Synthesis of 3-[(acetylamino)(aryl)methyl]-4-hydroxycoumarins

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One-pot, three-component reaction between aryl aldehydes, 4-hydroxycoumarin, and acetonitrile in the presence of chlorosulfonic acid affords 3-[(acetylamino)(aryl)methyl]-4-hydroxycoumarins in excellent yields.

Keywords: chlorosulfonic acid, 4-hydroxycoumarin, three-component reaction, aryl aldehydes

Today, with the emergence of high-speed parallel synthesis, the multi-component reaction (MCR) is widely employed for the rapid assembly of arrays with high molecular diversity.[1] Coupled with a post-condensation modification, the power of these reactions is increased even further, giving rise to a plethora of complex, pharmacologically relevant templates for screening purposes.[2]

The synthesis of coumarins and their derivatives has attracted considerable attention from organic and medicinal chemists for many years as a large number of natural products contain this heterocyclic nucleus. They are widely used as additives in food, perfumes, cosmetics, pharmaceuticals[3] and optical brighteners[4] and dispersed fluorescent and laser dyes.[5] Thus the synthesis of derivatives of this nucleus is of much interest.

Three-component reaction between an enolic system such as acetophenone or β-dicarbonyl compounds, an aryl aldehyde and acetonitrile is known as the modified Dakin–West reaction. This reaction is usually carried out in the presence of an excess amount of acetyl chloride and catalysed by an acid. A number of catalysts such as $CoCl_2$,[6] montmorilonit K-10 clay,[7] H_2SO_4/SiO_2,[8] Heteropoly acids,[9] and silica sulfuric acid[10] have been employed to effect this transformation. Here we report that the similar three-component reaction between 4-hydroxycoumarin, aryl aldehydes and acetonitrile in the presence of chlorosulfonic acid affords 3-[(acetylamino)(aryl) methyl]-4-hydroxycoumarins in nearly quantitative yields (Scheme 1). This reaction is carried out cleanly with no need to use any other activator or catalyst.

Results and discussion

The structure of compounds **3a–i** is deduced from their IR, ^{1}H, and ^{13}C NMR spectra. The mass spectra of compounds **3a–i** are fairly similar and display molecular ion peaks. The ^{1}H NMR spectrum of **3a** exhibits a sharp line at δ = 1.99 ppm for the protons of methyl group. The methine and NH protons couple each other and two doublets are observed for them at 6.59 and 8.49 ppm respectively. When the ^{1}H NMR spectrum is recorded after addition of some D_2O to the d_6-DMSO solution of **3a** the doublet related to NH proton was disappeared and the doublet related to methine proton was converted to a singlet. The proton of hydroxy group resonates in the range of 12.00–13.00 ppm as a very broad signal and in some cases it is so broadened that isn't observed. The ^{13}C NMR spectrum of compound **3a** shows 16 distinct signals in consistent with the proposed structure.

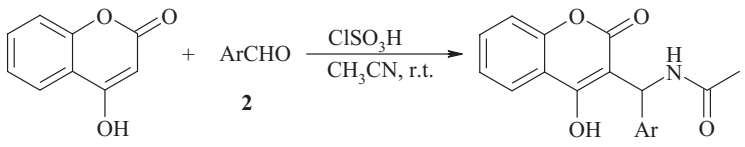
Four-component reaction between 2 equivalent of 4-hydroxycoumarin and terphthaldehyde **4** in acetonitrile in the presence of 2.2 equivalent of chlorosulfonic acid affords the addition product **5** in 98% yield. The ^{1}H NMR spectrum of compound **5** exhibits only a singlet at 7.24 ppm for the protons of the aldehyde subunit. ^{13}C NMR spectrum of this compound exhibits two signals at 126.69 and 139.66 ppm for aldehyde subunit. These observations are consistent with the symmetrical structure of compound **5**.

A reasonable mechanism for formation of compounds **3a–i** is presented in Scheme 3. Acetonitrile attacks to the condensation product of 4-hydroxycoumarin and aldehyde in the presence of chlorosulfonic acid to afford the cation **7** which hydrolyses to product **3**.

