Evaluation of Fragments of Window Glass
as Evidence in Criminal Cases
Thesis for the Degree of M.S.
Michigan State University

John P. Klosterman

1964

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# EVALUATION OF FRAGMENTS OF WINDOW GLASS AS EVIDENCE IN CRIMINAL CASES

by

John P. Klosterman

AN ABSTRACT OF A THESIS

Submitted to
Nichigan State University
in partial fulfillment of the requirements
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1964

APPROVED

F Tunnon (Chairm

Raymond T Galorn

P. Kaje-waras.
(Member)

#### ABSTRACT

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evidence in several types of investigations. They are frequently found in cases involving forceable entry such as burglary and are often found in traffic accidents, assaults, and explosions and they are occasionally involved in suspected poisonings.

Several studies have been made on the possibility of identifying the source of various types of glass fragments. Specific studies have been made on brown beer bottles, sealed beam headlights, and head-lamp lenses. This study is confined to window glass fragments from known sources and those collected from cases over a two year period.

Of the physical properties of glass that may be compared, most workers have found that refractive index and density were the most satisfactory for individualizing glass samples. Some held that refractive index was more discriminating while others

preferred density determinations or comparisons.

This study, although more detailed on refractive index, tends to support the latter conclusion.

The refractive indices of the samples were determined using a sodium lamp as the monochromatic (589 millimicrons) light source. The refractive indices were determined by the use of the Becke line observed through the microscope while the samples were immersed in an oil of known refractive index. Using a hot stage to increase the temperature and thereby change the refractive index of the oil, the Becke line was observed and then the refractive index of the glass was calculated using the temperature coefficient and refractive index of the oil used. Comparative refractive index determinations using a red filter (640-700 millimicrons) were not of value in differentiating between glass fragments having similar refractive indices as obtained using the sodium lamp.

Comparative density determinations using gradient density tubes were made and separations could be made on many samples having similar refractive indices, but not all samples could be individualized.

Examination for ultraviolet fluorescence was found to be of no value as none of the window glass samples exhibited any fluorescence.

Spectrographic examinations were not reproducible and so composition of glass fragments was not pursued as a method of analysis. In the absence of trace elements, it is doubtful whether spectrographic examinations would be of any value unless they were quantitative.

The study revealed that with refractive index determinations and density comparisons it was not possible to differentiate between fragments from one piece or the same batch of window glass, but fragments from different batches could be differentiated.

Of one hundred twenty-nine samples of window glass fragments collected over a two year period, it was possible to individualize 27 by refractive index alone and an additional 66 by a combination of refractive index and specific gravity. The 35 which could not be individualized fell into 10 groups of 2, 3 groups of 3, and one group of 7.

On the basis of refractive index and density, therefore, it was not possible to individualize all window glass fragments. Other factors such as surface pattern, color, thickness (if it can be measured), dispersion, hardness, and chemical compositon would probably make some further differentiations.

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#### CHAPTER I

#### THE PROBLEM AND DEFINITION OF TERMS USED

Glass fragments have been used as evidence in criminal cases for many years. Their use dates back to at least the early part of the 1900's. Dr. Cross' mentions comparison of color, ultraviolet fluorescence, density, refractive index, and hardness in the examination of glass fragments. The term 'glass fragments is used to describe small particles of glass such as might be found in a suspect's clothing. on tools, or in a variety of other locations. These usually are only a few millimeters in their longest dimension. Throughout this study the sample sizes used were kept within the size normally found in criminal investigations. That is, only about one or two millimeters in their longest dimension. This was done so as to duplicate any problems which might be encountered in actual cases.

Because most types of glass are easily broken and glass is so frequently used in a variety of objects, it is a very common type of evidence. As with other materials, almost any type of glass

Gross, Hans. Criminal Investigation, 4th ed. London: Sweet & Maxwell, 1950, p. 94

could become involved as evidence in a criminal matter. Some of the more common types of glass are automobile headlights and windows, bottles, drinking glasses, eye glasses, light bulbs, ornamental objects, and windows of buildings. Glass could become important in almost any type of case, however, it occurs most often in incidents such as assaults, homicides, and burglaries.

It was noted that during the period from 1957 to 1964 in the laboratories where the writer has been employed, that window glass (from buildings) constituted approximately ninety per cent of the glass cases.

More specifically, during the four year period from late 1960 to late 1964, out of 162 glass cases, 146 involved window glass. This calculates to a percentage of 90.1%. It was because of the commonness of glass as evidence, and more specifically window glass, that this subject was selected for study.

#### I. THE PROBLEM

Statement of the problem. This study consisted of an examination of 129 samples of glass collected in actual cases to determine how much duplication of physical properties such as refractive index, specific gravity, and ultraviolet fluorescence there might be in window glass fragments from various random sources.

In making this determination, two preliminary problems arose which required examination. First, what were the experimental errors in determining the physical properties and how much do these errors effect the reproducibility of the methods? Second, what range of variation might one expect in these properties within a single pane or sheet of glass: (Such as might come from a particular window.) These two questions had to be answered in order to properly evaluate the comparison of the random samples.

The basic problem might be expressed in two ways. That is, 'Is it always possible to distinguish between glass fragments from different sources?' or 'With how much certainty may it be stated that two samples of glass could have come from the same source?' Because of the limited number of samples involved, this study draws only general statistical conclusions; the basic conclusions were based on an evaluation of the samples examined,

Since suitable methods for determining the physical properties, such as refractive index and specific gravity, of glass are available, this study did not undertake the development of any new methods. Rather, it made use of methods and equipment which are readily available to most police laboratories.

Importance of the problem. There is need in the field of police laboratory work for more studies to be done on the evaluation of all types of physical evidence. There have been a few studies such as one by Burd and Kirk<sup>2</sup> in which they discuss the degree of match necessary in tool marks to make an identification. They stated that the proportion of matching lines is more important than the number of such lines. A statistical study was made on bullets by Biasotti<sup>3</sup> and he made some conclusions as to the basis for matching, but stated that the collection of much more data would be very desirable. A general review on the evaluation of this type of evidence is presented by Biasotti<sup>4</sup> and in this he restates the opinion that more data is necessary for useful statistical conclusions.

<sup>&</sup>lt;sup>2</sup>Burd, D. Q. & Kirk, P. L., "Tool Marks, Factors involved in Their Comparison and use as Evidence," The Journal of Criminal Law, Criminology, and Police Science, 32:679-636, 1942

<sup>&</sup>lt;sup>3</sup>Biasotti, A. A., "A Statistical Study of the Individual Characteristics of Fired Bullets," <u>Journal of Forensic Science</u>, 4:34-50, 1959.

<sup>&</sup>lt;sup>4</sup>Biasotti, A. A., "The Principles of Evidence Evaluation as Applied to Firearms and Tool Mark Identification," <u>Journal of Forensic Sciences</u>, 9:428-433.

A study was done on wool fibers by Burd and Kirk. This discusses the uses of probabilities to show identity of source of fibers. A statistical study of scale counts on human hairs was made by Gamble and Kirk. This was done with the idea of further individualizing hair and they state that the results, if properly obtained and used, increase the probability of showing common origin of hair samples.

Kirk<sup>7</sup> discuss the general area of evaluation in regard to most types of physical evidence, and he states that some previous studies have had short-comings and that there is much to be done on evaluation of physical evidence. He mentions that the greatest problem is the collection of large amounts of data; also a better relationship should be established between criminalists and statisticians.

<sup>&</sup>lt;sup>5</sup>Burd, D. Q. & Kirk, P. L., "Clothing Fibers as Evidence," <u>Journal of Criminal Law, Criminology</u>, and <u>Police Science</u>, 32:353-357, 1941.

Gamble, L. H. & Kirk, P. L., "Human Hair Studies," <u>Journal of Criminal Law</u>, <u>Criminology and Police Science</u>, 31:627-636, 1941.

Kirk, P. L., "Evidence Evaluation and Problems in General Criminalistics," <u>Journal of Forensic Sciences</u>, 9:434-444, 1964.

Because of this need for evidence evaluation and because window glass occurs frequently as evidence, it was felt that this was an area in need of further examination. It is hoped that this study will make a contribution to the knowledge about physical evidence, and about glass fragments in particular.

<u>Hypothesis</u>. The evaluation of window glass fragments may be better made with more information as to the variations and duplications in their physical properties.

#### II. DEFINITIONS OF TERMS USED

Refractive index. The refractive index of a material may be defined in two ways. In terms of velocity of light the refractive index of an isotropic substance is defined as the ratio of the velocity of light in air to its velocity in the substance being examined. The other definition is in terms of the sines of the angles of incidence and refraction. When light passes from one medium to another of different density, it is deviated from

Glasstone, Samuel, The Elements of Physical Chemistry, New York: D. Van Nostrand Co., 1946.

its original path. The refractive index is expressed as the ratio of the sine of the angle of incidence to the sine of the angle of refraction.

Since the refractive index varies with temperature, this must be stated when giving the refractive index of a substance. The amount of change with a change in temperature is described as the temperature coefficient. In virtually all instances, the temperature coefficient of liquids is much greater than that of solids. For instance, the temperature coefficient of the oils used in this study was  $0.0004/^{\circ}$ C while that of glass is only  $0.000001/^{\circ}$ C.

