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IDENTIFICATION OF SMALL GLASS FRAGMENTS FOR FORENSIC PURPOSES

By K.W.Terry, A.van Riessen and B.F. Lynch

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PREFACE

This publication forms the final report of the Criminology Research Council project 9/80 entitled "The Identification of Small Glass Fragments for Forensic Purposes". The project was funded over a two year period from August 1980 to August 1982 with grants totalling \$15,949.

The aim of the project was to implement a rapid and sensitive nondestructive method, based upon a scanning electron microscope, for the identification and comparison of small glass fragments. Full details of the various facets of the research programme have been recorded in two annual reports to the Criminology Research Council (Terry, van Riessen and Lynch 1981, 1982). These topics are listed in Appendix A of this report. This final report attempts to give an overall view of glass, glass analysis and the uses to which the analyses may be put. It is hoped that this will be of interest to law enforcement personnel and others in the criminal justice area as well as forensic scientists.

The three principal investigators have been Dr. K.W. Terry, Mr. A. van Riessen (both from the School of Physics and Geosciences, Western Australian Institute of Technology) and Mr. B.F. Lynch (Forensic Chemistry Division, Government Chemical Laboratories, Perth). Dr. K.W. Terry has acted as project leader but each of the principal investigators have been responsible for a clearly defined area of the programme as follows:

Mr. A. van Riessen X-ray induced elemental analysis in the SEM.

The salary component in the grant for a half-time graduate research assistant has been used to second David J. Vowles, Chief Technician, School of Physics and Geosciences, to the project for the two years.

We acknowledge the valuable contributions made to the project by the following people. Mr. D.J. Vowles for his dedicated assistance in all aspects of the project; Mr. N.G. Ware (A.N.U.) for his assistance in providing and help in installing his PIBS program; Dr. D.W. Reid and Ms. J. Posner (School of Mathematics and Computing) for their assistance in setting up the software system to enable the glass data to run on SPSS and CLUSTAN; Mr. A. Planken for his workmanship in constructing the two thin foil devices and other mechanical components; Ms. D. Hollingsworth for typing the manuscript.

We also acknowledge the cooperation of Pilkington ACI in providing samples, technical and commercial information. Finally we thank the Criminology Research Council for their financial support for the two years of funding.

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CHAPTER 1 INTRODUCTION

The term glass in its widest context refers to a physical state rather than to a composition. Glass is considered to be a non-crystalline solid which can be either inorganic or organic. The common usage of the term however implies an inorganic composition and an appropriate definition is that of the American Society for Testing and Materials (1978) which defines glass as "an inorganic product of fusion which has cooled to a rigid condition without freezing". To most people the term glass has an even narrower meaning which usually implies silicate glass. Silicate glasses are based on $SiO₂$ and may be modified by the addition of various agents to the melt to produce a number of basic types. The silicate glasses include soda-lime silica glass (by far the most common), borosilicate glass, aluminosilicate glass and lead silicate glasses. These groups can be further modified using such agents as MgO, PbO, $A1_2O_3$, BaO, K_2O , B_2O_3 , Fe_2O_3 , As_2O_3 and others. The non-silicate systems are very specialised glasses not in common use and would be easily identified but would be most unlikely to arise in a forensic context.

To any observer, glass is a very widely used commodity finding applications in buildings, containers, motor vehicles, electronics, lighting, tableware, cookware, scientific equipment and so on. A review of the sources and uses of glass in Australia is given in Chapter 2. The occurrence of glass fragments as physical evidence of crime is very probable from a wide variety of incidents, such as vandalism, breaking and entering offences, hit-run 'accidents' and assaults. In cases involving broken windows for example, it has been shown by Nelson and Revel (1967) that in addition to glass fragments being ejected in the direction of the breaking blow, glass fragments are also ejected in a backwards direction up to approximately 3 metres. Pounds and Smalldon (1978) have studied the deposition and distribution of such particles on the person of subjects standing nearby windows which were experimentally broken. Particles were recovered in greatest abundance from the surface of jackets and the hair. Few particles were recovered from subjects which exceeded 1 mm in size and most were in the size range $0 - 0.2$ mm. Interestingly, the subjects involved in these experiments were totally unaware that any glass particles had landed on them. In another paper, Pounds (1977) showed that although tiny fragments may persist on clothing for a considerable time, particles > 1 mm persist for only very short periods. In addition to glass particles being deposited by back fragmentation, there is also the probability of the criminal accumulating glass fragments in clothing or footware during subsequent entry to the premises. Clearly, the necessity in forensic glass analysis is to be able to analyse, preferably non-destructively, very small samples.

The major area of our work has been to commission techniques based on a scanning electron microscope equipped with an energy dispersive xray spectrometer. This enables accurate elemental analyses on small fragments to be obtained with little sample preparation or destruction. The initial technique was to use the conventional method of elemental analysis in a SEM/EDS system whereby the electron beam

induces x-ray fluorescence of the sample. The second technique was a recently reported method (Gould and Healey, 1975) in which an x-ray beam is generated within the SEM and then used to fluoresce the sample. The two techniques are complementary and have great significance to glass analysis. This is due to the electron beam technique, with >^ 1000 ppm detection limits, being most sensitive for low atomic number elements typical of the major and minor elements in glasses, while the x-ray beam technique, with its \geq 30 ppm detection limits, being most sensitive for higher atomic number elements,
typical of the trace elements in glasses. The details of these typical of the trace elements in glasses. analytical techniques are documented in Chapter 3. A collection has been made of 177 samples of glasses used within Australia which includes both locally produced and imported glasses. Quantitative elemental analyses of the glasses have been obtained using the electron induced method together with qualitative elemental analyses using the x-ray induced method. These analyses are presented in Appendices B and C.

The ubiquity of glass gives rise to the possibility of mistaking glass particles found on clothing in connection with a crime for those which might be there purely by chance. This problem was highlighted by Pearson, May and Dabbs (1971) when they examined 100 suits, obtained from a dry cleaning agency, for the presence of glass fragments. They found in 63 of these suits a total of 551 glass particles exceeding 0.1 mm in size. It is most essential therefore to be able to discriminate between different glass particles. Traditionally, the discrimination techniques are based on refractive index and/or density and these measures are usually quite effective. There exists however, the possibility that from a combination of effects, glasses with quite different compositions can have the same refractive index or density. An important goal in the study has been to evaluate the usefulness of elemental analyses obtained in the scanning electron micro-scope for discerning whether glass particles are closely related types. Details of this are presented in Chapter 4.

In many investigations, glass analyses are requested for fragments found at a crime scene but for which no comparison sample is available. In these cases a glass identification or classification is required. A statistical analysis of the quantitative elemental analyses and the refractive indices of all the glasses has been undertaken using the Statistical Package for the Social Sciences (SPSS) package by Nie (1975), in particular using DISCRIMINANT, and then with the CLUSTAN 1C package by Wishart (1978). These procedures and the subsequent findings are presented in Chapter 5.

The final aspect of the work has been to assess the usefulness of the various techniques developed to forensic case studies that involve glass fragments. In Chapter 6 the details of 9 case studies involving a total of 27 glass fragments are presented.

CHAPTER 2 SOURCES AND USES OF GLASS IN AUSTRALIA

Throughout the Western world there are some 538 companies listed in the Glass Industry Directory (1981) which manufacture glass or glass products starting from melting the raw ingredients. Needless to say, there are many further glass making facilities in Eastern bloc countries. There are also a large number of companies who do not melt glass, but further process purchased glass. In Australia, we have relatively few primary glass making companies. They include the following :

- i) Pilkington ACI which has a float glass plant at Dandenong, Victoria,and a plant for producing decorative figured rolled glass, wired cast glass, wired rolled glass and wired plate glass at Alexandria, New South Wales. Most of the products of these plants find use in the building industry, however some Australian float glass is further processed into windscreens and other products.
- ii) Australian Consolidated Industries (ACI) which has glass making plants in each Australian state for the manufacture of glass containers in flint, amber and green colours.
- iii) Glass Containers Ltd. with a plant at Penrith, NSW, for the manufacture of single use and returnable beer and beverage containers, food, milk and other containers.
- iv) Crown Corning with a plant at Waterloo, NSW, for the manufacture of domestic glassware, decorative giftware, glass tumblers for food packaging, glassware for the hotel industry and scientific products.

In addition there are a number of small concerns at Newcastle, NSW, for making specialised glasses such as light globes, lightingware and TV tubes.

The companies mentioned above produce the majority of glass in common use in Australia but there is a significant number of imports. The Australian Bureau of Statistics tabulates in its overseas trade statistics the imports of glass and glass products in terms of the source country, quantity and monetary value. Table 2.1 shows source country, quantity and monetary value. categories of glass imports extracted from this source for 1979.

