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SULPHONEPHTHALEINE INDICATORS, (U)
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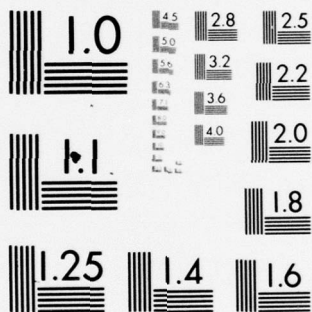
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FOREIGN TECHNOLOGY DIVISION



SULPHONEPHTHALEINE INDICATORS

by

O. Leminger



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FOREIGN TECHNOLOGY DIVISION
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SULPHONEPHTHALEINE INDICATORS

O. Leminger

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Sulphonephthaleine dyes have gradually become very important neutralizing and pH indicators. They were introduced into general analytical practice, became indispensable in industrial control, in food analysis and currently they are used in complexonometry (chelatometry). Processing of all these dyes as well as of basic raw materials for their preparation resulted from original research done by the Corporation for Chemical and Metallurgical Processing. This article presents a summary of existing literature and of obtained results.

Editors

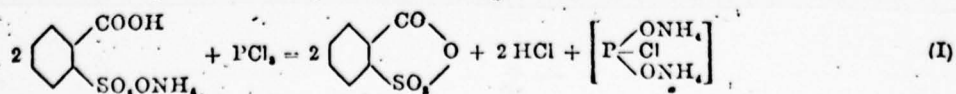
Some sulphonephthaleins^e represent important neutralizing and pH-indicators. They are especially important for the processing of pH-test papers and their importance is expected to grow even further. This article will briefly deal with technology and common properties of the most widely used sulphonephthaleins^e since the data on this subject are relatively sporadic and short while the literature on the analytical use as well as on physical - chemical data of sulphophthaleins^e is extensive and comprehensive. A special attention will be paid to our experiences, since we have technologically researched sulphonephthaleine indicators, introduced some new processing methods and for a certain time carried out their experimental production. At this time the most important sulphonphthaleine indicators are produced in larger quantities and of good quality by "Lachema" of Brno that obtained the results of our experiences. The promising production begins not only to satisfy our domestic consumption,

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but is also penetrating into foreign markets where to our expectation we will meet with success.

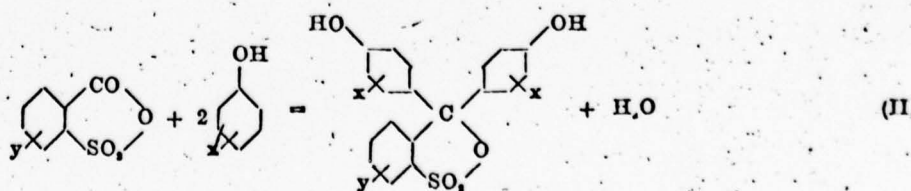
The most important raw material for the processing of sulphone-phthaleine indicators is presently an anhydride of o-sulphobenzoic acid, first obtained by Fahlberg and Barg while using acetylchloride on o-sulphobenzoic acid in benzene medium. Although the present literature describes a number of ways how to obtain the above anhydride, it is our opinion that of the truly technical importance are only those methods where the acid ammonium - or acid alkaline metal salt of o-sulphobenzoic acid is treated ^{with} suitable condensing agents. The above acid salts are relatively easily available; so f.e. the acid ammonium salt of a fairly high quantitative yield is obtained by breaking the ring of Fahlberg saccharin by means of hydrochloric acid²⁾. The literature on this subject describes the condensation combination of acid potassium salt with thionylchloride³⁾, with phosphorus pentaoxyde, with phosphorus pentachloride⁴⁾ at 400°C⁵⁾ and of acid ammonium salt with thionylchloride^{6) 7).}

