numerous, octahedral sites. However, with the presence of greater concentration of impurity boron, progressively more of the tetrahedral sites would also come to be filled. It would also follow that the octahedral boron would be more weakly bonded and hence more readily leached than the tetrahedral boron.

CONCLUSIONS

From the data presented here, it is seen that boron contamination from new platinum crucibles can be a major problem. It has also been shown that the amount of time required to render routinely-prepared platinum crucibles usable for boron analysis by successive alkaline fusions is excessive. Thus, the only effective solution to the problem seems to be to insist on delivery of boron-free crucibles. Communications from the principal U.S. manufacturers of platinum crucibles indicate that, while their routinely-prepared platinum crucibles contain small amounts of boron impurity, they are willing to supply "boron free" crucibles from spectroscopically-analyzed platinum for an additional fee. The detection limit for such analysis is about 2 ppm B.

In light of the approximately thousand-fold preference for leaching boron from platinum, even 2 ppm B or less may still present a problem in early fusions. Thus, it is also recommended that all crucibles to be used for alkaline fusion in boron analysis be thoroughly checked for contamination arising from leaching during the fusion.

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Recommendations for reporting thermal analysis data*

BECAUSE thermal analysis involves dynamic techniques, it is essential that all pertinent experimental detail accompany the actual experimental records to allow their critical assessment. This was emphasized by Newkirk and Simons (1963) who offered some suggestions for the information required with curves obtained by thermogravimetry (TG). Publication of data obtained by other dynamic thermal methods, particularly differential thermal analysis (DTA), requires equal but occasionally different detail, and this note is intended to present comprehensive recommendations regarding both DTA and TG.

In 1965 the First International Conference on Thermal Analysis (ICTA) established a Committee on Standardization charged with the task of studying how and where standardization might further the value of these methods. One area of concern was with the uniform reporting of data, in view of the profound lack of essential experimental information occurring in much of the thermal analysis literature. The following recommendations are now put forward by the Committee on Standardization, in the hope that authors, editors, and referees will be guided to give their readers full but concise detail. The actual format for communicating these details, of course, will depend upon a combination of the author's preference, the purpose for which the experiments are reported, and the policy of the particular publishing medium.

To accompany each DTA or TG record, the following information should be reported:

1. Identification of all substances (sample, reference, diluent) by a definitive name, an empirical formula, or equivalent compositional data.

2. A statement of the source of all substances, details of their histories, pre-treatments, and chemical purities, so far as these are known.

3. Measurement of the average rate of linear temperature change over the temperature range involving the phenomena of interest.

4. Identification of the sample atmosphere by pressure, composition, and purity; whether the atmosphere is static, self-generated, or dynamic through or over the sample. Where applicable the ambient atmospheric pressure and humidity should be specified. If the pressure is other than atmospheric, full details of the method of control should be given.

5. A statement of the dimensions, geometry, and materials of the sample holder; the method of loading the sample where applicable.

6. Identification of the abscissa scale in terms of time or of temperature at a specified location. Time or temperature should be plotted to increase from left to right.

7. A statement of the methods used to identify intermediates or final products.

8. Faithful reproduction of all original records.

9. Wherever possible, each thermal effect should be identified and supplementary supporting evidence stated.

In the reporting of TG data, the following additional details are also necessary:

10. Identification of the thermobalance, including the location of the temperature-measuring thermocouple.

11. A statement of the sample weight and weight scale for the ordinate. Weight loss should be plotted as a downward trend and deviations from this practice should be clearly marked. Additional scales (e.g., fractional decomposition, molecular composition) may be used for the ordinate where desired.

12. If derivative thermogravimetry is employed, the

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method of obtaining the derivative should be indicated and the units of the ordinate specified.

When reporting DTA traces, these specific details should also be presented:

10. Sample weight and dilution of the sample.

11. Identification of the apparatus, including the geometry and materials of the thermocouples and the locations of the differential and temperature-measuring thermocouples.

12. The ordinate scale should indicate deflection per degree Centrigrade at a specified temperature. Preferred plotting will indicate upward deflection as a positive temperature differential, and downward deflection as a negative temperature differential, with respect to the reference. Deviations from this practice should be clearly marked.

Members of the Committee on Standardization of ICTA are: Professor C. Mazieres (France), Professor T. Sudo (Japan), Mr. R. S. Forsyth (Sweden), Mr. H. G. Wiedemann (Switzerland), Dr. I. S. Rassonskaya (U.S.S.R.), Mr. C. J. Keattch (United Kingdom), and Dr. P. D. Garn (United States). Other delegates to the Committee include Professor L. G. Berg (U.S.S.R.), Dr. R. C. Mackenzie (United Kingdom), Dr. J. P. Redfern (United Kingdom) and Dr. S. Gordon (United States). The Chairman is Dr. H. G. McAdie from Canada.

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Newkirk, A. E., and Simons, E. L. (1963): *Talanta* **10**, 1199.

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Laboratory processing of halloysite*

(*Received* 24 *November* 1968)

INTRODUCTION

CLAY mineral analysis commonly involves X-ray diffractometry, yet no standards exist for preparation of the clay samples. Previous work clearly has shown that mounting techniques and preparation methods can affect X-ray results (Fenner, 1967; Figueiredo, 1965; Gibbs, 1965; Keller *et al.*, 1966; Parrish, 1960; Schultz, 1955; and Takahashi, 1957). Equipment and techniques used should be reported on to prevent results of sample preparation from being attributed to the minerals investigated.

We found that preparation differences change the X-ray diffraction peak intensities for samples, usually increasing the intensity up to a point in the comminution, and then decreasing the intensity. This change in intensity was caused primarily by decreasing particle size and sorting changes. Our methods did not bring about the amorphous state that Figueiredo (1965) achieved by other means. Mounting procedures also affect the results of X-ray diffractometry by influencing particle orientation within the clay sample. More electron microscope work is necessary; as more data are accumulated, possible standardization of analytical procedures for working with clay minerals (Fenner, 1966) can be proposed. Such standardization should ultimately serve as a means of communication rather than as a restriction among researches.†

Samples

API Project 49 reference samples no. 12 (Bedford, Indiana) and 13 (Eureka, Utah) were used in this study; their origin is discussed elsewhere (Wheeler and Burkhardt, 1950, p. 87).

Kerr *et al.* (1950) recognized two halloysite varieties. Both of our samples were placed in the 7 Å, $2H_2O$ group (their Table 7, p. 12). The $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$ and $Al_2O_3 \cdot 2SiO_2 \cdot 4H_2O$ structures and their tubular morphologies were described or explained in several conflicting reports (*e.g.*, Alexander *et al.*, 1943; Bates *et al.*, 1950; Miller and Keller, 1963; Pundsack, 1958), but the question of structure vs. morphology—hence more detailed classification—has not yet been resolved to the satisfaction of all authors (*e.g.*, Brindley and Santos, 1966; Chukhrov and Zvyagin, 1966). According to the classification scheme preferred by Brindley and Santos (their p. 8), our samples are halloysites—at least in part; the many platy grains also found would have to be considered kaolinites under their classification scheme.

*Modification of presentation entitled Halloysite Response To Preparation For X-Ray Diffractometry, given at the 16th Annual North American Conference on Clay Minerals.

†The Organisation de Coopération et de Développement Économiques (OECD) has recently embarked upon a project to create an information bank on availability of reference materials, and to make a study of the characterization of fourteen specimens of non-metallic minerals (including clays) utilizing ten categories of standardized measuring methods (unpublished documents for OECD by Fripiat and Jelli, 1967, and OECD Secretariat, 1968).

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