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Note

Stereoselective crossed-aldol condensation of 3-acetyl-2,5-dimethylthiophene/furan with aromatic aldehydes in water: Synthesis of (2*E*)-3-aryl-1-(thien-3-yl/fur-3-yl)-prop-2-en-1-ones

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Aldol condensation of 3-acetyl-2, 5-dimethylthiophene **1** and 3-acetyl-2, 5-dimethylfuran **2** with different aromatic aldehydes have been carried out in water in heterogeneous phases in the presence of cetyltrimethylammonium bromide as cationic surfactant at rt. All the reactions occur in short time with excellent yields of steroselective heteroarylpropenones with water as environmental friendly solvent.

Keywords: Stereoselective, crossed-aldol, cetyltrimethylammonium bromide, cationic surfactant, heteroarylpropenones, heterogeneous phases.

IPC Code: Int.Cl.[8] C 07D

Chalcones are α, β-unsaturated ketones which are abundant in the plant kingdom. It is well known that most natural or synthetic chalcones are highly active with extensive pharmaceutical and medicinal applications[1]. Recently, chalcones have been used as anti-AIDS[2], cytotoxic with antiangiogenic activity[3,4], antimalarial[5,6], anti-inflammatory[7,8] and antitumor[9, 10] agents.

The U.S. Environmental Protection Agency (EPA) has recommended a drastic reduction in the use and handling of more than ten hazardous common organic solvents for industrial production of chemicals. This paper presents a clean and safe method of production with high yield of stereoselective heteroarylpropenones as important biologically active compounds using water as a cheap solvent and environment friendly reaction medium.

Water is an attractive medium for many organic reactions[11]. The advantages of aqueous medium over organic solvents include lower cost, safety and environment friendliness. Also, it allows *p*H control and use of surfactants as micro aggregates.

The hydrophobic effect and large cohesive energy of water are considered to be the factors mainly responsible for enhancing reactivity and selectivity of these reactions[12,13].

Mixed or crossed aldol condensation is a base-catalyzed addition of different aldehydes and ketones and one of them must contain at least one α-hydrogen to form an aldol or ketol which can be dehydrated to α, β-unsaturated aldehydes or ketones (**Scheme I**).

The classical reaction conditions of aldol condensation are NaOH solution in hydroalcoholic medium which often yielded a mixture of (*E*) and (*Z*) chalcones[14,15].

Recently, aldol reaction has also been carried out in aqueous medium in the presence of catalysts to increase molecular aggregation and stereoselectivity[16-19].

Results and Discussion

The previous investigations[16-19] are now extended to carbon-carbon bond formation and this paper focuses on the crossed-aldol condensation of 3-acetyl-2, 5-dimethylthiophene **1** and 3-acetyl-2, 5-dimethylfuran **2** with a variety of different aromatic aldehydes, namely, 2-methoxybenzaldehyde, 4-methoxybenzaldehyde, 4-(N, N-dimethyl)aminobenzaldehyde, 2, 4-dimethoxybenzaldehyde, 3, 4-methylenedioxybenzaldehyde, 2-chlorobenzaldehyde, 4-chlorobenzaldehyde and 2-nitrobenzaldehyde in water at rt and in the presence of cetyltrimethylammonium bromide (CTABr) as the cationic surfactant. This results in the formation of (2*E*)-3-aryl-1-(thien-3-yl)prop-2-en-1-ones **3a-h**. On the other hand, 3-acetyl-2, 5-dimethylfuran **2** condenses with 4-methylbenzaldehyde (*p*-toulaldehyde), 2-methoxybenzaldehyde, 4-methoxybenzaldehyde (anisaldehyde), 2,4-dimethoxybenzaldehyde, 3,4-methylene-dioxybenzaldehyde (piperonal), 4-chlorobenzaldehyde and 2-nirobenzaldehyde in aqueous medium and under the same conditions to give (2*E*)-3-aryl-1-(fur-3-yl)prop-2-en-1-ones **4a-g**. In both cases, the products are obtained in excellent yields with high stereoselectivity and short reaction periods. Analytical gas chromatography and spectral data proved that only *E*-isomers were formed in all cases. The ^{1}H NMR coupling constants (*J*) of C2-H

