

**DEVELOPMENT OF AN ECO-FRIENDLY SOLID-PHASE
SPECTROPHOTOMETRIC METHOD FOR COBALT DETECTION BASED ON
IMMOBILIZED NITROSONAPHTHOL-BASED REAGENTS**

Inatova Makhsuda

*Doctor of Philosophy Chemistry, Associate Professor
Jizzakh State Pedagogical University Uzbekistan*

Ubaydullayeva Zilola

Independent researcher

MAQOLA MALUMOTI

ANNOTATSIYA:

MAQOLA TARIXI:

Received: 20.12.2025

Revised: 21.12.2025

Accepted: 22.12.2025

Immobilization of reagents was carried out, their kinetics were studied and the optimal conditions for immobilization and complex formation of cobalt ions were determined.

KALIT SO'ZLAR:

*immobilization, nitrosonaphthol
derivatives, heavy and toxic
metals*

The purpose of this work is to develop sorption-spectroscopic methods for determining cobalt that do not require the use of organic solvents, and are therefore safe for the environment. Immobilizing synthesized new organic reagents based on nitrosonaphthol derivatives on fibrous carriers, and reducing their consumption through metrological characteristics, makes it possible to determine cobalt in the analysis of natural objects and industrial materials.

Reducing the detection limit is achieved by preconcentrating the analyte by determining the integrals from the carrier after transferring a large volume of solution into the solid phase. Unlike extraction preconcentration, sorption-spectroscopic methods do not require the separation of the precipitate from the solution by filtration, which makes the analysis faster.

Nitrosonaphthol interacts with many metals, forming complexes, but among them cobalt is one of the most selective reagents. However, in order to improve selectivity, new reagents

were synthesized based on nitrosonaphthol derivatives: 2-hydroxy-3-nitroso-1-naphthaldehyde, 4-bromo-2-nitroso-1-naphthol, 4-hydroxy-3-nitroso naphthalene-1-sulfoxylic acid is introduced into the reagent molecule by bromine atoms, sulfo groups, and others. For example, when transitioning from nitrosonaphthol to halogen-substituted derivatives (such as 4-bromo-2-nitroso-1-naphthol) the new reagents become more specific and their selectivity improves, since they acquire the ability to interact in more acidic media and with fewer types of ions.

Similarly, introducing a sulfo group into the nitrosonaphthol molecule promotes increased solubility of the reagent in water. For example, the reagent 4-hydroxy 3-nitroso-2-naphthaldehyde-1-sulfonate synthesized in this way forms an intensely colored, water-soluble complex with cobalt (II) ions. Some transition metal cations form insoluble compounds with the reagent, preventing complex formation and thus not undergoing sulfonation.

The increase in selectivity during reagent modification is associated with changes in the steric factors of the nitrosonaphthol molecule. At pH 4–5, nitrosonaphthol reacts with many metals, including cobalt, nickel, iron, copper, and zinc, whereas the 2-hydroxy-3-nitroso-1-naphthaldehyde we synthesized does not interact with copper and zinc. At the same time, the new reagent 2-nitroso-4-isopropyl-1-naphthol-1 interacts with copper and zinc. The isopropyl group creates spatial hindrances for the placement of ions with large radii in the chelate complex. However, for metals that show stronger affinity to nitrogen, it reacts with 2-nitroso-4-isopropyl-1-naphthol-1 [4]. The assumption that steric hindrance is not solely caused by introducing an isopropyl group is confirmed by studies of the properties of 2-nitroso-4-isopropyl-1-naphthol-1. This reagent interacts with metals similarly to unsubstituted nitrosonaphthol, since the isopropyl group in this case is located fairly far from the heterocyclic nitrogen.

The improvement in selectivity is due to the fact that, during immobilization, organic reagents due to the geometric features of ligand fixation on the carrier surface can, in some cases, change their complexing properties, such as activity. It can be assumed that modified sorbents most effectively extract those metal ions that, when bound with the immobilized reagent, form ionic associates or complexes with a metal-to-ligand ratio of 1:1. In this case, steric hindrance is minimized, facilitating the process caused by the fixation of the ligand on the sorbent surface.

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Metal complexes with immobilized 2-hydroxy-3-nitroso-1-naphthaldehyde and 2-nitroso-4-isopropyl-1-naphthol differ from their reactions in solution in that they are more stable.

Selection of the optimal sorption value of cobalt (II) ions depending on pH. Since the ligands are weak anions, the complexing ability of nitrosonaphthol strongly depends on the pH of the medium. This is especially important for the selective precipitation of cobalt, because nitrosonaphthol forms complexes with cobalt that have lower stability constants than those of other metals. To achieve complete precipitation of cobalt from solution, its pH must be carefully regulated. Moreover, the degree of selectivity obtained by regular pH adjustment can be further increased by using appropriate masking reagents. At low pH values (in acidic media), cobalt (II) ions are in a hydrated form. This ensures a high sorption rate, but the degree of extraction on unmodified carriers is low. On modified carriers, the process of cobalt (II) sorption proceeds as follows: as the pH of the solution increases, cobalt (II) hydroxide begins to form. In this case, the sorption rate increases. Apparently, under these conditions, nucleation is facilitated, for example, in cobalt (II) hydroxide, which leads to the formation of a finer dispersed system. The proposed sorption mechanism becomes more effective when the maximum degree of cobalt ion extraction is achieved in a more alkaline medium on modified carriers. The sorbent obtained by immobilizing 2-nitroso-4-isopropyl-1-naphthol-1 on SMA-1 anionite, after contact with a cobalt solution, acquires a green color. The filtrate is transparent. However, starting from pH 8.5, the filtrate color becomes faintly bluish, and the sorbent's color intensity decreases significantly. The dynamics of cobalt ion sorption on SMA-1 anionite immobilized with 2-nitroso-4-isopropyl-1-naphthol-1 and 2-hydroxy-3-nitroso-1-naphthaldehyde, as well as the sorption rate of cobalt ions, were studied using a limited volume method. For this, a series of test tubes containing 0.1 g of sorbent was prepared, and 10 mL of a model cobalt solution of a given concentration was added. The tubes were stirred for periods ranging from 2 minutes to 1 hour under constant mixing. At certain time intervals, the liquid phase was subjected to analysis by the photometric method, as described earlier. The kinetics of sorption were studied at the selected optimal pH value. The pH of the solutions was adjusted with 0.1 N hydrochloric acid solution, acetic acid, and dilute ammonia solution. The experimental results showed that to achieve maximum cobalt extraction at the optimal pH value, 10–15 minutes is required, regardless of the initial concentration of cobalt ions in the liquid phase. With longer contact time, the degree of extraction remains constant, which

indicates the establishment of sorption equilibrium. The authors express their deep gratitude to Professor M.G. Mukhamediev and Doctor of Chemical Sciences D.A. Gafurova for providing fibrous sorbents, as well as to J. Nurmukhamadov and K. Tokhumkhamedov for synthesizing the reagents.

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