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Technical Report No. 18

POLAROGRAPHIC DETERMINATION OF NITRITE

Office of Naval Research
Contract N8onr-520/III
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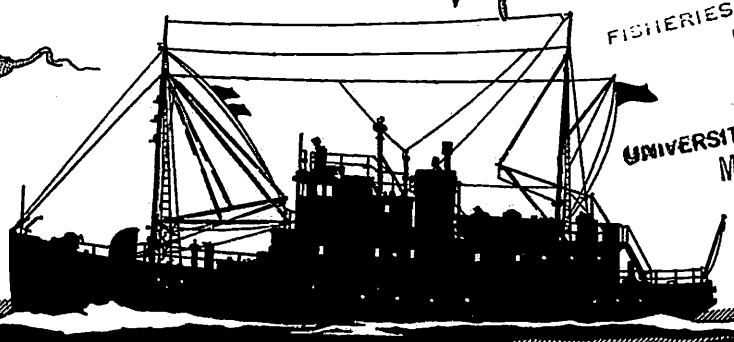
January 1954



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(Formerly Oceanographic Laboratories)
Seattle, Washington

POLAROGRAPHIC DETERMINATION OF NITRITE

by

David Tung-Whei Chow and Rex J. Robinson

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Richard H. Fleming
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Polarographic Determination of Nitrite

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This work was undertaken to investigate the polarographic reduction of molybdate in the presence of nitrite. It has been found that nitrite gives a current enhancement starting at zero applied potential which differs from that of nitrate recently reported. This current enhancement is a function of nitrite concentration and may be used as the basis for its determination. The molybdate-catalyzed reduction of nitrite in both sulfate and chloride solutions and the reduction of nitrate in chloride medium have been studied. It has been found that the current enhancement of both nitrite and nitrate is greater in chloride solutions. The method is suitable for the estimation of low concentrations of nitrite and also for the determination of both nitrite and nitrate ions in the presence of each other, for which a procedure has been proposed.

RECENTLY Johnson and Robinson (*4*) reported that molybdate gave an enhanced polarographic diffusion current when nitrate was present in the solution and that this enhancement in current was a function of the nitrate concentration. They were able to determine nitrate by measuring the current at the applied potential of −0.75 volt *vs.* SCE. Kolthoff, Harris, and Matsuyama (*7*) had used a potential of −1.20 volts *vs.* SCE for the determination of nitrate in uranyl acetate solutions. Likewise, Keilin and Otvos (*6*) had used the same potential for the determination of nitrite in the presence of uranyl ions. Further investigation of the polarographic reduction of molybdate has shown that the diffusion current is enhanced also by the presence of nitrite, and that this may be used as the basis for its determination.

APPARATUS

The polarograms presented in this paper have been obtained with a Sargent polarograph, Model XXI. An H-shaped electrolytic cell (*9*) with a saturated calomel reference electrode as one arm connected by a potassium chloride bridge was used. The capillary characteristics, $m^{2/3}t^{1/6}$, were 1.53 mg.$^{2/3}$ sec.$^{-1/2}$ The electrolytic cell was immersed in a thermostat adjusted to 25.0° ± 0.1° C. by a Merc-to-Merc thermoregulator and a Merc-to-Merc relay control box (Precision Scientific Co.).

REAGENTS

All chemicals used in this investigation were of reagent grade. The various solutions were prepared with distilled water.

EXPERIMENTAL

The oxygen dissolved in the various solutions was removed by washing with nitrogen gas. However, when the nitrogen gas was bubbled through the acidic nitrite solutions, there was appreciable loss of oxides of nitrogen. Keilin and Otvos (*6*) estimated that this error ranged up to 3% in their work. In the present investigation, loss of oxides of nitrogen was prevented by separately washing with nitrogen gas the sodium nitrite solution and the medium containing the indifferent electrolyte and the molybdate. The desired amount of nitrite was then added to the indifferent electrolyte medium, under a nitrogen atmosphere, and carefully mixed with a minimum of surface agitation. Polarographic analysis was made immediately after the preparation of this solution. Potentials reported in this work are referred to the saturated calomel electrode (SCE), and all currents have been corrected for residual current of molybdate.

