SUPPLEMENTARY INFORMATION

for

A new approach to the synthesis of nanocrystalline cobalt boride in the course of the thermal reduction of cobalt complexes with boron cluster anions

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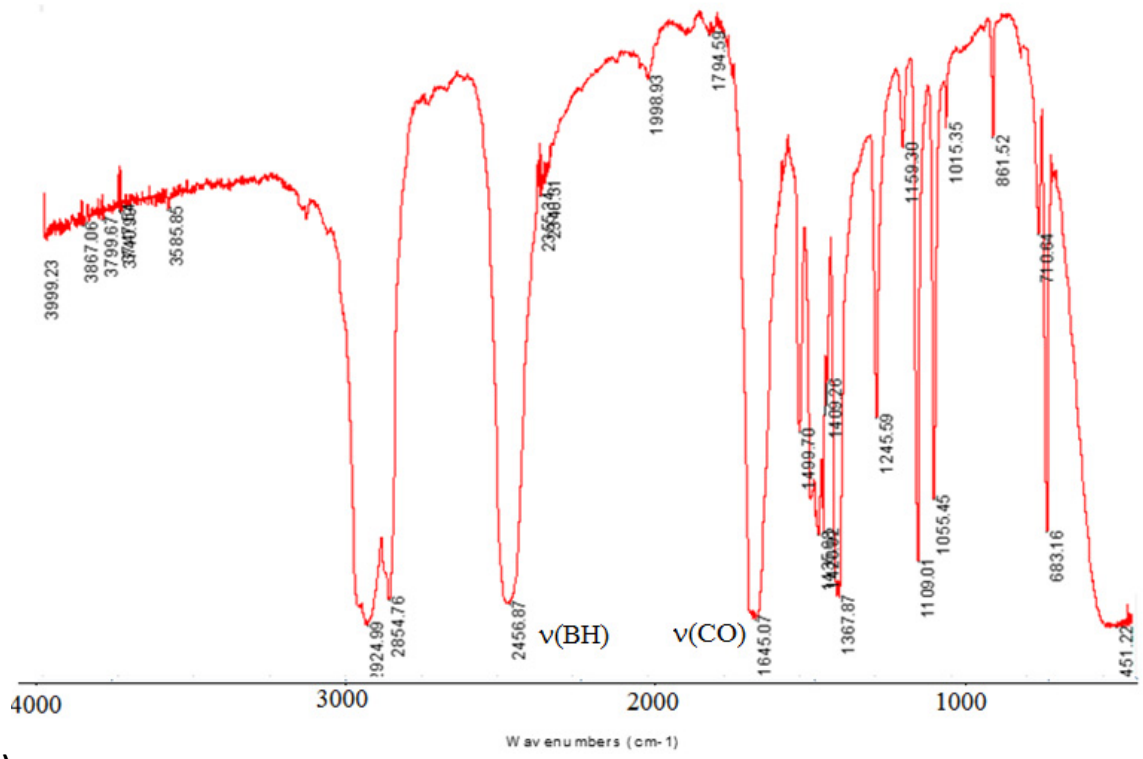
Table S1. Crystal data and structure refinement for complex **1**.

