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HISTORY OF ANALYTICAL CHEMISTRY

Ninety Years of Using Azo Compounds of the Pyridine Series as Analytical Reagents

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Abstract—The historical aspects of the synthesis, study, and application of heterocyclic azo compounds of the pyridine series in analytical chemistry are considered. The number of reagents and methods used for detection, separation, and preconcentration; the variety of samples; and the analytical ranges are increasing progressively.

The Griess reaction is rightly considered to be the first example of the targeted synthesis of an organic reagent for the analytical chemistry purposes (the detection of nitrite ions) [1, 2]. In the next 125 years, the number of synthesized azo compounds came to total several hundred. Diazotization is used not only to detect nitrite ions, but also to prepare analytical reagents for many ions, and in technology (dye industry) as well. Only six years later, Ilinski and Knorre [3] proposed 1-nitroso-2-naphthol for the detection of cobalt (later known as Ilinski reagent), and 20 years later, Tschugaev proposed dimethylglyoxime as a new analytical reagent for nickel [4]. A feature of the above reagents is their increasing value rather than their considerable age. In the following years, many analogs were synthesized, which made it possible to find out the regularities of the influence of the reagent structure on its reactivity and the analytical characteristics of the resulting complexes. The original parent reagents came to be used in various methods of analytical chemistry for determinations in wide concentration ranges.

Heterocyclic azo compounds belong to aromatic azo compounds, but they make up an individual group of organic reagents both by the peculiarities of their synthesis and by their reactivity and analytical characteristics. The credit for synthesizing of heterocyclic azo compounds belongs to A.E. Chichibabin with co-workers. They obtained 2-aminopyridine and 2-aminoquinoline and comprehensively studied diazotization of 2-aminopyridine and its azo coupling, leading to the synthesis of heterocyclic azo compounds.

Marckwald was the first who obtained 2-aminopyridine and studied its diazotization [5]. However, attempts at coupling the diazotization product with resorcinol and naphthol gave no satisfactory results, because the "diazotization" product was 2-chloropyridine in all cases. Substituting amyl nitrite for nitrous acid, which was successfully applied for the diazotization of aromatic amines—aniline, *p*-bromoaniline, and *o*-toluidine—also gave no satisfactory results [6]. Only 15 years later, Chichibabin and Ryazantsev [7] reported diazotization and studied the diazo reaction of 2-aminopyridine. Under the action of isoamyl nitrite in dehydrated ethanol in the presence of sodium alcoholate (or, better, sodium amide), the authors obtained and isolated a very stable solid diazotate and further coupled it with 2-naphthol, resorcinol, and 2,6-diaminopyridine. Later, the syntheses of 1-(2-pyridylazo)-2-naphthol (pyridylazonaphthol-2), 4-(2-pyridylazo)resorcinol (pyridylazoresorcinol), and 5-(2-pyridylazo)-2,6-diaminopyridine were described in detail [8]. The author [8] noted that azo coupling does not proceed in a closed airtight compartment, but by gradually passing small bubbles of carbon dioxide through the reaction mixture drastically accelerates the reaction. The resulting sodium carbonate precipitates and does not contaminate heterocyclic azo compounds in solution. The attempt at coupling pyridyldiazotate with nitrosonaphthols or *p*-nitrosodimethylaniline in glacial CH_3COOH gave negative results.

The data concerning synthesis of 1-(2-pyridylazo)-2-naphthol and 4-(2-pyridylazo)resorcinol and their application in analytical chemistry are separated by several decades [9]. Possible application of 1-(2-pyridylazo)-2-naphthol in analytical chemistry (qualitative reactions) was first mentioned in 1951 [10], its application as a chelatometric indicator was first mentioned in 1955 [11], and the application of 4-(2-pyridylazo)resorcinol for analysis purposes was first mentioned in 1957 [12]. Even brief enumeration of the fields of application of heterocyclic azo compounds in present-day analytical chemistry indicates the great variety of these fields, and each of them deserves discussion. In this paper, I present mainly the first publications on each method to show the priority of the authors and their schools of thought. The data concerning the number of publications and the number of heterocyclic azo compounds are given for statistical purposes only; they vary constantly.