Catalytic Reduction of Nitrite in Sulfate Medium. It has long been known that nitrite and nitrate give well-defined reduction waves only in the presence of certain polyvalent cations (*13, 14*), and also that molybdate can be reduced polarographically in acidic solution. Johnson and Robinson (*4*) observed that molybdate gave an enhanced diffusion current in the presence of nitrate and that this might be used as the basis for its determination. They used a solution of 0.1 *M* sulfuric acid, 0.2 *M* sodium sulfate, and 8.75 × 10^{-5} *M* sodium molybdate. In the present investigation, polarograms have been obtained for similar solutions containing varying concentrations of sodium nitrite ranging from 1 × 10^{-4} *M* to 5 × 10^{-3} *M*. Typical polarograms are shown in Figure 1, in which curve *a* was obtained with molybdate alone and curve *b* with nitrite present.

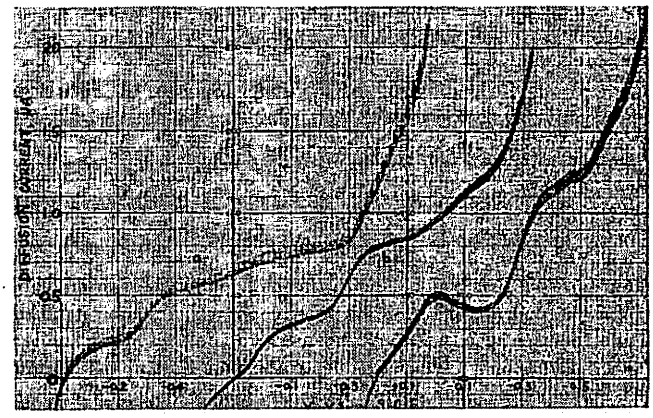

Figure 1. Polarograms of Sulfuric Acid Solutions Containing Varying Concentrations of Sodium Nitrite

a. 0.1 *M* sulfuric acid, 0.2 *M* sodium sulfate, and 8.75 × 10^{-5} *M* sodium molybdate
b. 0.1 *M* sulfuric acid, 0.2 *M* sodium sulfate, 8.75 × 10^{-5} *M* sodium molybdate, and 3.00 × 10^{-4} *M* sodium nitrite
c. 0.1 *M* sulfuric acid, 0.2 *M* sodium sulfate, 8.75 × 10^{-5} *M* sodium molybdate, and 2.00 × 10^{-3} *M* sodium nitrite

The reduction current of molybdate was enhanced as a function of nitrite concentration. It was also found that the current enhancement started at the applied potential where molybdenum (VI) was reduced to molybdenum(V). The increase in current

Table I. Catalytic Determination of Nitrite in Sulfuric Acid Solution[a]

Nitrite Molarity	Reduction Current for Nitrite, μa.	$K = i/C$, μa./Molarity
1.00 × 10^{-4}	0.033	330
2.00 × 10^{-4}	0.076	380
4.00 × 10^{-4}	0.130	325
6.00 × 10^{-4}	0.206	343
8.00 × 10^{-4}	0.270	338
1.00 × 10^{-3}	0.325	325
2.00 × 10^{-3}	0.612	306
3.00 × 10^{-3}	0.851	287
4.00 × 10^{-3}	1.097	274
5.00 × 10^{-3}	1.321	264

[a] 0.1 *M* sulfuric acid, 0.2 *M* sodium sulfate, 8.75 × 10^{-5} *M* sodium molybdate, $m^{2/3}t^{1/6}$ = 1.53 mg.$^{2/3}$ sec.$^{-1/2}$ Residual current at −0.5 volt *vs.* SCE = 0.508 μa.

could be conveniently measured at the applied potentials of −0.15 volt and −0.50 volt *vs.* SCE. These applied potentials were much less negative than reported by previous investigators for their work. The results obtained at −0.50 volt *vs.* SCE are given in Table I. It is seen that there is a definite relationship between the reduction current and nitrite concentration though it is not linear. Thus it is necessary to establish and use a calibration curve when determining nitrite.