TABLE 2.1: AUSTRALIAN GLASS IMPORTS (Source: Australian Bureau of Statistics, 1979)

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* 1. Argentina; 2. Austria; 3. Belgium-Luxembourg; 4. Canada; 5. China; 6. Czechoslovakia; 7. Denmark; 8. Finland; 9. France; 10. Germany; 11. Hong Kong; 12. India; 13. Indonesia; 14. Ireland; 15. Italy; 16. Japan; 17.Korea; 18. Netherlands; 19. New Zealand; 20. Norway; 21. Philippines; 22. Poland; 23. Portugal; 24. South Africa; 25. Spain; 26. Sweden; 27. Switzerland; 28. Taiwan; 29. Thailand; 30. Turkey; 31. United Kingdom; 32. United States of America; 33. Yugoslavia; 34. Hungary.

So far as forensic casework is concerned, the most important glass products are those derived from clear flat glass, which are used in windows, shopfronts, other building products, furniture and windscreens. The most common flat glasses are available in three types which vary in quality, quantity and price.

By far the most important flat glass now available is float glass. The float process is the latest technology for flat glassmaking and produces distortion free glass having perfectly flat, parallel surfaces with a bright fire polished finish. Float glass was invented by and first manufactured commercially by Pilkingtons in 1959 in the UK. Pilkington ACI first began manufacturing float glass in Australia
in 1974. Float glass is also imported into Australia from countries in 1974. Float glass is also imported into Australia from countries as shown in Table 2.1. Table 2.2 shows the quantity of import Table 2.2 shows the quantity of import compared to locally manufactured product based on 1979 figures.

Prior to 1974, Australian clear flat glass was manufactured by the drawn sheet process at plants at Alexandria, NSW and Dandenong, Victoria. With the introduction of float glass, sheet glass ceased being manufactured at Dandenong and in 1976 also ceased at Alexandria. As shown in Table 2.1, sheet glass is still imported from numerous countries. The quantities imported are shown in Table 2.2 and are compared with local and imported float glass.

Product (Thickness)	*Local Float	Imported Float square metres	Imported Sheet	
Less than 3 mm	1 487 200	307 336	223 909	
$3 \, \text{mm} - 4 \, \text{mm}$	4 088 267	463 278	901 661	
$5 \, \text{mm}$	1 825 840	52 094	76 897	
$6 \, \text{mm} - 7 \, \text{mm}$	1 028 066	289 290	23 619	
Greater than 7 mm	15 100	58 627	635	

TABLE **2.2:** IMPORTED AND LOCALLY MADE FLAT CLEAR GLASS (1979)

Based on tonnages supplied by Pilkington ACI assuming \star density $2\,$ 500 kg/m³

Figures 2.1 and 2.2 show trends for the import of float and sheet glass commodities into Australia during the period 1979-1982. These imports appear to fluctuate widely, however they are at such a level as to be a significant source of possible variability in forensic casework samples.

Figure 2.1 Import trends for imported clear float glass commodities (data extracted from Australian Bureau of Statistics overseas trade figures)

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Figure 2.2 Import trends for imported clear sheet glass commodities (data extracted from Australian Bureau of Statistics overseas trade figures)

The remaining flat glass type in common use in Australia is rolled glass which is manufactured at Alexandria NSW by Pilkington ACI. In this process glass in a molten state is passed between two rollers, one of which usually has a pattern on it so that the resulting sheet is patterned on one side and plain on the other. Clear unpatterned glass is also produced with this process by using two plain rollers. Glass of this type is used where only low quality optical properties are required for such applications as horticulture. In fact glass of this type bears the trade description "Horts". Table 2.3 shows comparative import and locally manufactured production levels for 1979.

TABLE **2.3:** IMPORTED AND LOCALLY MADE PATTERNED ROLLED GLASS (1979)

Product (Thickness)	*Local * - - square metres - -	Imported
Clear less than 5 mm	393 066	86 372
Clear 5 mm and above	326 653	25 106
Coloured less than 5 mm	84 799	121 270
Coloured 5 mm -6 mm	66 213	48 111
Coloured greater than 6 mm	8 680	13 458

 \bullet Based on tonnages supplied by Pilkington ACI assuming a density of 2 500 kg/m³.

The next most important glass product so far as forensic casework is concerned is windscreen and automotive glass products. Windscreens are manufacturedat Geelong, Victoria using Australian and imported flat glass. Table 2.4 shows comparative figures for imported and locally produced windscreens.

Headlamp glasses usually come from sealed beam headlights and in most cases they are made of borosilicate glass. All headlamps appear to be imported from countries as shown in Table 2.1.

The remaining major category of glass which is frequently considered in forensic casework is container and tableware glass. Glass is very widely distributed in the environment and broken or disused containers and/or tableware account for a large proportion of this. Particles of these glasses are most likely to be "naturally" present on articles of clothing etc. As mentioned earlier, the vast majority of container glass in use comes from Australian container glass plants located in all Australian states. As Table 2.1 shows however, there are imports from many countries. Table 2.5 shows quantities of these glass items imported.

Item	Number Imported (thousands)
Pharmaceutical, toiletry and cosmetic containers, $<$ 284 ml	117 030
Other glass containers < 284 ml	74 454
Glass containers 284 ml - 567 ml	9 085
Glass containers for beverages	7 491
Other glass containers	13 334
Tableware and kitchenware glass items	54 020

TABIE 2.5: IMPORTED CONTAINER AND TABI£WARE ITEMS (1979)

CHAPTER 3 ANALYTICAL TECHNIQUES

3.1 Introduction

The approach in this project has been to analyse quantitatively for the major and minor elements that constitutes the glass network formers and glass modifiers together with a qualitative assessment of the trace elements. This has been achieved using a scanning electron microscope equipped with an energy dispersive x-ray spectrometer. Results from conventional electron induced XRF are supplemented with those obtained from an x-ray induced XRF method that has been developed in our JEOL JSM 35C (van Riessen and Terry, 1981, 1982).

Refractive index determinations of each glass sample was also carried out and used together with the elemental analyses to determine discrimination of pairs of glasses or to help in classifying a glass according to use.

3.2 Experimental

3.2.1 Refractive Index Determination

The refractive index of each glass sample was determined using a Mettler FP5 heating stage controlled by a Mettler FP52 temperature programmer in conjunction with an optical microscope. This equipment is shown in Figure 3.la.

Fragments of each glass were immersed in Dow Corning DC 710 silcone oil and the mean temperature obtained of the disappearance and reappearance of the Becke line in sodium light. Reference to the calibration chart for the oil, provided the value of the refractive index.

Equipment used to determine refractive index allows the temperature to be read to 0.5⁰C, this in turn is reflected in an uncertainty in the refractive index of $+$ 0.0002, after using the appropriate calibration curve.

Samples of known refractive index used to check the technique provided either perfect matches or values within 1 in 10⁴ of the accepted value.

3.2.2 Sample Preparation

Once the refractive index was determined, each piece of glass was mounted in a 12.5 mm diameter acrylic tubing. The sample was then ground and polished prior to being coated with a vacuum evaporated thin film of carbon. The glass fragments in this form are still available as a court exhibit if necessary. Figure 3.1b shows the full set of glass museum samples as prepared for elemental analysis in the SEM.

Figure 3.1 (a) Mettler FP 5 programmer, FP 52 Heating stage and microscope used for refractive index measurement

Figure 3.1 (b) Glass museum samples prepared for elemental analysis in the S.E.M.

- 3.2.3 Scanning Electron Microscopy and Energy Dispersive X-ray Spectrometry
(i) Electro
	- *(T)* **Electron Induced**

The elemental analyses have been carried out using a JEOL JSM 35C scanning electron microscope equipped with an United Scientific lithium drifted silicon detector and asociated electronics (Figure 3.2a). The spectra are accummulated in a Tracor Northern TN 1705 multi-channel analyser (MCA). The adopted procedure for elemental analysis is to use 20 kv and $\overline{5}$ x 10^{-10} amps at normal incidence onto a 40 x 40 $\,$ m raster on a polished sample and a detector take-off angle of 35° (see Fig 3.3a). A typical count rate of 5 kcps is obtained. The spectra are collected in the MCA for 50s live time and are then stored on a stereo cassette for subsequent batch processing on a PDF 11/10 computer. The elemental analysis is then computed using the peak integration with background subtraction (PIBS) technique of Ware (1981). current program provides for an elemental analysis for Na, Mg, Al, Si, P, Cl, S or Pb, K, Ca, Ba or Ti, V, Cr, Fe, Mn and Zn expressed as oxides and has a sensitivity down to approximately 0.1%. The full set of data is presented in Appendix B.

