

AN ABSTRACT OF THE THESIS OF

Jenaro A. Acuna for the degree of Master of Science  
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Title: Elimination of sulfide interference with the  
colorimetric determination of nitrite.  
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Abstract approved:

Louis I. Gordón

The interference of sulfide with the colorimetric determination of nitrite, by the method of Bendschneider and Robinson (1952), can be eliminated by passing the samples through a cationic exchange resin, Dowex 50W-X8, in the zinc form. This resin retains sulfide without affecting nitrite and the technique can be applied to automated methods.

Elimination of sulfide interference with  
the colorimetric determination of nitrite

by

Jenaro A. Acuna

A THESIS

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## Preface

This thesis has been written in manuscript format, according to a recommendation of the School of Oceanography in order to facilitate publication in a scientific journal. Therefore, the order of the traditional thesis has been changed in that no lists of tables or figures appear, and tables are placed in order, followed by the figures, at the end of the main text. This thesis, in the form of a paper, will be submitted to Marine Chemistry with Jenaro A. Acuna as first author, Stephen J. Hawkes as second, and Louis I. Gordon as third.

## 1. Introduction

The analytical problem caused by the interference with colorimetric nitrite analyses by sulfide, is a serious one which has been noted or addressed by several works, for instance, Timmer-ten Hoor, 1974; Riley et al., 1975; and Koike et al., 1978. Not all of the solutions to the problem are simple and easy to apply at sea. We offer here such a solution.

Two important processes produce nitrite in the marine environment: nitrification and nitrate reduction. Nitrification, carried out by bacteria, occurs even at low dissolved oxygen concentrations (Carlucci and McNally, 1969; Deuser, 1975; Hattori et al., 1978). Nitrate reduction by bacteria also occurs when dissolved oxygen is low or absent (Goering and Cline, 1970; Wada and Hattori, 1972; Soerensen, 1978). These two processes, which can take place in the water column as well as in the sediments, can produce nitrite as a transitory intermediate. Thus, nitrite can be formed close to anoxic environments which are also favorable for sulfur bacteria to produce sulfide by reduction of sulfate (Cline and Richards, 1969; Jaeschke et al., 1978). Therefore, nitrite and sulfide may coexist in such environments (Richards, 1970; Cohen, 1978). Another instance where nitrite and sulfide may occur together is the case of submarine hydrothermal springs (Corliss et al., 1979), which introduce large concentrations of sulfide (20-160  $\mu\text{M}$ ) into well oxygenated bottom waters, and the gradients from low to high concentrations of oxygen and conversely of sulfide, indicate that here too nitrite may form and briefly coexist with sulfide. Nitrite concentrations in seawater may range from zero to 10  $\mu\text{M}$  (Deuser, 1975; Hattori et al., 1978) and sulfide concentrations in anoxic basins may range from zero to several

hundreds  $\mu\text{M}$  (Deuser, 1975) ( $\mu\text{M}$  stands for micro moles of constituent per liter of solution).

In order to measure accurately nitrite concentrations in such marine locations, where interest in study of the nitrogen system is very intense, a way to eliminate the sulfide interference is most desirable.

The colorimetric determination of nitrite applied to seawater was discussed by Bendschneider and Robinson (1952). The method produces an azo compound whose absorbance is proportional to the initial nitrite concentration. It gives low results when sulfide is present in even small concentrations (Timmer-ten Hoor, 1974). One of the reasons might be the replacement of the intermediate diazonium nitrogen by a sulfhydryl group. The result is a decrease of the amount of diazonium compound, available to couple subsequently with an aromatic amine to form the azo dye (Morrison and Boyd, 1973). Timmer-ten Hoor (1974) has reported that this interference may be overcome by addition prior to analysis of an excess of a cation whose sulfide is highly insoluble, eg.,  $\text{Hg}^{+2}$  and  $\text{Cd}^{+2}$ . A possible reaction that might take place was mentioned by Yost and Russell (1944):  $\text{NO}_2^- + 3 \text{HS}^- + 3\text{H}_2\text{O} \rightarrow \text{NH}_4^+ + 3\text{S} + 5 \text{OH}^-$ .

