Electronic Supplementary Information

Pentazolate coordination polymers self-assembled by *in situ* generated $[Pb_4(OH)_4]^{4+}$ cubic cations trapping *cyclo*-N_5^-

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1. Single-crystal X-ray diffraction analysis of CP **1**

Table S1. Crystal data, data collection, and refinement for CP **1**

$H_4N_{20}O_4Pb_4$	$F(000) = 1008$
$M_r = 1176.99$	$D_x = 4.537$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.6556$ (10) Å	Cell parameters from 6056 reflections
$b = 13.6507$ (17) Å	$\theta = 2.5$–$30.2°$
$c = 8.5962$ (11) Å	$\mu = 39.03$ mm^{-1}
$\beta = 106.440$ (4)°	$T = 296$ K
$V = 861.61$ (19) Å^3	Block, colourless
$Z = 2$	0.03 × 0.03 × 0.03 mm
Bruker D8 QUEST PHOTON 100 diffractometer	2850 reflections with $I > 2\sigma(I)$
Detector resolution: 10.42 pixels mm^{-1}	$R_{int} = 0.043$
ϕ and ω scans	$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
Absorption correction: multi-scan *SADABS*	$h = -9 \rightarrow 9$
$T_{min} = 0.407$, $T_{max} = 0.746$	$k = -16 \rightarrow 16$
9072 measured reflections	$l = -10 \rightarrow 10$
3013 independent reflections	
Refinement on F^2	Hydrogen site location: difference Fourier map
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.067$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.01$	$\Delta\rangle_{max} = 2.52$ e Å^{-3}
3013 reflections	$\Delta\rangle_{min} = -1.13$ e Å^{-3}
254 parameters	Absolute structure: Refined as an inversion twin.
41 restraints	Absolute structure parameter: 0.03 (3)

Table S2. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for **1**

	x	y	z	U_{iso}*/U_{eq}
Pb1	0.78330 (12)	0.62502 (6)	0.50629 (9)	0.0244 (2)
Pb2	0.94024 (12)	0.40915 (5)	0.80611 (9)	0.0251 (2)
Pb3	0.56478 (11)	0.57787 (6)	0.84131 (10)	0.0261 (2)
Pb4	1.04856 (12)	0.66947 (6)	0.95281 (9)	0.0262 (2)
O1	0.684 (2)	0.4953 (10)	0.6447 (16)	0.024 (3)

H1	0.600254	0.455301	0.597380	0.036*
O2	0.756 (2)	0.6877 (10)	0.7621 (16)	0.023 (3)
H2	0.740310	0.742912	0.714063	0.035*
O3	0.8636 (18)	0.5376 (10)	0.9777 (15)	0.020 (3)
H3	0.831273	0.499596	1.042238	0.030*
O4	1.0392 (18)	0.5639 (10)	0.7341 (16)	0.021 (3)
H4	1.097944	0.550980	0.667212	0.031*
N1	0.958 (3)	0.3791 (13)	0.499 (2)	0.028 (4)
N2	0.898 (3)	0.4515 (13)	0.404 (2)	0.027 (4)
N3	0.891 (3)	0.4232 (14)	0.258 (2)	0.032 (5)
N4	0.941 (3)	0.3295 (15)	0.264 (2)	0.036 (5)
N5	0.991 (3)	0.3050 (14)	0.416 (2)	0.032 (5)
N6	0.461 (3)	0.4410 (17)	0.320 (3)	0.046 (6)
N7	0.335 (3)	0.3884 (13)	0.220 (2)	0.032 (5)
N8	0.253 (3)	0.4425 (16)	0.097 (2)	0.039 (5)
N9	0.335 (3)	0.5260 (15)	0.121 (3)	0.045 (6)
N10	0.459 (3)	0.5257 (15)	0.253 (2)	0.037 (5)
N11	0.403 (5)	0.366 (2)	0.802 (3)	0.069 (8)
N12	0.560 (5)	0.352 (2)	0.898 (4)	0.073 (9)
N13	0.565 (3)	0.2685 (19)	0.975 (3)	0.054 (7)
N14	0.397 (4)	0.2361 (17)	0.927 (3)	0.056 (7)
N15	0.295 (3)	0.294 (3)	0.823 (3)	0.076 (10)
N16	0.389 (4)	0.724 (2)	0.444 (4)	0.069 (5)
N17	0.374 (4)	0.656 (2)	0.546 (3)	0.066 (5)
N18	0.216 (4)	0.614 (2)	0.488 (3)	0.067 (5)
N19	0.131 (4)	0.660 (2)	0.349 (3)	0.076 (6)
N20	0.245 (5)	0.725 (2)	0.328 (3)	0.071 (5)

Table S3. Atomic displacement parameters (Å^2) for **1**

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0390 (5)	0.0187 (4)	0.0147 (4)	-0.0016 (4)	0.0064 (3)	0.0027 (3)
Pb2	0.0422 (5)	0.0130 (4)	0.0177 (4)	0.0040 (4)	0.0047 (4)	0.0013 (3)
Pb3	0.0299 (5)	0.0265 (4)	0.0255 (4)	0.0009 (4)	0.0136 (4)	0.0028 (4)
Pb4	0.0400 (5)	0.0170 (4)	0.0171 (4)	-0.0051 (4)	0.0011 (4)	-0.0026 (3)
O1	0.030 (9)	0.019 (8)	0.018 (8)	0.000 (7)	-0.002 (6)	-0.004 (6)
O2	0.045 (9)	0.010 (7)	0.013 (7)	-0.002 (7)	0.005 (6)	-0.002 (6)
O3	0.033 (8)	0.019 (7)	0.008 (6)	-0.006 (7)	0.005 (6)	-0.001 (6)
O4	0.025 (7)	0.020 (7)	0.021 (7)	0.005 (6)	0.012 (6)	0.003 (6)

N1	0.039 (12)	0.018 (9)	0.027 (10)	0.011 (9)	0.013 (9)	0.006 (8)
N2	0.049 (12)	0.020 (9)	0.011 (8)	0.005 (9)	0.006 (8)	-0.007 (8)
N3	0.047 (13)	0.025 (11)	0.021 (10)	0.001 (9)	0.004 (9)	0.006 (8)
N4	0.054 (14)	0.032 (11)	0.027 (11)	0.018 (10)	0.019 (10)	-0.008 (9)
N5	0.050 (13)	0.022 (10)	0.023 (10)	0.013 (10)	0.008 (9)	0.002 (8)
N6	0.045 (14)	0.048 (14)	0.032 (12)	-0.011 (11)	-0.009 (10)	-0.007 (11)
N7	0.028 (10)	0.017 (10)	0.047 (13)	-0.001 (8)	0.005 (10)	-0.004 (9)
N8	0.042 (13)	0.046 (13)	0.020 (10)	0.010 (11)	-0.005 (9)	-0.005 (10)
N9	0.063 (15)	0.024 (11)	0.048 (15)	0.000 (12)	0.013 (13)	0.007 (10)
N10	0.045 (12)	0.034 (12)	0.029 (11)	-0.023 (11)	0.006 (10)	0.000 (9)
N11	0.10 (2)	0.056 (17)	0.047 (17)	0.017 (18)	0.010 (17)	0.007 (14)
N12	0.11 (3)	0.058 (19)	0.063 (19)	-0.041 (19)	0.038 (19)	-0.018 (16)
N13	0.036 (14)	0.059 (17)	0.053 (15)	0.012 (13)	-0.008 (11)	0.008 (13)
N14	0.09 (2)	0.037 (14)	0.054 (16)	-0.005 (14)	0.034 (16)	0.008 (12)
N15	0.031 (14)	0.14 (3)	0.056 (18)	-0.012 (19)	0.006 (13)	-0.04 (2)
N16	0.080 (12)	0.065 (11)	0.069 (11)	0.001 (10)	0.032 (10)	-0.003 (10)
N17	0.076 (11)	0.064 (12)	0.060 (11)	0.010 (10)	0.022 (9)	-0.004 (9)
N18	0.080 (12)	0.071 (12)	0.065 (11)	0.001 (10)	0.042 (10)	-0.009 (9)
N19	0.073 (12)	0.076 (12)	0.073 (11)	0.014 (10)	0.012 (10)	-0.019 (10)
N20	0.095 (13)	0.062 (12)	0.057 (11)	0.021 (10)	0.024 (10)	0.000 (9)

Table S4. Geometric parameters (Å, º) for **1**

Pb1—O1	2.377 (15)	N1—N5	1.30 (2)
Pb1—O2	2.422 (13)	N2—N3	1.30 (2)
Pb1—O4	2.489 (13)	N3—N4	1.33 (3)
Pb1—N2	2.759 (17)	N4—N5	1.30 (3)
Pb2—O1	2.372 (14)	N6—N10	1.29 (3)
Pb2—O4	2.384 (14)	N6—N7	1.31 (3)
Pb2—O3	2.466 (13)	N7—N8	1.30 (3)
Pb2—N1	2.715 (19)	N8—N9	1.29 (3)
Pb3—O3	2.322 (13)	N9—N10	1.26 (3)
Pb3—O2	2.331 (14)	N11—N12	1.26 (4)
Pb3—O1	2.414 (14)	N11—N15	1.33 (4)
Pb3—N17	2.76 (3)	N12—N13	1.31 (4)
Pb4—O3	2.338 (14)	N13—N14	1.31 (4)
Pb4—O4	2.354 (14)	N14—N15	1.28 (4)
Pb4—O2	2.382 (14)	N16—N20	1.25 (4)
O1—H1	0.8506	N16—N17	1.31 (4)

O2—H2	0.8509	N17—N18	1.30 (4)
O3—H3	0.8456	N18—N19	1.34 (4)
O4—H4	0.8425	N19—N20	1.29 (4)
N1—N2	1.28 (2)		
O1—Pb1—O2	71.8 (5)	Pb3—O3—H3	92.7
O1—Pb1—O4	69.5 (5)	Pb4—O3—H3	146.0
O2—Pb1—O4	69.8 (5)	Pb2—O3—H3	96.9
O1—Pb1—N2	72.4 (5)	Pb4—O4—Pb2	106.0 (5)
O2—Pb1—N2	135.3 (5)	Pb4—O4—Pb1	105.2 (5)
O4—Pb1—N2	73.3 (5)	Pb2—O4—Pb1	105.3 (5)
O1—Pb2—O4	71.4 (5)	Pb4—O4—H4	140.5
O1—Pb2—O3	72.5 (5)	Pb2—O4—H4	105.2
O4—Pb2—O3	70.9 (5)	Pb1—O4—H4	89.2
O1—Pb2—N1	76.5 (5)	N2—N1—N5	110.0 (17)
O4—Pb2—N1	77.0 (5)	N2—N1—Pb2	113.5 (13)
O3—Pb2—N1	140.8 (5)	N5—N1—Pb2	135.9 (14)
O3—Pb3—O2	71.8 (5)	N1—N2—N3	107.4 (17)
O3—Pb3—O1	74.4 (5)	N1—N2—Pb1	123.1 (13)
O2—Pb3—O1	72.7 (5)	N3—N2—Pb1	129.3 (13)
O3—Pb3—N17	138.2 (7)	N2—N3—N4	108.2 (17)
O2—Pb3—N17	72.3 (7)	N5—N4—N3	106.7 (18)
O1—Pb3—N17	75.0 (6)	N4—N5—N1	107.4 (18)
O3—Pb4—O4	73.7 (5)	N10—N6—N7	106.5 (19)
O3—Pb4—O2	70.7 (5)	N8—N7—N6	108.6 (19)
O4—Pb4—O2	72.8 (5)	N9—N8—N7	105.7 (18)
Pb2—O1—Pb1	109.4 (6)	N10—N9—N8	111 (2)
Pb2—O1—Pb3	103.6 (5)	N9—N10—N6	108.5 (19)
Pb1—O1—Pb3	103.9 (5)	N12—N11—N15	108 (3)
Pb2—O1—H1	110.3	N11—N12—N13	110 (3)
Pb1—O1—H1	122.9	N14—N13—N12	105 (2)
Pb3—O1—H1	104.7	N15—N14—N13	111 (3)
Pb3—O2—Pb4	106.1 (5)	N14—N15—N11	105 (2)
Pb3—O2—Pb1	105.1 (5)	N20—N16—N17	108 (3)
Pb4—O2—Pb1	106.5 (6)	N18—N17—N16	108 (3)
Pb3—O2—H2	133.9	N18—N17—Pb3	112 (2)
Pb4—O2—H2	114.0	N16—N17—Pb3	140 (2)
Pb1—O2—H2	84.9	N17—N18—N19	108 (3)
Pb3—O3—Pb4	107.9 (5)	N20—N19—N18	105 (3)

Pb3—O3—Pb2	103.5 (5)	N16—N20—N19	111 (3)
Pb4—O3—Pb2	103.9 (5)		

Table S5. Hydrogen bonds (Å, º) for **1**

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N6	0.85	2.33	2.92 (2)	127
O2—H2···N4^{i}	0.85	2.67	3.08 (2)	111
O2—H2···N7^{ii}	0.85	2.18	2.84 (2)	134
O3—H3···N3^{iii}	0.85	2.06	2.83 (2)	150
O4—H4···N18^{iv}	0.84	2.17	2.90 (3)	144

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

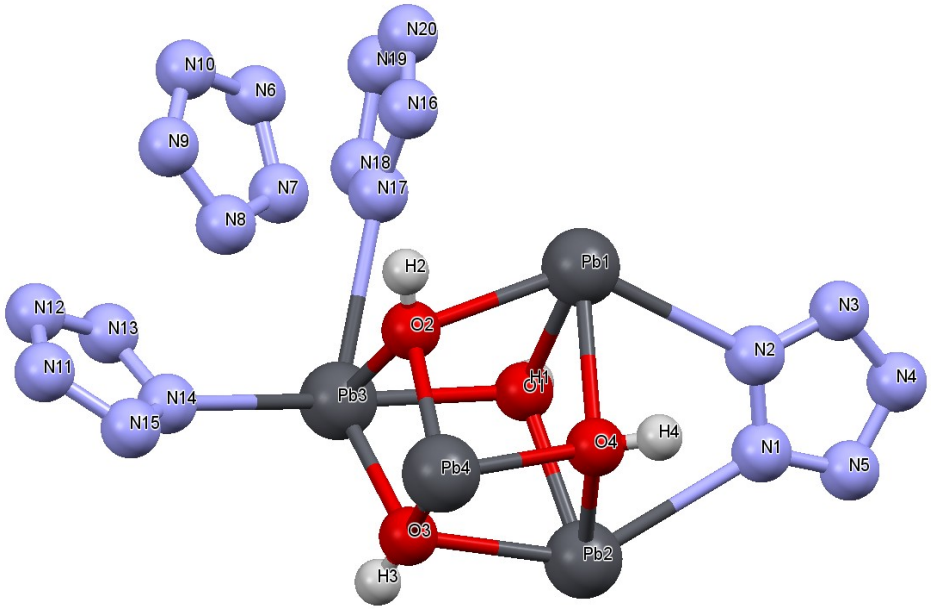

Fig. S1 The asymmetric unit in CP **1**.

