

Applications Outside the Field of Colorimetry. The expressions presented herewith are not limited to colorimetry. Completely analogous relations have been discerned in several protein adsorption phenomena and in the quantitative treatment of enzyme-substrate systems which do not show strict adherence to zero-order kinetics (2). Indeed, by appropriate approximations with suitable geometric progressions, a number of linear equations may be derived for enzyme systems acting in the zone of transition kinetics (2).

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Spectrophotometric Determination of Cobalt with 2-Nitroso-1-naphthol-4-sulfonic Acid

WARREN M. WISE and WARREN W. BRANDT

Department of Chemistry, Purdue University, Lafayette, Ind.

The investigation was undertaken to ascertain whether the color of the water-soluble red complex which is formed when cobalt(II) reacts with 2-nitroso-1-naphthol-4-sulfonic acid could be employed as the basis for a spectrophotometric method for the quantitative determination of the metal. The system was found to obey Beer's law between the concentration limits of 4.00×10^{-8} to $1.25 \times 10^{-4} M$ cobalt(II) and remain stable with respect to time and temperature. The effects of pH, reagent concentration, and diverse ions—chiefly iron(III), copper(II), and nickel(II)—were studied. Procedures for effectively removing nickel(II) and preventing the interferences of concentrations of iron(III) or copper(II) up to $1.00 \times 10^{-3} M$ were developed. The method is convenient, sensitive, reproducible, accurate, and precise.

THE most popular analytical procedure that exists for determining minute amounts of cobalt is based on the spectrophotometric measurement of the amount of color which is produced when cobalt(II) reacts with nitroso R salt (4). This method has been applied to many types of materials, including soils, grasses, steels, and carbides (7). However, if reproducible results are desired, appreciable quantities of iron(III) and copper(II) must first be removed (3). Also, the amount of time the solution is boiled after the reagent and nitric acid are added and the final concentration of the acid are important factors which can affect the intensity of the color (3).

Since 2-nitroso-1-naphthol-4-sulfonic acid has a chelating system which is almost identical with the one possessed by the nitroso R salt, it seemed desirable to investigate the known color reaction of the former with cobalt(II). The reaction was first mentioned by Hoffman (2). Some 50 years later Sarver (5) described the color reactions of the reagent with iron(III), iron(II), cobalt(II), nickel(II), and copper(II). These experiments were conducted to develop qualitative spot tests only, and no effort was made to adapt them for use as spectrophotometric procedures. Consequently, an investigation was undertaken to determine whether the color reaction of cobalt(II) with the reagent could be utilized as the basis for a convenient, sensitive, and quantitative spectrophotometric method.

APPARATUS AND REAGENTS

A General Electric automatic recording spectrophotometer

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operating on a band width of 10 m μ and equipped with two matched 1-cm. cells was used for all spectrophotometric measurements. A Beckman Model H-2 pH meter was utilized to indicate pH values. During the investigation the precipitates which were formed, because of the presence of diverse ions, were separated from the solutions with the aid of a centrifuge.

The reagent, 2-nitroso-1-naphthol-4-sulfonic acid, was prepared and purified from 1-naphthol-4-sulfonic acid (National Aniline), sodium nitrite, and hydrochloric acid according to published techniques (8). The required amount of purified 2-nitroso-1-naphthol-4-sulfonic acid was dissolved in distilled water so that the reagent concentration was $1.00 \times 10^{-2} M$. All other solutions were prepared from reagent grade compounds and distilled water. Citric acid was always utilized as the solid when it was used to complex diverse ions.

