frogs and fishes tartaric acid is much more toxic than Rochelle salts.

So far as the writer is aware, no series of systematic observations so extensive as those here recorded have ever been made. Only isolated experiments seem to have been conducted with no efforts at comparative results.

Our conclusions from these experiments were far different from our expectations in the beginning, which opinions were based on opinions of some eminent men in the field of investigation. The Referee Board of Consulting Scientific Experts, for example, laid great stress on the fact that sodium benzoate is not toxic to frogs as comparative evidence in drawing conclusions as to its toxicity to men. Such experiments as these in the past have been often relied on as evidence in court as to the poisonous character of substances used in foods, but after studying the data we have accumulated somewhat carefully, the writers have come to the conclusion that experiments of this nature, at least upon animals so distantly related to man, do not furnish conclusive evidence of the effects of such substances upon the human system.

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THE GRAVIMETRIC DETERMINATION OF REDUCING SUGARS IN CANE PRODUCTS

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The reference books devoted to sugar analysis all describe the determination of reducing sugars in considerable detail. The detail concerns the method of heating, the time of boiling, the filtration of the cuprous oxide, the determination of the copper by any of several methods, and the calculation of results. On the other hand, the preparation of the solution, the clarification and removal of excess lead are, as a rule, referred to very briefly and in the broadest terms: "Clarified if necessary with a minimum of neutral lead acetate solution—deleading by means of sodium carbonate, sodium sulfate, potassium oxalate or other means."¹ "In case clarification with lead acetate is used, the excess of lead must be removed; this may be done by the addition of a soluble carbonate, sulfate or oxalate."²

The present investigation was undertaken at the suggestion of G. L. Spencer, Chief Chemist of The Cuban-American Sugar Company, and carried on under his general direction. The work extended over a period of eighteen months. Failure to obtain concordant figures from several laboratories on exchange samples of final molasses called attention to the necessity for the revision of the routine method. The method of precipitation and the determination of the copper were under strict specifications in all the laboratories to which the exchange sample had been sent, so the indication was that the source of difference lay in the preparation of the solution for analysis.

¹ Browne's "Handbook of Sugar Analysis," p. 443.

² Deerr's "Cane Sugar," p. 468.

GENERAL METHOD

Throughout the investigation the Meissl and Hiller method was employed; 50 cc. of the solution for analysis and 50 cc. of mixed Fehling's solution were heated in a beaker to the boiling point for 4 minutes, and maintained at gentle ebullition exactly 2 minutes. A gasoline burner was the source of heat. At the end of the boiling period 100 cc. of cold, recently boiled distilled water were added and the solution filtered in an alundum crucible or fused silica Gooch crucible. The cuprous oxide was always burned to cupric oxide in an electric muffle at full red heat for at least 30 min. Other methods of determining the copper were employed at times as well, and comparisons are tabulated where this was done.

LEAD ACETATE SOLUTION—The lead solution employed was a 56° Brix solution of normal lead acetate, acidified faintly with acetic acid.

SAMPLE—For the first part of the work a sample of final molasses of the following analysis was used: Brix 87.2, polarization 36.7, Clerget number 42.43, reducing sugars about 11.00 per cent. This was warmed, mixed thoroughly and stored in an air-tight jar. Weights of this molasses were taken so that 50 cc. of solution contained 1 gram of molasses.

AMOUNT OF LEAD TO BE USED—To various portions of solution containing 1 gram of molasses to 50 cc. were added lead acetate solution—0.1, 0.2, 0.3, 0.4, etc., cc., respectively, per gram of molasses. The filtrates were tested with HCl. The filtrate from the portion using 0.5 cc. lead solution showed a slight precipitate. The portion containing 0.1 cc. lead solution filtered slowly, the filtrate showing a precipitate with hydrogen sulfide. This covered the range in which might be included "sufficient lead to clarify," as 0.1 cc. showed an excess of lead in the filtrate (with hydrogen sulfide), and 0.5 cc. was necessary to precipitate everything which could be brought down completely with normal lead acetate. The first series of tests was made using 0.5 cc. lead solution per gram of molasses.

