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ROLE OF THE THIN-LAYER CHROMATOGRAPHY METHOD IN THE CRIMINALISTICS RESEARCH ON IGNITION MIXTURES ON THE BASIS OF PETROLEUM PRODUCTS

The appearance and quality of the material evidence that comes to the study, often also not conducive to the use of complex methods of analysis. There are no separate techniques for forensic investigation of incendiary mixtures, and existing approaches to the study of petroleum products and fuels and lubricants do not allow the tasks set before the experts to be fully addressed.

To determine the applicability of the thin layer chromatography method and to interpret its results for the examination of incendiary mixtures, forensic modeling of incendiary incendiary incendiary mixtures was conducted. The study revealed the possibilities and limitations of thin layer chromatography in the analysis of complex compositions, including those containing polymeric thickeners. The results were interpreted for use in forensic practice. The study was performed by TLC in an octane solvent system: benzene with the detection of colored zones in UV rays, with the manifestation of iodine vapors and the processing of chromatograms of 2% formaldehyde in sulfuric acid. The results of thin-layer chromatography characterize the main constituent components and can be used in the primary study of petroleum-based ignition mixtures in expert practice. The use of UV detection of colored zones on a chromatographic plate allows to assess in more detail the nature and composition of the mixture under study, including to identify distillate and residual oil products.

Thin-layer chromatography can be recommended as a mandatory method of investigation for the ignition mixtures, which precedes the use of more complex Physics and Chemistry spectral and chromatographic approaches.

Keywords: oil products, combustion mixtures, multicomponent systems, component composition, thin-layer chromatography, forensic researches.

Formulation of Research Problem. Results of forensic investigations are important in establishing the evidence base in judicial investigations. Nowadays, there is no doubt that crime can be tackled on a scientific basis using a rich arsenal of scientific methods and technical means. Also, to ensure the effectiveness of this process, the areas of research need to be continually expanded and transformed, given the challenges of today.

Given that there is a continuing threat to personal safety, the number of terrorist attacks using explosive devices containing ignition mixtures is increasing, and the number of expert researches focusing on various based on Petroleum products ignition mixtures has increased. At the same time, available information on the use and preparation of which has not lost its relevance for decades[1].

The incoming combustion mixtures can be divided into three main groups:

– inflammatory mixtures based on Petroleum products;

– metal-containing ignition mixtures based on Petroleum products;

– termite ignition mixtures based on aluminum powder and iron scale.

Petroleum-based ignition mixtures are divided into liquid (non-thickened) and binder (thickened). The former are gasoline blends with heavy motor fuels of all types, diesel fuels and lubricants. In their preparation, the starting compounds may be in different ratios. The preparation of these mixtures does not require special qualification from the performer and the user. Thickened mixtures are gelatinous compounds that contain liquid fuels and thickeners. The light components of the mixtures improve its combustibility, the heavy ones increase the jet emission range and the duration of combustion. Special thickeners, such as polystyrene or polybutadiene are used to make viscous ignition mixtures.

The use of a large amount of polystyrene in the mixture significantly improves the retention of the combustible liquid on a variety of surfaces, even wet. Current techniques for the study of petroleum products and fuels and lubricants do not provide a separate scheme for the study of ignition mixtures.

This leads to the urgency of establishing the design scheme for the study of such mixtures using existing methods.

Analysis of recent research and publications. In foreign literature, the bulk of research on source, unmodified objects are devoted not to individual combustible mixtures, but to commercial fuels with

[1] Nastavlenie po strelkovomu delu [instruction on the shooting case]. Ruchnye oskolochnye i protivotankovye granaty i zazhigatel'nye butylki. Avtory neizv. Moskva : 1946, 94 s. URL: https://armyman.info/books/id–10775.html (data zvernennja 01.05.2019) [in Russian].

impurities of other types of oil, such as in papers[1],[2] and a review article[3].

This can be explained by the fact that the list of methods of investigation of such ignition mixtures is diverse and relates to the conventional methods for forensic research of petroleum products and fuels and lubricants, although the approaches mentioned in these works can also be used in the study of ignition mixtures. However, there are separate, more detailed studies of the latter.

Thus, the paper[4] proposes to use the method of capillary electrophoresis to determine the composition of the inorganic component of ignition mixtures and the paper[5] shows the possibility of using non-destructive Raman spectroscopy to control the composition of probable ignition mixtures.The use of instrumental methods, such as gas-liquid chromatography with different types of detection, in most cases require the preliminary analysis of relatively pure mixed samples or individual *light*»oil products, which causes some difficulties in determining the component composition of real objects. The appearance (Fig. 1) and the quality of packaging of physical evidence that comes to research, often do not contribute to the use of the above methods.

