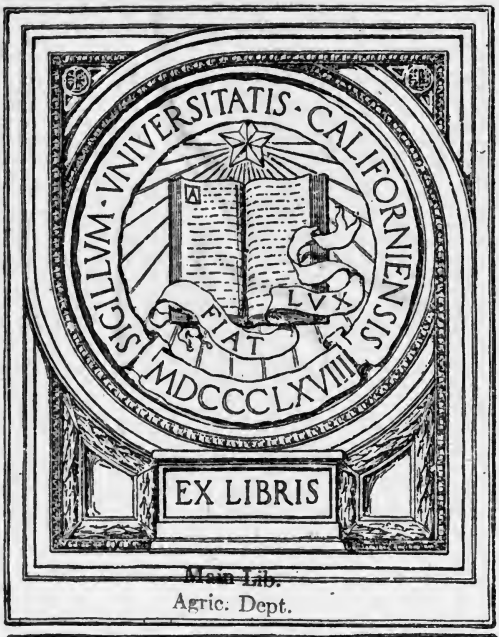

Issued October 30, 1909.

United States Department of Agriculture,

BUREAU OF CHEMISTRY—Circular No. 46.

H. W. WILEY, Chief of Bureau.

THE OCCURRENCE OF PINENE IN LEMON OIL.

By E. M. CHACE,
Chief, Food Technology Laboratory, Division of Foods.

INTRODUCTION.

In the winter and spring of 1907–8 extensive investigations were made by this Bureau in regard to the constituents of certain lemon oils imported into the United States from Sicily. The investigations made by the Bureau at that time led us to believe that the lemon oils referred to were not pure, but were adulterated either by the addition of turpentine or by some manipulation in the method of manufacture. At that time the importers made strong representations to the Bureau that the oils were absolutely genuine. They even went to the trouble and expense of sending a chemist from Messina, who made several visits to our laboratory and collaborated with our chemists and gave us the benefit of his skill. It was decided in order to settle the matter to send Mr. E. M. Chace to Sicily to investigate the method of handling the oil. Mr. Chace accordingly spent several months there making investigations and brought home samples which he could certify to be pure lemon oil. The results of his investigations fully sustain the original contentions of this Bureau.

I believe that the publication of the results of this investigation will be of great benefit not only to the people of the United States but to those of other countries importing or manufacturing lemon oil. I recommend that these results be published as Circular No. 46, Bureau of Chemistry.

Respectfully,

H. W. WILEY,
Chief of Bureau.

Approved:
W. M. HAYS,
Acting Secretary of Agriculture.

WASHINGTON, D. C., *September 15, 1909.*

9965—Cir. 46—09——1

THE OCCURRENCE OF PINENE IN LEMON OIL.

In 1907, in the enforcement of the food and drugs act of June 30, 1906, this Bureau was making examinations of all consignments of lemon oil offered for importation at the custom-houses throughout the United States. Nothing unusual had been observed in these examinations until May of that year, when certain lots of oil were offered for entry at New York which contained substantial amounts of pinene and showed greatly abnormal physical constants. The Board of Food and Drug Inspection detained the shipments in question and requested the importers to show reason why they should not be reshipped on the ground of adulteration.

A preliminary hearing was held in New York in July and a further one requested by the importers before the Board of Food and Drug Inspection at Washington. At the latter the testimony of an Italian chemist was offered on their behalf, the question being mainly whether or not the quantity of pinene found in the oils under discussion could occur in normal oils. The expert from abroad testified that he was an inspector attached to the Chamber of Commerce at Messina, Italy, and that it was his duty, in a general way, to inspect and supervise the manufacture of lemon oil; that he was thoroughly familiar with the situation with reference to the adulteration of oil and had made a study of the question of the presence of pinene in lemon oil for the past six or seven years. Pinene, according to his testimony, occurred in varying quantities, sometimes not being detectable by ordinary methods, at other times being present to such an extent as to be readily detected. He had examined much of the oil at Messina that year and had found pinene in almost all of it. He further stated that distilled oils entered into the final product exported from Messina to the extent of 3 or 4 per cent. It contained more pinene than hand-pressed oil, but would vary the total pinene content of the mixed oils but little. Other testimony by local chemists, and from literature, was offered to prove that pinene in considerable quantities was a normal constituent of lemon oil.

On behalf of this Bureau it was contended that while pinene was perhaps a normal constituent it did not occur in sufficient quantities to be detectable by methods using only ordinary means of distillation; that wherever it had been reported it had been found only after

repeated distillation with special forms of apparatus, and that for some time there had been considerable discussion among experts as to whether or not it ever occurred; that besides the presence of pinene in these oils many of them were otherwise abnormal. These consignments were confined to five importers; in fact, by far the larger part of them to one. The character of the oils had shown, during these months, a marked change; up to and including July 26 they had exhibited various other abnormalities aside from the presence of pinene. After that time, however, the physical constants became more or less normal and the amount of pinene found very much less, although considerable traces were always present.

A second hearing was later given by the Board of Food and Drug Inspection, at which the Secretary of Agriculture was present, and the importers then requested that such of the importations as were held merely because they contained pinene should be released and that the whole question be investigated by the Department at the place of production. The Board finally decided to release those oils which were not greatly abnormal and to send a representative of the Department to Sicily to investigate the conditions there during the season of 1907–8. The writer went to the island of Sicily under instructions from the Chief of the Bureau of Chemistry for the purpose of making an investigation of the methods of production of essential oils and to collect authentic samples from the several districts from which data could be derived on which to base future conclusions.

THE LEMON-PRODUCING DISTRICTS.

The island of Sicily contains five, more or less, distinct lemon-producing districts, and there is also a small area on the adjacent mainland in Italy where they are grown. The lemon belt in Sicily lies along the northern and southern coast lines, never extending inland for any great distance. The most important section lies at the foothills of Mount Etna, extending from Catania on the south to Giardini on the north, and will be called the Etna district. The second district of importance is a continuation of the first, extending from Giardini on the south to Messina on the north, and is named from the latter city. Palermo, the largest city of Sicily, is the center of the third district and gives it its name. The region begins on the east at Ficarazzi and extends westward to Partinico, including the valley of the Conco D' Oro. The fourth, or Syracuse, district lies on the south side of the island, from Avola on the south to Augusta on the north. It extends farther inland than any of the others, is not so mountainous, and has more mild climatic conditions. The fifth district lies between Messina and Palermo, on the north coast. It consists of groups of orchards around individual center towns, the largest of

which is Barcelona. The small mainland district of Calabria may be neglected for all practical purposes, as the output forms a very small

