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USE OF GRENZ RAYS IN THE CRIME LABORATORY

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V. Pavlovsky has been engaged in legal photography and related scientific investigation in European crime laboratories and has published a number of papers in foreign scientific journals. After studying at the Technological Institute of Kiev (Ukraine) where he received degrees of B.S. in Chemical Engineering and of Candidate of Chemical Science, he joined the staff of the Kiev Institute of Scientific Criminal Investigation in 1935 where he served until the German occupation in 1941. From 1945 to 1947, when he came to this country, he was a scientific assistant in Legal Chemistry and Photography at the Institute of Legal Medicine of the University of Heidelberg. Mr. Pavlovsky has been working with X-ray photography and the applications of Grenz rays in scientific police work for several years.—EDITOR.

By far the best known application of X-rays in the crime laboratory is their use in photography, or radiography. They have been used to trace a bullet’s course through the body, to determine an individual’s age from the degree of calcification of certain parts of his skeleton, to expose counterfeiting of antiques, etc. The methods employed in such work are very similar to those used in medical diagnostics and in industrial material testing. This paper describes a relatively new field for radiography, which opens up new possibilities in the crime laboratory, as well as in many other fields. It is the use of soft X-rays, known also as Grenz-rays and familiar in the field of medicine as Bucky-rays.

The penetrating power or “hardness” of X-rays is greatest for rays of the shortest wave length. Thus, for deep therapy and for inspection of large objects, very short wave lengths from less than 0.02 to 0.05 Å are used. For diagnosis, rays from 0.2 to 0.4 Å are more commonly used. Grenz-rays, which are rays with wave length from 1 to 2 Å and longer, have very little penetrating power. Indeed they are almost completely absorbed in the outer layers of the human skin; and for this reason Bucky first proposed to use them in treating skin diseases (3). The degree to which soft X-rays are absorbed by different materials varies greatly; hence they can be used to photograph objects as thin as histological sections and still bring out considerable detail. In fact, the photography of histological sections by Grenz-rays and subsequent enlargement of the microphotographs has been studied intensively by a number of workers and named micro-radiography or histo-radiography (4, 5, 6, 7, 9, 10, 13, 16, 17, 21, 33, 35).

The bibliography at the end of this paper lists a number of works dealing with Grenz-ray photography in the study of plant parts (3, 8, 9, 12, 18, 28, 36); insects (10, 14, 16, 28, 30); parts
of human and animal bodies \( (1, 2, 21, 22, 23, 25, 35) \); structure of metals \( (5, 6, 7, 13, 24) \); textile fabrics \( (29, 31) \); paper and cardboard \( (13, 26, 27, 32) \). Their possible utility in the crime laboratory was first pointed out in 1927 by G. Kögel (19).

The writer, in collaboration with B. Kirichinsky, began in 1939 to study the use of Grenz-rays in the crime laboratory. They found that the wave length range from 1.2 to about 3 Å was very useful for photographic work such as the comparison of samples of paper, cardboard, and even charred paper or cardboard. The comparison of pencil strokes and of ink writing, the detection of writing in invisible ink, and the deciphering of blotted-out characters was found possible. Other applications were found, such as the detection of powder grains in the body and clothing in cases where a shot was fired at close range, the detection of certain poisons in the bodily organs, and differentiation between printing on the same sheet of paper done with different inks of the same visual color.

While the above applications refer to photography with long-wave X-rays of mixed wave length, such as are obtained from the usual medical Bucky-ray tube, there is another property of Grenz-rays that deserves attention. This is the possibility of obtaining sharp contrasts in absorption by different chemical elements, even those whose atomic numbers are close together, by using monochromatic radiation of properly selected wave length. This technique can be used to carry out a type of qualitative chemical analysis. It can be used to determine accurately the distribution of a given element through an object under examination \( (5, 10, 24, 34) \). In this connection, one can often choose the wave length to avoid the disturbing influence of strong absorption by a heavier element that is also present.

