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## A PROVISIONAL METHOD OF ELIMINATING THE EFFECTS OF FILM SHRINKAGE AND IMPROPER CAMERA DIAMETER IN DEBYE PATTERNS

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## INTRODUCTION

When back reflection lines are present in Debye patterns, the Straumanis technique (Straumanis and Ievins, 1940) is generally used to correct for film shrinkage and improper camera diameter. When no back reflection lines are present, corrections may be made by using (a) the Wilson technique (Wilson, 1949), (b) fiducial marks (Bradley and Jay, 1932), which may be produced in a number of different ways, or (c) an internal standard (Davey, 1934).

When working with complex powders which do not give back reflections, but which give many lines close together, the internal standard method of correcting for film shrinkage and improper camera diameter falls short, since it is undesirable to add extra lines to the already complicated pattern. In this case the Wilson technique and the fiducial mark technique have in the past been the only suitable means of eliminating the error in Debye patterns due to film shrinkage and improper camera diameter. The purpose of this paper is to describe another method of correcting for film shrinkage and improper camera diameter which can be used when no back reflection lines are present.

## METHOD OF FILM SHRINKAGE CORRECTION

A picture is obtained from the sample in the usual way, the only modification being the placement of an appropriately thick brass screen over the back reflection region of the film. This gives lines in the front reflection region.

After this exposure, however, the radiation mask is now placed over the front reflection region of the film, the collimators are interchanged, and the camera is mounted on its track so that the  $x$ -rays are now going through the camera in the opposite direction. This exposure places the front reflections in the normal back reflection region. The film is developed, and since there are now lines in both the front and back reflection regions, the usual Straumanis technique can be used to make a film shrinkage correction. Furthermore, since the specimen and the film remain in the same positions throughout both exposures, the validity of this method depends only on the interchangeability of the collimators.

TABLE 1. FILM SHRINKAGE CORRECTION FACTORS

Film	Straumanis	Present Authors
1	1.003	1.004
2	1.004	1.004
3	1.000	0.999

This method was worked out using a Philips powder camera. With this camera, the following details are evident.

1. Since the incoming beam collimator has a peg to position the lead slit horizontally, a hole must be drilled in the outgoing beam collimator collar to assure the proper positioning when the collimators are interchanged.
2. A 180° rotation about either a horizontal or vertical axis may be used.

- a. Rotation about a vertical axis.

Such a rotation places the pulley shaft on the opposite side of the camera track. Consequently, if this type of rotation is to be used, another pulley motor must be installed.

- b. Rotation about a horizontal axis.

The Philips camera can be rotated about a horizontal axis by loosening the screw which holds the camera in its track mounting bracket. In order to make a complete 180° rotation, in this case, both the specimen centering pin and the film camp screw must be removed. In this case the film can be held in place with Scotch tape. It is preferable, however, to mill a slot in the camera base, thus avoiding the removal of the centering pin.

#### TEST OF COLLIMATOR INTERCHANGEABILITY

We tested the interchangeability of our collimators in the following way. Two exposures of finely powdered silicon, a compound which gives good back reflection lines, were made on the same film so that we could obtain film shrinkage correction factors by both the Straumanis method and our method. The first exposure was made with the camera in the normal position (no radiation mask covering the back reflection region). If the film were developed at this point, the normal pattern of silicon, with lines in both the front and back reflection regions, would be seen. However, the second exposure is now made with the camera in the rotated position, the collimators interchanged, and the radiation mask in place. After the second exposure, the film is developed, and it is seen that the front reflection lines are present not only in the front reflection region, but also in the back reflection region, intermingled with the normal back reflection lines. A correction factor using the Straumanis method is obtained in the usual manner, *i.e.*, by measuring the positions of pairs of front and back reflection lines. A correction factor using our technique is obtained by measuring the positions of pairs of front reflection lines pro-

duced with the camera in the normal position and pairs of front reflection lines produced with the camera in the rotated position. Table 1 shows a comparison of the correction factors obtained for three different patterns of silicon using both methods. It can be seen that the agreement in this case is good to one part per thousand.

## REFERENCES

- BRADLEY, A. J. AND A. H. JAY, (1932), A method for deducing accurate values of the lattice spacing from *x*-ray powder photographs taken by the Debye-Scherrer method. *Proc. Phys. Soc. (London)*, **44**, 563.
- DAVEY, W. P. (1934), A Study of Crystal Structure and Its Applications. McGraw-Hill Book Co., Inc., New York, 113-114, 158-163.
- STRAUMANIS, M. AND A. IEVINS, (1940), Die Präzisionsbestimmung von Gitterkonstanten nach der asymmetrischen Methode. Julius Springer, Berlin (reprinted by Edwards Brothers, Ann Arbor, Michigan, 1948), 27-30.
- WILSON, A. J. C. (1949), Straumanis method of film shrinkage correction modified for use without high angle lines. *Rev. Sci. Inst.* **20**, 831.

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## WAIRAKITE IN WEST INDIAN SPILITIC ROCKS

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## INTRODUCTION

An occurrence of wairakite in spilitic rocks from St. Thomas, Virgin Islands, is of special interest for several reasons. In the first place, this is the first record of this rare zeolite from spilitic rocks. Secondly, this is the first instance in which wairakite can be shown to be replacing albite and not a more calcic feldspar; therefore, the lime for the zeolite in this case was not derived from the plagioclase. Thirdly, if the hypothesis of an essentially primary origin for this mineral is accepted, then some rather interesting and important conclusions can be drawn regarding the chemical and physical conditions prevailing during the final stages of consolidation of this spilitite.

The oldest rocks of St. Thomas are the keratophyres and spilites of the Water Island Formation. This formation consists of about eighty per cent keratophyre (including quartz keratophyre) flows and pyroclastics and twenty per cent spilitite flows. The age is pre-Upper Cretaceous and the volcanic rocks appear to have escaped regional metamorphism. These rocks have been described elsewhere (Donnelly, 1959, and in MS). Wairakite was originally found in a poorly exposed spilitite