On the other hand, some forensic techniques for the study of inflammatory mixtures in the arsenal of forensic chemists are absent or do not allow to solve the tasks set before the experts. In this regard, the authors of the article have applied the method of thin-layer

[1] *Ferreiro-González M. at al.*Characterization of petroleum-based products in water samples by HS-MS. Fuel. Vol. 222, 2018. P. 506–512; *Babua V., Krishnaa R., Mani N.* Review on the Detection of Adulteration in Fuels through Computational Techniques. Materials Today: Proceedings. Vol. 4, 2017. P. 1723–1729; *Rajneesh K.Verma, Payal Suwalka, JatinYadav*. Detection of adulteration in diesel and petrol by kerosene using SPR based fiber optic technique. Optical Fiber Technology. Vol. 43, 2018. P. 95–100; *Pontes M.J. at al.* Screening analysis to detect adulteration in diesel/biodiesel blends using Near infrared spectrometry and multivariate classification. Talanta. Vol. 85(4), 2011. P. 2159–2165. *Nespeca M. G.* Rapid and sensitive method for detecting adulterants in gasoline using ultra-fast gas chromatography and Partial Least Square Discriminant Analysis. Fuel. Vol. 215, 2018. P. 204–211; *Speller N.C.* QCM virtual multisensor array for fuel discrimination and detection of gasoline adulteration. Fuel. Vol. 199, 2017. P. 38–46.

[2] *Mendes, G., Barbeira, P.J. S.* (2013) Detection and quantification of adulterants in gasoline using distillation curves and multivariate methods. Fuel. Vol. 112, 2013. P. 163–171.

[3] *Vempatapu B. P., Kanaujia P. K.* (2017) Monitoring petroleum fuel adulteration: A review of analytical methods. Trends in Analytical Chemistry. Vol. 92, 2017. P. 1–*11*.

[4] *Martín-Alberca, C., Ferrando, J. L., García-Ruiz, C.* (2013) Anionic markers for the forensic identification of Chemical ignition Molotov Cocktail composition. Science and Justice. Vol. 53, №o. 1, 2013. P. 49–54.

[5] *Martín-Alberca, C., López-López, M., García-Ruiz, C.* (2015) Analysis of pre-ignited improvised incendiary devices using portable Raman. Talanta. Vol. 144, 2015. P. 612–618.

chromatography to the study of the chemical composition of some components of *inflammatory mixtures*.

At the same time, the known possibilities of this method for the study of petroleum products and fuel and lubricants were taken into account[12].

Fig 1. Example of the appearance of combustible mixtures provided for research

Article Purpose.

The purpose of this work is to determine the suitability of thin layer chromatography for the study of combustion mixtures based on petroleum products and fuel and lubricants.

Research tasks include:

– Fixing chromatographic characteristics of the content of the components of the ignition mixture;

– detection of composition features by thin layer chromatography, which give reason to use other methods of investigation (in particular, IR spectroscopy).

Experimental part.

Objects and research methods. For the experiment, the following flammable mixtures models were made based on petroleum products:

1. Gasoline + diesel + fuel oil (10ml / 10ml / 2g).
2. Gasoline + diesel + synthetic oil (10ml / 10ml / 5ml).
3. Gasoline + Diesel + Spent Oil (10ml / 10ml / 5ml).
4. Petrol + fuel oil (10ml / 2g).
5. Petrol + spent oil (10ml / 5ml).
6. Gasoline + synthetic oil (10ml / 5ml).
7. Gasoline + diesel + mineral oil + fuel oil (10ml / 10ml / 5ml / 2g).
8. Gasoline + diesel + synthetic oil + fuel oil (10ml / 10ml / 5ml).
9. Gasoline + kerosene + spent oil (10ml / 10ml / 5ml).
10. Gasoline + diesel + paraffin (10ml / 10ml / 1.5g).
11. Petrol + polystyrene (10 ml / 2g).
12. Gasoline + diesel + polystyrene (10ml / 10ml / 2g).

Methodology and obtained results The research was performed by thin layer chromatography using Sorbfil chromatographic plates.

[1] Zolotarevskaya I. A. Vkazana pratsya.

