termined to try fluxing the brown-colored material with a mixture of pure sodium carbonate and potassium nitrate, as we had previously satisfied ourselves that it did not contain the alkali metals. The fusion was taken up with water, acidulated with hydrochloric acid, and evaporated to dryness. Tungstic acid separated; the residue was moistened with acid and again evaporated. It was then digested with acid and left pure tungsten trioxide which was filtered, washed, and weighed. It equaled 88.08 per cent. The chromium oxide in the filtrate, after precipitation and purification, equaled 12.10 per cent. Upon repeating this course with new portions of the substance we obtained results that agreed with those just given, so that we feel satisfied that they represent the true composition of the brown crystals. Taking the mean of the percentages of the two analyses given above as a basis of calculation we deduce as the most probable ratio, $1Cr_2O_3,5WO_3$ or $Cr_2O_3.5WO_3$. The theoretical requirements of this formula are:

	Per cent.
WO_3	88.40
Cr_2O_3	11.59

	Per cent. found.	
WO_3	88.37	88.08
Cr_2O_2	11.75	12.10

Tungstates of chromium that exist[1] are hydrous and have been prepared by double decomposition in the wet way. The salt we describe is, so far as we can ascertain, the first of its class.

UNIVERSITY OF PENNSYLVANIA,
June 15, 1893.

THE METHODS OF TESTING FATS AND OILS.

BY DR. ERNEST MILLIAU, DIRECTOR OF THE GOVERNMENT TESTING LABORATORY, MARSEILLES, FRANCE.

Read before the New York Section May 29, 1893.

THE difficulties of chemical analyses of fatty matters are greatly increased, not only by the slight differences which characterize the various glycerides, but also by the numerous cases of isomerism, and the phenomena of oxidation and fermentation which modify their molecular structures, the resins and the essen-

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[1] *Fremy's Encyclopedie Chimique*, 3. 166.

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tial oils which they contain, the foreign matters which spoil them, the composition of which varies infinitely, according to the nature of the soil from which the plant draws nourishment, the method of extraction, age, and a thousand other causes, needless to mention. Since the methods of communication have been facilitated and exchanges have become more numerous, the different markets of the world have been flooded with fatty matters presenting different characters, though of the same nature as those which industry and commerce have been accustomed to.

We have been led to seek methods of analysis for oils not by their impurities, which vary according to their origin, but by the examination of their principal constituents, which remain practically the same for the same species.

For example, it is now well known that the olive oil of Tunis, Morocco, and other provinces presents often the character of adulterated oils if analyzed without proper precautions or by old processes.

Accordingly most governments and large companies have modified their specifications. It is indispensable for obtaining precise results to submit the material to a purification, preceded and followed by a number of filtrations. By thus removing the impurities, which when present alter the results, we bring back the fatty bodies of the same species to a type which is always practically the same. To reach this end we work sometimes with washing by hot distilled water, sometimes by strong or dilute alcohol, sometimes we refine the fatty matter by the use of caustic soda lye, containing ten per cent. sodium hydrate, employed in the proportion of ten per cent. of the fat. The emulsion is poured upon a saturated solution of sodium chloride, and by the action of gentle heat, the different parts separate and the clear oil holding in suspension insoluble particles of soap rises quickly to the surface.

It is well to note that fatty matter, even when neutral, as has been demonstrated, dissolves notable quantities of alcohol, which must be eliminated if this method of washing has been employed. The nature of the operation can not be fixed definitely; it varies according to the nature of the impurities and the object of the research. In determining the amount of volatile acids we must be contented with simple filtration and when examining the oils of the cruciferae we must not use caustic soda, which changes organic sulphur to soluble sodium sulphide. Having finished the preliminary operations we can, according to circumstances. operate directly on the neutral fat or the fatty acids prepared from it.

The fatty acids collected in the nascent state, that is to say when they rise to the surface in a pasty mass, have much stronger chemical affinities than when melted and dehydrated. This difference is particularly remarkable in the examination of cotton-seed and sesame oils, the black and red colorations obtained very clearly with a mixture of five per cent. upon the nascent fatty acids are not visible with the same fatty acids when melted.

That purification skillfully made does not destroy the chemical characters of the oil, is easy to verify by treating comparatively the same oil containing five per cent. of the fatty matter the presence of which we wish to demonstrate. The reactions of the added material are in general intensified by these different treatments.

