CHICLE DEVELOPMENT CO., INC.

500 FIFTH AVENUE

NEW YORK

PLEASE ADDRESS ALL COMMUNICATIONS TO THE COMPANY

February 11th 1936

Messrs. Nelson L. Buck
J.C. Cox
P.L. Becker
W.C. Arkell
J.S. Ellithorp
C.W. Dunn
G.R. Cowgill
F. Thamann.

Gentlemen:-

I enclose herewith copy of a letter from Dr. Prescott, dated February 7th, together with a report signed by Mr. David Richardson, dated February 6th entitled "The Spectrographic Determination of Lead in Chicle" and also a report signed by Dr. Prescott "Progress Report on the Study of Methods for the Determination of Lead in Chicle" - undated.

Very truly yours,

CHICLE DEVELOPMENT COMPANY

S.S. Yates,
President

SSY-W
Encl. (3)

cc: SCP

CHICLE DEVELOPMENT COMPANY
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NEW YORK

Office of the Dean of Science

February 7, 1936.

Mr. E. S. Yates
Chicle Development Company
500 Fifth Avenue
New York City

Dear Mr. Yates:

I enclose herewith the report of the work carried out in my laboratory by Dr. Emerson and his associate and also a report from Mr. Richardson, who has made the spectrographic determination of lead on many of the same samples. These will, I think, present certain facts relative to the use of these methods for the determination of lead. I presume you will want to forward these to the laboratories concerned.

It is my belief that much of value has been found out here, but it is also clear that if we had known of the complications brought out by the use of solid red pins at the outset, the approach to these problems would have been somewhat different and more logical. Since we did not know that, it may now be necessary to keep this fact in mind in the consideration of the early results.

Very truly yours,

S. C. Prescott
Dean of Science.

SCP:F

Arrangement of Equipment

The instrument used in this investigation is a Hilger E 1 quarts prism spectrograph of the Littrow type. It is adjusted so that the spectrum from 2450 to 3400 A. U. is photographed upon a 10 inch Eastman 40 plate. A rotating disc with steps cut in its perimeter located just in front of the slit of the spectrograph causes the spectrum lines obtained to increase in intensity from bottom to top in such a way that each step represents twice the exposure of the one below it. This permits long exposures as were necessary a step in which the lines will not be overexposed. A quarts lens just in front of the disc is adjusted to focus the image of the arc on the collimator lens of the spectrograph. This arrangement permits the arc flame to wander without loss of light and results in each spectrum line being formed from light from all parts of the arc flame. These are both great advantages over the usual means of imaging the arc on the slit. The exact position of the electrodes and the distance between them is controlled by their image on a screen produced by an auxiliary lens.

Electrodes

The electrodes are of Acheson graphite rods which while not spectroscopically pure do not contain significant amounts of either bismuth or lead. The upper electrode is 3/16-inch in diameter while the lower one is 1/4 inch having a 0.15 cc. cup drilled in its upper end for the sample.

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Title

The details of the procedure for the determination of lead in chicle have been modified somewhat since my report of December 10th, 1935. I am therefore rewriting the entire paper with the new points of technique in this report.

The spectroscopic method and apparatus used in this work are much like those described by Cholak, (Ind. Eng. Chem. Anal. Ed. 7, (1935). The chief differences lie in the arrangement of the arc with respect to the spectrograph, the use of a Hilger E1 quartz prism spectrograph instead of the corresponding Beech and Lamb instrument, the method of making the standard plates, and the use of a hand lens rather than a microphotometer for the study of the spectrograms. The procedure depends upon the relation of the intensity of the bismuth line 2898.1 to that of the lead line 2833.1 where the bismuth line has been produced by the excitation of a known amount of this element and the lead line represents the unknown amount lead present in the arc.

Arc

The arc is started by bringing the two electrodes together momentarily and then quickly separating them to the distance indicated by the image on the screen. The arc current is maintained between 10 and 12 amperes. The lower electrode containing the sample is made positive. An exposure of 45 seconds is usually sufficient to permit all the lead present in the sample to enter the arc flame.

The sample

The sample is submitted either as the dry ash or in a solution of which 1 cc. represents the ash from 1 gram of the chicle. When the dry ash is used, the bismuth internal standard is added to the sample before ashing is begun. In the case of the wet sample it is added and mixed just before the application of the sample to the electrode. Successive .15 cc portions of the mixture are applied to the electrode and dried in the oven at 105°C.

