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Rapid Communication

Hydrothermal synthesis and structures of two novel chain-like heteropolymolybdate formed by Keggin cluster units☆

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Abstract

Two novel heteropolymolybdate, $[H_2bpy]_2$ $[Hbpy]$ $[PCuMo_{11}O_{39}] \cdot H_2O$ **1** and $[H_2bpy]_2$ $[Hbpy]$ $[PZnMo_{11}O_{39}] \cdot 2.75H_2O$ **2**, have been prepared under mild hydrothermal conditions and structurally characterized by single-crystal X-ray diffraction. The two compounds are isostructural and crystallize in the monoclinic space group $P2(1)/n$ (No. 14) with $a = 13.440(3)$ Å, $b = 26.660(5)$ Å, $c = 15.240(3)$ Å, $\beta = 99.55(3)°$, and $Z = 4$ for the compound **1**, and $a = 13.610(3)$ Å, $b = 26.781(5)$ Å, $c = 15.205(3)$ Å, $\beta = 100.40(3)°$, and $Z = 4$ for the compound **2**. Compound **1** and **2** exhibit a zigzag chain structure in which Keggin anions are connected through a common oxygen atom. They are the first characterized compounds containing 1D chains of transition-metal substituted Keggin heteropolymolybdate. Other characterizations by elemental analysis, IR, EPR, TG, and XRPD are also described.

Keywords: Hydrothermal synthesis; Heteropolymolybdate; Keggin cluster

1. Introduction

The self-assembly of organic and inorganic molecular building blocks into low-dimensional as well as three-dimensional structures is of great interest as a route to new microporous compound with potentially interesting intracrystalline reactivity and sorption properties [1–3]. During the past decade, a number of novel structures have been successfully designed and synthesized through judicious combination of various molecular building blocks and inorganic or organic linkers [4–9]. Polyoxometalates (POMs), as one kind of significant metal oxide clusters with nanosizes and abundant topologies, have recently been employed as inorganic building blocks for the design and prepared polyoxometalate based composition materials [10–22]. Moreover, several successful strategies have been developed to design materials constructed of POM building blocks. Pope's group has reported one-dimensional POMs with lanthanide or actinide cations as assembling groups [23]. Müller's group has synthesized several giant mixed-valence polyoxomolybdates constructed of $\{Mo_8\}$ building blocks with cyclic Mo_{176} [24], icosaderal Mo_{132} [25], capped cyclic Mo_{248} [26] and "basket" Mo_{116} [27] architectures. Zubieta and co-workers have employed the transition metal coordination complexes as inorganic bridging ligands linking polyoxomolybdate clusters into one-, two- and three-dimensional networks [28–30], and Khan and co-workers have produced a series of novel three-dimensional framework materials based on well-defined polyoxovanadate building blocks-$\{V_{18}O_{42}(XO_4)\}$ (X=S, V, Cl) [31–33].

Transition metal substituted polyoxometalates based on Keggin framework have received considerable attention for their catalytic properties in organic oxidation [34,35]. Although these transition metal substituted Keggin deviations are generally reported as discrete entities, several literatures have shown that they are capable of acting as inorganic building

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blocks to form one-dimensional chains through assembly, such as in compounds $(ET)_8[PMnW_{11}O_{39}]\cdot 2H_2O$ (ET = bis (ethylenedithio) tetrathiofulvalene) [36], $[NEt_3H]_5[XCoW_{11}O_{39}]\cdot 3H_2O$ (X = P, As) [37], and $[Co(dpa)_2(OH_2)_2]_2$ [Hdpa] $[PCoW_{11}O_{39}]$ (dpa = di—2-pyridylamine) [38]. However, all of these 1D chain-like compounds are constructed of transition metal substituted Keggin heteropolytungstate, and no chain-like transition metal substituted Keggin heteropolymolybdate has been reported.

