УДК 614.7:632.952.028
DOI 10.11603/mcch.2410-681X.2023.i1.13537

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DEVELOPMENT OF THE METHOD FOR DETERMINING THE CONTENT OF THE SYNTHETIC DYE DIAMOND BLUE FCF IN THE SORPTION MATERIAL BY THE HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY METHOD

Introduction. *To date, the use of unmanned aerial vehicles (UAVs) in Ukraine for the agricultural lands treatment is a new promising technology that requires detailed study and development of approaches to risk assessment and hygienic regulation. Development of a method that will allow measuring the content of diamond blue FCF in the sorption material (filter paper) is relevant.*

The aim of the study *– development of the method for determining the content of the synthetic dye diamond blue FCF in the sorption material by the high-performance liquid chromatography method.*

Research Methods. *The following materials were used: laboratory analytical balance Radwag® AS220.R2, rotary evaporator, cartridge Strata™ C18-e (55 µm, 70 °C) 500 mg/6 ml, f. Phenomenex, steel chromatographic column 150/4.6 Microsorb 100-5 C18, pre-column chromatographic 4/3 Microsorb 100-5 C18, liquid chromatograph "Shimadzu" with a diode array detector, Diamond blue FCF, standard, 87.7, paper filters de-ashed "red ribbon".*

Results and Discussion. *At the first stage of the research, samples were taken and prepared. For analysis, 2 parallel samples were taken. The next stage was the preparation of the sample for introduction into the chromatograph. The third stage was chromatography performing. At the final stage, the diamond blue FCF peak areas were determined and calculated on the chromatograms. The indicated method of determining the content of diamond blue FCF in the sorption material (filter paper) includes extraction of the dye from the sorption material (filter paper) with distilled water; solid-phase extraction and quantification of diamond blue FCF by reversed-phase HPLC with SF detection. This method differs from the known ones in that it makes it possible to determine the investigated dye in the sorption material.*

Conclusion. *The proposed method of containing diamond blue FCF in the sorption material (filter paper) will allow to evaluate the effectiveness and safety of the use of various models of UAVs in combination with various pesticide preparations when using different agrotechnical characteristics at the stage of pre-registration trials and scientific research.*

KEY WORDS: **diamond blue FCF; sorption material; high-performance liquid chromatography.**

INTRODUCTION. The introduction of advanced innovative agricultural technologies is the decisive factor in increasing the yield of agricultural crops and productivity of farms [1–3]. Precision farming seeks to use new technologies to increase crop yields and profitability, while reducing the traditional costs required to grow crops (land, water, fertilizers, pesticides, etc.). The introduction of systems based on the information technology in crop production usage gives positive economic results: monitoring the use of machinery and fuels and lubricants, control of fertilizers, plant protection products and seeds ensure the rational use of resources [4–6]. Systems of so-called precision agriculture, which are rapidly spreading in the leading world countries, are gradually being introduced, including in Ukraine.

To date, the use of unmanned aerial vehicles (UAVs) in Ukraine for the agricultural lands treatment is a new promising technology that requires detailed study and development of approaches to risk assessment and hygienic regulation. But it is already known that this technology allows to significantly reduce the risks for professional contingents, to reduce the loss of pesticide formulation outside the treated field, and therefore to reduce the negative impact on the population, to reduce the application rates, which has a positive effect both in toxicological and economic terms [1, 7–9].

Previously, a similar methodology for determining diamond blue FCF did not exist. There is a well-known method of determining the studied dye [10], chosen as a prototype, in which determination

is carried out by preparing food samples for extraction, carrying out extraction, purification and quantitative measurement. However, this method does not allow the determination of diamond blue FCF in the sorption material (filter paper) [11].

That is why the development of a method that will allow measuring the content of diamond blue FCF in the sorption material (filter paper) is relevant.

The aim of this study was development of the method for determining the content of the synthetic dye diamond blue FCF in the sorption material by the high-performance liquid chromatography method.

MATERIALS AND METHODS. The following measuring equipment, auxiliary equipment, reagents and materials were used for the development of the method: water bath, laboratory analytical balance Radwag® AS220.R2 with a measurement error of 0.0002 g, technical laboratory balance AXIS® AD1000, rotary evaporator, rubber pears for pipettes, chamber for solid-phase extraction, cartridge Strata™ C18-e (55 µm, 70 °C) 500 mg/6 ml, f. Phenomenex, steel chromatographic column 150/4.6 Microsorb 100-5 C18, pre-column chromatographic 4/3 Microsorb 100-5 C18, personal computer with software for processing chromatographic data “LabSolutions”, liquid chromatograph "Shimadzu" with a diode array detector, Diamond blue FCF, standard, 87.7 %, CAS RN 3844-45-9, aqueous ammonia 25 %, n.a.s., acetonitrile, for liquid chromatography, bidistilled water, orthophosphoric acid, n.a.s., sulfuric acid, H.C., indicator paper Acilit® pH 0–6.0, f. Merck, methyl alcohol, h.h., laboratory filter paper, 520×600 mm, 75 g/m^2, paper filters de-ashed “red ribbon”.

