Chemical research yesterday and today - selected aspects

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Forensics is an interdisciplinary field of science whose primary aim is to utilise all methods and techniques that can be used to detect and prove crimes for the purposes of criminal proceedings. From the very beginning, its development has been closely linked to advances in the natural and technical sciences, as well as to the growing demands of law enforcement authorities for the effective identification of perpetrators and the reconstruction of events based on traces preserved at the scene. The continuous and dynamic nature of developments in this field stems from the ongoing refinement of examination methods and techniques, the implementation of new technologies, and the integration of knowledge from a wide range of scientific disciplines.

The origins of forensics, dating back to the turn of the 19th and 20th centuries, were based primarily on advances in forensic medicine and the first attempts to apply scientific methods to the process of crime detection. That period marked the formation of the fundamental branches of forensics, such as dactyloscopy, anthropometry and custody photography, which played a significant role in identification of persons and documentation and analysis of traces.

In the early stages of forensics, the methods used based mainly on the investigators' observations and experience, as well as on relatively simple measurement and recording techniques. However, as the natural and technical sciences advanced, the range of examination tools used gradually expanded. An increasingly important role started to be played by analytical techniques that enabled examination of minute, often microscopic, traces, providing a wealth of information that is extremely valuable and crucial to the evidential process

Over the course of the following decades, forensics underwent a profound transformation – from methods based primarily on expert observation and experience to highly specialised analytical procedures utilising advanced testing equipment. This is perfectly illustrated by the example of chemical examination. Modern techniques, such as spectrometry, chromatography and electron microscopy, enable extremely precise examination of material structures and identification of chemical and elemental composition. This advancement has significantly enhanced the reliability and evidential value of forensic examinations, contributing to verification of investigative hypotheses, more accurate reconstruction of criminal incidents, thus helping in discovering the truth.

A special role in the area of chemical examination is played by examinations of microtraces and explosives. Microtraces, or traces that are invisible or barely visible to the naked eye, such as particles of metals and metal alloys, glass fragments or individual fibres, constitute evidence that can link the perpetrator to the crime scene or the victim. In turn, analysis of explosives, based on identification methods, makes it possible to determine the type of charge used, its composition and the method of initiation. Both of these areas of examination are very complex and highly sensitive to the quality of the analytical methods used.

The aim of this article is to outline the development of examination methods and techniques used in the analysis of microtraces and explosives – from their historical origins to modern day technological solutions. Special focus was put on changes in the examination approach, increased precision of analyses, and the role of modern equipment.

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Microtraces in forensic examinations in the past and today as demonstrated by the example of metal, metal alloy and glass traces

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Abstract

The article presents the evolution of methods for examining microtraces of metals, their alloys and glass in Polish forensics over the last few decades. It describes the transition from classical, destructive analytical techniques to modern instrumental analysis such as X-ray fluorescence. Modern physicochemical methods enable fast, precise and non-destructive examinations with high sensitivity, as illustrated by examples from expert practice in assessing the authenticity of jewelry products and detecting forgeries.

Keywords: microtaces, metal and their alloys, glass, jewelry products, analysis of elemental composition

When comparing analyses of interesting cases from the 1970s and 1980s presented by experts from the Forensic Department of the then National Police Headquarters (KG MO), with current papers published in “Problemy Kryminalistyki”, one can clearly see differences in the descriptions of the evidence submitted for examination. In the past, those descriptions were very detailed and employed specialist parlance specific to the field to which the subject belonged. This was often due to the fact that those commissioning an expert opinion required comprehensive examinations and requested that the method/technique used for manufacturing a given piece of jewellery or a coin be determined, or that the type of moulds, dyes and tools used be identified. Those opinions could be issued in collaboration with the Assay Offices and experts from the National Museum and the Issue Vault of the National Bank of Poland. Nowadays, these descriptions are more concise, often supplemented by extensive photographic documentation, with a significant portion of the opinion devoted to the description of the specialised instrument methods used for analysing a given trace. The number of methods available now is far greater, and they are more sensitive and accurate, while examinations are non-destructive.

Previously, in addition to a thorough preliminary examination of a coin, its specific gravity and the colour of the metal from which it was made were determined, along with its weight, diameter and thickness. These were then compared with tabulated weights and hallmarks of the most common original gold and silver coins. In many cases, the data enabled preliminary identification of type of metal or alloy from which a counterfeit coin was made. However, if the counterfeit was more complex, the PGS-2 emission spectrograph was usually employed (Fig. 1 and 2) to establish the elemental composition. It was a non-computerised

and time-consuming technique, involving destructive tests, and the sample size required for analysis was in the order of milligrams. It offered an advantage of determining trace elements [1].

Fig. 1. A spectrograph stand with electrodes mounted on it (a UBI-1 generator can be seen in the background)

Fig. 2. A Zeiss PGS-2 spectrograph

The samples were placed in graphite electrodes and burned in a direct current arc. When excited, the atoms of the elements in the substance under examination emitted radiation which, after passing through the cleaving system, produced a spectrum that was recorded on a glass plate coated with photographic emulsion and placed in a special cassette, which was then subjected to photographic processing. This involved removing the plate from the cassette and immersing it first in a tray with developer and next in a tray with fixer, followed by rinsing and drying in a room with no light access, known as a 'darkroom'. The exposed spectral plate was placed in a spectroprojector, where the enlarged image of the plate, projected onto the table, was compared with an atlas of spectral lines [2].

Fig. 3. Zeiss SP 2 spectroprojector (without the outer casing and curtains)

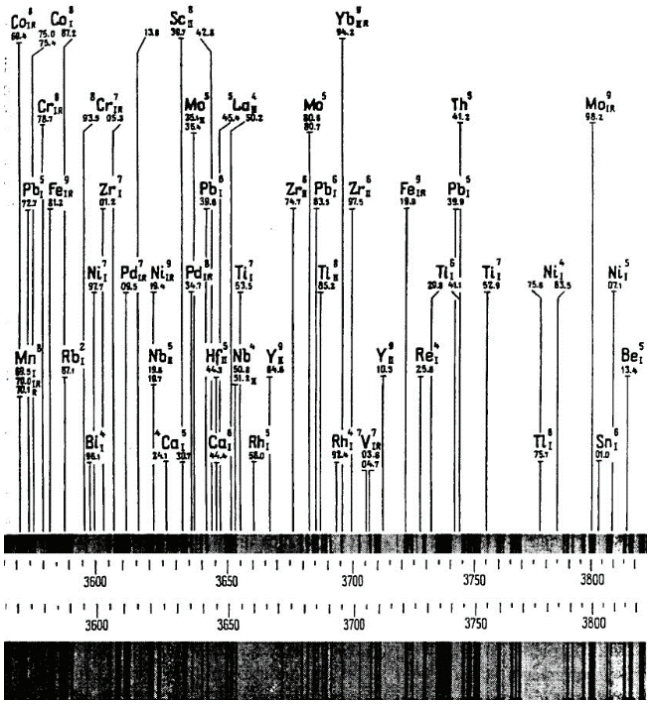

Fig. 4 A section of the spectrum from the spectral line atlas (above the scales) compared with a section of the sample's spectrum (below the scales)

At present, cases are reported that involve bars that resemble investment gold bars produced by various mints around the world, including ARGOR-HERAEUS S.A., PAMP, The Perth Mint Australia, among others. The very accurate finishing of those bars, combined with the requirement to place them in plastic packaging, means that buyers do not suspect them of being counterfeit.