The Determination

A standard plate of spectrograms made with all the conditions identical with those used when studying the sample is first prepared. In this plate the amount of both [illegible] and the lead introduced into the arc are accurately known.

Set [illegible] rodes [illegible] follows, G A 83 representing the ash from a sample [illegible] tetl [illegible] tile lead:

Electrode No.	cc. soln. G A 83	Bismuth	Lead
1	0.5 cc.	10 ✓	0
2	0.5	20	0
3	0.5	20	0.5 ✓
4	0.5	20	1.0
5	0.5	20	1.5
6	0.5	20	2.0
7	0.5	20	2.5

A spectrogram of the arc from each of these electrodes is made under the same conditions as are used when studying the unknown samples. The last six exposures should have the bismuth line of equal intensity while the lead

✓ = .001 mg = Milligram

should increase in intensity in successive spectra. The purpose of the first exposure with Bismuth is to permit the determination of the amount of lead in the 0.5 cc of G. A #3 used in each electrode. The ratio of this intensity of the bismuth line 2890.1 to that of the lead line 2833.1 is matched to the same ratio in one of the successive six spectra. To illustrate the way in which this gives us the amount of lead in G A #3 we will assume that the Bi:Pb ratio for #1 equals that for #4 and that the amount of lead in 0.5 cc. G A #3 equals x. We have:

$$\frac{10}{x} = \frac{20}{1.0+x}$$

$$x = 1.0$$

Thus the real Bi:Pb ratios represented by the seven spectra are actually

	1.	10:1.0	
2.	10:0.5	5	10:1.25
3.	10:0.75	6	10:1.5
4.	10:1.0	7	10:1.75

By comparing the ratio of the above two lines in the spectrum from an unknown sample with the ratio on the standard plate it is possible to determine the amount of lead when a known amount of bismuth has been added to the sample. Visual matching of the ratios does not increase the error of the method.

Results

Designation	Description of Material	Lead in micrograms per gram sample
Chicle Ash R2	Old (1933) dry chicle	5.0
Chicle Ash R2a	Another sample of same	1.8
Chicle Ash R2b	Same	1.8
Chicle Ash R2c	Same	1.6
R3 #1	10 g. R3 ashed	0.8
R3 #4	Another 10g. R3 ashed	1.6
R3 #10	Another 10g. R3 ashed	0.35

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N 7623.03

R3 (Dried) #1	Large amount of chicle R3 melted stirred and dried at 100°C before ashing 10 gm. samples	0.8
R3 (Dried) #2		3.1
R3 (Dried) #3		3.8
R3 (Dried) #4		3.8
R3 (Dried) #21		0.9
R3 (Dried) #36		0.9
R3 (Dried) #41		0.4
R3 (Dried) #79		0
R3 (Dried) #21a		1.0
R3 (Dried) #36a		0.5
R3 (Dried) #41a		0.7

R3 (Dried) #79a		1.9
R3 #3	10 g. ashed	1.0
R3 #7	10g. R3 ashed	0.6
R3 #85	10g. R3 with 2µgms. Pb	1.3
R3 #93	Same	1.4
R3 #21	10g. R3 with 10 µgms. Pb	8.2
R3 #36	Same	8.1
R3 #41	Same	8.8
KA #21	"Kahoe Ash"	4.7
KA #36	" "	4.7
KA #41	" "	5.2
KA #79	" "	5.6
CB #85	Control blank on reagents	0.25
R3 #3	10g. R3 ashed	1.2
R3 #7	10g. R3 ashed	1.0

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R3 #22	R3 with 2ppm. Pb. added before ashing	3.0
R3 #36	Same	3.5
R3 #41	R3 with 10 ppm. Pb added before ashing	11.0
R3 #79	Same	11.0
#85	Pb recovery from dish heated to 500°C	0.05
#93	Same	0.03
#3	Same	0
#4	Same	0
#7	Added 2 ppm. Pb	4.0
#21	Same	3.6
#36	Added 10 ppm. Pb	7.9
#41	Same	7.3
RA #36	Dry "Ashed Ash"	4.7
Comm. #1	5 g. R3 with 40 γ Bi added before ashing. Dry	1.0
Comm. #2	5 g. R3 with 80 γ Bi added before ashing. Dry	1.0
Latex #6	5 g. latex #6 with 20γBi	0.45
Latex #8	" " #6 " " "	0.25
Latex #10	" " #10 " " "	0.1
Dark #6	10 g. with 40 γ Bi	0.2
Dark #8	10 g. with 40 γ Bi	
Dark #10	10 g. with 40 γ Bi	

Many of the above determinations were made in an effort to check the ashing technique for loss of lead. They were also done for the development of the spectroscopic method for lead in chicle. The precision of the determination has increased considerably throughout this work and we are prepared to go ahead with the study of the many samples at hand as soon as we are confident that the ashing procedure does not introduce error.

