

Soybean Oil: Method I. (Adopted as tentative, May, 1942.)

Weigh 300 grams of refined oil into a refining cup; add 6% of Official Fuller's Earth and, using mechanical agitation at approximately 250 r.p.m., heat immediately to 120° C., taking not more than five minutes. Then stir mechanically at 250 r.p.m. (plus or minus 10) for five minutes, not allowing temperature to fall below 105° C.

Filter through an unused filter paper of fine texture. After sufficient oil has passed the filter to insure clearness, collect a sample for color reading. Cool and read color immediately as prescribed under **COLOR**.

Method II (for Refined Bleached Color on Crude Soybean Oil): Use 4% of Official Activated Clay under the same test conditions given in Method I.

NOTE—Method I is inapplicable to certain oils having an unusually high chlorophyll content (green types). Method II is not recommended for use on commercial refined soybean oil unless it has been refined within a few days. Method II is designed especially for use on green type and damaged crudes, after test refining. Method I and II do not give the same bleaching results.

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Report of the Refining Committee 1944-45

TWO meetings of the Refining Committee were held during the year, one in New Orleans on May 9, 1944, and the other in Chicago on October 24, 1944. Sub-Committees have been active as follows:

Sub-Committee on Refining of Extracted Soybean Oil—S. O. Sorensen, chairman.

Sub-Committee on Centrifugal Refining of Extracted Soybean Oil—E. M. James, chairman.

Sub-Committee on Modified Cup Refining of Extracted Soybean Oil—J. H. Sanders, chairman.

Sub-Committee on Expeller vs. Hydraulic Method for Hydraulic Oil—E. B. Freyer, chairman.

Copies of the minutes of the two meetings held and of the reports of the Sub-Committees for the year have been furnished the members of the main Committee. This report will constitute a brief review of the most important points covered at the meetings and in the Sub-Committee reports.

REFINING COMMITTEE MEETING, MAY 9, 1944, NEW ORLEANS:

Present: 13 members, 4 alternates, and 4 visitors.

Sub-Committee on Centrifugal Refining (R. R. King, chairman) recommended that work on this method be continued. Recommendation was adopted.

Sub-Committee on Expeller vs. Hydraulic Method for Hydraulic Oil (E. B. Freyer, chairman) reported as follows:

The substance of my report covers the refining of 32 samples of hydraulic oil by the laboratories of Swift and Company at Chicago and 11 samples of hydraulic oil by the laboratory of the South Texas Cotton Oil Co. at Houston, by both the method for expeller soybean oil and the former official method for hydraulic soybean oil, which it was voted by the Committee last October in Chicago should be abandoned. While the results of the tests show that 44% of the samples gave a lower loss by the expeller method, 49% by the hydraulic method and the balance showed no difference, the net average difference in losses shows the expeller method to be lower by 0.024%. Further, in the case of the widest loss differences between the two methods, the hydraulic results were the higher ones. The color of the refined oil is somewhat lighter by the expeller method although this point is possibly of small significance. On the whole, I consider the expeller method the better of the two, and its adoption has the advantage of making it possible to have only two standard procedures, one for hydraulic and expeller, and one for solvent extracted oil.

This report was unanimously accepted as confirmation of the previous year's recommendation of the Committee that the expeller method be made the official method for hydraulic oil.

Sub-Committee on Extracted Oil Refining (S. O. Sorensen, chairman) reported on new proposed kettle refining method for extracted oil. Messrs. Sorensen and James described their results which appeared to be favorable. It was decided to follow actively the investigation of the kettle method.

The 1943-44 report of the chairman covering the Refining Committee meetings at Peoria on July 7-8, 1943, and at Chicago on October 5, 1943, (Oil & Soap, February, 1945) was approved.

The following action was then taken with respect to a portion of the 1943-44 report:

It was moved and seconded that the paragraph in the 1943-44 report reading as follows: "Be it resolved that since the present tentative A.O.C.S. method has proved satisfactory on several additional extracted oils from this year's crop and seems satisfactory for general use it should be recommended as an official method" be rescinded. This motion was passed unanimously.

Another motion was then made, seconded, and passed unanimously as follows: "It is moved that since discrepancies in the analytical findings continue in the present method for the refining of various types of soybean oils that the present method for extracted oil be continued as a tentative method."

