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Supporting information for article:

Synthesis and investigation of the thermal properties of $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$ and $[Ir(NH_3)_6][Ir(C_2O_4)_3]$

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Table S1 Crystal data and structure refinement for $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$.

Identification code	$[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$
Empirical formula	$C_6H_{24}Co_2N_6O_{15}$
Formula weight	538.17
Temperature/K	293(2)
Crystal system	trigonal
Space group	*P*-3
a/Å	12.3686(2)
b/Å	12.3686(2)
c/Å	21.1962(4)
α/°	90
β/°	90
γ/°	120
Volume/Å3	2808.21(11)
Z	6
ρ_{calc}g/cm^3	1.909
μ/mm^{-1}	1.861
F(000)	1656.0
Crystal size/mm^3	0.200 × 0.180 × 0.160
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.26 to 56.53
Index ranges	-16 ≤ h ≤ 16, -16 ≤ k ≤ 16, -28 ≤ l ≤ 17
Reflections collected	23212
Independent reflections	4663 [R_{int} = 0.0409, R_{sigma} = 0.0310]
Data/restraints/parameters	4663/7/291
Goodness-of-fit on F^2	1.058
Final R indexes [I>=2σ (I)]	R_1 = 0.0318, wR_2 = 0.0795
Final R indexes [all data]	R_1 = 0.0474, wR_2 = 0.0843
Largest diff. peak/hole / e Å^{-3}	0.73/-0.57

Table S2 Bond Lengths for $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
Co1	N1	1.9763(16)	Co5	O52	1.9119(14)
Co1	N1[1]	1.9763(16)	Co6	O62	1.8881(14)
Co1	N1[2]	1.9763(16)	Co6	O62[5]	1.8881(14)
Co1	N1[3]	1.9763(16)	Co6	O62[3]	1.8881(14)
Co1	N1[4]	1.9764(16)	Co6	O61	1.8964(14)
Co1	N1[5]	1.9764(16)	Co6	O61[3]	1.8964(14)
Co2	N2	1.9697(17)	Co6	O61[5]	1.8964(14)
Co2	N2[6]	1.9697(17)	Co7	O71[9]	1.8860(14)
Co2	N2[3]	1.9697(17)	Co7	O71[10]	1.8860(14)
Co2	N2[7]	1.9697(17)	Co7	O71	1.8860(14)
Co2	N2[8]	1.9697(17)	Co7	O72[9]	1.8883(14)
Co2	N2[5]	1.9697(17)	Co7	O72	1.8883(14)
Co3	N32[9]	1.9577(17)	Co7	O72[10]	1.8883(14)
Co3	N32[10]	1.9577(17)	O51	C51	1.288(2)
Co3	N32	1.9577(17)	O52	C52	1.293(2)
Co3	N31[9]	1.9648(17)	O53	C51	1.221(3)
Co3	N31	1.9648(17)	O54	C52	1.212(3)
Co3	N31[10]	1.9648(17)	O61	C61	1.285(3)
Co4	N41[11]	1.9667(17)	O62	C62	1.281(2)
Co4	N41	1.9667(17)	O63	C61	1.213(3)
Co4	N41[12]	1.9668(17)	O64	C62	1.226(2)
Co4	N42[11]	1.9709(18)	O71	C71	1.292(3)
Co4	N42[12]	1.9709(18)	O72	C72	1.287(3)
Co4	N42	1.9709(18)	O73	C71	1.216(3)
Co5	O51	1.9096(14)	O74	C72	1.227(3)
Co5	O51[11]	1.9096(14)	C51	C52	1.548(3)
Co5	O51[12]	1.9096(14)	C61	C62	1.539(3)
Co5	O52[11]	1.9119(14)	C71	C72	1.537(3)
Co5	O52[12]	1.9119(14)			

[1]-X,-Y,-Z; [2]+Y,-X+Y,-Z; [3]-Y,+X-Y,+Z; [4]-Y+X,+X,-Z; [5]+Y-X,-X,+Z; [6]+Y,-X+Y,1-Z; [7]-X,-Y,1-Z; [8]-Y+X,+X,1-Z; [9]+Y-X,1-X,+Z; [10]1-Y,1+X-Y,+Z; [11]1-Y,+X-Y,+Z; [12]1+Y-X,1-X,+Z

Table S3 Bond Angles for $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
N1	Co1	N1[1]	180.0	O51[12]	Co5	O52[11]	174.48(6)
N1	Co1	N1[2]	91.01(7)	O51	Co5	O52[12]	174.48(6)
N1[1]	Co1	N1[2]	88.99(7)	O51[11]	Co5	O52[12]	92.98(6)
N1	Co1	N1[3]	88.99(7)	O51[12]	Co5	O52[12]	85.26(6)
N1[1]	Co1	N1[3]	91.01(7)	O52[11]	Co5	O52[12]	91.73(6)
N1[2]	Co1	N1[3]	180.00(11)	O51	Co5	O52	85.27(6)
N1	Co1	N1[4]	91.01(7)	O51[11]	Co5	O52	174.48(6)
N1[1]	Co1	N1[4]	88.99(7)	O51[12]	Co5	O52	92.99(6)
N1[2]	Co1	N1[4]	88.99(7)	O52[11]	Co5	O52	91.72(6)
N1[3]	Co1	N1[4]	91.01(7)	O52[12]	Co5	O52	91.73(6)
N1	Co1	N1[5]	88.99(7)	O62	Co6	O62[5]	90.52(6)
N1[1]	Co1	N1[5]	91.01(7)	O62	Co6	O62[3]	90.52(6)
N1[2]	Co1	N1[5]	91.01(7)	O62[5]	Co6	O62[3]	90.52(6)
N1[3]	Co1	N1[5]	88.99(7)	O62	Co6	O61	86.22(6)
N1[4]	Co1	N1[5]	180.00(11)	O62[5]	Co6	O61	92.01(6)
N2	Co2	N2[6]	89.99(7)	O62[3]	Co6	O61	175.89(6)
N2	Co2	N2[3]	90.01(7)	O62	Co6	O61[3]	92.01(6)
N2[6]	Co2	N2[3]	180.00(8)	O62[5]	Co6	O61[3]	175.89(6)
N2	Co2	N2[7]	180.0	O62[3]	Co6	O61[3]	86.22(6)
N2[6]	Co2	N2[7]	90.01(7)	O61	Co6	O61[3]	91.39(6)
N2[3]	Co2	N2[7]	89.99(7)	O62	Co6	O61[5]	175.89(6)
N2	Co2	N2[8]	89.99(7)	O62[5]	Co6	O61[5]	86.22(6)
N2[6]	Co2	N2[8]	90.01(7)	O62[3]	Co6	O61[5]	92.01(6)
N2[3]	Co2	N2[8]	89.99(7)	O61	Co6	O61[5]	91.39(6)
N2[7]	Co2	N2[8]	90.01(7)	O61[3]	Co6	O61[5]	91.39(6)
N2	Co2	N2[5]	90.01(7)	O71[9]	Co7	O71[10]	92.30(6)
N2[6]	Co2	N2[5]	89.99(7)	O71[9]	Co7	O71	92.30(6)
N2[3]	Co2	N2[5]	90.01(7)	O71[10]	Co7	O71	92.30(6)

