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(ПОЯСНЮВАЛЬНА ЗАПИСКА)**

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**Тема: «Розробка методики визначення 2-метил-4-хлорофеноксіацетатної
кислоти»**

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BACHELOR THESIS
(EXPLANATORY NOTE)
OF GRADUATING STUDENT OF EDUCATIONAL DEGREE BACHELOR
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EDUCATION PROGRAM “PHARMACEUTICAL BIOTECHNOLOGY”

Theme: « Development of methods for determining of 2-methyl-4-chlorophenoxyacetic acid »

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ЗАВДАННЯ

на виконання дипломної роботи

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3. Вихідні дані роботи: Літературні джерела, щодо будови та фізико-хімічних властивостей Розробка методики визначення 2-метил-4-хлорофеноксіацетатної кислоти.. Її токсичності, методів синтезу. Наукова інформація, щодо вилучення та методів аналізу МЦПА.

№	Завдання	Термін виконання	Підпис Керівника
1	Вибір теми дипломної роботи, узгодження змісту з дипломним керівником.	26.04.2021	
2	Складання схеми виконання бакалаврської дипломної роботи.	27.04.2021	
3	Підбір та обробка літератури по характеристиці 2-метил-4-хлорофеноксіацетатної кислоти та її застосуванню як діючої речовини гербіцидних препаратів.	28.04.2021 – 02.05.2021	
4	Підбір та обробка літератури по токсичності 2-метил-4-хлорофеноксіацетатної кислоти та гербіцидних препаратів до яких вона входить.	03.05.2021 – 04.05.2021	
6	Підбір та обробка літератури по методам вилучення 2-метил-4-хлорофеноксіацетатної кислоти.	09.05.2021 – 16.05.2021	
7	Оформлення дипломної роботи.	24.05.2021 – 29.05.2021	
8	Перевірка дипломної роботи керівником.	30.05.2021	

9	Коригування, підготовка доповіді та презентації	31.05.2021 -01.06.2021	
10	Передзахист дипломної роботи.	02.06.2021	
11	Захист дипломної роботи.	06.2021	

4. Зміст пояснювальної записки: ВСТУП; РОЗДІЛ 1 2-МЕТИЛ-4-ХЛОРОФЕНОКСІАЦЕТАТНОЇ КИСЛОТИ ТА ЇЇ ЗАСТОСУВАННЯ ЯК АКТИВНОЇ РЕЧОВИНИ ГЕРБИЦИДІВ; РОЗДІЛ 2. ХАРАКТЕРИСТИКА ТОКСИЧНОСТІ 2-МЕТИЛ-4-ХЛОРОФЕНОКСІАЦЕТАТНОЇ; РОЗДІЛ 3. МЕТОДИ ВИЗНАЧЕННЯ 2-МЕТИЛ-4-ХЛОРОФЕНОКСІАЦЕТАТНОЇ КИСЛОТИ; ВИСНОВКИ; ЛІТЕРАТУРА.

5. Перелік обов'язкового графічного (ілюстративного) матеріалу: 4 рисунок.

6. Календарний план-графік.

7. Дата видачі завдання: «26» квітня 2021 р.

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NATIONAL AVIATION UNIVERSITY

Faculty of environmental safety, engineering and technologies

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Specialty 162 “Biotechnology and bioengineering”

Education program “Pharmaceutical biotechnology”

APPROVED

Head of the Department

_____M.M.Baranovsky

«___» _____ 2021

TASK

FROM BACHELOR THESIS OF STUDENT

Rujinskiy M.O

1. The theme of the thesis: «Investigation of conditions for development of methods for determining of 2-methyl-4-chlorophenoxyacetic acid»

«11» may 2021 y. № 715/st

2. The term of the work: from 26 April till June 08, 2021.

3. Output data: Literature sources on the structure and physicochemical properties of Development of methods for determining of 2-methyl-4-chlorophenoxyacetic acid (MCPA). It`s toxicity, methods of synthesis. Scientific information on MCPA extraction and analysis methods.

4. Contents of the explanatory note: INTRODUCTION; CHAPTER 1. CHARACTERISTICS OF 2-METHYL-4-CHLOROPHENOXYACETIC ACID AND ITS USE AS AN ACTIVE SUBSTANCE OF HERBICIDES; CHAPTER 2. TOXICITY CHARACTERISTICS OF 2-METHYL-4 CHLOROPHENOXYACETIC ACID AND HERBICIDES WHICH IT INCLUDES;

CHAPTER 3. METHODS OF DETERMINATION OF 2-METHYL-4-CHLOROPHOXYACETIC ACID; CONCLUSIONS; REFERENCES:

5. List of compulsory graphic (illustrative) material: 5 figures.

6. Schedule.

№	Task	Execution term	Supervisor Signature
1	Selection of the theme of the thesis, agreement of the content with the supervisor.	26.04.2021	
2	Drawing up of the scheme of performance of the bachelor thesis.	27.04.2021	
3	Selection and processing of literature on the characteristics of 2-methyl-4-chlorophenoxyacetic acid and its use as an active ingredient in herbicides.	28.04.2021 – 02.05.2021	
4	Selection and processing of literature on the toxicity of 2-methyl-4-chlorophenoxyacetic acid	03.05.2021 – 04.05.2021	
5	Selection and processing of literature on methods of obtaining of 2-methyl-4-chlorophenoxyacetic acid.	05.05.2021 – 08.05.2021	
6	Selection and processing of literature on the methods of extraction of of 2-methyl-4-chlorophenoxyacetic acid	09.05.2021 – 16.05.2021	
7	Writing the thesis.	24.05.2021 – 29.05.2021	
8	Examination of the thesis by the supervisor.	30.05.2021	

9	Editing of the speech and final presentation	31.05.2021 - 01.06.2021	
10	Preliminary defence of graduating work.	02.06.2021	
11	Defence of graduating work	06.2021	

7. Date of task receiving: «12» May 2021

Supervisor of bachelor thesis _____ Maga I.M.
(sign of supervisor)

Task for execution was taken by _____ Rujinskiy M.O.
(sign of graduating student)

РЕФЕРАТ

1. Пояснювальна записка до дипломної роботи «Розробка методики визначення 2-метил-4-хлорофеноксіацетатної кислоти»: 44 сторінок, 4 рисунок, 20 використаних джерел.

2. Об'єкт дослідження – Розробка методики визначення 2-метил-4-хлорофеноксіацетатної кислоти. Важливість методів визначення МЦПА

3. Мета дипломної роботи – дослідження умов визначення Розробка методики визначення 2-метил-4-хлорофеноксіацетатної кислоти спектроскопічними та хроматографічними методами

4. Методи дослідження – аналітичні, синтетичні, математичні, порівняння.

5. Предмет дослідження – 2-метил-4-хлорофеноксіацетатної кислоти, гербіциди містять МЦПА, рослинні продукти, екологічні об'єкти, методики аналізу.

Ключові слова: :2-МЕТИЛ-4-ХЛОРОФЕНОКСІАЦЕТАТНА КИСЛОТА, ТОКСИЧНІСТЬ, МЕТОДИ ДОБУВАННЯ, ЕКСТРАКЦІЯ, ЕКОЛОГІЯ, ГАЗОРІДИННА ХРОМАТОГРАФІЯ, ТОНКОШАРОВА ХРОМАТОГРАФІЯ, МЕТОДИКИ ВИЗНАЧЕННЯ.

