

## Table-Top Oil Refinery For The Home Chemist

By RAYMOND B. WAILES

Mechanized Armies Make Oil Supply Vital," "Shortage of Fats Imperils Nation at War"—headlines like these spotlight what used to be routine items in a country's shopping list, and give them new interest for home-laboratory experimenters.

Envious eyes the world over turn toward our vast forests of oil derricks, and the rivers of crude oil that refineries turn into gasoline for vehicles of war and peace. What makes their enormous production possible is the modern "cracking" process for extracting gasoline. Once the crude oil was simply distilled to separate, in turn, the gasoline and other substances it contained. Today, in addition, by-products of the distillation are "cracked" or broken down into gasoline, doubling the total yield.

You can demonstrate for yourself how this is done, with a miniature cracking unit. Its furnace may be a test tube, preferably of heat-resisting glass. Fit the test tube with a one-hole stopper, and connect it with a small flask or a side-necked test tube. This second vessel catches the condensate from the cracking reaction. Finally, the condenser should be fitted with a straight glass delivery tube so that "refinery gas" formed in the reaction will issue from it and can be burned.

White mineral oil, the ordinary medicinal kind that you can buy at the corner drug store, will serve as your raw material. Place about five cubic centimeters (a teaspoonful and a half) of the oil in the first test tube. Fill the space in the tube above the oil with a wad of steel wool, to obtain a greater cracking area. Keeping the top of the test tube hot will heat the steel wool in turn, and oil vapors coming in contact with the hot material will crack, or decompose.

Start the experiment by slowly applying the flame of a Bunsen burner, fitted with a flame-spreading attachment, so as to heat the oil and the steel wool simultaneously. The oil will soon reach its boiling point. As the oil vapors pass through the hot steel wool, they break down into a number of lighter petroleum "fractions" or products, including gasoline and also combustible gas. You can light the gas at the end of the delivery tube.

When all the oil in the first test tube has been vaporized, let the apparatus cool. You will find an oily product in the flask or side-necked test tube that has served as a condenser. Its odor alone will tell you that something has happened to the mineral oil that you started with, and a simple experiment will confirm this.

Gently shake a teaspoonful of mineral oil with an equal quantity of strong sulphuric acid, in a test tube, and do the same with the residue from your condenser. Notice that nothing happens in the first case. The mineral oil consists of what a chemist calls paraffin hydrocarbons, which sulphuric acid does not attack. But sulphuric acid promptly imparts a brown discoloration to the residue from the cracking experiment, showing the presence

of “ole-fines” or “unsaturated hydrocarbons” such as are found in natural petroleum. In other words, you have changed back a refined product into one more akin to crude oil.

In commercial practice, this cracked oil is redistilled to separate the gasoline, kerosene, gas oil, and other “fractions” of which it is a mixture. Of course, pure white mineral oil is not the commercial raw material, but it illustrates the cracking principle nicely.

Instead of breaking down molecules of oil, you can build them up, by the method known as “hydrogenation.” This is the process used to produce well-known brands of lardlike shortening fats, glistening snowy-white solids, from liquid vegetable oils such as cottonseed oil. Chemically, it consists simply of adding hydrogen to the original molecules.

That isn’t quite as easy as it sounds, however, unless you know the trick. You must have the aid of a catalyst, one of those curious agents that promote reactions without undergoing any change themselves. In this case you can prepare your own catalyst.

FIRST, add a solution of sodium hydroxide or of potassium hydroxide to a solution of some nickel chemical—nickel sulphate, nickel nitrate, or nickel chloride, for example. The strengths of the solutions do not matter. Mixing them will yield a light-green precipitate of nickel hydroxide. Let it settle, and then pour off the clear liquid above it. Add fresh water; shake well; let the precipitate settle; and pour off the clear liquid again. Repeat this “washing by de-cantation” several times, to free the nickel hydroxide from other chemicals. Then filter out the precipitate and dry it. Place the dried product in a Rose’s crucible, or in an eight-inch length of glass tubing with an inside diameter of about half an inch. Pass hydrogen gas over the nickel hydroxide while you heat the outside of the tube or crucible. The hydrogen reduces the nickel hydroxide to metallic nickel. This reduction should be carried out at a temperature a little below red heat, and allowed to proceed for about fifteen minutes. The hydrogen may be supplied from a simple acid-metal generator, or illuminating gas, which contains free hydrogen, may be used. In either case, the gas that has passed over the contents of the tube or crucible should be led away and discharged outdoors.

When your homemade nickel catalyst is ready, place it in a distilling flask and add one or two fluid ounces of a vegetable oil, such as cottonseed oil or salad oil. Cork the flask with a two-hole stopper. Through one hole, a chemical thermometer should dip into the oil. The other hole carries a glass tube, also dipping into the oil, through which hydrogen gas can be delivered.

Heat the oil to about 175 or 200 degrees centigrade (350 or 375 degrees Fahrenheit) and keep it at this temperature while hydrogen gas bubbles through the oil for about twenty minutes. Here, again, ordinary illuminating gas may be substituted for hydrogen. If you use illuminating gas, you can lead the excess gas from the exit tube of the distilling flask to a Bunsen burner, and use it to heat the flask—just as byproducts are put to use in industrial processes. But if pure hydrogen gas is used, do not try to burn it. There would be danger that the flame would “strike back” into the flask, and explode any hydrogen-air

mixture that might be there. Out the window, through rubber tubing, is the best way to dispose of the left-over hydrogen.

