use of these reagents for clarification purposes, pigment removal, *etc.*, sometimes results in manipulative techniques that might better be replaced by simpler techniques that accomplish the same results. After washing once with water, the organic solution then was washed with 10 ml. of 20% of sodium phosphate monobasic and then once again with water. Sodium phosphate monobasic is an excellent way to remove the excess alkali in that it is much superior to acetic acid and permits the ready removal by washing of any excess of the acidifying agent. The removal of any excess acetic acid is most troublesome; for at this stage, if the ether methylene chloride solution is allowed to stand overnight, the separation of small amounts of glycosides takes place. However, the solvent was directly removed by distillation until a concentrate of about 20 ml. was obtained. The concentrate was impregnated on filter cell 4 Gm. and dried. The mixture was placed in a sintered-glass funnel and eluted slowly with 150 ml. of a mixture of 60 parts skellysolve B and 40 parts benzene. This solvent mixture removed some nonphenolic pigments and other resin-like substances that have not been characterized. The mixture then was eluted with 150–200 ml. of anhydrous ether. Upon concentration of the ether eluate to a small volume, acetyldigitoxin crystallized out directly in the distillation flask. The glycoside was collected by means of a sintered-glass filter. The filtrate was diluted with an equal volume of skellysolve B (hexane) and additional amounts of glycoside separated out that was collected by filtration. When the paper was examined chromatographically, the above two fractions contained chiefly acetyldigitoxin. The procedure in subsequent isolations was shortened by adding the skellysolve B directly to the ether concentrate and the total glycosides obtained by one filtration. Concentration of the ether–skellysolve filtrate to remove about half the ether caused the separation of further small quantities of acetyldigitoxin together with some green pigments. The filter cell finally was extracted with methylene dichloride to yield an additional small amount of glycosides which could have been extracted very slowly by additional quantities of ether. Of the solvents tested, ether gave the most satisfactory results and gave an initial glycoside preparation that had a very high glycoside concentration consisting almost entirely of acetyldigitoxin.

Acetyldigitoxin also can be eluted from the filter cell mixture by isopropyl ether, and upon concentration of this eluate the glycosides separated out as when ether was used.

When 250-Gm. samples of dry *D. mertonensis* were assayed by the above described techniques, values of the order of 0.55 Gm. of acetyldigitoxin were obtained, or 2.10 Gm. per Kg.

In the case of dried leaves in which some deacetylation had taken place during the drying, the same approximate weights of glycoside were obtained that were mixtures of digitoxin and acetyldigitoxin.

When fresh leaves of *D. mertonensis* were used, the only variation in the above techniques was the disintegration of the fresh with aqueous methanol of such a concentration that the final extract was 35%. With fresh leaves, as previously stated, one can be assured of very little or no deacetylation.

Although no effort was made to isolate or determine the acetylgitoxin or gitoxin fractions, all evidence obtained to date indicates that very small amounts of digilanide B are present in *D. mertonensis*. Contrast this with the large amounts of gitoxin (from purpurea glycoside B) that occur in *D. purpurea* grown in Minnesota.

The same techniques that were used for the isolation of acetyldigitoxin from *D. mertonensis* were applied to both the fresh and dried leaves of *D. siberica*. The results were essentially the same as those obtained with *D. mertonensis*, however, considerably fewer pigments were encountered, especially the flavone type. Quantitative studies will be carried out at a later date and reported.

The Isolation of Digitoxin from *D. purpurea*.—The isolation of digitoxin from *D. purpurea* was accomplished with ease by the same techniques used to isolate acetyldigitoxin from *D. mertonensis*. The gitoxin separated from the methylene dichloride ether solution both before and after concentration and very little gitoxin was encountered in the final digitoxin preparation.

When 250 Gm. samples of dry *D. purpurea* were assayed by the above described techniques, values of the order of 0.225 Gm. of digitoxin were obtained, or 0.90 Gm. per Kg. This is less than half of that obtainable from *D. mertonensis*.

REFERENCES

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ERRATUM

In the paper titled "Rheology of Thixotropic Montmorillonite Dispersions II. Kinetics of Structural Recovery" (1), the legend in the caption for Fig. 1 should read: △, 2 weeks; ●, 6 weeks; ○, 11 weeks; and □, 17 weeks. In addition, the reference to Fig. 3 in line 4, paragraph 5, column two, at page 953 should be a reference to Fig. 1.

(1) Levy, G., This Journal, **51**, 952(1962).