Here we report a simple and efficient one-pot synthesis of 3-[(acetylamino)(aryl)methyl]-4-hydroxycoumarins by three-component reaction between 4-hydroxycoumarin, aryl aldehydes and acetonitrile in the presence of chlorosulfonic acid. The advantages of this method are simply available starting materials, short reaction times, easy and clean work-up and excellent yields.

Experimental

Melting points were determined with an electrothermal 9100 apparatus. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyser. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionisation potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on Bruker DRX-500 Avance spectrometer at solution in d_6-DMSO using TMS as internal standard. The chemicals used in this work purchased from fluka (Buchs, Switzerland) and were used without further purification.



1 **3**

3	a	b	c	d	e	f	g	h	i
Ar	Ph	4-ClPh	4-BrPh	2-ClPh	3-NO_2Ph	4-NO_2Ph	3-CH_3OPh	2-CH_3OPh	2-HOPh
%Yield*	98	95	100	98	98	95	95	95	98

*Isolated yield

Scheme 1

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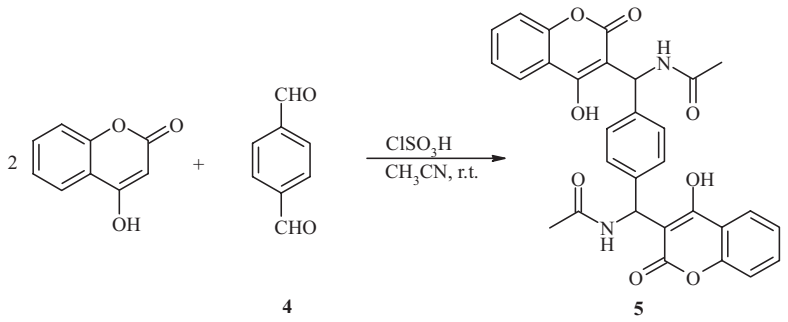
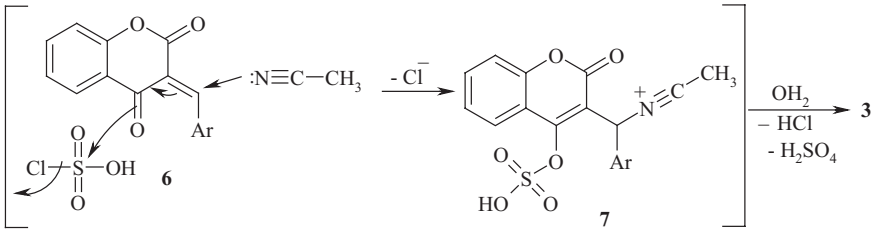


Scheme 2



Scheme 3

General procedure

To a magnetically stirred solution of 4-hydroxycoumarin (3 mmol) and aldehyde (3 mmol) in acetonitrile (15 ml) was added chlorosulfonic acid (3.3 mmol) at room temperature. The reaction mixture was then stirred for 1 h. The mixture was poured into 50 ml ice-water. The solid product was filtered, washed with ice-water and recrystalised from ethyl acetate/n-hexane to give the pure product.

N-[(4-Hydroxy-2-oxo-2H-chromen-3-yl)phenylmethyl]acetamide (**3a**): White powder, m.p. 184–186°C, IR (KBr) (ν_{max} cm^{-1}): 3320 (NH), 1674, 1629, (2 C=O). Analyses: Calcd. for $C_{18}H_{15}NO_4$: C, 69.89; H, 4.89; N, 4.53%. Found: C, 69.9; H, 4.6; N, 4.6. MS (*m/z*, %): 309 (6). ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.99 (3 H, s, C*H*$_3$), 6.59 (1 H, d, $^3J_{HH}$ = 9 Hz, C*H*), 7.20 (1 H, t, $^3J_{HH}$ = 7 Hz, C*H* of C_6H_5), 7.30 (4 H, m, 4 C*H* of C_6H_5), 7.40 (2 H, m, 2 C*H* of coumarin moiety), 7.65 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.05 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.49 (1 H, d, $^3J_{HH}$ = 9 Hz, N*H*), 12.49 (1 H, very broad, OH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 23.42 (*C*H$_3$), 47.98 (*C*H), 106.69, 117.00, 117.15, 124.65, 124.87, 133.24, 153.24, 162.28, and 170.55 (carbons of coumarin moiety), 126.94, 127.37, 128.87, and 141.38 (carbons of C_6H_5), 162.60 (N*C*=*O*).