In general, X-rays can be used successfully when certain parts of the objects under study contain chemical elements that absorb the rays more strongly than the matter in adjoining areas; or, where a chemical element is present in non-uniform concentration and its distribution is the subject of the study. The usual range of hard X-rays are useful in studies of the heavier elements such as copper, lead, tin, mercury, gold, barium, bromine, iodine, arsenic, and antimony. Grenz-rays, however, enable one to detect much smaller quantities of these elements, and also to detect and differentiate between the lighter elements such as aluminum, magnesium, phosphorus, silicon, and sulfur. The possibility of solving a given problem by the use of Grenz-rays depends on the chemical composition and homogeneity of the basic mass of the substance being examined,
TABLE I.

POSSIBLE USES OF GRENZ RAYS IN CRIMINAL INVESTIGATIONS

I. RADIOPHGRAPHS OF RATHER LARGE OBJECTS (MACRO-RADIOGRAPHS).

1. Examination of Documents
   a. Identification (comparison) of samples of paper or cardboard, even when charred.
   b. Identification (comparison) of pencil, ink, carbon paper, etc., strokes on paper.
   c. Deciphering of faded or discolored writing in ink.
   d. Detection of writing in invisible ink.
   e. Detection of the removal by chemicals, or any alterations (additions) of pen or pencil strokes.
   f. Comparative examination of adhesive agents in traces on paper, etc.
   g. Comparison of printing inks on paper.
   h. Detection of various soilures on paper, ink, etc., and the detection of their nature in view of detecting forgeries, etc.
   i. Restoration of writing or printing on charred paper.
   j. Deciphering of blotted out texts or these enclosed in an envelope.

2. Firearms Investigations
   a. Determination, by means of surrounding lead traces of the entrance hole made by a bullet in the skin, clothes, wood, etc., (when bullets without casings have been used).
   b. Proof of the firing of a gun at close range by means of traces of lead or mercury in the area surrounding the entrance hole of the bullet (for instance when Flobert cartridges with mercury fulminate or cartridges with "Synoxyd," lead-containing priming mixture, were used).
   c. Proof of the firing of a shot at close range by the presence of remnants of particles of gun powder on clothes or body.
   d. Detection of particles of glass imbedded in a body or clothes material carried along by a bullet that passed through glass, etc.
   e. Comparative examination of gun powder particles of various origin.
   f. Comparative examination of the materials of shot-gun wads.

3. Examination of Body Parts in Poisoning Cases, or for the Purpose of Identification of the Corpse
   a. The detection in the skin of traces and locations of the rubbing in of ointments, containing zinc, bismuth, lead, iodine, thallium and other medicinal substances having high atomic weights.
   b. The detection in the skin of deposits of copper, gold, tin, lead, zinc, etc., resulting from constantly worn adornments, or representing professional peculiarities.
   c. The detection of hairs dyed by salts of metals (lead, silver, copper, cobalt, etc.) or containing such salts as a professional peculiarity or as the result of chronic poisoning.
TABLE I (Cont’d)

4. Examination of Food Products
   a. The detection in food products of the admixture of sand, glass splinters, metal shavings, etc., and the determination of their distribution.
   b. The detection of weighting or diluting mineral admixtures to food products; the detection of the presence of mineral poisons in food.
   c. The detection of damage done by insects to seeds and grain products.
   d. The detection of the spraying of vegetables and fruits with heavy metal salts (such as salts of barium, copper, etc.), used for protection against agricultural pests.

5. Various Other Examinations
   a. Comparative examination of cigar and cigarette butts.
   b. Comparative examination of textile fabrics, ropes, leather, etc., as an auxiliary means of identifying their origin.
   c. Comparative examination of impregnated materials of various kinds, for instance, when investigating the remnants of matches, etc.
   d. The detection of traces of heavy soft metals or their salts in a pure state, or, for instance, in the form of pigments, medicinal preparations, etc., on packing materials, clothes materials, etc. in investigating of theft and similar.
   e. The examination of chromatograms on paper strips (capillary analysis), particularly in cases when the solutions examined, contain colorless salts of metals or other heavy elements.
   f. The examination of pictures or drawings.

