CHAPTER V

COLORIMETRIC DETERMINATION
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In the beginning of the Chapter, a review of the indicators reported in literature for the Colorimetric Determination of Iron is given. This is followed by an account of experimental work on substances investigated as new reagents. An attempt has then been made to correlate the molecular structure of the phenolic carbonyl compounds with their performance as reagents.
COLORIMETRIC DETERMINATION OF IRON
(A REVIEW)

Numerous reagents are available for the colorimetric determination of iron. These are divided into two classes according to their reaction with ferric iron or ferrous iron.

The colour reactions of many organic compounds with ferric ions form the basis of the processes suggested for the detection and colorimetric estimation of iron. As iron is generally found in nature in the ferric state, methods depending on the reaction with ferric ions are more convenient, but a drawback of such processes is that anions forming stable complexes with ferric ions (e.g., phosphates, citrates, cyanides, tartarates, fluorides) interfere and therefore prior to colorimetric determination of ferric ion, such anions have to be removed.

In the following pages, a brief summary of the reagents used for the determination of ferric ion is given. The article is divided into following sections:

(a) Inorganic substances,
(b) Carbonyl compounds and their derivatives,
(c) Amino polycarboxylic acids,
(d) Phenolic carbonyl compounds and their derivatives,
Phenolic carboxylic acids and their derivatives,
Hydroxamic acids,
Amines and aldimines,
Organic sulphur compounds,
Heterocyclic compounds,
Dyes,
Miscellaneous substances.

A brief summary of the characteristics of the reagents is given in Table 5-1, p. 209.

(a) Inorganic substances:
This class of reagents includes the classical thiocyanate method and the ferrocyanide method. Although the thiocyanate method has its limitations, e.g., it is not the easiest or the most reliable, it is a valuable method in applied analysis and is still widely used for determining iron. The reagent is cheap and the process is rapid, but the intensity of iron-complex is dependent on thiocyanate concentration, kind of acid used and time of standing.

The decided advantage of thiocyanate is that it can be used in strongly acidic medium however the system shows some deviation from Beer's law. The rate of fading thiocyanate complex increases with temperature. The colour is read promptly after development due to a gradual shift in value. Copper, silver, mercurous,
bismuth, titanium, uranium, molybdenum, cobalt, nickel, mercuric, cadmium, zinc, antimony, large amounts of manganese and anions forming complexes with ferric ions in acidic medium interfere in the estimation of ferric ion. In spite of the limitations of the thiocyanate method, the process has received the largest share of attention from various workers, as is evident from the numerous literature references on this process. A number of workers have studied the optimum conditions of the method.

The intensity of ferric-thiocyanate colour is increased by reduction of the dielectric constant of the solvent. For this, the colour may be extracted with a solvent of low polarity, or the aqueous solution may be diluted directly. For direct addition, acetone is generally used. The ferric-thiocyanate colour is stabilised by addition of ethylene glycol monobutyl ether.

Peters (13A) observed that hydrogen peroxide is a more satisfactory oxidant for iron than permanganate in the thiocyanate determination of iron. The red colour can be made stable for several minutes, depending on the amount of peroxide used and the faded colour may be restored, if necessary, by the addition of more peroxide. Too much peroxide may cause
a yellowish interfering colour due to oxidation products of thiocyanate.

Washmut (5A) reported that thiocyanate method gives reliable results for 3.6-4.9 mg iron. Vanossi (10A) observed that best results are obtained in 1.5-2.0N sulfuric acid. He stated that control of thiocyanate concentration is needed.

Brouckerey (7A) reported that KCl, NH₄Cl, KNO₃, Ca(NO₃)₂, BaCl₂ and AlCl₃ do not interfere in the colorimetric determination of iron by thiocyanate method. Iron can be determined colorimetrically by thiocyanate in the presence of Busmuth by masking bismuth with phosphate (96A).

Trutter (16A) in correspondingly numbered tables it is shown that (1) within the range 0.2-4.0 ml. of HCl and 0.5-6.0 ml of thiocyanate solution, maximum colour development was obtained with either 0.5 ml of HCl and 6.0 ml. of KCNS, or with 1.0 ml. HCl and 4.0 ml. KCNS soln. (2) As much as 0.5 ml. of sulfuric acid per tube, or its equiv. in sulfate plus 1.0 ml of HCl, does not interfere. (3) By maintaining iron in the ferric state hydrogen peroxide stabilizes the colour. (4) Between 5 and 18° colour depth increases but no further increase is noted in the 18-30° range. (5) In the presence of 0.01 mg Fe, 87 mg of P as the
orthophosphate does not interfere but with 0.025 mg of Fe, 52 mg of P or orthophosphate reduces the recovery (6) In the case of 0.01 and 0.025 mg. of Fe the corresponding limits of P as the pyrophosphate are 69 and 41 mg. respectively (7) Ca\(^{2+}\) may be present upto 400 mg. with HCl or to 200 mg. if sulfuric acid is used in the colour development and with either acid 20 mg of Ca\(^{2+}\) plus 17 mg. of P do not interfere but if P is increased to 52 mg., the recovery of Fe is 98.5% (8) Cu\(^{2+}\) may be present upto 0.1 mg.

Utsumi\(^{17}\) made a spectrophotometric study of the reaction between ferric and thiocyanate ions in the presence of sulfuric acid, beryllium sulphate and ammonium persulphate. The effects of acidity, temperature, light, added beryllium, thiocyanate ion concen., and persulfate ion concentration on the stability and colour intensity of the solution are described. For analytical applications in the absence of added beryllium and the best results are obtained in 1.5-2.0 N sulfuric acid while in the presence of 1.0M beryllium sulphate it is desirable to work in 3.0N sulfuric acid. Precise control of thiocyanate concentration and temperature is required. Undue exposure to daylight should be avoided. The solutions obey Beer's law, and an ultimate sensitivity of 0.035 p.p.m. Fe\(^{3+}\) is obtained when using a cm. cell, and
measurements are made at 475 μm with a Beckman photoelectric spectrophotometer.

Durand (1A) and Smith (4A) suggested the extraction of ferric thiocyanate complex by ether and amylalcohol. Steinhauser (9A) observed that extraction by either saturated with sulfurdioxide gives better results. Phosphates, fluorides, acetates, oxalic acid, citric acid and tartaric acid ought to be absent. Daniel (12A) reported that isoamylalcohol modification of thiocyanate method gives erratic results, fading of the ferric-thiocyanate complex occurs at all temperatures, and in the presence of calcium salts causes undesirable colours. The colour of the complex is intense and does not fade in solutions containing nitric acid but it does fail in solutions containing hydrochloric or sulfuric acids. The presence of large quantities of calcium chloride interferes with the accuracy of the method in HCl solution but does not produce any appreciable effect when calcium is in the form of nitrate and nitric acid is used as a medium.

Mellon (14A) made a critical evaluation of thiocyanate method and observed that ammonium thiocyanate is preferable to thiocyanic acid as a colour forming reagent. He confirmed the observation of Daniel (12A) that nitric acid is preferable to hydrochloric acid or sulfuric acid. According to Mellon (13A) in nitric acid, ferric-thiocyanate complex obeys Beer's law in the
pH range 1.2-1.5 but not at higher acidities. Amount of reagent, amount and kind of acid, used of excess oxidant, time of standing and dielectric constant of the solvent effect on the colour. Barium, calcium, lead, strontium, zirconium, silver, mercurous, antimonous, cadmium, mercuric, zinc, chromic, nickelous, uranyl, fluoride, oxalate, chlorostannate, iodide, nitrite, sulphite, thiosulphate, arsenite, chlorostannate, tetraborate, dichromate, vanadate, molybdate and tungstate ions interfere. By using a 60% acetone solution, sensitivity is improved by 100%. In general, thiocyanate method is inferior to ferron, salicylic acid, salicylaldoxime and other methods.

Peters (15A) reported that acidity is a factor in colour development of ferric thiocyanate. The optimum acidity is 0.01N. Increasing thiocyanate progressively increases the intensity of the colour. The extraction of the red colour by ether depends upon the thiocyanate ferric ion ratio and a high ratio is necessary for iron extraction.

Baily (28A) reported that ferric thiocyanate colour can be stabilised in aqueous solution by the addition of methylethylketone acetone. The colour is stable for at least 1 hour or more and is not affected by light exposure during this period. The optimum quantity is 0.2 p.p.m. Li (22A) reported that ethylmethyl ketone
stabilises the colour and increases the intensity.

Speaker (21A) separated iron from aluminium by extraction of ferric-thiocyanate complex with diethylether and tetrahydrofuran.

Ayres (24A) extracted iron in the presence of Cu$^{2+}$, Ni$^{2+}$, Co$^{2+}$, and Mn$^{2+}$ as pyridine thiocyanate complex. Phosphates, bromides, iodides, cyanides and cyanates interfere seriously. Okura (27A) suggested the extraction of the ferric thiocyanate complex by ethyl acetate.

Duyer (25A) reported that the chief disadvantages of the thiocyanate method as usually applied, are the sensitivity of the colour to excess of thiocyanate ion, its dependence on the amount of sulphate ion and certain other ions present and instability of the ferrithiocyanate complex. He suggested the extraction of red ferric triphenylmethyloxium thiocyanate complex in chloroform, acetone, ethylenechloride or o-dichloro-benzene. The procedure is suitable for 1-10 p.p.m. iron. Co$^{2+}$, Ni$^{2+}$, Ag$^+$, Hg$^+$, Sb$^{3+}$, Cd$^{2+}$, Hg$^{2+}$, Zn$^{2+}$ do not interfere but Cu$^{2+}$ does.

Winsor (26A) reported that 2 methoxyethanol is a superior medium in which to develop and compare the ferric thiocyanate colour; it gives a colour 85% more intense than that developed in water. Compared
with the most effective acetone-water mixture, it shows 27% greater intensity of colour and 96% less evaporation. The system confirms to Beer's Law.

Melnick (29A) extracted ferric thiocyanate complex with butyl phosphate. Acid concentration has a marked effect on iron extraction. Between 13°C-50°C temperature has no effect.

The thiocyanate method has been used for determination of iron content, of copper alloys (38A, 39A, 41A, 42A), copper (41A), silver (43A), calcium (51A), magnesium (48A, 50A, 51A) magnesium alloys (49A), aluminium (50A, 56A, 59A, 60A, 61A, 62A, 63A, 64A, 65A) aluminium alloys (57A, 58A, 59A), Zinc (50A, 52A, 53A), zinc alloys (55A), Zinc salts (50A), with lead (50A), lead compounds (50A), cadmium compounds (44A), indium (66A), titanium compounds (67A, 71A, 72A, 74A), titanium (68A, 69A, 70A, 73A), Zirconium compounds (75A), tin (76A, 77), Bismuth (79A, 81A), bismuth alloys (80A), tantalumpentoxide (82A), uranium (83A), tungsten ores (84A), Manganese (85A), nickel (86A, 88A, 90A, 91A, 92A), nickel salts (87A, 90A), nickel alloys (88A), cobalt (93A, 94A), platinum rhodium alloys (95A), uranium alloy (99A), ferrochrome (100A), Chromite (100A), metals (101A), alloys (101A), acids (101A), salts (101A), cermets (102A), Silicon carbide (103A), ores (104A), alumite rocks (105A), cement (106A), (107A), raw materials for cement (107A), glass (108A), clay (109A), silicic
acid (110A), waterglass (111A), cupola slag (112A, 113A).

Walker (2A) reported that results obtained in analysis of ferrous containing 110 p.p.m. Fe by ferrocyanide method and thiocyanate method show a good agreement.

Daniel (12A) modified thiocyanate method for the determination of iron in foods and reported that by using nitric acid as solvent, large quantities of Ca salts of pyrophosphates do not interfere and error due to the fading of colour is practically eliminated. Trutter (16A) determined iron content of blood, liver and sheep kidney by thiocyanate method. Winsor (26A) observed that extraction with 2 methoxy ethanol should prove especially useful for determination of minute amounts of iron in biological material.

The thiocyanate process has been applied for the determination of iron content of biological materials such as blood (114A, 116A, 117A, 120A), serum (115A, 116A, 119A), hemoglobin (118A), tissues (114A, 138A, 143A), urine (114A), bone (121A), food (122A, 123A, 124A), sugar (125A, 126A), meats (127A, 170A), dairy products (128A), Curd (129A), milk (130A), Cream (130A), butter (130A), chocolate (131A), wines (132A-136A), multivitamin preparations (137A), feces (139A), plant extracts (140A-142A, 145A), cellulose materials (144A).
The iron content of water (104A, 107A, 149A, 150A—151A, 152A, 153A, 154A, 155A), seawater (146A—148A) has been determined colorimetrically using thiocyanate as a reagent. The process has been applied to the determination of iron content of industrial waste (156A), crude oil (157A), lubricating oils (158A), plating baths (159A, 160A), tan liquors (161A—163A), dyes (164A), ferrocene (165A), organic acids (166A, 168A), organic compounds (167A, 169A).

The advantage of determination by $\text{K}_4\text{Fe(CN)}_6$ is that the method is applicable in the presence of orthophosphoric acid. However, large amounts of $\text{Cu}^{2+}$, $\text{Fe}^{2+}$, $(\text{NH}_4)_2\text{SO}_4$ interfere (5). Baba (171A) have investigated the optimum conditions of the method. The process has been applied to the determination of iron in tea ash (172A), aluminium (175A), wine (181A, 182A), tungsten (179A), soil (178A), biological materials (173A), water (180A).

