# PHYTOCHEMICAL NOTES.*

## No. 104. THE INORGANIC CONSTITUENTS OF ECHINACEA.

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While engaged in the chemical study of *Echinacea*, the precipitation method of the so-called simple bases by means of phosphotungstic acid was tried out as directed.[1] Having used 1500 Gm. of the reagent, the precipitation seemed to continue indefinitely, yet the precipitate, when worked up according to the conventional method, yielded no organic base, although the extractive from 100 lbs. of drug had been used.

Inasmuch as phosphotungstic acid forms an insoluble potassium salt, it seemed desirable to ascertain the amount present of this element. It was possible that because so much drug was being worked on, its potassium content interfered with the reagent as a precipitant of the organic bases. While making this determination, it seemed best to make a quantitative determination of other inorganic constituents as well.

Approximately 100 Gm. of the crude drug were heated until vapors ceased to be given off. The charred mass was lixiviated with boiling water after having been transferred to a filter. The filtrate, amounting to about $1^1/_2$ liters, was evaporated to dryness, leaving a brown-colored residue which constituted the water-soluble ash. The portion remaining on the filter was now heated to a higher temperature until all the carbon was burned leaving a grayish white ash which constituted the water-insoluble ash.

The water-soluble ash was analyzed for potassium by precipitation with $HClO_4$ in the presence of 96 p. c. alcohol,[2] after the sulphates had first been removed and determined by precipitation with $BaCl_2$ as given in the procedure.

The carbonates were determined by titration with $N/10$ $H_2SO_4$. Using the same sample after titration, the chlorides were precipitated with $AgNO_3$ in the presence of $HNO_3$.[3]

The water-insoluble ash was treated with 10 p. c. HCl and filtered as described under the "Procedure for the Determination of Calcium."[4] The insoluble residue was ignited to constant weight and weighed as $SiO_2$. The filtrate was analyzed for calcium by precipitation with ammonium oxalate and ignition to CaO. The magnesium remaining in solution was precipitated with sodium ammonium phosphate[5] and ignited to $Mg_2P_2O_7$.

During the analysis for calcium, when the solution was treated with $NH_4OH$, a reddish brown precipitate resulted. It was filtered off, ignited to constant weight and the iron content determined by titration with $N/10$ $KMnO_4$.[6] The difference between the total weight and the weight of iron thus determined was calculated as aluminum.

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[1] Barger, "The Simpler Natural Bases," page 118.

[2] G. N. Smith, "Quantitative Chemical Analysis," pages 68–69.

[3] *Ibid.*, page 55.

[4] *Ibid.*, page 75.

[5] *Ibid.*, page 79.

[6] *Ibid.*, page 134.

In order that the percentage of the various constituents found could be calculated on the basis of the total ash and crude drug, it was necessary to carry out an analysis on a small sample. The water-soluble, acid-soluble, water-insoluble and acid-insoluble ash were determined by treatment similar to that of the larger quantity which was used for analysis. The results of the several determinations are herewith tabulated:

| | | Average. |
|---|---|---|
| Total ash | 7.76 p. c.<br>7.72 p. c. | 7.74 p. c. |
| Water-soluble ash | 1.88 p. c.<br>1.82 p. c. | 1.85 p. c. |
| Water-insoluble ash | 5.88 p. c.<br>5.90 p. c. | 5.89 p. c. |
| Acid-insoluble ash | 3.21 p. c.<br>3.11 p. c. | 3.16 p. c. |
| Acid-soluble ash (by difference) | | 4.58 p. c. |

ANALYSIS OF WATER-SOLUBLE ASH.

| | Percentage with reference to. | | | |
|---|---|---|---|---|
| Ion. | Water-soluble ash. | | Total ash. | Crude drug. |
| K | 16.81<br>17.14 | 16.97 p. c. | 4.05 p. c. | 0.314 p. c. |
| $SO_4$ | 13.12<br>13.39 | 13.26 p. c. | 3.17 p. c. | 0.245 p. c. |
| $CO_3$ | 37.86<br>38.92 | 38.39 p. c. | 9.17 p. c. | 0.710 p. c. |
| Cl | 4.04<br>4.17 | 4.10 p. c. | 0.98 p. c. | 0.076 p. c. |

ANALYSIS OF WATER-INSOLUBLE ASH.

| | Percentage with reference to. | | | |
|---|---|---|---|---|
| Ion. | Water-insol. ash. | | Total ash. | Crude drug. |
| Ca | 13.50<br>12.88 | 13.19 p. c. | 10.03 p. c. | 0.776 p. c. |
| Mg | 1.98<br>2.02 | 2.00 p. c. | 1.52 p. c. | 0.117 p. c. |
| $SiO_3$ | 26.29<br>26.14 | 26.21 p. c. | 19.95 p. c. | 1.543 p. c. |
| $Fe'''$ | 0.747<br>0.907 | 0.827 p. c. | 0.628 p. c. | 0.048 p. c. |
| Al | 2.30<br>2.08 | 2.19 p. c. | 1.66 p. c. | 1.29 p. c. |

From the data tabulated above, it becomes apparent that the extract from 100 lb. of crude drug contains 142.5 Gm. of potassium which would require 4109.5 Gm. phosphotungstic acid for precipitation.

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## BILL FAVORS NATIONAL ANTHEM.

A sub-committee of the House Committee on Judiciary February 4th voted to make a favorable report to the Committee February 5th on a bill (H. R. 14), which would make the "Star Spangled Banner" the national anthem of the United States. The bill, however, as the sub-committee voted to report it to the Committee reads as follows: "That the composition, consisting of the words and music known as the "Star-Spangled Banner," is designated the national anthem of the United States." This wording was taken from the Army and Navy regulations of 1914 and was adopted by President Wilson. The anthem was composed by Francis Scott Key during the bombardment of Fort McHenry.