

an ion in common with that of borax, and a similar degree of alkalinity. All samples were filtered after 48 hours and set aside for observation. The details and results are summarized in Table VIII.

It appears that the sodium ion cannot be more than partly responsible for the reduced amount of precipitation following treatment with borax. Many similar experiments had previously been performed using small amounts of 10% sodium hydroxide solution, but the results were too erratic to be of value. In view of the known affinity of borax for complex formation, according to Dimroth and Faust (1), it would seem more likely that the borate ion was responsible. However, time did not permit us to extend these experiments sufficiently to prove the point. This is suggested as a topic for further study.

SUMMARY.

(a) It has been shown that treatment of fluidextract of senna with 2.0% borax and filtration after 48 hours markedly reduces precipitation in this preparation.

(b) There is apparently no direct relationship between total solids in the fluidextract of senna and the effectiveness of the borax.

(c) It was indicated that borate-KH₂PO₄ buffers might be helpful in reducing precipitation, when combined with the menstruum, replacing 10% of the water. The range of effectiveness was p_H 6.4 to 8.4.

(d) Evidence has been presented which indicates that the sodium ion of the borax can be only partly, if at all, responsible for reducing precipitation in fluidextract of senna.

REFERENCES.

- (1) Dimroth, O., and Faust, F., *Bull. soc. chim.*, 32, 720 (1922).
- (2) Kolthoff, I. M., *J. Biol. Chem.*, 63, 135 (1925).

STUDIES UPON PRECIPITATION IN FLUIDEXTRACT OF SENNA. IV.*

THE NATURE AND PROPERTIES OF THE PRECIPITATE IN FLUIDEXTRACT OF SENNA.¹
KARL L. KAUFMAN² AND C. O. LEE.³

The precipitate that commonly forms in fluidextract of senna is a dark brown, amorphous mass. It is lighter in color, and somewhat scaly when it forms in the cold.

A working quantity of the precipitate was obtained by making up a composite sample which was collected from many bottles of the fluidextract. These ranged in age from six months to three years. The precipitate was dried over calcium chloride and triturated to a fine powder. It was then subjected to various qualitative tests.

* Scientific Section, A. Ph. A., Dallas meeting, 1936.

¹ Based upon a thesis submitted to the Faculty of Purdue University by Karl L. Kaufman, in partial fulfilment of the requirements for the degree of Doctor of Philosophy.

² J. K. Lilly Fellow, Purdue University, School of Pharmacy, 1933-1936.

³ Professor of Pharmacy, Purdue University, School of Pharmacy.

SOLUBILITY.

Its solubility properties were studied according to the Dragendorff solvents. The averages of three such tests, expressed in per cent by weight, are as follows:

Petroleum ether	12.0%
Anhydrous ether	1.1%
Anhydrous alcohol	13.5%
Water	39.1%
0.15% NaOH solution	10.2%
0.10% HCl solution	15.1%
Total	91.0%

In addition to these, three other solvents were used and the solubility of the precipitate determined by weight differences. The results are as follows:

Water	50.0%
7% H ₂ SO ₃ solution	5.8%
2% NaOH solution	12.3%

Numerous other liquids were tested for their solvent effects upon the precipitate, but none were of sufficient interest to report.

ASH ANALYSIS.

The ash content of the precipitate averaged 22.6% which is about twice the ash content of the crude drug.

Samples of the precipitate, collected from fluidextracts, 48 hours after being treated with borax, gave an ash yield of 33.48%. These samples contained some borax, which may account for the larger ash content.

INORGANIC QUALITATIVE ANALYSIS.

Four fractions of the precipitate were subjected to a qualitative analysis. The results are summarized in Table IX.

Those portions of the precipitate which remained insoluble in H₂SO₃ and NaOH solutions were a dirty, fluffy residue. Upon microscopic examination, tiny bits of plant materials were to be seen. The diatom *arachnoidiscus Ehrenbergii* was found to be a contaminant, although we are unable to explain its presence.

TABLE IX.

Ions Present.	Water-Soluble Portion.	Sulfurous Acid-Soluble Portion.	NaOH-Soluble Portion.	Water Extract of Ash from Residue.
Cations	Al, K,* Mg, Na	K*	Al, K,* Ca, Na	Ca, K*
Anions	SO ₄ , PO ₄ , Cl, NO ₃ , Si ₂ O ₃	SO ₄ * K ₂ SO ₄ crystals	NO ₃ , SO ₄ , PO ₄ , CN, Si ₂ O ₃	CO ₃ , Si ₂ O ₃
Dialyzed portion	All of the above ions	All of the above ions
Dialysate	All of the above ions	All of the above ions except silicate

* Present in large amounts. The other ions listed were present in traces only.

CONSTITUENTS OF THE PRECIPITATE COMPARED WITH THOSE OF THE CRUDE DRUG.

As a procedure for examining the precipitate, the one given by Tutin (1) for the examination of the crude drug senna was used. In applying this technique

to the precipitate, certain minor adaptations were necessary, the details of which will not be discussed here. The presence of rhein, kaempferol, aloë-emodin and organic acids in the sediment of fluidextract of senna was indicated. These are also to be found in the crude drug.

A water-insoluble resinous substance was obtained from an alcoholic extract of the precipitate by subjecting the latter to steam distillation. In quantity it represented about one-sixth of the total solids extracted with alcohol.

About fifty per cent of this resinous substance was soluble in potassium hydroxide solution, the solution yielding a red color, characteristic of anthraquinone derivatives. According to its solubility in potassium hydroxide, this substance should consist of about one-half "acid resins," the remainder being "indifferent resin" or "resin anhydride." The alkali insoluble portion of this resinous mass was quite readily soluble in nitric acid.

THE PRECIPITATE FROM THE BORAX-TREATED SAMPLES OF THE FLUIDEXTRACT OF SENNA.

The precipitates from the borax-treated samples were subjected to solvent, and other tests. The results are summarized as follows:

1. The "borated" precipitates yielded about three per cent more alcohol-soluble extractive than the ordinary precipitates.
2. The "borated" precipitates yielded much more extraneous amorphous material than the regular precipitates. This made it hard to test for the constituents, but anthraquinone substances were in evidence.
3. Upon being subjected to steam distillation the "borated" precipitates were found to yield much less insoluble residue than the usual precipitates. The residue was not soluble in nitric acid and gave the hydroxyanthraquinone reaction.

REFERENCES.

- (1) Tutin, F., *Trans. Chem. Soc.*, 103, 2006 (1913).
- (2) Dragendorff, G., "Plant analysis," page 8, translated by H. G. Greenish.

NEW IODINE—BILE COMPOUND (IODOCHOLEATE).*

BY PAUL GOEDRICH.¹

THE PHYSICAL AND CHEMICAL PROPERTIES, GERMICIDAL AND PHARMACOLOGIC ACTION.

At the time that iodine was introduced to medicine as a germ-killing agent its toxic properties were known, and they caused very much concern. This situation has not changed very much.

The destructive action of iodine to the broken and unbroken skin, especially when covered by cloths or bandages, limits its field largely to First Aid; also in preparing the operative field, iodine must be carefully removed by alcohol to avoid tissue necrosis.

* Scientific Section, A. P. H. A., Dallas meeting, 1936. Abstracted by the author for publication.

¹ From the Research Laboratories of the New Jersey College of Pharmacy, Rutgers University.