Walker (2A) studied the thiocyanate and ferrocyanide methods for the colorimetric determination of ferric ions. He reported that thiocyanate process is generally preferable but unsatisfactory in the presence of $\text{Ag}^+$, $\text{Hg}^{2+}$, $\text{Co}^{2+}$, phosphoric acid, oxalic acid and hydrofluoric acid. Oxalates and fluorides must be removed in both methods but phosphoric acid does no harm in ferrocyanide method.
Kureshi (176A) compared thiocyanate and ferrocyanide methods for colorimetric determination of ferric ion. Thiocyanate method is ten times more sensitive (0.01 µg) than the ferrocyanide method (0.1 µg) but the excess thiocyanate increases colour and for ferric thiocyanate complex there is a slight deviation from Beer's law. However, ferrocyanide method does not have these disadvantages. He suggests the use of CNS-Fe(CN)₆ as a more selective reagent for the colorimetric determination of iron.

Fluotitanic acid-hydrogenperoxide reagent has also been employed for the colorimetric determination of iron (183A).

(b) Ketones as reagents for the colorimetric determination of ferric ions:

Very few ketones have been used as colorimetric reagents for ferric ions. These are acetylacetone, theonyltrifluoroacetone, dibenzoylmethane and acetoacetanilide. The keto-enol structure is responsible for the reaction of these compounds with ferric ions. Of the ketonic compounds studied so far the most sensitive one is dibenzoylemethane (12B).

The ferric acetylacetone colour is very stable, but does not confirm to Beer's Law (3). The process has been applied to the determination of iron in the presence of uranium (2B), and to iron in rock (3B),
and manganese agglomerates (4B). Theonyltrifluoroacetone has been investigated as a colorimetric reagent for the determination of ferric ions (5B-9B), in the presence of cerium (8B), or manganese (9B). Acetoacetanilide (10B,11B) and acetoacetic ester (13B), have also been investigated as reagents.

C. Polyaminocarboxylic acids as Reagents for the colorimetric determination of ferric ions:

More attention has been paid to the first compounds of this series viz., ethylenediamine tetraacetic acid (EDTA) (9C,11C,12C).

Lott (10C) reported that it is feasible to determine iron in clay and limestone samples directly without separating foreign ions. EDTA simultaneously prevents interference and acts as a colour forming agent.

The method has been applied to the analysis of iron in wine (12C), alloys (14C,16C,18C). Higher compounds of this group viz., diethylenetriamine pentaacetic acid (DTPA)(5C-7C), ethylene glycol tetraacetic acid (EGTA)(8C) as well as other analogous compounds viz., aminoacetone diacetic acid (1C), oxime of aminoacetone diacetic acid (2C), diaminocyclohexane tetraacetic acid (CYDTA)(3C,4C), EEDTA (17C), nitrilotriacetic acid, NTA (18C) have also been studied. However, a drawback of this class of compounds is that they can be used at pH values higher than 5 and at this pH many cations may interfere in the estimation of iron.
D. Phenols as reagents for the Colorimetric determination of ferric ions:

Phenols react with iron to give characteristic colours; however, all phenolic compounds cannot be used as reagents because they do not fulfil the criteria. In polyhydric phenols, catechol (2D,3D), resorcinol (24D) and pyrogallol (27D-29D) are investigated as colorimetric reagents.

Pyrocatechol forms inner complex compound in which the H atom of one OH group is replaced by one equivalent of the metal, which in turn is coordinated to the oxygen atom of the second-OH group giving compounds of the type.

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\text{Catechol has been used as a reagent for colorimetric determination of ferric iron in soil (3D).}
\]

1,2 dihydroxybenzene 3,5 disulfonate is a sensitive and selective reagent for iron (7D). It gives red colour in alkaline medium and blue colour in acidic medium. Greenburg (6D) reported that iron can be colorimetrically estimated with tiron in the presence of a large excess of copper and nickel.

Tiron method has been applied to the analysis of iron in copper (6D), nickel (6D,3D), coalash(13D), slag(13D),
boiler deposits (13D), quartz (15D), heavymetals (19D), sodium iodide Al₂O₃ whiskers (20D), body fluids (10D), brass (14D), spalarite (16D), NaI (17D), scintillation materials (20D), other compounds investigated are oamenspherel (28D), picramic acid (29D), ferricon (30D).

Isonitrosodimethylidihydroresorcinol is a sensitive and selective reagent for the determination of iron (26D). The colour formed is stable and pH need not be controlled closely. Both ferrous and ferric ion react with the reagent, so the colour formed is a function of total iron present. The sensitivity is as good as α phenanthroline or α χ′ bipyridyl.

Oaminophenol (30D), picramic acid (31D) and ferricon (32D) have also been investigated as reagents for the colorimetric determination of ferric ion.

(E) Phenolic carbonyl compounds and their derivatives as reagents:

The most important compounds of this group is salicylaldoxime 1E, 2E. Salicylaldoxime-ferric complex colour is highly influenced by pH, the colour being purple at pH 3, red orange at 7.0, yellow at 10.0. The hue is changed by a minor variation in pH 5. Compounds having o-phenolic oxime group give characteristic colours with ferric ions, but only substances investigated previously were salicylthiosanicarbazone (3E), protocatechualdehyde (6E), β-resorcylaldoxime (4E, 5E),
o-hydroxy acetophenone oxime (7E) and resacetophenone oxime. A study of phenolic ketones and their derivatives as analytical reagents for ferric ions had been undertaken in this laboratory with a view to develop new colorimetric reagents for iron and also to observe whether any correlation exists between molecular structure of the reagent and their performance. Thus, resacetophenone (10E), respropiophenone, resbutyrophenone, respropiophenone oxime, resbutyrophenone oxime (14E), o-hydroxy-propiophenone oxime (8E), and o-hydroxy-butyrophenone oxime (9E), 4-hydroxy-3-propionyl oxime benzoic acid (16E), 2,6 dihydroxy acetophenone and 2,4,6 tri hydroxy acetophenone have been studied. It was observed that all these compounds form 1:1 complex with iron. The optimum pH range and wavelength of maximum absorbance are the same for compounds having similar structure but with the increase in number of carbon atoms in the sidechain, the extinction coefficient and sensitivity decrease. However, selectivity increases with the number of carbon atoms of the side chain (15E). In the phenolic ketoximes, the introduction of extra -OH group in position four, changes the spectral characteristics of the complex (15E, 16E).

(F) Phenolic acids as Colorimetric Reagents: Compounds of this class also react with ferric ions due to the presence of two coordinating groups namely -OH and -COOH groups. Salicylic acid has been extensively
studied as a reagent for iron. The colour intensity is little affected in the pH range 3.5 to 4.0 but the colour is dependent on the concentration of buffer and the reagent as well as pH. Hence, the amount of the reagent added must be carefully controlled.

Mehlig (7F) evaluated salicylic acid as a reagent for the colorimetric determination of iron and applied the process to the analysis of iron in iron ores. Ferric salicylic acid complex is stable for 66 hours (5F).

Salicylic acid has been used for the determination of iron in copper (1F), zinc (3F), soil (4F-6F), ore (7F), boric acid (9F), blood (10F), used oil (14F), fats (16F), plants (15F), Cadmium (17F), other materials (3F, 11F, 12F, 13F, 17F).

Protocatechuic acid gives a blue green colour at pH 5.0. The amount of reagent used should be held to minimum. In presence of excess of reagent, colour darkens with 30 minutes, Cu^{2+} interferes (5, 29F).

The colour intensity of ferric complex with Kojic acid depends on the concentration of Kojic acid as well as concentration of ferric ion. Kojic acid method is (39F) relatively free of interferences. Al^{3+}, Zn^{2+}, and other metals interfere, the colour intensity is less than that of Fe-CNS. Kojic acid is used in the analysis of iron in ores (41F), 4-hydroxy
biphenyl-3-carboxylic acid is a highly specific reagent for iron, only vanadium interferes (6F, 42F).

Other hydroxy aromatic acids and related compounds investigated as reagents for colorimetric determination of iron are paminosalicylic acid (21F), diodosalicylic acid (19F), dinitro salicylic acid (23F), 3-resorcylic acid (24F-26F), gallic acid (27F-29F), pyrogallol carboxylic acid (30F), 2,3 dihydroxybenzoic acid (31F), cresotic acids (32A), 2-hydroxy-3-naphtoic acid (33F), 3-hydroxy-2-naphtoic acid (34F), 4-nitro-2-hydroxy-3-naphtoic acid (35F), 1-bromo-2-hydroxy-3-naphtoic acid (35F), Hacid (36F), chromotropic acid (37F), dichlorochromotropic acid (38F), 3-resorcyllamide (42F), salicylamidoxime (44F).

G. Hydroxamic acids as reagents for the colorimetric determination of ferric ion:

Hydroxamic acids have the structure R - C - N - H.

The possibilities of their use as analytical reagents were realised in 1940, but the subject was investigated in details only after a few years.

Three soluble species can be identified as the pH of an Fe$^{3+}$ benzohydroxamic acid system is varied (2G). At pH 1.0 or below a purple colour ($\lambda_{max}$ 510 nm, $E_{max}$ 1450) is formed. At pH 3.5, a red 1:2 complex ($\lambda_{max}$ 480 nm, $E_{max}$ 2860) is formed. At pH 8.0, a 1:3 complex ($\lambda_{max}$ 440 nm) is formed. The complex can be extracted by
by alcohols and esters. The complex of iron III acetohydroxamic acid are 1:1 complex, $\lambda_{max}$ 500 mp $E_{max}$ 1.235 x $10^3$; 1:2 complex, $\lambda_{max}$ 455 mp $E_{max}$ 2.17 x $10^3$; 1:3 complex, $\lambda_{max}$ 425 mp, $E_{max}$ 2.710 x $10^3$. The stepwise formation constants were 7 x $10^9$, 1 x $10^9$ and 3 x $10^7$ respectively.

The compounds investigated in this class are formo-hydroxamic acid (1G) p-methoxybenzothiohydroxamic acid (4G,5G) benzohydroxamic acid benzene sulfonamide (6G), diphenylacetophydroxamic acid (7G), phenylcinnamohydroxamic acid (8G), hydrocinnamylhydroxamic acid (9G), salicylaldehyde glycine hydroxamic acid (10G), salicylhydroxamic acid (11G), quinaldinohydroxamic acid (12G), nicotino hydroxamic acid (13G), isonicotinohydroxamic acid (14G), thiophene, 2-hydroxamic acid (15G), cupferron (16G).

H. Amines as reagents for the Colorimetric determination of ferric ions:

In this class of reagents, about 8 reagents have been investigated so far. Out of these reagents, ethylenediamine (bis, o-hydroxy phenylacetic acid) (9H), ethylenediamine (bis sulfosalicylaldehyde (3H) and o-phenylene diamine (1H) are workable upto pH 3.0. Other reagents are workable above pH 3.0 and thus are less preferable. Compounds also investigated are 4, amino 4, methoxydiphenylamine (4H), salicylaldehyde ethylenediamine (5H), N, N' ethylenediamine (4-methoxy
1,2-benzoquinone-1-oxime zimine) \(6\)H), N,Ni bis (\(\beta\)-hydroxypropyl o-phenylenediamine) \(7\)H), anthranilic acid \(8\)H), Schiff base from 3-aldehydesalicylic acid from ethylenediamine \(18\)H).

1. Organic Sulfur Compounds as reagents for colorimetric determination of ferric ions: Sulphosalicylic acid has been extensively investigated as a reagent of iron. The colour is stable for one day. Since the colour is acid solution is due to only ferric ions and the colour in alkaline solution is due to both \(\text{Fe}^{2+}\) and \(\text{Fe}^{3+}\) ions this reagent is useful for the determination of both \(\text{Fe}^{2+}\) and \(\text{Fe}^{3+}\) ions \(5\)H).

In acid solution, sulfosalicylic acid gives a red compound with ferric ion, but the depth of colour is influenced by acid concentration. Strong oxidising agents, \(\text{Cu}^{2+}\), \(\text{Ni}^{2+}\) and \(\text{Co}^{2+}\) should be absent \(5\)I)

For the determination of iron in chromium-plating baths, KCNS and 7-ido-8-HQ-quinoline sulfonic acid or ferron are unsuitable, whereas sulfosalicylic acid is satisfactory \(5\)I). The optimum conditions for the use of sulfosalicylic acid have been investigated \((11-11\)I). Sulfosalicylic acid has been used as a colorimetric reagent for the determination of iron in NaI \((12\)I), NaOH \((13\)I), copper alloys \((14\)I-18I), CaC\(_2\) \((18\)I), in beryllium \((19\)I), in aluminium \((20\)I-26I), babitt metal \((27\)I), crude tin \((28\)I, 29I), chromium baths \((30\)I-31I),

\[\text{1,2-benzoquinone-1-oxime zimine} \quad 6\text{H}, \quad \text{N,Ni bis (}\beta\text{-hydroxypropyl o-phenylenediamine}) \quad 7\text{H}, \quad \text{anthranilic acid} \quad 8\text{H}, \quad \text{Schiff base from 3-aldehydesalicylic acid from ethylenediamine} \quad 18\text{H}.\]
tin (32I), ore (33I), steel (34I-36I), noble metals (37I), sand (38I), minerals (39I), manganese rock (40I-42I), clay (42I), fire-clay (42I), refractories (42I-44I), silicates (43I-45I), phosphates (46I-47I), cement (47I-48I, 49I), slag (50I), blood (51I), milk (52I), wine (53I, 54I), food (55I), soil (56I, 57I), water (59I-60I), plating baths (61I), rubber (63I).