N2^{7}	Co2	N2^{5}	89.99(7)	O71^{9}	Co7	O72^{9}	86.05(6)
N2^{8}	Co2	N2^{5}	180.00(10)	O71^{10}	Co7	O72^{9}	89.92(7)
N32^{9}	Co3	N32^{10}	90.26(7)	O71	Co7	O72^{9}	177.29(6)
N32^{9}	Co3	N32	90.25(7)	O71^{9}	Co7	O72	89.92(7)
N32^{10}	Co3	N32	90.25(7)	O71^{10}	Co7	O72	177.29(6)
N32^{9}	Co3	N31^{9}	92.27(8)	O71	Co7	O72	86.06(6)
N32^{10}	Co3	N31^{9}	87.73(7)	O72^{9}	Co7	O72	91.79(6)
N32	Co3	N31^{9}	176.78(7)	O71^{9}	Co7	O72^{10}	177.29(6)
N32^{9}	Co3	N31	87.72(7)	O71^{10}	Co7	O72^{10}	86.05(6)
N32^{10}	Co3	N31	176.78(7)	O71	Co7	O72^{10}	89.92(7)
N32	Co3	N31	92.27(8)	O72^{9}	Co7	O72^{10}	91.79(6)
N31^{9}	Co3	N31	89.84(7)	O72	Co7	O72^{10}	91.79(6)
N32^{9}	Co3	N31^{10}	176.77(7)	C51	O51	Co5	113.22(13)
N32^{10}	Co3	N31^{10}	92.27(8)	C52	O52	Co5	113.38(13)
N32	Co3	N31^{10}	87.73(7)	C61	O61	Co6	112.47(13)
N31^{9}	Co3	N31^{10}	89.84(7)	C62	O62	Co6	113.12(13)
N31	Co3	N31^{10}	89.84(7)	C71	O71	Co7	113.05(13)
N41^{11}	Co4	N41	89.98(7)	C72	O72	Co7	112.84(13)
N41^{11}	Co4	N41^{12}	89.98(7)	O53	C51	O51	125.1(2)
N41	Co4	N41^{12}	89.98(7)	O53	C51	C52	120.64(19)
N41^{11}	Co4	N42^{11}	90.10(8)	O51	C51	C52	114.24(17)
N41	Co4	N42^{11}	88.67(7)	O54	C52	O52	125.8(2)
N41^{12}	Co4	N42^{11}	178.64(8)	O54	C52	C51	120.96(19)
N41^{11}	Co4	N42^{12}	88.67(7)	O52	C52	C51	113.27(17)
N41	Co4	N42^{12}	178.64(8)	O63	C61	O61	124.7(2)
N41^{12}	Co4	N42^{12}	90.10(8)	O63	C61	C62	121.0(2)
N42^{11}	Co4	N42^{12}	91.25(8)	O61	C61	C62	114.29(17)
N41^{11}	Co4	N42	178.64(8)	O64	C62	O62	124.6(2)
N41	Co4	N42	90.10(8)	O64	C62	C61	121.5(2)
N41^{12}	Co4	N42	88.67(7)	O62	C62	C61	113.89(17)
N42^{11}	Co4	N42	91.25(8)	O73	C71	O71	125.4(2)
N42^{12}	Co4	N42	91.25(8)	O73	C71	C72	121.4(2)

O51	Co5	O51[11]	90.27(6)	O71	C71	C72	113.17(18)
O51	Co5	O51[12]	90.27(6)	O74	C72	O72	124.3(2)
O51[11]	Co5	O51[12]	90.27(6)	O74	C72	C71	121.4(2)
O51	Co5	O52[11]	92.98(6)	O72	C72	C71	114.19(18)
O51[11]	Co5	O52[11]	85.26(6)				

[1]-X,-Y,-Z; [2]+Y,-X+Y,-Z; [3]-Y,+X-Y,+Z; [4]-Y+X,+X,-Z; [5]+Y-X,-X,+Z; [6]+Y,-X+Y,1-Z; [7]-X,-Y,1-Z; [8]-Y+X,+X,1-Z; [9]+Y-X,1-X,+Z; [10]1-Y,1+X-Y,+Z; [11]1-Y,+X-Y,+Z; [12]1+Y-X,1-X,+Z

Table S4 Crystal data and structure refinement for $[Ir(NH_3)_6][Ir(C_2O_4)_3]$.

Identification code	$[Ir(NH_3)_6][Ir(C_2O_4)_3]$
Empirical formula	$C_6H_{18}Ir_2N_6O_{12}$
Formula weight	750.66
Temperature/K	293(2)
Crystal system	triclinic
Space group	*P*-1
a/Å	7.5600(2)
b/Å	9.7304(3)
c/Å	11.9386(3)
α/°	84.3680(10)
β/°	87.5790(10)
γ/°	70.5340(10)
Volume/Å^3	823.99(4)
Z	2
ρ_{calc}g/cm^3	3.026
μ/mm^{-1}	16.211
F(000)	692.0
Crystal size/mm^3	0.100 × 0.070 × 0.040
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.428 to 56.64
Index ranges	$-10 \le h \le 9$, $-12 \le k \le 12$, $-15 \le l \le 15$
Reflections collected	7770
Independent reflections	4063 [R_{int} = 0.0340, R_{sigma} = 0.0474]
Data/restraints/parameters	4063/0/240

Goodness-of-fit on F^2	1.006
Final R indexes [I>=2σ (I)]	R_1 = 0.0221, wR_2 = 0.0544
Final R indexes [all data]	R_1 = 0.0274, wR_2 = 0.0557
Largest diff. peak/hole / e $Å^{-3}$	1.45/-1.29

Table S5 Bond Lengths for $[Ir(NH_3)_6][Ir(C_2O_4)_3]$.

