

Procaine	Immed.	1 Min.	10 Min.	Chlor. 10 Min.	Apomorph. 15 Min.
0.3 cc.	xxx	xxx	xxx	xxx	xxx
0.35	xxx	xxx	xxx	xxx	xxx
0.4	xxx	xxx	xxx	xxx	xxx
0.42	xxx	xxx	xxx	xxx	xxx
0.44	xxx	xxx	xxx	xxx	xxx
0.46	—	xx	xx	xx	xxx
0.47				x	xx
0.48	—	x	x	x	xx
0.48				x	xx
0.49				x	xx
0.5	—	—	—	?	x
0.5				?	x
0.51				—	—?
0.52				—	—
0.52	—	—	—	—	—
0.54	—	—	—	—	—

The results indicate that 1 cc. of *N*/100 bromine solution is decolorized by 0.5 cc. Procaine HCl *M*/200, as shown by the chloroform test, and by 0.505 cc. shown by the apomorphine test. Hence 0.682 mg. Procaine HCl absorbs 0.7992 mg. bromine.

SUMMARY.

1. Tenth normal bromine solution (Koppeschaar's solution) was used for the quantitative estimation of a number of alkaloids. Two general modifications of the method are described.

2. By this means quantitative estimations of a number of alkaloids may be made with an error of about 0.5 per cent in concentrations of about 1–1000.

3. The error in the quantitative estimation of substances in solutions of unknown concentration increases with the dilution, but antipyrine, amidopyrine and salicylic acid in concentrations of 1–1,000,000 may be determined with errors of from 5 to 10 per cent.

4. The estimations are made with controls in which attention must be paid to concentration, temperature, rate of reaction and other factors discussed in the paper.

REFERENCES.

- (1) Hart, W. B., *Soc. Chem. Ind. J.*, 13, 60, 72 (1921).
- (2) Weiss, S., and Hatcher, R. A., "Proc. Soc. Exptl. Biol. and Med.," 23, 33 (1925).
- (3) Haag, H. B., *Jour. A. Ph. A.*, 22, 21 (1933).

THE AIR-LIFT EXTRACTOR APPLIED TO THE ANALYSIS OF ALKALOIDAL DRUG EXTRACTS.

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The extraction of alkaloids from pharmaceutical preparations has always been time-consuming and many attempts have been made to accomplish the extraction by means of automatic devices. Most of these have been regarded as unsuitable because they depend upon refluxing of the solvent by heat (1) with a resultant possible decomposition of the alkaloid and because of the difficulty of determining when the extraction is complete (2). The air-lift extractor (3) ac-

compleishes the extraction at room temperature, thereby eliminating any error due to heating and it is provided with a stop-cock by means of which samples may be drawn off and tested for completeness of extraction.

In the original articles this extractor was used for the extraction of organic acid and its collection and measurement in standard alkali solution. In the present series of experiments the reverse of this operation is performed, an organic base is extracted and deposited in an acid solution. By this procedure two series of hand shake-outs are eliminated and it is then only necessary to remove the acid solution from the apparatus, make it alkaline with ammonia, proceed with the final extraction in a separatory funnel and titrate or weigh the alkaloid in the usual manner. Ammonia is used in the apparatus in place of caustic alkali because chloroform is not decomposed as readily by ammonia (4) and because in making the solution alkaline with ammonia the analysis more closely parallels the official methods for alkaloidal assays (5). A correction factor is not necessary since the acid solution itself is not titrated but must undergo another extraction before the alkaloid is titrated.

Method.—The large tube of the apparatus (3) is filled nearly to the overflow with chloroform, the preparation to be extracted is superimposed upon it and made alkaline with ammonia. A small quantity of chloroform is placed in the smaller tube, 40 cc. of 5% sulphuric acid is added and then chloroform until the top of the acid layer is almost to the inlet. The air (or nitrogen) is allowed to enter and extraction continued until all of the alkaloid is deposited in the acid layer. This point may be determined by removing a small quantity of chloroform solution through the stop-cock at the bottom of the tube and testing it in the usual manner with Mayer's Reagent. About four hours are required for the complete extraction of the alkaloid. The acid is then removed from the tube, the tube rinsed with water and the final extraction made with chloroform in a separator after making the acid solution alkaline with ammonia.

A series of extractions made by the air-lift extractor and checked by the hand shake-out method of the U. S. P. X shows the two methods to agree within the limits of experimental error (cf. table). The air-lift extractor method requires about one-half the actual working time of the other method although the total elapsed time is somewhat longer.

EXTRACTION OF ALKALOID FROM DRUG EXTRACTS BY THE AIR-LIFT EXTRACTOR AND
U. S. P. X METHODS.

Sample.	Air-Lift.	U. S. P. X.
Powd. Ext. Nux Vomica	12.77% alkaloids	12.83% alkaloids
Fluidextract Nux Vomica	(a) 2.55 Gm. alkaloids (b) 2.17 Gm. per 100 cc.	(a) 2.52 Gm. alkaloids (b) 2.20 Gm. per 100 cc.
Fluidextract Belladonna	0.363 Gm. per 100 cc.	0.390 Gm. per 100 cc.
Tincture Hyoscyamus	0.00765 Gm. per 100 cc.	0.00788 Gm. per 100 cc.

BIBLIOGRAPHY.

- (1) Paget, *J. Soc. Chem. Ind.*, 51, 190 (1932).
Palkin, Murray and Walkins, *Ind. Eng. Chem.*, 17, 612 (1925).
- (2) "Proc. Am. Drug. Manuf. Assoc." 160 (1932).
- (3) Chapman and Hammett, *Ind. Eng. Chem., Anal. Ed.*, 5, 346 (1933).
- (4) Desgrez, *Compt. rend.*, 125, 780 (1897).
- (5) U. S. Pharmacopœia X, page 452.