Other compounds investigated in this group are di-thiosalicylic acid (65I), thiosalicylamide (66I), thiomalic acid (67I), dinaphthiazone (68I), unithiol (69I), bithionol (70I, 71I), N-α-hydroxy iminobenzyl benzene sulfoamide (72I), thione (73I), 3-thianaphthenoyl trifluoroacetone (74I), sulphaanthranilic acid (75I, 76I), diethylthio carbamate (77I), dithiocarbamoyl-hydrazinium salt (80I), morpholinium 3-oxapentamethylene thiocarbamate (81I).

J. Heterocyclic compounds as reagents for the colorimetric determination of ferric ions:

The most important compounds of this group are 8-hydroxyquinoline and its derivative ferron. The dark green ferric-oxine complex can be extracted by organic solvents in the pH range 2-3(5). The oxine-Fe compound is soluble in chloroform whereas oxine-Mg compound is insoluble, making separation possible (1).

Oxine has been used as a reagent for the determination
Ferron has been investigated as a reagent for the colorimetric determination of ferric ions by Mellon (54J). Dey et. al investigated the Fe\(^{3+}\)-ferron system in details (65J). It instantly reacts with ferric ions in acidic medium to give a green colour, whereas ferrous ions give no colour (5J). Ferron has been used as a reagent in the determination of iron in humates (68J), copper (67J), nickel (67J), water (69J), limestone (70J), clinker (70J), cement (70J).

Other oxime derivatives investigated are 2-methyl 8-quinolinol (46J), 8-hydroxyquinoline 5-sulfonic acid (73J), 5-bromo-8-hydroxyquinoline 7-sulfonic acid (78J), 5,7-dibromooxine N-oxide (81J), 8-aminoquinoline (82J).

Other reagents investigated having heterocyclic nucleus in this are α-furoin oxime (1J), phenylfluorohe (3J-5J), 3-hydroxyl-phenyl 3-methyltriazine (7J), 3-hydroxy 1, 3-diphenyltriazine (8J), 2,3,5,6-tetra-pyridylpyrazine (9J), 1(o-carboxyphenyl) 3-hydroxy
3-phenyltriazine (11J), tropolone (12J), tropolone-sulfonic acid (13J), esculetin (14J), 2-methyl 3-hydroxy 4-pyrone (15J), 2,3-dihydroxypyridine (16J), 1-picolinoyl 3-thiosemicarbazide (18J), 1,2-di-2-pyridylethanediol (19J), 3-hydroxyflavone (20J), 1-hydroxyxanthone (21J), citrinin (22J), hydrazinophthalazine (23J), morrelin (24J), morin (25J), phenathroline (26J), 3-hydroxycromone (29J), diantipyryl-methane (30J), disulfodiantipyryl methane (31J), 6,5-(5-hydroxy 2-hydroxymethyl pyran 4-OH 6-yl ketone (32J), antipyrine (33J), 1-benzothiophene, 3-yl- 4,4,4-trifluorodione, 1,3-dione (34), 8-hydroxyquinaldine (76J).

K. Dyes as reagents for the colorimetric determination of ferric ions:

The compounds investigated in this class give high extinction coefficients with ferric ions, but the drawback of these reagents is that they are coloured. Moreover, their selectivity is less as compared to others.

Compounds investigated include methylthymol blue (1K, 2K), aluminon (3K-6K), PAR (7K), PAN (8K-10K), pyrocatechol violet (12K), xylenol orange (14K-21K), chromeazurole S(22K-26K) variamine blue (27K-30K), chromoxane violet R(31K), chrome yellow O(33K), arsenazo (34K), azomethine (35K), crystal violet (36K).
The compounds investigated are alloxantine (1L), pyramidon (2L), hematoxylin (4L), phenylpyruvate (5L), hesperetin (7L), meconic acid (8L), alumocreson (9L), cyanomalonic acid (10L), sodium benzillate (11L), nalidixic acid (12L-16L), n-monobutylmalate (13L), calcichrome (14L, 15L), potassium ethylxanthate (17L), salicylic acid formaldehyde polymer (18L), maltol (19L).
### TABLE V.1
SUMMARY OF REAGENTS SUGGESTED FOR THE COLORIMETRIC DETERMINATION OF FERRIC IONS

<table>
<thead>
<tr>
<th>Reagent</th>
<th>pH</th>
<th>Concentration, µg/cm²</th>
<th>pH</th>
<th>Beer's law</th>
<th>Interfering ions</th>
<th>Tolerated ions</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Thiocyanate</td>
<td>0.05-1.0N</td>
<td>480 (0.008)</td>
<td>Slight</td>
<td></td>
<td>F⁻, SO₄²⁻, PO₄³⁻, C₂O₄²⁻</td>
<td>Sn²⁺, Sb³⁺, Pb²⁺, Cu²⁺</td>
<td>9</td>
</tr>
<tr>
<td>2. CNS+acetone</td>
<td>0.05N</td>
<td>480 (0.004)</td>
<td>-do-</td>
<td></td>
<td>Acetate, Tartarate, Citrate, Ag⁺, Hg⁺, Cu²⁺, Bi³⁺, Ti⁴⁺, U⁶⁺</td>
<td>Mn²⁺, Zn²⁺, Ni²⁺, Al³⁺</td>
<td>9</td>
</tr>
<tr>
<td>3. K₄Fe (CN)₆</td>
<td>0-2.5</td>
<td>620 (0.1)</td>
<td>Obeyed</td>
<td>CrO₄²⁻, S₂O₃²⁻, S₂MnO⁻</td>
<td>Cd²⁺, Zn²⁺</td>
<td>Hg⁺, Bi³⁺, Ti⁴⁺, U⁶⁺, F⁻, SO₄²⁻, PO₄³⁻</td>
<td>176A</td>
</tr>
<tr>
<td>4. KCN+K₄Fe (CN)₆</td>
<td>0.2-0</td>
<td>700</td>
<td>-</td>
<td>5-50</td>
<td></td>
<td></td>
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</tbody>
</table>

**A. INORGANIC COMPOUNDS AS REAGENTS FOR COLORIMETRIC DETERMINATION OF FERRIC IONS:**

- **Reagents:** Thiocyanate, CNS+acetone, K₄Fe (CN)₆, KCN+K₄Fe (CN)₆
- **Concentration:** µg/cm²
- **Beer's law:** Slight, Obeyed
- **Interfering ions:** F⁻, SO₄²⁻, PO₄³⁻, C₂O₄²⁻, Acetate, Tartarate, Citrate, Ag⁺, Hg⁺, Cu²⁺, Bi³⁺, Ti⁴⁺, U⁶⁺, Mn²⁺, Zn²⁺, Ni²⁺, Al³⁺, Cd²⁺, Zn²⁺, Hg⁺, Bi³⁺, Ti⁴⁺, U⁶⁺, F⁻, SO₄²⁻, PO₄³⁻, Cr³⁺, Pt⁴⁺, Se⁶⁺, Mg²⁺, Te⁶⁺, Al³⁺, Ca²⁺, Hg⁺, Sr²⁺, Ba²⁺, Th⁴⁺, Pb²⁺, Sb³⁺, W⁶⁺, Pd²⁺, Zr⁴⁺, Ti⁺, Be²⁺, Sn²⁺, Bi³⁺, U⁶⁺, Tl⁶⁺, As³⁺, Mo⁶⁺, Co²⁺, V⁴⁺, Cu²⁺, I⁻, Br⁻, Cl⁻, Li⁺, K⁺, Na⁺, H⁺, Si⁺, C₂O₄²⁻, SO₄²⁻, PO₄³⁻, formate, citrate, tartarate.
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<thead>
<tr>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
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</thead>
<tbody>
<tr>
<td>5. Fluotitanic acid+H₂O₂</td>
<td>1N</td>
<td>420</td>
<td>240-</td>
<td>0.24-</td>
<td>-</td>
<td>-</td>
<td>183A</td>
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<td></td>
<td>HNO₃</td>
<td>1200</td>
<td>1.2</td>
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<td>B. KETONES AS REAGENTS FOR THE COLORIMETRIC DETERMINATION OF FERRIC IONS:</td>
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<tr>
<td>6. Acetylacetone</td>
<td>6-8</td>
<td>273</td>
<td>25-150</td>
<td>0.5-</td>
<td>-</td>
<td>Ni²⁺, Co²⁺, Mn²⁺, UO₂²⁺</td>
<td>1B, 2B</td>
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<tr>
<td></td>
<td>353</td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td>438</td>
<td></td>
<td></td>
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<td></td>
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</tr>
<tr>
<td>7. Thenoyltrifluoroacetone</td>
<td>2.0</td>
<td>460</td>
<td>1-12</td>
<td>1-10</td>
<td>UO₂²⁺, Co³⁺, Th⁴⁺, Zr⁴⁺, Hg²⁺, Co²⁺, Cr³⁺</td>
<td>6B, 7B</td>
<td></td>
</tr>
<tr>
<td></td>
<td>(4880)</td>
<td></td>
<td></td>
<td></td>
<td>Cu²⁺, Ag⁺, Ni²⁺</td>
<td></td>
<td></td>
</tr>
<tr>
<td>8. Acetoacetic acid methyl ester</td>
<td>2.3-</td>
<td>560</td>
<td>2.8</td>
<td>1-80</td>
<td>Mn²⁺, Bi³⁺, Al³⁺, PO₄³⁻, I⁻, SeN⁻, F⁻, citrate, tartarate</td>
<td>Common metals</td>
<td>10B, 11B</td>
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<tr>
<td></td>
<td>3.0 (1230)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>9. Dibenzoylmethane</td>
<td>2.5-</td>
<td>320</td>
<td>-</td>
<td>0-2</td>
<td>Mo³⁺, Ti⁴⁺, Cr³⁺</td>
<td>-</td>
<td>12B</td>
</tr>
<tr>
<td></td>
<td>3.0 (17000)</td>
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</tr>
<tr>
<td>10. Acetoacetic acid methylester</td>
<td>1-3</td>
<td>500</td>
<td>-</td>
<td>0-10</td>
<td>Ni²⁺, Cu²⁺, Co²⁺, Al³⁺, Zn²⁺</td>
<td>-</td>
<td>13B</td>
</tr>
<tr>
<td></td>
<td>0 (17000)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>11. Acetoacetic acid Ethylester</td>
<td>1-3</td>
<td>510</td>
<td>-</td>
<td>0-10</td>
<td>-</td>
<td>do-</td>
<td>13B</td>
</tr>
<tr>
<td></td>
<td>0 (17000)</td>
<td></td>
<td></td>
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<td>C. AMINOPOLYCARBOXYLIC ACIDS AS REAGENTS FOR THE COLORIMETRIC DETERMINATION OF FERRIC IONS</td>
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<td>12. Aminoaacetone NN' diacetid acid (ATA)+H₂O₂</td>
<td>5.5</td>
<td>370</td>
<td>50-500</td>
<td>0.20</td>
<td>At &gt;1 mole ratio with Fe⁺, PO₄³⁻, F⁻</td>
<td>1C</td>
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<tr>
<td></td>
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<td></td>
<td></td>
<td></td>
<td>concentrations, Cd²⁺, Zn²⁺, Al³⁺, Mn²⁺, Ni²⁺, Cr³⁺, Ph²⁺, Cu²⁺, interfere</td>
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<tr>
<td>13. Aminoaacetone dia- cetic acid oxime</td>
<td>5-6</td>
<td>420</td>
<td>0-25</td>
<td>-</td>
<td>Cu²⁺, F⁻, SO₄²⁻</td>
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<td>2C</td>
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<td>(2000)</td>
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<tr>
<td>14.1,2 dianinocyclo hexanetetraacetic 6.0</td>
<td>5-6</td>
<td>265</td>
<td>-</td>
<td>-</td>
<td>Cu²⁺, Hg²⁺</td>
<td>Ca²⁺, Mg²⁺, Cr³⁺, Zn²⁺</td>
<td>13C</td>
</tr>
<tr>
<td></td>
<td>(8440)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Co²⁺, Mn²⁺, Al³⁺</td>
<td>10I</td>
</tr>
<tr>
<td>No.</td>
<td>Phosphorus Compounds</td>
<td>Molar Absorbance (μM)</td>
<td>pH</td>
<td>Remarks</td>
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</tr>
<tr>
<td>15</td>
<td>1,2 diaminocyclohexane-1,2-tetraacetic acid (CDTA) + H₂O₂</td>
<td>10.8 - 530</td>
<td>0.5 - 60</td>
<td>-</td>
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<td>16</td>
<td>Diethylentriaminepentaacetic acid (DTPA) + H₂O₂</td>
<td>10.0 - 520</td>
<td>0.2</td>
<td>-</td>
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<td>17</td>
<td>Tetra-N,N-diethylglycylamine-tetraacetic acid (EDTA) + H₂O₂</td>
<td>3.0 - 450</td>
<td>0.13</td>
<td>obeyed Co²⁺/Cr³⁺</td>
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<tr>
<td>18</td>
<td>Di-(2-aminoethyl)glycol-2-1o</td>
<td>4.20 - 280</td>
<td>-</td>
<td>-</td>
<td></td>
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<tr>
<td>19</td>
<td>Ethylenediaminetetraacetic acid (EDTA) + H₂O₂</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td></td>
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<tr>
<td>20</td>
<td>Nitrilotriacetic acid (NTA)</td>
<td>3.0 - 285</td>
<td>8.99</td>
<td>-</td>
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<tr>
<td>21</td>
<td>Di-(2-aminoethyl)glycol N,N,N*,N*,N* (EEBTA)</td>
<td>4.5 - 386</td>
<td>7.0</td>
<td>-</td>
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</tr>
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</table>

