of the uterine muscle, we are forced to the conclusion that the drug is valueless in the conditions for which it is prescribed. We offer the suggestion that it be eliminated from our materia medica. As long as certain physicians prescribe it, deletion from the formulary may not be deemed advisable, but it would seem logical to urge strongly abandonment of any administration.

SUMMARY.

A proximate analysis of senecio was made, including moisture, ash, tannins, resins, etc. There was found 0.1 per cent of volatile oil whose constants are given, also about 8 per cent of inulin and no starch. No evidence could be found for the presence of alkaloids or glucosides.

Even with enormous doses, senecio caused no untoward effects in rats or rabbits. Numerous experiments on isolated uterine muscle and on normal uterine movements in intact animals clearly demonstrated the absence of any effect on the tone, rate or amplitude of this muscle.

REFERENCES.

(1) Barbour, *J. Pharmacol. and Exp. Therap.*, 7 (1915), 547.
(2) Pilcher, *Ibid.*, 8 (1916), 110–111.
(3) Kelly and Lynn, JOUR. A. PH. A., 20 (1931), 755–759.
(4) Manske, *Can. J. Research*, 5 (1931), 651–659.

THE ASSAY OF CAFFEINE SODIO-SALICYLATE AND ELIXIR OF SODIUM SALICYLATE.

BY EDWARD M. HOSHALL, DONALD C. GROVE AND GLENN L. JENKINS.

CAFFEINE SODIO-SALICYLATE.

This preparation has been recommended for admission into the National Formulary VI. A method of assay is proposed.

Assay for Caffeine.—Transfer 2.0 Gm. of caffeine sodio-salicylate, previously dried to constant weight at 80° C., and accurately weighed, to a 100-cc. volumetric flask and make to volume with distilled water. Transfer a 10-cc. aliquot to a separatory funnel, add 5 cc. sodium hydroxide T.S. and extract the caffeine with successive portions of chloroform until the residue gives no test for alkaloids with iodine (T.S.). Pass the chloroform solutions through a filter which has previously been moistened with chloroform and wash the stem of the funnel and filter with a few cc. of the solvent to remove any adhering caffeine. Evaporate the combined chloroform solutions on a water-bath, and dry the residue of anhydrous caffeine to constant weight at 80° C.

Assay for Sodium Salicylate.—Transfer the aqueous liquid from which the caffeine has been removed by the above assay for caffeine to a 500-cc. glass-stoppered Erlenmeyer flask, rinsing the separatory funnel with small portions of distilled water. Also wash the filter and funnel used in the caffeine determination with small portions of water, adding the washings to the 500-cc. flask. Add sufficient distilled water to make the volume in the flask about 100 cc. Add 50 cc. 0.1*N* Bromine solution, 10 cc. of hydrochloric acid, then stopper and shake for one minute, then at intervals for thirty minutes. Add 10 cc. of 15 per cent potassium iodide solution, stopper and shake for five minutes. Titrate the liberated iodine with 0.1*N* sodium thiosulphate solution, using starch T.S. as indicator.

Each cc. of 0.1*N* bromine is equivalent to 0.002668 Gm. $NaC_7H_5O_3$.

Experimental.—According to National Formulary VI *Bulletin*, page 325, the formula and

directions for the preparation of caffeine sodio-salicylate will be the same in the National Formulary VI, as they now are in the National Formulary V. Due to the efflorescent nature of hydrated caffeine, the water content will be extremely variable as shown by W. W. White.[1] Because of this fact it is recommended that the stable anhydrous caffeine (dried at 80° C.) be used for this preparation.

Accordingly the preparation was compounded by following the method as in the National Formulary *Bulletin*, page 525, substituting caffeine dried to constant weight at 80° C., for the hydrated caffeine. The same sodium salicylate as used in the elixir of sodium salicylate was used. The analysis appears in Table II.

The preparation was then assayed by the method given above, with the following results.

TABLE I.

	Analyst.	Amount Present Per Cent.	Amount Found Per Cent.		Error Per Cent.
Sodium Salicylate	A	49.82	49.71	49.74	−0.2
	B	49.82	49.64	49.67	−0.2
Caffeine (dried at 80° C.)	A	50.00	50.01	49.79	−0.2
	B	50.00	49.84	49.77	−0.4

CONCLUSION.

1. Suitable methods of assay for caffeine sodio-salicylate have been developed. It is recommended that the assay be adopted as official in the National Formulary VI.

2. It is recommended that caffeine (dried to constant weight at 80° C.), be substituted for caffeine hydrated, in this preparation.

3. It is recommended that the following tolerances be adopted:

The preparation shall contain not less than 47.5 per cent or more than 52.5 per cent of sodium salicylate.

The preparation shall contain not less than 47.5 per cent or more than 52.5 per cent of caffeine dried to constant weight at 80° C.

ELIXIR SODIUM SALICYLATE.

This preparation has been recommended for admission into the National Formulary VI. A method of assay is proposed.

Assay for Sodium Salicylate.—Transfer 10 cc. of the preparation to a 250-cc. volumetric flask and make to volume with distilled water. Transfer a 10-cc. aliquot to a 500-cc. glass-stoppered Erlenmeyer flask, add 100 cc. of distilled water, 50 cc. of 0.1*N* bromine solution and 10 cc. of hydrochloric acid. Stopper and shake for one minute, then at intervals for thirty minutes. Add 10 cc. of 15 per cent potassium iodide solution, stopper and shake for 5 minutes. Titrate the liberated iodine with 0.1*N* sodium thiosulphate solution, using starch T.S. as indicator.

Each cc. of 0.1*N* bromine = $(0.002668 \times \frac{250}{10} \times 10) = 0.667$ Gm. $NaC_7H_5O_3$ per 100 cc. of solution.

Experimental.—Sodium salicylate (a C.P. quality salt), was assayed by the above method, and also by the extraction and determination of the salicylic acid content. The results are given in the following table.

TABLE II.

	Analyst.	Assay by Proposed Method Per Cent.		Assay by Determination of Salicylic Acid Per Cent.	
Sodium Salicylate	A	99.62	99.62	99.65	99.38
	B	99.76	99.52	99.66	99.57
	Average	99.63%		99.57%	

[1] U. S. P. XI Circulars, General Committee, page 170.

Using the assayed salt and following the method of preparation of Elixir Sodium Salicylate as in the National Formulary VI *Bulletin*, the product was compounded and assayed by the method as presented above. The results appear in Table III.

TABLE III.

	Analyst.	Amount Present Gm./100 cc.	Amount Found Gm./100 cc.	Error Per Cent.
Sodium Salicylate	A		24.77	−0.6
		24.908	24.75	
	B		24.73	−0.6
			24.74	

CONCLUSIONS.

1. A suitable method of assay for Elixir of Sodium Salicylate has been developed. It is recommended that the method of assay herein proposed be adopted as official in the National Formulary VI.

2. It is recommended that the following tolerance be adopted for this preparation: 100 cc. of Elixir of Sodium Salicylate contains not less than 24.5 Gm. or more than 25.5 Gm. of sodium salicylate.

The Pharmacy Exhibit at the Century of Progress will probably be continued this year. Editorial comment is made in this issue of the JOURNAL.