Fig. 5. A bar resembling a PAMP-minted investment gold bar, complete with a certificate

Fig. 6. A bar resembling a PAMP-minted investment gold bar, complete with a certificate

Following a detailed description of the macro- and microscopic examinations of this type of traces (weight, dimensions, description of defects or surface abrasions on the bars found in the previously opened packages with certificates), instrumental examinations of the elemental composition are carried out using the micro-X--ray fluorescence (μXRF ED) method. This is a quick and non-destructive method.

Fig. 7. An EAGLE II μ-X-ray fluorescence spectrometer

Upon examination, the bars are often found to be made of a copper-nickel alloy coated with a thin layer of gold. In other words, their elemental composition does not match the specifications stated on their surfaces and in their certificates, according to which they should be made of fine gold.

Currently, the Chemical Examination Department's latest instrument for elemental composition analysis is the SPECTRO XEPOS XRF spectrometer, which is characterised by high sensitivity, resulting in a threefold improvement in measurement precision. This provides the basis for ensuring accuracy of analyses across a range of element concentrations, from trace levels to high concentrations.

This method is currently used for examining jewellery items secured in connection with fraud cases. Fraud is an offence that involves causing disadvantageous disposal of property to obtain a financial advantage by misleading the other party as to the material from which the jewellery offered for exchange was made.

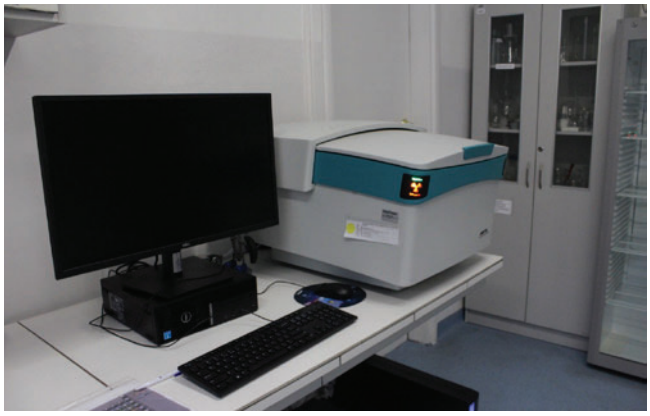

Fig. 8. A XEPOS X-ray fluorescence spectrometer

Based on quantitative examination of the elemental composition of a piece of jewellery, it is possible to estimate its fineness. Previously, this purpose was achieved by means of touchstone testing, which relied on the relationship between the colour of the alloy and its qualitative and quantitative composition.

Fig. 9. A test kit for determining fineness of precious metal products

The examinations involve comparing the intensity of the chemical reactions that occurred when test liquids are applied to the streaks. The analysis was carried out on a touchstone using the test specimen and a reference test needle with the closest matching alloy colour. "Streaks" were made on the clean surface of the touchstone using the object under examination and needles with matching colours with finenesses higher and lower than that suspected for the object under examination. Next, a drop of the test liquid appropriate for the suspected fineness (e.g. for 3-carat gold, this is a solution of gold(III) chloride, 18 g of 0.999 fine gold in the form of gold(III) chloride, water to the volume of 1000 cm^3) was applied to the streaks on the touchstone with a glass rod, and the course of the reaction was observed for 20 seconds. Next, the excess liquid had to be removed and the intensity, colour and quantity of the resulting precipitate compared. If the effect of the liquid on the "streaks" was the same, it indicated that the alloy contained the same proportion of precious metal. If not, the procedure had to be repeated with further needles until the matching effect of the liquid on the "streaks" was obtained [3].

Microscopic examination of such traces plays an important role, particularly in the observation of hallmarks and other symbols stamped on products. These marks are unique to individual manufacturers. There is no doubt that the ongoing development of microscopy and its advanced techniques have an impact on the quality of examinations. Furthermore, the availability of specialist literature online makes it easier to familiarise oneself with the subject of a particular field. In the case in question, the products were found to be made of a copper-zinc alloy, i.e. brass, coated with a thin layer of gold, while according to the hallmarks "585", which had most likely not been applied by the Assay Office, said items should primarily consist of gold, as the "585" hallmark indicates 58% gold content. In addition, the jewellery submitted for testing was found to bear the

stamped symbols “XP”. According to online sources, the XP symbol is the logo of Xuping, a Chinese manufacturer of gold-plated jewellery made from copper and electroplated with a layer of gold.

In examinations of glass traces, it is now possible to examine increasingly smaller glass particles by means of routine use of an electron microscope for qualitative and quantitative analysis of elemental composition using the energy-dispersive X-ray microanalysis (SEM/EDX) method.

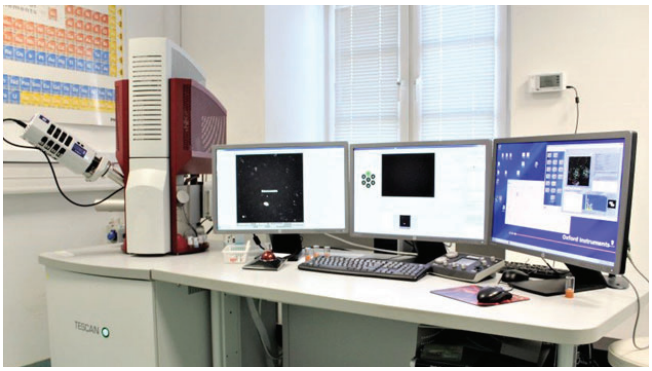

Fig. 10. A scanning electron microscope

The Chemical Examination Department at the Central Forensic Laboratory of the Police (CLKP) currently carries out examinations using an integrated analytical system: a *Tescan Mira 3* LMU scanning electron microscope coupled with an Oxford Instruments SSD-type X-Max^N energy-dispersive X-ray spectrometer with the Aztec EDS detector software package ver. 2.4. This method can be used for examining glass microfragments with a surface area of less than 0.2 mm ^{2}. It is a quick, non-destructive technique that does not require any complicated preparation of the test material. Traces with a similar elemental composition are passed to the next stage of testing, which involves measuring their refractive index. The refractive index (RI) of glass is measured with the thermo-immersion method and the GRIM III system from Foster & Freeman in monochromatic light (590 nm), employing B-series glass standards and type B immersion oil from Locke Scientific. This latest system offers measurements that are accurate to five decimal places, with a standard deviation of 0.00002 over a five-hour period and 0.00003 over a five-day period. The system can analyse up to four edges of a single fragment simultaneously, which significantly improves the speed of measurement and ensures greater statistical accuracy. The test results are archived and password protected along with such details as the case number and the operator’s identity. In addition, basic statistical data on the results is generated, including the mean, range and standard deviation. The software contains two clustering algorithms and two match-test options, offering the examiner the flexibility to choose the approach that is best suited to their laboratory’s quality requirements. The results can be displayed as charts and formatted for export to Microsoft Office for further analysis.