Atom	**Atom**	**Length/Å**	**Atom**	**Atom**	**Length/Å**
Ir1	N6	2.051(4)	O13	C11	1.226(6)
Ir1	N2	2.056(4)	O14	C12	1.217(6)
Ir1	N3	2.079(4)	O21	C21	1.308(6)
Ir1	N4	2.089(4)	O22	C22	1.296(6)
Ir1	N1	2.093(4)	O23	C21	1.212(5)
Ir1	N5	2.098(4)	O24	C22	1.218(6)
Ir2	O11	2.029(3)	O31	C31	1.298(6)
Ir2	O21	2.041(3)	O32	C32	1.287(6)
Ir2	O31	2.043(3)	O33	C31	1.219(6)
Ir2	O22	2.050(3)	O34	C32	1.211(6)
Ir2	O12	2.051(3)	C11	C12	1.553(7)
Ir2	O32	2.053(3)	C21	C22	1.550(7)
O11	C11	1.289(6)	C31	C32	1.545(7)
O12	C12	1.283(6)			

Table S6 Bond Angles for $[Ir(NH_3)_6][Ir(C_2O_4)_3]$.

Atom	**Atom**	**Atom**	**Angle/°**	**Atom**	**Atom**	**Atom**	**Angle/°**
N6	Ir1	N2	88.43(16)	O31	Ir2	O32	81.82(14)
N6	Ir1	N3	89.42(17)	O22	Ir2	O32	97.18(14)
N2	Ir1	N3	88.87(17)	O12	Ir2	O32	96.99(14)
N6	Ir1	N4	91.04(17)	C11	O11	Ir2	112.1(3)
N2	Ir1	N4	178.71(16)	C12	O12	Ir2	111.8(3)
N3	Ir1	N4	89.95(18)	C21	O21	Ir2	112.9(3)
N6	Ir1	N1	89.42(19)	C22	O22	Ir2	112.5(3)
N2	Ir1	N1	89.62(19)	C31	O31	Ir2	112.3(3)

N3	Ir1	N1	178.13(18)	C32	O32	Ir2	111.5(3)
N4	Ir1	N1	91.55(19)	O13	C11	O11	125.0(5)
N6	Ir1	N5	178.15(18)	O13	C11	C12	120.5(5)
N2	Ir1	N5	90.40(17)	O11	C11	C12	114.5(4)
N3	Ir1	N5	91.98(18)	O14	C12	O12	123.7(5)
N4	Ir1	N5	90.16(18)	O14	C12	C11	119.9(5)
N1	Ir1	N5	89.1(2)	O12	C12	C11	116.4(4)
O11	Ir2	O21	95.91(14)	O23	C21	O21	123.0(5)
O11	Ir2	O31	96.78(14)	O23	C21	C22	121.3(4)
O21	Ir2	O31	94.33(14)	O21	C21	C22	115.7(4)
O11	Ir2	O22	84.27(15)	O24	C22	O22	123.4(5)
O21	Ir2	O22	82.35(14)	O24	C22	C21	120.3(4)
O31	Ir2	O22	176.61(13)	O22	C22	C21	116.3(4)
O11	Ir2	O12	81.90(14)	O33	C31	O31	123.9(5)
O21	Ir2	O12	177.12(13)	O33	C31	C32	120.1(5)
O31	Ir2	O12	87.81(14)	O31	C31	C32	115.9(4)
O22	Ir2	O12	95.54(14)	O34	C32	O32	124.2(5)
O11	Ir2	O32	178.26(13)	O34	C32	C31	119.9(5)
O21	Ir2	O32	85.24(14)	O32	C32	C31	115.9(4)

Table S7 Crystal data and structure refinement for $K_3[Co(NH_3)_6][Ir(C_2O_4)_3]_2\cdot 6H_2O$.

Identification code	$K_3[Co(NH_3)_6][Ir(C_2O_4)_3]_2\cdot 6H_2O$
Empirical formula	$C_{12}H_{30}CoIr_2K_3N_6O_{30}$
Formula weight	1272.02
Temperature/K	293(2)
Crystal system	trigonal
Space group	*R*-3
a/Å	10.2003(8)
b/Å	10.2003(8)
c/Å	29.408(4)
α/°	90

β/°	90
γ/°	120
Volume/Å^3	2649.8(5)
Z	3
ρ_{calc}g/cm^3	2.391
μ/mm^{-1}	8.441
F(000)	1821.0
Crystal size/mm^3	0.120 × 0.080 × 0.040
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.38 to 52.76
Index ranges	-12 ≤ h ≤ 12, -12 ≤ k ≤ 12, -36 ≤ l ≤ 36
Reflections collected	7274
Independent reflections	1200 [R_{int} = 0.0424, R_{sigma} = 0.0253]
Data/restraints/parameters	1200/12/93
Goodness-of-fit on F^2	1.095
Final R indexes [I>=2σ (I)]	R_1 = 0.0191, wR_2 = 0.0461
Final R indexes [all data]	R_1 = 0.0213, wR_2 = 0.0469
Largest diff. peak/hole / e Å^{-3}	0.90/-0.59

Table S8 Bond Lengths for $K_3[Co(NH_3)_6][Ir(C_2O_4)_3]_2 \cdot 6H_2O$.

Atom	**Atom**	**Length/Å**	**Atom**	**Atom**	**Length/Å**
Ir1	O2	2.029(2)	K1	O2^{11}	2.943(3)
Ir1	O2^1	2.029(2)	K1	O2^3	2.943(3)
Ir1	O2^2	2.029(2)	K1	C2^9	3.468(3)
Ir1	O1^2	2.046(2)	K1	C2^{10}	3.468(3)
Ir1	O1^1	2.046(2)	K1	Ir1^3	3.4783(14)
Ir1	O1	2.046(2)	K2	O3^{12}	2.715(3)
Ir1	K1^3	3.4781(14)	K2	O3^{10}	2.715(3)
Co1	N1	1.966(3)	K2	O3^{13}	2.715(3)
Co1	N1^4	1.966(3)	K2	O3^{14}	2.715(3)
Co1	N1^5	1.966(3)	K2	O3^9	2.715(3)
Co1	N1^6	1.966(3)	K2	O3	2.715(3)

Co1	N1[7]	1.966(3)	K2	K1[12]	3.7769(14)
Co1	N1[8]	1.966(3)	O1	C1	1.296(4)
K1	O4[9]	2.723(3)	O2	C2	1.284(4)
K1	O4[10]	2.723(3)	O2	K1[3]	2.943(3)
K1	O4	2.723(3)	O3	C1	1.216(4)
K1	O3[10]	2.845(3)	O4	C2	1.223(4)
K1	O3[9]	2.845(3)	C1	C2	1.553(5)
K1	O3	2.845(3)	O1W	O2W	1.101(13)
K1	O2[5]	2.943(3)			

[1]1+Y-X,1-X,+Z; [2]1-Y,+X-Y,+Z; [3]1-X,1-Y,-Z; [4]-X,-Y,-Z; [5]-Y+X,+X,-Z; [6]-Y,+X-Y,+Z; [7]+Y,-X+Y,-Z; [8]+Y-X,-X,+Z; [9]1-Y,1+X-Y,+Z; [10]+Y-X,1-X,+Z; [11]+Y,1-X+Y,-Z; [12]2/3-X,4/3-Y,1/3-Z; [13]-1/3+Y,1/3-X+Y,1/3-Z; [14]2/3-Y+X,1/3+X,1/3-Z

Table S9 Bond Angles for $K_3[Co(NH_3)_6][Ir(C_2O_4)_3]_2\cdot 6H_2O$.