II. RADIOGRAHS REQUIRING FURTHER ENLARGEMENT (MICROOENTGENOGRAMS).

1. Examination of Documents
   a. Comparison of paper samples.
   b. The comparison of the structure of pencil and pen strokes on paper for the purpose of identification of writing material.
   c. Study of the chronological order of written strokes made by pencil, ink, etc., in places where they cross each other with possible use of stereomicrooentgenography.
   d. Study of the spreading of ions of chlorine and of sulphuric acid in paper, around pen strokes (for the purpose of estimating their age).

2. Firearms Examinations
   a. Examination of impurities on bullets’ surfaces and traces of metals on the skin, cloth, or wood in the area of the entrance hole of the bullet.
   b. The detection in body tissues of foreign matter particles carried along by the bullet into the wound and their close study.
TABLE I (Cont'd)

3. Examination of Body Parts in Poisoning Cases, Etc.
   a. Examination of the distribution of mineral salts deposits on hairs, in cases of artificial dyeing and of chronic or acute poisoning by heavy metals (particularly thallium).
   b. Examination of histological sections of various body organs (such as kidneys, liver, etc.) to establish the possible presence and distribution of heavy elements (such as mercury, lead, barium, antimony, arsenic, thallium, iodine, bismuth, copper) in the body tissues in poisoning cases.
   c. Examination of histological sections of lungs to determine the character of the dust in them, etc.

4. Other Examinations
   a. Examination of a test sample of dust or dirt from the clothes or in the pockets or underneath the nails, etc., to determine the presence of mineral particles in them and the study of the character of such particles.

and on the nature and relative concentration of the chemical element forming the admixture to be detected (5, 7, 15).

Although the use of Grenz-rays in the crime laboratory is very limited at present, they have proven adaptable and useful in a number of the problems that are encountered. They can be used to photograph rather large objects, for microradiography, and as a means of establishing the presence of chemical elements without damage to objects to be used later as evidence. The applications are classified in Table I.

Sources of Grenz Rays

The same principle is used in producing Grenz-rays, as in generating the more common range of X-rays: Electrons are emitted from the hot cathode of an X-ray tube, accelerated by a high voltage applied to the tube, and move with great speed in the vacuum until they impinge on an obstruction—the target. Most of the energy in the beam of electrons is dissipated in the target as heat, but some is transformed into X-rays (5, 34).

The tubes used to produce Grenz-rays are similar to the more usual type, but operate on much lower voltages, from about 4 kv to 12 kv; from 50 to 80 kv are employed in diagnostics, and much higher voltages are used in deep therapy and in materials testing. Grenz-ray tubes are also built with targets of different metals; tungsten, copper, nickel, iron, chromium, etc., in order to produce rays of different degrees of hardness or softness. As Grenz-rays are very easily absorbed, the tube windows are
constructed to minimize absorption. This may be done by using a special glass, such as lithium-beryllium-borate Lindemann-glass. A spherical bubble of ordinary glass, about 20 microns thick, has also been used in commercial tubes as well as thin beryllium sheet. Experimental tubes have been built with windows of cellophane, aluminum foil, and metallic lithium sheet, all serving the same purpose of transmitting a maximum of the Grenz-rays.

**Radiation of Mixed Wave Length.** The majority of Grenz-ray applications listed in Table I can be carried out using medical Grenz-ray tubes. These tubes generally are made with tungsten targets, and their radiation is of mixed wave lengths. Typical patterns, showing the relative intensity of rays of different wave lengths, are given by the solid curves in Figure 1. Curves for three tube voltages are given, and it will be noted that the minimum wave length decreases as tube voltage increases.

In general, it may be said that in the beam of emitted rays, the upper limit of wave length is set by the absorbing characteristics of the tube window, and the minimum wave length is fixed by the maximum voltage applied to the tube. For photography, one is most interested in the wave length at which
the maximum intensity of radiation occurs. It can be seen from Figure 1 that this is somewhat longer than the minimum. As the beam of Grenz-rays passes through air, the rays of longer wave length are absorbed more readily, and the maximum intensity shifts slightly to shorter wave lengths.

