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Drug Extraction. XXIV. The Effect of the Length of Drug Column on the Efficiency of Percolation of Cinchona*

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Recent studies of drug extraction have shown that the method of forced percolation through a long column of drug has given more efficient extraction of several drugs than could be obtained by ordinary percolation (1, 2, 3). However, little information has been available regarding the effect of variations in the length of the drug column on the efficiency of extraction. Hence some research has been carried out on this point, using cinchona as representing an important drug which offers difficulties in extraction.

EXPERIMENTAL

Materials Used.—Yellow cinchona, U. S. P., in moderately coarse (No. 40) powder, assaying 7.31% alkaloids and 8.87% moisture was used. The menstrua employed were those prescribed in the U. S. P. X for fluidextract of cinchona.

Analytical Methods.—Alkaloidal content of the percolates was determined by the U. S .P. XI method for Compound Tincture of Cinchona, with a modification in the amount of sample taken. Asbestos fiber was used as the adsorbent. Moisture was determined by the U. S. P. XI method for drugs containing no constituents volatile at 100° C. To determine total extractive, 10 cc. of the liquid were evaporated to dryness on a water bath and then heated in an oven at 105° C. until the difference between two successive weighings did not exceed 10 mg.

Apparatus and Procedure.—The apparatus described by Husa and Huyck (1) was employed, the length of the drug column being increased or decreased by varying the number of tubes and elbows. The tubes were of Pyrex glass, each tube having a length of 91 cm. and an internal diameter of 2.5 cm. The drug was moistened with 25 cc. of Menstruum I per 100 Gm. of drug and packed, in sections, with moderately firm pressure, using 20 to 25 Gm. of drug for each packing portion. After being packed with drug, the tubes and elbows were joined together by means of rubber jackets and metal joint flanges. One end of the drug column was connected to a storage tank containing Menstruum II. This menstruum was forced through the drug column by means of air pressure obtained from the compressed air line, using a reducing valve and gage to secure the desired pressure. The percolate was collected in four equal fractions, the volume of each fraction amounting to 0.5 cc. for each Gm. of drug used. Each experiment was repeated and the data in the tables represent the averages of the duplicate experiments. No breaks in the drug column were observed in any of the experiments. The experimental conditions are given in Table I and the results are summarized in Table II.

DISCUSSION OF RESULTS

Table III shows the per cent of total alkaloids extracted in the first fraction, second fraction, and total of the first two fractions of percolate. Table IV shows the amount of total extractive obtained in 100 cc. of percolate in the first and second fractions, and also the total amount obtained in these two fractions.

The results in Tables III and IV indicate that the efficiency of extraction of alkaloids and total extractive is about the same for one and two tubes. The efficiency of extraction is greater when the drug column is lengthened by using three and five tubes, respectively.

Table I.—Experimental Data

	Experiments			
	A	B	C	D
Number of tubes used	1	2	3	5
Weight of drug in Gm.	300	600	950	1650
Approximate volume of packed drug in cc.	630	1295	1955	3325
Air pressure used	Up to 3 lb.	Up to 4 lb.	Up to 4 lb.	Up to 8 lb.
Average temperature	24.5° C.	24.5° C.	24.5° C.	23.5° C.
Time required for experiment (in hours)	29.5	98.1	210.5	449.2

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Table II.—Assay Results

Fraction	Gm. Total Extractive in Fraction	Gm. Alkaloids in Fraction	Per Cent Alkaloids Extracted
Experiment A			
1 (150 cc.)	39.22	6.45	29.43
2 (150 cc.)	28.87	4.84	22.08
3 (150 cc.)	19.80	3.41	15.56
4 (150 cc.)	11.31	2.20	10.04
Total (600 cc.)	99.20	16.90	77.11
Experiment B			
1 (300 cc.)	88.04	13.40	30.57
2 (300 cc.)	50.00	8.72	19.89
3 (300 cc.)	33.83	5.64	12.87
4 (300 cc.)	23.97	4.56	10.40
Total (1200 cc.)	195.84	32.32	73.73
Experiment C			
1 (475 cc.)	141.48	22.09	31.83
2 (475 cc.)	93.84	16.20	23.34
3 (475 cc.)	62.35	10.26	14.78
4 (475 cc.)	36.44	6.77	9.75
Total (1900 cc.)	334.11	55.32	79.70
Experiment D			
1 (825 cc.)	262.81	41.21	34.19
2 (825 cc.)	169.37	29.17	24.20
3 (825 cc.)	105.69	17.70	14.68
4 (825 cc.)	36.26	8.42	6.98
Total (3300 cc.)	574.13	96.50	80.05

Table III) may be considered as experimental error. However, some investigators have considered such variations as being real fluctuations, which were not readily explainable but which might be due to surface phenomena of adsorption, etc. (4, 5, 6).

The general results of the present study, indicating a somewhat greater efficiency of extraction with the longer drug columns, are in agreement with the results of J. U. Lloyd (7), who found that, in the extraction of cimicifuga, the longer the drug column the greater was the amount of extractive matter in the reserve percolate. Similarly Lefevre and Lee (8) found that increasing the length of the drug column increased the efficiency of extraction of krameria when the N. F. VI process of percolation was employed.

SUMMARY

A study was made of the effect of variations in the length of the drug column on the efficiency of extraction of cinchona in the process of forced percolation through a long column of drug. The general results indicate a somewhat greater efficiency of extraction with the longer drug columns.

The longest drug column used yielded only about 58% of the total alkaloids in a percolate representing 1 cc. for each Gm. of drug. In similar experiments with belladonna root, podophyllum and ipomea, more than 99% of the active constituents were obtained in a corresponding proportion of percolate.