Atom	**Atom**	**Atom**	**Angle/°**	**Atom**	**Atom**	**Atom**	**Angle/°**
O2	Ir1	O2[1]	94.04(9)	O3	K1	O2[3]	101.10(7)
O2	Ir1	O2[2]	94.04(9)	O2[5]	K1	O2[3]	60.58(8)
O2[1]	Ir1	O2[2]	94.03(9)	O2[11]	K1	O2[3]	60.58(8)
O2	Ir1	O1[2]	175.98(9)	O4[9]	K1	C2[9]	18.15(7)
O2[1]	Ir1	O1[2]	86.42(10)	O4[10]	K1	C2[9]	135.00(7)
O2[2]	Ir1	O1[2]	81.95(9)	O4	K1	C2[9]	105.09(8)
O2	Ir1	O1[1]	86.42(10)	O3[10]	K1	C2[9]	86.94(8)
O2[1]	Ir1	O1[1]	81.95(9)	O3[9]	K1	C2[9]	43.40(8)
O2[2]	Ir1	O1[1]	175.98(9)	O3	K1	C2[9]	120.11(9)
O1[2]	Ir1	O1[1]	97.60(9)	O2[5]	K1	C2[9]	83.23(7)
O2	Ir1	O1	81.95(9)	O2[11]	K1	C2[9]	76.17(7)
O2[1]	Ir1	O1	175.98(9)	O2[3]	K1	C2[9]	132.92(8)
O2[2]	Ir1	O1	86.43(10)	O4[9]	K1	C2[10]	105.09(8)
O1[2]	Ir1	O1	97.59(9)	O4[10]	K1	C2[10]	18.14(7)
O1[1]	Ir1	O1	97.59(9)	O4	K1	C2[10]	135.00(7)
O2	Ir1	K1[3]	57.65(7)	O3[10]	K1	C2[10]	43.40(8)
O2[1]	Ir1	K1[3]	57.64(7)	O3[9]	K1	C2[10]	120.11(9)
O2[2]	Ir1	K1[3]	57.64(7)	O3	K1	C2[10]	86.94(8)

O1^{2}	Ir1	K1^{3}	119.68(7)	O2^{5}	K1	C2^{10}	76.18(7)
O1^{1}	Ir1	K1^{3}	119.68(7)	O2^{11}	K1	C2^{10}	132.92(8)
O1	Ir1	K1^{3}	119.69(7)	O2^{3}	K1	C2^{10}	83.23(7)
N1	Co1	N1^{4}	180.0	C2^{9}	K1	C2^{10}	118.26(3)
N1	Co1	N1^{5}	89.36(14)	O4^{9}	K1	Ir1^{3}	87.53(6)
N1^{4}	Co1	N1^{5}	90.64(14)	O4^{10}	K1	Ir1^{3}	87.53(6)
N1	Co1	N1^{6}	90.64(14)	O4	K1	Ir1^{3}	87.53(6)
N1^{4}	Co1	N1^{6}	89.36(14)	O3^{10}	K1	Ir1^{3}	134.22(6)
N1^{5}	Co1	N1^{6}	89.36(14)	O3^{9}	K1	Ir1^{3}	134.22(6)
N1	Co1	N1^{7}	89.36(14)	O3	K1	Ir1^{3}	134.22(6)
N1^{4}	Co1	N1^{7}	90.64(14)	O2^{5}	K1	Ir1^{3}	35.62(5)
N1^{5}	Co1	N1^{7}	90.64(14)	O2^{11}	K1	Ir1^{3}	35.62(5)
N1^{6}	Co1	N1^{7}	180.00(19)	O2^{3}	K1	Ir1^{3}	35.62(5)
N1	Co1	N1^{8}	90.64(14)	C2^{9}	K1	Ir1^{3}	97.64(6)
N1^{4}	Co1	N1^{8}	89.36(14)	C2^{10}	K1	Ir1^{3}	97.64(6)
N1^{5}	Co1	N1^{8}	180.0(2)	O3^{12}	K2	O3^{10}	98.87(9)
N1^{6}	Co1	N1^{8}	90.64(14)	O3^{12}	K2	O3^{13}	81.13(9)
N1^{7}	Co1	N1^{8}	89.36(14)	O3^{10}	K2	O3^{13}	98.87(9)
O4^{9}	K1	O4^{10}	119.815(10)	O3^{12}	K2	O3^{14}	81.13(9)
O4^{9}	K1	O4	119.817(10)	O3^{10}	K2	O3^{14}	180.0
O4^{10}	K1	O4	119.817(10)	O3^{13}	K2	O3^{14}	81.13(9)
O4^{9}	K1	O3^{10}	83.23(9)	O3^{12}	K2	O3^{9}	98.87(9)
O4^{10}	K1	O3^{10}	59.82(8)	O3^{10}	K2	O3^{9}	81.13(9)
O4	K1	O3^{10}	135.29(9)	O3^{13}	K2	O3^{9}	180.0
O4^{9}	K1	O3^{9}	59.82(8)	O3^{14}	K2	O3^{9}	98.87(9)
O4^{10}	K1	O3^{9}	135.29(9)	O3^{12}	K2	O3	180.0
O4	K1	O3^{9}	83.23(9)	O3^{10}	K2	O3	81.13(9)
O3^{10}	K1	O3^{9}	76.72(9)	O3^{13}	K2	O3	98.87(9)
O4^{9}	K1	O3	135.29(9)	O3^{14}	K2	O3	98.87(9)
O4^{10}	K1	O3	83.23(9)	O3^{9}	K2	O3	81.13(9)
O4	K1	O3	59.82(8)	O3^{12}	K2	K1^{12}	48.67(6)
O3^{10}	K1	O3	76.72(9)	O3^{10}	K2	K1^{12}	131.33(6)