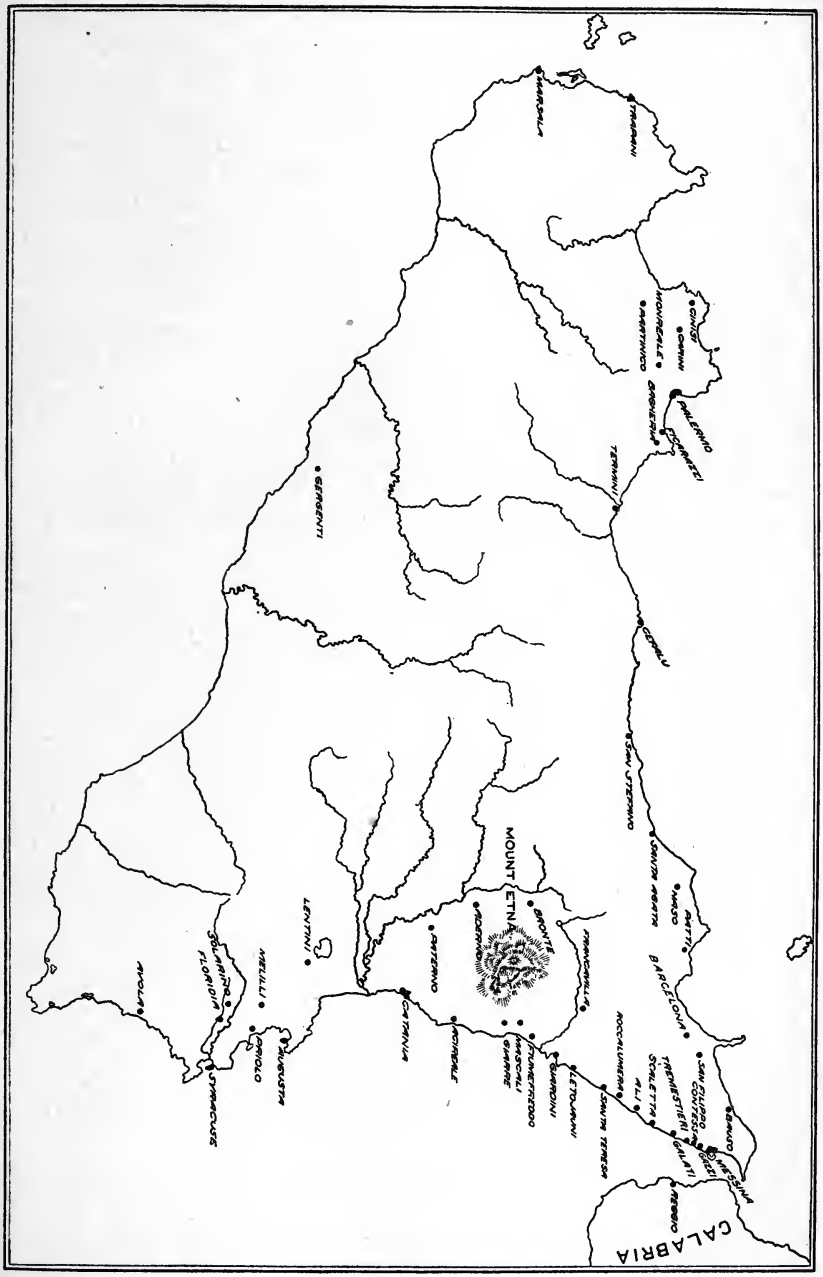

Fig. 1.—Map of Sicily showing the lemon producing districts.

part of the total crop and is only interesting for the reason that the oil is made by machine instead of hand pressed.

COLLECTION OF SAMPLES.

The problem of collecting authentic samples in sufficient number to cover adequately not only the territory, but also the different periods of the season, was one of considerable difficulty. There seemed three possible ways to make satisfactory collections: First, by purchasing the fruit and hiring workmen to produce the oil under such supervision as would preclude the possibility of anything but genuine samples; second, by arranging with the factories to produce, under closest personal supervision, samples of oil from the fruit which was on hand; third, by visiting the factories unannounced and requesting samples at a stage in the manufacturing process where, beyond all reasonable doubt, no adulteration could have taken place.

It would seem, of course, that the first method was preferable to the others, but experience soon showed that it had many disadvantages. It was found practically impossible to hire workmen and space in which to manufacture the samples in other cities than Messina, except at excessive cost in both time and money. If either of the other methods would give the desired results they were much preferable on this account, and by using them it would be possible to collect two or more samples a day. If the first method were employed, the samples would average at most five or six a week, even if the oil were made at a central point. Moreover, the fruit is always somewhat damaged in shipping and very often delayed for some days, and thus abnormal oil might result. The second and third methods were open to the objection that no authentic information could be obtained relative to the fruit used, although indications of abnormality might be revealed by the peel, which could always be examined. It was seldom possible, however, to have samples prepared in the factories. Owners did not care to change the routine of their work for such a purpose, and where such samples were obtained the fruit used was generally that prepared the day before. Fruit cut and worked up immediately yields a much smaller quantity of oil than that prepared in the ordinary way, and this fact makes the possibility of obtaining abnormal results greater. Furthermore, samples of oil taken during the process of manufacture would more nearly represent the average output of a district, as the fruit would be more thoroughly mixed, and samples taken from several bowls in different parts of the pressing room would probably represent lemons from different lots purchased by the factory.

After considering all sides of the question, it was decided to carry out the following plan: First, a large number of samples were collected from the bowls of the factory workmen while the factory was in operation. This assured a sample taken at a stage of the process of manufacture where intelligent adulteration would be impossible, for only very skillful sophistication can be practiced successfully by the

producer at this point, since all the oil must undergo examination by the chemist of the purchasing broker. As such sophistication could only take place after the oil had been separated and measured, it would seem that only genuine samples would be collected in this way, unless, of course, the factory operators had been warned of the inspection and had deliberately adulterated the oil. Since, however, the prevailing opinion among the producers was that this investigation, if fairly conducted, would vindicate them, a great majority of manufacturers were anxious to see that samples obtained from their places were representative and genuine beyond all possible doubt. Many insisted on drawing a sample from their storage tank in addition to the one taken from the working room in order to show that the oils were in no way different.

The factories were visited immediately upon arriving at the town in which they were situated and samples collected as soon as possible, so that the news of what was being done would have little time to spread. Close watch was kept on every move of the factory operators after arrival, and where suspicions were aroused the circumstances were noted, and the sample classed as not authentic in considering the final results. The greater part of the samples were taken in this way, the analyses of 130 of them being included in the average of bowl and authentic samples given in Table XII.

In addition to these, a considerable number of samples were collected which were made in factories under close personal supervision from selected peel. This method was especially followed in the north coast and Palermo districts, owing to the peculiar methods of manufacture employed. Fifteen samples were taken in this way, the analyses being given in Table IV under the head of authentic samples made at factories. In addition to these, nine samples were made by hired workmen in a small laboratory established for the purpose at Messina. Fruit was shipped from the Syracuse, Etna, Messina, and north coast districts and worked up in a manner excluding to an unusual degree the possibility of contamination This work was done late in December and early in January and was not repeated later on account of the length of time required and the injury caused to the fruit by shipment in small parcels. The results of the analyses of these samples are not included in the averages for the reason that they can not be discussed as fully as desired, and the average result is affected by several abnormal oils. All were examined for pinene, however, and the absence of that body in detectable quantities proven in every case. Whenever samples could not be obtained under conditions which seemed to justify their classification as authentic, they were purchased and classed as commercial. The 33 samples thus obtained are given in Table XI.

The inspection work began early in December, and a complete circuit of the district was made in that and the two following months.

After the 15th of March the late Dr. A. S. Cheney, then consul at Messina, continued the collection of samples until the season closed, in May. Doctor Cheney had unusual qualifications for doing this work, being himself a trained chemist, and after a very successful collection had been made his samples were forwarded to Washington and are included with the others.

METHODS OF ANALYSIS.

The same methods of examination were used in the preliminary analysis of these samples as had been used on the oils under dispute,

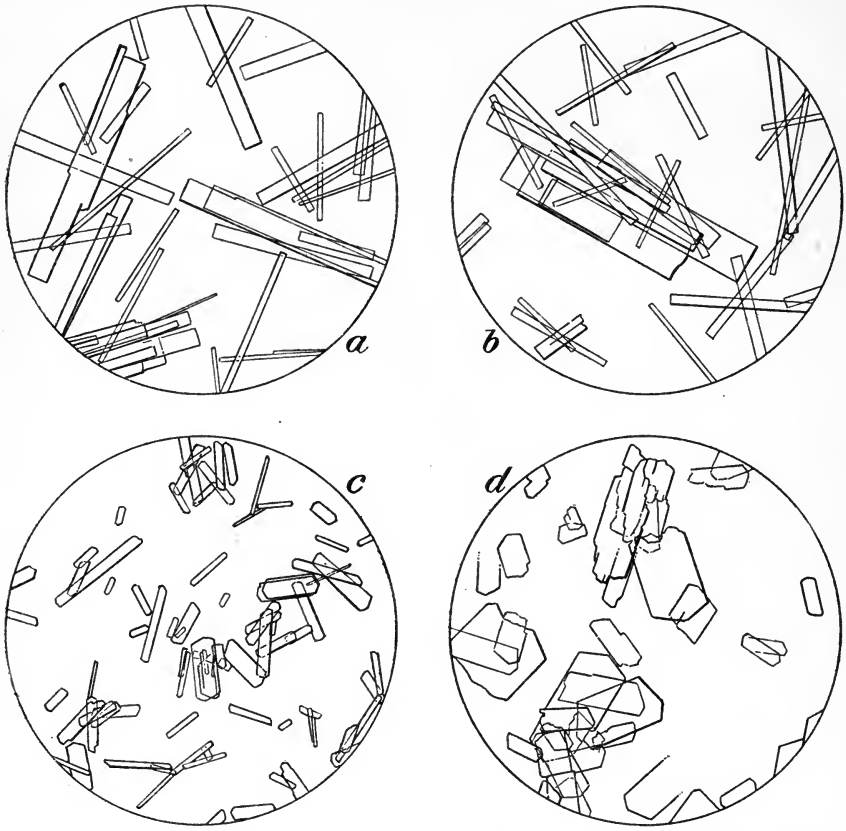

FIG. 2.—Photomicrographs of crystals from lemon oil (× 100). *a*, *b*, Limonene nitroso chlorid crystals from lemon oil; *c*, limonene and pinene nitroso chlorid crystals from a lemon oil mixed with 5 per cent of turpentine; *d*, Pinene nitroso chlorid crystals from turpentine.