COMPARISON OF ORDINARY DELEADING AGENTS AND VARYING AMOUNTS OF LEAD

TABLE I—WEIGHTS OF CuO FROM 1 GRAM OF MOLASSES
0.5 cc. Lead Solution per Gram of Molasses, Delead with Various Deleading Agents in Solutions, the Amount Indicated Being per Gram of Molasses

Sodium carbonate 2 cc. 10% solution	Potassium oxalate 2 cc. 10% solution	Sodium sulfate 2 cc. saturated solution
0.2547	0.2646	0.2517
0.2547	0.2647	0.2500
0.2561	0.2649	0.2549
0.2525	0.2644	..
0.2515	0.2605	..
0.2521	0.2616	..
Av., 0.2534	0.2634	0.2522

Spaces separate determinations made on different weighings.

The carbonate and sulfate check, but the oxalate gives weights of CuO 10 mg. higher. As the deleading agents all gave considerable precipitates with 0.5 cc. of lead solution, a second set of tests was made using 0.25 cc. of lead solution per gram, one-half the amount used above. This was about the minimum of lead giving a rapid filtration. As sodium sulfate did not form any precipitate with this smaller

amount of lead, its use was discontinued, and a series with dry sodium oxalate was substituted.

TABLE II—WEIGHTS OF CuO FROM 1 GRAM OF MOLASSES
0.25 cc. of Lead Solution per Gram of Molasses, with Decreased
Amounts of Deleading Agents as Shown

Sodium carbonate 1.5 cc. 10% solution	Potassium oxalate 1 cc. 10% solution	Sodium oxalate (dry) 0.1 gram
0.2635	0.2710	0.2703
	0.2704	0.2709
	0.2723	..
0.2658	0.2717	..
0.2664	0.2732	..
0.2689	..	..
0.2638	..	..
Av., 0.2657	0.2715	0.2706

The dry sodium oxalate checks the potassium oxalate in solution, and as before the carbonate figures are much lower than those with oxalates. Comparing the average results with 0.5 cc. of lead solution per gram and with 0.25 cc. lead solution per gram we have:

	Sodium carbonate	Potassium oxalate
0.5 cc. lead solution.....	0.2534	0.2634
0.25 cc. lead solution.....	0.2657	0.2715

The smaller amount of lead gives much higher results with both deleading agents.

Another comparison of the effect of varying the lead, deleading with potassium oxalate gave similar results, as here shown.

TABLE III—DIFFERENT AMOUNTS OF LEAD SOLUTION, DELEADED WITH POTASSIUM OXALATE IN SOLUTION

0.5 cc. lead solution per gram molasses	0.25 cc. lead solution per gram molasses
0.2637	0.2748
0.2626	0.2744
Av., 0.2632	0.2746

THE EFFECT OF KIESELGUHR AS AN AID TO FILTRATION, and a comparison of dry reagents with previous figures with reagents in solution was brought out in the following:

TABLE IV—DRY DELEADING AGENTS, WITH AND WITHOUT KIESELGUHR
All with 0.25 cc. Lead Solution per Gram of Molasses

0.15 gram sodium carbonate, dry per gram molasses		0.1 gram sodium oxalate, dry per gram molasses	
No Kieselguhr	Kieselguhr	No Kieselguhr	Kieselguhr
0.2670	0.2669	0.2748	0.2743
0.2652	0.2642	0.2740	0.2745
0.2670	0.2672		0.2742
Av., 0.2664	0.2663	0.2744	0.2743
Av. with wet reagent,	0.2657		0.2746

The kieselguhr aids filtration and does not affect the results. Dry reagents give check figures with the same reagents in solution. This tabulation again emphasizes the difference between deleading with carbonate and with oxalates.

SUMMARY—Considering now the possibilities under the options offered in "sufficient lead to clarify" and "deleading with carbonate, sulfate or oxalate," the following figures are taken from the foregoing work:

0.5 cc. lead per gram of molasses, deleading with carbonate; average of five check results, weight CuO 0.2534, per cent glucose 10.64.

0.25 cc. lead per gram of molasses, deleading with oxalate; average of seven check results, weight CuO 0.2744, per cent glucose 11.54, showing a difference of about 8.5 per cent on the glucose content of the molasses, using methods both of which are permissible under the directions as ordinarily given.