M.I.T. Spectroscopy Laboratory
Cambridge, Mass.
February 6, 1936.

(Signed) David Richardson.

KE 0006512

matter Drs. Cowgill and Rohde and myself were chosen to consider the problem to consider the problems of lead-determination in chicle by chemical and spectroscopic methods, and in a later conference it was decided that the work in my laboratory would be directed toward a study of the two principal methods in use with a view to finding the principal obstacles to accurate work and securing an evaluation of the two methods or a combination embodying certain features of each, with the hope of developing a method which would have a high degree of accuracy and give concordant results even when extremely small quantities of lead were present. As a final and convincing test of such a method, it should be possible to add known quantities of lead to chicle and to secure analytical results which showed at least a 95% accurate measurement of the whole amount so added.

KE 0006513

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PROGRESS REPORT ON STUDY OF METHODS FOR THE DETERMINATION OF LEAD IN CHICLE

The problem of estimation of lead in chicle has been one of acute interest for several months. Inquiry as to methods for the quantitative determination of this element brought out the fact that several relatively new procedures based on the use of "Dithizone" have been developed, chiefly with reference to the occurrence of lead in bones, organs and excretions of the body, but had not been specially devised for the study of chicle or for general use. It had been assumed that these methods could be adopted for the analysis of chicle, but the results obtained showed such variations in range as to demand investigation. Obviously it is useless to employ methods the accuracy of which may be seriously questioned, and a critical study of the procedures is therefore desirable.

At a meeting in November of those especially interested in this

The dithizone test for lead depends upon the removal by suitable means of other metallic elements or conflicting substances and the formation of a brilliant cherry red colored solution of lead and dithizone. This complex is separated, decomposed with acid and the lead can then be titrated with dithizone to yield a direct reading convertible into a percentage fig . Two modifications of the process have found use in clinical work, one which has been used extensively in the medical laboratories at the University of Pennsylvania the other a method employed by Dr. Cowgill of Yale Medical School and modified by Dr. Hervitt, Dr. Cowgill's assistant. The first involves the destruction of organic matter by the use of wet chemicals and the consequent fixation of the metallic elements in acid solutions; the second utilizes a slow carbonization and then combustion of the organic matter leaving the salts in a dry condition so that they may be further treated with the required solvents.

Attention is directed at the outset to the extremely small quantity of lead probably to be found in the material (from 0.5 to 2.0 parts per million) and to the significance of the necessity of great care to prevent loss through the technique of preparation. It is also well to bear in mind the limitations of methods depending on a color change of endpoint and the necessity for the use of "blanks" derived from the reagents. Equally important is the certainty that all chemicals and apparatus employed are as free from lead as possible and cannot give up or absorb this element during the procedure.

With these fundamental premises in mind a series of analysis was carried out in which chicle from a single lot was used as the material under test.

From this point on, and for the sake of brevity, the conclusions reached will be given without all the minute details of procedure, but essential facts will be stated.

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TESTS ON MEXICAN CHICLE

Mexican chicle was prepared for analysis by both wet and dry digestions followed by the so-called Philadelphia Method.

Coors and Sillimanite porcelain casseroles were both tried and the latter were adopted for regular use.

The original ashing method consisted of drying and burning 5-10 gms. chicle on a hot plate followed by muffling for one hour. The sample was then removed from the muffle, dissolved in 5 cc. 1:1 nitric acid, and returned to the muffle for another hour. The muffle temperature was 450°- 480°C., determined by thermocouple.

On the basis of the common practice of reducing organic materials to a carbon free ash before analysis, numerous variations of the above ashing procedure were tried, some of which were:

There were several types of hot plates tried, some of which were found too hot, and might cause loss of lead by volatilization. Therefore it has been concluded that the melted chicle should be "dried" only to a black charred mass on the hot plate, and the final burning to ash done in the muffle.

One method tried was to burn to an ash on the hot plate, add 5 cc. 1:1 nitric acid, dry and muffle for an hour; remove from the muffle, cool add another 5 cc. 1:1 nitric acid and muffle for another hour. This method gave a white ash that could be completely dissolved in acid and could be extracted with the dithizone reagent before precipitation of the calcium or other inorganic salts. There were many variations using both nitric acid and hydrochloric acid. No difference in the action of the acids was observed. It is now my conviction that for complete recovery of lead, the ashing process should not be so severe but whether to remove all the carbon or not has not as yet been definitely settled.

