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DIFFERENTIAL THERMAL ANALYSIS OF KIDNEY AND BLADDER STONES*

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ABSTRACT

Differential Thermal Analysis (DTA) has been used to investigate the thermal decomposition of several different stones collected from various patients. The stones were washed with distilled water, methanol, and acetone then air dried and ground to −100, +200 mesh. Heating rates of 5, 10, and 25 °C were used, along with both static air and flowing argon atmospheres. An evaluation of the effect of heating rate and furnace atmosphere, along with possible stone composition and structure, is discussed.

INTRODUCTION

This study was undertaken in order to better understand the exact composition and structure of several different types of stones collected from patients of McLennan County. Along with this better understanding of the composition and morphology it was hoped that a rapid and inexpensive method for the screening of stones from patients could be developed that might lead to a course of treatment that would eliminate the need for further surgical removal of like stones. These stones were collected over a period of several months with no reference kept as to the patient, the doctor, the exact location, or date of removal of the said stone.

EXPERIMENTAL PROCEDURE

The stones once collected were washed in distilled water, then in methanol and finally in acetone. The washed stones were then allowed to air dry and then they were ground with a morter and pestel to approximately 100–200 mesh. The samples were then transferred to stoppered sample bottles for storage until analyzed. The DTA analysis was performed on a commercial instrument, the Fisher Differential Thermalyzer using a Brown 1MV Strip Chart Recorder. The X-ray diffraction analysis was

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performed on a Norelco X-Ray Unit with a Debye-Scherrer Camera using CuK radiation and 40 kV, 35 mamp, Nickel filter, NS-392 film, 3-h exposure and a developing time of 5 min.

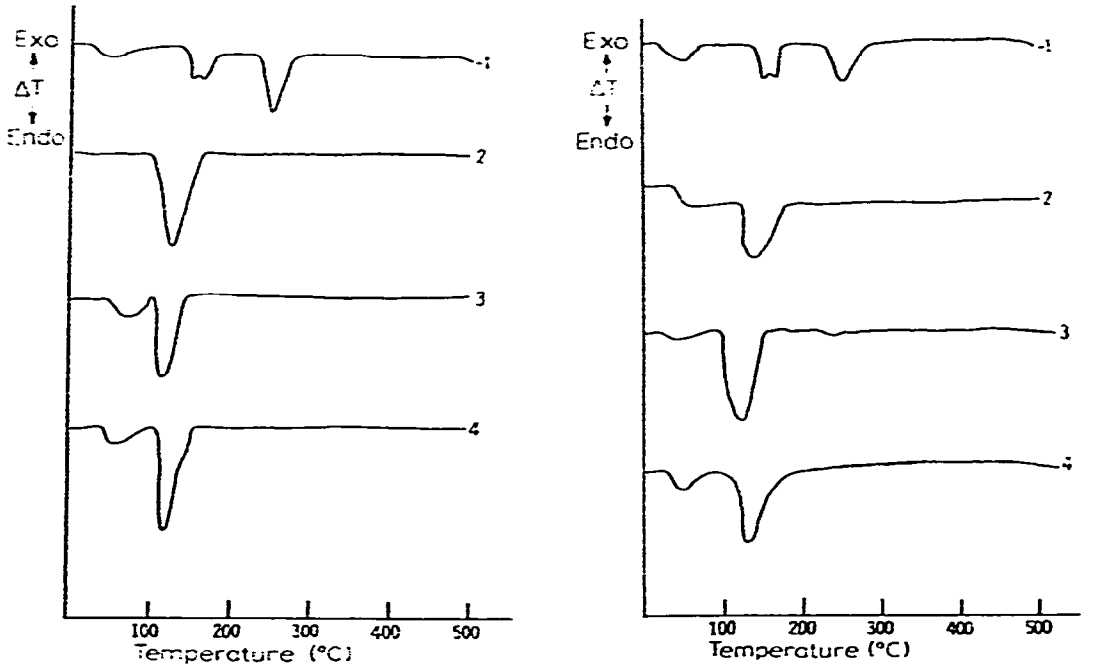

Fig. 1. DTA curves recorded at a heating rate of 5 °C min^{-1} in argon flow.

Fig. 2. DTA curves recorded at a heating rate of 5 °C min^{-1} in static air.

