

COMPARISON OF METAL CORROSION INHIBITION BY GRAVIMETRIC AND LINEAR POLARIZATION RESISTANCE METHODS

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ABSTRACT

Studies were conducted to evaluate the effectiveness of various dosages of the selected silicate and phosphate compounds applied for corrosion inhibition of cast iron, copper, lead, and galvanized steel specimens. The compounds selected for study were zinc polyphosphate (Calgon C-39), zinc orthophosphate (Virchem V-931), sodium metasilicate and glassy silicate. The effectiveness of these compounds for corrosion inhibition were studied under differing water quality conditions using gravimetric and electrochemical corrosion test methods.

INTRODUCTION

Corrosion in potable water distribution systems is a continuous problem faced by municipalities and water utilities. The problems created as a result of corrosion can be grouped into three categories: economics, aesthetics and health. Corrosion may result in the deterioration of water quality and may significantly decrease the hydraulic capacity of water mains by promoting pipe wall pitting and the growth of tubercles. If corrosion is not inhibited, costly main replacements are inevitable. Corrosion wastes resources and can cost industrialized countries 3 percent of their gross national product (1).

Aside from the economic problems mentioned above, the excessive dissolution of iron and copper from plumbing and distribution systems can cause aesthetic problems with respect to taste, color, or staining characteristics (2). In addition, excessive lead levels resulting primarily from the corrosive action of water on lead pipes and joints can cause health problems such as learning disabilities and mental retardation (3). Consequently, the U.S. Environmental Protection Agency has recently reduced the maximum contamination level (MCL) of lead to 0.015 mg/L (4). The main source of lead in the drinking water is from dissolution of lead from old lead pipes, solders and plumbing fixtures. Thus, municipalities that have lead problems in their water supplies will be investigating treatment techniques to reduce corrosion and lead dissolution. Corrosion control for lead and copper pipes using silicate and phosphate compounds may be possible for some water supplies (5). In the past, orthophosphate addition to water has been successful in reducing the corrosion of galvanized pipes (6). Blends of ortho- and polyphosphates have been particularly effective in reducing the corrosion of iron and steel (7). Sodium silicate has been used to reduce the corrosion of galvanized iron (8). There are numerous other

publications where phosphate and silicate compounds have been used for corrosion protection (9) (10).

Metal corrosion rate has been measured for quite some time by a standard test procedure in which the weight loss of a sample coupon is evaluated after exposing it to the test solution for a set period (11). The test in effect measures the average cumulative corrosion rate for the exposed period. Others have suggested the use of electrochemical techniques to determine the instantaneous corrosion rate at the surface of a probe (2) (12). In these systems, the corrosion rate is measured by polarizing the test electrode where the corrosion rate under freely corroding conditions is found to be proportional to the applied current needed to maintain the polarization shift.

The research reported herein investigated the effectiveness of silicate and phosphate compounds for corrosion control in water systems using two commonly used methods for measuring corrosion rates of four types of metals used in water distribution systems; viz., cast iron, copper, lead and galvanized iron. The aim of the research was to see if these two methods give similar results under similar experimental conditions in the presence of these inhibitor compounds. Thus, the water quality parameters that affect the metal corrosion rates, such as pH, dissolved oxygen, total dissolved solids, alkalinity, hardness, temperature, etc., were kept the same for both systems.

MATERIALS AND METHODS

Gravimetric Corrosion Test

For evaluating the adequacy of silicate and phosphate compounds for corrosion control, a corrosion test apparatus was constructed. Figure 1 shows a schematic of this apparatus.

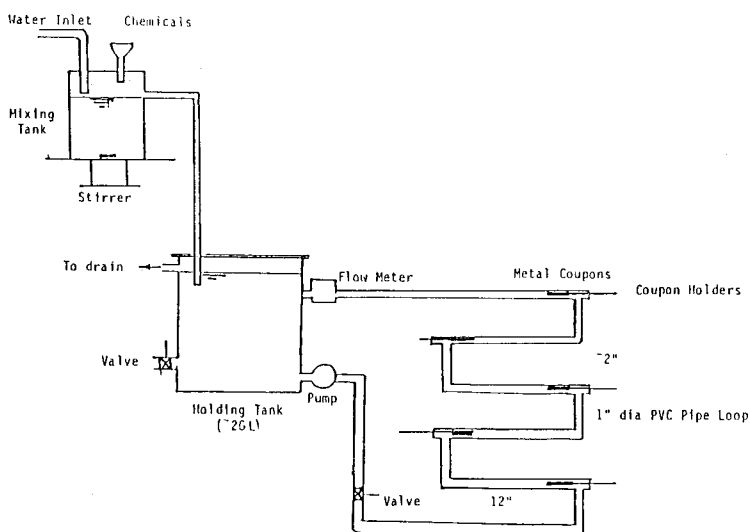


Fig. 1: Corrosion Test Pipe Loop System.