**PHENOLS AS REAGENTS FOR COLORIMETRIC DETERMINATION OF FERRIC IONS:**

Catechol 5.3

V⁺, V⁺⁺, Cu²⁺, Cr³⁺, Ni²⁺, Mn²⁺, Al³⁺, Fe²⁺, Fe³⁺, Br⁻, Cl⁻, NO₃⁻, Acetate.
<table>
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<td>23.</td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Tiron(1,2</td>
<td>8.5</td>
<td>480</td>
<td>(0.009)</td>
<td>0.2</td>
<td>0.2-</td>
<td>Cu^{2+},UO_{2}^{2+},Ti^{4+},MoO_{4}^{2-},O_{2}^{2-}</td>
<td></td>
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<tr>
<td>dihydroxy</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Be^{2+},Fe^{2+},NH_{4}^{-},Ni^{2+}</td>
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<tr>
<td>benzene</td>
<td>9.5</td>
<td>66</td>
<td></td>
<td>10</td>
<td>0.25-</td>
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<td></td>
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<tr>
<td>3,5 disulfonate</td>
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<td></td>
<td></td>
<td></td>
<td>1.42</td>
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</tr>
<tr>
<td>24.</td>
<td>2.9</td>
<td>450</td>
<td>-</td>
<td></td>
<td>0.25-</td>
<td>Cations</td>
<td>24D</td>
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<td>Resorcinol</td>
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<td></td>
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<td>Fe^{2+}</td>
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</tr>
<tr>
<td>25.</td>
<td>3-11</td>
<td>620</td>
<td>(0.02)</td>
<td>1-6</td>
<td>1-6</td>
<td>Cr^{3+},Cu^{2+},Co^{2+},Ni^{2+}</td>
<td>25D</td>
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<td>Isonitroso</td>
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<td>Al^{3+},Zn^{2+},Mn^{2+},UO_{2}^{2+}</td>
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<tr>
<td>dimethyl</td>
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<td>620</td>
<td>(0.02)</td>
<td>1-6</td>
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<td>dihydro</td>
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<td>Pyrogallol</td>
<td></td>
<td>550</td>
<td>-</td>
<td></td>
<td>10</td>
<td>Cb^{5+},Ti^{4+}</td>
<td>28D,29D</td>
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<td>27.</td>
<td>1.2-</td>
<td>430</td>
<td>50-750</td>
<td></td>
<td>2.2-</td>
<td>Cr^{3+},V^{5+},Ce^{3+},BrO_{3}^{-},H_{2}O_{2}</td>
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<td>O-aminophenol</td>
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<td>28.5</td>
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<tr>
<td>28.</td>
<td>12N</td>
<td>428</td>
<td>15</td>
<td>-</td>
<td>-</td>
<td>Non oxidising ions and ions not absorbing at 428 mp</td>
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<td>Picramic acid</td>
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<td></td>
<td>Li^{+},Fe^{2+},Sb^{3+},Be^{2+},Ce^{3+}</td>
<td>32D</td>
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<tr>
<td>HCl</td>
<td></td>
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<td></td>
<td></td>
<td>VO_{3}^{-} in limited amounts</td>
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<tr>
<td>29.</td>
<td>2.5</td>
<td>-</td>
<td>-</td>
<td></td>
<td>0.4-</td>
<td>F^{-},B_{2}O_{7}^{2-},H_{2}PO_{4} ,</td>
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<td>Ferricon(diNa,</td>
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<td>20</td>
<td>C_{6}O_{4}^{2-},tartarate</td>
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<tr>
<td>2 naptho 3,6 disulfonate</td>
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<td>citrate</td>
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<td>E. PHENOLIC CARBONYL COMPOUNDS AS REAGENTS FOR COLORIMETRIC DETERMINATION OF FERRIC IONS:</td>
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<tr>
<td>30.</td>
<td>7.0</td>
<td>480</td>
<td>0.011</td>
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<tr>
<td>Salicylaldoxime</td>
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<td>1E</td>
</tr>
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<td>Salicylthio</td>
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<td>3E</td>
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<td>2,4 dihydroxy benzaldoxime</td>
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<td>4 dihydroxy</td>
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<td>34. 2,4 dihydroxy acetophenone</td>
<td>2.9-470</td>
<td>5.3-1-56</td>
<td>PO₄³⁻, F⁻, C₂O₄²⁻, citrate, tartarate</td>
<td>Al³⁺, Ba²⁺, Be²⁺, Ca²⁺, Ce³⁺, Ca²⁺, Mg²⁺, Mn²⁺, K⁺, Na⁺, Sr²⁺, Th⁴⁺, UO₂²⁺, Zn²⁺, Zr⁴⁺, NH₄⁺, Cl⁻, C₁₀⁴⁻, N₃⁻, S₄²⁻</td>
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<td>35. 2,4 dihydroxy propiophenone</td>
<td>3.0(1390)</td>
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<td>36. 2,4 dihydroxy butyrophenone</td>
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<td>8.1-1.56</td>
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<td>37. 2,6 dihydroxy acetophenone</td>
<td>1.8-500</td>
<td>1.56</td>
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<td>38. 2,4,6 trihydroxy acetophenone</td>
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<td>39. 2,4 dihydroxy acetophenone oxime</td>
<td>2.95(1225)</td>
<td>4.9-1-10</td>
<td>Co²⁺, UO₂²⁺, MoO₄²⁻, F⁻, PO₄³⁻, citrate, UO₂</td>
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<td>42. 2-hydroxy acetophenone oxime</td>
<td>2.0-420</td>
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<td>F-, C₂O₄²⁻, PO₄³⁻, vanadate, citrate, tungstate, molybdate</td>
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<td>2.85-510</td>
<td>4.9-17.0</td>
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<td>F⁻, C₂O₄²⁻, PO₄³⁻, tartarate, oxalate</td>
<td>Al³⁺, Ba²⁺, Be²⁺, Cd²⁺, Cs⁺, Ca²⁺, Mg²⁺, Mn²⁺, Kb⁺, Na⁺, Sr²⁺, Th⁴⁺, UO₂²⁺, Zn²⁺, Zr⁴⁺, NH₄⁺, Cl⁻, ClO₄⁻, NO₃⁻, SO₄²⁻</td>
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<td>2,hydroxy buty-</td>
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<td>5.6-</td>
<td>1-56</td>
<td>do-</td>
<td>do-</td>
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<td>19.5</td>
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<td>1-13</td>
<td>do-</td>
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<td>4.6 (2500)</td>
<td>(0.040)</td>
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<td>PHENOLIC CARBOXYLIC ACIDS AND RELATED COMPOUNDS AS REAGENTS FOR COLORIMETRIC DETERMINATION OF FERRIC IONS:</td>
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<td>(0.03)</td>
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<td>C₂O₄²⁻, PO₄³⁻, F⁻, Cu²⁺, NO₂⁻, I⁻, tartarate, oxalate, tungstate, arsenate</td>
<td>NH₄⁺, Ba²⁺, Cd²⁺, Ca²⁺, 5,9, Pb²⁺, Li⁺, Mg²⁺, Mn²⁺, K⁺, Na⁺, Sr²⁺, Zn²⁺</td>
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<td>NO$_3^-$, Cl$^-$, SO$_4^{2-}$, CNS$^-$,</td>
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<td>Mn$_2^+$, Co$_3^+$, Th$_4^+$, Ti$_4^+$</td>
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<td>Zn$^{2+}$, Mn$_2^+$, Ni$^{2+}$, Mg$^{2+}$,</td>
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<td>Mg²⁺, Ca²⁺, Cu²⁺, Na⁺, Mn²⁺</td>
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* G. HYDROXAMIC ACIDS AS REAGENTS FOR COLORIMETRIC DETERMINATION OF FERRIC IONS:*

* 0
H. AMINES AS REAGENTS FOR THE COLORIMETRIC DETERMINATION OF FERRIC IONS:

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<td>1-8</td>
<td>Ti$^{4+}$, UO$_2$$^{2+}$, Cu$^{2+}$, Ni$^{2+}$</td>
<td>-</td>
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<tr>
<td>84. Cupferron</td>
<td>-</td>
<td>460</td>
<td>20-50</td>
<td>-</td>
<td>Ti$^{4+}$, V$^{5+}$</td>
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85. 0-phenylene diamine

86. Anthranilic acid 4.5 465 - - Cu$^{2+}$, Cr$^{3+}$ - 8H

87. Ethylenediamine (bis salicylaldehyde) 5.0 495 0-10 0-10 Cu$^{2+}$, Cr$^-$, F$^-$ - 5H

88. Ethylenediamine (bis 0-hydroxy-phenylacetic acid) 3.7- 480 3.2-8.0 0-14 Cu$^{2+}$, Hg$^{2+}$ Pb$^{2+}$, Al$^{3+}$, Mn$^{2+}$, Ca$^{2+}$, Ni$^{2+}$ 6H

89. Ethylenediamine (bis sulfosalicylic acid) 2.8- 510- - 0.25- $F^-$, $BO_3^{2-}$, $PO_4^{3-}$, $MoO_4^{2-}$ 3H
I. ORGANIC SULFUR COMPOUNDS AS REAGENTS FOR THE COLORIMETRIC DETERMINATION OF FERRIC IONS

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<tr>
<td>90</td>
<td>0-phenylene diamine N,N, (bis α-hydroxy propyl)</td>
<td>-</td>
<td>525</td>
<td>-</td>
<td>0.5-15.0</td>
<td>Ag⁺⁺Hg⁺⁺Cu⁺⁺Ni⁺⁺Co⁺⁺, oxidizing agents, anions</td>
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<td>91</td>
<td>Anthranilic acid 5.0 (bis carboxy methyl)+H₂O₂</td>
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<td>Mn²⁺</td>
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<td>92</td>
<td>Sulfosalicylic acid</td>
<td>1.0-2.5</td>
<td>510</td>
<td>-</td>
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<td>obeyed</td>
<td>-</td>
<td>9,9I</td>
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<td>93</td>
<td>5,5' dithiodisalicylic acid</td>
<td>4.5-6.5</td>
<td>498-2860</td>
<td>8-9.8</td>
<td>-</td>
<td>-</td>
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<td>94</td>
<td>Thiosalicylaldehyde</td>
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<td>350</td>
<td>1-8</td>
<td>-</td>
<td>Cu²⁺</td>
<td>Fe⁺⁺,Co⁺⁺Ni⁺⁺Cd⁺⁺, Al⁺⁺⁺,Mn⁺⁺⁺,Bi⁺⁺⁺</td>
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<td>95</td>
<td>Thiomalic acid</td>
<td>8.0-10.5</td>
<td>530</td>
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<td>100-1000</td>
<td>-</td>
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<td>67I</td>
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<td>96</td>
<td>Unithiol</td>
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<td>-</td>
<td>-</td>
<td>0.5-100 Bi³⁺,Cr³⁺,Au³⁺,Cu²⁺</td>
<td>Co²⁺,Ni²⁺⁺Mn²⁺⁺UO₂²⁺⁺, Pd²⁺⁺V⁵⁺</td>
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<td>97</td>
<td>Bithionol</td>
<td>4.7</td>
<td>484 (2200)</td>
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<td>-</td>
<td>Cu²⁺⁺,Ni²⁺⁺Co²⁺</td>
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<td>N,α hydroxy benzene sulfonamide</td>
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<td>540 (2650)</td>
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<td>0.2-60</td>
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<td>99.</td>
<td>Thione</td>
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<td>550</td>
<td>(0.02)</td>
<td>-</td>
<td>Cu²⁺,NO₂⁻</td>
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<td>100.</td>
<td>3,thianaphthenoyltrifluoroacetone</td>
<td>1.5-</td>
<td>516</td>
<td>(5000)</td>
<td>1-14</td>
<td>Oxalate,phosphate, EDTA,citrate</td>
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<td>74l</td>
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<td>101.</td>
<td>5-sulfoanthrаниlic acid</td>
<td>4.0</td>
<td>455</td>
<td>-</td>
<td>5-150</td>
<td>Cr³⁺,Al³⁺,Co²⁺,Cu²⁺</td>
<td>Cd²⁺,Zn²⁺,Mn²⁺,Pb²⁺</td>
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<td>102.</td>
<td>Dithiocarbamoylhydrazine</td>
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<td>570</td>
<td>-</td>
<td>-</td>
<td>-</td>
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<tr>
<td>103.</td>
<td>Morpholinium 3,oxapentamethylenethiocarbamate</td>
<td>1.5</td>
<td>510</td>
<td>(0.016)</td>
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<td>-</td>
<td>-</td>
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J. HETEROCYCLIC COMPOUNDS AS REAGENTS FOR COLORIMETRIC DETERMINATION OF FERRIC IONS:

