THE PRODUCTION OF OIL OF AMERICAN WORMSEED, U. S. P., IN ILLINOIS.*

BY W. A. KONANTZ.[1]

The opinion seems to be generally held that Oil of American Wormseed, or Chenopodium, conforming to the specifications of the Pharmacopœia of the United States, can be produced only from plants (*Chenopodium ambrosioides var. anthelminticum*) cultivated in Carroll County, Maryland. The oil obtained from plants grown in the Middle West—known as "western" oil—is commonly thought to be inferior in quality, being of too low specific gravity and, therefore, containing too little ascaridol, the active constituent. The specifications of the Pharmacopœia are that the oil shall have a specific gravity of 0.955 to 0.980 at 25° C., shall be soluble in 8 volumes of 70% alcohol, and shall have an optical rotation between −4° and −10° in a 100-mm. tube at 25° C.

The opinion that "western" oil is inferior to Baltimore oil seems to be based upon an article written in 1854,[2] and apparently substantiated by the work of Wirth[3] on oil distilled from authentic wormseed plants grown at the University of Michigan Botanical Gardens. Wirth concluded that the "western" oil agrees in composition with the Maryland oil except in the amount of ascaridol; that it is impracticable to bring the "western" oil up to standard by fractionation, due to the waste involved; and that a more logical method of attack would be a study of the climate and soil conditions under which the "western" plants are grown as compared with the others.

Wirth describes his method of distillation as follows:

"A copper still with a capacity of 2 to 3 Kg. of fruits, equipped with a false bottom so arranged as to allow the passage of steam upward but to prevent any vegetable matter falling into the lower compartment, was employed. The lower compartment had a capacity of from five to six liters, and was equipped with an inlet for steam. The entire still was surrounded by a copper jacket, through which superheated steam could be conducted. This was used as a source of heat. The condensing surface was small, the temperature of distillate in the receiver being from 45° to 50° C.

"The fruits from the sun-dried plants were stripped from the stems (which served to bruise them), and macerated with water for twenty-four hours. Three liters of water were placed in the lower compartment, and the moist fruits in the upper.

When all the water in the lower compartment was evaporated, steam of exterior generation was introduced. The flow of steam through the jacket was so regulated that no vapors evolved from the end of the condenser, the distillate in the receiver being about 45° C. The time of distillation was in every case less than one and one-half hours. This method of procedure eliminated (1) excess reflux action (prevented by the steam jacket surrounding the compartment containing the fruits); (2) unnecessary heating of the oil (which is apt to cause its decomposition),

* This investigation was undertaken because the supply of wormseed oil has been for several years very inadequate to the demand, and this condition has prevented extensive use of the oil in the treatment of domestic animals—a field in which it is much needed. Owing to the low individual value of such animals as hogs, for example, it is not economical to treat them with a drug of such high price as wormseed oil. This paper is a report of the first year's work in producing this oil at the Experiment Station of the Moorman Mfg. Co., Quincy, Ill. It is planned to continue the work on a much larger scale, covering every phase of the problem. Anyone wishing to produce the oil can obtain the full coöperation of this company on request.

[1] Chief of Research Department, Moorman Mfg. Co., Quincy, Ill.

[2] *Amer. Jour. Pharm.*, 26, p. 503.

[3] JOUR. A. PH. A., 9, 127, 1920.

and gave (3) a better separation of the oil from the water (due to "hot-running" the distillate) as well as (4) a larger yield of oil (the bruising and macerating of fruits allowing a more efficient escape of oil), and (5) the short period of subjection to heat prevented the decomposition of the oil. . . . . "

The results of an average distillation were as follows: Fruits (dried), 2000 grams; total distillate, 4000 cc.; oil, 49.5 cc. × 0.9343 (sp. gr.) = 46.035 grams = 2.3% yield of oil. Total time of distillation, 1 hour, 20 minutes. Constants of oil: d (15.56°), 0.9343; α_D (100-mm. tube), 25°−6.21°; not soluble in 10 vol. of 70% (vol.) alcohol; ascaridol, 42 to 45% (by fractionation).