Accuracy

The PIBS program of Ware that has been used to calculate the quantitative elemental analysis of each glass requires certain calibration factors to be determined using standard samples. Four glasses from the range of NBS Standard Reference Materials and certain mineral samples have been used to calibrate the program. The mean of the two analyses obtained from each of the four NBS glasses subsequent to the calibration procedure is given in Table 3.1.

TABLE 3.1: MEAN ANALYSES OF NBS STANDARD GLASSES COMPARED TO THEIR QUOTED COMPOSITIONS IN WT.%

GLASS		Na2O	MgO	A1203	SiO ₂	SO3	K20	CaO	Fe203
NBS 621	STD MEAN	12.71 12.8	0.27 0.3	2.77 2.8	71.14 71.3	0.13 0.2	2.01 2.0	10.71 10.7	
NBS K411 STD	MEAN		14.67 14.8		54.3 55.2	0.0 0.1		15.47 15.4	14.42 14.5
NBS K412 STD	MEAN		19.33 19.3	9.27 9.7	45.35 45.9			15.29 14.9	9.96 10.0
NBS 620	STD MEAN	14.4 14.5	3.6 3.6	1.8 1.8	72.1 72.2	0.0 0.3	0.4 0.4	7.1 7.0	

Figure 3.2 (a) JEOL JSM 35 C Scanning Electron Microscope and energy dispersive X-Ray analysis equipment.

Figure 3.2 (b) Control for manipulating the thin foil device in place on the column of the S.E.M.

Figure 3.2 (c) Thin Foil Device.

Precision

The duplicate analyses of the 134 glass museum samples has taken place over a period of three months. During each analysis session NBS glass SRM 621 has been included as an internal control. The precision of the fifteen sets of data over this period is presented in Table 3.2. The resulting mean and standard deviation indicates a satisfactory level of performance of the technique.

TABLE 3.2: PRECISION OF RESULTS ON GLASS NBS 621 OVER THREE MONTHS IN WT.% (Electron induced technique)

	Na20	MgO	A1203	SiO ₂	SO3	K20	CaO
STANDARD NBS VALUE	12.7	0.27	2.77	71.14	0.13	2.01	10.71
EXPERIMENTAL $MEAN (N=15)$	12.6	0.2	2.8	71.3	0.1	2.0	10.8
STANDARD DEVIATION OF MEAN	0.3	0.1	0.1	0.3	0.1	0.0	0.2

(ii) X-ray Induced

The x-ray induced method that has been adopted is to focus the electron beam (obtained from using 39kV and 200nA) onto a silver foil of 10 μ m thickness which acts as a transmission x-ray target (see Fig. 3.3b). The resultant primary x-ray beam, impinging on the sample, consists of the characteristic silver peaks and associated Compton peaks together with a continuum that has been self-filtered by the foil. Figure 3.2b shows the thin foil device in place on the column of the SEM, while the thin foil device itself is presented in Figure 3.2c. The incident primary x-ray beam causes secondary x-ray fluorescence in the sample so that characteristic xrays of the elements in the sample are subsequently detected in the Si (Li) detector. With the sample tilted and rotated to directly face the x-ray detector the x-ray spot size on the sample is an ellipse with dimensions of approximately 1.5 x 2.5 mm. The technique inherently has a very low background so that sensitivities of better than 30 ppm are achieved for certain elements.

Figure 3 3(a) Schematic diagram of electron induced technique

Figure 3.3 (b) Schematic diagram of x -ray induced technique

Although better minimum detection levels are achieved considerably longer counting times (typically 1000s) are required to accumulate sufficient counts. Peak minus background counts extracted from spectra provide qualitative information regarding the amount of a particular element present in the sample. The full set of data is presented in Appendix C.

In glass analysis the x-ray induced method complements the electron induced method. As is shown in Fig. 3.4, the electron induced method is most sensitive for low atomic number elements, typical of the major and minor elements in glasses, while the x-ray induced technique is most sensitive for higher atomic number elements, typical of the trace elements in glasses.

Accuracy

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As the x-ray induced work was used in a qualitative fashion, a determination of accuracy for the technique is of little consequence. However, the glass museum samples have been analysed, by atomic absorption, for certain elements and this enables a comparison to be made between the two techniques. Similarly the electron induced and x-ray induced techniques could be compared for common elements. Limited comparisons for SrO between the x-ray induced technique (P-B counts for Sr) and atomic absorption (ppm of SrO) provide a linear relationship with a correlation coefficient of 0.986.

Precision

During the analyses of the 134 glass museum samples, glass NBS SRM 621 was included as an internal control. The precision of twelve sets of data over this period is presented in Table 3.3. The largest standard deviation occurred for iron which has the lowest counts and thus the worst counting statistics.

FIGURE 3.4 • TYPICAL X-RAY SPECTRA USING THE ELECTRON AND X-RAY INDUCED METHODS.

3.3 X-ray Induced Elemental Analysis Data

As mentioned previously a full set of data obtained using the thin foil technique is presented in Appendix C. As presented the data is qualitative, although for some elements supporting information from atomic absorption measurements enables semiquantitative analyses to be made.

During the batch analysis it was apparent that for some elements the choice of accelerating voltage, foil type and foil thickness were not at their optimum values for minimum detection levels. For future case work, where only a few samples at a time would be analysed, the best conditions possible (for elements of interest) would be selected.

Peak minus background counts of 20 different trace elements were made from the glass museum samples. Other elements, constituting the glass network formers and modifiers were present but were not measured as better sensitivity was achieved for these elements using the electron induced approach. Certain elements measured (Fe, Cr and Zn) were detected by both the electron and x-ray induced techniques and acted as a check on the reproducibility of the techniques.

Of most interest were the trace elements, as these could not be observed with the electron induced technique. The detection of these additional elements aided in discriminating between two similar glasses when other approaches had failed (for an example see section 4.5).

The development and trial of the thin foil device has demonstrated the value of the technique in supporting electron induced data. An obvious advantage is the ability to obtain data from both techniques, consecutively, using the same equipment (SEM) on the same sample. The non destructive nature of the technique and its inherent sensitivity for a number of elements augments well for its further use and development in the field of forensic science.

CHAPTER 4 COMPARATIVE GLASS ANALYSIS

4.1 Introduction

A frequent casework requirement is the comparison of glass from a crime scene with glass particles found to be associated with a suspect. Such glass particles are often exceedingly small, examples of which are shown in Figure 4.9. The SEM/EDS technique offers a means by which certain compositional information may be obtained, virtually non-destructively, to supplement other information. In this chapter, quantitative SEM/EDS has been utilised in a two sample comparison in order to determine whether a pair falls within a close class relationship. In making these determinations variability associated with the SEM/EDS measurements is examined.

4.2 Data Analysis

As previously outlined in chapter 3, the quantitative analysis gives the oxide concentration for up to 15 elements. In most instances however only the oxides of sodium, magnesium, aluminimum, silicon, potassium and calcium occur at levels high enough for measurement.

In assessing multiple measurements, the technique proposed by Smalldon and Brown (1980) offers quite a convenient approach. In this treatment, a test parameter \bar{R} is calculated by obtaining the ratio of an observed difference (D) in concentration to an expected difference (E) in concentration if the samples were from the same origin.

i.e.
$$
R = \frac{D}{E}
$$

which extended to i dimensions becomes

$$
R = \sqrt{\sum_{i=1}^{n} (\frac{D_i}{EI})^2}
$$

The expected differences were determined from 134 duplicate analyses. The observed difference between each duplicate was plotted against the mean of the duplicate. (Figures $4.1 - 6$). plotted against the mean of the duplicate. NOTE: These plots were computer plotted and not all 134 points appear since

(i) in some cases the particular element was not detected, and (ii) points occurring in a defined area are printed only once.

Certain parts of the scale are non linear to show the range of values.

These plots were tested by regression analysis to determine if any systematic relationship existed between the mean value and the difference between duplicates. No such relationship was detected and it was determined that the mean of the difference between duplicates was a suitable expected difference for each

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Fig. 4.1 Mean $Na₂O$ vs difference between duplicates.

Fig.4.2 **Mean** MgO vs difference between duplicates.

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Fig. 4.4 Mean SiO₂ vs difference between duplicates.

Fig.4.6 Mean CaO vs difference between duplicates.

component. The expected difference for the six major oxides are given in Table 4.1.

Component	Expected difference (per cent)	
Na20	0.423	
MgO A1203	0.210 0.204	
SiO ₂	0.406	
K20 CaO	0.203 0.286	

TABLE 4.1: EXPECTED DIFFERENCES BETWEEN DUPLICATES

A computer program was written to

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- (i) Calculate a R value for all duplicates
- (ii) Calculate a R value for all duplicate pairs excluding duplicates
- (iii) Print a histrogram of (i) and (ii)

Frequency distribution of R values for the 134 duplicates are shown in Table 4.2.