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<td>104.</td>
<td>Furoin α oxime</td>
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<td>105.</td>
<td>Phenyl fluorone</td>
<td>5-6</td>
<td>610</td>
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<td>106.</td>
<td>2 Acetyl,10,3, dimethylamino propyl phenothiazine maleate</td>
<td>512</td>
<td>1-8</td>
<td>-</td>
<td>EDTA</td>
<td>-</td>
<td>-</td>
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<td>107.</td>
<td>2 chloro,10,3, dimethyl amino propyl phenothiazine maleate</td>
<td>512</td>
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<td>-do-</td>
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<td>3,hydroxy1,1, phenyl 3, methyl triazine</td>
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<td>625</td>
<td>0.1</td>
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<td>109. (0-carboxy phenyl)3, hydroxy, 3, phenyltriazine</td>
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<td>110. Tropolone</td>
<td>0.2N HCl</td>
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<td>111. Tropolone, 5, sulfonic acid</td>
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<td>113. 2 methyl, 3, hydroxy 4, pyrone</td>
<td>1.0-6.0</td>
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<td>-</td>
<td>405</td>
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<td>0.5-5.0</td>
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<td>116. 1,2 di 2,pyridyl ethanediol</td>
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<td>-</td>
<td>1-11</td>
<td>-</td>
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<td>21J</td>
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*Cr^3+, Ce^2+, Ni^2+, Bi^3+, Sb^3*
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<td>Citrinin</td>
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<td>Hydrazinophthalazine</td>
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<td>535</td>
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<td>Co$^{2+}$, Ni$^{2+}$, Mn$^{2+}$, Cr$^{3+}$,</td>
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<td>Ce$^{3+}$, MnO$_4^-$, CrO$_4^{2-}$</td>
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<td>121</td>
<td>Morellin</td>
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<td>615</td>
<td>-</td>
<td>not</td>
<td>Cu&lt;sup&gt;2+&lt;/sup&gt;, Co&lt;sup&gt;2+&lt;/sup&gt;, Ni&lt;sup&gt;2+&lt;/sup&gt;, Al&lt;sup&gt;3+&lt;/sup&gt;</td>
<td>NH&lt;sub&gt;4&lt;/sub&gt;&lt;sup&gt;+&lt;/sup&gt;, Sb&lt;sup&gt;3+&lt;/sup&gt;, Ba&lt;sup&gt;2+&lt;/sup&gt;, Be&lt;sup&gt;2+&lt;/sup&gt;</td>
<td>58J</td>
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<td>beyond</td>
<td>P&lt;sub&gt;6&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;&lt;sup&gt;-&lt;/sup&gt;, CN&lt;sup&gt;-&lt;/sup&gt;, oxalate</td>
<td>Ca&lt;sup&gt;2+&lt;/sup&gt;, Co&lt;sup&gt;2+&lt;/sup&gt;, Li&lt;sup&gt;+&lt;/sup&gt;, Mg&lt;sup&gt;2+&lt;/sup&gt;,</td>
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<td>sulphonic acid</td>
<td>7.7</td>
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<td>citrate, tartrate</td>
<td>Mn&lt;sup&gt;2+&lt;/sup&gt;, K&lt;sup&gt;+&lt;/sup&gt;, Ag&lt;sup&gt;+&lt;/sup&gt;, Na&lt;sup&gt;+&lt;/sup&gt;,</td>
<td>Sr&lt;sup&gt;2+&lt;/sup&gt;, Zn&lt;sup&gt;2+&lt;/sup&gt;</td>
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<td>132. 5, bromo 8,hydroxy</td>
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<td>-</td>
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<td>Co&lt;sup&gt;2+&lt;/sup&gt;, UO&lt;sub&gt;2&lt;/sub&gt;&lt;sup&gt;2+&lt;/sup&gt;, V&lt;sup&gt;3+&lt;/sup&gt;,</td>
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<td>80J</td>
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<td>134. 5,7 dibromo oxine N-oxide</td>
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<td>-</td>
<td>0.22-</td>
<td>UO&lt;sub&gt;2&lt;/sub&gt;&lt;sup&gt;2+&lt;/sup&gt;, Ce&lt;sup&gt;3+&lt;/sup&gt;, PO&lt;sub&gt;4&lt;/sub&gt;&lt;sup&gt;3-&lt;/sup&gt;,</td>
<td>-</td>
<td>81J</td>
<td></td>
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<tr>
<td>oxine N-oxide</td>
<td></td>
<td></td>
<td></td>
<td>3.08</td>
<td>WO&lt;sub&gt;4&lt;/sub&gt;&lt;sup&gt;2-&lt;/sup&gt;, oxalate</td>
<td>-</td>
<td></td>
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<tr>
<td>135. 8, a minoqui-</td>
<td>1.6-2.0</td>
<td>550</td>
<td>-</td>
<td>1.5-</td>
<td>-</td>
<td>-</td>
<td>82J</td>
<td></td>
</tr>
<tr>
<td>noline</td>
<td></td>
<td></td>
<td></td>
<td>150</td>
<td></td>
<td>-</td>
<td>90J</td>
<td></td>
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<tr>
<td>136. Quinolpyridine</td>
<td>-</td>
<td>450</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>83J</td>
<td></td>
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<td>DYES AS REAGENTS FOR COLORIMETRIC DETERMINATION OF FERRIC IONS:</td>
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<tr>
<td>137. Methylthymol blue</td>
<td>4.5-520</td>
<td>-</td>
<td>0.2-</td>
<td>-</td>
<td>-</td>
<td>2K</td>
<td></td>
<td></td>
</tr>
<tr>
<td>138. Aluminon</td>
<td>3.2-560</td>
<td>1.1-</td>
<td>not Cu²⁺,Fe²⁺,Be²⁺UO₂²⁺,</td>
<td>-</td>
<td>Cu²⁺,Fe²⁺,Be²⁺UO₂²⁺,</td>
<td>4K</td>
<td></td>
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<tr>
<td></td>
<td>5.4</td>
<td>60</td>
<td>obeyed A1³⁺,Cr³⁺,Ce³⁺,Co²⁺,</td>
<td>-</td>
<td>Th⁴⁺,Zr⁴⁺</td>
<td>6K</td>
<td></td>
<td></td>
</tr>
<tr>
<td>139. PAR</td>
<td>8-9.3 536</td>
<td>-</td>
<td>Cu²⁺,Ga²⁺,Ti⁴⁺,Hf⁴⁺,Cr³⁺</td>
<td>-</td>
<td>Cu²⁺,Ga²⁺,Ti⁴⁺,Hf²⁺,</td>
<td>7K</td>
<td></td>
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</tr>
<tr>
<td></td>
<td>(60,400)</td>
<td>Cu²⁺,Sn²⁺,Ta³⁺,Au³⁺,</td>
<td>-</td>
<td>UO₂²⁺,Sn²⁺,Ta³⁺,Au³⁺,</td>
<td>-</td>
<td>Bι³⁺,Zr⁴⁺</td>
<td></td>
<td></td>
</tr>
<tr>
<td>140. PAN</td>
<td>4-8 560</td>
<td>(0.4)</td>
<td>10</td>
<td>-</td>
<td>-</td>
<td>8K,10K</td>
<td></td>
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<tr>
<td></td>
<td>(15,500)</td>
<td></td>
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<tr>
<td>141. Pyrocatechol violet</td>
<td>-</td>
<td>610</td>
<td>(0.002)</td>
<td>Cu²⁺,Mg²⁺,Al³⁺,Sb³⁺,</td>
<td>-</td>
<td>Cu²⁺,Mg²⁺,Al³⁺,Sb³⁺,</td>
<td>13K</td>
<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td>Mo³⁺,EDTA</td>
<td></td>
<td>Mo³⁺,EDTA</td>
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<tr>
<td>142. Xylenol orange</td>
<td>-</td>
<td>575</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>16K</td>
<td></td>
<td></td>
</tr>
<tr>
<td>143. Chromeazurol S+Zephriramine</td>
<td>4.3 640</td>
<td>-</td>
<td>0.014-</td>
<td>-</td>
<td>-</td>
<td>22K</td>
<td></td>
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<tr>
<td></td>
<td>(85,000)</td>
<td></td>
<td>0.35</td>
<td></td>
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<tr>
<td>144. Chromeazurol S+cetyltrimethylammonium chloride</td>
<td>3.1-630</td>
<td>-</td>
<td>not Sn²⁺,Be²⁺,UO₂²⁺,Au³⁺</td>
<td>-</td>
<td>Sn²⁺,Be²⁺,UO₂²⁺,Au³⁺</td>
<td>22K</td>
<td></td>
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<tr>
<td></td>
<td>3.8 (14,700)</td>
<td>obeyed Al³⁺,Hf³⁺,Ti⁴⁺,Th⁴⁺</td>
<td>-</td>
<td>Al³⁺,Hf³⁺,Ti⁴⁺,Th⁴⁺</td>
<td>-</td>
<td></td>
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<tr>
<td>145. Chrome azurolS</td>
<td>5.5-630</td>
<td>-</td>
<td>0.8-</td>
<td>-</td>
<td>-</td>
<td>23K</td>
<td></td>
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<tr>
<td></td>
<td>5.9 (55000)</td>
<td>18.0</td>
<td>-</td>
<td></td>
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<tr>
<td>146.</td>
<td>Variamine blue</td>
<td>1.0-</td>
<td>610</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>Ni²⁺, Co²⁺, Zn²⁺, Cu²⁺, Al³⁺, PO₄³⁻, CO₃²⁻</td>
<td>28K</td>
</tr>
<tr>
<td>147.</td>
<td>Chrome yellow</td>
<td>3.3</td>
<td>-</td>
<td>0.8-5</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>148.</td>
<td>Rhodamine B</td>
<td>-</td>
<td>550-</td>
<td>(0.0021,</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>32K</td>
</tr>
<tr>
<td>149.</td>
<td>Azomethine hypophosphorous acid</td>
<td>3-8</td>
<td>495</td>
<td>-</td>
<td>0.15</td>
<td>-</td>
<td>36K</td>
<td></td>
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</tbody>
</table>

<p>| L. MISCELLANEOUS REAGENTS FOR COLORIMETRIC DETERMINATION OF FERRIC IONS |
|-----------------------------|------------------|------------------|------------------|
| 150. Alloxantine            | -                | -                | 1.0              | -    | -                                      | 1L                |
| 151. Pyramidone             | 0.1              | -                | 0.5-3            | -    | -                                      | 2L                |
| 152. Hematoxylin            | -                | -                | (0.30)           | -    | -                                      | 4L                |
| 153. Sodium phenyl pyruvate | 3.5              | -                | 2.5              | -    | -                                      | 5L                |
| 154. Hesperetin             | 6.5              | 480 (13800)      | 1-10             | -    | Cu²⁺, Be²⁺, UO₂²⁺, Al³⁺, PO₄³⁻, CO₃²⁻ | 6L                |
| 155. Meconic acid           | 1.0              | -                | (0.5)            | -    | -                                      | 7L                |</p>
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<tr>
<td>156.</td>
<td>Alumocreson</td>
<td>4.4</td>
<td>530</td>
<td>-</td>
<td>2-20</td>
<td>-</td>
<td>-</td>
<td>8L</td>
</tr>
<tr>
<td>157.</td>
<td>Ethylcyano malonate</td>
<td>2-3</td>
<td>504</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>9L</td>
</tr>
<tr>
<td></td>
<td>(3600)</td>
<td></td>
<td></td>
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<td></td>
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</tr>
<tr>
<td>158.</td>
<td>Nalidixic acid (Negram)</td>
<td>3-4</td>
<td>410</td>
<td>-</td>
<td>0.43-</td>
<td>-</td>
<td>17.05</td>
<td>12L</td>
</tr>
<tr>
<td>159.</td>
<td>Sodium benzilate</td>
<td>-</td>
<td>315</td>
<td>-</td>
<td>0.05-</td>
<td>-</td>
<td>0.112</td>
<td>14L</td>
</tr>
<tr>
<td>160.</td>
<td>Salicylic acid formaldehyde polymer</td>
<td>5.5</td>
<td>500</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>18L</td>
</tr>
<tr>
<td>161.</td>
<td>Potassium ethylxanthate</td>
<td>10</td>
<td>428</td>
<td>-</td>
<td>3.27</td>
<td>-</td>
<td>-</td>
<td>17L</td>
</tr>
<tr>
<td></td>
<td>or</td>
<td></td>
<td>465</td>
<td></td>
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1. Organic analytical reagents I.  
   F.J. Welcher,  

2. Organic analytical reagents. II.  
   F.J. Welcher,  

3. Organic analytical reagents, III.  
   F.J. Welcher,  

4. Organic analytical reagents, IV.  
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M. Grat Cabanac,

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L.H. Li,

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R.W. Ramette,

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L.Z. Beizerman,
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E. Jackwerth, E.L. Schneider,

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W. Kemula, K. Brajter, S. Cieślik,

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E.A. Brown,

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R.E. Kitson,

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29K. Varianmine blue as a colorimetric reagent III. The
determination of vanadium and chromium.
L. Erdey, F. Szabadvary,

30K. Spectrophotometric determination of ferric ions in
surface waters with varianmine blue.
Z. Gregorowicz, T. Sunwinksa,

31K. Chromoxane violet R. reagent for total and separate
determination of microgram quantities of aluminium
and iron I. Determination of aluminium and iron in
natural waters.
N.F. Lisenko, I.S. Mustafin, L.A. Molot,

32K. Analytical characteristics of phenolic acids of the
triphenylmethane series. Determination of aluminum
and iron in certain metals.
I.S. Mustafin, N.F. Lisenko,

33K. New Microchemical method for specific research and
the colorimetric determination of the ferric ion.
C. Ott,

34K. Use of arsenazo reagent for determining rate ele­
ments.
A.F. Kuteinikov,
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S.C. Liang,

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A. Korkuc,
8L. Colorimetric determination of iron with meconic acid.
G. Mannelli, R. Biffoli,

9L. Alumocreson, an improved reagent of the aluminum type.
L.M. Kul'tberg, L.A. molot,

10L. Spectrophotometric investigation of the cyanomalonic ion. Application to the spectrophotometric determination of Fe$^{3+}$.
G. Mignonac, R. Miguel, C. Bonnemaison,

11L. Spectrophotometric determination of iron III.
S.S. Gupta, D. Mukerjee,

12L. Spectrophotometric determination of iron(III) with nalidixic acid in the presence of the most frequently accompanying elements.
I. Dick, N. Murgu,
Revta Chim. 16(10), 515-17 (1965); A.A. 14, 698 (1967).

