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CHICLE DEVELOPMENT CO., INC.

500 FIFTH AVENUE

NEW YORK

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PART ONE

INVESTIGATION of the METHODS, MATERIALS, and CONDITIONS of CHICLE PRODUCTION

1. Introductory Statement - ORIGIN:

Chicle, as used in chewing gum, is the latex of the Achras Sapote Tree, solidified by boiling. This tree is found in sufficient quantities to exploit chicle, in the Yucatan Peninsula which comprises the northern half of the Territory of Peten in Guatemala, the northern half of British Honduras, the Mexican States of Campeche and Yucatan, and the Mexican Territory of Quintana Roo. Small amounts of chicle are also obtained in the Mexican State of Vera Cruz. The Achras Sapote tree is found extensively in this entire territory but always in a forest mixed with other trees. There are no solid Achras Sapote forests. This area is composed of limestone, which in many places is exposed on the surface of the ground. There is some clay in the soil overlying the limestone and in almost every place a heavy layer of vegetable humus, formed by the decay of the forests through many centuries. The limestone is porous, and in many parts of the Peninsula there are no surface rivers or streams and all rain-water escapes underground through crevices in the limestone.

There is a rainy season, lasting from six to seven months and a dry season during the balance of the year. The rainy season starts about the 1st of July and ends somewhere between January and March.

11. Description of PRODUCTION PROCESS:

The Achras Sapote Tree contains a latex, which is found in the young bark growing outside of the cambium. As you cut through the bark with a knife, you sever the latex vessels and the latex escapes. These vessels are not continuous pipes up the trunk of the tree but consist of short disconnected reservoirs containing the latex. Consequently, in bleeding the tree commercially to obtain the latex, it is necessary to cut the bark through to the cambium in zigzag gashes from the ground up to the first branches.

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11. Description of PRODUCTION PROCESS: (continued)

The latex that exudes through the severed vessels flows down the channel made by the cutting of the bark and is collected at the foot of the tree. This latex will only run during the wet weather when there is moisture in the soil. Thefefore, collection of the chicle is undertaken from July to the end of the wet season. The chiclero, of course, has to climb the tree during this process of cutting. He supports himself, while cutting, with a rope which passes around the trunk of the tree and around his body at the same time. A palm-leaf acts as a spout to carry the latex from the cut in the tree to a canvas bag which is placed at the foot of the tree to collect the latex. A tree will bleed for a period of four or five hours and then the cuts gradually close up and no more latex flows. A chiclero usually cuts from four to six trees during a morning and then waits until all the latex has flowed and been collected in a canvas bag at the foot of the tree. He may get from one to three quarts of latex per tree on an average. He then collects the latex from all the trees that he has tapped during the morning and brings it back to camp in a large canvas sack strapped on his back. The latex is collected for a period of several days and kept in camp in the form of latex in these canvas containers. In this form it is a sticky milky fluid.

When sufficient latex has been collected, the chiclero takes a large iron kettle, places it in the open, and builds a fire under it. He then pours the latex into the kettle, with the idea of boiling off sufficient moisture so that the chicle will solidify and be cast in blocks, which can be handled commercially. During this cooking process, nothing is added. The man continuously stirs the mass with a wooden stick to hasten the cooking and to prevent burning. When the cooking has been carried along for a sufficient length of time, determined by the experience of the chiclero, the kettle is removed from the fire and the chicle is then pressed into wooden molds where it is allowed to cool. These molds are greased on the inside so that the chicle will not stick to the wood. It is then transported in henequen bags to our warehouses where each block is cut for inspection with a steel machete and then it is stored in the warehouses until ready for shipment. It is shipped in cotton-lined henequen sacks and comes directly to the warehouses in the United States.

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III. COMMERCIAL DEVELOPMENT of Chicle Production:

The extraction of chicle requires the investment of large amounts of money before deliveries of the finished chicle can be made at the port of shipment. Contractors, operating on government lands (or, at times, on private lands) place chicleros in the woods at the beginning of the season and furnish them with supplies and equipment and provide the mules necessary for the transportation of the chicle. All these expenses must, in a large part, be financed by the buyer.

The Chicle Development Company and the Wm. Wrigley Jr. Company are both prepared and organized to take the necessary financial risks involved in making these advances, and, for this reason, they purchase jointly probably 90% of the chicle imported into the United States. The balance of the importations is handled by various brokers who deal in this material on a relatively small scale and at irregular intervals.

IV. INVESTIGATION UNDERTAKEN FOR DISCOVERY of the SOURCE of CONTAMINATION:

Two methods of obtaining the facts as to the contamination of chicle suggested themselves. The first was to observe the various steps in the process of preparation of the chicle in order to find out where the contamination could occur. The second consisted in collecting samples of all materials which came in contact with the chicle at all stages of the process and, by analysis of these samples, either to locate the contaminant or to disprove its existence as such. Both methods were put to use and both yielded results.

