A STUDY OF THE ELECTROLYTIC METHODS FOR DETERMINING LEAD IN BAKING POWDERS.

THESIS FOR DEGREE OF M. S.

ARNOLD AWOTIN

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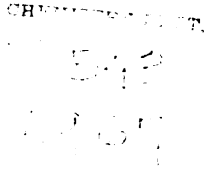

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Table of Contents.

1. Introduction and purpose.
2. Literature review.
3. Methods.
4. Separation of lead from metals electrolytically.
5. Baking powders.
6. A Comparative Study of Some Different Methods.
    a. Deposition of lead in nitric acid solution.
    b. Deposition of lead in hydrolyzed starch solution.
    c. Deposition of lead in sucrose solution.
    d. Incineration of baking powder.
    e. Modification of the Corper Method.
    f. Modification of the Wichmann Method.
7. General discussion.
8. Summary.
9. Literature cited.

Introduction and Purpose.

Baking powders have no food value in themselves but are substances which by chemical reactions bring about, under certain conditions, the comparatively quick liberation of gas and the consequent aeration of biscuit, bread, and cake. No substance that comes within the domain of food inspection is the subject of so much controversy as baking powder. Unless a law forbids the use of a particular ingredient or class of ingredients, or in some manner regulates the labelling of the package, no baking powder of any kind can be considered adulterated under the food law, unless it can be proved to be injurious to health, or unless it contains inert and useless mineral matter.

The maximum amount of lead permissible in a baking powder is 20 parts per million, because the lead salts are injurious to health. The intention of the author is to find some accurate electrolytic method for determining lead in baking powders. This method should be of commercial use.

Literature Review.

Very little has been done in the determination of lead in baking powders. The Bureau of Chemistry, Washington, D.C., started this problem in 1915. Lead was determined quantitatively by gravimetric method in comparatively simple solutions and the results were satisfactory. In 1916, the Referee on Baking Powders of Bureau of Chemistry continued the study of the determination of lead in baking powders. The samples consisted of

a synthetic baking powder mixed from ingredients in which the lead contents had been previously determined. Each sample weighed 100 grams and consisted of 56 gm. mono-calcium phosphate, 25.5 grams of sodium bi-carbonate, and 18.5 grams starch. The lead content of the baking powder, calculated from the determinations on the individual ingredients as made by Mr. Secker and Mr. Chittick, was 7 parts per million, or 0.0007 grams in each 100 gram sample.

The methods used in the collaboration were the Exner and the Wichmann methods. The following results were obtained by different collaborators:

Collaborator and (Analyst)	Exner Method		Wichmann Method	
	Mg. of Lead per 100 g.	: Parts per : million	Mg. of Lead per 100 g.	: Parts per : million
R. E. Stallings	2.0	20	1.4	14
	3.1	31	0.8	8
(J. O. Clarke)			0.6	6
			0.4	4
			0.3	3
C. B. Morey	0.0	0	0.0	0.0
(L. F. Hoyt)	0.0	0	0.0	0.0
T. J. Bryan	1.2	12	0.5	5
(J. R. Davies)	1.4	14	1.0	10
R. E. Doolittle	1.8	18	0.4	4
(A. L. Burns)	1.0	10	1.2	12
E. R. Lyman	0.0	0	0.0	0
	0.0		0.0	0

From an examination of the above table it will be seen that the results vary greatly, not only as a whole, but in duplicate samples determined by the same analyst. The results with the Exner method run about twice as high as those with the Wichmann method and about twice the actual lead content. The average of

the results with the Wichmann method agrees fairly well with the actual content of lead in the sample but due to wide variations in individual results the average cannot be considered of much importance. The time consumed varies greatly, that of Exner method is from two to three times that of Wichmann method.

The following table shows the inaccurate results which were obtained in the determination of lead in baking powders by Seeker-Clayton, Remington, and Exner methods:

Solutions contained 400 mg. of lead per liter.

Method	Lead in milligrams per liter of solution		
	Solution A	B	C
Seeker-Clayton	448	320	384
	704	192	194
Remington	410	179	102
	397	38	140
Exner	137	121	313
	281	294	

Solutions contained 20 parts of lead per million.

