

## SECTION -7: EXAMINATION OF GLASS FRAGMENTS

### 7.1 Scope:

Glass, as a physical clue, is frequently encountered in various crimes, such as burglary, road accidents, murder, sexual assault, shooting incidents, arson and vandalism. The chips of broken glass from a window may be lodged in suspects shoes or garments during the act of burglary or 'particles of headlight glass found at the scene of hit and run accident may offer clues that confirm the identity of a suspected vehicle or glass may also be found on the clothing of an alleged assailant, where a bottle is used as weapon. Further, anytime, when there is a violence, bottles, window pane glass, mirrors, eyeglasses and other glass objects can be accidentally scattered and fragments of these can also adhere to the criminal's clothing or shoes. Thus, glass forms one of the evidentiary materials in many criminals investigations. The method of comparison of control and crime exhibits of glass fragments for source correspondence and study of various types of glass fractures are described.

### 7.2 Methods of examination

The forensic examination of glass may be undertaken in two steps. One is the physical comparison of suspected and control glass fragments for their identity and source correspondence and secondly, the examination of fractures on broken glass fragments and their reconstruction for determining the direction, angle and sequence of impact of projectile.

#### 7.3.1 Physical comparison

- 1) Appearance: Transparent/milky/ground/colored.  
Flat mirror/curved/patterned/polished.
- 2) Type of glass: Ordinary/ Pyrex/ borosilicate/laminated/ tempered/toughened/wired glass. Window pan/ glassware/automobile glass/ head light glass/ optical glass bottle glass etc

#### 7.3.2 Physical measurements

This include mainly two parameters namely, the thickness and curvature of the glass pieces under comparison. It is observed that the thickness of glass sheet vary significantly from one place to the other and do not have uniform thickness throughout. Therefore, it is very much desirable to obtain larger pieces of at least, the control glass samples and study the variations in their thickness before comparing the thickness of the control sample with that of the crime exhibit.

- a) Edge thickness: A micrometer is used to measure accurately the edge thickness of the glass fragments. Readings should be taken all around the broken edged to find out at which point, the crime exhibits matches with any portion of the broken glass. This will further help during the matching of surface patterns and other identifying characteristics, along the broken edges.
- b) Curvature: A spherometer is used to measure the radius of curvature of the glass fragments having curved surface. The radius of curvature of the fragment is calculated using the formulae.



$$R = (l^2/6h) + (h/2)$$

Where

l = the mean distance between the legs of the spherometer.  
h = height of the curved surface

### 7.3.3 Fluorescence under UV radiation

Some types of glass fluorescence under ultraviolet radiation with different colours which may be brown, violet, purple, blue or green etc., This examination has to be conducted in a dark room and the glass pieces to be exposed to UV radiation should be of similar size and thickness and they are to be thoroughly washed with acetone or any other suitable solvent to remove any grease or dirt. When there is a clear difference in the fluorescence of the two glasses, it indicates different sources of their origin. On the other hand, the similarity in fluorescence by itself cannot be a conclusive proof of source correspondence and further tests are to be conducted to arrive at the possible commonness of origin.

### 7.3.4 Physical matching

This is most conclusive proof of source correspondence, since no two fractures will ever be identical over any appreciable length. A complimentary lateral fit along the broken edges over a length of quarter inch (1/4) or more establishes that the two glass fragments were continuous before breakage. By naked eye or under a microscope, we should search carefully the edges of the samples, which will exactly fit into each other, taking into consideration, the factors contributing to the matching, such as general appearance, colour, edge thickness, shape of breakage, all the irregularities and striations near the broken surfaces. Lateral pressure has to be applied, in order to see that the small irregularities intermesh and hold the pieces together, so that we can feel the sense of exact fit along the broken edges. This is especially important for matching of smaller fragments.

In case of non continuous glass fragments, source correspondence is attempted by matching the invisible glass ream patterns, manufacturing marks, polish marks, striation on the surface of the glass fragments. Proper illuminations are to be used, such as point light source (2 mm<sup>2</sup>), diffused light transmitted light, incident or oblique light in order to obtain the exact patterns for comparison. A comparison microscope with lower magnification, could be used for matching these patterns. While glass pieces of the size of about 1 sq. cm could be sufficient for such comparisons. Larger pieces increase the probability of source correspondence.