Only one of the four pipe loop systems are shown in the figure. The pipe loop system was a recirculation type, which reduced the cost of chemical additions, and was similar to that proposed in the Method B ASTM-D2688-83 (11). At each specimen holder location duplicate coupons were attached with a Teflon screw. The pipe loop was constructed of polyvinyl chloride pipe in order to avoid any external galvanic corrosion problems during testing. Table 1 shows the concentrations of the test tap water used for running the corrosion tests. The specific conductance of the tap water was about 700 micro mho/cm levels at 23° C and the total dissolved solids were about 326 mg/L. The dissolved oxygen levels were close to saturation values during the tests.

Table 1
Concentrations of various elements and Ions in Tap Water at University of Missouri Laboratories.

Elements and Ions	Chemistry Department mg/L	College of Engineering mg/L
Ag	<0.01	<0.01
Al	<0.1	<0.05
As	0.1	0.1
B	0.33	0.3
Ba	0.005	0.057
Be	<0.001	<0.001
Ca	57.0	59.0
Cd	<0.01	<0.01
Co	<0.01	<0.01
Cr	<0.01	<0.01
Cu	<0.01	<0.01
Fe	<0.02	<0.02
K	5.6	4.0
Li	0.042	0.04
Mg	26.0	26.0
Mn	0.001	0.006

which was used in the polarization resistance measurement. The Princeton Applied Research Corporation corrosion cell kit (PAR9700) is basically a 1-liter flat bottom flask with 3 ground glass joints and one ball-and socket joint. The two graphite electrodes are placed on opposite sides of the corrosion flask to carry the current. The reference electrode is used to measure the potential across the surface of the working metal electrode. A saturated calomel electrode (SCE) is used for the reference electrode.

In order to minimize the resistance between the reference electrode and the working electrode, an electrode bridge tube is used. The bridge tube has a ball-and -socket type joint so that its Vycor tip can be adjusted to a position as close as possible to the working electrode. The bridge tube is filled with a solution of 50:50 v/v saturated with KCl and water. The working electrode is the corroding metal electrode. It is a metal cylinder which is attached to a stainless steel rod which is connected to the potentiostat. The rod is insulated from the corrosive solution by a glass tube and Teflon washers. The only metal in contact with the solution is the metal sample.

The distilled deionized (DDI) water was prepared by distilling water which had been deionized by commercial cation and anion exchange columns. The specific conductance of the DDI water was 5.4 micro mho/cm at 23° C. The tap water (pH = 8), which was used in the corrosion tests, has been tested before, and found to be low in organics, chlorine and fluoride. Electrochemical tests were conducted using University of Missouri tap water, whose quality is shown in Table 1.

The cast iron samples were the same as used in the gravimetric corrosion evaluation method, but were cut into small cylinders with surface areas around 0.7 square in. (10.96 square cm) instead of flat specimens. Later, smaller cylinders were cut from the same pipe, and these were mounted differently in the apparatus. Lead, galvanized steel, and copper were of this smaller size.

Initially, the polarization resistance corrosion test runs were made using nitrogen as a purge gas. Since corrosion rates are so dependent on dissolved oxygen concentrations, it is standard procedure to use some type of purge gas, such as hydrogen, carbon dioxide, or nitrogen. The purge gasses used in this study were bubbled through vanadous solutions to remove any oxygen.

When it was discovered that purging with nitrogen changed the pH of a solution, a mixture of oxygen-free carbon dioxide and nitrogen was used. When no purge gas was desired, the sample was left open to the atmosphere. After 1 hour, the dissolved oxygen was found to be 8.5 ppm at 23° C. ASTM methods suggested that, when a purge gas is used, the corrosion cell solution should be purged for a minimum of half an hour prior to immersion of the test specimen (13).