Method	Solution A	B	C
Seeker-Clayton	35	16	19
	22	10	10
Remington	22	9	5
	20	2	7
Exner	7	6	16
	14	15	

The Remington modification method shows a much closer agreement of results than the Seeker-Clayton. With this method the larger weight of sample taken (100 grams) reduces the error in weighing. Still closer agreement is found in the results with the Exner method.

The deposition of lead in baking powder by electrolysis offers a means of shortening the determination. Dr. Bryan has submitted a modification of the Corper method for electrolytic determination of lead which he has found very satisfactory when applied to phosphate baking powders. It was recommended that this method be studied during 1916 and 1917.

A few experiments were made by Dr. Patten on both clear solutions containing known quantities of lead, and upon solutions of baking powders themselves. In clear synthetic solutions by depositing lead peroxide from nitric acid solution upon the anode no deposit was obtained from lower quantities than 24 parts per million, with 24 parts per million in solution, 60% of total lead was deposited after 18 hours electrolysis with a $C.D_{100}=1.8$ A. From dilute phosphoric acid solution, containing 50 parts per million of lead, 95% of the total amount was deposited over night with a $C.D_{100}=0.2$ A.

By adding dilute phosphoric acid to baking powder a pasty mixture was obtained, from which with varying currents lead was not deposited. Upon addition of a small amount of nitric acid which partly hydrolyzed the starch, a deposit was obtained. This was so crusted over with starch and salts that it could not be handled. When baking powder was completely neutralized with nitric acid and the starch hydrolyzed until a clear solution was obtained, no deposit was secured upon the anode with prolonged electrolysis and varying the current density.

Further suggestions were made by Dr. Fiske. He states that the temperature of the solution during electrolysis should not exceed 60° C, and the time of electrolysis should exceed 7

hours, 14 to 15 hours used preferably. To eliminate starch, Dr. Fiske prefers the Catlin method, in which the baking powder is ignited with a strong solution of magnesium nitrate and nitric acid instead of the hydrolysis with hydrochloric acid.

Methods.

The gravimetric methods for determination of lead in baking powders are found in "Journal of Association of Official Agricultural Chemists", Feb. 1917. The methods are as follows:

1. Seeker-Clayton Method.
2. Remington Modification of the Seeker-Clayton Method.
3. Exner Method.
4. Wichmann Method.

Separation of Lead from Metals Electrolytically.

The precipitation of lead as dioxide upon anode affords a good method by which to separate it from other metals.

The electrolytic separation of lead is easily obtained from the following metals: Barium, Strontium, Calcium, Magnesium, the alkali metals, Beryllium, Cadmium, Chromium, Iron, Uranium, Zirconium, Zinc, Nickel, and Cobalt. In this case the solution should be made strongly acid, by adding 20 c.c. of concentrated nitric acid, sp. gr. 1.35 - 1.38, to 100 c.c. of solution, with amperage of 1.5 to 1.7, and voltage of 2.30 - 2.5. Chlorides must be absent.

Separation of Lead from Copper.- This separation is made in the presence of free nitric acid.

Separation of Lead from mercury.- By electrolyzing a solution of these two metals in 20% nitric acid solution the separation may be obtained.

Separation of Lead from Aluminum.- The separation may be made by using 20% nitric acid solution, because Aluminum is not precipitated electrolytically from a nitric acid solution.

Separation of Lead from Arsenic.- The electrolytic separation of these two metals is not known.

Separation of Lead from Antimony.- A purely electrolytic separation is at present not known. This separation may be obtained so: Dissolve Lead and Antimony in nitric acid (4 c.c.), water 15 c.c., 10 grams tartaric acid, add sulphuric acid (4 c.c.), dilute to 250 c.c. On filtering from the lead sulphate, which has separated, the filtrate will contain all the Antimony. The lead sulphate should be digested for a few minutes with ammonia water. Add to the lead hydroxide nitric acid and electrolyze.