O3[9]	K1	O3	76.72(9)	O3[13]	K2	K1[12]	48.67(6)
O4[9]	K1	O2[5]	66.77(7)	O3[14]	K2	K1[12]	48.67(6)
O4[10]	K1	O2[5]	75.43(8)	O3[9]	K2	K1[12]	131.33(6)
O4	K1	O2[5]	122.75(8)	O3	K2	K1[12]	131.33(6)
O3[10]	K1	O2[5]	101.10(7)	C1	O1	Ir1	112.5(2)
O3[9]	K1	O2[5]	126.47(7)	C2	O2	Ir1	113.4(2)
O3	K1	O2[5]	156.05(8)	C2	O2	K1[3]	124.5(2)
O4[9]	K1	O2[11]	75.43(8)	Ir1	O2	K1[3]	86.74(8)
O4[10]	K1	O2[11]	122.75(8)	C1	O3	K2	144.3(3)
O4	K1	O2[11]	66.77(7)	C1	O3	K1	114.8(2)
O3[10]	K1	O2[11]	156.05(8)	K2	O3	K1	85.55(8)
O3[9]	K1	O2[11]	101.10(7)	C2	O4	K1	118.0(2)
O3	K1	O2[11]	126.47(7)	O3	C1	O1	124.4(3)
O2[5]	K1	O2[11]	60.58(8)	O3	C1	C2	120.1(3)
O4[9]	K1	O2[3]	122.75(8)	O1	C1	C2	115.5(3)
O4[10]	K1	O2[3]	66.77(7)	O4	C2	O2	123.9(3)
O4	K1	O2[3]	75.43(8)	O4	C2	C1	120.2(3)
O3[10]	K1	O2[3]	126.47(7)	O2	C2	C1	115.9(3)
O3[9]	K1	O2[3]	156.05(8)				

[1]1+Y-X,1-X,+Z; [2]1-Y,+X-Y,+Z; [3]1-X,1-Y,-Z; [4]-X,-Y,-Z; [5]-Y+X,+X,-Z; [6]-Y,+X-Y,+Z; [7]+Y,-X+Y,-Z; [8]+Y-X,-X,+Z; [9]1-Y,1+X-Y,+Z; [10]+Y-X,1-X,+Z; [11]+Y,1-X+Y,-Z; [12]2/3-X,4/3-Y,1/3-Z; [13]-1/3+Y,1/3-X+Y,1/3-Z; [14]2/3-Y+X,1/3+X,1/3-Z

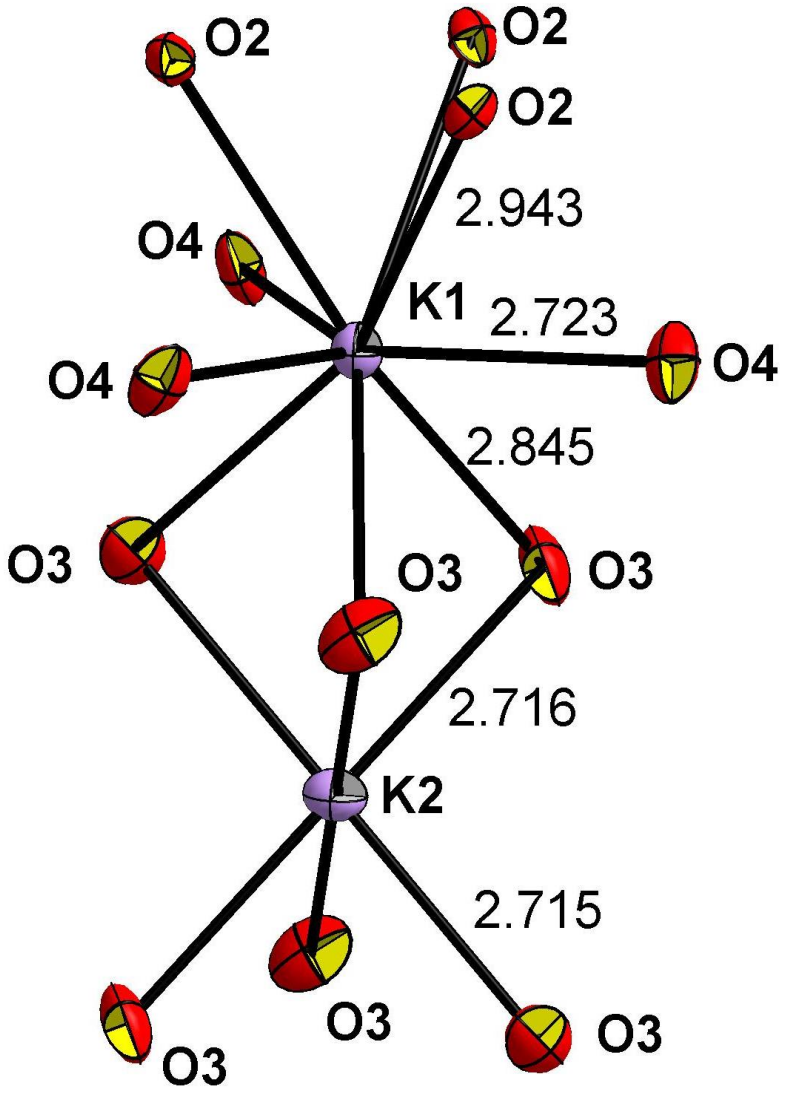

Figure S1 The crystal environment of the potassium cations in the $K_3[Co(NH_3)_6][Ir(C_2O_4)_3]_2{\cdot}6H_2O$ complex salt.

