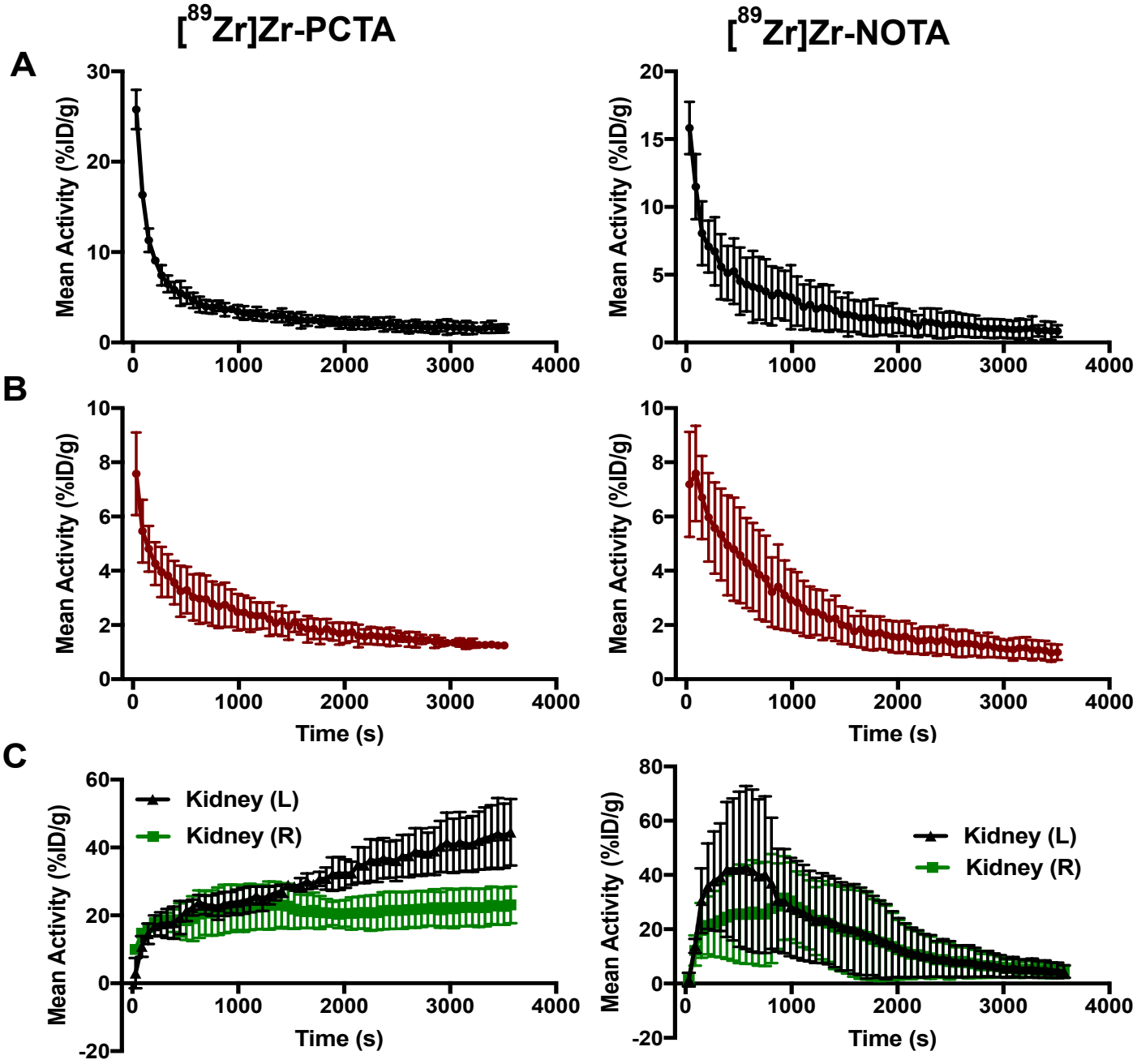


Figure S59. Dynamic PET data of [^{89}Zr]Zr-PCTA and [^{89}Zr]Zr-NOTA collected over the course of 1 h in specific tissues. Rapid perfusion and subsequent washout is represent via the blood (A) and liver (B). Slow accumulation is shown in the kidneys (C) to enable rapid clearance, which is further depicted in static PET images at later time points.