Table I. Effect of Ammonium Ion Concentration on Absorbance of the System

[Cobalt(II) = $5.00 \times 10^{-5} M$; reagent = $6.00 \times 10^{-4} M$; pH = 7.0]

Molar Concn. of (NH ₄) ⁺	Absorbance of System at 525 M μ
0.00	0.720
0.01	0.660
0.10	0.610
1.00	0.600

EXPERIMENTAL

Effect of pH on System. Cobalt(II) will react with 2-nitroso-1-naphthol-4-sulfonic acid to produce a water-soluble red complex. Figure 1 shows that at 525 m μ the absorbance of an aqueous solution of cobalt(II) and reagent, when measured against a reagent blank, increases as the pH is increased from 4.5 to 6.0; it then remains constant between 6.0 and 10.0. Figure 2 exhibits the effect of pH on the spectrophotometric properties of an aqueous solution of the reagent. In the latter case a distilled water blank was employed. Examination of Figures 1 and 2 revealed that pH 7.0 is most suitable. At this value the absorbance at 525 m μ which is due to the cobalt complex is developed to a maximum and that of the reagent is a minimum. Dilute sodium hydroxide instead of ammonium hydroxide was chosen for increasing the pH, for an increase in the concentration of the ammonium ion at constant pH was found to cause a decrease in the absorbance of the system at 525 m μ (Table I). This effect can be attributed to the formation of complexes of cobalt and ammonia.

Effect of Reagent Concentration. A study was undertaken to examine the effect of the concentration of the reagent on the

absorbance of the system. It was found that if enough reagent is present to complex all of the cobalt(II), a further increase in the concentration of the reagent will not produce a significant change in the absorbance value at 525 $m\mu$ (Table II). On the basis of the results of this study the conclusion was reached that at pH 7.0 a reagent concentration of $6.00 \times 10^{-4}M$ is sufficient to complex any concentration of cobalt(II), up to $1.25 \times 10^{-4}M$.

Stability of System with Respect to Time and Temperature. An examination of the stability of the system with respect to time was conducted by allowing the sample and its blank to stand in the laboratory, and it was found that the absorbance at 525 $m\mu$ remained constant for at least 3 months. The solution used contained $1.00 \times 10^{-3}M$ cobalt(II) and $6.00 \times 10^{-4}M$ reagent at pH 7.0.

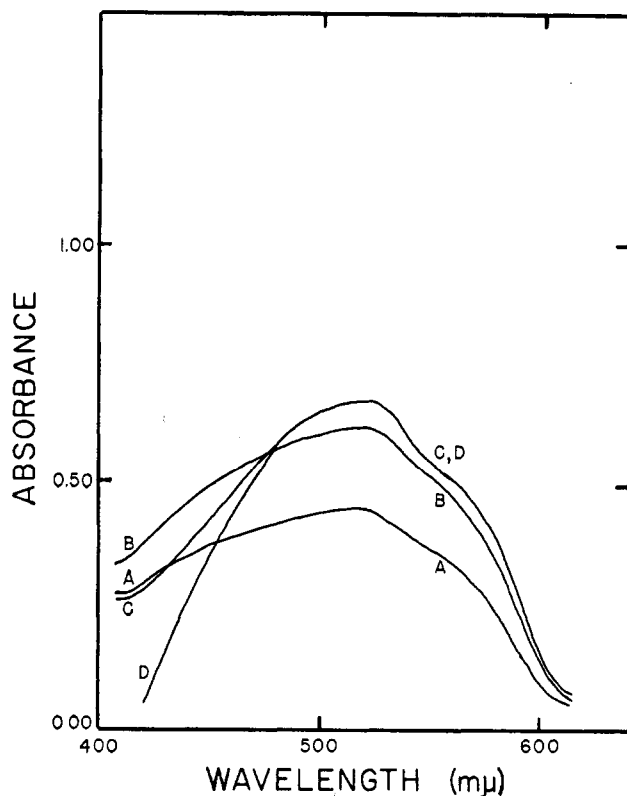


Figure 1. Effect of pH on Color of Cobalt Complex

Sample, $5.00 \times 10^{-3}M$ cobalt(II) and $2.00 \times 10^{-4}M$ reagent
Blank, $2.00 \times 10^{-4}M$ reagent
A. pH 4.5
B. pH 5.0
C. pH 6.0
D. pH 7.0-10.0

