

Review of Electrolytic Conductance Standards

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Measurements of aqueous electrolytic conductance are performed routinely in a variety of disciplines and industries. Conductivity is a measure of the ionic content in solution and thus has applications in pharmaceuticals, power plants, rainwater, lake surveys, and oceanography, to name a few. A thorough review of the measurement of and standards for aqueous electrolytic conductance is herein presented. At present, the most precise and accurate standards have been set forth by the International Organization of Legal Metrology (OIML), and have been adopted by most other standards organizations. However, the uncertainty assigned to these standards, especially the secondary standards, is somewhat larger than would be expected from the physical aspects of the measurement. Several changes in the units and measurement scales, including temperature, volume, molar mass, resistance, and concentration obfuscate the accuracy of these standards. In addition to the review, research is proposed, using a conductance cell with variable length, to establish new standards for aqueous electrolytic conductance.

KEY WORDS: Aqueous solution; cell constant; conductance; electrolytic conductivity; potassium chloride; primary; resistance; resistivity, secondary, standards, and temperature scale.

1. INTRODUCTION

Electrolyte conductance has long been recognized as an important property of electrolyte solutions. It plays a significant role in the development of modern theory on ionic interaction and also serves as a powerful tool for determining ionic dissociation upon which many chemical reactions depend.

The specific conductance of an electrolyte solution, κ , is determined by the ratio G/R , where the cell constant G is the ratio of the ef-

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fective cell length l to the electrode area a (i.e., l/a), and R is the resistance of the solution contained in this cell. The unit for κ is reciprocal ohm per centimeter, or in SI units, Siemen per meter, $S\text{-m}^{-1}$. The measurement of electrolytic conductance with an A.C. bridge began with Kohlrausch in 1869.⁽¹⁾ Kohlrausch determined the cell constant by measuring l and a , and measured the solution resistance with a modified Wheatstone bridge. At the end of the 19th century, he published a set of specific conductances for KCl and other solutions.⁽²⁾ His data were considered the best at the time, and his values for KCl solutions were adopted as standards because of the stability of these solutions and the ease with which KCl could be purified.

Some twenty years later, Parker and Parker,⁽³⁾ using a method similar to that of Kohlrausch, redetermined the values for the KCl standards, and defined a new unit, the 'demal' (D), to be a gram mole of KCl dissolved in 1000 cm^3 instead of 1 liter of solution as was used by Kohlrausch. Parker and Parker's values were adopted by the International Critical Tables,⁽⁴⁾ and were used in the recalculation of Kohlrausch's values for equivalent conductances.

In the late 1920s and early 1930s, Jones and his coworkers⁽⁵⁻⁷⁾ critically evaluated the two sets of standards and the previous experimental techniques and apparatus. They found it desirable to redetermine the values for specific conductance of KCl solutions, and carefully designed the necessary apparatus. They redefined the 'demal' unit on a fixed mass of KCl in 1000 g of aqueous solution (weight basis in vacuo). Mercury was used for determined their primary cell constant (the resistance of a fixed column of mercury was at that time the international standard of resistance). In 1933, a new set of values for the specific conductances of KCl solutions was published.⁽⁷⁾ These new values together with those of Kohlrausch and Parker are listed in Table I.

Jones and Bradshaw's values were adopted in 1981, with corrections for the change in the temperature scale and for the conversion to the absolute ohm, by the Organisation Internationale de Metrologie Legale (OIML) as primary standards.⁽⁹⁾

Concurrent with Jones' work, Shedlovsky⁽¹⁰⁾ made careful measurements of κ at 25°C for KCl solutions from 3×10^{-5} to 0.1 mol-L^{-1} (herein after abbreviated with the letter c), using Parker and Parker's values to obtain his cell constants. His equivalent conductances, $\Lambda = 1000\kappa/c$, $S\text{-cm}^2\text{-mol}^{-1}$ were subjected to curve fitting to obtain the specific conductances at nominal concentrations of 0.001, 0.01, and 0.1 c . These values were adopted by OIML as secondary standards, again

Table I. Specific Conductance of KCl at 25°C

Conc.	Kohlrausch ^a		Parker & Parker ^b		J. & B. ^b	
	g KCl per kg soln(vac)	κ S-cm ⁻¹	g KCl per kg H ₂ O(air)	κ S-cm ⁻¹	g KCl per kg soln(vac)	κ S-cm ⁻¹
1D	71.3828	0.11180	76.6276	0.111322	71.1352	0.11134 ₂
0.1D	7.43344	0.01288	7.47896	0.01285 ₄	7.41913	0.012856
0.01D	0.746558	0.001413	0.746253	0.0014078 ₉	0.745263	0.001408

^a From Ref. 8. ^b Jones and Bradshaw, Ref. 7.

after corrections were made to the Jones standard, the absolute ohm, and the 1968 temperature scale.

