Fraction 7 does not fit into any pattern for a binary mixture, but does fit fairly well into the pattern for a ternary mixture which is mostly C_{32} and C_{34} with small amounts of C_{36}. Fraction 8 is virtually 100 per cent C_{32} alcohol but with small amounts of higher homologs as shown by the high melting points of the ethyl ester and acetate.

Carbon and hydrogen analyses were run on Fractions 3, 4, and 8 as shown in Table VI below:

TABLE VI.

Carbon, Hydrogen Analyses of Alcohols*

	Calcd. for $C_{28}H_{57}OH$	Found Fr. 3	Calcd. for $C_{30}H_{61}OH$	Found Fr. 4	Calcd. for $C_{32}H_{65}OH$	Found Fr. 8
% C	81.87	81.70	82.11	82.32	82.32	82.79
% H	14.23	14.38	14.24	14.17	14.25	14.27

*We are indebted to J. E. Varner of the Chemistry Department for these analyses.

Discussion

Fractional distillation of the free alcohols can, therefore, be used to separate the higher homologs of the n-aliphatic alcohols. It does have limitations in that slight decomposition does occur and the recrystallization of the fractions is necessary. However, with refinement in apparatus and technique these difficulties could be minimized.

Despite its limitations, this method has made possible for the first time the isolation from carnauba wax of the C_{28} alcohol, the C_{30} alcohol, and the C_{32} alcohol, in states of purity of 95 per cent or better. Moreover, by this method further evidence has been found to support the inference of Chibnall (2) that the alcohols of the wax include the C_{34} alcohol, and perhaps even higher homologs.

Finally, although previous workers had believed that the C_{30} alcohol was the principal alcohol of carnauba wax, it has been possible to show by means of this method that the C_{32} alcohol is even more abundant in the wax than is the C_{30} alcohol.

Summary and Conclusions

1. Fractional crystallization alone is not an adequate method for isolating pure alcohols from carnauba wax.

2. More than 50 per cent of the alcohols of carnauba wax can be distilled at 0.5 mm. pressure without serious decomposition.

3. Fractional distillation of the free wax alcohols at 0.5 mm. is a useful method of separating these compounds, but recrystallization of the resulting fractions is necessary.

4. There are substances in the unsaponifiable portion of carnauba wax which are of an unknown nature but which are probably not n-aliphatic alcohols.

5. For the first time, three alcohols, octacosanol (C_{28}), triacontanol (C_{30}), and dotriacontanol (C_{32}) have been isolated from carnauba wax in states of purity of 95 per cent or better.

6. Dotriacontanol is even more abundant in the wax than is triacontanol.

LITERATURE CITED

1. Brown, J. B., and Koonce, S. D. (soon to be published).
2. Chibnall, A. C., Piper, S. H., Pollard, A., Williams, E. F., and Sakai, P. H., Biochem. J. *28*, 2189 (1934).
3. Koonce, S. D., and Brown, J. B., Oil and Soap *21*, 167.
4. Longenecker, H., J. Soc. Chem. Ind. *56*, 199 T (1937).
5. Piper, S. H., Chibnall, A. C., and Williams, E. F., Biochem. J. *28*, 2175 (1934).
6. Pollard, A., Chibnall, A. C., and Piper, S. H., Biochem. J. *25*, 2111 (1931).
7. Shriner, R. L., and Fuson, R., The Systematic Identification of Organic Compounds (1935). John Wiley & Sons, New York.

Oil From Tumbling Mustard Seed

W. H. GOSS and J. E. RUCKMAN

Northern Regional Research Laboratory,[1] Peoria, Illinois

ALTHOUGH not a major item in our fat and oil commerce, rapeseed oil has performed a variable but significant role during the past decade in our domestic oil industries. In 1935 and 1936, it was used in considerable amounts in shortening, and to a lesser extent in soaps. In addition, there has been a fairly constant consumption of this oil in the manufacture of lubricants and rubber substitutes. Tariff restrictions were imposed in 1936 and 1938 which resulted in a discontinuance of large-scale use of the oil for food purposes, but denatured rapeseed oil for use in lubricants and rubber substitutes was exempted. Importations for these latter purposes continued, therefore, until war developments cut off supplies, which had been obtained almost entirely from Japan.