Separation of Lead from Tin.- The gravimetric separation of the two metals is preferable.

Lead may be obtained as metal on cathode by electrolyzing solutions of double oxalates, the acetate, the oxide in sodium hydroxide, or the phosphate dissolved in the latter reagent or in phosphoric acid of 1.7 sp. gr. While the metal separates well from either of those solutions, difficulty is experienced in drying the deposit, the most metal almost invariably suffers a partial oxidation, thus rendering the results high. It may be dried in atmosphere of hydrogen, but this is expensive and tedious.

Phosphoric acid, mercury, Arsenic, Chlorides, and Manganese interfere with the deposition of lead electrolytically, these

metals should be removed gravimetrically before electrolyzing lead solution.

Classes of Baking Powder.

Baking powders are divided naturally into three main classes, with reference to the acid principle:

1. Tartrate powders, wherein the acid principle is bitartrate of potassium or tartaric acid, typified by the following reactions:

(a) $KHC_4H_4O_6 + NaHCO_3 = KNaC_4H_4O_6 + CO_2 + H_2O$

(b) $H_2C_4H_4O_6 + 2NaHCO_3 = Na_2C_4H_4O_6 \cdot 2H_2O + 2CO_2$

In tartrate powders available carbon dioxide is about 12.6%.

2. Phosphate powders, in which calcium acid phosphate is the acid principle:

$$CaH_4(PO_4)_2 + 2NaHCO_3 = CaHPO_4 + Na_2HPO_4 + 2CO_2 + 2H_2O$$

This baking powder contains about 13% available carbon dioxide.

3. Alum powders, wherein the acidity is due wholly or in part to sulphate of aluminum as it occurs in potash or ammonia alum, or in the double sulphate of aluminum and sodium.

The reaction should be as follows:

$$K_2Al_2(SO_4)_4 + 6NaHCO_3 = 2Al(OH)_3 + 3Na_2SO_4 + K_2SO_4 + 6CO_2$$

Available carbon dioxide is about 8%.

Naturally many baking powders of complex composition are met with, embodying various mixtures of the above classes.

Composition of Various Baking Powders.

Following are analyses of typical baking powders of the above classes:

Cream of Tartar Baking Powder:

Total carbon dioxide, CO_2	13.21
Sodium oxide, Na_2O	13.28
Potassium oxide, K_2O	14.93
Calcium oxide, CaO	0.18
Tartaric acid, $C_4H_4O_5$	41.60
Sulphuric acid, SO_3	0.42
Starch	7.42
Water of combination and association by difference	8.98
	100.00

Phosphate Baking Powder:

Total carbon dioxide, CO_2	13.47
Sodium oxide, Na_2O	12.66
Potassium oxide, K_2O	0.31
Calcium oxide, CaO	10.27
Phosphoric acid, P_2O_5	21.83
Starch	26.41
Water of combination and association by difference	15.05
	100.00

Alum Baking Powder:

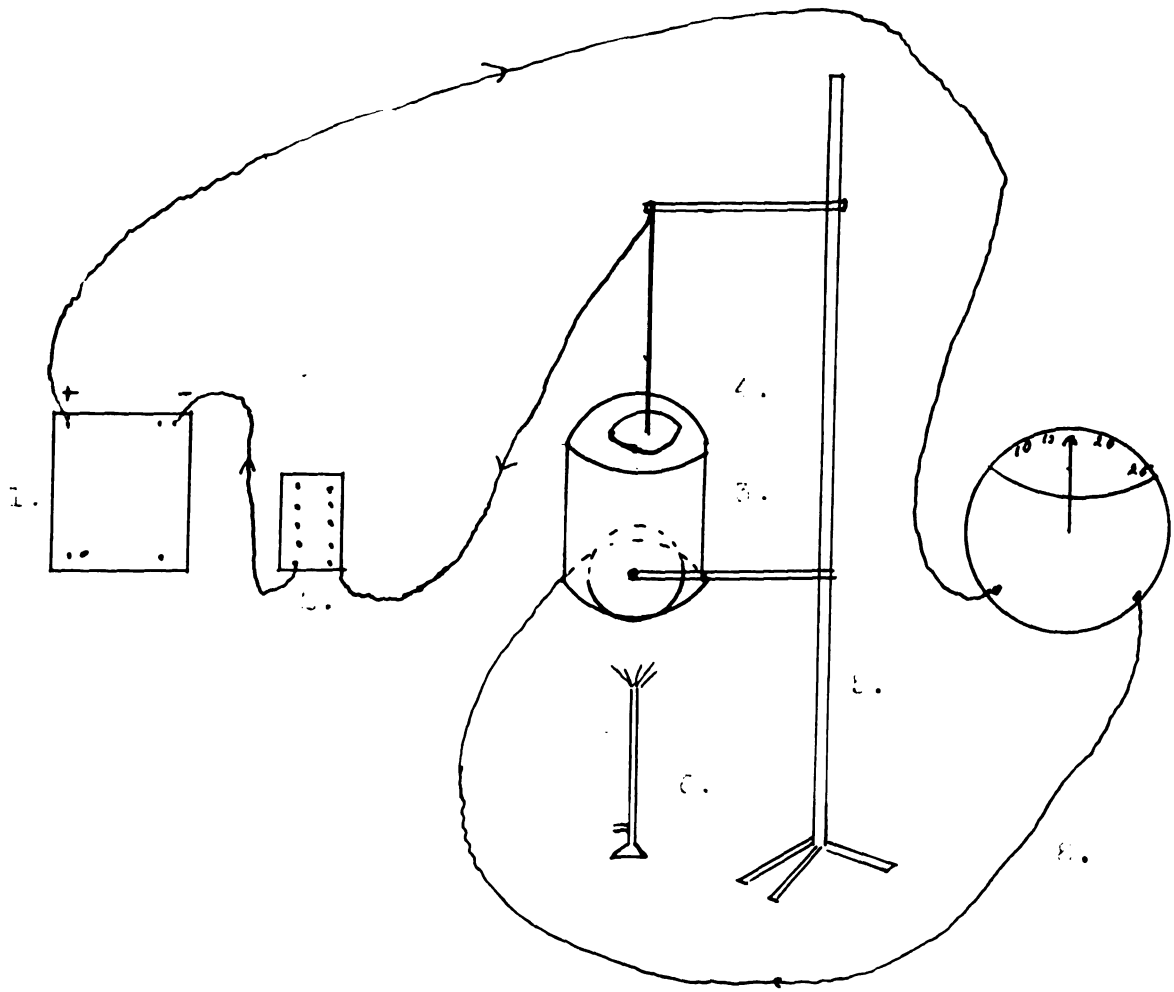
Total carbon dioxide, CO_2	9.45
Sodium oxide, Na_2O	9.52
Aluminum oxide, Al_2O_3	3.73
Ammonia, NO_3	1.07
Sulphuric acid, SO_3	10.71
Starch	43.25
Water of combination and association by difference	22.27
	100.00

Mixed Powders:

Total carbon dioxide, CO_2	10.68
Sodium oxide, Na_2O	14.04
Calcium oxide, CaO	1.29
Aluminum oxide, Al_2O_3	4.59
Ammonia, NO_3	1.13
Phosphoric acid, P_2O_5	3.38
Sulphuric acid, SO_3	11.57
Starch	42.93
Water of combination and association by difference	10.39
	100.00

A Comparative Study of Some Different Methods.

The necessary apparatus is shown on next page and consists of a storage battery, the potential of the current at the battery is 6.4 volts. For measuring the current a voltmeter and ammeter, graduated to 0.1 volts and 0.1 amperes respectively, is used. The resistance box is used for varying the current. A deposition bath consists of platinum dish which at the same time is anode, the cathode consists of platinum electrode. The electrode is supported in the proper position by ringstand and clamps. The platinum dish is supported by ringstand so that it may be heated by a Bunsen burner. Connections are made to the platinum dish, electrode, resistance box, and ammeter, as shown in the accompanying diagram. The temperature during electrolysis was 50-60°C.