By working upon the fatty acids of the neutral oils I have been able to demonstrate the purity of certain fatty matters which appeared adulterated and in which the presence of cotton-seed and sesame oils was indicated by the old processes, which I have been able to revise. (*Circulaire du ministre de la marine français, en date du 26 julliet, 1892. Rapport du Resident général de Tunisie, en date du 9 mai, 1892. Circulaires des ministres du commerce et de l'agriculture. etc.*)

I well know that in practice these preliminary operations may appear very tedious, but I do not advise their use except in cases where adulterations have been indicated by the usual methods, and it is desired to make the proof positive.

Besides I cannot understand why a chemist has always been expected in the analysis of such delicate materials as fats to discover an infallible reagent. a single drop of which will immediately turn olive oil green, arachide black, sesame red, etc., so that any inexperienced man could discover adulteration and bring the perpetrator to justice. This requirement of the most difficult part of chemical verification, is absolutely incomprehensible and is only to be excused by the ignorance of those who ask it.

We will now proceed to the study of each oil in particular. We will pass rapidly over those which possess only a secondary interest so as to lay greater stress upon those which are the object of a great number of sophistications as for example, olive oil. But in the first place we will indicate rapidly the general processes physical and chemical which are used for the identification of the various fats.

GENERAL METHODS.

(1) *Specific Gravity.*—Is taken with accuracy by the Mohr balance, which is too well known to need description. It must be borne in mind that the specific gravity of the same fat varies greatly with individual samples, and this physical character is not sufficient to allow us to decide on purity or adulteration.

(2) *Action of Nitrous Fumes.*—We owe to a Marseilles gentleman, M. Pontet, the first serious process to recognize certain adulterations of olive oil. It was supposed especially to discover the presence of poppy-seed oil which at that time was a very frequent adulteration and for which the process gave good results.

It is based upon the transformation of the oleine to its isomeric modification elaidine by the action of nitrous fumes. It has been modified by other chemists, Messrs. Boudet, Fauré, and Cailletet. The process of M. Cailletet was most sure and simple and we use it in the following manner:

We take a tube ten cm. long, 2.5 cm. wide, in which are twenty cc. of the fat to be analyzed. Six drops of pure sulphuric acid at 66° B., are added, the tube shaken one minute, and then nine drops of nitric acid C. P. 40° B. are added, after which the tube is again shaken and then plunged in boiling water, where it is left exactly five minutes, after which it is cooled in a water bath at 8°–10° C., whence it is taken at the end of two hours, and the condition of the mass observed. It is well to note the different colorations obtained, first after the addition of the sulphuric acid, second after the addition of nitric acid, third after removal from the water bath and fourth after chilling.

(3) *Sulphuric Saponification.*—M. Maumené proposed to observe the rise of temperature produced by rapidly mixing sulphuric acid with oil. The manner of applying the process in our laboratory is as follows: Fifty grams of the fat for analysis are weighed into a conical glass of 100 cc. capacity. The temperature is noted and ten cc. sulphuric acid 66° B. at the same temperature as the oil is added. The two liquids are stirred together for one minute. An accurate thermometer is then immersed in the upper portion of the mass, stirred slowly, and the maximum temperature noted. The initial temperature must be at least 20° C. In order to obtain exact results it is well to make several determinations and take their mean when the variation does not exceed 2° C.

To obtain the relative sulphuric saponification, we note the rise of temperature obtained with fifty grams of distilled water at the same temperature as the oil and ten cc. of the same sulphuric acid. The number of degrees obtained by the oil is multiplied by 100 and the product divided by the number of degrees obtained with water. This process has the advantage of giving results, nearly constant, with acids of somewhat different strength. I have tried the application of the sulphuric saponification with semi-solid vegetable oils by working at 2°–3° above their melting points, and the results have been very satisfactory, especially for palm-nut and cocoanut oil, because the differences between them and the fluid oils often amount to 45° or more,—as between palm-nut oil and sesame oil, for example.

(4) *Iodine Number.*—In the fatty bodies there are members of unsaturated series, as for example oleic acid, which can absorb into its molecule as many atoms of the halogens as there are lacking atoms of hydrogen for complete saturation.

The iodine number of olive oil varies between eighty and eighty-four, that of arachide or peanut oil is ninety-seven, while that of cotton-seed oil is 108. These differences then allow, within certain limits, the determination of the purity of the various fats. We apply the Hubl method as follows:

Solutions required:

	Grams per liter.
Alcoholic solution of iodine	50
Solution of sodium thiosulphate	24.8
" " mercuric chloride	60
" " potassium iodide	100

Five (5) grams of fatty acids are weighed out, diluted to 100 cc. with ninety-two per cent. alcohol, ten cc. of the solution are taken, and to it added twenty cc. of the iodine solution and fifteen cc. to twenty cc. of the solution of mercuric chloride. The flask is closed and allowed to stand three hours. Twenty cc. of the potassium iodide solution is added and the excess of iodine titrated. When the brown coloration begins to disappear a few drops of starch solution are added and then thiosulphate until the color disappears.