13L. Spectrophotometric study of the iron(III) complex with nalidixic acid (Negram).
I. Dick, N. Murgu,

14L. Reaction of n-monobutyl maleate with metallic iron.
A.Kh. Uzbekova, A.M. Frost,

15L. Spectrophotometric determination of Fe(III).
S.S. Gupta, D. Mukerjee,

16L. Studies of calcichrome as a spectrophotometric reagent II. Determination of iron (III).
H. Ishii, H. Einaga,

17L. Spectrophotometric study of the system iron (III) - Potassium ethylxanthate.
C. Isvoranu-Panait, D. Negoin, M. Marica,
18L. Spectrophotometric determination of ferric ion with salicylic acid-formaldehyde polymer.
P. Umapathy, A.M. Hundekar, D.N. Sen,

19L. Reaction of iron III with maltol,
A. Stefanonic, J. Havel, L. Sommer,
A solution of the reagents in 40% ethyl alcohol was used to investigate the characteristics of ferric complexes by spectrophotometric methods and to investigate the possibilities of using the title compounds as reagents for the colorimetric determination of iron.

(a) Effect of pH:
All the four compounds give a violet colour with ferric ions at pH values below 3.0, whereas above pH 3.0, the complexes are reddish brown, the latter being unstable. The complexes were investigated below pH 3.0 and adjustments of pH were made by addition of dilute nitric acid. The absorption characteristics of the complexes in the pH range 2.0 to 3.0 are shown in Fig. JV-1, page 305. It can be seen that the maximum colour of the ferric salicylaldehyde complex is shown in the pH range 2.6 to 2.9, the corresponding range for orthohydroxy ketones being 2.5 to 2.8. The maximum absorption of light is shown by ferric salicylaldehyde complex at 520 mu and by remaining complexes at 530 mu. Between pH 2.0 and 3.0, the complexes are stable for:
The depth of the colours is not affected by sequence of adding the reagent.

(b) Absorption measurements:
The absorption spectra of the mixtures of equimolecular solutions of ferric nitrate and reagents in the ratio 1:1, 1:2 and 1:3 were measured between 400 and 600 μm. After adjusting the desired pH of the mixtures, the absorbancies were measured after half an hour. At any pH between 2.0 and 3.0, the wavelength of maximum absorbance is 520 μm for salicylaldehyde and 530 μm for orthohydroxy ketones. This suggests that under the conditions of the study, only one complex between ferric ions and reagent is formed. The absorption characteristics of these mixtures at optimum pH are given in Fig. V-2, p 305.

(c) Composition of the complex:
The composition of ferric complexes was studied by mole ratio method, method of continuous variation, and the slope ratio method.

(i) Mole Ratio Method:
A series of solutions were prepared from equimolar
solutions of ferric nitrate and the reagent. The concentration of ferric solution was kept constant and concentration of the reagent was varied from 1:0.2 to 1:20.0. The ionic strength was maintained at 0.1M by adding sodium perchlorate solution and the concentration of alcohol in final dilution was maintained at 16%. The pH was adjusted to 2.7 for salicylaldehyde and 2.6 in case of the other reagents and after allowing the colour to stabilise for 15 minutes, the absorbance of the mixture was measured at the wave length at which maximum absorption is shown. Results are shown in Fig. \textit{V-3}, page 290.

For all the four compounds, the graph is a straight line up to molar composition 1.0:1.0 and then it bends. A large excess of the reagents is needed to bring about the colour saturation, which indicates the highly dissociating nature of the complexes. The extinction coefficients calculated from maximum absorbance are given in Table \textit{V-2}, page 290.

(ii) Method of continuous variation:

A series of solutions were prepared by mixing x ml. of 0.01M ferric nitrate solution with (12-x) ml. of the reagent solution. The ionic strength was maintained at 0.1M by adding sodium perchlorate solution. The pH was adjusted at optimum value, and final volume was made up to 50 ml. Fig. \textit{V-4}, page 290 shows the results.
obtained by using equimolecular and non-equimolecular solutions. In all the cases, the maximum absorbance is shown by the mixtures corresponding to 1:1 proportion suggesting the formation of 1:1 complexes.

(iii) Slope Ratio Method:

Two series of solutions were prepared at optimum pH and ionic strength was maintained at 0.1M. In the first series, the concentration of the reagent was kept constant and in sufficient excess to make the dissociation of complex negligible and the concentration of ferric ions was varied. In the second set, the concentration of ferric ions was kept constant and that of the reagent was varied. (Due corrections were made for the absorbance of unreacted iron). Fig. V-5, page 367 shows a plot of absorbance of the complex against the concentration of variable component. The lines are parallel to each other, thus having the same values for the slope. This shows that the molar composition of the complexes is 1:1. The values of molecular extinction coefficients calculated from the intercepts of plots are given in Table V-2, page 386.

(d) Evaluation of Stability Constants:
The data obtained in mole ratio method and Job's method were used to calculate the stability constants of the complex. The stability constant of the complex may be calculated using the relation,
Where \( \alpha \) = degree of dissociation,
\( c \) = concentration,
\( m \) and \( n \) are integral numbers.

The values of \( m \) and \( n \) having been determined, \( \alpha \) may be calculated by the relation,

\[
\alpha = \frac{E_m - E_s}{E_m}
\]

Where \( E_m \) = maximum absorbance
\( E_s \) = absorbance at the stoichiometric ratio.

The stability constant of the complex may also be calculated using the relation,

\[
k = \frac{x}{(a - mx)^m (b - nx)^n}
\]

Where \( a \) = concentration of the ferric ions,
\( b \) = concentration of the reagent,
\( x \) = concentration of the complex calculated from the observed absorbance and the molecular extinction coefficient,
\( m \) and \( n \) are integral numbers.

The molar absorptivity and the stability constant can also be evaluated by the relation,
\[ A = E_c b_k \frac{(M)(R)}{1, k(R)} \]  \hspace{1cm} (4)

Where \( E_c \) = molar absorptivity of the complex,
\( c \) = concentration of the complex,
\( b \) = optical path length,
\( M \) = molar concentration of ferric ions,
\( R \) = molar concentration of the reagent,
\( k \) = stability constant of the complex.

The relation (4) used in the form of the equation,
\[ \frac{M(b)}{A} = \frac{1}{E_c} + \frac{1}{k \cdot E_0 \cdot R} \]  \hspace{1cm} (5)

is valid for 1:1 mole ratio complexes and for the systems in which the absorbance of uncomplexed metal ions at the wavelength used is negligible. If the values of \( \frac{M(b)}{A} \) calculated from the mole ratio data are plotted against \( 1/R \) the values of \( E_c \) and \( k \) can be calculated respectively from the intercept and the slope.

From Fig. \( V-6 \), page 307, the values of \( E_m \) and \( E_s \) are obtained. The value of \( n \) for the complex having been determined the values of \( k \) and \( E_c \) can be calculated by relations (1) and (2) respectively. Results are given in Table \( V-3 \), page 309.

The stability constants were calculated by assuming the
values of \( n = 1, \ n = 2 \) or \( n = 3 \). Results are shown in Table IV-4, page 310. The values are fairly constant for \( n = 1 \) indicating the formation of 1:1 complexes. The mole ratio data was also utilised for graphical evaluation of the stability constant and molar absorptivity with the help of equation 5 (Fig. IV-6, page 307). From the graph, the values of \( E_c \) and \( K \) were calculated (Table IV-3, page 309). The stability constants calculated from the data obtained in the method of continuous variation using the relations 1 and 2 are given in Table IV-6, page 312. It can be seen that the values of stability constants show a fair agreement.

(e) Sensitivity and optimum concentration range:

The sensitivity of a coloured complex can be observed by preparing a series of solutions in which the concentration of ferric ions is varied. A known excess of the reagent is added, and the pH value is adjusted to the optimum. The lowest concentration of ferric ions, which gives a difference of 0.2% in transmittance (\( \Delta \)) in the blank and sample solutions is termed sensitivity. The sensitivity values are as follows:

(1) Salicylaldehyde - 2.24 p.p.m.; (2) o-hydroxyacetophenone - 2.80 p.p.m.; (3) o-hydroxypropiophenone - 3.36 p.p.m.; (4) o-hydroxybutyrothenone - 4.2 p.p.m.
The optimum concentrations (p.p.m.) are as follows:

(1) Salicylaldehyde: 9.08 - 34.8; (2) o-hydroxyacetophenone: 10.6 - 37.2; (3) o-hydroxypropiophenone: 11.2 - 39.2; (4) o-hydroxybutyrophenone: 11.5 - 41.1.

(f) Beer's law:
The system obeys Beer's Law in the range 1-56 p.p.m. Higher and lower concentrations were not investigated.

(g) Effect of Diverse Ions:
To determine whether diverse ions would interfere, tolerance limits were established for the process described. To 2.0 ml. of a 0.005M solution of ferric nitrate, 25 ml. of 0.01M reagent were added. pH was adjusted to optimum value, and the final volume was made up to 50 ml. with distilled water. The absorbance was measured against a blank containing corresponding concentration of ferric ions at λ max.

A limit of 3.0% change in absorbance was arbitrarily taken as a limiting concentration. Then, if the absorbance of any sample containing 11.2 p.p.m. iron and a diverse ion had an absorbance beyond the range this ion was said to interfere. The tolerable amount was calculated on the assumption that Beer's Law was valid for the interfering ions. Corresponding blanks
at optimum pH values were always carried out and due corrections made. The diverse ions were added to give a final concentration of 1000 p.p.m. If an ion interfered seriously at this concentration, it was again studied at a lower concentration, and calculations were made. This was continued till the tolerance limit was obtained. All solutions were prepared exactly as in the procedure described above, except that the diverse ion was added to the 50 ml. volumetric flask just after the ferric solutions. Effect of diverse ions on the absorbance of the ferric complexes is given in Table \( V-7 \), page 313.

(h) Standard deviation:
Six determinations were made in the above-mentioned manner and the mean absorbances were calculated. The standard deviation was calculated by the formula:

\[
\sigma = \sqrt{\frac{\sum d^2}{n - 1}}
\]

The standard deviations are as follows:

Salicylaldehyde - ±0.9\%; o-hydroxyacetophenone - ±0.8\%; o-hydroxypropiophenone - ±0.7\%; o-hydroxybutyrophenone - ±0.4\%.

(i) Applications:
The reagents investigated were applied to the analysis of the synthetic solutions. Results are given in
The reagents were also studied for analysing the iron content of an indigenous ore. Results are given in Table V-9, page 317.
CORRELATION BETWEEN MOLECULAR STRUCTURE OF PHENOLIC COMPOUNDS AND THEIR CHELATING ACTION TOWARDS FERRIC IONS

It is well known that phenols, phenolic aldehydes and ketones, phenolic aldoximes and ketoximes, phenolic carboxylic acids, and their derivatives give distinct colours with ferric salts. However, a systematic data on the correlation of molecular structure with the performance of the substance as a colorimetric reagent for iron is lacking in literature. The work in this laboratory has been planned, with a view to

1. observe the effect of modification of molecular structure of the substance on its performance as a colorimetric reagent, and

2. search for new analytical reagents.

With this aim, the following compounds have been investigated as reagents:

2,4 dihydroxyacetophenone;1,8,9 2,4 dihydroxy propiophenone;2,8,9 2,4 dihydroxybutyrophenone;3,8,9
2,4 dihydroxypropiophenone oxime;4,8,9 2,4 dihydroxybutyrophenone oxime;5,8,9 2,hydroxypropiophenone oxime;6,8,9 2,hydroxy butyrophenone oxime7,8,9
2,6 dihydroxy acetophenone;10 2,4,6 trihydroxy
acetophenone; salicylaldehyde; o-hydroxyacetophenone; o-hydroxypropiophenone; and o-hydroxy butyrophenone; \( \beta \) resorcylande 2,5 dihydroxyacetophenone; 4 hydroxy 3 acetylbenzoic acid; 4 hydroxy 3 propionylbenzoic acid; 4 hydroxy 3 butyryl benzoic acid; 4 hydroxy 3 acetyloxime benzoic acid; 4 hydroxy 3 propionyloxime benzoic acid; 4 hydroxy 3 butyryl oxime benzoic acid.

A summary of the ferric complexes investigated is given in Table IV-10. A comparison of the results obtained with salicylaldehyde (X); and orthohydroxyacetophenone (I) indicates that the replacement of H atom of aldehyde group by a methyl group does not influence extinction coefficient and sensitivity remarkably, but it does increase the selectivity.