ABSTRACT

1. Explanatory note to the thesis Investigation of conditions for Development of methods for determining of 2-methyl-4-chlorophenoxyacetic acid , 44 pages, 4 figures, 20 sources used.

2. Object of investigation: Investigation of methods for determination and extraction of 2-methyl-4-chlorophenoxyacetic acid. The importance of developing methods for determining MCPA.

3. Purpose of the work - study of conditions for determination of 2-methyl-4-chlorophenoxyacetic acid by spectroscopic and chromatographic methods.

4. Methods of research – Analytical, synthetic, mathematical, comparative.

5. Subject of investigations: 2-methyl-4-chlorophenoxyacetic acid, herbicides contain MCPA, plant products, ecological objects, method of analysis.

Key words: 2-METHYL-4-CHLOROPHENOXY ACETATE ACID, TOXICITY, EXTRACTION METHODS, EXTRACTION, ECOLOGY, GAS-LIQUID CHROMATOGRAPHY, TONOCHART CHROMATOGRAPHY, METHODS OF DETERMINATION.

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ВСТУП

Мета дипломної роботи. Розробка методики визначення 2-метил-4-хлорофеноксіацетатної кислоти та умов її вилучення .

Для досягнення цієї мети було поставлено наступні **завдання**:

1. Проаналізувати стан та властивості 2-метил-4-хлорофеноксіацетатної кислоти її властивості як сполуки та як активного інгредієнту гербіцидів.

2. Визначити токсичні характеристики МЦПА.

3. Дослідити схеми синтезу МЦПА.

4. Дослідити методики виділення та методи визначення МЦПА.

Об'єкт дослідження: дослідження методів визначення та вилучення МЦПА. Важливість визначення МЦПА.

Предмет дослідження: 2-метил-4-хлорофеноксіацетатної кислота, гербіциди містять МЦПА. Продукти рослинного походження, екологічні об'єкти.

Методи дослідження: аналітичні синтетичні, математичні, порівняння.

Наукова новизна отриманих результатів. Проведено нові теоретичні дослідження стану та властивостей 2-метил-4-хлорофеноксіацетатної кислоти, гербіцидної активності, токсичності, методів добування, методів виділення, а також різних методів аналізу МЦПА.

Практичне значення отриманих результатів. Описані важливі методи добування, синтезу 2-метил-4-хлорофеноксіацетатної кислоти, способи її виділення з різних матриці, чутливі методики визначення МЦПА хроматографічними методами, які можуть лягти в основу розробки нових високочутливих методик аналізу 2-метил-4-хлорофеноксіацетатної кислоти.

Особистий внесок випускника. Весь обсяг теоретичних досліджень за темою дипломної роботи, аналіз літературних даних, опис та обробка проаналізованих матеріалів виконані випускником особисто під керівництвом к.х.н., доцента

Мага І.М.

INTRODUCTION

Actuality. « 2-methyl-4-chlorophenoxyacetic acid (MCPA) is an important and selective systemic herbicide. It is effective against annual and perennial broad-leaved weeds and brushed species in cereals, maize, sorghum, sugarcane, turf, pastures, range land and non-crop areas. MCPA is absorbed through roots as well as leaves and translocates throughout the plant. It mimics auxin, a plant growth regulator and at adequate concentrations, is known to increase plant growth rate that outgrows its nutrient supplies leading to death of the plant. MCPA in combination with phenoxy or other herbicides is used in pastures, range land, and non-crop areas to control weeds.

The purpose of the work is investigation of the conditions for determination of 2-methyl-4-chlorophenoxyacetic acid.

To achieve this goal, the following tasks were set:

1. Analyze the status and properties of 2-methyl-4-chlorophenoxyacetic acid and its properties as a compound and as an active ingredient in herbicides.
2. Determine the toxic characteristics of MCPA.
3. Investigate the schemes of MCPA synthesis.
4. Establish methods for extracting MCPA from the studied objects.
5. Investigate recovery techniques and methods for determining MCPA.

Object of investigation: investigation of methods for the determination and extraction of 2-methyl-4-chlorophenoxyacetic acid. The importance of MCPA determination.

Subject of investigations: 2-methyl-4-chlorophenoxyacetic acid, Herbicide preparations containing MCPA, Food of plant origin, Environmental objects.

Research methods: analytical, synthetic, mathematical, comparative.

Scientific novelty of the results. New theoretical studies of the reserches and properties of 2-methyl-4-chlorophenoxyacetic acid, herbicidal activity, toxicity, extraction methods, isolation methods, as well as various methods of analysis of dicamba.

The practical significance of the results. Descriptive important methods of extraction, synthesis 2-methyl-4-chlorophenoxyacetic acid, sensitive methods for determining MCPA by spectroscopic and chromatographic methods.

Graduate's personal contribution. The entire volume of theoretical research on the topic of the thesis, analysis of literature data, description and processing of the analyzed materials were performed by the graduate personally under the guidance of Ph.D., Associate Professor Maga I.M.

CHAPTER 1

CHARACTERISTICS OF 2-METHYL-4-CHLOROPHENOXYACETIC ACID AND ITS USE AS AN ACTIVE SUBSTANCE OF HERBICIDES

1.1. Overall characteristic of pesticides

For high crop yields along Using fertilizers is of great importance to the use of chemical means of protection against pests, diseases and weed plants, i.e. pesticides. Without them, modern cultivation technologies are unthinkable [1 – 2]. In some cases, the use of pesticides gives a greater effect, How applying fertilizers. The use of pesticides, in addition to their main actions can also improve the absorption of nutrients with plants, What promotes harvest. Thus, widespread use Pesticides in Western Europe made it possible to improve grain crops In the 90s, twice as compared with the 70s. [3].

Pesticides include chemicals, as well as biological Means used to combat various harmful organisms: insects (insecticides), bacteria (bactericides), mushrooms (fungicides), higher plants (herbicides), vegetative ticks (acaricides), Mollusks (Limacides), round worms (non-Natocides), TLEY (AFADI), larvae and caterpillars (Larvicides), with rodents (zoocides), for Destroying algae and other weed vegetation in reservoirs (algicides) and others. This group of substances also include antiseptics that Used to protect wooden and other non-metallic materials from destruction by microorganisms; substances used for Pre-gauge removal of leaves from plants (defoliates); Caller dehydration of plant tissues, which speeds up their ripening and facilitates harvesting (desiccants), pre-sowing seed processing (rulators seeds) [4].

Some chemical drugs can act on harmful Organisms only with direct contact with the object Impact (contact pesticides). Systemic pesticides are capable move along the vascular system of the plant and, in some cases, Vascular animal system. They often turn out to be more efficient than contact proceedings [5].

Many pesticides have toxic properties, may accumulate in environmental facilities and enter the human body through the respiratory tract, the gastrointestinal tract, skin and mucous Shell [6 – 7].

The following chemical classes have the greatest application. Compounds: chlororganic and phosphorodorganic pesticides, Dithiocarbamic acid derivatives and chlorophenoxy cells, Dinitrophenol and mercury compounds, copper-containing Preparations and others. Passed state registration of pesticides more than 20 Chemical classes based on 1000 chemical compounds. In the world More than 100 thousand pesticides are used [8 – 7].

