

The Spectrophotometric Determination of Scandium(III) with Chromazurol S^{*1}

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(Received August 8, 1966)

Chromazurol S reacts with scandium(III) in a weakly acidic medium to form one of two kinds of complexes according to the pH value of the medium. Their compositions were confirmed; one had a 1 : 2 combining ratio of scandium(III) and chromazurol S at pH 5.2 or higher, and the other, a 1 : 1 ratio at pH below 4.0. The color development of the 1 : 2 complex is more sensitive than the other. Therefore, it was applied to the determination of scandium(III). Several conditions for the color development were investigated, a calibration curve was made under the optimum conditions, and the influences of coexisting ions were investigated. The maximum absorption wavelength of the complex is 550 m μ against the reagent blank. The optimum pH for the color development is about 5.6. By adding a reagent in an amount four times the scandium concentration, the maximum color intensity is given. The color intensity decreases gradually during the first 1.5 hr, and then it remains constant for several hours. A linear relationship between the absorbance and the scandium concentration is obtained in the concentration range from 4 to 27 μ g per 10 ml. The value of the molar extinction coefficient of the complex at 550 m μ is 27000, and the sensitivity of the color reaction is 0.0017 μ g/cm² for log I_0/I = 0.001. The formation constant, K , of the complex is calculated to be 9.5×10^{11} under these conditions. Anions such as fluoride, phosphate, oxalate, tartrate, citrate, and EDTA interfere with the color development of the complex. Cations such as Al(III), Be(II), U(VI), Fe(II), Th(IV), Cu(II), Y(III), Fe(III), Zr(IV), and W(VI) interfere, too.

In recent years a number of chromogenic reagents have been used for the colorimetric determination of scandium(III), for example, pontachrome azure blue B,¹⁾ eriochrome cyanine R,^{2,3)} pyrocatechol violet,⁴⁾ xylenol orange,⁵⁻⁷⁾ α -(2,4-dihydroxyphenylazo)-pyridine,⁸⁾ arsenazo (neo-thorin),^{9,10)} arsenazo III,^{11,12)} glyoxal-bis-(2-hydroxyanil),¹³⁾

sulfonazo,¹⁴⁾ quercetin,¹⁵⁾ oxine and its derivatives,^{16,17)} alizarin red S,¹⁸⁻²¹⁾ tiron,²²⁾ propyl-fluorone,²³⁾ stilbazochrome,²⁴⁾ p -nitrobenzene azo-chromotropic acid,²⁵⁾ and anthrarufin-2,6-disulfonic acid (disodium salt).²⁶⁾

This paper will present a spectrophotometric method for the determination of scandium(III) based on a sensitive color reaction of scandium(III) with chromazurol S, which has a structure similar

^{*1} Presented at the 19th Annual Meeting of the Chemical Society of Japan, Yokohama, April, 1966.

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to pontachrome azure blue B and eriochrome cyanine R. Recently Dey and his co-workers have studied much about the use of chromazurol S as metallochromic indicator in complexometric titrations and as a reagent for the colorimetric determination of metallic ions.²⁷⁻³⁹ Chromazurol S, the sodium salt of 3''-sulfo-2'', 6''-dichloro-3, 3'-dimethyl-4-hydroxyfuchson-5, 5'-dicarboxylic acid, reacts with scandium(III) to form a stable reddish violet complex in a weakly acidic solution. Several conditions for the color development, the composition of the colored complex, and the influences of coexisting ions will be investigated.

Experimental

Apparatus. A Shimadzu model QR-50 spectrophotometer with 1-cm glass cells was used for all the absorbance measurements.

A glass electrode pH meter (Hitachi-Horiba model M-4) was employed for all the pH measurements. It was calibrated with certified standard buffer solutions.

Reagents. *Standard Scandium Solution.* A 1.0×10^{-3} M scandium solution was prepared by dissolving scandium nitrate anhydrate (G. R. grade, Mitsuwa Chemical Co., Ltd.) in distilled water. The solution contained 45 μ g of scandium(III) per milliliter.

Chromazurol S Solution. A 1.0×10^{-3} M solution of chromazurol S was prepared by dissolving Dotite Chromazurol S (Dojindo & Co., Kumamoto) in distilled water and by then filtering off the floating residue. According to Ueno,⁴⁰ this solution is very stable.

Buffer Solution. A 1.0 M hexamethylenetetramine solution was prepared by dissolving crystalline hexamethylenetetramine (Kanto Chemical Co., Inc.) in distilled water. Buffer solutions of various pH values were prepared by mixing 1.0 M hexamethylenetetramine and 1.0 N perchloric acid solutions in different ratios.