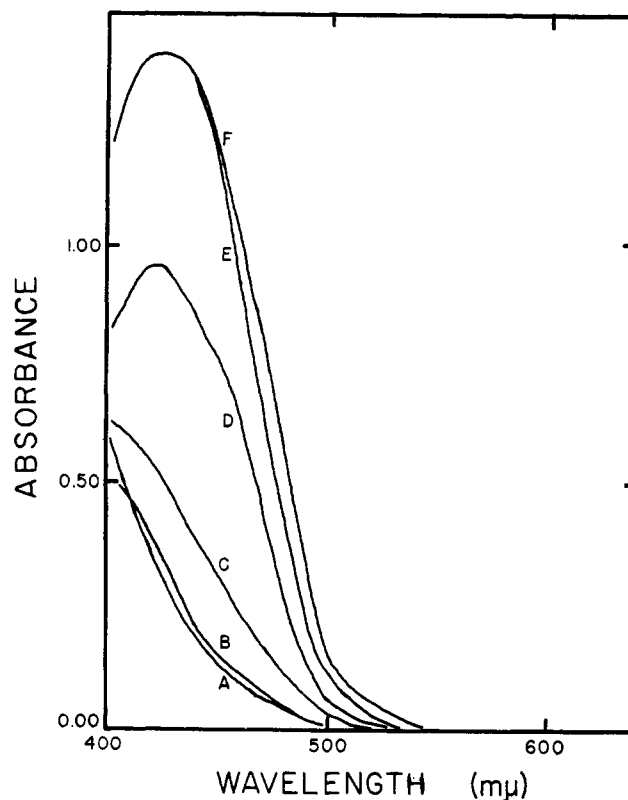


Figure 2. Effect of pH on Color of Reagent

Sample, $2.00 \times 10^{-4}M$ reagent
Blank, distilled water
A. pH 4.5
B. pH 5.0
C. pH 6.0
D. pH 7.0
E. pH 8.0
F. pH 9.0-10.0

The temperature of the cobalt(II) solution when the reagent is added and the pH adjusted is not a factor which will alter the absorbance of the system at 525 $m\mu$ so long as the temperatures of the solutions are brought to the same value before the respective absorbance values are measured. The reaction temperature range between the limits of 10° and 95° C. was investigated, and this variation did not affect the spectrophotometric properties of the system.

Preparation of a Calibration Curve. In order to determine whether the system obeys Beer's law, a calibration curve was prepared. It was found that in the absence of interfering ions the system conforms to Beer's law between the concentration limits of 4.00×10^{-6} and $1.25 \times 10^{-4}M$ cobalt(II) (0.24 to 7.5 p.p.m. cobalt).

Table II. Effect of Reagent Concentration on Absorbance of System

Molar Concn. of Cobalt(II) $\times 10^5$	Molar Concn. of Reagent $\times 10^4$	Moles of Reagent per Mole Cobalt(II)	Absorbance of System at 525 $m\mu$
1.00	6.00	60.0	0.160
1.00	14.0	140	0.160
2.50	3.00	12.0	0.375
2.50	6.00	24.0	0.380
2.50	14.0	56.0	0.370
5.00	3.00	6.00	0.715
5.00	6.00	12.0	0.725
5.00	14.0	28.0	0.720
7.50	6.00	8.00	1.055
7.50	14.0	18.7	1.060
10.0	6.00	6.00	1.375
10.0	14.0	14.0	1.370
12.5	6.00	6.80	1.680
12.5	14.0	11.2	1.690

SUGGESTED METHOD FOR DETERMINING COBALT(II) IN ABSENCE OF INTERFERING IONS

Pipet 3.00 ml. of $1.00 \times 10^{-2}M$ reagent to the solution which contains cobalt(II).

Dilute to almost 50 ml. with distilled water and adjust the pH to 7.0 with 0.01M sodium hydroxide. Finish diluting the sample to exactly 50 ml.

Prepare a blank by diluting 3.00 ml. of $1.00 \times 10^{-2}M$ reagent to 50 ml. with distilled water, and adjust the pH of the solution to 7.0 with 0.01M sodium hydroxide.