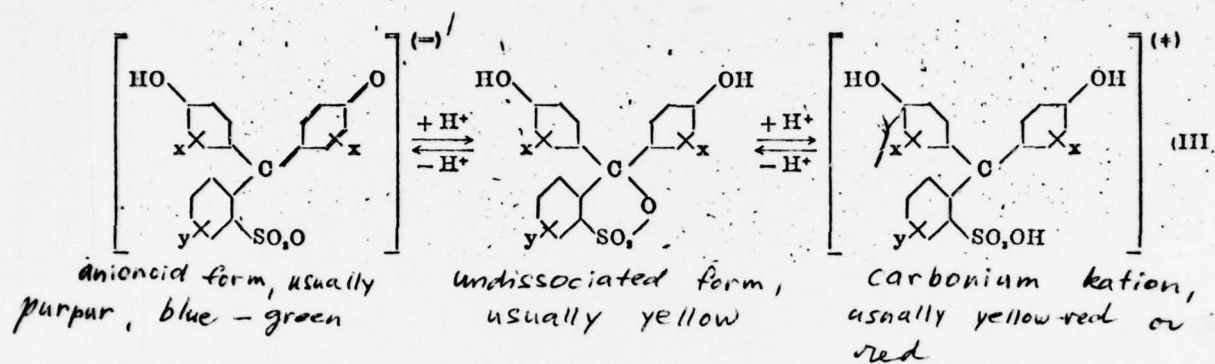
We have found that these methods require a relatively long reaction time and result in average yield (max. 65%) of o-sulphobenzoic anhydride of average quality, which usually requires its further purification by vacuum distillation. We have found, however,^{8), 9)} that the considerably higher yields can be obtained when treating the acid ammonia salt of o-sulphobenzoic acid with phosphorus trichloride in the non-polar solvent medium, such as benzene, at higher temperatures (60-110°C). The respective reaction can be expressed



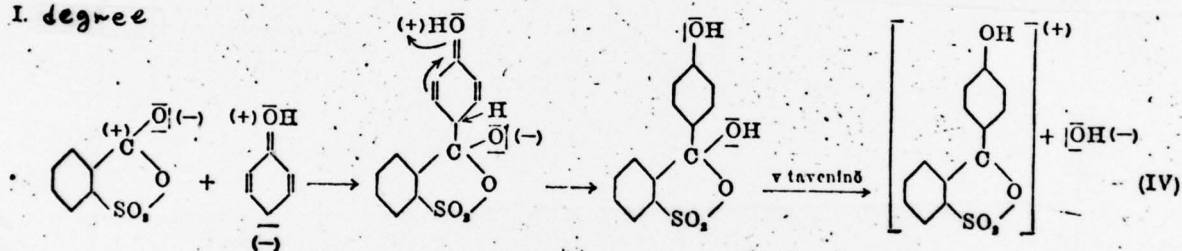
by the equation (I), that corresponds to the ratio of raw materials taken at highest yields of o-sulphobenzoic anhydride. The reaction is terminated in about 3 hours; then the non-soluble yellow phosphanic polymer, the composition of which is not yet fully analyzed, is sucked off and from the thickened filtrates then crystallizes a relatively very pure (about 98,5%) o-sulphobenzoic anhydride in the shape of large, colorless prisms, of 123-126°C melting point, corrected at the theoretical yield of about 86%. Aparature for this process is very simple, it consists of an enclosed enamelled vessel with a mixer, provided with a thermometer combined with reverse and stage cooler and with absorption equipment for reactive HCl, and further consists of an enclosed nutch filter and of a vacuum dryer.

O-sulphobenzoic anhydride is a very reactive substance, that combines with a great number of chemical individua. This feature resembles an anhydride of a phthalic acid. Its most important ability is to condense with phenols into sulphonphthaleins (usually in para-form), see II.