Figure 2 is given as a guide for the radiographer using a Grenz-ray tube with tungsten target. This shows the relation between minimum wave length of emitted radiation, and tube voltage. For tubes operating on alternating current, this is the maximum instantaneous value of voltage. The same figure shows the wave length of maximum intensity after the Grenz-ray beam has passed through 20 cm (8 inches) of air. By changing tube voltage, the investigator can vary the wave length at which the major part of the photographic effect is produced, and obtain a considerable range of penetrating power or hardness with a single tube. By changing tube current, he can vary the intensity of radiation and thus influence exposure time.

Monochromatic Radiation (5, 10, 20, 24). For some applications, it is desirable to use substantially monochromatic Grenz-rays. One way of producing such radiation is to use tubes with targets of different metals.

It was described above how the minimum wave length of mixed radiation decreases as tube voltage is increased. When tube voltage reaches a critical value, the target begins to emit radiation with a wave length characteristic of the target element. The intensity of this characteristic radiation increases sharply
upon relatively slight increase in tube voltage; so much, indeed, that the resulting beam of Grenz-rays is practically monochromatic.

A second, often more convenient method of producing monochromatic radiation is to direct a primary beam of hard X-rays from a standard tube, at a target element (10, 20). This beam produces secondary radiation that is also substantially monochromatic, and at very nearly the same characteristic wave lengths as for X-ray tubes with special targets. The most effective primary radiation for this purpose is produced by tubes operating at 30 to 50 kv, a normal range for X-ray tubes used in medical diagnostics. On the whole, the intensity of secondary radiation is quite low, but becomes somewhat larger with increase in the atomic number of the radiator.

The technique of using secondary radiation for Grenz-ray studies offers several advantages. A single medical X-ray tube suffices for work at a number of wave lengths. The apparatus eliminates the need for protecting the photographic emulsion from light rays. It is easily adapted to use vacuum or a hydrogen atmosphere, which is necessary when working with rays of wave length over 3 or 3.5 A. Figure 3 (after Dershem) shows one type of apparatus. The primary X-ray beam enters through a window, strikes a radiator made of the desired chemical element, and produces a beam of secondary radiation which is directed at the object under study and the photographic plate. The apparatus is air tight and can either be evacuated or filled with dry hydrogen. It can be equipped with interchangeable radiators made of different materials.
Absorption of Grenz-Rays

The absorption of X-rays varies in an exponential manner with the cube of their wave length, the fourth power of the atomic number of the absorbing element, and the thickness of the layer of absorbing material. The ease with which long wave length rays are absorbed accounts for the large change in the intensity pattern of mixed wave length radiation shown in Figure 1. The effect of thickness of the absorbing layer is very large. It determines the thickness of histological cross-sections and material samples which may be examined. Its influence is indicated by the special window constructions used in Grenz-ray tubes. Closely allied to thickness of a layer of uniform absorbing material, is the concentration of an absorbing element dispersed in a medium of lesser absorbing power.

Figure 4 shows quantitatively how the intensity of a Grenz-ray beam is decreased in its passage through air, between the window of the X-ray tube and the object studied. The influence of wave length is clearly evident. Operation in air generally becomes impractical when intensity is decreased to about 20% of its initial value. For example, a working distance of 5 inches would limit work in air to wave lengths 3.5 Å and less. Hydrogen, on the other hand, causes only about 0.1% decrease in intensity of 5Å rays over an 8 inch path. In practice, a hydrogen atmosphere may be of advantage at wave lengths above 2.5 Å,
and either hydrogen or a vacuum is found necessary when working above 3.5Å.

Mass absorption coefficients denote the decrease in intensity suffered by a beam of X-rays 1 sq. cm. in area, on passing through 1 gram of the substance in question. They change considerably with the wave length of radiation. Their values for a number of materials have been calculated or determined experimentally by different workers and are useful as a guide in differentiating structures or searching for admixtures of suspected materials. Coefficients for a number of elements are shown in Figure 5, plotted against wave length in the Grenz-ray region. One immediately notes the sudden jumps in absorption coefficients. The wave lengths at which these jumps occur, correspond closely to the wave lengths of characteristic radiation from targets of these same elements. With copper, for instance, the jump in absorption occurs at 1.38 Å. These wave lengths are independent of the physical and chemical state in which the element is present. Heavy elements, like barium, have K-jumps at much shorter wave lengths but can also have L-jumps in the range of Grenz-rays.