namely: (1) Specific gravity at 15.5° C., by means of a picnometer or Sprengel tube; (2) rotation at 20° C., on a Smith and Haensch instrument in a water-jacketed 50 mm tube and conversion of the readings to angular degrees; (3) refractive index on an Abbe refractometer at 20° C.

A 50 cc portion of the oil was then distilled as described in Schimmel & Co.'s Reports for 1898, page 41. The rotation of this distillate was determined at 20° C., and it was then tested for pinene as follows:

The distillate was mixed with an equal volume of glacial acetic acid in a 2-ounce Erlenmeyer flask and immersed in a freezing mixture. Ten cubic centimeters of ethyl nitrite were next added and finally, slowly with constant stirring, 2 cc of a mixture of two parts of concentrated hydrochloric acid and one part of water, all previously cooled. The whole was allowed to remain fifteen minutes in the freezing bath, then rapidly filtered on a Gooch crucible provided with a filter paper disk, using vacuum. The resulting crystals of nitroso chlorid of limonene were dissolved in the smallest possible amount of chloroform and reprecipitated with methyl alcohol. After filtering off these crystals they were mounted with olive oil and examined under the microscope, using a magnification of 100. (See fig. 2.) If present, pinene nitroso chlorid is easily detected by its comparatively broad crystals having irregular pyramidal ends, limonene nitroso chlorid crystallizing in needle shapes or columns.

The citral was determined by the fuchsin sulphite colorimetric method, which at the time of making the analyses was the most accurate method available. The method, in the hands of an expert manipulator, gives results within two-tenths of a per cent of the actual amount present, the error being uniformly positive.

EXPLANATION OF TABLES.

Table I contains the analyses of oils adjudged by the Bureau to be adulterated which were imported between May 17 and July 26, 1907. Aside from the presence of considerable quantities of pinene, the oils are abnormal in other respects.

Table II contains the analyses of oils imported between July 30 and November 17, 1907. They were adjudged to be adulterated chiefly because of the presence of pinene, although, as is noted under "Remarks" in this table, many of the oils are abnormal in other respects.

Table III contains analyses of oils imported between May 29 and October 11, 1907, which were adjudged to be unadulterated, and it was largely on these analyses that the judgment condemning the oils in Tables I and II was based.

Table IV contains analyses of all of the authentic factory samples made as described on page 7.

Table V contains analyses of the oils collected in the Syracuse district from the bowls of the workingmen, as described on page 6.

Table VI contains analyses of oils collected in the same manner in the Etna district, in which is situated the Mascali region. The oils

from this region were reported by the Italian expert as highly abnormal.

Table VII contains analyses of oils collected in the same manner in the Messina district. The oils collected at Bauso on the north coast are included here, although their composition is more nearly like that of Barcelona and Patti oils.

Table VIII contains analyses of oils collected in the same manner in the Palermo district. Both distilled oil and oil from Bastardoni lemons are produced in this district. Presence of the latter may account for the abnormal physical constants observed in some of these samples.

Table IX contains the analyses of oils collected in the same manner in the Barcelona district. Oils from this district were also reported as abnormal and of high pinene content.

Table X contains analyses of oils collected in the same manner at Patti. The oils are similar to those of the Barcelona district.

Table XI contains samples of commercial oils collected throughout Sicily.

Table XII contains analyses of miscellaneous samples of oils which were collected on account of their general interest and the light which they throw on the question at issue.

The analytical work reported in these tables was done by Messrs. H. S. Bailey and C. O. Dodge, with the exception of the determinations given in Tables I, II, and III, which were made in the New York Food and Drug Inspection Laboratory by Messrs. A. F. Seeker and L. D. Havenhill.

TABLE I.—*Analyses of oils adjudged adulterated.*

(Imported between May 17 and July 26, 1907.)

New York port No.	Date received.	Specific gravity at 15.6/15.6.	Rotation of oil at 25° C.	Rotation of 10 per cent distillate.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.	Remarks.
7550	May 17, 1907	0.8610	59.4	56.6	2.8	1.4732	3.1	Very strongly positive.	Refractive index also below normal.
7681	May 29, 1907	.8614	59.6	.....	.....	1.4733	3.5	.....do.....	Do.
7838	June 15, 1907	.8601	58.6	55.3	3.3	1.4729	3.6	.....do.....	Do.
7842	June 18, 1907	.8604	58.1	55.5	2.6	1.4728	4.1	.....do.....	Do.
7840	June 19, 1907	.8591	58.6	56.5	2.1	1.4734	4.7	.....do.....	Do.
7839	.....do.....	.8600	58.6	56.2	2.4	1.4736	4.7	.....do.....	Do.
7941	June 22, 1907	.8596	59.4	56.4	3.0	1.4735	4.7	.....do.....	Do.
8134	July 9, 1907	.8613	[a] 40.6	23.8	16.8	1.4738	[a] 6.6	.....do.....	Grossly adulterated with turpentine.
8152	.....do.....	.8594	58.1	53.3	4.8	1.4740	4.5	.....do.....	Refractive index also below normal.
8153	July 15, 1907	.8605	59.2	55.8	3.4	1.4732	7.1	.....do.....	Do.
8311	July 26, 1907	.8594	59.2	56.3	2.9	1.4740	.....	.....do.....	Do.
Average	.....	.8602	58.8	52.6	4.4	1.4734	4.0		

[a] Omitted from average.

TABLE II.—*Analyses of oils adjudged adulterated.*

(Imported between July 30 and November 17, 1907.)

New York port No.	Date received.	Specific gravity at 15.6/15.6.	Rotation of oil at 25° C.	Rotation of 10 per cent distillate.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.	Remarks.
8355	July 30, 1907	0.8595	58.3	54.4	3.9	1.4745	.....	Positive	Normal but for presence of pinene.
8469	Aug. 8, 1907	.8597	58 5	55.4	3.1	1.4747	.....	do	Do.
8470	do	.8594	59.1	54.0	5.1	1.4746	.....	do	Abnormal as to the 10 per cent distillate.
8492	Aug. 12, 1907	.8589	58.9	53.5	5.4	1.4745	.....	do	Do.
8543	do	.8591	58.9	53.5	5.4	1.4745	.....	do	Do.
8510	Aug. 13, 1907	.8593	59.6	54.7	4.9	1.4747	.....	do	Normal but for presence of pinene.
8511	do	.8589	59.2	54.5	4.7	1.4747	.....	do	Do.
8557	Aug. 19, 1907	.8589	58.9	55.2	4.7	1.4748	4.2	do	Do.
8531	do	.8593	57.6	54.5	3.1	1.4748	4.1	do	Rotation low.
8558	do	.8595	58.8	57.3	1.5	1.4748	4.5	do	Normal but for presence of pinene.
8642	Aug. 22, 1907	.8591	58.9	53.7	5.2	1.4747	3.8	do	Abnormally low citral and low rotation of 10 per cent distillate.
8698	Aug. 26, 1907	.8595	57.4	55.2	2.2	1.4743	3.9	do	Abnormally low citral.
8697	do	.8591	58.3	55.4	2.9	1.4750	4.2	do	Normal but for presence of pinene.
8673	do	.8579	59.1	57.2	1.9	1.4749	4.7	Traces	Do.
8699	do	.8600	57.4	55.1	2.3	1.4743	4.2	Positive	Rotation low.
8700	do	.8599	57.5	54.9	2.6	1.4742	4.2	do	Do.
8701	do	.8595	57.6	54.5	3.1	1.4744	4.5	do	Do.
8764	do	.8603	57.1	55.8	1.3	1.4744	4.6	do	Do.
8858	Sept. 4, 1907	.8579	60.3	57.8	2.5	1.4748	4.6	do	Normal but for presence of pinene.
8859	do	.8579	60.3	58.0	2.3	1.4748	4.6	do	Do.
8872	Sept. 7, 1907	.8580	57.9	56.9	1.0	1.4750	4.9	do	Do.
8873	do	.8587	58.1	57.1	1.0	1.4758	4.5	do	Do.
8833	Sept. 10, 1907	.8593	57.6	54.7	2.9	1.4747	3.7	do	Low citral and rotation.
9079	Sept. 17, 1907	.8585	59.1	55.8	3.3	1.4748	4.8	Traces	Normal but for traces of pinene.
9435	Oct. 7, 1907	.8587	58.6	56.5	2.1	1.4747	4.0	do	Do.
9967	Nov. 17, 1907	.8585	57.7	53.8	3.9	1.4746	.....	do	Normal but for traces of pinene and low rotation.
Average	.....	.8591	58.5	55.4	3.2	1.4747	4.3		