I. degree

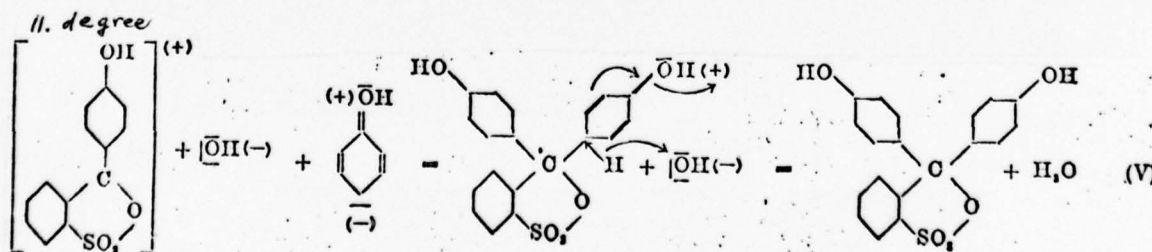


Sulphonphthaleins.

As follows from the above equation, sulphonphthaleins represent variously substituted anhydrized p, p'-dihydroxytriphenylcarbinol-o''-sulphonic acid. The sulphonphthaleins are especially characterized by constitutional changes due to various pH in aqueous solutions. These changes are expressed by a different and very intensive coloring, see III.

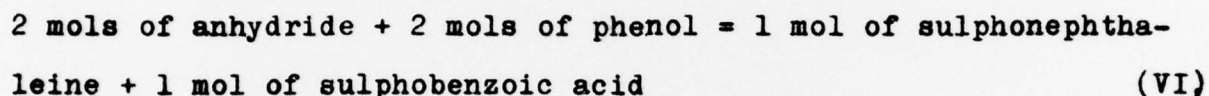
The above characteristics are generally used in volumetric analysis (color indicator titration) and in pH-metry. Sulphonphthaleins can be obtained by different methods, as f.e. by condensation of Fahlberg saccharin with phenols under the influence of concentrated H_2SO_4 ¹⁰⁾, though of the technical importance is primarily the above condensation of anhydride o-sulphobenzoic with phenols (II), that can be expressed by equations (IV) and (V) with electrons.

However here we deal only with an approximate interpretation



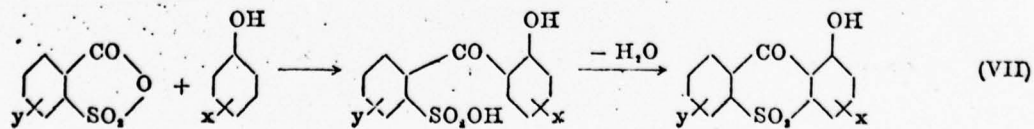
of a reaction, not yet experimentally supported.

In order to speed up the condensation, it is necessary to bond the reaction water in some suitable manner. The most important, almost universal method is to directly use the next mol of o-sulphobenzoic anhydride in accordance with the equation (VI) .



This method however has ^{the} disadvantage that the one half of expensive o-sulphobenzoic anhydride is wasted. Use of condensing agents, such as ZnCl_2 , AlCl_3 is not recommended since that only contaminates the sulphonaphthaleine with an undesirable and almost unremovable kation. For the preparation of chelatometric indicators this method is especially not suitable.

During the research of condensations of o-sulphobenzoic anhydride with phenols we have found that for some sulphonaphthaleins it is convenient to use the azeotropic method of condensation, f.e. in the presence of xylene.¹¹⁾



At the same time, considerable yields are being obtained (theoretical-

ly up to 70%, based on added anhydride), since water separates and reaction basically follows the equation (II). Beside this, the azeotropic method of sulphonephthaleine preparation has still another advantage, namely that the resulting indicator separates in crystalline form. It is usually dispersed in the auxiliary liquid medium and it is only necessary to suck it off, wash it, etc. In this way it is possible to prepare phenolsulphonephthaleine, o-cresolsulphonephthaleine and pyrogallolsulphonephthaleine. For some sulphonephthaleines, as f.e. for thymolsulphonephthaleine this azeotropic method of preparation is not suitable since the reaction results in predominantly useless resinuous products.

It can be added that, almost never, the mentioned reactions of o-sulphobenzoic anhydride with phenols are of quantitative character since several secondary reactions are taking place simultaneously. Thus used phenol can be etherified into a corresponding phenol-ether or can be esterified into sulphobenzoic ester; likely is also analogous synthesis of oxyanthraquinones, see (VII).