Fig. 11. A refractive index measurement kit GRIM III

In the 1970s, the absolute thermo-immersion method, which had been developed at the Forensic Department of the then National Police Headquarters, was also used for comparative glass analysis. The examinations were carried out using a Boetius microscope fitted with a heating table. The refractive index results were given with the accuracy to four decimal places [4].

A relative thermo-immersion method was also employed with the use of the aforementioned microscope, in which an immersion fluid with the refractive index of $n_D^{25} = 1.5241$ was applied. The temperatures (in degrees Celsius) at which the refractive indices of the immersion fluid and the test samples were equalised were recorded. The test results indicated that the temperatures at which the refractive indices of the reference samples and the test samples became equal differed by less than 2^{o}C (which was the margin of error) or showed no difference at all. The similarity between the traces was confirmed through X-ray fluorescence analysis on a Finnigan QM-905 spectrometer [5].

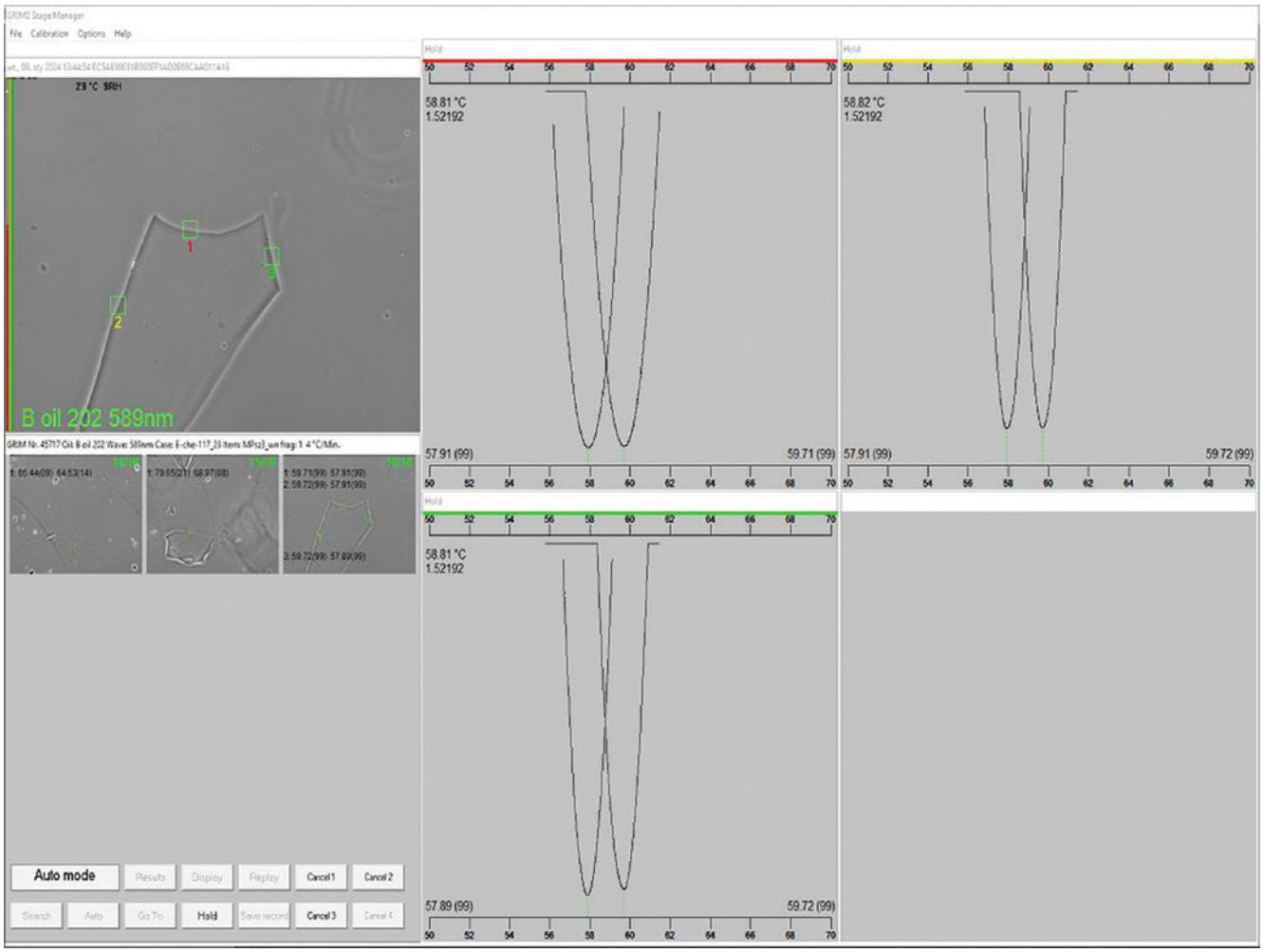

Fig. 12, 13, 14. Results of refractive index measurements carried out using the system GRIM III

Used for comparative examination of glass traces was also the spectral emission analysis method mentioned above, in which a glass sample had to be ground into a powder (approx. 30 mg), mixed in a 1:3 ratio with graphite, and then placed between graphite electrodes. The subsequent course of action was identical to the procedure described earlier. However, it was rare to have enough evidence material to apply that method. Examinations were often abandoned due to an insufficient amount of material.

Given as an example of a highly specialised and comprehensive forensic evidence involving dactyloscopic, physicochemical and mechanoscopic examinations in the 1980s can be the case of the burglary of the Primatial Cathedral Basilica in Gniezno which took place on the night of 19–20 March 1986. In the incident, the coffin of St. Adalbert and the saint's body were destroyed. The reference material was preserved from the fragments remaining on the destroyed coffin. The silver ingots found during the subsequent investigation were also secured. Physicochemical examinations were also carried out on metal filings and particles found in a bag belonging to the perpetrators, as well as on a screw and filings recovered from the coffin, metal particles sampled from the tools, and metal particles found in the suspects' clothing pockets. The elemental composition examination was carried out using X-ray fluorescence and emission spectroscopy. The Assay Office in Warsaw determined the silver content using the touchstone assay method and conventional quantitative analysis. Based on the results, it was established that the elemental composition of the evidence was similar to that of the reference material. Forensic examination of the physical evidence secured in connection with the burglary of the Primatial Cathedral Basilica in Gniezno contributed to the drafting of the indictment and finding the suspects guilty by the court [6].