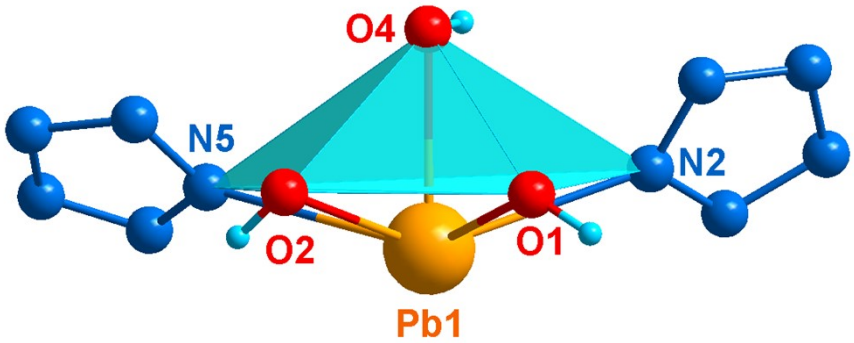

Fig. S2 Coordination environment of the $Pb1^{2+}$ ion in CP **1**.

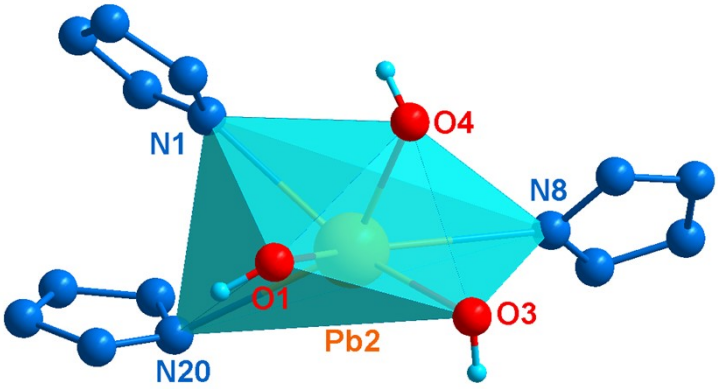

Fig. S3 Coordination environment of the $Pb2^{2+}$ ion in CP **1**.

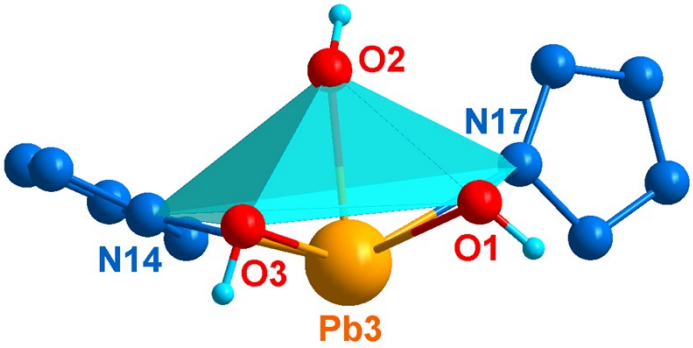

Fig. S4 Coordination environment of the $Pb3^{2+}$ ion in CP **1**.

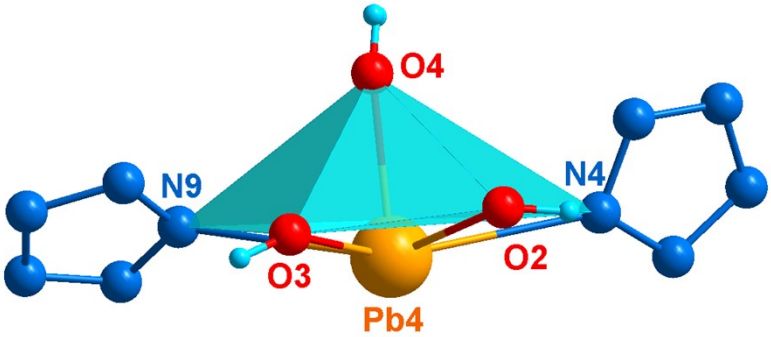

Fig. S5 Coordination environment of the $Pb4^{2+}$ ion in CP **1**.

2. Single-crystal X-ray diffraction analysis of CP **2**

Table S6. Crystal data, data collection, and refinement for CP **2**

$2(H_{18}N_{16}O_{12}Pb_3)\cdot 4(N_5)\cdot H_3O_{1.50}\cdot 2(O_{0.50})\cdot 2(H)$	D_x = 2.852 Mg m^{-3}
M_r = 2436.99	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hexagonal, $P6_3/m$	Cell parameters from 7396 reflections
a = 14.9624 (5) Å	θ = 2.7–27.5°

c = 7.3194 (2) Å	μ = 17.85 mm^{-1}
V = 1419.09 (10) Å^3	T = 296 K
Z = 1	Needle, colourless
F(000) = 1109	0.20 × 0.01 × 0.01 mm
Bruker D8 QUEST PHOTON 100 diffractometer	1039 reflections with $I > 2\sigma(I)$
Detector resolution: 10.42 pixels mm^{-1}	R_{int} = 0.037
ϕ and ω scans	θ_{max} = 27.5°, θ_{min} = 3.1°
Absorption correction: multi-scan *SADABS*	h = -16→19
T_{min} = 0.368, T_{max} = 0.746	k = -19→19
14760 measured reflections	l = -9→9
1172 independent reflections	
Refinement on F^2	0 restraints
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.021	H-atom parameters constrained
$wR(F^2)$ = 0.041	$w = 1/[\sigma^2(F_o^2) + (0.016P)^2 + 3.8176P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001
1172 reflections	$\Delta\rangle_{max}$ = 1.77 e Å^{-3}
86 parameters	$\Delta\rangle_{min}$ = -1.05 e Å^{-3}

Table S7. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for **2**

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
Pb1	0.49955 (2)	0.58062 (2)	0.250000	0.02420 (8)	
N1	0.5042 (2)	0.3426 (2)	0.3957 (4)	0.0227 (6)	
N2	0.4943 (3)	0.2542 (2)	0.3403 (4)	0.0272 (7)	
N3	0.5106 (3)	0.3963 (3)	0.250000	0.0196 (9)	
N4	0.333333	0.666667	0.250000	0.0219 (16)	
O3	0.3066 (3)	0.5729 (3)	0.250000	0.0326 (9)	
O1	0.6646 (4)	0.7653 (4)	0.250000	0.0607 (14)	
H1A	0.646631	0.810998	0.250000	0.091*	
H1B	0.696981	0.774728	0.149970	0.091*	0.5
O2	0.37214 (19)	0.4332 (2)	0.0053 (4)	0.0276 (6)	
H2A	0.324731	0.376466	0.071517	0.041*	
H2B	0.328741	0.449936	-0.044883	0.041*	
N5	0.1397 (9)	0.2239 (10)	0.750000	0.073 (3)	0.6667
N6	0.0239 (5)	0.2581 (6)	0.6640 (11)	0.0547 (18)	0.6667

N7	0.0988 (7)	0.2400 (7)	0.6077 (12)	0.082 (3)	0.6667
O4	-0.0212 (18)	0.2581 (16)	0.750000	0.048 (5)	0.25
H4	-0.034378	0.224183	0.849080	0.072*	0.25
O5	1.000000	1.000000	0.500000	0.126 (12)	0.5
H5A	1.036790	0.973530	0.528920	0.188*	0.0833
H5B	0.938230	0.950990	0.512490	0.188*	0.0833

Table S8. Atomic displacement parameters (Å^2) for **2**

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.02827 (12)	0.02611 (12)	0.02014 (11)	0.01503 (9)	0.000	0.000
N1	0.0332 (17)	0.0234 (15)	0.0171 (15)	0.0184 (14)	0.0003 (14)	0.0008 (13)
N2	0.0444 (19)	0.0235 (16)	0.0195 (15)	0.0212 (15)	0.0008 (15)	0.0015 (14)
N3	0.026 (2)	0.019 (2)	0.016 (2)	0.0131 (19)	0.000	0.000
N4	0.025 (2)	0.025 (2)	0.016 (4)	0.0124 (12)	0.000	0.000
O3	0.039 (2)	0.022 (2)	0.038 (2)	0.0159 (18)	0.000	0.000
O1	0.063 (3)	0.038 (3)	0.070 (4)	0.017 (3)	0.000	0.000
O2	0.0249 (13)	0.0299 (13)	0.0324 (15)	0.0170 (11)	-0.0004 (13)	0.0061 (12)
N5	0.071 (7)	0.108 (9)	0.077 (8)	0.073 (8)	0.000	0.000
N6	0.056 (5)	0.067 (5)	0.057 (4)	0.041 (4)	-0.003 (4)	0.005 (4)
N7	0.093 (6)	0.123 (7)	0.050 (5)	0.069 (6)	0.012 (4)	0.004 (5)
O4	0.057 (15)	0.035 (11)	0.062 (16)	0.030 (12)	0.000	0.000
O5	0.060 (8)	0.060 (8)	0.26 (4)	0.030 (4)	0.000	0.000

Table S9. Geometric parameters (Å, º) for **2**

Pb1—O1	2.629 (5)	N6—N7	1.344 (10)
Pb1—O2^{i}	2.739 (3)	N6—H4^{iv}	0.76 (2)
Pb1—O2	2.739 (3)	O4—H4	0.8500
N1—N3	1.310 (4)	O4—H4^{iv}	0.8501
N1—N2	1.319 (4)	O5—H5A	0.8501
N2—N2^{i}	1.322 (6)	O5—H5B	0.8500
N4—O3^{ii}	1.252 (4)	O5—H5A^{v}	0.8502
N4—O3^{iii}	1.252 (4)	O5—H5A^{vi}	0.8502
N4—O3	1.252 (4)	O5—H5A^{vii}	0.8502
O1—H1A	0.8500	O5—H5A^{viii}	0.8502
O1—H1B	0.8500	O5—H5A^{ix}	0.8502
O1—H1B^{i}	0.8501	O5—H5B^{v}	0.8500
O2—H2A	0.9254	O5—H5B^{vi}	0.8500
O2—H2B	0.8843	O5—H5B^{vii}	0.8500

N5—N7	1.290 (10)	O5—H5B^{viii}	0.8500
N5—N7^{iv}	1.290 (10)	O5—H5B^{ix}	0.8500
N6—N6^{iv}	1.258 (15)		
O1—Pb1—O2^{i}	139.14 (6)	H5B—O5—H5B^{v}	180.0
O1—Pb1—O2	139.14 (6)	H5A^{v}—O5—H5B^{v}	104.4
O2^{i}—Pb1—O2	81.66 (11)	H5A^{vi}—O5—H5B^{v}	50.6
N3—N1—N2	107.6 (3)	H5A^{vii}—O5—H5B^{v}	129.4
N1—N2—N2^{i}	107.91 (19)	H5A^{viii}—O5—H5B^{v}	164.5
N1^{i}—N3—N1	109.0 (4)	H5A^{ix}—O5—H5B^{v}	15.5
O3^{ii}—N4—O3^{iii}	119.996 (1)	H5A—O5—H5B^{vi}	15.5
O3^{ii}—N4—O3	120.001 (1)	H5B—O5—H5B^{vi}	118.9
O3^{iii}—N4—O3	120.003 (1)	H5A^{v}—O5—H5B^{vi}	164.5
Pb1—O1—H1A	109.7	H5A^{vi}—O5—H5B^{vi}	104.4
Pb1—O1—H1B	109.4	H5A^{vii}—O5—H5B^{vi}	75.6
H1A—O1—H1B	104.5	H5A^{viii}—O5—H5B^{vi}	129.4
Pb1—O1—H1B^{i}	109.39 (5)	H5A^{ix}—O5—H5B^{vi}	50.6
H1A—O1—H1B^{i}	104.5	H5B^{v}—O5—H5B^{vi}	61.1
H1B—O1—H1B^{i}	118.9	H5A—O5—H5B^{vii}	164.5
Pb1—O2—H2A	107.6	H5B—O5—H5B^{vii}	61.1
Pb1—O2—H2B	111.0	H5A^{v}—O5—H5B^{vii}	15.5
H2A—O2—H2B	98.2	H5A^{vi}—O5—H5B^{vii}	75.6
N7—N5—N7^{iv}	107.7 (10)	H5A^{vii}—O5—H5B^{vii}	104.4
N6^{iv}—N6—N7	107.9 (5)	H5A^{viii}—O5—H5B^{vii}	50.6
N6^{iv}—N6—H4^{iv}	97.2 (5)	H5A^{ix}—O5—H5B^{vii}	129.4
N7—N6—H4^{iv}	128.1 (16)	H5B^{v}—O5—H5B^{vii}	118.9
N5—N7—N6	108.1 (8)	H5B^{vi}—O5—H5B^{vii}	180.0
H5A—O5—H5B	104.4	H5A—O5—H5B^{viii}	129.4
H5A—O5—H5A^{v}	180.0	H5B—O5—H5B^{viii}	118.9
H5B—O5—H5A^{v}	75.6	H5A^{v}—O5—H5B^{viii}	50.6
H5A—O5—H5A^{vi}	114.0	H5A^{vi}—O5—H5B^{viii}	15.5
H5B—O5—H5A^{vi}	129.4	H5A^{vii}—O5—H5B^{viii}	164.5
H5A^{v}—O5—H5A^{vi}	66.0	H5A^{viii}—O5—H5B^{viii}	104.4
H5A—O5—H5A^{vii}	66.0	H5A^{ix}—O5—H5B^{viii}	75.6
H5B—O5—H5A^{vii}	50.6	H5B^{v}—O5—H5B^{viii}	61.1
H5A^{v}—O5—H5A^{vii}	114.0	H5B^{vi}—O5—H5B^{viii}	118.9
H5A^{vi}—O5—H5A^{vii}	180.0	H5B^{vii}—O5—H5B^{viii}	61.1
H5A—O5—H5A^{viii}	114.0	H5A—O5—H5B^{ix}	50.6
H5B—O5—H5A^{viii}	15.5	H5B—O5—H5B^{ix}	61.1