On or about December 1st, 1935, both the Chicle Development Company and the Wm. Wrigley Jr. Company arranged to send experienced and competent employees into the chicle camps to inspect and study the operations of the production of chicle as they are actually carried on. The following men were

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IV. INVESTIGATION UNDERTAKEN FOR DISCOVERY of the SOURCE of CONTAMINATION: (Continued)

selected and sent into the territories indicated:

Mr. Edwin Murray into a section of Quintana Roo (a journey covering a distance of four or five days on mule from Peto, Yucatan.

Mr. J.C. Brydon into a region in the eastern part of Yucatan, known as "El Cuyo".

Mr. Edward Moore into the lands of The Laguna Corporation of the Partido del Carmen in the State of Campeche.

Mr. J.G. Aznar into the same lands as Mr. Moore and then into the Tuxpena Section of the Partido de Champoton in the State of Campeche

Mr. R.C. Porter into the lands known as Santa Maria in the northeastern portion of Quintana Roo.

Mr. A.B. Perlin and Mr. R.P. Dyckman into the lands of The Mexican Gulf, Land and Lumber Company and adjacent lands in the Partida del Carmen in the State of Campeche.

These men were given a questionnaire, consisting of the list of questions shown on EXHIBIT "A" attached herewith. Their answers are summarized in full on this EXHIBIT "A".

These seven inspectors were also given instructions to collect a sample of each material or utensil in contact with chicle from the time the latex left the tree until it was cooked and cast in blocks.

The work of these seven inspectors covered the States of Campeche, Yucatan, and the northern portion of the Territory of Quintana Roo.

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IV. INVESTIGATION UNDERTAKEN FOR DISCOVERY of the SOURCE of CONTAMINATION: (Continued)

These investigations were later supplemented by additional investigations carried on by regular employees of the companies in the Territory of Peten, Guatemala; in the Territory of British Honduras; and, in the State of Vera Cruz, Mexico. Corrective action, taken at a later date, covered all territories alike.

V. REPORTS of FINDINGS:

A study of EXHIBIT "A" shows clearly that certain of the kettles, which were used for the cooking of the chicle, were responsible for the contamination. These kettles consisted of iron-plates riveted together with solder composed of lead and tin in the joints and, in some cases, with a coating of this same solder on the inside of the kettle. This coating was put on to prevent the rusting of the iron. This solder was in direct contact with the chicle latex during the process of cooking. No other significant sources of contamination were discovered by inspection, although it is obvious that small additional quantities of lead might find their way into the chicle or to the surface of the chicle block through the soil, by direct or indirect contact.

It was subsequently found that most of the kettles used in Guatemala and in British Honduras - and many of the kettles used in Tuxpam - were of cast iron with no seams and no lead or other contaminant in contact with the chicle.

VI. Samples of the following material were obtained and sent to the United States for test:

Fresh latex - as it came from the tree.
Samples of the outer bark of the Achras Sapote tree.
Samples of the inner bark of the Achras Sapote tree.
Foliage of the Achras Sapote (contaminated with soil).
Soil from various areas.
Wood ashes from the chicle camps (on surface of camp ground).

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V1. (continued)

Water as used for various purposes in the camps.
Nails used to make wooden molds in Cozumel District.
Soap used to lubricate the molds in Merida District.
Soap used to lubricate the molds in Tuxpena District.
Soap used to lubricate the molds in Laguna District.
Soap used to lubricate the molds in Campeche District.
"Sosquil" used to apply soap to the molds
Grease used to lubricate the molds
Kerosene used to lubricate the molds
Palm Leaves used as a mat in molding
Jute Mat used in molding
Hemp Bag used as mat or container (soiled) for blocks.
Dry Dye (used in dilute solution to label bags)
Hennequin Bag used as container for chicle blocks (soiled)
Plaster from warehouse wall.
Sweepings from floor of warehouse.
Numerous blocks of chicle prepared under the inspection of our inspectors.

The findings in respect to these samples are discussed in Part 2. of this report.

V11. CORRECTIVE ACTION TAKEN:

As soon as we located the source of the trouble as the solder in the riveted kettles, we immediately commenced an investigation to determine the best material for new kettles and the best type of kettle to be chosen. We considered the various materials, which were available for the manufacture of such kettles, and we made a careful study to determine the size and the shape of the kettle desired, and whether or not an improvement could be made by having a kettle with a double bottom in order to further improve the quality of the chicle by prevention of burning.

