



The effect of hydrogen ion concentration and ionic strength on suspensibility of commercial acid lead arsenate  
by Louis A Spain

A Thesis Submitted to the Graduate Committee in partial fulfillment of the requirements for the Degree of Master of Science In Entomology  
Montana State University  
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SUSPENSIBILITY OF COMMERCIAL ACID LEAD ARSENATE

by

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Submitted to the Graduate Committee in partial  
fulfillment of the requirements for the  
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THE EFFECT OF HYDROGEN ION CONCENTRATION AND IONIC STRENGTH ON  
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INTRODUCTION

It is a long established fact that a good suspension of any spray material is highly desirable to insure uniform coverage and distribution on foliage. There is a large number of spreading materials that have been used with lead arsenates to obtain this result. Another factor which enters into this problem is the fact that lead arsenate is often used in spray mixtures with other spray materials such as Bordeaux mixture, nicotine sulphate, soaps, etc. A third factor is the great variability of the waters used in making sprays. This results in lead arsenate being subjected to a wide variety of conditions due to the great difference in salts and salt concentrations present.

It has been recognized that the quality of the spray, with respect to spreading, adhesiveness and settling, varies when used under different conditions. It has been assumed that these differences are due to various factors such as acidity, alkalinity, salt concentration, etc., in the spray mixture.

Preliminary studies at the Montana Experiment Station indicate that hydrogen ion concentration is one of the factors affecting suspensibility of lead arsenate. Although no attempt has been made to determine the effect of varying salt concentration, it is assumed that this is also a factor in suspensibility.

Due to cloudiness of the mixture when a good suspension of lead

arsenate is obtained, colorimetric determinations of pH are inaccurate and unsatisfactory. When either hydrogen or quinhydrone electrodes are used to determine the pH of a lead arsenate suspension, the soluble arsenic ( $\text{As}_2\text{O}_5$ ) causes a poisoning of the electrode metals which introduces large errors in the determinations made. After an electrode has been used for two or three determinations it is impossible to obtain further readings. This handicap in measuring accurately the pH of lead arsenate suspensions has now been overcome by the use of the glass electrode which is not affected by soluble arsenic.

In some of the previous work on the effects of hydrogen ion concentration on acid lead arsenate, hydrochloric acid and ammonium hydroxide were used to vary pH. Since hydrochloric acid is used to dissolve lead arsenate and ammonium hydroxide is used in the manufacture of basic lead arsenate from the acid form, these reagents were not used. Sorensen's phosphate buffer was considered more desirable for adjusting and holding any desired pH. Potassium chloride was used to adjust the ionic strength.

In the preliminary work on this problem much trouble was experienced in obtaining checks on the rate of settling for a given lead arsenate. Density determinations showed that there was quite a wide variation not only between different brands but also in samples from a single brand. These variations were thought to be due to either the difference in methods of manufacture of lead arsenate or to the 1% inert material which is present in most of the brands. The results embodied in this paper were obtained by the study of a brand of lead arsenate which was considered to be relatively consistent as judged from density determinations.

The problem discussed in this paper had for its objectives:

- (1) The determination of the effect of varying hydrogen ion concentration at constant ionic strength, and;
- (2) The determination of the effect of varying ionic strength at constant pH on the suspensibility of a selected sample of commercial lead arsenate.

#### REVIEW OF PREVIOUS WORK

Hengel and Reckendorfer (2) tested a number of methods and types of apparatus for the determination of fineness and suspensibility of particles. They found that the best results were obtained with a "revolver" apparatus devised by Hengel for use in the mechanical analysis of soils. This apparatus consists of a 120 cm. glass tube of 20 mm. bore, the lower end of which gradually narrows in the last 8 cm. to terminate with a bore of 6 mm. The upper end of the tube is ground and closed by a pear-shaped flask 10 cm. high and 6 cm. in the greatest inner diameter. In use, the tube is fixed in an upright position in a stand at the bottom of which is arranged a turntable carrying a series of small glass dishes, and so contrived that it can be quickly rotated to bring the dishes in succession under the tip of the tube, just as the successive chambers in a revolver cylinder are aligned with the barrel. The Schweinfurt green or other material to be tested for suspensibility is placed in the flask, which is filled to the neck with water and fitted over the previously filled tube. Then with simultaneous release of a stop watch, the whole is inverted and fixed in the stand so that the tapered end of the tube is immersed in one of the dishes, which were previously filled with water. The sediment settles through the column of water and collects in the

dish. After a desired interval, another dish is quickly shifted under the tube, allowed to remain for the determined interval and the process repeated for as long as desired.