[2] Dhole V. R., Ghosal G. K. J. (1994) Rapid detection and characterization of petroleum products and residues by TLC: Part 1 / V. R. Dhole, G. K. J. Ghosal // Planar Chromatogr. Mod. TLC. 1994. 7 (6). 469–471 p.

Samples of mixtures in the amount of ~ 2-0 μl without additional dissolution were applied to the starting line of two chromatographic plates.

As the mobile phase used a mixture of solvents octane: benzene (5: 1).

After raising the eluent front by 10 cm, the plates were removed from the chromatographic chamber and dried in the open air, examined in UV rays of mercury-quartz lamp at two wavelengths, showed iodine vapors and 2% formalin solution in concentrated sulfuric acid (CSA).

The results are shown in Tables 1-4 and are presented in Figure 2-9.

Table # 1.

The nature of the chromatographic separation of samples of mixtures of NP in the case of vapor iodine

Mixture No	Chromatographic picture	Value: R_f
1	The strip is yellow	R_f =0, 90
2	Elongated saturated yellow strip. Spot is brown-green in color, the top is bifurcated.	R_f=0, 60–0, 90 R_f=0, 10–0, 15
3	The spot is yellow-brown.	R_f =0, 65–0, 80
4	Elongated strip with alternating colors: yellow, light brown	R_f =0, 15–0, 90
5	Spot light brown	R_f =0, 80–0, 90
6	The strip is yellow-brown in color. Spot is brown-green in color, the top is bifurcated.	R_f =0, 75–0, 80 R_f = 0, 11
7	Elongated strip with alternating colors: yellow, light brown	to R_f= 0, 90
8	Spot light brown. Spot is brown-green in color, the top is bifurcated.	R_f= 0, 70 -0, 90 R_f = 0, 11
9	The spot is light brown in color	R_f= 0, 50–0, 85
10	Spot light brown	R_f= 0, 70 -0, 90
11	Spot light brown	R_f= 0, 50–0, 85
12	Spot light brown	R_f= 0, 80–0, 90

Table # 2.

The nature of the chromatographic separation of samples of mixtures of NP in UV rays at λ = 265 nm

Mixture No	Chromatographic picture	Value: R_f
1	The luminescent strip is purple, the top is bifurcated. Stained dark purple.	R_f=0, 10–0, 70 From start to Rf = 15
2	The luminescent strip is purple, the top is bifurcated. Spot purple color Rf = 0,55-0,85	R_f=0, 12–0, 60 R_f=0, 12
3	The luminescent strip is purple, the top is bifurcated	R_f=0, 10–0, 60
4	Luminescent strip of lilac color. The strip is dark purple in color.	From start to R_f=0, 80 From start to R_f=0, 15
5	Fuzzy (blurred) spot of lilac color	R_f=0, 55–0, 85
6	The luminescent strip is purple, the top is bifurcated. Spot purple color Rf = 0,55-0,85	R_f=0, 55–0, 85 R_f =0, 11
7	Luminescent strip of lilac color. Dark purple spot	R_f=0, 12–0, 85 R_f =0, 12
8	The luminescent strip is purple, the top is bifurcated. Spot purple color Rf = 0,55-0,85	R_f=0, 55–0, 85 R_f = 0, 11
9	Luminescent fuzzy lilac strip	R_f=0, 50–0, 75
10	Luminescent fuzzy lilac strip. The top is forked.	R_f=0, 70–0, 75
11	Any stains are missing	–
12	Llac spot. The top is forked.	R_f=0, 65–0, 67

Table 3.

The nature of the chromatographic separation of mixtures of NPs when UV rays at λ = 365nm

№ Mixtures	The color of the spots on the starting line	Color features of spots with R_f=0, 1–0, 2	R_f	Color, features of spots and zones
1	Dark	Up to 0, 90 Fluorescent strip with alternating colors: yellow-green, blue, top bifurcated		
2	Luminescent, blue	Dark	0, 45–0, 75	Luminescent stain of blue color, yard shape
3	Luminescent, light yellow	–	0, 45–0, 75	Luminescent stain of blue color, yard shape