Here, we reported the synthesis and structures of the first characterized compounds containing 1D chains of transition metal substituted Keggin heteropolymolybdate: $[H_2bpy]_2$ [Hbpy] $[PCuMo_{11}O_{39}]\cdot H_2O$ **1**, $[H_2bpy]_2$ [Hbpy] $[PZnMo_{11}O_{39}]\cdot 2.75H_2O$ **2** (bpy = 4, 4′-bipyridine). Compounds **1** and **2** show a novel zigzag chain structure, which is different to the straight chain structure in the reported 1D transition metal substituted Keggin heteropolytungstate. Furthermore, the zigzag chains of compounds **1** and **2** form layers through short inter-species contact and these layers are stacked parallel and form a three-dimensional structure with dumbbell-like 1D channel.

2. Experimental

2.1. Synthesis and characterization

Synthesis of $[H_2bipy]_2$ [Hbipy] $[PCuMo_{11}O_{39}]\cdot H_2O$, **1**. A mixture of $Na_2MoO_4\cdot 2H_2O$ (0.27 g), H_3PO_4 (0.195 g), $CuCl_2\cdot 2H_2O$ (0.095 g), 4, 4′-bipy (0.08 g) and H_2O (10 mL) was stirred for 20 min in air. The mixture was then transferred to a Teflon-lined autoclave (20 mL) and kept at 160°C for 6 days. After slow cooling to room temperature, yellow crystals were filtered off, washed with distilled water and dried in a desiccator at room temperature to give a yield of 60% based on Mo. The ICP analysis showed that compound **1** contained 46.2% Mo, 2.6% Cu and 1.2% P (calcd: Mo, 46.6%; Cu, 2.8%; P, 1.4%). The elemental analysis found: C, 15.6%; H, 1.6%; N, 3.6% (calcd: C, 15.9%; H, 1.4%; N, 3.7%).

Synthesis of $[H_2bipy]_2$ [Hbipy] $[PZnMo_{11}O_{39}]\cdot 2.75H_2O$, **2**. A mixture of $Na_2MoO_4\cdot 2H_2O$ (0.27 g), H_3PO_4 (0.195 g), $ZnCl_2$ (0.075 g), 4, 4′-bipy (0.08 g) and H_2O (10 mL) was stirred for 20 min in air. The mixture was then transferred to a Teflon-lined autoclave (20 mL) and kept at 160°C for 6 days. After slow cooling to room temperature, green crystals were filtered off, washed with distilled water and dried in a desiccator at room temperature to give a yield of 50% based on Mo. The ICP analysis showed that compound **2** contained 45.6% Mo, 2.6% Zn and 1.1% P (calcd: Mo, 45.9%; Zn, 2.8%; P, 1.3%). The elemental analysis found: C, 15.5%; H, 1.8%; N, 3.5% (calcd: C, 15.7%; H, 1.5%; N, 3.7%).

Elemental analyses (C, H and N) were performed on a Perkin-Elmer 2400 CHN Elemental Analyzer. Mo, Cu, Zn and P were determined by a Leeman inductively coupled plasma (ICP) spectrometer. The infrared spectrum was obtained on an Alpha Centaurt FT/IR spectrometer with pressed KBr pellet in the 4000–400 cm^{-1} region. EPR spectrum was recorded on a Brucker ER 200D spectrometer at room temperature. A Perkin-Elmer TGA7 thermogravimetric analyzer was used to obtain TGA curve in N_2 with a temperature increasing rate of 10°C min^{-1}. X-ray powder diffraction (XRPD) patterns were recorded on a Siemens D5005 diffractometer with Cu$K\alpha$ ($\lambda = 1.5418$ Å) radiation.

2.2. Single-crystal X-ray diffraction

A yellow single crystal of **1** and a green single crystal of **2** were carefully selected under a polarizing microscope and glued at the tip of a thin glass fiber with cyanoacrylate (super glue) adhesive respectively. Single-crystal structure determination by X-ray diffraction was performed on a R-axis RAPID IP diffractometer equipped with a normal focus, 18 kW sealed tube X-ray source (Mo$K\alpha$ radiation, $\lambda = 0.71073$ Å) operating at 50 kV and 200 mA. Data processing was accomplished with the RAXWISH processing program. Empirical absorption correction was applied. The structure was solved by the direct method and refined by full-matrix least squares on F^2 using the SHELXL 97 software [39]. All of the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were located from difference Fourier maps. Further details of the X-ray structural analysis are given in Table 1. The atomic positional and displacement parameters, bond lengths and angles for compounds 1 and 2 are listed in the tables of Supplementary materials.