According to Robinson (4), chemical analysis of commercial brands of lead arsenate, of which more than 95% are the acid lead arsenate form ( $\text{PbHAsO}_4$ ) shows very little variation between the brands. Such variation as does occur is due mainly to the presence of small amounts of basic lead arsenate ( $\text{Pb}_3(\text{AsO}_4)_2$ ). Examination of the various commercial brands indicates that they differ widely in their physical properties. Some are finely ground, fluffy and bulky; others are coarse grained and compact. The density of a sample was determined by ascertaining the weight of toluene displaced by a known quantity of the lead arsenate. Care was taken to remove occluded air by placing the pycnometer in a dessicator and exhausting it. The density of twelve brands tested varied only from 5.69 to 6.01 with apparently no correlation between the specific gravity and the dry volume, as some brands occupy more than twice the dry volume of certain other brands. It was shown that one brand of lead arsenate will remain in suspension in water longer than another and to show the actual amounts that remain in suspension after five minutes, two methods were employed:

(1) Graduated glass oil tubes containing 1 gm. of the arsenate and 100 cc. of water, were thoroughly shaken and allowed to stand five minutes. The particles which remained in suspension were siphoned off with the supernatant liquid, evaporated to dryness at  $100^\circ\text{C}$ ., and weighed. The amount that settled was calculated by difference.

(2) The second method was more rapid but less accurate. A 1 gm. sample was introduced into the graduated oil tube containing 100 cc. of water.



After shaking thoroughly, the tube was centrifuged for one minute at 1200 r.p.m. and the volume of the settled arsenate was noted. The tube was again shaken, allowed to stand five minutes, and the supernatant liquid and particles in suspension siphoned off. The tube was again centrifuged and a second reading taken of the settled arsenate. From the two readings the percentages of lead arsenate which settled and which remained in suspension were calculated. The results indicate a perceptible difference in the various brands of lead arsenate with respect to settling. Since only the very fine particles remain in suspension, the percentages reflect to a certain extent the relative amounts of large and small particles.

Beeman (1) used a Becker "Chainomatic" balance fitted with a plummet suitable for measuring densities with a fairly high degree of accuracy, for determining the rate of change in density of clay suspensions during sedimentation. As a result of the investigation of several suspensions in solutions of different hydrogen-ion concentration, he found that small quantities of hydrochloric acid were effective flocculating agents, the maximum effect being produced by N/100, N/200 and N/1000 hydrochloric acid, while corresponding concentrations of sodium hydroxide were without effect. N/10 sodium hydroxide did produce flocculation, but the effect was less than that produced by N/2000 hydrochloric acid. Stoke's law was assumed to hold under the conditions of the experiments and the increased rate of sedimentation was interpreted as an increase in particle size. In effect, the addition of an acid to a clay suspension usually causes rapid sedimentation, while the addition of a base was without effect. These experiments showed that the effect of hydrogen-ion concentration is relatively great and explains why changes in the pH values

produce such noticeable changes in plasticity and viscosity. They show also that elutriation is an uncertain process without pH control.

#### EXPERIMENTAL METHODS AND APPARATUS

To determine the rate of settling of lead arsenate, a modification of the Hengel-Reckendorfer apparatus was used. This apparatus consists of a 120 cm. glass tube of 30 mm. bore, the lower end of which gradually tapers in the last 6 cm. The diameter of the tapered end is 10 mm. and the capacity of the tube is 750 cc. The upper end of the tube is closed with a rubber stopper. The tube is fixed in a vertical position in a stand at the bottom of which is a small shelf on which are placed the sedimentation dishes. This shelf is so constructed that it can be lowered against a spring to permit of the substitution of successive dishes as required, the spring pushing the shelf with the substituted dish into the proper position. The glass dishes are 4 cm. in diameter and 1 cm. in depth.