We will pass in silence the other indices, such as that of acetyl, which is of use only to discover castor oil.

(5) *Freezing Point.*—The freezing point is easy to determine by means of a thermometer and a freezing mixture.

(6) *Melting Points of Fatty Acids.*—The dry and melted fatty acids are sucked into a capillary tube. After solidification the tube is placed beside the bulb of a sensitive thermometer and immersed in a beaker of water, the temperature of which is raised very slowly The reading of the thermometer is taken the instant the body passes from the solid to the liquid state.

(7) *Solidification Point of Fatty Acids (Titer).*—The dried and melted fatty acids are placed in a tube fifteen cm. long and two cm. in diameter, which is suspended in a wide-mouthed bottle with a perforated stopper. A thermometer graduated to tenths is inserted so that the bulb reaches the center of the material. At the moment solidification commences a circular movement is given to the thermometer, stirring the whole mass; the thermometer is then left at rest and carefully watched till the mercury ceases to rise, the reading then taken gives the point of solidification or titer conventionally adopted.

(8) *Saturation.*—We operate on five grams of the melted and dried fatty acid with a solution of normal caustic soda. The number of cc. absorbed gives the saturation number.

(9) *Solubility in Absolute Alcohol.*—We determine this in the

following manner: The fat is neutralized by agitating in a closed separating funnel for thirty minutes with twice its weight of ninety-five per cent. alcohol; after settling it is drawn off and the alcohol held in solution driven off at low temperature, after which it is agitated at 15° C. or at a few degrees above the melting point, if solid at 15° with twice its weight of absolute alcohol. A known quantity of this alcohol is evaporated, the residue weighed, and the quantity of oil dissolved by 1,000 grams of alcohol is calculated.

FLUID VEGETABLE OILS.

Olive Oil.—Olive oil, like other vegetable oils, is a mixture of neutral glycerides, oleine, stearine, palmitine, etc., and a variable quantity of free fatty acids. It is the edible oil *par excellence*, and the south of France owes a portion of its prosperity to the cultivation of the olive tree. This culture is to-day almost precarious in certain countries, though the consumption continually increases, and olive oil always enjoys the just title of uncontested superiority. We can only attribute these remarkable results to the adulterations made for the purpose of selling under the name of olive oil, seed oils, or adulterated olive oil. We would be easily freed from these adulterations if chemists had at their disposition absolutely sure means to recognize them. Unfortunately the similarity of composition and reaction of the various vegetable oils render the demonstration of their presence extremely difficult. In face of the prejudice caused by adulteration the greatest efforts have been made to solve the problem, and although the results have not been sterile, they have not yet reached a satisfactory solution. It is necessary to say that if certain adulterators succeed in selling seed oils disguised as olive, they reverse the problem for the chemist by making him seek olive oil in seed oil. I may remark that the fraud is not always so great; very often the proportion of seed oil is not higher than ten per cent. Consequently for our method of analysis to be considered absolutely good it must give results, without recourse to comparisons, which satisfactorily show five per cent. in a mixture. Under these conditions we are certain to discover adulteration when made in the proportion of ten per cent.

Let us rapidly review the general processes for the recognition of peanut, sesame, cotton-seed, and poppy oils in olive oil, and we must acknowledge that none of them demonstrate less than ten per cent.

Specific Gravity.—The specific gravity of olive oil varies from 0.915 to 0.918. We have 0.917 to 0.918 for undecorticated peanut, 0.921 for decorticated peanut, 0.923 for sesame, 0.921 to 0.924 for cotton-seed oil, 0.924 for poppy oil. These variations of density in the olive oil do not allow us to recognize a mixture with any degree of certainty.

Action of Nitrous Fumes.—When taken from the water bath, if the oil contains a fairly large proportion of peanut oil, it will appear wine red. Pure olive oil is, on the contrary, lemon yellow. When taken from the cold bath a complete solidification is observed with pure olive oil, which has the appearance of very light fresh butter. With a mixture of fifteen per cent. of all other oils there is no solidification. This method gives good means of detecting peanut and poppy oils in edible, but is of less use when applied to oils used for industrial purposes, which may not solidify though pure.

