

J. Andrasko, ¹ Ph.D. and A. C. Maehly, ¹ Ph.D.

The Discrimination Between Samples of Window Glass by Combining Physical and Chemical Techniques

In forensic science laboratories it is frequently necessary to compare properties of glass fragments. The forensic scientist is asked to determine if a glass fragment came from a certain automobile headlight, from a certain window, and so forth. The comparison of glass fragments by means of several physical methods is a routine operation in forensic science applications.

Physical Methods

The physical methods for glass investigation show generally high reproducibility and precision, are convenient and rapid, and do not require expensive instrumentation. Discrimination between large fragments of glass can sometimes be achieved by comparing color and by measuring the thickness of glass samples if they have smooth and undamaged surfaces. Certain glass specimens also fluoresce and can thus be distinguished from other specimens [1].²

For many years, determinations of density and refractive index have been the generally accepted and widely used methods for discriminating between samples of glass. These methods are applicable to small sample sizes and exhibit high accuracy of measurement. Both absolute measurements and comparative measurements between pairs of samples are carried out. Recent improvements in density determination have been published by Dabbs and Pearson [2] and others. Refractive index methods have been refined in later years by Ojena and De Forest [3] and others. Many authors recommend measurements at various wavelengths (dispersion).

One inherent weakness of these determinations is the well-known fact that density and refractive index are mutually dependent properties. The higher the density, the greater (in general) the refractive index. Plots of these parameters have been assembled and discussed by Dabbs and Pearson [1] and Cobb [4].

A second weakness is the narrow range of the values of these properties for the same type of glass. Thus, modern window glass (manufactured since 1945) has a refractive index in the range of 1.515 to 1.518 [1] and a density in the range of 2.43 to 2.52 [3]. Furthermore, because of the inhomogeneity of glass, the variation of refractive index and density within one sample is often greater than the error in the measurements.

To increase the evidential value of discrimination between glass samples further means of comparison are desirable. In recent years the application of chemical analysis to the discrimination of glass specimens has been shown to be promising.

Chemical Analysis

The basic elemental composition of glass samples can vary considerably and should be

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¹ Research associate and director, respectively, The National Laboratory of Forensic Science, Linköping, Sweden.

² L. Carlsson, personal communication.

examined in forensic science laboratories. The presence of trace elements arising from the raw materials and the manufacturing processes may further characterize glass specimens. Various analytical techniques have been suggested for the analysis of glass by trace element determination. Glass analyses have been done with spark source mass spectrometry [5-7], neutron activation analysis [8-10], X-ray fluorescence [11], atomic absorption spectroscopy [12, 13], emission spectrography [14], and energy dispersive X-ray analysis in a scanning electron microscope [15]. With these methods different samples of glass may be distinguished from each other even though they possess the same physical properties.

Complete analysis of the major and minor elements in glass presents a challenging problem. In this work an attempt was made to discriminate between samples of window glass by combining two methods of chemical analysis. The main effort was centered on comparative analyses using energy dispersive X-ray analysis in a scanning electron microscope. When this analysis failed to distinguish between the samples the glass fragments were analyzed for trace elements with emission spectrography.

Methods

Density and Refractive Index Determinations

Absolute values of densities for glass samples can be determined by using Archimedes' principle [1]. Comparison of glasses sent to our laboratory is, however, usually carried out by varying the density of a liquid as a function of temperature.

Refractive index was measured with a Mettler hot-stage and the Becke line technique. Clove oil and MS 710 silicon oil were used as immersion liquids. The refractive index of the immersion liquids was calibrated as a function of temperature with standard glass samples. Usually, three readings were taken on each specimen of glass. The apparatus was equipped with a monochromator, enabling the measurement of dispersion of refractive index. Our experiences as well as the data reported in the literature indicate, however, that the studies of dispersion of refractive index generally do not distinguish between glass samples with the same refractive index.

Energy Dispersive X-Ray Analysis

Energy dispersive X-ray analysis of glass samples was carried out in a Japan Electronic Optic Laboratory JSM-35 scanning electron microscope equipped with a PGT-1000 X-ray analyzer. The accelerating voltage was 25 kV.