H5A^{v}—O5—H5A^{viii}	66.0	H5A^{v}—O5—H5B^{ix}	129.4
H5A^{vi}—O5—H5A^{viii}	114.0	H5A^{vi}—O5—H5B^{ix}	164.5
H5A^{vii}—O5—H5A^{viii}	66.0	H5A^{vii}—O5—H5B^{ix}	15.5
H5A—O5—H5A^{ix}	66.0	H5A^{viii}—O5—H5B^{ix}	75.6
H5B—O5—H5A^{ix}	164.5	H5A^{ix}—O5—H5B^{ix}	104.4
H5A^{v}—O5—H5A^{ix}	114.0	H5B^{v}—O5—H5B^{ix}	118.9
H5A^{vi}—O5—H5A^{ix}	66.0	H5B^{vi}—O5—H5B^{ix}	61.1
H5A^{vii}—O5—H5A^{ix}	114.0	H5B^{vii}—O5—H5B^{ix}	118.9
H5A^{viii}—O5—H5A^{ix}	180.0	H5B^{viii}—O5—H5B^{ix}	180.0
H5A—O5—H5B^{v}	75.6		
N3—N1—N2—N2^{i}	-0.3 (3)	N7^{iv}—N5—N7—N6	-5.3 (16)
N2—N1—N3—N1^{i}	0.4 (5)	N6^{iv}—N6—N7—N5	3.3 (10)

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

Table S10. Hydrogen bonds (Å, º) for **2**

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···O4	0.85	2.18	2.87(3)	138
O1—H1B···N5	0.85	2.54	2.851(17)	103
O1—H1B···N6	0.85	2.42	3.242(9)	164
O1—H1B···N7	0.85	2.20	2.975(10)	152
O2—H2A···N6	0.93	1.96	2.837(10)	156
O2—H2A···O4	0.93	1.83	2.74(2)	164
O2—H2B···N2	0.88	1.95	2.829(6)	177

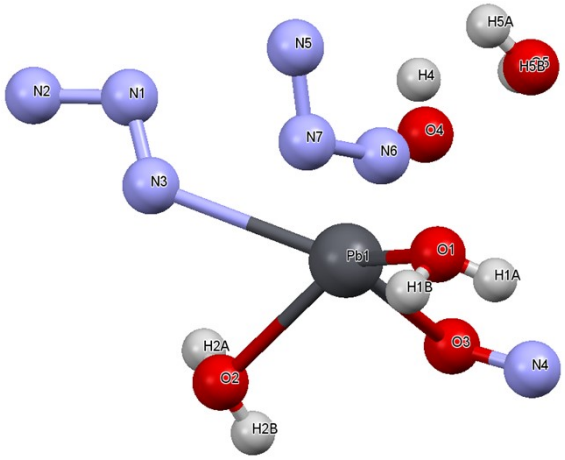

Fig. S6 The asymmetric unit in CP **2**.

3. Single-crystal X-ray diffraction analysis of CP **3**

Table S11. Crystal data, data collection, and refinement for CP **3**

$H_{10}N_{16}O_{10}Pb_4$	D_x = 3.913 Mg m^{-3}
M_r = 1223.00	Mo $K\alpha$ radiation, λ = 0.71073 Å
Trigonal, *R*3	Cell parameters from 3470 reflections
a = 8.9224 (2) Å	θ = 2.7–28.3°
c = 22.5833 (4) Å	μ = 32.42 mm^{-1}
V = 1556.97 (8) Å^3	*T* = 296 K
Z = 3	Block, colourless
F(000) = 1590	0.08 × 0.07 × 0.07 mm
Bruker D8 QUEST PHOTON 100 diffractometer	1156 reflections with $I > 2\sigma(I)$
Detector resolution: 10.42 pixels mm^{-1}	R_{int} = 0.082
ϕ and ω scans	θ_{max} = 24.9°, θ_{min} = 2.7°
Absorption correction: multi-scan *SADABS*	*h* = -9→10
T_{min} = 0.029, T_{max} = 0.096	*k* = -10→9
3927 measured reflections	*l* = -26→26
1206 independent reflections	
Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.045	$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.114	$(\Delta/\sigma)_{max} < 0.001$
S = 1.12	$\Delta\rangle_{max}$ = 1.12 e Å^{-3}
1206 reflections	$\Delta\rangle_{min}$ = -1.57 e Å^{-3}
91 parameters	Absolute structure: Flack x determined using 536 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons, Flack and Wagner, Acta Cryst. B69 (2013) 249-259).
1 restraint	Absolute structure parameter: 0.09 (4)

Table S12. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for **3**

	x	*y*	*z*	U_{iso}*/U_{eq}
Pb1	0.333333	0.666667	0.66633 (6)	0.0303 (5)
Pb2	0.08311 (14)	0.66408 (14)	0.52963 (7)	0.0334 (4)
O1	0.333333	0.666667	0.4873 (14)	0.027 (7)
H1	0.333332	0.666667	0.443868	0.040*

O2	0.330 (2)	0.846 (2)	0.5892 (8)	0.028 (4)
H2	0.328920	0.948889	0.603981	0.041*
O3	0.146 (3)	0.011 (3)	0.5223 (12)	0.064 (8)
O4	0.889 (4)	0.361 (4)	0.4598 (16)	0.080 (9)
H4A	0.959068	0.358592	0.434927	0.119*
H4B	0.886496	0.298192	0.488177	0.119*
N1	-0.204 (5)	0.683 (5)	0.7089 (15)	0.072 (11)
N2	-0.067 (5)	0.681 (5)	0.7321 (13)	0.060 (8)
N3	-0.205 (4)	0.669 (5)	0.6517 (13)	0.058 (9)
N4	-0.069 (4)	0.657 (5)	0.6381 (12)	0.053 (7)
N5	0.017 (3)	0.669 (4)	0.6865 (12)	0.036 (6)
N6	0.000000	0.000000	0.525 (2)	0.044 (12)

Table S13. Atomic displacement parameters (Å^2) for **3**

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0308 (7)	0.0308 (7)	0.0293 (10)	0.0154 (3)	0.000	0.000
Pb2	0.0313 (7)	0.0377 (7)	0.0354 (6)	0.0203 (5)	-0.0018 (5)	0.0010 (5)
O1	0.035 (12)	0.035 (12)	0.009 (16)	0.018 (6)	0.000	0.000
O2	0.033 (11)	0.026 (10)	0.025 (9)	0.016 (8)	-0.004 (7)	0.002 (7)
O3	0.033 (14)	0.057 (16)	0.10 (2)	0.020 (13)	-0.006 (14)	-0.010 (14)
O4	0.09 (2)	0.060 (19)	0.10 (3)	0.04 (2)	0.005 (19)	0.001 (16)
N1	0.07 (2)	0.13 (4)	0.046 (19)	0.08 (3)	-0.006 (17)	-0.02 (2)
N2	0.07 (2)	0.08 (2)	0.044 (17)	0.05 (2)	-0.007 (15)	-0.011 (16)
N3	0.030 (15)	0.11 (3)	0.046 (16)	0.042 (17)	-0.003 (13)	0.002 (17)
N4	0.07 (2)	0.08 (2)	0.035 (15)	0.056 (19)	0.013 (14)	0.010 (15)
N5	0.017 (12)	0.046 (17)	0.044 (15)	0.014 (11)	-0.006 (10)	0.002 (11)
N6	0.021 (14)	0.021 (14)	0.09 (4)	0.011 (7)	0.000	0.000

Table S14. Geometric parameters (Å, º) for **3**

Pb1—O2	2.377 (18)	O3—N6	1.26 (3)
Pb1—O2^{i}	2.377 (18)	O4—H4A	0.8509
Pb1—O2^{ii}	2.377 (18)	O4—H4B	0.8455
Pb2—O2	2.393 (19)	N1—N3	1.30 (4)
Pb2—O2^{i}	2.409 (19)	N1—N2	1.33 (5)
Pb2—O1	2.418 (12)	N2—N5	1.31 (4)
Pb2—N4	2.79 (3)	N3—N4	1.31 (4)
O1—H1	0.9800	N4—N5	1.31 (4)
O2—H2	0.9800		

O2—Pb1—O2^{i}	72.2 (7)	Pb1—O2—Pb2^{ii}	105.2 (7)
O2—Pb1—O2^{ii}	72.2 (7)	Pb2—O2—Pb2^{ii}	106.5 (7)
O2^{i}—Pb1—O2^{ii}	72.2 (7)	Pb1—O2—H2	112.9
O2—Pb2—O2^{i}	71.3 (9)	Pb2—O2—H2	112.9
O2—Pb2—O1	71.5 (7)	Pb2^{ii}—O2—H2	112.9
O2^{i}—Pb2—O1	71.3 (7)	H4A—O4—H4B	104.8
O2—Pb2—N4	78.1 (8)	N3—N1—N2	111 (3)
O2^{i}—Pb2—N4	77.3 (7)	N5—N2—N1	105 (3)
O1—Pb2—N4	141.7 (9)	N1—N3—N4	106 (3)
Pb2^{ii}—O1—Pb2^{i}	105.4 (8)	N5—N4—N3	109 (3)
Pb2^{ii}—O1—Pb2	105.4 (8)	N5—N4—Pb2	118 (2)
Pb2^{i}—O1—Pb2	105.4 (8)	N3—N4—Pb2	132 (2)
Pb2^{ii}—O1—H1	113.3	N4—N5—N2	109 (3)
Pb2^{i}—O1—H1	113.3	O3^{iii}—N6—O3	119.8 (5)
Pb2—O1—H1	113.3	O3^{iii}—N6—O3^{iv}	119.8 (5)
Pb1—O2—Pb2	105.7 (7)	O3—N6—O3^{iv}	119.8 (5)
N3—N1—N2—N5	1 (5)	N3—N4—N5—N2	2 (4)
N2—N1—N3—N4	0 (5)	Pb2—N4—N5—N2	173 (3)
N1—N3—N4—N5	-1 (5)	N1—N2—N5—N4	-2 (4)
N1—N3—N4—Pb2	-171 (3)		

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

Table S15. Hydrogen bonds (Å, º) for **3**

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N3^{v}	0.98	1.91	2.86 (3)	166
O4—H4*A*···N1^{vi}	0.85	2.06	2.91 (5)	179
O4—H4*B*···O3^{vii}	0.85	2.21	2.94 (4)	145

Symmetry codes: (v) -*y*+1, *x*-*y*+2, *z*; (vi) *x*+4/3, *y*-1/3, *z*-1/3; (vii) -*y*+1, *x*-*y*, *z*.

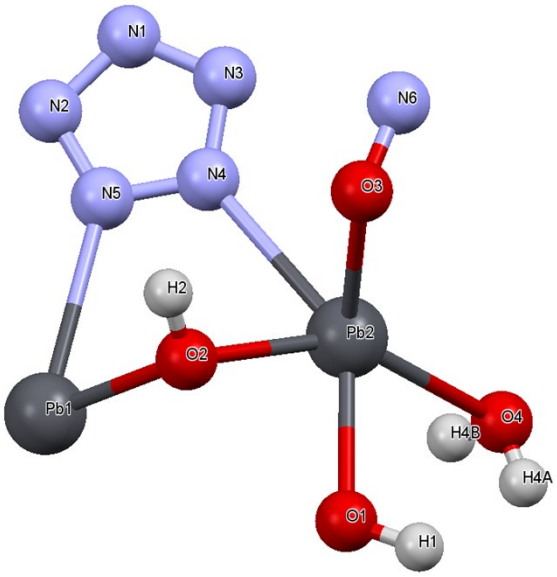

Fig. S7 The asymmetric unit in CP **3**.