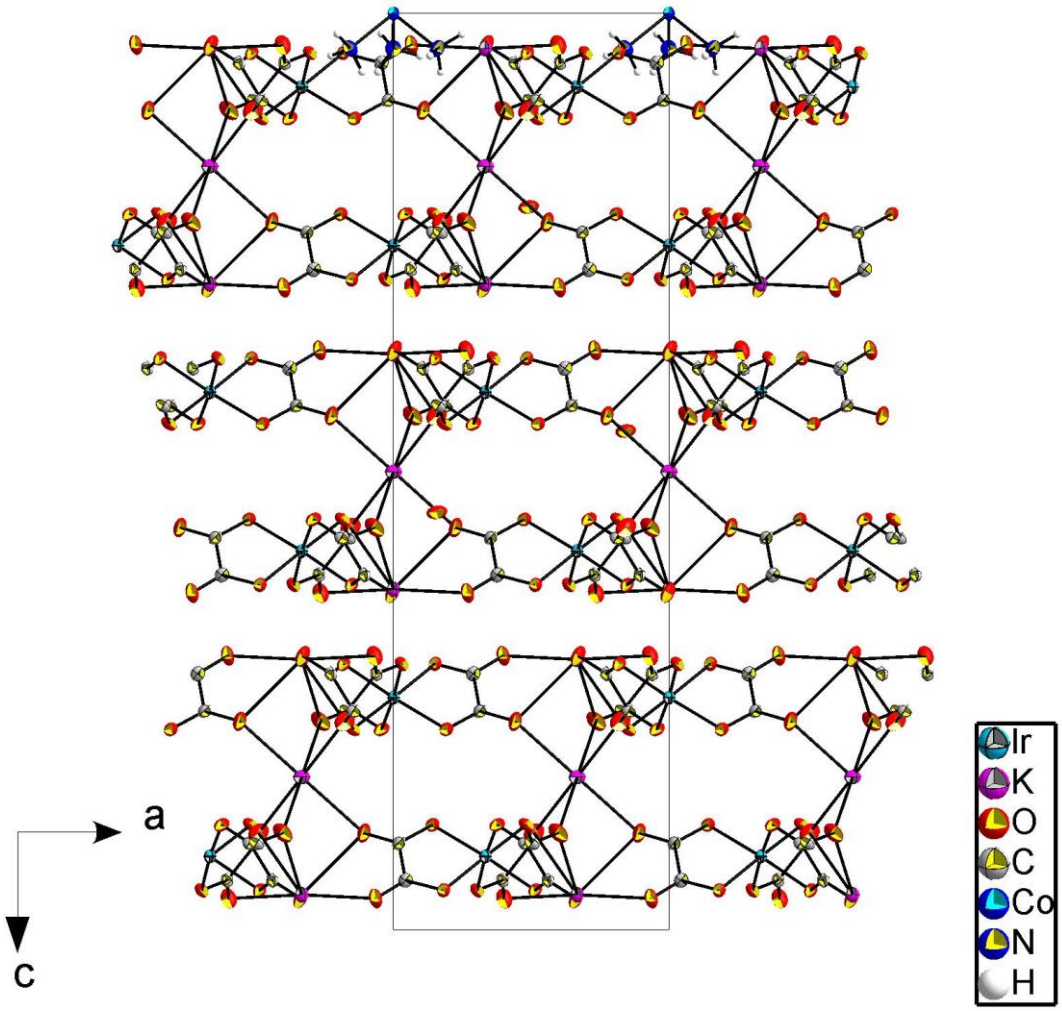

Figure S2 The anionic substructure layers in the $K_3[Co(NH_3)_6][Ir(C_2O_4)_3]_2{\cdot}6H_2O$ compound.

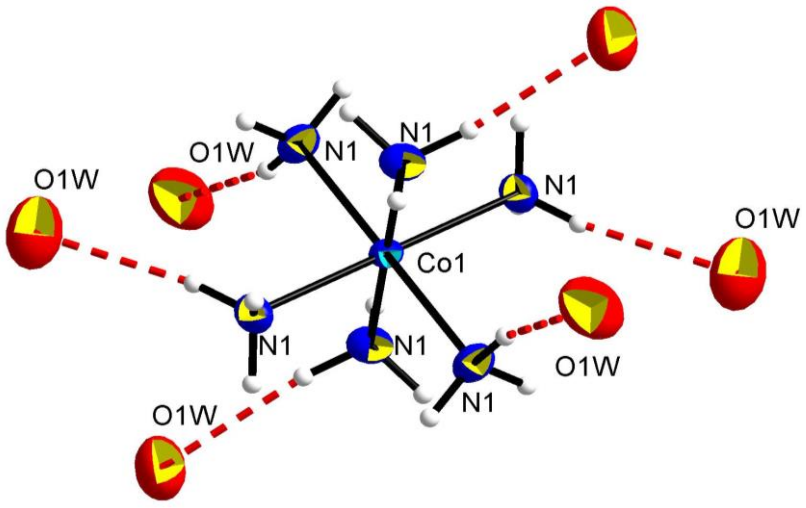

Figure S3 The $[Co(NH_3)_6]^{3+}$ cation and solvating water molecules in the $K_3[Co(NH_3)_6][Ir(C_2O_4)_3]_2{\cdot}6H_2O$.

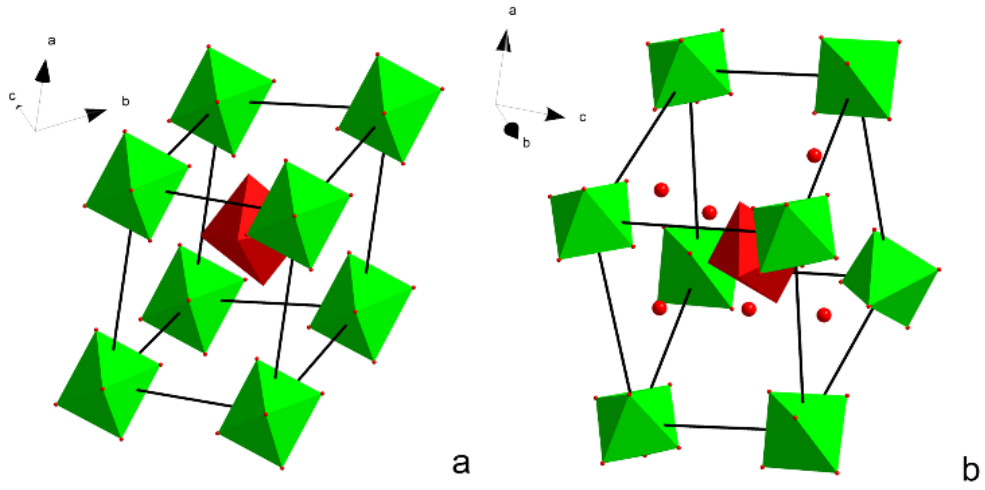

Figure S4 a) distorted cubic unit in the structure of [Ir-Ir] DCS (*P*-1), b) distorted cubic unit in the structure of [Co-Co] DCS (*P*-3), oxygen atoms of solvate water molecules are shown in red. Green polyhedrons corresponds to $[M(C_2O_4)_3]^{3-}$ anions, red polyhedron – cation of $[M(NH_3)_6]^{3+}$.