To develop a technique for removal of sulfide in an automated continuous analyzer, the possibility of using a short ion exchange column to supply a cation that retains sulfide was considered. Among the possible cations, zinc (II) was preferred to cadmium (II) and mercury (II) because it is less toxic. A useful technique was developed which critically depends for its success upon the proper pretreatment of the column. The technique is simple, convenient and applicable to manual or automated methods ashore or at sea.



## 2. Materials and Procedures

In our work nitrite was determined by the method of Bendschneider and Robinson (1952) as reported in Strickland and Parsons (1972), in which it is converted into the diazo compound by reacting in an acid solution with sulfanilamide. The diazo compound is then reacted with N-1-naphthylethylene-diamide, forming an azo dye whose absorbance was measured at 543 nm in 1 cm rectangular cuvettes. A double beam Heath Spectrophotometer, model EU 700-56, was used at 300 microns slit width. For the continuous automated work we used a Technicon<sup>R</sup> AutoAnalyzer<sup>R</sup>, according to the method given by Atlas et al. (1971).

All reagents used were Analytical Reagent quality. Distilled water free of dissolved oxygen was obtained by boiling water for one-half hour. The starting water was the laboratory supply which had been distilled and then deionized. Finally, the water was stripped with "Ultra-Pure" nitrogen while cooling.

Standard sulfide solutions, 2.0 mM and 550  $\mu\text{M}$ , were prepared by dissolving sodium sulfide ( $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ ) in distilled water free of dissolved oxygen (OFDW). The solutions were kept under nitrogen atmosphere in dark serum cap bottles and were standardized iodometrically, according to the method of Broderius and Smith (1976). Determination of dissolved oxygen was carried out by Carpenter's modified Winkler technique (Carpenter, 1965).

Stock nitrite solutions, 500  $\mu\text{M}$ , were prepared by dissolving potassium nitrite in OFDW. The solutions were kept under nitrogen and in dark serum cap bottles to avoid photolysis (Zafiriou and True, 1979).

Standard nitrite solutions, 5.0  $\mu\text{M}$ , were prepared from the stock solution immediately before use by dilution with natural seawater, stripped with nitrogen for one hour, and kept under nitrogen atmosphere.

Experimental nitrite solutions, 4.8  $\mu\text{M}$ , were prepared by mixing 40.0 ml of standard nitrite solution with 2.0 ml of either distilled water or one of the standard sulfide solutions, and were analyzed immediately to avoid reduction of nitrite by sulfide (Yost and Russell, 1944).

We first performed experiments to verify Timmer-ten Hoor's (1974) results, but included zinc. Zinc (II) chloride, cadmium (II) chloride and mercury (II) chloride solutions were added to 5.0  $\mu\text{M}$  nitrite standards to produce a concentration of 100  $\mu\text{M}$  of cation. The nitrite standards contained sulfide in concentrations of zero, 25 and 50  $\mu\text{M}$ . The samples were filtered on 0.45  $\mu$  membrane filters to remove the sulfide precipitates and the reagents for the nitrite determination added. After a half hour the absorbance was measured. As references, samples of nitrite with no addition of heavy metals but of the same sulfide concentrations were run.

For the ion exchange work a strong cation exchange resin was chosen, Dowex 50W-X8, 60-140 mesh. Conditioning of the resin was done in the usual way, as given by Saville (1968), i.e., HCl and NaOH washes and treatment with  $\text{ZnCl}_2$  to put the resin into the zinc form. It will be shown later that this procedure was not adequate; so the next method utilized was basically that of Rieman and Walton (1970) which consisted in washing the resin successively with 1 M hydrochloric acid, 95% ethanol and 1 M sodium hydroxide, rinsing between reagents with enough distilled water to remove residues of the previous reagents. Qualitative tests with

phenolphthalein and methyl orange were applied to the effluents to check this. This sequence was repeated three times. The last washing was made with 2 M hydrochloric acid and the rinsing with distilled water was done until the effluent was neutral to methyl orange. The resin was placed in a column and a 1.5 M  $ZnCl_2$  solution was passed through it. Rinsings with distilled water were done until the effluent gave negative qualitative tests for zinc (Feigl et al., 1972). For the manual methods, burettes of 25 ml and 100 ml served as columns. Approximately 15 g of resin were used in each of the columns.

