A CLASSIFICATION OF GLASS MICROTRACES

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ABSTRACT: A scheme of glass classification is proposed. The scheme is based on results of the quantitative analysis of 153 glass objects, which belonged to the following categories: car windows (cw), car headlamps (h), external glass of car bulbs (ecb), internal glass of car bulbs (icb), external glass of ordinary light bulbs (eob), internal glass of ordinary light bulbs (iob) and window sheets (w). With the use of SEM-EDX method concentrations of aluminium, barium, calcium, iron, lead, magnesium, potassium, sodium and zinc were determined. The concentration ranges for each element in each category of glass were found and the elements for which the concentration ranges did not overlap were selected. With this non-statistical approach to the problem of glass microtraces classification a collection of 153 glass samples was divided into six sets. Then the cluster analysis was applied for classification within these separated sets. In the latter approach raw analytical results were normalised and Tukey HSD test was used to choose significant variables. The presented approach leads to the correct classification of most of the glass samples studied.

KEY WORDS: SEM-EDX; Glass microtraces classification; Cluster analysis.

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INTRODUCTION

The importance of glass as evidence was recognised many years ago [3]. In the case of lack of a comparative material, the evidence material is studied in order to classify it into an use-category of glass objects. When both, evidence and comparative materials are available the task of a forensic chemist is to answer, whether they could have come from the same object.

Glass as evidence material often occurs in very small quantities. Thus, investigations of glass samples require sensitive analytical methods providing satisfactory results from small amounts of the examined material. One of such methods is the quantitative elemental analysis using a scanning electron microscope with an energy dispersive X-ray spectrometer (SEM-EDX) [4].

It is a method which allows to determine elements present at concentrations of the order of at least 0.1% by weight. Although SEM-EDX does not provide information on trace elements, is useful for examination of very small objects. In Poland the systematic research of glass microtraces using the SEM-EDX method for forensic purposes begun in the Institute of Forensic Research, Cracow in 1995 [1, 2].

MATERIALS AND METHODOLOGY

So far examined 153 glass samples which were collected in Poland were examined. The numbers of samples representing certain use-category are presented in Table I.

TABLE I. THE ANALYSED GLASS SAMPLES

Glass group	Notation	Number of samples
Car windows	cx	69
Windows	w	38
Headlamps	h	19
External part of car bulbs	ecb	6
Internal part of car bulbs	icb	7
External part of ordinary light bulbs	eob	8
Internal part of ordinary light bulbs	iob	6

Examinations were carried out using a scanning electron microscope JSM-5800, Jeol with an energy dispersive X-ray spectrometer Link ISIS 300, Oxford Instruments Ltd., United Kingdom.

Glass samples selected for the examination were washed first in acetone (p.a.) or hexane (p.a.) and later in distilled water, and crushed. For each glass sample at least three fragments with possibly smooth and flat surfaces, and about 1 mm in length, were chosen with the help of an optical microscope, and placed on SEM stubs with self – adhesive carbon tabs. They were covered with carbon using a sputtering unit – SCD 050, BAL-TECH, Switzerland, and mounted in the sample chamber of the scanning electron microscope. The quantitative elemental analysis was carried out in the SEMQuant option.

The following measurement conditions were applied: accelerating voltage 20 kV, life time 50 s, magnification 1000–1500 times, the analysed area – about 0.01–0.005 mm^2.

All elements were determined except for Li and B. However, the concentrations of aluminium, barium, calcium, iron, lead, magnesium, potassium, sodium and zinc were considered. They were determined with a precision of 0.1 weight percents.

DISCUSION

Inspection into the obtained results allowed for finding elements, whose ranges of concentration in the considered groups did not overlap. The effect of such a non-statistical approach to the problem of classification of glass microtraces, which were collected in Poland, is presented in Figure 1. Concentrations of Zn, Pb, Ba and Fe were considered as critical values in this scheme.

