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Reactivity of $Co_4(CO)_{12}$ with polydentate ligands. Fragmentation kinetic studies of the substituted derivatives

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Abstract

The reaction of $Co_4(CO)_{12}$ with the polydentate phosphines Ph_2Ppy (2-(diphenylphosphino)pyridine), *t*-dppv (*trans*-1,2-vinylenebis(diphenylphosphine) and dpmp (bis((diphenylphosphino)methyl)phenylphosphine) yield the tetrasubstituted $Co_4(CO)_8L_4$ complexes (L = Ph_2Ppy and *t*-dppv) in which the ligand behaves as monodentate, trisubstituted $Co_4(CO)_9$(dpmp) complex in which the ligand behaves as tridentate, and pentasubstituted $Co_4(CO_7$(dpmp)$_2$ in which the ligands behave as di- and tridentate. All the compounds have been characterized by elemental analysis and IR, ^1H and ^{31}P NMR spectroscopy. The kinetics of fragmentation in DCE (dichloroethane) of $Co_4(CO)_8(PPh_2Py)_4$ to form $Co_2(CO)_6(Ph_2Ppy)_2$ has been studied under a N_2 or CO atmosphere. The rate constants are independent of the concentration of Ph_2Ppy and are lower under CO. This suggests that the reaction proceeds by a CO-dissociative (D) mechanism. The complexes $Co_4(CO)_8$(*t*-dppv)$_4$, $Co_4(CO)_9$(dpmp) and $Co_4(CO)_7$(dpmp)$_2$ are stable towards fragmentation.

Introduction

Reactions of metal carbonyl clusters often involve scission of one (or more) metal–metal bond leading to fragmentation of the cluster. Metal carbonyl clusters containing polydentate ligands are of interest since the bridging ligands may be capable of stabilizing the clusters against fragmentation under a variety of conditions under which the unsubstituted cluster undergoes fragmentation [1–4]. Although the kinetic behavior of the $M_3(CO)_{12}$ triad of clusters (M = Fe, Ru, Os) has been thoroughly studied [5] the same cannot be said of the series $M_4(CO)_{12}$ (M = Co, Rh, Ir) for which, until very recently, quantitative data were available only for CO exchange and fragmentation reactions of $Co_4(CO)_{12}$ [6,7] and substitution reactions of $Rh_4(CO)_{12}$ [8] and $Ir_4(CO)_{12}$ [9]. We report here an investigation of the substitution reactions of $Co_4(CO)_{12}$ with potentially polydentate phosphines such as Ph_2Ppy, *t*-dppv and dpmp and a study of the kinetics of the fragmentation reactions.

Results and discussion

Addition under N_2 of a dichloroethane (DCE) solution of L (L = Ph_2Ppy or *t*-dppv) to a solution of $Co_4(CO)_{12}$ in the same solvent results in loss of CO to give the tetrasubstituted products; the monosubstituted complexes were only observable during the reaction.

The nature of the product in DCE was further examined by recording the IR spectra of solutions of $Co_4(CO)_{12}$ which had been allowed to react to completion with various amounts of Ph_2Ppy or *t*-dppv. The intensity of the bands due to $Co_4(CO)_{11}L$ grew to a maximum with increasing concentration of L and then decreased as bands due to the final product grew. As is shown in Fig. 1, for the Ph_2Ppy, slightly more than four times as much ligand was required to reduce the intensity of the $Co_4(CO)_{11}L$ bands to zero, than was required to form $Co_4(CO)_{11}L$ without the presence appreciable bands due to $Co_4(CO)_{12}$ or the final product. The spectra of the solutions remained unchanged in the presence of larger amounts of L at 25 °C. However, for L = Ph_2Ppy, as the temperature was raised to $\gtrsim 35\,°C$ further spectroscopic changes occurred and eventually a orange precipitate was formed. After the precipitate was separated and washed with hexane its IR spectrum showed two strong bands at 1973 and 1950 cm^{-1} characteristic of $Co_2(CO)_6(Ph_2Ppy)_2$ [10].

Addition of DCE solution of dpmp to a solution of $Co_4(CO)_{12}$ under N_2 in the same solvent resulted in loss of CO to give $Co_4(CO)_9(dpmp)$ and $Co_4(CO)_7(dpmp)_2$ when the reactant ratio was 1 : 1 or 1 : 2 respectively.