3. Results and discussions

3.1. Description of structure

The chemistry of Keggin polyoxoanion has been studied for many years and is in general quite well-known [40]. It consists of four M_3O_{13} groups (M = Mo, W) surrounding a central heteroatom in a tetrahedral cavity. Each M_3O_{13} group is formed by three octahedral sharing edges and having a common oxygen atom which is also shared with the central heteroatom. By removing one, two, or three M atoms in the Keggin structure it is possible to obtain the mono-, di-, and trivacant Keggin polyoxometalates. These lacunary species easily incorporate first-row transition metals resulting in mono-, di- and trisubstituted Keggin polyoxoanions. Although

Table 1
Crystal data and structure refinement for **1** and **2**

	1	**2**
Empirical formula	$C_{30}H_{31}CuMo_{11}N_6O_{40}P$	$C_{30}H_{34.50}ZnMo_{11}N_6O_{41.75}P$
F_w	2265.45	2298.82
Crystal system	Monoclinic	Monoclinic
Space group	$P2(1)/n$	$P2(1)/n$
a (Å)	13.440(3)	13.610(3)
b (Å)	26.660(5)	26.781(5)
c (Å)	15.240(3)	15.205(3)
β (deg)	99.55(3)	100.40(3)
V (Å^3)	5385.0(19)	5451.0(19)
Z	4	4
D_{calcd} (g/cm^3)	2.793	2.801
μ (mm^{-1})	2.999	3.017
T (K)	293(2)	293(2)
λ (Å)	0.71073	0.71073
Final R_1, wR_2[$I>2\sigma(I)$]	0.0664, 0.1304	0.0751, 0.1088
Final R_1, wR_2 (all data)	0.1331, 0.1449	0.1352, 0.1248

Note. $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$; w$R_2 = \sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]^{1/2}$.

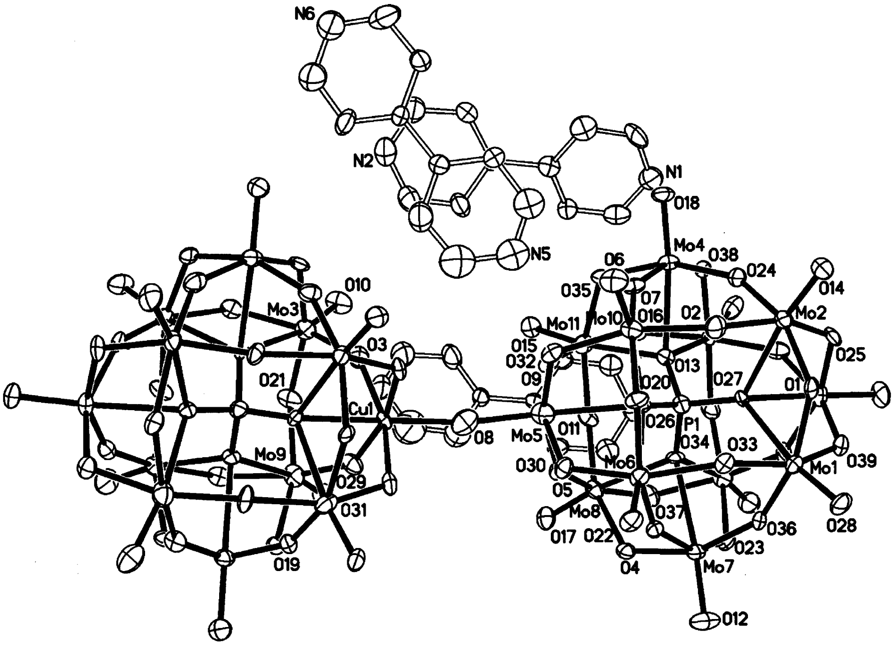

Fig. 1. ORTEP drawing of **1** showing the labeling of atoms with thermal ellipsoids at 50% probability. Hydrogen atoms are omitted for clarity.

most of them show discrete structures, some monosubstituted Keggin polyoxoanions can be polymerized into dimer [41], trimer [42] and chain-like polymer [36–38].