Procedure

Throughout this study, the ASTM methods were closely followed (13). In addition to these guidelines, many of the steps followed in the polarization resistance runs were standardized so the results would be more reproducible. The PAR 173 and 175 units were warmed-up for a minimum of one hour prior to the runs. The metal sample was wet-

sanded and then wet-polished with 600-grit SiC paper until all visible corrosion products and scratches were removed. It was then soaked in boiling benzene for 5 minutes to remove any grease. The sample's dimensions were measured by a micrometer which was accurate to 0.001 in. (0.00255 cm). It was then weighed and mounted as soon as possible in the test cell, so that it could soak in the solution for half an hour prior to the run. The test solution had already been stirring, in the cell, at a rate of about 100 RPM, so that it had a full hour to equilibrate before the run. Any gas purging also lasted, for at least an hour. Once the metal sample was mounted, the recorder was allowed to warm up for half an hour. The minutes before the run was made, the instrument was calibrated, using the dummy cell of the PAR 173 unit, so that full scale was 1 amp and the 5 decades, which were printed on the PAR paper, corresponded to the current indicated by the PAR 376 settings. The y-axis was calibrated by connecting the PAR 175 directly to the recorder and manually changing the voltage. Usually -1V to +2V was expanded full scale. Five minutes before the run, the pH was measured. The corrosion potential was then found manually, by using the PAR 173 potential settings to adjust its meter to zero. The run began one hour from the time the metal was placed in the boiling benzene. The temperature was then recorded. A run was made by starting at some potential below the corrosion potential (usually -1V) and sweeping at 1 mV/sec to a potential above the corrosion potential (usually +2V). The recorder plotted log current as a function of the potential. As the potential approached the corrosion potential, the log current decreased. The line obtained, corresponds to the cathodic Tafel line. Once the potential was above the corrosion potential, the log current increased with increasing potential. The line formed corresponds to the anodic Tafel line. During the run, the PAR 173 meter was used to verify that both the x-axis and y-axis were calibrated properly. The gas purging and stirring of solution were maintained during the run.

An explanation of how the Tafel plot obtained from the electrochemical corrosion test apparatus gives the instantaneous corrosion rate of the sample metal is worthwhile. When iron corrodes, in the absence of any applied external voltage, both the oxidation and the reduction reactions take place, on the equipotential surface, at the same rate. The iron surface can be thought of as containing a network of short-circuited galvanic cells (14). The cathodic and anodic reactions are taking place simultaneously, on the same surface at the same potential which is called the corrosion potential. Current may be thought of as flowing from the sites of oxidation (anodic current) to the sites of reduction (cathodic current). On the freely corroding iron surface, the anodic and cathodic currents are equal and opposite in sign. The magnitude of these currents is called the exchange current or corrosion current, and is directly related, by Faraday's Law, to the corrosion rate of iron. However, since the cells are short-circuited, no net current flows. Therefore, at the corrosion potential, the anodic and cathodic currents can not be measured directly. An impressed current can be used to lower the potential of the iron to a point below the corrosion potential, where the entire surface of the iron acts as a cathode. Under these conditions, the current flowing out of the iron (I_a -anodic current) is negligible, and the cathodic current is equal to the impressed current. At potentials, higher than the corrosion potential, the entire surface of the iron can be made to act as an anode. Under these conditions, the current flowing into the iron (I_c -Cathodic current) is negligible, and the anodic current is equal to the impressed current.

When the corrosion rate is controlled by a slow step requiring an activation energy, the relationship between current and over-voltage may be expressed by the Tafel equations (15).

$$n = B_a \log \frac{I_a}{I_{\text{corr}}} \quad \text{and} \quad n = -B_c \log \frac{I_c}{I_{\text{corr}}}$$

n = over-voltage = difference between the potential of the iron and the corrosion potential; B_a and B_c = the anodic and cathodic currents, respectively; I_{corr} = the corrosion current. Thus, plots of potential vs. \log (current) should give straight lines. The line which represents the anodic equation will have a slope $+B$ (positive slope means current increases with increasing potential), and the line which represents the cathodic equation will have a slope of $-B$ (negative slope means current decreases with increasing potential). However, the linear relationships break down at small over-voltages because it is the observed current (the sum of the anodic and cathodic currents) which is actually plotted. At low over-voltages, neither of the currents are negligible, and the observed current is not longer approximately equal to the anodic or cathodic currents.