Compound	**1**
Empirical formula	$C_{18}H_{54}B_{12}CoN_6O_6$
Formula weight	639.32
Temperature/K	150.15
Crystal system	triclinic
Space group	P-1
a/Å	9.5857(5)
b/Å	18.9576(11)
c/Å	19.1748(11)
α/°	89.9010(10)
β/°	85.0580(10)
γ/°	89.9280(10)
Volume/Å^3	3471.5(3)
Z	4
ρ_{calc}g/cm^3	1.223
μ/mm^{-1}	0.535
F(000)	1356.0
Radiation	MoKα (λ = 0.71073)
Index ranges	$-13 \le h \le 13$ $-25 \le k \le 25$ $-25 \le l \le 26$
Reflections collected	38452
Independent reflections	18096 [R_{int} = 0.0531, R_{sigma} = 0.0906]
Data/restraints/parameters	18096/0/770
Goodness-of-fit on F^2	0.984
Final R indexes [I>=2σ (I)]	R_1 = 0.0510, wR_2 = 0.1001
Final R indexes [all data]	R_1 = 0.1092, wR_2 = 0.1201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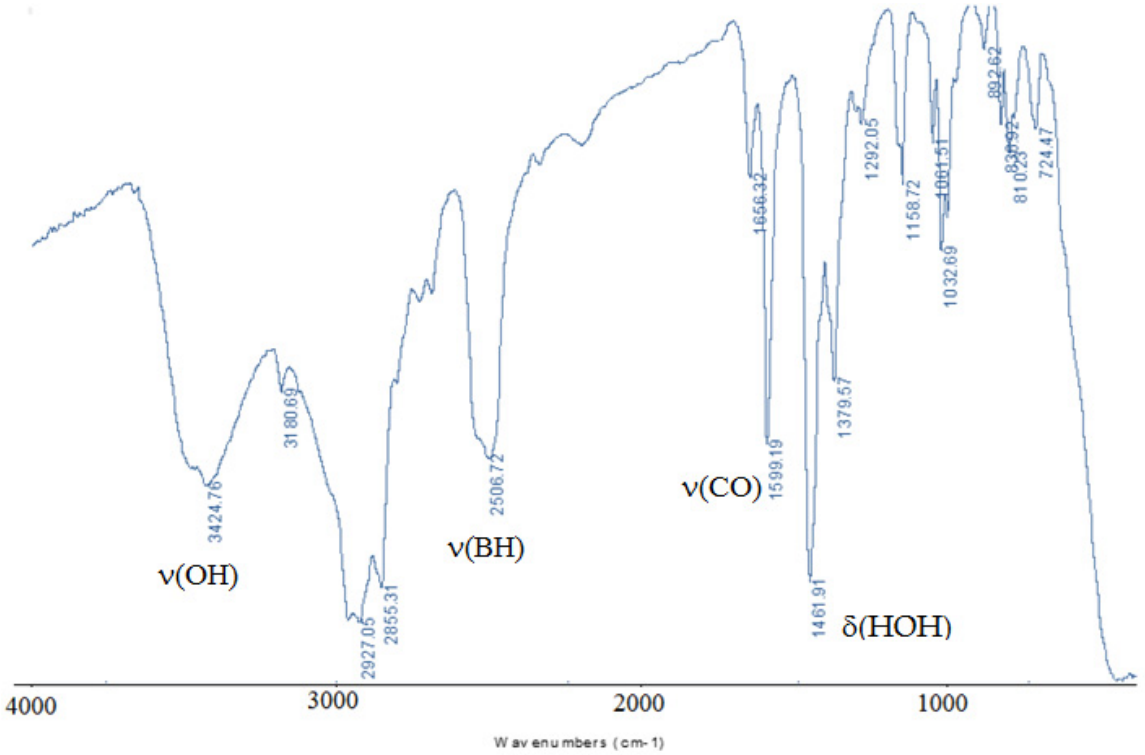
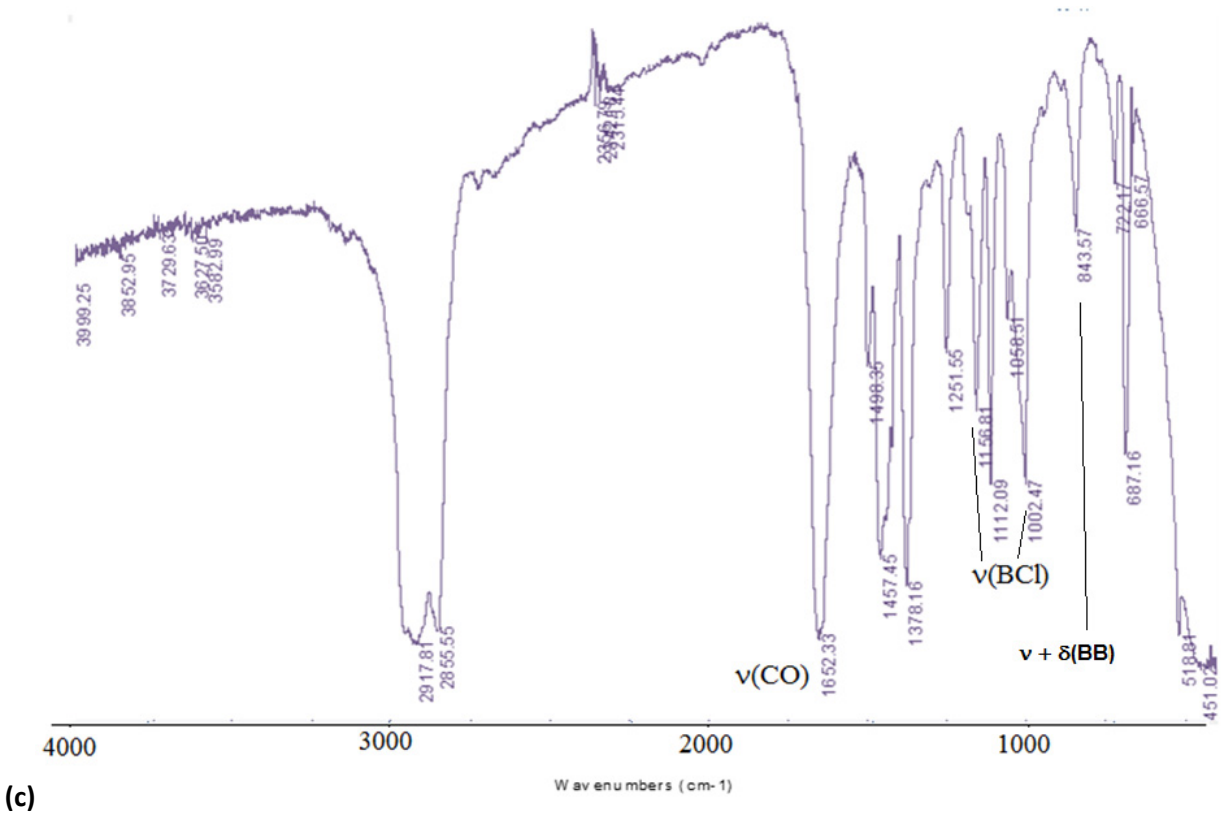
3867.06
3799.67
3585.85
2456.87
2854.76
1998.93
1794.59
1645.07
1409.70
1367.87
1245.59
1159.30
1109.01
1055.45
1015.35
861.52
683.16
ν(BH)
ν(CO)
4000
3000
2000
1000
Wavenumbers (cm-1)