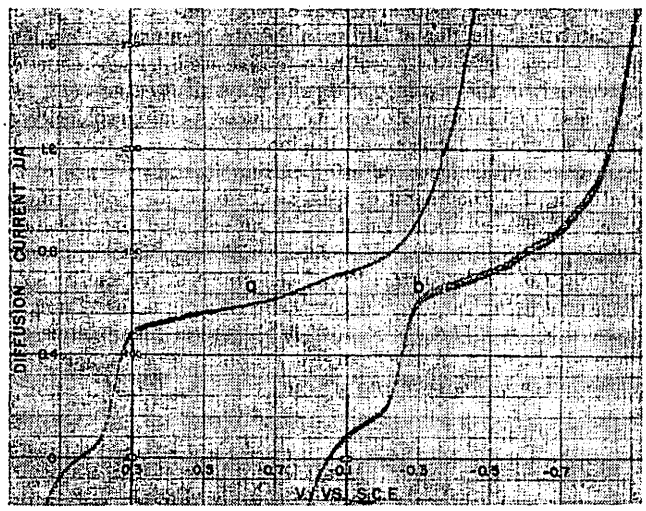

Figure 2. Polarograms of Hydrochloric Acid Solutions Containing Varying Concentrations of Sodium Nitrite

a. **0.1 *M* hydrochloric acid, 0.2 *M* sodium chloride, and 8.75 × 10^{-5} *M* sodium molybdate**
b. **0.1 *M* hydrochloric acid, 0.2 *M* sodium chloride, 8.75 × 10^{-5} *M* sodium molybdate, and 1 × 10^{-4} *M* sodium nitrite**

By comparing the reduction currents of nitrate as obtained by Johnson and Robinson (*4*) with those of nitrite as given in Table I, it was readily apparent that nitrite, for a given concentration, gave the greater current enhancement in the presence of molybdate. This may have been due to the fact that nitrite is more easily reducible than is nitrate. Greater ease of reduction of nitrite also accounts for the catalytic determination of nitrite at a smaller negative applied potential.

From the results in Table I, it is obvious that, as in the case of nitrate, the current-molarity ratio for nitrite was not constant and the reduction current was apparently not diffusion controlled. Consequently, the Ilkovič (*3*) equation could not be applied here to calculate the number of electrons involved in the reduction process and thus to determine the reduction products.

It has been proposed by Johnson and Robinson (*4*) that the current enhancement with nitrate is due to the chemical reaction between nitrate and the molybdenum(III) formed at the dropping mercury electrode. Nitrate oxidizes molybdenum(III) to molybdenum(V) which is then reduced again at the cathode, resulting in an increase in current. The molybdate-catalyzed reduction of nitrite can also be explained by this type of mechanism, with the difference that the current enhancement starts at the applied potential where molybdenum(VI) is reduced to molybdenum(V). This suggests that molybdenum(V), formed at the cathode, is oxidized by nitrite to molybdenum(VI) which is reduced again at the dropping mercury electrode. The oxidation of molybdenum (V) to molybdenum(VI) by nitrite but not by nitrate also indicates that nitrite is more easily reducible than is nitrate.

Since the increase in current is dependent upon chemical reaction, a higher current value is expected with a faster reaction rate, which is in agreement with the observed current enhancement with nitrite. When the applied potential is more negative than −0.3 volt *vs.* SCE, molybdenum(VI) is reduced at the electrode to molybdenum(III), and the chemical reaction between nitrite and molybdenum,(III) likewise gives an increase in current at these potentials.

When the nitrite concentration was greater than 2 × 10^{-3} *M*, a minimum was observed in the polarogram at a potential of about −0.18 volt *vs.* SCE with a preceding maximum at zero applied potential. A typical polarogram is shown in Figure 1, *c*. The cause of this maximum and minimum is unknown. It is different from the usual maximum with the dropping mercury electrode, caused by stirring effect of the growing drop or by adsorption of electroactive material on the electrode surface, since it is quite reproducible and is a function of the nitrite concentration. Kolthoff and Parry (*8*) also observed minima, probably of a similar nature, in the polarographic reduction of molybdate and hydrogen peroxide. They too offered no explanation as to the cause.