N-[(4-Chlorophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl) methyl]acetamide (**3b**): White powder, m.p. 175–177°C, IR (KBr) (ν_{max} cm^{-1}): 3325 (NH), 1677, 1637, (2 C=O). Analyses: Calcd. for $C_{18}H_{14}ClNO_4$: C, 62.89; H, 4.10; N, 4.07%. Found: C, 62.8; H, 4.2; N, 4.1. MS (*m/z*, %): 343 (5). ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.99 (3 H, s, C*H*$_3$), 6.61 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H*), 7.38 (6 H, m, 2 CH of coumarin moiety and 4 CH of C_6H_4Cl), 7.65 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.04 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.53 (1 H, d, $^3J_{HH}$ = 8 Hz, N*H*), 12.50 (1 H, very broad, OH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 22.96 (*C*H$_3$), 46.97 (*C*H), 105.87, 116.53, 116.81, 124.30, 124.54, 132.98, 152.85, 162.05, and 170.14 (carbons of coumarin moiety), 128.37, 128.45 131.52, and 140.24 (carbons of C_6H_4Cl), 162.63 (N*C*=*O*).

N-[(4-Bromophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl) methyl]acetamide (**3c**): White powder, m.p. 172–174°C, IR (KBr) (ν_{max} cm^{-1}): 3235 (NH), 1668, 1625, (2 C=O). Analyses: Calcd. for $C_{18}H_{14}BrNO_4$: C, 55.69; H, 3.63; N, 3.61%. Found: C, 55.8; H, 3.6; N, 3.5. MS (*m/z*, %): 387 (7). ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.98 (3 H, s, C*H*$_3$), 6.55 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H*), 7.26 (2 H, d, $^3J_{HH}$ = 8 Hz, 2 CH of C_6H_4Br), 7.40 (2 H, m, 2 C*H* of coumarin moiety), 7.49 (2 H, d, $^3J_{HH}$ = 8 Hz, 2 CH of C_6H_4Br), 7.66 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.04 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.49 (1 H, d, $^3J_{HH}$ = 8 Hz, N*H*), 12.35 (1 H, very broad, OH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 23.35 (*C*H$_3$), 47.40 (*C*H), 106.25, 116.89, 117.23, 124.69, 124.97, 133.42, 153.24, 162.40, and 170.52 (carbons of coumarin moiety), 120.37, 129.22 131.67, and 141.09 (carbons of C_6H_4Br), 162.44 (N*C*=*O*).

N-[(2-Chlorophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl) methyl]acetamide (**3d**): White powder, m.p. 208–210°C, IR (KBr) (ν_{max} cm^{-1}): 3225 (NH), 1662, 1625, (2 C=O). Analyses: Calcd. for $C_{18}H_{14}ClNO_4$: C, 62.89; H, 4.10; N, 4.07%. Found: C, 62.9; H, 4.2; N, 4.2. MS (*m/z*, %): 343 (11). ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.95 (3 H, s, C*H*$_3$), 6.49 (1 H, d, $^3J_{HH}$ = 7 Hz, C*H*), 7.19–7.39 (6 H, m, 2 CH of coumarin moiety and 4 CH of C_6H_4Cl), 7.65 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.03 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.56 (1 H, d, $^3J_{HH}$ = 7 Hz, N*H*), 12.50 (1 H, very broad, OH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 23.22 (*C*H$_3$), 47.40 (*C*H), 105.03, 116.82, 117.21, 124.54, 124.84, 132.56, 153.29, 162.09, and 170.17 (carbons of coumarin moiety), 127.09, 129.07, 129.92, 130.62, 133.36, and 138.90 (carbons of C_6H_4Cl), 162.80 (N*C*=*O*).