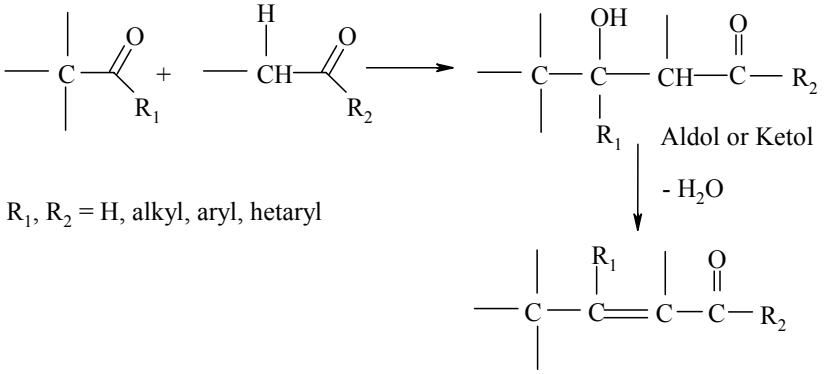
and C3-H of the isolated heteroarylpropenones are in the range of 15.5-16.0 Hz which is characteristic of *E*-propenones **(Table I)**.

It is observed from **Table II** that electron donating substituents on the aromatic aldehydes decrease the reaction period and increase the yield of heteroarylchalcones. No chalcones are obtained in the absence of cetyltrimethyl ammonium bromide (CTABr).

It is expected that the synthesized heteroarylpropenones might have biological and medicinal activity analogous to the biologically active quinolinyl[9] and some ferrocenyl chalcones[15].

Experimental Section

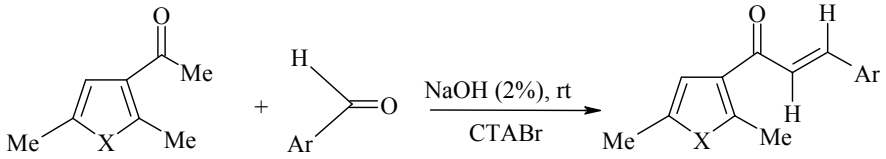
All melting points reported are uncorrected. IR spectra were recorded using Perkin-Elmer Spectrum RXIFT-IR spectrophotometer. The ^{1}H NMR spectra



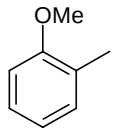
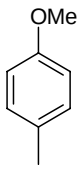
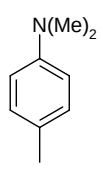
Scheme I

Table I—Crossed-Aldol condensation of 3-acetyl-2, 5-dimethylthiophene/furan **1, 2** with aromatic aldehydes

Synthesis of (2*E*)-3-aryl-1-(thien/furyl-3-yl)prop-2-en-1-ones **3a-h** and **4a-g**.



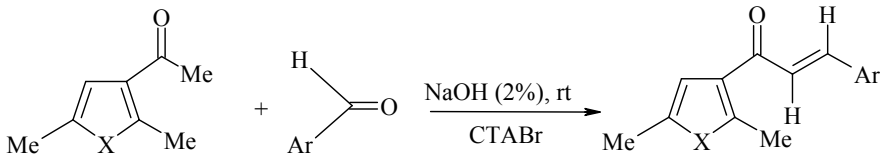
1: X= S **; 2:** X= O **3a-h:** X= S **; 4a-g:** X= O