We were obliged to make prompt decisions in order to eliminate the contamination as soon as possible and we did not have time to make proper experiments to actually prove which type of kettle would be the best. We have installed new kettles of stainless steel, black iron, and of cast-iron. Almost all of the stainless steel kettles purchased are welded without the use of solder and were made in the United States under our direct supervision. The balance of the stainless steel kettles are stamped

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VII. CORRECTIVE ACTION TAKEN: (Continued)

out of one piece of metal without joints of any kind. Some of these have double bottoms and some have not. About one thousand black iron kettles were made of one piece stamped metal without joints of any kind and with a false double bottom which is removable. These were made in the United States under our direct supervision. The balance of the black iron kettles were made by a manufacturer in Merida with riveted and welded joints and with a false bottom riveted and welded to the kettle. During the manufacture of these kettles, we had our own inspector present in the factory in order to see that no solder was introduced into the kettle. All of these kettles have handles distinctive from those formerly used so that, at a glance, our inspectors can determine whether or not the kettle is lead-free.

Both the Wm. Wrigley Jr. Company and the Chicle Development Company have advised all their contractors that the companies will replace free of cost any of the old riveted and soldered kettles, which the contractors may have in their camps, whether used for the cooking of chicle or for the storage of water. Our own employees are personally present when the transfer of the new kettles is made to the contractors, and for each new kettle delivered, they personally see that an old kettle is destroyed. We are, therefore, confident that no old contaminated kettles will be in existence in the chicle camps during the season which is just starting. We have told the contractors that the finding in any of their camps of one single kettle, containing lead, would be sufficient cause for the cancellation of contract and we have from each contractor a letter agreeing to pay a heavy financial penalty if such kettles are found. In addition to giving these kettles to the contractors, we are sending out at our expense into all the Indian Villages in these regions, and installing without cost to the inhabitants, new kettles for any old ones which we may find in these villages and on the small surrounding farms. Many of these individual owners of isolated kettles do not use them at all for the cooking of chicle but own them for the manufacture of lard, soap, etc. They may, however, a few times during the season cook a few blocks of chicle in these kettles and sell the blocks through one of our contractors, and we are making this replacement in order to eliminate any possibility of future contamination.

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VII. CORRECTIVE ACTION TAKEN: (Continued)

We have already replaced approximately 1850 kettles at a total cost of $ 49,000.00.

We have advised all other importers of chicle of the source of this contamination and we have urged them to take similar action with the people from whom they buy chicle. The largest of these other dealers, F. Huber & Company of New York, has already purchased and sent to Tuxpan a number of kettles similar to ours.

VIII. FUTURE POLICY:

Both the Chicle Development Company and the Wm. Wrigley Jr. Company have organized staffs of inspectors, selected from men trained in the chicle business, familiar with the country and accustomed to the life of the chicle camps. There are eight or ten of these inspectors, who will have no other duties than to continuously travel from camp to camp, and the work has been planned so that they will visit every camp at intervals of between thirty and sixty days during the coming season. They are instructed to inspect every kettle and to see that all are of the new type. They will break up any kettles which they find that contain contamination. This inspection system extends throughout the chicle territory and is carefully organized.

We are confident that no more chicle will be introduced into the United States which is contaminated by lead.

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PART TWO

SOURCES OF LEAD IN COMMERCIAL CHICLE

A report on the samples and materials received and the results obtained upon analysis

The presence of lead in commercial chicle in quantities ranging from 0.15 parts per million up to 54.00 parts per million pointed to the likelihood of some source of contamination in the handling of the raw material. This likelihood was strengthened by the fact that chicle from certain areas of production showed only minute amounts of lead, while from others the amounts, although exceedingly variable, were frequently high. A previous study of the lead content of vegetation, taken from Mexican fields far away from cities and highroads, and uncontaminated by deposits from chimneys and factory fume and smoke-stacks had been made by Dr. R.A. Kehoe and his associates of the Kettering Laboratory, College of Medicine, University of Cincinnati. This study had failed to show a plant or plant product with a lead concentration in excess of 0.9 p.p.m. Moreover, such soil samples from various parts of the world as they had analyzed had not given results in excess of 10 p.p.m. It seemed unlikely, therefore, that any part of a plant would selectively absorb lead from the soil to such an extent as would be required to give the results obtained on chicle. Moreover, experience had shown that the opportunities for the accidental introduction of lead into processed materials are very numerous and very widespread.

As stated in Part One of this report, a survey of the chicle camps disclosed the fact that in certain areas the kettles for cooking the latex were fabricated in such a way as to permit solder to be in contact with the contents of the kettle. No other significant sources of contamination were discovered by inspection, although it was obvious that small additional quantities of lead might find their way into the chicle, or to the surface of the chicle blocks, through the soil, by direct or indirect contact.

The evidence obtained by the systematic collection of samples of materials associated with all the processes of producing chicle, together with the analyses of these materials, is presented in the following paragraphs.