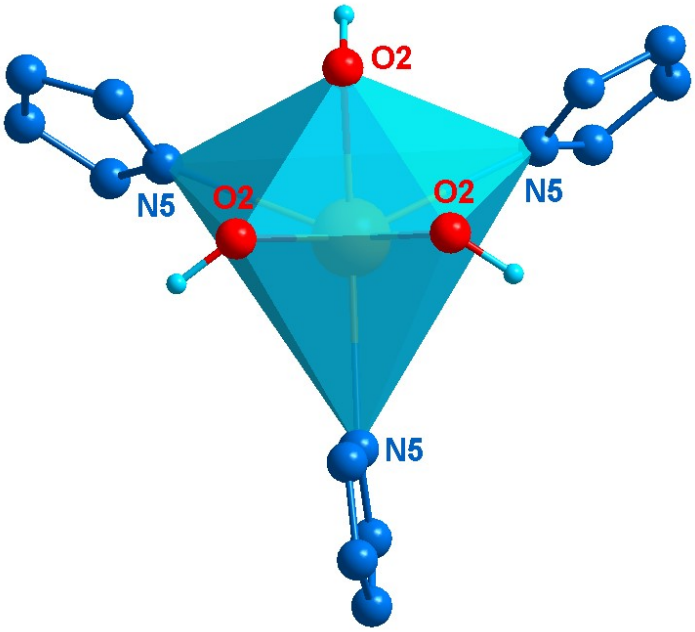

Fig. S8 Coordination environment of the $Pb1^{2+}$ ion in CP **3**.

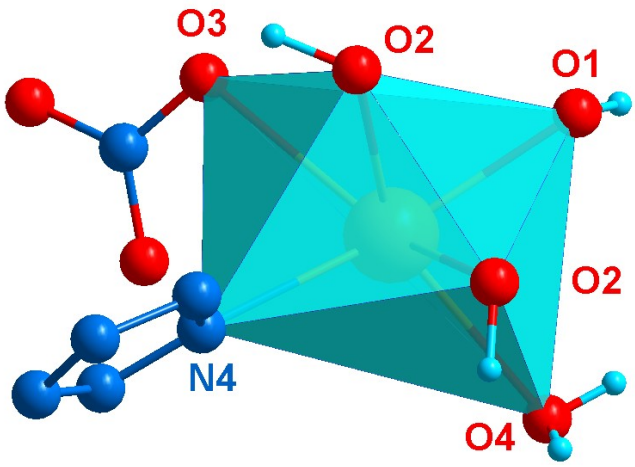

Fig. S9 Coordination environment of the $Pb2^{2+}$ ion in CP **3**.

4. Single-crystal X-ray diffraction analysis of CP **4**

Table S16. Crystal data, data collection, and refinement for CP **4**

$ClH_6N_{15}O_9Pb_4$	D_x = 4.417 Mg m^{-3}
M_r = 1224.41	Mo $K\alpha$ radiation, λ = 0.71073 Å
Orthorhombic, $Pca2_1$	Cell parameters from 9944 reflections
a = 10.1088 (8) Å	θ = 2.5–27.5°
b = 13.5520 (11) Å	μ = 36.69 mm^{-1}
c = 13.4401 (9) Å	T = 296 K
V = 1841.2 (2) Å^3	Block, colourless
Z = 4	0.02 × 0.02 × 0.02 mm
$F(000)$ = 2112	
Bruker D8 QUEST PHOTON 100 diffractometer	3669 reflections with $I > 2\sigma(I)$
Detector resolution: 10.42 pixels mm^{-1}	R_{int} = 0.066
ϕ and ω scans	θ_{max} = 27.6°, θ_{min} = 2.9°
Absorption correction: multi-scan *SADABS*	h = -12→13
T_{min} = 0.565, T_{max} = 0.746	k = -17→15
17903 measured reflections	l = -14→17
3982 independent reflections	
Refinement on F^2	Hydrogen site location: mixed
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.028	$w = 1/[\sigma^2(F_o^2) + (0.0178P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.054	$(\Delta/\sigma)_{max}$ = 0.001

S = 1.01	$\Delta\rangle_{max}$ = 1.19 e Å^{-3}
3982 reflections	$\Delta\rangle_{min}$ = -1.25 e Å^{-3}
263 parameters	Absolute structure: Refined as an inversion twin.
1 restraint	Absolute structure parameter: 0.034 (15)

Table S17. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for **4**

	x	y	z	U_{iso}*/U_{eq}
Pb1	0.78990 (5)	0.35207 (3)	0.25098 (4)	0.02531 (13)
Pb2	1.14069 (5)	0.29182 (4)	0.34914 (4)	0.02577 (13)
Pb3	1.03794 (5)	0.20994 (4)	0.08328 (4)	0.02620 (13)
Pb4	0.88389 (5)	0.08651 (4)	0.31274 (4)	0.02916 (14)
N1	0.9138 (12)	0.4833 (9)	0.3774 (9)	0.031 (3)
N2	1.0437 (12)	0.4845 (8)	0.3793 (8)	0.030 (3)
N3	1.0820 (13)	0.5640 (9)	0.4256 (10)	0.035 (3)
N4	0.9761 (13)	0.6118 (9)	0.4533 (10)	0.035 (3)
N5	0.8705 (12)	0.5628 (9)	0.4234 (10)	0.034 (3)
N6	0.5469 (12)	0.5923 (10)	0.5891 (11)	0.038 (3)
N7	0.6086 (16)	0.5530 (12)	0.6657 (12)	0.055 (4)
N8	0.7196 (14)	0.6040 (11)	0.6772 (11)	0.049 (4)
N9	0.7265 (12)	0.6730 (10)	0.6086 (9)	0.031 (3)
N10	0.6185 (13)	0.6642 (10)	0.5550 (10)	0.037 (3)
N11	0.8475 (15)	0.8835 (10)	-0.0192 (10)	0.043 (3)
N12	0.9438 (16)	0.9482 (10)	-0.0238 (10)	0.045 (4)
N13	0.9310 (16)	1.0099 (10)	0.0516 (10)	0.048 (4)
N14	0.8270 (16)	0.9802 (11)	0.1021 (12)	0.057 (4)
N15	0.7765 (14)	0.9036 (9)	0.0591 (12)	0.050 (4)
O1	0.8287 (9)	0.1915 (6)	0.1798 (7)	0.028 (2)
H1A	0.754509	0.167209	0.139679	0.034*
O2	0.9144 (9)	0.2498 (6)	0.3722 (7)	0.025 (2)
H2A	0.884236	0.258711	0.440925	0.031*
O3	1.0061 (9)	0.3422 (6)	0.2020 (7)	0.024 (2)
H3A	1.036881	0.405342	0.174963	0.028*
O4	1.0837 (9)	0.1451 (6)	0.2443 (7)	0.0235 (19)
H4A	1.153605	0.095008	0.243719	0.028*
Cl1	0.4716 (4)	0.8347 (3)	0.8044 (3)	0.0366 (8)
O5	0.4762 (14)	0.7583 (11)	0.7321 (13)	0.077 (5)
O6	0.5978 (10)	0.8460 (9)	0.8512 (11)	0.052 (3)

O7	0.4331 (12)	0.9264 (9)	0.7599 (11)	0.054 (3)
O8	0.3730 (11)	0.8098 (9)	0.8789 (10)	0.053 (3)
O9	0.7222 (11)	0.3540 (9)	0.0524 (9)	0.052 (3)
H9A	0.781053	0.378356	0.014507	0.077*
H9B	0.654513	0.389956	0.042907	0.077*

Table S18. Atomic displacement parameters (Å^2) for **4**

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pb1	0.0258 (3)	0.0216 (2)	0.0285 (3)	0.0054 (2)	0.0003 (2)	-0.0034 (2)
Pb2	0.0241 (3)	0.0275 (3)	0.0257 (3)	-0.0007 (2)	-0.0017 (2)	-0.0033 (2)
Pb3	0.0365 (3)	0.0219 (3)	0.0202 (2)	0.0024 (2)	0.0019 (2)	-0.0015 (2)
Pb4	0.0345 (3)	0.0181 (3)	0.0349 (3)	-0.0034 (2)	0.0012 (2)	0.0061 (2)
N1	0.036 (7)	0.027 (7)	0.031 (7)	-0.001 (6)	-0.007 (6)	-0.002 (5)
N2	0.044 (8)	0.023 (6)	0.024 (6)	-0.001 (5)	-0.005 (5)	-0.004 (5)
N3	0.030 (7)	0.028 (7)	0.047 (8)	0.002 (5)	0.001 (6)	0.004 (6)
N4	0.041 (8)	0.020 (7)	0.045 (8)	0.007 (6)	0.000 (6)	0.002 (5)
N5	0.028 (7)	0.031 (7)	0.042 (8)	-0.004 (5)	0.002 (6)	-0.007 (6)
N6	0.037 (7)	0.042 (8)	0.035 (7)	-0.012 (6)	-0.002 (6)	0.000 (6)
N7	0.075 (11)	0.049 (9)	0.041 (9)	-0.026 (8)	-0.018 (8)	0.006 (7)
N8	0.049 (9)	0.057 (9)	0.041 (9)	-0.016 (7)	-0.015 (7)	0.026 (7)
N9	0.029 (7)	0.043 (7)	0.023 (6)	0.002 (6)	-0.005 (5)	0.005 (5)
N10	0.040 (8)	0.033 (7)	0.037 (8)	-0.005 (6)	0.002 (6)	0.007 (6)
N11	0.054 (9)	0.038 (8)	0.038 (8)	-0.014 (7)	0.014 (7)	-0.020 (7)
N12	0.075 (11)	0.032 (8)	0.029 (8)	-0.017 (7)	0.019 (7)	-0.003 (6)
N13	0.072 (10)	0.035 (8)	0.037 (8)	-0.016 (7)	0.012 (7)	-0.013 (6)
N14	0.072 (10)	0.030 (8)	0.068 (11)	-0.013 (7)	0.032 (9)	-0.020 (7)
N15	0.055 (10)	0.024 (7)	0.070 (11)	-0.011 (6)	0.011 (8)	0.000 (7)
O1	0.031 (5)	0.020 (5)	0.033 (6)	0.003 (4)	-0.003 (5)	-0.005 (4)
O2	0.028 (5)	0.019 (5)	0.029 (5)	-0.001 (4)	0.004 (4)	-0.003 (4)
O3	0.028 (5)	0.009 (4)	0.034 (5)	0.000 (4)	0.000 (4)	-0.001 (4)
O4	0.025 (5)	0.023 (5)	0.023 (5)	0.006 (4)	-0.002 (4)	0.003 (4)
Cl1	0.036 (2)	0.0279 (19)	0.046 (2)	-0.0027 (14)	-0.0046 (17)	-0.0005 (17)
O5	0.063 (9)	0.072 (10)	0.095 (13)	-0.012 (7)	0.012 (8)	-0.046 (10)
O6	0.031 (6)	0.056 (8)	0.069 (8)	-0.006 (5)	-0.011 (6)	0.018 (6)
O7	0.058 (8)	0.040 (7)	0.064 (8)	-0.004 (5)	-0.009 (7)	0.020 (6)
O8	0.045 (7)	0.053 (7)	0.061 (9)	-0.010 (6)	0.003 (6)	0.015 (6)
O9	0.042 (7)	0.063 (9)	0.049 (7)	0.004 (6)	0.003 (6)	0.030 (6)

Table S19. Geometric parameters (Å, º) for **4**

Pb1—O3	2.286 (9)	N3—N4	1.306 (17)
Pb1—O1	2.410 (9)	N4—N5	1.320 (17)
Pb1—O2	2.482 (9)	N6—N10	1.298 (18)
Pb1—O9	2.755 (12)	N6—N7	1.32 (2)
Pb1—N1	2.760 (12)	N7—N8	1.326 (19)
Pb2—O2	2.378 (9)	N8—N9	1.314 (18)
Pb2—O3	2.496 (9)	N9—N10	1.314 (17)
Pb2—O4	2.504 (9)	N11—N15	1.303 (19)
Pb3—O4	2.381 (9)	N11—N12	1.312 (19)
Pb3—O3	2.422 (9)	N12—N13	1.320 (18)
Pb3—O1	2.494 (10)	N13—N14	1.314 (19)
Pb4—O1	2.351 (10)	N14—N15	1.292 (19)
Pb4—O4	2.357 (9)	Cl1—O5	1.419 (14)
Pb4—O2	2.373 (9)	Cl1—O6	1.432 (12)
N1—N2	1.313 (16)	Cl1—O7	1.434 (12)
N1—N5	1.318 (17)	Cl1—O8	1.453 (12)
N2—N3	1.303 (17)		
O3—Pb1—O1	71.2 (3)	N6—N7—N8	106.3 (13)
O3—Pb1—O2	70.8 (3)	N9—N8—N7	109.5 (13)
O1—Pb1—O2	71.0 (3)	N10—N9—N8	106.1 (12)
O3—Pb1—O9	87.7 (3)	N6—N10—N9	109.7 (12)
O1—Pb1—O9	70.4 (3)	N15—N11—N12	107.9 (12)
O2—Pb1—O9	140.1 (3)	N11—N12—N13	108.3 (13)
O3—Pb1—N1	77.3 (4)	N14—N13—N12	106.3 (13)
O1—Pb1—N1	138.8 (3)	N15—N14—N13	109.4 (14)
O2—Pb1—N1	74.1 (3)	N14—N15—N11	108.2 (13)
O9—Pb1—N1	134.7 (3)	Pb4—O1—Pb1	106.4 (4)
O2—Pb2—O3	69.2 (3)	Pb4—O1—Pb3	104.8 (3)
O2—Pb2—O4	70.2 (3)	Pb1—O1—Pb3	104.7 (3)
O3—Pb2—O4	69.3 (3)	Pb4—O2—Pb2	107.7 (3)
O4—Pb3—O3	72.6 (3)	Pb4—O2—Pb1	103.5 (3)
O4—Pb3—O1	69.8 (3)	Pb2—O2—Pb1	105.6 (3)
O3—Pb3—O1	67.6 (3)	Pb1—O3—Pb3	111.1 (4)
O1—Pb4—O4	72.7 (3)	Pb1—O3—Pb2	108.0 (4)
O1—Pb4—O2	73.9 (3)	Pb3—O3—Pb2	104.3 (3)
O4—Pb4—O2	72.9 (3)	Pb4—O4—Pb3	108.2 (3)
N2—N1—N5	108.2 (11)	Pb4—O4—Pb2	104.2 (3)
N2—N1—Pb1	118.3 (8)	Pb3—O4—Pb2	105.2 (3)