4	Dark	Up to 0, 85 Fluorescent strip with alternating colors: yellow-green, blue		
5	Luminescent, light yellow	–	0, 55–0, 85	Slightly luminescent stain blue-violet colors
6	Luminescent, blue	Dark	0, 55–0, 85	Slightly luminescent stain blue-violet colors
7	Dark	Up to 0, 80 Fluorescent strip with alternating colors: yellow-green, blue, top bifurcated		
8	Luminescent, yellow	Dark	0, 10–0, 80	Up to Fluorescent strip with alternating colors: yellow-green, blue, top bifurcated
9	Luminescent, light yellow	–	0, 80–0, 90	Not clear slightly luminescent stain blue-violet colors, forked
10	Luminescent, light yellow	–	0, 50–0, 60 0, 75–0, 85	Not clear, slightly luminescent, yellowish, arcuate spot Not clear, slightly luminescent, yellowish, arcuate spot
11	Luminescent, light yellow	–	0, 50–0, 60 0, 75–0, 85	Not clear, slightly luminescent, yellowish, arcuate spot Not clear, slightly luminescent, yellowish, arcuate spot
12	Luminescent, light yellow	–	0, 50–0, 60 0, 75–0, 85	Not clear, slightly luminescent, yellowish, arcuate spot Not clear, slightly luminescent, yellowish, arcuate spot

Note: Luminesc. means luminescent.

As a result of the study of samples of mixtures of NP based on it is established that the obtained chromatograms show clear spots of the corresponding substances contained in the composition of the mixtures, namely:

– diesel fuel – the presence of bifurcated limb spots or elongated spots in the form of «torches» in the UV rays at λ = *254 nm* and *365 nm*) in mixtures containing it (№№. 1-3, 7, 8, 10);

— black oil – spots of dark purple color with corresponding R_f=0, 1–0, 15 at λ = 254 nm and elongated spot in the form of *torch* almost from

the start to the region with R_f = 0,80 in the UV rays at λ = *254 nm* and *365 nm* in mixtures containing it (№№. 1, 4, 7, 8);

– synthetic oil (synthetic component); spots of brown color when iodine vapors with corresponding R_f=0, 1–0, 2, containing (№ 2, 6, 8);

– polystyrene; the absence of any spots in the UV rays at λ = *254 nm* on the chromatogram of the mixture containing it (№. 11).

It should be noted that the values of R_f for certain types of petroleum products may depend on the number and variety of components of the mixtures. The brightness of the color and the clarity of the separation of the zones depend on the concentration of the petroleum product in the mixture and the amount of sample mixture applied to the chromatographic plate.

Table №4.

The nature of the chromatographic separation of samples of mixtures of NPs in the manifestation of stains 2% solution of FGC

Sample number	R_f	Color	R_f	Color	R_f	Color
1	0, 10–0, 20 0, 35–0, 45	Light brown Brown	0, 55	Brown-green	0, 90	Brown-pink
2	0, 10–0, 20 0, 35–0, 45	Light brown Brown	0, 40–0, 60	Brown-green	0, 60–0, 80	Brown-pink
3	0, 10–0, 20 0, 35–0, 45	Light brown Brown	0, 45–0, 55	Brown-green	0, 55–075	Brown-pink
4	0, 10–0, 40	Brown strip	0, 60–0, 75	Brown-green	0, 80–0, 90	Brown-pink
5	0, 10–0, 40	Brown strip	0, 50–0, 75	Brown-green	0, 75–0, 85	Brown-pink
6	0, 10–0, 20	Brown	0, 6–0, 7	Blue-green	0, 8–0, 9	Dark red
7	0, 10 0, 35–0, 40	Brown Brown	0, 40–0, 50 0, 50–0, 65	Blue-green Blue	0, 80–0, 90	Brown-pink
8	0, 10 0, 25–0, 35	Brown Brown	0, 40–0, 50 0, 50–0, 65	Blue-green Blue	0, 80–0, 90	Brown-pink

9	0, 10–0, 30 0, 30–0, 40	– Brown	0, 40– 0, 60	Blue and black	0, 65– 0, 90	Brown-pink
10	0, 10–0, 30 0, 30–0, 40	– Brown	0, 50– 0, 60 0, 60– 0, 75	Brown-blue Blue	0, 80– 0, 90	Brown-pink
11	0, 10–0, 30 0, 35–0, 50	– Brown	0, 50– 0, 60	Brown-blue	0, 70– 0, 80	Brown-pink
12	0, 10–0, 30 0, 35–0, 45	– Brown	0, 50– 0, 70	Blue-green	0, 80– 0, 90	Brown-pink

Comparing the obtained pictures of the chromatographic separation of mixtures when considered in UV rays at different wavelengths λ = *254 nm* and *365 nm*, it should be noted that the picture that is obtained at λ = *254 nm* is more informative and preferable. The appearance of the chromatographic picture in UV rays at λ = *254 nm* is shown in Fig. 2, 3.