Currently, the global pesticide market is estimate at about 30.5 billion dollars. In Russia in 2006, pesticides were applie approximately by 380 million dollars, at the same time in Germany this indicator in Value terms amounted to 1.4 billion dollars. In 2007, the volume Sales in the USA 6.077 billion dollars, the share of herbicides accounted for 64.4% Market. In our republic, the volume of pesticides used in 2009 amounted to 12948.7 thousand tons, including herbicides - 9793.2 thousand tons. The volume of application of pesticides in Russia in Russia is growing every year, The increase in 2012 slowed down regarding the previous year and amounted to 8% [9, 10, 11].

1.2. Herbicides

Herbicides (from Lat. Herba - Grass and Caedo - I kill) - Chemical Substances used to destroy weed vegetation. IN Agricultural Encyclopedia of 1974 given the following interpretation weeds: "Plants whose growing in this place for any The reason is undesirable. " State standard of our country (GOST 16265-89) established such definition: "Weighing plants - wild Plants living on agricultural land and reduce The magnitude and quality of products. " In each soil and climatic zone. The greatest harm to agriculture is a caused several hundreds, and on Separate fields are no more than ten species of weeds [13 – 12].

Reduction of the use of herbicides in the 90s associated with the economic difficulties of the country led to an increase in the tones of crops. Thus, the loss of harvesting from weed plants for several years amounted to approximately 20% [14].

Pesticides by the nature of their effect on plants are divided into herbicides of total action, killing all types of plants, and herbicides of selective (selective) action, destroying some types of plants and not damaging others. In most cases, one and the same pesticide, depending on the concentration and conditions of application, can manifest itself as a herbicide of continuous or selective action [15]. A distinction is made between biochemical and topographic selectivity of a herbicide. At biochemical selectivity herbicide action is associated with its interference in the metabolism of plants sensitive to it. B resistant plants, the herbicide is detoxified. Topographic Herbicide selectivity is associated with differences in the anatomical and morphological structure of plants and the method of herbicide application. Topographic selectivity explains the increased sensitivity to herbicides of plants growing under certain conditions, for example, in the shade on fertile moist soil [16 – 17].

Herbicides can be broadly or narrowly selective. MCPA, which destroys all dicotyledons, is broadly selective, while propanide, for example, which destroys millet in rice crops, is narrowly selective. Many herbicides can be used both for treatment of above-ground parts of weeds (leaf weeds) and for application to the soil surface (soil or root weeds) [19 – 17].

Modern herbicides are divided into several large groups by chemical composition: ethers, substituted phenols, quaternary ammonium compounds, aryloxyalkane carboxylic acids and their derivatives, benzoic acids, halogenated aliphatic acids, carbamates, thiocarbamates, amides, urea derivatives, phosphonic derivatives pyridine, bipyridylum, imidazolinones, thiadiazines and other organic compounds [20].

More than 1000 compounds with herbicidal properties are known, but for more than 150 are used for weed control. Herbicides are produced mainly in the form of solutions, powders (soluble in water), pastes, concentrates, and emulsions.

In addition to the active substance, the preparation contains a filler - any neutral substance (talc, chalk, kaolin), as well as surfactants, wetting agents (OP-7, OP-10), adhesives and other additives that increase the effectiveness of the herbicide [20].

The main method of using herbicides is the treatment of plants using ground sprayers and from aircraft with aqueous solutions, water emulsions and suspensions. The timing of herbicide application: before or after fall plowing, at different times before sowing an

agricultural crop, in the period from sowing to germination and after emergence of cultivated plants in different phases of their development [11 – 8].

The dose of herbicides depends on the degree of contamination of the fields, characteristics culture, soil and climatic conditions and agricultural techniques. At the same doses, herbicides are generally weaker with a decrease in temperature (below 8-12 ° C), and with an increase, they are stronger. On light soils, less herbicides are required than on heavy ones, rich in humus [6].

Chemical methods of weed control are usually use in combination with agrotechnical. Biological methods of weed control are preferred over chemical ones, but they are not yet widely used. The use of herbicides leads to significant savings manual labor [7].

1.3. General characteristics of MCPA

MCPA (2-methyl-4-chlorophenoxyacetic acid) is a powerful, selective, widely used phenoxy herbicide. His chemical formula is (Fig 1.1.). The pure compound is a brown powder. MCPA has been widely used in agriculture

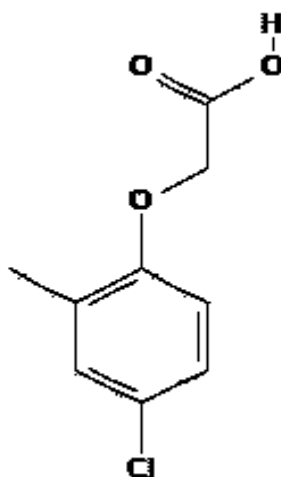


Fig 1.1. The chemical structure of MCPA.

to control broadleaf weeds as a growth regulator, mainly in pastures and grain fields since 1945. MCPA acts like auxin, a growth hormone that is naturally found in plants. An

overdose of MCPA acts as a herbicide and results in abnormal growth [17]. His chemical formula is (Fig.2)

Synonyms: Agritox; **Agroxon; Agroxone; Anicon Kombi; Anicon M;** BH MCPA; Bordermaster; Brominal M and Plus; B-Selecton M; Chiptox; 4-chloro-o-cresolexyacetic acid; 4-chloro- 2-methylphenoxyacetic acid; 4-chloro-o-toloxycetic acid; Hvastox; Cornox; Kornox-M; Translate even faster with DeepL for Windows Synonyms: Agritox;

Agroxon; Agroxone; Anicon Kombi; Anicon M; BH MCPA; Bordermaster; Brominal M and Plus; B-Selecton M; Chiptox; 4-chloro-o-cresolexyacetic acid;

MCPA concentrate; Weedone; Weedone MCPA ester; Weedrhap; Zelan.

1.4. Environmental Fate of MCPA

Biological. Extracts of characters isolated from pseudomonas SP. Basically, the MCPA saline medium has decomposed to 4-chloro-o-cresol and glyoxylic acid (Gamar and Gant, 1971).

The soil. Residual activity in soil is limited to about 3-4 months (Hartley and Kidd, 1987). Plant. Penetration, translocation and metabolism of radioactive labeled MCPA in graceful corn weeds (Galium Aparine) were studied in leaves (1962). Carbon dioxide is defined as a metabolite, but only 7% of the applied MCPA. Although no additional compounds have been found, it has been suggested that MCPA is detoxified in weeds due to the loss of both side chain carbon atoms (Leaves, 1962).