Samples of 50 ml of a nitrite standard (5.0  $\mu M$ ) were passed through a column prepared according to Saville (1968) under sufficient air pressure to obtain a flow rate of 10 ml/min. After 30 ml had passed, 20.0 ml were taken for the determination of nitrite. For reference 20.0 ml samples of nitrite standard not passed through the column were also analyzed. More samples of nitrite now containing sulfide concentrations of 23.0  $\mu M$  and 96  $\mu M$  were passed through the column and then analyzed for nitrite. Again, for reference, similar samples not passed through the column were analyzed. In a subsequent experiment, experimental nitrite solutions of zero, 25  $\mu M$  and 100  $\mu M$  sulfide were similarly passed through a column prepared according to Rieman and Walton (1970). Again, similar samples, not passed through the column, were also analyzed for nitrite.

For tests of the ion exchange technique with the automated method, columns 3.5 mm in diameter and 12 cm length (1 to 1.2 cc) were inserted ahead of the sample inlet to the AutoAnalyzer, using a three-way stopcock

to facilitate bypassing of the column. To avoid channeling, complete debubbling was necessary. Nitrite working solutions of zero and 100  $\mu\text{M}$  sulfide were determined after passing them through the column. Similar samples, not passed through the column, were also analyzed.

### 3. Results and Discussion

Our first experiments to confirm the observations of Timmer-ten Hoor (1974) are summarized in Table 1 and Figure 1. Comparable decreases in absorbance were obtained and precipitation of the sulfide by cadmium and mercury eliminated the interference. The less toxic ion, zinc (II), also was effective for this purpose. The single line shown for the cation-added data was fitted to the combined data from all three cation precipitations. It is important to note that the zinc, also, does not interfere with the nitrite analysis.

Having experimentally shown that zinc (II) is as effective as cadmium (II) or mercury (II), we proceeded to the cation exchange column work described earlier. Typical early results are shown in Table 2 and Figure 2. It is clear that sulfide removal by this column was only partially complete. However, there is again little if any adverse effect of the column itself, as shown by the data for samples 1 and 2. Further experiments with other batches of resin conditioned in the same way gave no significant retention of sulfide.

At this point, it was suspected that the resin might contain organic impurities which would impede the loading of the resin with cations; therefore, a new batch was conditioned including the 95% ethanol washings described in the preceding section. The results with the first column so prepared are given in Table 3, run 1; and Figure 3a. The resin now completely removed the sulfide and again, the column did not interfere with the analysis. This experiment was repeated two weeks later with a freshly prepared batch of resin (but with the same, and apparently somewhat decomposed, stock nitrite standard). The results confirm the first

run (Table 3, run 2; Figure 3b). This procedure appears to be quite useful for the manual colorimetric determination of nitrite in seawaters containing sulfide.

The results obtained using the zinc form cation exchange column ahead of the AutoAnalyzer are given in Table 4 and Figure 4. As before, the zinc column eliminates the sulfide interference but does not interfere in the nitrite analysis. The columns are easy to prepare and being small do not add appreciable dead volume to the AutoAnalyzer flow system. An important detail is to insure complete debubbling of the stream before passage through the column. Bubbles reaching the column caused serious channeling, in turn necessitating interruption of flow and off-line bubble removal. This was simple to do but interrupts the AutoAnalyzer operation. With care in this regard, the method offers a simple and reliable method of sulfide removal. In typical AutoAnalyzer methodology, one should be able to analyze approximately 3000 or more 100  $\mu\text{M}$  sulfide samples per small column before sulfide breakthrough occurs.

#### 4. Conclusions

1. Sulfide interference with the colorimetric determination of nitrite can be eliminated by passing the sample through a suitably prepared cation exchange resin in the zinc (II) form. Washing the resin with alcohol appears to be the critical pre-treatment step.
2. This column does not interfere with the nitrite analysis.
3. The technique can be applied to AutoAnalyzer or other continuous automated methods.

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Table 1. Sulfide interference with and without elimination by precipitation with three different cations. The 5.0  $\mu\text{M}$  nitrite standard was prepared in seawater of 28.3‰ salinity. See text for solution preparation and other details.  $C_1$  is the nominal nitrite concentration for sample No. 1;  $C_i$  is the apparent nitrite concentration of sample No. i.