REFINING COMMITTEE MEETING, OCTOBER 24, 1944, CHICAGO:

Present: 11 members, 2 alternates, and 7 visitors.

Dr. Freyer's Sub-Committee to define the term "concordant results" as used in the soybean oil refining methods presented a report which suggested a definition. This report was accepted, and the chairman was instructed to poll the committee as to whether the proposed definition should be presented to the Society. A definition revised by Dr. Freyer for clarification was later submitted. The two proposed definitions for concordancy as follows were submitted to the Refining Committee. The preferred definition is to be suggested for incorporation in the Methods of Analysis of the A.O.C.S. immediately after the two lines of the last paragraph of page 16D:

I. Original Proposed Definition

On the test to be reported the chemists' report must be based upon the average of duplicate tests with the same lye agreeing within a tolerance of 0.3% on losses up to 6.0%, or within a tolerance of 5.0% of the lower loss when losses are above 6.0%.

II. Modification by Dr. Freyer

The result to be reported shall be the lower loss of the two obtained using different lyes, and it shall be based upon the

average of duplicate tests with the same lye agreeing within a tolerance of 0.3% on losses up to 6.0% or within a tolerance of 5.0% of the lower loss when losses are above 6.0%.

Fourteen members of the committee preferred the modified definition for concordancy as given under II above. Two members had other suggestions which will be considered at the next meeting of the committee.

Mr. James presented the report of the Sub-Committee on Centrifugal Refining and described the method developed by A. U. Ayres of the Sharples Corporation as well as the centrifuge which has been built for special test work. The method suggested by Mr. Ayres and used in his preliminary test work was as follows:

Problem Laboratory Centrifuge Refining

The Refining Committee of the American Oil Chemists' Society is anxious to find some method of refining certain types of extracted soybean oils that show very erratic figures by the A.O.C.S. standard refining method.

Work done at the Northern Regional Laboratories, using a centrifugal method in conjunction with an International machine showed great improvement over the standard method on such oils and a reasonable check from one refining to another although a study of all the data indicates the existence of a good deal of variation.

It has seemed to us that the one thing necessary to get better uniformity in results is an increase in centrifugal force, and this almost inevitably carries with it the expectancy of lower loss figures than those previously shown. The use of higher centrifugal force should more closely approximate the use of a similar force in continuous centrifugal refining, and it would seem feasible, therefore, to develop a laboratory centrifugal method that should reasonably well predict the results that will be obtained by continuous centrifugal refining.

Apparatus

The Sharples Analytical Laboratory Centrifuge consists of a heavy forged yoke driven at speed of 7500 R.P.M. by means of a 3 H.P. motor. In the yoke is provision for the fitting of four steel casings in which are mounted rubber cushions to take the centrifugal thrust of pear-shaped glass tubes, built in the general proportions of A.S.T.M. tubes. The casing is surrounded by a cooling coil to offset the increase in temperature due to air friction at high speed. The tubes have a capacity of slightly over 50 milliliters each. Centrifugal force at the tips is approximately 10,000 times gravity.

Agitation is accomplished by means of a special agitator, designed to fit into the cone shaped tubes, and driven by a Sargent cone drive stirring motor, style No. S-76445.

Refining Method for Expeller Soybean Oil

Weigh into a tube 45 grams of crude oil, and add caustic representing .25 excess of 20% lye. Dip the agitator blade and cork in crude oil and allow to drain for 5 minutes. Agitate at 1000 R.P.M. for 60 minutes at room temperature, then immerse in a water bath at 65° centigrade for 5 minutes while agitation is continued. Stop agitation, allow agitator to drain into the tube for 5 minutes, then place the tube in the centrifuge and spin for 20 minutes. Pour off the oil by inverting the tube for 5 minutes and weigh, adding to the weight figure .36 grams, which has been determined as an average of the amount of oil that clings to the tube and will not drain.

Refining Method for Extracted Soybean Oil

Same as above, except agitate at room temperature for 15 minutes at 1000 R.P.M. and heat for 30 minutes at 100 R.P.M.