Photographic. When MCPA in dilute aqueous solution was exposed to summer sun or internal photoreactor (290 nm), 2-methyl-4-chlorophenol formed as the main product, as well as o-cresol and 4-chloro-2-formal. and Crosby, 1975.). Klelki et al. (1986) studied the photo content of an aqueous MCPA solution (120 ppm, pH 5.4, 25 ° C) in a photoreactor equipped with a high pressure mercury lamp. After 3 minutes irradiation, 4-chloro- and 2-methylphenol is formed as an intermediate product that decomposes to 2-methylphenol. Both compounds were not detected after 6 minutes of irradiation; However, 1, 4-dihydroxy-2-methylbenzene and 2-methyl-2, 5-cyclohexade-1, 4-dione formed as major and minor

photodepressant products, respectively. The same experiment was carried out using simulated sunlight (300 nm) in the presence of riboflavin, a known photosensitizer. 4-chloro-2-methylphenol and 4-chloro-2-methylbenzyl format are formed as primary and secondary products, respectively (Claps et al, 1986). MCPA, degraded by ozone, in dilute aqueous solution with and without ultraviolet light (300 nm) (Benua-Gayo et al., 1986).

1.5. Physical and chemical properties

Physical and chemical properties: in pure form - a white crystalline substance, has a smell of chlorocrisol or odorless. The technical product has a part of 0.3% chlorocrisol. The substance is poorly soluble in water, well - in ether, alcohol, benzene and other organic solvents. The drug is stable with prolonged storage in solutions and solid form in a metal sealed container, the shelf life is practically not limited.

Description: The mechanisms of action of MCPA are practically identical. The selectivity of their actions is associated with the accumulation of weeds of exchange, in particular, nitrogen (above the norm is 20 times), while in cultivated plants the amount of this element does not significantly increase. These drugs violate in the weeds of the process of oxidative phosphorylation. In the dicotyledonous breath intensity, the energy formed is not accumulated, but is lost. When spraying MCPA small drops fall on stems and leaves. To show phytotoxic action, they must overcome the barriers of protective tissue and penetrate inside the sheet. Preparations can penetrate through the stomachs or a cuticle, permeable to lipophilic and hydrophilic compounds due to the presence of micropores - ectodes.

Oil solutions, having a dissolving ability, are better penetrated through a cuticle. Water-soluble concentrates, etc. Working with sufficient moisture of plants when water fills the micropores of a cuticle.

Cell shells are not an obstacle to penetration of the drug, through them well and lipophilic, and hydrophilic substances, and in young leaves, have more subtle cover tissues and open stomachs, they penetrate better with beneficial weather.

MCPA substances cause hypertrophied distribution of meristemic cells, [6] deformation of stems and leaves, formation of air roots. In the damaged plants of the molds grow tissues, the plant is distorted, he stops.

In the root part, thickening is form, from which additional roots appear. Young roots die. There are other morphological changes. In weeds treated with herbicides, the intensity of breathing is intensified, the process of photosynthesis as a result of the destruction of chlorophyll, is subjected to hydrolytic decomposition of complex organic substances - protein, starch, inulin, the synthesis processes are weakened, the content of moving forms of carbohydrates and sugars increases, the precipitate of nitrogen is increased, Phosphorus and potassium, the synthesizing activity of the root system is stopped. As result of a violation of water exchange, plants lose turgor and begin to fade.

1.6. The synthesis of MCPA

In 1959-1961, experimental production of MCPA the amount of 5 tons per year from 30% of the preparation "Khvastoks R-30" was initiate at IPO-Warsaw. This allowed extensive agricultural research, including studies on 15500 flax plantations. In 1962, the first technical plant of 1000 tons of Khvastoks R-30 preparation was launch in Nowa Sarzhina (Moszczyński et al, 1963).

The synthesis was based on chlorination of 2-methylphenol with chlorine gas. Technical 4-chloro-2-methylphenol was condens with monochloroacetic acid (MCAA) without purification. The reaction scheme is show below (Fig 1.2).

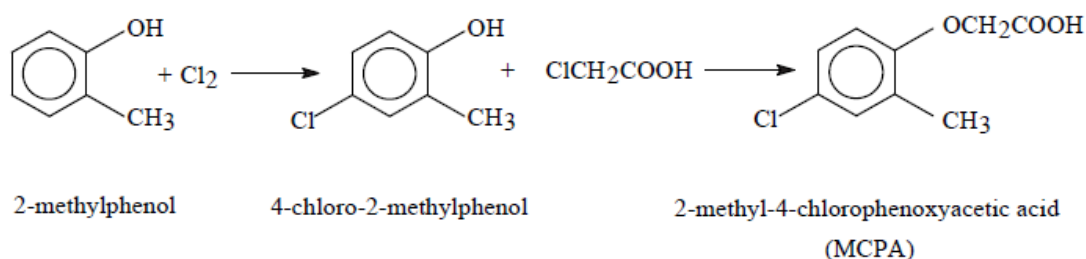


Fig 1.2. Synthesis of MCPA

The chlorination reaction of molten 2-methylphenol had a very low selectivity of 60 to 70%. Technical 4-chloro-2-methylphenol was a mixture of chlorophenols of the following composition:

4-chloro-2-methylphenol 60-70%

6-chloro - 12-20%

4,6-dichloro- 3-9%

After condensation of technical 4-chloro-2-methylphenol with chloroacetic acid, a mixture of a mixture of chloromethylphenoxyacetic acids containing 60 to 70% MCPA was obtained.

Such a product is referred to below as "MCPA 70". During the condensation process, the efficiency varied widely, and there was always a few percent of unreacted chloromethylphenols.

1.7. MCPA effect on weed and cultural plants

Derivatives of aryloxyalkancarboxylic acids, including MCPA preparations, belong to the group of herbicides with auxin-like activity, since they have much in common with natural auxins. Synthetic auxins, in contrast to natural ones, do not lend themselves to endogenous regulation and do not lose activity when they are excessively supplied to plants. The consequence of this is an excessive intensity of cell division and an increase in respiratory processes in which ATP is not formed (uncoupling of oxidative phosphorylation), a weakening of the supply of nutrients and a depletion of the carbohydrate supply. The effect of herbicides on plants is realized at the level of mechanisms of nucleic acid metabolism, synthesis of proteins, various enzymes [12].

The mechanisms of action of 2,4-D and MCPA are almost identical. The selectivity of their action is associated with the accumulation of metabolic products in weeds, in particular, nitrogen (20 times higher than the norm), while in cultivated plants the amount of this element does not noticeably increase. These drugs disrupt the process of oxidative

phosphorylation in weeds. In dicotyledons, the intensity of respiration increases, the resulting energy is not accumulate, but is lost. The latter leads to the fact that plants lose adenosine triphosphoric acid (ATP), a compound rich in energy [8].

When spraying 2,4-D and MCPA, small drops fall on the stems and leaves. To be phytotoxic, they must overcome the barriers of the protective tissue and penetrate into the leaf. [4] The drugs can penetrate through the stomata or cuticle, which is permeable to lipophilic and hydrophilic compounds due to the presence of micropores - ectodesmus [4].

1.8. Industrial receipt

In industry, 2-methyl-chlorophenoxyacetic acid is obtain two methods: condensation of MCA with salts of monochloroacetic acid and chlorination of 2-methyl-phenoxyacetic acid. The first reaction is carried out in an alkaline medium ($\text{pH} \approx 10$) at $103 - 105^\circ\text{C}$ on (Fig 1.3).



Fig 1.3. Condensation of MCA with salts of monochloroacetic acid

Both pure 2-methyl-4-chlorophenol and technical product containing 6-chloro- and 4,6-chloro-2-methylphenols can be used as a starting product. However, phenol is chlorine, sodium hypochlorite or sulfuryl chloride.