(a)


3424.76
3180.69
2927.05
2855.31
2506.72
1656.32
1599.19
1461.91
1379.57
1292.05
1158.72
1032.69
892.62
810.23
724.47
ν(OH)
ν(BH)
ν(CO)
δ(HOH)
4000
3000
2000
1000
Wavenumbers (cm-1)

(b)



(c)

Figure S1. IR spectra of compounds (**a**) **1**, (**b**) **2**·3H_2O, and (**c**) **3**.

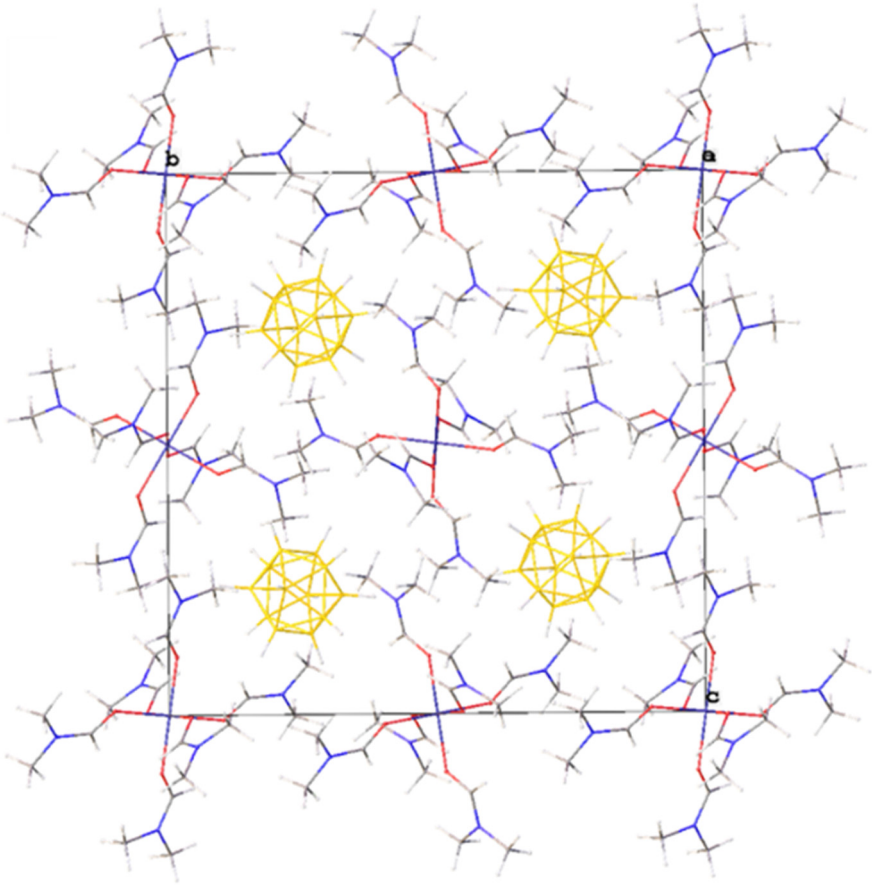
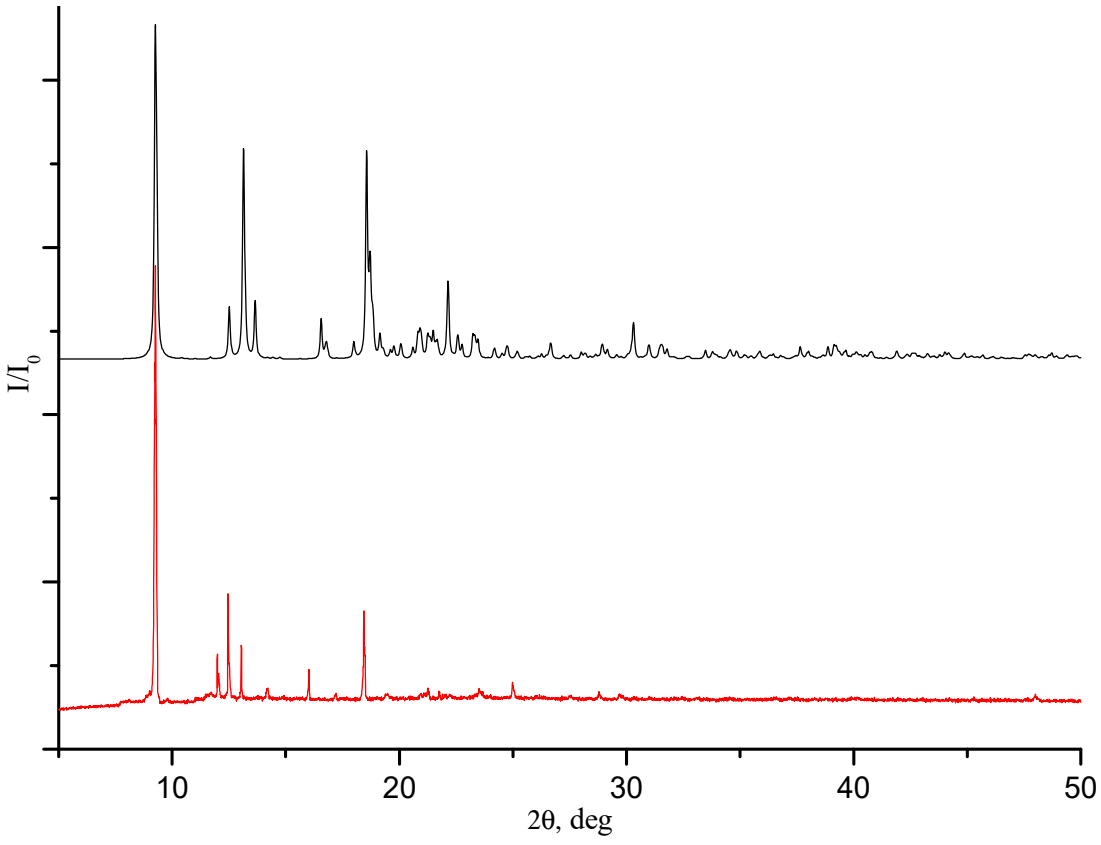
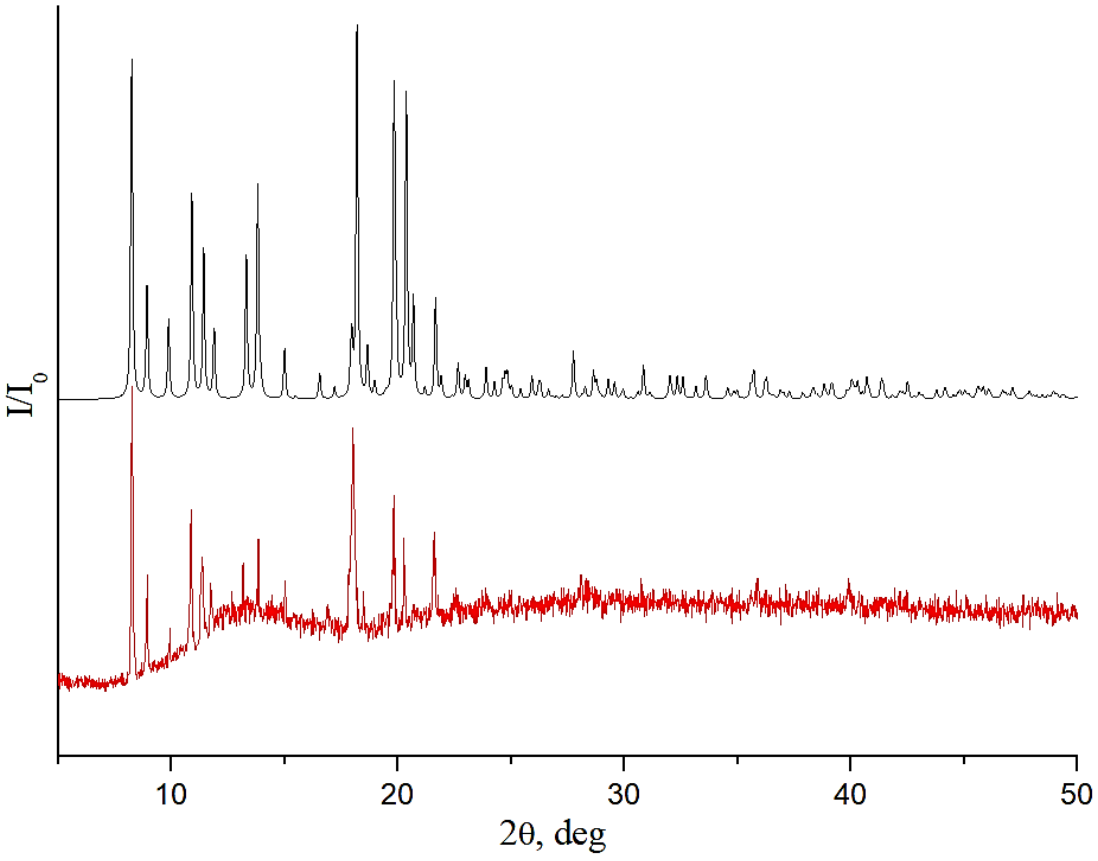