In general, the majority of the glass evidence presented for forensic comparison is either too fragmentary or too minute, to permit comparison of this type. Hence, the physical properties of density and refractive index are used most successfully for characterizing glass particles. The characterizing properties of a glass depend not only on the constituent elements, but also on the manner in which the glass has been treated during manufacture. Glass of similar composition posses different properties depending upon the mechanical and heat treatments, which they have received. It is for this reason, that considerable importance is attached to physical properties of density and refractive index, as a means of comparing glass samples. These two fundamental parameters, though class characteristic in nature, provide sufficient data to the analyst, to exclude glass fragments that originate from different sources.

### 7.3.5 Density or Specific gravity

Glass from various sources such as windowpanes, automobile headlights, bottles and plate glass doors, all may have slightly different densities. The densities of various glass and related materials are given below:

Window glass, flat	=	2.47 to 2.56
Head light glass	=	2.47 to 2.63
Mica	=	2.6 to 3.2
Quartz	=	2.65
Glass, flint	=	2.9 to 5.9
Diamond	=	3.01 to 3.52

#### 7.4 Density measurements for bigger fragments of glass

i) Use a laboratory balance with sensitivity  $\pm 0.01$  gms or better, along with support rack provision, a 250 ml. capacity beaker and a piece of string about 1 meter in length.

ii) Tie the string around the glass fragment and suspend it from the pan support hook of the balance, after the preliminary adjustments are made with the balance, before weighing.

iii) Weigh the glass fragment in air to the nearest 0.01gm and record its value ( $W_1$ ).

iv) Place the 250 ml. beaker nearly filled with water on the beaker. Support and suspend the glass in the water. Adjust the glass height, so that it does not touch the walls of the beaker, inside the water. Weigh the glass fragment suspended in water to the nearest 0.01gm and record this value ( $W_2$ ).

v) Density of glass fragment is determined by the following relationship:

$$\text{Density of object} = (\text{Weight of object in Air}) / (\text{Loss of weight of object in water})$$

$$\text{Density of glass} = (W_1) / (W_1 - W_2)$$

Exhibit Glass fragments marked	$W_1$ (in gm)	$W_2$ (in gm)	$(W_1 - W_2)$ (in gm)	Density $(W_1) / (W_1 - W_2)$
Suspected -				
A				
B				
C				
D				
Control-				
1				
2				
3				
4				

vii) Based on the results obtained above, match the fragments lettered A,B,C,D with that of the fragments marked 1,2,3, and 4.



- ii) Turn on the sodium vapour lamp and adjust the microscope. If sodium lamp is not available, yellow filter, having maximum transmission of 590nm may be used to provide an approximate equivalent of sodium light (589nm).
- iii) Add one drop of one of the standard liquids on to the cavity slide. Place the suspected glass fragment in it and cover with a small cover glass. The cover glass ensures that the chip is completely immersed and minimizes any spurious lens effect from the surface of the drop.
- iv) Bring the glass fragment into focus. Most microscopes have a 16mm (2/3 inch) objective, which should be used with an eye piece of 10x or 20x magnification.
- v) Observe the Becke line or the bright halo surrounding the edges. If the halo seems to be indistinct, it may be more pronounced by closing down the condenser diaphragm.
- vi) Focus upward and downward to know the movement of Becke line in order to determine whether the glass fragment or the solution has the higher refractive index. When the refractive indices of the glass and the immersion liquid are the same, the chip will become almost invisible and it may be difficult to find and bring into focus the edges of the chip. By focusing up or down and following the Becke line, the examiner will be able to ascertain, whether the refractive index of the fragment is the same, as the liquid. If there is difference, to make an appropriate assessment, the next immersion liquid may be selected.
- vii) The first liquid may be removed with filter paper and next liquid flowed into the cover glass, which enters the cavity by capillary attraction. Any surplus can be absorbed by the filter paper. With little experience, the investigator should obtain the refractive index of the average glass sample after trying three or four immersion Liquids.
- viii) Repeat this process, until the refractive index of the glass is between the closest two immersion mixtures.
- ix) Record the refractive index range for this piece of glass.
- x) Repeat the entire process for the remaining fragments of glass and tabulate the results as shown:-
  - a) Index of refraction of standard solution and proportions used:

Liquid Mixtures	Index of refraction	Drops of olive/ castor oil	Drops of clove oil
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			

- b) Index of refraction of glass samples:-

Suspect: A _____	Control: 1 _____
B _____	2. _____
C _____	3 _____
D _____	4 _____

- c) Matches of glass fragments based on the index of refraction and conclusions, there on.