Compd	Ar	^{1}H NMR ($CDCl_3$)	MS *m/z* (%)
3a		2.45 (s, 3H, CH_3), 2.71 (s, 3H, CH_3), 3.91 (s, 3H, OCH_3), 6.95 (m, 2H, Ph-H), 7.08 (s, 1H, C4-H), 7.35 (m, 1H, Ph-H), 7.37 (d, 1H, C2-H, *J* = 15.6 Hz), 7.60 (d, 1H, thien-H), 8.03 (d, 1H, C3-H, *J* = 15.6 Hz).	276 (5, M+3), 272(14, $M^{+\cdot}$276 (5, M+3), 272(14, $M^{+\cdot}$),), 241 (100, M-OMe), 165 (16), 163(5), 151 (16) 121 (30, $^{+\cdot}C_7H_7OCH_3$), 110 (27) 105 (16, C_6H_3 OCH_3), 79 (24), 71 (15), 51(28, C_4H_3).
3b		2.44 (s, 3H, CH_3), 2.70 (s, 3H, CH_3), 3.84 (s, 3H, OCH_3) 6.91(d, 2H, Ph-H), 7.06 (s, 1H, thien-H), 7.14 (d, 1H, C2-H, *J* = 15.8 Hz), 7.55 (d, 2H, Ph-H), 7.66 (d, 1H, C3-H, *J* = 15.7 Hz).	
3c		2.44 (s, 3H, CH_3), 2.69 (s, 3H, CH_3), 3.01 (s, 6H, N $(CH_3)_2$), 6.67 (d, 2H, Ph- H), 7.05 (s, 1H, thien-H), 7.07 (d, 1H, C2-H, *J* = 15.69 Hz), 7.51 (d, 2H, Ph-H), 7.67 (d, 1H, C3-H, *J* = 15.62 Hz).	275 [22, (M+2)-CH_3], 274 [31, (M+3)- CH_3], 243 [16, (M+2)-NC_2H_6], 171 (8), 138 (33), 111 (100), 83(38) 77 (34, $C_6H_5^+$), 51 (43, C_4H_3).

Contd

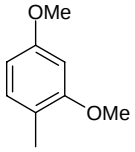
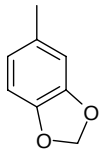
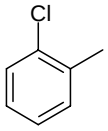
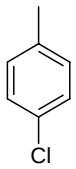
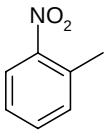
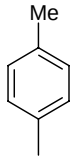
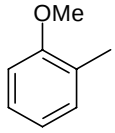
Table I—Crossed-Aldol condensation of 3-acetyl-2, 5-dimethylthiophene/furan **1, 2** with aromatic aldehydes

Synthesis of (2*E*)-3-aryl-1-(thien/furyl-3-yl)prop-2-en-1-ones **3a-h** and **4a-g**.—*Contd*