A comparison of the results obtained with o-hydroxyacetophenone (I); 2,4 dihydroxyacetophenone (IV); 2,5 dihydroxyacetophenone (VII); 2,6 dihydroxyacetophenone (VIII); and 2,4,6 trihydroxyacetophenone (IX) shows that, introduction of another hydroxy group in positions 5 and 6 is unfavourable. 2,5 dihydroxy acetophenone gives an unstable green colour with ferric ions, which decomposes in a few minutes, whereas 2,6 dihydroxy acetophenone gives a less sensitive
colour than orthohydroxyacetophenone. Moreover, it was observed that 2,6 dihydroxyacetophenone is less selective than orthohydroxyacetophenone.

In contrast, the substitution of the H atom in position No. 4 of 2,4 dihydroxyacetophenone casts a very favourable influence, as shown by the fact that 2,4 dihydroxyacetophenone is a more selective and sensitive reagent than orthohydroxy acetophenone. However, introduction of third OH group in the nucleus does not seem to be very beneficial as 2,4,6 trihydroxyacetophenone is less sensitive and selective reagent than 2,4 dihydroxyacetophenone. Thus, the introduction of the third OH group in position No. 6 off sets the beneficial action of the section OH group in position No. 4.
The beneficial action of the substitution of H atom in position No. 4 with OH group is confirmed by a comparison of the results obtained with (1) o-hydroxypropiophenone and 2,4 dihydroxypropiophenone and (2) o-hydroxybutyrophenone and 2,4 dihydroxybutyrophenone.

It is interesting to note that the substitution of H atom in position No. 4 of orthohydroxyacetophenone by a -COOH group is unfavourable. 4 hydroxy 3 acetyl benzoic acid, 4 hydroxy 3 propionyl benzoic acid and 4 hydroxy 3 butyryl benzoic acid give unstable colours of very low intensity with ferric ions whereas the corresponding oximes tend to give precipitates with higher concentrations of ferric ions.

The present work confirms an earlier observation that homologous compounds of the same series show the same optimum pH range and wave length of maximum absorbance, but with the increase in the number of carbon atoms in the side chain, the extinction coefficient and sensitivity decreases, whereas selectivity increases. This can be easily seen by a comparison of the results obtained with

(1) o-hydroxyacetophenone, o-hydroxypropiophenone and o-hydroxybutyrophenone

(2) 2,4 dihydroxyacetophenone, 1,8 2,4 dihydroxypropiophenone, 2,8 and 2,4 dihydroxybutyrophenone 3,8
FIG. V - 1  Spectral Characteristics of the Ferric Complexes at various pH values.
(2 ml. 0.01M ferric nitrate + 20 ml. 0.01M reagent + 5 ml. 1M Sodium perchlorate, final volume 50 ml.)

<table>
<thead>
<tr>
<th>Compound</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>Salicylaldehyde</td>
<td>A 2.6-2.9</td>
</tr>
<tr>
<td></td>
<td>B 2.5</td>
</tr>
<tr>
<td></td>
<td>C 2.1</td>
</tr>
<tr>
<td>o-hydroxyacetophenone</td>
<td>D 2.5-2.8</td>
</tr>
<tr>
<td></td>
<td>E 2.3</td>
</tr>
<tr>
<td></td>
<td>F 2.0</td>
</tr>
<tr>
<td>o-hydroxypropiophenone</td>
<td>G 2.5-2.8</td>
</tr>
<tr>
<td></td>
<td>H 2.3</td>
</tr>
<tr>
<td></td>
<td>I 2.0</td>
</tr>
<tr>
<td>o-hydroxybutyrophenone</td>
<td>J 2.5-2.8</td>
</tr>
<tr>
<td></td>
<td>K 2.3</td>
</tr>
<tr>
<td></td>
<td>L 2.0</td>
</tr>
</tbody>
</table>

FIG. V - 2  Absorption spectra of ferric complexes.

Salicylaldehyde - pH 2.7, o-hydroxyketones - pH 2.6

<table>
<thead>
<tr>
<th>Spectra</th>
<th>Solution details</th>
</tr>
</thead>
<tbody>
<tr>
<td>A, D, G, J</td>
<td>2 ml. 0.005M Ferric nitrate + 2 ml. 0.005M reagent</td>
</tr>
<tr>
<td>B, E, H, K</td>
<td>2 ml. 0.005M ferric nitrate + 4 ml. 0.005M reagent</td>
</tr>
<tr>
<td>C, F, I, L</td>
<td>2 ml. 0.005M ferric nitrate + 6 ml. 0.005M reagent</td>
</tr>
</tbody>
</table>

(μ = 0.1M NaClO₄, final volume 50 ml.)
Fig. V-3 Composition of the ferric complexes by the mole ratio method:

Salicylaldehyde - pH 2.7, $\lambda_{\text{max}}$ 520 nm

$\alpha$-hydroxyketones - pH 2.6, $\lambda_{\text{max}}$ 530 nm

(2 ml 0.005M ferric nitrate + varying amounts of 0.005M reagent + 5 ml 1.0M sodium perchlorate, final volume 50 ml.)

Fig. V-4 Composition of ferric complexes by the method of continuous variation:

Salicylaldehyde - pH 2.7, $\lambda_{\text{max}}$ 520 nm

$\alpha$-hydroxyketones - pH 2.6, $\lambda_{\text{max}}$ 530 nm

x ml 0.005M ferric nitrate + 12 - x ml reagent (0.01M in plots A,D,G,J; 0.005M in plots B,E,H,K; 0.0025M in plots C,F, I,L) + 5 ml 1.0M NaClO$_4$, final volume 50 ml.)
FIG. V-5 Composition of ferric complexes by the slope ratio method.

Salicylaldehyde - pH 2.7, λ 520 μm
o-hydroxyketones - pH 2.6, λ 530 μm

A,C,E,G - 15 ml. 0.01M reagent + varying amounts of 0.005M reagent + 5 ml. 1M sodium perchlorate.

B,D,F,G - 15 ml. 0.01M ferric nitrate + varying amounts of 0.005M reagent + 5 ml. 1M sodium perchlorate, final volume 50 ml.

FIG. V-6 Evaluation of Stability Constants.
<table>
<thead>
<tr>
<th></th>
<th>From mole ratio data</th>
<th>From slope ratio method</th>
<th>From mole ratio method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Salicylaldehyde</td>
<td>1125</td>
<td>1100</td>
<td>1114</td>
</tr>
<tr>
<td>o-hydroxyacetophenone</td>
<td>1050</td>
<td>1020</td>
<td>1053</td>
</tr>
<tr>
<td>o-hydroxypropiophenone</td>
<td>1000</td>
<td>990</td>
<td>1000</td>
</tr>
<tr>
<td>o-hydroxybutyrophenone</td>
<td>975</td>
<td>960</td>
<td>952</td>
</tr>
</tbody>
</table>
TABLE V-3

GRAPHICAL EVALUATION OF EXTINCTION COEFFICIENTS AND STABILITY CONSTANTS USING THE RELATION

\[
\frac{M(b)}{A} = \frac{1}{E_c} \cdot \frac{1}{K \cdot E_c \cdot r}
\]

<table>
<thead>
<tr>
<th></th>
<th>(E_c)</th>
<th>(\kappa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Salicylaldehyde</td>
<td>(1.114 \times 10^3)</td>
<td>(1.591 \times 10^3)</td>
</tr>
<tr>
<td>o-hydroxy acetophenone</td>
<td>(1.053 \times 10^3)</td>
<td>(1.170 \times 10^3)</td>
</tr>
<tr>
<td>o-hydroxy propiophenone</td>
<td>(1.000 \times 10^3)</td>
<td>(1.060 \times 10^3)</td>
</tr>
<tr>
<td>o-hydroxy butyrophenone</td>
<td>(0.952 \times 10^3)</td>
<td>(0.805 \times 10^3)</td>
</tr>
<tr>
<td>Ratio</td>
<td>$n = 1$</td>
<td>$n = 2$</td>
</tr>
<tr>
<td>-------</td>
<td>--------</td>
<td>--------</td>
</tr>
<tr>
<td>Fe:R</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1:0.2</td>
<td>1.49x10³</td>
<td>9.42x10⁷</td>
</tr>
<tr>
<td>1:0.4</td>
<td>1.35x10³</td>
<td>7.77x10⁷</td>
</tr>
<tr>
<td>1:0.6</td>
<td>1.57x10³</td>
<td>3.16x10⁷</td>
</tr>
<tr>
<td>1:0.8</td>
<td>1.74x10³</td>
<td>2.74x10⁷</td>
</tr>
<tr>
<td>1:1.0</td>
<td>1.75x10³</td>
<td>2.60x10⁷</td>
</tr>
<tr>
<td>1:1.2</td>
<td>1.68x10³</td>
<td>8.30x10⁶</td>
</tr>
<tr>
<td>1:1.3</td>
<td>1.63x10³</td>
<td>4.73x10⁶</td>
</tr>
<tr>
<td>1:1.4</td>
<td>1.47x10³</td>
<td>2.87x10⁶</td>
</tr>
<tr>
<td>1:1.5</td>
<td>1.41x10³</td>
<td>2.66x10⁶</td>
</tr>
<tr>
<td>1:1.8</td>
<td>1.62x10³</td>
<td>1.36x10⁶</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>o-hydroxyacetophenone</th>
</tr>
</thead>
<tbody>
<tr>
<td>$n = 1$</td>
</tr>
<tr>
<td>1:0.2</td>
</tr>
<tr>
<td>1:0.4</td>
</tr>
<tr>
<td>1:0.6</td>
</tr>
<tr>
<td>1:0.8</td>
</tr>
<tr>
<td>1:1.0</td>
</tr>
<tr>
<td>1:1.2</td>
</tr>
<tr>
<td>1:1.3</td>
</tr>
<tr>
<td>1:1.4</td>
</tr>
<tr>
<td>1:1.5</td>
</tr>
<tr>
<td>1:1.8</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>o-hydroxypropiophenone</th>
</tr>
</thead>
<tbody>
<tr>
<td>$n = 1$</td>
</tr>
<tr>
<td>1:0.2</td>
</tr>
<tr>
<td>1:0.4</td>
</tr>
<tr>
<td>1:0.6</td>
</tr>
<tr>
<td>1:0.8</td>
</tr>
<tr>
<td>1:1.0</td>
</tr>
<tr>
<td>1:1.2</td>
</tr>
<tr>
<td>1:1.3</td>
</tr>
<tr>
<td>1:1.4</td>
</tr>
<tr>
<td>1:1.5</td>
</tr>
<tr>
<td>1:1.8</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>o-hydroxybutyrophenone</th>
</tr>
</thead>
<tbody>
<tr>
<td>$n = 1$</td>
</tr>
<tr>
<td>1:0.2</td>
</tr>
<tr>
<td>1:0.4</td>
</tr>
<tr>
<td>1:0.6</td>
</tr>
<tr>
<td>1:0.8</td>
</tr>
<tr>
<td>1:1.0</td>
</tr>
<tr>
<td>1:1.2</td>
</tr>
<tr>
<td>1:1.3</td>
</tr>
<tr>
<td>1:1.4</td>
</tr>
<tr>
<td>1:1.5</td>
</tr>
<tr>
<td>1:1.8</td>
</tr>
</tbody>
</table>
TABLE IV-5
STABILITY CONSTANTS FROM THE MOLE RATIO DATA USING THE RELATION

\[ k = \frac{1 - \alpha}{\alpha^2 C} \]

<table>
<thead>
<tr>
<th>Reagent</th>
<th>Em</th>
<th>Es</th>
<th>( \alpha )</th>
<th>( k )</th>
</tr>
</thead>
<tbody>
<tr>
<td>o-hydroxy acetophenone</td>
<td>0.210</td>
<td>0.045</td>
<td>0.786</td>
<td>1.73 \times 10^3</td>
</tr>
<tr>
<td>o-hydroxy propiophenone</td>
<td>0.200</td>
<td>0.040</td>
<td>0.800</td>
<td>1.56 \times 10^3</td>
</tr>
<tr>
<td>o-hydroxy butyrophenone</td>
<td>0.195</td>
<td>0.035</td>
<td>0.820</td>
<td>1.37 \times 10^3</td>
</tr>
<tr>
<td>Salicylaldehyde</td>
<td>0.225</td>
<td>0.052</td>
<td>0.769</td>
<td>1.77 \times 10^3</td>
</tr>
</tbody>
</table>
TABLE Jjfo-0

STABILITY CONSTANTS FROM THE CONTINUOUS VARIATION DATA USING THE RELATION

\[ k = \frac{1 - \alpha}{\alpha^2 c} \]