Catalytic Reduction of Nitrite and Nitrate in Chloride Medium. It was found that in hydrochloric acid–sodium chloride medium the current enhancement caused by nitrite in the presence of molybdate was greater than that in sulfate medium. Polarograms were obtained for a series of different nitrite solutions in a medium containing 0.1 *M* hydrochloric acid, 0.2 *M* sodium chloride, and 8.75 × 10^{-5} *M* sodium molybdate, as shown in Figure 2, in which curve *a* was obtained with molybdate only and curve *b* with nitrite present. The current enhancement also started at the applied potential where molybdenum(VI) was reduced to molybdenum(V), as in the case of the sulfate medium. The minimum phenomenon was also observed when the nitrite concentration was greater than 4 × 10^{-3} *M*.

The currents could be measured at the potentials of −0.15 and −0.40 volt *vs.* SCE. The results obtained at −0.40 volt *vs.* SCE are given in Table II. Because of the greater current enhancement in chloride medium, it was possible to determine smaller nitrite concentrations in chloride than in sulfate solutions.

Other concentrations of sodium chloride, hydrochloric acid, and sodium molybdate were investigated regarding their effects on the current. While variations in the sodium chloride concentration were found to have no effect, an increase in hydrogen ion concentration increased the current enhancement considerably. Although the sensitivity of the method may be increased by increasing the hydrogen ion concentration, it was felt undesirable to operate at a higher hydrogen ion concentration for nitrite solutions, because of the resulting decrease in stability of the nitrous acid solutions and because ample sensitivity was obtained at a hydrogen ion concentration of 0.1 *M*.

It was also found that varying molybdate concentration did not affect the current enhancement when the nitrite concentration was small, but, for high nitrite concentrations, higher current enhancement values were obtained with higher molybdate concentrations. It was determined that a molybdate concentration of 8.75 × 10^{-5} *M* was the most suitable for this work since the

Table II. Catalytic Determination of Nitrite in Hydrochloric Acid Solution[a]

Nitrite Molarity	Reduction Current for Nitrite, μa.	$K = i/C$, μa./Molarity
1.50 × 10^{-4}	0.025	1667
2.50 × 10^{-4}	0.063	2520
5.00 × 10^{-4}	0.080	1600
7.50 × 10^{-4}	0.129	1720
1.00 × 10^{-3}	0.178	1780
2.00 × 10^{-3}	0.238	1190
4.00 × 10^{-3}	0.324	810
6.00 × 10^{-3}	0.387	645
8.00 × 10^{-3}	0.475	594
1.00 × 10^{-2}	0.550	550
2.00 × 10^{-2}	0.891	446
3.00 × 10^{-2}	1.290	430
4.00 × 10^{-2}	1.645	411
5.00 × 10^{-2}	2.015	403

[a] 0.1 *M* hydrochloric acid, 0.2 *M* sodium chloride, 8.75 × 10^{-5} *M* sodium molybdate. $m^{2/3}t^{1/6}$ = 1.53 mg.$^{2/3}$ sec.$^{-1/2}$ Residual current at −0.4 volt *vs.* SCE = 0.425 μa.

Table III. Catalytic Determination of Nitrate in Chloride Solution[a]

Nitrate Molarity	Reduction Current for Nitrate, μa.	$K = i/C$, μa./Molarity	Reduction Current for Nitrate[b], μa.
5.00×10^{-5}	0.050	1000	...
1.00×10^{-4}	0.083	830	0.028
2.00×10^{-4}	0.153	765	0.042
4.00×10^{-4}	0.240	600	0.080
6.00×10^{-4}	0.303	505	...
8.00×10^{-4}	0.360	450	...
1.00×10^{-3}	0.408	408	0.165
2.00×10^{-3}	0.641	321	...
3.50×10^{-3}	0.893	255	...
5.00×10^{-3}	1.085	217	0.598

[a] 0.1 *M* hydrochloric acid, 0.2 *M* sodium chloride, 8.75×10^{-5} *M* sodium molybdate. $m^{2/3}t^{1/6} = 1.53$ mg.$^{2/3}$ sec.$^{-1/2}$ Residual current at −0.75 volt *vs.* SCE = 0.583 μa.
[b] Data of Johnson and Robinson (*4*) for nitrate in 0.1 *M* sulfuric acid, 0.2 *M* sodium sulfate, 8.75×10^{-5} *M* sodium molybdate, at −0.75 volt. *vs.* SCE $m^{2/3}t^{1/6} = 1.30$ mg.$^{2/3}$ sec.$^{-1/2}$

maximum galvanometer sensitivity could be utilized for low concentrations of nitrite.