Refractive index also varies with the wavelength of the light used. This is the reason that
monochromatic light must be used for accurate results.
The most commonly used wavelength is the sodium D-line.
This variation of refractive index with wavelength is
made use of in the measurement of dispersion.

<sup>90</sup> Hara, C. E. & Osterburg, J. W., An Introduction to Ciminalistics, New York: Macmillan, 1949, p.561

Dispersion. Transparent objects slow short wavelengths of light more than long wavelengths.

This property is described as dispersion. Because of this dispersion, glass will show a greater refractive index toward the shorter wavelengths. Some glasses will show more variation than others so that if dispersion can be measured, it is a valuable property. 10

Specific gravity. Specific gravity is the ratio of the weight of a specific volume of a substance to the weight of the same volume of water. 11 Because of the coefficient of expansion of all materials, it is necessary to specify temperatures for both water and the substance being described except when using approximate values.

 $<sup>^{10} \</sup>text{Kirk. P. L., Crime Investigation. New York:}$  Interscience Publishers, Inc., 1953, p. 246.

<sup>11</sup>Kirk, P. L., <u>Density and Refractive Index</u>, Springfield: Charles C. Thomas, 1951, pp. 13-14.

#### CHAPTER II

#### REVIEW OF THE LITERATURE

The review of the literature on glass as related to this study is divided into three parts. The first covers glass in general and includes information pertaining to the manufacture and composition of glass. The second covers the literature on physical properties and spectrographic comparison, and the last, the literature on evaluation of glass as evidence. A comparison of the results of this study with the studies mentioned in this review is found in Chapter VIII.

## I. LITERATURE ON MANUFACTURE AND COMPOSITION OF GLASS

Originally glass was made in batches using 'pots' for the melting and mixing of the components. At the present time, this method is used mainly for small batches of special production glasses.

Modern glass factories use large tank furnaces for the mixing and melting of the components.

 $<sup>^{1}\</sup>text{Morey.}$  George W., The Properties of Glass. New York: Reinhold Publishing Co., 1938, p. 25.

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 $<sup>\</sup>frac{1}{25}$  Morey, George W., The Properties of Glass, New York: Reinhold Publishing Co., 1938, p. 25.

As this process is virtually a continuous one, the variations in modern glass manufacturing are much less than with older methods.<sup>2</sup> This is especially true of mass produced glass such as window glass.

Basically glass is made up of the oxides of silica, sodium, and calcium with smaller amounts of the oxides of aluminum, iron, magnesium, and sulphur. In addition, there are trace elements which occur accidentially or have been added for specific purposes, such as color or strength. Kirk states that in the manufacture of glass it is difficult to obtain a completely uniform mixture and this coupled with the presence of elements other than the basic components form the basis for comparisons in criminalistics laboratories. Nickolls cites Home Dickerson's figures that these variations are not too great. He states that the specific gravity range of glass from a tank furnace varied only from 2,503 to 2,515 over a period of a month and a half.

Lindquist, Frank (Ed.), Methods of Forensic Science, Vol. I. New York: Interscience Publishers, 1962, p. 364.

<sup>30°</sup>Hara, C. & Osterburg, J., An Introduction to Criminalistics. New York: Macmillan, 1949, pp. 302-305.

<sup>&</sup>lt;sup>4</sup>Kirk, P. L., <u>Crime Investigation</u>, New York: Interscience Publishers, 1953, pp. 234-235.

<sup>&</sup>lt;sup>5</sup>Lindquist, op. cit., p. 365,

# II. LITERATURE ON PHYSICAL PROPERTIES AND SPECTROGRAPHIC COMPARISON

Refractive index. The simplest method for the determination of refractive index is described by Tryhorn. 6 A fragment of the glass to be examined is placed in a few drops of a liquid, such a monobromo-nathalene (refractive index 1.66), which has a refractive index higher than glass. A liquid with a refractive index lower than glass, such as alcohol (refractive index 1.37), is added dropwise with mixing until the glass becomes invisible in the mixture. No mention is made of measuring the amounts of liquids added. The sample or samples to be compared with the first sample are then added to the liquid mixture and if they too become invisible. they are of the same refractive index. A sample of the liquid mixture may be removed at this point and the actual refractive index read on a refractometer. This measurement is quite subject to error by evaporation. A more sensitive method of determining the refractive index of glass fragments is described by Kirk<sup>7</sup> as the 'Oblique Illumination Method.'

Tryhorn, F. G., "The Examination of Glass,"

<u>Journal of Criminal Law, Criminology, and Police</u>

<u>Science</u>, 30:414-415, 1939.

<sup>&</sup>lt;sup>7</sup>Kirk, Crime Investigation, op. cit., pp. 563-564.

In this method the glass fragment is immersed in a liquid which has a refractive index close to that of the sample and observed under a microscope using a half illuminated field. The liquid is then varied in refractive index by the addition of another liquid. No mention is made of measuring the amount of liquids used. When the dark side of the fragment is on the same side as the dark side of the field, the fragment has a higher index than the liquid; when the dark and light sides reverse, the liquid has the higher value. The intensity of the two sides will be equal and the glass fragment will essentially disappear when the refractive index of the glass and the liquid are the same. This method is rather tedious and so the method which is usually preferred is the use of the Becke Line.

The 'Beck Line Method' is the most commonly used method for determining the refractive index of small fragments of glass. This method was first described by F. Becke in 1893 when he pointed out that if two adjacent minerals having different refractive indices are illuminated by a narrow cone of light and the focus of the microscope is raised, a bright line appeared within the border of the mineral having the higher refractive index;

lowering the focus reversed this line. This method is also referred to as the Normal Illumination Method.

The usual method of using the Becke line to determine the refractive index of a substance is described by Chamot and Mason. 10 This involves using two liquids; one with a refractive index higher than the sample, and the other lower. These two liquids are mixed in measured proportions until they are within the refractive index range of glass. (1.500-1.550; this might vary depending upon the type of glass.) The sample is then immersed in the mixture and observed under the microscope. The refractive index of the liquid mixture is varied by adding measured amounts of either the higher or lower refractive index liquid until the Becke line just disappears. When this occurs, the liquid mixture has the same refractive index as the glass. The refractive index of the liquid mixture, hence the glass, is then calculated from the measured amounts of the liquids used or it is read on a refractometer.

<sup>8</sup>Allen, Roy, <u>Fractical Refractometry by Means of the Microscope</u>, New York: Cargille Laboratorics, 1954, p. 6.

<sup>9</sup>Kirk, Crime Investigation, op. cit., p. 559

<sup>10</sup>Chamot, E. & Mason, C., <u>Handbook of Chemical Microscopy</u>, Vol. I, 2rd Ed., New York: Wiley, 1959 pp. 314-317.

A variation of this method is described by Roche and Kirk. 11 Their method uses for the liquid with the lower refractive index one which is fairly volatile. The mixture is then adjusted to have a refractive index slightly less than the glass sample and then the more volatile component is allowed to evaporate slowly while observing the Becke line. It is necessary, of course, to have the samples being compared in the liquid at the same time. If the Becke line changes direction in both fragments at the same time, they have the same refractive index. Using the evaporation method, it is not possible to determine numerical values for refractive index.

The stated accuracy for refractive index using the mixing methods is only to the third decimal place,  $^{12}$  so all of these methods are used in the direct comparison manner.

A scheme for speeding up the adjustment of the mixture is described by Davis.  $^{13}$  This uses the

Roche, G. & Kirk, P., "Differentiation of Similar Glass Fragments by Physical Properties,"

Journal of Criminal Law, Criminology and Police

Science, 38:169-170, 1947.

<sup>&</sup>lt;sup>12</sup>Ibid., p. 170

<sup>13</sup>Davis, J., "Refractive Index Determination of Glass Fragments-A Simplified Procedure," <u>Journal of Criminal Law, Criminology</u>, and <u>Police Science</u>, 47:380-386, 1956.

dispersion colors to roughly estimate the differences between the refractive index of the liquid and of the glass, so as to better determine how much adjustment is necessary. When the disappearance point of the Becke line nears, a sodium lamp replaces the white light as the light source, as the monochromatic light allows a more precise determination of the refractive index.

Another variation of the Becke line method is known as the 'Emmons Double Variation Method.' In this method a set of liquids having known refractive indicies is used. The glass is immersed in a liquid having a refractive index slightly higher than the glass; the refractive index of the liquid is lowered by gentle heating until the liquid and the glass have the same refractive index. This is determined by the usual observation of the Becke line. The temperature is noted and the refractive index is calculated using the temperature coefficient of the liquid. This method is based on the fact that while the temperature coefficient of glass is very small

<sup>14</sup>Allen, op. cit., p. 9

 $(0.000001/^{9}C)$ , that of the oils is proportionally much higher  $(0.0004/^{9}C)$ . A detailed description of this method is found in Chapter V since this was the method used for this study.