These two sets of standards (Jones and Shedlovsky) have generally been accepted for the past fifty years. However, during this period, as has already been noted, the resistance unit has changed from the international to the absolute ohm (1948), the temperature scale has changed (1968) slightly,⁽¹¹⁾ the liter has been redefined as the cubic decimeter (1964),⁽¹²⁾ and the atomic weights of potassium and chlorine have been redetermined. Although these changes have affected the values of standards to 0.1% or less, this is significantly greater than the claimed accuracy of the original measurements. Moreover, the 'demal' unit is not a customary unit of concentration in solution chemistry, and fifty years is a long time for any given standard to go without remeasurement and verification. For these reasons, we have critically examined the old values subjected to the noted changes, and have drawn conclusions about the need for new experiments.

2. THE EVALUATION OF THE PRIMARY STANDARD

Jones and Bradshaw's results converted from the international to the absolute ohm (1 international ohm = 1.000495 ohms, January 1, 1948) are given in Table II.

In 1968, a change was made in the international practical temperature scale (IPTS) of 1948.⁽¹¹⁾ This scale had been formalized in 1948, but was, in fact, in usage before then and was the one used by Jones and Bradshaw in the 1930's. The differences in these scales are given in Table III.

At 18°C and 25°C the differences are -0.0065°C and -0.008°C, respectively.

The effect of the change of temperature scale on κ can be seen by

Table II. Specific Conductances of Standard Aqueous Solutions of KCl, per Jones and Bradshaw (converted to the absolute ohm basis)

Demal	$\kappa(\text{S}\cdot\text{cm}^{-1})$		
	0°C	18°C	25°C
1.0	0.065144	0.097790	0.111287
0.1	0.0071344	0.0111612	0.0128496
0.01	0.00077326	0.00121992	0.00140807

Table III. Changes in the International Practical Temperature Scale (IPTS)

$t_{68}^{\circ}\text{C}$	$(t_{68} - t_{48})^{\circ}\text{C}$	$t_{68}^{\circ}\text{C}$	$(t_{68} - t_{48})^{\circ}\text{C}$
0	0		
10	-0.004	60	-0.010
20	-0.007	70	-0.008
30	-0.009	80	-0.006
40	-0.010	90	-0.003
50	-0.010	100	0

comparing data in Table IV with those in Table II. The data in Table IV were obtained by least squares fitting the data of Jones and Bradshaw at various temperatures, and corrects $\kappa(\text{S}\cdot\text{cm}^{-1})$ for the 1968 temperature scale according to the following polynomials.

$$\kappa_{1\text{D}} = 6.5144 \times 10^{-2} + 1.73198 \times 10^{-3}t + 4.5752 \times 10^{-6}t^2 \quad (1)$$

$$\kappa_{0.1\text{D}} = 7.1344 \times 10^{-3} + 2.11221 \times 10^{-4}t + 6.98508 \times 10^{-7}t^2 \quad (2)$$

$$\kappa_{0.01\text{D}} = 7.7326 \times 10^{-4} + 2.33404 \times 10^{-5}t + 8.24156 \times 10^{-8}t^2 \quad (3)$$

The temperature dependence equations for $\kappa_{0.1\text{D}}$ and $\kappa_{0.01\text{D}}$ give essentially the same results as the conductance-temperature data of Bremner and Thompson.⁽¹³⁾ Juhasz and Marsh,⁽¹⁴⁾ in 1981, also made temperature scale change corrections to the values of Jones and Bradshaw and their values agree with those in Table IV.