Other Reagents. All the other reagents were of reagent grade and were used without further purifications.

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Standard Procedure for Scandium Determination. An aliquot of a standard scandium solution containing from 5 to 15 μ g of scandium(III) was transferred into a 10-ml volumetric flask, and then the solution was filled up to about 5 ml by adding distilled water. The pH value was adjusted to 5.6 with 1 ml of a buffer solution, and then 2.0 ml of a chromazurol S solution were added. The volume of the solution was made up to the mark with distilled water. After gentle mixing, the solution was allowed to stand for 1.5 hr. The absorbance of the solution was measured at 550 m μ against a reagent blank obtained in a similar way.

Results and Discussion

Absorption Spectra. A series of solutions was prepared so as to have the same amounts of scandium(III) and chromazurol S at different pH values. The absorption spectrum of each solution was measured against the reagent blank. The results are shown in Fig. 1. (Chromazurol

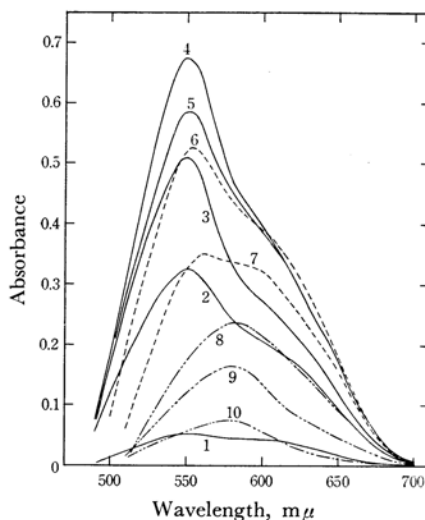


Fig. 1. Absorption spectra of Sc-ChS complex at various pH values.

Sc: 2.5×10^{-5} M, ChS: 1.0×10^{-4} M

1: pH 7.9, 2: pH 6.8, 3: pH 6.2, 4: pH 5.6
5: pH 5.2, 6: pH 5.0, 7: pH 4.4, 8: pH 3.8
9: pH 3.5, 10: pH 3.1

S is abbreviated as ChS in all the figures.) The maximum absorption wavelength at pH values of about 5.2 or higher is found at 550 m μ , and the absorption spectra of the complex indicate similar shapes. The absorbance disappeared at a pH value of about 8. At pH 5.0 or lower, each peak of the spectrum is shifted toward a longer wavelength, in accordance with the decreasing pH value of the solution, and each absorption spectrum changes its shape, while at pH below 4.0, an absorption spectrum of the same shape is newly obtained, with a maximum at 580 m μ .

The relation between the wavelength of the

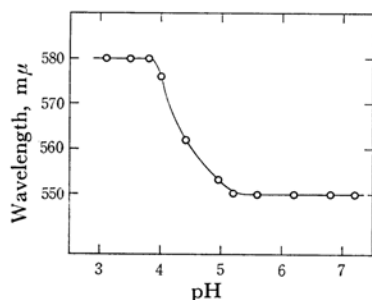


Fig. 2. Relation between wavelength of absorption maximum and pH of the medium.

Sc: 2.5×10^{-5} M, ChS: 1.0×10^{-4} M

maximum absorption and the pH of the medium is shown in Fig. 2.

The results in Figs. 1 and 2, consequently, indicate that two complexes, one a species with its absorption maximum at 550 mμ and at pH 5.2 or higher, the other with its absorption maximum at 580 mμ and at pH below 4.0, are probably formed. Furthermore, it is estimated that the two species coexist in the pH range between 4.0 and 5.2.

This paper will chiefly be concerned with the complex which gives the higher absorbance and which has its absorption peak at 550 mμ, at pH 5.2 or higher.

Though not illustrated in the figure, the absorption spectra of solutions containing 0.8 μmol of chromazurol S and scandium(III) added in various amounts up to 4.0×10^{-5} M, show an isobestic point at about 485 mμ against the water blank at pH 5.6. The occurrence of an isobestic point indicates that there is only one kind of complex.