Figure 3 shows what a Tafel Plot should theoretically look like. Notice that the curve also deviates from linear behavior at high currents. This is caused by concentration polarization and resistance polarization. Concentration polarization is brought about when

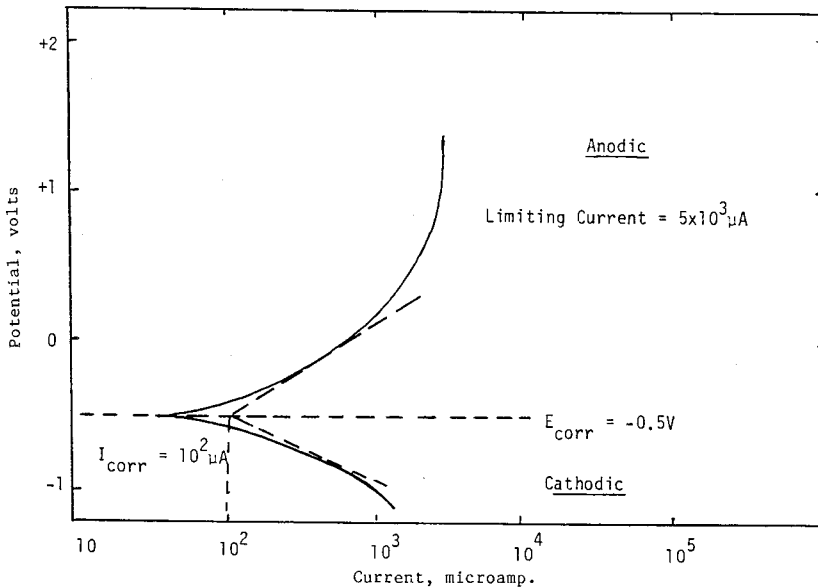


Fig. 3: Theoretical Tafel Plot

the corrosion rates get so fast that diffusion cannot keep up with it. The corrosion products build-up around the surface of the anode and exert a back EMF. Further increases

in the corrosion rate, therefore, require a disproportionate rise in over-voltage. The limiting current will then be dependent on the stirring rate. Resistance polarization is brought about because of solution resistance to current flow between electrodes. This can be minimized by using a supporting electrolyte to carry the current. However, it is often difficult to find an electrolyte which does not affect the corrosion rate. Many of the instruments used for polarization resistance measurements have an IR compensator which minimizes resistance problems.

Even though the corrosion current, which is related to the instantaneous corrosion rate of a metal, cannot be measured directly at the corrosion potential, it can be estimated by looking at the two extreme cases in which the metal surface is either predominantly anodic or cathodic. If the straight line portions of the anodic and cathodic reaction lines in Figure 3 are extrapolated to the line corresponding to the corrosion potential, all three lines should theoretically intersect at the corrosion current. Unfortunately, the linear portions of the Tafel Plots are not always as obvious as in Figure 3. However, the cathodic portion is usually pronounced enough to determine the corrosion current without extrapolating the anodic portion. Once the corrosion current is determined, it can be converted into the corrosion rate by an equation such as the following for cast iron samples:

Corrosion Rate in mils per year (mpy)

$$= \frac{8.56 \times I_{\text{corr}} \times \text{Volume}}{\text{Mass} \times \text{Area}}$$

where volume = volume of the cast iron samples in³

area = surface area of the cast iron samples in²

mass = cast iron sample mass in grams

The corrosion rate is millimeters per year (mmpy) can be obtained by multiplying mpy data by 0.0254. Thus, Tafel Plots can be used to determine the instantaneous corrosion rate of different metals in the presence of various corrosion inhibitors.

The above procedure was checked by making standard runs with #430 stainless steel samples. The results obtained were within the range of acceptability given by the ASTM method (13).