Samples of window glass were washed with distilled water and acetone and subsequently crushed into small pieces. Thin fragments of a maximum of 1 mm in any direction and with a plane surface were selected for the analysis. The fragments were mounted on a carbon block by using paper glue. The mounting was performed while the fragments were being observed in a stereo light microscope. Care was taken to insure that the surface to be analyzed was plane and mounted in parallel with the plane of the carbon holder. After this procedure specimens were directly analyzed (uncoated) in the scanning electron microscope. To obtain reproducible results, all geometric parameters and instrumental settings were kept as constant as possible. Of particular importance was that the position of the X-ray detector, the position of the samples in the microscope chamber, and the surface tilting angle (45 deg in this work) were kept constant. The topography of the analyzed surface was further controlled by observing the backscattered electron image. The samples were then analyzed with the electron beam sweeping rapidly through a selected area of 80 by 80 μm . By performing the analysis with the electron beam in the scanning mode the effect of contamination of the glass, such as observed in the spot analysis mode [16], was not serious.

The analyses were run until the silicon peak had reached a standard height (25 000 counts, with 5 eV per channel). The form of background and the total X-ray counting rate were controlled in each analysis as these parameters should be similar for all the specimens investigated.

The following elements were found in the investigated glass samples by energy dispersive X-ray analysis: silicon, calcium, sodium, magnesium, aluminum, potassium, sulfur, chlorine, iron, and in some instances barium. Three fragments from each sample were analyzed. The best agreement between the results of these analyses on different fragments from the sample was obtained by comparing ratios of the concentrations of the elements. Therefore, the experimental concentration ratios sodium/magnesium, sodium/aluminum, magnesium/aluminum, calcium/sodium, calcium/potassium, and sometimes barium/calcium were calculated and the mean values employed for discriminating between different glass samples. The elemental ratios were computed for peak net heights of the K lines for sodium, magnesium, and aluminum; the K_{α} lines for potassium and calcium; the L_{α} line for barium. The signals for sodium, magnesium, and aluminum were not completely resolved and the level at 900 eV was taken as background for all three elements. The elemental net ratios of sodium/magnesium, sodium/aluminum, and magnesium/aluminum calculated here are consequently lower than the values obtained by more sophisticated background correction.

Emission Spectrography

For measuring the trace element content of the glass samples emission spectrography was carried out on an Ritzl, Seitner & Veith 3.5-m emission spectrograph (with a resolution of 75 600). Approximately 2 mg of glass fragments were ground in an agate mortar and blended with graphite powder. The mixture was transferred to a cup-type electrode (Ringsdorff Werke RW0052). The top electrode was RW0025, the current 8 A, analytical gap³ 2 mm, and exposure 10 to 20 s. The arcing process was carried out by direct current in an oxygen atmosphere (O_2 flow \sim 7 litres/min) with a glass cup attached to the bottom electrode. After development the resulting photographic plates were examined in a Steinheil "Doppel-Projektor." The densities of elemental lines were measured automatically by using a minicomputer and printer attached to the comparator.

Generally, only specimens that could not be distinguished by energy dispersive X-ray analysis were analyzed. In addition to the elements found in the samples at higher concentrations, manganese, titanium, chromium, arsenic, and copper could be detected by emission spectrography. Semiquantitative analyses of these trace elements were performed and the elemental ratios calculated from the intensities of the elemental lines. When necessary, the intensities of some of the weaker lines for magnesium, aluminum, and potassium were also measured. The densities of the lines given in Table 1 were determined.

Results

Material

Group A consisted of samples from two panes (about 60 by 80 cm) of window glass. One of the panes was manufactured about 15 years ago, and the second pane was new. (The panes came from two different factories.)

Group B comprised 29 samples of window glass collected in one Swedish town (about 100 000 inhabitants) over a period of several weeks. Glass from broken windows in both new and older houses were represented in this collection. The maximum dimensions of

³The gap increased throughout the burn.

TABLE 1—*Wavelengths of elemental lines.*

Element	Line, nm
Aluminum	308.22
Arsenic	234.98
Barium	350.11
Chromium	425.43
Potassium	404.72
Magnesium	277.67
Manganese	280.11
Titanium	365.35

the glass fragments varied from 2 to 20 cm and the thicknesses between about 1.8 and 3.7 mm.

Group C comprised ten samples of show window glass with thicknesses of around 6 mm and similar densities as well as refractive indexes. They were selected from a larger collection acquired from casework in our laboratory in 1952 and 1953.

Group D consisted of three glass samples from an actual case, indistinguishable with respect to refractive index and density. One of the samples was a comparison window glass, and the other two were glass fragments collected from suspects.