*For small glass fragments, any one of the comparative density techniques- namely flotation methods or 'density gradient methods' could be used. These methods are based on the observation that a solid particle will float in liquid medium of greater density, sink in a liquid of low density or remain suspended in a liquid of equal density. These methods are found to be most sensitive and can adequately distinguish glass particles that differ in density by 0.001 gms/cc.*

### 7.5 Density comparison by flotation

i) For the heavier liquid, methylene iodide or Bromoform can be used. For the lighter liquid, Xylene, Bromobenzene, Nitrobenzene, Benzene or Kerosene can be used. For this method, Bromoform ( $d=2.89$ ) and Bromobenzene ( $d=1.52$ ) are selected.

ii) The crime and control glass piece samples are to be crushed to comparable sizes with similar shape. Each piece of glass is briefly sketched and marked for reference to return it to its original packet after examination.

iii) A cleaned and dried sample of crime glass particle is placed in a small beaker containing bromoform. The glass will float on the liquid surface. This indicates that the density of the liquid is greater than that of the glass.

iv) Slowly add the less denser liquid, Bromobenzene, drop wise with stirring, until the particle is exactly suspended. If the addition of Bromobenzene is in excess, which is indicated by the sinking of the glass particle, then bromoform is added, until the glass chip remains suspended in the liquid medium. Care is taken to see that the mixture is stirred with each addition and the air bubbles, if any, are removed.

v) Add similar size, clean and dry sample of control glass. If both the crime and the control glass particles remain suspended in the liquid, then, their densities are equal to each other and to that of the liquid mixture. Particles of different densities will either sink or float, depending on whether they are more dense or less dense than the liquid medium.

vi) The density value of the particles of glass can be determined by calculating the density/specific gravity of the flotation mixture using specific gravity bottle or a pycnometer.

### 7.6 Density comparison by density gradient tubes

An alternative technique of comparing densities of glass particles is to use density gradient tubes having length of 30cms and diameter 1 cm. A standard density gradient tube is made up of layers of two liquids, mixed in varying proportions so that each layer has a different density value. When completed, a density gradient tube will usually have 6 to 10 layers, in which bottom layers have higher density. In this experiment, we not only compare glass particles but also calibrate the density column, so that a fairly accurate estimate of density can be made. This is done by adding small crystals of ionic salts to the column, whose densities are known. List of a few crystals of different densities ( $\text{gms/cm}^3$ ) are furnished below:

TABLE-C1

Compound	Density	Compound	Density
		KF	2.48



$\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$	1.73	$\text{NaClO}_3$	2.49
$\text{Na}_2\text{C}_4\text{H}_4\text{O}_6 \cdot 2\text{H}_2\text{O}$	1.82	$\text{KClO}_4$	2.52
$\text{K}_2\text{Fe}(\text{CN})_6$	1.85	$\text{NaF}$	2.56
$\text{KNO}_2$	1.91	$\text{MgSO}_4$	2.66
$\text{KCl}$	1.98	$\text{K}_2\text{Cr}_2\text{O}_7$	2.67
$\text{KNO}_3$	2.10	$\text{KMnO}_4$	2.70
$\text{NaCl}$	2.16	$\text{Na}_2\text{CrO}_4$	2.72
$\text{NaNO}_3$	2.26	$\text{AgClO}_4$	2.80
$\text{KClO}_3$	2.32		

The liquids that are being used for preparation of the gradient mixture, as mentioned in various literatures, are as shown:-

TABLE-C-2

Heavier Liquid	Density gm/cc	Lighter Liquid	Density gm/cc
1. Bromoform	2.89	i. Benzene	0.875
2. Sym-Tetrabromoethane	2.96	ii Kerosene	0.80
3. Methylene iodide	3.32	iii Xylene	0.88
		iv Nitrobenzene	1.20
		v. Bromobenzene	1.52

However, in this method also, Bromoform and Bromobenzene are selected for preparing density gradient columns.