RESULTS AND DISCUSSIONS

Cast Iron Corrosion Rates:

The effect of zinc polyphosphates (Calgon C-39) inhibitor on the cast iron corrosion rate was determined by the gravimetric as well as the electrochemical method. These data are presented in Table 2. It can be seen that the electrochemical corrosion rate of cast iron was much higher than that measured by the gravimetric corrosion rate measured after 7 days. The electrochemical corrosion rate measures the corrosion rate of cast iron at time

Table 2

Comparison of Corrosion Rate Determination of Cast Iron in Presence of Zinc Polyphosphate (Calgon C-39) by Gravimetric and Electrochemical Methods.

System (tap water)	Corrosion Rate, mmpy	
	Gravimetric (7 day)	Electrochemical
Blank	0.73	2.9
4.36 mg/L P	0.79	1.6
8.72 mg/L P	0.40	1.0
13.10 mg/L P	0.07	0.5
Temperature °C	30 ± 2.5	22 ± 1

zero (approximately 45 minutes after the start), whereas the gravimetric corrosion rate measures the average corrosion rate for the exposure period. The gravimetric corrosion rate is continuously changing with time and reaches a steady state after 30-40 days, while the electrochemical method measures the maximum initial corrosion rate. With time corrosion products accumulate on the surface of the sample specimen which progressively reduces the corrosion rate of the specimen. Thus, even though the initial corrosion rate for the cast iron specimen in the gravimetric method may have been high but with time it averaged out to the values reported.

However, the trend for the corrosion rates measured by the two methods at different inhibitor concentration was similar. The system with 13.10 mg/L as P zinc polyphosphate had the least corrosion measured by both the methods. But, the degree of corrosion protection at different inhibitor doses was not found to be the same by the two methods. For example, at 7 -days exposure, 13.10 mg/L as P system reduced the specimen corrosion rate by 90% as per the gravimetric method, but only about 83% by the electrochemical method when compared to the blank (control) corrosion rate. Therefore, quantitative differences in corrosion protection predictions will remain depending upon the method used for measuring the corrosion rate.

Table 3 shows a comparison of the effectiveness of the inhibitor zinc orthophosphate (Virchem V-931) on cast iron corrosion by the gravimetric and the electrochemical method. Again, the corrosion rate values determined by the electrochemical method were larger, but the trend was similar except for the blank data. The 8.72 mg/L as P system had the least corrosion rate measured by both the methods. It is not clear why there were increases in the corrosion rates for the systems dosed with 2.18 mg/L P and 4.36 mg/L P as compared to the blank using the gravimetric method.

Table 4 and 5 shows the comparison of the corrosion rate of cast iron in the presence of sodium silicate and glassy silicate respectively by the two methods. The gravimetric

Table 3

Comparison of Corrosion Rate Determination of Cast Iron in Presence of Zinc Phosphate (Virchem V-931) by Gravimetric and Electrochemical Methods.

System (tap water)	Corrosion Rate, mmpy	
	Gravimetric (3 day)	Electrochemical
Blank	0.78	2.4
4.36 mg/L P	1.31	2.1
8.72 mg/L P	1.0	2.1
13.10 mg/L P	0.28	1.4
Temperature °C	22 ± 1	22 ± 1

Table 4

Comparison of Corrosion Rate Determination of Cast Iron in Presence of Na₂SiO₃ by Gravimetric and Electrochemical Methods.

System (tap water)	Corrosion Rate, mmpy	
	Gravimetric (3 day)	Electrochemical
Blank	1.19	2.9
5 mg/L as SiO ₂	1.18	2.7
15 mg/L as SiO ₂	0.89	2.9
30 mg/L as SiO ₂	0.72	2.8
Temperature °C	20 ± 2	22 ± 1

method indicated that at 3 -day exposure time, sodium silicate at 30 mg/L as SiO₂ reduced the corrosion rate of cast iron by about 40% whereas the reduction measured by the electrochemical method was negligible. Thus, the electrochemical method could not measure differences in corrosion rates of cast iron in the presense of sodium silicate (except at very high doses), whereas the gravimetric method clearly showed some improved corrosion protection at 15 and 30 mg/L as SiO₂. The data on cast iron corrosion in the presence of glassy silicate inhibitor were different. The gravimetric method found the cast iron corrosion rate to decrease by 13% at a 15 mg/L as SiO₂ dose and to increase by 5% at a 12.5% decrease at 20 mg/L as SiO₂ and 25% decrease at 30 mg/L as SiO₂. Thus, there was a poor correlation between the cast iron corrosion rate

Table 5

Comparison of Corrosion Rate Determination of Cast Iron in Presence of Glassy Silicate by Gravimetric and Electrochemical Methods.