In summary, it can be said that the methods and instruments used in the forensic examination of microtraces of metals, metal alloys and glass in the past by Polish forensic experts were advanced and modern compared to the methods and instruments in use around the world at that time. The Forensics Department was well-equipped and staffed by the best experts.

Nowadays, thanks to advances in science, instruments offer greater levels of sensitivity, precision and automation, making it possible to examine larger numbers of objects and leading to greater efficiency.

What links the two eras is the human factor. In theory, human involvement is now reduced due to the automation of many processes. In practice, however, the input of an expert who demonstrates perceptiveness, patience and composure was, and still is, essential in forensic examination of traces such as microtraces.

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Microtraces in forensic examinations in the past and today as demonstrated by the example of paint coatings and fibres

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Abstract

The article presents the development of methods for examining microtraces such as paints and fibers, in Polish forensic since 1970s to the present day. It describes the transition from classical optical microscopy, based on visual observation, to advanced structural analysis. The application of digital, comparison and electron microscopy, as well as spectroscopic techniques is discussed. The combination of these methods enables comprehensive physicochemical identification of the collected material, increasing the accuracy and evidential value of mictrotraces analysis and making them an essential component of modern forensic examination.

Keywords: microtraces, paints, fibers, microscopy, spectroscopy

Forensic evidence examination has been practised since at least the late 19th century. A type of forensic evidence, microtraces are defined as par ticles of matter which are invisible or barely visible to the naked eye that the perpetrator leaves at the crime scene. As microtraces cannot be avoided or removed, they are a valuable source of information about an incident, its course and persons involved that can help in apprehending the perpetrator [1, 2]. Chemical microtraces include mainly fibres, paint coat particles, plastic particles and micro-shards of glass, metals and metal alloys. Microtraces became particularly important in forensics with the introduction of new sensitive and non-destructive analytical techniques and the development of the measuring instruments used in examinations.

Microscopic methods are among the oldest non-destructive methods of observing microtraces. The use of microscopic observation of specimens with various techniques made it possible to determine a number of physical parameters based on the microscopic images obtained, albeit in a subjective manner and without the aid of result recording equipment.

However, microscopic methods were used in an objective manner as early as in the 1970s. Among those was the interference-polarisation method, used for determining the refractive index and birefringence in synthetic fibres. The method used the MPI-5 microscope (Fig. 1) as the measuring instrument. A combination of the MB-30 biological microscope and the MPI-3 polarisation-interference device, the instrument opened up a range of examination and measurement possibilities, including for anisotropic bodies.

The MPI-5 microscope consisted of the following key components:

- a slit condenser with a rotating polariser,
- an MPI-3 polarisation-interference device,
- a set of lenses with birefringent prisms,
- an OK 15 KM measuring eyepiece.

During examination, measurements of the optical path difference were taken in an interference fringe field, given the known thickness of the object. The data made it possible to calculate the birefringence value and the refractive index [12].

The method for determining the refractive index and birefringence of synthetic fibres had a number of advantages, including the ease of measurement and the ability to examine minute quantities of material and identify fibres without having to destroy them. These advantages enabled widespread use of that method for comparison and group identification of synthetic fibres [12].

Presented below are examples of cases from the 1970s in which polarisation-interference method was applied to determine the refractive index.

The Forensic Department of the then Voivodship Police Headquarters (KW MO) carried out micro-trace analysis for a 1973 preliminary investigation into a burglary of a flat. The perpetrators entered the living quarters through the roof. Evidence in the form of multicoloured fibres was secured from a plank near the manhole cover on the roof of the building. Fibres from a jumper belonging to the suspect were secured for the investigation as reference material. The authority conducting the criminal proceedings requested determination of: "Do the fibres recovered at the scene of the burglary come from the suspect's jumper?"

Fig. 1. A polarisation-interference microscope

It was established in the course of the examination that the jumper was made of polyacrylonitrile fibres. Detailed examinations of the evidence and reference fibres were carried out using, among others, the polarisation-interference microscopy method, and the refractive index of the fibres was determined. In addition, a microspectrophotometer was used for comparing the dyes used to colour the fibres during the manufacturing process. Based on the examinations carried out, it was found that the green and light brown evidence fibres showed resemblance to the polyacrylonitrile fibres from which the jumper had been made and that they probably came from it [5].

In another case from 1974 concerning a fatal road traffic accident in which a lorry hit two women, the polarisation-interference method was also applied. Evidence material in the form of red and beige fibres, which were found and secured during the inspection of the vehicle, was submitted for examination.

The following question was asked in the order: "Could the red fibres found on the vehicle have come from the deceased's scarf, and the beige fibres from her tights?"

It was established in the course of the examination that the scarf was made from a blend of red wool and polyacrylonitrile fibres, while the tights were made from beige polyamide fibres. It was further established that the red fibres recovered from the vehicle were also a blend of wool and polyacrylonitrile fibres, while the beige fibres were polyamide fibres.

Measurements of the refractive indices of the polyacrylonitrile and polyamide fibres, both evidence and reference ones, carried out using the polarisation-interference method, demonstrated a match. In addition, the dyes used for colouring the fibres were compared. The findings constituted the main evidence in the criminal proceedings and led to a guilty verdict and the conviction of the perpetrator [5].

In recent years, advances in microscopy have expanded the microtrace examination capability, including for fibres and paint coatings, while the design and parameters of microscopes have also evolved. Microscopes were connected to a computer, which made it possible to capture images, save the results and compile them into catalogues for use in comparative examinations.

The Chemical Examination Department of the Central Forensic Laboratory of the Police (CLKP) is currently equipped with microscopes that enable a more detailed analysis of evidence and reference material. In addition to stereomicroscopes using reflection and

transmission techniques, which determine the subsequent examinations, their types and sequence, there are also: comparative microscopes, examination microscopes, digital imaging microscopes and scanning electron microscopes (Fig. 2-5, Fig. 10).

Using an examination or digital microscope, one can observe the morphological details of fibres under various lighting conditions, such as white transmitted light (BF), phase contrast (PH), polarised light (PO) and ultraviolet light (UV). In the case of paint coatings, examinations are carried out in white (BF) and dark (DF) fields of reflected light.

By analysing paint coatings, it is possible to determine shape and type of pigments used and the number of layers, and to measure the thickness of individual layers of the paint coating under examination. In fibre examinations, one can determine the shape and size of the cross-section and analyse the longitudinal view, which, in the case of natural fibres, enables identification of the fibre type.

When conducting examinations under polarised light, it is possible to observe the distribution of crystalline and amorphous areas within the fibres. On this basis, an initial identification can be made of the most common types of fibres, particularly synthetic chemical fibres such as polyamide, polyester or polyacrylonitrile fibres [6].