2-Methyl-4-chlorophenol is obtained by chlorination of phenol with chlorine, sodium hypochlorite or sulfur chloride. In the latter case, the more pure 2-methyl-4-chlorophenol is formed. Other chlorinating agents give a mixture of chlorocresols containing 65-70% 2-

methyl-4-chlorophenol. The isomeric 2-methyl-6-chlorophenol can be used to make wood preservatives. The second method of obtaining 2M-4X by chlorination is of great practical importance (Fig 1.4.).

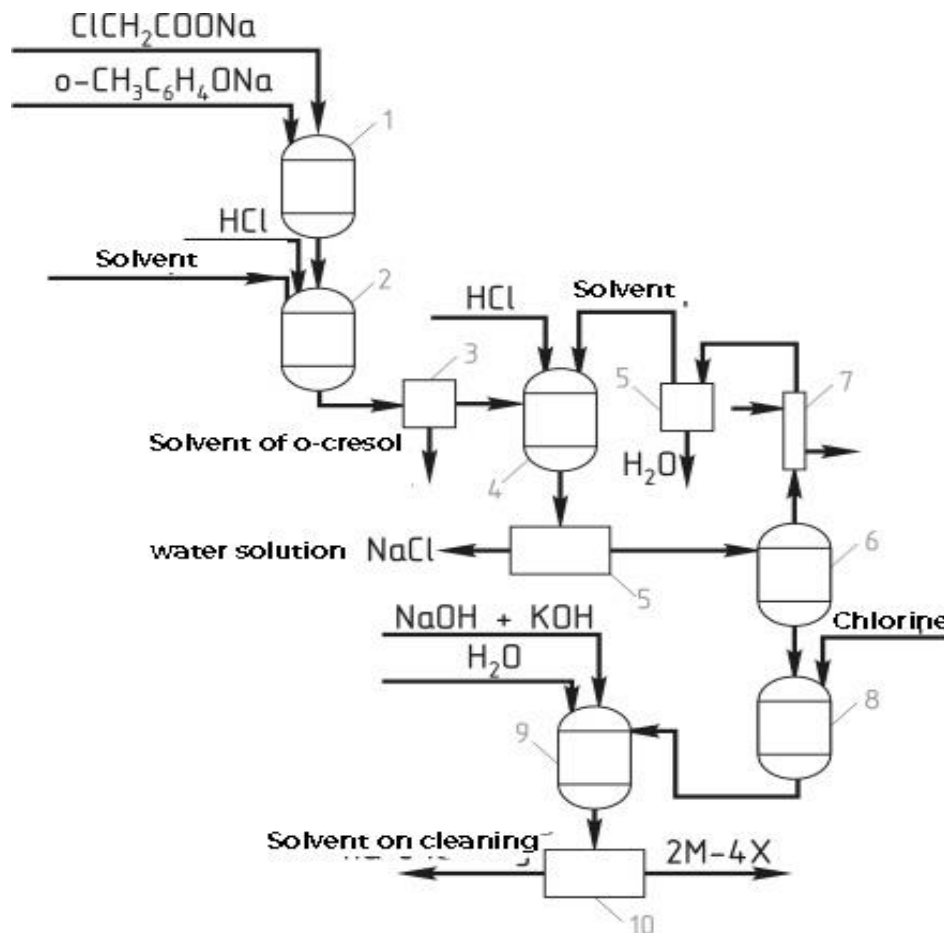


Fig 1.4. Principlesal technological scheme of deeds 2M-4X: 1— Sodium salt fusion reactor 2-methylphenoxic acid; 2 — o-cresol extraction machine; 3 — o-cresol separation machine; 4 — Apparatus for extraction 2-methyl phenoxyacetic acid; 5, 10 — Separates for separating water and solvent 6 — The device for drying acid; 7 — heat exchanger; 8 — chlorinator; 9 — Apparatus for extraction 2M-4X

1.10. Conclusion to chapter

2-methyl-4-chlorophenoxyacetic acid (MCPA) belongs to the synthetic auxin class and exhibits herbicidal activity. Chemically, MCPA is a weak organic acid. The most commonly used forms of herbicides are dimethylamine and sodium salt. Its effect is manifested in increasing the rate of RNA synthesis and its concentration, accelerating the synthesis of lipids and protein, increasing the extensibility of membranes and cell growth in length.

CHAPTER 2

TOXICOLOGICAL PROPERTIES AND CHARACTERISTICS OF 2-METHYL -4 -CHLOROPHENOXYACETIC ACID

2.1. Toxicological properties and characteristics

In the soil, MCPA preparations are rapidly decomposed. Half-life - 14 days - 1 month, [7] 4-6 weeks. [14] In case of drought - two to three months [14].

In plants, MCPA is identical to 2,4-D. The substance also gives conjugants with the waste products of organisms [9].

Useful species and entomophagy MCPA preparations are low toxic for bees, birds and wild animals [10].

Warm-blooded. MCPA compounds can enter the body of warm-blooded animals and humans through the respiratory system, gastrointestinal tract and skin. The drugs have a pronounced skin-resorptive toxicity, cause changes in the skin and mucous membranes [8].

Human. The substance is moderately dangerous for humans, has a pronounced skin-resorptive effect [11].

Accidental ingestion of 22 g of MCPA potassium salt (300 mg / kg) caused death 20 hours later. The pathomorphological examination of the deceased showed enlargement of the heart, pallor of the myocardium, venous congestion and fatty degeneration of the liver with foci of necrosis. Significant amounts of the herbicide were found in internal organs (liver, brain, stomach), as well as in blood and urine [8].

Poisoning symptoms. The clinical picture of poisoning upon admission through the gastrointestinal tract is characterized by odor from the mouth resembling carbolic acid, burning sensation in the mouth and stomach, nausea, vomiting and diarrhea, cyanosis of the lips, fingertips, ears, and attacks of clonic-tonic seizures. The urine is green [8].

A local irritating and general toxic effect was observed in a woman who worked with the drug without personal protective equipment. Already on the first day there were complaints of burning of the skin of the hands, paresthesia, an unpleasant sensation in the

pharynx, dizziness, and nausea. After working for three days, the patient was admitted to the clinic with complaints of vomiting, nausea, pain in the epigastric region and right hypochondrium, itching, burning of the skin of the limbs and trunk, discomfort in the heart, occasionally headache, general weakness. Objectively revealed hyperemia of the skin, generalized rash (petechial, confluent, vesicular), enlarged liver, hypotension. The tongue is dryish, coated with a white bloom, from the mouth there is a sharp specific odor. ECG shows sinus arrhythmia. 2.5 months after poisoning - pain in the right hypochondrium, facial flushing, pale pigmentation at the sites of the former vesicular rash, serum bilirubin with direct diazoreaction [8].

Subject to the precautions taken, the herbicide workers did not show noticeable changes in health [8].