N-[(3-nitrophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl] acetamide (**3e**): White powder, m.p. 195–196°C, IR (KBr) (ν_{max} cm^{-1}): 3315 (NH), 1676, 1634, (2 C=O). Analyses: Calcd. for $C_{18}H_{14}N_2O_6$: C, 61.02; H, 3.98; N, 7.91%. Found: C, 60.9; H, 4.1; N, 7.9. MS (*m/z*, %): 354 (10). ^{1}H NMR (500 MHz, d_6-DMSO): δ 2.01 (3 H, s, C*H*$_3$), 6.49 (1 H, d, $^3J_{HH}$ = 7 Hz, C*H*), 7.42 (2 H, m, 2 CH of coumarin moiety), 7.56 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of $C_6H_4NO_2$), 7.65 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 7.78 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of $C_6H_4NO_2$), 8.03 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.10 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of $C_6H_4NO_2$), 8.17 (1 H, s, C*H* of $C_6H_4NO_2$), 8.69 (1 H, d, $^3J_{HH}$ = 7 Hz, N*H*). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 22.91 (*C*H$_3$), 47.13 (*C*H), 105.46, 116.45, 116.85, 124.37, 124.56, 133.09, 152.90, 162.01, and 170.29 (carbons of coumarin moiety), 121.15, 122.04, 129.97, 133.43, 143.95, and 148.14 (carbons of $C_6H_4NO_2$), 162.40 (N*C*=*O*).

N-[(4-nitrophenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl]acetamide (**3f**): White powder, m.p. 179–182°C, IR (KBr) (ν_{max} cm^{-1}): 3350 (NH), 1691, 1636, (2 C=O). Analyses: Calcd. for $C_{18}H_{14}N_2O_6$: C, 61.02; H, 3.98; N, 7.91%. Found: C, 61.0; H, 4.1; N, 7.9. MS (*m/z*, %): 354 (13). ^{1}H NMR (500 MHz, d_6-DMSO): δ 2.02 (3 H, s, C*H*$_3$), 6.69 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H*), 7.42 (2 H, m, 2 CH of coumarin moiety), 7.52 (2 H, d, $^3J_{HH}$ = 8 Hz, 2 C*H* of $C_6H_4NO_2$), 7.65 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.03 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.22 (2 H, d, $^3J_{HH}$ = 8 Hz, 2 C*H* of $C_6H_4NO_2$), 8.62 (1 H, d, $^3J_{HH}$ = 8 Hz, N*H*). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 22.91 (*C*H$_3$), 47.39 (*C*H), 105.47, 116.17, 116.43, 124.38, 124.64, 133.19, 152.97, 162.01, and 170.29 (carbons of coumarin moiety), 123.63, 127.77, 133.19, and 149.54 (carbons of $C_6H_4NO_2$), 162.40 (N*C*=*O*).

N-[(3-methoxyphenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl) methyl]acetamide (**3g**): White powder, m.p. 203–206°C, IR (KBr) (ν_{max} cm^{-1}): 3300 (NH), 1677, 1631, (2 C=O). Analyses: Calcd. for $C_{19}H_{17}NO_5$: C, 67.25; H, 5.05; N, 4.13%. Found: C, 67.2; H, 5.2; N, 4.2. MS (*m/z*, %): 339 (7). ^{1}H NMR (500 MHz, d_6-DMSO): δ 2.00 (3 H, s, C*H*$_3$), 3.72 (3 H, s, OC*H*$_3$), 6.60 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H*),

6.78 (1 H, m, CH of C_6H_4OMe), 6.90 (1 H, s, CH of C_6H_4OMe), 7.19 (1 H, m, CH of C_6H_4OMe), 7.31 (3 H, m, 2 CH of coumarin moiety and CH of C_6H_4OMe), 7.65 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.03 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.69 (1 H, d, $^3J_{HH}$ = 8 Hz, N*H*). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 22.99(*C*H_3), 47.36*C*H), 55.43 (O*C*H_3), 106.33, 116.50, 116.80, 124.24, 124.54, 132.91, 152.79, 161.77, and 170.08 (carbons of coumarine moiety), 111.85, 112.84, 118.86, 129.54, 142.69, 159.62 (carbons of $C_6H_4NO_2$), 162.11 (N*C*=*O*).