TABLE III.—*Analyses of oils adjudged unadulterated.*

(Imported between May 29 and October 11, 1907.)

New York port No.	Date received.	Specific gravity at 15.6/15.6.	Rotation of oil at 25° C.	Rotation of 10 per cent distillate.	Difference.	Refractive index of oil at 25° C.	Citral.	Pinene test.	Remarks.
7679	May 29, 1907	0.8561	59.5	.....	.....	1.4750	5.1	.....	No pinene test made.
7680	do	.8577	58.9	.....	.....	1.4747	4.6	.....	Do.
7682	do	.8567	60.5	.....	.....	1.4748	4.4	.....	Do.
7782	June 7, 1907	.8593	60.4	.....	.....	1.4737	4.8	.....	Low refractive index. No pinene test made.
7835	June 14, 1907	.8586	58.6	56.6	2.0	1.4747	5.9	.....	No pinene test made.
7836	June 18, 1907	.8577	58.1	56.1	2.0	1.4751	4.9	.....	Do.
7937	June 20, 1907	.8570	60.5	59.3	1.2	1.4748	5.6	.....	Do.
7957	June 21, 1907	.8584	59.3	57.4	1.9	1.4749	6.2	.....	Do.
7958	do	.8576	59.4	57.7	1.7	1.4750	6.7	.....	Do.
8018	June 28, 1907	.8585	59.9	58.1	1.8	1.4745	6.0	.....	Do.
8030	July 5, 1907	.8580	59.7	56.0	3.7	1.4751	[a]7.3	Negative	
8078	do	.8583	59.8	.....	.....	1.4750	6.1	do	
8079	July 6, 1907	.8585	59.3	54.7	4.6	1.4751	6.9	do	
8116	July 5, 1907	.8590	59.6	.....	.....	1.4750	6.2	do	
8117	do	.8581	59.4	.....	.....	1.4750	6.4	do	
8136	July 9, 1907	.8574	60.0	56.3	3.7	1.4745	.....	do	
8137	July 15, 1907	.8585	59.6	56.2	3.4	1.4742	.....	do	
8150	July 9, 1907	.8590	58.3	53.9	4.4	1.4745	.....	do	
8192	July 22, 1907	.8578	58.5	53.6	4.9	1.4745	[a]7.7 (?)	do	
8215	July 11, 1907	.8584	60.3	56.4	3.9	1.4743	.....	do	
8232	July 15, 1907	.8578	59.9	55.6	4.3	1.4743	.....	do	
8266	July 18, 1907	.8589	58.6	55.1	3.5	1.4751	.....	do	
8317	July 26, 1907	.8579	60.0	56.5	3.5	1.4745	[a]8.6 (?)	do	
8335	July 30, 1907	.8585	59.4	56.5	2.9	1.4745	.....	do	
8372	do	.8586	57.6	54.7	2.9	1.4743	.....	do	
8403	Aug. 2, 1907	.8588	60.2	55.9	4.3	1.4750	.....	do	
8452	do	.8585	59.3	54.3	5.0	1.4750	.....	do	
8467	Aug. 12, 1907	.8581	59.4	56.3	3.1	1.4750	.....	do	
8468	Aug. 8, 1907	.8583	59.4	55.2	4.2	1.4749	.....	do	
8530	Aug. 17, 1907	.8588	58.1	56.2	1.9	1.4754	4.3	do	
8674	Aug. 22, 1907	.8585	59.5	58.5	1.0	1.4750	4.5	do	
8763	Aug. 26, 1907	.8587	56.8	54.7	2.1	1.4749	6.5	do	
8831	Sept. 10, 1907	.8584	58.9	58.3	.6	1.4754	4.7	do	
9000	Sept. 11, 1907	.8579	59.8	58.0	1.8	1.4753	4.8	do	
9001	do	.8580	60.0	58.9	1.1	1.4753	4.9	do	
Average		.8582	59.3	56.3	2.9	1.4748	5.48		

[a] Omitted from averages.

TABLE IV.—*Analyses of all of the authentic factory samples.*

Sample No.	District, subdistrict, or town.	Date made.	Date examined.	Condition of sample.	Specific gravity at 15.6/15.6.	Rotation of oil at 20° C.	Rotation of 10 per cent distillate at 20° C.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.
31	Etna, Fiumefreddo	Dec. 28, 1907	July, 1908	Poor	0.8575	61.55	57.93	3.62	1.4754	4.35	Negative.
4	Messina, city of Messina	Dec. 7, 1907	Dec., 1908	Good	.8596	60.57	57.36	3.21	1.4758	6.62	Do.
5	do	do	do	do	.8591	59.37	55.49	3.88	1.4756	6.99	Do.
6	do	do	do	do	.8589	58.54	54.38	4.16	1.4754	7.03	Do.
38	Barcelona	Dec. 31, 1907	do	do	.8566	63.03	58.75	4.28	1.4748	4.99	Do.
39	do	Jan. 3, 1908	do	do	.8557	60.38	54.68	5.70	1.4745	5.35	Do.
104	do	Feb. 4, 1908	do	do	.8575	61.84	57.08	4.76	1.4749	4.77	Do.
56	Palermo, city	Jan. 13, 1908	do	do	.8575	59.34	54.35	4.99	1.4748	5.25	Do.
58	Palermo, Monreale	Jan. 14, 1908	do	do	.8578	59.30	54.24	5.06	1.4749	5.62	Do.
60	Palermo, Ficarazzi	do	do	do	.8586	59.10	54.71	4.39	1.4751	5.58	Do.
63	Palermo, Partinico	Jan. 16, 1908	do	do	.8577	60.18	56.49	3.69	1.4750	6.20	Do.
65	Palermo, city	Jan. 17, 1908	do	do	.8583	59.84	56.68	3.16	1.4752	5.46	Do.
109	Palermo, Ficarazzi	Feb. 12, 1908	do	do	8587	59.97	56.28	3.69	1.4751	5.78	Do.
113	Palermo, Monreale	Feb. 13, 1908	do	do	.8581	58.58	54.00	4.58	1.4750	4.53	Do.
243	Palermo, city	May 18, 1908	do	do	.8586	57.96	53.70	4.26	1.4747	4.96	Do.
	Average				.8580	59.97	55.74	4.23	1.4751	5.57	

TABLE V.—*Analyses of oils collected from bowls of workingmen in factories of the Syracuse district.*