The Wet Digestion : 5 gms. of chicle were put in a 300 cc. Kjeldahl flask. It was then heated with concentrated nitric acid followed by 15 cc concentrated sulphuric acid, followed by more nitric acid until digest was white. A final addition of 2 cc 10% volume hydrogen peroxide was added to oxidize any remaining organic material. The process is slow but with experience 5 or 6 digestions can be made simultaneously in 4 hours. The results of this digestion were satisfactory, but because of the time and constant attention required this method was discarded as not being sufficiently practical for a rapid factory control method, although it is suitable for analysis where time is not a factor.

Twelve portions of the same sample of Bolivian chicle were run, six by wet and six by dry digestions, followed by the Philadelphia method of lead estimation from this point on. These analyses should have shown reasonable agreement. Such widely varying results were found in this series, however, that it was concluded that the method was not very reliable and therefore not to be recommended. The quantitative results obtained varied from 3.2 to 13.2 ppms. of lead. This indicated either lack of uniformity of distribution of lead or a serious defect in the method. Later work proved that this sample varied considerably in distribution of lead. In all of this work the reagent blank was near 0.005 mg. of lead per 10 gm. sample.

Because of the above results (and because the hypothesis of solder contamination had not yet been suggested) it was felt advisable to check reagents and solutions. This was done by use of known quantities of standard lead salts.

Standards. At this time there were two different standard salts, (acetate and nitrate) being used in the laboratory by two different operators. These standards were cross-checked with each standardized dithizone solution and were found to be identical for this test.

which had been recovered and purified by distillation, caused oxidation of the dithizone. The cause of this oxidation was perplexing and not immediately evident but after considerable investigation these facts were established.

(1) On redistillation of used chloroform the distillate is unsatisfactory for use with dithizone because the dye is decomposed. (2) Chloroform is decomposed in the presence of chemicals used in the method of analysis, and forms phosgene, chlorine and possibly peroxides. (3) These decomposition products of chloroform all react on dithizone. (4) Phosgene and chlorine are not removed by distillation, but may be concentrated thereby. (5) Distillation of over mercury does not remove the chlorine. The presence of phosgene was determined by the benzidine test. (x) Dithizone was found to be a more sensitive test for phosgene than is benzidine. (6) The usual concentrated sulphuric acid treatment, followed by concentrated sodium hydroxide, for the removal of impurities in chloroform, was not found to render the chloroform suitable for preparation of the dithizone reagent. Fresh

(x) "Organic Solvents" by Weissberger and Proskauer.

Double Distilled Water. All water used in the analytical work was first distilled in a Barnstead still followed by a second distillation in Pyrex glass. 0.05 p.p.m. of lead was shown by the Dithizone titration after this treatment. A third distillation was found not to improve the water as shown by titration involving so little dithizone, it is necessary to saturate the water phase with chloroform in order that the dithizone solution will not be wholly dissolved on successive additions if it until after the end point has been reached.

Beginning of the analytical work no unsatisfactory conditions were caused by the chloroform but later, as quantities needed greatly increased, a 25 lb. can of U.S.P. chloroform was purchased. This chloroform immediately caused high blanks and was found to contain lead in quantities too large for satisfactory use. In an attempt to purify this chloroform for use it was distilled in Pyrex. This distilled chloroform as well as any used chloroform

U. S. P. grade chloroform, daily used and stored in dark glass, seems to be the only grade that can be used satisfactorily.

Dithizone. Eastman dithizone has been used and found to be satisfactory. It is purified for the final titration by dissolving in 0.5% ammonia and precipitating with nitric acid. At present even this purification is not thought to be necessary. The standard dithizone solution is kept in Pyrex glass stoppered flasks covered with black paper. By these precautions the standard dithizone has been kept for as long as two to three weeks with only slight variations (0.0095 to 0.0120). The solution is of course standardized each time it is used for titration of the final solutions.

Potassium cyanide. The 10% potassium cyanide solution has given some trouble when kept too long but if made up fresh every 3 or 4 days no difficulty is experienced.

Phenol Red. In the Philadelphia method where there is an adjustment to pH 7.5 with phenol red, until recently we have used the solution supplied with LaMotte pH Block." When this solution was used up the phenol red solution was made up according to Clark. This Clark solution was much stronger and for the larger quantities (30-40 cc.) of solution to be adjusted this Clark solution of phenol red was much more satisfactory. The same quantities (2 drops) of dye solution were used at all times.

