•*Technical*

Solvent Hydrogenation of Cottonseed Oil

LYLE F. ALBRIGHT, CHIN-HSUAN WEI,[1] and JOHN M. WOODS,
Purdue University, Lafayette, Indiana

Refined and bleached cottonseed oil was dissolved in a solvent (hexane, isopropyl alcohol, or di-isopropyl ether) and was then hydrogenated in a dead-end hydrogenator. Hydrogenation runs were conducted at temperatures from 115 to 145°C., at hydrogen partial pressures from 44 to 74 p.s.i.a., with catalyst concentrations varying from 0.05 to 0.40% nickel, and at high rates of agitation to eliminate mass-transfer resistances. A series of hydrogenation runs was also made in which no solvent was used.

The rates of hydrogenation for the various series of runs were in the same order of magnitude but decreased in the following order: nonsolvent, hexane, isopropyl alcohol, and di-isopropyl ether runs. Selectivity and isomerization were low in all cases and essentially identical for solvent and nonsolvent runs.

The rate of hydrogenation increased in all cases with higher catalyst concentrations. For the isopropanol runs, the reaction rate was maximum as a function of temperature at about 135°C. In the case of the other solvents, the rate of hydrogenation increased with increased temperature in the range from 115 to 145°C., but the rate increases of the solvent runs were less than those of the nonsolvent runs.

HYDROGENATION of triglyceride oils dissolved in various solvents has been practiced for many years in the laboratory, and recently a continuous flow process, using solvents, has been patented by Sanders (7). Higher selectivity and less isomerization are claimed with the solvent type of hydrogenation as compared to nonsolvent type of hydrogenation. At low temperatures the rate of hydrogenation was also improved significantly. Solvents which are inert and which are relatively volatile in reaction conditions are necessary; methanol, ethanol, isopropyl alcohol, cyclohexanol, acetone, and ethyl ether were, in particular, recommended. Unpurified commercial hexane, petroleum ether, and dioxane were reported to decrease the rate of reaction however.

Dissolving the triglyceride in a solvent may change several properties of the system that could account for the characteristics of the hydrogenation as claimed in the patent (7). Some of these factors are as follows.

a) Decrease the viscosity of the liquid phase so that mass-transfer resistances of the reactants and products to and from the catalyst are substantially reduced. As a result, concentrations of reactants during the hydrogenation may be increased on the catalyst surfaces.

b) Modify the solubility of hydrogen in the liquid phase. The use of polar solvents, such as alcohols and acetone, may increase hydrogen concentrations possible in the liquid.

c) Change the adsorption characteristics and concentrations of reactants on the catalyst surfaces. The solvents, as well as the reactants, probably will be adsorbed to some extent on the catalyst; as a result, the number of "active sites" available for hydrogenation reactions may be reduced.

d) The solvent may enter into reactions, *e.g.*, under some conditions, alcohols are reduced to aldehydes with the concomitant release of hydrogen (4, 5, 6). The aldehydes could subsequently be hydrogenated back to alcohols, continuing a reaction cycle.

e) Reduce the concentration of triglyceride oil (and of the double bonds). In many cases the rate of hydrogenation is directly proportional to the iodine value of the mixture.

f) Modify the physical characteristics of the system by internal refluxing of the solvent during hydrogenation. If the top of the reactor is colder than the main body of the oil, the solvent will condense on the top surface and drop back into the liquid phase. The continuous boiling of the solvent may affect both the heat and mass-transfer effects within the system.

Sanders' patent (7) does not indicate whether the operating conditions for his solvent and nonsolvent hydrogenation runs were directly comparable. For example, the total pressure in a solvent run has little theoretical importance since the gas phase is a mixture of volatile solvent plus hydrogen. The total absolute pressure is the sum of the partial pressures of the solvent and hydrogen. The partial pressure of the hydrogen will be a function of the temperature, the type of solvent, and composition of the liquid phase. The hydrogen partial pressure in solvent runs should be compared to the total absolute pressure in nonsolvent runs, but apparently Sanders did not do this. Although he used high rates of agitation in his investigation, there is no specific evidence that mass-transfer resistances were eliminated. As a result, modifications of the selectivity and isomerization characteristics of the hydrogenated oils may have been caused primarily by variations of the reactant concentrations on the catalyst.

Sokol'skiĭ *et al.* (8) have recently reported on the kinetics of the solvent hydrogenation of cottonseed oil. Their investigation was limited to total pressures of one atmosphere. Since the partial pressure of the solvent varies with temperature, their studies at various temperatures were consequently at different hydrogen partial pressures, and the results are of limited value. The most rapid rates of reaction occurred at hydrogen pressures of about 600 mm. Hg regardless of the temperature. Solvents studied included aromatics, alcohols, dioxane, and dichloroethane.

Solvent hydrogenation has, possibly, industrial importance. For example, cottonseed or soybean oil, as obtained by solvent extraction, might be refined, bleached, and hydrogenated in the original solvent solution. The present investigation was made to characterize more completely solvent hydrogenation. Hydrogenation runs were made with cottonseed oil dissolved in isopropyl alcohol, hexane, or di-isopropyl ether; these runs were compared to runs with undissolved cottonseed oil at similar temperatures, hydrogen partial pressures, and nickel catalyst concentrations, and with sufficient agitation to eliminate most mass-transfer resistances.