These sudden changes in absorption can be used to study the distribution of chemical elements in plant and animal tissues. Dershem (10) made use of the K-jump in calcium absorption to study the distribution of calcium in extremely thin sections of bone. This jump occurs at 3.06 Å, so he used scandium radiation at 2.75Å to make micro-radiographs. At this wave length absorption by calcium is near its maximum value in the Grenz-ray region, and it affords maximum contrast to the absorption of other elements present in bone tissues: Hydrogen, carbon, oxygen, nitrogen, and phosphorus.

By making a series of photographs with monochromatic radiation of properly chosen wave lengths, this principle can be used to detect or to differentiate between such elements as iron, manganese, and copper.

**Radiography with Grenz Rays**

Radiography with Grenz-rays presents a problem in protecting the photographic emulsion from visible light, while at the same time avoiding any unnecessary decrease in intensity of the rays, which are already very weak in comparison with the usual range of X-rays. The preparation of objects for photography, especially for micro-radiography, is somewhat critical. The exposure time, the choice of photographic emulsions, their processing, and methods for later enlargement of the image are also important.
Mass Absorption Coefficients in the Grenz Ray Region for Representative Elements and Materials.

The initial intensity of a beam of rays $I_0$ decreases to a value $I$ in passage through a piece of material of $d$ cm. thickness in accordance with the equation:

$$I = I_0 e^{-\mu d}$$

where $\mu$ is the linear absorption coefficient, a function of the wavelength and $\rho$ is the specific gravity of the given element or material.

A number of methods for protecting the photographic emulsion from visible light can be used. In many cases, the window
of the X-ray tube can be covered by a filter which does not pass photographically active light, the tube itself enclosed, and the entire operation conducted in a darkroom under red light for working illumination. As a Lindemann-glass window of a Bucky-ray tube must be protected from the atmosphere by a coating of lacquer, one merely paints over this with another coat containing a suspension of lamp black. Large objects can then be placed directly on the photographic emulsion and irradiated. Another convenient method, useful when working with relatively hard radiation from 1 to 1.2 Å, is to enclose the film or plate in an envelope of thin photographic black paper on which the object can be placed. The paper should always be tested to insure that it produces no irregular shadows, graininess, or other structural details. For work with somewhat softer rays, whose absorption by the paper would be too great, a window may be cut in the envelope and covered with a gelatin foil filter of the kind used in photography.

Suitable filters for radiation from ordinary Grenz-ray tubes may be made from foils of dyed cellophane, celluloid, or gelatine. Red or orange photographic filters of gelatine are satisfactory. Still better is the Wratten No. 87 infra-red filter recommended by Sherwood. These filters are generally between 0.012 and 0.020 mm (0.0005 to 0.0008 inches) thick. Thicknesses up to 0.030 mm (0.0012 inches) may be used; Figure 6 shows the absorption of Grenz-rays of different wave lengths by various thicknesses of cellophane filters.

A simple construction for a film holder suitable for macro- or micro-radiography is shown in Figure 7. One takes a cardboard box in which photographic plates are packed, cuts a hole in the cover, and covers the hole with a suitable film to act as a light filter. In the darkroom, one places a plate in the box emul-

**Figure 6.** Absorption of Grenz Rays by Cellophane Light Filters.

**Figure 7.** Film and Object Holder for Grenz Ray Photography.
sion side up, places the object to be photographed on the plate, and closes the box. The holder is carefully moved under the Grenz-ray tube and irradiated from above. To prevent the specimen from slipping when using a horizontal X-ray beam, one places a springy material under the plate, which holds the object in place by pressure between the plate and the light filter.

Investigators working with Grenz-rays in the range from 3 to 12 Å and above have to eliminate all possible absorption between the source of radiation and the object being photographed. Dershem's apparatus for working with secondary radiation has already been described. Lamarque (22) went further by building his X-ray tube and camera as a single unit and evacuating the whole assembly. To shield the emulsion from the light of the glowing cathode, he covered it with aluminum or lithium foil, several microns thick. These special precautions are unnecessary when working with ordinary Grenz-ray tubes, which produce radiation largely under 3 Å.