#### 7.6.1 Procedure:

- i) Place seven test tubes in a test tube rack.
- ii) Prepare the mixtures of bromobenzene and bromoform in the following ratios, by pipetting out the respective liquids into the test tubes.
  - a) Pure bromoform-6ml (density -2.89)
  - b) 1 ml of bromobenzene 5 ml of bromoform
  - c) 2 ml of bromobenzene 4 ml of bromoform
  - d) 3 ml of bromobenzene 3 ml of bromoform
  - e) 4 ml of bromobenzene 2 ml of bromoform
  - f) 5 ml of bromobenzene 1 ml of bromoform
  - g) Pure bromobenzene-6ml (density -1.52)
- iii) Mark off seven equal spaces-say 4 cms apart-along each of the two glass tubes of length 30 cms and place them in the tube stand.
- iv. Carefully add the solutions in the order listed in step (ii) in to the tubes (Bromoform at the bottom).
- v. With a forceps, carefully select one of the heavier crystals listed in table C-1 and add it to one of the columns. Repeat this process until at least seven different crystals are added to both the columns and record them.
- vi. Allow the crystals to settle for 10 minutes and measure their initial heights from the bottom of the tubes.
- vii Carefully add the crime and control glass fragments to their respective columns and allow the columns to stand overnight.
- viii. Record the final heights of marked crystals in each column.
- ix. Plot density Vs height for the marked crystals in each column.
- x. Record the height of the glass particles in each column.
- xi. From the graph plotted in step (ix), determine the density of the glass particles in each column. The data of the crystals used and graphs plotted as shown below:
  - a) Record of the crystals used for calibration.

COLUMN-1

COLUMN-2

Crystal	Initial Height	Final Height	Crystal	Initial Height	Final Height
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					

b) Plot density versus height for each column

c) Indicate on the graphs, the heights of glass particles and find out the corresponding densities from columns there on.

### 7.7 Refractive Index:

Every material has its own characteristic refractive index. Further the refractive index varies with temperature as well as wavelength of the light source. A super script is used to indicate the temperature and a subscript is used to indicate the wavelength, at which the refractive index is measured. Thus, if the RI of any medium is expressed as  $N_D^{20}$  it indicates that refractive index is measured at 20 degree and for sodium D line (5893A or 589 nm). Generally, Sodium lamp is used to obtain a monochromatic light source. Alternatively, a tungsten lamp with appropriate filter can also be used.

Although the refractive indices of glasses may vary considerably, the refractive indices of most glass samples encountered in practice lie between 1.51 and 1.53. Automobile head light glasses generally have refractive index range from 1.47 to 1.49. The index of refraction for several glasses is furnished below:

TYPES OF GLASS	$N_D^{20}$
Headlight glass	1.47 - 1.49
Television glass	1.49 - 1.51
Window glass	1.51 - 1.52
Bottles	1.51 - 1.52
Ophthalmic lenses	1.52 - 1.53
Common flat glasses	1.51 - 1.53

#### 7.7.1 Refractive index by immersion method:

The refractive index of the glass pieces could be studied by immersion of glass pieces in a liquid medium. When a transparent object, such as glass, is immersed in a liquid of same refractive index, it will be invisible because of the optical homogeneity of the system. With fairly large samples, the use of microscope is not necessary in the initial determinations. A simple procedure in approximating the refractive index of glass is as follows:

- (i) Place the glass fragment in a small beaker, after cleaning and drying.
- (ii) Select suitable liquids from the following:
  - a) Di-n - butyl carbonate:  $N_D^{20} = 1.411$
  - b) Tri-n butyl citrate:  $N_D^{20} = 1.455$
  - c) Alpha - bromonaphthalene:  $N_D^{20} = 1.658$



- d) Methylene iodide:  $N_D^{20} = 1.742$
- (iii) Add liquid of lower index than glass in sufficient quantity to cover the glass piece.
- (iv) Now, add in small amounts, the liquid of higher index of refraction, until the glass becomes invisible.
- (v) Remove the sample of liquid and determine its index of refraction using an Abbe refractometer. Abbe refractometers are available in the measuring ranges from 1.30 to 1.70 and from 1.45 to 1.84.
- (vi) For measuring the refractive index of the liquid mixture using Abbe refractometer, place a drop of liquid carefully on the polished surface of the upper refracting prism of the instrument. Lock the upper and lower prisms and keep them in position. The lower prism has a rough ground surface which diffuse light and permits it to enter the liquid in all directions. Select a sodium light source to fall on the reflector, below the assembly. Adjust the reflector so as to direct the light into the lower prism. The two prisms are attached to a rotating arm, which follows a curved scale, giving direct readings of the refractive index. Rotate the prism arm until the dark border, as seen in the telescope of the refractometer is set on the cross hairs. Measure the reading at this position, which gives the refractive index of the liquid directly.
- (vii) The refractive index of the mixture of two liquids can also be calculated approximately, using the following equation.