Table S26. Comparative stability of [^{89}Zr]Zr-Complexes in exogenous ligand challenge study

[^{89}Zr]Zr-Complexes	Challenging Ligand	pH	Time Point	% Intact of ^{89}Zr-complexes
[^{89}Zr]Zr-L4[11]	EDTA (1000-fold)	7.0	6 d	87 ± 1
[^{89}Zr]Zr-TAM-1[9]	DTPA (1000-fold)	7.0	7 d	100
[^{89}Zr]Zr-TAM-2[9]	DTPA (1000-fold)	7.0	7 d	100
[^{89}Zr]Zr-2,3-HOPO[12]	DTPA (1000-fold)	7.0	7 d	78
[^{89}Zr]Zr-C7[13]	EDTA (1750-fold)	7.0	7 d	87 ± 3
[^{89}Zr]Zr-CP256[14]			Data Not Reported	
[^{89}Zr]Zr-TAFC[15]	EDTA (1000-fold)	7.0	7 d	97.2 ± 0.2
		6.0	7 d	94.4 ± 0.5
[^{89}Zr]Zr-DFO*[16]			Data Not Reported	
[^{89}Zr]Zr-DFOSq-Taur[17]	EDTA (500-fold)	7.0	1 d	88 ± 3.2
[^{89}Zr]Zr-(oxinate)$_4$[18]			Data Not Reported	
[^{89}Zr]Zr-(Me-AHA)$_4$[19]			Data Not Reported	
[^{89}Zr]Zr-THPN[20]	EDTA (100-fold)	7.0	7 d	100
		5.0	7 d	41.6 ± 11.1
[^{89}Zr]Zr-DFO-HOPO[21]	EDTA (100-fold)	7.0	7 d	> 99
[^{89}Zr]Zr-4HMS[22]	DTPA (1000-fold)	7.0	7 d	91.9 ± 0.1
		5.0	7 d	80.8 ± 1.8
[^{89}Zr]Zr-(DFO2)[23]	EDTA (100-fold)	7.0	7 d	85.3 ± 2.7
		5.0	7 d	0.6 ± 0.2
[^{89}Zr]Zr-HOPO[24]	EDTA (100-fold)	7.0	7 d	100
		5.0	7 d	99.2 ± 1.5
[^{89}Zr]Zr-DFO[8]	EDTA (1000-fold)	7.0	7 d	20.3 ± 0.5
		5.0	7 d	0
[^{89}Zr]Zr-DOTA[8]	EDTA (1000-fold)	7.0	7 d	100
		5.0	7 d	100
[^{89}Zr]Zr-PCTA (Current Study)	EDTA (1000-fold)	7.0	7 d	100
		5.0	7 d	100
[^{89}Zr]Zr-NOTA (Current Study)	EDTA (1000-fold)	7.0	7 d	70.7 ± 1.0
		5.0	7 d	65.3 ± 1.2
[^{89}Zr]Zr-TRITA (Current Study)	EDTA (1000-fold)	7.0	7 d	0
		5.0	7 d	9.6 ± 0.1

Table S27. Comparative stability of [^{89}Zr]Zr-Complexes with various metals in competition study

[^{89}Zr]Zr-Complexes	Time Point	% Intact of [^{89}Zr]Zr-Complexes						
		Fe^{3+}	Zn^{2+}	Co^{2+}	Cu^{2+}	Mg^{2+}	Gd^{3+}	Ga^{3+}
[^{89}Zr]Zr-L4[11]		Data Not Reported						
[^{89}Zr]Zr-TAM-1[9]		Data Not Reported						
[^{89}Zr]Zr-TAM-2[9]		Data Not Reported						
[^{89}Zr]Zr-2,3-HOPO[12]		Data Not Reported						
[^{89}Zr]Zr-C7[13]		Data Not Reported						
[^{89}Zr]Zr-CP256[14]	20 min	14	Data Not Reported					
[^{89}Zr]Zr-TAFC[15]		Data Not Reported						
[^{89}Zr]Zr-DFO*[16]		Data Not Reported						
[^{89}Zr]Zr-DFOSq-Taur[17]		Data Not Reported						
[^{89}Zr]Zr-(oxinate)$_4$[18]		Data Not Reported						
[^{89}Zr]Zr-(Me-AHA)$_4$[19]		Data Not Reported						
[^{89}Zr]Zr-THPN[20]		Data Not Reported						
[^{89}Zr]Zr-DFO-HOPO[21]		Data Not Reported						
[^{89}Zr]Zr-4HMS[22]	7 d	97.5 ± 1.0	-	99.0 ± 0.7	99.6 ± 0.4	99.6 ± 0.4	-	-
[^{89}Zr]Zr-(DFO2)[23]	7 d	~ 30	Data Not Reported					
[^{89}Zr]Zr-HOPO[24]	7 d	83.0 ± 4.2	98.3 ± 2.4	98.9 ± 1.6	98.4 ± 2.3	98.7 ± 1.8	94.5 ± 4.1	96.4 ± 0.6
[^{89}Zr]Zr-DFO[8]	7 d	33.9 ± 1.5	95.9 ± 0.7	95.4 ± 0.9	96.0 ± 1.0	96.8 ± 1.2	96.8 ± 0.2	72.6 ± 1.3
[^{89}Zr]Zr-DOTA[8]	7 d	100	100	100	100	100	100	100
[^{89}Zr]Zr-PCTA (Current Study)	7 d	100	100	100	100	100	100	100
[^{89}Zr]Zr-NOTA (Current Study)	7 d	96.2 ± 0.2	90.4 ± 0.4	92.9 ± 0.7	87.8 ± 0.9	90.6 ± 0.4	96.3 ± 0.4	89.5 ± 1.5
[^{89}Zr]Zr-TRITA (Current Study)	7 d	70.4 ± 0.8	9.4 ± 0.7	7.3 ± 0.6	59.7 ± 0.5	83.9 ± 0.5	57.2 ± 0.6	61.8 ± 1.2