N5—N1—Pb1	131.7 (9)	O5—Cl1—O6	110.5 (9)
N3—N2—N1	108.5 (11)	O5—Cl1—O7	110.8 (10)
N2—N3—N4	107.6 (12)	O6—Cl1—O7	109.4 (7)
N3—N4—N5	109.1 (12)	O5—Cl1—O8	109.0 (8)
N1—N5—N4	106.6 (11)	O6—Cl1—O8	109.5 (8)
N10—N6—N7	108.4 (13)	O7—Cl1—O8	107.6 (8)
N5—N1—N2—N3	-0.3 (15)	N7—N8—N9—N10	0.2 (18)
Pb1—N1—N2—N3	166.3 (9)	N7—N6—N10—N9	-0.2 (18)
N1—N2—N3—N4	0.6 (15)	N8—N9—N10—N6	0.0 (17)
N2—N3—N4—N5	-0.7 (16)	N15—N11—N12—N13	-1 (2)
N2—N1—N5—N4	-0.1 (15)	N11—N12—N13—N14	1 (2)
Pb1—N1—N5—N4	-164.2 (9)	N12—N13—N14—N15	-1 (2)
N3—N4—N5—N1	0.5 (16)	N13—N14—N15—N11	0 (2)
N10—N6—N7—N8	0.3 (19)	N12—N11—N15—N14	1 (2)
N6—N7—N8—N9	-0.3 (19)		

Table S20. Hydrogen bonds (Å, º) for **4**

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4A···N1	0.91(4)	2.18(4)	3.009(3)	153(4)
O4—H4B···N1	0.97(7)	2.55(6)	3.508(4)	168(5)
N6—H6···O3	0.95(4)	1.74(4)	2.671(3)	167(3)
N7—H7A···N5	0.88	2.16	3.025(3)	167
N7—H7B···O3	0.88	2.06	2.725(3)	132
N7—H7B···O4	0.88	2.22	2.865(3)	130

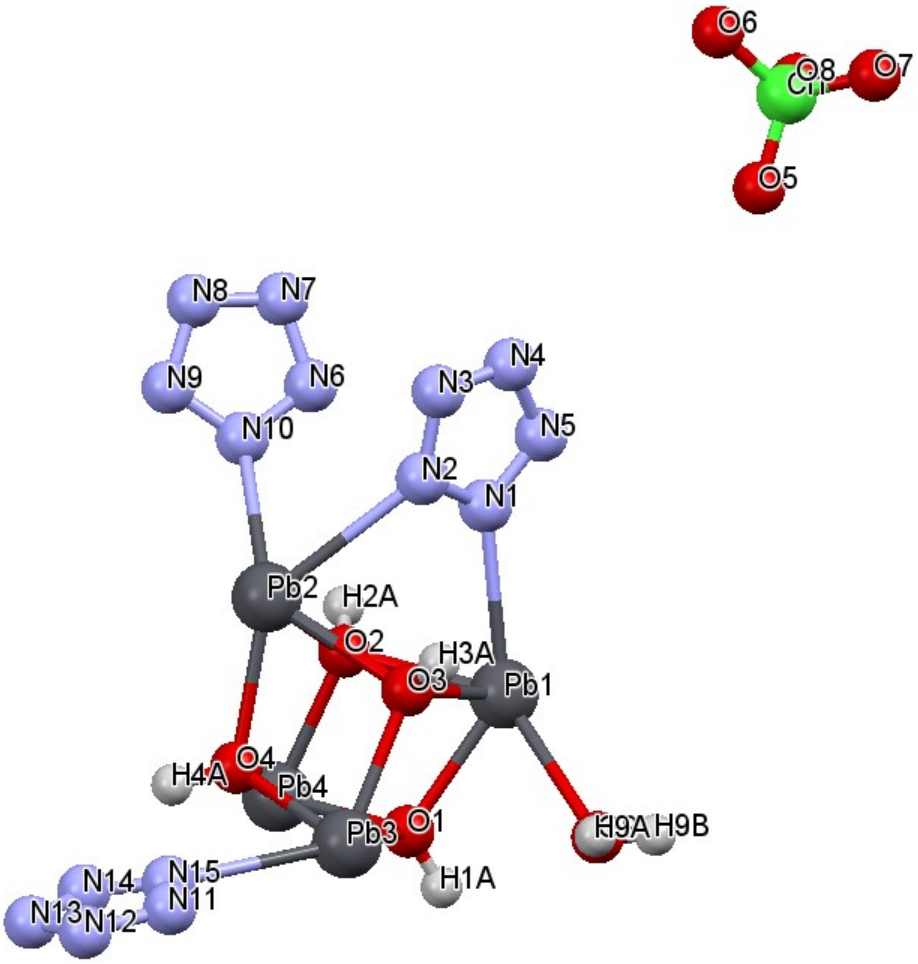

Fig. S10 The asymmetric unit in CP **4**.

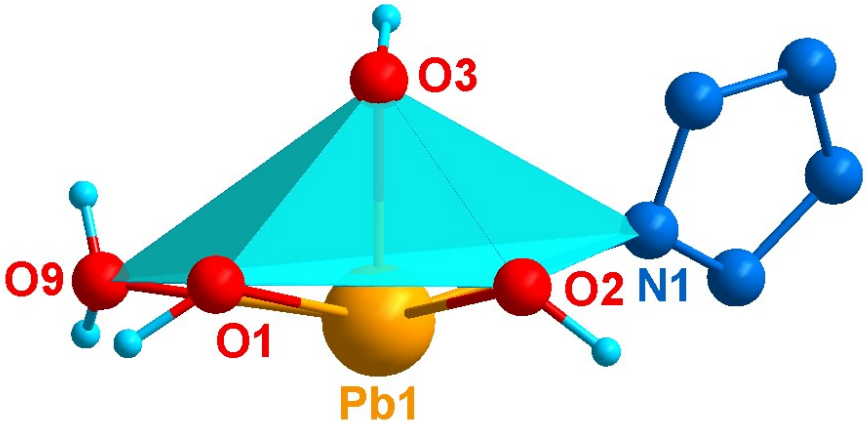

Fig. S11 Coordination environment of the $Pb1^{2+}$ ion in CP **4**.

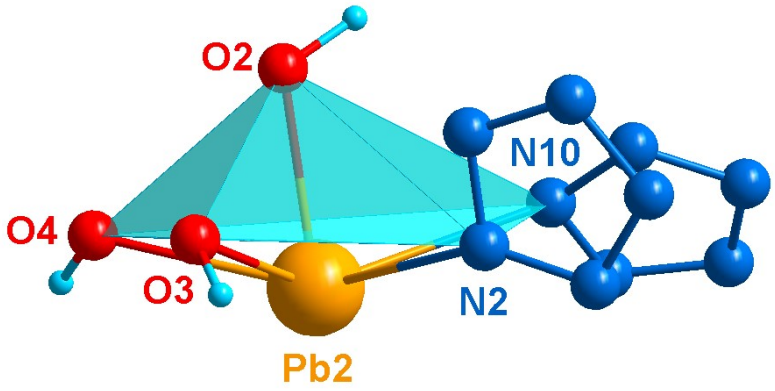

Fig. S12 Coordination environment of the $Pb2^{2+}$ ion in CP **4**.

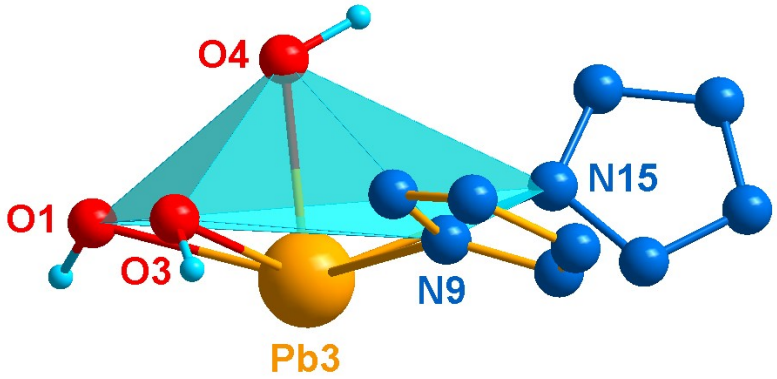

Fig. S13 Coordination environment of the $Pb3^{2+}$ ion in CP **4**.

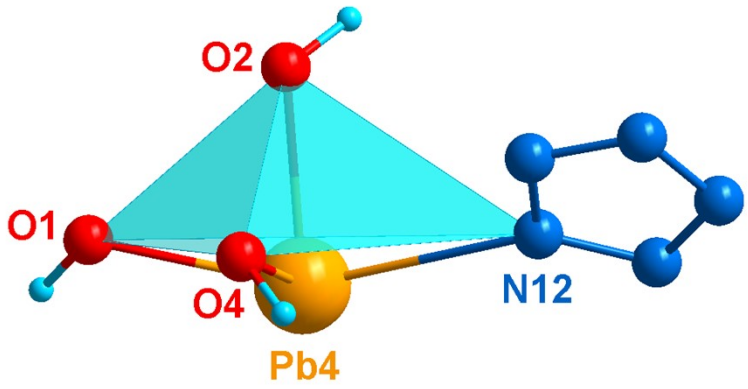

Fig. S14 Coordination environment of the $Pb4^{2+}$ ion in CP **4**.

5. High-resolution mass spectra of CPs **1-4**

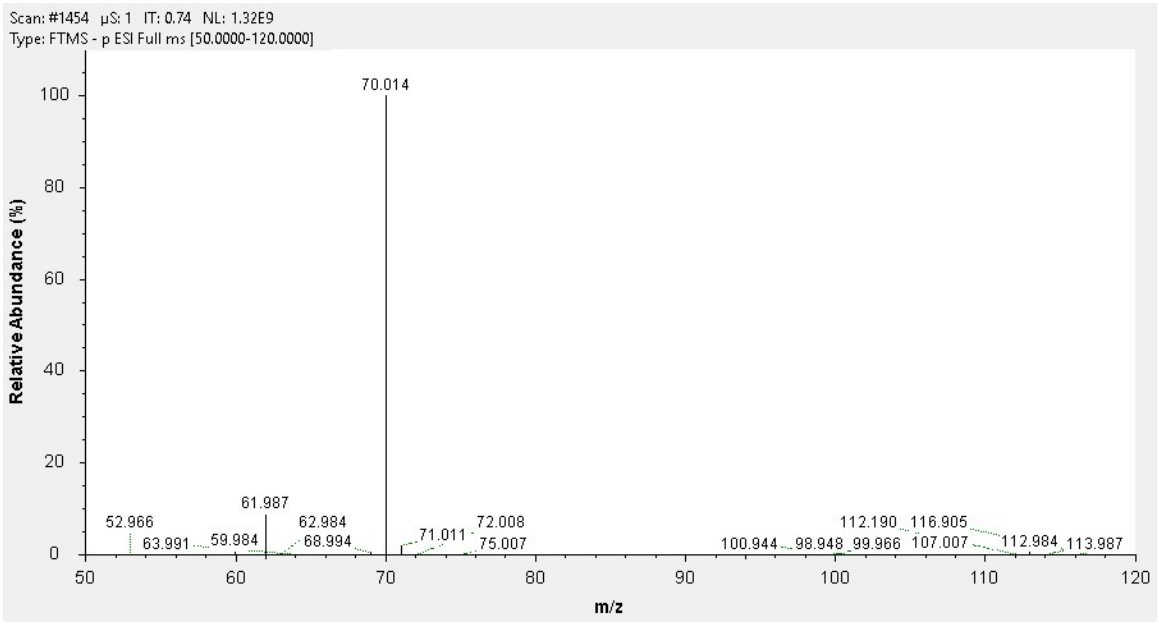

Fig. S15 High-resolution mass spectrum of CP **1** (negative ion mode).

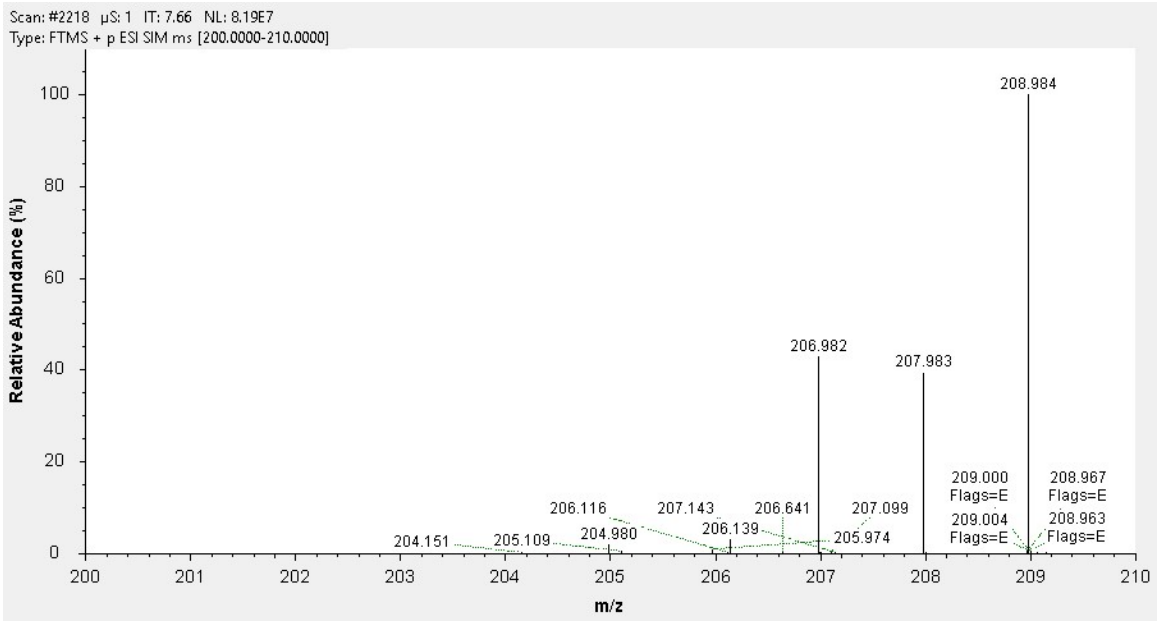

Fig. S16 High-resolution mass spectrum of CP **1** (positive ion mode).