TABLE 4.2: FREQUENCY DISTRIBUTION OF R VALUES FOR DUPLICATE SAMPLES

Fig.4.7 Histogram showing distribution of R values.

From this we could set a critical value of say $R = 5$ above which any pair of samples would not be expected to be in the same class relationship. Figure 4.7 shows the distributions of R for duplicates and all possible pairs. It can be seen that the vast majority of R values for dissimilar pairs exceeds value range (i.e. $R = 0-5$) of the R duplicates.

4.3 Identification of dissimilar pairs

Thirty pairs of glasses from the collection from different classes which were found to have similar refractive indices (+0.0001) were compared using this technique. The results are summarised in Table 4.3.

$RI + 0.0001$	TYPES	\mathbf{R}
1.5198	Float, container	19
1.5233	Float, spectacle	82
1.5237	Float, spectacle	52
1.5192	Float, tableware	17 ²
1.5187	Float, tableware	18
1.5198	Float, patterned	5.5
1.5196	Float, patterned	3.9
1.5143	Sheet, lampbulb	7
1.5164	Sheet, container	19
1.5163	Sheet, tableware	17 ²
1.5246	Patterned, container	12 ²
1.5196	Patterned, container	18
1.5199	Patterned, container	19
1.5247	Patterned, container	26
1.5200	Patterned, container	15 ₁₅
1.5200	Patterned, container	17
1.5193	Patterned, container	16 [°]
1.5229	Patterned, spectacle	39
1.5247	Patterned, spectacle	51
1.5229	Patterned, spectacle	72
1.5188	Patterned, headlamp	22
1.5231	Wired, container	9
1.5228	Wired, spectacle	42
1.5235	Container, container	18
1.5203	Container, headlamp	28

TABLE 4.3 R VALUES FOR DISSIMILAR PAIRS WITH SIMILAR RI

Of these, all were identified except for one pair $(R = 3.9)$. This pair was patterned Belgian glass and an American float glass. the next closest comparison for a dissimilar pair $(R =$ 5.5) also featured patterned - float glasses. These are both window type glasses.

4.4 Discrimination of window glasses

As noted in Chapter 2, the bulk of window glass now being installed is float glass. The analysis of the six mentioned components obtained for 20 float glass samples is shown in Appendix B.2.

It can be seen that there is a remarkable uniformity in composition for the six major-minor elements. Information from Pilkington ACI is that the composition of their float glass is controlled within very rigid limits. Quite clearly the important question of discriminating such window glasses cannot be resolved from quantitative major-minor elemental comparison. An analysis of trace elements would be required to tackle this problem.

So far as trace element analysis in the SEM is concerned, the xray spectra obtained from the thin foil device may be of use in discriminating certain cases. As noted in the previous section, two window glasses not separable on the basis of refractive index or major-minor composition were resolvable with the thin foil spectra. These spectra are shown in Figure 4.8 below. A full listing of thin foil spectra for the glass collection is given in Appendix C

FIGURE 4.8 • X-RAY INDUCED X-RAY SPECTRA FROM AN AMERIC-AN FLOAT AND A BELGIUM PATTERNED GLASS

Figure 4.9 (a) Glass particles lodged in crevice in hammer handle (x 20)

Figure 4.9 (b) Glass particles from hammer handle
CHAPTER 5 STATISTICAL ANALYSIS OF THE GLASS DATA BANK

5.1 Introduction

One of the aims of this investigation has been explored in the previous chapter, namely, to predict whether two fragments of glass are of a close class relationship. In this chapter an account is given of attempts to classify correctly a single fragment of glass based upon the information measured on a data bank of the first 134 samples collected.

The 134 glass samples were initially catalogued into eleven categories, namely, float, sheet, patterned, wired, windscreen, container, light bulb, spectacle, tableware, labware and headlamp. For each glass sample data has been acquired comprising the refractive index and the elemental composition of up to fifteen elements expressed as oxides. The full set of data is presented in Appendix B.

In this chapter the data is intially assessed using basic statistical methods of means, standard deviations and histograms. It is then assessed using the Statistical Package for the Social Sciences (SPSS) package by Nie (1975), in particular using DISCRIMINANT, and then with the CLUSTAN 1C package by Wishart (1978).

5.2 Basic Statistical Methods

A summary of the data of the refractive index and the most commonly occurring oxides is presented in Table 5.1. The mean, and standard deviation fromthe mean, is provided for the appropriate subgroups such as country of origin, or type of glass, for each glass classification.

There are many significant features highlighted in Table 5.1. The float glasses from the three represented countries have a remarkable consistency about their data including the virtual absence of alumina and potash. The sheet glasses from Japan have consistent data and hence dominate the mean results for this classification. Each source of patterned glass has its own characteristic data. The Australian wired glass differs from the Japanese imports. The fourteen windscreens have come from eight different world wide car manufacturers so a common composition is not expected. Most containers used within Australia are manufactured locally in each State by the same national company. The two Victorian containers are repeated samples from a pharmaceutical pack and has an unusual composition. The thirteen overseas containers originate from nine countries and hence again there is a variability of data. The borosilicate headlamps have a fairly uniform set of data but the two soda-lime-silica headlamps are quite different.

It can be seen that the borosilicate labware differs mainly in potash and lime to the borosilicate headlamp. A characteristic of the soda-lime-silica spectacle glasses is their potash content in excess of 5.0%, the only other glasses with such a potash THE TANK THE TANK OF DATA FROM GLASSES USED WITHIN AUSTRALIA

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A:ORIGINAL CLASSIFICATIONS

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TABLE 5.1 Cont..

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B: FINAL FLAT GLASS CLASSIFICATIONS

composition being lead glasses. It is difficult to make further comments upon the light bulbs, spectacles, tableware and labware due to the lack of sample numbers in each subgroup.

The data obtained is also presented in histogram form so as to visually display the similarities and differences of the various chosen glass classifications.

The differences in the various flat glasses is displayed in Figure 5.1 in which the refractive index is plotted for all the flat glasses, and for each of the five subgroups as well as for
the entire glass museum. The float glasses have all very The float glasses have all very constant values apart from three American samples. The distribution of the data for sheet glasses reflect the country of origins, besides being all lower than the value of float glass. The wired glasses despite being mainly from one country indicate a bimodal distribution, which could suggest two sources from within Japan. The patterned glasses again reflect the fact that the origin of the samples is mainly from two countries. The windscreens have a wider distribution due to their multinational origin. A check across classifications suggests that the single Japanese patterned glass is from the same source as one set of the Japanese wired glasses. The small number of samples make this cross interpretation suspect but the histograms themselves support the concept of maintaining the five classifications of flat glasses.

Recognising the lack of samples in each classification the histograms for the other parameters have been restricted to those classifications or groups with 20, or more, samples. This limits the plots to four categories, namely, float, container, all the flat glasses and the total collection.

The data for the soda content does not reveal in the initial plots, Figure 5.2 (a), (b), (c), (d), much differences between flat, float and containers. With the expanded plots in Figure 5.2 (e) and (f) the uniformity of the float glass compositions is again emphasised.

The magnesia data in Figure 5.3 demonstrates the usual magnesia content in flat glasses and its absence in containers, although of course exceptions exist. The magnesia content in floats do not appear to differ much from the bulk of other flat glasses.

The alumina data is plotted in Figure 5.4. These figures clearly reveal float glasses as being unique in having zero, or practically zero, content of alumina. However, other flat glasses have a wide range of alumina content while many containers are seen to have a fairly restricted range of alumina content.

The silica plots of Figure 5.5 do not help distinguish between flat, floats or container classifications.

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a-Total Glass Museum

- b-Total Flat Glasses
- c-Float
- d-Sheet
- e-Wired
- f Patterned
- g Windscreen

a - Total Glass Mus<mark>eum</mark> b - Total Flat Glass c - Float d - Container

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- a-Total Glass Museum
- b-Total Flat Glass
- c-Float d- Container
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- a, b Total Glass Museum c - Total Flat Glass d- Float
	- e -Container

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- - d Container

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There is a wide range of potash content in the various glasses as shown in Figure 5.6 (a). However most of the glasses have a potash content of less than 1% so the accompanying plots of Figure 5.6 (b), (c), (d) and (e) are for this restricted range. The floats all have negligible, or zero, potash content but are not alone in that feature as some other flats and some containers also have this same lack of potash.

Finally Figure 5.7 displays the lime data and indicates that the lime content in soda-lime-silica glass is in the range 0 to 12.2%. A reasonably wide range of lime composition occurs in the flat and container classifications but its content in float glasses is again in a very restricted range.