Effect of pH Value. The effect of the pH value on the color development of the complex was investigated at 550 mμ by measuring the absorbance of colored mixtures containing 0.25 μmol of scandium(III) and 1.0 μmol of the reagent

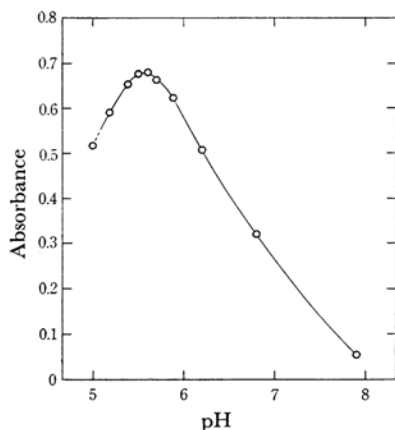


Fig. 3. Effect of pH on color development.

Sc: 2.5×10^{-5} M, ChS: 1.0×10^{-4} M
Wavelength: 550 mμ

at different pH values from 5 to 8; the results are shown in Fig. 3. The maximum and nearly constant absorbance was obtained at pH 5.5–5.6. The optimum pH range for the color development is narrow, so the accuracy of the pH adjustments were made sure by using a glass electrode.

Effect of the Addition of Chromazurol S.

The effect of the reagent concentration on the color development was studied with solutions containing 0.25 μmol (11.2 μg) of scandium(III) and various amounts of a 1.0×10^{-3} M chromazurol S solution. The absorbance measurements were made at 550 mμ. The results are shown in Fig. 4. The maximum color intensity is obtained by adding 1.0 ml of the reagent (the mole ratio of scandium(III) to chromazurol S being 1:4). More than a 4-fold excess of the reagent tends to decrease the absorbance gradually. It seems that

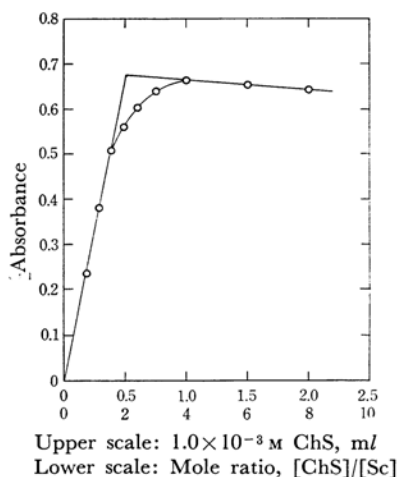


Fig. 4. Effect of addition of chromazurol S. (Mole ratio method.)

Sc: 0.25 μmol (11.2 μg), pH: 5.6
Wavelength: 550 mμ

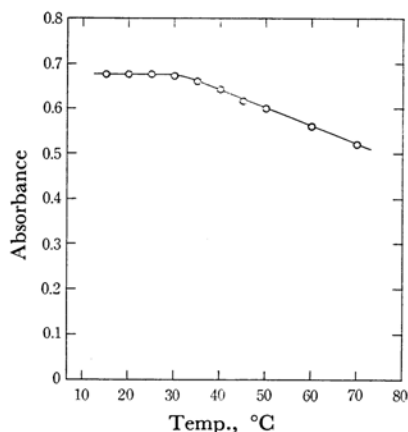


Fig. 5. Effect of temperature on color intensity.

Sc: 11.2 μg ChS: 1.0×10^{-4} M
pH: 5.6 Wavelength: 550 mμ

a complex of other species may form in the presence of a large excess of the reagent. In fact, a greater excess of the reagent causes a shift of the absorption peak to the longer wavelengths.

Effect of Temperature. The absorbance of a colored solution was measured at various temperatures. As is shown in Fig. 5, there was no variation in the absorbance of the color system within the range of temperature from 15 to 30°C, and over 30°C the color intensity decreased. Hence, normal variations in the temperature in laboratory seem to have no effect.

Effect of Time of Standing. At room temperature the color intensity decreased during the first 1.5 hr, and then remained constant for at least several hours (Fig. 6).

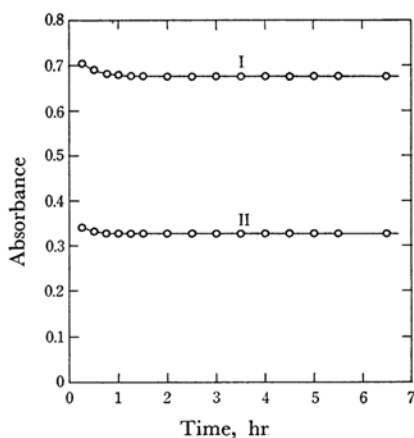


Fig. 6. Stability of color.
Sc: I, 11.2 μg ; II, 5.6 μg
ChS: $1.0 \times 10^{-4} \text{ M}$ pH: 5.6
Wavelength: 550 $\text{m}\mu$

Effect of the Amounts of the Buffer Solution and the Choice of Buffer. According to Otomo,⁴¹⁾ the buffer system of hexamethylenetetramine-perchloric acid has a great buffering power in the pH range from 4 to 7. At a constant pH, the absorbance of the colored solution was measured by varying the amounts of the buffer solution. As is shown in Fig. 7, the maximum color development is obtained by using from 0.6 to 1.2 ml of a 1.0 M buffer solution per 10 ml. In this series of experiments 1.0 ml of the buffer solution was always used.