[1] Present address, University of Wisconsin, Madison, Wis.

Equipment and Operating Procedure

The dead-end hydrogenator and auxiliary equipment used are shown schematically in Figure 1. The hydrogenator was 6 in. i.d. and had a capacity of about 6 liters. It was provided with a 4-in. turbine impeller manufactured by the Mixing Equipment

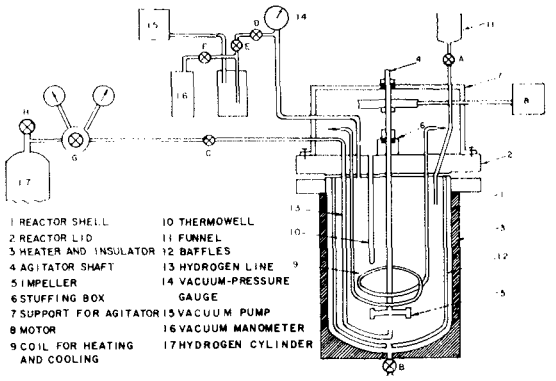

FIG. 1. Scheme of assembled apparatus.

Company and with four baffles, 5/8 in. wide. Agitator speeds could be varied from several hundred up to about 1,700 r.p.m.

The cottonseed oil or oil-solvent mixture and the catalyst were introduced to the hydrogenator through the funnel (11). In the case of nonsolvent runs, 1,500 or 2,000 cc. of oil were added for each run; for solvent runs, the mixtures contained 1,000 cc. of oil and 3.84 g. moles of solvent. This amount of solvent is equivalent to approximately 500, 300, and 540 cc. of hexane, isopropyl alcohol, and di-isopropyl ether, respectively. Vapor pressures of the solvents in these mixtures were measured in the hydrogenator at several temperatures, and the results are shown in Table I. It should be emphasized that the liquid composition varied slightly with temperature since a relatively large vapor volume was present above the liquid. The partial pressure of hydrogen during a hydrogenation run was assumed to be equal to the total pressure minus the vapor pressure of the solvent (Table I).

After the hydrogenator was filled with the oil mixture, the system was evacuated for several minutes by means of the vacuum pump (15). The hydrogenator was heated to the desired temperature, using the steam-heated coil (9) and/or electrical resistance wiring wrapped around the reactor shell. Temperature measurements were made with a thermocouple (10) and potentiometer. When the temperature of the hydrogenator was raised to within 10°C. of the desired operating temperature, hydrogen was introduced and maintained at the desired pressure, and the agitator motor was turned on to start the run.

The temperature generally rose to the desired level within the first few minutes of a run. The heaters were turned down or off; in some cases low steam pressure was provided in the coil to maintain the desired temperature. With experience the temperature was controlled manually to within at least ± 3°C. The agitator speed was measured at frequent intervals with a tachometer.

Six or seven oil samples generally were taken during a run through Valve B. Since most runs were about 1 hr. long, the time between samples was approximately 10 min. The first 10 ml. of the sample collected were discarded, and the next 10 cc. were filtered to remove the catalyst. When solvent was present, it was evaporated from the oil; and the oil was analyzed by Procter and Gamble Company, according to conventional procedures.

TABLE I

Vapor Pressure of Mixtures of Cottonseed Oil and Solvent

Solvent	Pressure, p.s.i.a.			
	115°C.	125°C.	135°C.	145°C.
Hexane	38	46	56	67
Isopropyl alcohol	39	50	63	77
Di-isopropyl ether	37	45	54	65

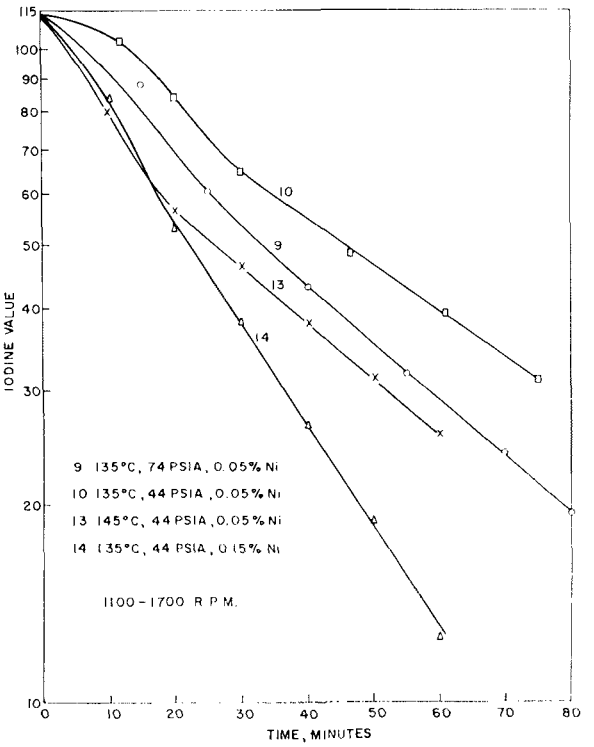

FIG. 2. Hydrogenation rate of cottonseed oil without solvent.