Group A Samples

In these samples, the variations in refractive index and elemental composition over the panes were determined. Seven locations on each pane were chosen at random. From each location four fragments were taken for refractive index determination, one fragment for energy dispersive X-ray analysis, and one fragment for emission spectrography. The results of these measurements are shown in Table 2, which also includes the results of energy dispersive X-ray analysis performed repeatedly on a single fragment.

Group B Samples

The refractive index distribution of the glass specimens is shown in Fig. 1. The distribution curve exhibits a maximum in the range 1.515 to 1.517. This curve has an appearance similar to the refractive index distribution observed by Dabbs and Pearson [1] for a large number of window glasses. Tables 3 and 4 list the refractive index (in increasing order) and the elemental composition for the samples in this group.

Groups C and D Samples

The physical properties and the elemental composition of the samples of Group C are shown in Tables 5 and 6, while Table 7 lists the refractive index and the elemental composition of the samples of Group D.

Discussion

Reproducibility of the Measurements

The results shown in Table 2 indicate a good reproducibility in elemental net ratios obtained by energy dispersive X-ray analysis of a single glass fragment. Slightly higher standard deviations were observed in the analysis of different samples from the same glass pane. This difference might be due to variations in chemical composition over the panes but more likely it stems from variations in surface geometry of the specimens analyzed.

TABLE 2—*The homogeneity of sheet glass.*

Measurement	Single Fragment	Pane A (Old)	Pane B (New)
Refractive index			
Standard deviation	0.00002 ^a	0.00004 ^b	0.000035 ^b
Observed range	0.00007	0.00023	0.00021
Energy dispersive X-ray analysis (7 determinations)			
Sodium/magnesium			
Mean	1.85	2.25	1.81
Coefficient of variation, %	1.3	1.8	2.0
Sodium/aluminum			
Mean	2.51	3.10	3.51
Coefficient of variation, %	2.3	3.2	3.4
Magnesium/aluminum			
Mean	1.36	1.38	1.94
Coefficient of variation, %	1.6	1.95	2.3
Calcium/sodium			
Mean	1.09	1.15	1.23
Coefficient of variation, %	6.6	10.1	9.2
Calcium/potassium			
Mean	6.77	13.2	16.5
Coefficient of variation, %	2.2	3.1	3.6
Emission spectrography (7 determinations) ^c			
Titanium/manganese			
Mean	...	0.27	...
Coefficient of variation, %	...	8.1	...
Potassium/manganese			
Mean	...	0.56	1.25
Coefficient of variation, %	...	10.4	14.1
Potassium/titanium			
Mean	...	1.85	...
Coefficient of variation, %	...	13.5	...
Barium/manganese			
Mean	0.14
Coefficient of variation, %	10.4
Magnesium/manganese			
Mean	0.77
Coefficient of variation, %	14.6

^a Twelve readings.

^b Four fragments each from seven locations.

^c Note that these values express the ratio of line intensities and are not proportional to absolute concentrations.

The following experiment demonstrates the dependence of measured elemental composition on one geometrical parameter—the surface tilting angle φ . A single glass fragment with a plane surface was analyzed. Figure 2 shows the intensities⁴ of the sodium and magnesium signals and the calculated elemental ratios sodium/magnesium and magnesium/aluminum as functions of φ . For making comparisons, the values of the parameters depicted in Fig. 2 were all normalized to $\varphi = 60$ deg. It is obvious that the signal intensities obtained from the analysis of several unpolished fragments from the same sample may vary considerably. The ratios between the intensities of adjacent elemental signals depend, however, much less on the tilting angle. Thus, Fig. 2 illustrates the advantage of calculating elemental ratios (rather than elemental concentrations) in such analyses. Nevertheless,

⁴ The intensities of the sodium and magnesium signals were expressed as the percentage of peak height of each element: peak height = (peak height of element/sum of all peak heights) 100.