Bleaching

If a bleach test is desired, without checking the color before bleach, the oil that is poured off from the refining tube is poured directly into another similar tube and the proper proportion of bleaching clay is added directly into the tube after the oil has been heated to 105° centigrade. This is then agitated with the same stirrer as used for the refining step for 5 minutes, and the tube is transferred to the centrifuge and spun for 20 minutes. At the end of that time the clay is tightly packed at the bottom of the tube and the oil is clear so that it can be poured into a tintometer tube and the color read without filtration.

In the data shown at the end of this report all of the bleaches from laboratory centrifugal refining were made in this manner except where otherwise noted. Bleaches on all raffinings were made with 4% of Special Filtrol at 105°C.

Results Obtained

The following covers all of the runs made in conformity with the above technique, giving comparative standard A.O.C.S. results where they are obtainable:

Oil	Lye Used	Method Used	Loss	F.F.A.
Degummed Expeller	2.5%—20°	Pilot Plant Centrifugal Refining	3.84	0.4
Degummed Expeller	2.5%—20°	Laboratory Centrifugal Refining	3.98	0.4
Extracted Oil	2/3 Max.—14°	A.O.C.S. Refining	5.8	0.3
Extracted Oil	1.49%—30°	Laboratory Centrifugal Refining	2.43	
	1.49%—30°		2.60	0.3
Extracted Oil	2.43%—30°	Laboratory Centrifugal Refining	3.08	
	2.43%—30°		2.99	0.3
Expeller	2/3 Max.—12°	A.O.C.S. Refining	3.3	0.43
Expeller	2.18%—20°	Laboratory Centrifugal Refining	2.78	0.43
	2.18%—20°		2.89	
	2.18%—20°		2.88	
	2.18%—20°		2.99	
	2.18%—20°		3.13	
Extracted	2/3 Max.—14°	A.O.C.S. Refining	5.3	0.67
Extracted	2.43%—20°	Laboratory Centrifugal Refining	3.48	0.67
	2.43%—20°		3.59	
	2.43%—20°		3.25	
	2.43%—20°		3.93	
	2.43%—20°		3.52	
	2.43%—20°		3.33	
	2.43%—20°		3.38	
Expeller	2/3 Max.—12°	A.O.C.S. Refining	6.9	0.70
Expeller	2.48%—20°	Laboratory Centrifugal Refining	5.79	0.70
	2.48%—20°		4.96	
	2.48%—20°		5.13	
	2.48%—20°		5.12	
	2.48%—20°		4.89	
	2.48%—20°		5.04	
Expeller	Max.—12°	A.O.C.S. Refining	5.5	0.60
Expeller	2.31%—20°	Laboratory Centrifugal Refining	3.41	0.60
	2.31%—20°		3.55	
	2.31%—20°		3.51	
	2.31%—20°		3.69	

Conclusion

The procedure as outlined appears to permit quite a reasonable check on those oils that have been tried.

Too much importance must not be assigned to the variations in bleach color between the A.O.C.S. refining and centrifugal refining. The colors reported for the standard laboratory tests are read by personnel in the Lever Brothers Laboratory who are accustomed to checking colors within this range. It is possible that our operator is not as skilled in this matter, which is indicated by the fact that we ran comparative A.O.C.S. tests and checked reasonably well on losses but did not check well on colors.

Not a great deal of information is available on these oils as to their performance in continuous centrifugal refining, but a comparison of the laboratory centrifuge refining results with the A.O.C.S. results indicates that the centrifugal laboratory refining method will probably approach the results of plant refining.

A number of variations were made in agitation time and in lye strength and excess in working toward the procedure described above, but of all those tried the selected procedure seemed to be the best and has been utilized for all of the oils tested.

The fact that the samples are small does not militate against reasonably accurate checks in a relatively short time, and the contents of one tube are sufficient for a refining and a final bleach color reading if handled by the method described above.

It is admitted that a complete knowledge of the value of this method will require study of a greater number of oils than we have had an opportunity to run, and the next step would seem to be the placing of these units in some plant where a variety of oils are encountered, and where the results can be currently checked against A.O.C.S. refining and against continuous centrifugal refining. In this study it may be necessary to re-examine the matter of lye strength and excess and agitation time, when studying different oils, although there is a marked

advantage in having a standardized method if it can be accomplished as outlined above.