Another possible result is undesirable condensation into ortho-form towards both hydrogroups. And finally, complex resinous products may result.

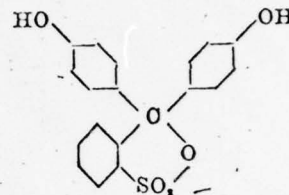
Equipment for preparation of sulphonephthaleine dyes is relatively simple. The mentioned condensations take place in an enamelled enclosed vessel provided with reverse cooler, or with the extension for azeotropic condensation while using a powerful mixer, with the

best results in combination with baffles, since the condensation melt is usually very thick. It is necessary to maintain the empirically found optimal temperature (100-140°C) with the accuracy of one degree, otherwise will result lesser yields and lesser quality. The separation of prepared indicator is determined by its character; in the case of azeotropic condensation the reactive compound has to be merely sucked off, the dye cake washed and distilled with water vapour. The thick melt has to be then adequately diluted and the dye triturated with suitable solvent. The water-soluble sulphonephthaleins (f.e. the chlorphenol and bromphenol red) have to be separated by suitable solvents from the diluted melt that is treated to predetermined pH.

Relatively uncomplicated is the preparation of halogenated sulphonephthaleins by direct halogenation. Halogen is known to have a relatively strong auxochromous effect on sulphonephthaleins and that is why these halogenderivates are widely used as indicators. If a direct halogenation is possible, the resulting sulphonephthaleine then dissolves or disperses in a suitable liquid medium, whereupon it is directly effected by a respective halogen. As the best liquid medium proved to be glacial acetic acid; with other liquids (such as water, chloroform, carbontetrachloride) less satisfactory results were obtained. It is interesting that in the case of the above mentioned direct halogenation a certain additive compound is temporarily formed that decomposes into a desired halogen sulphonephthaleine and respective hydrogen halide only(only) when the temperature is raised.

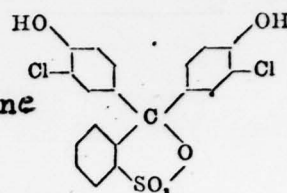
Following is a brief description of the most important sulphone-phthaleine indicators. Their respective melting points that are usually high and not very distinct are not stated here since we have not finished investigation^{on} whether they could be taken as characteristic constants.

Phenolsulphonephthaleine
(phenol red)



is the simplest and the oldest sulphonephthaleine indicator, discovered by Sohon¹²⁾. It forms short red needles with slight metallic lustre of lighter or darker shade, according to their size. In water they dissolve with great difficulty, alcohol is better for this purpose. They do not dissolve in non-polar solvents. Their solutions in alkali are of intensive red color. It is easily halogenated into tetrahalogen derivatives. Color transition is between pH 6,8 (yellow) and pH 8,4 (red). Phenol red is prepared practically by direct condensation of o-sulphobenzoic anhydride with phenol; with¹³⁾ or without addition of condensing agents¹⁴⁾. Yields at the same time do not exceed 50%, based on anhydride added into the reaction vessels. On the other hand we have observed¹¹⁾ that for the preparation of phenol red is very suitable the azeotropic condensation. Yields reach theoretically 70%, based on added anhydride and at the same time the indicator results into easily workable suspension in xylene. Phenol red represents a very general neutralising and pH indicator and it also serves as a basic raw material for other indicators, such as bromphenol blue and tetrachlorophenolsulphonephthaleine.