Reproductions exposed to MCPA (95% A.I.) at 0, 3.3, 10, or 30 mg / kg body weight per day in the two-generation diet were not detected in rats [14]. After oral administration of MCPA (75% Ai) at 0, 5, 25 or 100 mg / kg body weight per day to mice on days 6-15 of pregnancy, significantly reduced fetal weight and skeletal ossification delay were observed at the highest doses [15]. MCPA (purity not specified) was administered (0, 20, 50 or 125 mg / kg body weight per day) by gavage to pregnant CD rats (16-38 per dose) on days 6-15 of pregnancy. No maternal or fetal toxicity or teratogenic effects were observed [16]. Intragastric administration of technical MCPA (700 mg / kg) on days 9 or 10 of pregnancy to Wistar rats caused an increase in the frequency of resorption, a decrease in fetal weight and the appearance of major malformations [12]. After administration of MCPA (0, 5, 12, 30 or 75 mg / kg body weight per day) by gavage to rabbits on days 6-18 of pregnancy, no fetotoxicity or teratogenicity was observed at any dosage level. Body weights were not significantly reduced in the group of 75 mg / kg body weight per day. Fetal N oael 75 mg / kg body weight per day was detected, and maternal noal 30 mg / kg body weight per day [13].

Table 2.1

Toxicological data

(ADI) (mg / kg human body weight)	0,002
(APC) in soil (mg / kg)	0,04
(MPC) in water of reservoirs (mg / dm ³)	0,003
(TAC) in water of reservoirs (mg / dm ³)	(org.)
(MPC) in the air of the working area (mg / m ³)	10. 0
ATMOSPHERIC OBS (mg/m ³)	0,001
MRL in products (mg / kg):	
in legumes	0.05
in cereal grain	0.05
in potatoes	0.05
in sunflower (oil)	0.05
in millet	0.05
In rice	0.05

2.2. Subjects and methods

THC examines the production of MCPA from 1947 to 1982. The process entailed the condensation of orthoosquol with sodium monochloroacetate to form sodium caresoxyacetate, which was then acidified and reacted with chlorine gas to produce MCPA. The production was carried out at the same site with the majority of the labor taken from the local rural population. Several other agricultural chemicals were produced at the plant during the same period, the most important being copper oxychloride (1943-1962), dinitro Orthozole (1943-1962), as well as a variety of organophosphorus insecticides (1948) and

Chlortriline herbicides (1964 on the). In addition, in many other products, 2,4,5-t and other phenoxy acids have been purchased and formulated, including secondarily. However, at Com Prison with MCPA, 2,4,5-t was processed only on a small scale [12].

Until 1972, the Company also operated an aerial and tractor spraying service from a warehouse scattered across a wide area of rural UK [14].

Until 1972, the company "ROKA company TORZHOZHO" carried out a bulk on the position "TRADE with windmills" on tractors of the warehouse "Z". Roughs in wide areas of the British Sailles. The assortment for the alignment of chemicals was an analogue of Tim, which knocked down in the form of formists, the alley gave it to be flavored. The phenochemical (mainly MSR) is widely separated from the perigodes of birch in Sherry, Alila 2,4,5-t was not practical verbal. J. Bulka Z Tim, Shako Robot Bula Seasonal, Bagato Z Oproskuvachiv walked Mesen Six Mes. Sii Choloriki was included in Slobudnitskaya, the group was included in the subprommic in Perigord from 1 s) 1947 Roca on December 31, 1975 Roca on reliable Yoy Fabdari ABO warehouses. MI visited the coils of the personnel, and for spells of the skin they took narrative information, the date of BigPa, addresses, the date of work of Catherine and the end and the robot of the Torzoza Congobox. Based on the Board, plaques with names (and immediately operator ruppy - Datia to work) classified Lawkiroviks replied and the melting power of feathersislings. Bulas Violet three steps of fit - 'Temple', 'low' and 'backgrounds'. To the profession of Visokim Rivan, it is part of the main, main, chemical manufacturer, ruppilators and laboratory laboratory laboratories. To the profess of the lowest level, the maintenance staff is included in the middle of the state, the composition of the anniversary and transport of chemical rumbo.

The backgrounds of the exposition were delayed for the commercial personnel of the II, and the manager for the manager, the porch did not sign in the zone of chief-maker. In opera, spindle scabs occurs in the spring of the first summer period. Acting vipads when pantils changed mysce robot In those waxes, the catchers of the mineral thing is to work with the holiness of the highness of its work, with a frame document in vain Make information of boots about the rest of the profession. Under the guidance of Ecposikii, I realized that through the whole work machine Bob Cholevik Bulks are similar (point zero pussy) is a well-known occupation. Cleaning cereal Buli tracks in the central register of the

National School of Ocoron is a healthy (NSW) in the port of South (with problems that require national parties in Newcastle-on-Tiny), as well as visitors' budget, the status of 31 breasts 1983. The management of ash and polls Nemovoxal submit us a copy of the deaths of men, as died, the main reason for the death of the Minzimi-male classification of the Jewer (ICB). Kimm, Mi, they twisted details of information on the canopy of the BE-POW Zarovujan, which Bullets Zareston Seals Mission World for a period of avenue. Expecting the number of deaths from reasons of roses was the method of " Luba-Rokki " from wave roads of national maintenance of decompositions (according to Hinina and Welsh) for men would be in ICU and calendar dust. OSKILKI VALIDA PODSKIVNIKOV PID HOUR ROBOTI Lived in Rural Marsost, MI Deleted Otriki KDirkuvniy Numbers, Crucifixion for Standardization of Mortgage Mortgage on Concrete (SMR) for Silskiy District Angle Aggregates and Wels for PPIG 1968-1978. No live conscience does not include, Alla, in the context of mortality from crayfish at most, substrate, and the manual, lying in the interage of them. Results of statistical knowledge by the method and Dovirch and Kordonu Buli Rujanovanovi to Zvitch, singing by the Poisson method. ZVITCH methods, singing on Poisson dispensing [15].

2.3. Development of healing

Healing technology is reported, in fact, it is effective to reduce MCPA in drink consumption Water connects activated carbon adsorption and / or ionization, membrane filtering, UV irradiation and advanced oxidation processes (AOPS). Showed full and pilot studies. Actually that Stock Focusing MCPA is important below Mac 0.1 mg / l (100 µg / l) Reachable with the introduction of methods outlined below. Choosing the appropriate process of healing for a certain water supply will be dependent on a variety of moments, covering the properties of raw materials Water supply and operational circumstances of a certain method of healing.

2.3. Conclusion to chapter

A total of 5,784 employees met criteria for participating in the study, but 30 (0.5%) were not included in the analysis, since the date of birth or date of coming to the company were not received from personnel documents. Another 98 men (1.7%) could not be tracked in the central registration of NHS, and they could only be traced to the date of their departure from the company. Two hundred and fifty six people emigrated during the observation period and were traced to the date of their departure.

Of the 5,754 employees included in the analysis, 4,078 were recognized have a higher level than the 'rear plan' exposure to phenoxic acids (TABLE. 2.2). More than half of these people worked on potentially dangerous work at least six months. The total mortality in this cohort was lower than expected on the basis of national indicators, as well as mortality from all neoplasms, cardiovascular diseases and diseases of the respiratory system. Mortality from injuries and poisoning, as well as gastrointestinal diseases slightly increased, but not to the level of statistical significance. OP Application.

The coefficient of rural correlation, deficiency of deaths from Cancer is a slight excess, but again it is was not statistically significant.