Table III.—Efficiency of Extraction of Alkaloids

Experiment	Tubes and Elbows Used	Per Cent Alkaloids Extracted		
		Fraction 1	Fraction 2	Total Per Cent
A	1	29.43	22.08	51.51
B	2	30.57	19.89	50.46
C	3	31.83	23.34	55.17
D	5	34.19	24.20	58.39

Table IV.—Efficiency of Extraction of Total Extractive

Experiment	Tubes and Elbows Used	Gm. in 100 Cc. of Percolate		Total in First Two Fractions in Gm.
		Fraction 1	Fraction 2	
A	1	26.14	19.24	45.38
B	2	29.35	16.67	46.02
C	3	29.79	19.76	49.55
D	5	31.86	20.53	52.39

In general, the greater the length of drug column the higher is the per cent of total alkaloids extracted. It would seem that the length of the drug column is not the most important factor in the extraction of cinchona, since the longest drug column used yielded only about 58% of the total alkaloids in a percolate representing 1 cc. for each 1 Gm. of drug. In similar experiments with belladonna root (1), podophyllum (2) and ipomea (3), more than 99 per cent of the active constituents were obtained in the percolate representing 1 cc. per 1 Gm. of drug. The possibility of a combination between the alkaloids and tannins present in the drug, and subsequent precipitation in the tubes, may have some bearing on the results obtained.

The slightly lower efficiency of extraction of alkaloids with two tubes as compared with one tube (see

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Book Reviews

Organic Reagents in Inorganic Analysis, by IBERT MELLAN, Ph.G., M.Sc., F.A.I.C. xxiii + 682 pages, 15 x 23 cm., index and appendix included. Structural formulas, graphs and tables. The Blakiston Company, Philadelphia, 1941. Price, $9.00.

Superior sensitivity and specificity of organic reagents over those of inorganic reagents used in various phases of qualitative and quantitative analysis have brought rapidly increasing interest on the part of the analyst. As a prelude to the main text of this book the author discusses, in twenty or more pages, fundamental theories and principles of organic reagents as they apply to the testing of inorganic radicles and compounds. Such topics as acidic hydrogen, coördination bond, chelated compounds, oxime and hydroxyl groups, etc., are considered in this short discussion.

Following the theoretical treatment is a group of approximately two hundred organic reagents, alphabetically listed, for testing inorganic chemicals. A short description of some physical and chemical properties is given under each reagent along with a bibliography, which, in some instances, contains as many as forty to fifty references.

In the final section of the book there are, alphabetically arranged, cations, anions and some inorganic compounds, all in alphabetical order, to which the organic reagents, previously presented, have application. Procedure for testing are given together with a bibliography at the end of each procedure.

In general the book is well organized, bibliographies are quite complete and up-to-date and the print is easy to read. However, the author has left much of the detail out of test procedures. The bibliographies are expected to take care of this deficiency. As for instance, in the test for small quantities of lead by the dithizone method the fact that special precaution as to purification of reagents, particularly dithizone, and cleansing apparatus before it is used in the test with a hot aqueous nitric acid solution has been entirely ignored. Although the author has stressed the sensitivity of some of these reagents in the first part of the text he has lost sight of it in a lack of detailed specifications where necessary. Some of these reagents can be used not only in spot tests and colorimetric qualitative analysis but have been adapted to very accurate quantitative work, again citing the dithizone as an example where quantities of lead have been accurately determined between four or five gamma.

This book serves only as a guide to the literature on the subject of organic reagents. Judging from the extent of the bibliographies this is what the author intended for it to be. In such a wide field he had no room for extra details in so small a space.—E. C. B.

The Glass Electrode. Methods, Applications, and Theory, by MALCOLM DOLE. First Edition, xv + 332 pages, illustrations. John Wiley and Sons, Inc. 440 Fourth Avenue, New York, New York, 1941. Price, $4.50.

This is the first book of its kind. With the introduction and perfection of thermionic tubes, the glass electrode has become a very useful tool in industrial work and in the determination of pH of those materials whose pH cannot be determined by hydrogen electrodes.

The book is both timely and authoritative. The author, from his own experience, has written a fine treatise with an extended bibliography of the construction, theory, advantages, limitations and application of the glass electrode which should be a valuable reference book for years. The chapters on the pH of living tissues and natural products are interesting and informative. Several chapters are devoted to the pH of unbuffered solutions, micro methods and automatic pH control which the author expertly employs to illustrate the wide application of the glass electrode.

Professor Dole devotes several chapters to the standardization of the pH scale by various methods, which though not a complete and ultimate answer, is nevertheless a valuable contribution to the efforts to arrive at a uniform practice in pH methods and applications. He suggests several materials, especially potassium acid phthalate which is supplied by the pH Standards Section of the National Bureau of Standards, for use in the calibration of glass electrodes. He also recommends for the same purpose borax, potassium tetraoxalate, and sodium and potassium phosphates, which are also being established as pH standards at the National Bureau of Standards, by potentiometric, colorimetric and conductometric methods. Professor Dole is to be congratulated on the completeness and unusual clarity of this book.—W. J. Hamor

pH and Electrotitrations, by I. M. KOLTHOFF and H. A. LAITINEN. Second Edition, ix + 190 pages, illustrations. John Wiley and Sons, Inc., 440 Fourth Avenue, New York, 1941. Price, $3.00.

This is the second edition of a well-known text intended for college seniors and graduate students in chemistry. It is noteworthy in that it brings together in one book four related subjects of importance in pure and applied chemistry. The first three parts deal with the applications of colorimetric, potentiometric and conductometric methods to a