3. Langmuir's equation represents the adsorption curves better than does Freundlich's equation.

4. The ability of Charcoals No. 1 and No. 2 to adsorb strychnine sulphate from solution is less when the salt is dissolved in alcohol than when it is dissolved in water.

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THE STABILIZATION OF SOLUTION OF IRON AND AMMONIUM ACETATE, U. S. P. X.[1]

BY WILLIAM J. HUSA[2] AND LYELL J. KLOTZ.

Husa and Birmingham (1) found that the stability of Solution of Iron and Ammonium Acetate is affected by various factors such as light, heat and variations in the proportions of the ingredients. In the present investigation, further experimentation was undertaken with a view toward increasing the stability of Solution of Iron and Ammonium Acetate, commonly known as Basham's Mixture.

Order of Mixing.—In the preparation of colloidal solutions, the order of mixing is often an important factor in the stability of the finished product, particularly when substances capable of acting as protective colloids are present. Birmingham (2) prepared Basham's Mixture by 12 different orders of mixing and stored the resulting preparations under three conditions, *i. e.*, sunlight, darkness and diffused light. Order of mixing appeared to have no effect upon the stability, except in a few cases of diffused light.

Since the tests carried out by Birmingham in diffused light gave some indication that order of mixing might be a factor to consider, further tests were carried out on this point. The materials used were of U. S. P. grade, the ammonium carbonate assaying 31.2% ammonia and the diluted acetic acid being adjusted to 6.0%. Solutions were prepared by two methods: (*a*) hand mixing, shaking well after the addition of each ingredient, and (*b*) mechanical mixing, adding each ingredient drop by drop from a burette into a beaker fitted with an electric stirrer, five minutes being allowed after the final addition of each ingredient and efficient mechanical stirring being maintained throughout the process.

In order to designate the orders of mixing concisely, the following numbers are assigned to the various ingredients:

1. Solution of Ammonium Acetate.
2. Diluted Acetic Acid.
3. Tincture of Ferric Chloride.
4. Aromatic Elixir.
5. Glycerin.
6. Water.

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The orders of mixing were as follows, certain ingredients being omitted in some cases as indicated:

MECHANICAL AGITATION.

1 + 2 + 3 + 4 + 5 + 6; 3 + 5 + 4 + 1 + 2 + 6; 3 + 5 + 4 + 2 + 1 + 6; 3 + 5 + 2 + 1 + 4 + 6; 3 + 5 + 1 + 2 + 4 + 6; 3 + 1 + 2 + 5 + 4 + 6; 1 + 5 + 3 + 2 4 + 6; 1 + 3 + 2 + 5 + 4 + 6; 1 + 2 + 5 + 4 + 3 + 6; 3 + 4 + 1 + 2 + 5 + 6; 3 + 4 + 2 + 1 + 5 + 6; 1 + 3 + 4 + 5 + 6 (2 omitted); 1 + 3 + 5 + 4 + 6.

HAND AGITATION.

1 + 2 + 3 + 4 + 5 + 6; 1 + 3 + 5 + 4 + 6 (2 omitted).

The solutions were stored under various conditions, 25-cc. portions being placed in 1-oz. prescription bottles. The solutions were observed daily for evidences of precipitation.

TABLE I.—THE EFFECT OF VARIOUS CONCENTRATIONS OF DILUTED ACETIC ACID ON THE STABILITY OF SOLUTION IRON AND AMMONIUM ACETATE.

Diluted Acetic Acid.	Time of Ppt., Days.	Diluted Acetic Acid.	Time of Ppt., Days.
0% (*s*)	9	8% (*s*)	19
0%	9	8%	19
2% (*s*)	11	12% (*s*)	51
2%	11	12%	52
4% (*s*)	11	18% (*s*)	*
4%	11	18%	*
6% (*s*)	14		
6%	14		

* Did not precipitate during the test period of 60 days.

Mechanically mixed samples following the U. S. P. X order of mixing precipitated in 22 days in diffused light and in 29 days in darkness; hand-mixed samples precipitated after practically the same interval. The samples prepared by various other orders of mixing precipitated in 13 to 26 days in diffused light and in 18 to 31 days in darkness. Since differences of a few days might be attributed to experimental error and in any case would not be of much practical significance, it may be safely concluded that the variations in order of mixing showed no advantage over the U. S. P. method. It is further evident that mechanical mixing offers no advantage over hand mixing as far as stability is concerned. Samples of all the above solutions were also stored in a refrigerator at about 6° C.; after 18 months, all the samples were still perfectly free from precipitation. It is thus apparent that storage of the solution at refrigerator temperature constitutes an excellent method of avoiding the troublesome deterioration.