The hexamethylenetetramine-perchloric acid buffer (pH 5.6) gave a clear color development. For the sake of comparison, were also used such buffers, as acetic acid-sodium acetate (pH 5.2₅), disodium citrate-sodium hydroxide (pH 5.5₅), sodium phosphate(dibasic)-potassium phosphate(monobasic) (pH 5.7), citric acid-sodium phosphate(dibasic) (pH 5.6), veronal sodium-hydro-

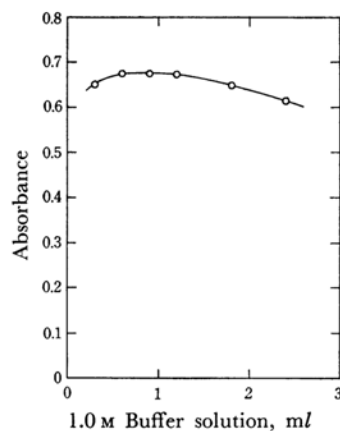


Fig. 7. Effect of concentration of buffer solution.
Sc: 11.2 μg ChS: $1.0 \times 10^{-4} \text{ M}$
pH: 5.6 Wavelength: 550 $\text{m}\mu$

chloric acid (pH 6.8), and pyridine-hydrochloric acid (pH 5.3) buffers. The observed absorbance, when the acetic acid-sodium acetate buffer was used, was lower than what was obtained with the hexamethylenetetramine-perchloric acid buffer, and the pyridine-hydrochloric acid buffer was unsatisfactory since the complex was precipitated when it was used. The other buffers gave almost no color development.

Calibration Curve. The relationship between the absorbance of a scandium-chromazurol S complex and the scandium concentration was studied by varying the scandium concentration and by measuring the absorbances at a wavelength of 550 $\text{m}\mu$ and at pH 5.6. The results are shown in Fig. 8. In the lower range of scandium concentration a bit of flection is observed, but a linear concentration-absorbance relationship is obtained in the scandium-concentration range between 4 and 27 μg (Beer's law holds). The optimum concentration range of scandium(III) determined

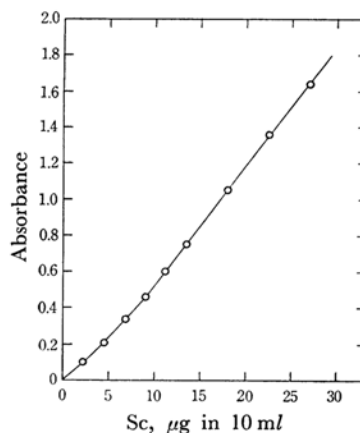


Fig. 8. Calibration curve.
pH: 5.6 Wavelength: 550 $\text{m}\mu$

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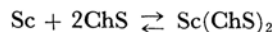
by Ringbom's plot⁴²⁾ was between 5 and 15 μg per 10 ml. According to the notation of Sandell,⁴³⁾ the sensitivity of this color reaction is 0.0017 $\mu\text{g}/\text{cm}^2$ for $\log I_0/I=0.001$. The molar extinction coefficient is about 27000 at 550 m μ .

When this molar extinction coefficient is compared with those of other reagents, its sensitivity is slightly lower than that of the pontachrome azure blue B (molar extinction coefficient, $\epsilon=31500$)¹⁾ and that of the xylenol orange ($\epsilon=29410$)⁴⁾ reagents; it is, however, much higher than those of the α -(2, 4-dihydroxyphenylazo)-pyridine ($\epsilon=22100$),⁸⁾ eriochrome cyanine R ($\epsilon=19000$),²⁾ pyrocatechol violet ($\epsilon=17300$),⁴⁾ arsenazo ($\epsilon=17000$),⁹⁾ glyoxal-bis-(2-hydroxyanil) ($\epsilon=14000$),¹³⁾ pontacyl violet 4BSN ($\epsilon=13700$),⁴⁴⁾ quercetin ($\epsilon=12800$),¹⁵⁾ 5, 7-dibromoxine ($\epsilon=12000$),¹⁷⁾ 5, 7-dichloroxine ($\epsilon=10000$),¹⁷⁾ tiron ($\epsilon=8000$),²²⁾ carboxyarsenazo ($\epsilon=8000$),⁴⁵⁾ and oxine ($\epsilon=6900$)¹⁶⁾ reagents.