The main physicochemical properties of diamond blue FCF are given in Table [12].

RESULTS AND DISCUSSION. At the first stage of the research, samples were taken and prepared. For analysis, 2 parallel samples were taken.

To carry out the extraction, the sorption material (filter paper) with an area of 2 dm^2 was crushed with scissors and introduced into a conical flask with a capacity of 250 ml. 50 ml of distilled water was added and vigorously shaken by hand for 1 minute. The obtained extract was decanted into another conical flask with a capacity of 250 ml. The extraction was repeated. The flask with the sorption material was washed with 10 ml of distilled water, which was added to the main extract.

To perform solid-phase extraction [13] on a Strata™ C18-e cartridge, (2–3) ml of a 1 M aqueous solution of sulfuric acid was added to the combined extract obtained earlier until the pH value was set to ≤ 2, the pH control was carried out according to the indicator paper. The extract was applied to the prepared Strata™ C18-e cartridge.

The cartridge was washed with 10 ml of bidistilled water. Solvents that passed through the cartridge were discarded. Diamond blue FCF was eluted from the sorbent layer with 3 mL of 2 % (v/v) ammonia solution in methanol. The eluate was collected in a pear-shaped solvent distillation flask with a capacity of 25 ml and evaporated on a rotary evaporator at a water bath temperature not higher than 40 °C to a volume of (0.2–0.3) ml. The remaining solvent was evaporated in air.

The next stage was the preparation of the sample for introduction into the chromatograph. To do this, the dry residue was dissolved in 1 ml of bidistilled water and transferred to a vial with a capacity of 1.5 ml. The final volume of the sample extract is 1 ml.

The third stage was chromatography performing, the conditions of which are: liquid chromatograph with diode array detector; chromatographic steel column 150/4.6 Microsorb 100-5 C18; pre-column chromatographic steel 4/3 Microsorb 100-5 C18; mobile phase – gradient mode in the system of two eluents: eluent A – acetonitrile; eluent B – 0.1 % (by volume) aqueous solution of orthophosphoric acid; volumetric flow rate of the mobile phase – 1.0 ml/min; the wavelength of the diode array detector is 620 nm; the temperature of the column thermostat is 30 °C; injection volume – 10 µl.

The retention time of diamond blue FCF under these conditions is (5.5±0.1) minutes.

At the final stage, the diamond blue FCF peak areas were determined and calculated on the chromatograms. Samples in which the Diamond Blue FCF peak had an area greater than that of the Diamond Blue FCF in the 2.5 µg/mL mass concentration calibration solution were diluted with bidistilled water.

Using the gradient dependence, the results of measurements and calculations of the average value of the area of the chromatographic peaks of diamond blue FCF in the samples, the content (ρ_n), µg/dm^2, of diamond blue FCF was calculated for each of the parallel samples according to the formula:

$$\rho_n = \frac{\overline{S}_n - a}{b} \cdot \frac{V_{пр}}{S},$$

where n is the parallel sample number (n=1, 2);

$\overline{S}_n$ – is the average value of the diamond blue FCF peak area, c.u.;

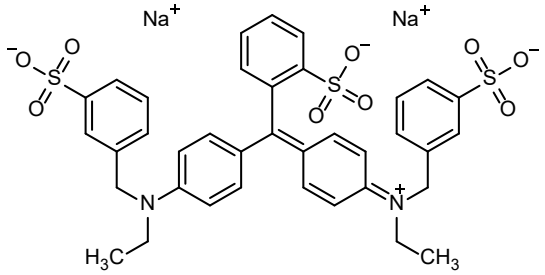
a, b – regression coefficients;

V_{pr} – the final volume of the sample extract, ml;

S – the sample area of the sorption material (filter paper), dm^2.

The calculation result is approximated to the second significant digit.

Table – **Physico-chemical characteristics of diamond blue FCF**

Characteristics	Value for diamond blue FCF
Chemical name (IUPAC)	disodium 2-[{4-[ethyl(3-sulfonatobenzyl)amino]phenyl} {4-[ethyl(3-sulfonatobenzyl)iminio]cyclohexa-2,5-dien-1-ylidene}methyl] benzenesulfonate
Structure formula	
Empiric formula	$C_{37}H_{34}Na_2N_2O_9S_3$
Molecular weight	792.9
Physical state, color	blue crystalline powder
Melting temperature, °C	283 (with decomposition)
Partition coefficient n-octanol/water	log P=-4,9 (estimated)
Water solubility, g/l	30
Solubility in organic solvents, g/l	ethanol – 3

The stages of the methodology for determining the content of diamond blue FCF in the sorption material (filter paper) by the method of high-performance liquid chromatography are shown in Fig.