Reagents. At present, the number of heterocyclic azo compounds used in analytical chemistry has

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increased substantially. It totals several hundred compounds, and some of them are of special interest as analytical reagents. In addition to 2-aminopyridine, 2-amine derivatives of pyrazole, imidazole, antipyrine, triazole, thiazole, quinoline, and other heterocycles (more than 15) are used. The number of azo components is even more (more than 30): phenols, diphenols, alkylphenols, aniline, and diaminobenzenes. However, only 2-aminopyridine and 2-aminoquinoline are diazotized under special conditions. All other amines, including 2-aminothiazole, can be diazotized by sodium nitrite in acid. At present, the compounds containing a heteroatom only in diazo component [1-(2-pyridylazo)-2-naphthol, 4-(2-pyridylazo)resorcinol, etc.] or in azo and diazo components [7-(2-pyridylazo)-8-hydroxyquinoline, rhodanine-(5-azo-2)thiazole, etc.] are referred to as heterocyclic azo compounds. Finally, a hetarylazo group can be considered analytically active in the azo compounds containing an aromatic ring with functional groups for certain ions (7-(arylazo)-8-hydroxyquinoline [13], thiazolylazopyrocatechol [14], etc.) as an azo component. There are known azo compounds that contain heteroatoms both in the diazo and azo components, which form a common analytical functional group [7-(2-thiazolylazo)-8-hydroxyquinoline, 5-(2-thiazolylazo)-2,6-diaminopyridine, 5-(2-thiazolylazo)-2,6-dihydroxypyridine, etc.]. The heterocyclic azo compounds based on 2-amines are of greatest interest, because this heteroatom takes part in the complex formation and substantially affects both the reagent and complex properties.

Phenols are commonly used as azo components; in this case, the hydroxy group in the *ortho* position to the azo group enters the analytical functional group.

Azo derivatives of 3-amino- and 4-aminopyridine also can be referred to as heterocyclic azo compounds, but they have no analytical functional groups of heterocyclic azo compounds and are therefore of no interest as analytical reagents. 2-(2-Pyridylazo)-1-naphthol (pyridylazonaphthol-1) isomeric to 1-(2-pyridylazo)-2-naphthol is referred to as a heterocyclic azo compound, but it is used only for theoretical purposes in analytical chemistry. 4-(2-Pyridylazo)-4-naphthol (pyridylazonaphthol-4), a structural analog of pyridylazonaphthol-1, is of no interest to analytical chemistry; neither are as azo derivatives of 3-amino- and 4-aminopyridine.

Finally, azo derivatives of heterocycles with two and more similar nitrogen atoms in a five-membered ring (pyrazole, imidazole, antipyrine, triazole, and tetrazole) or in a six-membered ring (pyrimidine and indazole) as well as with different heteroatoms, such as nitrogen and sulfur (thiazole and benzothiazole), can be referred to as heterocyclic azo compounds. Because the nitrogen atom of the heterocycle, being more electronegative than the sulfur atom, enters the analytical functional group, all heterocyclic azo compounds are traditionally classed into nitrogen-containing (pyridine, anabasine, and quinoline series) and sulfur-containing ones, in spite of the fact that the latter also contain a nitrogen atom in the heterocycle. With the electronegativity of the nitrogen atom and its coordinative ability, the sulfur atom in sulfur-containing heterocyclic azo compounds strongly affects the acid properties of the hydroxy group, the protonation constant of the nitrogen atom in the heterocycle and, as a consequence, the stability constants of the complexes. Additionally, as mentioned above, the synthesis of sulfur-containing heterocyclic azo compounds is much simpler and is similar to the diazotization of aromatic amines.

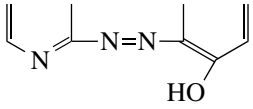
Literature. According to the data of monograph [9] and review [15], the number of references varied over time as follows (a cumulative series is given):

Year	1964	1967	1968	1979	1985	1990	2000
Number of references	156	323	443	>1400	2050	2750	>3000

The sharp increase in the number of publications after 1968 can be attributed to the synthesis and wide application of anabasine, antipyrine, and thiazole series azo compounds [16] as well as the variety of analytical methods used for the study of complex formation and practical application of heterocyclic azo compounds.

The reasons for the high reactivity of heterocyclic azo compounds. Heterocyclic azo compounds interact with all elements existing in solution as cations. Alkali metals are the exception. It is reported that 1-(2-pyridylazo)-2-naphthol-2 interacts with alkaline-earth elements to form extractable complexes. Platinum group elements make up a special group. They exist in solution as anionic complexes (in the presence of halogen acid anions) but interact with heterocyclic azo compounds in the presence of carboxylates accelerating complex formation when heated. Molybdenum(VI) forms mixed-ligand complexes in the presence of hydroxylamine; in this case, molybdenyl ions take part in the complex formation [17]. Tungsten(VI) complexes are not formed, although the conditions are known of the formation of tungstenyl cations. At the same time, vanadium(V), niobium(V) and tantalum(V) existing as anions (especially in the presence of tartaric, citric, oxalic, and other organic acids) interact with heterocyclic azo compounds and are widely used in the analytical chemistry of these elements [18]. Permanganate and dichromate oxidize heterocyclic azo compounds to colorless compounds, and strong reducing agents, such as vanadium(II), chromium(II), and titanium(III), reduce the reagents to two amines. These reactions are also used in analytical chemistry.