The CLKP's Chemical Examination Department uses a Keyence VHX-6000 digital microscope. Used for high-resolution observation and analysis of samples, the device combines top-quality optics with digital image processing. It enables detailed observation of very small objects and their morphology. The microscope has a large depth of field, enabling observation of uneven surfaces from any angle. It is fitted with an automatic lens system, providing for smooth switching between magnifications ranging from 20x to 2000x. The device facilitates measurements, fast image capturing and report generation. In addition, the microscope enables 3D imaging [7].

Photographs of the microscopes used by the CLKP's Chemical Examination Department are shown below.

Fig. 2. Stereoscopic microscope

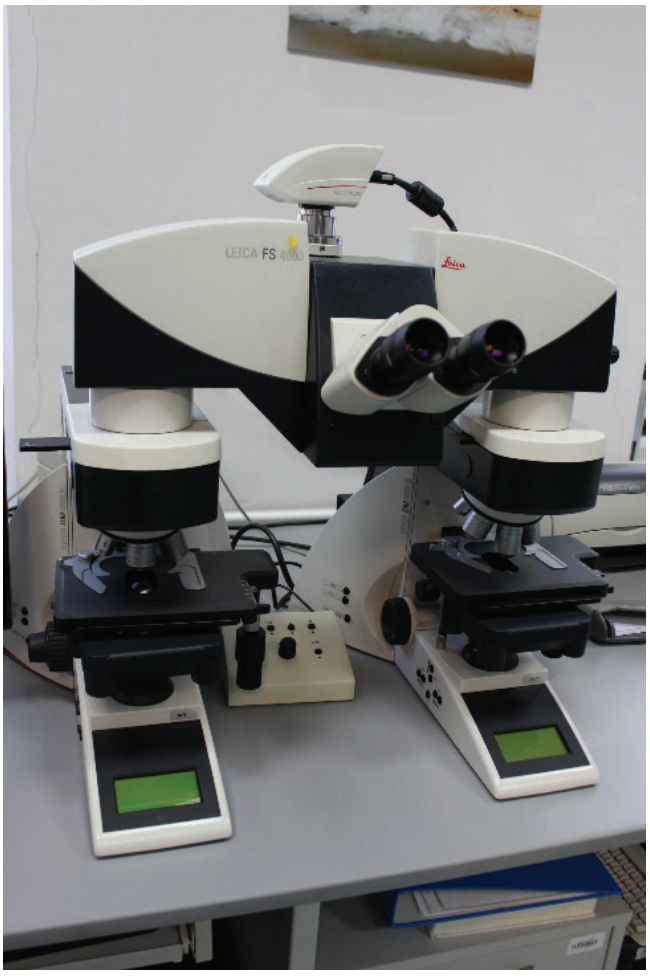


Fig. 3. A comparative microscope

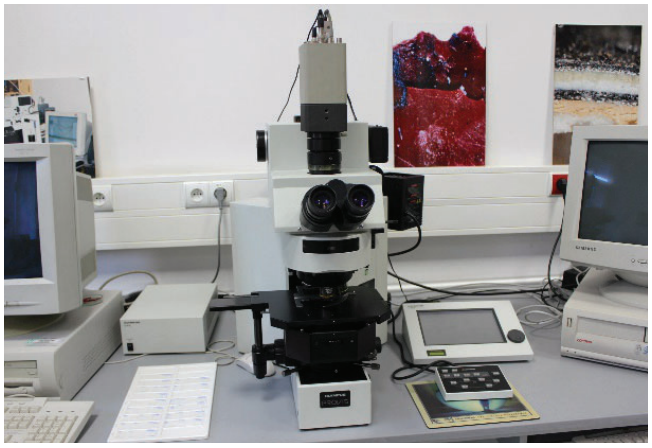


Fig. 4. An examination microscope

Fig. 5. A digital microscope

A microscopic image of bicoloured, colourless-red, synthetic fibres under white light (BF) and ultraviolet light (UV) (Fig. 6, 7), and of a black pearlescent paint coating and its cross-section (Fig. 8, 9) obtained with a digital microscope (Fig. 5)

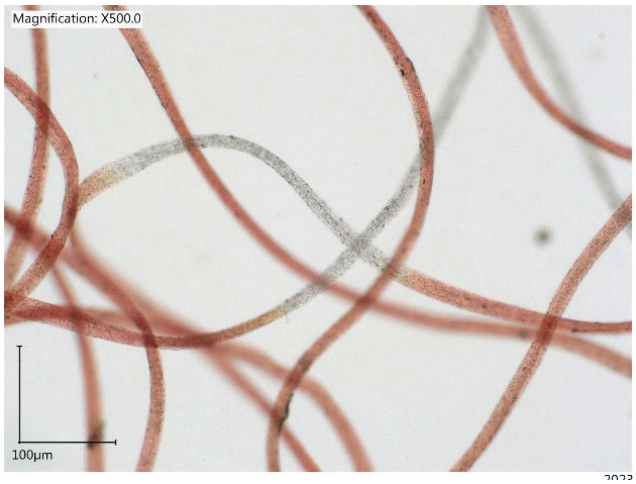


Fig. 6. A microscopic image of fibres under white light (BF)

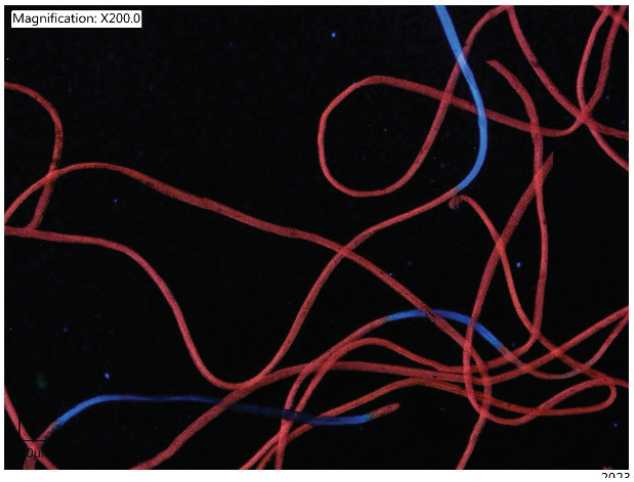
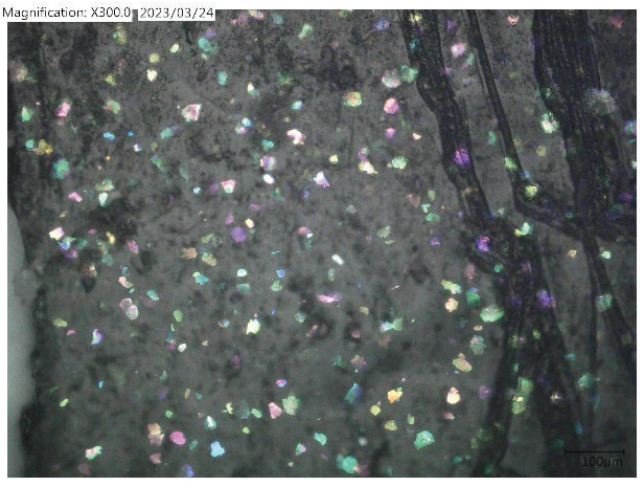