Effect of Various Concentrations of Acetic Acid.—Samples of Solution of Iron and Ammonium Acetate were prepared containing 0%, 2%, 4%, 6%, 8%, 12% and 18% of Diluted Acetic Acid in the finished preparation. Two 30-cc. portions of each sample were placed in 4-oz. prescription bottles in diffused light. One sample was shaken daily and the other allowed to remain undisturbed. Table I shows the time of precipitation of these solutions. Samples marked (*s*) were shaken daily.

It was concluded from the data in Table I, that the time of precipitation is inversely proportional to the quantity of acetic acid present, and that daily agitation has no effect upon the time of precipitate formation.

Effect of Addition of Alkali.—Specified quantities of a normal solution of sodium hydroxide were added to samples of Solution of Iron and Ammonium Acetate before diluting the mixture to volume with distilled water. Samples were stored in diffused light. Data are shown in Table II.

TABLE II.—THE EFFECT OF ADDITION OF ALKALI UPON THE STABILITY OF SOLUTION OF IRON AND AMMONIUM ACETATE.

N Alkali in Solution.	Time of Ppt., Days.	N Alkali in Solution.	Time of Ppt., Days.
0.5% (*s*)	7	5.0% (*s*)	9–27*
0.5%	7	5.0%	10–29*
1.0% (*s*)	6	10.0% (*s*)	**
1.0%	6	10.0%	**
2.0% (*s*)	5		
2.0%	7		

* Precipitated at the end of 9 and 10 days, respectively; the precipitate redissolved within a few hours and did not reappear until the expiration of 27 and 29 days, respectively.

** Did not precipitate during the test period of 60 days.

It was concluded from the data in Table II, that additions of alkali in concentrations of 5% or more of normal solution exert a stabilizing influence upon this preparation. This observation is in accord with theory in that it is well known that ferric hydroxide is more readily peptized in neutral or alkaline than in acid media (3).

Effect of Concentration of Ingredients.—Husa and Birmingham (1) have shown that an increase in the concentration of certain ingredients resulted in increased stability of Solution of Iron and Ammonium Acetate. Walter Taylor (4) stated that a double strength preparation is stable, his statement being based upon the U. S. P. formula of 1890. Although the present U. S. P. formula cannot be doubled in strength by the use of the ingredients contained, it is possible to perform the concentration by substituting the proper amount of acetic acid or glacial acetic acid for the required quantity of Diluted Acetic Acid.

In order to determine the stability of a preparation made exactly twice as strong as the present official solution, samples of solution were prepared according to the following formula:

Solution of Iron and Ammonium Acetate.

Tincture of Ferric Chloride	80 cc.
Glacial Acetic Acid	65 cc.
Ammonium Carbonate	50 Gm.
Aromatic Elixir	240 cc.
Glycerin	240 cc.
Distilled Water, *q. s.*	1000 cc.

Mix the glacial acetic acid with 350 cc. of distilled water and to this mixture, gradually add the ammonium carbonate. After effervescence has ceased, add, successively, the tincture of ferric chloride, the aromatic elixir, and the glycerin, and lastly, enough distilled water to make the product measure 1000 cc.

This solution was then stored under the usual conditions in 1-oz. prescription bottles, portions of the solutions also being placed in the electric oven at approximately 50° C. In addition, 8-oz. bottles of the concentrated preparation were stored under the usual conditions and at the end of each month, 1 fl. oz. of the concentrate was diluted with an equal volume of distilled water and stored for comparison with a control sample. Data follow in Table III.

TABLE III.—THE EFFECT OF DOUBLING THE CONCENTRATION OF ALL INGREDIENTS IN SOLUTION OF IRON AND AMMONIUM ACETATE.

	No. of Days before Ppt. Occurred.			
Sample.	Sunlight.	Diffused Light.	Darkness.	Oven.
U. S. P. Control	5	55	60	1
Concentrate	26	No ppt. 6 months	No ppt. 6 months	10

In addition, samples prepared by diluting the concentrate were fully as stable as freshly prepared control specimens, no difference being observed in any case between the times of precipitation.

It was concluded from the data in Table III, that a stable preparation can be prepared by doubling the quantities of active ingredients in Solution of Iron and Ammonium Acetate.

SUMMARY.

1. The deterioration of Basham's Mixture may be avoided by storing the solution at refrigerator temperature.

2. Basham's Mixture is very stable when prepared double strength; this concentrated preparation may be diluted to double its volume with distilled water just before it is dispensed.

3. The stability of Basham's Mixture may be improved by increasing the proportion of acetic acid or by the addition of alkali, but since these changes would alter the product somewhat, they are not considered as practical as the methods listed under 1 and 2.

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ANALYSIS OF MAGNESIUM CARBONATE.*

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Samples of Magnesium Carbonate were obtained from eight sources. These were subjected to routine analysis to discover how closely they compared, particularly as to MgO content and to find out how wide a range existed. Also to

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