$$RI \text{ of Mixture} = (N_1 V_1 + N_2 V_2) / (V_1 + V_2)$$

Where  $V_1$  and  $V_2$  are the volumes of the liquids added and  $N_1$  and  $N_2$  are their respective refractive indices. The theoretical value obtained from the above equation may be verified by comparing with the index value obtained using the refractometer.

- viii) Alternatively, we can select a series of liquids of known index of refraction and immerse the glass fragments in several of these in turn, until the visibility indicates the liquid of nearest index. An adequate series of liquids is furnished below:

TABLE D-1

Liquid	$N_D^{20}$	Liquid	$N_D^{20}$
Methyl alcohol	1.3288	Anisole	1.5178
Water	1.3330	Trimethylene bromide	1.5238
Acetone	1.3592	Chloro benzene	1.5250
Ethyl acetate	1.3727	Methyl iodide	1.5310
N-Hexane	1.3755	Ethylene bromide	1.5383
N-Heptane	1.3872	Clove oil	1.5430
N-Butyl alcohol	1.4022	O-Nitro toluene	1.5466
Methyl cyclo Hexane	1.4235	Nitro benzene	1.5526
Ethylene glycol	1.4318	Tri-O-Cresyl Phosphate	1.5582
Ethyl citrate	1.4434	Bromobenzene	1.5602

Ethylene chloride	1.4453	O-Toluidine	1.5725
Cyclo Hexane	1.4507	Aniline	1.5864
Olive Oil	1.4667	Bromoform	1.5973
Cyclo hexanol	1.4678	O-Iodotoluene	1.6095

Diethanolamine	1.4782	Quinaldine	1.6120
Castor oil	1.4820	Iodo benzene	1.6205
Tricthanolamine	1.4853	Quinoline	1.6272
S-Tetrachloroethane	1.4853	S-Tetrabromoethane	1.6378
Toluene	1.4957	$\alpha$ -Bromonaphthalene	1.6585
Benzene	1.5017	Methylene iodide	1.7420
Ethyl iodide	1.5138		

### 7.7.2 BECKE LINE CONCEPT:

The image of a transparent object observed through a microscope is formed by refraction and reflection. Due to this behaviour of light, a dark boundary line is observed at the borders of the object. Many objects are surrounded by a narrow band of light, when immersed in a liquid. This band is called "Becke line". Thus, Becke line is the contrast (halo or bright border), which outlines the transparent irregular particle, immersed in a liquid of different refractive index. This halo disappears, when the liquid medium and the transparent object have the same refractive index. Thus, when glass particles are immersed in a liquid medium, the Becke lines will appear due to the difference in the refractive indices of the glass and liquid. When the indices are equal, the Becke lines will disappear and this point is known as 'match point.'

An important advantage of the Becke line is that, it not only indicates a difference between the indices of the glass and liquid, but also indicates, which possesses the higher value. Thus, when the focus of the microscope is raised, the Becke line moves towards the medium of higher refractive index and if the focus is lowered, it moves towards the medium of lower refractive index. This observation allows the examiner to properly select a liquid that most closely matches the refractive index of glass.

When a colourless transparent object, such as glass, is being examined with a microscope by transmitted light, visibility of the object is enhanced, as the condenser diaphragm is closed, although the resolution is sacrificed. Further more, reducing the aperture of the optical system of the microscope enhances the visibility, by emphasizing the RI difference between the glass and the mounting medium. Therefore, a minimum numerical aperture should be used in examining the glass chips for refractive index.

Standard immersion liquids may be prepared from two miscible liquids. (Refer TABLE D-1) one of which should have the lower and the other higher refractive index than that of the glass samples to be examined. A series of mixture is made to cover the expected range of glasses, in steps of 0.001 or 0.002. Alternatively standard refractive index liquids may be used. The liquids chosen should be of low volatility, chemically stable and of congenial smell. Castor oil (1.48) and clove oil (1.54) are quite suitable and readily available. Mixture of these liquids are reasonably stable in refractive index. The standardized liquid mixtures are stored in small screw capped vials in a rack. Their glass rods may be used to transfer drops of liquids from the vials to microscopic slide.