Sample No.	Town or subdistrict.	Date collected.	Date examined.	Condition of sample.	Specific gravity at 15.6/15.6.	Rotation of oil at 20° C.	Rotation of 10 per cent distillate at 20° C.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.
7	Syracuse	Dec. 13, 1907	Oct., 1908	Good	0.8566	61.34	58.71	2.63	1.4753	5.42	Negative.
10	do	do	July, 1908	do	.8571	63.34	60.45	2.89	1.4754	5.05	Do.
14	Avola	Dec. 14, 1907	do	do	.8572	64.99	63.98	1.01	1.4751	5.81	Do.
15	do	do	do	do	.8574	65.34	63.34	2.00	1.4754	5.51	Do.
16	Melilli	Dec. 16, 1907	do	do	.8584	62.08	60.54	1.54	1.4752	5.29	Do.
17	Priolo	do	do	do	.8577	63.09	60.91	2.18	1.4753	4.76	Do.
18	do	do	do	do	.8571	63.66	60.40	3.26	1.4754	5.33	Do.
19	Augusta	Dec. 17, 1907	do	do	.8580	61.78	58.89	2.89	1.4756	5.29	Do.
66	Avola	Jan. 20, 1908	Oct., 1908	do	.8579	62.15	59.58	2.57	1.4748	5.46	Do.
68	do	do	do	do	.8592	63.62	61.19	2.43	1.4744	6.43	Do.
70	Floridia	Jan. 21, 1908	do	do	.8570	64.53	62.80	1.73	1.4744	5.88	Do.
71	Augusta	Jan. 22, 1908	do	do	.8578	64.39	62.53	1.86	1.4747	5.49	Do.
118	Floridia	Feb. 18, 1908	do	do	.8571	62.59	59.18	3.41	1.4748	5.37	Do.
119	do	do	do	do	.8567	61.58	57.87	3.71	1.4745	6.06	Do.
120	do	do	do	do	.8563	64.09	61.14	2.95	1.4748	6.13	Do.
121	do	do	do	do	.8573	62.27	58.47	3.80	1.4748	5.78	Do.
122	Augusta	Feb. 19, 1908	do	do	.8582	64.53	63.25	1.28	1.4750	5.33	Do.
125	Syracuse	Feb. 20, 1908	do	do	.8583	[a]55.90	52.06	[a]3.84	1.4747	6.13	Do.
126	Avola	do	do	do	.8601	62.49	59.01	3.48	1.4745	5.28	Do.
127	do	do	do	do	.8577	59.65	56.13	3.52	1.4747	.....	Do.
128	do	do	do	do	.8561	61.62	58.31	3.31	1.4746	5.56	Do.
201	Syracuse	Mar. 21, 1908	do	do	.8589	[a]55.68	51.96	[a]3.72	1.4750	5.31	Do.
202	Floridia	do	do	do	.8568	62.78	59.55	3.23	1.4746	5.45	Do.
203	do	do	do	do	.8561	63.02	58.94	4.08	1.4744	5.79	Do.
204	Avola	Mar. 23, 1908	do	Poor	.8573	61.91	58.83	3.08	1.4748	5.98	Do.
205	do	do	do	Good	.8577	64.03	62.45	1.58	1.4746	6.10	Do.
206	Augusta	Mar. 24, 1908	do	do	.8577	62.20	59.94	2.26	1.4746	6.00	Do.
230	Syracuse	Apr. 27, 1908	do	do	.....	58.26	54.21	4.05	1.4747	5.48	Do.
231	Floridia	do	do	do	.8584	60.76	56.60	4.16	1.4744	4.59	Do.
232	do	do	do	Poor	.8570	63.00	59.73	3.27	1.4744	5.15	Do.
233	Avola	Apr. 28, 1908	do	Good	.8557	66.28	63.98	2.30	1.4743	5.59	Do.
234	do	do	do	do	.8580	61.41	58.64	2.77	1.4748	5.72	Do.
235	Augusta	Apr. 30, 1908	do	do	.8576	60.60	57.39	3.21	1.4749	4.88	Do.
	Average				.8575	62.69	.....	2.79	1.4748	5.54	

[a] Omitted from averages.

TABLE VI.—*Analyses of oils collected from bowls of workingmen in factories of the Etna district.*

Sample No.	Town or subdistrict.	Date collected.	Date examined.	Condition of sample.	Specific gravity at 15.6/15.6.	Rotation of oil at 20° C.	Rotation of 10 per cent distillate at 20° C.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.
22	Catania	Dec. 19, 1907	July, 1908	Good	0.8572	60.70	57.17	3.53	1.4754	5.38	Negative.
23	Aci Reale	Dec. 22, 1907	Oct., 1908	do	.8571	60.61	56.11	4.50	1.4751	5.27	Do.
24	do	do	do	do	.8576	60.76	57.22	3.54	1.4750	5.72	Do.
25	Carrubba	Dec. 23, 1907	July, 1908	do	.8573	60.73	57.62	3.11	1.4754	4.86	Do.
26	do	do	do	do	.8572	59.57	55.03	4.54	1.4754	5.19	Do.
30	Giarre	Dec. 28, 1907	do	Poor	.8573	59.91	54.97	4.94	1.4753	4.82	Do.
72	Aci Reale	Jan. 23, 1908	Oct., 1908	Good	.8572	61.18	57.04	4.14	1.4745	5.44	Do.
73	do	do	do	do	.8582	61.19	56.75	4.44	1.4745	5.43	Do.
76	Giarre	Jan. 24, 1908	do	do	.8579	60.76	56.46	4.30	1.4744	4.99	Do.
77	Carrubba	do	do	do	.8574	60.51	56.76	3.75	1.4745	5.18	Do.
78	do	do	do	do	.8563	63.14	59.43	3.71	1.4747	5.15	Do.
81	Catania	Jan. 25, 1908	do	do	.8578	61.37	59.01	2.36	1.4748	5.35	Do.
82	do	do	do	do	.8572	61.73	58.90	2.83	1.4748	5.61	Do.
83	Fiumefreddo	do	do	do	.8576	58.65	55.11	3.54	1.4746	4.37	Do.
84	do	do	do	do	.8566	59.94	55.21	4.73	1.4747	5.40	Do.
85	Acarntare	do	do	do	.8572	61.19	57.72	3.47	1.4748	5.43	Do.
87	Giardini	do	do	do	.....	63.87	60.79	3.08	1.4746	5.17	Do.
129	Aci Reale	Feb. 21, 1908	Nov., 1908	do	.8573	62.48	59.51	2.97	1.4750	4.89	Do.
131	Aci Catena	do	do	do	.8588	60.87	56.83	4.04	1.4749	5.67	Do.
132	do	do	do	do	.8583	59.48	54.68	4.80	1.4749	5.64	Do.
133	Aci Reale	do	do	do	.8578	60.75	56.86	3.89	1.4750	5.45	Do.
134	Catania	Feb. 22, 1908	do	do	.8578	60.19	55.95	4.24	1.4748	4.89	Do.
135	do	do	do	do	.8581	61.86	58.82	3.04	1.4751	4.68	Do.
136	do	do	do	do	.8573	60.44	58.62	1.82	1.4750	5.17	Do.
137	Giarre	Feb. 25, 1908	do	do	.8569	60.95	55.97	4.98	1.4746	4.85	Do.
138	Mascali	do	Oct., 1908	do	.8594	59.47	55.70	3.77	1.4748	5.24	Do.
140	Fiumefreddo	do	do	do	.8570	58.73	53.34	5.39	1.4746	4.67	Do.
141	do	do	Nov., 1908	do	.8575	59.93	55.60	4.33	1.4748	4.33	Do.
143	Carrubba	Feb. 26, 1908	do	do	.8576	59.34	54.59	4.75	1.4749	6.10	Do.
145	Giardini	Feb. 27, 1908	do	do	.8575	59.26	55.26	4.00	1.4749	5.79	Do.
146	do	do	do	do	.8567	59.69	55.88	3.81	1.4747	5.14	Do.
208	Carrubba	Apr. 3, 1908	Oct., 1908	Fair	.....	58.43	53.86	4.57	1.4748	.....	Do.
209	Fiumefreddo	do	Nov., 1908	do	.8588	59.87	54.79	5.08	1.4748	5.17	Do.
210	Giardini	do	do	Good	.8572	59.77	55.83	3.94	1.4747	4.45	Do.
224	Catania	Apr. 15, 1908	do	do	.8577	62.48	59.50	2.98	1.4748	4.80	Do.
	Average				.8575	60.56	56.65	3.91	1.4748	5.16	

