STUDIES UPON PRECIPITATION IN FLUIDEXTRACT OF SENNA. III.*

THE PREVENTION OF PRECIPITATION IN FLUIDEXTRACT OF SENNA BY THE ADDITION OF BORAX AND OTHER AGENTS.[1]

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In a previous paper it was noted that borax, when added to fluidextract of senna, markedly reduced the amount of precipitate. Accordingly, numerous samples of the preparation were prepared and treated with borax for study. It was our purpose to determine the optimum concentration of borax necessary to yield a minimum of the precipitate in the fluidextract. To this end two series of six samples each of the fluidextract were prepared. One sample of each series served as the control. To the other samples varying amounts of borax were added. After 48 hours all samples were filtered and set aside for later observations. The details are recorded in Table VI following.

TABLE VI.

Sample Number.	Gm of Borax Added.	Precipitation after 128 Days.	322 Days.
71-A	None	++	+++
71-B	2.5	+	+
71-C	4.0	++	+++
71-D	5.0	++	+++
71-E	7.5	++	++++
71-F	None[1]	+	++
		87 Days.	120 Days.
191-a	None	+++	+++
191-b	1.25 (1/2%)	++	+++
191-c	2.50 (1%)	++	++
191-d	3.75 (1 1/2%)	+	+ to ++
191-e	5.00 (2%)	++	++
191-f	6.25 (2 1/2%)	++	++

[1] Adjusted to p_H 6.54 by the addition of NaOH and HCl solutions.

From the results of this experiment it was concluded that borax, as an agent for reducing precipitation in fluidextract of senna, was most effective in concentrations of 1.5 to 2.5 per cent. Furthermore, it seemed quite as effective, in this respect, after a period of 48 hours, as it did after 30 days.

THE EFFECTIVENESS OF BORAX IN RELATION TO TOTAL SOLIDS IN THE FLUIDEXTRACT.

Samples of fluidextract of senna, containing borax in concentrations of 2%, occasionally had somewhat more than a plus (+) deposit. It was thought that this variation in quantity of precipitate might be due to the differences in total solids in the samples of the fluidextract.

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[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.

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To determine this, portions of a sample of a fluidextract, having a density of 1.067 and containing 22.4% W/W of solids, were increasingly diluted with the official menstruum. To each sample, except the control, there was added 2% W/V of borax. The samples were all filtered after 48 hours and set aside for observation. The results are given in Table VII, following.

TABLE VII.

Sample Number.	Cubic Centimeters. Sample.	Menstruum Added.	Gm. of Borax Added.	Precipitation after 77 Days.	126 Days.
181-1	250	None	None	++	+++
181-2	250	None	5	+	+
181-3	225	25	5	++	++
181-4	200	50	5	++	+++
181-5	175	75	5	+	+
181-6	150	100	5	++	++
181-7	125	125	5	++	++

No definite relationship could be noted between the total solids and the effectiveness of borax.

THE USE OF BORAX BUFFERS IN THE MENSTRUUM FOR FLUIDEXTRACT OF SENNA.

The effectiveness of borax as an agent in reducing precipitation in fluidextract of senna, when added directly to the preparation, has been shown. Efforts were next made to test its value when added to the menstruum rather than to the finished product.

Buffers were made up, consisting of borax-succinic acid, and borax-phosphate combinations according to Kolthoff (2). They ranged from p_H 4.0 to 8.4, inclusive, at intervals of 0.4 p_H. They were added in quantities equivalent to 10% of the menstruum.

Forty-three samples were prepared and studied over a considerable period of time. It was concluded that the borax-phosphate buffers, in the range p_H 6.4 to 8.4, were somewhat effective in reducing precipitation in fluidextract of senna.

BORAX AND OTHER BASIC SALTS.

The alkalinity of borax suggests itself as the mechanism by which the salt acts in reducing sedimentation in fluidextract of senna. If the answer is not in the alkalinity, but in the ions furnished by the borax, it then becomes a problem to determine the effective ion.

TABLE VIII.

Sample Number.	Reagent Added.	Precipitation after 116 Days.	172 Days.
151-2	2% $Na_2B_4O_7.10H_2O$	++	++
151-3	2% NaCN	+++++	++++
151-4	2% Na_2HPO_4	++++	++++
151-5	Control	++++	++++
		After 76 Days.	112 Days.
221-1	Control	+++	+++
221-2	2% CH_3COONa	++	+++

To test out these conjectures, six samples of fluidextract of senna were prepared. Two served as controls. To each of the other four were added basic salts having

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-KH_2PO_4 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]
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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.

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