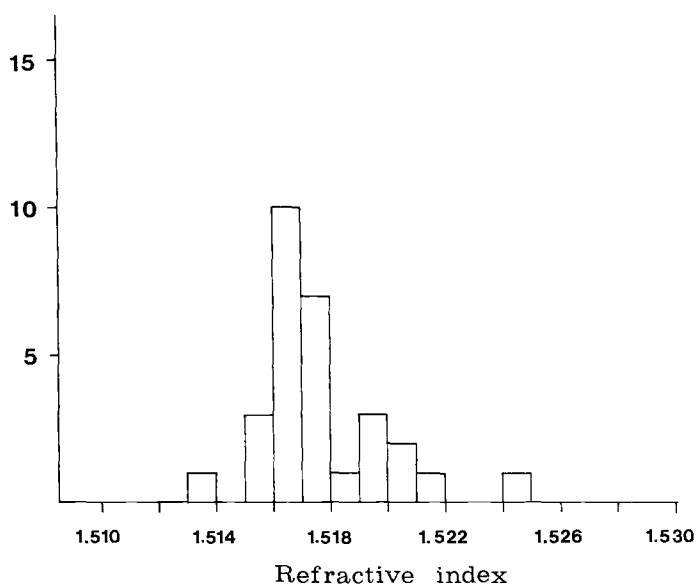


FIG. 1—The distribution of refractive index values for the glass samples of Group B.

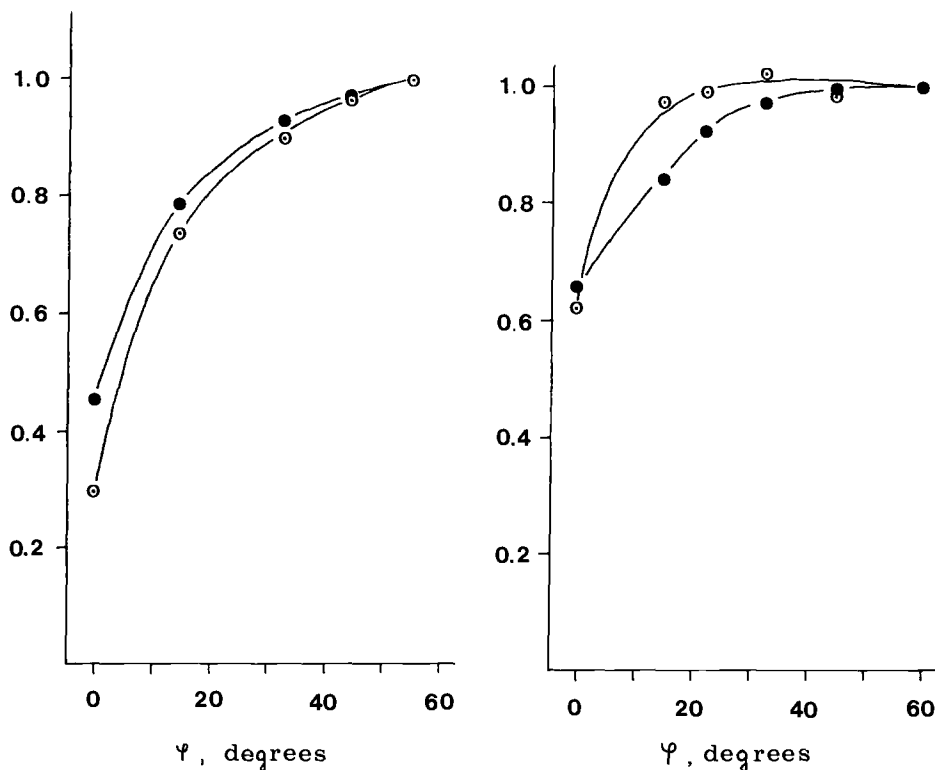


FIG. 2—The variation of measured elemental composition for one glass sample with sample geometry. (left) The dependence of the sodium (—○—) and magnesium (—●—) net peak heights on the tilting angle ψ . (right) The dependence of the elemental ratios sodium/magnesium (—○—) and magnesium/aluminum (—●—) on the tilting angle ψ .

TABLE 3—Energy dispersive X-ray analysis of window glass samples in Group B.