Complex Formation. The composition of the scandium-chromazurol S complex was determined by Job's continuous-variation method.⁴⁶⁾ The experiment was carried out at $25.0 \pm 0.1^\circ\text{C}$. Solutions of the same concentration of scandium(III) and chromazurol S were mixed in various ratios, the pH of the solutions thus prepared was adjusted to 5.6, and the absorbance was measured at 550

m μ . The results are illustrated in Fig. 9. This figure shows that the combining ratio of scandium(III) and chromazurol S is 1 : 2. The mole-ratio method of Yoe and Jones⁴⁷⁾ also suggests the presence of the 1 : 2 species in the solution at pH 5.6 (Fig. 4).

As the combining ratio of the scandium-chromazurol S complex is 1 : 2, the formation of the complex can be expressed as:



The formation constant, K , was then calculated from the curves of the continuous-variation plots on the basis of the following relationships, expressed by Harvey and Manning⁴⁸⁾:

$$K = C(1 - \alpha)/(\alpha C)(2\alpha C)^2 \quad (1)$$

$$\alpha = (E_m - E_s)/E_m \quad (2)$$

where C is the total concentration of the complex assuming no dissociation; α , the degree of dissociation of the complex; E_m , the maximum absorbance of the complex when it does not dissociate into metal ions and free ligands; and E_s , the actual absorbance of the complex. E_m and E_s are obtained from the curve shown in Fig. 9; that is, E_m and E_s correspond to PR or PR' and PQ or PQ' respectively. The formation constant of the scandium-chromazurol S complex at pH 5.6 and $25.0 \pm 0.1^\circ\text{C}$ was thus calculated to be 9.5×10^{11} .

To estimate the composition of the complex (wavelength of absorption peak: 580 m μ) formed at pH values below 4, the continuous-variation method and the mole-ratio method were also applied. The results at pH 3.8 are shown in Figs. 10 and 11. It is evident that scandium(III) forms a 1 : 1 complex with chromazurol S.

The results presented above confirm the presence of two types of complex in this pH range.

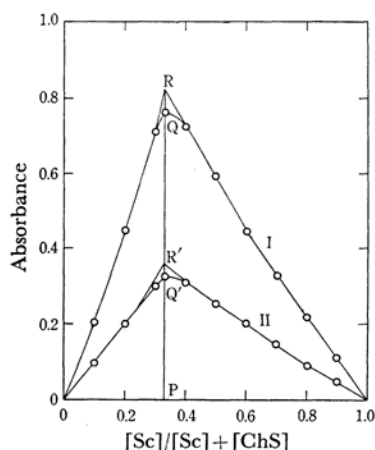


Fig. 9. Continuous variation method.
pH: 5.6 Wavelength: 550 m μ
Temperature: $25 \pm 0.1^\circ\text{C}$
I: $[\text{Sc}] + [\text{ChS}] = 1.0 \times 10^{-4} \text{ M}$
II: $[\text{Sc}] + [\text{ChS}] = 5.0 \times 10^{-5} \text{ M}$

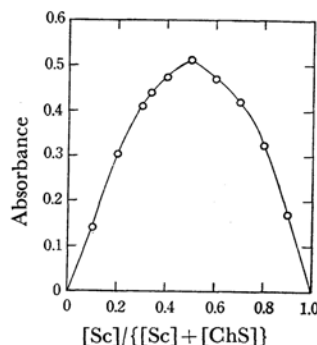


Fig. 10. Continuous variation method at pH 3.8.
Wavelength: 580 m μ
 $[\text{Sc}] + [\text{ChS}] = 1.5 \times 10^{-4} \text{ M}$

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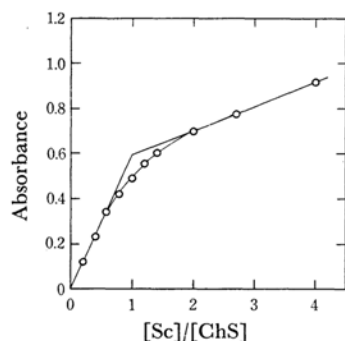


Fig. 11. Mole ratio method at pH 3.8.