It is evident that concordant results can be obtained only when the amount of lead is accurately measured, and when the deleading agent is specified. Pending further work, we adopted the use of 0.25 cc. of lead solution per gram of molasses, as this was the minimum amount of lead giving a rapid filtration, and showing an appreciable precipitate with the deleading agent, removing the excess lead with 1 cc. of 10 per cent potassium oxalate solution, or 0.1 gram of dry sodium oxalate, with kieselguhr added to aid filtration. The oxalates were preferred for several reasons. Boerntraeger¹ had raised objections to carbonate, particularly in excess; Sawyer² considered the oxalate preferable; solutions deleading with carbonate, on standing a short time, frequently developed a cloud which settles on the glassware; and finally the oxalate gives the higher figures.

WEIGHING AS CUPROUS OXIDE—Before igniting to cupric oxide, in most cases the cuprous oxide was dried at 105° C. and weighed. Several determinations of the copper present in the precipitates were made by Low's modifications of the volumetric iodide method.³

TABLE V

Cu ₂ O	Cu (calculated)	CuO	Cu (calculated)	Cu by Low's method
0.2532	0.2248	0.2732	0.2183	0.2192
0.2522	0.2240	0.2723	0.2175	0.2176
0.2550	0.2264	0.2742	0.2191	0.2184
0.2449	0.2175	0.2642	0.2111	0.2084
0.2548	0.2263	0.2745	0.2193	0.2187
0.2567	0.2279	0.2740	0.2189	0.2184
Av.,	0.2245		0.2174	0.2168

Weighing as cuprous oxide is evidently worthless. The checks between the copper as calculated from cupric oxide and as determined by Low's titration, show the reliability of the cupric oxide weight.

CLARIFICATION WITH KIESELGUHR ONLY, WITHOUT THE USE OF LEAD

With solutions which are free from suspended matter it has long been the practice with many chemists to dispense with the use of lead or other clarifying reagents. This practice has been objected to on the ground that there might be copper-reducing substances, other than sugars, present in the solutions which would be removed by the use of lead. Noel Deerr⁴ says: "With the use of no clarifying agent, bodies other than reducing sugars which act on copper may be present. Zerban and Naquin have shown that with cane molasses a little more copper is precipitated with unclarified solutions than with solutions clarified with neutral acetate of lead, and the writer is of the opinion that results closest to the truth are obtained with the use of this material."

In the present investigation we have shown that tests made with neutral lead solution may vary between wide limits depending on the amount of lead and the class of deleading agent used. It is possible that most of the work comparing the use of lead and no lead has been done using sodium carbonate to de-

¹ *Z. angew. Chem.*, **1892**, 333.

² *J. Am. Chem. Soc.*, **26**, 1631.

³ Browne's "Handbook," p. 411.

⁴ "Cane Sugar," p. 467.

lead, which gives much lower figures than the oxalate, as we have brought out.

The second phase of the work took up the comparison of the lead and oxalate method already developed, as against preparation of the solution without the use of reagents, using kieselguhr for the removal of suspended matter. Experiment showed that the addition of a few teaspoonfuls of kieselguhr to the molasses solution, after making to the mark, gave a solution on filtering which was quite bright and clear. The filtration may also be effected quite as well by adding the kieselguhr to the filter paper before pouring on the solution.

A new sample of molasses was used, polarization 34.24, Clerget number 40.31, reducing sugars about 21.00 per cent. This sample was warmed, mixed and stored as before. Amounts were weighed to give 0.5 g. of molasses per 50 cc. of solution.

TABLE VI—COMPARISON BETWEEN A SET OF TESTS CLARIFIED WITH NORMAL LEAD ACETATE SOLUTION
0.25 cc. per Gram of Molasses with 1 cc. of 10 Per Cent Potassium Oxalate Solution per Gram of Molasses, and Three Sets of Tests Using Kieselguhr Only, with No Lead or Other Reagent

Lead and potassium oxalate	No lead		
	A	B	C
0.2518	0.2517	0.2478	0.2481
0.2520	0.2517	0.2513	0.2478
0.2538	0.2500	0.2488	0.2505
0.2534	0.2486	0.2509	0.2512
0.2509	0.2491	0.2501	0.2492
..	..	0.2500	0.2496
..	..	0.2494	0.2494
..	..	0.2503	0.2493
..	..	0.2492	..
..	..	0.2510	..
Av., 0.2523	0.2501	0.2499	0.2494

The lead and oxalate gives results higher than the tests where no lead is used. The three sets using no lead agree well with one another and within themselves.