Sample	Refractive Index	Sodium/ Magnesium	Sodium/ Aluminum	Magnesium/ Aluminum	Sodium/ Sodium	Calcium/ Potassium ^a	Barium/ Calcium
1	1.5133	3.17	1.32	0.415	0.51	1.66	0.32
2	1.5150	1.78	3.31	1.86	0.95	31.5	...
3	1.5151	16.9	2.19	0.135	0.92	4.99	0.015
4	1.5152	13.4	2.45	0.18	0.79	7.01	...
5	1.5161	2.11	2.61	1.23	1.00	20.3	...
6	1.5162	2.04	2.55	1.25	1.15	22.0	...
7	1.5162	2.17	2.99	1.38	1.16	19.4	...
8	1.5162	1.89	2.36	1.25	1.16	15.7	...
9	1.5165	2.10	2.77	1.32	0.91	16.9	...
10	1.5165	2.20	2.63	1.19	1.12	15.3	...
11	1.5169	2.41	4.20	1.74	0.73	∞	...
12	1.5169	2.33	3.88	1.66	1.09	68	...
13	1.5170	1.81	2.18	1.20	1.16	9.2	...
14	1.5170	1.78	2.66	1.50	1.00	17.6	...
15	1.5173	2.30	3.86	1.68	1.14	70	...
16	1.5176	3.26	2.19	0.67	1.26	∞	0.022
17	1.5178	2.33	3.87	1.66	1.21	73	...
18	1.5178	2.03	2.66	1.31	1.03	26.5	...
19	1.5179	2.73	3.33	1.22	1.25	40	...
20	1.5179	2.45	3.51	1.43	1.30	25.5	...
21	1.5179	2.06	3.39	1.65	1.09	31	...
22	1.5181	8.37	1.95	0.23	1.47	11.6	0.009
23	1.5198	3.40	2.27	0.67	1.11	16.1	...
24	1.5198	3.25	4.52	1.39	1.22	∞	...
25	1.5199	2.29	3.80	1.66	1.31	55	...
26	1.5203	10.7	2.73	0.26	1.13	13.9	...
27	1.5204	2.65	2.87	1.08	1.41	26	...
28	1.5216	9.4	2.49	0.265	1.29	22.7	...
29	1.5241	3.32	1.88	0.56	1.51	∞	...

^a When the ratio exceeds ~ 100, the value ∞ is used.

TABLE 4—Emission spectrography of the samples of Group B indistinguishable by energy dispersive X-ray analysis.^a

Ratio	Set 1		Set 2		
	Sample 5	Sample 6	Sample 12	Sample 15	Sample 17
Chromium/manganese	0.18	0.15
Potassium/manganese	2.1	2.3
Aluminum/manganese	2.8	2.75
Magnesium/manganese	1.9	1.8
Titanium/manganese	3.0	0.4	0.25
Barium/manganese	0.23
Potassium/manganese	1.8	0.8	0.4
Arsenic/manganese	0.4

^aNote that these values express the ratio of line intensities and are not proportional to absolute concentrations.

careful mounting of samples and choice of a suitable surface to be analyzed are necessary for obtaining good reproducibility of the analyses. The variations in sample geometry are presumably the reason why the calcium/sodium ratio exhibits the largest standard deviations: the calcium and sodium signals lie relatively far from each other in the spectrum.

The analysis of glass samples by emission spectrography shows lower reproducibility in measured elemental ratios (Table 2). On the other hand, this method is more sensitive than energy dispersive X-ray analysis and can detect trace elements at concentrations in the parts per million range. The reproducibility for iron is poor, probably because of contamination [14], and this element was therefore not included in our experimental results.

Variations over Single Glass Panes

No variation in chemical composition could be detected over two panes of sheet glass. By contrast, a variation in refractive index over the panes was observed. The analysis of variance showed a statistically significant difference in the refractive index of samples taken from different points in the panes. This finding is in agreement with previous reports [2]. No evidence of a gradual change of refractive index over the panes was obtained.

Discrimination Between the Samples of Window Glass

The glass samples listed in Tables 3 through 6 all originated from different windows. Because of the variation in the physical parameters within the same sample, many of the specimens could not be distinguished on the basis of their physical properties alone. On the other hand, the elemental ratios obtained by energy dispersive X-ray analysis showed large variations between the samples. The ratios were mean values of three separate measurements. Nearly all of the samples could be distinguished by this method. In this discrimination, a greater uncertainty in the high values of elemental ratios was considered when the concentration of one of the elements was near the detection limit of the method.

Figure 3 illustrates clear differences in chemical composition of two of the samples with identical refractive index. A short analysis of such samples would be quite sufficient to notice the differences.

Energy dispersive X-ray analysis did not distinguish between the glass Specimens 5 and 6; 12, 15, and 17; and 32 and 34. Glass Samples 12, 15, and 17 had, however, quite different refractive indexes. Finally, emission spectrography showed that Samples 12, 15, and 17 were different from each other, as were Samples 32 and 34. Only Samples 5 and 6 were indistinguishable by all the methods employed in this study.