O-chlorphenolsulphonephthaleine
(chlorphenol red)

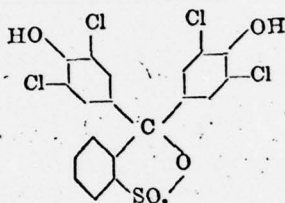


This desirable indicator that could be prepared only with great difficulty was first described by Cohen ¹⁶⁾. Crystallization of this substance is extraordinarily difficult (still best from glacial acetic acid) and it is not easy to eliminate resinous impurities. Due to this the products are of varying composition and (of different) quality. In the past it used to be the most expensive sulphonephthaleine indicator - 1kg was worth over 30.000 Kčs. By suitable modification of conditions in the case of condensation of o-sulphobenzoic anhydride with pure o-chlorphenol and by isolation of the resulting preparation we have succeeded in considerably higher yield (theoretically up to 70%) and in constant quality product.¹⁶⁾ Since the chlorphenol red is easily soluble we can separate it from the diluted melt modified on pH 4 by means of n-butanol. Our o-chlorphenol red is ^asodium salt in the form of pseudocrystalline, a fiery orange powder very easily soluble in water and alcohol and insoluble in non-polar solvents. Its aqueous solutions are distinguished by the brilliancy and intensity of their color transitions: pH 4,8(yellow) up to pH 6,75 (red-purple)¹⁷⁾. O-chlorphenol red is used predominantly for the preparation of lactophenol indicator test papers for quality testing of milk and other food products and also for developping of paper chromatograms ^{for} in identification of organic acids. O-bromphenolsulphonephthaleins (bromphenol red) character resembles the character of the former indicator. It is prepared in a quite similar way using o-bromphenol. It is an orange powder, easily watersoluble with color changes

of pH 5,2 (yellow) up to pH 6,8 (red-purple).

Tetrachlorphenolsulphonephthaleine¹⁸⁾.

This indicator has been little described until now. We have obtained it easily and in satisfactory quantity by direct chlorination of phenolsulphonephthaleine suspension in glacial acetic acid at the laboratory temperature; chlorination in water



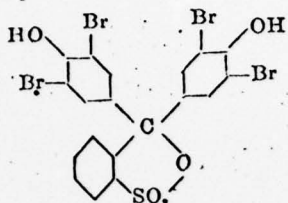
or chloroform does not bring adequate results. Following the action of theoretical amount of chlorine, orange-yellow lustrous needles will crystallize out with one molecule of acetic acid. From this preparation acetic acid is eliminated only by drying in a vacuum, while it changes into yellow powder. Tetrachlorphenolsulphonephthaleine is slightly soluble in water, more soluble in alcohol; its color transitions are between pH 3,0 (yellow) and pH 4,6 (blue-purple). In our opinion this is a neutralizing and pH-indicator of a good applicability. We expect that the tetrachlorphenolsulphonephthaleine production will be initiated in a foreseeable time.

Tetrabromphenolsulphonephthaleine

(bromphenol blue)

forms slightly yellowish or pinkish needles of a low solubility

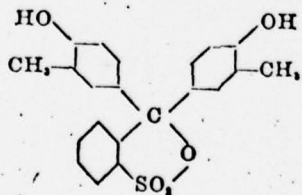
in water, better in alcohol. It is easily prepared by direct bromination of phenolsulphonephthaleine in glacial acetic acid,



when an additive bromhydrate is formed temporarily. Color changes are between pH 3,0 (yellow) and pH 3,6 (blue). Bromphenol blue is a common^{neutralizing} and pH indicator that is lately often used as a developer in paper chromatography of mixture of organic acids.

O-cresolsulphonephthalein (o-cresol red)

forms short red-brown needles difficult to dissolve in water, easier in alcohol ¹⁴⁾. It has a slight metallic lustre and it is of darker coloring than phenol red. It is easily prepared by azeotropic condensation of o-cresol with o-sulphobenzoic anhydride in xylene medium at theoretical yield of 65% (based on anhydride)¹¹⁾.

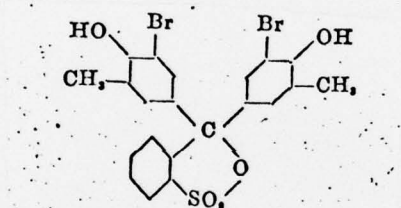


When directly melting both components, the yields are lower and the hardened melt is difficult to remove from the reactor. O-cresol red is a very common neutralizing and pH-indicator, whose upper

color transition is between 7,2 pH (yellow) and pH 8,8 (carmine). It also serves^{as} a basic raw material for the bromcresol purple preparation.