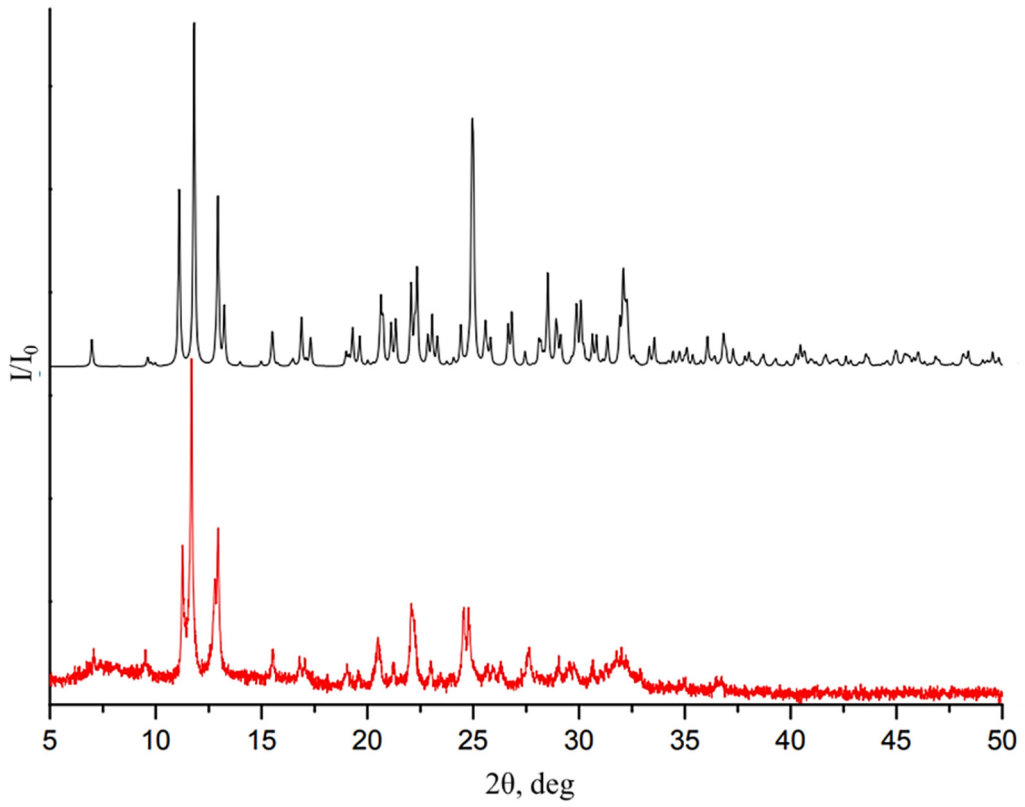
Figure S2. Projection of structure of complex **1** along the *a* axis.



(a)



(b)



(c)

Figure S3. Calculated (black) and experimental (red) X-ray powder diffraction patterns for (**a**) compound **1**, (**b**) compound **2**, and (**c**) compound **3**.