In the light of the present writer's work it seems that Wirth's failure to obtain a satisfactory quality of oil from the "western" plants was largely due to faulty methods of distillation. By his methods the steam pressure is too low, the time of distillation too long, and the temperature of the distillate too high. That "the method of distillation is a factor which causes great change in the oils" has been amply demonstrated by several investigators, including the present writer.

Schimmel & Co.[1] performed several experiments involving various methods of distillation, but after trying several methods came to no definite conclusions as to the best method, but advised that the oil should be subjected to heat for as little time as possible.

Nelson[2] laid emphasis on the method of distillation, stating: ". . . . . the valuable and chief ingredient, is unstable, and is decomposed gradually on boiling with water. Consequently, the distillation must be carried on rapidly with steam at a good pressure, the condenser kept warm and the warm distillation water separating from the oil in the receiver, discarded."

Russell,[3] who investigated the influence of methods of distillation on the commercial value of oil of wormseed, states that the second precaution mentioned by Nelson has no effect on the composition of the oil but does tend to effect a better separation of the oil in the receiver, and that the third precaution is not applicable for no producer of wormseed oil is returning distillate water to the retorts.

Russell cultivated the so-called "tall variety" of Chenopodium at the Government Experimental Farm, Arlington, Va. The accompanying data were obtained, oil

	Oil No. 1.	Oil No. 2.
Weight of herb distilled	150 kilos	150 kilos
Weight of oil secured	275 grams	185 grams
Yield of oil	0.183%	0.123%
Steam pressure at inlet to retort	60 lb.	60 lb.
Temperature of cooling water entering condenser	18° C.	18° C.
Temperature of cooling water leaving condenser	72–85° C.	35° C.
Temperature of distillate	60–74° C.	22° C.
Time of distillation	30 min.	60 min.
Steam condensed per minute	3.65 lb.	1.82 lb.
Weight of distillate water	110 lb.	110 lb.
Specific gravity of oil, 25° C.	0.9631	0.9338
Angle of rotation in 100-mm. tube at 25° C.	−5° 30′	−6° 55′
Solubility 70% alcohol	8 vols.	Insoluble
Ascaridol	73%	60%

[1] *Schimmel & Co. Report*, April 1908, p. 109.

[2] *Jour. Am. Chem. Soc.*, 42, 1204, 1920.

[3] Jour. A. Ph. A., 11, 255, 1922.

No. 1 being produced by rapid distillation with condenser warm, and oil No. 2 by slow distillation with condenser cold.

Russell states that "the method of distillation is a factor which causes great change in the oils" and that "with rapid distillation, that is, with a good flow of steam, an oil was secured which passed all the U. S. P. requirements and contained a high percentage of ascaridol." Also, a better yield of oil was obtained by distillation with a good flow of steam. These laboratory results he later confirmed in Maryland on the commercial-sized stills. He states that no differences in yield and specific gravity of the oil were noted when the steam pressure at the retort was 80 or 100 pounds. When the pressure was reduced to 40–60 pounds the specific gravity was lowered. The time of distillation (from appearance of distillate at discharge end of condenser) was 8 to 10 minutes. With a slower method of distillation the specific gravity was reduced. With a distillation time of thirty minutes and a steam pressure of about 40 pounds, oil of low specific gravity was obtained (0.938–0.946). Russell made the following recommendations: The use of a short supply steam line from the boiler to the retort, not less than 2″ in diameter, with a 1″ pipe distributing the steam through a "spider" in the retort, and a steam pressure of 80 pounds at the boiler gage. The steam should be admitted promptly and at the full capacity of the 1″ inlet pipe (not less than 20 pounds of steam per minute and the total time should not exceed 15 minutes). Water should not be added to the retorts and these should be drained of condensed steam whenever necessary. The condensate, a mixture of oil and water, should flow out at a relatively high temperature.