Mr. Sorensen presented the report of the Sub-Committee on the glass kettle method. For the purpose of the record the method developed and used by this Sub-Committee in the preliminary work done to date is given below:

Glass Kettle Refining Loss Method for Extracted Soybean Oil

I. Apparatus

A. *Glass Kettle*—An elongated Pyrex glass vessel capable of holding 500 grams of oil plus 50 grams of water, completely surrounded by a glass jacket with an opening in the bottom to drain the kettle, and constructed especially for this work. It is approximately 9 inches long and 2¾ inches inside diameter. The bottom of the kettle is cone shaped with an angle of approximately 45 degrees. The kettle jacket is equipped with two side arms, the lower side arm being placed on the opposite side from the upper arm and at the lowest practical point near the bottom, while the upper side arm is placed about ¾ inch from the top of the kettle.

B. *Stirrers or Agitators*—Made by securely fastening to a ¼-inch iron or steel rod, 12 inches long, two paddle blades set at right angles to each other. These paddle blades are made of heavy gauge sheet metal ¼-inch thick, ½-inch wide, and 1½ inches long. The lower paddle is fastened to the rod by cutting a slot in the tip of the rod deep enough to accommodate the paddle. The paddle is then soldered or brazed into the slot. This is done to retain the over-all length of the stirrer at 12 inches. The second paddle is securely fastened, by soldering or brazing, at right angles to the bottom paddle at a distance of 3 inches from the bottom of the lower paddle to the bottom of the upper paddle. This upper paddle must measure 1½ inches across from tip to tip.

The stirrer is held in the chuck of a variable speed motor. It is advisable to use a cork bearing on the shaft of the stirrer held in place by means of a clamp to the upright stand. This cork guide or bearing is located just above the top of the kettle in order to minimize any tendency of the long shaft to whip. When installed in the glass kettle, the bottom of the lower paddle is 1½ inches above the bottom of the kettle.

C. *Variable Speed Motor*—Any variable speed motor that can be adjusted to 350 R.P.M. and then reduced to a known speed of 200 R.P.M. while still in operation. A Cenco stirrer (Catalog No. 18805) variable speed motor, type NS1-12, has been found satisfactory.

D. *Constant Temperature Bath and Circulating Motor*—Any constant temperature setup capable of maintaining the oil in the kettle at 80°C. or 176°F. with an outlet at the bottom which can be attached to a small circulating pump so that the water from the bath can be pumped into the lower side arm of the kettle jacket, circulated within the jacket, and then returned to the constant temperature bath.

E. *Wash Bottle With Spray Nozzle*—A 500-ml. laboratory wash bottle is equipped with a glass spray nozzle. The nozzle is made by blowing a bulb (approximately ½-inch diameter) on the end of a piece of Pyrex tubing (same outside diameter as tubing used in wash bottle). While still hot the bulb is flattened to form face of nozzle. The face is then drilled with a sharp, long tapered piece of tungsten wire heated to white heat. Approximately seven holes are made in the face of the nozzle large enough so that the stream from each hole is separate and distinct; a very fine spray is to be avoided since this tends towards the formation of emulsions.

F. *Centrifuge*—Since the purpose of the centrifuge is to separate the entrained oil from the foots, any centrifuge that will accomplish this purpose is satisfactory. An International size 1, type SB centrifuge, head No. 234 (6-place, 4 tube) using a 50 ml. tube at 3350 R.P.M. (50th step) with a reported maximum relative centrifugal force at the tip of 2470 has been used. About 5 minutes under the foregoing conditions was found to accomplish this purpose.

II. Sodium Hydroxide Solution

Eight percent by weight of sodium hydroxide (12° Baumé at 15°C.) in the proper weight of distilled water.

III. Calculation of Caustic Solution for Refining

The amount of 8% caustic solution required for refining an oil is computed from the following general formula:

$$\frac{(\% \text{ F.F.A.} \times .142 + .15) \times 5}{.08} = \text{Number of grams of } 12^\circ \text{ Baumé lye required for 500 grams of oil.}$$

The various constituents of the foregoing formula may be briefly explained as follows:

% F.F.A. = % free fatty acid—obtained by analysis.

.142 = Factor obtained by dividing the molecular weight of sodium hydroxide by the molecular weight of oleic acid

$$\frac{\text{NaOH (40.0)}}{\text{Oleic acid (282)}} = .142$$

.15 = Excess of alkali to be added (dry basis).