SAMPLE No.	NUMBER OF MEASUREMENTS	CATION ADDED (100 $\mu\text{M}$ )	SULFIDE CONCN. ( $\mu\text{M}$ )	ABSORBANCE (AVERAGE $\pm$ STD.DEV.)	C <sub>i</sub> APPARENT NITRITE CONCN. ( $\mu\text{M}$ )	
						$\frac{C_i}{C_1}$
1	2	NONE	0	0.257 $\pm$ 0.001	5.0	1.00
2	2	Zn	0	0.258 $\pm$ 0.000	5.02	1.00
3	2	Cd	0	0.258 $\pm$ 0.000	5.02	1.00
4	2	Hg	0	0.258 $\pm$ 0.000	5.02	1.00
5	2	NONE	25.0	0.086 $\pm$ 0.001	1.67	0.33
6	3	Zn	25.0	0.258 $\pm$ 0.004	5.02	1.00
7	3	Cd	25.0	0.261 $\pm$ 0.003	5.08	1.02
8	3	Hg	25.0	0.264 $\pm$ 0.006	5.14	1.03
9	2	NONE	51.0	0.040 $\pm$ 0.002	0.78	0.16
10	3	Zn	51.0	.257 $\pm$ 0.004	5.00	1.00
11	3	Cd	51.0	0.258 $\pm$ 0.002	5.02	1.00
12	3	Hg	51.0	0.256 $\pm$ 0.003	4.98	1.00

Table 2. Partial elimination of sulfide from a 5.0  $\mu\text{M}$  nitrite solution, by means of a cation exchange column in the zinc form. The solutions were prepared in seawater as described in the text. Column characteristics: Length: 11.5 cm; internal diameter: 1.6 cm; flow rate: 10 ml/min. The column is described further in the text.  $C_1$  is the nominal nitrite concentration for sample No. 1;  $C_i$  is the apparent nitrite concentration of sample No. i.

SAMPLE No.	NUMBER OF MEASUREMENTS	ZINC COLUMN USED	SULFIDE CONCN. ( $\mu\text{M}$ )	ABSORBANCE		Ci APPARENT NITRITE CONCN. ( $\mu\text{M}$ )	$\frac{C_i}{C_1}$
				(AVERAGE $\pm$ STD.DEV.)			
1	3	NO	0	0.253	0.001	5.00 (Nominal)	1.00
2	3	YES	0	0.249	0.006	4.92	0.98
3	3	NO	23.0	0.134	0.009	2.65	0.53
4	3	YES	23.0	0.211	0.002	4.17	0.83
5	3	NO	96	0.035	0.005	0.69	0.14
6	3	YES	96	0.124	0.022	2.45	0.49

Table 3. Complete elimination of sulfide from a 4.8  $\mu\text{M}$  nitrite solution, using a cation exchange column in the zinc form, prepared by the method of Rieman and Walton (1970). The solutions were prepared in seawater as described in the text. Column characteristics: Length: 12 cm; internal diameter: 1.6 cm; flow rate: 10 ml/min.  $C_1$  is the nominal nitrite concentration for sample No. 1;  $C_i$  is the apparent nitrite concentration of sample No. i.

RUN	SAMPLE No.	NUMBER OF MEASUREMENTS	ZINC(II) COLUMN USED	SULFIDE CONC. ( $\mu\text{M}$ )	ABSORBANCE (AVERAGE $\pm$ STD.DEV.)	C <sub>i</sub>	$\frac{C_i}{C_1}$
						APPARENT NITRITE CONC. ( $\mu\text{M}$ )	
1	1	2	NO	0	0.240 $\pm$ 0.000	4.80	1.00
1	2	3	YES	0	0.243 $\pm$ 0.004	4.86	1.01
1	3	3	NO	26.0	0.083 $\pm$ 0.001	1.66	0.35
1	4	3	YES	26.0	0.239 $\pm$ 0.002	4.78	1.00
1	5	3	NO	92	0.000 $\pm$ 0.000	0.00	0.00
1	6	3	YES	92	0.240 $\pm$ 0.001	4.80	1.00
2	1	3	NO	0	0.196 $\pm$ 0.002	4.80	1.00
2	2	3	YES	0	0.197 $\pm$ 0.001	4.82	1.00
2	3	3	NO	23.4	0.049 $\pm$ 0.006	1.20	0.25
2	4	3	YES	23.4	0.197 $\pm$ 0.000	4.82	1.00
2	5	3	NO	97	0.000 $\pm$ 0.000	0.00	0.00
2	6	3	YES	97	0.197 $\pm$ 0.001	4.82	1.00