5.3 Discriminant Analysis

The glass data bank of the initial 134 glasses has been examined using the SPSS subprogram DISCRIMINANT. Seven of the original samples have been excluded from this procedure so as to maintain the original eleven classifications with closely defined elemental compositions. The excluded glasses are the lead glasses (2), high alumina glass (1), soda-lime headlamps (2), and the soda-lime labware (1). The variables used have been limited to the refractive index and the oxide compositions of Na, Mg, Al, Si, K and Ca.

Beforeproceeding to discuss the results of the discriminant analysis it is appropriate to outline the information available during the execution of the subprogram DISCRIMINANT. For full details refer to Nie (1975).

5.3.1 Introduction to Discriminant Analysis

The aim of discriminant analysis is to statistically distinguish between two or more groups of cases by relying upon discriminating variables that measures characteristics on which the groups are expected to differ. It is necessary to weight and linearly combine the discriminating variables in some fashion so that the groups are forced to be as statistically distinct as possible.

Discriminant analysis attempts to do this by forming one or more linear combination of discriminating variables. These "discriminant functions" are of the form

$$
D_i = d_{i1} z_1 + d_{i2} z_2 + \dots + d_{ip} z_p
$$

where D_i is the score on discriminant function i, d's are weighting coefficients and Z's are standardised values of the p discriminating variables. The maximum number of discriminant variables is one less than the number of groups, or equal to the number of variables, whichever is smaller.

The program includes tools for the interpretation of data.

Statistical tests are included for measuring the success with which the discriminating variables actually discriminate when combined into discriminant functions. It may be possible to obtain satisfactory discrimination with fewer functions. The program enables the original set of cases itself to be classified so as to see how many cases are correctly classified by the variables used, and to classify new cases with unknown membership.

The standardised canonical discriminant functions coefficients are tabulated during the running of DISCRIMINANT. The coefficients corresponds to the discriminant score for a case in which the original
discriminating variables are in standard form. The discriminating variables are in standard form. discriminant score is computed by multiplying each discriminating variable by its corresponding coefficient
and adding together these products. There will be a and adding together these products. separate score for each case on each function. The coefficients have been derived in such a way that the discriminant scores produced are in standard form. means that, over all cases in the analysis, the score from one function will have a mean of zero and a standard deviation of one. Thus, any single score represents the number of standard deviations that case is away from the mean for all cases on the given discriminant function. If there are several discriminant functions, each case will have a score on each function. By averaging the scores for the cases within a particular group, a group mean on the respective function is obtained. For a single group, the means on all the functions are referred to as the group centroid, which is the most typical location of a case from that group in the discriminant function space. A comparison of the group means on each function tells us how far apart the groups are along that dimension. The functions are arranged in order of decreasing importance, so that a given difference between group means on the higher number function is not as meaningful as the same difference on a lower number function.

The standardised discriminant function coefficients are of great analytic importance in and of themselves. Each coefficient represents the relative contribution of its
asociated variable to that function. The sign merely asociated variable to that function. denotes whether the variable is making a positive or negative contribution.

Having derived discriminant functions it is possible to use them in a classification procedure. By classification is meant the process of identifying the likely group membership of a case when the only information known is the case's values on the discriminating variables. Alternatively the adequacy of the derived discriminant functions may be determined by classifying the cases used to derive the functions in the first place and comparing predicted group membership with actual group membership. The measure of success is the proportion of correct classifications.

5.3.2 Choice of Classification Groups using Discriminant Analysis

The subprogram DISCRIMINANT of the SPSS package has been run on 127 samples taken from the initial glass museum of 134 specimens. The seven excluded glasses are the lead glasses (3), a high alumina glass (1), the soda-lime headlamps (2) and the soda-lime labware glasses (1). These 127 samples had originally been grouped into eleven classifications. As a result of running the DISCRIMINANT procedure two other groupings of the samples have also been adopted. The details of these three sets of groupings are shown in Table 5.2.

TABLE 5.2

CLASSIFICATION GROUPINGS USED IN DISCRIMINANT

The discriminant functions and the standard scores of each classification's mean that resulted using Grouping No. 1 are shown in Table 5.3. Although only four of the Although only four of the functions are included in Table 5.3 all seven functions were used in the subsequent classification procedure. The important variables in Function 1 appear to be the refractive index and lime, while potash and alumina emerge as the extreme variables in Function 2. Soda and magnesia then begin to play an important role in Function 3.

TABLE 5.3: DATA APPROPRIATE TO USING GROUPING NO. 1 WITH DISCRIMINANT

A. STANDARD SCORES OF CLASSIFICATIONS¹ MEANS

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B. STANDARD CANONICAL DISCRIMINANT FUNCTION COEFFICIENTS

C. CANONICAL DISCRIMINANT FUNCTIONS EVALUATED AT GROUP MEANS (GROUP CENTROIDS)

The functions evaluated at the group means result in the borosilicates (headlamp and labware) being well separated from the other classifications in Function 1, and
spectacles being separated by Function 2. The high spectacles being separated by Function 2. negative values of Function 1 for the borosilicates is mainly due to their low values of soda, magnesia and lime. Surprisingly the low refractive index associated with borosilicates is not a significant variable since it would yield a positive component to the function. The high negative values of Function 2 for spectacles is due to all the variables, apart from the refractive index, contributing negative values although the potash component dominates. Function 3 is starting to distinguish tableware and containers on the one side and the flat glasses on the other side. This is due to the magnesia and alumina content. Finally the fourth function highlights the lightbulbs due to their high soda and magnesia levels.

The discriminant functions derived from Grouping No. 1 were used to classify the original 127 samples used to derive the functions. The success of correct predictions within both first choice only and first and second choices is shown in Table 5.4, along with details for the other two groupings. The success rate of predictions with Grouping No. 1 was not that good. Since the borosilicates are readily identified from the experimental derived data by their low values of refractive index, soda and lime they were excluded from subsequent groupings. The hope was that better success 'rates could be obtained on classifying more similar glasses. In addition the windscreens were excluded since they were obviously made from a range of flat glass categories and hence spoiling the singularity of the other nominated flat glass classi-It was also decided to exclude the two Victorian containers from the data bank since they were from a pharmaceutical pack that had an unusual composition.

	PERCENTAGE CORRECT	
GROUPING NO.	1ST CHOICE ONLY	1ST AND 2ND CHOICE
	68.5	
	92.1	99.0
	88.3	96.8

TABLE 5.4 ACCURACY *OF* PREDICTIONS USING DISCRIMINANT

After several runs of DISCRIMINANT using various groups of classification it became apparent that the patterned and wired glasses should be grouped together as rolled glass and four subgroups be introduced. The first two subgroups are for some Japanese glasses with a low magnesia content

(~0.5%) and for Belgium glasses with a high magnesia content (~4.5%). The other two subgroups are for some Australian glasses with magnesia content of 3.1% and 3.6% respectively. Since our understanding of the Australian scene is that no sheet glass is currently manufactured, the term Non Float Flat (Australian) has been adopted for these two subgroups. It was found that no single grouping of classifications by itself yielded total prediction
success. The proposed final scheme is a compromise The proposed final scheme is a compromise involving two sets of classification groupings and then inspecting these results before making a final decision
upon the classification. The two sets of groupings are upon the classification. The two sets of groupings shown in Table 5.2.

It is seen in Table 5.5 that only three discriminant functions result from using the four categories of Grouping No. 2. Upon considering these functions and the standard scores of each classifications' mean it is seen that Function 1 yields a high negative value for This is because all variables result in a negative contribution to the discriminant function
evaluated at the group mean. The magnesia content evaluated at the group mean. dominates Function 2 so that this function distinguishes the flat glasses from the containers/tableware. Finally light bulbs are highlighted in Function 3 by their high soda and low refractive index values which has more than compensated for the negative contribution due to the low Grouping No. 2 is a very coarse set of classifications but leads to the reasonable accuracy of predictions shown in Table 5.4. It is seen that there is a 99.0% probability of the correct grouping being among the first and second probability.

Inthe third grouping spectacles were excluded since their potash content singularly weight the standard scoresfor this variable. The discriminant functions and the standard scores of each classification's mean that resulted from using Grouping No. 3 are shown in Table 5.6. Again only four functions are shown although all seven were used in the subsequent classification procedure. The important variables in Function 1 are the magnesia and alumina content. The extreme variables in Function 2 are refractive index and alumina whilst refractive index and silica emerge as the extremes in Function 3. Finally refractive index and lime become the extreme variables in Function 4.

