Studies on the Analysis of Vitamins D

6. A NOTE ON THE PREPARATION OF FLORIDIN EARTH FOR VITAMIN D CHROMATOGRAPHY

By J. GREEN

Walton Oaks Experimental Station, Vitamins Ltd., Tadworth, Surrey

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Further work on the use of floridin earth for the chromatographic analysis of materials containing vitamin D, as described in earlier papers (Green, 1951*a-c*), has shown that the preparation of the earth before use is of a more critical nature than had originally been thought. The adsorbent used in these earlier papers was 'prepared for Emmerie's test' supplied by British Drug Houses Ltd. The preparation process consists essentially of an activation with boiling hydrochloric acid under controlled conditions; activation appears to take place first by the removal of carbonates and other soluble materials and, secondly, by the breakdown of the surface structure of the earth. Trials on recently received samples of earth show that this preliminary preparation can 'over-activate' the earth by carrying the breakdown process too far. If this extensive structural breakdown (the process is an irreversible one) should occur, the earth is then unsuitable for chromatography, destroying 20–40 % of the vitamin D during the adsorption procedure. The earth is, of course, still quite suitable for vitamin E chromatography.

Whilst it is hoped that suitably prepared samples of floridin may be available in the future, a satisfactory laboratory method of preparation has been found to be as follows: 100 g. of the crude untreated earth (obtainable from British Drug Houses Ltd.) is made into a slurry with 260 g. of concentrated hydrochloric acid and 40 g. of water. The mixture is boiled for 30 min. under reflux. After cooling, the floridin is washed with water, first by decantation and then on a filter at the pump, until free from acid. The floridin is then dried at 37–40° in thin layers, turning the layers frequently. When dry, the earth is passed through a 160-mesh sieve; the retained portion is suitable for use and remains so indefinitely.

Although the length of time for the activation as stated above appears to be satisfactory, it is possible that this may have to be varied slightly from batch to batch of untreated earth. It is therefore recommended that the prepared sample be tested for quantitative adsorption and elution.

REFERENCES

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- Green, J. (1951*b*). *Biochem. J.* **49**, 232.
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