TABLE VII.—*Analyses of oils collected from bowls of workingmen in factories of the Messina district.*

Sample No.	Town or subdistrict.	Date collected.	Date examined.	Condition of sample.	Specific gravity at 15.6/15.6.	Rotation of oil at 20° C.	Rotation of 10 per cent distillate at 20° C.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.
33	Santa Teresa	Dec. 30, 1907	Aug., 1908	Good	0.8578	61.76	58.13	3.63	1.4757	5.08	Negative.
34	Roccalumera	do	do	do	.8577	61.04	57.26	3.78	1.4753	5.67	Do.
35	do	do	do	Fair	.8576	61.70	57.08	4.62	1.4754	5.28	Do.
88	Bauso	Jan. 27, 1908	Oct., 1908	Good	.8576	61.73	57.68	4.05	1.4748	5.35	Do.
91	Santa Teresa	Jan. 29, 1908	do	do	.8557	63.26	59.65	3.61	1.4744	5.42	Do.
92	do	do	July, 1908	do	.8567	59.02	56.42	2.60	1.4758	5.10	Do.
93	do	do	do	do	.8580	58.96	56.80	2.16	1.4751	6.40	Do.
94	Gazzi	Jan. 30, 1908	Oct., 1908	do	.8596	59.06	56.36	2.70	1.4752	5.70	Do.
95	Tremestieri	do	do	do	.8577	57.25	53.49	3.76	1.4746	6.00	Do.
98	Messina	Jan. 31, 1908	do	do	.8577	61.22	57.04	4.18	1.4745	5.88	Do.
100	Mili	do	do	Fair	.8606	58.37	55.54	2.83	1.4750	5.36	Do.
101	Galati	do	do	Good	.8580	60.00	55.49	4.51	1.4751	4.21	Do.
147	Santa Teresa	Feb. 27, 1908	do	do	.8592	62.09	59.00	3.09	1.4748	5.45	Do.
148	do	do	Nov., 1908	do	.8573	62.67	58.39	4.28	1.4746	5.83	Do.
149	do	do	do	do	.8596	57.82	53.63	4.19	1.4748	5.87	Do.
150	Roccalumera	do	do	do	.8595	56.49	51.98	4.51	1.4751	6.00	Do.
151	do	do	do	do	.8575	57.44	53.46	3.98	1.4749	6.18	Do.
155	Contesse	Feb. 28, 1908	do	do	.8579	57.85	53.55	4.30	1.4745	6.22	Do.
156	Gazzi	do	do	do	.8599	58.69	53.77	4.92	1.4749	5.83	Do.
157	Santa Andre	Feb. 29, 1908		do	.8576	59.38	56.50	2.88	1.4754	5.59	Do.
158	do	do	Nov., 1908	do	.8579	58.96	54.00	4.96	1.4748	5.24	Do
159	Bauso	do	do	do	.8575	62.35	58.35	4.00	1.4746	5.25	Do.
211	Gazzi	Apr. 4, 1908			.8587	58.47	53.92	4.55	1.4751	5.34	Do.
212	Tremestieri	do	Dec., 1908	Good	.8568	60.91	56.72	4.19	1.4744	6.37	Do.
215	Messina	do	do	do	.8576	59.77	54.68	5.09	1.4744	5.54	Do.
222	Bauso	Apr. 14, 1908	do	Fair	.8571	60.84	56.74	4.10	1.4748	4.94	Do.
236	Contesse	May 12, 1908	do	Good	.8592	a 55.70	a 51.19	a 4.51	a 1.4750	5.76	Do.
238	Gazzi	May 14, 1908	do	do	.8585	a 55.99	a 50.98	a 5.01	1.4748	5.89	Do.
239	Tremestieri	do	do	Poor	.8579	57.16	51.92	5.24	1.4745	4.99	Do.
240	Messina	do	do	do	.8576	59.61	55.02	4.59	1.4743	4.65	Do.
245	Letojauni	May 23, 1908	do	Good	.8592	a 54.16	a 49.08	a 5.08	1.4749	6.28	Do.
	Average				.8581	59.78	55.81	3.98	1.4748	5.57	

a Omitted from averages.

TABLE VIII.—*Analyses of oils collected from bowls of workingmen in factories of the Palermo district.*

Sample No.	Town or subdistrict.	Date collected.	Date examined.	Condition of sample.	Specific gravity at 15.6/15.6.	Rotation of oil at 20° C.	Rotation of 10 per cent distillate at 20° C.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.
55	Palermo	Jan. 12, 1908	Aug., 1908	Good	0.8569	59.32	55.39	3.93	1.4752	5.40	Negative.
111	Monreale	Feb. 13, 1908	Oct., 1908	do	.8588	56.83	52.42	4.41	1.4751	4.92	Do.
117	Palermo	Feb. 16, 1908	do	Fair	.8588	58.25	54.38	3.87	1.4750	5.62	Do.
216	Ficarazzi	Apr. 7, 1908	Nov., 1908	Good	.8581	57.35	52.51	4.84	1.4748	5.40	Do.
218	Palermo	do	do	do	.8577	59.32	54.24	5.08	1.4744	5.54	Do.
219	Monreale	Apr. 8, 1908	do	Fair	.8584	58.55	55.07	3.48	1.4749	5.71	Do.
220	Partinico	Apr. 9, 1908	do	Poor	.8579	61.69	58.29	3.40	1.4747	5.53	Do.
221	do	do	do	Good	.8580	61.33	56.40	4.93	1.4745	4.72	Do.
241	Monreale	May 16, 1908	do	do	.8573	54.93	50.01	4.92	1.4747	4.62	Do.
244	Palermo	May 19, 1908	do	do	.8584	56.74	51.33	5.41	1.4744	4.11	Do.
261	do	June 24, 1908	Dec., 1908	do	.8578	58.05	53.67	4.38	1.4744	5.13	Do.
	Average				.8580	58.40	53.97	4.42	1.4747	5.15	

TABLE IX.—*Analyses of oils collected from bowls of workingmen in factories of the Barcelona district.*

Sample No.	Town or subdistrict.	Date collected.	Date examined.	Condition of sample.	Specific gravity at 15.6/15.6.	Rotation of oil at 20° C.	Rotation of 10 per cent distillate at 20° C.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.
40	Barcelona	Jan. 3, 1908	Aug., 1908	Poor	0.8566	64.21	60.73	3.48	1.4750	5.02	Negative.
102	do	Feb. 3, 1908	Oct., 1908	Good	.8570	57.79	51.62	6.17	1.4745	4.34	Do.
166	do	Mar. 7, 1908	Nov., 1908	do	.8579	60.97	55.92	5.05	1.4748	5.10	Do.
167	do	do	do	Poor	.8569	63.12	60.09	3.03	1.4746	4.96	Do.
168	do	do	do	Fair	.8569	59.79	54.93	4.86	1.4745	5.12	Do.
227	do	Apr. 21, 1908	do	Good	.8573	59.33	54.52	4.81	1.4746	5.28	Do.
228	do	do	do	do	.8577	60.76	56.46	4.30	1.4747	6.29	Do.
248	do	May 27, 1908	do	do	.8567	59.73	55.18	4.55	1.4747	4.81	Do.
249	do	do	do	do	.8577	58.29	53.57	4.72	1.4749	4.43	Do.
250	do	do	do	do	.8564	60.07	54.18	5.89	1.4748	4.41	Do.
251	do	do	do	do	.8567	57.54	51.49	6.05	1.4747	4.85	Do.
	Average				.8571	60.14	55.33	4.81	1.4747	4.96	

TABLE X.—*Analyses of oils collected from bowls of workingmen in factories of the Patti district.*