The functions evaluated at the group means results in the container like categories, namely containers, tableware and rolled (Japan, low MgO), being well separated from the flat glasses by Function 1. This is due mainly to the magnesia content. The lightbulb and sheet glasses are separated from the flat glasses by Function 2 due mainly to the refractive index and alumina while tableware is diverging away from containers.

TABLE 5.5 DATA APPROPRIATE TO USING GROUPING NO. 2 WITH DISCRIMINANT

GROUP Flat Lightbulb Spectacle Container/ Tableware Mean Std.Dev. RI -0.1 -2.1 0.9 $\mathbf{0}$ 1.5203 0.0036 Na20 0.1 2.0 -2.8 0.2 13.5 1.3 MgO 0.8 0.8 -1.1 -0.8 2.0 1.6 A1203 -0.4 0.7 -0.1 0.6 1.2 0.9 Si02 0.3 -0.3 -1.6 0.0 72.4 1.2 K20 -0.3 -0.1 3.4 -0.2 0.8 1.9 CaO -0.3 -2.3 -0.9 0.6 9.4 1.5

A. STANDARD SCORES OF CLASSIFICATIONS' MEANS

B. STANDARD CANONICAL DISCRIMINANT FUNCTION COEFFICIENTS

C. CANONICAL DISCRIMINANT FUNCTIONS EVALUATED AT GROUP MEANS (GROUP CEOTHOIDS)

TABLE 5.6 DATA APPROPRIATE TO USING GROUPING NO. 3 WITH DISCRIMINANT

GROUP Float Sheet Rolled (Other) Non Float Flat (Aus 3.6% MgO) Non Float Flat (Aus 3.1% MgO) Rolled (Japan) Rolled (Belgium) Lightbulb Tableware Container Mean Std.Dev. RI 0.1 -1.7 0.5 -0.4 0 0.9 1.3 -2.4 -0.8 0.3 1.5199 0.0031 Na20 Ω -0.2 -0.3 -0.3 0.1 -1.7 -0.3 2.6 1.3 0 13.8 0.9 MgO 0.9 0.9 0.4 0.9 0.6 -1.0 1.1 0.7 -0.9 -0.9 2.2 1.6 A1203 -1.2 0.6 0.3 -0.2 0.3 0.6 -0.9 0.7 0.2 0.7 1.2 0.9 Si02 0.5 0.5 -1.0 0.4 -0.5 -0.1 -0.7 -0.6 0.3 -0.4 72.6 1.0 K20 -0.8 0.6 0.6 -0.4 -0.6 0.4 -0.4 0.6 -0.4 0.2 0.4 0.5 CaO -0.4 -1.6 0.1 -0.7 -0.5 1.5 0.2 -0.4 -0.3 0.5 9.5 1.5

A. STANDARD SCORES OF CLASSIFICATIONS' MEANS

B. STANDARD CANONICAL DISCRIMINANT EWCT1CN COEFFICIENTS

C. CANONICAL DISCRIMINANT FUNCTIONS EVALOATED AT GROUP MEANS (GROUP CENTROIDS)

Tableware continues to diverge from containers in Function 3 due to their refractive index and lime values. These same variables contribute to the float and rolled (Belgium) glasses being separated by Function 3. Certain fine tuning of the data is being contributed by Function 4. It is seen that rolled (other) has remained fairly close to the means of the values of each function since its standard scores are all fairly low. Likewise the contributions due to the potash variable are also close to the mean values of each function.

The success of Grouping No. 3 is shown in Table 5.4. An increase in the prediction success can be achieved by applying the following rules.

- The correct classification is one of the two choices from Grouping No. 2.
- The correct classification is one of the two choices from Grouping No. 3.
- . A flat glass needs to have two flat categories named in Grouping No. 3 except for rolled (Japan, low MgO) which are always associated with container. The first choice of the two named flat categories is taken as the correct classification.
- . The non-flat category should be taken if it occurs associated with a flat category, except for rolled (Japan, low MgO) when associated with container.
- When two non-flat categories occur then use the first choice.
- . All final decisions on classifications should be made after checking the original values of the variables, especially the magnesia content of flat glasses and high soda and low refractive index for tableware compared with container.

Using this approach the success rate of prediction of the correct classification of the 94 cases in Grouping No. 3 is 91.5%. However, if some uncertainty between tableware and containers, and between which flat glass, can be tolerated then the prediction rate is raised to 95.7%. The other four samples then not correctly classified are

- (i) a Japanese wired glass containing 1.8% magnesia,
- a West German tableware,
- (iii) the Filipino sheet glass, and
- (iv) a South African container.

Using this procedure on the fourteen windscreens that were originally excluded from the original 127 samples yields a 100% success in the prediction that the glasses are from a flat category. Since the spectacle and borosilicate glasses are also correctly predicted then the overall success rate of prediction of the classification of the original 127 samples is 96.9%.

5.3.3 Discriminant Analysis on New Samples

The initial glass museum of 134 samples has been extended by an additional sixteen samples of known origin so these were examined using DISCRIMINANT and the Groupings No. 2 and 3. The results are summarised in Table 5.7. Sample GM135 was excluded because it was another specimen of the container from Victoria which is from a pharmaceutical pack of unusual composition. Of the remaining fifteen, eleven were correctly predicted, two (GM137 and GM146) were partly predicted, one (GM145) predicted as an unusual glass and the fourth (GM150) predicted as a rolled (other) whilst it was labware. Sample GM145 was in fact a sodalime labware, a classification not in the groupings used. The use of discriminant analysis on this limited sample of fifteen additional glasses give encouragement to its usefulness as a means of classification of glasses. Its use on case study glasses is discussed in a later chapter.

5.4 Cluster Analysis

5.4.1 Introduction

A second approach to the statistical analysis of the glass data has been to use the cluster analysis program CLUSTAN 1C produced at University College, London, and described by Wishart (1978). In particular the data file has been obtained from that used in the discriminant analysis using procedure SPSS and the distance matrix generated using
procedure CORREL. The procedure HIERARCHY has been used The procedure HIERARCHY has been used using the transformation method suggested by Ward (1963), and this then followed by the procedure TREE.

In this program the N objects, each of which is measured on each of p variables, are expressed in a distance matrix. The hierarchical technique then proceeds to fuse individuals, or groups of individuals, which are closest to each other. By a series of successive fusions the N individual entities can eventually be reduced to a single group. The results may be presented in a form of The results may be presented in a form of dendrogram,or tree, which is a two dimensional diagram illustrating the fusion which have been made at each successive level.

The method is expected to place similar glasses alongside each other on one of the branches of the tree. However, it should be remembered that only a two dimensional diagram is being used, and a fusion once made is irrevocable, so that alternate ordering in the tree structure can result from slight variation in data or additional samples.

TABLE 5.7: CLASSIFICATION OF NEW SAMPLES OF NOWN ORIGIN USING DISCRIMINANT

5.4.2 CLUSTAN on Initial Data Bank

The same original data used as that used for DISCRIMINANT has been used for CLUSTAN. This means that seven glasses This means that seven glasses have been excluded from the original 134 glass samples and the variables limited to seven namely, refractive index and the oxide compositions of Na, Mg, Al, Si, K and Ca. An inspection of the dendrogram obtained using these 127 qlasses is upon first glance rather confusing. However, glasses is upon first glance rather confusing. upon closer examination it is possible to distinguish five main groups that can be broadly identified as non-float flat glasses, float glasses, containers, spectacles and borosilicate glasses. The apparent mismatches within these five groups reveal significant subgroups so that there is an extension to the number of classifications that can be identified.

A simplified version of the dendrogram is shown in Figure 5.8. The group with the major number of subgroups is seen to be the non-float flat glasses. The five Japanese sheet glasses, the two containers from Victoria together with a lightbulb, and the two flat glasses with magnesia content of 2.3%, each form distinct subgroups. There are two subgroups of Australian flat glasses with a magnesia content of 3.1% and 3.6% respectively. A further subgroup of four members results from two lightbulb, the Filipino sheet and a West German tableware. There then only sheet and a West German tableware. remains three containers (South African, British, and French) and an Italian tableware that are mismatches among the total number of forty three glasses within this group of non-float flat glasses.

The group of float glasses contain a total of 27 samples. At one end of the group there occurs a subgroup of rolled glass from Belgium having a magnesia content of 4.5%. Neighbouring this subgroup are two USA float glasses that upon inspection of their original data have higher
refractive indices than the other float glasses. In fact refractive indices than the other float glasses. their refractive indices match closely with those of this Belgium subgroup. The only non-float glass located within this group is a rolled glass from Belgium that has a magnesia content of 3.9%.