Grabar and Principe  $^{15}$  used this method to determine refractive index and dispersion of glass fragments. They did not have oils available which were calibrated for the red and blue wavelengths so they give a method for making measurements of refractive index at these wavelengths by using a correction factor. They give the normal accuracy of obtaining temperature changes in refractive index determinations by this method as  $\neq 1.0^{\circ}$ C. However, they do state that with careful procedure, an accuracy of  $\neq 0.5^{\circ}$ C is possible.

Roche and Kirk<sup>16</sup> found that refractive index was a more delicate factor for making differentiations than specific gravity. They followed a general procedure of specific gravity first and then refractive index determination on samples which could not be individualized. This was also found to be the case in

<sup>15</sup>Grabar, D. G. & Principe, A. H., "Identification of Glass Fragments by Measurement of Refractive Index and Dispersion," <u>Journal of Forensic Sciences</u>, 8:54-67, 1963.

<sup>&</sup>lt;sup>16</sup>Roche & Kirk, op. cit., pp. 169-171.

the work done by Gamble et. al.  $^{17}$  and Greene and Burd.  $^{18}$ 

Marris  $^{19}$  and Nelson  $^{20}$  found that specific gravity was of greater value than refractive index in individualizing glass fragments.

Specific gravity. Tryhorn<sup>21</sup> describes the simplest method of comparing the specific gravities of glass fragments as floating them on bromoform or methylene iodide about one half inch deep in a test tube, and then adding dropwise, with mixing, alcohol until a mixture is formed in which the samples are suspended. If they are suspended in the same mixture, the fragments are all of the same specific gravity.

<sup>17</sup>Gamble, et. al., "Glass Fragments as Evidence," <u>Journal of Criminal Law, Criminology</u>, and <u>Police Science</u>, 33:419, 1943.

<sup>18</sup> Green, R. & Burd, D., "Headlight Glass as Evidence," <u>Journal of Criminal Law</u>, <u>Criminology and Police Science</u>, 40:87, 1949.

<sup>19</sup>Marris, N. A., "Identification of Glass Splinters," The Analyst, 59:687, 1934.

 $<sup>$^{20}\</sup>rm{Nelson}$$ , D. F., "The Identification of Lucas 700 Headlamp-glass Fragments by their Physical Properties," Analyst, 84:390, 1959.

<sup>&</sup>lt;sup>21</sup>Tryhorn, <u>op</u>, <u>cit</u>, p, 417,

A method of determining the exact specific gravity using this method is described by Kirk and Russcll. 22 When the sample or samples are suspended in the liquid mixture, a sample is taken of the liquid and its specific gravity determined by the use of a pycnometer.

The necessity for very close temperature control in determining absolute values for specific gravity is pointed out by Beeman. <sup>23</sup> He also points out the need for using similar sized fragments in making specific gravity comparisons. When making comparisons, he suggests adjusting the liquid mixture close to the specific gravity of the glass and then using small changes in temperature to make final comparisons. <sup>24</sup>

 ${ t McCall}^{25}$  observed that the specific gravity of some glasses varied with a change in temperature.

<sup>22</sup>Kirk, P. & Russell, R., "Microdetermination of Specific Gravity in Forensic Chemistry," <u>Journal of Criminal Law, Criminology</u>, & <u>Police Science</u>, 32:118, 1941.

Variations on the Determination of Specific Gravity of Glass Fragments." Journal of Criminal Law, Criminology, & Police Science, 36:298-299, 1945.

<sup>24</sup>Beeman, <u>Ibid.</u>, p. 229

<sup>25</sup>McCall, D., "Temperature Variations with Respect to the Specific Gravity of Class Fragments," <u>Journal of Criminal Law, Criminology & Police Science</u>, 39:113-117, 1948,

Since the specific gravity increased as the temperature increased, the change could not be explained by the thermal expansion of glass. His only conclusion was that further work should be done along these lines. However, no further references to this were found in the literature.

Roche and Kirk<sup>26</sup> describe the adaptation of gradient density tubes for comparing the specific gravity of glass fragments. The tubes were made by layering nitrobenzene over methylene iodide, stirring slightly, and allowing a density gradient to form. A more refined description of this method is given by Kirk.<sup>27</sup> In this description, the tubes are filled with five different density liquids rather than only two. A detailed description of this method is found in Chapter V, since it is the method used for specific gravity comparisons in this study.

<sup>26</sup>Roche, G. & Kirk, P., "Differentiation of Similar Glass Fragments by Physical Properties,"

Journal of Criminal Law, Criminology & Police

Science, 38:169, 1947.

<sup>27</sup>Kirk, P. L., <u>Density and Refractive Index</u>, Springfield: Charles C. Thomas, 1951.

Fluorescence. The ultraviolet fluorescence of glass is mentioned by  $Tryhorn^{20}$  as a preliminary examination which differentiates between glass samples in only a few cases since most glass shows little or no difference in fluorescence.

Apparently most writers in the field have found it not to be too useful since it is mentioned only in passing in two books.  $^{29.30}$  and only a few mentions are made of it in the periodical references.  $^{31.32}$  with the general opinion being that its value is not too great. Marris  $^{33}$  reports the use of fluorescence and mentions obtaining better results using a microscope to make the comparisons.

<sup>&</sup>lt;sup>28</sup>Tryhorn, op. cit., pp. 416-417

<sup>29</sup>Kirk, Crime Investigation, op. cit., pp. 245-246.

<sup>300°</sup>Hara & Osterburg, op. cit., p. 259

<sup>31</sup> Lindquist, op. cit., p. 367

<sup>32&</sup>lt;sub>Nelson, on, cit., p. 388</sub>

<sup>33&</sup>lt;sub>Marris, op. cit., p. 686</sub>

Spectrographic comparisons. The use of the spectrograph is only briefly mentioned in most of the references on the examination and evaluation of glass fragments, 34,35,36,37,38

Kirk<sup>39</sup> states that spectrographic comparisons should be based on trace elements since the major constituants vary little from one glass sample to another unless they are of different types and then physical properties such as refractive index and specific gravity will differentiate them more easily. He also points out the disadvantage of consumption of the sample.

Nickolls\* opinion is that spectrographic analysis of window glass is uscless since variations in composition are too small to be accurately determined. He suggests a method which may be

<sup>&</sup>lt;sup>34</sup>Camble, et. al., <u>op. cit.</u>, p. 416

<sup>&</sup>lt;sup>35</sup>Greene & Burd, <u>op</u>, <u>cit</u>, p, 89

<sup>36</sup> MacDonell, H., "Identification of Glass Fragments," <u>Journal of Forensic Sciences</u>, 9:246, 1964.

<sup>&</sup>lt;sup>37</sup>Tryhorn, <u>op</u>, <u>cit</u>, p, 417.

<sup>33</sup>McCall, op. cit., p. 113.

<sup>&</sup>lt;sup>39</sup>Kirk, Crime Investigation, op. cit., p. 247

<sup>40</sup> Nickolls, L. C., The Scientific Investigation of Crime, London: Butterworth & Co., 1956, p. 68.

used on other types of glass. The glass is finely powdered and mixed with an equal volume of finely powdered aluminum chloride and twice the volume of Specpure graphite. This is then arced using a current of four to six amperes.

O'llara and Osterburg<sup>41</sup> state that spectrographic comparisons are of greater value in proving differences than identity because of the similarities in composition of glass.

General. MacDonell<sup>42</sup> describes methods for analysis to determine the chemical composition of glass and for physical properties such as refractive index, density, and coefficient of thermal expansion. Some of these methods give greater accuracy than the usual ones, however, most of them require larger samples than are available in most criminal cases. The development of these methods for use on glass fragments would be quite helpful.

<sup>410</sup> Hara & Osterburg, op. cit., pp. 304, 600
42 MacDonell, op. cit., pp. 244-254.

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The most promising new development for analysis in the field of criminalistics is 'Neutron Activation Analysis.' Basically this method consists of preparing the sample, irradiation in an atomic pile, and then the detection of the radioactive elements by a Geiger counter. Much greater sensitivities are possible than with standard analytical methods. 43

For the most part, this work is still in the experimental stage and the equipment is available in only a few locations. Preliminary studies on glass show very promising results; on a limited number of samples, this method showed very marked differences. 44

<sup>43</sup> Smith, H., "Estimation of Arsenic in Biological Tissue by Activation Analysis,"

Analytical Chemistry, 31:1361-1363, 1959.

<sup>44</sup>Ruch, R., et. al., "Neutron Activation Analysis in Scientific Crime Detection," <u>Journal of Forensic Sciences</u>, 9:123-129, 1964

## III. LITERATURE ON EVALUATION OF GLASS

Tryhorn comments on the evaluation of glass fragments and states that only by performing a great variety of tests and analyses on the samples being compared "can any opinion expressed as to their being of common origin be raised to a high probability value of correctness."45

The earliest reported study on evaluation was

46

He reports examining

sixty-five glass samples using refractive index,

specific gravity, and ultraviolet fluorescence.

He was attempting to determine if any of the sixty
three samples could not be distinguished from a known

and unknown in a particular case. (These had

previously been found to be similar in the above

properties.) In comparing the refractive index of

these two samples with the indices of twenty-two of

the sixty-three, it was found that four had similar

indices while twelve were lower and six higher.