In 1979, Saulnier and Barthel (SB)⁽¹⁵⁾ made an independent, absolute measurement of κ for a 0.01D KCl solution at three temperatures. However, as pointed out by Marsh,⁽¹⁶⁾ they used the wrong sign in the

Table IV. Specific Conductances of Standard Aqueous Solutions of KCl, per Jones and Bradshaw (converted to the absolute ohm and the 1968 Temperature Scale)

Demal	$\kappa(\text{S}\cdot\text{cm}^{-1})$		
	0°C	18°C	25°C
1.0	0.065144	0.097802	0.111303
0.1	0.0071344	0.0111627	0.0128516
0.01	0.00077326	0.00122009	0.00140828

correction of the Jones and Bradshaw conductance values to the 1968 temperature scale. When the data are corrected properly, the two sets of data agree within 0.07%, which is the stated uncertainty in the Saulnier and Barthel data.

The International Recommendation No. 56, June 1986, (1st Edition 1981) by OIML⁽⁹⁾ recommended the rounded values shown in Table V, to which we have added the SB values in the last row for comparison.

Table V. Primary Standards for Specific Conductance

	Demal	$\kappa(\text{S}\cdot\text{cm}^{-1})$		
		0°C	18°C	25°C
OIML	1.0	0.065144	0.09781	0.11131
	0.1	0.007134	0.011163	0.012852
	0.01	0.0007733	0.0012201	0.0014083
SB	0.01	0.0007731 ₇	0.0012207 ₆	0.0014093 ₆

There are several factors incorporated in the current recommended values of the primary standards that support an improved, redetermination and redefinition, *viz.*, the temperature range (0, 18, and 25°C) is limited, the demal unit is inconvenient, the accuracy could be improved by modern techniques and instrumentation, and the present primary standard is based on the resistivity of mercury. It would be very useful if the primary standards covered the temperature range 0 to 60°C at 5°C increments. This would reduce errors caused by non-linear interpolations for temperatures within the range. A molality scale, with a specified mass of KCl per kilogram of water, would be preferable to the demal scale, since it would be consistent with the customary units

used in physical chemistry and thermodynamics of solutions. (The argument that the molality scale is sensitive to changes in atomic masses, but that the demal scale is not, is fallacious. Both scales are affected by the isotopic composition of the KCl used in a given experiment. Furthermore, the atomic masses for potassium and chlorine have changed less than 0.01% over the last 50 years.) When these factors and concerns are appropriately addressed through refined experiments, the primary standards will be definitely improved.

3. CRITIQUE OF THE SECONDARY STANDARDS

The secondary standards recommended by OIML (1981) are shown in Table VI, and are based on the data of Shedlovsky.⁽¹⁰⁾ There is no concern with the quality of Shedlovsky's original data. However, the recommended values for the secondary standards have been affected by several manipulations of the original data to obtain the values given in Table VI, thus compromising the accuracy of these standards, as explained below.

Table VI. Specific Conductance of Secondary Standards at 25°C

Mass of KCl, g per 1000 g of solution	κ (S-cm ⁻¹)
0.37329	0.0007182
0.14932	0.0002916
0.07466	0.0001469

Shedlovsky did not obtain his results by absolute measurement but by calibrating his cell constant with Parker and Parker's (PP) 0.1D KCl standard solution at 25°C (Table I). In order to convert his conductance values to the primary standard, it is necessary to correct his conductance values to the Jones and Bradshaw's (JB) standard (Table I). This can be accomplished by correcting his masses for air-buoyancy. (We have assumed the following densities in g-cm⁻³; air, 0.00118; brass, 8.44; KCl, 1.984; and H₂O, 0.997043). The conductance is multiplied by 1.000280 for conversion from PP to JB, by 1/1.000495 for conversion from (int. Ω)⁻¹ to (abs. Ω)⁻¹, and by 1.000154 for conversion from 1948 IPTS to 1968 IPTS. Thus, 0.012852₄ (int. Ω)⁻¹-cm⁻¹ (1948 IPTS) becomes 0.012851₆ (abs. Ω)⁻¹-cm⁻¹ at 25°C (1968 IPTS).