Sample No.	Town or subdistrict.	Date collected.	Date examined.	Condition of sample.	Specific gravity at 15.6/15.6.	Rotation of oil at 20° C.	Rotation of 10 per cent distillate at 20° C.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.
41	Patti	Jan. 3, 1908	Aug., 1908	Good	0.8575	62.73	58.75	3.98	1.4751	5.02	Negative.
42	do	do	do	do	.8572	62.10	57.57	4.53	1.47.2	4.44	Do.
105	do	Feb. 5, 1908	Oct., 1908	do	.8575	60.48	56.56	3.92	1.4746	5.70	Do.
106	do	do	do	do	.8565	63.02	58.44	4.58	1.4747	4.51	Do.
164	do	Mar. 6, 1908	do	Fair	.8567	62.94	59.51	3.43	1.4744	4.23	Do.
165	do	do	Nov., 1908	Good	.8576	60.44	55.50	4.94	1.4746	4.15	Do.
229	do	Apr. 21, 1908	do	do	.8579	59.25	54.41	4.84	1.4749	4.07	Do.
252	do	May 27, 1908	do	do	.8578	58.66	54.38	4.28	1.4746	4.86	Do.
253	do	do	do	do	.8569	60.34	55.49	4.85	1.4745	5.00	Do.
	Average				.8573	61.11	56.73	4.37	1.4747	4.66	

TABLE XI.—*Analyses of samples of commercial oils collected throughout Sicily.*

Sample No.	District, subdistrict, or town.	Date purchased.	Date examined.	Condition of sample.	Specific gravity at 20° C.	Rotation of oil at 20° C.	Rotation of 10 per cent distillate at 20° C.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.	Dealer.
11	Syracuse, Floridia	Dec. 13, 1907	July, 1908	Good	0.8571	63.76	61.95	1.81	1.4752	5.41	Negative	Carneelo Pezzinga.
12	do	do	do	do	.8569	63.56	60.38	3.18	1.4751	4.56	do	Guiseppe Sciontí.
13	Syracuse, Avola	Dec. 14, 1907	do	do	.8564	64.07	61.94	2.13	1.4755	5.97	do	Giovanni de Luca & Sons.
124	Syracuse	Feb. 20, 1908	June, 1908	do	.8578	59.64	56.32	3.32	1.4755	4.49	do	Guiseppe de Luca.
32	Etna, Fiumefreddo	Dec. 28, 1907	July, 1908	do	.8575	61.36	57.72	3.64	1.4754	5.06	do	Papandrea Bros.
79	Etna, Mascali	Jan. 24, 1908	June, 1908	do	.8558	61.00	56.12	4.88	1.4759	5.29	do	Francesco Cucinotta & Son.
80	Etna, Catania	Jan. 25, 1908	Oct., 1908	do	.8581	59.09	54.88	4.21	1.4746	5.94	do	Cucinotta Bros.
139	Etna, Mascali	Feb. 25, 1908	Nov., 1908	do	.8577	60.04	55.38	4.66	1.4748	5.03	do	L. Mauro Giardina.
142	Etna, Fiumefreddo	do	May, 1908	do	a .8605	a 58.58	56.64	a 1.94	a 1.4753	a 4.53	do	Leonardi & Cucinotta.
223	Etna, Catania	Apr. 15, 1908	Oct., 1908	do	.8608	60.32	56.94	3.38	1.4750	4.94	do	Cucinotta Bros.
263	Etna, Giardini	June 24, 1908	Dec., 1908	do	.8576	60.25	55.74	4.51	1.4746	5.62	do	De Pasquali.
264	do	do	do	do	.8579	56.22	50.70	5.52	1.4741	5.39	do	A. Roberto.
265	do	do	do	do	.8575	62.58	59.11	3.47	1.4749	5.11	do	M. Giordano.
89	Messina, Bauso	Jan. 27, 1908	Oct., 1908	do	.8582	61.18	57.57	3.61	1.4746	5.83	Positive	Rosario Ferigno.
90	Messina, Rometta	do	do	do	.8572	61.79	58.28	3.51	1.4746	6.01	Negative	Dominico Revici.
152	Messina, Scaletta	Feb. 27, 1908	Nov., 1908	do	.8597	58.53	54.95	3.58	1.4750	5.68	do	Antonio de Blase.
153	do	do	Dec., 1908	do	.8595	58.32	54.63	3.69	1.4753	5.34	do	Salvate Auditore.
213	Messina, Galati	Apr. 4, 1908	do	do	.8575	59.62	55.57	4.05	1.4746	4.48	do	Vincenzo Rizzo.
214	Messina, Mili	do	do	do	.8589	57.57	53.96	3.61	1.4753	6.46	do	Giovanni Brescullo.
237	Messina, Gazzi	May 12, 1908	do	do	.8592	55.64	50.64	5.00	1.4749	4.81	do	Onifero Garufi.
37	Barcelona	Dec. 31, 1907	Oct., 1908	do	.8560	62.24	57.84	4.40	1.4749	4.92	do	Guiseppe Genovesse.
170	do	Mar. 7, 1908	do	do	.8582	60.47	56.51	3.96	1.4748	5.08	do	Antonino Campo.
226	do	Apr. 21, 1908	do	do	.8581	59.97	56.10	3.87	1.4744	5.56	do	Guiseppe Genovesse.
249	do	May 27, 1908	Nov., 1908	do	.8577	58.29	53.57	4.72	1.4749	4.43	do	Antonio Perdichizi.
62	Palermo, Ficarazzi	Jan. 14, 1908	Oct., 1908	do	.8570	59.82	57.01	2.81	1.4750	5.76	do	Antonio Canizzaro.
114	Palermo, Terrasini	Feb. 13, 1908	do	do	.8585	59.04	55.20	3.84	1.4749	5.05	do	Marco Carollo.
115	Palermo, Partinico	Feb. 14, 1908	June, 1908	do	.8581	59.90	57.27	2.63	1.4758	4.51	do	Girlamo Guercia.
242	Palermo, Ficarazzi	May 18, 1908	Nov., 1908	do	.8601	57.97	54.02	3.95	1.4747	4.80	do	Antonio Canizzaro.
260	do	June 24, 1908	Dec., 1908	do	.8588	55.83	50.85	4.98	1.4746	4.53	do	Do.
262	Palermo, Monreale	June 25, 1908	do	do	.8580	57.12	52.99	4.13	1.4745	5.58	do	Guiseppe Nicolosa.
107	Santa Agata	Feb. 6, 1908	Oct., 1908	do	.8583	62.34	58.83	3.51	1.4748	5.71	do	Constantino Speciale.
108	do	do	do	do	.8584	58.86	54.93	3.93	1.4745	5.09	do	André Speciale.
174	Etna, Aci Reale	Jan. 23, 1908	do	do	.8574	61.19	57.31	3.88	1.4748	5.77	do	Angelo Bello Russo.
	Average				.8580	59.92	56.12	3.82	1.4749	5.26		

a Excluded from average.

TABLE XII.—*Analyses of miscellaneous samples of oils.*

Sample No.	Description.	Date collected.	Date examined.	Condition.	Specific gravity, 15.6/15.6.	Rotation of oil at 20° C.	Rotation of 10 per cent distillate at 20° C.	Difference.	Refractive index of oil at 20° C.	Citral.	Pinene test.	Remarks
161	Calabrian machine-made oil.	Mar. 5, 1908	Dec., 1905	Poor....	0.8575	60.73	59.26	1.47	1.4746	5.58	Negative .	Sample taken from filters.
162	.....do.....	.....do.....	...do.....	Fair	.8586	55.52	52.31	3.21	1.4747	6.73	...do.....	Do.
163	.....do.....	.....do.....	...do.....	Good ...	.8589	58.75	57.04	1.71	1.4747	6.34	...do.....	Sample from stock on hand.
	Average	.....	.....	.....	.8583	58.33	56.20	2.13	1.4747	6.22		
160	Calabrian hand-pressed oil.	Mar. 5, 1908	Dec., 1908	...do	.8583	58.93	54.77	4.16	1.4749	5.29	...do.....	Commercial sample.
	Distilled oils.											
36	Barcelona.....	Dec. 31, 1907	Oct., 1908	Good ...	.8510	64.60	53.64	10.96	1.4729	1.58	...do.....	Sample taken from still.
103	.....do.....	Feb. 3, 1908	...do.....	...do	.8523	60.84	48.19	12.65	1.4725	.84	...do.....	Do.
110	Palermo	Feb. 12, 1908	...do.....	...do	.8522	61.01	51.58	9.73	1.4727	1.58	...do.....	Do.
61	.....do.....	Jan. 14, 1908	...do.....	...do	.8515	61.12	50.87	10.25	1.4727	1.32	...do.....	Do.
	Average	.....	.....	.....	.8518	61.89	51.07	10.90	1.4727	1.33		
	Oils pressed from fece.											
75	Etna, Aci Reale.....	Jan. 23, 1908	Dec., 1908	Good ...	.8575	61.65	57.65	4.00	1.4749	6.20	...do.....	Sample taken from filter press.
99	Messina.....	Jan. 31, 1908	Oct., 1908	...do	.8589	61.11	57.75	3.36	1.4746	5.40	...do.....	Do.
	Oil from "Bastardoni" lemons.											
57	Palermo, Monreale ...	Jan. 13, 1908	Dec., 1908	Good ...	.8574	56.60	50.63	5.97	1.4746	4.59	...do.....	Authentic sample.
	Old oil.											
29	Etna, Carrubba	Dec. 23, 1908	Dec., 1908	Fair	.8610	61.24	56.85	4.39	1.4754	.....	...do.....	Commercial sample prepared at the Di Mauro factory in 1892.
	[a] Average of bowl and authentic samples.	.....	.....	.....	.8577	60.72	.....	3.85	1.4748	5.33		
	Maximum	.....	.....	.....	.8606	66.28	.....	6.17	1.4758	7.03		
	Minimum.....	.....	.....	.....	.8557	54.16	.....	1.01	1.4743	4.07		