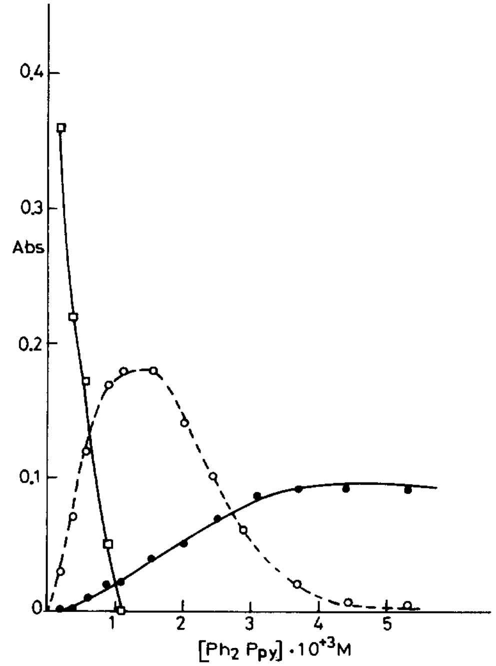
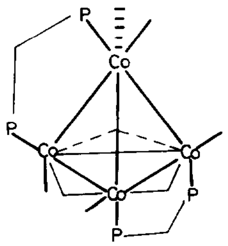


Fig. 1. Dependence on $[Ph_2Ppy]$ of the absorbance of IR bands at 2050 cm^{-1} (□) due to $Co_4(CO)_{12}$, 2021 cm^{-1} (○) due to $Co_4(CO)_{11}(Ph_2Ppy)$ and 1976 cm^{-1} (●) due to $Co_4(CO)_8(Ph_2PPy)_4$ after reaction of ca. 1×10^{-3} *M* solutions of $Co_4(CO)_{12}$ in DCE with Ph_2Ppy.

Table 1

IR spectral data for complexes (relative peak heights are shown in parentheses)

Complex	Solvent	$\nu(CO)_t$ (cm^{-1})				$\nu(CO)_{sb}$ (cm^{-1})		$\nu(CO)_b$ (cm^{-1})		
$Co_4(CO)_9(dpmp)$	Nujol	2046 sh	2037 vs	1994 vs	1975 vs	1961 s	–	1827 s	1783 s	
	$CHCl_3$	2053 (10)		2006 (8.2)		1977 (2.8)	–	1828 (0.5)	1784 (2.8)	
	CH_2Cl_2	2052 (10)		2004 (8.9)		1974 (2.9)	–	1828 (0.6)	1785 (3.1)	
	DCE	2050 (10)		2002 (9.5)		1970 (3.0)	–	1827 (0.7)	1787 (3.5)	
$Co_4(CO)_8(Ph_2Ppy)_4$	Nujol	2010 s	1980 vs	1950 vs	1931 sh		1886 m	1817 m	1783 s	1763 s
	$CHCl_3$	2011 (3.5)	1975 (6.6)	1954 (10)	1926 sh		1887 (2.9)		1786 (3.2)(br)	
	CH_2Cl_2	2014 (2.5)	1975 (4.0)	1954 (10)			1889 (9.1)		1768 (3.3)(br)	
	DCE	2015 (2.2)	(178 (4.3)	1942 (10)			1888 (12.3)		1769 (3.2)	
$Co_4(CO)_8(t\text{-dppv})_4$	Nujol	2008 s	1984 sh	1957 vs	1915 s		1883 vs			
	$CHCl_3$	2012 (3.6)	1991 sh	1963 (10)	1916 (5.7)		1890 (6.9)			
	CH_2Cl_2	2012 (3.1)	1990 (3.1)	1963 (10)	1917 (4.15)		1883 (6.7)			
	DCE	2011 (3.0)	1990 (3.1)	1963 (10)	1918 (4.0)		1888 (6.5)			
$Co_4(CO)_7(dpmp)_2$	Nujol	1991 s	1953 vs	1941 sh	1930 sh		–	1780 sh	1757 s	1714 s
	$CHCl_3$	1997 (10)	1968(10)	1956 (5.0)	1930 sh		–	1797 (0.6)	1765 (3.4)	1723 (2.5)
	CH_2Cl_2	1996 (10)	1963 (10)	1953 (5.0)	1928 sh		–	1797 (0.5)	1765 (3.5)	1723 (2.5)
	DCE	1995 (10)	1959 (9.9)	1948 (2.7)	1926 sh		–	1797 (0.9)	1765 (3.8)	1724 (2.6)