Since the current enhancement from nitrite was greater in chloride than in sulfate solutions, it was thought desirable to obtain data for nitrate in chloride medium. A typical polarogram is shown in Figure 3. From the results, it was found that any applied potential more negative than −0.40 volt *vs.* SCE could be used for measuring the current enhancement from nitrate. The results at −0.75 volt *vs.* SCE are shown in Table III. The data of Johnson and Robinson (*4*) for nitrate in sulfate medium are also given in Table III for comparison. It is seen that in chloride medium, nitrate like nitrite gave a greater current enhancement than in sulfate solutions. This suggests that the reaction rate between molybdate and nitrate or nitrite was faster in chloride than in sulfate medium. In a chloride solution, a minimum nitrate concentration of 2×10^{-5} *M* may be detected, as compared to 1×10^{-4} *M* in sulfate medium.

Catalytic Determination of Nitrate and Nitrite in a Mixture. By utilizing the fact that nitrite gives a current enhancement at the applied potential where molybdenum(VI) is reduced to molybdenum(V) whereas nitrate does not, it was possible to determine nitrite and nitrate in a mixture. The concentration of nitrite can be estimated by determining the current at −0.15 volt *vs.* SCE in either a sulfate or a chloride solution. However, chloride medium is recommended because of the higher sensitivity. Nitrate in the mixture could not be determined directly even at a potential great enough to form molybdenum(III) because of the lack of additivity of the current enhancements caused by nitrate and nitrite. However, by oxidizing nitrite to nitrate with hydrogen peroxide, as described by Keilin and Otvos (*6*), the total concentration of nitrate could be estimated. Then by subtracting the nitrite concentration previously determined, nitrate in the original mixture could be obtained.

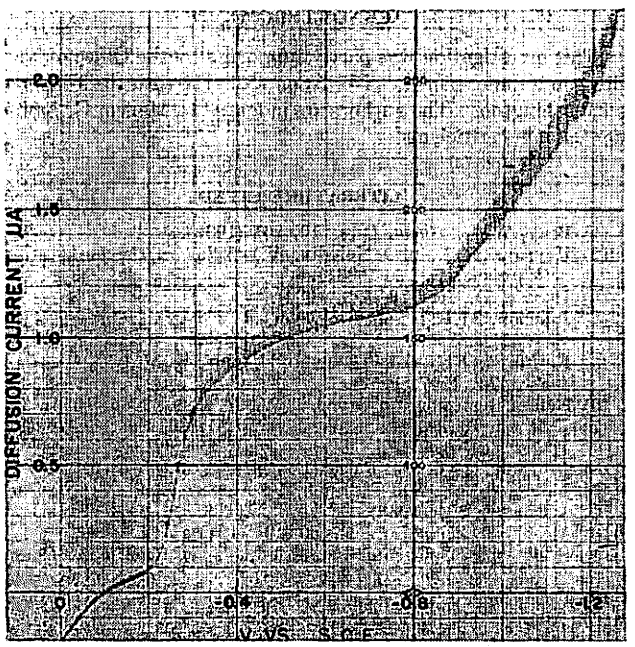

Figure 3. Polarogram of Solution Containing Nitrate in Chloride Solution

0.1 *M* hydrochloric acid, 0.2 *M* sodium chloride, and 1.50×10^{-3} *M* sodium nitrate

Molybdate in the Presence of Nitrite. The enhancement in current was also applied to the determination of molybdate in the presence of nitrite. In a 2×10^{-3} *M* solution in nitrite, the lower limit of detectable molybdate concentration was found to be about 1×10^{-6} *M* and 5×10^{-6} *M* in sulfate and chloride solutions, respectively. This is considerably less sensitive than the determination of molybdate in the presence of nitrate, for which Johnson and Robinson (*4*) reported that as little as 4×10^{-7} *M* could be estimated.