System (tap water)	Corrosion Rate, mmpy	
	Gravimetric (10 day)	Electrochemical
Blank	0.415	2.4
5 mg/L as SiO ₂	0.41	2.1
15 mg/L as SiO ₂	0.36	2.1 (20 mg/L as SiO ₂)
30 mg/L as SiO ₂	0.435	2.8
Temperature °C	19	22 ± 1

data in the presence of glassy silicate by the two methods.

Table 6 shows the comparison of the corrosion rate data of copper, lead and galvanized steel in the presence of zinc polyphosphate inhibitor by the two methods.

The blank copper corrosion rate was again found to be higher as measured by the electrochemical method that obtained by the gravimetric method. Reiber *et al.*, (16) had observed that the copper corrosion rates measured by the electrochemical method was about 25% lower than the gravimetric method. The water used in the tests reported here was hard water (hardness 253 mg/L as CaCO₃) with a pH of 7.8 and total alkalinity of 252 mg/L as CaCO₃ while the Seattle, WA water used in Reiber *et al's* experiment was soft (hardness < 50 mg/L as CaCO₃), pH range 7.8 - 8.3 and total alkalinity of about 16 mg/L as CaCO₃. The water quality differences could be a factor causing differences with corrosion measurements.

The two methods gave different results to measure the effects of zinc polyphosphate additions for reducing copper corrosion rate. The electrochemical method indicated that zinc polyphosphate at doses ≥ 4.36 mg/L as P was effective in reducing the corrosion rate of copper, but the data from the gravimetric method was different. It showed that there was some protection at 4.36 mg/L as P dose, but practically none at higher doses. Reiber (17) found that orthophosphate additions in the range of 1-5 mg/L as P decreased the corrosion rate of copper by three-to fivefold. Thus, the data from the electrochemical measurements are supported by these findings.

For lead specimens, the additions of 13.10 mg/L as P zinc polyphosphate reduced the corrosion rate four-fold as measured by the electrochemical method while the reduction in corrosion rate was not as drastic when measured by the gravimetric method. However, the trends were generally similar. In the case of galvanized steel specimens, there was a good correlation between the corrosion rates measured in presence of different doses of zinc

Table 6

Comparison of Corrosion Rate Determination of Copper, Lead and Galvanized Steel in Presence of Zinc Polyphosphate by Gravimetric and Electrochemical Methods.

System (test water)	Corrosion Rate, mmpy					
	Copper		Lead		Galvanized Steel	
	Gravimetric (7 day)	Electrochemical	Gravimetric (5 day)	Electrochemical	Gravimetric (7 day)	Electrochemical
Blank	0.26	0.9	0.071	0.17	1.49	0.54
4.36 mg/L P	0.19	0.2	0.071	0.17	0.89	0.42
8.75 mg/L P	0.265	0.2	0.03	0.07	0.76	0.20
13.10 mg/L P	0.25	0.2	0.05	0.04	1.31	0.45
Temperature °C	21 ± 1	22 ± 1	21 ± 1	22 ± 1	19.5 ± 0.5	22 ± 1

phosphate by the two methods.

It has been reported that in systems where there is a uniform corrosion of a surface, such as in copper and zinc specimens, there is a good correlation between the corrosion rate measurements by the electrochemical and the gravimetric methods (12). But where the surfaces are non uniformly corroded, forming pits overgrown by tubercles of corrosion products, such as in iron and steel surfaces, there may not be a good correlation between the two methods (12).

CONCLUSIONS

In summary, it was found that the electrochemical corrosion rates were generally higher than the gravimetric corrosion rates measured on the same metal specimen. In many instances the corrosion inhibition trends measured by these two methods in the presence of various corrosion inhibitors for different metal specimens were similar; e.g. cast iron, lead and galvanized steel in the presence of zinc polyphosphate. However, there were systems where the corrosion rate measurement data by the two methods did not correlate well; e.g. cast iron in the presence of sodium silicate and glassy silicate, and copper in the presence of zinc polyphosphate. Therefore, caution is needed to interpret metal corrosion rate inhibition data as measured by these two methods.

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