Rapeseed oil is of particular value in certain lubricants and in factice because of its content of erucic acid, a mono-unsaturated fatty acid containing 22 carbon atoms, which is present to the extent of about 50 percent of the component fatty acids in the oil. Few oils contain appreciable proportions of erucic acid, and that obtained from rapeseed is the only one that has been consistently available in any quantity. For use in marine lubricants, for which purpose it is blown to achieve a high viscosity, and in rubber substitutes, rapeseed oil is a difficultly replaceable item.

With the loss of its normal supplies of rapeseed oil, it was necessary for the United States to resort to whatever substitutes or new sources could be found. Initially, mustardseed oils from domestically grown seed were used to augment stocks, for these are quite similar to rapeseed oil both in composition and in their physical properties. More recently, however, it has been possible to procure substantial quantities of rapeseed oil from Argentina.

During the months when the rapeseed oil shortage threatened to assume serious proportions, considerable thought was given to the production of substitutes from still other oilseeds which had heretofore

[1] This is one of four regional research laboratories operated by the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

received little or no attention. A noteworthy example is fanweed seed, the oil of which was recently described by Clopton and Triebold (2) and by Schrader (5). Fanweed grows prolifically in the north-central and northwestern parts of the United States, and in southwestern Canada, and produces an abundance of seeds which are rich in oil. The fatty acid composition of fanweed seed oil is remarkably similar to that of rapeseed oil.

FANWEED is only one member of a large group of weeds which grow in the western wheat belt, and the seeds of which occur in varying proportions in grain screenings. Like rape, fanweed is a member of the mustard family. The various mustard and other seeds in grain screenings are frequently crushed to produce "grain screenings oil", and this material has received some consideration as a rapeseed oil substitute or extender. However, there appear to be no published data on its fatty-acid composition, and its properties would undoubtedly vary greatly, depending on the source of the screenings and proportions in which the many kinds of mustard and other seeds are present. Anderson *et al.* (1), for example, have recently published statistical data on the variation in contents of eight oilseeds and six other weed seeds in grain shipments from Fort William and Port Arthur, Ontario, Canada.

One of the commonest weed seeds screened out of wheat, particularly in Idaho, Montana, and certain other western areas, is tumbling mustard, *Sysimbrium altissimum* L. (*Norta altissima* Britton), which is also widely known as Jim Hill mustard because its introduction into these regions was a direct result of the transportation of mustard-containing grain over the Great Northern Railroad. It is a serious weed pest in grain and hay fields and grows abundantly along railroad right-of-ways and highways, in waste ground, and over a large part of the western range country, According to the Range Plant Handbook (3), a single plant will produce 1,500,000 seeds which are widely disseminated during the winter while the plants are being blown about over the countryside.

Tumbling mustard is a nuisance, not only because of its adverse effect on grain yields in contaminated fields but also because of its unpalatability to most livestock, with the exception of sheep. The seeds are extremely small, and during the harvesting of wheat they pass through the screens in the combines and fall back on the soil where they grow during the following season. If it were profitable to collect these seeds, along with the wheat, subsequently separating them for oil crushing or some other use, reseeding would be prevented to a large extent.

Experimental

A SAMPLE of tumbling mustard seeds obtained from wheat screenings in vicinity of American Falls, Idaho, was cleaned and analyzed. The results are shown below:

Moisture	5.46 percent
Oil	32.60 percent
Iodine value of oil (Wijs)	151.00
Free fatty acid in oil	0.42 percent
Nitrogen in air-dry oil-free cake	7.22 percent

The seeds were too small for satisfactory grinding in the Intermediate-Model Wiley mill, for a large proportion of them passed through both the 20- and 26-mesh screens. A small Labcono attrition mill, specially designed for grinding wheat, proved satisfactory and was used to prepare a relatively large sample of the seed for extraction. A medium-size Soxhlet apparatus was used to extract six successive batches, totalling 570 grams, from which 172.5 grams of oil was obtained.