Table S28. Comparative biodistribution results of [^{89}Zr]Zr-Complexes

[^{89}Zr]Zr-Complexes	Time Point	% ID/g			
		Blood	Liver	Kidney	Bone
[^{89}Zr]Zr-L4[11]	24 h	0.09 ± 0.01	0.40 ± 0.14	2.76 ± 0.40	0.60 ± 0.19
[^{89}Zr]Zr-TAM-1[9]	24 h	0.003 ± 0.002	0.449 ± 0.037	8.214 ± 1.018	0.100 ± 0.030
[^{89}Zr]Zr-TAM-2[9]	24 h	0.010 ± 0.003	1.244 ± 0.180	46.095 ± 7.788	0.274 ± 0.100
[^{89}Zr]Zr-2,3-HOPO[12]	24 h	0.004 ± 0.001	0.650 ± 0.080	29.191 ± 6.989	0.272 ± 0.066
[^{89}Zr]Zr-C7[13]	Data Not Reported				
[^{89}Zr]Zr-CP256[14]	Data Not Reported				
[^{89}Zr]Zr-TAFC[15]	6 h	0.05 ± 0.01	-	0.86 ± 0.48	0.04 ± 0.02
[^{89}Zr]Zr-DFO*[16]	Data Not Reported				
[^{89}Zr]Zr-DFOSq-Taur[17]	Data Not Reported				
[^{89}Zr]Zr-(oxinate)$_4$[18]	Data Not Reported				
[^{89}Zr]Zr-(Me-AHA)$_4$[19]	Data Not Reported				
[^{89}Zr]Zr-THPN[20]	24 h	0.01 ± 0.01	1.08 ± 1.25	3.49 ± 0.28	011 ± 0.01
[^{89}Zr]Zr-DFO-HOPO[21]	24 h	0.00 ± 0.00	0.02 ± 0.00	0.08 ± 0.01	0.01 ± 0.00
[^{89}Zr]Zr-4HMS[22]	24 h	0.00 ± 0.00	0.04 ± 0.00	0.80 ± 0.20	0.01 ± 0.00
[^{89}Zr]Zr-(DFO2)[23]	Data Not Reported				
[^{89}Zr]Zr-HOPO[24]	24 h	0.02 ± 0.00	0.06 ± 0.03	0.51 ± 0.29	0.17 ± 0.03
[^{89}Zr]Zr-DFO[8]	24 h	0.00 ± 0.00	0.08 ± 0.01	1.34 ± 0.14	0.08 ± 0.02
[^{89}Zr]Zr-DOTA[8]	24 h	0.00 ± 0.00	0.07 ± 0.00	0.32 ± 0.05	0.04 ± 0.01
[^{89}Zr]Zr-PCTA (Current Study)	24 h	0.00 ± 0.00	0.06 ± 0.00	0.42 ± 0.02	0.11 ± 0.01
[^{89}Zr]Zr-NOTA (Current Study)	24 h	0.00 ± 0.00	0.16 ± 0.01	0.47 ± 0.02	0.08 ± 0.01
[^{89}Zr]Zr-TRITA (Current Study)	24 h	0.04 ± 0.01	0.32 ± 0.05	0.68 ± 0.02	6.04 ± 0.73

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