Compounds **1** and **2** are novel examples of chain-like polymer of monosubstituted Keggin polyoxoanions. The structure of **1** contains Keggin anions $[PCuMo_{11}O_{39}]^{5-}$, H_2bipy^{2+}, $Hbipy^{+}$ cations and water molecules (Fig. 1). In compound **1**, Mo–O distances were 1.682(8) Å for terminal oxygen (Mo–Ot), 1.917(8) Å for O bonded to two Mo atoms (corner-sharing Mo–Oc and edging-sharing Mo–Oe), 2.421(7) Å for O bonded to three Mo atoms and one P atoms (Mo–Oi), 1.859(8) Å for O bonded to one Cu atom and one Mo atom. P–O distances varied between 1.514(8) and 1.541(9) Å, average 1.527(9) Å. The bond lengths of monosubstituted Cu(1) are quite different from those of Mo atoms. Five of the Cu(1)–O lengths are in the range of 1.905(8)–2.004(9) Å. The sixth corresponding to Mo–Oi is very long (2.367(8) Å).

The Keggin anions are connected through a common oxygen atom to give a one-dimensional chain as shown in Fig. 2. This bridging oxygen atom connects two interval positions of the Keggin unit occupied by Cu(II) and Mo(IV), rather than two opposite position as reported in compounds $(ET)_8[PMnW_{11}O_{39}]\cdot 2H_2O$ [22],

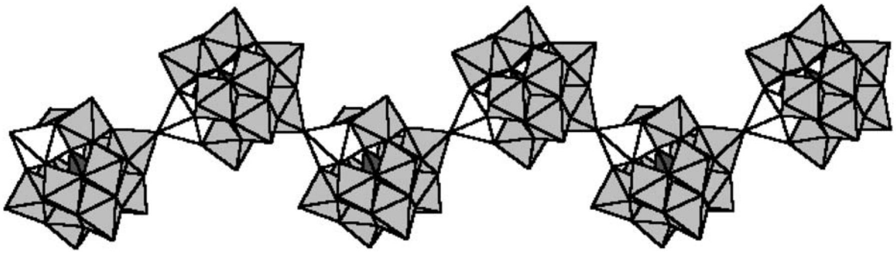

Fig. 2. A polyhedral view of the 1D chain in **1**. The white octahedra are CuO_6, light gray octahedra are MoO_6 and dark gray tetrahedra are PO_4.

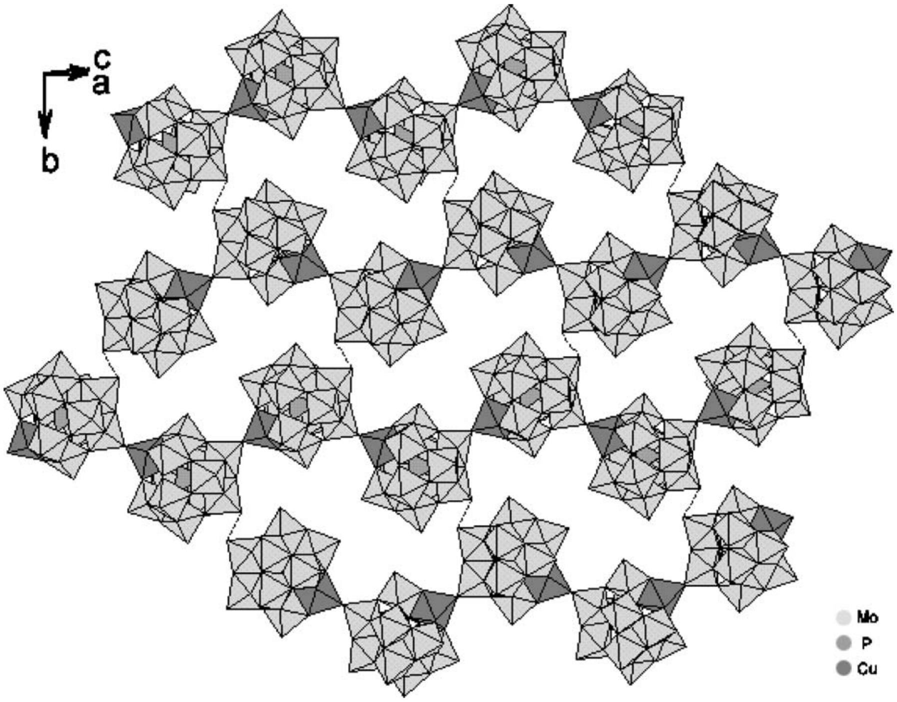

Fig. 3. View of the 2D layer in **1**. All C, H and N atoms are omitted for clarity.