Measure the absorbance of the system at 525 $m\mu$ with a spectrophotometer and convert absorbance to cobalt(II) concentration with the aid of a previously prepared calibration curve.

Sensitivity and Applicability of Method. At

Table III. Results of Several Determinations

(Reagent = $6.00 \times 10^{-4} M$; pH = 7.0)

Molar Concn. of Cobalt(II) $\times 10^6$		Error, %
Taken	Found	
1.00	0.80	-20
2.00	1.85	-7.5
4.00	4.00	...
6.00	6.10	+1.7
8.00	7.85	-1.9
10.0	10.0	...
20.0	20.0	...
40.0	40.0	...
60.0	60.5	+0.8
80.0	80.0	...
100	101	+1.0
125	127	+1.6

525 $m\mu$ the molar absorptivity of the system is 1.44×10^4 liter-mole⁻¹-cm.⁻¹. Such a large value permits this method to be used for determining minute amounts of cobalt.

Table III contains the results of a series of twelve determinations which was undertaken to investigate the applicability of the method. On the basis of the data in Table III, it can be assumed that if the cobalt(II) concentration is between 4.00×10^{-6} and $1.25 \times 10^{-4} M$ and if no interfering ions are present, the method is accurate to $\pm 2\%$. However, under the same circumstances cobalt(II) concentrations as low as $1.00 \times 10^{-6} M$ (0.06 p.p.m.) can be determined with an accuracy of $\pm 20\%$. Using this method systems containing concentrations of cobalt(II) greater than $1.25 \times 10^{-4} M$ would have transmittances which are too small for spectrophotometric measurement in 1.00-cm. cells.

Accuracy and Precision of Method. Upon investigating the data in Table III it is evident that the ideal concentration range for this method is 1.00×10^{-5} to $1.00 \times 10^{-4} M$ cobalt(II) (0.6 to 6.0 p.p.m. cobalt). In order to demonstrate the accuracy and precision which can be obtained within these limits, determinations were conducted on ten solutions containing $1.00 \times 10^{-5} M$ cobalt(II) and ten solutions which contained $1.00 \times 10^{-4} M$ cobalt(II). The results are tabulated in Table IV. They show that the standard deviation (σ) is 0.005 of an absorbance unit at both of these concentrations. This corresponds to 0.02 p.p.m. cobalt.

Table IV. Results of Precision and Accuracy Determinations

(Reagent concn. = $6.00 \times 10^{-4} M$; pH = 7.0)

Expected Absorbance Value at 525 $m\mu$	
For system with $1.00 \times 10^{-5} M$ cobalt(II) = 0.160, obsvd. values	For system with $1.00 \times 10^{-4} M$ cobalt(II) = 1.370, obsvd. values
0.155	1.365
0.160	1.360
0.160	1.370
0.165	1.365
0.160	1.365
0.160	1.375
0.150	1.370
0.165	1.365
0.160	1.365
0.155	1.370
Std. deviation 0.005	0.005

Effects of Diverse Ions. A study was undertaken to determine which of the more common ions will interfere with this method. The procedure that was employed to examine these effects was the same as the one outlined earlier except that the diverse ion was added to the original cobalt(II) solution before carrying out the procedure. If a precipitate formed during this process it was settled by centrifuging. After the supernatant liquid was removed distilled water was added to the precipitate, and the precipitate was thoroughly dispersed by shaking. The mixture was again centrifuged and the liquid was decanted and added to the main solution. This practice was repeated until the

supernatant liquid was void of any color. Then the final pH and volume adjustments were made and the absorbance of the system was measured at 525 $m\mu$. Sometimes such precipitates will cause low results by adsorbing the red-colored cobalt-2-nitroso-1-naphthol-4-sulfonic acid complex, and repeated shakings with distilled water and subsequent centrifugings will not remove this adsorbed color. Aluminum(III), cadmium(II), and lead(II) hydroxides are very prone to do this. Table V lists the effects of some of the more common ions. An interference is defined as an alteration of at least $\pm 2\%$ in the absorbance value of a solution