[a] 130 bowl samples and the 15 authentic samples made in factories are included.

DISCUSSION OF RESULTS.

In questioning the purity of the samples reported in Tables I and II it would seem that the first point to be considered is the presence of pinene. It will be noted from perusal of the remainder of the tables that the other samples of oil imported during this period gave negative results when tested for pinene. Absolutely no traces of it were found in the 15 authentic samples manufactured under personal supervision at the factories in Sicily, nor was it detected in the 130 samples, designated as bowl samples, from the six districts of Sicily. Moreover, of the 33 commercial samples, it was found in but one, No. 89, purchased at Bauso. The dealer claimed that this sample had been produced the same day and had been taken from the factory to the storeroom situated in another part of the town, but had not been mixed with oil held in stock. This is the only instance in which pinene was detected in the samples collected, and it would seem that the only conclusion possible to draw is that the oil had been adulterated by the dealer. This had been very skillfully done, as the sample is normal in every respect except the considerable quantities of pinene, which class it as on a par with the oils of Table I.

Of the miscellaneous samples, none was found to contain pinene. Nevertheless, four of these were distilled oils, which, according to the testimony of the Italian expert who appeared on behalf of the importers at the hearings above referred to, should be found to contain considerably larger quantities of pinene than the hand-pressed samples. This fact is not borne out by the analyses; for while the oils are highly abnormal in other respects, no traces of pinene were found in any of them.

Aside from the strongly positive tests for pinene, the next most striking feature of the oils of Table I are their abnormally low refractive indices. It will be seen from the entire average of all the samples examined (Table XII) that the average for this determination is 1.4748, the minimum 1.4743, and the maximum 1.4758. For all of the oils in Table I, the refractive index is below the minimum figure in Table XII. None of them is over 1.4740, and the average is 1.4734, or 0.0009 below the lower limit for the genuine oils. The next abnormal point in the oils in Table I is the uniformly low percentage of citral present. But two of the eleven oils have a good content of citral, and to one of these that substance was undoubtedly added, as the other data conclusively show that the sample had been grossly adulterated with turpentine, to such an extent that it could not contain 6 per cent of natural citral. It will be seen from the tables that the average percentage of citral in all of the samples examined was 5.33 per cent, with a maximum of 7.03 per cent and a minimum of 4.07 per cent. The amount of this constituent seems to vary somewhat with the district and the season of the year in which the oil

is produced. In the Messina and Syracuse districts the average citral content is slightly over 5.5 per cent, while in the Etna and Palermo districts it is but little over 5 per cent. The samples from Barcelona and Patti contain less than 5 per cent of citral, the percentage in the latter case being but slightly over 4.5.

In considering the citral content of the oils in question it should be remembered that it is not usual to export from Sicily a consignment of oil produced in one district solely, such consignments, in a great majority of cases, being made up of mixtures of oil from several districts. Three of the oils in Table I contain less than 4 per cent of citral and could have been rejected solely on this ground. A fourth oil containing 4.1 per cent should also be classed with them. Three of the oils contained 4.7 per cent and one other 4.5 per cent, which is an unusually low figure except for oils of the Barcelona and Patti districts.

The specific gravity of the oils, while in every case within the figures usually given for genuine oils, is uniformly high, the average of the 11 being 0.8602, compared with the entire average of 0.8577 and a maximum of 0.8606 and minimum of 0.8557, this figure being unusually uniform on the authentic samples, as the district averages show.

Considering the rotation of the oils, it will be seen that four of them would fall below 58° if determined at 20° C.[a] This figure does not, however, seem to be unusual in the case of oils from the Messina and Palermo districts. The entire average rotation is above 60°, while the lowest district average, that of Palermo, is 58.4°. Individual samples often show lower rotations, although in many instances explanation of their abnormal character is not wanting, as in the case of Palermo oils, where the oil from Bastardoni lemons is largely responsible for low rotations; oils produced late in the season are also often abnormal in this respect.

Turning to Table II, the chief cause of complaint against the oils is the presence of pinene. Of the 26 oils, all but 4 contain pinene in what was considered by the analyst as more than traces. With one exception the refractive indices of these oils are normal and the average is approximately that of the entire average for authentic samples. The citral content is again unusual and uniformly low; three of the samples having below 4 per cent could have been rejected on this abnormality alone. The average of the 18 samples upon which citral was determined is but 4.3 per cent. The specific gravity is again uniformly high, the minimum figure, 0.8579, being slightly above the average of the authentic samples. Some of the samples are unusually low in rotation; in comparing these figures with those on the authentic samples, however, it is to be noted that the rotation of imported samples was determined at 25° C., while that on the authentic samples was determined at 20° C., so that a correction of −1.1° should be made.

[a] Apply correction of −1.1° C, as determinations were made at 25° C.

Comparing the oils of Tables I and II with those of Table III, which are the importations of other firms offered for entry during the same period of time, it will be seen that but two of the latter samples are abnormally low in refraction. One of these oils had not been tested for pinene, and it is possible, of course, that had this test been made it would have been included in the other tables. The second sample is but 1 point in the fourth decimal place below the minimum obtained on the authentic samples. The citral is uniformly much higher, but one sample falling below 4.5 per cent, while the average is approximately that obtained on the authentic samples. The specific gravity is not uniformly so high, the average being but 5 points in the fourth decimal place above that obtained on the authentic samples.

It is not the purpose in this circular to discuss the data on the genuine samples given herewith except as it bears on the question under discussion, and, indeed, it would seem that the case presented is sufficiently strong to need very little discussion. The matter will therefore be closed with a summary of the evidence submitted.

In all, there were 37 lots of oil detained at the port of New York pending the settlement of this controversy. Eleven of these oils, offered for entry between May 17 and June 26, gave very strong positive tests for pinene and were otherwise abnormal. The remaining 26 lots offered for entry between July 30 and November 17, contained considerable pinene, except in four cases, where the analysts reported traces only and were otherwise abnormal.

During this same period of time 35 samples of lemon oil were offered for entry by other importers at New York, 25 of which contained no pinene, the remainder not having been tested for that substance. In practically every case these oils presented normal physical and chemical constants. Fifteen samples of oil manufactured under the personal supervision of the writer in several districts throughout Sicily not only gave no test for pinene, but showed none of the abnormal physical constants which the oils in question gave. Analyses of 130 bowl samples are given, all of which gave negative results in the tests for pinene, and while in some cases certain of the physical constants are abnormal, the great majority of them are closely uniform and in no way present the abnormal figures obtained on the other oils under discussion. Wherever the miscellaneous samples bear on the question at issue, their testimony undoubtedly goes to prove that the oils were adulterated.

The only reasonable conclusion would seem to be that where pinene is found in lemon oil, using only ordinary means of distillation, it is prima facie proof of adulteration, whether or not the physical constants of the oil are abnormal.

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