Five hundred cc. of buffer of the desired pH and ionic strength and 1 gm. of lead arsenate were placed in the tube and mixed for three minutes by alternate inversions of the tube. More buffer was added in three installments with mixing until the tube was completely filled at the conclusion of a ten minute mixing period. Then, with simultaneous release of a stop watch, the tube was fixed in the stand so that the tapered end of the tube was immersed in one of the dishes which was filled with 8 cc. of the same buffer as used in the tube. After intervals of two to five minutes, successive dishes were shifted under the tip of the tube until over half of the lead arsenate had settled out. The dishes were dried to constant weight

in an oven at 95°C. and weighed to determine the amount of lead arsenate settled in each. Several dishes containing only buffer were dried each time to correct for the weight of salt in the buffer.

Several commercial brands of acid lead arsenate tested, without added defluoculent, were found to be not only inconsistent between brands but inconsistent within samples of the same single brand. The figures herein contained are based on a standard commercial brand of acid lead arsenate, referred to as simply lead arsenate, containing 0.5% water soluble arsenic and 2% inert ingredients and found to be relatively uniform by density determinations.

Stock solutions of 0.0022 M secondary sodium phosphate (0.7963 gm.  $\text{Na}_2\text{HPO}_4 \cdot 12\text{H}_2\text{O}$  per liter) and 0.0022 M primary potassium phosphate (0.3026 gm.  $\text{KH}_2\text{PO}_4$  per liter) were prepared. These were used in proportions as given by Sorensen (5) to obtain a desired pH. Ionic strength was maintained in buffers of varying pH and varied in buffers of constant pH by addition of potassium chloride. Ionic strength ( $\mu$ ) was calculated according to Lewis and Randall (3) by multiplying the concentration of each ion (in gm. molecular weights per 1000 gms. of solvent) by the square of its valence; all these quantities are added together and divided by two. The factor two is included because both positive and negative ions are involved. An example of a sample calculation is shown herewith for a buffer of pH value of 6.0 and a desired ionic strength of  $6.66 \times 10^{-3}$ . The buffer was made by mixing 123 cc. of 0.0022 M sodium phosphate with 877 cc. of 0.0022 M potassium phosphate and adding 0.2930 gm. of KCl. The amount of KCl added was calculated as follows:

$$\text{Conc. of Na}^+ = \frac{123}{1000} \times 0.0022 \times 2 = 0.0005412$$

$$\text{Conc. of } \text{HPO}_4^- = \frac{123}{1000} \times 0.0022 = 0.0002706$$

$$\text{Conc. of } \text{K}^+ = \frac{877}{1000} \times 0.0022 = 0.00192$$

$$\text{Conc. of } \text{H}_2\text{PO}_4^- = \frac{877}{1000} \times 0.0022 = 0.00192$$

$$u = \frac{0.0005412 \times 1^2 + 0.0002706 \times 2^2 + 0.00192 \times 1^2 + 0.00192 \times 1^2}{2} = 2.73 \times 10^{-3}$$

$$(6.66 \times 10^{-3}) - (2.73 \times 10^{-3}) = 3.93 \times 10^{-3}$$

$$(3.93 \times 10^{-3}) \times 74.56 = 0.2930 \text{ gms. of KCl per liter of buffer}$$

which must be added to obtain an ionic strength of  $6.66 \times 10^{-3}$ .

The pH of a liter of buffer when once made up could not be adjusted by adding sodium or potassium phosphate as this would alter the ionic strength. Therefore, the buffers were made up as nearly as possible to the desired pH and the hydrogen ion concentration measured.

All measurements of hydrogen ion concentration were made by means of a glass electrode with a potentiometer and a Compton quadrant electrometer. A galvanometer was used to obtain the approximate reading and the electrometer to obtain the final balance. The pH of the lead arsenate suspension of each buffer was measured at the time when one half of the lead arsenate had settled out of the sedimentation tube. This time was obtained from graphs on which the per cent of the lead arsenate settled was plotted against time.

The density of the samples was determined by ascertaining the volume of a known weight of the lead arsenate in toluene. Care was taken to remove occluded air by placing the specific gravity bottles filled with lead arsenate and toluene in a chamber from which the air was exhausted. A constant temperature water bath at 25°C. was used in all density determinations.