Table V. Results of Investigation of Effects of Diverse Ions

No Interferences at Concentrations $10^{-2} M^a$ or Smaller	Interferences at Concentrations $10^{-3} M^b$ or Larger
F ⁻	Ce ^{+++c}
Cl ⁻	Fe ^{++c}
Br ⁻	Fe ^{+++c}
I ⁻	Ni ^{++c}
BO ₃ ⁻	Cu ^{++c}
CO ₃ ⁻⁻	Pb ^{++c}
HPO ₄ ⁻⁻	Al ^{+++c}
NO ₃ ⁻	Cd ^{++c}
NO ₂ ⁻	Hg ^{+++c}
SCN ⁻	Bi ^{+++c}
SO ₄ ⁻⁻	Sn ^{++++c}
SO ₃ ⁻⁻	Sb ^{+++c}
VO ₃ ⁻	As ^{+++c}
WO ₄ ⁻⁻	UO ₂ ⁺⁺
Oxalate ⁻⁻	CrO ₄ ⁻⁻
Citrate ⁻⁻	Cr ₂ O ₇ ⁻⁻
Tartrate ⁻⁻	CN ⁻
Mg ⁺⁺	Mn ⁺⁺⁺
Ca ⁺⁺	
Sr ⁺⁺	
Ba ⁺⁺	
Zn ^{++c}	

^a Highest limit investigated.
^b Lowest limit investigated.
^c Indicates the formation of a precipitate.
^d Mn⁺⁺ interferes if its concentration is $10^{-2} M$, but does not if its concentration is $10^{-3} M$.

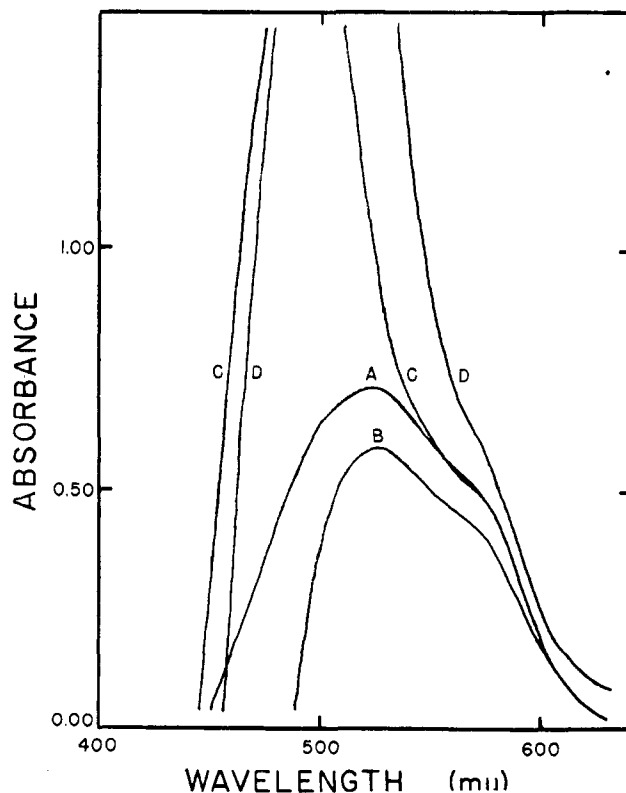


Figure 3. Interferences Caused by Iron(III), Nickel(II), and Copper(II)

- A. Cobalt(II), no interfering ions
- B. Interference caused by iron(III)
- C. Interference caused by nickel(II)
- D. Interference caused by copper(II)

at pH 7.0 containing $5.00 \times 10^{-5}M$ cobalt(II) and $6.00 \times 10^{-4}M$ reagent.