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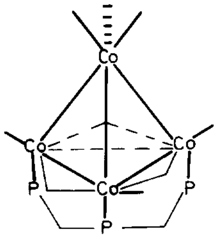

II

All the compounds were characterized by elemental analysis and IR, ^{1}H and ^{31}P NMR spectroscopy. Table 1 lists the carbonyl stretches of the substituted products.

The IR spectra (Table 1) of $Co_4(CO)_8L_4$ (L = Ph_2Ppy and *t*-dppv), $Co_4(CO)_9$(dpmp) and $Co_4(CO)_7(dpmp)_2$ are very similar to those of $Co_4(CO)_8(PPh_3)_4$ [2], $Co_4(CO)_9(HC(PPh_2)_3)$, and $Co_4(CO)_7[HC(PPh_2)_3](PMe_3)_2$ [11], respectively, and are consistent with the structures shown schematically in I and II. These are the structures adopted by $Rh_4(CO)_8(DPM)_2$ [12] and $Co_4(CO)_9(HC(PPh_2)_3)$ [11], respectively in the solid state. The shifts of CO to lower frequencies for the new compounds are consistent with the degree of substitution.

The main features of the IR spectra of $Co_4(CO)_8(Ph_2Ppy)_4$ and $Co_4(CO)_8$(*t*-dppv)$_4$ that distinguish them from the other $Co_4(CO)_9$(dpmp) and $Co_4(CO)_9(dpmp)_2$ complexes are the presence of a band at ca. 1888 cm^{-1}, the weakness of the band at 1817 cm^{-1} in $Co_4(CO)_8(Ph_2Ppy)_4$ complex, and the absence of the band from bridging carbonyls in $Co_4(CO)_8$(*t*-dppv)$_4$. The band at ca. 1888 cm^{-1} can be attributed to semibridging carbonyls [13] and is also seen, as a very weak band, for $Co_4(CO)_{12}$ [14], $Co_4(CO)_8[P(OMe)_3]_4$ and $Co_4(CO)_8(PPh_3)_4$ [2]. The intensity of this band for $Co_4(CO)_8(Ph_2Ppy)_4$ increases as the polarity of the solvent increases. This suggests that the compound $Co_4(CO)_8(Ph_2Ppy)_4$ probably exists in various forms in equilibrium. In solution the isomers with semibridging carbonyls become more preponderant when the solvent is changed from $CHCl_3$ to DCE, while $Co_4(CO)_8$(t-dppv)$_4$ complex probably exist only as a semibridging form.

For all the compounds the bands corresponding to ν(P–C) [15,16] appear in the range 690–672 cm^{-1}. In the case of complex $Co_4(CO)_8(Ph_2Ppy)_4$ the vibrations ν(C=N) [17] (1570 and 1560 cm^{-1}) and δ(py) [18] (619 cm^{-1}) are not shifted toward higher energies with respect to the free ligand, and so Ph_2Ppy must function as a monodentate ligand, with the coordination taking place throught the P-atom, whose base strength is lower than that of the N-atom. This is consistent with cobalt being a class b or soft metal [19].

Table 2

Maxima in the electronic spectra of the complexes in CH_2Cl_2

Complex	λ (cm^{-1})
$Co_4(CO)_8(Ph_2Ppy)_4$	25707
$Co_4(CO)_8$(*t*-dppv)$_4$	26178
$Co_4(CO)_9$(dpmp)	26178
$Co_4(CO)_7(dpmp)_2$	25125
$Co_4(CO)_{12}$	26700

The ^1H and ^{31}P NMR spectra of these clusters are reported in the Experimental section. The usual down-field shift of the ^{31}P resonance is observed upon coordination of the phosphorus to cobalt. For $CO_4(CO)_8(Ph_2Ppy)_4$ one signal is observed in the range for coordinated P. For the complex $CO_4(CO)_8$(*t*-dppv)$_4$ two signals are observed, in the ranges for coordinated P and uncoordinated P*. Finally in the case of the complexes containing dpmp, whereas $Co_4(CO)_9$(dpmp) shows two multiplets in the range for coordinated P, indicating that the three P-atoms are coordinated to the cobalt, for $Co_4(CO)_7(dpmp)_2$ the ^{31}P NMR spectrum indicates that the central P-atom in one of the ligands is uncoordinated.