RESULTS

In the DTA scans made at 5 °C min^{-1}, and with a flowing argon atmosphere, stone 1 has its first endotherm appearing at 25 °C and a doublet at 142 °C with the third endotherm beginning at 225 °C and the starting of an endotherm at 480 °C; while stone 2 has an endotherm at 44 °C and another at 102 °C. Stone 3's endotherm begins at 25 °C and 108 °C. Finally, stone 4 has endotherms at 25 °C and 112 °C. These are shown in Fig. 1.

The DTA scans shown in Fig. 2 made at 5 °C min^{-1} under static air environment show much the same general pattern of endotherms as were found with flowing argon. The endotherms of stone 1 begin at 25 °C, a doublet beginning at 147 °C and two endotherms beginning at 231 °C and 475 °C, respectively. Stone 2 has endotherms beginning at 33 °C and 113 °C, while stone 3 has endotherms beginning at 35 °C and 118 °C. Finally, stone 4 has an endotherm at 40 °C and one at 100 °C.

In order to determine if a more rapid DTA analysis could be performed on stones such as these, the same specimen were run at heating rates of 10 °C min^{-1} and 25 °C min^{-1}. If a DTA method can be used for a diagnostic tool, the method must be

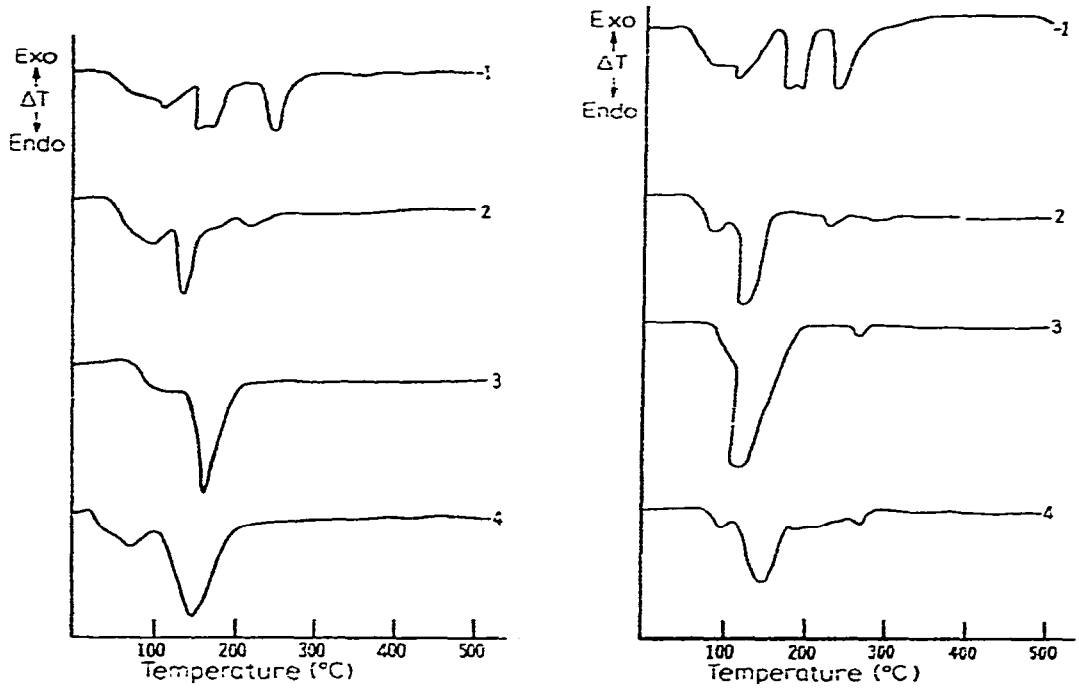

Fig. 3. DTA curves recorded at a heating rate of 10 °C min^{-1} in argon flow.

Fig. 4. DTA curves recorded at a heating rate of 10 °C min^{-1} in static air.