<sup>45</sup>Tryhorn, op. cit., p. 419.

<sup>46</sup> Marris, op. cit., pp. 636-687.

Examination of flucrescence showed four of these twenty-two to be similar to the pair in the case. Combining these two properties, only one of the twenty-two could not be differentiated. However, upon comparing these samples by specific gravity, it was found that none of the twenty-two were similar. The other forty-one samples were also found to be different in specific gravity from the samples in the case, so that all of the samples could be differentiated from the two in the case. Apparently no attempt was made to determine if all of the remaining sixty-three could be individualized.

Another study was made in New Zealand on head-lamp-glass by Nelson. 47 He examined refractive index, specific gravity, color, ultraviolet fluorescence, dispersion, and hardness to make the differentiations. Out of fifty samples, only two could not be individualized.

Gamble et. al.  $^{48}$  used refractive index and specific gravity to make comparisons of glass from

<sup>47&</sup>lt;sub>Nelson, op. cit.</sub>, p. 388.

<sup>48</sup> Gamble et. al., op. cit., p. 421.

various sources such as bottles, windows, automobiles, eyeglasses, lights, household, and mirrors. Their conclusion was that all one hundred samples could be individualized by the use of refractive index and specific gravity.

Another study was made by Roche and Kirk<sup>49</sup> on fifty samples of brown bottle glass. This study also used only refractive index and specific gravity, and their results were that all but two of the fifty samples could be differentiated by these two properties. Similar results were reported by Green and Burd<sup>50</sup> in that after examination of fifty samples of glass from headlights by refractive index and specific gravity, only two could not be individualized.

A report in the FBI Law Enforcement Bulletin<sup>51</sup> shows graphically the results of the examination of two hundred samples of window glass in relation to refractive index and specific gravity. A great percentage of these samples are apparently individualized while some of them are not. Unfortunately there is no numerical description of the results.

<sup>&</sup>lt;sup>49</sup>Roche & Kirk, <u>op</u>. <u>cit</u>., p. 171.

<sup>&</sup>lt;sup>50</sup>Greene & Burd, <u>on</u>, <u>cit</u>., p. 89.

<sup>51</sup>Anon., "Don't Overlook Evidentiary Value of Glass Fragments," FBI Law Enforcement Bulletin, 33:19-22, October, 1964.

#### CHAPTER III

#### COLLECTION OF SAMPLES

Two types of samples were used in this study. The first was used to determine how much variation there was in the physical properties, such as refractive index, specific gravity, and ultraviolet fluorescence, of fragments taken from different parts of one piece of glass. The second type was random samples used to determine the variations in the above properties of window glass when the glass comes from different sources.

## I. VARIATIONS IN ONE PIECE

The samples used to determine the variations in physical properties of fragments from one piece of glass were obtained from two commercial glass sources. Stripes from two to four inches wide and about three feet long were obtained. It was felt that only rarely would a questioned and known sample come from a greater distance apart than three feet. This is because the average window size is under three feet, and when a large display type window is involved, seldom is the entire window broken; usually it is only a small area.

Some of these samples were assumed to be from the same batch. This assumption was based on the

facts that the samples were trimmings from pieces which came from the factory packaged together and all of the strips were found to have the same refractive index and specific gravity. Two other groups were obtained so as to have a high degree of probability of being from different batches. These were obtained at different times, about two months apart, and were of different strengths (thickness). All of these samples were smooth finished, uncolored window glass.

#### II. VARIATIONS FROM RANDOM SOURCES

The samples examined to determine the differences in physical properties in glass from different sources were taken from window glass submitted to the St. Louis Police Laboratory in actual cases (mostly burglaries).

It was felt that this method of collecting the samples would produce as nearly a random sample as possible. This should also be representative of the situation as it would occur in cases examined by a laboratory.

Precautions were taken to prevent any duplication of samples. For instance, the samples were collected over a two year period and when more than one sample of glass was submitted in a case, only one was selected as a sample.

Basically the samples consisted of smooth surfaced, colorless window glass of varying thicknesses. There were a few exceptions to this in that there were two samples which had one side that was not smooth; these were the type of glass used to reduce visibility. Two other samples were clear glass with wire reinforcement to increase the strength of the glass.

#### CHAPTER IV

#### EQUIPMENT USED

#### I. REFRACTIVE INDEX

Refractometer. An American Optical Abbe type refractometer was used to determine the refractive index of the oils. The light sources mentioned below were used with this refractometer.

Microscope and hot stage. The microscope used was a Leitz Ortholux equipped with a 10% objective and 10% eyepieces. However, the microscope had a tube factor of 1.25 so that the total magnification used was 125%. The hot stage was a Leitz hot stage fitted with a centigrade thermometer which was graduated in 0.5 degree increments so that it could be read to 0.1 or 0.2 degree accuracy. This hot stage was equipped for cooling water so a reversible syphon was set up to facilitate cooling the stage between runs.

<u>Light sources</u>. A sodium lamp<sup>1</sup> was used as the basic light source. This furnishes the standard solium lines of 588.9 and 589.5 millimicrons. Light sources of other wavelengths were obtained by the

 $<sup>^{\</sup>mathrm{l}}$  Manufactured by the George W. Gates Co. and available from most laboratory supply houses.

use of filters with a standard microscope lamp which is furnished as an accessory to the above microscope.

The blue filter used was a Klett number 42 which transmits light from 400 to 450 millimicrons. The red filter was a Klett number 66 which transmits light from 640 to 700 millimicrons. Plots of the actual transmission of these filters run on a spectrophotometer<sup>2</sup> are found in Appendix A.

Refractive index oils. The oils used in this study  $^3$  ranged in refractive index from 1.512 to 1.538 in increments of 0.002. The accuracy of the labeled refractive index (at  $25^{\circ}$ C with sodium light) is given as  $\frac{1}{2}$  0.0002 and the temperature coefficient for the above range of oils is 0.0004 per degree centigrade.

#### II. SPECIFIC GRAVITY

Only comparisons of specific gravity were to be made, not absolute measurements, so gradient density tubes were used. These were made from glass tubing, OD 7mm., about one foot long according to the method described by Kirk. The procedure varied from this method in that the specific gravity range of the

<sup>&</sup>lt;sup>2</sup> Beckman DU spectrophotometer.

<sup>&</sup>lt;sup>3</sup> R. P. Cargille Laboratories, New York 6, N. Y.

<sup>4</sup> Kirk, Paul L., <u>Density and Refractive Index</u>, Springfield: Charles C. Thomas, 1951, pp. 18-22.

liquids was narrowed from 1.499.2.890 to 2.40-2.60 to provide greater sensitivity. There were five liquids in increments of 0.05 used to cover this range.

The sections of tubing were sealed at one end by heating and the liquids<sup>5</sup> were layered into the tubes starting with the heaviest liquid. Layers 2 1/2 inches deep were used for the heaviest and lightest liquids and 1 1/2 inch layers were used for the intermediate liquids. The tubes were then allowed to stand overnight before using.

## III. ULTRAVIOLET FLUORESCENCE

The ultraviolet lights used for observing the fluorescence of the samples were two Mineralights; one equipped with a model SL3660 filter which transmits long wave ultraviolet, and the other with a model SL2537 filter which transmits short wave ultraviolet light.

Also used for the examination of fluorescence was the Leitz Ortholux microscope mentioned above.

This microscope was equipped with hydrogen discharge lamps as ultraviolet light sources for incident and transmitted light.

 $<sup>^5\</sup>mbox{Available from Microchemical Specialties,}$  Berkeley, California

## IV. SPECTROGRAPHIC COMPARISON

The spectrographic comparisons were made using a Bausch and Lomb Large Littrow spectrograph. This unit used DC arc with the power supplied by a DC generator. The spectrographic plates were Kodak Spectrum Analysis #1 plates, and the electrodes were Ultra Carbon's numbers 1992 and 2509 which were a semi-micro type.

#### CHAPTER V

#### EXPERIMENTAL METHODS

A description of the specific methods used in this study is given in this chapter and the procedures used for examining the samples are given in Chapter VI.

## I. REFRACTIVE INDEX

Determination of numerical value. The method used for the determination of the refractive index values is described by Allen as a modification of the Emmons Double Variation Method. This method is based on the fact that while the refractive index of the oils changes relatively rapidly with temperature  $(0.0004/^{\circ}C)$ , the refractive index of glass changes very slowly  $(0.00001/^{\circ}C)$  at temperatures between 25 and  $100^{\circ}C$ . Therefore it is possible to use an increase in temperature to decrease the refractive index of the medium in which the glass is immersed rather than the classic method of mixing two liquids. Since the temperature coefficient of the oils is constant, the temperature change may be used to calculate the refractive index of the glass sample.

<sup>1</sup>Allen, Roy M., <u>Practical Refractometry</u>. New York; R. P. Cargille Laboratories, 1954, pp. 9-10

Several drops of an oil of known refractive index were placed in the depression of a glass slide and the glass fragment to be examined was then immersed in the oil; it should be completely immersed in the oil. If the glass could not be completely covered, care had to be taken to make the readings using a portion of the glass which was well covered by the oil so as to obtain proper results. In the determination of refractive index by this method it was found that sample size was not critical other than the sample should be capable of being covered by three or four drops of oil.