Electronic spectral data for the complexes in CH_2Cl_2 are shown in Table 2. Each cluster displays an intense low-energy UV absorption at ~ 25100–25710 cm^{-1} and a long absorption tail into the visible. The electronic absorption spectra are similar to those of $Co_4(CO)_{12}$ and $Co_4(CO)_9[HC(PPh_2)_3]$ [20]. For these complexes the

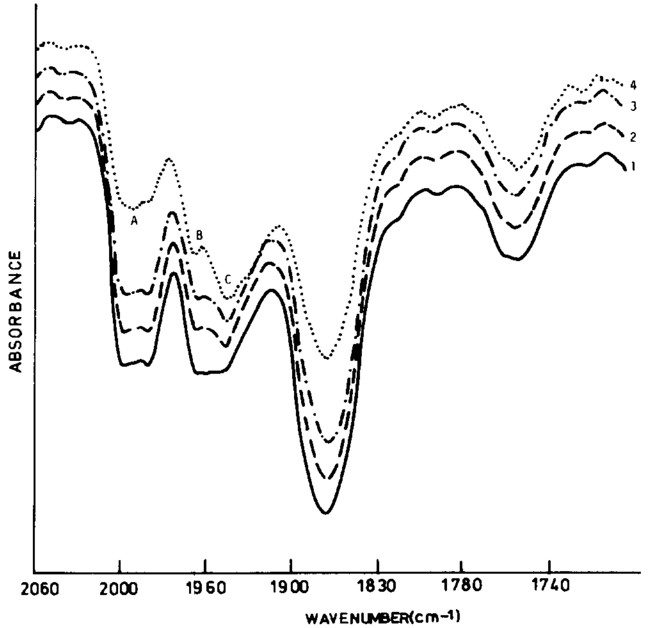

Fig. 2. Infrared spectral changes in the fragmentation reaction $Co_4(CO)_8(Ph_2Ppy)_4 \rightarrow Co_2(CO)_6(Ph_2Ppy)_2$ in DCE at 50 °C. (1) $t = 20$ s; (2) $t = 860$ s; (3) $t = 2673$ s; (4) $t = 6489$ s. Band A assigned to $Co_4(CO)_8(PPh_2Ppy)_4$; Bands B and C assigned to $Co_2(CO)_6(Ph_2Ppy)_2$ [10].

Table 3

Rate constants (in DCE) and activation parameters for the reaction

$Co_4(CO)_8(Ph_2Ppy)_4 \xrightarrow{Ph_2Ppy} Co_2(CO)_6(Ph_2Ppy)_2$

T (°C)	Ratio	$k_{obs}\,10^4$ (s^{-1})	
		N_2 (100%)	CO (100%)
35	1:10	0.8097	
45	1:10	3.8721	
50	1:0	5.2277	
50	1:4	5.3752	0.9742
50	1:10	5.9410	0.9007
50	1:30	5.3521	0.9438
50	1:40	5.8794	
50	1:76	5.5350	
65	1:10	37.8864	

$\Delta H^{\ddagger} = 26.11 \pm 0.09$ kcal·mol^{-1} $\Delta S^{\ddagger} = 5.9 \pm 0.3$ eu.

low-energy UV absorption involves a transition between cobalt–cobalt bonding and antibonding orbitals (σ–$\sigma^{\star}$), and the intense peaks around 42000 cm^{-1} are due to the charge transfer transitions from the metal to the ligand. Phosphine substitution gives rise to a red-shift of the $\sigma \rightarrow \sigma^{\star}$ transition, indicating that coordination of donor ligands increases the electron density in the Co_4 core.