It may be of interest here to note that Series A and B were filtered through alundum crucibles and Series C. through silica Gooch crucibles with porcelain discs. The agreement is good. We found that if alundum crucibles were given a proper treatment the weight remained practically constant through the duration of the test. New crucibles were soaked with nitric acid, washed with hot water, then treated with hot Fehling's solution and again washed thoroughly and finally ignited at red heat in a muffle furnace for at least 30 mins. After use the copper precipitate was removed with nitric acid, the crucible washed thoroughly with very hot water and the ignition carried out as before. Prepared crucibles must be stored in a desiccator to avoid absorption of moisture. The greater part of our work was done with alundum, and in the numerous instances where comparative tests were made using both Gooches and alundum crucibles, the agreement was always very good.

DETERMINING THE BOILING POINT WITH A THERMOMETER—In the tabulations given so far it will be seen that, almost without exception, there is a maximum variation of 3 mg. of CuO among tests made on the same solution. The only phase of the precipitation and collection of the copper precipitate which seemed capable of causing this variation was the determination of the beginning of the 2-minute boiling period. The

usual directions read: "Heat the contents of the beaker to the boiling point in 4 minutes and continue the heating for exactly 2 minutes, maintaining gentle ebullition." Some directions define the beginning of boiling as "full ebullition of the solution." Frequently the solution comes to a boil all at once; sometimes it boils locally for a few seconds. To place this part of the operation on a definite basis, so as to limit the variations in check tests, one of us (Harris) suggested the use of a thermometer to determine the beginning of boiling. The thermometer was thrust through a hole in a metal disc which served as a cover for the beaker in place of the usual watch glass. The lower end of the thermometer reached into the liquid to within 4 or 5 mm. of the bottom of the beaker. The solution was heated at such a rate that the thermometer registered 100° C. in four minutes. The heating was continued for exactly two minutes from this point, a gentle ebullition being maintained as usual. We employed this device in all of our succeeding work, and obtained closer checks than with the old method. Not only is the two-minute boiling period made more definite by the use of the thermometer, but it is much easier to regulate the pre-heating to the required 4 minutes. Local boiling frequently takes place before 100° C. is reached, and occasionally full boiling does not begin until after that point is passed. Under any circumstances, the 2-minute period was timed from the 100° point.

Further work with no lead as compared to lead and oxalate was carried out to determine, if possible, the reason for the higher results obtained where lead and oxalate were used.

TABLE VII—CuO PRECIPITATED BY 0.5 GRAM MOLASSES, TESTS WITHOUT LEAD

With 0.25 cc. Lead Solution and 1 cc. 10 Per Cent Potassium Oxalate per Gram of Molasses; and with 0.25 cc. Lead Solution and 1 g. (Large Excess) of Dry Sodium Oxalate per Gram of Molasses

No lead	Lead and potassium oxalate	Lead and sodium oxalate
0.2492	0.2498	0.2515
0.2486	0.2500	0.2509
0.2492	0.2497	0.2528
0.2481	0.2506	0.2514
0.2482	0.2515	0.2527
Av., 0.2487	0.2503	0.2519

As in Table VI, the use of lead and oxalate gives higher tests than no lead. The excess of oxalate gives still higher figures. Later work, however, did not bear this out.

(The difference between the no lead and the lead and oxalate methods is so slight that frequently there is a greater maximum difference within a series than between the averages of two series. Our conclusions are justified by the constant tendency of the one method to give higher figures than the other.)