The average size of the particles of lead arsenate in each suspension

was determined by use of Stoke's law for the settling of particles. A sample calculation of particle size for a buffer with a pH value of 6.8 and an ionic strength of  $4.39 \times 10^{-3}$  is shown herewith in which the time for half the sample to settle was 354 seconds, and the average distance each particle settled was 59.25 cm. (one-half the height of the tube):

$$\text{Stoke's law: } V = 2 g R^2 \frac{(d_1 - d_2)}{9 N}$$

V = velocity of settling =  $59.25 \div 354 = 0.167$  cm. per sec.

g = gravitational constant for latitude and elevation of Bozeman which is 980.065

R = radius of particle in centimeters

$d_1$  = density of the particle = 5.8916

$d_2$  = density of the medium = 0.9974

N = viscosity of the medium = 0.00894

$$0.167 = 2 \times 980.065 \times R^2 \frac{(5.8916 - 0.9974)}{9 \times 0.00894}$$

R = 0.001183 cm. = 11.83 microns

The diameter is equal to 23.66 microns which is the average size of the particles settled out during the time for half the sample to settle in a buffer with a pH value of 6.8 and an ionic strength of  $4.39 \times 10^{-3}$ .

#### DATA AND RESULTS

Table I shows the results of settling experiments for lead arsenate in phosphate buffer solutions of various pH values with ionic strength kept constant at  $6.66 \times 10^{-3}$ . There was a flocculation of the lead arsenate particles in all the buffers, but the data show that there are considerable

differences in the behavior of the lead arsenate in buffers of different pH. Decreasing the pH from 7.72 to 7.09 decreased the rate of settling, while decreasing the pH from 7.09 to 4.64 increased the rate of settling. Figure 1 shows three typical curves in which the per cent of the sample settled is plotted against the time in minutes. From these curves, the time for half the sample to settle was obtained.

TABLE I.

pH	Ionic strength	Time	% Settled
7.72	$6.66 \times 10^{-3}$	3 min.	16.21
		5	42.30
		7	64.20
		10	73.41
		15	77.96
		7.45	$6.66 \times 10^{-3}$
5	39.65		
7	60.68		
10	70.39		
15	75.56		
7.09	$6.66 \times 10^{-3}$		
		5	36.50
		7	57.79
		10	70.03
		15	75.73
		6.67	$6.66 \times 10^{-3}$
5	44.27		
7	68.00		
10	78.71		
15	82.90		
5.93	$6.66 \times 10^{-3}$		
		5	41.20
		7	64.88
		10	78.89
		15	83.49
		4.64	$6.66 \times 10^{-3}$
5	42.64		
7	67.29		
10	80.77		
15	84.54		

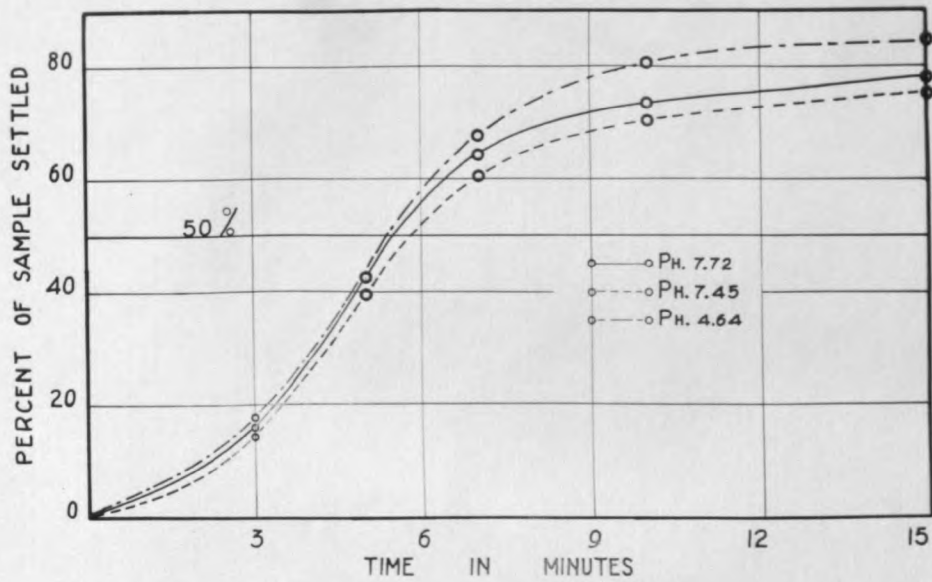


Fig.1. Per cent settled plotted against time for pH values of 7.72, 7.45, 4.64, keeping ionic strength at a constant value of  $6.66 \times 10^{-3}$ .