1. The natural lead content of the raw material
Samples of latex together with various parts of the corresponding trees were obtained from various localities, and samples of soil were taken from the root area of the trees. Table 1. shows

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the analytical data on 68 samples of latex, 11 from the Cozumel District, 13 from Merida, 12 from Laguna, 12 from Tuxpena and 20 from three camps in the State of Campeche. These samples were collected by slightly different methods, and only a few were so obtained as certainly to exclude the possibility of slight contamination from contact with bark or implements which were unclean in a chemical sense. Several were collected directly into chemically clean pyrex tubes, which were sealed on the spot and kept closed until the time of analysis; others were taken from the wanvas bag normally used to collect latex from the tree; others were taken from the larger canvas bag used to collect latex from many trees; two otherswere taken from the still larger bags used to convey the collections for several days into the cooking camp. Some were treated with formaldehyde, some with ammonia, and others were heated by dipping the containers in a boiling water bath. (These measures were employed to prevent fermentation and loss of material in shipment). A few had no treatment of any kind. It is apparent that the latex contained only such minimal traces of natural lead as have been found in other vegetation. The variation in the results, while slight, is not due entirely, nor, perhaps, appreciably, to variation in the natural lead content of different samples. On the contrary, the higher findings are associated with opportunities for slight and hygienically insignificant contamination, in that such samples contained foreign material (e.g. bark), or they had been taken from larger collections of latex which had been transferred from one canvas bag to another. In general, it may be said of these samples, as of every other type of material collected for analytical study, that the more they had been handled, the greater was their lead content. Doubtless, however, some small quantity of lead (up to 0.08 p.p.m., perhaps) is a natural constituent of the latex.

In view of the minute quantities of lead in latex, the lead content of the soil and of the foliage, bark and trunk of the tree loses in primary importance and has significance only so far as these materials may be contaminants of the latex as it is collected. Accordingly the analytical results on these substances are given under the next heading.

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2. The lead contents of various materials with which latex comes in contact exclusive of those associated with cooking.

Without again entering upon a step-by-step description of the procedures, suffice it to say that the collection and handling of latex are associated with some degree of contact with a number of materials. Table 2. lists these materials, approximately in the chronological order of their use or occurrence, and gives the lead content of samples obtained on the spot. These data disclose no facts of vital importance. Kerosene often contains significant quantities of lead as a result of refinery "sweetening" processes, and is thus ill chosen as a lubricant for molds. This will be eliminated in future operations. The lead found in other materials is of little practical importance. (The dye, high in lead, is used on the bags with a stencil in very dilute solution. It would not influence the lead content of blocks of chicle carried within such bags.) It is probable that a proportion of the lead found in these samples was derived from contact with the soil. Obviously reasonable care should be taken against the inclusion of soil or dirt of all kinds in the latex or in the chicle blocks.

3. The significant source of the lead contamination of chicle.

Pursuant to the observation that soldered kettles were in use in certain camps, samples of the solder were obtained and analyzed and found to run from 25 to 50 per cent of lead, with a remainder of tin. Blocks were made from fresh chicle in certain of these kettles under the supervision of an investigator, these blocks being identified and preserved for analytical examination. The details of a single experiment will serve to illustrate the source of the contamination and the possibilities for variation in the lead content of chicle so contaminated.

One lot of chicle latex designated as "Cuyo" and found subsequently to contain lead in the amount of 0.05 p.p.m. was divided into 2 parts, one of which was cooked in a black iron unsoldered kettle, the other in a galvanized iron kettle with soldered seams and a solder coating on the inside of the kettle. The block resulting from cooking in the first kettle yielded 0.20 p.p.m. on analysis, while the second showed variable results up to 200 p.p.m. (The reason for variable results will appear later.) A second lot of chicle latex from the same source (but not analysed), was divided and cooked in two other kettles, one a galzanized, soldered kettle which had been used for two months, and the other a similar kettle

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that had been used for a long time. The resultant chicle blocks showed respectively 75.0 p.p.m. and 0.70 p.p.m. of lead. The spectrograms of these samples showed the presence of tin in quantities proportionate to the quantities of lead.

It is apparent from these observations that a soldered kettle gives up its solder to the cooking latex, gradually losing it and thereby becoming a less significant source of contamination. Some considerable variation in lead content may be expected among blocks of chicle prepared in soldered kettles which have been in use for variable periods of time. Table 3. shows a wide range of variability in a series of blocks prepared for experimental purposes in a series of cooking camps.