In addition, Shedlovsky's concentrations are subjected to two other corrections, the molar mass from 74.553 (1932) to 74.5510

(1986);⁽¹⁷⁾ and volume unit, from 1.000027 to 1.000000 dm³.⁽¹²⁾ His concentrations expressed in today's units of mol-L⁻¹, increase by 1.000054. Since the equivalent conductance is proportional to the square root of concentration, the following relationship may be used to calculate the change of conductance due to concentration changes.

$$\Delta\Lambda \approx -100 \Delta\sqrt{c} \quad (14)$$

Thus, Shedlovsky's conductance values, when they are normalized to today's concentration scale, will be reduced by a factor of 1/1.000018.

The combination of all of these corrections to Shedlovsky's data yields a final correction factor of 1/1.00008. This amounts to 0.008% which is within his estimated experimental uncertainty. Nevertheless, the overall uncertainty in the values is necessarily increased through the combination of uncertainties involved in the corrections.

Furthermore, there is concern about the way in which Shedlovsky treated his data.⁽¹⁰⁾ First, he plotted the equivalent conductances of the dilute solutions (0.00003 to .0033 *c*) against the square root of *c* (mol-L⁻¹) to obtain values at nominal concentration from 0.0001 to 0.002 *c*. For the more concentrated solutions he fitted the data to the following equation

$$\Lambda_o = (\Lambda + 59.79 \sqrt{c}) / (1 - 0.2274 \sqrt{c}) - Bc \quad (5)$$

where $\Lambda_o = 149.82$, the equivalent conductance at infinite dilution and $B = 94.9$, an empirical constant. His calculated values fitted the experimental values with a standard deviation of 0.029, and a maximum deviation of 0.13. It is apparent that Eq. (5) is not the best form for a fitting equation.

In 1959, Lind, Zwolenik, and Fuoss (LZF)⁽¹⁸⁾ published a comprehensive analysis of nine sets of data^(10,19-27) for concentrations of KCl below 0.012 *c*, including the data of Shedlovsky. Their analysis was made in terms of international electrical units and all the concentrations conformed to a molecular weight of 74.557 for KCl. The equation used for their analysis was

$$\Lambda + 94.64 \sqrt{c} - 58.74 \text{ clog} c = \Lambda_o + Jc \quad (6)$$

Λ_o and J were found from a linear plot of Eq. (6) to be 149.93 and 198.4, respectively, from a weighted average of the nine sets of data they used in their analysis.

Chiu and Fuoss (CF)⁽²⁸⁾ extended the measurements to 0.10 *c* and used the equation

$$\Lambda = 149.93_6 - 94.88 \sqrt{c} + 25.48 \operatorname{clnc} + 221c - 229c^{3/2} \quad (7)$$

to express their results, having added one more concentration term to the LZF Eq. (6). Justice (J)⁽²⁹⁾ expressed his measurements up to 0.04 c at 25°C (1948 IPTS) by the equation

$$\Lambda = 149.89 - 94.87 \sqrt{c} + 58.63 \operatorname{clog}c + 229c - 264.3c^{3/2} \quad (8)$$

These three equations [Eqs. (6-8)] are expressed in the old units (*i.e.*, Int. ohms, 1948 IPTS, old liter, and 74.557 for the molar mass of KCl). To convert to the present units, their numerical constants must be divided by a factor of 1.00038 [*i.e.*, (1/1000495) (1.000154) (1/1.00001) (1/1.000027)], producing

$$\Lambda = 149.873 - 94.60c + 58.72 \operatorname{clog}c + 198.3c \quad (6a)$$

$$\Lambda = 149.879 - 94.84 \sqrt{c} + 58.65 \operatorname{clog}c + 220.9c - 228.9c^{3/2} \quad (7a)$$

$$\Lambda = 149.833 - 94.83 \sqrt{c} + 58.60 \operatorname{clog}c + 228.9c - 264.2c^{3/2} \quad (8a)$$

In 1977, Janz and Tompkins⁽³⁰⁾ cited an equation from the Rostock school

$$\Lambda = 150.000 - 99.282 \sqrt{c} + 135.798c - 120.788c^{3/2} + 57.891c^2 - 11.725c^{5/2} \quad (9)$$

which was converted to the abs. ohm and 1968 IPTS by Juhasz, *et al.*⁽¹⁴⁾ as follows

$$\Lambda = 149.95 - 99.25 \sqrt{c} + 135.75c - 120.75c^{3/2} + 57.87c^2 - 11.7c^{5/2} \quad (10)$$