Dibrom-o-cresolsulphonephthaleine
(bromcresol purpur)

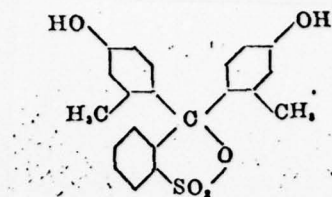
is easily prepared by direct bromination of o-cresol red in glacial acetic acid medium, during which an additive bromhydrate is temporarily formed. Bromcresol purpur forms pinkish needles



relatively well soluble in water (excessive washing in water in the course of its preparation considerably lowers its yields), and still better soluble in alcohol. Color changes are between pH 5,2 (yellow) and pH 6,8 (purpur). It is a common neutralizing and pH-indicator.

M-cresolsulphonephthaleine
(m-cresol purpur)

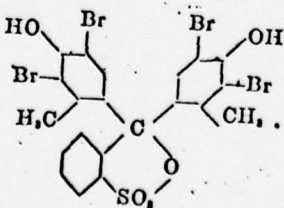
can always be obtained only in low yields by direct melting of o-sulphobenzoic anhydride with m-cresol¹⁹⁾. For its isolation it is recommended to carefully macerate the melt with water and then to filter the separated indicator²⁰⁾.



In this case condensation is predominantly into ortho-form towards both hydroxygroups, so that not even the azeotropic method can be applied here well. It could be said that any substantial increase in yields of m-cresol purpur still remains to be an open problem. M-cresol forms bronze prisms, little soluble in water, better in alcohol. Its color changes are on one hand between pH 1,2 (red) and pH 2,8 (yellow) and on the other between pH 7,4 (yellow) and 9,0 (purpur). M-cresol purpur is a neutralizing and pH-indicator, ^{cf} mainly however a basic raw material for preparation ~~for~~ bromcresol green.

Tetrabrom-m-cresolsulphonephthaleine
(bromcresol green)

could be prepared in satisfactory yield by direct bromination of m-cresol purpur in glacial acetic acid medium ^{19),20)}, during which again, an additive bromhydrate is temporarily formed.



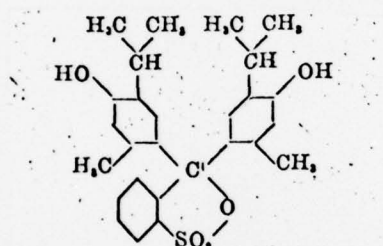
Bromcresol green is in a form of light yellowish needles, not very soluble in water, better in alcohol. Color transi-

tion is between pH 3,8 (yellow) and pH 5,4 (blue). It is a common neutralising and pH-indicator, used mainly in organic analysis.

Thymolsulphonephthaleine

(thymol blue)

is obtained by direct thymol melting of dichloride or anhydride of o-sulphobenzoic acid with thymol²¹⁾. This indicator is rather sensitive to different chemical and physical influences.



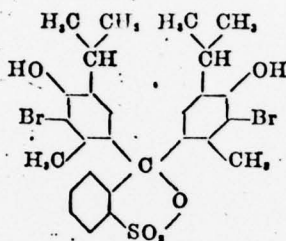
In order to reach satisfactory yields, it is necessary to exactly maintain the empirically determined optimal conditions.²²⁾

Azeotropic condensation cannot be here well applied since the indicator during this method of preparation transforms into resinous form. Thymol blue forms beautiful green short needles of metallic luster, difficult to dissolve in water, better in alcohol. The lower color transition lies between pH 1,2 (red) and 2,8) (yellow), the upper between pH 8,0 (yellow) and 9,6 (blue). Thymolsulphonephthaleine is a popular neutralising and pH-indicator, since a transition from yellow to blue can be noticed very easily. Beside that it is a basic raw material for the preparation of bromthymol blue. Of a similar character is also isometric carvacrolsulphonephthaleine.