Cluster fragmentation kinetics

Fragmentation of $Co_4(CO)_8(Ph_2Ppy)_4$ leads to $Co_2(CO)_6(Ph_2Ppy)_2$, as shown by the IR changes in the carbonyl region (Fig. 2). The kinetics were studied for reactions under N_2 and CO atmosphere in the presence of varying amounts of Ph_2Ppy (1 : 0–1 : 76). The rate constants (Table 3) are independent of the Ph_2Ppy-concentration and are much lower in the presence of CO. This suggests that a CO-dissociative step is rate determining. The activation parameters were derived by least squares analysis of the dependence of $\log(k/T)$ on $1/T$ (Table 3). The values of $\Delta S^{\ddagger}$ and $\Delta H^{\ddagger}$ are also consistent with a CO dissociative (D) mechanism, and are similar to those for $Co_4(CO)_8(PPh_3)_4$ [2].

$Co_4(CO)_9$(dpmp), $Co_4(CO)_7$(dpmp)$_2$ and $Co_4(CO)_8$(*t*-dppv)$_4$ are very resistant to fragmentation under forcing conditions ($T < 80$ °C for long periods). In the absence of cone angle data for these ligands it is not possible to make generalization about the effects of substituents on the kinetic stability of the Co_4 clusters.

Experimental

All manipulations were carried out by standard Schlenk techniques under oxygen-free N_2. Dichloroethane (DCE), CH_2Cl_2 and $CHCl_3$, were dried over and distilled from P_4O_{10}. Hexane, pentane, and tetrahydrofuran (THF) were dried over and distilled from sodium benzophenone ketyl. Deuteriated chloroform was degassed (3 freeze-pump-thaw cycles) and stored over type 4 Å molecular sieve.

The compound $Co_4(CO)_{12}$ was prepared a by published procedure [2] and characterized by IR spectroscopy.

The reagents Ph_2Ppy [22], *t*-dppv [23] and dpmp [24] were prepared by published procedures and characterized by IR and NMR spectroscopy. The melting points and elemental analyses were in good agreement with the previously reported data.

The cobalt was determined by titration of the Co-EDTA complex in the presence of NET as indicator. The microanalysis were performed by the Microanalytical Laboratory of this Department.

The IR spectra were recorded in the range 4000–200 cm^{-1} on a Nicolet 5DX FT-IR spectrometer using Nujol and hexachlorobutadiene mulls between CsI windows, KBr plates or in CH_2Cl_2, $CHCl_3$ and DCE solution. ^1H and ^{31}P NMR spectra were recorded on a Bruker WH-200-SY instrument. All ^1H NMR chemical shifts are relative to internal TMS, and ^{31}P shifts are relative to extent 85% aqueous H_3PO_4. The visible spectra were recorded on a Pye Unicam SP 8-100 ultraviolet spectrophotometer.

Preparation of $Co_4(CO)_8(Ph_2Ppy)_4$

To a solution of $Co_4(CO)_{12}$ (2 g, 3.50 mmol) in DCE (30 ml) in a nitrogen-filled 100 ml Schlenk flask was added solution of Ph_2Ppy (3.68 g, 13.99 mmol) in the same solvent. The mixture was stirred at room temperature and the progress of the reaction monitored by IR spectroscopy. After ca. 3 h the reaction was complete. The orange-red solid was filtered off and identified as $Co_2(CO)_6(Ph_2Ppy)_2$ [10], and the solvent was removed from the filtrate under vacuum. The residue, of $Co_4(CO)_8(Ph_2Ppy)_4$, was chromatographed under N_2 on a silica gel column (70–230 mesh) with a 1:1 mixture of CH_2Cl_2 and hexane as eluant. The first band was evaporated to dryness under vacuum. Recrystallization of the residual powder from THF/hexane gave brown crystals (0.95 g, 18%). Anal. Found: C, 59.91; H, 3.80; N, 3.61; Co, 15.63. $C_{76}H_{68}N_4Co_4$ calcd.: C, 60.28; H, 3.70; Co, 15.58%. ^1H NMR ($CDCl_3$): δ 6.62 (m, 5-py, 4H); 7.35 (s, br, 40H, C_6H_5); 7.82 (m, 3 and 4-py, 8H); 8.72 (m, 6-py, 4H). ^{31}P NMR ($CDCl_3$): δ 21.1 (s, coordinated P).