Table II shows the pH values in terms of hydrogen ion concentration and the time in minutes for one-half the lead arsenate sample to settle from the buffers of various pH and constant ionic strength. Fig. 2 shows graphically the time for half the sample to settle plotted against hydrogen ion concentration, showing that as the hydrogen ion concentration is increased from  $1.91$  to  $8.13 \times 10^{-8}$ , the time increases for half the sample to settle, and as the hydrogen ion concentration is increased from  $8.13$  to  $2290 \times 10^{-8}$ , the time decreases for half the sample to settle. Table II also shows the particle sizes of the lead arsenate calculated from the data obtained in the settling experiments using buffers of different hydrogen ion concentration. The assumption was made that Stoke's law holds for the conditions of these experiments and that flocculation is merely the aggregation of smaller to form larger particles under the influence of the environment. Fig. 3, in which particle size is plotted against hydrogen ion concentration, shows that as the latter is increased from  $1.91$  to  $8.13 \times 10^{-8}$ , the size of the particles decreases, and as the hydrogen ion concentration is increased from  $8.13$  to  $2290 \times 10^{-8}$ , there is an increase in the size of the particles.

TABLE II.

pH	H. ion conc.	Ionic strength	Time for half sample to settle in minutes	Diameter of particles in microns
7.72	$1.91 \times 10^{-8}$	$6.66 \times 10^{-3}$	5.55	24.4
7.45	3.55 "	"	5.80	23.9
7.09	8.13 "	"	6.13	23.2
6.67	21.4 "	"	5.32	24.94
5.93	117.5 "	"	5.60	24.32
4.64	2290 "	"	5.45	24.64

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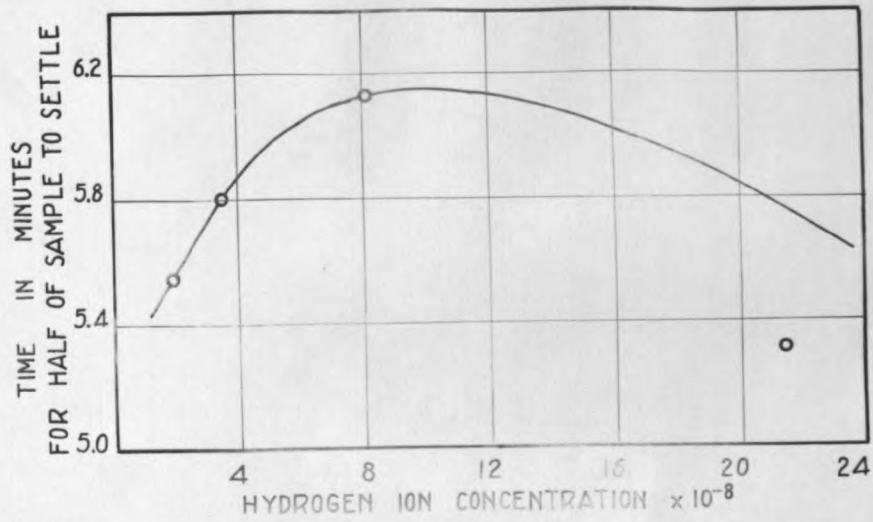


Fig.2. Time for half of the sample to settle plotted against hydrogen ion concentration keeping ionic strength at a constant value of  $6.66 \times 10^{-3}$ .

