

ASSAY METHODS FOR SOME N. F. VI PREPARATIONS CONTAINING BROMIDES.*

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There are six preparations containing bromides proposed for admission to the N. F. VI, namely, Elixir of Ammonium Bromide, Elixir of Potassium Bromide, Elixir of Sodium Bromide, Elixir of Three Bromides, Elixir of Five Bromides and Syrup of Five Bromides. As these preparations are quite frequently prescribed by physicians, there is certainly no question as to the need of accurate methods of analysis.

The method of assay proposed below is the U. S. P. X method for the determination of bromides, which has been modified to apply to the N. F. preparations. All quantities given in the assay method are based on the formulas of these preparations as they will appear in the N. F. VI.

(a) As Elixir of Potassium Bromide, Elixir of Ammonium Bromide and Elixir of Sodium Bromide are all to contain the same quantity of the respective bromide (25 Gm. per 100 cc.) the following method may be applied to each.

Assay Method.—Dilute 10 cc. of the elixir to 200 cc. Introduce into an Erlenmeyer flask 25 cc. of the diluted elixir, 50 cc. of 0.1*N* silver nitrate, 2 cc. of nitric acid and 2 cc. of ferric ammonium sulphate T.S. in the order named and determine the excess silver nitrate by titrating with 0.1*N* ammonium thiocyanate until the supernatant liquid remains reddish brown in color after it is well shaken. The difference between the number of cc. of 0.1*N* silver nitrate and the number of cc. of 0.1*N* ammonium thiocyanate multiplied by the equivalent of the respective bromide represents the number of Gm. of bromide in 100 cc. of the elixir.

$$\text{Equivalent for Elixir of Ammonium Bromide} = (0.009796 \times \frac{200}{25} \times 10) = 0.78368.$$

$$\text{Equivalent for Elixir of Potassium Bromide} = (0.011902 \times \frac{200}{25} \times 10) = 0.95216.$$

$$\text{Equivalent for Elixir of Sodium Bromide} = (0.010292 \times \frac{200}{25} \times 10) = 0.82336.$$

EXPERIMENTAL.

Before making up any of the preparations, it was necessary to run an analysis on each salt so that from the per cent purity of the salt, the exact amount of bromide that should be recovered in the preparation could be calculated. All the salts were assayed for bromide by the U. S. P. X method with the following results:

TABLE I.

Salt.	Analyst A. Total Bromide.			Analyst B. Total Bromide.			Ave. A & B.
	1.	2.	Ave.	1.	2.	Ave.	
NH ₄ Br	100.38%	100.14%	100.26%	100.41%	100.36%	100.39%	100.32%
KBr	99.68%	99.64%	99.66%	99.58%	99.48%	99.53%	99.60%
NaBr	101.20%	101.32%	101.26%	101.43%	101.50%	101.47%	101.36%

The high results obtained in the case of ammonium bromide and sodium bromide were due to the presence of small amounts of chloride. Most manufacturers allow a tolerance of 0.6% chloride in C.P. and reagent bromides, some as

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high as 1.5%. Theoretically a sample of sodium bromide containing 0.6% sodium chloride assayed by the U. S. P. X method should assay 100.46% sodium bromide, or if it contained 1.5% sodium chloride it should assay 101.44% sodium bromide.

Elixir of Ammonium Bromide, Elixir of Potassium Bromide and Elixir of Sodium Bromide were carefully prepared with the assayed salts and were assayed by the method given above. The results of analysis are as follows:

TABLE II.

Preparation.	Analyst A.					Analyst B.				
	Total Bromide.		Ave.	Theoretical Gm./- 100 cc.	% error.	Total Bromide.		Ave.	Theoretical Gm./- 100 cc.	% error.
	1. Gm./- 100 cc.	2. Gm./- 100 cc.				1. Gm./- 100 cc.	2. Gm./- 100 cc.			
Elixir KBr	24.891	24.851	24.871	24.900	-0.1	24.802	24.742	24.772	24.900	-0.8
Elixir NaBr	25.216	25.241	25.229	25.340	-0.4	25.148	25.173	25.161	25.340	-0.7
Elixir NH ₄ Br	24.849	24.865	24.857	25.080	-0.9	24.963	24.898	24.931	25.080	-0.6

The average result of both analysts A & B for the salts (see Table I) was used to calculate the theoretical number of Gm. of bromide per 100 cc. In all cases the error was less than 1% for each analyst.