The next group is that of containers consisting of 35 members. At one end next to the float glasses are three flat glasses that can be identified as rolled glass from Japan with a magnesia content of 0.5%. The only other subgroup is located centrally within the group and contains four of the tableware samples. Significantly all four are Australian products while the other tableware is dispersed throughout the dendrogram. The only further non-container in the group is an Italian tableware glass.

The fourth group contains initially the seven spectacles all next to each other. At one end are positioned five

Figure 5.8 Simplified dendrogram of original samples using clustan

imported containers from various countries and then an Argentine tableware followed by a borosilicate headlamp.

The fifth and final group contains the remaining eight borosilicates with no other intruder.

The dendrogram produced in this work has been reasonably successful in sorting the original data into groups. Duplicate samples from the same source of glass have been placed alongside each other. Likewise the five Japanese
sheet glasses have been located together. It has sheet glasses have been located together. supported the existence of the rolled glasses from Belgium with a magnesia content of 4.5% and the rolled glasses from Japan with a magnesia content of 0.5%. It has highlighted the non float flat categories of Australian flat glasses with a magnesia content of 3.1% and 3.6% respectively. These four groupings have arisen out of the discriminant analysis. It has also revealed the existence of rolled glasses with a magnesia content of 2.3%, and a group of Australian tableware. Also it has shown the existence of the subgroup of USA float glasses that have a higher refractive index than other float glasses.

An extra test of the procedure is by inspecting the placement of the windscreen samples that occur in the original data. All fourteen samples are located within the two flat glass categories. Furthermore, they occur distributed as follows: Float (3), Sheet (3), Non float flat (Australian, 3.6% MgO) (5), Rolled (Others) (2), Rolled (Others, 2.3% MgO) (1).

5.4.3 CLUSTAN on New Samples

The sixteen samples of known origin that have been added to the initial glass museum have each in turn been added to the original 127 samples and run on CLUSTAN. Running eachsample separately results in the minimum amount of change in the original ordering within the dendrogram. The location of the addition within the dendrogram has been noted and interpreted on the basis of the original
data. The results are summarised in Table 5.8. The results are summarised in Table 5.8.

Of the sixteen samples thirteen have been correctly predicted. Of the others, sample GM145 is predicted as a Sheet (Japan) but it differs quite significantly from this group. This sample has a lowrefractiveindex andhigh potash content so does not fit any of the established classes. In fact the sample is a soda-lime labware, a classification not in the groupings used. Sample GM147 although a rolled glass from Belgium only contains 4.1% magnesia so does not fit the special group of rolled (Belgium, 4.5% MgO). Consequently it fits better into the float glass category. Finally GM150 is a soda-lime Finally GM150 is a soda-lime labware so again, like GM145, this is a classification not in the groupings used.

TABLE 5.8 CLASSIFICATION OF NEW SAMPLES OF MOONN ORIGIN USING CLUSTAN

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CHAPTER 6 CASE STUDIES INVOLVING GLASS FRAGMENTS

6.1 Introduction

The collection of the 177 samples for the glass museum has included 27 items from forensic and non-forensic case studies. The results of processing them through the various techniques developed in this work are reported upon below.

6.2 Case 1 Glass Nos. GC151-159

This involved a murder in which the victim's throat was cut. Investigating police wished to determine the possible movements of the victim prior to his death. Sweepings from a suspect's car revealed a number of glass particles. The classifications given by DISCRIMINANT and CLUSTAN are shown in Table 6.1. The predicted classification of lightbulbs glass for six of the fragments was interesting since the victim's job was to repair faulty pin ball machines and he was known to carry spare bulbs
for the purpose. Reference to the original data for refractive Reference to the original data for refractive indices and elemental analyses clearly reveals that the fragments are from more than one broken lightbulb. The decision to select for GC153 the classification of Rolled (Other) was based upon the fact that it is a flat glass which contains 0.6% K20. The other two fragments were predicted as container glass.

6.3 Case 2 Glass Nos. 160-161

This case involved a wilful damage incident in which a wired glass screen was alleged to have been smashed with a mattock. Particles in the handle of the mattock near the head were presented for comparison with the screen. The elemental analyses showed dissimilarity in that the glass from the mattock contained
no potash compared to 0.7% K20 in the screen glass. The two no potash compared to 0.7% K20 in the screen glass. glasses were predicted by DISCRIMINANT to be different flat Their actual predictions are in doubt due to the potash content of GC160 and the magnesia content of GC161. Both glasses caused a rearrangement of many of the flat glasses in the dendrogram using CLUSTAN, but they each finished up in a different subgroup. The conclusion was that they are both unusual glasses of different composition, especially potash.

6.4 Case _3 Glass Nos. QC162-163

This case concerned breaking, entering and stealing from a motor vehicle. Glass particles from a suspect were submitted for comparison with glass from the victim's car. No differences in refractive index or chemical composition were detected and both glasses were clearly classified as float glass. See Table 6.1. The trace elements revealed by the x-ray induced technique are also very similar so suggesting that the two samples are from common sources. It has to be remembered, however, that many car windows are made from float glass.

6.5 Case 4 Glass No. GC164 The Interest in this case was to attempt to locate the classification and hence origin of a glass fragment found in a loaf of bread. From Table 6.1 it is seen that both statistical

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procedures predicted the fragment to be of lightbulb glass.

6.6 Case 5 Glass Nos. GC165-168

The requirement in this case was to compare glass fragments on a breaking and entering suspect with windows from broken-in premises. A comparison of the data showed distinct differences in refractive index and composition between the samples from the suspect and those from the crime scenes. The classifications predicted for these samples are shown in Table 6.1. The DISCRIMINANT analysis clearly indicate that the fragments were all flat glasses. Those on the suspect were similar to each other but different to those from the broken windows. In addition the two window fragments are different to each other. Interestingly CLUSTAN had difficulty in separating QC165,166 and 168 despite their refractive index differences. Sample GC168 also has a lower magnesia content from that associated with the classification predicted for it on both statistical procedures. The chemical composition of sample GC167 indicates the presence of alumina and potash so is not that expected for a float glass. Consequently the non float flat (Aus, 3.6% MgO) classification has been chosen as the final prediction. The two fragments from the suspect (GC165,166) have had the final prediction of rolled (other) assigned to them due to their potash content of 0.5%.

6.7 Case 6 Glass No. GC169 A sliver of glass that had been found in a sandwich needed to be classified in this inquiry. The results from both statistical procedures, as shown in Table 6.1, was for a container glass.

6.8 Case 7 Glass Nos. 170-172

Particles from the boot of a breaking and entering suspect were submitted for comparison with windows from two broken premises. No differences in refractive index or chemical composition were detected between the suspect and shop 1 glass samples. Glass from shop 2 was distinctly dissimilar in these respects. The DISCRIMINANT analysis yielded a first choice of rolled (other) and second choice of tableware for samples GC170 and 172. Using the rules developed during this project this leads to a prediction of tableware despite the fact that GC170 is known to be a flat glass. The third glass, GC171, is predicted as a flat glass of the float classification. The use of CLUSTAN puts GC170 and GC172 in the identical place at one end of the non float flat (Australian, 3.6% MgO) category, while GC171 is confirmed as float glass. It appears that the glass from shop 1 window and the shoe of suspect are from a similar source of a flat glass not within our original glass museum.

- 6.9 Case 8 Glass No. GC173 Fractured glass particles found in sugar were submitted for identification. They were shown to *be* pure silica glass particles.
- 6.10 Case 9 Glass Nos. GC174-175 This case involved an assault with a hammer on a detective in a remote location. The offender then damaged the police vehicles's

windscreen and fled. The hammer was subsequently recovered from the suspect. Glass particles lodged in the hammer handle were compared with the damaged windscreen glass and no differences in RI or chemical analysis could be detected. The statistical analysis for both glasses gives the category of float from DISCRIMINANT while CLUSTAN places them at the end of the nonfloat flat grouping neighbouring the float glasses. The final prediction was that the glass fragments were from a similar source of flat glass not within our original glass museum. This and other evidence led to conviction.

6.11 Case 10 Glass Nos. GC176-177

The premises of a car rental company were burgled at night after thieves had gained entry via a broken window. The window was covered with adhesive tape prior to being broken. Suspects were interviewed the following day and police took possession of, amongst other things, a wooden handled claw hammer. Some fine particles of glass impacted into a crevice in the hammer handle were revealed by examination under an optical microscope. An inspection of the RI and chemical composition clearly reveal that the two samples are from similar sources. It is interesting to see the predicted classifications. DISCRIMINANT yields the prediction of non-float flat (Australian, 3.1% MgO) while CLUSTAN places then at the end of the non-float flat grouping next to the float glass. The conclusion was that they were glass fragments from a similar source of flat glass not within our original museum. The suspects subsequently pleaded guilty in this case.