Determining the correct oil to use on a specific sample was done by trial and error. From the direction of movement of the Becke line one could determine whether the refractive index of the oil was higher or lower than that of the glass; the brightness of this line was used as an indication of how much too low or too high. The direction of travel of the Becke line was toward the material of higher refractive index when the distance between the sample and the objective is increased. Since the refractive index of the oils decreases with an increase in temperature, the correct oil to use was one whose refractive index was slightly higher than that of the glass. It was found that for the best results, the oil used should

equal the glass fragment in refractive index with no more than a five to ten degree rise in temperature. Since the temperature coefficient of the oils was  $0.0004/^{\circ}$ C, the correct oil was one whose refractive index was no more than 0.004 higher than the glass.

It was essential for accurate results to use a hot stage which was capable of accurately showing on the thermometer the temperature of the oil. It was found that it was necessary to control the heating rate so that it was not too fast as this would not allow the entire mass of the hot stage including the oil to be at the temperature shown on the thermometer; also an error in reading the thermometer results in that there would be a temperature rise between the time the Becke line disappeared and the thermometer was read. A heating rate of one degree centigrade per 35 to 40 seconds was found to be quite satisfactory.

The temperature of the hot stage was raised and when the Becke line completely disappeared, the temperature was noted again when the Becke line first reappeared. The average of these two temperatures was used to calculate the refractive index of the glass fragment in the following manner: The temperature rise in degrees above 25°C was multiplied by 0.0004 (the temperature coefficient of the oil) and the result subtracted from the refractive

index value of the oil used.

Comparative determination of refractive index.

The samples of glass to be compared were immersed in a few drops of oil in the depression of a glass slide.

The temperature was then increased, using the same rate as above, until the Becke lines disappeared and changed direction. If this occurred simultaneously in the samples being compared, they were of the same refractive index.

## II. SPECIFIC GRAVITY

Chapter IV were used for specific gravity comparisons. All of the samples to be compared were placed in a tube at one time. However, they had to be added a short time apart to avoid confusion. Approximately equal sized samples were used; samples weighing about one half to one milligram were found most suitable. Time had to be allowed for the samples to reach a stable point in the tube; from five to ten minutes was found to be adequate. Gentle tapping of the tube usually dislodged any fragments which stuck to the side of the tube or to other fragments of glass. By the use of duplicate samples added a short time apart, it was possible to be certain that the glass had reached an equilibrium point with the gradient rather

than sticking to the side of the tube or to another fragment of glass.

## III. ULTRAVIOLET FLUORESCENCE

The fluorescence of the samples was observed using both the short and long wavelength lights. It was necessary to make the observations in a room that was completely darkened and against a background which was non-fluorescent. The few samples which appeared to have any fluorescence were then examined under the microscope using the hydrogen discharge lamp as the light source. Since the filtering was much better than with the hand lamps, visible light was virtually eliminated. This minimized the problem of confusing reflection of visible light with fluorescence.

#### IV. SPECTROGRAPHIC COMPARISON

Samples which were approximately the same size as is usually found in cases (under two millimeters in the longest dimension) were used. These were weighed so as to have a more accurate record of sample size for future reference, placed in the electrodes, and arced for ten seconds at four to five amperes using position V on the large Littrow spectrograph.

The plates were developed in Kodak D-3 developer for four minutes at approximately  $25^{\circ}\text{C}_{\bullet}$ 

rinsed in a stop bath, fixed in Kodak Rapid Fix for five minutes, washed for thirty minutes and allowed to dry.

The comparisons were made by direct visual comparison of the spectra. It was found that spectrographic comparison did not supply sufficient information to continue its use as an experimental method.

#### CHAPTER VI

#### EXPERIMENTAL PROCEDURE

The basic purpose of this study was to obtain more information about the blue of window glass fragments as evidence by determining how much duplication there is in the physical properties (such as refractive index, specific gravity, and ultraviolet fluorescence) of fragments from random sources. It was found that to properly evaluate these properties, it was necessary to determine the reproducibility, or experimental variation, of the methods used. Also it was necessary to determine how much variation one might expect to find in these properties within a single pane or sheet of glass. With this information as a background, the random samples were examined and the results evaluated.

#### I. EXPERIMENTAL VARIATION IN

#### THE METHODS

Refractive index. The only property for which numerical values were determined was refractive index.

Therefore a study was made as to the experimental variation and reproducibility of the method used.

The refractive indices of the oils used were measured at  $25^{\circ}\text{C}\pm0.5^{\circ}$  with an Abbe refractometer

using a sodium light for the light source. These measured values were compared with the labeled values to insure that there had been no contamination or deterioration of the oils. The results are shown in Table IA, Chapter VII.

Since an attempt was made to determine the refractive index of glass fragments using the red and blue portions of the spectrum, and no oils calibrated for these wavelengths were available, the refractive indices of the oils used in this study were determined using the red (#66) filter with a tungsten lamp. An attempt was made to use the blue (#42) filter in the same manner, but the line on the refractometer could not be established clearly enough to allow accurate measurements.

Several oils were available which were two to three years old and comparable new oils were also available, so for general information, a comparison was made of these using the Abbe refractometer. The results of this comparison are shown in Table IC, Chapter VII.

The experimental variation or reproducibility of the refractive index method used was determined in the following manner. Twelve of the random samples were selected over the most common range of indices

(1.5159 to 1.5258) and their indices determined at three different times; each time working without knowledge of the previously obtained results. These results are shown in Table II. Chapter VII.

The validity of using absolute values of refractive index to compare glass fragments rather than the more commonly used direct comparison method (with the samples in the oil at the same time) was determined by running direct comparisons of glass fragments having close values of refractive index. This was done to determine if samples which could not be differentiated by absolute value comparison could be differentiated by direct comparison. It was found that the direct comparison method was no more discriminating than the absolute value method.

In determining refractive index values using the filtered light, it was found that with the red filter, although the refractive indices were different from those determined with the sodium light, the values were not reproducible within usable limits. Therefore, only direct comparisons were made with this light source. With the blue filter it was found that the Becke line faded and changed direction over such a wide temperature range that this filter was not used further in experimental work. These

problems were probably due to the filters not producing monochromatic light.

Specific gravity. Since the specific gravity method was one of comparison the only reproducibility involved was to determine if duplicate samples would level exactly in the tubes within a reasonable time. This was done by placing in a tube duplicate samples of three glasses known to equilibriate with the gradient in widely different levels in the tube, and being certain that the duplicate samples leveled within a reasonable time.

Ultraviolet fluorescence. The observation of ultraviolet fluorescence involved only visual examination and comparison. The main point was to be objective in the observations and through examination of a number of samples determine what the differences in fluorescence actually were in regard to color and intenseness.

II. RANGE OF VARIATION IN PHYSICAL PROPERTIES
OF FRAGMENTS FROM ONE PIECE OF GLASS

The samples collected as described in Chapter III were examined by refractive index, specific gravity, and ultraviolet fluorescence. One group of samples consisted of seventy-five strips of glass assumed to be from one factory batch. About ten per cent, seven pieces, of these were selected at random. Samples were taken from each of the seven and from both ends of three of the pieces making a total of ten samples. These were then compared by refractive index, specific gravity, and ultraviolet fluorescence to determine the variations in these properties.

The other two groups of six were sampled and examined in the same manner. That is, samples were taken from each of the six and from both ends of three. Each set of nine samples was then subjected to the same three examinations and comparisons.

These results are found in Section II, Chapter VII.

# III. DIFFERENCES IN CLASS FROM RANDOM SOURCES

One hundred twenty-nine samples collected as described in Chapter III were examined in the following manner. The refractive index of each of the samples was determined using a sodium lamp as the light source. Refractive index was selected as the first property to be compared since it was the only one with a numerical value that could be cataloged for subdividing the samples into groups for further comparison.

Examination of the refractive index data showed that many of the samples tended to group together by refractive index. Most of the samples which could not be differentiated fell into six groups containing from five to ten samples each for a total of forty-three samples.

Each of these six groups was subjected to comparison by specific gravity and fluorescence. Any samples in the groups which still could not be differentiated were then subjected to comparative determination of refractive index at a second wavelength, specifically the red region of the spectrum. With this filter it was found that an oil 0.004 higher in refractive index than the oil

used with the sodium lamp was the correct one to use.

Of the remaining eighty-six samples those which could not be individualized by refractive index were compared in the same manner as above.

## CHAPTER VII

## EXPERIMENTAL RESULTS

The results of the experimental procedures.

Chapter VI, are given in this chapter and the conclusions drawn as a result of this study are given in Chapter VIII.

## I. EXPERIMENTAL VARIATION IN

#### THE METHODS

Refractive index. Table IA gives the comparison of the labeled values for the refractive index oils with the values determined with the Abbe refractometer using a sodium lamp as the light source. With the equipment used it was possible to control the temperature at  $25.0^{\circ}$ C  $\neq 0.5^{\circ}$ C.