Spectrographic and Chemical Methods Worked Together

In working with both spectrographic and chemical procedures on the chicle samples it was found advisable to run both tests on the same sample. Since the spectrographic test requires a concentrated solution of the ash (the equivalent of a gram of chicle per cc) the following procedure was devised:

h is prepared in the manner described above. The ash is
nitric acid and the solution is made up to 10 cc. in a Pyrex
. About 3 cc. are sufficient for several spectroscopic plates
an r is used for chemical determination. By this method the
two pro pared favorably. In the work on perfecting dry digestions
ocurring ixwoks this method was used exclusively.

The spectroscopic procedure cannot handle the product of the wet digestion because of the large quantity of sulphuric acid present.

In the light of later work the dissolving of the ash of 10 gms. of chicle and making it up to so small a volume was found to be unsatisfactory as th

Since the chemical method needed more solution and the amounts of lead were not large enough to require an aliquot determination, the above procedure was discarded. This procedure also requires a carbon free ash which is undesirable since some of the lead may be lost. Further, the spectroscopic method could handle the dry ash very well if the "internal standard" is added to the thick before ashing.

Porcelain and Pyrex dishes. A good many tests were run using 50 x 45 mm Pyrex evaporating dishes. No very definite advantage from the analytical point of view could be found with these over the porcelain casseroles.

The Pyrex dish is somewhat better for the dissolving of the ash since the small amount of white residue that is difficult to get into solution is more easily observed. The Pyrex glass on continued use becomes badly etched and after several times in the muffle there was some breakage due to the extremes of heat to which it was exposed. Silica dishes are open to the same objections as to the etching.

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Recovered lead. At various times tests to recover known quantities of lead have been made. The most extensive work in this line was done near the beginning of the problem. The recovery in certain cases was unsatisfactory. By wet digestion methods the recovery was better than 90% in all runs.

The dry digestion has given more difficulty. In the presence of chicle the recovery has been at times good but even the Hewitt-Cowgill mild ashing in the few tests for recovering added lead run by that method it has varied from 80% to slightly 93% recovery. By the more severe ashing methods the recovery has at times been less.

The following results are typical of the recovery we have experienced by a severe ashing method.

To specimens of chicle shown by previous examination to contain an amount of lead of from 0.6 to 0.8 p.p.m., amounts of a lead solution were added such that the increment should be 2. p. p. m. and 10 p.p.m. The analysis was then made in the usual manner.

No.	Added p.p.m.	Found p.p.m.	Found less Blank Recovered p.p.m.	% Recovered
#8	2	2.6	1.9	95
#9	2	2.8	2	100
#10	10	8.9	8.2	82
#5	10	9.5	8.8	88
#6	10	10.6	9.9	99

From this table it will be noted that recovery is more difficult with the larger amounts of lead that were added. In order to discover the reasons for the discrepancy analysis of a lead salt alone was carried out.

When trying to recover lead after muffling in the absence of any chicle, no satisfactory results have as yet been obtained. Our examinations show that larger percentage losses occur if the amount of lead salt is large. In fact, in some cases all the lead nitrate solution originally evaporated in the casserole was lost. This was the case occasionally even though the muffle temperature was always kept below 500°C. This work is being continued until a satisfactory procedure is developed and a reliable method can be recommended.

[illegible] SAMPLE

As a check to find if there were anything unique to the current samples, determinations were run on a [illegible] sample [illegible] chicle already in the laboratory. The character of this sample was no different from any of the samples examined except that it was somewhat drier. It showed less than 1 p. p. m. of lead.

[illegible] ASH

As a routine of comparing methods used in different laboratories, the plan was proposed that Dr. Kehoe should prepare a large sample of chicle ash, thoroughly mix it and send portions to the laboratories concerned. The "Kehoe Ash" sample was analyzed in this laboratory by the Philadelphia method. At the time this ash was analyzed it was thought necessary to reduce all ashes to a white powder. The "Kehoe Ash" was treated with nitric acid, dried, muffled, again treated with acid, dried and muffled to attain a carbon free ash.

The white ash was dissolved in nitric acid and made to 10 cc. with water. From this volume two chemicals and one spectrographic determinations were made on each of four samples.