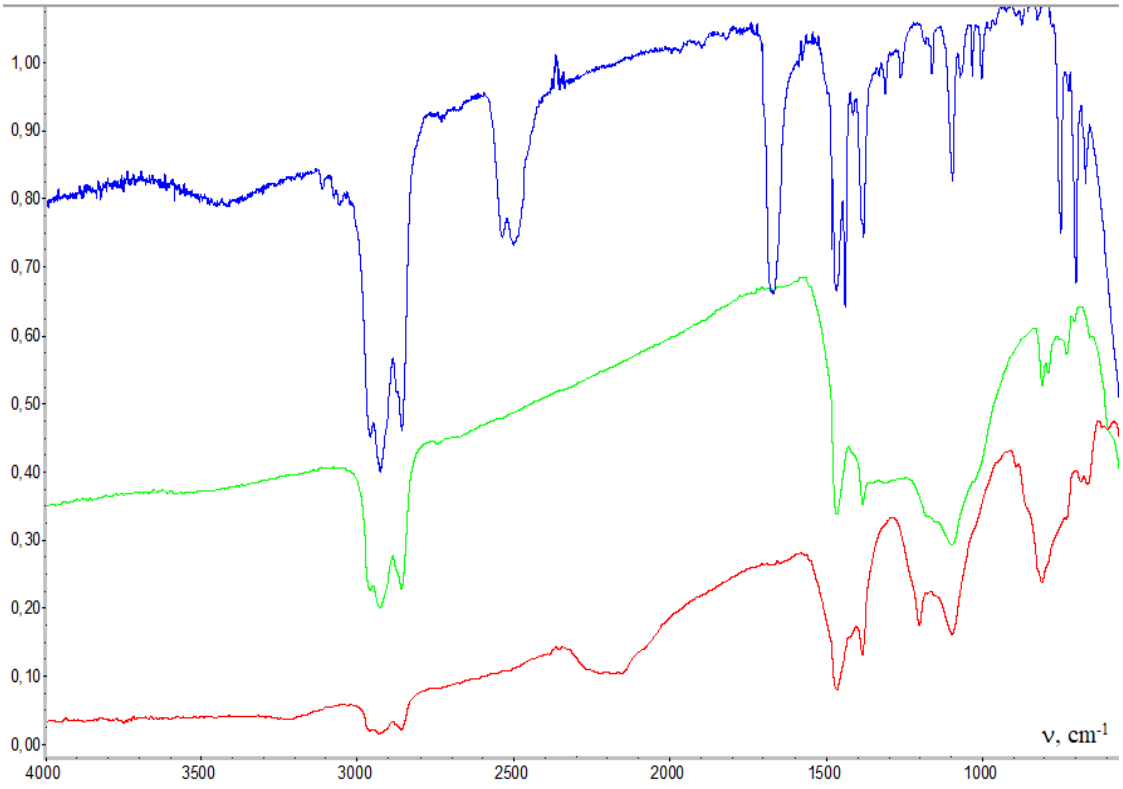


Figure S4. IR spectra of complex **2**·*n*H_2O (blue), sample **2a** (green) and amorphous boron (red).