4. The particulate nature of lead in chicle.

In the course of numerous analyses of commercial chicle, it had been observed that check results were not obtained on samples from the same blocks, even when attempts were taken to obtain uniform samples from contiguous areas. This apparently uneven distribution of lead through the blocks, when coupled with the facts as to the source of contamination, indicated that the lead was present, in part, at least in particulate matter. On the assumption that some portion might be in the form of visible droplets of solder, X-ray examinations were made of blocks known to be high in lead. The films demonstrated the presence of discrete particles of variable size, in numbers roughly proportional to the magnitude of the lead contamination as indicated by analyses. Experiments dealing with this subject will be described in another section of this report; mention is made here in order to draw attention to the fact that the physical state of the lead and its irregular distribution through chicle blocks make it quite impossible to obtain uniform samples of chicle for analysis. It is to be expected, therefore, that any series of analytical results on chicle samples from the same source will vary within wide limits. Such analyses may be counted on to portray only the general order of magnitude of the lead content of the material. The importance of this fact, for present purposes, relates to the difficulty of comparing analytical results obtained in the various laboratories concerned with this matter. In order to combine and utilize the data being obtained, it was necessary to compare analytical results on samples of uniform lead content.

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5. A comparison of various analytical methods.
Since all the laboratories engaged in this study were concerned primarily with chicle it seemed best to use commercial chicle as the material for analysis and to so treat it as to render it homogeneous. Accordingly a large sample was prepared by compositing the ash of 12 one-third pound portions of chopped chicle, each of which had been burned over night in a muffle furnace at 450°C. in a 500 cc quartz evaporating dish. The ash was sieved until it passed the standard 200 mesh sieve, after which it was thoroughly mixed and subdivided. In order to check its homogeneity, a series of samples were analysed in this laboratory by three methods, a spectrographic method (which has been published together with its sensitivity and accuracy) a dithizone method (the details of which are unpublished), and a chemical method, the latter, described elsewhere, has been in use over a period of years. It consists essentially of standard analytical procedures which result in the isolation of pure lead chromate. It gives uniformly low results by reason of loss in the numerous separatory steps.) When repeated analyses were found to check within the known limits of accuracy of the three methods, adequate quantities of the ash for triplicate analyses were sent to various analysts. The results appear in Table 4. The spectrographic results obtained in two laboratories correspond closely and give somewhat higher results than two of the dithizone methods. The chemical (analytical) method confirms the higher values of the spectrographic analysis when due allowance is made for its known error on the low side.

1. Summary

Commercial chicle has been found to contain lead as a contaminant. The natural lead content of the raw latex is not in excess of that found in many other types of vegetable products.

2. The source of contamination has been found in the solder employed in the fabrication of certain of the kettles used to cook the latex.

3. The solder is given up from the kettles largely in the form of globules which distribute themselves unevenly through the cooking mass.

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4. The analytical methods employed in the study of the problem yield substantially uniform and satisfactory results when applied to homogenous samples of lead-containing material. They do not, and cannot be expected to give concordant results on commercial chicle because of the impossibility of obtaining samples which are homogenous with respect to their lead content.

5. Obviously, the solution of the problem of future lead contamination of chicle consists in the permanent elimination of soldered or otherwise lead-bearing kettles.

Table 1

Distribution of Samples of Fresh Chicle Latex * According to Lead Content

Lead in parts per million	Frequency
0 - 0.019	0
0.02 - 0.039	4
0.04 - 0.059	24
0.06 - 0.079	16
0.08 - 0.099	12
0.10 - 0.119	6
0.12 - 0.139	3
0.14 - 0.159	1
0.16 - 0.179	2
Total	68

* The results on samples stabilized with Formaldehyde and Ammonia do not differ measurably from those obtained on untreated samples. The ammonia contained lead to the extent of 0.013 p.p.m.

Table 2

The Lead Content of Certain Potential Contaminants of Chicle

Articles Analyzed	Number of Samples Analyzed	Lead in parts per million
		Minimum
Outer bark of Zapote tree (may fall into latex)	12	0.10
Inner bark of Zapote tree (may fall into latex)		0.10
Foliage of Zapote tree (contaminated with soil)	1	
Soil from various areas	to be analyzed	
Wood ashes from camps (on surface of camp ground)	2	7.50
Water as used for various purposes in camps	13	0.015
Nails used to make wooden moulds in Cozumel	6	0.00
Soap used to lubricate moulds in Merida	4	1.40
Soap used to lubricate moulds in Cozumel	5	0.80
Soap used to lubricate moulds in Tuxpena	4	0.20
Soap used to lubricate moulds in Laguna	1	
Soap used to lubricate moulds in Campeche	2	0.60
"Sosquil" used to apply soap to moulds	10	2.00
Grease used to lubricate moulds	1	
Kerosene used to lubricate moulds	1	
Palm leaves used as mat in moulding	1	
Jute mat used in moulding	1	
Hemp bag used as mat or as container (soiled) for blocks	3	3.0
Hennequin bag used as container for chicle blocks (soiled)	2	6.6
Dry Dye (used in dilute solution to label bags)	1	
Plaster from warehouse wall	2	4.00
Sweepings from floor of warehouse	3	10.40
Total number of analyses	76	

Table 3.