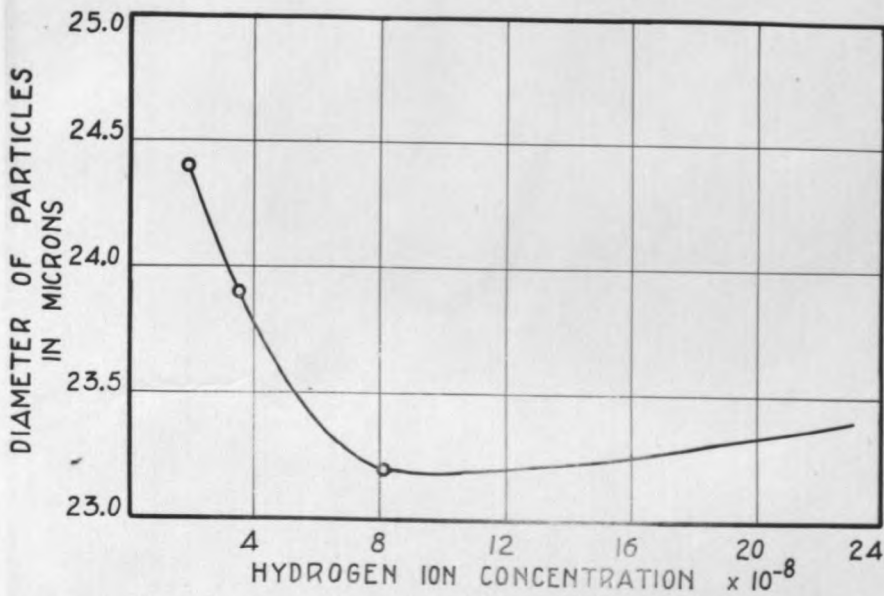


Fig.3. Particle size plotted against hydrogen ion concentration keeping ionic strength at a constant value of  $6.66 \times 10^{-3}$ .

Table III shows the results of settling experiments for lead arsenate in phosphate buffer solutions of varying ionic strength with pH kept constant at a value of 6.8. Here again, the lead arsenate flocculated in the buffer but there is a striking difference in the rate at which it settled at different ionic strengths. As the ionic strength was increased from  $4.39$  to  $25.0 \times 10^{-3}$  the rate of settling decreased, and as the ionic strength was increased from  $25.0$  to  $200 \times 10^{-3}$ , the rate of settling increased. Fig. 4 shows three curves of the per cent of the lead arsenate settled plotted against time. These curves were used to obtain the time for half the sample to settle.

TABLE III

pH	Ionic strength	Time	% Settled
6.8	$4.39 \times 10^{-3}$	3 min.	12.19
		5	36.29
		7	60.96
		11	75.61
		15	79.19
6.8	$7.69 \times 10^{-3}$	3	10.06
		5	26.87
		7	46.25
		10	64.64
		15	73.58
6.8	$10 \times 10^{-3}$	3	6.01
		5	21.95
		7	41.31
		10	62.57
		15	71.12
6.8	$14.3 \times 10^{-3}$	3	4.49
		5	16.72
		7	33.15
		10	56.11
		15	68.67

TABLE III, Cont'd.

<u>pH</u>	<u>Ionic strength</u>	<u>Time</u>	<u>% Settled</u>
6.8	$25.0 \times 10^{-3}$	3	2.64
		5	10.95
		7	25.99
		10	50.87
		15	68.54
6.8	$100 \times 10^{-3}$	3	3.29
		5	11.77
		7	27.79
		10	52.64
		15	69.92
6.8	$200 \times 10^{-3}$	3	3.86
		5	12.38
		7	28.53
		10	53.66
		15	68.58

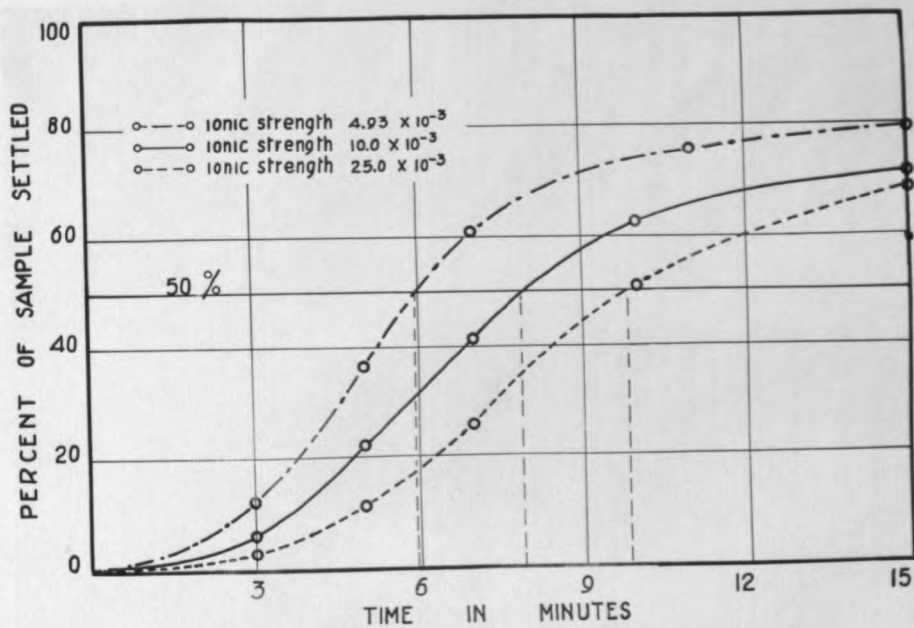


Fig.4. Per cent of the sample settled plotted against time for buffers of ionic strength values of  $4.39$ ,  $10.0$  and  $25.0 \times 10^{-3}$ , keeping the pH equal to  $6.8$ .