In the proposed formulas for the above elixirs the N. F. allows the dispenser to color the elixir if he so desires by adding 1.5 cc. of compound tincture of cudbear per 100 cc. Accordingly, samples of the three elixirs containing cudbear were assayed and results similar to those above were obtained, all within 1% of error showing that the color does not interfere with the assay method.

(b) Elixir of Three Bromides, Elixir of Five Bromides and Syrup of Five Bromides may all be assayed by the method given below.

Assay Method.—Dilute 10 cc. of the elixir or syrup to 300 cc. Use an aliquot of 50 cc. and continue the assay as in (a) beginning with the words "introduce into an Erlenmeyer flask."

$$\text{Equivalent for Elixir of Three Bromides} = 0.01059 \times \frac{300}{50} \times 10 = 0.6354.$$

$$\text{Equivalent for Elixir of Five Bromides} = 0.01037 \times \frac{300}{50} \times 10 = 0.6222.$$

$$\text{Equivalent for Syrup of Five Bromides} = 0.01058 \times \frac{300}{50} \times 10 = 0.6348.$$

EXPERIMENTAL.

In making the above preparations it was necessary to analyze two more salts. The results of analysis of calcium bromide and lithium bromide are as follows:

TABLE III.

Salt.	Analyst A.			Analyst B.			Ave. A & B.
	1.	Total Bromide. 2.	Ave.	1.	Total Bromide. 2.	Ave.	
CaBr ₂	81.40%	81.34%	81.37%	81.27%	81.47%	81.37%	81.37%
LiBr	85.21%	85.27%	85.24%	85.23%	85.19%	85.21%	85.23%

Elixir of Three Bromides, Elixir of Five Bromides and Syrup of Five Bromides were carefully prepared with the assayed salts, and assayed by the method above. The results of analysis are as follows:

TABLE IV.

Preparation.	Analyst A.					Analyst B.				
	Total Bromide.		Ave.	Theoretical Gm./- 100 cc.	% error.	Total Bromide.		Ave.	Theoretical Gm./- 100 cc.	% error.
	1. Gm./- 100 cc.	2. Gm./- 100 cc.				1. Gm./- 100 cc.	2. Gm./- 100 cc.			
Elixir 3 Br	23.955	23.968	23.962	24.102	-0.6	23.968	23.949	23.959	24.102	-0.6
Elixir 5 Br	24.969	24.932	24.952	24.852	-0.4	24.950	24.925	24.938	24.852	+0.3
Syrup 5 Br	23.629	23.603	23.616	23.809	-0.8	23.603	23.654	23.629	23.809	-0.8

Both analysts obtained results under one per cent of error.

CALCULATIONS.

In calculating the amount of bromide equivalent to one cc. of 0.1N silver nitrate in Elixir of Three Bromides, Elixir of Five Bromides and Syrup of Five Bromides the following general formula was developed and used:

$$F = \frac{a}{\frac{b_1}{c_1} + \frac{b_2}{c_2} + \frac{b_3}{c_3} + \dots}$$

Where F = number of Gm. of mixed bromides equivalent to 1 cc. 0.1N AgNO_3 .

a = total number of Gm. of bromides (based on 100% purity) in mixture.

b_1, b_2, b_3 , etc. = number of Gm. of individual bromides (100% pure) in mixture.

c_1, c_2, c_3 , etc. = number of Gm. of the bromide represented by b_1, b_2, b_3 , etc., equal to 1 cc. N/10 AgNO_3 .