1. Storage battery of three cells, capacity = 2.1, 4.2, and 6.4 volts.
2. Resistance box.
3. Platinum dish for anode.
4. Platinum electrode for cathode.
5. Ringstand with asbestus and copper triangle.
6. Bunsen burner.
7. Ammeter and voltmeter.
8. Wire connections.

Deposition of Lead in Nitric Acid Solution.

Prepare a lead nitrate solution containing 0.2 gm. and 0.01 gm. of lead per liter. Measure 50 c.c. of each solution, add 10 c.c. concentrated nitric acid sp. gr. 1.34 and electrolyze for three hours with CD_{100}= 1.5 and a voltage of about 4.2. The temperature of the bath should be from 50-60°C. Upon completion of the electrolysis the solution is siphoned out with constant adding of distilled water. The platinum dish is washed and dried for two hours in oven at a temperature from 200-210°C. The lead is weighed as lead peroxide and multiplied by factor 0.8661 to obtain the weight of lead.

Results:

Wt. of lead in solution	Wt. of lead found
0.01 gm.	0.01 gm.
0.005 gm.	0.005 gm.

This experiment proves that all lead can be deposited as PbO_2 during 3 hours at 50-60°C and with 1.5 amperes.

Deposition of Lead in Hydrolyzed Starch Solutions.

A 10 gram sample of starch is weighed out, 20 c.c. of concentrated nitric acid and 40 c.c. of water are added, the solution is heated until the starch is hydrolyzed; it is filtered and lead nitrate solution added in different amount. The solution is made up to 100 c.c. The experiment is run for 3 hours with 1.5 amperes and 4.2 volts at temperature from 50-60°C. The lead is deposited as peroxide on anode.

The following results were obtained:

Grams of lead added to 10 gm. starch solution.	Parts per million.	Grams of lead found.
0.001	10	0.0
0.002	20	0.0
0.005	50	0.0
0.01	100	0.0
0.02	200	0.0
0.04	400	0.0
0.08	800	0.0
0.10	1000	0.0

The experiment was further carried out by adding to 0.5 gm. of hydrolyzed starch solution 0.008 gm., 0.004 gm., 0.001 gm., 0.0005 gm., and 0.0001 gm. of lead as lead nitrate, the solution was made up to 100 c.c. and electrolyzed as in the preceding experiment.

Results:

Grams of starch in solution.	Grams of lead added.	Grams of lead found after 3 hours.
0.5	0.008	0.0011
0.5	0.004	0.0003
0.5	0.001	0.0001
0.5	0.0005	0.0
0.5	0.0001	0.0

The above two tables show that hydrolyzed starch interferes with the deposition of lead as peroxide on anode; if the amount of it in the solution is high, as in this case 10 gm. of starch in 100 c.c. of solution, no deposit of lead peroxide is obtained, although the solution may have 1000 parts of lead per million. Smaller amounts of hydrolyzed starch retard the complete deposition, but some lead peroxide can be obtained on the anode.

Deposition of Lead in Sucrose Solution.

The experiment was repeated with sucrose solutions. Five grams of sucrose are dissolved in distilled water, 0.001 gm., 0.0025 gm., 0.02 gm., and 0.08 gm. of lead added as lead nitrate, 20 c.c. of concentrated nitric acid added, the solution is made up to 100 c.c. and electrolyzed for 3 hours with 1.5 amperes and 4.2 volts. The temperature of the bath is 50-60°C. The washing, drying, and weighing of lead peroxide were done as in the preceding experiments.

Results:

Grams of sucrose in solution.	Grams of lead added.	Grams of Pb found.
5	0.001	0.0
5	0.0025	0.0
5	0.020	0.0
5	0.080	0.0

The following data were obtained by electrolyzing lead nitrate in 1% sucrose solution.

Grams of sucrose in 100 c.c. solution.	Grams of lead.	Grams of lead found.
1 gm.	0.0250	0.0091
1 "	0.0005	0.0
1 "	0.0010	0.0
1 "	0.0035	0.0

The above two tables prove that sucrose, like invert sugar (hydrolyzed starch), interferes with the deposition of lead. No deposit is obtained in 5% sucrose solution, although the quantity of lead is 800 parts per million. In 1% sucrose solution a slight deposit is obtained when the content of lead is 250 parts per million.