TABLE 5—*Refractive index, density, and composition by energy dispersive X-ray analysis of glass samples in Group C.*

Sample	Refractive Index	Density, g/cm ³	Sodium/Magnesium	Sodium/Aluminum	Magnesium/Aluminum	Calcium/Sodium	Calcium/Potassium
30	1.5251	2.5199	2.37	3.40	1.43	1.36	42
31	1.5252	2.5185	2.57	3.88	1.51	1.41	60
32	1.5253	2.5190	2.37	3.66	1.54	1.47	∞
33	1.5253	2.5219	2.55	4.00	1.56	0.99	80
34	1.5253	2.5218	2.32	3.64	1.57	1.55	∞
35	1.5255	2.5228	6.0	3.41	0.56	1.36	∞
36	1.5257	2.5239	7.5	3.36	0.44	1.61	∞
37	1.5257	2.5242	7.7	3.01	0.39	1.60	60
38	1.5261	2.5255	7.6	3.13	0.41	1.96	∞
39	1.5262	2.5251	2.71	3.20	1.18	1.75	∞

TABLE 6—Emission spectrography of the samples of Group C indistinguishable by energy dispersive X-ray analysis.^a

Ratio	Sample 32	Sample 34
Titanium/manganese	0.8	0.7
Barium/manganese	0.2	...
Potassium/manganese	0.6	0.95
Chromium/manganese	...	0.35

^aNote that these values express the ratio of line intensities and are not proportional to absolute concentrations.

TABLE 7—Elemental analysis of three glass samples from an actual case. The samples were indistinguishable by density measurements.

Analysis	Sample A	Sample B	Sample C
Refractive index	1.5174	1.5174	1.5175
Sodium/magnesium	2.11	1.89	1.91
Sodium/aluminum	2.38	2.45	3.82
Magnesium/aluminum	1.13	1.30	2.00
Calcium/sodium	1.12	1.12	1.23
Calcium/potassium	15.6	51	∞

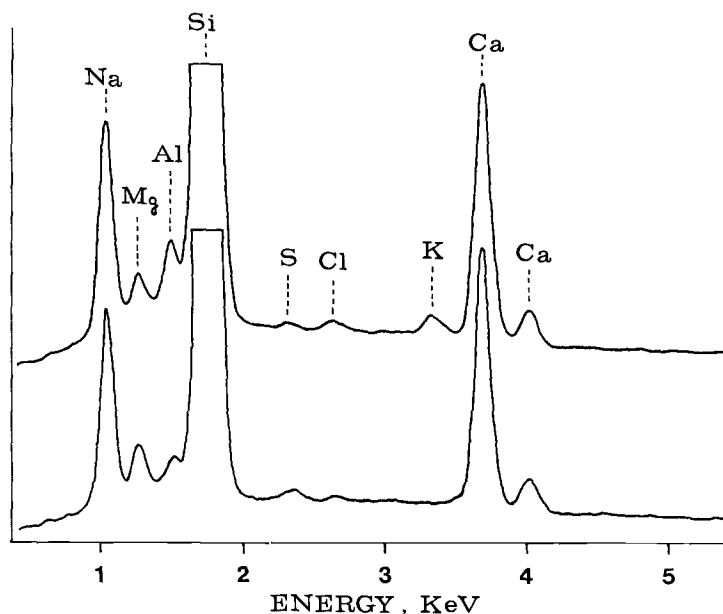


FIG. 3—X-ray spectrum resulting from a 400-s analysis of glass Samples 23 and 24 of Group B. The refractive index of both of these samples was 1.5198. The height of the silicon peaks exceeds the intensity scale in the figure.

Table 7 shows the results of an actual case. Eight glass samples were sent to our laboratory for investigation. Three of the samples were indistinguishable with respect to refractive index and density. By using energy dispersive X-ray analysis, all these samples were found to be clearly different from each other.

A recent report on quantitative analysis of glass by emission spectrography [14] revealed a correlation between refractive index and calcium concentration. The number of glass specimens investigated in this study was too low to draw any definitive conclusions. However, it seems that the calcium/sodium ratio of the samples increased with increasing refractive index. This ratio also only rarely discriminated between glasses with similar refractive index. The frequency distribution of the other elemental ratios for window glass is depicted in Fig. 4.

Conclusions

The comparison of glass fragments may be performed by four methods in our laboratory. The measurement of refractive index and density of samples is a routine operation. In this study, two semiquantitative methods for chemical analysis of glass have been added. By combining these methods, 38 of 40 window glass specimens could be distinguished from each other. All the methods, with the exception of emission spectrography, are non-destructive and can be used for a single glass fragment. The whole procedure for the examination of actual cases in our laboratory is as follows.