TABLE VIII—TESTS WITHOUT LEAD
With 0.25 cc. Lead Solution and 1 cc. 10 Per Cent Potassium Oxalate Solution per Gram of Molasses; and with No Lead, but 1 cc. 10 Per Cent Potassium Oxalate per Gram of Molasses

No lead	Lead and potassium oxalate	Potassium oxalate but no lead
0.2488	0.2512	0.2501
0.2498	0.2497	0.2505
0.2491	0.2493	0.2521
0.2491	0.2503	0.2522
0.2491	0.2492	0.2511
Av., 0.2492	0.2500	0.2512

Again the use of lead and oxalate gives figures higher than the tests in which no lead is used (although in

this particular case the two methods agree more closely than before), but the addition of oxalate alone gives results higher than where no reagent is used. The indication here is that the lead is not the disturbing factor.

The combined precipitates in the second set of tests (lead and oxalate) were dissolved in nitric acid and evaporated with sulfuric acid, but showed no lead.¹

To 100 cc. portions of a solution to which had been added 0.25 cc. lead solution per gram of molasses, and filtered, were added 0.2, 0.5, 1 and 2 g. of dry sodium oxalate. After standing three hours these were filtered and tested with hydrogen sulfide. The lead was in direct proportion to the amount of oxalate used. In the filtrate from the 0.2 gram of oxalate only a trace of lead was shown, but in the filtrate from 2 grams oxalate the precipitate of lead sulfide was quite appreciable. Treadwell-Hall² says, "In an excess of alkali oxalate, many of the insoluble oxalates dissolved, forming double salts."

To determine whether increasing the oxalate with no lead present would increase the results proportionately, a series of tests were made as follows: Four portions of molasses of 5 g. each were made up to 500 cc. with quantities of potassium oxalate in solution as indicated in the tabulation. The lime salts formed a precipitate which was filtered out, using kieselguhr. Tests were run in duplicate. After weighing the ignited cupric oxide, this was reduced to metallic copper in alcohol vapor according to Wedderburn,³ and this copper in turn was dissolved and determined by Low's volumetric iodide method.

TABLE IX—TESTS USING NO LEAD, BUT WITH INCREASING QUANTITIES OF POTASSIUM OXALATE IN SOLUTION

Grams potassium oxalate per 100 cc.	CuO	Cu (calculated)	Cu reduced by alcohol	Low's titration
None.....	0.2473	0.1975	0.1975	0.1977
None.....	0.2467	0.1970	0.1979	0.1974
0.1.....	0.2501	0.1998	0.1995	0.2018
0.1.....	0.2510	0.2005	0.2011	0.2012
0.4.....	0.2499	0.1996	0.2007	0.2208
0.4.....	0.2487	0.1992	0.1992	0.2005
2.0.....	0.2502	0.1999	0.2008	0.2008
2.0.....	0.2500	0.1997	0.2009	0.2002
With lead and delead with potassium oxalate.....	0.2500	..	..	..

The presence of the oxalate gives higher figures than where none is present, but increasing the amount of oxalate does not increase the results. Judging from the volumetric copper, the greater weight where oxalate is present is due to copper actually precipitated and not to some extraneous material precipitated with the copper. The oxalate without lead gives figures corresponding to those where lead is used and the excess removed with oxalate.

From the figures obtained with lead and oxalate, oxalate and no lead, and no lead or other reagent, it seems safe to say that the molasses with which we

¹ The work with no lead covered a period of several months, during which time the molasses sample was not remixed. Stratification and absorption of moisture may account for slight variations between sets of tests by the same method in the different tables. Each table contains all the data for the conclusions drawn from it, and comparisons must not be made between one method as shown in one table and another method as shown in another. Each table should be considered as dealing with a different sample of molasses.

² "Analytical Chemistry," Vol. 1, p. 349.

³ THIS JOURNAL, July, 1915.

were dealing contained no non-sugar copper-reducing substance removable by lead. Since the oxalate without lead gives just as high figures as the lead and oxalate together, the deleading agent and not the lead is the cause of the difference between the no lead method and the method with lead and deleading agent. As suggested earlier in this article, the probable reason for the conclusion that the use of lead for clarification was necessary because it removed copper-reducing non-sugars, arose from the fact that the comparisons with the no lead method were made using lead and sodium carbonate, which gives low results.