Incineration of Baking Powder to Ashes.

Weigh out 100 grams of baking powder into a casserole, add 50 c.c. concentrated nitric acid and 20 c.c. concentrated sulphuric acid, heat in hood on small flame to avoid spattering; heat until dry, place in a furnace at 600°C and heat to light redness. The lead will be in the form of sulphate. Leach the ash in hot saturated ammonium acetate solution (several leachings are necessary), filter, neutralize filtrate with ammonia, and pass hydrogen sulphide until saturation, allow to settle, filter and dissolve the precipitate in hot 20% nitric acid. Electrolyze the solution in weighed platinum dish, using platinum dish as anode and electrode as cathode. The temperature of bath is kept from 50-60°C. The current is run for 5 hours with amperage of 1.5.

After electrolysis, remove the liquid with a siphon tube in the usual manner. Dry and weigh.

With this method no deposit was secured upon the anode. The quantity of ash is large and bulky, and practically impossible to extract the lead sulphate with ammonium acetate. This method seems to give very low results, sometimes no deposit was detected.

Modification of the Corper Method.

The depositing electrode, anode, is made of a piece of platinum foil (2 inches in diameter) welded to a platinum wire about 6 inches long, the cathode is made of platinum wire wound into a spiral. The beaker used for electrolysis is a 400 c.c. Jena beaker with lip.

In use the two electrodes are brought close together (the anode above the spiral cathode 1 cm. apart) and within 2 cm. from the bottom of the beaker. Siphon tube is essential for removing the liquid after electrolysis is complete and consists of a bent glass tube. The apparatus is arranged as on diagram on page 11.

A 100 gram sample of baking powder is weighed out into a 400 c.c. beaker and 55 c.c. of concentrated nitric acid carefully added, the beaker is heated in the water bath until the starch is hydrolyzed. The solution is diluted to 350 c.c. and electrolyzed for 6 hours with amperage of about 1.5 CD_{100} and voltage 6.4. The temperature of the bath is kept from 50-60°C. The platinum electrode, anode, is removed, washed in distilled water, dried in oven at 200°C, and weighed as lead peroxide.

No deposit was secured upon the anode with six hour electrolysis. The experiment was repeated by adding to the hydrolyzed baking powder solution different quantities of lead nitrate, but all the results were negative.

Data:

Grams of baking powder in solution.	Grams of Pb added.	Grams of lead found.
100 gms.	0.001	0.0
" "	0.005	0.0
" "	0.100	0.0
" "	0.200	0.0

Modification of the Wichmann Method.

100 grams of baking powder are weighed out into a 1.2 liter beaker and 80 c.c. of hydrochloric acid (50 c.c. concen. + 30 water) added in small portions with constant stirring to avoid frothing. The mixture of acid and baking powder is heated until the starch is hydrolyzed and the solution is quite limpid. After cooling, 200 c.c. 50% ammonium citrate solution are added. The solution is kept cold while a slight excess of ammonia is slowly and carefully added. If a precipitate forms, sufficient ammonium citrate is added to dissolve it. 15 c.c. of 10% mercuric chloride are added and the solution diluted to about 1000 c.c. The solution is saturated with hydrogen sulphide and set aside. The precipitate settles rapidly and can be readily filtered. There is no need of standing over night as the heavy mercuric sulphide will carry down the lead sulphide. If the solution stands over night a precipitate of calcium citrate is apt to form.

The filter paper and sulphides are placed in a small casserole. 10 c.c. of concentrated nitric acid and 2 c.c. of concentrated sulphuric acid are added. The nitric acid is evaporated and the sulphuric acid slowly fumed off. The casserole is heated on a wire gauze to a light redness. The mercury salts will vaporize and any ferric sulphate present will be broken up to the oxide. After cooling, add about 20 c.c. of saturated ammonium acetate solution, heat on water bath and filter by decantation, repeat this three times. Wash the filtrate with hot water. The filtrate is made neutral or slightly alkaline with ammonia and warmed, hydrogen sulphide

is passed till precipitation is complete, the precipitate is allowed to settle and is then filtered.