Recently, Barthel *et al.* (BFNW)⁽³¹⁾ made measurements up to 0.05 c at 0, 10, 18, and 25°C and expressed their results for 25°C by

$$\Lambda = 149.875 - 95.01 \sqrt{c} + 38.48 \operatorname{clog}c + 183.1c - 176.4c^{3/2} \quad (11)$$

In this equation, the second and third coefficients were computed theoretically and included a correction for the recently developed Chen effect.⁽³²⁾ Their measurements were based on JB 0.01 D standard which is in int. ohm and 1948 IPTS units. Their molar mass KCl was 74.555. Their equivalent conductance values, when expressed in abs. ohms, 1968 IPTS, and molar mass of 74.5510 will be reduced by a factor of 1/1.000361 *i.e.*, [(1/1.000495) (1.000154) (1/1.00002)]. Thus Eq. (11) becomes

$$\Lambda = 149.819 - 94.98 \sqrt{c} + 38.47 \operatorname{clog}c + 183.0c - 176.3c^{3/2} \quad (12)$$

Table VII. Comparison of Specific Conductivities at 25°C

Author	Eq.	$\lambda(\text{S}\cdot\text{cm}^2\cdot\text{mol}^{-1})$						$0.0099718(0.01D)$		$0.0996921(0.1D)$	
		0.001	0.002	0.005	0.01	0.05	λ	$10^3\kappa(\text{S}\cdot\text{cm}^{-1})$	λ	$10^2\kappa$	
S	obs	146.93	145.79	143.64	141.32	133.33	141.33	1.40932	128.93	1.2853 ₃	
S	4	146.95	145.81	143.65	141.36	133.33	141.37	1.40971	128.97	1.2857 ₃	
LZF	6a	146.903	145.721	143.501	141.221	-	141.231	1.40833	-	-	
CF	7a	146.918	145.743	143.522	141.202	133.340	141.213	1.40815	128.897	1.2850 ₀	
J	8a	146.879	145.710	143.506	141.203	133.307	141.214	1.40816	128.545	1.2814 ₉	
R	10	146.943	145.772	143.569	141.268	133.333	141.279	1.40881	128.883	1.2848 ₆	
BFNW	11a	146.878	145.713	143.514	141.205	133.258	141.213	1.40815	128.685	1.2828 ₆	
JB	obs							1.40828		1.2851 ₆	
SB	obs							1.40936			
Mean		146.914	145.751	143.557	141.254	133.316	141.253	1.40870	128.818	1.2843 ₅	
R.S.D. %		0.020	0.027	0.445	0.045	0.023	0.044	0.044	0.128	0.121	

It should be noted that only the Λ values of the above equations should be converted by those factors, the second and third parameters of Eq. (12) have their theoretical significance and should not be altered. The adjustable parameters, Λ_0 and the last two parameters must be readjusted by a least squares fit. The above adjustments are used only for comparison with the final results that are computed with those equations. The results are listed in Table VII.

The last 4 columns of Table VII are the equivalent Λ and specific κ conductances for 0.01 and 0.1D KCl solutions as specified by Jones and Bradshaw (JB) for which the molalities are 0.01000414 and 0.1002614, respectively. The molarities of these solutions are 0.00997184 and 0.0996921, as calculated from the following equation

$$c/m = d_0 - Am \quad (13)$$

where d_0 , the density of water is 0.997045,⁽³³⁾ and $A = 0.02716$, is obtained from a least squares fitting of the density of KCl solutions given in I.C.T.⁽³⁴⁾ Both Λ and κ are calculated from equations as references in the second column. Also included are three absolute measurement values by Jones and Bradshaw (JB) and by Saulnier and Barthel (SB). It is interesting to note, that for 0.01 D solution, Saulnier and Barthel's absolute value is in excellent agreement with that of Shedlovsky's. The Rostock's value is within 0.035%. The rest of the values agree with JB value to within 0.01%. As for 0.1D solution, Shedlovsky's observed value and Chiu and Fuoss's calculated value agree with JB's to about 0.01%, while the rest differ from JB by .03% or more.