The Lead Content of Chicle Prepared Experimentally in Camps in which Soldered Kettles were in Use. *

Description of Sample	Lead Content of Chicle Expressed in parts per million.	Tin Line in Spectrogram of
Block made in Merida Camp #1	3.60	present
" " " " " #2	0.30	?
" " " " " #3	13.00	dense
" " " " " #4	18.00	"
" " " Cozumel " M	5.80	present
" " " " " P 1	2.40	"
" " " " " P 2	1.80	"
" " " " " P 3	1.60	"
" " " " " P 4	1.30	"
" " " " " P 5	1.30	"
" " " " " P 6	1.20	"
" " " Tuxpena " C	0.50	"
" " " " " E.M.	0.50	"
" " " " " S.F.	0.15	?
" " " " " F.C.	0.20	?
" " " Laguna " A	0.20	?
" " " Campeche " Q-1	0.30	?
" " " " " M-1	0.10	?
" " " " " M 2	0.20	?

* Two blocks made from fresh chicle in new stainless steel kettles gave results of 0.05 p.p.m. and 0.05 respectively.

Another sample block cooked in a clay pot showed 0.20 p.p.m.

Table 4

Analytical Method	Location of Laboratory	Grams Ash Used	Gram Equivalent of Raw Chicle	Lead found Mgs.	Res in les raw
Dithizone	Yale University	0.560	10.0	0.0502	
Dithizone	Yale University	0.560	10.0	0.0508	
Dithizone	Massachusetts Institute of Technology	0.560	10.0	0.045	
Dithizone	Massachusetts Institute of Technology	1.120	20.0	0.086	
Spectrographic	Massachusetts Institute of Technology	0.560	10.0	0.0475	
Spectrographic	Massachusetts Institute of Technology	1.120	20.0	0.1074	
Dithizone	Kettering Laboratory	0.280	5.0	0.028	
Spectrographic	University of Cincinnati " " "	0.280	5.0	0.027	
Anal. Chem.	" " "	5.600	100.0	0.47	
Dithizone	Beech-Nut Packing Co., Canajoharie	?	?	?	

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METHODS FOR THE CONTROL OF THE LEAD CONTENT OF CHICLE IN THE MANUFACTURE OF CHEWING GUM

General Methods of Plant Control

Since certain lots of chicle had been found to contain lead in excess of the present tolerance, it became necessary to select that which was entering into the production of chewing gum so as to exclude unsatisfactory material. This required routine analyses of chicle in the manufacturing plants. Accordingly laboratory facilities were provided or expanded in the manufacturing plants and analytical methods were made the subject of collaboration between the laboratories operated by the industry and those engaged in the general investigation, (i.e. The Kettering Laboratory, College of Medicine, University of Cincinnati, the biochemical Laboratory at Yale, and that at Massachusetts Institute of Technology). Results were obtained and exchanged on samples of known lead content so as to provide a basis for comparing the various series of results.

By reason of the observed lack of homogeneity of the chicle, with respect to its lead content, some method of sampling had to be devised which would permit the analyses to give information as to the general order of magnitude of the lead content of batches of ground chicle entering into the manufacturing processes. Various plans for grinding, mixing, and sampling the chicle were developed in the plants of the American Chicle Company, the Beech-Nut Packing Company, and the Wm. Wrigley Jr. Company, as described in Appendix B to this report. The analysis of such samples provided the criterion for eliminating chicle of high lead content from manufacturing procedures.

From what is now known of the physical state of lead in chicle it is not to be supposed that the samples of chicle obtained by the methods as described were entirely homogeneous, or completely representative of the lots used in making up the batches for production. On the other hand some considerable amount of information developed whereby the selection of lots could be made with assurance that they would prove to be low in lead. In order to demonstrate the character of this information it is necessary to discuss briefly the results of certain experimental observations.

1. The physical state of lead in chicle.
Mention has been made in Part II. of this report of the fact that

PART THREE

certain samples of chicle experimentally produced in kettles fabricated with a tin-lead solder, contained appreciable quantities of tin together with abnormal amounts of lead. In fact, in the spectrographic analysis of commercial chicle, tin was always found to be present when the lead content was abnormally high. Thus, it appeared, early in the investigation, that solder was in all likelihood the chief source of the lead contamination, and, perhaps the only significant one. (It developed later that solder was the only significant source of contamination.) In order to study the distribution of lead in the chicle, several methods were employed. Chicle known by analysis to contain excessive quantities of lead, was dissolved in various organic solvents, whereupon a sediment was obtained in which coarse metallic particles could be seen microscopically, while many spherical particles of variable size(from 0.0005 in. to 0.12 in. in diam.) could be seen with the aid of a microscope. Radiographic examination of chicle blocks also revealed the presence of dense spherical particles of variable size, irregularly distributed through the chicle. Qualitative analyses of the particles isolated wither from the sediment of the chicle solutions, or located by X-ray and removed from chicle blocks showed the presence of lead and tin.