Table 4. Elimination of sulfide from a 4.8  $\mu\text{M}$  nitrite solution in seawater, using a cation exchange resin in the zinc form before the Auto-Analyzer inlet. Column characteristics: Length: 9.7 cm; internal diameter: 3.5 mm; flow rate: 3.9 ml/min. See text for column preparation and other experimental details.  $C_1$  is the nominal nitrite concentration for sample No. 1;  $C_i$  is the apparent nitrite concentration of sample No. i.

SAMPLE No.	NUMBER OF MEASUREMENTS	ZINC (II) COLUMN USED	SULFIDE CONCN. ( $\mu\text{M}$ )	ABSORBANCE (AVERAGE $\pm$ STD.DEV.)	C <sub>i</sub> APPARENT NITRITE CONCN. ( $\mu\text{M}$ )		$\frac{C_i}{C_1}$
1	4	NO	0	0.070 $\pm$ 0.002	4.80	1.00	1.00
2	4	YES	0	0.069 $\pm$ 0.003	4.73	0.99	0.99
3	4	NO	99	0.059 $\pm$ 0.002	4.05	0.84	0.84
4	4	YES	99	0.070 $\pm$ 0.004	4.80	1.00	1.00

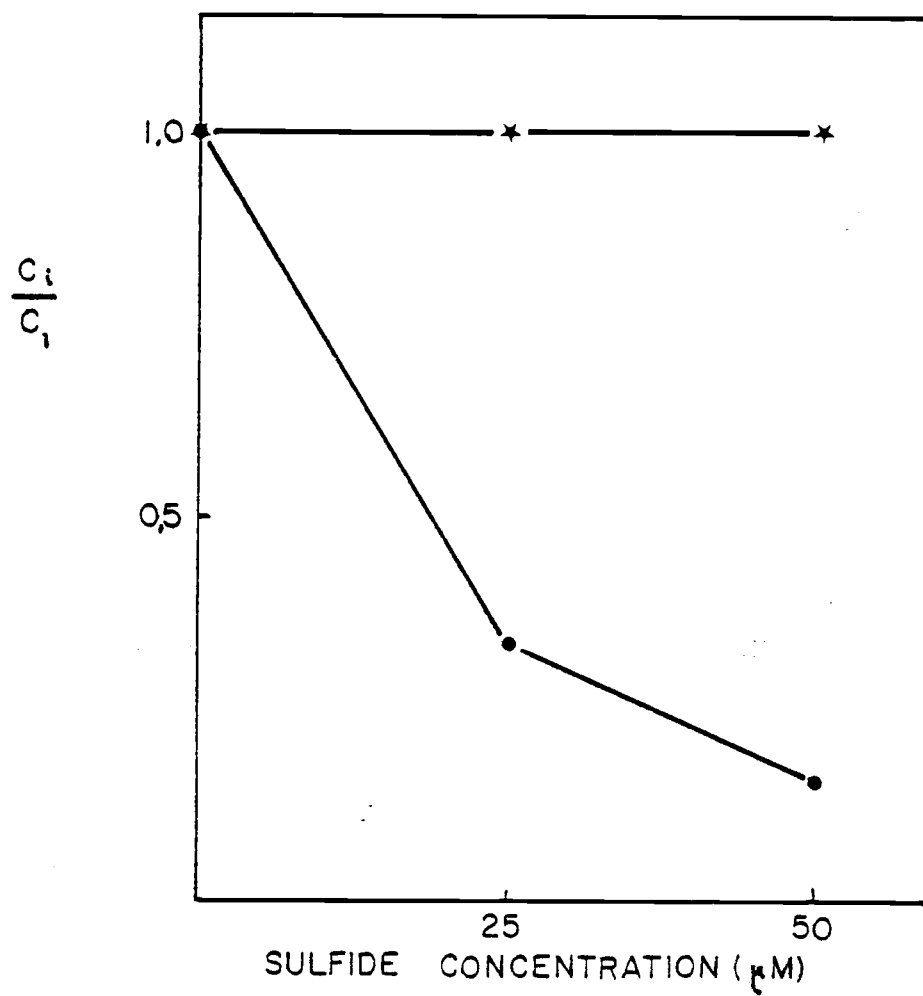


Figure 1. Elimination of the sulfide interference with the colorimetric determination of a 5.0  $\mu\text{M}$  nitrite solution in sea water, by means of three different cations.