Figure 3 illustrates the interfering effects caused by $1.00 \times 10^{-3}M$ iron(III), copper(II), or nickel(II) upon a system containing $5.00 \times 10^{-5}M$ cobalt(II) and $6.00 \times 10^{-4}M$ reagent. Since the iron(III) and copper(II) distort the entire spectrophotometric curve of the cobalt-2-nitroso-1-naphthol-4-sulfonic acid complex, methods of complexing or removing these ions had to be found to determine satisfactorily the amount of cobalt(II) present.

SUGGESTED METHOD FOR MASKING CONCENTRATIONS OF COPPER(II) OR IRON(III) LESS THAN $10^{-3}M$

Attempts were made to complex iron(III) or copper(II) with fluoride, oxalate, tartrate, or citrate ions, and then determine the amount of cobalt(II).

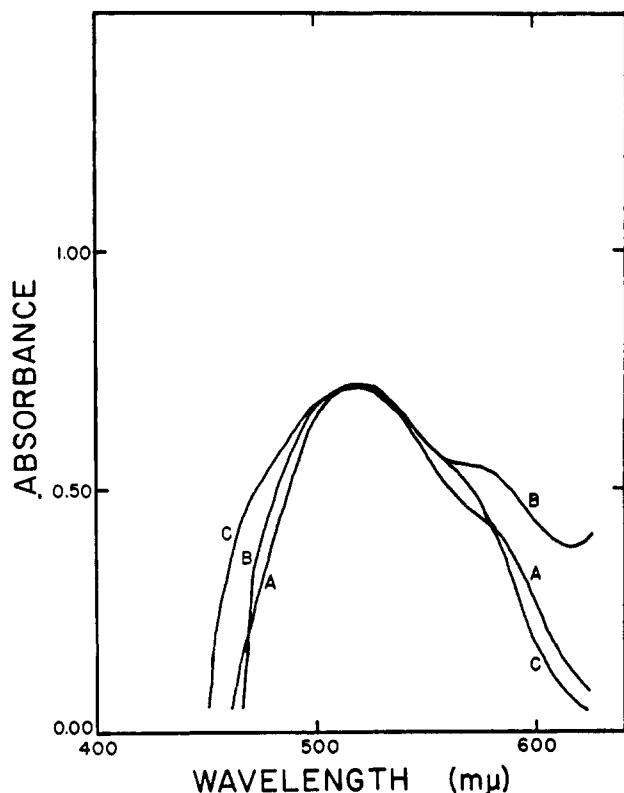


Figure 4. Removal of Interfering Effects of Iron(III) and Copper(II) with Citrate Ion

- A. Cobalt(II), no interfering ions
- B. Iron(III) complexed with citrate ion
- C. Copper(II) complexed with citrate ion

The citrate ion at a concentration of $0.1M$ appears to be the most efficient agent for complexing either $10^{-3}M$ iron(III) or $10^{-3}M$ copper(II); it will permit accurate absorbance measurements at $525 m\mu$ for a system containing $6.00 \times 10^{-4}M$ reagent and $5.00 \times 10^{-5}M$ cobalt(II) (Figure 4). However, it was found that if the cobalt(II) concentration is increased to $1.00 \times 10^{-4}M$ in the presence of $10^{-3}M$ iron(III) or $10^{-3}M$ copper(II), the reagent concentration must be increased to $1.00 \times 10^{-3}M$ in order to obtain the calibration curve value for the absorbance corresponding to $1.00 \times 10^{-4}M$ cobalt(II). This is attributed to the fact that in spite of the citrate ion the iron(III) or copper(II) can react with the 2-nitroso-1-naphthol-4-sulfonic acid and not enough reagent remains to complex all of the cobalt(II) and develop the color.

Removal of Iron(III) and Copper(II). If the concentration of iron(III) or copper(II) is greater than $1.00 \times 10^{-3}M$, $0.1M$ citrate ion will not serve as an efficient agent for completely alleviating the undesirable effects caused by these ions. Consequently, they must be removed before the reagent solution is added if accurate results are desired for the determination of cobalt(II). Iron(III) may be extracted from a $7.5N$ hydrochloric acid solution with the aid of isopropyl ether (1), and copper(II) is precipitated from an acidic solution with hydrogen sulfide. The hydrogen sulfide is then removed by boiling the acidic solution before the reagent is added and the pH increased. After these procedures the pH must be adjusted to approximately 3 by boiling or by adding base to prevent the destruction of the reagent when it is introduced into the solution containing the cobalt(II).