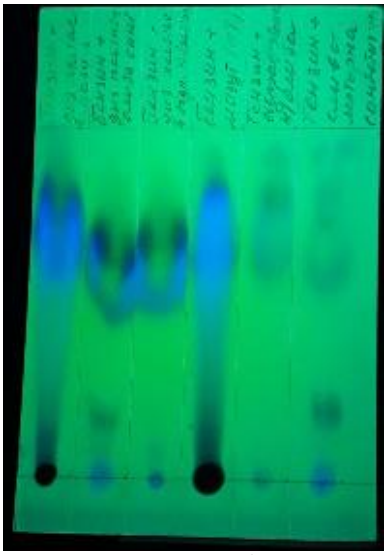

Fig. 2 Chromatogram of mixtures № 1-6 (from left to right) in UV rays at λ = 254 nm.

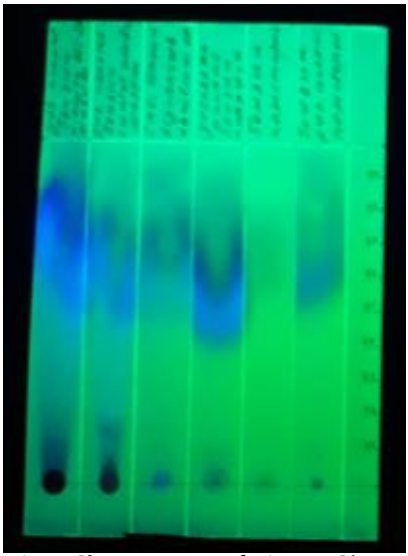

Fig. 3 Chromatogram of mixtures № 1-7 (from left to right) in UV rays at λ = 254 nm

The nature of the chromatographic separation of mixtures when UV is used (λ = 365 nm) is shown in Fig. 4, 5.

Fig. 4 Chromatogram of mixtures № 1-6 (from left to right) in UV rays at λ = 365 nm.

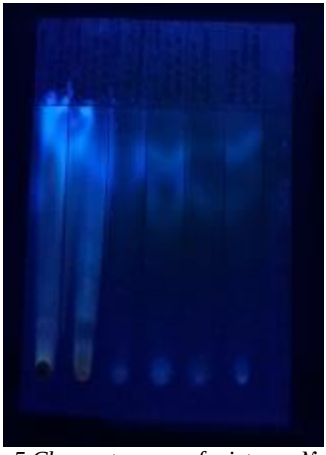

Fig. 5 Chromatogram of mixtures № 1-7 (from left to right) in UV rays at λ = 365 nm

At the manifestation of the plates with a 2 % solution of FGC, there was an alternation of colored spots: light brown, brown, blue-green and brown-pink, which corresponds to the presence of a complex of hydrocarbons of paraffin, naphthenic, aromatic series and hybrid hydrocarbons of different structure (Fig. 8-9) .

The nature of the chromatographic separation of mixtures with the manifestation of iodine vapors and 2 % FGC solution is shown in Fig. 6, 7 and 8, 9, respectively.

Fig. 6 Pattern of iodine vapor manifestation of chromatogram of mixtures №1-6 (from left to right)

Fig. 7 Pattern of iodine vapor manifestation of chromatogram of mixtures №1-12 (from left to right)

Fig. 8 Picture of 2 % solution of FSK chromatogram of mixtures №1-6 (from left to right)

Fig. 9 Picture of the manifestation of 2 % solution of FSK chromatogram of mixtures № 7-12 (from left to right)

Analyzed the nature of the chromatographic separation of the studied mixtures, it is possible to pre-determine the probable groups of inflammatory mixtures, such as:

– with residual petroleum product;
– with synthetic oil (component);
– with probably a polymer component (polystyrene).

The IR spectroscopy method was used to confirm the presence of polystyrene in mixture № 11. The absorption spectra were recorded using a *NICOLET – 380* spectrometers manufactured by *Thermo Electron Corporation*, USA, at room temperature, and the full reflection method was excited. Software – EZOmnic v. 7.4. IR spectra were recorded in the wavenumber range from 650 to 4000 cm^{-1}.

The parameters of the analysis:

– measurement area: 650–4000 cm^{-1};
– resolution: 4;
– recording speed: 0, 6329;
– strengthening: 4;
– number of scans: 32.

