

## Advanced Chromatography 2020: Chromatographic analysis of Chloranilines in aqueous environments

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Analysis of aniline and its chlorine derivatives in aqueous environments is a complex process: Determination of micro-concentrations with low sensitivity (0.01-0.1 mg / dm<sup>3</sup>). It is difficult to separate aniline and chloraniline from water, and the methods of separation of these compounds are not fully universal. Most liquid extracts are used for aniline and its derivatives. Excavators used for liquid extraction must meet the following requirements:

- Must be able to remove the analyzed component or group of substances well.
- Must have minimal solubility in water.
- The density of the excavator should be as different as possible from the density of the analysis product.

The effective solvents for the extraction of aniline are given as follows: saturated k.h <unsaturated k.h <aromatic k.h <ethers <alcohols. The introduction of inorganic solvents (NaCl, Na<sub>2</sub>SO<sub>4</sub>) will significantly increase the distribution coefficients and the efficiency of the extraction concentration of anilines. There are two main approaches to determining the micronutrient content of chloranilins in water by chromatographic methods by Direct determination of chloranilines and determination of chloranilines in the form of derivatives. Chromatography is a laboratory technique for the separation of a mixture. The mixture is dissolved in a fluid called the mobile phase, which carries it through a structure holding another material called the stationary phase. The various constituents of the mixture travel at different speeds, causing them to separate. The separation is based on differential partitioning between the mobile and stationary phases. Subtle differences in a compound's partition coefficient result in differential retention on the stationary phase and thus affect the separation. Chromatography may be preparative or analytical. The purpose of preparative chromatography is to separate the components of a mixture for later use, and is thus a form of purification. Analytical chromatography is done normally with smaller amounts of material and is for establishing the presence or measuring the relative proportions of analytes in a mixture.

With the help of highly efficient capillary columns and methods of modern concentrations of selective detectors (ECD, NPD), chloranilines can usually be determined directly from the required sensitivity level (0.05 µg / dm<sup>3</sup> and 0.5-5 µg / dm<sup>3</sup>).

The reason for this unsatisfactory sensitivity is that the presence of an amine group in chloranilines interferes with the sample and causes erosion and asymmetry of individual chromatographic peaks. The NH<sub>2</sub> group, on the other hand, is highly reactive to modify anilines. Using this, the removal of the

amine group will have an equally positive effect on both the extraction concentration of anilines and their chromatographic determination.

The reaction of obtaining nitrogen derivatives used for the determination of this class of compounds in gas chromatography is as follows. They can be divided into two types: cilia and assimilation reactions. Siliceous organic compounds are also one of the most universal methods for the deactivation of polar functional groups. Silica derivatives of single and double amines are prepared using the following reagents.

MSTFA: N-methyl-N (trimethylcyl) – trifluoroacetamide

BSTFA: N, O - bis (trimethylcyl) – trifluoroacetamide

Trimethylchlorosilane (TMCS) or trimethylsilyl-imidazole (TMSIM) is used as the catalyst. Trimethylsilyl chloride, also known as chlorotrimethylsilane. It is an unstable fluid that is steady without water. It is generally utilized in natural science. 1-(Trimethylsilyl) imidazole (TMSI) was utilized for derivatization of sugars into trimethylsilyl ethers. It was additionally used to integrate polysubstituted chiral spiro tetrahydropyrans and as silylating reagent for the assurance of hydroxyl bunches within the sight of amine functionalities.

Silylating reagent for the protection of hydroxyl groups in the presence of amine functionalities It should be noted that the activity of different classes of organic compounds in cilia reactions varies and decreases as follows: alcohols> phenols> carboxylic acids> single amines> double amines> amides.

The reaction is carried out only between organic solvents, because both reagents and reaction products are easily hydrolyzed even in small amounts of water. It has been found that tetrabutyl dimethylsilyl derivatives (TBDMS) are more resistant to hydrolysis than trimethylcyl derivatives (TMS), and that MTBSTFA, which is reactive for their preparation, can be stored for a long time. Another problem with silage is the replacement of both H atoms with a mixture of mono and di-TMS derivatives. MTBSTFA also has advantages here, as di-TBDMS derivatives are practically not formed due to steric barriers created by mass TBDMS groups.

Oxidation reactions are more efficient than cilification because some derivatives show hydrolytic and thermal resistance to N-silicon derivatives, respectively, and this requires more stringent conditions for gas chromatographic analysis. The acidification reactions are carried out in organic solvents, using pyridine, trimethyl or triethylamine as catalysts and solvents for by-products.

Although acetyl amine products are more resistant to hydrolysis

than silic derivatives, acidification occurs after extraction in the liquid and solid phases. The combination of acidification with solid-phase extraction to increase the rate of removal of amines from water is described using various reagents. Derivatives of anhydrides and acylides containing F and Cl atoms significantly increase the sensitivity of their determination when using ECD or GC in the chemical ionization mode with the combination of ions. In this case, the detection sensitivity of DEZ increases in the order  $F < Cl < Br < I$ , so derivatives of anilines containing mono, di and trichloroacetyl anhydride are more sensitively determined by ECD than derivatives of tri, penta and heptafluorine reagents.

Gas chromatography (GC) is a typical kind of chromatography utilized in diagnostic science for isolating and dissecting intensifies that can be disintegrated without decay. Run of the mill employments of GC incorporate testing the immaculateness of a specific substance, or isolating the various parts of a blend (the overall measures of such segments can likewise be resolved). In certain circumstances, GC may help in distinguishing a compound. In preparative chromatography, GC can be utilized to plan unadulterated mixes from a blend. In any case, polyhalogenated reagents (PFPA, HFBA, TCA-Cl, HFB-Cl, HFB-Cl). Accompanying use with ECD requires the expulsion of overabundance reagents and results (polyhalogenic carbonic acids). Since these mixes meddle with the investigation of gas chromatography.

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