The oil was converted to methyl esters, 100 grams of which was fractionally distilled in a concentric-tube column at a fairly high reflux ratio. Twelve fractions were obtained, but these were composited on the basis of iodine number into eight portions, for which iodine numbers and saponification equivalents were determined. The recovery from the initial charge of 100 grams was 99.3 grams.

The composition of each of the eight fractions was calculated from the iodine number and saponification equivalent, assuming that only three constituents were present in each. The average molecular weight furnished the basis for assuming which esters were present in any given fraction. This procedure resulted in the estimated overall composition of the oil shown in Table 1 as percentages of the total mixed acids. The compositions reported by Hilditch, Riley, and Vidyarthi (4) for typical rapeseed and mustard-seed oils, and by previously mentioned investigators for fanweed seed oil, are also listed for comparison.

TABLE 1

Estimated Composition of Tumbling Mustard Seed Oil, Compared With Reported Analyses of Other Erucic Acid-Containing Oils.

Component	Tumbling mustard	Rapeseed[1]	Black mustard-seed[1]	Fanweed[2]	Fanweed[3]
	percent	*percent*	*percent*	*percent*	*percent*
Myristic acid ($C_{14}H_{28}O_2$)	.....	.....	.....	trace	.....
Palmitoleic acid ($C_{16}H_{30}O_2$)	1.0	.....	.....	.....	.....
Palmitic acid ($C_{16}H_{32}O_2$)	14.1	1.0	2.0	1.5	.....
Oleic acid ($C_{18}H_{34}O_2$)	5.2	32.0	24.5	12.5	41.7
Linoleic acid ($C_{18}H_{32}O_2$)	19.0	15.0	19.5	33.0	21.2
Linolenic acid ($C_{18}H_{30}O_2$)	34.9	1.0	2.0	0.5	2.8
Erucic acid ($C_{22}H_{42}O_2$)	25.3	50.0	50.0	49.0	22.8
Lignoceric acid ($C_{24}H_{48}O_2$)	.....	1.0	2.0	3.5	.....
Unsaponifiable	0.5	.....	.....	.....	.....

[1] Hilditch, Riley, and Vidyarthi, loc. cit.
[2] Clopton and Triebold, loc. cit.
[3] Incomplete analysis, Schrader, loc. cit.

Discussion

OF the analyses listed in the table, only that of black mustardseed oil as reported by Hilditch *et al.* and that of fanweed seed oil, as reported by Clopton and Triebold, approach rapeseed oil in erucic acid content. The fractional distillation procedure employed by Clopton and Triebold is ordinarily more accurate for determining erucic acid than is the lead salt-ether method resorted to by Schrader.

In the case of tumbling mustard seed oil, erucic acid is present in appreciable quantity, but it falls far short of being an adequate replacement for rapeseed oil in this respect. The present trend in the oil and synthetic chemical industries is to consider the component fatty acids of an oil as potential raw

materials for industrial products, and means for fractionating the oils and their acids to obtain technically pure materials are now being made available. In view of these developments, it will be of interest, and of considerable value to the oil trade, to procure information on the compositions of the oils in the several other types of weed seeds available as waste products of the grain-processing industries.

LITERATURE CITED

(1) Anderson, J. A., Meredith, W. O. S., Eva, W. J., and Heise, A. C. Oil Seeds in Western Canadian Grain Screenings. Can. J. Res. *22F* (1) 19-27 (1944).
(2) Clopton, J. R., and Triebold, H. O. Fanweed Seed Oil. Ind. Eng. Chem. *36*, 218-9 (1944).
(3) Forest Service, U. S. Department of Agriculture. Range Plant Handbook, 1937.
(4) Hilditch, T. P., Riley, J., and Vidyarthi, N. L. The fatty acids of seed oils of Brassica species. The composition of rape, ravison, and the mustard seed oils. J. Soc. Chem. Ind. *46*, 457-62 T (1927).
(5) Schrader, Paul G. Penny Cress, a Promising New Plant Vehicle. Am. Paint J. *28* (23), 54, 56-7, 60, 64 (1944).