Sulphuric Saponification.—The rise of temperature of olive oil is 35°, the relative 94°. A rise of temperature above 35° usually indicates adulteration. Equal or lower results do not absolutely indicate purity because certain pure olive oils give only 31°, 32°, and 33°, and in consequence after the addition of seed oils show only 34°-35°.

Indices.—The iodine number varies from eighty to eighty-five, and in a certain measure helps us to discover seed oils. It likewise offers us a quantitative method with a mixture of known oils. The variation in composition of the same oil spoils its sensitiveness for determining mixtures from five to ten per cent.

We will not mention the other general processes, but will dwell particularly on the special reactions which characterize the presence of the three oils most frequently used to adulterate olive oil, to wit, peanut, sesame and cotton-seed.

These processes have a much greater analytical value than the general ones; for if in the course of analysis we find unsatisfactory results with the latter, we can detect adulterations with certainty by the former.

Examination for Peanut Oil.—The density is almost the same. The sulphuric saponification, and above all the Cailletet process give us good results, but only the presence of arachidic acid $C_{22}H_{44}O_2$, melting point 75°, allows the sure recognition of the presence of peanut oil. We have adopted with slight modifications the method of Renard. Twenty grams of the oil are saponified by 20 cc. of a caustic soda solution 36° B., diluted in 100 cc. alcohol ninety per cent. The soap formed is precipitated by a fifty per cent. solution in alcohol of lead acetate, which must be neutral. After complete precipitation decant while warm and wash the residue with alcohol, which after being ground in a mortar is agitated with 200 cc. of ether. This operation is repeated three times to remove the last traces of lead oleate, soluble in ether. The residue is then put in a porcelain dish containing two or three liters of distilled water and fifty cc. hydrochloric acid. When decomposition is completed the solution is decanted and the fatty acids washed with distilled water, after which they are dried in an oven to remove the last traces of water, when they are dissolved in forty cc. of ninety per cent. alcohol. A drop of hydrochloric acid is added, and the mixture chilled to 15°. Peanut oil gives a generous deposition of arachidic acid crystals. These are washed twice, using twenty cc. each time of ninety per cent. alcohol, then three times with twenty cc. each time of seventy per cent. alcohol, in which arachidic acid is completely insoluble. The washing is complete when a few drops give no residue on evaporation. The acids are warmed slightly and treated with boiling absolute alcohol. After filtration the alcohol is evaporated in the oven at 100° till the weight of the residne remains constant. If the melting point of the residue is between 73° and 75° we can affirm the presence of peanut oil. We must be sure of the freedom of the fatty acids from all traces of oleic acid, which prevents their crystallization. This elaborate but truly scientific process must be carried out only by skilled hands.

Examination for Sesame Oil.—To recognize sesame oil in olive oil we can use the specific gravity, the sulphuric saponification and Cailletet process, but especially the method which we use in our laboratory, and which consists of the reaction of hydrochloric acid and sugar upon the fat.

We do not operate on the glyceride, but on the derived fatty acids. If the oil is operated on directly we can obtain a red or pink coloration with a perfectly pure olive oil. I have frequently observed this coloration in my laboratory with olive oils from Tunis, Algiers, Molfetta, Bitonto (Italy), and more rarely with those from Provence. This coloration comes from the coloring matter dissolved in the juice which flows out along with the oil from the presses. This can be shown by treating the separated juice with hydrochloric acid and sugar with which it gives a coloration exactly resembling that given by sesame oil. It is, therefore, of the highest importance to work with pure fatty acids according to the process I am about to describe and which has been called the Millian process.

Method of Operating.—We saponify fifteen cc. of the fat under examination with ten cc. of the solution of caustic soda, 36° B., with the addition of ten cc. ninety-two per cent. alcohol. When the boiling mixture becomes clear we add 200 cc. hot distilled water and boil to expel the alcohol. Then decompose with ten per cent. sulphuric acid. The fatty acids are removed from the surface in the pasty state, washed by shaking in a test tube with cold distilled water, after which they are heated in an oven to 105°. When the greater part of the water is eliminated and they commence to melt, we pour them on half their volume of pure hydrochloric acid which has been saturated in the cold with finely pulverized sugar. The mixture is shaken violently in the test tube. The presence of sesame is always distinctly indicated by the rose or red coloration of the acid solution. Other oils leave the acid colorless or communicate to it a slightly yellowish tinge. This reaction is extremely delicate and permits the sure recognition of the presence of one per cent. of sesame oil, not only in olive oil, but in all fatty mixtures as well as in soap.