Table IV shows the time for half the sample of lead arsenate to settle and the ionic strength of the various buffers used at constant pH. From this table it can be seen that the time for half the sample to settle increased as the ionic strength was increased from 4.39 to  $25.0 \times 10^{-3}$ , and decreased as the ionic strength was increased from 25.0 to  $200 \times 10^{-3}$ . Fig. 5, showing time for half the sample to settle plotted against ionic strength, graphically illustrates this effect of increasing the ionic strength from 4.39 to  $25.0 \times 10^{-3}$ . Table IV also shows the variation of particle size as calculated by Stoke's law for buffer solutions of different ionic strength. As the ionic strength was increased from 4.39 to  $25.0 \times 10^{-3}$ , the size of the particles decreased, and as the ionic strength was increased from 25.0 to  $200 \times 10^{-3}$ , the size of the particles increased. Fig. 6 illustrates this relationship of particle size to ionic strength as the latter was increased from 4.39 to  $25.0 \times 10^{-3}$ .

TABLE IV.

pH	H. ion conc.	Ionic strength	Time for half sample to settle in minutes	Diameter of particles in microns
6.8	$15.8 \times 10^{-8}$	$4.39 \times 10^{-3}$	5.9	23.66
"	"	7.69	7.3	21.30
"	"	10.0	7.8	20.60
"	"	14.3	8.7	19.56
"	"	25.0	9.8	18.38
"	"	100	9.5	18.70
"	"	200	9.3	18.86

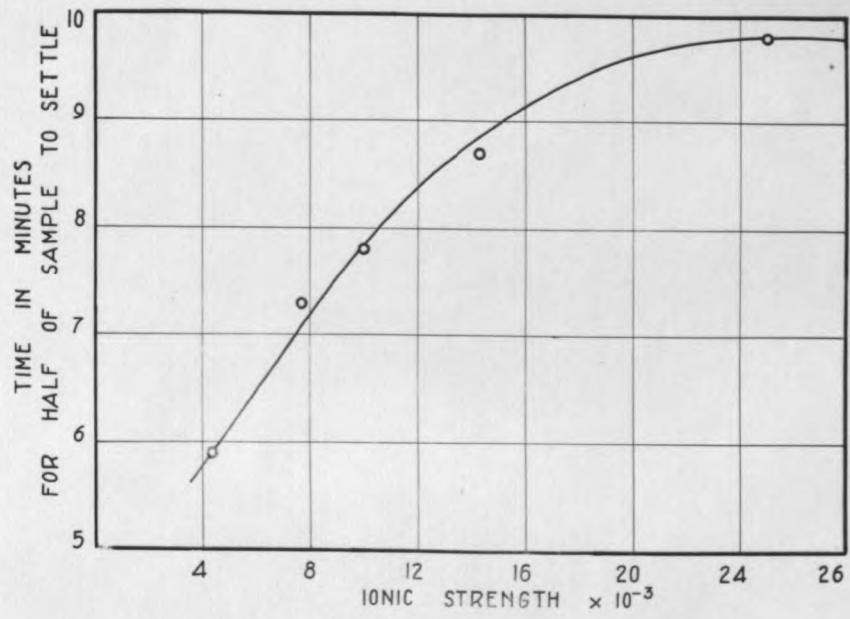


Fig.5. Time for half of the sample to settle plotted against ionic strength, keeping the pH equal to 6.8.