Report of the Cellulose Yield Committee

DURING the past year linter samples were sent out seven times by the committee to 13 laboratories. Each set consisted of three samples, A and B of lint and C of hull fiber. Nine laboratories completed all seven sets sent out; four laboratories completed five sets. A different type of lint or hull fiber was sent out each time; however, sample A always represented a good grade of second cut linters and B a poorer grade in regard to yield. Sample C, which was the hull fiber sample, was an average hull fiber grade.

The results were averaged by laboratories and are presented in the table below. Only the results of the laboratories which completed all seven sets were included in the overall average.

Lab. No.	Number sets of samples tested	Samples			Overall average year
		A Linters	B Linters	C Fiber	
1	7	77.3	72.9	69.6	73.3
2	7	77.9	73.2	70.1	73.7
3	7	77.8	73.4	70.5	73.9
4	5(1)	77.9	73.9	71.6	.....
5	7	78.1	73.5	70.8	74.1
6	7	78.3	73.4	70.7	74.1
7	7	77.6	73.0	70.2	73.6
8	5(1)	78.1	74.1	70.8	.....
9	7	79.0	74.4	71.2	74.9
10	5(1)	77.5	74.0	70.9	.....
11	7	78.4	74.1	71.1	74.5
12	7	78.6	73.8	70.8	74.4
13	5(1)	78.7	74.1	70.7	.....
Avg. (seven sets only)		78.1	73.5	70.6	74.1

(1) Five sets run, not included in average.

In the case of Laboratory No. 9, the results reported in one case were high. The reason for this was found and corrected. These analyses were included in the average above.

It was found during the past year as in previous years that it is very essential to watch the water pressure at which the samples are washed. Several cases of poor results have been due to this cause during the past year.

As no method of car sampling of linters and hull fiber has been adopted, the following procedure has been worked out and is recommended for adoption.

Car Sampling of Linters and Hull Fiber

LINTER samples should be taken in the dry. That is, if it is raining, take sample inside of car unless unloading under a shed which prevents the bales from getting wet.

One handful, 25 grams ± 5 grams, of linters is taken from each bale as it is unloaded. It should be taken at different positions on bales to insure a representative sample. The sample is taken as follows: Cut the bagging to remove the sample from underneath if necessary. Run the hand underneath the first layer of linters as far as possible, whenever possible, and obtain a handful. Where the bale is too tightly compressed to do this, pull off a small piece of the bale and throw away the outside portion.

The linters are put in as near an air-tight container as possible, 5 gallons ± 1 gallon, with a close fitting air-tight cover. A five-gallon G. I. can or other close fitting lid cans should be used. The container should be closed after each handful is put in, keeping the linters pressed down.

The container should be closed as near air-tight as possible when sent to the laboratory for analyses. By taking approximately 25 grams per handful, the car sample should weigh from 4 to 6 pounds.

If the sample of linters is to be sent some distance for analysis, a separate sample, of approximately 100 grams ± 10 grams, in a small air-tight quart size container, should be included for actual moisture determination. By some distance it is meant to another city where the sample has to be shipped by express or parcel post.

Note: It is thought advisable to include the extra sample for moisture if the sample is to undergo high temperature during shipment, as it is hard to obtain a large container which is air-tight under wide temperature changes. The moisture from the small sample is to be used for calculating the yield to the moisture as unloaded.

Recommendations

(1) That samples be sent out to laboratories for yield checks at least once every two months.

(2) That the car sampling procedure be adopted as a standard procedure for sampling linters and hull fiber.

E. H. TENENT — M. G. BOULWARE
E. C. AINSLIE — W. S. HUDE
C. H. COX — L. N. ROGERS, *Chairman.*