This process was presented before the Academy of Sciences by M. Debray, February 20, 1888, and was awarded the gold medal by the Société d'Encouragement (Report of M. Muntz in the name of the Agricultural Committee, February 20, 1889).

Examination for Cotton-Seed Oil.—Till recently the detection of cotton seed in olive oil was considered impossible, and accordingly the different scientific societies and several chambers of commerce promised great prizes to the inventor of a process which would detect this adulterant. Two simultaneous methods were proposed, one by an Italian savant, M. Bechi, the other by myself. These two processes, which at first sight appear quite analogous and based upon the same reaction, differ completely from the scientific standpoint as well as in the exactness of the results obtained. M. Bechi has based his process upon the direct action of silver nitrate upon the oils. As long ago as 1878 we made some experiments with M. Puget of a similar nature, and the uncertainty of the reactions led us to abandon them entirely. M. Bechi's process possesses the serious inconvenience of causing us at times to reject, as adulterated, oils which are absolutely pure, and thus work injustice and create a considerable prejudice against the firms which have sold them. From results obtained at L'Institut National Agronomique de Paris as well as in other laboratories a deep coloration has been found with oils absolutely pure. Outside of these inconveniences, which are sufficient to cause us to throw it aside, it has the defect of being based upon a coloration having no well-defined chemical character, and it produces effects with different substances the action of which it is impossible to explain. The use of colza oil has the serious fault of bringing into the reaction a second oil which may be impure and completely nullify the results. Finally, the conclusions reached by the Italian scientific commission instituted at Rome by act of the ministry to study the Bechi process show that it is not certain on quantities of less than fifteen per cent. It solves the problem of finding less than ten per cent. no better than the Cailletet process, which shows the presence of fifteen per cent. The same commission declared also that olive oil containing glycerine, free fatty acid, formic and acetic acids, does not give a sure reaction with the Bechi reagent. Since all olive oils contain free fatty acid from several tenths of one per cent. for virgin oil to 100 per cent. in some industrial oils, what conclusions can we arrive at?

The Bechi process will give, from fifteen per cent. up, a brown coloration of variable intensity. But in oil containing cotton-seed oil, which is fresh and well-refined, the only sort used for edible purposes, we have a very weak reaction; while with an olive oil perfectly pure, but containing organic or mineral matters in suspension or solution which have an action upon the reagents, we would have a pronounced coloration. That is why I gave up these uncertain and variable results obtained in 1878 and studied the action of nitrate of silver, not upon the oil itself, but upon the products of saponification derived from it. The results obtained exceeded my expectations and have been published by the press as the Milliau process.

Milliau Process, Mode of Procedure.—In a porcelain dish of 250 cc. capacity fifteen cc. of the oil to be investigated is heated to about 110°; a mixture of ten cc. of a solution of caustic soda at 36° B., and ten cc. of alcohol at 90° B. is then slowly poured on the oil. When the mass boils it becomes clear and homogeneous; 150 cc. of hot distilled water is added and boiling continued to expel the alcohol. The fatty acids are displaced with ten per cent. sulphuric acid solution added in slight excess, and the pasty acids immediately collected with a small platinum spoon. They are washed by shaking several times in a test tube with an equal volume of cold distilled water, the drops of water are drained off and they are then poured into a tube 2.5 cm. in diameter and nine cm. long; fifteen cc. of alcohol, ninety-five per cent., and two cc. of three per cent. nitrate of silver solution are added. The tube is protected from the light in a water bath at 90° C. till about one-third of the alcohol is expelled, which is replaced by ten cc. distilled water. The heating is continued a few minutes and the coloration of the insoluble fatty acids is observed. The presence of cotton-seed oil in any proportion whatever causes a mirror-like precipitate of metallic silver, which blackens the fatty acid of the mixture.

The members of the agricultural committee, composed of the greatest savants of France, MM. Pasteur, Eug. Tisserand, Boitel, Chatin, Heuzé, Hardi, Rissler, Schloesing, Ronna, Lavalard, Müntz, Prillisux, Muret, Bon Chénard, Liebaut, after having tested the process on 128 samples from different provinces have had the kindness to confer upon me the gold medal for this method of analysis, and I will take the liberty to present the conclusions of their secretary, M. Müntz, director of the laboratories of L'Institut National Agronomique: "The preliminary saponification applied to the examination of fatty matters constitutes a new method on which we can confidently rely, not only in the analysis of edible products, but besides of those used for industrial purposes, and also for soaps. It has for effect not only to give an extremely sensitive reaction, but it also eliminates accidental and secondary products which can mask the reaction and deprive it of precision. Feb. 1889."