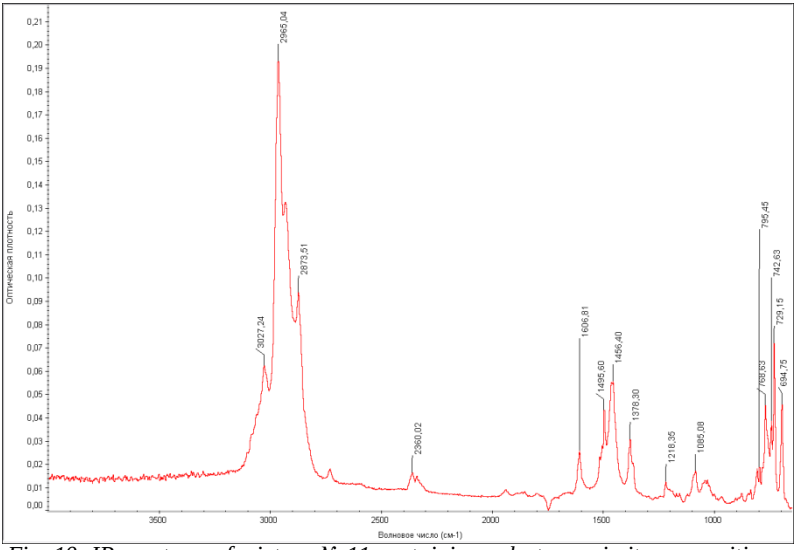

Fig. 10: IR spectrum of mixture № 11 containing polystyrene in its composition

The IR spectra of mixture № 11 (with polystyrene) contain the characteristic vibrational bands in the wavenumber range υ = 3000-2800 cm^{-1}, which are characterized by the valence vibrations of the metal and methylene groups, and the characteristic vibrational bands with the wavenumber values 500, 1460, 750- 700 cm^{-1}, which coincide with the spectrum of polystyrene from the database of applied software in terms of location, quantity and relative intensity (Fig. 10).

It should be noted that use of the band at about 1460 cm^{-1} when installing polystyrene should only be in combination with other bands, since in the spectrum of most petroleum products in the range 1470-1440 cm^{-1} there is also a double absorption band (*with arm*), characteristic of asymmetric absorption bands of methyl absorption and symmetric absorption bands of methylene groups.

Conclusions. Studies have shown that thin-layer chromatography can be useful in the initial investigation of NP mixtures in expert practice and provides general information on the main constituents of combustion mixtures if they relate to different types of petroleum products. Further research should be carried out using methods that include more detailed information on individual components, such as IR spectroscopy. In the study, it is recommended to use the conventional scheme of thin-layer chromatography in the octane: benzene system with a volume ratio of 5: 1.

Processing of chromatographic plates includes:

– UV scan that is desirable to be carried out consistently at two wavelengths of *365 and 254 nm.*

This review allows to differentiate the distillate and residual nature oil in the composition of the mixture, as well as to conclude on their possible compatible presence;

In the presence of a high proportion of unsaturated compounds, the products of thermal refining processes in the object on the chromatographic plate, iodine absorption is observed at their locations with the appearance of white spots at the locations of saturated hydrocarbon structures.

In addition, steam treatment of iodine makes it possible to determine the presence of oils with a synthetic component (synthetic and semi-synthetic motor oils), which may be contained in combustible mixtures, for example, in the form of waste oil;

– treatment with a 2 % solution of FGC, which provides an additional possibility of differentiation of petroleum products in the composition of the ignition mixtures due to the transition of the colors observed in this case, and their localization (R_f).

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РОЛЬ МЕТОДУ ТОНКОШАРОВОЇ ХРОМАТОГРАФІЇ ПРИ КРИМІНАЛІСТИЧНОМУ ДОСЛІДЖЕННІ ЗАПАЛЮВАЛЬНИХ СУМІШЕЙ НА ОСНОВІ НАФТОПРОДУКТІВ

У статті наведені можливості методу тонкошарової хроматографії при дослідженні запалювальних сумішей на основі нафтопродуктів, у тому числі із вмістом полімерних згущувачів, та інтерпретація отриманих результатів для використання в криміналістичній практиці. Дослідження проводилося методом ТШХ в системі розчинників октан: бензол з детектуванням забарвлених зон в УФ-променях, при проявленні парами йоду та оброблення 2% розчином формальдегіду в сірчаній кислоті. Отримані результати дають інформацію щодо основних компонентів та можуть застосовуватися при первинному дослідженні запалювальних сумішей.

Ключові слова: нафтопродукти, запалювальні суміші, багатокомпонентні системи, компонентний склад, тонкошарова хроматографія, експертні дослідження.

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