N-[(2-methoxyphenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl) methyl]acetamide (**3h**): White powder, m.p. 190–191°C, IR (KBr) (ν_{max} cm^{-1}): 3405, 3300 (NH), 1677, 1631, (2 C=O). Analyses: Calcd. for $C_{19}H_{17}NO_5$: C, 67.25; H, 5.05; N, 4.13%. Found: C, 67.2; H, 5.1; N, 4.2. MS (*m/z*, %): 339 (10). ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.91 (3 H, s, C*H*$_3$), 3.67 (3 H, s, OC*H*$_3$), 6.53 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H*), 6.89 (2 H, m, 2 CH of C_6H_4OMe), 7.21 (1 H, m, CH of C_6H_4OMe), 7.31 (2 H, m, 2 CH of coumarin moiety), 7.46 (1 H, d, $^3J_{HH}$ = 7 H$_Z$, CH of C_6H_4OMe), 7.65 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.00 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.43 (1 H, d, $^3J_{HH}$ = 8 Hz, N*H*), 12.50 (1 H, very broad, OH). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 23.22 (*C*H_3), 44.89 (*C*H), 56.78 (O*C*H_3), 106.06, 116.76, 116.97, 124.51, 124.76, 132.99, 153.08, 161.84, and 170.53 (carbons of coumarin moiety), 111.65, 120.47, 128.20, 128.74, 137.25, and 157.32 (carbons of C_6H_4OMe), 162.30 (N*C*=*O*).

N-[(2-hydroxyphenyl)(4-hydroxy-2-oxo-2H-chromen-3-yl) methyl]acetamide (**3i**): White powder, m.p. 146–148°C, IR (KBr) (ν_{max} cm^{-1}): 3300 (NH), 1670, 1635, (2 C=O). Analyses: Calcd. for $C_{18}H_{15}NO_5$: C, 66.46; H, 4.65; N, 4.31%. Found: C, 66.3; H, 4.5; N, 4.3. MS (*m/z*, %): 325 (15). ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.96 (3 H, s, C*H*$_3$), 6.48 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H*), 6.71 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of C_6H_4OH), 6.77 (1 H, t, $^3J_{HH}$ = 7 Hz, C*H* of C_6H_4OH), 7.04 (1 H, t, $^3J_{HH}$ = 7 Hz, C*H* of C_6H_4OH), 7.37 (3 H, m, 2 CH of coumarin moiety and CH of C_6H_4OH), 7.62 (1 H, t, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 7.97 (1 H, d, $^3J_{HH}$ = 8 Hz, C*H* of coumarin moiety), 8.58 (1 H, d, $^3J_{HH}$ = 8 Hz, N*H*). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 23.29 (*C*H_3), 45.03 (*C*H), 106.16, 116.98, 117.26, 124.46, 124.77, 132.98, 153.08, 162.02, and 170.75 (carbons of coumarin moiety), 115.68, 119.05, 126.81, 128.44, 129.06, and 155.18 (carbons of C_6H_4OH), 162.36 (N*C*=*O*).

N-{4-[Acetylamino(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl] phenyl}(4-hydroxy-2-oxo-2H-chromen-3-yl)methyl]acetamide (**5**): White powder, m.p. 230–233°C, IR (KBr) (ν_{max} cm^{-1}): 3295 (NH), 1666, 1635, (2 C=O). Analyses: Calcd. for $C_{30}H_{24}N_2O_8$: C, 66.66; H, 4.48; N, 5.18%. Found: C, 66.5; H, 4.5; N, 5.3. MS (*m/z*, %): 540 (3). ^{1}H NMR (500 MHz, d_6-DMSO): δ 1.91 (6 H, s, 2 C*H*$_3$), 6.53 (2 H, d, $^3J_{HH}$ = 8 Hz, 2 C*H*), 7.24 (4 H, s, 4 CH of C_6H_4), 7.39 (4 H, m, 4 CH of coumarin moieties), 7.64 (2 H, t, $^3J_{HH}$ = 7 Hz, 2 C*H* of coumarin moieties), 8.01 (2 H, d, $^3J_{HH}$ = 8 Hz, 2 C*H* of coumarin moieties), 8.48 (2 H, d, $^3J_{HH}$ = 7 Hz, 2 N*H*). ^{13}C NMR (125.8 MHz, d_6-DMSO): δ 23.37 (*C*H_3), 47.71 (*C*H), 106.55, 116.92, 117.16, 124.61, 124.90, 133.28, 153.18, 162.20, and 172.88 (carbons of coumarin moieties), 126.69 and 139.66 (carbons of C_6H_4), 162.57 (N*C*=*O*).

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