A more detailed analysis showed that most of the main types of cancer are rarely rarely among labor than among the population. A small excess of prostate carcinoma (18 deaths, expected 13.5) and brain cancer (11 deaths, 8.7 expected) were statistically significant. Observation of 14 deaths from leukemia (8.0 expected) was more impressive, but five of these cases occurred in men with the background influence of phenoxits, and the distribution of leukemia for histological type was non-armamented (four chronic lymphatic leukemia, four acute myeloid leukemia, three Chronic myeloid leukemia, three acute myeloid leukemia).

Chronic myeloid leukemia, two acute monocyte leukemias and one unspecified leukemia). There were three deaths from the nasal cancer (0.7 expected), all in men with rn by the influence of phenoxic acids. Two of them worked as spray operators (for two and three months, respectively), and the third third worked for six months by the operator of the technological process in chemical production.

Table 2.2

Potential exposure to phenoxy acids among Subjects included in the analysis of mortality and cancer incidence

Type of potential exposure	<1 month	1-6 months	>6 months	Total
Manufacture and formulation	137	388	1020	1545
Application	322	1126	1111	2561
Any ^a	450	1502	2126	4078

a -Twenty-eight men were potentially exposed both in manufacture and spraying. A further 1 676 subjects were considered 10 have had only background exposure.

CHAPTER 3

METHODS DETERMINATION OF MCPA

3.1. Gas-chromatographic method

Chromatographic methods of analysis based on cyclic acts of sorption-desorption occurring between the mobile phase (eluent) with a dissolved sample and a stationary sorbent. The components of complex mixtures have different sorption properties, and passing along the stationary phase, they are absorbed at different rates and in different quantities. The subsequent study of the results and their comparison with the standard allows you to establish the exact composition of the reagent.

The procedure developed in Section 3.3.7 was applied to the analysis of MCPA in a water sample taken from Sydney Harbour at Botanical Gardens. Two samples were taken from the same location. A 2.5 litre Winchester bottle and a 2 litre volumetric flask, which had been cleaned carefully and contained 5ml of H₂so₄ (18'1), were used. The water sample was collected by holding the mouth of the bottle 2-3cm below the water surface which allowed the sea water to be drawn into the bottle until full. The sampling date was 1 June 1981, which was a public holiday. The samples were taken to the laboratory and two 500ml sea water extracted, esterified and processed as described in Section 3.3.7. Where the gas chromatogram indicated the presence of the MCPA herbicide, the residual ester was examined by thin layer chromatography on silica gel plates [16].

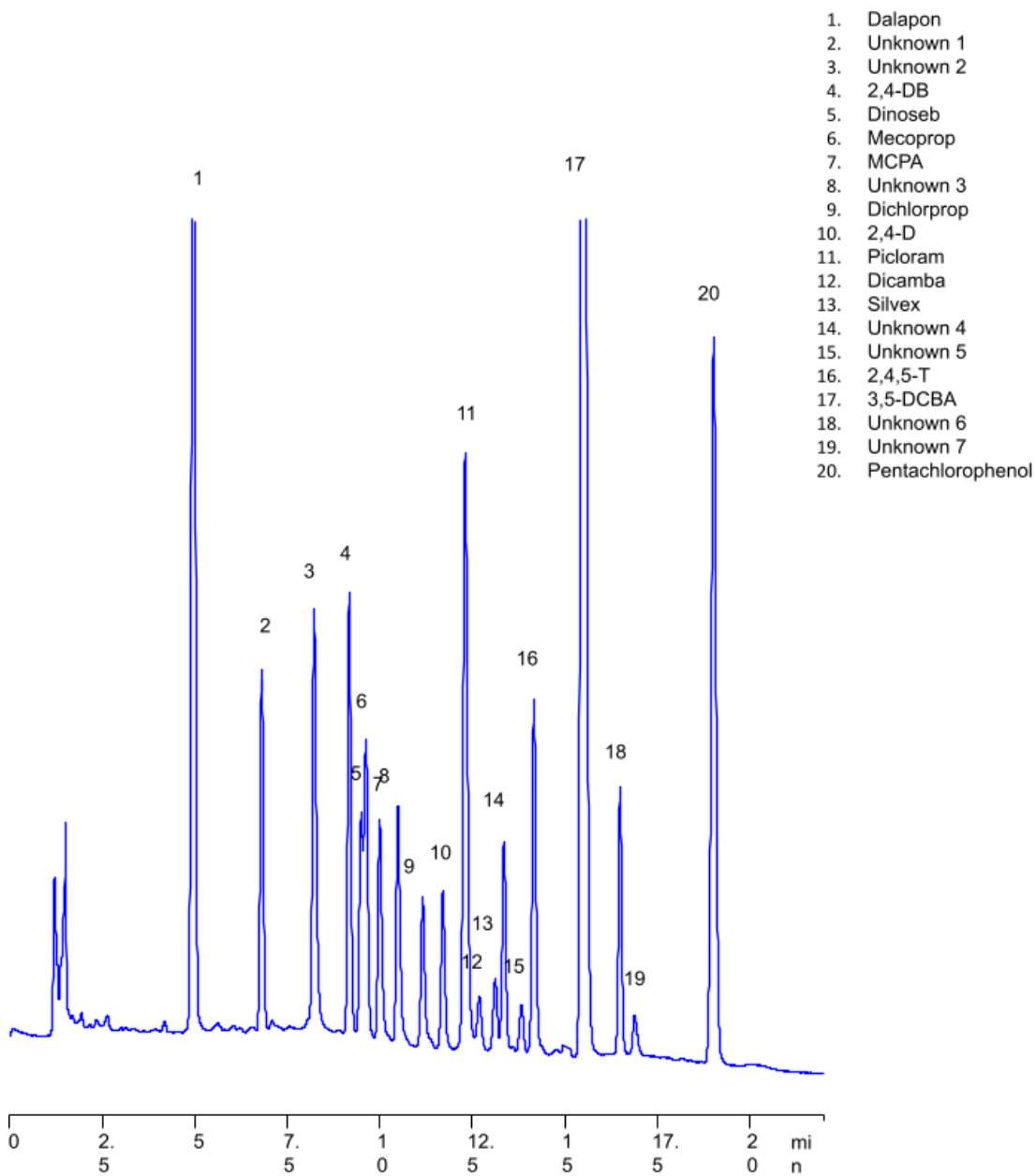
3.2. Gas-liquid chromatographic method

Gas-liquid chromatography is based on the physicochemical separation of the analyzed components in the gas phase, with their passing along the non-leisure fluid applied to the solid sorbent. This is one of the most promising analysis methods. The widespread and prospects of GLC methods are due to the fact that they allow to divide and quantify substances in a complex mixture even in cases where they are similar to chemical properties,

and boiling temperatures W differ in the tenths of degrees. For analysis requires very small amounts of matter, and the definition time is usually calculated minutes. The separation of the analyzed substances occurs in columns (tubes) filled with a solid porous sorbent, to which the liquid non-volatile stationary phase is applied. The pairs of analyzed substances mixed with the carrier gas are moving through the column. In this case, there is a repeated establishment of equilibrium between the moving gas and liquid stationary phases, due to the repeated repetition of the dissolution and evaporation processes. Substances, better soluble in the stationary phase, are held longer than it. Due to this, the analyzed mixture is separated into individual components, which come out of the column separately and recorded at the output.