Obviously chemical analyses of small samples of such material, no matter how selected, could be expected to give erratic results, and such was the case. On the other hand, the radiographic examination of ground chicle might be expected to give useful information as to the number, size, and distribution of the solder particles, thereby providing additional means for the elimination of heavily contaminated material. This proved to be a useful procedure.

2. The localization of chicle contamination.

As analytical results accumulated on various lots of chicle imported prior to the elimination of soldered kettles in the chicle camps, it became apparent that there was no appreciable factor of lead contamination in chicle from a number of sources. Table 1. clearly demonstrates the essentially satisfactory character of the chicle imported from Tuxpena, the Cozumel Bank Lands, the Carmen District of the State of Campeche, and from Belize. The chief area supplying contaminated chicle was obviously Merida. Thus by elimination of the Merida chicle from production the greater portion of undesirable chicle could be avoided.

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PART THREE

The Effectiveness of the Plant Control

By means of routine analyses of chicle going into production, combined with care in the selection of lots presumably low in lead (on the basis of source or x-ray examination), the use of raw material containing lead in excess of the tolerance has been avoided. (There can be little doubt that the filtration of molten chicle as carried out by standard methods in the manufacturing plants served to remove some of the lead. This point will receive attention later in a further report. This fact is demonstrated by the analytical data on finished chewing gum as carried out routinely in the manufacturing plants. In no case has the lead content of the product exceeded the tolerance. The available data of the plant laboratories are shown in Table 2., while the results of a smaller number of analyses carried out in this laboratory are given in Table 3. It may be seen that the distribution of the results corresponds in the two sets of results, thereby providing presumptive evidence of uniformity in the analytical work.

APPENDIX "B"

BEECH NUT PACKING COMPANY

METHOD TO INSURE COMPLETE MIXING OF CHICLE BEFORE MELTING

1. One block, or equivalent small pieces from each of 8 different bags from each of 8 different lots ground in the Everetts grinder.

2. Run through Everetts grinder.

3. Fill enough trucks at each plant in this way for one full day's run.

4. Place either 4 or 6 trucks in the most efficient positions - a horseshoe or "U" is suggested - to be shoveled into "E" grinder truck, a shovelful at a time from each truck.

5. Put through grinder and again fill 4 or 6 trucks.

6. Again fill one truck by taking one shovelful at a time from each of four trucks.

7. Again run through Everetts grinder.

8. Place in barrels by taking a scoopful from each truck.

9. Test by taking one ounce from each barrel.

10. Spread 1 scoopful on a tray from each barrel of a day's run for drying.

11. Dump in barrels after drying.
Dump barrels in boxes for day drier. If two charges are run, dump one-half barrel at a time.

12. Number barrels consecutively, dividing for each day's run.

13. Carry 4 days ahead at Canajoharie.

14. Carry 5 days ahead at Brooklyn.

15. Must be sugar or virgin barrels.

16. Brooklyn sample s ent by first-class mail at noon to Canajoharie Control Laboratory for test.

17. Chicle not found satisfactory is set aside and not used in production.

APPENDIX "B"
continued

AMERICAN CHICLE COMPANY

METHOD OF CONTROL PROCEDURE TO INSURE COMPLETE MIXING OF CHICLE

The Lead Control Procedure in effect in this plant is briefly as follows:

1. Chicle from each shipment from each source is ground separately in small lots of from 20 to 30 bags and dried on trays in a dry room.

2. When the Chicle has cooled sufficiently after drying, it is loaded by hand into barrels in such a manner that each barrel contains substantially equal amounts of chicle from each bag in the lot ground.

   Before the Chicle is dumped from the drying trays, a sample is taken and "quartered down" to about 1 lb. This is sent the Laboratory for analysis.

3. The barrels of blended Chicle are held in a specified space until the Laboratory report has been received. If the use of the lot is not approved, the barrels are removed to a location on another floor where there will be no danger of using by mistake.

4. When a barrel of Chicle is dumped prior to the weighing out of individual filter press batches, it is mixed with a shovel.

5. The method of analysis for lead is that developed by Dr. Horwitt. However, the method of ashing the sample prior to analysis is that used at Dr. Kehoe's Laboratory, since this is more rapid and permits tests to be started and completed the same day.

6. In addition to the Chicle, samples of finished gum and base from the filter presses are being tested.

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WM. WRIGLEY JR. COMPANY

METHODS OF CHICLE BLENDING FOR LEAD CONTROL AND METHODS OF SAMPLING AND TESTING

BLENDING DURING DESICCATION:

1. Each importation of chicle is designated as a separate lot when received at the plant and each such lot is stored separately.