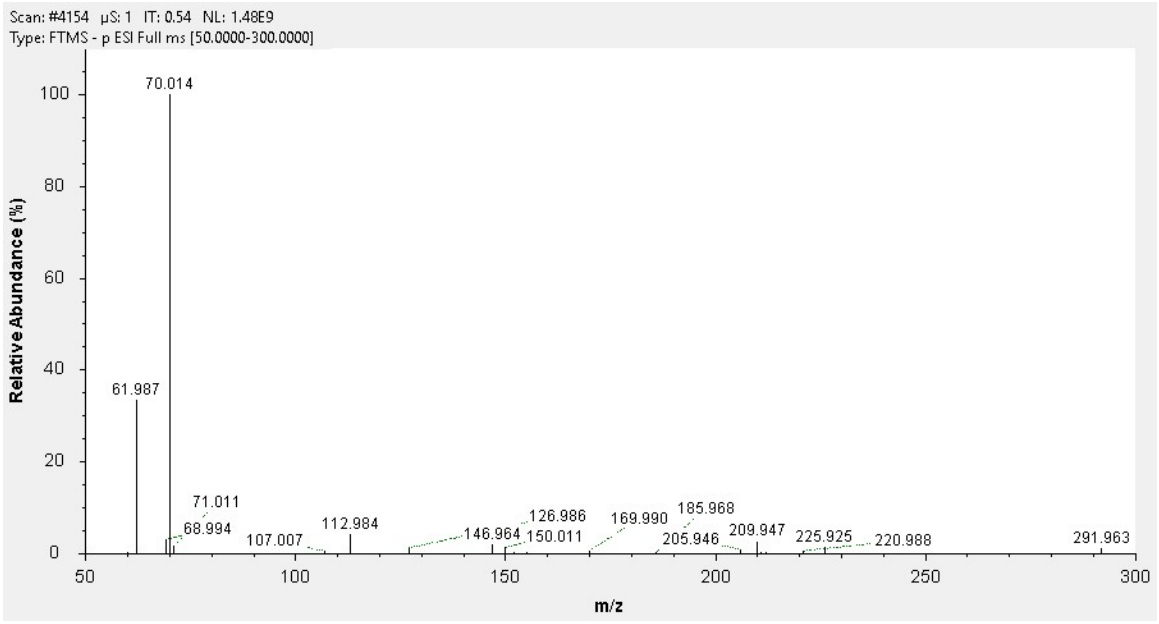

Fig. S17 High-resolution mass spectrum of CP **2** (negative ion mode).

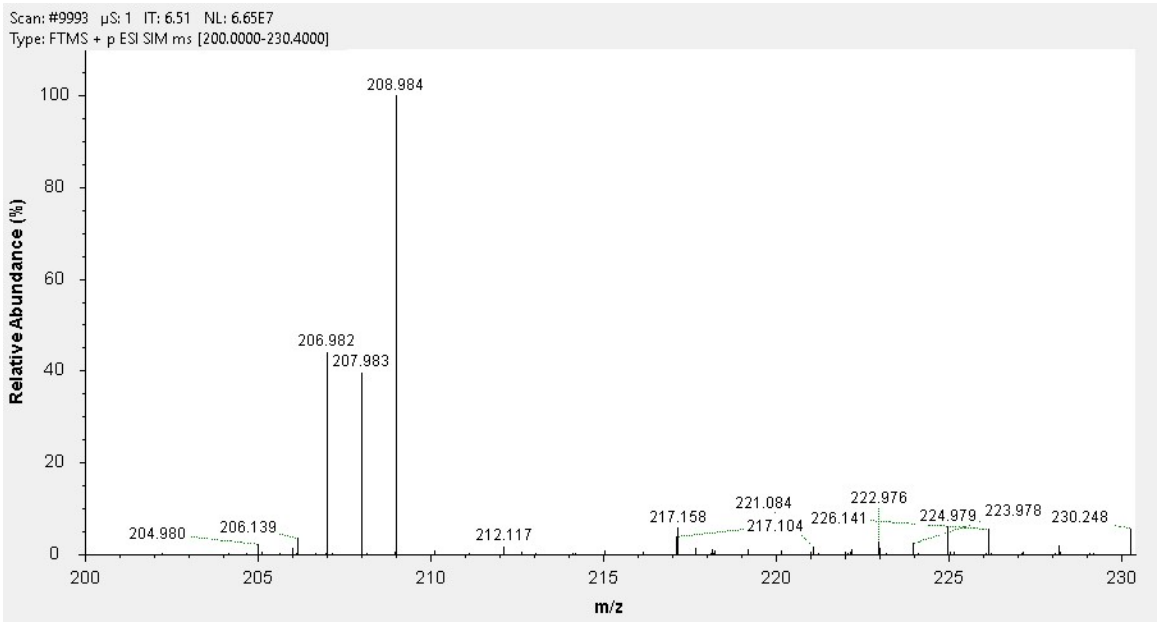

Fig. S18 High-resolution mass spectrum of CP **2** (positive ion mode).

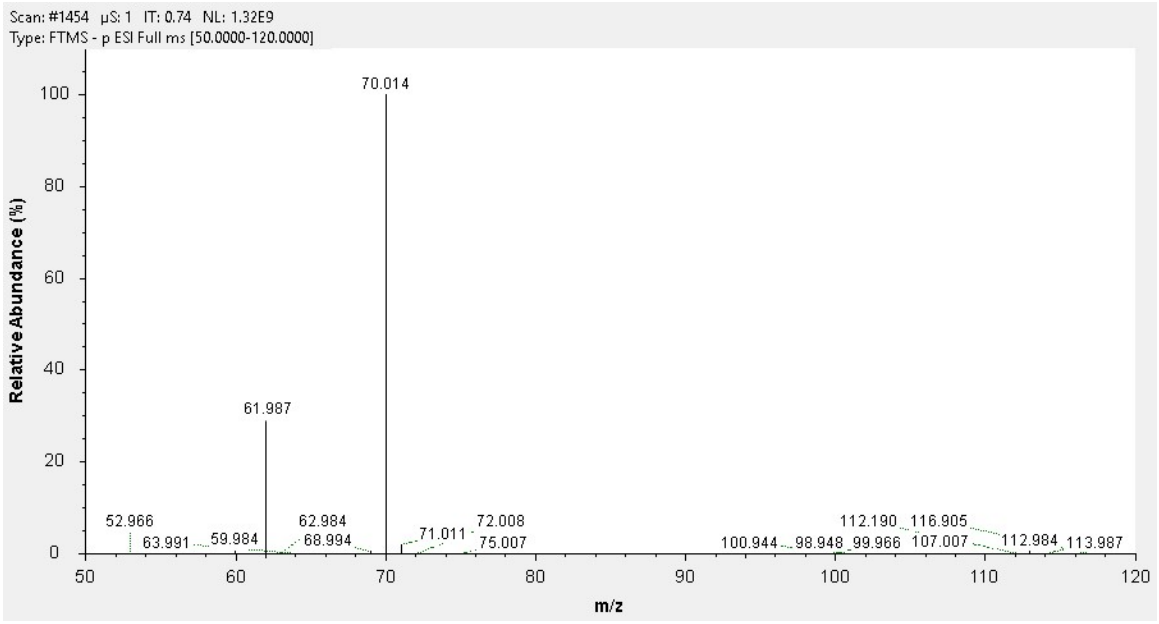

Fig. S19 High-resolution mass spectrum of CP **3** (negative ion mode).

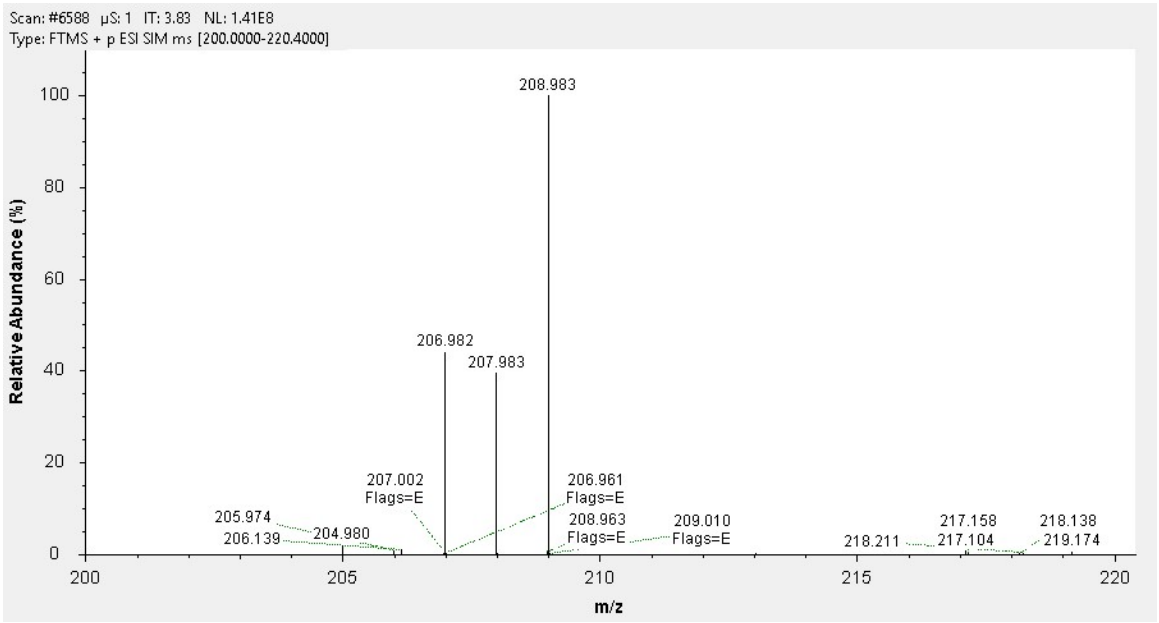

Fig. S20 High-resolution mass spectrum of CP **3** (positive ion mode).

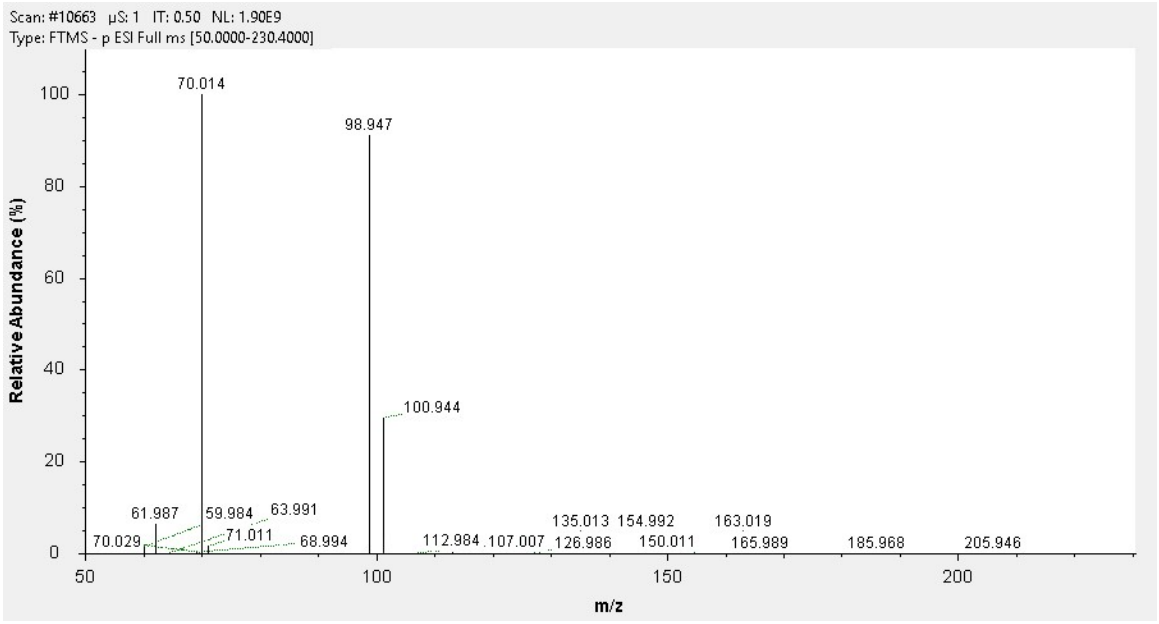

Fig. S21 High-resolution mass spectrum of CP **4** (negative ion mode).