.08 = Strength of alkali (equivalent to 8%).

The foregoing equation is multiplied by 5 to obtain the required number of grams of 8% (12° Baumé) lye required for 500 grams of oil.

Example:

Given an extracted oil with .5% F.F.A.

Then by substituting in the formula

$$\frac{(.5 \times .142 + .15) \times 5}{.08} = 13.8 \text{ grams } 8\% \text{ (12}^\circ \text{ Baumé) lye required for 500 grams of the oil.}$$

IV. Refining Procedure

1. Shake sample vigorously to mix thoroughly. Sample may be heated in hot water (in original container) to a temperature not to exceed 50°C. (122°F.) and then mixed.
2. Determining the percent F.F.A. of the oil by the regular method outlined in the A.O.C.S.
3. Fill dead space in the draw-off line from the kettle with water so that the water level just reaches the top of the opening or is flush with the bottom of the kettle.
4. Transfer 500 grams of oil to the glass refining kettle. This is done by weighing out 502 grams in a tared Pyrex beaker (600 ml.), carefully pouring the oil into the kettle and allowing the beaker to drain for about 2 minutes. This results in a charge of exactly 500 grams.
5. Start agitation at 350 R.P.M. and allow the oil to reach a constant temperature of 80°C. (176°F.).
6. Upon reaching a constant temperature of 80°C. (176°F.), quickly add the calculated amount of sodium hydroxide solution to the oil. The lye solution can be easily added by weighing out the required amount of alkali in a small beaker (30-50 ml.) and pouring it into the oil.
7. Agitate for 30 seconds at 350 R.P.M., then slow stirrer down to 200 R.P.M. and allow agitation to continue for 10 minutes at 80°C. (176°F.).
8. At the end of 10 minutes stop the agitation and allow the oil and foots mixture to settle for 15 minutes at 80°C. (176°F.).
9. After settling for 15 minutes, introduce by means of a spray from a wash bottle approximately 10% hot wash water (50 ml.). This amount can be judged fairly closely after the first addition of water and will fill the kettle almost to the top. The temperature of the water is almost at the boiling point (approximately 95°C.). After adding the water, allow the mixture to settle for 5 minutes. The temperature of the oil will rise slightly due to the addition of the hot water, but it will return quickly to the 80°C. (176°F.) constant temperature. The bulk of the water usually collects below the foots layer.
10. On completion of the 5-minute settling period, draw off the wash water to the foots layer.
Note: Occasionally the water tends to stratify between the foots layer and the oil layer and upon drawing off the foots will come out first. In this case they can be collected in a centrifuge tube and then the water can be drawn off.
11. Spray a second 10% portion of hot wash water on the surface of the oil and allow the mixture to settle for 5 minutes at 80°C. (176°F.) as before.

12. As soon as the 5-minute settling period is completed, draw off the wash water which can now be discarded.
13. The foots layer, containing entrained oil, is now drawn off and collected in a centrifuge tube or beaker, care being taken to prevent undue agitation of the mixture as this tends toward the formation of emulsions. The mixture is then centrifuged. Upon centrifuging, the entrained or occluded oil is found in a layer on top of the foots. This oil layer is removed by means of a pipette or medicine dropper, and weighed. The weight obtained is then added to the weight of refined oil.
14. Now drain the refined oil, which may be slightly cloudy, into a previously tared beaker, allowing the kettle to drain for 10 minutes, and then weigh.
15. The weighed refined oil is then thoroughly mixed and a sample is poured off for a moisture determination. The moisture is determined by weighing 5 grams of the oil into a suitably desiccated 30 ml. beaker. The sample is then heated in a forced air oven at 110°C. for 2 hours. At the end of the two hours the sample is removed, cooled in a desiccator, weighed and the percentage of moisture or volatile calculated from the difference in weight. If preferred, any of the methods set forth in the Official and Tentative Methods of the A.O.C.S. may be used.
16. Finally, 10 grams of kieselguhr (i.e. Hyflo Super-Cel) may be added to the oil to dry it. It is then filtered through 32 cm. No. 230 fluted Reeve-Angel filter paper and the filtered oil used for a color and bleach determination.

V. Calculations

Weight refined oil + weight of oil recovered from centrifuge = total weight refined oil.