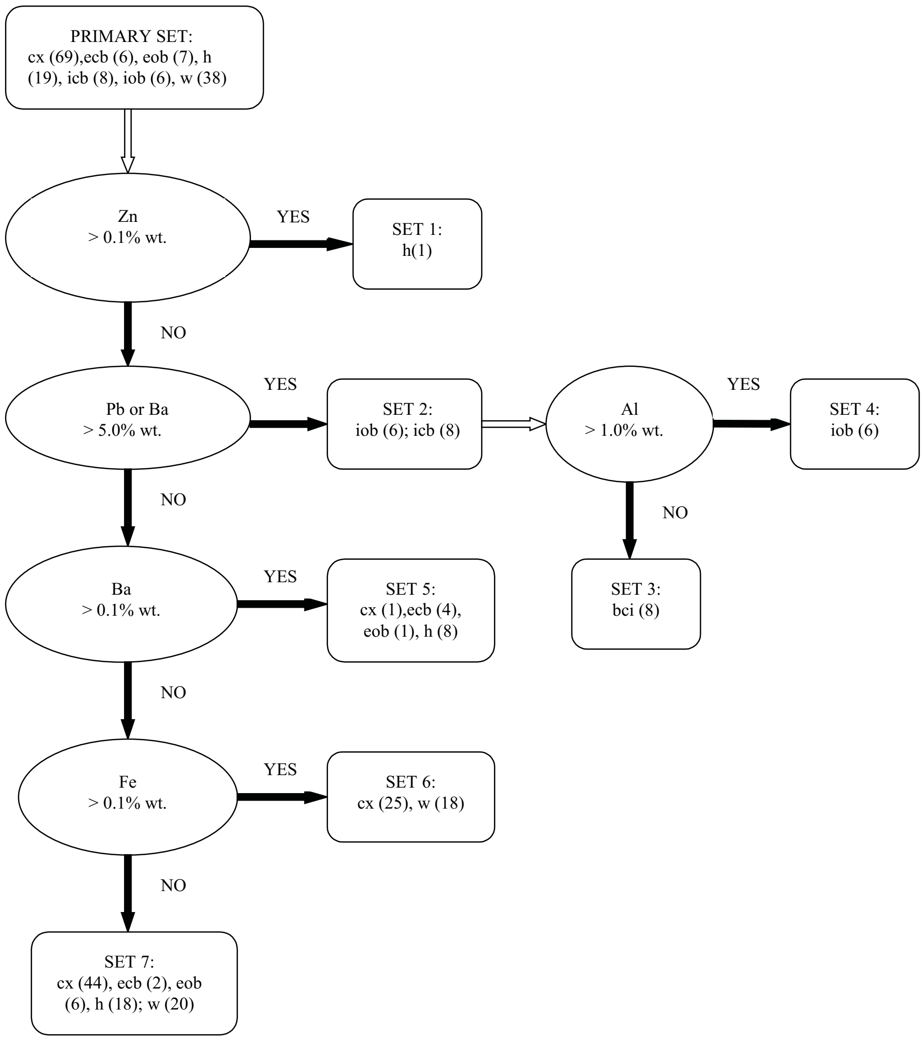

Fig. 1. A glass classification scheme – a non-statistical approach.

Most of the considered glass categories were not homogenous populations, e. g., the headlamps glass samples were classified into two sets (set 5 and set 7 – Figure 1), only groups of internal part of car bulbs (icb – set 3) and internal part of ordinary light bulbs (iob – set 4) were homogenous. The classification of objects in set 5, 6 and 7 was not possible with the non-statistical approach. None of the element concentrations was significantly different for glass groups creating these sets. A method of multidimensional analysis of data, i.e. the cluster analysis was used to solve the problem of classification in the case of sets: 5, 6 and 7.

Raw data were normalised according to the following formula {1}:

$$Z_{X-i} = \frac{c_{X-i} - \min c_X}{\max c_X - \min c_X}, \qquad \{1\}$$

where: Z_{X-i} – a normalised value of i-th measurement of X-th element; $c_{X\text{-}i}$ – a concentration of X-th element obtained at i-th mesurement; max c_X, min c_X – a minimum value and a maximum value of concentration of X-th element.

In order to choose the suitable variables the Tukey HSD method was used. The distance between points in multidimensional space were calculated as Czebyszew distance {2}:

$$d_{A-B} = \max\left|Z_{A_i} - Z_{B_i}\right|, \qquad \{2\}$$

and the Ward's method was chosen as the clustering method {3}.

$$d(s_a, s_k) = \{(n_i + n_k)\, d(s_i, s_k) + (n_j + n_k)\, d(s_j, s_k) - n_k\, d(s_i, s_k)\} \,/\, (n_i + n_j + n_k) \qquad \{3\}$$

where: $d(s_a, s_k)$ – a distance between a new cluster (s_a) and compared cluster (s_k); s_a – a new cluster obtained from an agglomeration of clusters s_i and s_j; s_k – a cluster, from which the distance to cluster s_a is calculated; n_i – number of objects belonging to cluster s_i; n_j – number of objects belonging to cluster s_j.

Figures 2–4 present results of the cluster analysis within the following sets:

– set 5 (Figure 2) – a separation of external part of car bulbs (ecb) and external part of ordinary bulbs (eob) from headlamps (h);
– set 6 (Figure 3) – a separation of car windows (cx) from ordinary window glass (w);
– set 7 (Figure 4) – a separation of external part of car bulbs (ecb), external part of ordinary bulbs (eob) and headlamps (h) from car windows (cx) and ordinary windows glass (w).