Dibromthymolsulphonephthaleine

(bromthymol blue)

is prepared easily and with satisfactory yield by direct bromination of thymolsulphonephthaleine in glacial acetic acid²³⁾, during which an unstable additive bromhydrate is temporarily formed.

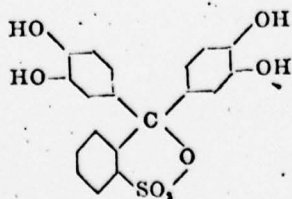


Bromthymol blue is in form of slightly purplish needles of low water solubility. In alcohol its solubility is better. Color changes occur between pH 6,0 (yellow) and pH 7,6 (blue). Bromthymol blue is a widely used neutralising and pH-indicator, since the easily noticable transition is at pH 7. It is also used as an indicator of water purity in aquariums.

Pyrocatecholsulphonephthaleine

(pyrocatechol violet)

was first mentioned by Moir²⁴⁾ and Wood²⁵⁾. We have succeeded for the first time in preparation of this compound in pure crystalline form



We have observed that in aqueous solutions with different pH it forms with some kations characteristically colored complexes⁸⁾. The Research Fellows of Charles University to whom we have passed the prepared pyrocatecholsulphonephthaleines for further study have found that this sulphoph^{neph}thaleine has a character of a typically chelatometric indicator and that it represents a long-sought-for complexometric indicator for acid medium. Based on this, the elaborated methods for determining of vismut, thorium and for establishing factors of normal solutions of complexons^{26),27)} will remain classical. It was further observed that with the help of pyrocatechinsulphonephthaleine it is also possible to complexometrically determine Mg, Ca, Zn, Ni, Co, Mn, Cn....²⁸⁾. Further research is under way to determine some anions. It can be said that, for the time being, pyrocatechol violet is the most universal complexometric indicator. At the same time it is a neutralising indicator, whose lower transition is at about pH 1 (light red-yellow), the upper between pH 6,5 (yellow) and pH 8,5 (violet). Its purple colored alkaline solutions become brown after awhile. Pyrocatechol violet is best prepared by careful condensation melting of a o-sulphobenzoic anhydride with pyrocatechol while stirring and by separation of a resulting reaction melt with glacial acetic acid²⁹⁾. It is necessary to exactly maintain the empirically found optimal conditions, since pyrocatechol violet is sensitive to different physical and chemical effects when it easily transforms into the so called pyrocatechol green (see later). Pyrocatecholsulphonephthaleine forms bronze prisms of a good solubility in water and

in aqueous alcohol but of a difficult solubility in cold acetic acid and in absolute alcohol. They are non-soluble in non-polar solvents. From the solution it crystallizes with crystallically bound solvent, that it maintains persistently. As a result of a prolonged warming of preparation containing acetic acid to the temperature of 115°C, best azeotropically in xylene, the so called pyrocatechol green is obtained by a simultaneous separation of water.

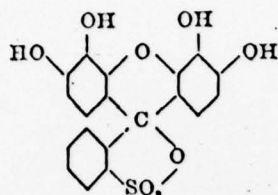
Pyrocatechol green⁸⁾

is a hygroscopic, microcrystalline black powder of a slightly purple metallic luster that is very well soluble in water, alcohol and in glacial acetic acid; it is however non-soluble in non-polar solvents. From solutions it crystallizes only with extraordinary difficulty. Its slightly acidic or neutral aqueous solutions are mostly of brown-yellow coloring, ^{the} alkaline solutions are intensively green. It does not form color complexes with metals. Application of pyrocatechol green as a neutralizing indicator is still in the experimental stage. Constitution of pyrocatechol green is not yet exactly solved; we assume however, that monoacetyl derivative of pyrocatechol green is involved here. We have further prepared some other pyrocatechol greens, whose composition is not known to us presently.

Pyrogallolsulphonephthaleine

was first obtained by Orndorf and Fuchs³⁰⁾. We have succeeded in

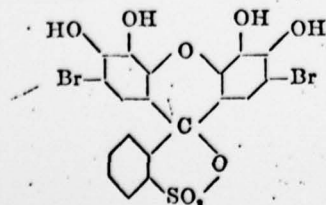
simplifying its preparation and simultaneously in substantial ¹¹⁾ raise ^{of} in its yields by means of azeotropic condensation .