Total weight refined oil × % moisture = weight moisture in oil.

Total weight refined oil — weight moisture in oil = total weight dry refined oil.

Weight of crude oil — total weight of dry refined oil = weight material lost in refining.

Then:

$$\frac{\text{Weight of material lost in refining}}{\text{Weight of crude oil}} \times 100 = \% \text{ refining Loss}$$

Example:

Weight of refined oil (grams).....	480.8
% volatile89
Weight of oil recovered from centrifuging of foots (grams)	5.5
480.8 + 5.5 =	486.3 grams.
486.3 × .0089 =	4.3 grams.
486.3 — 4.3 =	482.0 grams.
500.0 — 482.0 =	18.0 grams.
$\frac{18.0}{500} \times 100 =$	3.60% Refining Loss.

Results on one collaborative sample were presented by Mr. Sorensen. The agreement between collaborators was not too satisfactory, but the consensus of the committee was that work on this method should be continued. Mr. Barrow reported that the standard cup refining method had been run on the sample used in the kettle refining collaborative work and firm soap stock was obtained in all cups.

R. C. Stillman reported for Mr. Sanders, chairman of the Sub-Committee to study the modified cup method. In view of the interest of a number of the Refining Committee members in the possibility of the modified cup method for degummed extracted soybean oil the report of this Sub-Committee is given in its entirety.

A Modified Cup Method for Refining Degummed Extracted Crude Soybean Oil

A Subcommittee Report

These objections have been raised against the present tentative cup procedure for extracted crude soybean oil, when it is applied to *degummed*, extracted, crude soybean oil.

1. A third phase of free lye frequently appears during the refining and interferes with the accurate separation of refined oil and soap stock.
2. The soapstock is usually too fluid to permit clean decantation of oil.
3. Refining losses obtained are well above those sustained in plant kettle refinings of such oils, whereas with other crude soybean oils there is reasonably good agreement between laboratory cup and plant kettle results.

As a means of eliminating these objections, the current tentative procedure and modifications involving the amount and strength of lye, the temperature and time cycle, and the addition of absorbents for free lye were applied to degummed, extracted, crude oil from four different suppliers.

The current tentative procedure gave easily manageable foots on the four oils and presented no significant problems of lye separation. Refining losses, however, were substantially higher than those usually obtained on such oils in commercial operations.

Simply reducing the amount of lye to about one-half the currently prescribed value reduced the cup losses on all samples from an actual value of about 4.0% to about 1.5%. Lye separation was no significant problem. Where refining loss was low, the foots on two of the oils were very good, and on the other two, were soft but manageable. On one of the four oils, the lowest loss occurred under conditions where the remelts were higher than proper for good refining practice. Refined and/or bleach colors showed no serious variations, although in seasons of dark oils reduced lye usages could be expected to produce darker colors.

The four degummed, extracted crudes varied from less than .01% to as much as 0.5% acetone insoluble. This spread is probably near the limit that can be handled by a procedure designed for "degummed crudes."

Based on the results obtained here, the feeling of the Sub-Committee is that a modified cup procedure would be satisfactory for refining degummed, crude soybean oils, without regard for the method by which the oil was removed from the seed. Further cooperative work on a cross-section of degummed, crude oils should be done.

Conclusions and Suggestions for Next Year's Activities

The extensive experimental program carried on this year by the Sub-Committees did not lead to the development of a single suitable method for the several types of extracted soybean oil. Progress has been made, however, and several procedures show considerable promise. The following program is indicated for next year:

1. A complete report of this year's work carried out on the glass kettle refining method by Mr. Sorensen's Sub-Committee will be available before the next meeting of the Committee, *which is now tentatively planned for June, 1945*. A decision should be made at that time as to whether further attention should be given to this method.
2. Work should be continued on the Centrifugal Method.
3. Attempts should be continued to work out a modified cup method which will be applicable to the several types of extracted soybean oil.

The Refining Committee is appreciative of the splendid work done by the Sub-Committees made up as follows:

Kettle Refining Method—Sorensen, chairman; James, Milner, Kruse, Mitchell.
 Centrifugal Method—James, chairman; Ayres.
 Modified Cup Refining Method—Sanders, chairman; Barrow, Freyer.
 Expeller vs. Hydraulic Method for Hydraulic Oil—Freyer.

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