Elements which normalised concentrations were used as variables, are placed in the obtained schemes. The effect of classification within set 5 and 7 was satisfactory as only two samples from *cx* category (within set 5.2 and 7.1) were classified incorrectly. The separation of car windows (cx) and ordi-

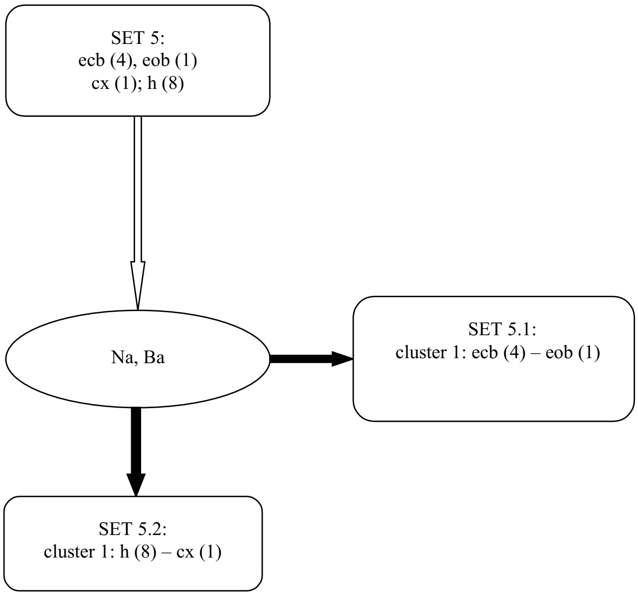

Fig. 2. The result of a separation of external part of car bulbs (ecb) and external part of ordinary bulbs (eob) from headlamps (h).

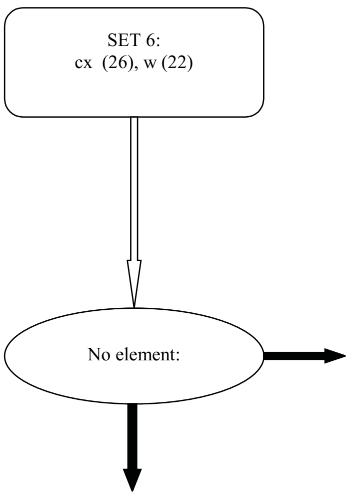

Fig. 3. The result of a separation of car windows (cx) and ordinary window glass (w).

nary window glass (w) was not possible (set 6 and set 7.2). No element could be used as a variable in the cluster analysis. Samples originated from categories ecb and eob created not only common clusters (set 5.1, set 7.1 – cluster 5), also separate cluster (set 7.1 – cluster 4). Due to a small number of the ex-

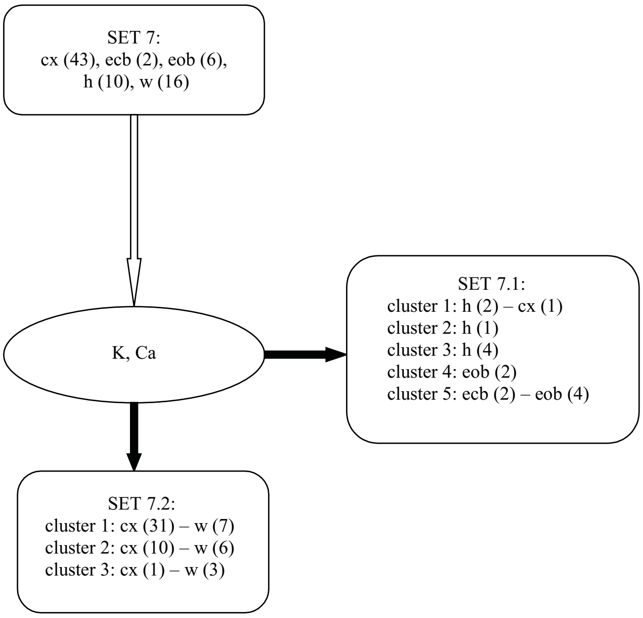

Fig. 4. The result of a separation of external part of car bulbs (ecb), external part of ordinary bulbs (eob) and headlamps (h) from car window (cx) and ordinary window glass (w).

amined objects of there groups it is impossible to establish whether their separation can be performed taking into account results of the elemental analysis with SEM-EDX method.

CONCLUSIONS

Most of the glass microtraces examined can be classified by means of the elemental analysis with SEM-EDX method. Glass microtraces originating from car windows (cx) and ordinary windows (w) should be treated as one group for they reveal the same qualitative and very closed quantitative elemental composition.

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