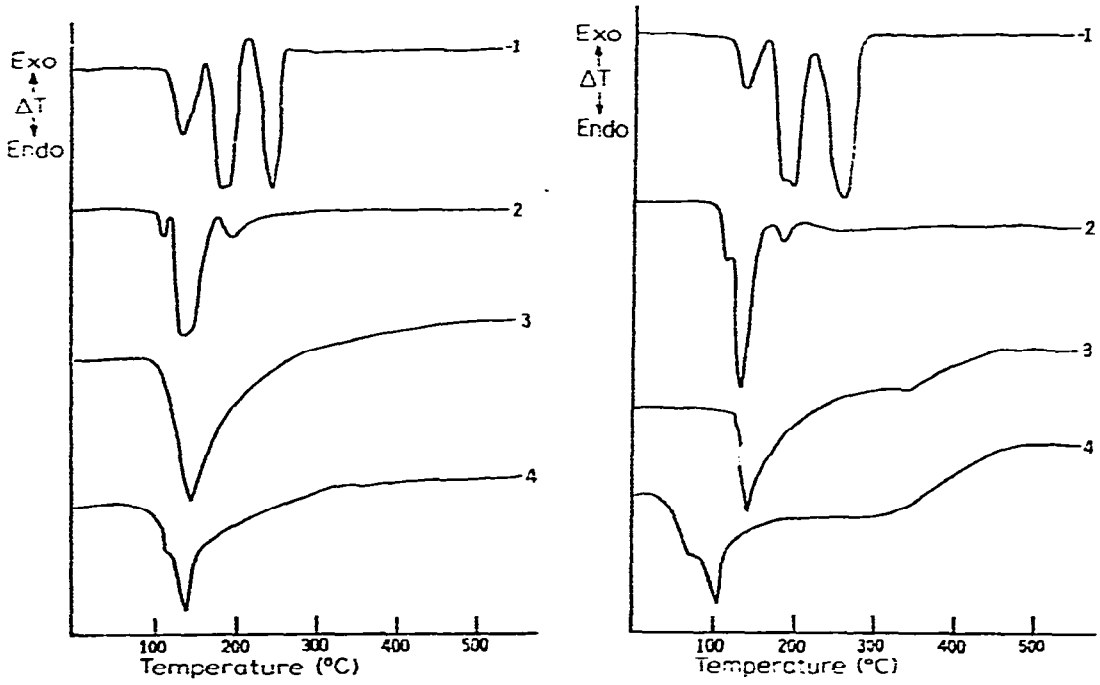

Fig. 5. DTA curves recorded at a heating rate of 25 °C min^{-1} in argon flow.

Fig. 6. DTA curves recorded at a heating rate of 25 °C min^{-1} in static air.