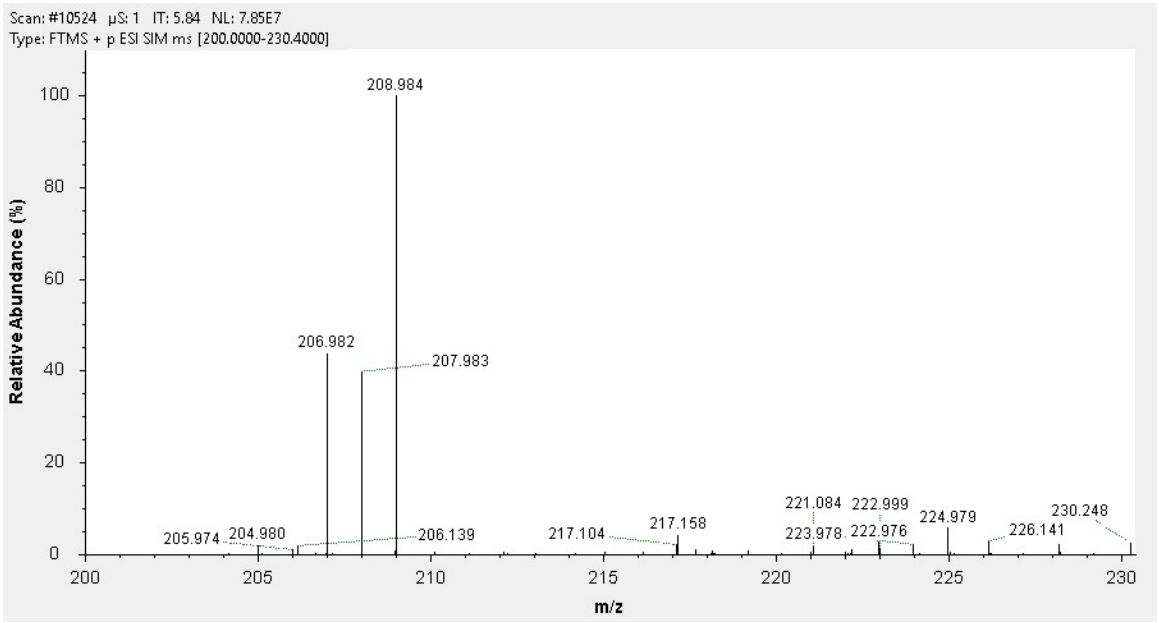

Fig. S22 High-resolution mass spectrum of CP **4** (positive ion mode).

6. PXRD patterns of CPs **1-4**

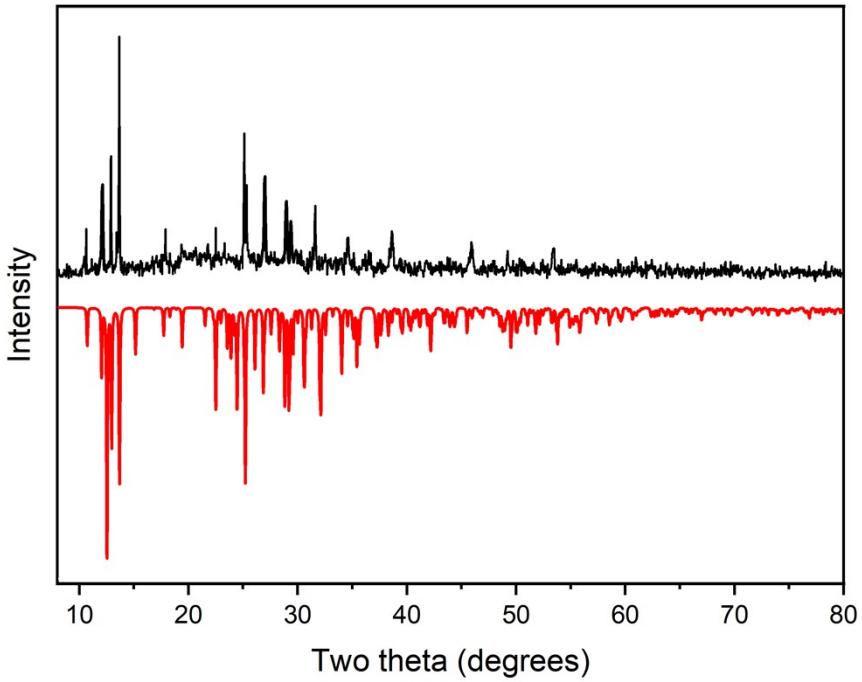

Fig. S23 The PXRD pattern (in black) of CP **1** and the calculated pattern (in red) from its single-crystal structure.

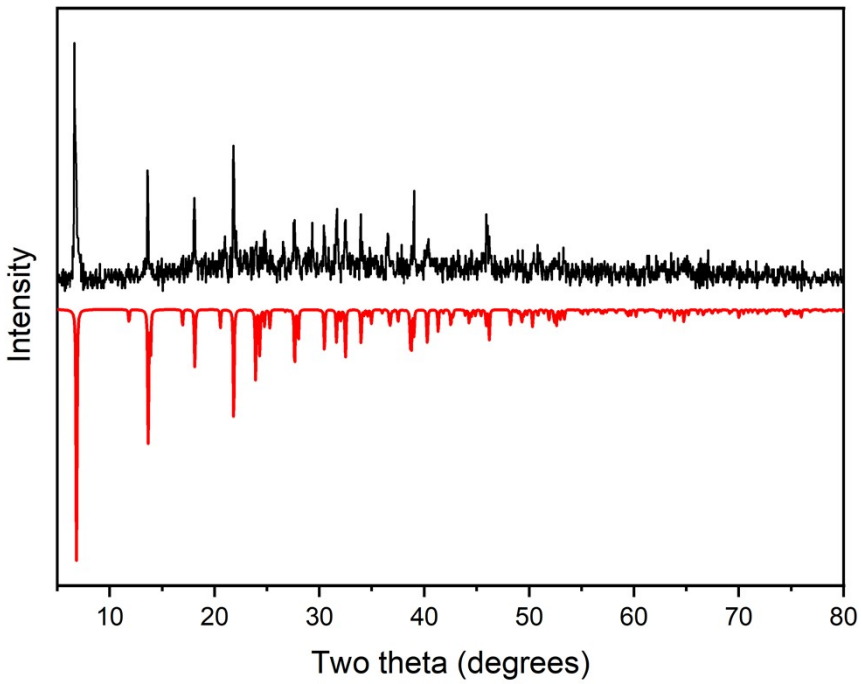

Fig. S24 The PXRD pattern (in black) of CP **2** and the calculated pattern (in red) from its single-crystal structure.

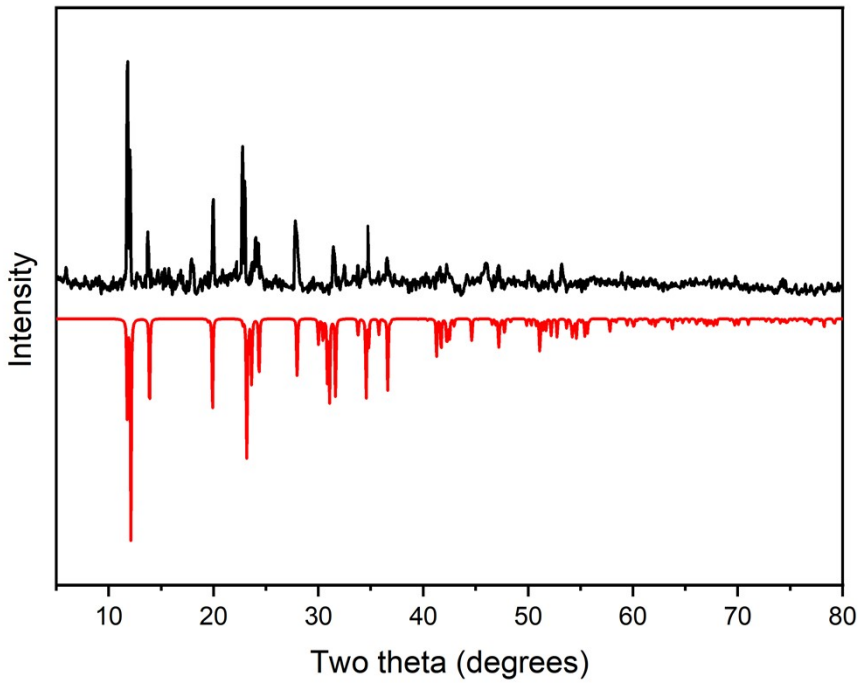

Fig. S25 The PXRD pattern (in black) of CP **3** and the calculated pattern (in red) from its single-crystal structure.

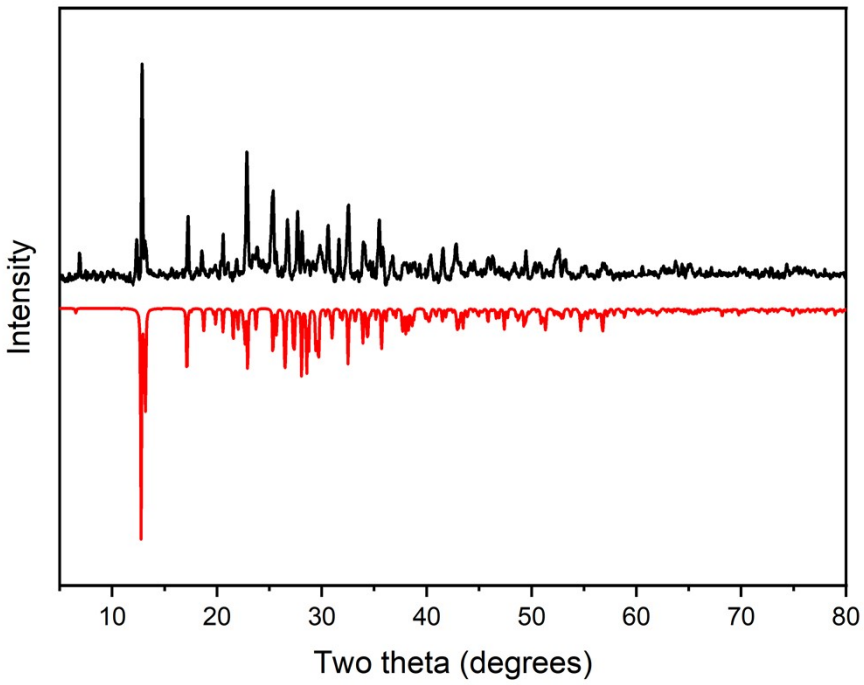

Fig. S26 The PXRD pattern (in black) of CP **4** and the calculated pattern (in red) from its single-crystal structure.

7. Non-isothermal kinetic analysis

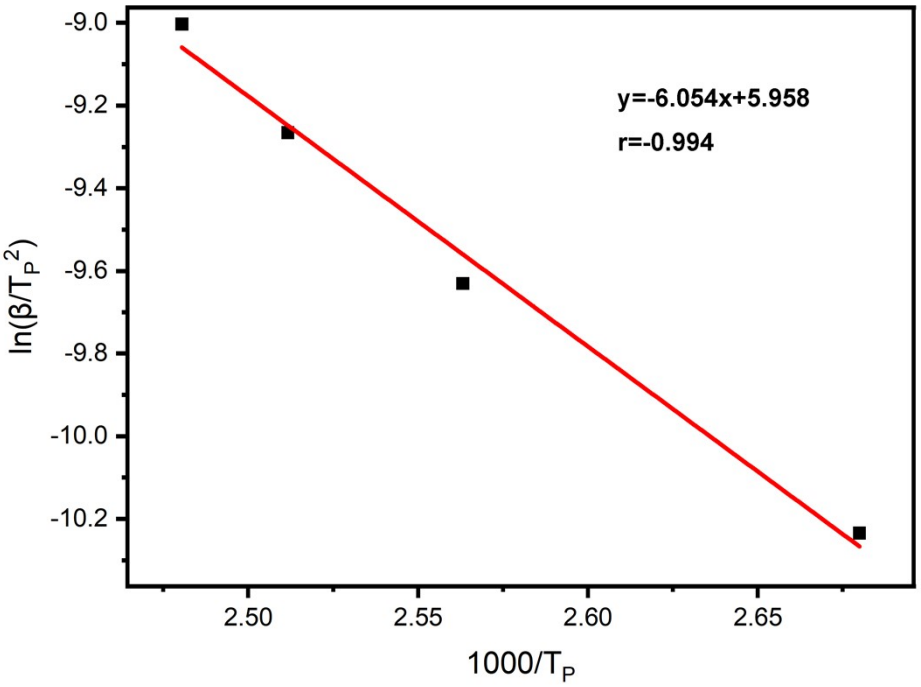

Fig. S27 Dependence of $\ln(\beta/T_p^2)$ on $1/T_p$ for CP **1** (Scatter points are the experimental data and lines denote the linear fitting results).

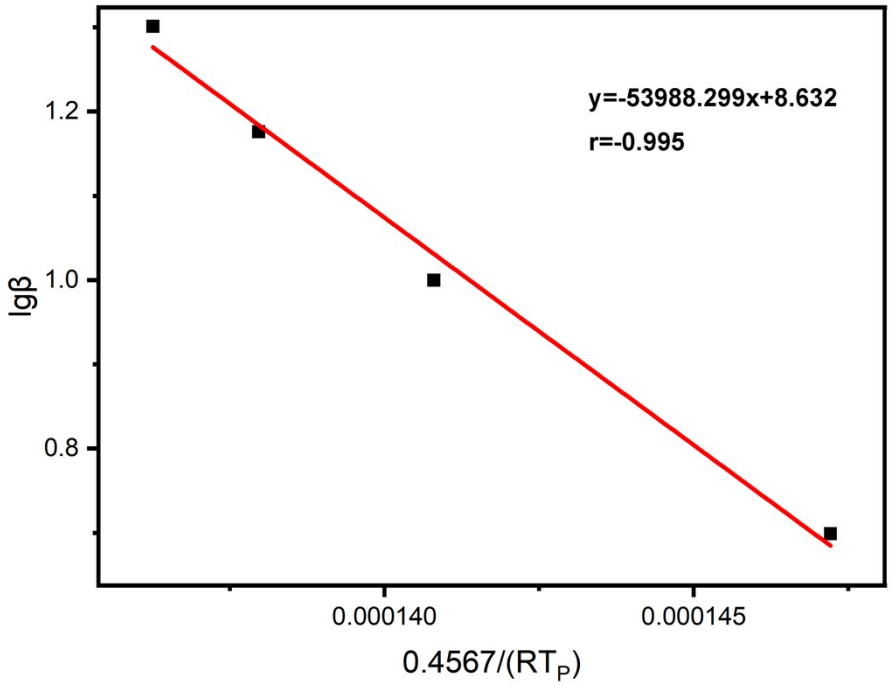

Fig. S28 Dependence of $\lg\beta$ on $0.4567/RT_p$ for CP **1** (Scatter points are the experimental data and lines denote the linear fitting results).