*Preparation of $Co_4(CO)_8($*t*-dppv$)_4$*

By a similar procedure brown crystals were obtained from $Co_4(CO)_{12}$ (2 g, 3.50 mmol) and *t*-dppv (5.55 g, 13.99 mmol). The yield was 1.43 g (20%). Anal. Found: C, 65.48; H, 4.42; Co, 11.25. $C_{112}H_{88}O_8P_8Co_4$ calcd.: C, 65.71; H, 4.30; Co, 11.52%. ^1H NMR ($CDCl_3$): δ 7.18 (s, br, 8H, CH=CH); 7.8 (m, 80H, C_6H_5). ^{31}P NMR ($CDCl_3$): δ 22.0 (m, 1P, coordinated P); −5.3 (m, 1P, uncoordinated P^*).

Preparation of $Co_4(CO)_9(dpmp)$

By a similar procedure black crystals were obtained from $Co_4(CO)_{12}$ (1 g, 1.75 mmol) and dpmp (0.88 g, 1.75 mmol). The yield was 1.39 g (80%). Anal. Found: C, 49.60; H, 3.06; Co, 23.63. $C_{41}H_{29}O_9P_3Co_4$ calcd.: C, 49.49, H, 2.92; Co, 23.71%. ^1H NMR ($CDCl_3$): δ 2.86 (m, 2H, CH_2); 3.12 (m, 2H, CH_2); 7.55 (m, 25H, C_6H_5). ^{31}P NMR ($CDCl_3$): δ 26.0 (m, 2P, PPh_2, coordinated P); 29.1 (m, 1P, PPh, coordinated P).

Preparation of $Co_4(CO)_7(dpmp)_2$

(a) By a similar procedure green crystals were obtained from $Co_4(CO)_{12}$ (1 g, 1.75 mmol) and dpmp (1.77 g, 3.50 mmol). The second band was evaporated to dryness under vacuum. Recrystallization of the residual powder from THF/hexane gave green crystals (1.94 g, 77%).

(b) The compound was also prepared directly from the trisubstituted $Co_4(CO)_9$(dpmp) (1 g, 1.01 mmol) and dpmp (0.51 g, 1.01 mmol). The solution was concentrated and then chromatographed. Elution with a mixture of CH_2Cl_2/hexane (1/6) afforded two bands violet and green respectively. The first was changed $Co_4(CO)_9$(dpmp) and the second was the new compound $Co_4(CO)_7(\text{dpmp})_2$. Recrystallization of the residue from THF/hexane gave green crystals (1.24 g, 85%). Anal. Found: C, 58.88; H, 3.97; Co, 16.34. $C_{71}H_{58}O_7P_6Co_4$ calcd.: C, 58.99; H, 4.02; Co, 16.32%. ^{1}H NMR ($CDCl_3$): δ 2.36 (m, 2H, CH_2); 2.83 (m, 2H, CH_2); 3.57 (m, 4H, CH_2); 7.38 (m, 50H, C_6H_5). ^{31}P NMR ($CDCl_3$): δ 22.0 (m, 2P, PPh_2, coordinated P); 26.6 (m, 2P, PPh_2); 30.3 (d, 1P, PPh, coordinated P); −25.5 (m, 1P, PPh, uncoordinated P*).

Kinetic measurements

A DCE solution containing the ligand and the complex was placed under nitrogen in an aluminum foil-wrapped Schlenk tube (the DCE was degassed by several freeze-pump-thaw cycles and stored under N_2). The solution of phosphine was added from a syringe with a stainless steel, which was inserted through the rubber septum caps sealing the Schlenk tubes. The Schlenk tubes were then immersed in a constant temperature ($\pm 0.2°$C) bath and samples were withdrawn at intervals through a rubber septum with the use of a syringe and transferred to a 0.5 mm NaCl cell; the 6–10 samples were withdrawn during 1 or 2 half lives of the reaction. The IR cells was flushed with N_2 and sealed with rubber septa before use.

Reactions were monitored by observing the disappearance of the highest frequency carbonyl stretching band of the parent complex. The absorbance mode was used. Plots of ln A against time were linear for more than one half-life, and the value of k_{obs} was determined from the slope of this line by the least-squares method. The correlation coefficient for the least-square line ($R^2 > 0.997$) was very good.

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