SUMMARY

Summarizing the work on final molasses without lead we find that:

Using kieselguhr only, without lead or any other reagent, gives a clear filtrate and consistent results.

The no lead method offers no mechanical difficulties, either in preparing the solution for analysis or in the precipitation and collection of the copper precipitate.¹ It eliminates all chance of the gross errors which we have shown to be possible under the options offered as to the quantity of lead and class of deleading agent. The only objection offered, that there may be present copper-reducing non-sugars which make the use of lead essential, seems to be contradicted by our work with oxalate and lead, and oxalate alone.

Compared with tests made with lead solution and delead with potassium oxalate, the results without lead are slightly lower, 1.5 to 2 mg. of CuO.

Oxalate alone, without lead, gives figures corresponding to those with lead and oxalate, showing that the oxalate is the disturbing factor, and not the lead.

COMPARISON BETWEEN VARIOUS DELEADING AGENTS—

Oxalic acid has been suggested as a deleading agent, as well as phosphoric acid and sodium phosphate. Tests were made with these in comparison with the no lead method. In order to corroborate the earlier work with carbonate and oxalate, these reagents were also included in the comparison.

TABLE X—COMPARISON BETWEEN NO LEAD METHOD AND VARIOUS DELEADING AGENTS. LEAD SOLUTION 0.25 CC. PER GRAM OF MOLASSES

No lead	Lead and 1 cc. 10% potassium oxalate	Lead and 1.5 cc. 10% sodium carbonate	Lead and 1 cc. 10% oxalic acid
0.2507	0.2532	0.2439	0.2538
0.2505	0.2527	0.2424	0.2531
0.2513	0.2516	0.2447	0.2545
..	0.2516	0.2444	0.2541
..	0.2533	0.2418	0.2540
Average, 0.2508	0.2525	0.2434	0.2540

The carbonate method again gives results much lower than the potassium oxalate, and the relation between the no lead and oxalate is as before. The oxalic acid results are higher than any so far obtained. The high inverting power of oxalic acid (18.7 to 100 for HCl) is sufficient to condemn it for routine work.

The solution delead with carbonate was slightly alkaline but showed considerable lead when tested with hydrogen sulfide. That from the potassium oxalate showed a trace of lead. The combined precipitates from the tests using potassium oxalate showed no lead when dissolved and evaporated with sulfuric acid.

¹ With some samples of molasses requiring 1 g. for a test it has since been found that the filtration of the cuprous oxide is somewhat slower than where lead is used.

TABLE XI—COMPARISON BETWEEN NO LEAD METHOD, LEAD AND POTASSIUM OXALATE, LEAD AND PHOSPHORIC ACID, AND LEAD, AND LEAD AND SODIUM PHOSPHATE. LEAD SOLUTION 0.25 CC. PER GRAM OF MOLASSES

No lead	Lead and 1 cc. 10% potassium oxalate	Lead and 0.7 cc. 10% phosphoric acid	Lead and 3 cc. 10% disodium phosphate
0.2483	0.2501	0.2478	0.2505
0.2482	0.2510	0.2479	0.2504
0.2471	..	0.2482	0.2504
..	..	0.2474	0.2491
Av., 0.2479	0.2505	0.2479	0.2498

The agreement between no lead, and lead and phosphoric acid is very close. The sodium phosphate results check those with potassium oxalate. The solutions delead with phosphoric acid and with sodium phosphate were tested with hydrogen sulfide, and neither showed any lead whatever. These are the only cases in which delead solutions were found quite free from lead.

The results using phosphoric acid to delead are so good that the method seems worthy of a more extended investigation than we have been able to give it so far.

Since so many of the deleading agents failed to delead completely, we thought it might be of interest to run a few tests using lead without any deleading agent, *i. e.*, leaving the excess of lead in the solution. The precipitation showed no abnormality and the cuprous oxide was the usual color and filtered without difficulty. Tests were made on two separate weighings of two different molasses samples.