Dissolve the precipitate in 20% nitric acid solution (10 c.c. concen. HNO_3 + 50 c.c. water) and hydrolyze for 4 hours with amperage of 1.5 CD_{100} and 4.2 volts. The temperature should be 50-60°C. Siphon out the solution properly. Dry in oven at 220°C for 2 hours, weigh as lead peroxide and multiply the weight by factor 0.8661 to obtain the weight of lead.

Data:

Baking Powders	Grams of Pb per 100 gms. of baking powder.	Parts per Million.
Cream of Tartrate	0.0005	5
Phosphate	0.0012	12
Alum	0.0010	10
Mixed powders	0.0009	9
	0.0008	8

The Referee on Baking Powder sent six samples of baking powder for collaborative work:

No. 1702, Monocalcium Phosphate Baking Powder.

No. 1712, Sodium Aluminum Sulphate Baking Powder.

No. 1722, Sodium Aluminum Sulphate and Monocalcium Phosphate Baking Powder.

No. 1711, Sodium Aluminum Sulphate Baking Powder.

No. 1721, Combination Phosphate and Sodium Aluminum Baking Powder.

No. 1701, Monocalcium Phosphate Baking Powder.

The lead was determined by Wichmann Modified Method and the following results were obtained:

Baking Powder.	Gms. of Pb per 100 g.	Parts per Million.
No. 1702	0.0048	48
	0.0057	57
	0.0055	55
No. 1712	0.0081	81
	0.0080	80
No. 1722	0.0084	84
	0.0085	85
No. 1711	0.0	0
	0.0	0
No. 1721	0.0	0
	0.0	0
No. 1701	0.0	0

General Discussion.

The deposition of lead in baking powder by electrolysis makes the determination shorter and more accurate. The precipitation of lead as dioxide upon anode affords an excellent method by which to separate it from the other metals. Only three metals are deposited on the anode: lead, manganese and thallium; but strong nitric acid solution prevents the deposition of the last two. Starch and sugars interfere with the deposition of lead. The author's intention is to remove starch gravimetrically and deposit the lead as peroxide upon the anode. He selected Wichmann's Method and modified it by reprecipitating the lead acetate with hydrogen sulphide, dissolving PbS in 20% nitric acid and electrolyzing the solution.

The deposition of lead as metal is not desirable, the drying is impossible due to rapid oxidation of lead by oxygen, the results will be usually higher. Lead as metal does not adhere firmly to the electrode. The deposit is spongy and may easily be washed away. There may be carried upon the cathode other metals if they are present.

The method is not very accurate. The complete removal of arsenic, tin, and zinc is not possible. The experiment cannot be accomplished in one day. The method is not accurate for commercial use.

Summary.

The gravimetric determination of lead in baking powders is very unsatisfactory; the results vary greatly, not only as a whole but in duplicate determinations.

The Remington modification method shows much closer checking than the Seeker-Clayton. The Exner method is very tedious and long and gives great variation in duplicate samples by the same analyst. The average of the results with Wichmann method agrees fairly well with actual content of lead in the sample, but due to wide variations in individual results the average cannot be considered of much importance.

The Bryan modification of the Corper method has been found very satisfactory when applied to phosphate baking powders.

A few experiments were made by Dr. Patten upon determination of lead in baking powders electrolytically, but the results were unsatisfactory.

The deposition of lead as dioxide upon anode affords a good method by which to separate it from other metals.

The deposition of lead as metal is not desirable, the results will be usually higher due to rapid oxidation of lead in drying.

Starches and sugars interfere with the deposition of lead upon the anode and should be removed gravimetrically.

The two methods, "Incineration of Baking Powder" and "Modification of the Corper Method", are undesirable. No lead was detected by these methods.

The author's modification of the Wichmann method gives the results of duplicate samples fairly satisfactory.

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