Solvents Committee Revises Specifications

Pentane Type Product Containing Minimum Amount of Hexane and Iso- Types Shown Most Desirable for Oil Extractions

By R. H. FASH, *Chairman*

THE present specifications of the American Oil Chemists' Society for petroleum ether are based upon a distillation test. Therefore, various mixtures of hydrocarbons can be made which would comply with the specifications, but, owing to the varying composition of these mixtures, there is a possibility of different amounts of extractive material being obtained on the same sample using different petroleum ethers. The investigation was first conducted to study the effect of varying the composition of the petroleum ether. The results obtained were such as to indicate that the temperature at which the extraction occurred influenced the amount of extracted material obtained. Therefore an investigation was made to determine the effect of varying the temperature at which extraction occurred.

Table I gives the results obtained upon extracting samples of cottonseed meal and cottonseed with various solvents. Table II gives the composition of some of the solvents.

Table I

Cottonseed Meal				Av.
Louisiana Gas	6.09%	6.06%	6.01%	6.05%
Skellysolve B	6.71 "	6.81 "	6.92 "	6.81 "
Skellysolve G	6.01 "	6.22 "	6.09 "	6.11 "
Skellysolve B 50% Skellysolve G 50%	6.49 "	6.40 "	6.33 "	6.41 "
Cottonseed				
Louisiana Gas	18.00%*	18.77%	18.68%	18.72%
Skellysolve B	19 53 "	19.58 "	19.45 "	19.52 "
Skellysolve G	18.58 "	18.18 " *	18.60 "	18.59 "
Skellysolve B 50% Skellysolve G 50%	18.91 "	18.70 "	18.69 "	18.77 "

Note: *—not considered in average.

Table II

	Skellysolve A	B	G	F	Louisiana Gas
Iso-pentane	0.71%		Trace	3.39%	36.09%
Normal pentane	97.73		28.04	70.36	25.75
Iso-hexane	1.56	0	5.06	5.05	5.02
Normal hexane		100	60.55	21.20	28.92
Heptane			5.81		4.22

Table III gives the results obtained upon extracting cottonseed meal with various mixtures of Skellysolve A and Skellysolve B compared with Louisiana Gas.

Table III

Skellysolve A	B		Oil %		Av.
100%	0%	5.25	5.22	5.18	5.18
80	20	5.24	5.25	5.24	5.24
60	40	5.25	5.29	5.20	5.25
40	60	5.41	5.20	5.30	5.30
20	80	5.44	5.45	5.33	5.41
0	100	5.50	5.47	5.42	5.46
Louisiana Gas		5.03	5.05	5.04	5.04

Table IV gives the results obtained on extracting cottonseed with mixtures of Skellysolve A and Skellysolve B, the cottonseed being handled in different ways with regard to preheating the seed in order to determine the effect of preheating upon the amount of ex-

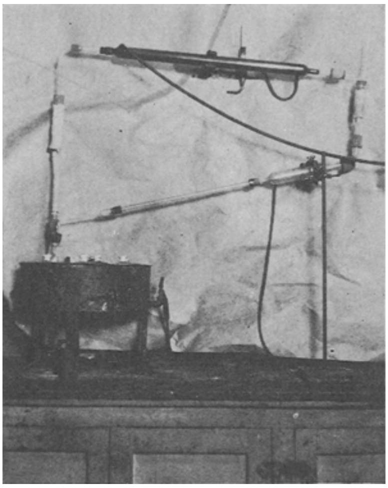

Figure I

tracted material obtained. The question had been raised as to whether in the preheating of the seed as carried out in practice, where the seed are in a mass, the desired polymerization of resinous materials was obtained. The preheating of the seed where the seed were spread out in a layer only one seed thick insured that the seed in this sample received the desired heating. The results indicate very little difference between the present procedure and heating in a thin layer.

In studying the results obtained using various solvents, A. K. Schwartz suggested that possibly the temperature at which extraction occurred and not the composition of the solvent was the deciding factor in determining the amount of extracted material obtained. If this were so, then, by extracting cottonseed with different solvents at the same temperature, the same amount of extracted material should be obtained, and the amount obtained should be greater the higher the temperature of extraction. In order to do this, the apparatus shown in figure I was arranged.

The temperature at which extraction occurs is regulated by the temperature of the water in the long condenser. The tube carrying the condensed solvent dips below the level of the liquid in the flask, while the small condenser prevents vapor from the flask entering the extraction tube. The tube carrying the vapor to the long condenser is fitted on the outside with nichrome wire through which an electric current is passed to heat the tube so that with the higher boiling point solvents sufficient solvent will be carried to the condenser to maintain the drops per minute required. The tube from the condenser to the extractor must be insulated to maintain the temperatures desired.

The results obtained using this equipment are given in Table V. These results confirm the theory that the amount of extracted material obtained is dependent upon the temperature of extraction and not the chemical composition of the paraffin hydrocarbons used as a solvent.