$[NEt_3H]_5[XCoW_{11}O_{39}] \cdot 3H_2O$ ($X = P$, As) [23] and $[Co(dpa)_2(OH_2)_2]_2[Hdpa]$ $[PCoW_{11}O_{39}]$ [24]. Owing to the difference in connecting site, zigzag chain is formed in compound **1**, while straight chain is shown in previously reported compound $(ET)_8[PMnW_{11}O_{39}] \cdot 2H_2O$ [36], $[NEt_3H]_5[XCoW_{11}O_{39}] \cdot 3H_2O$ ($X = P$, As) [37] and $[Co(dpa)_2\ (OH_2)_2]_2$ $[Hdpa]$ $[PCoW_{11}O_{39}]$ [38].

The zigzag chains of compound **1** form layers through short inter-species contact O22⋯O22 (at $-x, 1-y, -z$) 2.819 Å as shown in Fig. 3. Each $[PCuMo_{11}O_{39}]^{5-}$ unit joins with three adjacent $[PCuMo_{11}O_{39}]^{5-}$ units through covalent bond and short inter-species contact respectively, thus resulting in dumbbell-liked voids circumscribed by six $[PCuMo_{11}O_{39}]^{5-}$ units. All such networks are piled in parallel to constitute a three-dimensional crystal structure. The voids left by stacking the layers form 1D channels in the direction perpendicular to the layers with dimensions of $18.867 \times 0.861\,Å^2$ (Fig. 4).

There are 4, 4′-bipy cations and crystal water molecule situated in the dumbbell-like channels. According to the consideration of the change balance, the monoprotonated $Hbipy^+$ and diprotonated H_2bipy^{2+} co-exist in compound **1**, which is a common feature in many reported literatures [43,44]. These cations, polyanions and crystal water molecules are contacted with each other by N–H(*bpy*)⋯N(*bpy*), N–H(*bpy*)⋯O(polyanion), N–H(*bpy*)⋯O(crystal water) and O(crystal water)⋯O(polyanion) hydrogen bonds with distances of 2.632–3.045 Å as listed in Table 2.

Compound **2** is isostructural with **1**, consequently, the structure description will not be repeated here.

3.2. Characterization

The experimental and simulated XRPD patterns of compounds **1** and **2** are shown in Fig. S1. Their peak positions are in good agreement with each other, indicating the phase purity of the products. The differences in intensity may be due to the preferred orientation of the powder samples.