TABLE IA

COMPARISON OF MEASURED REFRACTIVE INDICES

WITH LABELED VALUES

Labeled RI (25.0°C)	Measured RI (25.0° <u>7</u> 0.5°C)	Variation
1.5120	1.5119	-0.0001
1.5140	1.5139	-0 <sub>•</sub> 0001
1.5160	1.5160	0.0000
1.5180	1,5181	<b>4</b> 0.0001
1.5200	1.5198	-0.0002
1.5220	1.5218	-0.0002
1.5240	1.5238	<b>-0.</b> 0002
1.5260	1.5260	0.0000
1.5280	1.5277	-0.0003
1.5300	1.5297	-0.0003
1.5340	1.5338	-0.0002
1.5390	1.5381	<b>≠</b> 0.0001

The stated accuracy of the oils is  $\neq 0.0002$ , so when the temperature variation is considered. (The temperature coefficient of the oils is  $0.0004/^{\circ}C_{\bullet}$ ) the refractive index oils were found to be as labeled. There apparently had been no contamination or deterioration.

Table IB gives the comparison of the labeled refractive index values of the oils with the values obtained using the red (#66) filter. Again the values were measured at  $25.0^{\circ}\text{C}\neq0.5^{\circ}\text{C}$ .

TABLE IB
REFRACTIVE INDICES OF THE OILS WITH
SODIUM LAMP AND WITH RED FILTER

Red Filter RI	Variation	
1.5121	<b>≠</b> 0 <b>,</b> 0001	
1.5163	40.0003	
1,5199	-0.0001	
1.5237	-0.0003	
1.5260	0,0000	
1.5299	-0.0001	
1.5378	-0.0002	
	RI 1.5121 1.5163 1.5199 1.5237 1.5260 1.5299	

As may be seen by examination of these values, there was little difference in the values of the oils at two different wavelengths. A similar comparison was attempted using the blue (#42) filter, but the line

on the refractometer could not be established clearly enough to allow accurate measurement. These results do not agree with the expected results for refractive indices at different wavelengths; this is because the filters did not give monochromatic light. However, when these filters were used to determine the refractive index of glass, the values obtained were different from the sodium light values, as would be expected.

Table IC shows the effect of the age of the oils on their stability. Compared are refractive index values of oils which have been recently purchased with similar oils about two to three years old.

TABLE IC
EFFECT OF AGE ON OILS

Labeled RI	New Oils RI	Old Oils RI	Variation
. 1.5160	1.5160	1.5162	<b>≠</b> 0.0002
1.5180	1.5181	1.5177	-0.0004
1.5220	1.5218	1.5215	-0.0003

From this limited sample it would appear that the oils are reasonably stable over a period of time.

When making comparisons for similarity of refractive index, it is essential to use the same oil for all samples. This automatically compensates for

the above-mentioned sources of error. These variations in the refractive indices of the oils must be taken into consideration, however, when making comparisons over a wide range of refractive index values.

Since the oils were found to be in good condition—within the stated accuracy—the labeled values were used for the following results.

Table II gives the reproducibility or experimental variation of the refractive index method used. The reproducibility is a much more important factor than any error in the absolute value for refractive index since the accuracy of comparing samples rests on the ability to obtain reproducible results.

TABLE II

REPRODUCIBILITY OF REFRACTIVE INDEX MEASUREMENTS

Sample		Temp.	(°)	Refra	active Ind	dex
No.	1	2		1	2	3
15	29.3	29.4	29.3	1.5163	1.5162	1.5163
57	30.2	30.2	30.5	1.5159	1.5159	1.5158
85	29.8	29.7	29.9	1.5161	1.5161	1.5160
54	30.0	29.8	29.9	1.5180	1.5181	1.5180
63	29.7	29.6	29.5	1.5181	1.5182	1,5182
72	27.8	27.8	27.6	1.5189	1.5189	1,5190
96 .	29.4	29.2	29.1	1.5222	1.5223	1.5224
110	29.0	29.1	29.2	1.5224	1.5224	1,5223
115	29.0	28.9	28.9	1.5224	1.5224	1.5224
82	31.2	31.0	31.2	1.5255	1.5256	1.5255
123	31.5	31.6	31.5	1.5254	1.5254	1.5254
127	30.7	30.6	30.8	1.5257	1.5258	1.5257

From the above table it may be seen that the experimental variation in temperature in determining refractive index may be as much as  $0.3^{\circ}$ C. However, this amount of temperature variation changes the refractive index only 0.00012.

In examining Table II it will be noted that in many instances a  $0.1^{\circ}$ C temperature variation resulted in a refractive index variation of 0.0001 when the variation should only be 0.00004, or a  $0.3^{\circ}$ C variation changed the refractive index 0.0002 when the variation should only be 0.0001. For instance, with sample 15 the first temperature is  $29.3^{\circ}$  and the second is  $29.4^{\circ}$ . When converting the temperature rise over  $25.0^{\circ}$ C (4.3 and  $4.4^{\circ}$  respectively) to refractive index change, the following occurs:

For 4.3° temperature rise---

4.3x0.0004 = 0.00172 Considering significant figures, this becomes 0.0017. Subtracting this from the RI of the oil, 1.5180, the calculated RI is 1.5163.

For 4.4° temperature rise--

4.4x0.0004 = 0.00176Considering significant figures, this becomes 0.0018. Subtracting this from 1.5180 the calculated RI is 1.5162.

Because of this, temperatures make better comparison values than the calculated refractive indices.

There are three basic reasons for this variation in temperature. One is that the thermometer is graduated only to 0.5 degree so that the reading accuracy is  $\neq 0.1$  degree. Another reason is the time lag between the Becke line disappearing and the reading of the thermometer; this may be one or two tenths of a degree. The third reason for the variation is the range over which the Becke line disappears and reappears. With a sample which gives a sharp, clear Becke line, the line will reappear within 0.3 to 0.5 degree. This allows for good reproducibility. Sometimes, however, it may not be possible to find an edge on a fragment which will give a sharp, clear Becke line and then the range may be as much as 0.7to 0.8 degree for the disappearance and reappearance of the Becke line.

Specific gravity. Checking of the gradient density tubes showed that duplicate samples leveled exactly. It was found necessary to consider sample size as with small fragments (less than half a milligram) well over fifteen minutes was necessary for the samples to reach equilibrium with the gradient. This was also true when tubes of greater sensitivity than given in Chapter VI were used; the samples took an unreasonable length of time to reach a stable point

in the tube. It was also difficult in these instances to determine exactly when the sample had reached equilibrium with the gradient. The more sensitive tubes were also very subject to outside factors such as drafts of air. When using tubes as described in Chapter IV and samples weighing from one half to one milligram, the time necessary for the fragments to reach a stable point in the tube was from five to ten minutes.

Ultraviolet fluorescence. It was found that none of the window glass samples examined showed any fluorescence. Some of the samples showed what appeared to be faint fluorescence, but closer examination showed this to be surface reflection. Using the microscope to examine these samples was found quite useful since the filtering was much better and visible light was virtually eliminated.

II. RANGE OF VARIATION IN PHYSICAL PROPERTIES

OF FRACMENTS FROM ONE PIECE OF GLASS

Refractive index. The refractive index values determined for the three groups of samples used in this portion of the study are shown in Tables IIIA, IIIB, and IIIC. The sample numbers indicate separate strips of glass and the attached 'a' after 1, 2, and 3 indicate the samples taken from the opposite end of that particular strip of glass.

TABLE IIIA

REFRACTIVE INDICES OF GROUP A

Sample No.	Temperature (°C)	Refractive Index
1	29.8	1.5181
la	<b>29.</b> 8	1.5181
2	29.3	1.5181
2 <b>a</b>	29.9	1.5130
3	29.7	1.5132
3a	29.9	1.5190
4	29.3	1.5181
5	30.0	1.5180
6	29.9	1.5180
7	29.7	1.5182

TABLE IIIB
REFRACTIVE INDICES OF GROUP B

Sample No.	Temperature (°C)	Refractive Index
1	32.5	1.5190
la	32.6	1.5190
2	32.4	1.5190
2a	32.4	1.5190
3	32,5	1.5190
3a	32.7	1.5139
4	32.7	1.5189
5	32.6	1.5190
6	32.7	1.5189

TABLE IIIC

REFRACTIVE INDICES OF GROUP C

Sample No.	Temperature ( <sup>O</sup> C)	Refractive Index
1	28.3	1.5247
la	28.2	1,5247
2	28.3	1.5247
2 <b>a</b>	28.4	1.5246
3	28.2	1.5247
3 <b>a</b>	28.3	1.5247
4	23.3	1.5247
5	28 <b>.3</b>	1,5247
6	$28  extstyle{\bullet}4$	1.5246

The refractive index values for groups A, B, and C show that the variation in refractive index for fragments from different parts of a single piece are no greater than the experimental variation. From the limited sample run, it would appear that this holds true not only for one piece, but also for glass from a single batch.

This lack of significant variation in different fragments from a single piece and pieces from the same batch would mean that any variation slightly greater than the experimental variation would be significant. It was determined that a temperature variation of 0.5 degree in the determination of the refractive index of a given set of samples would be sufficient to show non-identity or difference of source when the same oil is used for all samples.