TABLE XII—TESTS USING LEAD SOLUTION, 0.25 CC. PER GRAM OF MOLASSES, WITHOUT DELEADING REAGENT. NO LEAD AND LEAD AND OXALATE FOR COMPARISON

Molasses A	No lead	Lead and potassium oxalate	Lead, without removing excess
	0.2483	0.2499	0.2448
	0.2482	0.2502	0.2459
	0.2471	..	..
Average.....	0.2479	0.2501	0.2454
Molasses B.....	0.3282	0.3298	0.3266

The lower figures where lead is not removed are directly opposite to what had been expected. A possible explanation might be that lead salts of reducing sugars are formed, which the oxalate breaks up, but which are not broken up by the boiling with Fehling's solution. Herein also may lie the explanation for the low results obtained with sodium carbonate. The lead salts of reducing sugars may be precipitated by the alkaline carbonate.

REDUCING SUGARS IN RAW SUGARS

The percentage of reducing sugars (or "glucose") in raw sugars is so low as a rule that fairly large variations in the weight of the proper precipitate make little apparent difference in the result. On the other hand, the amount of raw sugar handled, both in the factory and the refinery, is so much greater than the amount of molasses produced that small percentage differences in the raw sugar glucose content affect the "glucose balance" perceptibly. The desirability of using the same method for both products is evident. The only difficulty which we thought might be encountered with raw sugar was that of filtering a solution of such density (usually 20 grams to 100 cc.) without defecation. However, we found on experimenting with several sugars, including molasses sugars and raws which had deteriorated, that with

kieselguhr, with solutions of the strength necessary for the glucose test, the filtration was sufficiently rapid for practical purposes, and the resulting filtrate quite clear.

A test was made on a raw sugar with fairly high glucose content, comparing the no lead method with clarification with 0.25 cc. lead solution and 1 cc. 10 per cent potassium oxalate solution per 10 g. of sugar. As it has been claimed that weighing as cuprous oxide is sufficiently accurate for raw sugar determinations, the precipitates were first dried and weighed and then ignited and reweighed, in order to obtain some figures on this point.

TABLE XIII—5 GRAMS OF RAW SUGAR TO 50 CC. OF SOLUTION

A—Clarified with 0.25 cc. Lead Solution and 1 cc. of 10 Per Cent Potassium Oxalate per 10 g. of Sugar			
Cu ₂ O	Cu (calculated)	CuO	Cu (calculated)
0.2192	0.1947	0.2342	0.1874
0.2189	0.1944	0.2329	0.1863
0.2212	0.1965	0.2361	0.1890
Average.....	0.1953		0.1876
Per cent glucose..	1.79		1.69

B—Clarified with Kieselguhr Only, without Lead or Other Reagent

Cu ₂ O	Cu (calculated)	CuO	Cu (calculated)
0.2174	0.1932	0.2328	0.1862
0.2179	0.1936	0.2337	0.1870
0.2195	0.1950	0.2353	0.1882
Average.....	0.1939		0.1871
Per cent glucose..	1.79		1.68

The difference between the no lead method and the lead and oxalate method is so slight as to be immaterial. The copper by Cu₂O is appreciably higher than by CuO, the difference being practically as great, calculated to percentages of glucose content, as in the case of final molasses.

Two series of tests with kieselguhr using no lead were run on another sugar. No further comparison with other methods was considered necessary, in view of the extended work already done with molasses.

TABLE XIV—WEIGHTS OF CuO FROM 5 GRAMS OF SUGAR

5 g. of Raw Sugar to 50 cc. of Solution, using Kieselguhr and no Lead	
Two Series of Tests on the Same Sugar	
0.2619	0.2619
0.2615	0.2633
0.2641	0.2661
0.2645	0.2657
0.2656	0.2647
Average.....	0.2635
Per cent glucose..	1.96
Minimum per cent glucose in 10 tests.....	1.94
Maximum per cent glucose in 10 tests.....	1.98

SUMMARY—Clarification with kieselguhr only, using no lead, is quite as practicable with raw sugars as with final molasses, the greater density of the solution offering no difficulty in the filtration. The desirability of having the same method for both products is sufficient to recommend it.

Weighing the cuprous oxide direct without igniting is no more advisable in the case of raw sugars than with molasses.

CONCLUSIONS

1—Results differ with varying amounts of neutral lead acetate solution.