Table IV. Diffusion Current of Nitrous Acid[a]

Nitrous Acid Molarity	Diffusion Current, μa.	$K = i/C$, μa./Molarity ($\times 10^{-3}$)
1.00×10^{-5}	0.114	11.4
4.00×10^{-5}	0.470	11.8
8.00×10^{-5}	0.902	11.3
1.00×10^{-4}	1.143	11.4
2.00×10^{-4}	2.238	11.2
5.00×10^{-4}	5.715	11.4
1.00×10^{-3}	11.43	11.4
2.00×10^{-3}	22.91	11.5
3.00×10^{-3}	34.13	11.4
4.00×10^{-3}	45.32	11.3
5.00×10^{-3}	56.98	11.4
	Average value	11.4

[a] 0.1 *M* sulfuric acid, 0.2 *M* sodium sulfate. $m^{2/3}t^{1/6} = 1.53$ mg.$^{2/3}$ sec.$^{-1/2}$. Residual current at −1.25 volts *vs.* SCE = 0.366 μa.

Polarography of Nitrous Acid. In the polarographic determination of nitrite in the presence of molybdate, a wave other than that of molybdate was observed to start at an applied potential of about −0.76 volt *vs.* SCE. This differed from the polarographic reduction of nitrite in the presence of uranyl in which no such wave had been observed by Keilin and Otvos (*6*). Heyrovský and Nejedly (*2*) had observed the same wave with an acidic solution of nitrite. This they attributed to the reduction of nitric oxide because they obtained the same wave when an alkaline solution, through which hydrogen gas had been bubbled after passing first through an acidic solution of nitrite, was acidified. They concluded that nitric oxide was reduced to ammonia by comparing the wave height with that of a thallous solution of the same concentration. They did not, however, report the half-wave potential nor the potential at which the current was measured.

Further studies regarding this wave were made in this investigation. It was determined that this wave occurred even in the absence of molybdate. Consequently, the use of molybdate was eliminated in later work and polarograms were obtained with acidic solutions of nitrite. It was found that this wave was independent of the nature of the mineral acid used and also of the supporting electrolytes. Studies were made of 0.1 and 0.2 *M* hydrochloric acid, 0.1 and 0.2 *M* sulfuric acid, mixtures of hydrochloric acid and sodium chloride, and of sulfuric acid and sodium sulfate solutions. Virtually the same results were obtained with only a slight difference in the half-wave potentials in sulfate and chloride media.

The applied potential of −1.25 volts *vs.* SCE was used to measure the reduction currents. The results are recorded in Table IV. It was observed that a well-defined wave was obtained only

when the concentration of nitrite was higher than 1×10^{-4} *M*, and that the wave was rather drawn out, extending from about −0.76 to −1.1 volts *vs.* SCE. A typical polarogram is shown in Figure 4. Despite the fact that the waves were not well-defined with low concentrations of nitrite, the current at −1.25 volts *vs.* SCE could be used as well for estimating the nitrite concentration as shown by the results in Table IV.

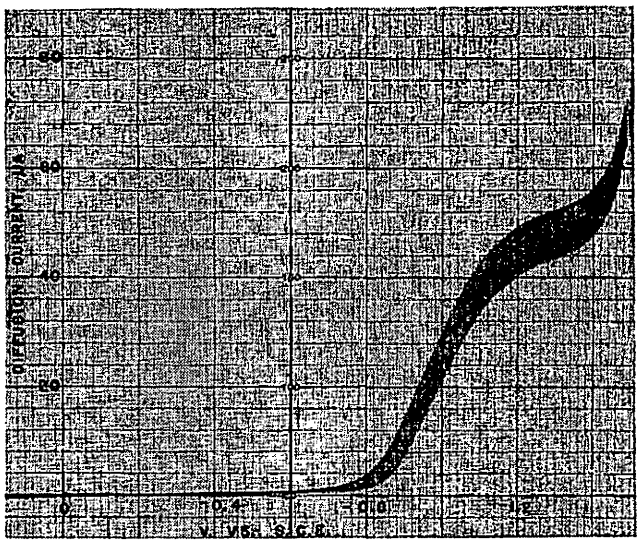

Figure 4. Polarogram of Solution Containing Nitrite in Sulfate Solution

0.1 *M* sulfuric acid, 0.2 *M* sodium sulfate, and 4.00×10^{-3} *M* sodium nitrite

The half-wave potential was determined to be −0.98 volt *vs.* SCE in a sulfate medium by plotting $\log \frac{i}{i_d - i}$ against the applied potential as illustrated in Figure 5. A value of $n = 0.4$ was determined from the slope of this straight line, indicating that the reduction was irreversible. A value of −0.96 volt *vs.* SCE was found as the half-wave potential in a chloride medium by a similar plot.