In 1922 and 1923 the present writer made a thorough study of the methods of growing and distilling wormseed as practiced in Maryland. Most of the retorts used in Maryland are processing kettles such as are used in canning factories. The steam is usually generated in a stationary boiler, but in some instances in the boiler of a threshing engine. A steam pressure of 80 to 100 pounds at the boiler gage is employed. The condensers are uniformly of one type and consist of straight pipes laid in a trough of running water and connected to the retort in a suitable manner just under the cover. Each distilling outfit is set up near a small stream so that by building a dam across the stream the water can be diverted to the condenser trough and an abundant supply be assured at all times. These condensers can be made very efficient since no limit need be set to the length of pipe through which the steam from the retort must pass. The temperature of the cooling water entering the condenser is in most cases about 15° C. and that leaving the condenser 27° C. The temperature of the distillate is from 32–38° C. The time of distillation varies between 12 and 20 minutes.

EXPERIMENTAL.

The plants upon which the present writer's work was conducted were grown at the Moorman Experiment Station, 10 miles northeast of Quincy, in Adams County, Illinois, on soil of the type known as yellow-gray silt loam. This soil is low in lime, phosphorus, nitrogen and organic matter. A 2–8–2 fertilizer was applied at the rate of 600 pounds to the acre. No trouble was experienced in growing the plants. As nearly as can be calculated a yield of oil was obtained from these plants corresponding to between 40 and 50 pounds per acre.

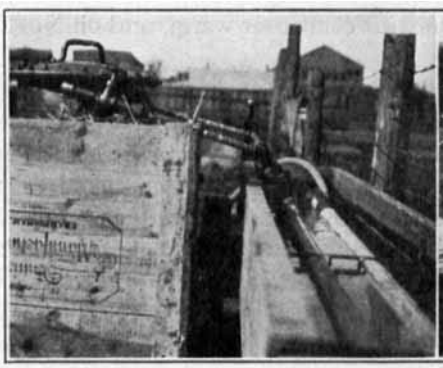

Fig. 1.—Retort used in distilling oil of wormseed, showing connection with condenser.

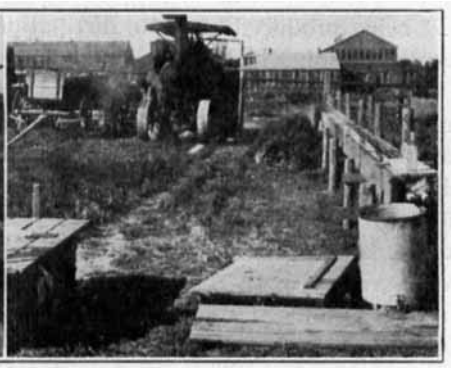

Fig. 2.—Complete outfit used in distilling oil of wormseed; steam generated in threshing engine.

The retort used is shown in Fig. 1. It was surrounded by a wooden box, packed with straw, to prevent condensation of the steam. This was thought necessary on account of the small size of the retort and the cold weather. The retort is of 3 cu. ft. capacity and holds 35 to 45 pounds of plants. In our experiments we nearly always ran two batches, obtaining from 4 to 10 fluid ounces of oil, a quantity sufficient for testing. Figure 2 shows the complete outfit. Fig. 3 is a close-up view of the discharge end of the condenser.

Fig. 3.—Discharge end of condenser.

At first we used a $^3/_4$" steam inlet pipe to the retort. With this we found that the steam rushed through the plants so rapidly that practically no oil was gotten out. It was evident that it would be necessary to reduce the size of the pipe so that the plants would be subjected to more of a "cooking effect." We then reduced the inlet to $^1/_8$"; this proved to give the best results in all our experiments. Later we tried $^1/_{16}$" and $^1/_4$" inlets. With the $^1/_{16}$" inlet the flow of steam was so slow that very little oil was obtained, whereas with the $^1/_4$" inlet we experienced some of the same trouble that we had with the $^3/_4$" opening; *i. e.*, steam rushing through too rapidly, resulting in a low yield of oil, and an oil much darker in color, although the specific gravity was good (0.969). Table I shows the effect

TABLE I.—EFFECT OF VARYING SIZE OF INLET.