It is of course well understood that the same processes can be used for the detection of peanut, sesame, and cotton-seed oils in all other fatty mixtures.

Peanut Oil.—Peanut oil apart from the arachidic acid which it contains presents no special characters.

Sesame Oil.—The purity of sesame oil may be demonstrated by shaking ten cc. of oil, first with five drops of sulphuric acid at 53° B., then with five drops of nitric acid at 28° B. The oil undergoes a progressive change of color, through various shades from light-green to red; the final red coloring matter obtained by this oxidation process turns yellow by the action of alkalies and returns to its original color with acids. This curious phenomenon does not take place with other oils, but is obtained sometimes when working on sesame oils slightly adulterated; besides the industrial oils from hot pressing may give, though pure, negative results. I have devised a rapid process, especially applicable for the recognition of castor oil in the industrial sesame oils.

Milliau Process.—For the rapid identification of castor oil and sesame ten grams of the oil are shaken up with four drops of sulphuric acid at 66° B., a drop of nitric acid at 40° is added and shaken violently; pure sesame oil blackens immediately, while that containing castor oil remains turbid yellow.

Cotton-seed Oil.—From a chemical standpoint we have nothing particular to say. From the industrial point of view we may state that this product becomes more useful every day, that its cheapness allows everybody to use it, and this fat material daily augments its important place in the markets of the world.

We will go rapidly over the drying oils, poppy-seed, linseed, walnut, cameline, over those of the cruciferae, colza, rape, and mustard, as well as the oils of the sweet almond, hazel nut, and castor.

I will take the opportunity, however, to mention a rapid process which I have found for detecting the presence of castor oil in other oils. It is well known that the oils in general are soluble in petroleum ether, while castor oil, which distinguishes itself by its solubility in alcohol is likewise remarkable for its insolubility in petroleum ether. Unfortunately this insolubility disappears at the ordinary temperature when castor oil is adulterated wlth a small proportion of a soluble oil.

It is only necessary to demonstrate its insolubility by shaking in a test tube one volume of the oil to be examined with two volumes of petroleum ether, and cooling the mixture to minus 16° C., at the end of a few minutes the mass coagulates and the oil separates if castor oil is present, while the liquid remains homogeneous if it is pure. This phenomenon is especially curious because it will be noted that the freezing point of castor oil unmixed with petroleum ether is much lower than the majority of other oils.

SOLID VEGETABLE OILS.

Cocoanut and Palm Kernel Oils.—The production of these oils, which is continually increasing on account of their great value for the manufacture of soap during the last few years, has been made the object of several adulterations. Independently of the general, chemical, and physical characters of these oils, as well as the sulphuric saponification which I have mentioned, we can employ with good results the determination of the saturation equivalent with normal caustic soda solution.

These oils, containing a greater amount of the lower fatty acids, require, for their saturation, a larger quantity of sodium oxide. It is only necessary to determine the number of cc. of normal solution required for the saturation of five grams of the pure fatty acid to recognize these oils,—cocoanut oil 24.1, palm kernel oil 22.5, while the average of the other liquids is seventeen or eighteen cc. only. This process presents some inconveniences, especially when trying to discover small quantities of adulteration, because it must be noted that the adulteration of these oils is only practiced within certain limits, otherwise their appearance would lead to its detection. Besides, when an oil is made from old or spoiled seeds the saturation is a little less.

Since the detection of adulterations in these oils depends on variations of a few tenths of a centimeter which might be caused by the variation of the oils themselves, or the method of operating, it is necessary to be very circumspect in the use of this truly scientific process.

With palm kernel oil this error might be greater because it is customary to balance the low saturation of the seed oils by the addition of cocoanut oil. As, for example, five grams of fatty acids from a mixture containing forty-five per cent. of palm kernel oil, forty-five per cent. cocoanut oil, and ten per cent. peanut oil, require precisely 22.5 cc. normal solution for their saturation.

Iodine Number.—This determination is very useful because these oils being very rich in saturated acids absorb much smaller quantities of iodine than most other oils. For example, peanut gives ninety-seven while cocoanut and palm kernel show only nine to sixteen. Unfortunately this difference of seven between the maximum and minimum throws us out of the way for small quantities of admixture, so much so that different authors fail to agree upon the average variation—some giving eight to nine only, while we are accustomed to find from thirteen to fifteen.