Table IV

Solvent Skellysolve A	B	Regular Official L	No Preheating M	1 Seed layer preheating P
100%	0%	16.67 16.58 16.31 Av. 16.52	16.33 16.81 Av. 16.57	16.62 16.44 16.60 Av. 16.55
80	20	16.14 16.16 16.22 Av. 16.17	16.81 16.66 16.67 Av. 16.71	16.73 16.43 16.70 Av. 16.62
60	40	15.97* 16.54 16.37 Av. 16.46	17.07 17.08 16.42* Av. 17.07	16.17 16.61 16.34 Av. 16.38
40	60	16.46 16.42 16.35 Av. 16.41	17.07 16.89 16.87 Av. 16.93	16.85* 16.34 16.47 Av. 16.40
20	80	—— 16.59 16.42 Av. 16.50	17.07 16.74 16.77 Av. 16.86	15.86* 16.60 16.49 Av. 16.55
0	100	16.91 16.56 16.72 Av. 16.73	17.18 17.09 16.73* Av. 17.14	16.68 16.63 16.35 Av. 16.55
Louisiana Gas		16.21 16.37 16.58 Av. 16.39	16.83 16.33 16.54 Av. 16.34	16.96* 16.34 16.23 Av. 16.28
Moisture, Ground		2.72%	7.47%	1.76%
Original Moisture		10.18 "	10.00 "	9.93 "

All figures calculated to 10% moisture basis

Note: * not considered in average

Table V

Temp. ° F.	Skellysolve A	Skellysolve B	Louisiana Gas
55	18.47%—18.53% av. 18.50%	18.55%—18.42% av. 18.49%	18.37%—18.32% av. 18.35%
80	18.85 "—18.77 " av. 18.81 "	18.60 "—18.70 " av. 18.65 "	18.92 "—18.75 " av. 18.83 "
105	Too warm	19.01 "—18.93 " av. 18.97 "	19.03 "—19.17 " av. 19.10 "
130	Too warm	19.26 "—19.21 " av. 19.24 "	Too warm

(*Turn to page* 317)

Kapok Oil

(*From page* 305)

In all these tests, the color developed by the kapok oil is much deeper than that developed by the same percentage contamination of cottonseed oil. Further, the color produced by any given mixture with kapok oil is equivalent to that given by mixtures containing cottonseed oil in quantities ten or more times as large as the particular kapok admixture. Mixtures of oils containing cottonseed oil in amounts of 5% or more give colors of such deep red, that the depth of color is of very little value, as indicating the percentage of contamination. This is also true of kapok mixtures containing 1% kapok oil, or more.

These tests, of course, were made on cottonseed oil, which had not been subjected to any heat that would vitiate the Halphen reaction.

◆

Solvent Specifications

(*From page* 312)

The fact that the temperature of extraction determines the amount of extracted material obtained leads to some interesting conclusions regarding the determination of oil in cottonseed products by different laboratories. In South Texas, cottonseed start moving in July. Therefore, the laboratories have to make analyses during the hottest part of the year. The petroleum ether which has been used contains a large amount of iso-pentane quickly lost when extracting in hot weather, necessitating the refilling of the extraction flask two and three times, thereby materially increasing the amount of hexane in the solvent, which would tend to give a higher percent of oil than that obtained by laboratories operating in cooler climates.

Complaint was made against the oil results obtained by one laboratory being materially higher than those obtained by another laboratory, both using the same petroleum ether and operating in the same city. On investigation, the fact was found that the laboratory obtaining the lower results was using a Smalley extraction tube, while the other laboratory was using a Butts tube. Both tubes are of the percolating type, but the Smalley tube has a side arm which carries the hot vapors of the solvent direct to the condenser without the vapors coming in contact with the material being extracted, which contact does occur in the Butts tube. On substituting a Butts tube for the Smalley tube, the results obtained by the laboratory obtaining the lower results checked the results obtained by the laboratory obtaining the higher results. The conclusion was reached, independent of the work of this committee, that the differences between the two laboratories was due to the difference in temperature at which extraction occurred.

A number of the samples of cottonseed which had been extracted at a temperature of 80° C., to obtain the results given in Table V, were re-extracted in the ordinary manner. The material extracted was solid and reddish-brown in color, apparently of a resinous nature.

Conclusions

The solvent used should be of the pentane type, containing a minimum amount of iso-pentane, iso-hexane, and hexane. The following specifications would give a product meeting the above requirements:

Initial boiling temperature —not less than 35° C.
" " " — " over 40° C.
Dry flask end point — " " 60° C.
" " " " — " less than 50° C.
At least 95% distilling under—55° C.
Not over 85% " " —40° C.
Specific gravity at 60° F. —.630 to .675
Color—water white
Evaporation residue—not over .002% by weight
Doctor test—sweet
Copper strip corrosion test—non-corrosive
Unsaturated compounds—trace only permitted

This product would have approximately the following composition:

Iso-pentane	0%	to	5%	or about	2%
Normal pentane	70%	"	85%	" "	80%
Iso-hexane	5%	"	20%	" "	9%
Normal hexane	5%	"	20%	" "	9%

◆

Linseed Crush

According to preliminary figures of the Census Bureau there were 25 mills in the United States which crushed flaxseed during the quarter ending June 30, 1931, reporting a crush of 201,223 tons of flaxseed and a production of 130,324,839 pounds of linseed oil. These figures compared with 203,568 tons of seed crushed and 130,863,405 pounds of oil produced for the corresponding quarter in 1930, and 278,933 tons of seed and 187,018,538 pounds of oil in 1929.

Stocks of flaxseed at the mills on June 30, 1931, amounted to 33,535 tons, compared with 65,173 tons for the same date in 1930 and 100,928 tons in 1929. Stocks of linseed oil reported by the crushers were 47,156,191 pounds on June 30, 1931, compared with 71,222,982 pounds for the same date in 1930, and with 83,544,918 in 1929.