<table>
<thead>
<tr>
<th>Reagent</th>
<th>p</th>
<th>Theoretical absorbance</th>
<th>Observed absorbance</th>
<th>( \alpha )</th>
<th>( k )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Salicylaldehyde</td>
<td>2.0</td>
<td>0.90</td>
<td>0.38</td>
<td>0.577</td>
<td>( 1.59 \times 10^3 )</td>
</tr>
<tr>
<td>-do-</td>
<td>1.0</td>
<td>0.675</td>
<td>0.25</td>
<td>0.629</td>
<td>( 1.56 \times 10^3 )</td>
</tr>
<tr>
<td>-do-</td>
<td>0.5</td>
<td>0.45</td>
<td>0.15</td>
<td>0.668</td>
<td>( 1.86 \times 10^3 )</td>
</tr>
<tr>
<td>o-hydroxy acetophenone</td>
<td>2.0</td>
<td>0.84</td>
<td>0.32</td>
<td>0.619</td>
<td>( 1.24 \times 10^3 )</td>
</tr>
<tr>
<td>-do-</td>
<td>1.0</td>
<td>0.63</td>
<td>0.22</td>
<td>0.650</td>
<td>( 1.38 \times 10^3 )</td>
</tr>
<tr>
<td>-do-</td>
<td>0.5</td>
<td>0.42</td>
<td>0.12</td>
<td>0.714</td>
<td>( 1.40 \times 10^3 )</td>
</tr>
<tr>
<td>o-hydroxy propiophenone</td>
<td>2.0</td>
<td>0.80</td>
<td>0.30</td>
<td>0.625</td>
<td>( 1.22 \times 10^3 )</td>
</tr>
<tr>
<td>-do-</td>
<td>1.0</td>
<td>0.60</td>
<td>0.190</td>
<td>0.683</td>
<td>( 1.13 \times 10^3 )</td>
</tr>
<tr>
<td>-do-</td>
<td>0.5</td>
<td>0.40</td>
<td>0.11</td>
<td>0.725</td>
<td>( 1.31 \times 10^3 )</td>
</tr>
<tr>
<td>o-hydroxy butyrophenone</td>
<td>2.0</td>
<td>0.78</td>
<td>0.29</td>
<td>0.628</td>
<td>( 1.18 \times 10^3 )</td>
</tr>
<tr>
<td>-do-</td>
<td>1.0</td>
<td>0.585</td>
<td>0.18</td>
<td>0.692</td>
<td>( 1.07 \times 10^3 )</td>
</tr>
<tr>
<td>-do-</td>
<td>0.5</td>
<td>0.39</td>
<td>0.10</td>
<td>0.718</td>
<td>( 1.37 \times 10^3 )</td>
</tr>
</tbody>
</table>
### Table V-7

**Influence of Diverse Ions on the Absorbance of Ferric Complexes**

<table>
<thead>
<tr>
<th>Ion Added as</th>
<th>Concentration of Iron (p.p.m.)</th>
<th>Change Tolerance in Limit (p.p.m.)</th>
<th>Change Tolerance in Limit (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al&lt;sup&gt;+++&lt;/sup&gt; sulphate</td>
<td>150</td>
<td>-0.5</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>200</td>
<td>-</td>
<td>200</td>
</tr>
<tr>
<td>Ba&lt;sup&gt;++&lt;/sup&gt; nitrate</td>
<td>400</td>
<td>0.0</td>
<td>400</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Be&lt;sup&gt;++&lt;/sup&gt; nitrate</td>
<td>400</td>
<td>0.0</td>
<td>400</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Cd&lt;sup&gt;++&lt;/sup&gt; chloride</td>
<td>150</td>
<td>+1.5</td>
<td>150</td>
</tr>
<tr>
<td></td>
<td>500</td>
<td>0.0</td>
<td>500</td>
</tr>
<tr>
<td>Cs&lt;sup&gt;+&lt;/sup&gt; nitrate</td>
<td>500</td>
<td>0.0</td>
<td>500</td>
</tr>
<tr>
<td>Ca&lt;sup&gt;++&lt;/sup&gt; nitrate</td>
<td>400</td>
<td>+2.0</td>
<td>400</td>
</tr>
<tr>
<td>Cr&lt;sup&gt;+++&lt;/sup&gt; nitrate</td>
<td>300</td>
<td>0.0</td>
<td>300</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

Reagent = 0.006 M

Salicylaldehyde (I) 0.225 (Mean absorbance) o-hydroxyacetophenone (II) 0.210 (Mean absorbance) o-hydroxypropiophenone (III) 0.200 (Mean absorbance) o-hydroxybutyrophenone (IV) 0.195 (Mean absorbance)
<table>
<thead>
<tr>
<th>Ion Added as</th>
<th>Change Tolerance in Limit Absorbance (p.p.m.)</th>
<th>Concentration (p.p.m.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Co** nitrate</td>
<td>+0.5</td>
<td>200</td>
</tr>
<tr>
<td>Fe** nitrate</td>
<td>100</td>
<td>+0.5</td>
</tr>
<tr>
<td>Pb** nitrate</td>
<td>200</td>
<td>+0.5</td>
</tr>
<tr>
<td>Mg** nitrate</td>
<td>400</td>
<td>+0.5</td>
</tr>
<tr>
<td>Mn** chloride</td>
<td>200</td>
<td>+0.5</td>
</tr>
<tr>
<td>K+ chloride</td>
<td>1000</td>
<td>+0.5</td>
</tr>
<tr>
<td>Ag+ chloride</td>
<td>1000</td>
<td>+0.5</td>
</tr>
<tr>
<td>Sr+ chloride</td>
<td>400</td>
<td>+0.5</td>
</tr>
<tr>
<td>Th+++ nitrate</td>
<td>400</td>
<td>+0.5</td>
</tr>
<tr>
<td>Ion</td>
<td>Added as</td>
<td>Concentration of iron (p.p.m.)</td>
</tr>
<tr>
<td>---------</td>
<td>----------</td>
<td>-------------------------------</td>
</tr>
<tr>
<td></td>
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<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zr⁺⁺⁺⁺</td>
<td>nitrate</td>
<td>400</td>
</tr>
<tr>
<td></td>
<td>nitrate</td>
<td>200</td>
</tr>
<tr>
<td>Zn⁺⁺</td>
<td>sulfate</td>
<td>400</td>
</tr>
<tr>
<td>NH₄⁺⁺</td>
<td>chloride</td>
<td>1000</td>
</tr>
<tr>
<td>Pd⁺⁺</td>
<td>chloride</td>
<td>200</td>
</tr>
<tr>
<td>Br⁻</td>
<td>KBr</td>
<td>50</td>
</tr>
<tr>
<td>Cl⁻</td>
<td>NaCl</td>
<td>1000</td>
</tr>
<tr>
<td>SO₄²⁻</td>
<td>Na₂SO₄</td>
<td>1000</td>
</tr>
<tr>
<td>I⁻</td>
<td>KI</td>
<td>20</td>
</tr>
<tr>
<td></td>
<td>10</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>+1.0</td>
</tr>
<tr>
<td>PO₄³⁻</td>
<td>Na₃PO₄</td>
<td>5</td>
</tr>
</tbody>
</table>
### Table

#### Analysis of Synthetic Solutions

<table>
<thead>
<tr>
<th>Sample, p.p.m.</th>
<th>Salicylaldehyde Absorbance</th>
<th>Fe+++ Found p.p.m.</th>
<th>o-Hydroxyacetophenone Absorbance</th>
<th>Fe+++ Found p.p.m.</th>
<th>o-Hydroxypropiophenone Absorbance</th>
<th>Fe+++ Found p.p.m.</th>
<th>o-Hydroxybutyrophenone Absorbance</th>
<th>Fe+++ Found p.p.m.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe+++ 11.2 + Ca++ 200 + Sr+++ 200</td>
<td>0.225 11.20</td>
<td>0.211 11.25</td>
<td>0.200 11.20</td>
<td>0.196 11.25</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe+++ 11.2 + Cu++ 2 + Ni+++ 50</td>
<td>0.226 11.25</td>
<td>0.211 11.25</td>
<td>0.200 11.20</td>
<td>0.195 11.20</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe+++ 11.2 + Cu++ 2 + Co+++ 100</td>
<td>0.225 11.20</td>
<td>0.210 11.20</td>
<td>0.199 11.15</td>
<td>0.195 11.20</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe+++ 11.2 + UO2+++ 100 + Th+++ 200</td>
<td>0.224 11.15</td>
<td>0.210 11.20</td>
<td>0.199 11.15</td>
<td>0.194 11.15</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe+++ 11.2 + Mn+++ 100 + Ni+++ 50</td>
<td>0.225 11.20</td>
<td>0.210 11.20</td>
<td>0.201 11.25</td>
<td>0.195 11.20</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Fe+++ 11.2 + Al+++ + Na+ 1000</td>
<td>0.225 11.20</td>
<td>0.209 11.15</td>
<td>0.200 11.20</td>
<td>0.195 11.20</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
An indigenous iron ore with the following percentage composition was analysed with the reagents investigated. \( \text{Fe}_2\text{O}_3 \) - 92.8; \( \text{Al}_2\text{O}_3 \) - 1.54; \( \text{SiO}_2 \) - 3.07; \( \text{CaO} \) - 0.3; \( \text{TiO} \) - 0.0; \( \text{MgO} \) - 0.28; \( \text{PO}_4 \) - 0.10; S - 0.03; 1.84 gms of the ore was brought into solution by standard method and diluted to 250 ml. A 2 ml aliquot of this stock solution was diluted to 100 ml, and 2 ml aliquots of the diluted solution was taken for colour development.

<table>
<thead>
<tr>
<th>Reagent</th>
<th>Observed absorbance</th>
<th>( \text{Fe}^{3+} ) (found) mg.</th>
<th>( \text{Fe}^{3+} ) (expected) mg.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Salicylaldehyde</td>
<td>0.075</td>
<td>3.73</td>
<td>3.82</td>
</tr>
<tr>
<td>2. o-hydroxy acetophenone</td>
<td>0.072</td>
<td>3.84</td>
<td>3.82</td>
</tr>
<tr>
<td>3. o-hydroxy propiophenone</td>
<td>0.070</td>
<td>3.92</td>
<td>3.82</td>
</tr>
<tr>
<td>4. o-hydroxy butyrophenone</td>
<td>0.068</td>
<td>3.90</td>
<td>3.82</td>
</tr>
<tr>
<td>Name of the complex</td>
<td>Optimum pH range</td>
<td>λ_{max} (m,\mu)</td>
<td>E_{max}</td>
</tr>
<tr>
<td>------------------------------</td>
<td>------------------</td>
<td>-----------------</td>
<td>--------</td>
</tr>
<tr>
<td>Ferric-salicylaldehyde</td>
<td>2.6 - 2.9</td>
<td>520</td>
<td>1.125x10³</td>
</tr>
<tr>
<td>Ferric-ortho hydroxy acetophenone</td>
<td>2.5 - 2.9</td>
<td>530</td>
<td>1.05x10³</td>
</tr>
<tr>
<td>Ferric-ortho hydroxy propiophenone</td>
<td>2.5 - 2.9</td>
<td>530</td>
<td>1.000x10³</td>
</tr>
<tr>
<td>Ferric-ortho hydroxy butyrophenone</td>
<td>2.5 - 2.9</td>
<td>530</td>
<td>0.975x10³</td>
</tr>
<tr>
<td>Ferric-2,4 dihydroxy acetophenone</td>
<td>2.9 - 3.0</td>
<td>470</td>
<td>2.104x10³</td>
</tr>
<tr>
<td>Ferric-2,4 dihydroxy propiophenone</td>
<td>2.9 - 3.0</td>
<td>470</td>
<td>1.378x10³</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>3</td>
<td>4</td>
</tr>
<tr>
<td>----------------------</td>
<td>-------</td>
<td>-------</td>
<td>------------</td>
</tr>
<tr>
<td>Ferric-2,4 dihydroxy</td>
<td>2.9 -</td>
<td>2.6</td>
<td>470</td>
</tr>
<tr>
<td>butyrophenone</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferric-2,6 dihydroxy</td>
<td>1.8 -</td>
<td>2.6</td>
<td>500</td>
</tr>
<tr>
<td>acetophenone</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferric-2,4,6 trihydroxy</td>
<td>2.0 -</td>
<td>2.96</td>
<td>510</td>
</tr>
<tr>
<td>acetophenone</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferric-2,hydroxy</td>
<td>2.0 -</td>
<td>2.5</td>
<td>420</td>
</tr>
<tr>
<td>acetophenone oxime</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferric-2,hydroxy</td>
<td>2.85 -</td>
<td>2.95</td>
<td>470</td>
</tr>
<tr>
<td>propiophenone oxime</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferric-2,hydroxy</td>
<td>2.85 -</td>
<td>2.95</td>
<td>510</td>
</tr>
<tr>
<td>butyrophenone oxime</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferric-2,4 dihydroxy</td>
<td>7.0</td>
<td></td>
<td>475</td>
</tr>
<tr>
<td>acetophenone oxime</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferric-2,4 dihydroxy</td>
<td>2.6 -</td>
<td>2.8</td>
<td>510</td>
</tr>
<tr>
<td>propiophenone oxime</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ferric-2,4 dihydroxy</td>
<td>2.6 -</td>
<td>2.8</td>
<td>510</td>
</tr>
<tr>
<td>butyrophenone oxime</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*General Absorption
<table>
<thead>
<tr>
<th>pH of 0.01M ferric nitrate solution</th>
<th>Reagent</th>
<th>pH of 0.1M Reagent</th>
<th>pH of Mixture</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.6</td>
<td>Salicylaldehyde</td>
<td>5.2</td>
<td>2.50</td>
</tr>
<tr>
<td>2.6</td>
<td>o-hydroxyacetophenone</td>
<td>5.0</td>
<td>2.48</td>
</tr>
<tr>
<td>2.6</td>
<td>o-hydroxypropiophenone</td>
<td>5.1</td>
<td>2.50</td>
</tr>
<tr>
<td>2.6</td>
<td>o-hydroxybutyrophenone</td>
<td>5.0</td>
<td>2.52</td>
</tr>
</tbody>
</table>