Pyrogallolsulphonephthaleine crystallizes with one molecule of water in the form of reddish brown prisms of light metallic luster. Water-free preparation obtained by drying at 200°C or azeotropically in xylene is a brown powder. Pyrogallolsulphonephthaleine is of low solubility in common solvents. In diluted aqueous solutions at different pH it forms with numerous kations characteristically colored (purple to red) complexes and it will be thus possible ³¹⁾ to use it as a suitable chelatometric indicator . It can be used also as a neutralizing indicator since its alkalic solutions are of violet-red color, while the neutral or the slightly acidic ones are of yellow-orange color. Detailed research of the above possibilities of pyrogallolsulphonephthaleine application is far from being concluded.

Dibromopyroga llolsulphonephthaleine

is a product of a direct bromination of pyrogallolsulphonephthaleine in ethanol medium. It forms deep green metallicly lustrous prisms of low solubility in water, better in alcohol.



sulphonephthaleine

We have found that dibrompyrogallol_Λ can be used also as a neutralizing indicator (with some kations it forms characteristically blue to red colored complexes). The diluted alkalic solutions of dibrompyrogallolsulphonephthaleine are purple and the neutral or the slightly acidic ones are orange. Detailed research of character of this sulphonephthaleine indicator is not concluded and it is expected to bring many new points of interest ³¹⁾. So, f.e. uranium, when combined with this indicator in slightly acidic medium, forms an intensively purple complex that is titratable by a complexon into a well noticable color transition. Resorcin-sulphonephthaleine and phloroglucinsulphonephthaleine are of only a theorethical interest since their color changes (f.e. yellow-orange) are less outstanding. Research of other sulphonephthaleins_Λ such as oxyhydrochinosulphonephthaleines, xylenosulphophthaleines and naftosulphonephthaleines is still under way.

Evaluation of sulphonephthaleine indicators

Already simple tests, such as determination of ashes, of moisture and of insoluble residue in alcohol present ^{some idea of} a value of defined marketed sulphonephthaleine indicators. From the sulphur content, determined f.e. by well known explosion method with Na_2O_2 we can determine the percentual content of sulphonephthaleine. Content of halogen, metoxyle and so on, determined by some known method can serve as a relatively exact criterion for evaluation of sulphonephthaleine.

The most authoritative criterion of quality and purity of

sulphonophthaleine indicators is a visual, spectroscopic and colorimetric definition of their color transition and coloring intensity at various pH, thus in buffer solutions, in comparison with the chosen type. For the types we use especially the pure, several times crystallized sulphonophthaleins whose elementary analysis is in the agreement with the theory. Such preparations can be considered as chemical individuals. A satisfactory sulphonophthaleine indicator should not have greater deviations in its color transition than the hundredth order of pH, similarly intensity of color and its solutions should not noticeably differ from the defined type.

In the conclusion it remains to draw attention of those interested in sulphonophthaleine indicators, until now very difficult to obtain, to the fact that, due to the domestic production they begin to appear in sufficient quantity and in excellent purity at low prices on domestic market. This gives opportunity to the chemical workers in all branches of industry to apply many modern exact analytical methods.

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Research of sulphonophthalein^e indicators was conducted by Zdr. Vodák and Dr. Ing. Otakar Leminger in Corporation for Chemical and Metallurgical Processing, Ústí nad Labem. Of great help was also Dr. Jaroslav Schneider. During first experimental production were present comrades F. Prokop, F. Šindler, V. Dobrý. For start-up of production of sulphonophthaleines on larger scale according to ^{the} new methods should be credited Ing. Bedřich Lebl from MCHP. This production is in the process in "Lachema"

in Brno under the leadership of a collective of Comrades headed by Dr. Vízďala, Dr. Pláteník and Zářybnický.

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