1: X= S **; 2:** X= O

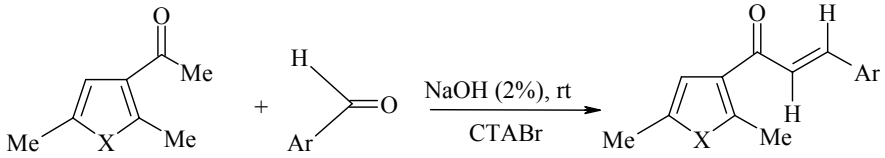
3a-h: X= S **; 4a-g:** X= O

Compd	Ar	^{1}H NMR ($CDCl_3$)	MS *m/z* (%)
3d		2.31 (s, 3H, CH_3), 2.57 (s, 3H, CH_3), 3.73 (s, 3H, OCH_3), 3.76 (s, 3H, OMe) 6.35 (s, 1H, Ph-H), 6.40 (d, 1H, Ph-H) 6.93 (s, 1H, thien-H), 7.15 (d, 1H, C2-H, *J* = 15.92 Hz), 7.40 (d, 1H, Ph-H), 7.83 (d, 1H, C3-H, *J* = 15.83 Hz).	286 [22, (M+1)-CH_3], 165 (12), 135 (100, $C_6H_3(OCH_3)_2^+$), 110 (18). 89 (81, $C_7H_5^+$), 67 (20), 59 (46).
3e		2.43 (s, 3H, CH3), 2.69 (s, 3H, CH_3), 6.00 (s, 2H, O_2CH_2), 6.82 (d, 1H, Ph-H), 7.05 (s, 1H, thien- H), 7.08 (d, 1H, Ph-H), 7.09 (d, 1H, C2-H; *J* = 14.7 Hz) 7.12 (s, 1H, Ph-H), 7.61 (d, 1H, C3-H; *J* = 14.8 Hz).	213 (13, M- (CO+ O_2CH_2), 111 (100), 83 (18).
3f		2.44 (s, 3H, CH_3), 2.72 (s, 3H, CH_3), 7.07 (s, 1H, thien-H), 7.24 (d, 1H, C2-H, *J* = 15.7 Hz), 7.29 (m, 2H, Ph-H), 7.43(d, 1H, Ph-H), 7.70 (d, 1H, Ph-H), 8.08 (d, 1H, C3-H; *J* = 15.7 Hz).	
3g		2.43 (s, 3H, CH_3), 2.70 (s, 3H, CH_3), 7.06 (s, 1H, thien-H), 7.23 (d, 1H, C2-H, *J* = 15.2 Hz), 7.37 (d, 2H, Ph-H), 7.53 (d, 2H, Ph-H), 7.64 (d, 1H, C3-H, *J* = 15.8 Hz).	207 (5), 165 (5, M-C_6H_4Cl or C_6H_4Cl $CH=CHCO^+$), 151 (27), 139 (21), 111 (10, C_6H_4Cl), 102 (26, C_6H_4–CH=$CH^{+\bullet}$), 77 (7, $C_6H_5^{+\bullet}$), 67 (20), 59 (60), 51 (100, C_4H_3).
3h		2.44 (s, 3H, CH_3), 2.71 (s, 3H, CH_3), 7.09 (d, 2H, Ph-H), 7.55 (m, 1H, Ph-H), 7.67 (s, 1H, thien-H), 7.68 (d, 1H, C2-H, *J* = 15.6Hz), 8.03 (s, 1H, Ph-H), 8.04 (d, 1H, C3-H, *J* = 15.7 Hz).	287 (0.5, $M^{+\bullet}$), 252 (15), 240 (10, M- NO_2), 224 (14), 155 (21), 139 (99), 111 (52), 102 (38, $C_6H_4CH=CH^{+\bullet}$), 89(31, $C_7H_5^{+\bullet}$) 76 (29, $C_6H_4^+$), 67 (79, C_5H_7), 59 (100), 51 (64).
4a		2.28 (s, 3H, CH_3), 2.38 (s, 3H, CH_3), 2.60 (s, 3H, Ph-CH_3), 6.33 (s, 1H, furyl-H), 7.12 (d, 1H, C2-H, *J* = 16.1 Hz), 7.21 (d, 2H, Ph-H), 7.49 (d, 2H, Ph-H), 7.69 (d, 1H, C3-H, *J* = 15.8 Hz).	240 (44, $M^{+\bullet}$), 225 (12, M-CH_3), 197 [21, M-(CH_3+ CO)], 169 (14), 149 (24, M-$C_6H_4CH_3$), 145 (12, $C_6H_4CH_3$-CH=CH-CO^+), 123 (34), 115 (55, CH_3-C_6H_3CH=CH), 94(17), 91 (59, C_6H_4-CH_3), 81 (17), 65 (26), 53 (100).
4b		2.28 (s, 3H, CH_3), 2.61 (s, 3H, CH_3), 3.90 (s, 3H, OCH_3), 6.32 (s, 1H, furyl-H), 6.95 (m, 2H, Ph-H)), 7.26 (d, 1H, C2-H, *J* = 15.6 Hz), 7.35 (t, 1H, Ph-H), 7.58 (d, 1H, Ph-H), 8.02 (d, 1H, C3-H), *J* = 15.6 Hz)..	256 (26, $M^{+\bullet}$), 225 (64, M-OCH_3), 149 (9, M-C_6H_4O- CH_3), 123 (29), 118 (15, $C_7H_4OCH_3$), 94 (21), 89 ($C_7H_5^{+\bullet}$), 77 (30, $C_6H_5^{+\bullet}$) 53 (100, HC_2CO^+).

Contd

Table I—Crossed-Aldol condensation of 3-acetyl-2, 5-dimethylthiophene/furan **1, 2** with aromatic aldehydes

Synthesis of (2*E*)-3-aryl-1-(thien/furyl-3-yl)prop-2-en-1-ones **3a-h** and **4a-g**—*Contd*