- \* Addition of 100  $\mu\text{M}$  in Zn(II), Cd(II) and Hg(II)
- No cation added

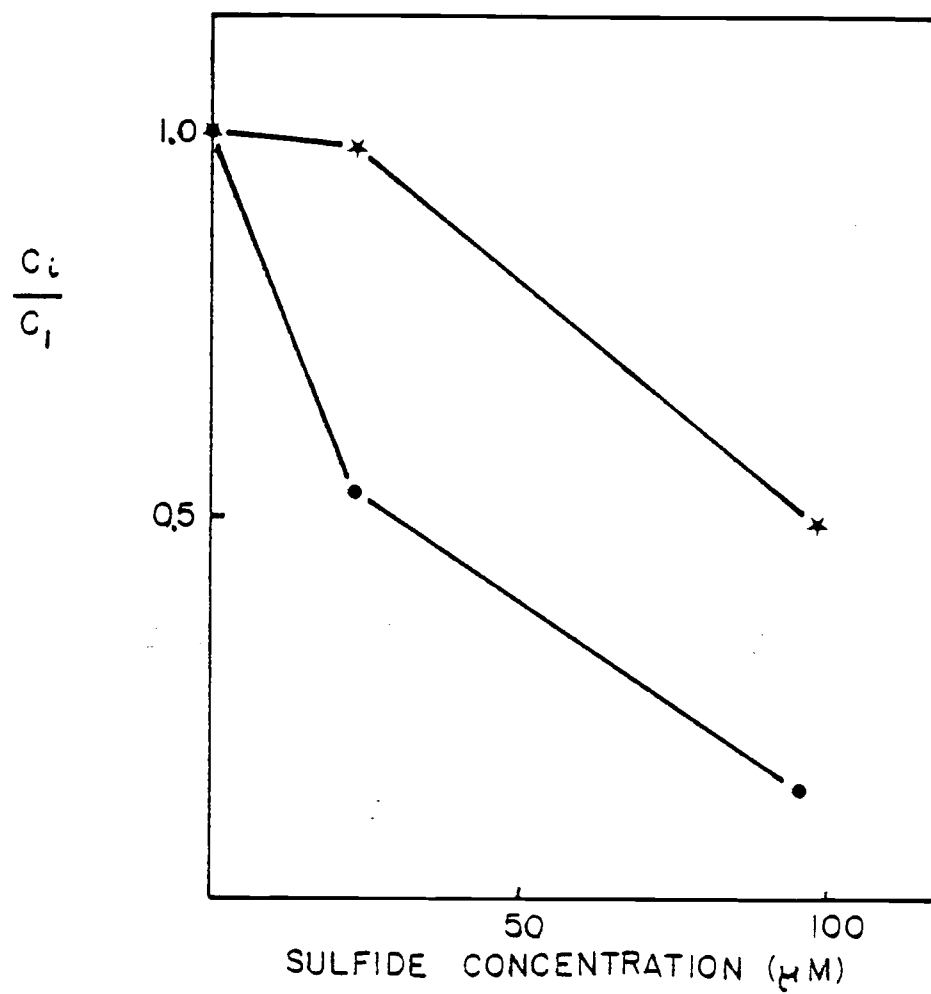


Figure 2. Performance of a simple cation exchange column, in the zinc form, in the elimination of sulfide from a 5.0 $\mu\text{M}$  nitrite solution.