The physical properties of samples are examined first. Density, refractive index and, when possible, the thickness of glass fragments are measured. A rapid discrimination between samples with sufficiently different refractive index may be done on the Mettler hot stage equipped with a monochromator. At constant temperature, the Becke line will disappear at different wavelengths for such samples.

When the physical methods do not discriminate between the glass samples, they are analyzed by energy dispersive X-ray analysis. This method is nondestructive, applicable to extremely small fragments (submilligram samples), and rapid in comparison with, for example, emission spectrography. A complete analysis (that is, sample preparation, analysis, and calculation of elemental ratios) of two glass samples requires approximately 1 h. Two or more analyses performed on each sample increase the analysis time a little, but the precision is improved. A short analysis time and small sample size (such as samples obtained by vacuuming) are the greatest advantages of this method.

Emission spectrography is a more laborious method. It is used if energy dispersive X-ray analysis finds samples indistinguishable. Because of its longer analysis time, this method is employed as a last resort for discriminating between the samples. The analysis is destructive and requires at least 1 mg of glass for each measurement.

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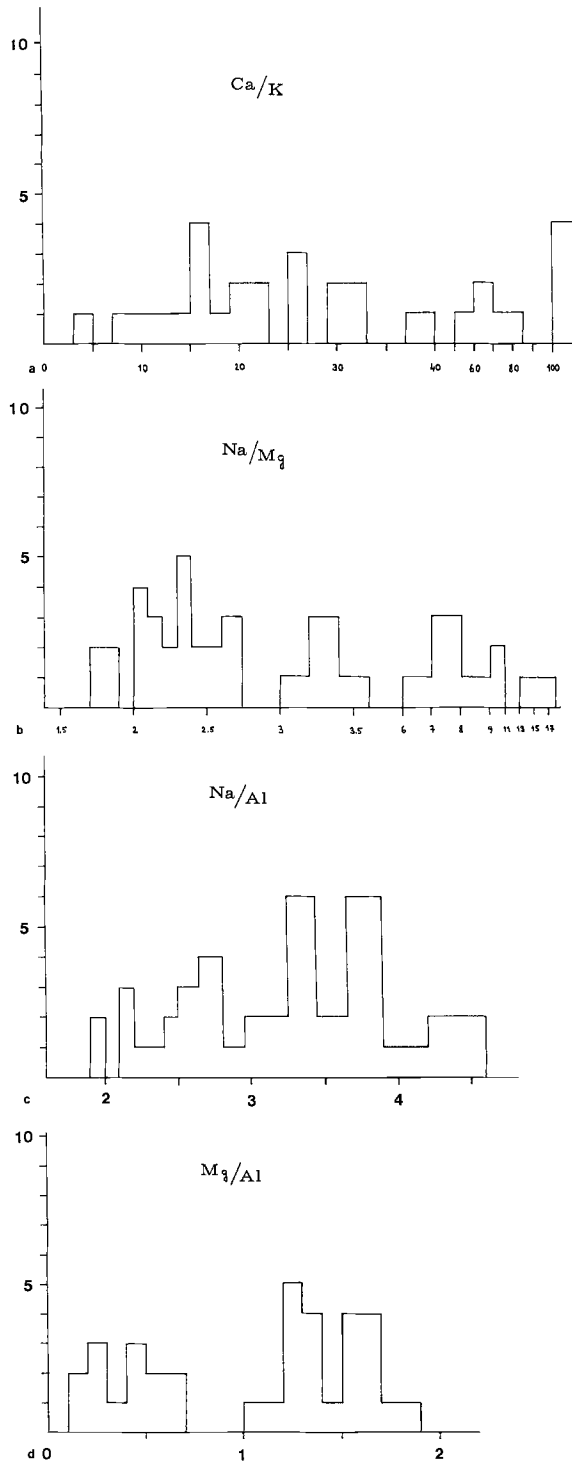


FIG. 4—The frequency distribution of the elemental ratios calcium/potassium (a), sodium/magnesium (b), sodium/aluminum (c), and magnesium/aluminum (d) for 39 specimens of window glass from Groups B and C.

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Address requests for reprints or additional information to
Andreas Maehly, Ph.D., Director
Statens Kriminaltekniska Laboratorium
Fack, S-581 01
Linköping, Sweden