To resume this process, which. if it gave constant results, would be excellent, fails to give sufficiently accurate indications to detect adulteration of cocoanut oil when in small proportions. It is worse with palm kernel oil and of no value to distinguish between the two.

Continuing our investigations in another direction we have fortunately found a process which gives constant results when used upon these oils after a preliminary neutralization. We have discovered that cocoanut and palm kernel oils are entirely soluble in absolute alcohol. At a temperature of 30°–31° C. the former requires two volumes and the latter four volumes for complete solution.

It is a curious phenomenon that the smallest addition of vegetable or animal oils destroys this solubility in the same quantities of absolute alcohol. The solubility of the mixture is not proportional to its composition, but the mixture acts entirely like a distinct body.

Mode of Procedure.—First operation: Twenty cc. of the oil are shaken in a test tube with forty cc. of ninety-five per cent. alcohol. This indispensable preliminary treatment may give certain indications. Oil soluble in ninety-five per cent. alcohol, castor, resin oil, etc., are thus discovered, while Mowrah and Karité oils give a milky turbidity to the alcoholic stratum.

Second operation: Five cc. of the neutralized cocoanut oil are measured with a pipette into a graduated test tube, and ten cc. absolute alcohol added. The temperature is raised to 31°, the tube shaken violently for half a minute and then immersed in a water bath, kept at a temperature slightly above that of the tube. Pure cocoanut oil dissolves completely, and the solution remains clear. Any addition of another fatty matter causes precipitation. The material in solution being in a state of molecular equilibrium which is destroyed by the slightest modification.

Cocoanut oil, containing palm kernel oil, precipitates when the proportion of the mixture amounts to twenty per cent.; below this the mass remains turbid.

The verification of palm kernel oil is made in the same manner, only using twenty cc. absolute alcohol instead of ten, temperature remaining the same, 30–31°: five cc. of palm kernel oil, containing twenty per cent. of cocoanut oil, and above, dissolves in fifteen cc. absolute alcohol. In the same proportions pure oil does not dissolve and the mixture remains turbid.

A mixture of cocoanut, palm kernel, and peanut oil in such proportions that the oil would appear pure by the indices of saturation would be easily discovered by this process.

If we work at a lower temperature the proportion of absolute alcohol must be increased. For example, at 25–26° it is necessary to double the quantity; for five cc. of cocoanut oil, twenty cc. of alcohol. and for the palm kernel nut forty cc.

The same method may be used to determine the purity of cocoanut and palm kernel cakes by first extracting the oil by means of a solvent and then operating on it in the manner described.

This process, which I have had the honor to describe, carefully executed will allow us to determine in a few minutes adulterations of these oils which might cause bad results in soap making, which employs hundreds of millions of kilograms per annum, and in agriculture which uses the cakes for cattle food. This process was presented to the Academy of Sciences, Oct. 10, 1892, by M. L. Troost, and has been the object of a favorable report to the minister of agriculture.

Let us pass rapidly over palm oil whose principal chemical properties possess nothing in particular except its partial solubility in absolute alcohol, which is ten per cent. Also, we will pass over the Mowrah, which, as I have already mentioned, is identified by the milky white turbidity which it communicates to alcohol by shaking.

Illepe oil, whose titer is 52.5°, and cotton-seed margarine or stearine, as it is called here, whose chemical reactions are the same as the oil itself, will not long detain us.

I wish to mention, while closing the description of the solid vegetable oils, Karité oil, which comes from the French province, Soudan, which I have been especially directed to investigate by the Chamber of Commerce, of Paris, and the Chamber of the Syndicate for the manufacture of soap and candles.

It was very important for us to know how to utilize the great forest which covered the largest part of French Soudan. It is composed chiefly of the wild acacia and the Karité or butter tree. The natives collect the nuts which fall from the trees, crush the kernels in a mortar, treat the pasty mass with boiling water, and skim off the fat which comes to the surface. This fat, which is solid at the ordinary temperature, is used as an edible butter.

Without speaking about the great uses to civilization of this product, I must mention its remarkable chemical properties. The fatty acids having a titer of 52.5° C. combine with soda to produce an extremely hard soap. For candle making they would probably give better results after preliminary treatment to remove the resinoid matters which they contain and which hinder the crystallization of the fatty acids.

The presence of these resins lower the saturation to 14.9, and like the Mowrah this oil causes turbidity by shaking with alcohol (communication of M. Milliau to the Société d'Encouragement de Paris in 1892).