Removal of Nickel(II). None of the previously mentioned complexing agents were found to be satisfactory for preventing the interference of nickel(II). The only method which was found for this is to remove it. The procedure used to accomplish this is based on the facts that both nickel(II) and cobalt(II) form complexes with dimethylglyoxime, but the nickel complex is extremely insoluble in basic solutions while the corresponding cobalt complex is very soluble.

Nickel(II) is removed by precipitating it with an excess of dimethylglyoxime in the presence of ammonium hydroxide and filtering the mixture through a medium-pored sintered-glass crucible. The cobalt complex and excess dimethylglyoxime contained in the filtrate are then destroyed by adding concentrated sulfuric acid, evaporating to fumes of sulfur trioxide, and heating the fuming mixture until no evidence of any undestroyed carbon remains. More sulfuric acid is added when necessary. The solution is allowed to cool, diluted to a suitable volume, and the pH adjusted to about 3. The reagent is added and the usual procedure of developing and measuring the color is followed. During this process two important sources of error occur. A sufficient excess of dimethylglyoxime must be used to complex all of the cobalt(II) before it can be assumed that all of the nickel(II) has been precipitated (6); and if both cobalt(II) and iron(III) are present, a brown complex with the composition $FeCoC_{12}H_{19}N_6O_6$ will be precipitated. The latter can be prevented by removing the iron, as previously mentioned, or by reducing it with sulfur dioxide and carrying out the precipitation of the nickel(II) in the presence of tartrate ion (6).

SUGGESTED METHOD FOR DETERMINING COBALT(II) IN PRESENCE OF SMALL QUANTITIES OF NICKEL(II)

Figure 3 shows that the presence of nickel(II) does not cause the spectrophotometric curve of the cobalt-2-nitroso-1-naphthol-4-sulfonic acid complex to become distorted in the vicinity of $560 m\mu$. Consequently, if a molar ratio of nickel(II) to cobalt(II) of only 20 to 1 exists, a new procedure for determining cobalt could be developed by measuring the absorbance of the system at $560 m\mu$. The sensitivity of this spectrophotometric method for cobalt(II) would be about 20% smaller than if the absorbance measurements were taken at $525 m\mu$, but this sacrifice would be very insignificant in comparison to the advantages which would be gained by eliminating the time-consuming procedure of removing the nickel(II).

CONCLUSIONS

The results of this investigation reveal that the reaction of cobalt(II) with 2-nitroso-1-naphthol-4-sulfonic acid produces a water-soluble red complex which has an absorbance maximum at $525 m\mu$ and provides a useful method for the quantitative spectrophotometric determination of the element. Since the normal eye has a maximum sensitivity at about $550 m\mu$, this color could be used as the basis for a visual comparison method. The procedure given can be used to determine cobalt(II) in the

presence of small quantities of iron(III) or copper(II) after these foreign ions have been complexed with citrate ion. However, large quantities of iron(III), copper(II), and nickel(II) must be removed. The system is stable, independent of reagent concentration, and obeys Beer's law. The method is sensitive, reproducible, accurate, and precise. Consequently, its advantages render it very useful for determining minute amounts of cobalt.

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Measuring Wet Gel Strength of Coagulant-Dipped Films

Paper Dip Method

A. I. MEDALIA, H. B. TOWNSEND, and H. N. GROVER

Boston University, Boston, Mass., and General Latex and Chemical Corp., Cambridge, Mass.