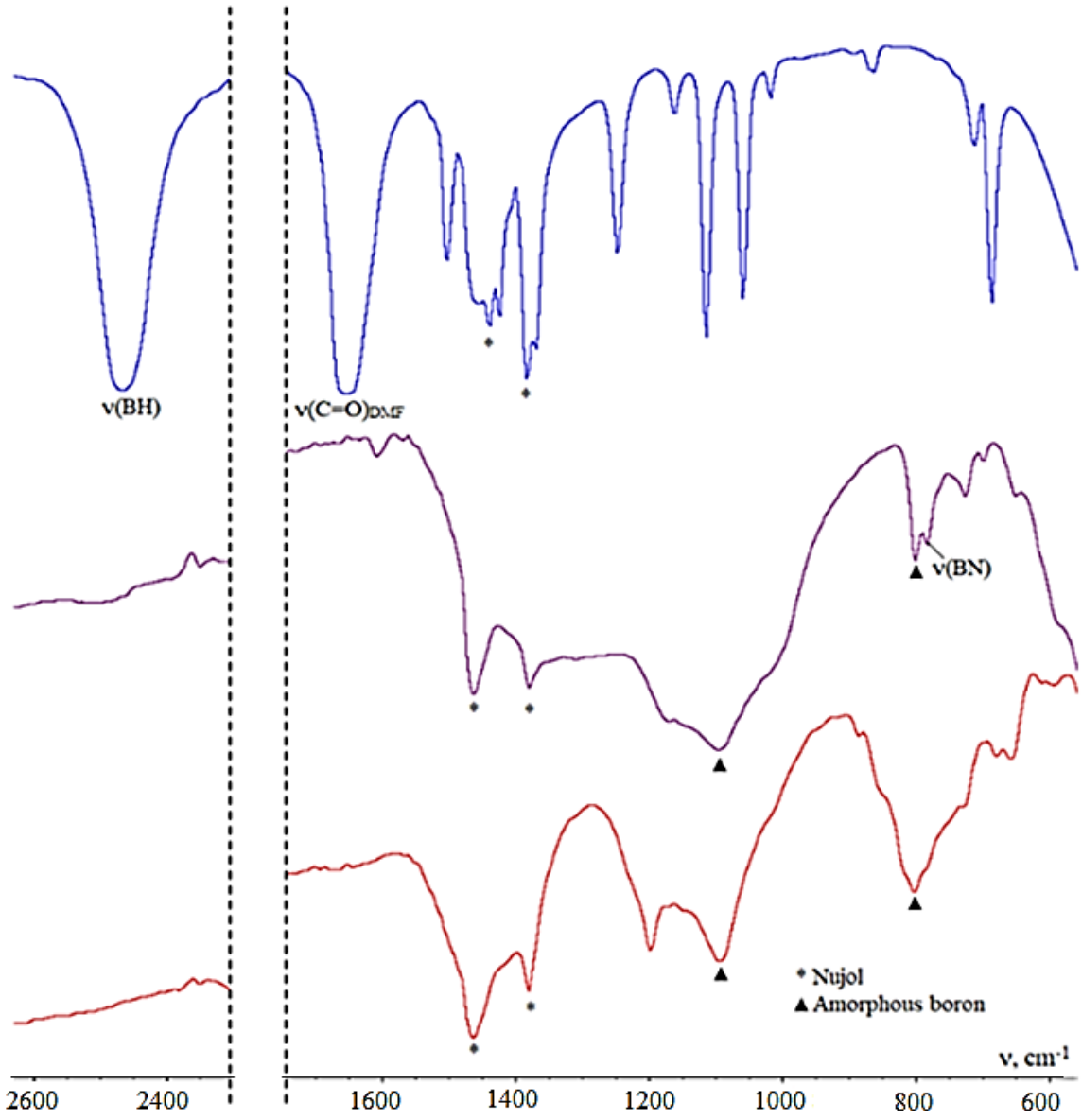


Figure S5. Fragments of IR spectra of complex **1** (blue), sample **1a** (purple) and amorphous boron (red).