Fig. 7. A microscopic image of fibres under UV light



Ryc. 8. A microscopic image of a black pearlescent paint coating under white light (BF)

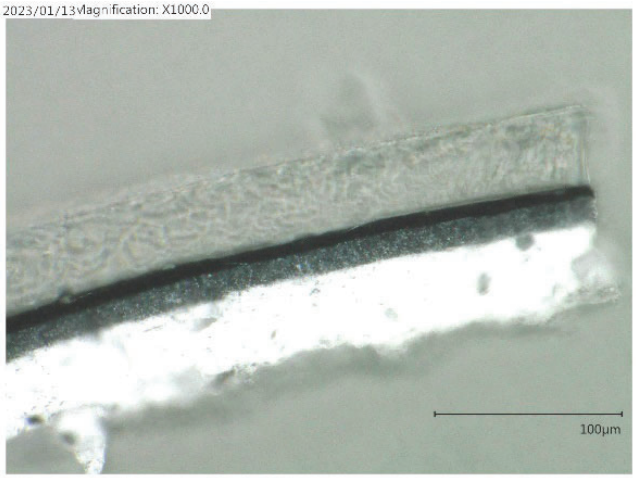


Fig. 9. A microscopic image of a cross-section of a paint coating under white light (BF)

Microtrace imaging is also carried out with a scanning electron microscope coupled with an energy-dispersive X-ray spectrometer (Fig. 10) which enables observation of objects at a magnification of up to tens of thousands of times. It provides for obtaining a large depth of field, and its high resolution facilitates analysis at very high magnifications that cannot be achieved with optical microscopes. Using a scanning electron microscope, one can identify morphological details, areas of degradation and the nature of damage, e.g. charring/melting of fibres, exposure to chemicals, ruptures and cuts that may have been caused by various tools [8, 9]. In the case of paints, it enables detailed observation of the shape and type of pigments used in automotive paint coatings. Using this microscope, one can also determine the elemental composition of microtraces, leading to identification of, among others, inorganic pigments and fillers present in e.g. paint.

A microscopic image of selected fibres and a cross-section of a paint coating (Fig. 11, 12) obtained with a scanning electron microscope (Fig. 10) is shown below.

Fig. 10. An electron microscope

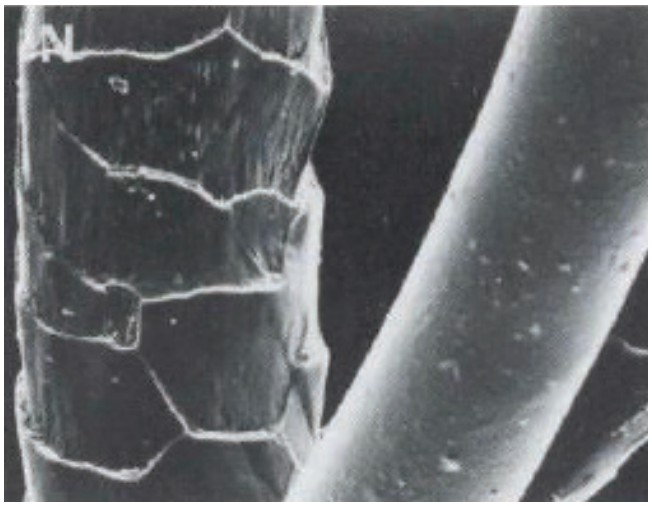


Fig. 11. A microscopic image – a longitudinal view of a wool and polyester fibres

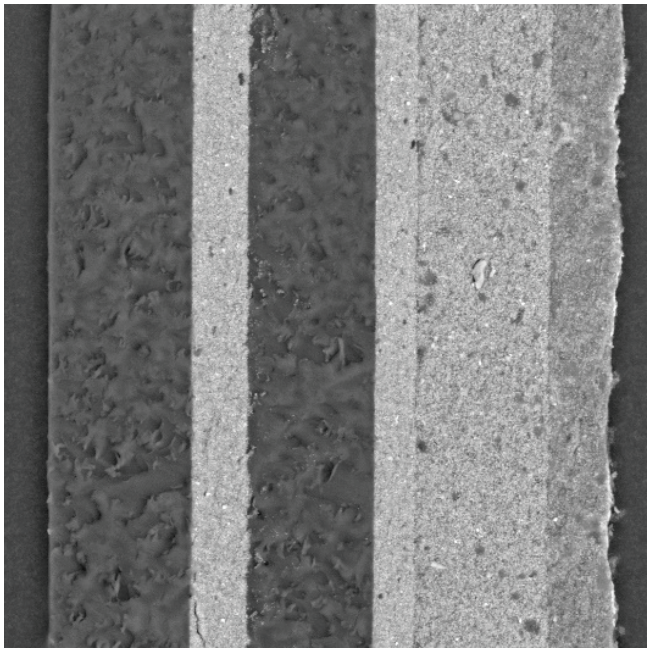


Fig. 12. A microscopic image of a cross-section of a paint coating

Significant progress in examination of paint traces or fibres was made when two or more basic analytical techniques were employed. Microspectrometry, which combines optical microscopy with spectrometry, is currently particularly useful in forensic trace examinations. It provides for comparing colours, morphological structure and chemical composition of minute samples of paint or fibres in a non-destructive manner [1, 3].

Among methods that enable determination of the chemical composition of microtraces is Fourier-transform infrared spectroscopy – FTIR (Fig. 13). It enables identification of the polymer type in fibres and paints with the use of a microscope fitted with a diamond cell (Fig. 14). This tool facilitates measurements and enables analysis and identification of small samples.

Microspectrophotometry is also used in the UV-VIS range for the objective determination of colour. In the 1990s, the CLKP's Chemical Examination Department was equipped with a Leitz MPV-SP (Combi) microspectrophotometer (operating in the 220–780 nm

Fig. 13, 14. Fourier-transform infrared spectroscopy with a diamond cell

range) coupled with a quartz optics examination microscope in a fully automated setup that enables archiving of measurement data. The measurement results are presented in the form of relative and corrected as well as graphical or numerical spectra. The method is used mainly in comparative examinations of forensic traces [10, 11] (Fig. 15). The chemical lab is also equipped with an uSight 2100 series MSP VIS microspectrophotometer with a Nikon Ci Series microscope operating in the visible light range of 380–780 nm (Fig. 16). Microspectrophotometry provides for measuring and comparing colours in even minute samples, such as varnishes, paints, fragments of individual fibres no longer than one millimetre and with a diameter of a few micrometres, and cross-sections of multi-layer paint coatings. It is an analytical method that combines optical microscopy and UV-VIS spectrophotometry. The purpose of the former is to enable observation of an object in transmitted and reflected light. Spectrophotometry, as a measurement method, involves measuring the intensity of radiation transmitted through and/or reflected from the test sample as a function of wavelength [4].