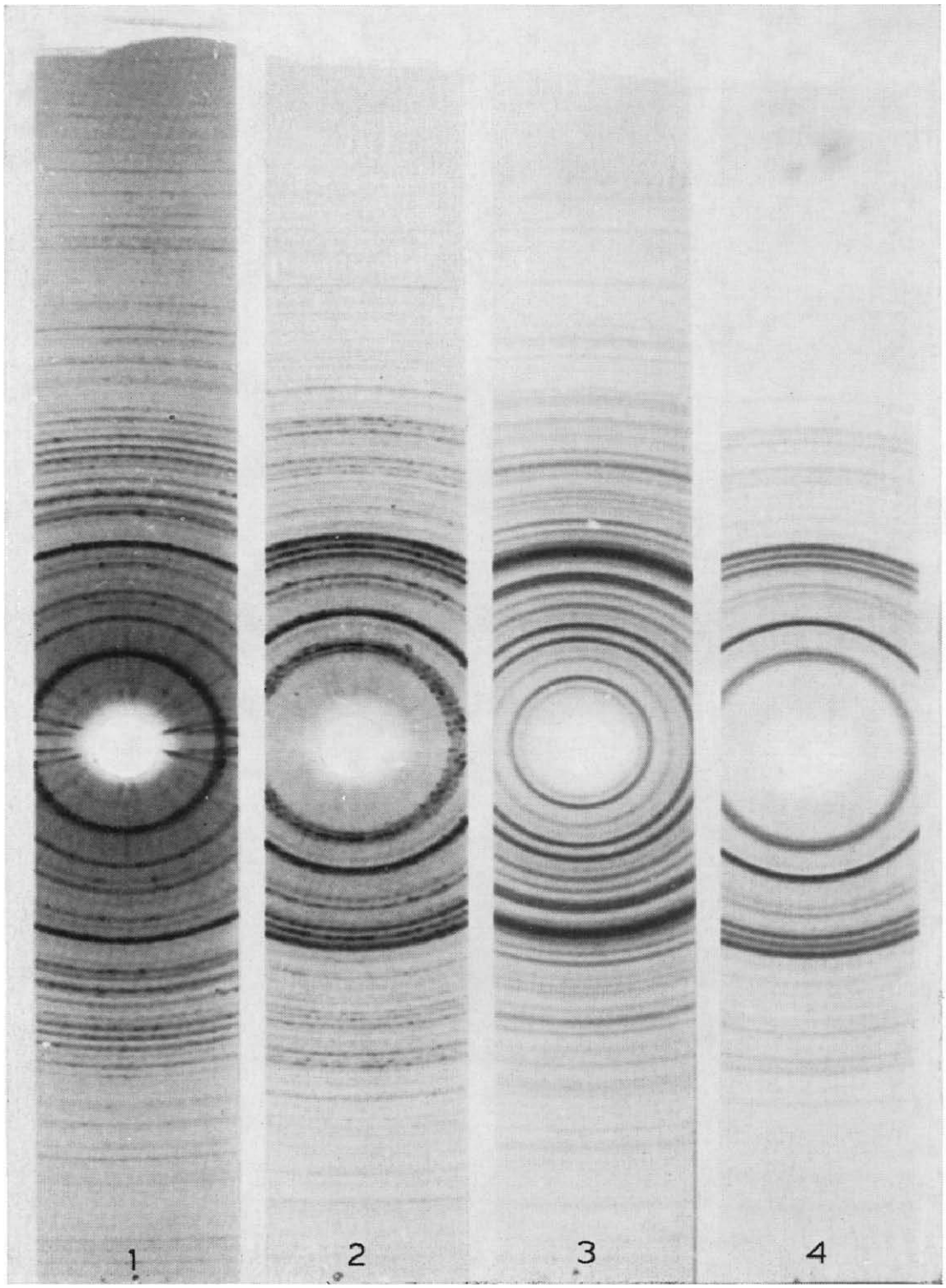

Fig. 7. X-ray diffraction patterns of stones studied by DTA.

as rapid as possible and yet give the necessary precision. Fig. 3 shows the DTA patterns for runs made at 10 °C min^{-1} with a flowing-argon atmosphere. Under these conditions stone 1 gives an endotherm at 35 °C with another at 95 °C, and a doublet at 157 °C, and endotherms at 225 °C and 470 °C. With stone 2 the peaks appear at 45 °C, 108 °C and, possibly, one at 190 °C. Stone 3 has endothermic peaks at 67 °C and 115 °C, and stone 4 has the endotherms at 35 °C and 113 °C.

The DTA scans made at 10 °C min^{-1} and in a static-air environment are given in Fig. 4. Endothermic peaks at 45 °C, 100 °C, a doublet at 150 °C, and other endotherms at 225 °C and one beginning at 472 °C are shown for stone 1. Stone 2 has endotherms at 45 °C, 115 °C, and 200 °C, while stone 3 has endothermic peaks at 45 °C, 110 °C, and 225 °C. Under these conditions stone 4 has endotherms at 45 °C, 112 °C, and 245 °C.

For the conditions of 25 °C min^{-1} and flowing argon (Fig. 5), stone 1 has endothermic peaks at 40 °C, a doublet at 170 °C and endotherms at 225 °C and 470 °C. Stone 2 has endotherms at 40 °C, 125 °C, and 190 °C while stone 3 has endothermic peaks at 40 °C and 100 °C. Stone 4 has peaks at 40 °C and 125 °C.

Fig. 6 shows the set of runs made at 25 °C min^{-1} and with static air. Under these conditions, stone 1 has peaks at 125 °C, a doublet at 178 °C and endotherms at 225 °C and 470 °C. Stone 2 has endotherms appearing at 35 °C, 115 °C, 185 °C and 400 °C, while stone 3 endotherms appear at 140 °C and, possibly, one beginning at 300 °C. Finally, stone 4 has an endothermic peak at 40 °C and one at 120 °C.

A comparison of the various DTA patterns shows that a fast heating rate can be used without reducing the accuracy of the analysis. In general, it can be concluded that stone 1 is considerably different from the others, and that stones 2, 3, and 4 must have only minor differences.

Fig. 7 shows the X-ray diffraction patterns of all stones studied by DTA. The diffraction pattern of stone 1 was found to agree with the ASTM card file of calcium oxalate, form D. The other three stones are believed to be mixed calcium–magnesium carbonates, but exact correlation with ASTM cards could not be made. Complete chemical analysis will be made in the future for possible correlation with the DTA and X-ray patterns.

ACKNOWLEDGEMENT

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REFERENCES

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