The results of the chemical analysis were as follows:

Sample	1st Detn.	2nd Detn.
	p.p.m.	p.p.m.
#1	4.8	4.6
#2	4.1	4.5
#3	4.1	4.6
#4	4.3	4.1
Average	4.4	

The variations at the first decimal place is probably due to the small quantity of solution handled. It is also interesting to note that although the "Kehoe Ash" was given a more severe ashing treatment than may be thought desirable there is no apparent loss of lead.

LOS CHENES CHICLE

In an endeavor to get a large amount of more or less uniform sample we used a duplicate sample of some Los Chenes Chicle which we had received. In 15 preliminary determinations on various lumps of the chicle results varied from 0.5 p.p.m. to 2.3 p.p.m.

By sampling different parts of a large lump (such as a block) no relation was found between the quantity of lead and the position on the inside or outside of the lump from which the sample had been taken.

By holding 150 gms. of chicle at 100°C. with frequent stirring of the viscous mass over a period of 3 days we hoped to obtain a more uniform sample. In spite of this treatment no more uniform results were obtained. These variations are now probably explainable from our knowledge of the use of soldered pans in the region from which the chicle was obtained.

Finally, the remaining Los Chenes chicle was cut up in very small pieces by hand and thoroughly mixed. All of our experimental work for the past three weeks has been on this material. This work has included the study

of lead found when known quantities of lead were added; variations of the ashing procedure, both of which have been previously discussed, and analyses using the Horwitt-Cowgill Method.

THE HORWITT-COWGILL METHOD

A total of 23 determinations by the Horwitt-Cowgill method have been run in this laboratory up to the present. With only this number of determinations and since some difficulties with the method have not yet been worked out, we do not regard ourselves in a position to make an extended critique of this method. Nevertheless, we have had the following experience, which may be of service to the plant laboratories.

Preparation of Sample. The Horwitt procedure of drying the chicle to a black charred mass on a stove and doing all of the burning in the muffle furnace without the addition of any acid is apparently superior to the dry method we had been using. By this treatment there is very ~~xxxxxxx~~ considerable carbonaceous material left with the ash. The strong solution of citric and hydrochloric acids is very effective in removing the lead from this material. The residual carbon requires a filtration which of course is a manipulation that takes additional time and is a possible source of either contamination and loss, both of which are undesirable for a factory control method.

Dithizone. The Horwitt-Cowgill method uses a minimum of chloroform dithizone mixture. This is desirable, if difficulty with the stability of dithizone has been experienced.

The Titration. In the final titration where the dithizone is removed from the chloroform by standard lead solution it has been found difficult to reach a final end point. The fading of the pink color is not easy to follow and there is a tendency of the chloroform and dilute potassium cyanide to emulsify.

The advantage of the preliminary end point is of course recognized but an experienced operator of the Philadelphia method can get a fairly accurate idea of what the second titration will be from the first titration. This is the case with chicle more than with some other materials because of the absence of large amounts of interfering materials such as ferric iron, bismuth, etc.

The Horwitt-Coegill method has 6 steps against the Philadelphia method's 5 steps. Aside from the final titrations there are two steps in the Horwitt-Coegill method, the first extraction of lead and the removal of the excess dithizone, that require considerable care and experience.

The concentrated citric and hydrochloric acid solution used by Horwitt and Coegill has been found very satisfactory for the removal of the ash and preventing the precipitation of the calcium salts. It has also been very useful for the cleansing of glassware.

No preference has been found in the use of hydrochloric acid and nitric acid.

Latex and Raw Smoke.

Determinations run on the Latex Sample #6, #8 and #10 in 50 gm. quantities showed less than 0.1 p.p.m. of lead in all cases.

Moisture. Moisture determinations on the latex were as follows:

#6 (formalin treated)	33.2%
#8 (boiled 1 hr.)	41.8%
#10 (" 1 ")	46.7%

These were dried at 100°C to constant weight.

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The Bark samples were run on 10 gms. each and gave the following results.

#6	0.15 p.p.m.
#8	0.15 p.p.m.
#10	0.6 p.p.m.

We have not as yet had the opportunity to check these results.

REFERENCES

The "Philadelphia Method" refers to Determination of Minute Amount of lead in Biological Materials" by Wilkins, Willoughby, Kramer and Smith, Ind. & Eng. Chem., Analytical Ed., V. 4, 33 - Jan. '35.

The "Merritt-Cargill Method refers to a Confidential Report entitled "Merritt-Cargill Modification of the Mitchrone Method for the Determination of Minute Amounts of Lead."

(Signed) H. C. Prescott.