### 7.7.3 Refractive index by using the mixture of miscible liquids.

- i) Prepare a series of solutions ranging from either pure olive oil (1.46) or castor oil (1.48) to pure clove oil (1.54). Eight to ten solutions having refractive index range between 1.47 to 1.53 are prepared and the indices of refraction of these solutions are determined by Abbe refractometer, as described earlier. Then, store the solutions in small screw cap bottle with proper labelling.



- ii) Turn on the sodium vapour lamp and adjust the microscope. If sodium lamp is not available, yellow filter, having maximum transmission of 590nm may be used to provide an approximate equivalent of sodium light (589nm).
- iii) Add one drop of one of the standard liquids on to the cavity slide. Place the suspected glass fragment in it and cover with a small cover glass. The cover glass ensures that the chip is completely immersed and minimizes any spurious lens effect from the surface of the drop.
- iv) Bring the glass fragment into focus. Most microscopes have a 16mm (2/3 inch) objective, which should be used with an eye piece of 10x or 20x magnification.
- v) Observe the Becke line or the bright halo surrounding the edges. If the halo seems to be indistinct, it may be more pronounced by closing down the condenser diaphragm.
- vi) Focus upward and downward to know the movement of Becke line in order to determine whether the glass fragment or the solution has the higher refractive index. When the refractive indices of the glass and the immersion liquid are the same, the chip will become almost invisible and it may be difficult to find and bring into focus the edges of the chip. By focusing up or down and following the Becke line, the examiner will be able to ascertain, whether the refractive index of the fragment is the same, as the liquid. If there is difference, to make an appropriate assessment, the next immersion liquid may be selected.
- vii) The first liquid may be removed with filter paper and next liquid flowed into the cover glass, which enters the cavity by capillary attraction. Any surplus can be absorbed by the filter paper. With little experience, the investigator should obtain the refractive index of the average glass sample after trying three or four immersion Liquids.
- viii) Repeat this process, until the refractive index of the glass is between the closest two immersion mixtures.
- ix) Record the refractive index range for this piece of glass.
- x) Repeat the entire process for the remaining fragments of glass and tabulate the results as shown:-
  - a) Index of refraction of standard solution and proportions used:

Liquid Mixtures	Index of refraction	Drops of olive/ castor oil	Drops of clove oil
1.			
2.			
3.			
4.			
5.			
6.			
7.			
8.			
9.			
10.			

- b) Index of refraction of glass samples:-

Suspect: A \_\_\_\_\_ Control: 1 \_\_\_\_\_  
 B \_\_\_\_\_ 2. \_\_\_\_\_  
 C \_\_\_\_\_ 3 \_\_\_\_\_  
 D \_\_\_\_\_ 4 \_\_\_\_\_

- c) Matches of glass fragments based on the index of refraction and conclusions, there on.

- xi) The visibility of the Becke line depends to a larger extent on the shape of the particle. The strongest lines are shown on flat-like edges. Hence, this type of pieces are chosen, if possible, for tests.

#### 7.7.4 Refractive index using hot stage microscope.

In the previous method, the change in the refractive index of the liquid medium was accomplished by mixing the two miscible liquids of lower and higher index values to match with the refractive index of the glass particle, at constant temperature (say, room temperature). Alternatively, we can vary the temperature of immersion fluid using a special apparatus known as a "hot stage". In this method, the glass is immersed in high boiling liquid. Usually a silicone oil and heated at a uniform rate until the match point is reached. Mettler hot stage is commonly used for varying and maintaining the exact temperatures ( $\pm 0.1^\circ\text{C}$ ) of particles in an immersion liquid on a glass slide. The rate of change of RI of glass with respect to temperature is approximately 0.00001 per degree Celsius and is negligible. Where as, the RI of liquid medium decreases at the rate of approximately 0.0004 per degree Celsius. The immersion liquid to be used must have two properties. Its change in RI with temperature must be large in comparison to that of the glass.