2—Carbonate, sulfates and oxalates are not interchangeable as deleading agents, oxalates giving results from 4 to 5 per cent higher on the weight of copper than where either of the others is used.

3—Kieselguhr only, without the use of lead or other

reagent, gives a clear filtrate, both with final molasses and raw sugar, and the solution offers no mechanical difficulty in the precipitation and collection of the copper precipitate. Without lead the results are slightly lower than where lead and oxalate are used.

4—The use of a thermometer to determine the beginning of the two-minute boiling period reduced the variations between tests on the same solution.

5—Weighing as cuprous oxide gives results about 5 per cent higher, on the weight of copper, than those obtained by igniting to cupric oxide, whether working with final molasses or raw sugars. Volumetric iodide determinations of the copper check the cupric oxide.

6—Under strict specifications as to the quantity and class of reagents, any method for the preparation of the solution for analysis will give results which check within themselves.

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THE DETERMINATION OF THE GELATINIZING TEMPERATURE OF THE STARCHES FROM THE GRAIN SORGHUMS BY MEANS OF A THERMO-SLIDE

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The usual method for determining the gelatinizing temperature of starches requires that small quantities of the material be placed in test tubes immersed in a water bath, the temperature of which is gradually raised; then portions of the starch are removed at intervals for microscopical examination. This has been found a tedious method and the results not so accurate as desired.

The apparatus designed for our work on the starches obtained from the various grain sorghums permitted direct readings so that a large number of tests were made in a comparatively short time; triplicate determinations checked well within one degree. The thermo-slide, Fig. I, was made in this laboratory and may easily be constructed by any one having a little ability to handle tools. The material consists of:

(1) Three pieces of steel $\frac{5}{8}$ in. long, $1\frac{3}{8}$ in. wide and $\frac{1}{8}$ in. thick, with a central opening $\frac{7}{8} \times 2\frac{3}{4}$ in. cut in each piece, approximately 1 in. from each end and $\frac{1}{4}$ in. from each side; two $\frac{1}{4}$ in. nipples are fitted into the steel piece, No. 1, the holes for these being drilled $\frac{3}{4}$ in. from each end; two openings, $\frac{1}{4}$ in. wide, are cut in steel piece No. 2, as indicated in the top view in Fig. II. (2) Two glass slides 1×3 in. (3) Six rubber gaskets, $\frac{1}{8} \times 5$, cut from $\frac{1}{16}$ in. rubber sheets: two of these gaskets are cut to match steel plate No. 2.

The pieces of steel and rubber have two $\frac{1}{4}$ in. holes drilled in each end through which bolts pass to clamp the entire system together. The $\frac{1}{4}$ in. piping is connected by means of nipples fitted into the top plate. The thermometer is small enough to fit inside the T opposite the exit pipe and the opening made water-tight by slipping a piece of tubing over the thermometer and the nipple. The general arrangement of the micro-polariscope, the thermo-slide and

connections for the warm water are illustrated in Fig. I, and further details of the thermo-slide are shown in the drawing, Fig. II.

PROCEDURE—The apparatus is arranged as shown in Fig. I, and the syphon started, then the flow adjusted by means of the pinch-cock so that the water will run at the rate of not over 100 cc. a minute; a second pinch-cock may be used as a total cut-off. The temperature in the flask is brought to $60^{\circ}\text{C}.$, then the heat controlled so that the thermometer in the flask will indicate a rise of 1° every minute. The water bath (flask) and the microscope are placed not over 18 in. apart so that there will be but little difference in the temperatures recorded in the flask and in the slide. This difference should not be over 3° .

A small quantity of dry starch is placed on the thermo-slide, moistened with an excess of water, and a cover

FIG. I

glass placed over it. The low power (16 mm.) objective is adjusted and focused, then the analyzer put in place and turned to the dark field. The water is now turned on and the temperature noted when all the starch granules have lost their polarizing properties. This temperature should not be considered the correct one, but is to be used as a guide.

The slide is now cleaned and a second portion prepared in the same way as has been described. The water in the flask is heated to the same degree which was recorded for the preliminary test; this gives a temperature in the thermo-slide about $2-3^{\circ}$ below that first found, and, consequently, below the gelatinization point. The temperature of the water is