The definitions are based on the extraction of herbicides from the acidic samples of water, ekstrakty by diethyl ether, the release of the MCP is submitted using the boron of the tropfluoride complex of the complex when determining by version 1 or ethanol according to version 2. MTP ester is removed from a mixture of n-hexane and quantitatively determined by gas-liquid chromatography with detector Capture electrons (Fig.4).

Identification. The Esters of the MCPA are carry out at the time of holding on chromatograms and esterified samples of hyrolides; by heighten their chromatographic peaks on the chromatograms of a standard sample solution [11].



Column: Coresep SB
Dimensions: 4.6x150 mm, 2.7 um, 90A
Mobile phase: ACN from 40% to 60% in 15 min, 5 min hold, 0.1% H₂SO₄
Flow: 1 ml/min
Detection: 220 nm

Fig 3.5. Gas -liquid chromatography with detector Capture electrons

3.3. Thin-layer chromatography method

Thin-layer chromatography (ros. Thin-layer chromatography, English). As a fixed phase is used silica gel, aluminum oxide, kieselgur, cellulose, polyamide materials, ion-exchange synthetic resins, mineral-organic jet, as a moving phase - organic solvents (eg alcohols, ketones, phenols, mixtures thereof). Methods of T.X. Allows to divide and identify substances in amounts of 10⁻⁹-10⁻⁶ g, perform analyzes from the capacity of up to 10⁻⁶ g. Use with a qualitative analysis of minerals. Particularly valuable use of T.X. In analyzing ores and minerals with close chem. Properties, for example. rare earth elements, zirconium and hafnium, niobium and tantalum and others.

Thin layer chromatography. Dry residue after evaporation of essential extract is dissolved in 1 ml of acetone and quantitatively applied to Chromatographic plate with a thin layer of silica gel or plate "Silufol". 2, 5 and 10 µg 2m-4x in the form of a solution are applied to the same plate in acetone and conduct chromatography in the system of solvents of n-hexane Diethyl ester-acetic acid (50: 50: 2). For chromatography Use a saturated chamber. To saturate the wall of the camera, look filter paper that concerns the mixture of solvents and soaked. 2 hours quickly open the camera lid and put Plate for chromatogram development. After graduation from chromatography The plate is removed from the chamber and dried in the air in the exhaust cabinet. For The discovery of the 2M-4X plate is treated with the manifestory reagent N 1, dried and They irradiate UV light within 10 - 15 minutes. If there are 2m-4x in sample The plate appears a gray-black stain on a white background with a magnitude R 0.30 +/- 0.01. As a second manifesting reagent may be F.

Bromocresole green. After the development of chromatograms, the record dried in air and placed in a drying cabinet at 110 ° C for 20 minutes. for removing traces of acetic acid and then processed by manifest Reagent N 2. The 2M-4x preparation is manifested in the form of a yellow spot on blue background. The minimum detectable amount on chromatograms of 1 µg. To improve the reliability of identification, the second can be used. Mobile solvent system: benzene acetic acid (5: 1). Value R 2M-4X in this system 0.54 +/- 0.01. f

It is possible to determine 2 m-4x in the presence of 2,4-dichlorophenoxyacetic acid 2,4-d and 2,4-dichlorophenol. R values for these connections in the system F. H-hexane-diethyl ester-acetic acid (50: 50: 2) - 0.25 and 0.50, in Benzol-acetic acid system (5: 1) - 0.48 and 0.65, respectively. Processing analysis results. The content of 2m-4x in the sample is assessed visually, comparing the size and intensity of the color of the stains of the standard compound with the size and intensity of the sample stains. To determine the content of 2M-4x in samples (x, mg / l or mg / kg), use the formula:

$$X = \frac{A}{PR},$$

Where:

A - the amount of 2m-4x in the sample found by comparison with the standard, μg ;

P - of sample, ml or g;

R is the definition percentage found.

Safety requirements. Security requirements complied with, usually recommended for work with chemical reagents [11].

3.4. Conclusions to chapter

Chromatographic methods are mainly use to determine MCPA. The method of gas chromatography, using capillary columns, is widely used, which is effective and highly sensitive. The definition of MCPA requires derivatization. Primary aliphatic alcohols are often use for derivatization. The method of TNL chromatography is also of great importance. Although the TLC method is significantly inferior in sensitivity to gas chromatographic methods, its relative cheapness, as well as the ability to determine MCPA at a level below the maximum permissible concentration, makes it available to a wide range of laboratories. As eluents for TLC H-hexane-diethyl ester-acetic acid or Benzol-acetic acid systems in various ratios of organic solvents. For a more accurate determination, a gas chromatography method used in combination with a very spectrometric detector (GC/MS/MS).

CONCLUSIONS

1. 2-methyl-4-chlorophenoxyacetic acid is a powerful, selective, widely used phenoxy herbicide. The pure compound is a brown powder. MCPA has been widely used in agriculture to control broadleaf weeds as a growth regulator, mainly in pastures and grain fields since 1945. MCPA acts like auxin, a growth hormone that is naturally found in plants. An overdose of MCPA acts as a herbicide and results in abnormal growth

2. The selectivity of the action of 2-methyl-4-chlorophenoxyacetic acid is associated with the accumulation of metabolism by weeds, in particular nitrogen (20 times higher than the norm), while in cultivated plants the amount of this element does not increase significantly. This drug disrupts the process of oxidative phosphorylation in weeds. With the intensity of respiration of dicotyledonous plants, the generated energy is not accumulated, but lost. The drugs penetrate the stomach or cuticle, permeable to lipophilic and hydrophilic compounds due to the presence of micropores - ectodes.

3. The synthesis of 2-methyl-4-chlorophenoxyacetic acid was based on chlorination of 2-methylphenol with chlorine gas. Technical 4-chloro-2-methylphenol was condensed with monochloroacetic acid. After condensation of technical 4-chloro-2-methylphenol with chloroacetic acid, a mixture of a mixture of chloromethylphenoxyacetic acids containing 60 to 70% MCPA was obtained.

4. MCPA substances cause hypertrophied distribution of meristemic cells deformation of stems and leaves, formation of air roots. In the damaged plants of the molds grow tissues, the plant is distorted, he stops.

5. MCPA compounds can enter the body of warm-blooded animals and humans through the respiratory system, gastrointestinal tract and skin. The drugs have a pronounced skin-resorptive toxicity, cause changes in the skin and mucous membranes. The substance is moderately dangerous for humans, has a pronounced skin-resorptive effect.

6. The definitions are based on the extraction of herbicides from the acidic samples of water, extracted by diethyl ether, the release of the MCP is submitted using the boron of the tropfluoride complex of the complex when determining by version 1 or ethanol according

to version 2. MTP ester is removed from a mixture of n-hexane and quantitatively determined by gas-liquid chromatography with detector Capture electrons.

7. Thin-layer chromatography As a fixed phase is used silica gel or aluminum oxide, Allows to divide and identify 3,6-dichloro-2-methoxybenzoic acid in amounts of 10^{-9} - 10^{-6} g, perform analyzes from the capacity of up to 10^{-6} g.

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