After this twenty-minute treatment of the oil, turn off the gas supply and let the flask cool. If all has gone well, you will find that the oil has thickened considerably or has actually turned solid—depending on the degree of activity of your homemade catalyst, and other conditions in carrying out the experiment.

You can make your product a little purer by filtering it, while hot, through a pledget of absorbent cotton placed in a funnel. This removes the nickel catalyst mixed with it. Both the oil and the funnel must be warm during this filtration, or the oil will harden in the funnel and refuse to flow.

Another oil that lends itself to home-chemistry experiments is the oil from the seed of the flax plant, or linseed oil. Used in making paints, linoleum, printing inks, and putty, linseed oil has the unusual ability to combine with the oxygen of the air and form a stiff, rubberlike film. The thick scum or skin that you see on the surface of paint in a can is a film produced by the oxidation of linseed or other oils of the paint, forming compounds that are termed linoxins.

You can easily show how such an oil combines with the oxygen of the air, and at the same time demonstrate the first step in the manufacture of linoleum. Cut several pieces of cotton cloth, an inch or more wide and six or eight inches long. Hang the strips vertically in a warm place, with a noncombustible tray beneath them. Pour linseed oil over the strips, letting it trickle down and saturate the cloth. To each fluid ounce of the linseed oil that you apply, there should previously have been added several drops of a liquid drier (a metallic resinate or borate), which may be obtained at any paint store. The drier acts as a catalyst to speed up the oxidation of the linseed oil film. At the end of several days or weeks, the cotton strips will be so stiff that they can be held horizontally by one end without drooping.

By repeated treatments of this kind, the cloth may be given a thick layer of oxidized oil or linoxins. In making linoleum, such strips are prepared in warm buildings; the strips are ground; gums, resins, coloring pigments, and cork dust are added; and the whole is fabricated into sheets of linoleum for your kitchen and other floors.

Linseed oil also combines with sulphur, as do other vegetable oils, notably rapeseed oil. A rubbery product is the result. Try heating a tablespoonful of linseed oil with a pinch of ordinary flowers of sulphur, stirring until the sulphur has melted. The mass can be drawn out like rubber and even has the odor of rubber. Under the name of "factice," it is used as a rubber substitute.

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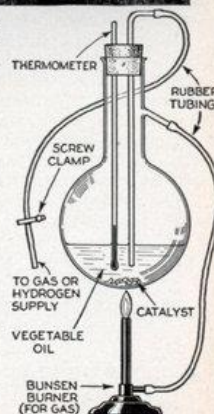
You can demonstrate for yourself how this is done, with a miniature cracking unit. Its furnace may be a test tube, preferably of heat-resisting glass. Fit the test tube with a one-hole stopper, and connect it with a small flask or a side-necked test tube. This second vessel catches the condensate from the cracking reaction. Finally, the condenser should be fitted with a straight glass delivery tube so that "refinery gas" formed in the reaction will issue from it and can be burned.

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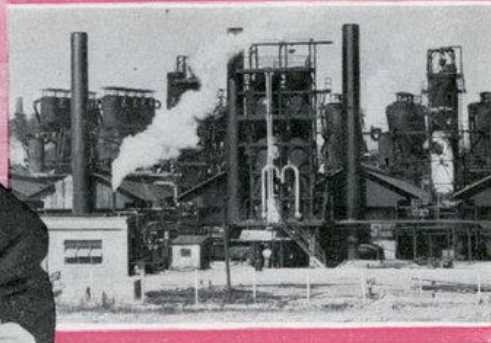
Start the experiment by slowly applying the flame of a Bunsen



Converting a liquid oil into a solid fat, by the process used in making vegetable shortening. The apparatus is shown below



In the simple experiment illustrated at the lower left, you can duplicate the process employed in great industrial plants in "cracking" oils for petroleum products



burner, fitted with a flame-spreading attachment, so as to heat the oil and the steel wool simultaneously. The oil will soon reach its boiling point. As the oil vapors pass through the hot steel wool, they break down into a number of lighter petroleum "fractions" or products, including gasoline and also combustible gas. You can light the gas at the end of the delivery tube.

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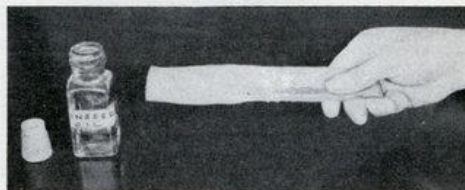
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Linoleum in the making: a strip of cloth on which linseed oil has been allowed to oxidize. Already it is stiff enough to be held horizontally



## ● EXPERIMENTS WITH OILS AND FATS REPRODUCE

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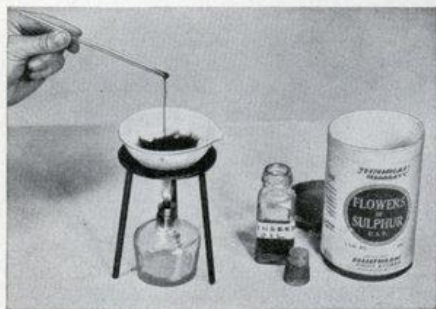
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After this twenty-minute treatment of the oil, turn off the gas supply and let the flask cool. If all has (Continued on page 245)



Hydroquinone, added to a vegetable oil used as a heating bath, will keep the oil from becoming rancid. Left, making a rubber substitute by heating sulphur and linseed oil

## IMPORTANT PROCESSES USED IN INDUSTRY •

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(Continued from page 211)

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