Direct comparison of the refractive indices within each of the three groups using red filtered light showed no significant differences in the refractive indices at this wavelength within any of the three groups.

Comparisons of the specific Specific gravity. gravities within each of the groups A. B. and C showed no significant variations in fragments from the same piece or even in pieces from the same batch. Although there were slight variations in level within the groups, the variations were no greater than the experimental variation. This means that a difference in specific gravity would be cause for reporting non-identity of a given set of samples. A difference in the levels of the samples in the tube shows a difference in the specific gravity of the samples. This means that there must be a space between the samples even if it is only very slight. Because of this it was essential to use samples which were not too large (around one milligram) or the bulk of the samples would cause overlapping.

<u>Ultraviolet fluorescence</u> No differences were noted in ultraviolet fluorescence in any of these samples since none of them exhibited any fluorescence.

# III. DIFFERENCES IN GLASS FROM RANDOM SOURCES

Refractive index using sodium light. The refractive indices of the 129 random samples as determined using the sodium lamp are found in Appendix B. Of these 129 samples, only 27 could be completely individualized on the basis of their refractive index alone.

Specific gravity. As mentioned in the procedure, 43 samples in 6 groups were separated from the 102 samples which could not be individualized on the basis of refractive index. Each of these 6 groups covered only a narrow range of refractive indices, but it was possible to individualize an additional 23 samples by the use of the gradient density tubes. There were 20 which could not be individualized; these 20 were made up of 5 groups of 2, one group of 3, and one group of 7.

The 59 samples which were not included in the above groups were also compared in the gradient density tubes. As a result, an additional 43 samples could be individualized. The 16 which could not fall into 5 groups of 2 and 2 groups of 3.

To summarize, of 102 samples which could not be individualized by refractive index, 66 could be individualized by specific gravity. The 36 which

could not be individualized fell into 10 groups of 2.

3 groups of 3. and one group of 7.

Refractive index using filtered red light.

The samples which could not be differentiated by specific gravity were compared as to refractive index using the red (#66) filter and a tungsten lamp as the light source. None of the samples could be further differentiated with two exceptions. In both of these instances, there was slight overlapping of the fragments in the gradient density tubes, but not exact leveling. As a result, the refractive index comparisons served as a confirmation of the results of the specific gravity comparisons rather than a means of differentiation.

<u>Ultraviolet fluorescence.</u> Since none of the samples exhibited any fluorescence, it was not possible to make any differentiations on this basis.

#### CHAPTER VIII

#### SUMMARY AND CONCLUSIONS

First discussed in this Chapter are the conclusions formed regarding the examination of the samples by the determination of refractive index, comparison of specific gravity, and examination under ultraviolet light. Following this, the general conclusions of the study with regard to the use of window glass fragments as evidence are given.

#### I. REFRACTIVE INDEX

From the distribution of the random samples as shown in Figure II. Appendix C. it may be seen that glass of some refractive indices is more common than others.

It was also noted from this distribution that the samples tend to fall into two major groups with a lack of samples having refractive indices between 1.5197 and 1.5214. That is, there is one group of samples having refractive indices from 1.5112 to 1.5197 and another group with values from 1.5214 to 1.5295, with two single samples of higher refractive index. This is shown graphically in Figure II. Appendix C. This graph shows the two groups as two almost normal distributions.

An examination of Figure 1 in "Don't Overlook Evidentiary Value of Glass Fragments" shows this same effect of grouping by refractive index in that no samples are shown between refractive index values of 1.5190 and 1.5204.

From the limited information available about the samples, no conclusions could be drawn as to the reason or reasons for this effect.

There is apparently little correlation between refractive index and thickness of glass sheets.

(Whether it is single, double, or triple strength.)

This is shown in Figure III, Appendix D, which is a plot of refractive index versus the thickness of the glass samples. There is a slight correlation in that only the thinnest samples tend to have the lowest refractive indices and the thickest samples tend to have a greater proportion of the higher refractive indices. The middle range of thicknesses, three to six millimeters, are the most common and are represented in almost the entire range of refractive indices.

<sup>&</sup>lt;sup>1</sup>Anon., "Don't Overlook Evidentiary Value of Glass Fragments," FBI Law Enforcement Bulletin, 33:19-22, October, 1964.

It was noted that wire reinforced or special type of glasses such as those having irregular surfaces tended to have refractive indices in the higher group. There were four samples of this type and all had refractive indices of around 1.524.

The range of refractive indices of window glass was naturally found to be much narrower than the 1.469 to 1.566 range for assorted glasses reported by Gamble et. al.<sup>2</sup> The range for the window glass samples was from 1.511 to 1.535, with most of the samples between 1.512 and 1.530. This corresponds very closely to the 1.513 to 1.529 range reported by the Federal Bureau of Investigation.

The refractive index comparisons using red filtered light were found to be of little value for this study since these comparisons were not responsible for any separations which were not indicated by either the refractive index or specific gravity comparisons. This was probably due to the transmission range of the filter. This is an area which is deserving of further study because with monochromatic light at various

<sup>&</sup>lt;sup>2</sup>Gamble et. al., "Glass Fragments as Evidence,"

<u>Journal of Criminal Law, Criminelogy & Police Science,</u>

33:419. 1943.

P. 20. Bulletin, op. cit.

wavelengths, it would be possible to make further differentiations as is described by Grabar and  $\frac{4}{2}$ 

# II. SPECIFIC GRAVITY

It was noted that within each of the six groups the samples which could not be differentiated by specific gravity also were close in refractive index. For instance, samples 82 and 105 could not be separated, and these two had a difference of only 0.0001 in their refractive indices. The same was true of samples 104, 106, and 128. In all cases where samples were at the exact same level in the gradient density tube, there was also no possibility of differentiation by refractive index, within the limits of the method used.

Specific gravity was found to be the more sensitive method for making separations since many samples which could not be individualized by refractive index could be by specific gravity. This agrees with

<sup>&</sup>lt;sup>4</sup>Grabar, D. G. & Principe, A. H., "Identification of Glass Fragments by Measurements of Refractive Index and Dispersion," <u>Journal of Forensic Sciences</u>, 8:54-67, 1963.

the findings of Kirk and Roche.<sup>5</sup> It is however, necessary to compare both refractive index and specific gravity since it was noted that some of the groups overlapped so that some samples with different refractive indices did have similar specific gravities.

Tubes having a specific gravity range of from 2.40 to 2.60 and a liquid depth of 9.1/2 inches were found to be quite satisfactory for window glass. One disadvantage is that this range will not cover all types of glass so it is necessary to have more than one type of tube available.

When comparing small numbers of samples
(such as evidence in cases) specific gravity forms
the best screening method for comparing a given set
of samples. This is because it takes very little
time and provides separations in most cases in which
the glasses are not similar. If the samples do not
level in exactly the same spot in the tube, they
should be considered not similar. Any samples found
to be similar should then be compared as to refractive
index. When making comparisons of large numbers of

<sup>&</sup>lt;sup>5</sup>Roche, G. & Kirk, P. L., "Applications of Microchemical Techniques," <u>Journal of Criminal Law, Criminology</u>, and <u>Police Science</u>, 33:169, 1947.

samples, such as in this study, it is more practical to determine refractive index first since it is difficult to keep track of large numbers of samples in a specific gravity tube, especially if they are relatively close in specific gravity.

# III. ULTRAVIOLET FLUORESCENCE

Ultraviolet examinations were found to be of no value in comparing samples of window glass since none of the samples exhibited any fluorescence. Its principle value would be in making original eliminations since it was found that certain non-window glass samples such as optical glass and some ashtrays did fluoresce. However, in small samples such as might be found in actual cases, it was difficult to detect fluorescence. This would create some doubt as to the usefulness of fluorescence in comparing small samples.

A possible area for future study would be to determine if this absence of fluorescence is typical of window glass and if so, would this form a means of narrowing the type of a questioned sample of glass.

#### IV. GENERAL

of the 129 random samples examined in this study, 27 could be individualized by the determination of refractive index using a sodium lamp as the light source. The remaining 102 samples were spread over a range of index values such that while an individual pair having refractive index differences of around 0.0002 could be differentiated, there was enough overlapping of these values that the samples could not be individualized. The differentiation of a specific pair of samples by refractive index was based on their having at least a 0.5°C difference in the temperature rise in the determination of the index values.

An additional 66 samples could be individualized after placing samples with close refractive indices in gradient density tubes. Duplicate samples were found to level exactly so a difference in level in the gradient tube was considered to show a difference in source.

The total number of differentiations which were possible was 93 out of 129. This means that out of the 129 samples, 36 could not be individualized.

Most of these, 20 samples, were in groups of 2. One group of 7 could not be differentiated; this group

had a refractive index which was in a very common range of indices.

This number which could not be individualized is greater than that reported in other studies. Gamble et. al. reported no duplication out of one hundred samples. However, this was on a wide variety of samples which included only 25 samples of window glass. This would increase the chance for complete differentiation. Roche and Kirk and Greene and Eurd each repoted only two duplications out of fifty samples of bottle glass and headlight glass respectively. Nelson reported two pairs indistinguishable out of fifty samples of headlamp-glass. From this it would appear that there is greater duplication in window glass than in these three types.