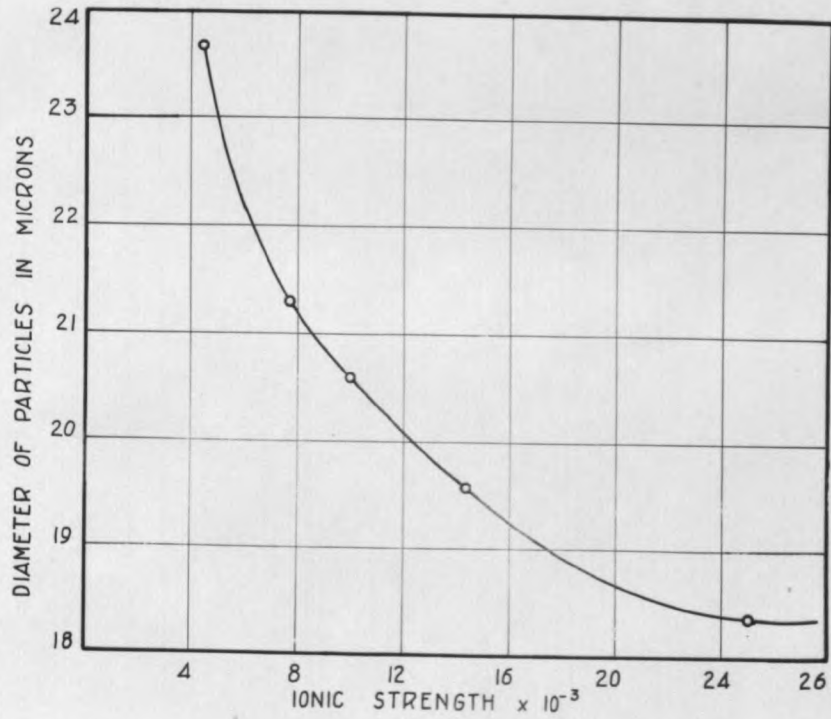


Fig.6. Particle size plotted against ionic strength, keeping the pH equal to 6.8.



DISCUSSION OF RESULTS

The settling tests were run in duplicate and found to check within the range of experimental error for the brand of lead arsenate used in these experiments. With some brands of lead arsenate which varied considerably in density, accurate checks could not be obtained.

In determining the density of lead arsenate, the density of toluene was first arrived at. The results of five determinations showed a variation of only 0.01% for the density of toluene, indicating that the method was satisfactorily accurate. The lead arsenate used was found to have an average density of 5.8916, the greatest variation from the mean being 0.08%. For another brand of lead arsenate tested, the average density was 5.8430, the greatest deviation from the mean being 1.0%.

The commercial lead arsenate used in these experiments was flocculated in every case and thus the effects of pH and ionic strength were minimized to the fullest extent. If in some cases a fine suspension had been attained, there would be shown a much greater contrast for different pH and ionic strength values.

However, it can be readily seen from fig.2 that hydrogen ion concentration has considerable effect on the settling of the lead arsenate. It also shows the optimum hydrogen ion concentration for suspensibility at the ionic strength used ( $6.66 \times 10^{-3}$ ). Fig. 4 and fig. 5 illustrate the striking effect of ionic strength on the suspensibility of the lead arsenate. Table V shows the ionic strength which gave the best suspension for the pH involved (6.8).

Another commercial brand of acid lead arsenate tested and found to

be rather inconsistent, showed exactly these same trends. That is, as the hydrogen ion concentration was increased there was first a decrease and then an increase in the rate of settling. The same trend was observed for changes in ionic strength in that the rate of settling decreased and later increased as the ionic strength was increased. As might be expected, the pH and ionic strength for optimum suspensibility were somewhat different for this brand of lead arsenate. In the case of this particular brand for which there are no data recorded, some of the buffers produced an excellent suspension and the effects of hydrogen ion concentration and ionic strength were much greater. Because samples of this brand were most inconsistent, checks on the settling could not be made and the data were not recorded.

Fig. 3 and fig. 6 show that both hydrogen ion concentration and ionic strength have a profound effect on particle size.

#### SUMMARY AND CONCLUSIONS


1. Experimental evidence shows that hydrogen ion concentration affects the suspensibility of lead arsenate.
2. It also shows that ionic strength has a decided effect on suspensibility.
3. The results of this paper provide further evidence that particle size is directly proportional to the rate of settling.
4. Assuming that large particles are formed by coalescence of smaller particles, it is evident that the surface covered by a given amount of lead arsenate is largely dependent on the hydrogen ion concentration and ionic strength of the medium.

5. The variation of spray waters due to salt concentration is expected to be much greater than the variation due to hydrogen ion concentration. Therefore, it might be concluded that ionic strength has more influence on suspensibility than hydrogen ion concentration.

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