**Objects for Radiography.** Objects for radiography may be from fractions of a millimeter up to several millimeters thick. The thickness to be used will depend on the material and the wave length of radiation. For example, rays of 1.0 to 1.2 Å will penetrate readily through a ¼ inch pine board, or through dried skin. Rays of 2.3 to 2.5 Å, however, will be considerably absorbed by a sheet of writing paper about 0.1 mm. (0.004 inches) thick. Objects containing considerable water absorb Grenz-rays far more than when dry.

Objects for histological sectioning and micro-radiography may be fixed with formaldehyde or alcohol but should not be treated with fixatives containing heavy metal salts (2, 21, 35). Sections imbedded in paraffin may be fastened directly to the photographic emulsion with water or alcohol. However, Grenz-rays are strongly absorbed by paraffin, and it must be washed out by successive extractions with xylene (under red light) before the exposure can be made. Perhaps the best method is to use the freezing technique and to place the frozen sections directly on the photographic emulsion. Lamarque and Turchini found that damp specimens adhered well, and dried rapidly without noticeable structural changes. After making the exposure, one may immerse both plate and section in the developer; the section generally frees itself without difficulty. To show fine structure in histological sections, their thickness must be not over 0.003 to 0.005 mm. To determine the distribution of heavy metals in different parts of an organ, thicker sections up to 0.02 or 0.05 mm. may be used.
Photographic Materials. The usual types of X-ray films and plates are well suited to Grenz-ray photography. Films coated on both surfaces offer no advantages, however, as the rays are completely absorbed in the first emulsion layer. Intensifying shields are not suitable; they are opaque to Grenz-rays.

For micro-radiographs, to be subsequently enlarged from 10 to 30 times, one can use fine grain development on ordinary process plates or films or special Eastman fine grain film Type V-O spectroscopic plates (11). Greater enlargements, up to 40 or 50 times, require finer grain emulsions, such as Kodalith Halftone. Still greater enlargements require Eastman Type 548-O Spectroscopic Plates, or similar emulsions.

The sensitivity of the almost grainless materials to Grenz-rays is unfortunately very low, and therefore a very long exposure period is required. The relative speed of Kodalith Halftone is 1/10 of the speed of Eastman Type V-O spectroscopic plates, and the relative speed of Eastman Type 548-O spectroscopic plates is only 1/200.

Time of Exposure. There are a number of factors which are of importance in determining the time of exposure: Tube amperage, tube voltage, distance between tube and plate, the length of the path of the rays through the air, characteristics of the membrane protecting the emulsion from light, characteristics of the objects under investigation, and the relative speed of plates or films.

With increase in tube amperage, increases the general intensity of the rays without change of their wavelength (of their hardness). With increase in tube voltage the wavelength of the rays decreases and they become harder. It means, they are not weakened as much by various media in their path: By the window of the X-ray tube, by the air (see Figure 4), by the membrane protecting the photographic emulsion against light (see Figure 6), and by the object itself. Therefore harder rays cause more intensive exposure even with the same intensity of radiation. But as the tube voltage increases, the general intensity of the radiation emitted increases quite rapidly. In some cases the increase of the intensity is proportional to the fourth power of the increase in tube voltage.

When we change the distance between the tube and the object, the intensity of the rays at the object changes in an inverse proportion to the square of the ratio of new and old distances. The change of absorption of the rays by air in their passage through the new distance must also be calculated in addition.

Concerning the influence of the thickness of objects for radiography, their chemical composition, and their water content
on the exposure, and concerning the relative speed of photographic materials see above.

All these factors combine to cause an extremely wide range in the exposure times used in practice. If some changes are made, it is necessary to calculate the new exposure on the base of old data and then test the calculated exposure experimentally.

The finished negatives of microradiographs are printed by projection with the usual enlarger for small size negatives. For enlargements of more than 10 or 12 times, it is advisable to treat the negative like a microscopical specimen: To mount it in Canada balsam under a cover glass, and to make a microphotograph of it in the usual way.

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