1: X= S **; 2:** X= O **3a-h:** X= S **; 4a-g:** X= O

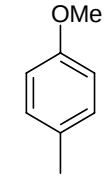
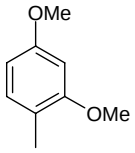
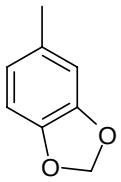
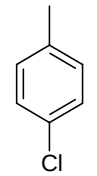
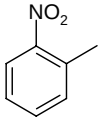
Compd	Ar	^{1}H NMR ($CDCl_3$)	MS *m/z* (%)
4c		2.30 (s, 3H, CH_3), 2.62 (s, 3H, CH_3), 3.87 (s, 3H, OCH_3), 6.34 (s, 1H, furyl-H), 6.93 (d, 2H, Ph-H), 7.07 (d, 1H, C2-H, *J* = 15.6 Hz), 7.57 (d, 2H, Ph-H), 7.70 (d, 1H, C3-H, *J* =15.6 Hz).	256 (14, $M^{+\cdot}$), 241(8, M-CH_3), 225 (6, M-OCH_3), 213 [20, M-($CO+CH_3$)], 185 [10, M-(HC_2CO+CH_3)], 123 (34), 121 (39, $C_7H_6OCH_3$), 94 (32), 89 (42, $C_7H_5^{+\cdot}$), 53 (100, HC_2CO).
4d		2.28 (s, 3H, CH_3), 2.60 (s, 3H, CH_3), 3.88 (s, 3H, OCH_3), 3.90 (s, 3H, OCH_3), 6.31 (s, 1H, furyl-H), 6.46 (s, 1H, Ph-H), 6.51 (d, 1H, Ph-H), 7.19 (d, 1H, C2-H, *J* = 15.8 Hz), 7.51 (d, 1H, Ph-H), 7.95 (d, 1H, C3-H, *J* = 15.5 Hz).	274 [57, (M+2)- CH_3], 273 [18, (M+1)-CH_3], 259 (22, M-CO), 243 [29, M- ($CO+CH_3$)], 171 (21), 138 (42, $C_6H_4(OCH_3)_2$), 111 (79), 89 (23, $C_7H_5^{+\cdot}$), 83 (36) 77 (23, $C_6H_5^{+\cdot}$), 51 (100, HC_2CO).
4e		2.28 (s, 3H, CH_3), 2.60 (s, 3H, CH_3), 6.00 (s, 2H, O_2CH_2), 6.31 (s, 1H, furyl-H), 6.81 (d, 1H, Ph-H), 7.00 (d, 1H, C2-H, *J* = 15.4 Hz), 7.08 (d, 1H, Ph-H), 7.10 (s, 1H, Ph-H), 7.62 (d, 1H, C3-H, *J* = 15.4 Hz).	270 (29, $M^{+\cdot}$), 135 (42, $C_7H_5O_2CH_2$), 123 (39), 94 (30), 89 (87, $C_7H_5^+$), 63 (78), 53 (100, HC_2CO^+).
4f		2.29 (s, 3H, CH_3), 2.61 (s, 3H, CH_3), 6.32 (s, 1H, furyl-H), 7.13 (d, 1H, C2-H, *J* = 15.5 Hz), 7.36 (d, 2H, Ph-H), 7.52 (d, 2H, Ph-H), 7.65 (d, 1H, C3- H, *J* = 157 Hz).	260 (26, $M^{+\cdot}$), 217 (9, M-($CO+CH_3$), 165 (10, ClC_6H_4-CH=CH-CO^+), 149(33), 135 (19), 123 (40), 112 (11, C_6H_5Cl), 101 (54, C_6H_4-C≡$CH^{+\cdot}$), 81 (17), 75 (28, $C_6H_3^+$), 53 (100, HC_2CO^+).
4g		2.28 (s, 3H, CH_3), 2.59 (s, 3H, CH_3), 6.33 (s, 1H, furyl-H), 6.99 (d, 1H, C2-H, *J* = 15.7 Hz), 7.55 (m, 1H, Ph-H), 7.68 (m, 2H, Ph-H), 8.04 (s, 1H, Ph-H), 8.05 (d, 1H, C3-H, *J* = 15.3 Hz).	276 (25), 165 (39), 151 (57), 139 (45), 111 (25), 101 (49), 67 (36), 59 (84), 51 (100).

Table II—Characterization data of heteroarylpropenones **3** and **4**.

