Journal of Criminal Law and Criminology

Volume 38 | Issue 2 Article 14

1947

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APPLICATIONS OF MICROCHEMICAL TECHNIQUES

Differentiation of Similar Glass Fragments by Physical Properties*

G. W. Roche and Paul L. Kirk

Paul L. Kirk, Professor of Biochemistry, Medical School, University of California, Berkeley, is well known to our readers for his articles on practical laboratory methods and techniques. Much of the research for these papers has been inspired by problems encountered in connection with criminal investigations for which Prof. Kirk has served as a consultant. In an earlier article, "Glass Fragments as Evidence" (*33*:416-21 (1943)), he and his associates developed a technique for rapid differentiation between glass fragments of the kind found at crime scenes. His present contribution discusses the result of additional investigation into this problem.

G. W. Roche, a graduate in Technical Criminology (1942) from the University of California, is at present a crime laboratory analyst with the Minnesota State Bureau of Criminal Apprehension. From 1942-45 he served with the U. S. Army and was separated from the service in the rank of Captain. Before assuming his present position Mr. Roche carried on graduate studies at the University of California during which time he worked with Prof. Kirk on the problem of differentiating glass samples.—EDITOR.

It has been shown previously that the physical properties of glass are capable of detecting minute differences in composition. The most extensive study of such differentiations was made by Gamble, Burd, and Kirk (1) who found it possible to distinguish each of 100 samples of glass from all of the others on the basis of refractive index and specific gravity comparisons. The glass studied by these authors was of all types, including only limited numbers of very similar glasses. In connection with a criminal investigation in which brown glass, probably from a beer bottle was found on the sole of a suspect's shoe, the question arose as to whether the manufacture of mass-produced glass items such as brown bottles would actually differ significantly from each other, as has been previously found for miscellaneous glasses.

This study reports the findings when each of 50 samples of brown bottle glass, chiefly from beer bottles were compared as to their specific gravity and refractive index. A rapid technique for carrying out such comparisons is also given, in which specific gravity comparisons are made by means of a gradient tube as described previously for other purposes (2).

EXPERIMENTAL

Absolute measurements of either specific gravity or refrac-

* Aided by a grant from the Research Board of the University of California.

tive index involve careful control of temperature and composition of liquids used for flotation or immersion. With any practical degree of control of these variables there is still a definite uncertainty in the final determination of a value. Thus, such results are accurate for refractive index only to the third decimal unless exceptional precautions are taken. It is possible but not simple to evaluate specific gravity with a slight uncertainty in the fourth decimal, and in practice it is not easy to reproduce values past the third. It is, however, quite easy to compare two or more solid fragments for either of these properties with much better accuracy than this, or rather, to distinguish differences in the values far more sensitively than the absolute value can be obtained.

Specific gravity comparisons were made rapidly and conveniently as follows: A glass tube, about 8 mm. internal diameter and 1 foot long was sealed off in the form of a test tube. To it was added enough methylene iodide to fill it approximately 1/3 full. A little less nitrobenzene was added over the methylene iodide, and the mixture stirred with a long glass rod until partially mixed. On standing a short time, a rather uniform density gradient was set up in the tube. To the tube were then added the glass fragments which were to be compared. Further adjustment of the gradient was then possible on the basis of the difference in level at which the fragments came to rest. After about 15 minutes, each fragment had stopped sinking or rising, and had reached the point at which its specific gravity was exactly equal to the liquid in immediate contact with it. The amount of separation was indicative of the difference in specific gravity between the particles concerned and the value of the gradient. If insufficient spread was obtained further stirring of the mixture was necessary.

Some care was necessary to ascertain that particles were not adhering to each other or to the sides of the tube. Two successive observations of the position of a particle were necessary to make sure that the true equilibrium positions had been reached. Only those particles which floated at exactly the same level could be said to have the same specific gravity.

Refractive index comparisons were somewhat more laborious than specific gravity comparisons, and were only employed for those samples that had the same specific gravity, since only these could in any case be identical. Two particles having the same specific gravity were removed from the gradient tube and placed on a microscope slide. Immersion media having approximately the correct refractive index (range-1.50 to 1.53) was

added, and the particles placed close together in the drop so that both were visible in the field of the microscope under about 110X. The medium was adjusted by the usual Becke line method (3) until its refractive index was very slightly less than that of either glass fragment. By constant observation as the more volatile constituent evaporated, it could be determined with great accuracy whether the Becke line reversed at the same moment with both pieces of glass, in which case the refractive indices were identical. If the movement of the Becke line occurred at a different time with the two fragments, their refractive indices were not identical. Previous studies had shown that when various fragments taken from a single piece of glass were compared, both specific gravity and refractive index were identical unless the fragments came from widely different locations in a large piece of glass in which case, occasional small differences were noted (1).

Results

A single fragment from each of 50 brown glass bottles was dropped into a gradient tube as described. The particles assumed 40 distinct levels, 32 of which were represented by one fragment only, 6 by two fragments and 2 by three fragments. Thus, only 18 of the 50 samples required further checking, which was performed as described for refractive index. Of these 18 only two fragments were indistinguishable on the basis of refractive index, all other being definitely resolved. Thus, out of 50 samples of very similar glass, only two showed identical values of the two physical properties studied within the limits of detection.

As an independent check on the reliability of the gradient tube method, 25 of the samples were compared in all possible combinations of pairs for specific gravity, by adjusting the medium until one sample would rise and the other sink when they represented different values of specific gravity. The order of these checked the order found in the gradient tube.

As an investigative technique the value of the above described technique lies in the fact that if a considerable number of small glass fragments are found, for example in a suspect's clothing, they can be rapidly divided into groups or shown to have possible single origin. A known sample from glass broken in the commission of the crime may then be added, and if it equilibrates at the same level as any group of glass found on the suspect, it need then be compared for other factors such as re-

fractive index only with the glass of that group, thus saving a great deal of needless comparison.

Refractive index is inherently a considerably more delicate factor for distinguishing differences in glass than is specific gravity, but it also is a much more laborious process. By combining it in the manner described with specific gravity determination only a minimum number of refractive index comparisons need be made.

Summary

Fifty samples of similar brown bottle glass, most of which originated from broken beer bottles were compared with respect to specific gravity and refractive index. Of the entire number only two samples were indistinguishable on the basis of these two properties.

A rapid technique is described whereby a large number of samples may be checked against each other. Its application in criminal investigation is indicated.

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