The factor for Elixir of Three Bromides, which contains 80 Gm. of sodium bromide, 80 Gm. of ammonium bromide and 80 Gm. of potassium bromide per liter of solution, is calculated thus:

$$F = \frac{240}{\frac{80}{0.010292} + \frac{80}{0.011902} + \frac{80}{0.009796}} = 0.01059$$

The factor for Syrup of Five Bromides is calculated as follows: The syrup contains 80 Gm. of potassium bromide, 80 Gm. of sodium bromide, 50 Gm. of ammonium bromide, 25 Gm. of calcium bromide and 8 Gm. of lithium bromide per liter of solution. Thus substituting in the formula:

$$F = \frac{237.8}{\frac{80}{0.011902} + \frac{80}{0.010292} + \frac{50}{0.009796} + \frac{25 \times 0.84}{0.009996} + \frac{8 \times 0.85}{0.008686}} = 0.01058$$

According to the U. S. P. X, calcium bromide should contain at least 84% of CaBr_2 , thus we have to multiply 25 Gm. by 84% to get the amount of 100% CaBr_2 present. The same applies to lithium bromide, the N. F. V allows a minimum of 85% LiBr .

The calculations for Elixir of Five Bromides are carried out similar to those above, F being equal to 0.01037.

SUGGESTED STANDARDS.

Elixir of Potassium Bromide, Elixir of Sodium Bromide and Elixir of Ammonium Bromide each should contain not less than 24.5 Gm. nor more than 25.5 Gm. of the respective bromide per 100 cc. of elixir. This allows a tolerance of 2% above and 2% below the amount specified in the formula.

Elixir of Three Bromides should contain not less than 23.4 Gm. nor more than 24.6 Gm. of total bromide per 100 cc. of elixir. This allows a tolerance of 2.5% above and 2.5% below the amount specified in the formula.

Elixir of Five Bromides should contain not less than 24.52 Gm. or more than 25.78 Gm. of total bromide per 100 cc. of elixir. This allows a tolerance of 2.5% above and 2.5% below the amount of pure bromides specified in the formula.

Syrup of Five Bromides should contain not less than 23.19 Gm. nor more than 24.37 Gm. of total bromides per 100 cc. of elixir. This allows a tolerance of 2.5% above and 2.5% below the amount of pure bromides specified in the formula.

CONCLUSION.

Elixir of Potassium Bromide, Elixir of Sodium Bromide, Elixir of Ammonium Bromide, Elixir of Three Bromides, Elixir of Five Bromides and Syrup of Five Bromides can be accurately assayed by the U. S. P. X (Volhard) method for assay of bromides.

It is recommended that the assay methods as given above, with the suggested standards be adopted in the N. F. VI.

DETAILING THE DENTIST FOR HIS PRESCRIPTIONS.*

BY A. O. MICKELSEN.¹

Dentistry has made astounding advancements. It is now a recognized branch of the medical profession, requiring at the present time five years of college training—four years of which must be devoted to the study of subjects relating directly to the profession.

Such subjects as anatomy, histology, bacteriology, pathology, bio-chemistry, surgical anatomy, anesthesia, principles of surgery, clinical medicine, materia medica, pharmacology, physiology and physical diagnosis have given the dentist a good foundation for the diagnosis and treatment of most pathological conditions of the oral cavity. The disturbances of the oral cavity that lie within the province of the clinical practitioner are primarily of an infectious nature, requiring a more or less specific treatment for each individual case. Directed care is necessary as the mouth, throat and nose are the avenues through which pathogenic bacteria gain entrance into the body, causing serious diseases.

Instead of directed medication by the dentist, the patient is instructed to purchase some drug or patent preparation and treat himself. However, the dentist is not solely to blame for this condition; there are numerous preparations on the market prepared especially for the convenience of the dentist and the patient. These are the directions accompanying such a preparation, not mentioning the trade name: Quoting—“*Directions*—to relieve distress and discomfort due to simple headache, head colds, simple neuralgia and muscular aches and pains, also after tooth extraction, and for painful menstruation. In such conditions 1 and 2 tablets should be taken with a half glass of water and repeated 1 tablet in an hour if necessary.” Those are the directions accompanying this sample to aid the

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