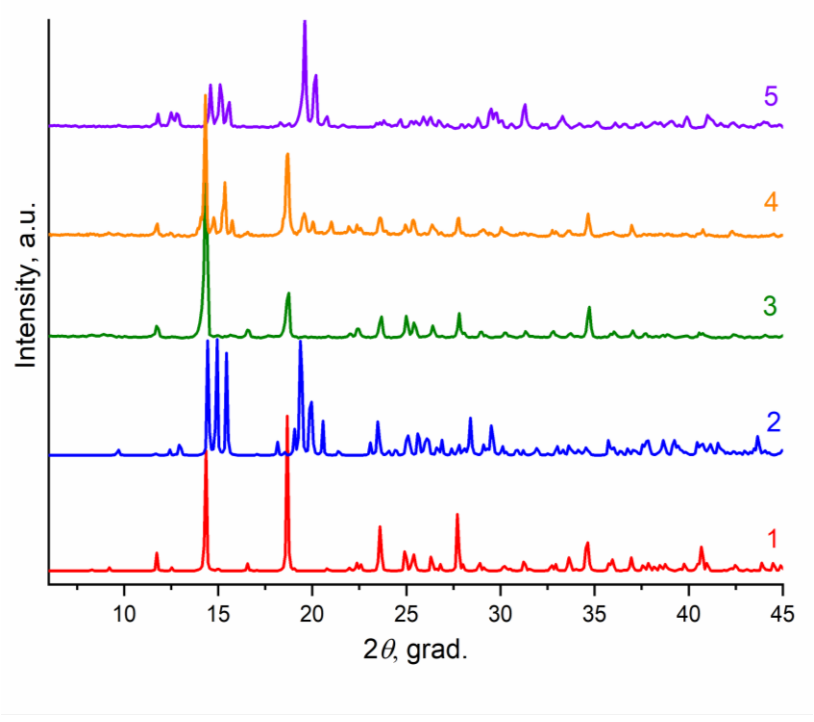

Figure S5 1 – $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$, calculated from single crystal data; 2 – $[Ir(NH_3)_6][Ir(C_2O_4)_3]$, calculated from single crystal data; 3 – $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$ (XRD, as prepared); 4 – $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$ (XRD, 20 days after synthesis); 5 – $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$ (XRD, 30 days after synthesis).

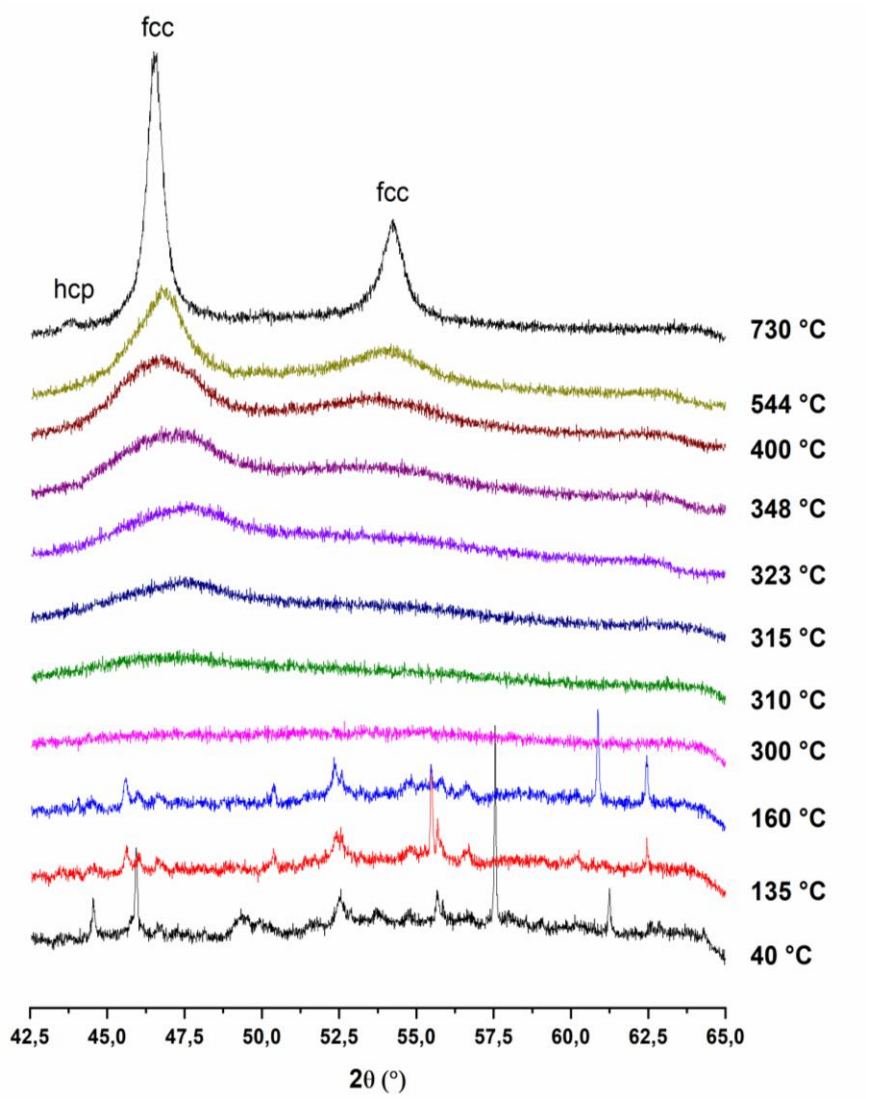

Figure S6 *In situ* X-ray diffraction patterns of $[Co(NH_3)_6][Ir(C_2O_4)_3]$ under different temperatures in the hydrogen atmosphere.