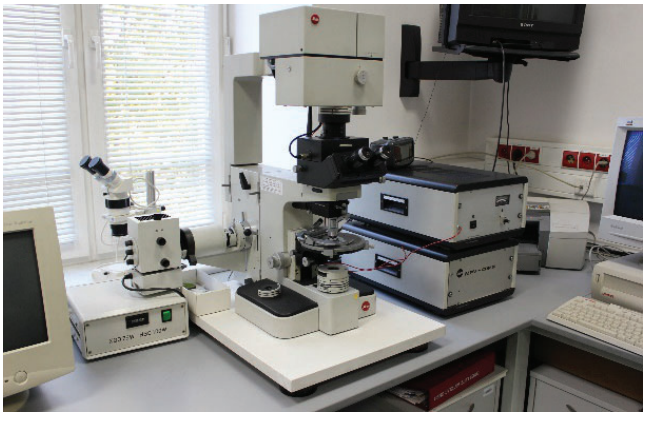


Fig. 15. A UV-VIS microspectrophotometer

Fig. 16. A VIS microspectrophotometer

Over the years, the methodology used in microtrace examination has remained largely unchanged, although there have been significant advances in the equipment used. The miniaturisation of measuring instruments and their connection to computers have provided for automation of examinations. The use of graphics and measurement software has made it possible to record the test results and measure the analysed microtraces.

New methods have expanded examination capabilities, provided information on the structure and composition of the examined trace, enabled faster acquisition of results and provided for detection and identification of minute traces.

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Explosive materials and devices in forensic examinations over the years

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Abstract

This article presents the development of methods for examining explosive materials and explosive devices in Poland from the late 1950s to the present day. It discusses the evolution of post-blast residue identification techniques, from classical chemical reaction to modern instrumental methods such as gas chromatography with mass spectrometry (GC-MS). The article also describes changes in the design of explosive devices, the impact of advances in information technology and legal regulations on forensic practice. Scientific and technological progress has significantly enhanced the effectiveness of forensic investigations of incidents involving explosive materials and the identification of post-blast residues.

Keywords: explosive material, post-blast residue, explosive devices

The origins of forensic examination of explosives in Poland date back to 1959-1960

Earlier on, post-explosion traces – apart from examination of gunshot residues – had been analysed primarily from an ordnance disposal perspective. Initially, though often successfully, aimed at identification of the type of explosive used in an incident. This mainly concerned situations where unconsumed particles of material were detected. The examination methods used at the time did not provide for carrying out chemical analyses at the level of sensitivity required for detection and identification of explosive traces in the debris left after a detonation. Consequently, investigators usually limited themselves to a general identification of the type of explosive, for example, based on the intensity of soot stains in the vicinity of the blast epicentre. Numerous incidents, ranging from accidents involving the handling of wartime unexploded ordnance to bomb attacks, led to increasing attention being paid to issues relating to post-explosion investigations.

At this point, it is worth mentioning Dr. Tadeusz Baran, an expert at the Forensic Department of the National Police Headquarters in the People's Republic of Poland (KG MO), a lecturer at the Academy of Internal Affairs and the author of numerous forensic opinions and specialist publications on the examination of explosives, whose professional achievements have greatly contributed to the development of this field of forensics in our country.

A review of archive expert reports compiled by T. Baran suggests that the underlying motives and modi operandi of perpetrators of crimes involving explosives during the People's Republic of Poland did not differ greatly from those of today: the most common incidents were ones involving reckless handling of found wartime ammunition, experiments with production of home-made explosives, and bomb attacks motivated by personal vendettas or acts of vandalism. Occasionally, however, there were incidents that one would describe today as highly „media-friendly", such as the blowing up of the Higher School of Education in Opole in 1971 with the use of a charge of approximately 70 kg of

TNT, or the tragic attempt by Rudolf Olma to hijack an aeroplane in 1970 with the use of an improvised explosive device hidden in a cake (Photo 1).

Fig. 1. A reconstruction of the explosive device used by Rudolf Olma in 1970

When analysing the design solutions of the explosive devices themselves, one can say that they were similar to the ones made at present. They were characterised by significant diversity, primarily due to the varying levels of knowledge and skill among the makers, ranging from solutions that were practically unusable and blatantly demonstrated engineering incompetence, to simple yet ingenious devices, e.g. ones disguised as weights (Photo 2) or household items, right up to highly sophisticated designs containing electronic timing mechanisms or radio-controlled trigger systems.

The range of explosives examined in the cases at that time was similar to that of today and included: - military-grade materials, such as TNT, hexogen, pentrite and their compounds, acquired through theft or obtained from wartime ammunition, - mining explosives, pyrotechnic mixtures obtained from commonly available products, such as matches or caps for cap guns, - home-made materials.

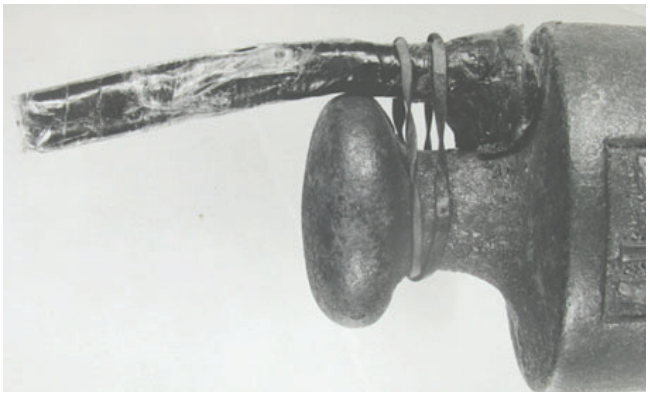

Fig. 2. An improvised grenade made from a 1-kilogram weight (1970)

Dominating in the latter group of materials, just as today, were pyrotechnic mixtures home-made by combining various fuels and oxidisers. Between 1969 and 1972, out of the approximately 200 post-explosion cases compiled by T. Baran, 50% involved a detonation of pyrotechnic products, mainly home-made ones.

Despite many similarities, there are significant differences between forensic examinations of explosives of yesteryear and those of today, which are particularly evident in the field of chemical examination. The chemical analysis methods once used in the past for detection and identification of explosives during laboratory tests included mainly classic analysis based on colour tests and other characteristic reactions, and thin-layer chromatography (TLC). Nowadays, these methods, if used at all, tend to be employed for preliminary or supplementary examinations. In the past, however, they formed the basis of the entire analytical process. Although instrumental techniques, such as gas chromatography and UV-Vis spectrophotometry, were available in forensic laboratories since the 1970s, their sensitivity was insufficient for the purposes of post-explosion trace analysis. Particular attention is drawn to the wide range of TLC and classic analytical techniques used at the time, which are now largely a forgotten art, yet which, although being highly labour-intensive, enabled detection levels that were quite good even by today's standards, in the range of 0.2–0.5 micrograms for most of the explosives encountered at that time.