- i) The glass particle to be examined (whole or crushed) is mounted in Silicon oil on the microscope slide, on the hot stage.
- ii) The stage is illuminated with a monochromatic source and the glass fragment is brought into focus and edge is selected.
- iii) The hot stage provides a stable heating and cooling and can be set to  $\pm 2^\circ\text{C}$ ,  $\pm 1^\circ\text{C}$  or  $\pm 0.5^\circ\text{C}/\text{minute}$ .
- iv) As the temperature of the hot-stage is varied, constant observation is made to see the 'point', where the edge contrast is at minimum and this temperature is known as "match temperature". It is the temperature at which the glass and oil will have the same refractive index. As the temperature is changed at a continuous, constant rate and there will be a small temperature lag between the hot-stage and the glass, and the match temperature is taken as the mean of the temperatures at which the glass disappears and then re-appears.
- v) The oil is calibrated using standard optical glass of known RI, to establish the variation of refractive index of the oil with temperature. After calibration, the match temperature gives the refractive index of the glass fragment directly.
- vi) When making an RI measurement, the operator is required to concentrate for several minutes on an image set against a bright yellow background. Since, it is the points of disappearance and re-appearances which is recorded, it is necessary to focus on very faint images or at the match point on the residual blank area of the field of view.
- vii) This method is found to be very precise and reproducible results are obtained, when repeated measurements were carried out on the same edge, with immediate succession. Since, the refractive index is wave length dependent, it is usually measured at the sodium 'D' line (589nm). In modern instruments, this is achieved by the use of filters rather than by a sodium discharge lamp.



- viii) Depending upon the visual response of the operators, there may be small discrepancies, in recording the match temperature and it may impose considerable strain on the operator, if too many particles are to be examined at a stretch. To eliminate this fatigue on the operator, automatic systems for measuring refractive index of glass are commercially available. These are microprocessor controlled, which operates on Microsoft windows environment and has the following advantages:
- Full control of the hot stage.
  - Automatic RI match point detection by image processing.
  - Immersion oil calibration.
  - Match temperature to RI conversion using stored calibration data.
  - Documented records of every measurement.
  - Formatted casework print out and graphical plots for future presentations.

## 7.8 Elemental analysis of glass:

Subject to availability of the instrumental analysis techniques, elemental composition of the glass samples can also be studied for identifying the type of glass-such as sheet glass, container glass etc., indication of the manufacturer, batch of glass and such other factors for probable source correspondence. A number of efforts have been made to evaluate the capabilities of various analytical technique- where methods are well known- such as emission spectrography, Atomic absorption, Neutron activation, Mass spectrography, Inductively coupled plasma Mass spectrometry (ICP/MS), Inductively coupled plasma Emission spectrometry (ICP/AES), Laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS), Scanning electron microscope energy dispersive X-ray fluorescence (XRF), SEM/EDX and so on, for the trace elemental analysis of the glass fragments and it has been observed that the trace element profiling of glass fragments offered additional discrimination between the glass fragments that were indistinguishable on the basis of their physical properties such as density and refractive index.

The elements of interest, while comparing the glass samples are the minor and trace elements, namely Aluminium (Al), Iron(Fe), Magnesium(Mg), Manganese(Mn), Strontium(Sr), Zirconium(Zr) Calcium(Ca), Barium(Ba), Titanium(Ti), Arsenic(As) cobalt(Co) and lead(Pb). These elements may be deliberately added in small amounts or may be present as accidental traces in the raw materials. The major elements do not vary enough among sources of soda-lime-silica glass or borosilicate glass and will not be of much use for discrimination.

Initially, the elemental analysis of the glass fragments was used to study the population data i.e. to know the presence or absence of trace elements to ascertain their source correspondence. This type of qualitative elemental analysis for the population studies of the trace elements leads to a definite conclusion only in cases, where some very uncommon elements are detected in the samples. In practice, each of these techniques possess its own particular strength and weaknesses. In any event, whether elemental analysis is used or not, the various methods set out in this manual for density and refractive index determinations, will always be useful as sensitive methods for matching tiny particles with suspect source and the opinion of the Forensic glass examiner could be formulated as under:-

- A positive opinion of identity- that the submitted specimens came from the same pane or object and no other.
- An opinion, on the basis of certain physical properties, that the submitted specimens most probably came from or consistent with or similar to or indistinguishable from a common source.
- A positive opinion of non-identity, specimens could not have come from a common source.
- An opinion that no conclusion could be reached as to a common source.