★ After passing through the column

● Without the column

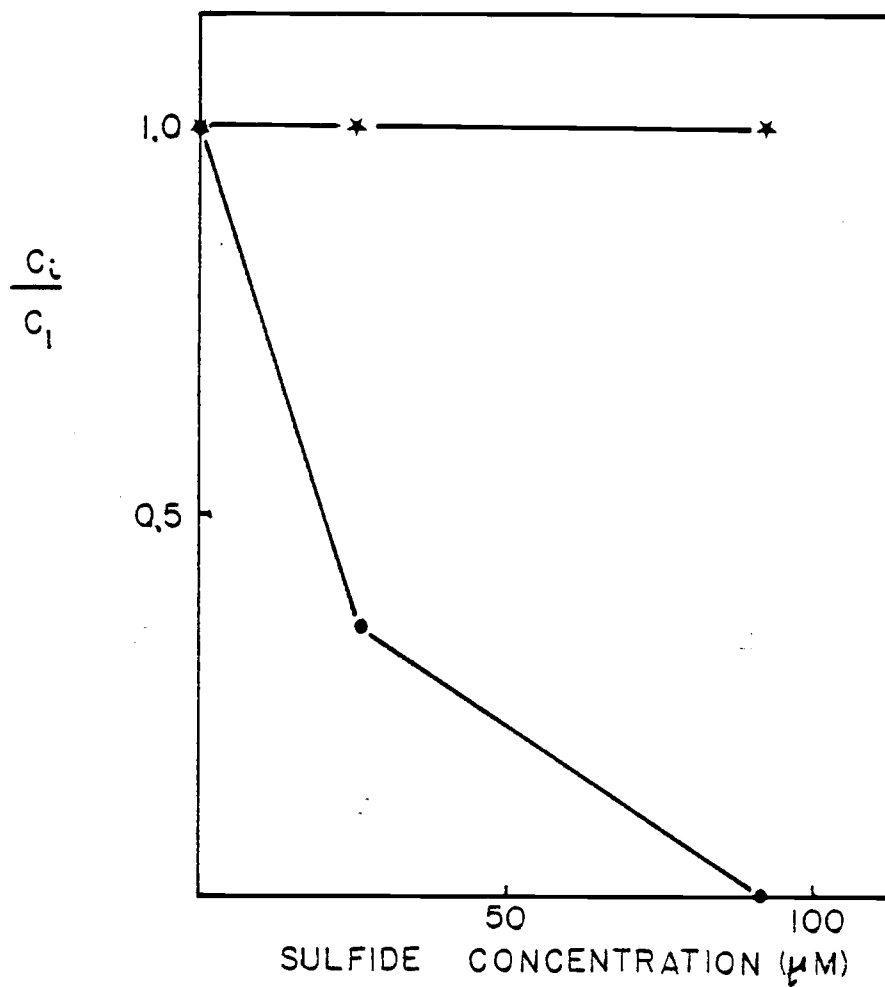


Figure 3a. Elimination of a sulfide interference with the colorimetric nitrite determination in sea water, by means of a zinc(II) cationic exchange column. Run 1.