A property of latex which is considered of fundamental importance in the latex industry is the so-called wet gel strength. This term connotes the tensile strength of the freshly set film, but hitherto there has been no adequate method of measurement of this property, since the film is so weak that it cannot readily be removed from the surface on which it is deposited. The present method is based upon the use of absorbent paper of low wet strength, impregnated with calcium nitrate, as a base for the deposition of a latex film. The strip of paper, which may be in a dumbbell shape, is dipped for 30 seconds and then removed from the latex, together with the adhering coagulant dipped film. This film is rinsed in water and then mounted on a special tensile testing machine designed for high elongation and low tensile. Elongation and tensile are measured at a standard rate of jaw separation of 20 inches per minute. These properties have been studied using natural and various synthetic latices. The effects of preulcanization, compounding, and dilution of these latices have been determined. Tensile properties of the film from a given latex have also been measured as a function of the time and conditions of drying of the film after deposition.

IN THE majority of applications of latex, such as dip goods, foam rubber, and castings, the first step is the formation of a continuous film of rubber in the desired shape. Some form of treatment is used—for example, chemical coagulation, heating or evaporation—whereby the individual rubber particles of the latex are brought in contact with one another, resulting in the establishment of a continuous latticelike phase of rubber suspended in, or permeated by, a continuous phase of water or serum. This system of two continuous phases is termed a wet gel. The tensile properties of this wet gel are far inferior to those of the dried polymer, and it is universally recognized in the latex industry (3) that the weakness of the wet gel imposes a severe limitation on the applicability of latex.

Despite the interest of the latex industry in the so-called wet gel strength, no adequate method has hitherto appeared by which this property can be measured and characterized. This fact is due primarily to the inherent weakness of the wet gel, which makes it difficult to remove a freshly dipped or coagulated film from the form on which it is cast or dipped—for example, a strip may be coagulant-dipped on a glass slide, but distortion of the film usually takes place when the film is removed from the slide.

The film may be cast on mercury, but it is difficult to obtain a film of definite shape in this manner.

In the present method, a latex film is deposited directly on a strip of paper of low wet strength by a coagulant dip process. The film assumes the shape of the paper, which can be made in a dumbbell shape for tensile testing. The moist strip of paper contained between the two layers of wet gel tears readily and so does not affect the measurement of the tensile properties. Owing to the low tensile strength of the wet gel, a testing machine of much greater sensitivity than that ordinarily employed in rubber testing must be used.

By this procedure, it is possible to study the tensile properties of fresh coagulant-dipped latex films. These films can also be carried through various procedures such as leaching and drying, and the tensile properties can be measured at any stage. When these properties are known it should be possible for the engineer

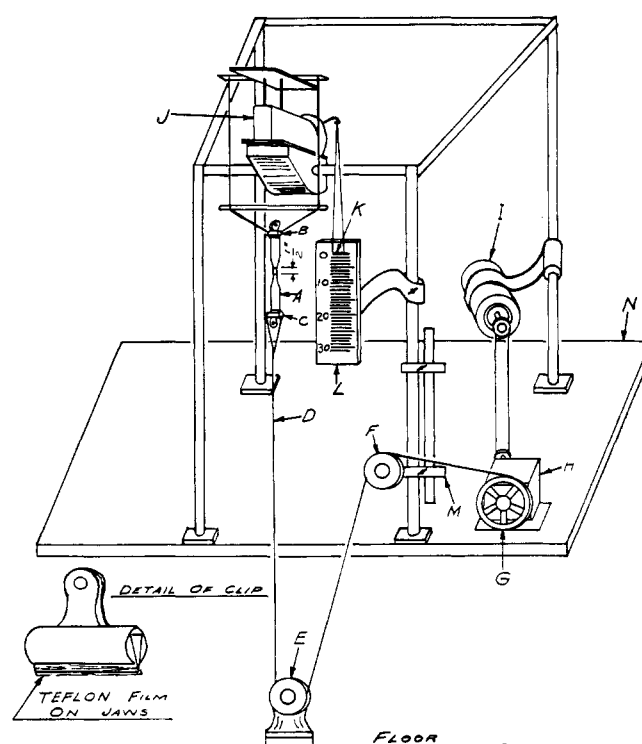


Figure 1. Tensile Test Apparatus