Wavelength: 580 m μ [ChS] = 7.5×10^{-5} M

Effects of Diverse Ions. The effects of many ions on the determination of scandium(III) were studied under the conditions used in the proposed procedure.

The influences of 15 anions were examined. These anions were added individually to a solution containing 11.2 μ g of scandium(III) in the form of sodium salt. The results are summarized in Table 1.

TABLE 1. EFFECTS OF ANIONS ON THE DETERMINATION OF SCANDIUM(III)
Scandium(III) taken: 11.2 μ g (0.25 μ mol)

Anion added μ mol	Scandium(III) found, μ g
Chloride	10
Sulfate	10
Thiosulfate	10
Carbonate	5
	10
Bicarbonate	5
	10
Cyanide	1.25
	10
Acetate	1.25
	10
Fluoride	0.25
	5
Orthophosphates	PO_4^{3-} 0.25
	HPO_4^{2-} 0.25
	H_2PO_4^- 0.25
Oxalate	0.25
Tartrate	0.25
Citrate	0.25
EDTA	0.25

Chloride, sulfate, and thiosulfate ions do not interfere. Carbonate, bicarbonate, cyanide, and acetate ions have no effect at lower concentrations (0.125–0.5 mm), however, these ions give negative errors at higher concentrations (1 mm). Fluoride,

orthophosphate, oxalate, tartrate, citrate, and EDTA ions bleach the color of the complex, even when only a very small amount is present.

Next, the effects of various cations were studied by using various solutions, containing 0.25 μ mol of scandium(III) and the same amount of a particular cation. The results are shown in Table 2.

TABLE 2. EFFECTS OF CATIONS ON THE DETERMINATION OF SCANDIUM(III)
Scandium(III) taken: 11.2 μ g (0.25 μ mol)

Cation	Added as	Scandium(III) found, μ g
Ag(I)	AgNO_3	11.2
Hg ₂ (II)	$\text{Hg}_2(\text{NO}_3)_2$	11.3
Pb(II)	$\text{Pb}(\text{NO}_3)_2$	11.4
Hg(II)	$\text{Hg}(\text{NO}_3)_2$	11.2
Cd(II)	$\text{Cd}(\text{NO}_3)_2$	11.3
Bi(III)	$\text{Bi}(\text{NO}_3)_3$	11.2
Sn(II)	SnCl_2	11.5
Zn(II)	ZnSO_4	11.2
Co(II)	$\text{Co}(\text{NO}_3)_2$	11.4
Ni(II)	$\text{Ni}(\text{NO}_3)_2$	11.5
Mn(II)	$\text{Mn}(\text{NO}_3)_2$	11.6
Ca(II)	CaCl_2	11.3
Sr(II)	$\text{Sr}(\text{NO}_3)_2$	11.4
Ba(II)	$\text{Ba}(\text{NO}_3)_2$	11.3
Mg(II)	MgSO_4	11.3
NH ₄ (I)	$(\text{NH}_4)_2\text{SO}_4$	11.2
Ce(III)	$\text{Ce}(\text{NO}_3)_3$	11.4
Mo(VI)	Na_2MoO_4	11.3
Cu(II)	$\text{Cu}(\text{NO}_3)_2$	12.4
Al(III)	$\text{K}_2\text{SO}_4 \cdot \text{Al}_2(\text{SO}_4)_3$	18.9
Fe(II)	$\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4$	15.2
Be(II)	$\text{Be}(\text{NO}_3)_2$	16.9
Y(III)	$\text{Y}(\text{NO}_3)_3$	12.3
Th(IV)	$\text{Th}(\text{NO}_3)_4$	12.6
U(VI)	$\text{UO}_2(\text{CH}_3\text{COO})_2$	16.3
Fe(III)	FeCl_3	9.9
W(VI)	Na_2WO_4	2.3
Zr(IV)	$\text{ZrO}(\text{NO}_3)_2$	7.2

With chromazurol S, Al(III), Be(II), U(VI), and Fe(II) ions form colored complexes to give the highest positive error in the determination of scandium (III). Th(IV), Cu(II), and Y(III) ions cause also an overestimation of scandium(III). In nature scandium almost always occurs mixed with yttrium, thorium, and rare earth elements; these other elements always give an overestimation of scandium(III). Therefore, these metallic ions must be separated completely, by the ion exchange method²⁶⁾ or the solvent extraction method,⁹⁾ prior to the determination of scandium(III). Fe(III), Zr(IV), and W(VI) ions precipitate under these conditions and interfere, they should be removed by other suitable procedures.