It was concluded that this wave was due to the reduction of nitrous acid rather than of nitric oxide, as proposed by Heyrovský and Nejedly (*2*), for several reasons.

According to Bray (*1*), nitrous acid decomposes according to either of the following equations.

$$3HNO_2 \longrightarrow 2NO + H^+ + NO_3^- + H_2O \quad (1)$$

$$2HNO_2 \longrightarrow N_2O_3 + H_2O \quad (2)$$

In the discussion of the decomposition of nitrous acid, Bray stated that Reaction 2 was the first step to occur. Thus, in the experiment of Heyrovský and Nejedly, both NO and N_2O_3 gases would be carried out of the acidic nitrite solution by the hydrogen gas. The latter oxide of nitrogen, when passed through an alkaline solution, would react with the alkali to form nitrite again. Upon acidification, the wave of nitrous acid would be obtained.

Nitric oxide is not very soluble in water, acidic or alkaline solutions. However, it reacts, though slowly, with alkali to form nitrite and nitrogen gas (*12*). If nitric oxide remained only as dissolved gas in the alkaline solution, it would certainly be washed out upon prolonged passing of an inert gas through the solution. In a repetition of the Heyrovský and Nejedly experiment, nitrogen gas was passed through a similar solution for 13 hours. It was assumed then that no nitric oxide remained dissolved. However, the wave was still obtained after the acidification of the alkaline solution. Furthermore, by colorimetric determination (*11*), nitrite was found to exist in this alkaline solution. Apparently, Heyrovský and Nejedly had not appreciated the fact that nitric oxide is only slightly soluble in alkaline solutions.

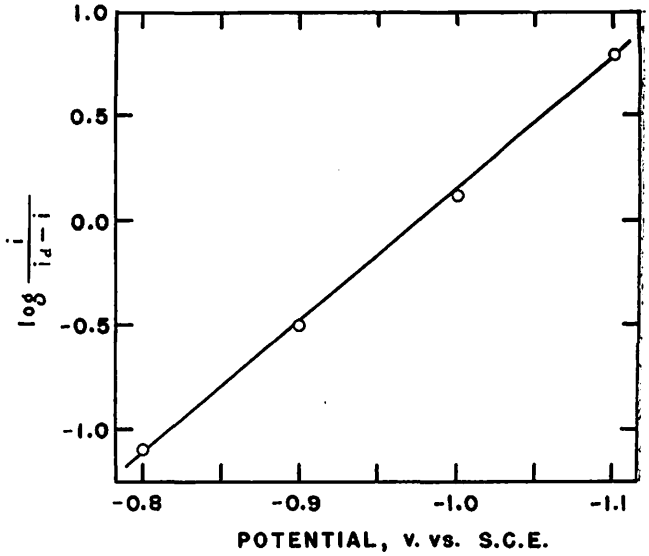

Figure 5. Analysis of Nitrous Acid Reduction Wave

0.1 *M* sulfuric acid and 0.2 *M* sodium sulfate

According to Moser (*10*), even water may slowly react with nitric oxide forming nitrous acid.

In view of the above considerations, it was concluded that the wave actually was due to nitrous acid. By assuming the diffusion coefficient for nitrous acid to be the same as that for nitrite ions, 1.92×10^{-5} cm.^{2} sec.$^{-1}$ (*5*), and by using the Ilkovič equation (*3*), a value of 2.8 was obtained for *n*. This agrees with the value obtained for the reduction of nitrite in the presence of uranyl ion by Keilin and Otvos (*6*) who interpreted this as indicating that the nitrite was reduced to nitrogen. Since this investigation was completed, Kaufman *et al.* (*5*) have reported a value of 3.4 for *n* or nitrite in the presence of uranyl ion.

ACKNOWLEDGMENT

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