★ After passing through the Zn(II) column

● Without the column



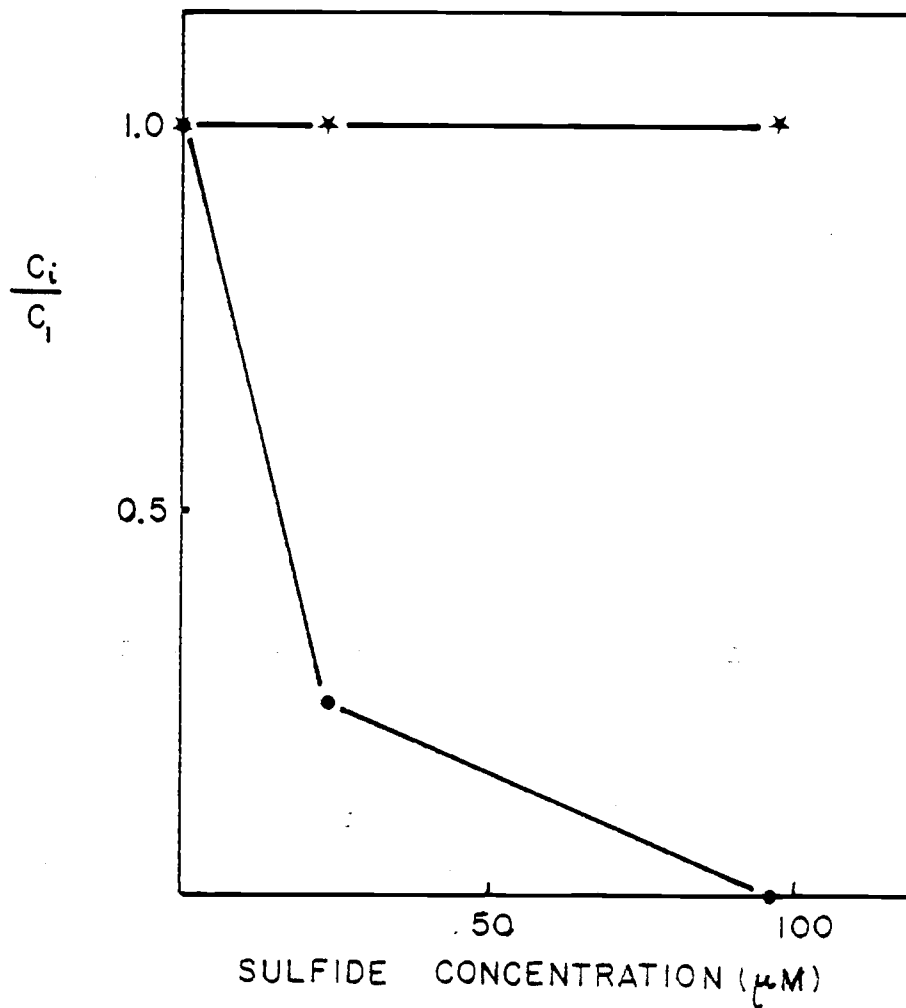


Figure 3b. Run 2. See text for details.

- ★ After passing through the zinc(II) column
- Without the column

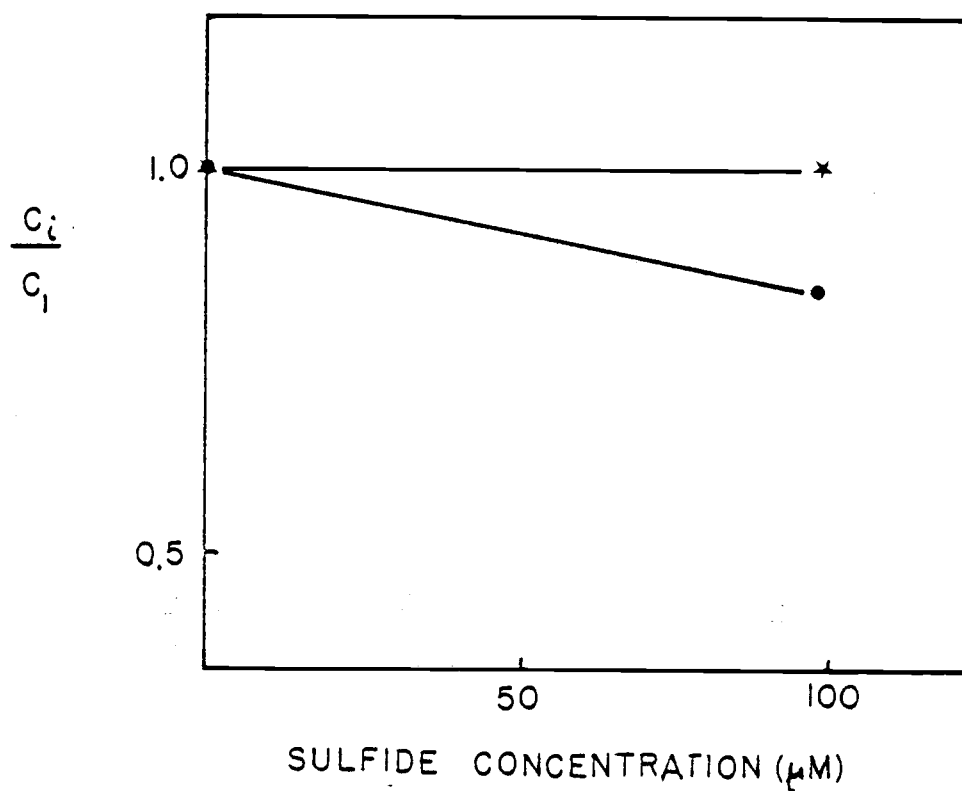


Figure 4. Elimination of a sulfide interference from a 4.8  $\mu\text{M}$  nitrite solution, by means of a zinc(II) cation exchange column. See text for details.

- ★ After passing through the zinc(II) column
- Without the column