Gamble et. al., op. cit., p. 419.

<sup>7</sup> Roche & Kirk, op. cit., p. 171.

Greene, R. & Burd, D., "Headlight Glass as Evidence," <u>Journal of Criminal Law, Criminology</u>, and <u>Police Science</u>, 40:89, 1949.

Nolson, D. F., "The Identification of Lucas 700 Headlamp-glass Fragments by their Physical Properties," Analyst, 84:363, 1959.

As is shown in Figure II. Appendix C. window glasses of certain refractive indices are more common than others. This is also true of the specific gravities, with the extreme light and heavy samples being less common. With refractive index there is an odd situation in that not only are the extreme high and low indices less common, but also there is a group in the center of the range which is also uncommon. The sample size in this study is too small to draw any valid conclusions as to which refractive indices are more rare, although a definite pattern has developed. This is an area that would benefit from further study. That is. to determine which indices are common and which ones are rare. This is a fact which should be considred when making refractive index comparisons of specific samples since more value could probably be placed on samples which have an uncommon refractive index.

Another area which would warrant further study would be to obtain more data on the variations within batches; then any methods which could be developed to exploit these variations would indeed be helpful.

A major problem is that most fragments are too small to determine their type (window, bottle, auto, etc.). Any information such as shape or thickness

which would help to determine the type of glass from which the fragments came, would also be very useful and helpful. The fact that the window glass fragments did not fluoresce while some other types (optical glass and some ashtrays) did is a factor which might be worthy of further study.

As to the basic question of how much value to place on a particular set of samples, the answer will vary somewhat with each case as it does with virtually all types of evidence. There are a number of factors which enter into the final decision: Is it possible from examining the samples to determine its type? How well does it lend itself to refractive index and specific gravity determinations? Can these be accurately determined on this particular sample? Are these values of a common or uncommon group? Is there anything unusual or unique about the samples, such as color or surface shape? These are all questions which must be answered before an evaluation is made.

In general from this study it would appear that any evaluation which must be based solely upon comparison of refractive indices and specific gravities could not be on a very positive basis. That is, it should not be stated that there is a high degree of

probability of the samples having come from the same piece of glass.

#### V. SUMMARY

Fragments from three different batches of window glass were examined and while it was possible to differentiate between each of the batches by either gradient density comparisons or refractive index determinations, samples within the same batch or from the same piece of window glass could not be differentiated.

Out of 129 random samples of window glass fragments, it was possible to individualize 93 by refractive index determination and gradient density comparison. The 36 samples which could not be individualized fell into 10 groups of 2, 3 groups of 3, and one group of 7.

The basic conclusion of the study was that any evaluation which must be made solely upon refractive index and specific gravity could not be on a completely positive basis.

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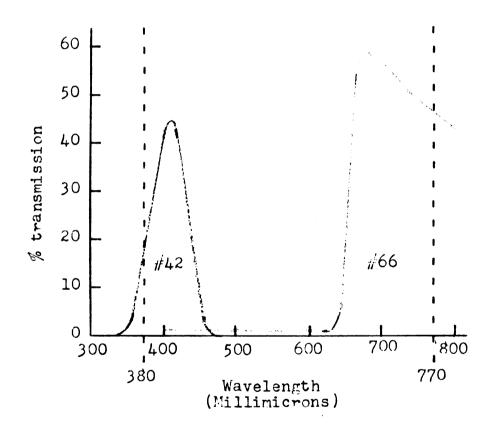
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# APPENDICES

# APPENDIX A

# TRANSMISSION OF FILTERS

Figure I shows the per cent transmission of the two filters used for the wavelengths range covering visible light. These curves were run on the actual filters using a Beckman DU spectrophotometer to measure the transmission values.



FICURE I

Per cent transmission vs. wavelength for Klett #42 (blue) filter and for Klett #66 (red) filter.

The #42 filter shows maximum transmission at 410 millimicrons and the #66 filter shows maximum transmission at 670 millimicrons. The dotted lines at 300 and 770 millimicrons show the limits for visible light as given by Judd. 1

The wavelength ranges given by the manufacturer are 400 to 450 for the #42 filter and 640 to 700 for the #66 filter.

Judd, Dean. <u>Color in Science and Industry.</u>
New York: John Wiley & Sons. 1953, p. 30.

APPENDIX B

REFRACTIVE INDEX OF THE RANDOM SAMPLES

No.	RI	ī	No.	RI -	No.	RI
1	1.5136		14	1.5153	87	1.5262
2	1.5227		45	1.5124	83	1.5256
3	1.5232		46	1.5134	89	1.5176
4	1.5163	i	47	1.5248	99	1.5246
5	1.5248	2	48	1,5354	91	1.5151
6	1.5150	4	49	1.5184	92	1.5233
7	1.5163	!	50	1.5247	23	1.5154
3	1.5242		51.	1.5270	94	1.5227
9	1.5197		52	1.5258	95	1.5264
10	1.5172		53	1.5159	96	1.5222
11	1.5197		54	1.5194	97	1.5224
1 <b>2</b>	1.5232		55	1.5245	<del>9</del> 8	1.5242
13	1.5228		56	1.5159	99	1.5229
14	1.5150		57	1.5159	100	1.5247
15	1.5163		53	1.5232	101	1.5223
16	1.5232		59	1.5190	102	1.5112
17	1.5180		60	1.5227	103	1.5278
18	1.5157		61	1.5230	104	1.5257
19	1.5228		<b>62</b>	1.5244	105	1.5256
<b>2</b> 0	1.5233		63	1.5191	106	1.5258
21	1.5156		64	1,5146	107	1.5240
22	1.5190		65	1.5192	108	1.5158
23	1.5127		66	1.5155	109	1.5262
24	1.5235		67	1.5153	110	1.5224
25	1.5161		68	1.5163	111	1.5132
26	1.5247		69	1.5169	112	1.5236
27	1.5227		70	1.5172	113	1.5241
28	1.5189		71	1.5131	114	1.5269
29	1.5183		72	1.5189	115	1.5224
30	1.5223		73	1.5144	116	1.5157
31	1.5139		74	1.5214	117	1.5153
32	1.5190		<b>7</b> 5	1.5292	118	1.5152
33	1.5188		76	1.5295	119	1.5152
34	1.5313		77 70	1.5130	120	1.5232
35 26	1.5177		78 70	1.5239	121	1.5244
36	1.5220		79	1.5162	122	1.5193
37 30	1.5220		30	1.5164 1.5189	123 124	1.5254 1.5244
36 20	1.5157		3 <b>1</b> 3 <b>2</b>		125	1.5189
39 40	1.5217 1.5154		3 <b>3</b>	1.5255 1.5243	126	1.5228
41	1.5134		34	1.5243	127	1.5257
42	1.5173		9 <del>1</del> 85	1.5161	128	1.5258
$\frac{42}{43}$	1.5239		36	1.5188	129	1.5181
49	1.0207	•	JU	T.0100	14/	1.0101

# APPENDIX C

# DISTRIBUTION OF THE RANDOM SAMPLES

# BY REFRACTIVE INDEX

The distribution of the 129 random samples listed in Appendix B is shown as a function of refractive index in Figure II.

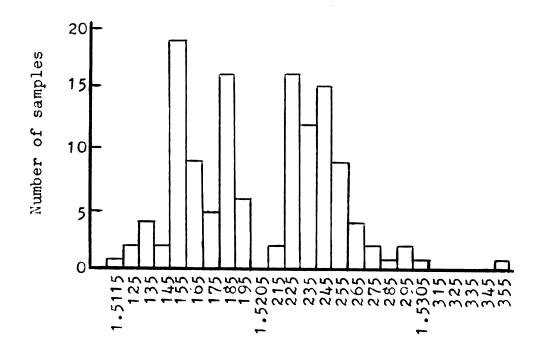


FIGURE II

Distribution showing number of samples vs. refractive index.

The refractive index values shown are those determined on the random samples collected for this study. These values were divided into intervals of 0.0010 with the 0.005 values as midpoints of the intervals. The 'Number of samples' is the number of samples in that particular interval.

APPENDIX D

CORRELATION OF REFRACTIVE INDEX

AND THICKNESS OF GLASS

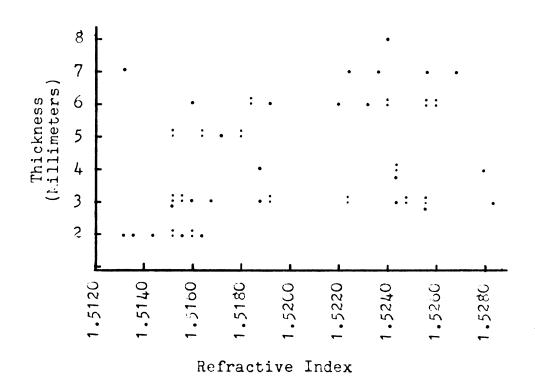


FIGURE III

Plot of refractive index vs. thickness in mm. for random samples of glass.

The thickness was measured, to the nearest millimeter, on a number of the random samples and plotted as above. Each sample is indicated by (•).

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