Steam pressure, 120 lb.

Inlet size.	Distillation time.*	Yield from 100 lb. plants.	Specific gravity.	Per cent. ascaridol.
$^3/_4$"	....	None	....	...
$^1/_4$"	13 min.	170 cc.	0.969	78.7
$^1/_8$"	16 min.	268 cc.	0.969	78.1
$^1/_{16}$"	21 min.	Very little	....	...

* From appearance of distillate at discharge end of condenser until no globules of oil came over.

of varying the size of the inlet. It is evident that there is an optimum rate of flow of steam, that it can be too slow or too rapid; for our size still the best opening used was $^1/_8$".

We also tried different steam pressures, namely, 80, 100, 120 and 140 pounds, and found that the specific gravity of the oil was not affected except at 140 lbs., when it fell to 0.966 as compared with 0.969 for the other pressures. The yield, however, was affected as shown in the following table. The highest yield was obtained with a steam pressure of 120 pounds.

TABLE II.—EFFECT OF VARYING STEAM PRESSURE.

$^1/_8$" inlet.

Steam pressure.	Distillation time.	Yield from 100 lb. plants.	Specific gravity.	Per cent. ascaridol.
80 lb.	26 min.	168 cc.	0.969	79.2
100 lb.	19 min.	220 cc.	0.969	79.2
120 lb.	17 min.	344 cc.	0.969	79.1
140 lb.	23 min.	233 cc.	0.966	76.5

Both Nelson and Russell recommend the use of a warm condenser. In studying our data we noted that oil obtained in the first runs of the day was a little higher in specific gravity than that obtained later in the day when the water in the condenser had warmed up. To prove this we ran batches with a warm and with a cold condenser. With the warm condenser the specific gravity was 0.960 and with the cold condenser 0.969. The yield was practically the same in both cases. Because of the unstable nature of ascaridol we feel that it is important to have an efficient condenser that will cool the mixture of steam and oil as quickly as possible.

TABLE III.—EFFECT OF VARYING DISTILLATE TEMPERATURE.

$^1/_8$" inlet, 120 lb. pressure.

Temperature.	Distillation time.	Yield from 100 lb. plants.	Specific gravity.	Per cent. ascaridol.
50–55° C.	22 min.	260 cc.	0.960	71.5
35–45° C.	24 min.	268 cc.	0.969	78.1
20–25° C.	22 min.	282 cc.	0.970	80.1

We also distilled stalks alone and seeds alone. No oil was obtained from the stalks. The seeds alone gave an excellent yield of the highest grade of oil we obtained in our experiments; namely, 0.975. Table IV gives the comparative data.

TABLE IV.—DISTILLATION OF DIFFERENT PARTS OF PLANT.

$^1/_8$" inlet, 120 lb. pressure.

Part.	Distillation time.	Yield from 100 lb. plants.	Specific gravity.	Per cent. ascaridol.
Seeds	22 min.	438 cc.	0.975	85.5
Stalks	....	None	...	...
Whole plants	20 min.	282 cc.	0.970	80.1

We also set out and cultivated some wild wormseed plants found in our locality and obtained an oil having a specific gravity of 0.964. The yield was about 60% of that obtained from the other plants. This would indicate that the wild plants as found here could by cultivation and selection be made to yield oil of wormseed of good quality. We also distilled plants that had been shipped to us from Carroll County, Maryland, and obtained an oil which had a specific gravity of 0.966. Oil from our own plants, obtained under the same conditions of distillation, had a specific gravity of 0.970.