2. Each lot is ground separately just before desiccation.

3. In grinding, use three (3) bags of block chicle simultaneously.

4. After desiccation, eight (8) trays holding dry chicle are dumped simultaneously from each of three (3) loads of trays into a mixing hopper, from which the dried chicle is carried away to be bagged. (Each bag of desiccated chicle, therefore, will contain chicle from a minimum of nine (9) different bags of block chicle.)

BLENDING OF CHICLE AT TIME OF USE:

1. When weighing out filter press batches, use four (4) lots of desiccated chicle for each batch.

2. The filtered chicle is mixed in a blending tank so that a unit batch, when drawn from this tank, represents chicle from at least four (4) filtered batches. A unit batch of chicle as drawn from the blender will contain chicle from 100-200 different bags of block chicle.

SAMPLING AND TESTING CHICLE:

1. Sampling for Preliminary Test of Crude Block Chicle as Received: Upon receipt at factory of each lot of crude chicle, a sample lot, consisting of ten (10) bags taken at random, is immediately sent up to the desiccating department and there separately ground, bag by bag. A two (2) pound sample of ground chicle is removed from each bag lot and finally the whole twenty (20) pounds is quartered down to one two (2) p ound sample, which is sent to the laboratory. This sample (in a kraft paper bag provided for this purpose) is marked as to date, lot number, and initials from the separate blocks of chicle, which entered into the sample.

2. Sampling for Test of Desiccated Chicle:
(Desiccation is completed at least two weeks before chicle is used.)

At the time of desiccation of a lot, a sample is taken from each bag of desiccated chicle at time of filling into bags, on the following basis:

Using 6-1/2 gram unit bronze sampler, if lot contains about 40,000 pounds crude weight, or less.

Using 5-1/2 gram unit bronze sampler, if lot contains approximately 60,000 pounds crude weight.

Using 3-1/2 gram unit bronze sampler, if lot contains approximately 80,000 pounds or over, crude weight.

These samples are collected in fibreboard boxes and as soon as the lot is completely bagged, sent down to the laboratory, marked with date, lot number, and number of bags (desiccated) in lot.

3. Sampling for Check or Control Test (Made at time of use of Chicle)

When using desiccated chicle, a sample is taken of each different lot of chicle as it is used in each filter press batch made up, on the basis given below:

If the lot of chicle had an original crude weight of about 80,000 pounds, or over, use the 1-1/2 gram bronze sampler.

If the lot was smaller than 80,000 pounds, use the 3-1/2 gram bronze sampler.

Upon completion of a lot of chicle, the sample representing the lot is sent down to the laboratory in the fibre box in which it has been collected. This box is marked with the date, lot number, and number of filter press batches in which the lot of chicle was used. (These samples average about four (4) pounds in weight, as sent to the laboratory.)

4. The samples of chicle sent down to the laboratory are analyzed for lead by a method which is essentially that of Dr. Herwitt, except for a slight different in the method of ashing the sample.

5. If a lot of crude block chicle is found to be substandard as regards content of lead, this lot shall be plainly marked with "Do Not Use" signs and shall be stored on a floor where only chicle of this nature will be kept.

6. In addition to the sampling and analyses outlined above, daily random samples of chewing gum are being tested for lead.

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Table 1.

Distribution of Analytical Results on Commercial Chicle from Various Production Areas According to Lead Content.

Lead in p.p.m.	Belize	Campeche	Carmen	Cozumel	Laguna	Lox Chenes	Merida	Tuxpena	All
0-0.49	5	0	7	0	2	1	1	4	2
0.50-0.99	5	0	1	0	1	0	2	3	1
1.00-1.49	6	1	3	3	2	0	0	1	1
1.50-1.99	4	0	3	2	2	0	3	0	1
2.00-2.49	1	0	0	1	1	0	0	0	
2.50-2.99	1	0	0	0	0	0	2	0	
3.00-3.49	1	1	1	0	1	0	1	0	
3.50-3.99	0	0	1	0	0	0	4	0	
4.00-4.49	0	0	0	0	1	0	1	0	
4.50-4.99	0	0	0	0	0	0	0	0	
5.00-5.49	0	0	1	0	1	0	1	0	
5.50-5.99	0	0	0	1	0	0	0	0	
6.00-54.00	0	0	1	1	1	0	6	0	
All Results	23	2	18	8	12	1	21	8	9

Table 2

Distribution of Results of Analyses of Finished Gum in Laboratories of Manufacturing Plants

Lead in p.p.m.	Frequency
0 - 0.19	5
0.2 - 0.39	67
0.4 - 0.59	154
0.6 - 0.79	102
0.8 - 0.99	54
1.0 - 1.19	18
1.2 - 1.39	8
1.4 - 1.59	5
1.6 - 1.79	3
1.8 - 1.99	1
	417

Mean = 0.6