Nowadays, instrumental methods play an essential role in explosives examination labs, particularly those from the area of coupled methods, which combine separation techniques,-which enable separation of complex chemical mixtures, with spectroscopic techniques, which provide information on the molecular structure of the detected substances. Examples include gas chromatography coupled with mass spectrometry (GC-MS) or capillary electrophoresis with a PDA detector that records spectra in the UV-Vis range. From today's perspective, the analytical achievements of the 1970s no longer seem so impressive: the gas chromatography method with a chemiluminescent detector (GC-TEA) used at CLKP provides for detecting explosives from the nitro compound group in quantities as low as 0.1 nanograms, i.e. several thousand times smaller than the detection limit for the TLC technique cited above. The costs associated with purchasing and operating modern analytical equipment are incomparably higher than those of the old, simple methods.

Advances in laboratory techniques extended also to methods of preparing samples for testing. The simplest approach still in use, which involves extracting the sample with a suitably chosen solvent and then concentrating the extract by partially evaporating it, was supplemented by such techniques as solid-phase extraction (SPE) and solid-phase microextraction (SPME). Invented in the 1990s and used primarily in the analysis of post-fire residues, SPME proved particularly useful in the post-explosion analysis of triacetone triperoxide (TATP), an example of a new, home-made explosive that both domestic and Western forensic services had to confront in the second half of the 1990s.

Continuous scientific and technological progress, as well as the revolutionary changes in the country's economic landscape brought about by the political

transition, brought benefits not only to the services combating bomb terrorism, but also to criminals. In the past, anyone who did not have access to factory-made explosives but intended to construct an explosive device had to make ingenious use of the limited options available on the market, e.g. by converting miniature light bulbs into electric detonators. Building a more sophisticated device, e.g. a radio-triggered one, required specialist knowledge. At present, the general retail market offers pyrotechnic cords, electric fuses and even radio-controlled firework kits that do not require a licence. Despite their seemingly harmless purpose, these items are used by criminals in such enterprises as, for example, explosive ATM break-ins. This is an example of a new type of incident, unknown at the time of the People's Republic of Poland. Another new development involves the use of mobile phones as explosion trigger or fire devices, which can act both as a delay mechanism and as receivers enabling remote detonation of a charge from virtually anywhere in the world. The most technically complex trigger systems found in modern-day cases use programmable, miniature electronic circuits equipped with a microprocessor. An example of this is shown in Photo 3: a microcontroller-based electronic device that reacts to movement, equipped with a digital accelerometer and a lithium-ion battery with a USB-C charging port. Building such a device is well within the capabilities of a skilled amateur today.

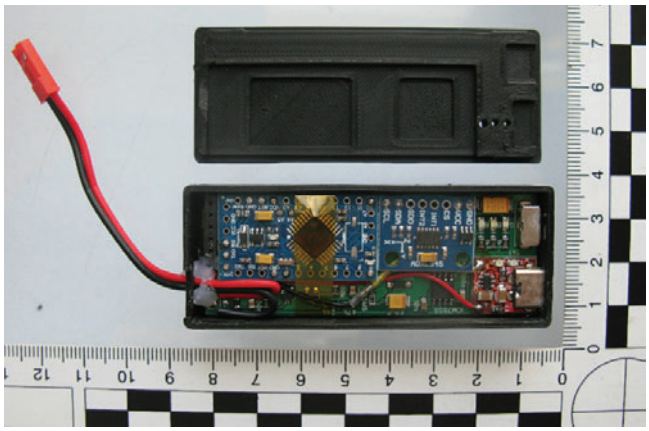

Fig. 3. A microcontroller-based motion-activated device (2025)

Today's economic freedom means, among other things, easy access to a wide range of chemicals that were previously difficult for the average consumer to obtain, and which can be used for making explosives. Despite efforts to introduce legislation restricting access to key precursors for explosives, forensic practice shows that the range and quantities of chemicals found at sites where explosives are made illegally are often significantly greater than they were under the planned economy. However, the change is most evident in the segment of entertainment pyrotechnics, which once existed in a rudimentary form - comprising practically only toy caps, sparklers and the aforementioned „cap gun caps" - but now constitutes a significant part of the economy and ranks Poland third in the global ranking of pyrotechnics exporters (data for 2024). There is a vast range of such products available on the market, and many of them - particularly flash bangs - have become a popular source of pyrotechnic mixtures for the construction of improvised explosive devices, having virtually completely replaced the once-common matchstick mixture.

The profound social changes brought about by the development of information technology, notably the advent of the Internet, have had an impact both on the activities of criminals who use explosives and on the work of persons who combat crime. Easy access to technical information has led to a relative decline in its value. In the past, anyone seeking to learn about making and detonating explosives had to rely either on specialist publications, available only in print in specialised reading rooms, or on the results of their own risky experiments. Nowadays, it is easy to obtain information on the properties and synthesis methods of various explosive substances. The ease with which information is shared means that various „novelties" in the field of making improvised explosive devices (IED) rapidly gain international popularity. A case in point is the „rediscovery" among amateur circles of erythritol tetranitrate (ETN), a long-known powerful explosive which gained in appeal following the widespread use of erythritol, a sugar substitute, which is its precursor. This material has also been found in Poland on several occasions in recent years.

The obvious benefits of easy access to information are enjoyed also by experts. Nowadays, thanks to online resources, it takes just a few minutes to find the properties of any chemical substance or to identify an unknown product from a photograph alone – a task that used to require spending hours poring over hard-to-find reference books and catalogues.

From the perspective of an expert's work, major changes have also taken place over the years in the legal framework regulating access to explosives. Between 1961 and 2000, all matters in this regard were governed by the Act of 31 January 1961 on weapons, ammunition and explosives, which was in force at the time. Over time, that Act, which ran to four pages on the day of its promulgation, was replaced by a series of legislative acts, namely: the Act of 21 May 1999 on weapons and ammunition, the Act of 21 June 2002 on explosives intended for civilian use, the Act of 13 April 2016 on the safety of trade in explosive precursors, the Act of 13 June 2019 on the conduct of business activities relating to the manufacture of and trade in explosives, weapons, ammunition and products and technology intended for military or police use. In addition to the above regulations, there are many other implementing acts.

Nowadays, an expert specialising in the examination of explosive materials and devices should have a sound understanding of the broader field of high-energy materials chemistry, explosive device electronics, as well as the legal aspects unique to their specialisms. In the above light, it can be concluded that the process of training experts in this field is becoming increasingly

demanding. Candidates acquire theoretical knowledge and undertake numerous practical training sessions in the field (training at a training ground). An invaluable aspect is the exchange of knowledge and experience among experts, particularly those with many years of experience, who actively participate in numerous training courses.

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