Compd	Mol. formula (Mol. wt.)	m.p.ºC (Colour)	Reaction time (hr) (Yield%)	Calcd.(Found) % C	H	N
3a	$C_{16}H_{16}O_2S$ (272.36)	87-9 (yellow)	8 (94)	70.56 (70.49	5.92 5.88	—)
3b	$C_{16}H_{16}O_2S$ (272.36)	73-4 (dark yellow)	24 (72)	70.56 (70.43	5.92 5.85	—)
3c	$C_{17}H_{19}NOS$ (285.40)	97-8 (orange)	5 (84)	71.54 (71.36	6.71 6.64	4.91 4.77)

Contd

Table II—Characterization data of heteroarylpropenones **3,4**.—*Contd*

Compd	Mol. formula (Mol. wt.)	m.p.ºC (Color)	Reaction time (hr) (Yield%)	Calcd.(found) % C	H	N
3d	$C_{17}H_{18}O_3S$ (302.39)	103-04 (dark yellow)	4 (65)	67.52 (67.43	6.00 5.92	—)
3e	$C_{16}H_{14}O_3S$ (286.35)	99-100 (yellow)	2 (94)	67.11 (67.00	4.93 4.87	—)
3f	$C_{15}H_{13}ClOS$ (276.78)	118-20 (yellow)	3.5 (97)	65.09 (64.94	4.73 4.67	—)
3g	$C_{15}H_{13}ClOS$ (276.78)	122-24 (pale yellow)	1.5 (82)	65.09 (64.88	4.73 4.63	—)
3h	$C_{15}H_{13}NO_3S$ (287.33)	147-49 (buff)	3 (95)	62.70 (62.53	4.56 4.47	4.87 4.81)
4a	$C_{16}H_{16}O_2$ (240.30)	70-2 (yellow)	2 (81)	79.97 (79.81	6.71 6.66	—)
4b	$C_{16}H_{16}O_3$ (256.30)	77-8 (yellow)	6 (83)	74.98 (74.82	6.29 6.21	—)
4c	$C_{16}H_{16}O_3$ (256.30)	84-6 (yellow)	11 (85)	74.98 (74.78	6.29 6.17	—)
4d	$C_{17}H_{18}O_4$ (286.32)	88-90 (yellow)	5 (61)	71.31 (71.19	6.34 6.27	—)
4e	$C_{16}H_{14}O_4$ (270.28)	113-15 (dark yellow)	3 (75)	71.10 (70.63	5.22 5.14	—)
4f	$C_{15}H_{13}ClO_2$ (260.72)	105-07 (yellow)	7 (66)	69.10 (68.33	5.03 4.89	—)
4g	$C_{15}H_{13}NO_4$ (271.27)	135-36 (yellow)	5 (69)	66.41 (66.23	4.83 4.71	5.16 4.87)

were recorded on Bruker Avance DPX400 spectrometer, using $CDCl_3$ as solvent and TMS as internal standard (chemical shifts in δ, ppm; *J* in Hz). The mass spectra were recorded using Shimadzu GC-17A gas chromatograph coupled with QP-5000 mass spectrometer. Elemental analyses were preformed on Perkin-Elmer 2400, Series II microanalyzer. 3-Acetyl-2,5-dimethylthiophene and 3-acetyl-2,5-dimethylfuran were obtained from Aldrich and used without further purification.

General procedure for the synthesis of (2*E*)-3-aryl-1-(thien/furyl-3-yl)prop-2-en-1-ones 3a-h and 4a-g.

3-Acetyl-2, 5-dimethylthiophene **1**, or 3-acetyl-2, 5-dimethylfuran **2**, (100 mmoles), aromatic aldehydes (100 mmoles) and cetyltrimethylammonium bromide (CTABr, 5.46 g, 15 mmoles) were added to an aqueous solution of NaOH (200 mL, 0.5 *M*). The mixture was vigorously stirred at 20°C for the duration reported in **Table II**. The reaction was monitored by TLC and GC by using a solution of the reaction mixture in CH_2Cl_2. The solid products were filtered off, washed with water (3 × 25 mL), dried and purified by recrystallization from the appropriate solvent. The characterization data of the purified compounds, reaction period and percentage yield are listed in **Table II**.

The present method offers the following significant advantages over conventional procedures:

(i) improved reaction rates and increased yields through suppression of side reactions,
(ii) clean, safe and simple methodology,
(iii) enhanced stereo-selectivity,
(iv) expensive and hazardous organic solvents are totally eliminated,
(v) aqueous alkali metal hydroxides replace alkoxides, and
(vi) lower reaction temperatures and easier work-up.

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