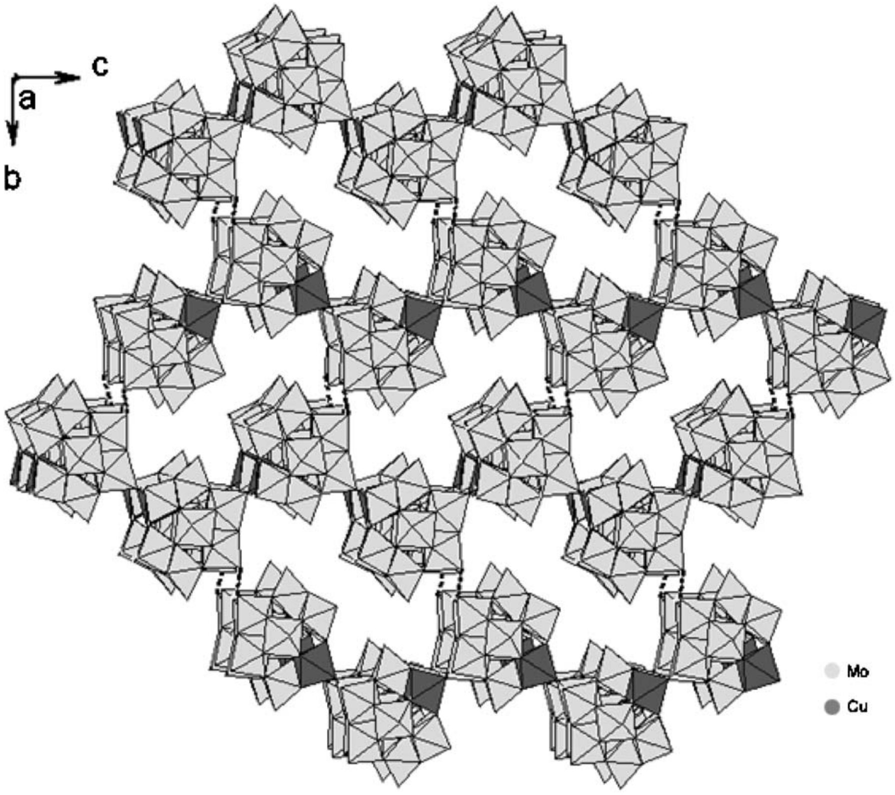

Fig. 4. Packing of the 2D layers showing the formation of 1D channel in **1**. All C, H and N atoms are omitted for clarity.

Table 2
Hydrogen Bonds (Å and deg) in the compound **1**

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠(DHA)
N(1)–H(1B)···O32#1	0.89	2.086	2.926	165.71
N(1)–H(1B)···O20#1	0.89	2.715	3.045	104.44
N(2)–H(2B)···N4#2	0.89	1.900	2.760	178.59
N(3)–H(3A)···O(1W)	0.89	1.782	2.631	168.47
N(4)–H(4B)···N2#2	0.89	1.918	2.760	165.73
N(6)–H(6B)···O1#3	0.89	2.408	2.817	109.70
N(6)–H(6B)···O2#3	0.89	2.321	3.038	141.00
N(6)–H(6B)···O33#3	0.89	2.285	2.916	130.37
O(1W)···O17#1			2.856	
O(1W)···O35#4			2.980	

Symmetry transformations used to generate equivalent atoms. #1: $x+1/2,\ -y+1/2,\ z+1/2$; #2: $-x+1, -y+1, -z+1$; #3: $x+1,\ y, z$; #4: $-x+1/2, y+1/2,\ -z+3/2$.

The IR spectra of compound **1** and **2** exhibit a series of bands in the 1200–1500 cm^{-1} region associated with the 4,4′-bipyridine; the compounds also possess strong bands in the 1000–1100 cm^{-1} region attributed to ν(P–O) and a band at 942 (**1**) and 939 (**2**) attributed to $\nu(\mathrm{Mo{-}O_t})$ (Fig. S2). The EPR spectrum of **1** at room temperature shows a Cu^{2+} signal with $g_{\perp} = 2.289$, $g_{\parallel} = 2.131$ (Fig. S3).

The thermal analysis of **1** gives a total loss of 23.0% in the range of 50–540°C, which agrees with the calculated weight loss of 21.7%. The weight loss of 1.0% at 50–200°C is attributed to the loss of physical water in the crystal (calc. 0.8%). The weight loss of 22.0% at 230–540°C corresponds to the loss of 4, 4′-bipy (calc. 20.9%). For compound **2**, mass losses at 50–200°C (2.3%) and 240–550°C (21.7%) are attributed to the release of physical water in the crystal (calc. 2.2%) and the loss of 4,4′-bipy (20.6%) (Fig. S4).

4. Conclusions

In summary, we have prepared two novel chain-like heteropolymolybdate formed by Keggin cluster units. Compounds **1** and **2** show a zigzag chain structure, which is different from the straight chain structure formed in one-dimensional transition metal substituted Keggin heterotungstate. This work shows that the hydrothermal technique is a powerful method for the synthesis of novel structural polyoxometalate compounds.

5. Supplementary materials

Crystallographic data (excluding structure factors) for the structure(s) reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-201814 and 201815. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (44) 1223 336-033; e-mail: deposit@ccdc.cam.ac.uk).

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