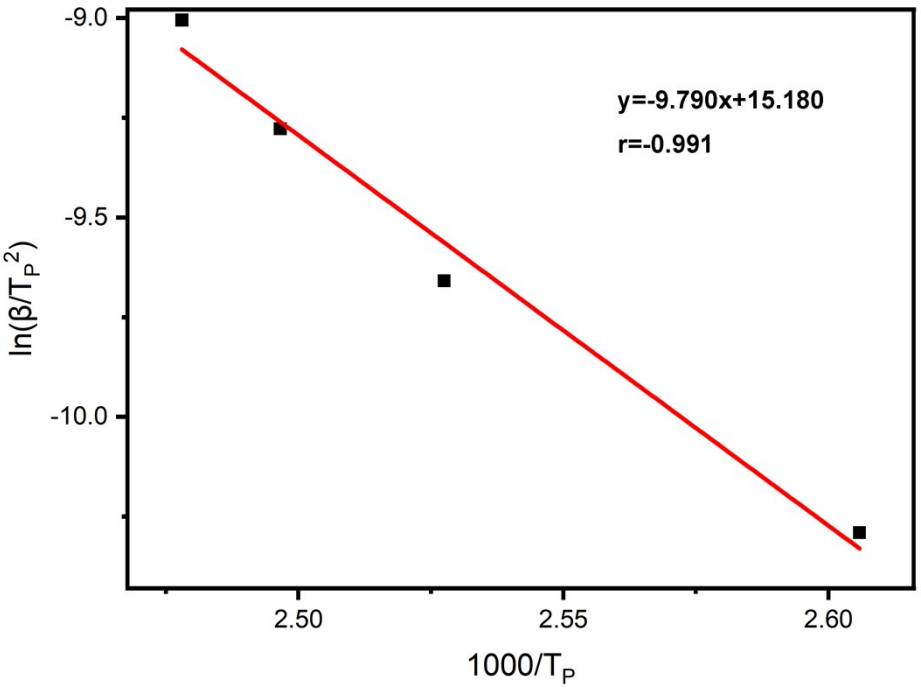

Fig. S29 Dependence of $\ln(\beta/T_p^2)$ on $1/T_p$ for CP **2** (Scatter points are the experimental data and lines denote the linear fitting results).

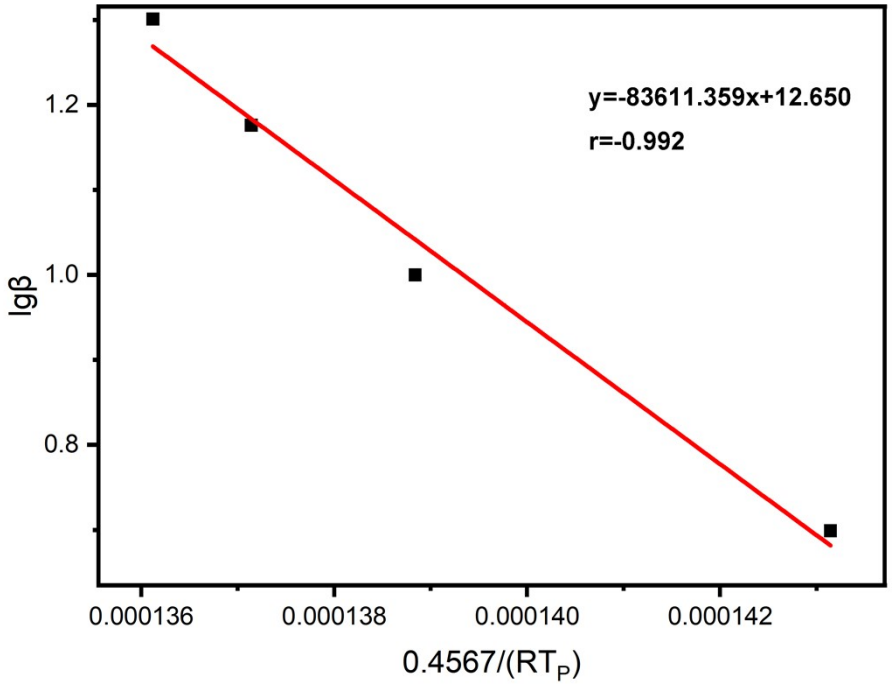

Fig. S30 Dependence of $\lg\beta$ on $0.4567/RT_p$ for CP **2** (Scatter points are the experimental data and lines denote the linear fitting results).

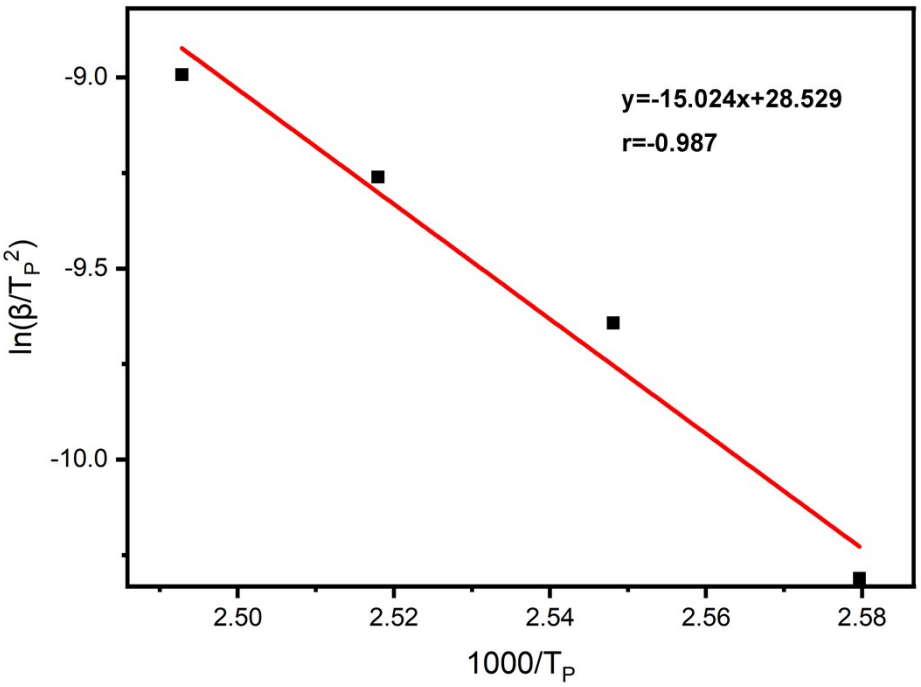

Fig. S31 Dependence of $\ln(\beta/T_p^2)$ on $1/T_p$ for CP **3** (Scatter points are the experimental data and lines denote the linear fitting results).

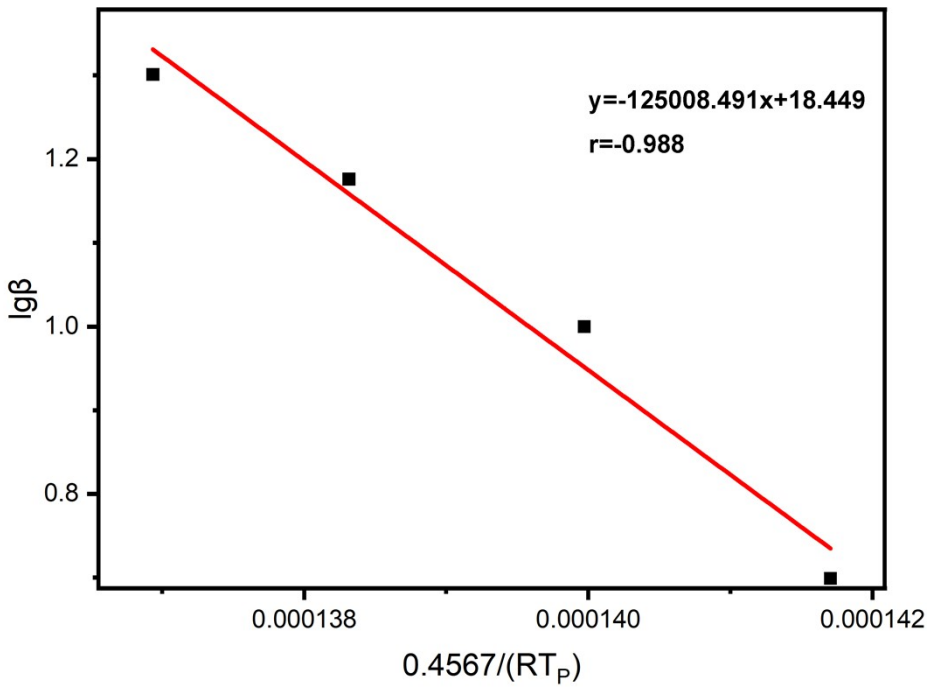

Fig. S32 Dependence of $\lg\beta$ on $0.4567/RT_p$ for CP **3** (Scatter points are the experimental data and lines denote the linear fitting results).

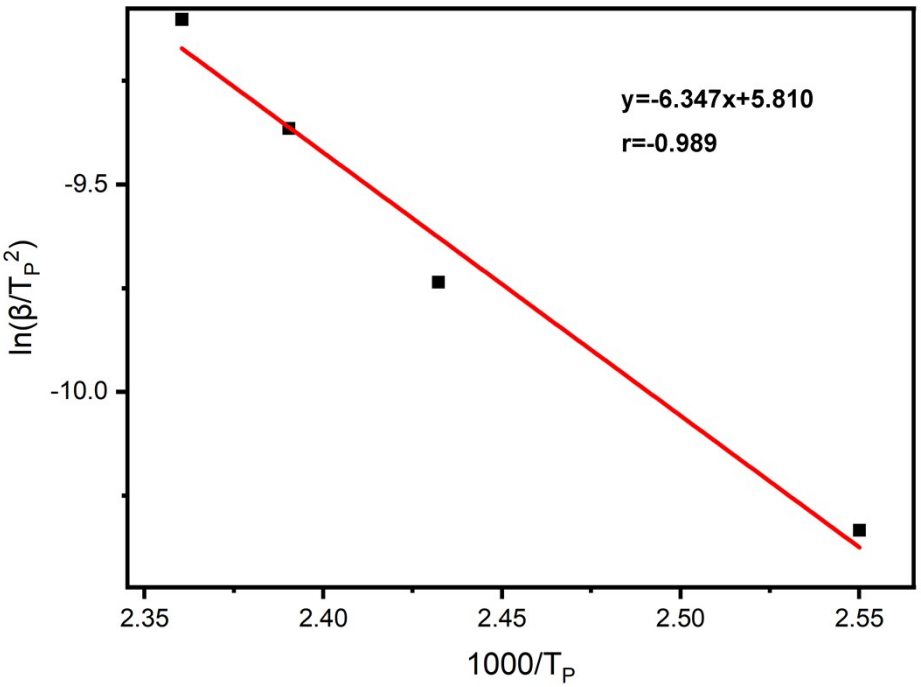

Fig. S33 Dependence of $\ln(\beta/T_p^2)$ on $1/T_p$ for CP **4** (Scatter points are the experimental data and lines denote the linear fitting results).

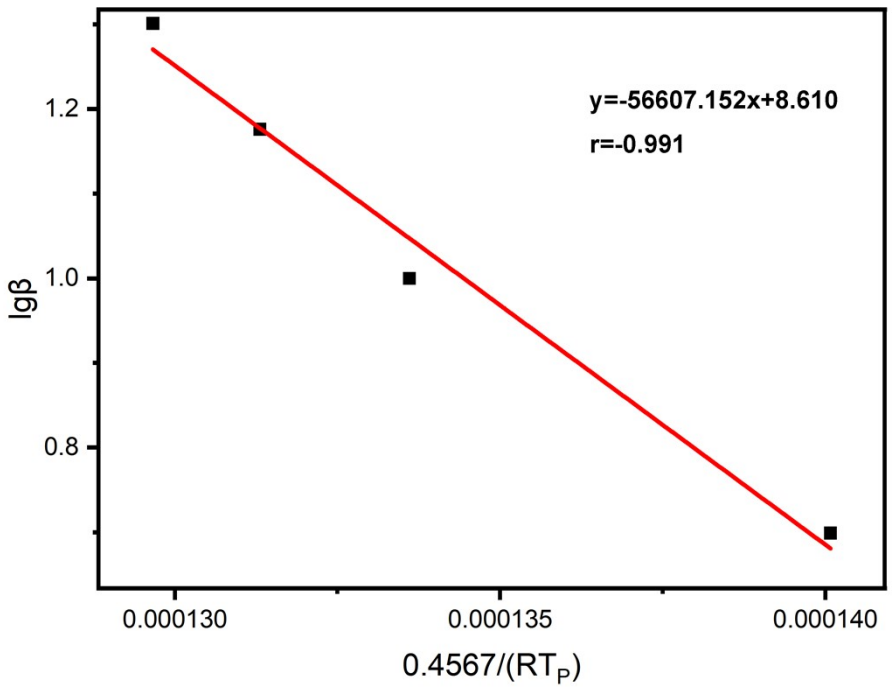

Fig. S34 Dependence of $\lg\beta$ on $0.4567/RT_p$ for CP **4** (Scatter points are the experimental data and lines denote the linear fitting results).