CHAPTER 7 CONCLUSIONS

The activities of the project have been aimed at implementing a rapid and sensitive non-destructive method for identifying and comparing small glass fragments that may be significant in a forensic context. The tendencies of previous workers in glass analysis have been to supplement the refractive index information with the analyses of the trace element components of the glass. The approach in this work has been primarily to analyse for the major and minor constituents of the glass matrix. The trace elements have only been used to provide extra discriminating information when necessary. This approach has been based upon using a SEM/EDS system, a facility that is becoming more
widely available and commonly used by forensic scientists. In widely available and commonly used by forensic scientists. previous chapters the results from this method have been reported and they illustrate the potential of this approach.

In assessing the success, or otherwise, of this work it is worth considering the various activities which were initially put forward in the proposed outline of the research.

With the stated aim to obtain data from glass fragments that may be significant in a forensic context it was necessary to know the likely range of glass products that might be encountered. Consequently a review was undertaken on the uses of glass within Australia and their sources. This has been documented in Chapter 2 where it can be seen that although the majority of flat and container glass used within Australia is locally made there is a significant amount imported. Furthermore the imports come from a large number of countries so potentially causing considerable difficulties to the Australian forensic scientist.

A collection of 177 glass samples has been made from a variety of sources and initially classified under the headings of float, sheet, patterned, wired, windscreen, lightbulb, headlamps, spectacles, tableware, labware and containers. The museum is clearly not representative of the overall Australian scene but reflects those glasses more readily obtained in Western Australia. Continuing effort and resources are required to constantly update and expand the museum. It had been hoped to collect manufacturer's specifications on these glasses but this is one area in which the companies insisted upon confidentiality of information. Consequently all elemental data expressed in this document is that determined by the research team.

A major activity of the project has been in improving the detection limits of the SEM/EDS system. Quantitative elemental analysis are now possible using the conventional electron beam technique with a detection limit of 0.1%. In addition, the x-ray induced technique has been developed and implemented so resulting in semi-quantitative analysis with a detection limit down to 30 ppm for certain elements. Consequently the major and minor elements can be quantitatively analysed while the trace elements analyses are semi-quantitative. The accuracy of the electron induced analysis have been tested using a range of standard MBS glasses. A satisfactory precision has been obtained.

Data has been obtained from both techniques for all the 177 glasses in the museum. This data together with refractive indices has been used This data together with refractive indices has been used in the three statistical analyses of the data.

For comparative analyses, quantitative electron induced major-minor elemental data has been shown effective in highlighting glasses from different classifications. This greatly enhances the predictions achieved by refractive index measurement alone. The "trace" surveys obtained with the x-ray induced technique further facilitates classification. Further work remains to fully assess this
information. The DISCRIMINANT procedure of SPSS and the CLUSTAN The DISCRIMINANT procedure of SPSS and the CLUSTAN package have both been found to be very useful in predicting the classification of a single glass fragment. The data bank used as a reference contains only 134 samples so that prediction successes are impressive. Even when the unknown fragment is of a classification not within the original selections there is a good chance that this will be highlighted by the two statistical packages. This is shown by each procedure predicting a different classification since they both operate on different selection criteria.

The use of DISCRIMINANT and CLUSTAN has lead to a revision of the classifications used. The changes are all associated with the glasses originally classified as patterned or wired. The collective term of rolled glass has been used for these glasses. However, since sheet rolled glass has been used for these glasses. glasses are no longer produced in Australia the term non-float flat has been adopted for such glasses of known Australian origin. The final list of classifications is now float, sheet, rolled (other), non-float flat (Australian, 3.6% MgO), non-float flat (Australian, 3.1% MgO), rolled (Japan), rolled (Belgium), lightbulb, spectacles, tableware, container, and borosilicates.

These three statistical procedures have given great confidence in the approach adopted to use the major and minor elements only as a means to identify closely related samples, and classify, glasses. This is significant to other workers since it eliminates the needs for trace element analyses that may be time consuming. In the SEM/EDS system all the elements are detected simultaneously in an analysing time of 50s.

The various procedures developed in this work have been applied to several fragments of glasses from case studies. In some instances the data obtained from the glass fragments reveal directly whether they are similar or not. The use of the statistical package to predict the classification of a glass has been successful on many samples. Doubts in the predicted classifications have arisen on a few samples and it appears that the fragments are from glasses not within the original collection. Such problems are expected to occur until the glass data bank itself is much larger but at least the statistical procedures do tend to highlight when this occurs.

Activities have been carried out on all the various facets of the original research proposal. This document contains the status of this work at the end of the funding period. There exists the potential, and the need, to extend the glass data bank and to refine some of the procedures that have been implemented.

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APPENDIX A

INTERIM REPORTS TO THE CRIMINOLOGY RESEARCH COUNCIL

- TOPIC A: Optimum voltage for maximising peak to background ratio,
- TOPIC B: Variation of total background with accelerating voltage.
- TOPIC C: Variation in x-ray spectra with input count rate.
- TOCIC D: Variation of detector count rates with sample-detector distance.
- TOPIC E: Reduction of extraneous x-rays in a Jeol JSM 35C scanning electron microscope. (Available as School of Physics and Geosciences Internal Report SPG 267/1981/ AP 11.)
- TOPIC F: X-ray spectra handling and library facility. (Available as School of Physics and Geosciences Internal Report SPG 257/1981/AP 2.)
- TOPIC G: Homogeneity of sample and reproducibility of results.
- TOPIC H: Effects of tilting polished samples.
- TOPIC I: Refractive index determinations for soda-lime-silica glass.
- TOPIC J: X-ray spot size determination.
- TOPIC K: Performance of the amplifier.
- TOPIC L: Dead-time correction of the EDS system.
- TOPIC M: Atomic absorption analysis of glasses.
- TOPIC N: Quantitative electron induced x-ray fluorescence analysis of glasses in the SEM. (Available as School of Physics and Geosciences Internal Report SPG 272/ 1981/AP 16.)
- TOPIC 0: Accumulated data on glass museum samples.
- TOPIC P: Homogeneity of glass across thickness.
- TOPIC Q: Comparative glass analysis using multiple continuous measurements - data analysis.
- TOPIC R: An initial study of a discriminative analysis to predict glass categories.
- TOPIC S: Inter-laboratory comparison of quantitative elemental analysis of glass.
- TOPIC T: X-ray induced x-ray data on glass museum samples.

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TABLE 3.1

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TABLE B.1

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Only flat window glass are accurate thickness
measurements. Remainder are nearest approximations.
No entry indicates sample unsuitable for thickness
measurement.

Pluorescence with 253.7 mm light (U.V.).

NOTE 3.

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NOTE 2.

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TABLE B.1

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TABLE B.2 FLOAT GLASSES

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TABLE B.3 SHEET GLASSES

TABLE B.4 ROLLED (OTHERS)

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TABLE B.7 ROLLED (JAPAN, 0.58 Mg0)

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TABLE B.10 HEADLAND CLASSES

TABLE B.11 LIGIT BULB GASSES

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TRELEWARE CLASSES **TABLE B.13**

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TABLE B.14 LABWARE GLASSES

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TABLE B.15 (CONT'D) CONTAINER CLASSES

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GLASSES2. RWT

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APPENDIX C

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X-PAY INDUCED ELEMENTAL ANALYSIS DATA

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ACCIMULATED DATA OF GLASS MUSEUM SAMPLES 1-172

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THE C.1 CONDUCTED DATE OF GLASS MUSEUM SAMPLES 1-172

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ACCUNULATED DATA OF GLASS MUSEUM SAMPLES 1-172 TABLE C.1

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Only flat window glass are accurate thickness
measurements. Remainder are nearest approximations.
No entry indicates sample unsuitable for thickness
measurement.

NOTE 2.

When the ratio of Sr(K)/Sr(K) falls below a value
of 3 it indicates the presence of $2r$ as the $2r$ (K)
overlaps the Sr K peak.

Pluorescence with 253.7 mm light (U.V.).

NOTE 3. NOTE₄. l,

TABLE C.2 FLOAT GLASSES

TRIE C.3 SHET GASSES

TABLE C.4 ROLLED (OTHERS)

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TABLE C.9 WINDSCREEN CLASSES

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TRIE C.10 HEADLAND GASSES

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TRUE C.12 SPECTACLE CLASSES

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TABLE C.13 TABLEWARE GLASSES

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TABLE C.14 LABWARE GLASSES

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TABLE C.15 CONTAINER GLASSES

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TABLE C.15 (CONT'D) CONTAINER GLASSES

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TABLE C.15 (CONT'D) CONTAINER GLASSES

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CRIMINOLOGY RESEARCH COUNCIL PROJECT 9/80

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