Heterocyclic azo compounds contain the analytical functional group



Complexes are formed through the coordination bonds with the donor nitrogen atom of the heterocycle and the other (more electronegative) nitrogen atom of the azo group and through substituting the proton of the hydroxy group in the *ortho* position to the azo group. In this case, the bond with the negatively charged oxygen is formed, and this charge can be changed by introducing various substituents to the azo component. Two five-membered rings are formed in complex formation. Virtually no stepwise complex formation occurs, because even elements with the coordination number 6 mainly form 1 : 1 complexes. This, combined with the high electronegativity of the donor atoms, high $pK_{o\text{-}OH}$, and three binding sites in the analytical functional group make for the high stability constants. This accounts for the wide application of these reagents in analytical chemistry.

Heterocyclic azo compounds have the following advantages: a high sensitivity with respect to many ions; a substantial bathochromic shift of the absorption maximum due to complex formation; a low reagent excess necessary to complete complex formation; the linearity of calibration plots in wide concentration ranges for many methods; the lack of the effect of buffer solution components on the complex absorptivity; the stability of the reagent and complex absorbances over time; a wide acidity range where the absorbance is a maximum and remains virtually constant; the low absorbance of the blank solution; the lack of the association of the reagent and complex molecules and ions in solutions; and the possibility of extraction, adsorption, and ion-exchange preconcentration.

Use of heterocyclic azo compounds. Methods for studying the reagents and complex formation are very diversified. The kinetics and thermodynamics of complex formation are discussed in detail in the monograph [9] and the review [15]. In recent years, the number of determination and, especially, preconcentration and separation methods has increased. The most frequently used methods are given below, and the peculiarities of some of them are discussed in regard to heterocyclic azo compounds.

Methods of detection: spot tests and test methods. The differences in the colors and hues of the complexes in the pH of their formation and existence, and in their behavior with respect to masking agents as well as the good water solubility of 4-(2-pyridylazo)resorcinol and its complexes, are used for the detection of various ions [9]. These features also form a basis for test methods [19]. The immobilization of heterocyclic azo compounds, mainly, 1-(2-pyridylazo)-2-naphthol, on various supports resulted in the creation of test forms and, because of preconcentration that proceeds in the course of test determinations, in a substantial increase in sensitivity.

Titrimetric methods: complexometric, precipitation, and acid–base titration. The wide use of chelatometry in the late 1950s did not go beyond metallochromic indicators. Eriochrome Black T or murexide are suitable for the determination of a limited number of elements (mostly, alkaline-earth metals, copper, and zinc). Low color contrast in the titration end-point required the high skill of the operator, and the instability of solutions of these indicators required the use of solid mixtures with NaCl. Xylenol orange and pyrocatechol violet, which came into use for the determination of many ions, extended the number of analyte elements. However, they were insufficiently stable in solutions and depended on the solution ionic strength. The use of 1-(2-pyridylazo)-2-naphthol [11] and, later, 4-(2-pyridylazo)resorcinol [12] eliminated these defects. All versions of chelatometry (direct titration, back-titration, indirect titration, and displacement titration) are used for determinations. In this connection, the determination of cations for which no metallochromic indicators exist as well as the determination of anions became possible.

The example of a selective titration in a most acidic medium is the determination of bismuth at pH 1 using 1-(2-pyridylazo)-2-naphthol [20]. To determine low amounts of erbium (0.33–0.84 mg), 4-(2-pyridylazo)resorcinol was used as a metallochromic indicator, and the titration end-point was detected by photometry [21].

Spectroscopic methods: photometry and extraction–photometry, solid-phase spectrophotometry, kinetic methods, flow-injection and continuous flow analyses with photometric detection, colorimetry, fluorimetric methods, atomic absorption, X-ray emission analysis, and thermal lens spectroscopy. 4-(2-Pyridylazo)resorcinol was the first water-soluble azo compound whose sensitivity with respect to uranium ($\varepsilon = 3.87 \times 10^4$ [9, p. 48]) was higher than that of arsenazo I ($\varepsilon = \sim 2 \times 10^4$) and the most sensitive among the known reagents for cobalt ($\varepsilon = 5.5 \times 10^4$ [22]).

In extraction photometry [23], substituting 1-(2-pyridylazo)-2-naphthol solution in the corresponding solvent for an organic reagent in the extract is of interest. The use of organic extracting reagents (trioctylamine, trioctylphosphine oxide, diaryl-, and dialkyldithiophosphoric acids) to extract easily hydrolyzable elements from strongly acidic media eliminates side reactions of hydrolysis and polymerization. The addition of heterocyclic azo compounds to the organic phase and complex formation in it make efficient photometric determination possible.