Shedlovsky's observed values are higher than those of all the later workers, possibly due to unit and scale changes. His equivalent conductances at 0.001, 0.002, and 0.005 c , after conversion to specific conductances, are recommended by OIML as secondary standards. However, we were unable to duplicate the calculations of OIML resulting in the mass of KCl per kg of solution. All calculated values from Eq. (13) are compared with the OIML values in Table VIII.

The difference of 0.15% in the mass per kg of solution shown in Table VIII is surprisingly large. However, it should be noted that the effect of this large difference on the specific conductance for the KCl solutions at the listed concentrations is very small, and is within the stated uncertainty of 0.1% of the conductance values.

From Tables VII and VIII, it appears that the secondary standards for cell calibration as described by OIML cannot be recommended.

Table VIII. Interconversion of Concentration Scale for KCl Solution^a

mol-L ⁻¹	mol·kg ⁻¹ (H ₂ O)	mol·kg ⁻¹ (soln)	mass-(kg soln) ⁻¹	
			Eq. (13)	OIML
0.001	0.0010030	0.0010029 ₃	0.074775	0.07466
0.002	0.0020060 ₄	0.0020057 ₄	0.149955 ₂	0.14932
0.005	0.0050155	0.0050136 ₃	0.37391	0.37329
Average Δ = 0.15%				

^a Molar Mass = 74.5510; 25°C (1968 IPTS); L = 1 dm³; and $d_{\text{H}_2\text{O}} = 0.997043 \text{ g}\cdot\text{cm}^{-3}$

4. DISCUSSION AND CONCLUSION

The prime purpose of conductance standards is the calibration of conductivity cell constants. The factors which affect the accuracy are 1. stability and purity of the materials, KCl and H₂O in this case; 2. instrumentation and measuring technique; 3. temperature control; and 4. cell design.

The data in Table VII indicates a wide spread among the values from various authors and casts doubt on the use of these secondary standards to calibrate cell constants. It is conceded that for routine work not requiring high accuracy, the OIML recommended values for primary standards will suffice. However, for those measurements requiring high precisions and accuracies (better than 0.1%), the variance in the values is unsettling. Moreover, the many changes in temperature, volume, molar mass, and resistance complicates the use of the primary JB standard. As noted before, the primary standard of JB is based on the resistivity of mercury. It would be desirable to eliminate this dependency and to redetermine a primary standard with greater precision and accuracy using modern technology. An additional enhancement to the knowledge and measurement of aqueous electrolytic conductance would be an extension of the temperature range for standards to include several points from 0 to 60°C, rather than the current limited values at 0, 18, and 25°C.

To this end, we have designed an all glass conductance cell with a well designed geometry. By carefully measuring the cell dimensions, the cell constant can be determined accurately to 0.02%. A portion of the distance between the electrodes can be adjusted by inserting precision-bore borosilicate glass tubes whose diameter and length have been accurately determined.

The theory behind this approach is as follows. The specific conductance for a known concentration of electrolyte is a constant. The total effective cross-sectional area is a fixed value for a given cell and is composed of two parts of the NBS cell: A_o , due to the portion of the cell between the electrodes and the connecting flanges; and A_i , due to the diameter of the replaceable center tube. A_i must be accurately known and it is expedient if it is constant for all the center tubes. The distance l has two components: l_o , the portion of the fixed but unknown length between the electrodes and the connecting flanges; and l_i , the accurately known length of the i 'th center tube. The resistance R_i is the measured quantity, and can be separated into two components: r_o , due to l_o and A_o ; and r_i , due to the i 'th center tube. Thus.

$$R_1 = (1/\kappa)(r_o + r_1) = (1/\kappa)(r_o + l_1/A_1) \quad (14)$$

and

$$R_2 = (1/\kappa)(r_o + r_2) = (1/\kappa)(r_o + l_2/A_2) \quad (15)$$

By performing the experiment with two center tubes of different lengths, (but with $A_1 = A_2 = A$), and solving the simultaneous equations, κ can be determined

$$\kappa = (l_1 - l_2)/(R_1 - R_2)A \quad (16)$$

Preliminary experiments using potassium chloride have been completed successfully. Agreement with the OIML recommended value for 0.1*D* KCl is within 0.04%. Refinements to the cell and the procedure are being made to improve the accuracy and precision. The concentration of KCl to be studied will be from 0.005 to 1.0 m, over the temperature range of 0 to 60°C.

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