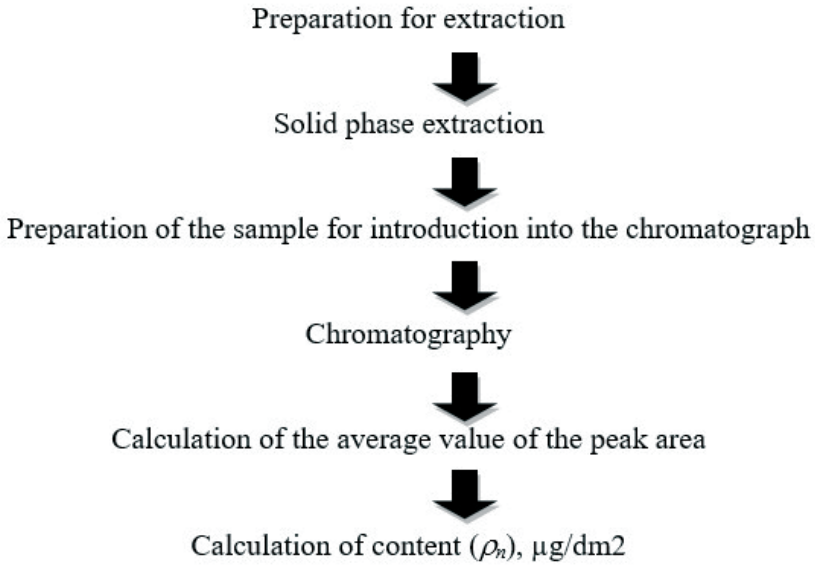


Fig. Method for determining the content of diamond blue FCF in the sorption material (filter paper).

CONCLUSION. Therefore, the optimal conditions for the analytical determination of diamond blue FCF in the sorption material (filter paper) have been established.

The developed method allows measuring the content of diamond blue FCF in the sorption material (filter paper) in the range from 0.125 to 1.25 µg/dm^2 by the method of reversed-phase high-performance liquid chromatography (HPLC) with spectrophotometric (SP) detection.

The proposed method of containing diamond blue FCF in the sorption material (filter paper) will allow to evaluate the effectiveness and safety of the use of various models of UAVs in combination with various pesticide preparations when using different agrotechnical characteristics at the stage of pre-registration trials and scientific research.

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ІНСТИТУТ ГІГІЄНИ ТА ЕКОЛОГІЇ НАЦІОНАЛЬНОГО МЕДИЧНОГО УНІВЕРСИТЕТУ ІМЕНІ О. О. БОГОМОЛЬЦЯ, КИЇВ

РОЗРОБКА СПОСОБУ ВИЗНАЧЕННЯ ВМІСТУ СИНТЕТИЧНОГО БАРВНИКА ДІАМАНТОВОГО СИНЬОГО FCF У СОРБЦІЙНОМУ МАТЕРІАЛІ МЕТОДОМ ВИСОКОЕФЕКТИВНОЇ РІДИННОЇ ХРОМАТОГРАФІЇ

Резюме

Вступ. *На сьогодні використання в Україні безпілотних літальних апаратів для обробки земель сільськогосподарського призначення – нова перспективна технологія, що потребує детального вивчення і розробки підходів до оцінки ризиків та гігієнічного нормування. Актуальною є розробка методу, який дозволить визначати вміст діамантового синього FCF у сорбційному матеріалі (фільтрувальному папері).*

Мета дослідження *– розробити спосіб визначення вмісту синтетичного барвника діамантового синього FCF у сорбційному матеріалі методом високоефективної рідинної хроматографії.*

Методи дослідження. *Використовували такі матеріали: лабораторні аналітичні ваги Radwag® AS220.R2, роторний випаровувач, картридж Strata™ C18-e (55 мкм, 70 °C, 500 мг/6 мл), сталеву хроматографічну колонку 150/4.6 Microsorb 100-5 C18 (фірма "Феноменекс"), передколонку хроматографічну 4/3 Microsorb 100-5 C18, рідинний хроматограф "Shimadzu" з діодноматричним детектором, діамантовий синій FCF, стандарт, фільтри паперові знезолені "червона стрічка" (87,7 %).*

Результати й обговорення. *На першому етапі досліджень відібрали та підготували проби. Для аналізу відібрали дві паралельні проби. Наступним етапом була підготовка проб до введення у хроматограф. На третьому етапі проводили хроматографію. На завершальному етапі визначали площу піків діамантового синього FCF та розраховували на хроматограмах. Зазначений спосіб визначення вмісту діамантового синього FCF у сорбційному матеріалі (фільтрувальному папері) включав екстракцію барвника з фільтрувального паперу дистильованою водою, твердофазну екстракцію та кількісне визначення діамантового синього FCF за допомогою високоефективної рідинної хроматографії з оберненою фазою з використанням СФ-детектування. Цей спосіб відрізняється від відомих тим, що дає змогу визначати досліджуваний барвник у фільтрувальному папері.*

Висновок. *Запропонована методика визначення діамантового синього FCF у сорбційному матеріалі (фільтрувальному папері) дозволить оцінити ефективність і безпеку використання різних моделей безпілотних літальних апаратів у комплексі з різними пестицидними препаратами при застосуванні різних агротехнічних показників на етапі передреєстраційних випробувань та наукових досліджень.*

КЛЮЧОВІ СЛОВА: **діамантовий синій FCF; сорбційний матеріал; фільтрувальний папір; високоефективна рідинна хроматографія.**

Received 12.01.23

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