SOLID ANIMAL FATS, COLORED BROWN BY A STREAM OF CHLORINE GAS.

Butter.—The analysis of this material, which has attracted so much attention during the last few years, is not so difficult as some folks imagine, but requires to be carried on by the skill of a practical and experienced analyst. The specific gravity of butter is notably lower than that of tallow and lard. It is the same with the iodine number and the freezing point of the neutral fat. The saponification and the solubility in absolute alcohol on the contrary are notably higher.

We can combine with these different characters microscopical examination, melted butters showing under the microscope a collection of small regular spheres. Adulterated butter shows on the contrary abnormal figures as well as crystals which appear brilliant in the dark field of the polarizer.

By determining the fixed and volatile fatty acids and the solubility in alcoholic toluene, a skilled analyst familiar with these processes can easily determine admixtures above ten per cent. Below this point I do not believe any adulteration would be profitable. Natural butter contains about eleven per cent. soluble and volatile fatty acids, and eighty-seven per cent. fixed fatty acids. Besides alcoholic toluene dissolves it almost entirely.

Tallow.—The properties of tallow are so well known that they require no description. The distinction between beef and mutton tallow is a matter of interest, and presents great difficulties from the chemical standpoint, and we can only observe differences by taking the melting and solidifying points of the neutral fat and those of the fatty acids.

Lard.—Lard adulterated with cotton-seed oil is easily tested with nitrate of silver.

If the lard has been altered by time or other causes, it is necessary before using this reagent upon the fatty acids to take the precaution of purifying the fat in the manner in which I have indicated at the beginning of this article. We will thus avoid the slight reduction which is produced by decomposition products.

Some authors strongly believe in the use of the iodine number, which will give good results in the cases of large admixtures, as well as for quantitative analysis of such a mixture. But it is quite illusory for the detection of a small proportion of cotton-seed oil, for we must consider that the iodine number of pure lard varies within considerable limits, and it would be possible to correct for the high iodine number of cotton-seed oil by the addition of tallow, which will lower it appreciably. In such a mixture the action of nitrate of silver on the fatty acids allows us to discover adulteration even below five per cent. Tallow is detected by observing the crystallization of the fat from ethereal solution under the microscope.

The following oils, which frequently are used for the adulteration of agricultural fatty materials, are easily detected:

Fish Oil.—By its iodine number, its solubility in absolute alcohol, the presence of cholesterine, and finally the brown coloration by chlorine and red by caustic soda and phosphoric acid.

Resins.—By their density, their saturation, their solubility in absolute alcohol, and the deviation of the plane of polarization. Besides, it is easy to isolate them because the majority of compounds which they form with metallic salts are soluble in ether, while those of the fatty acids are insoluble.

Resin Oils.—By their property of not saponifying with caustic soda as well as by their insolubility in glacial acetic acid, and the characteristic purple coloration which they give with fuming stannic chloride.

Oleine Oil of Commerce.—By its solubility in alcohol and by its specific gravity.

Finally, *the mineral oils*, by their iodine numbers, their indifference to the action of caustic soda, and their insolubility in absolute alcohol at 15° C.

The processes which I have just enumerated are applicable indiscriminately to fatty matters both edible and industrial, for the recognition of their purity with an approximation sufficient for the majority of cases.

The results obtained will be more conclusive if they are compared with those from products of the same origin and known purity.

Although the majority of the processes which I have just indicated were devised and perfected in my laboratory and have been adopted by several governments, I have not, however, the presumption to feel that we have definitely finished this question, but consider on the contrary that in the vast unexplored region of the fatty series we have only taken a few steps. But I feel at the same time that we are now in the possession of sensitive and scientific methods which permit us to detect mixtures, the determination of which has appeared impossible up to the last few years.

The mission with which I have been charged by the French and Tunisian governments at the demands of commerce and industry shows you to what extent these countries are desirous of facilitating the exportation of pure and satisfactory products. France furnishes the best growth of virgin olive oil from Aix en Provence and Nice. On its side Tunis furnishes as well, but at a less price, olive oils of excellent quality which we can certify without analysis, because the government imposes such heavy export and import duties upon seed and seed oils that it would be impossible to practice the smallest amount of sophistication.

Often people who deal in olive oils make the remark that pure oil is too strong and it is improved in flavor by a mixture with a seed oil. Without discussing this inexact statement I believe that it would be preferable in every instance for the dealer to buy pure olive oil and to make his own mixtures. He would thus profit by the difference in price, which averages about sixty francs per 100 kilograms.