Solid-phase spectrophotometry [24] increased the sensitivity of determination by several orders of magnitude because of high preconcentration factors. Organ-

opolymer cation- and anion-exchangers are used as supports.

Sensitivity in photometry is increased by adding surfactants and using the most sensitive surfactant known, 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol. In this case, molar absorptivities reach $(1–4) \times 10^5$ [15].

Colorimetry offers new potentialities [25]. The adsorption version of colorimetry increases the sensitivity by 1.5–2.5 orders of magnitude as compared to spectrophotometry and offers additional information about the reagent and complex characteristics. The bifunctional version of colorimetry is of special interest [26]. This method is based on the difference between the slopes of the calibration plots in the colorimetric function–analyte concentration coordinates.

In addition to common methods of pre-separation and masking, the selectivity of determinations in photometric methods is increased by using derivative spectrophotometry [27] and even more by using the Fierordt method [28] combined with variation of pH [29].

Among the kinetic methods, determination of 5×10^{-9}–2×10^{-7} M silver based on its catalytic effect on the oxidation of antipyrylazo-8-hydroxyquinoline by persulfate is worthy of notice because of its sensitivity [30].

Electrochemical methods: voltammetry [31], amperometry, and ion-selective electrodes. Various versions of voltammetry are used: adsorptive stripping voltammetry, differential pulse adsorptive stripping voltammetry, linear sweep adsorptive stripping voltammetry, voltammetry with a stationary mercury electrode and adsorption preconcentration, and catalytic adsorptive stripping voltammetry [15, 31]. Very high sensitivity ($\geq 10^{-11}$ M) and wide linearity range of the calibration plots are attractive. The method is especially promising for analysis of environmental samples (ultratrace amounts) and biological samples (small portions).

Separation and preconcentration methods: extraction, adsorption, extraction chromatography, thin-layer chromatography, HPLC, detection in chromatographic methods, and capillary electrophoresis. Extraction was used to isolate colored complexes with heterocyclic azo compounds formed in aqueous solutions or to extract metal ions as complexes with 1-(2-pyridylazo)-2-naphthol in an organic solvent immiscible with water [23]. Except for the complexes of 4-(2-pyridylazo)resorcinol, the complexes of other heterocyclic azo compounds of pyridine series are well extracted. In the case of complexes of 4-(2-pyridylazo)resorcinol, the charge of the *p*-hydroxy group in the azo component is compensated by large organic cations, such as diphenylguanidinium. To determine elements in the extract, atomic absorption can be used in addition to photometry [32].

Heterocyclic azo compounds are used in adsorption preconcentration to prepare modified adsorbents, to prepare metal complexes with heterocyclic azo compounds for further preconcentration on adsorbents unmodified with heterocyclic azo compounds, and to adsorb heterocyclic azo compounds by adsorbents with further complex formation on the adsorbent. The elements are determined either in the adsorbate phase by nondestructive methods (diffuse reflection spectroscopy or colorimetry) or after elution. 1-(2-Pyridylazo)-2-naphthol is commonly used as a modifier; and polyacrylonitrile fiber, organopolymer ion-exchangers, naphthalene, silica gels, xerogels, silochromes, and polyurethane foams are commonly used as supports [15].

Extraction column chromatography was applied to the preconcentration of palladium from chloride solutions using fluoroplastic as a support and 1-(2-pyridylazo)-2-naphthol in chloroform or isopentanol as stationary phases [33]. When using EDTA as a masking reagent at 85–98°C, 10^4–10^5-fold amounts of nonferrous metals, Fe(III), Pb, Mn, and Al can be masked [34].

In thin-layer chromatography, 1-(2-pyridylazo)-2-naphthol [35] and 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol were used as reagents [36]. The method was mainly applied to the study of the chelate retention mechanism on silica gel and to the evaluation of the number of bonds with active sites of silica gel.

Among the chromatographic methods, both the normal-phase and reversed-phase versions of HPLC employing mostly 1-(2-pyridylazo)-2-naphthol and 4-(2-pyridylazo)resorcinol as reagents are, probably, most frequently used [37]. In the case of 4-(2-pyridylazo)resorcinol, ion-pair reversed-phase high-performance liquid chromatography is of special interest. This method is promising for the separation of mixtures of ionic compounds, because it allows varying the separation selectivity: the adsorbate retention depends on the nature of the stationary phase; the nature and concentration of the ion-pair reagent, organic solvent, and buffer solution; pH; ionic strength; and temperature of the mobile phase [37, 38].

Other separation and preconcentration methods mentioned above are only coming into use.

Among all the heterocyclic azo compounds, 1-(2-pyridylazo)-2-naphthol, 4-(2-pyridylazo)resorcinol, and 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol are still most widely used. Chemical industry of many countries manufactures these reagents of good purity grade, and their price makes them available for any laboratory.

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