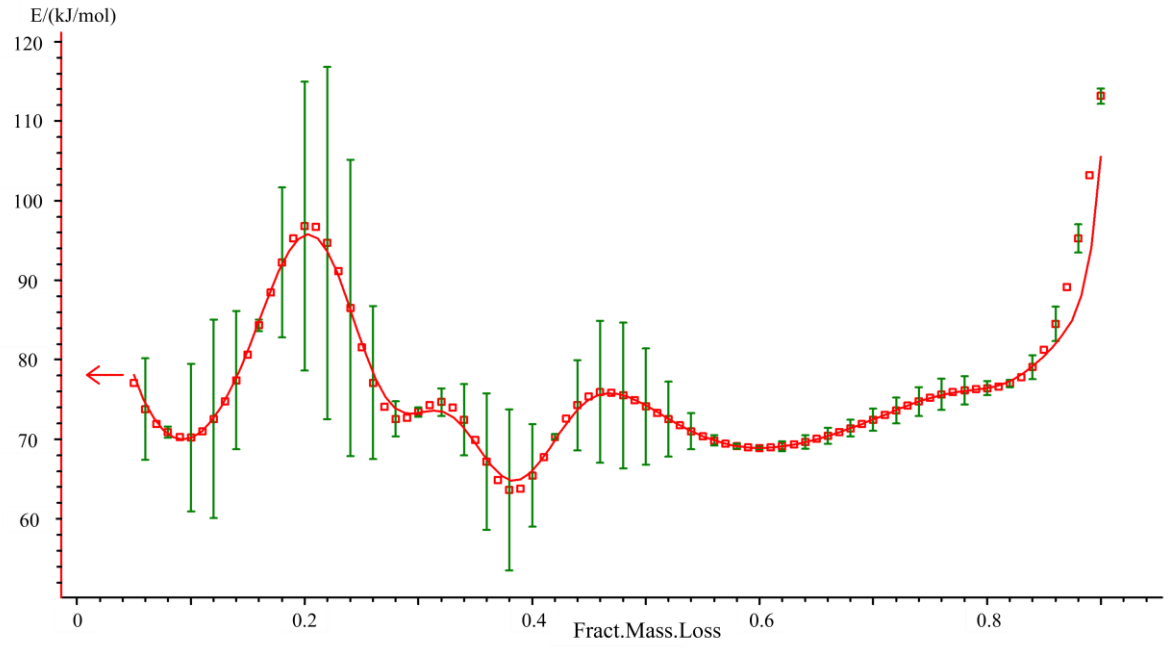

Figure S7 Friedman analysis of the dehydration of $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$: activation energies depend on the degree of conversion. Perpendicular lines denote standard deviations.

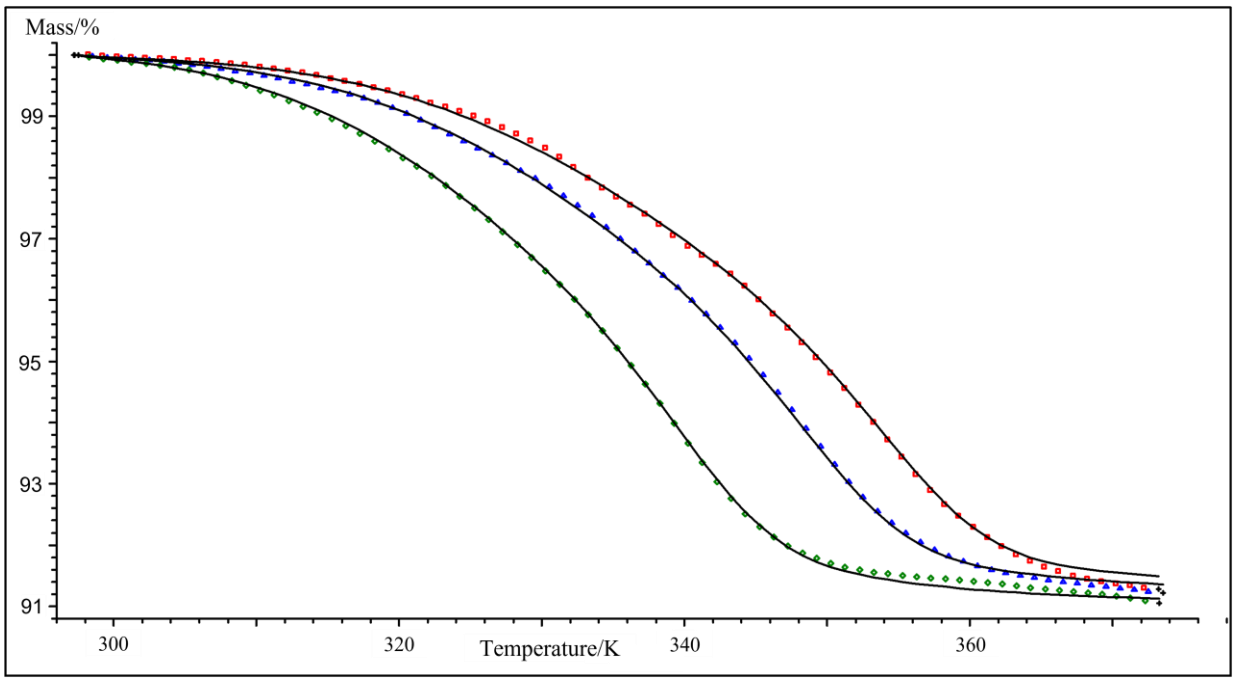

Figure S8 Thermal dehydration of $[Co(NH_3)_6][Co(C_2O_4)_3]\cdot 3H_2O$. Data processing: non-linear regression curve fitting simulated with two consecutive reactions A $\rightarrow$ B $\rightarrow$ C; equations Fn and An. The points are the experimental data; the lines are the calculated data.

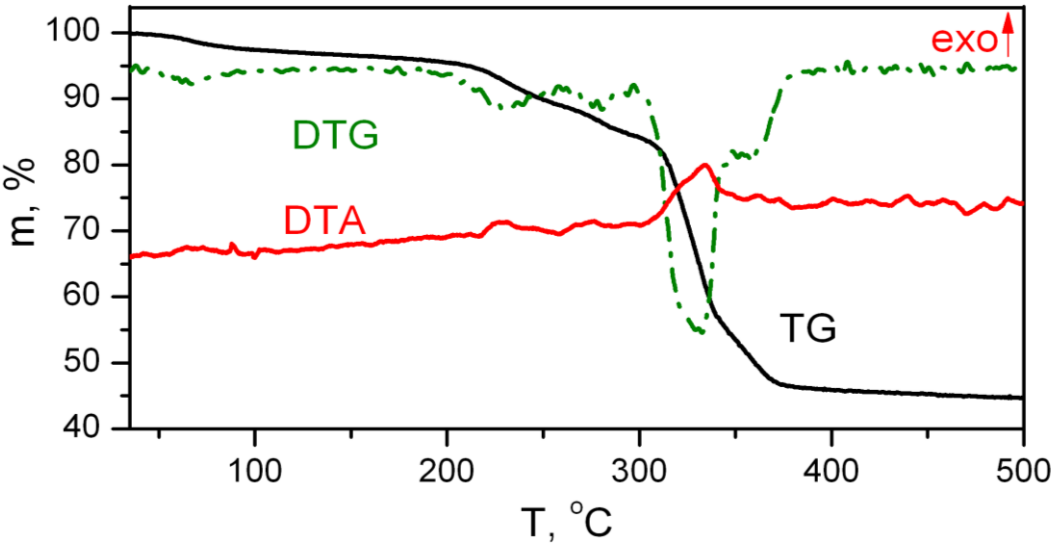

Figure S9 TG-, DTG- and DTA curves for $K_3[Co(NH_3)_6][Ir(C_2O_4)_3]_2 \cdot 6H_2O$ in an inert atmosphere.

Table S10 Data on the F-test of fit quality/to identify the best kinetic description, α = 0.005–0.995.

F_{exp}	F_{crit}	f-act	Equation A → B	Equation B → C
1.00	1.25	218	Fn	An
2.42	1.25	219	Fn	Fn
3.23	1.25	218	Fn	Fn
3.41	1.25	218	An	An
3.88	1.25	218	An	Fn