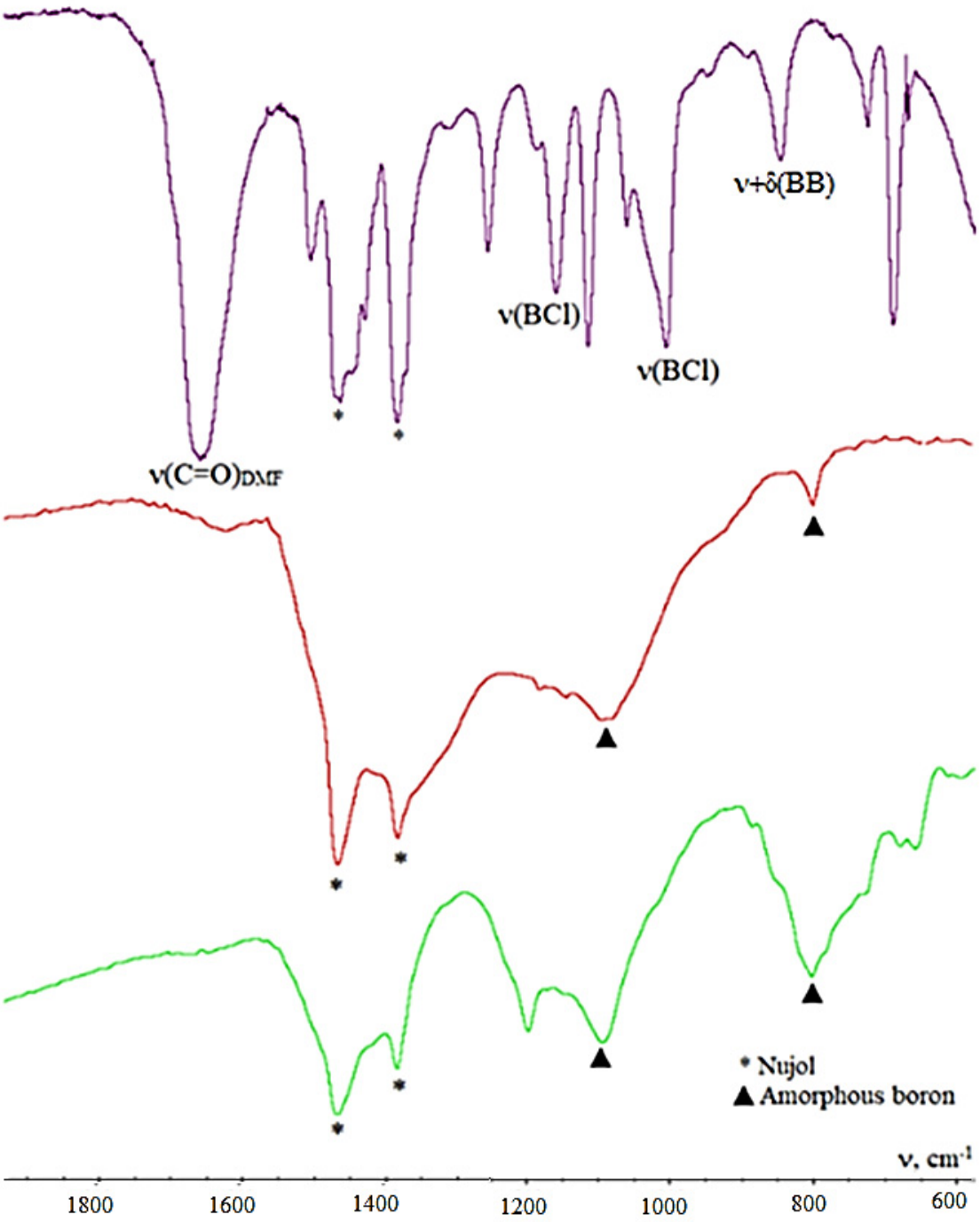


Figure S6. Fragments of IR spectra of complex **3** (purple), sample **3a** (red) and amorphous boron (green).

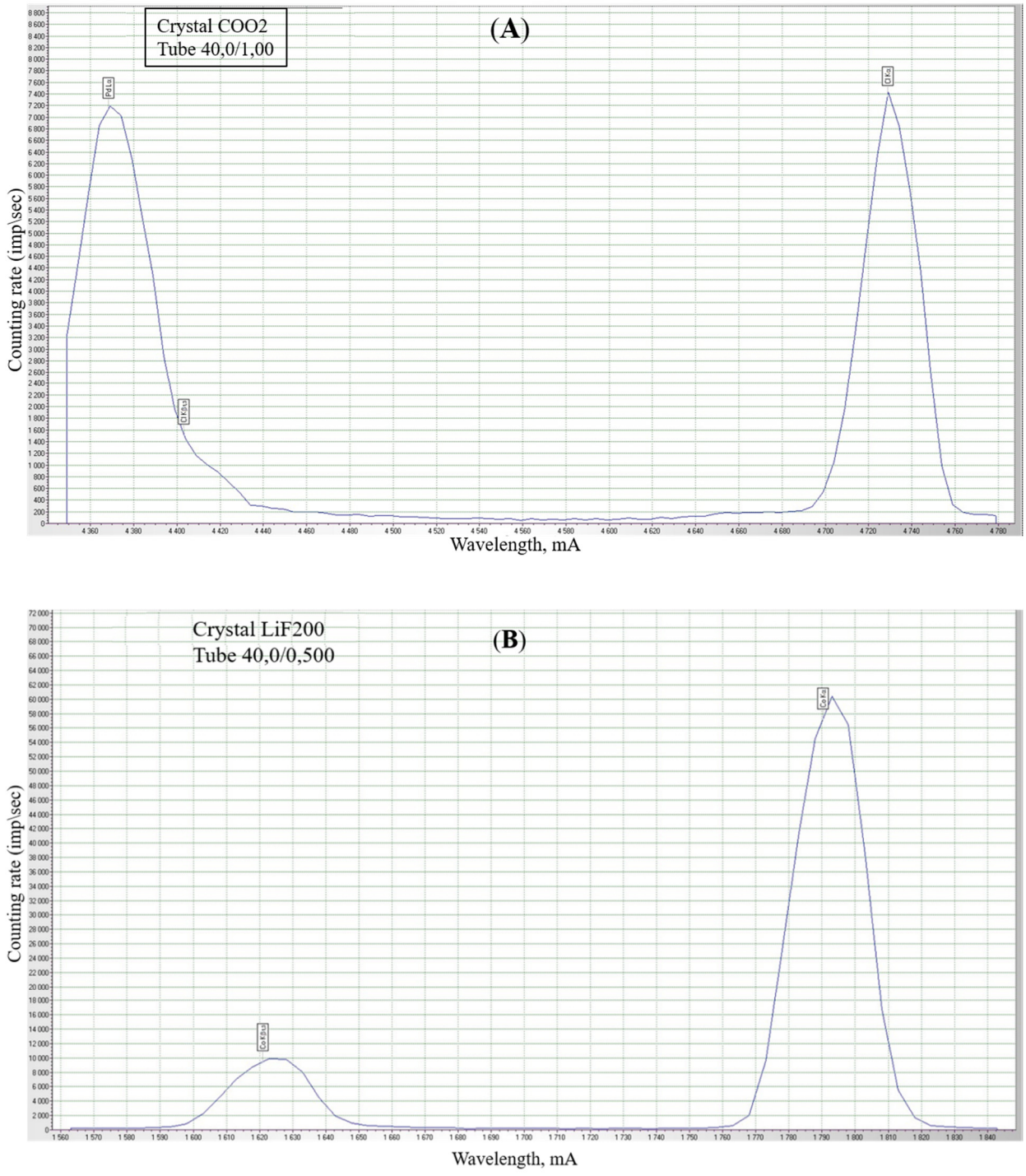


Figure S7. X-ray spectral fluorescence spectrum of sample **3a**: (**A**) determination of Cl, (**B**) determination of Co.