During runs with solvents, small leaks occurred frequently at the packing gland of the agitator. Presumably the hot solvent condensate was removing the lubricant from the packing gland. Such a leak is of special importance for solvent runs because solvent vapors were being lost in addition to hydrogen. Since the total pressure remained constant during a run, the partial pressure of the hydrogen increased as solvent was lost. Efforts to repair the packing gland and reduce the solvent losses were, as a rule, only partly successful. The initial and final ratios of the solvent and oil were measured in several cases, and up to about 30% of the solvent was lost in a few runs.

The refined and bleached cottonseed oil for the main series of runs was furnished by the Procter and Gamble Company. Rufert flake catalyst containing 25.3% nickel was obtained from the Harshaw Chemical Company. The electrolytic type of hydrogen was procured from the Linde Air Products Company. Commercial grade hexane (Phillips Petroleum Company), 99% isopropyl alcohol (Union Carbide Chemical Company), and commercial-grade of di-isopropyl ether (Union Carbide Chemical Company) were employed as solvents.

Results

Nonsolvent Hydrogenation Runs. A series of nonsolvent hydrogenation runs was made as a comparison for the solvent runs and, in addition, to establish operating conditions at which increased agitation did not change the rate of hydrogenation, *i.e.*, mass-transfer resistances were essentially eliminated. The rate of hydrogenation for several typical runs is indicated in Figure 2, which is a plot of the logarithm of the iodine value *versus* time. The data for each run generally are represented by straight lines at iodine values less than 60, at which value the linoleic acid content of the oil was approximated zero (Figure 3).

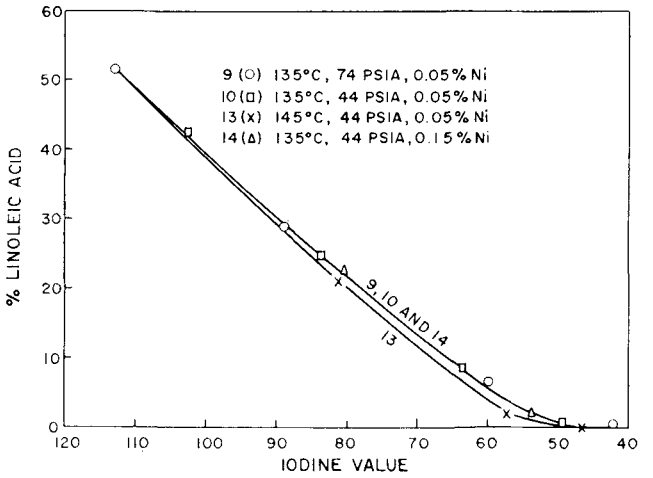

FIG. 3. Selectivity during nonsolvent hydrogenation of cottonseed oil.

The straight line relationships indicate a pseudo-first-order over-all reaction, the rate of which is represented by the following equation:

(1) $r = d(I.V.)/dt = k(I.V.)$

where

r = rate of hydrogenation
t = time
I.V. = iodine value
k = pseudo-reaction-rate constant, which is equal to the negative slope of the straight line divided by 2.3 (since the logarithm plots were to the base 10).

Table II reports the k values of several runs. The reproducibility of these values is thought to be within about 5%, as was demonstrated by repeating Runs 1 and 3. In some cases agitator speeds of 1,000 r.p.m. did not give k values as high as comparable runs at 1,700 r.p.m. (see Runs 1, 3, 8, and 9). The k values of these runs were relatively high, being about 0.020 min.$^{-1}$, and it was concluded that 1,000 r.p.m. did not give sufficient agitation to eliminate mass-transfer variables at these high rates of hydrogenation. At lower rates however, as indicated by Runs 10, 11, and 12, an agitator speed of 1,000 r.p.m. appears sufficient to eliminate most mass-transfer resistances. Increased temperature (compare Runs 10, 11, and 12), higher catalyst concentrations (Runs 10, 11, and 14), and increased pressures (Runs 8, 9, 10, 11, and 12) all increased the rate of hydrogenation. The k values did not vary linearly with catalyst concentration; therefore it is concluded that mass-transfer resistances were significant at the rates accompanying the higher

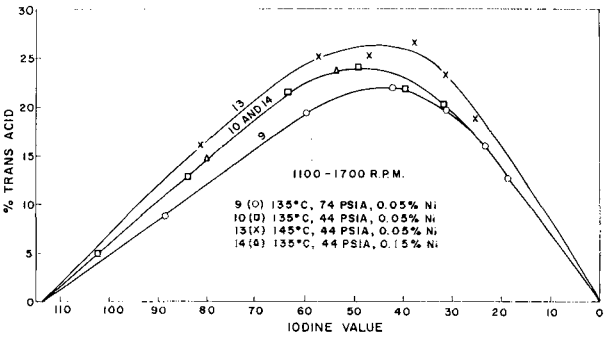

FIG. 4. *trans*-Isomerization during nonsolvent hydrogenation of cottonseed oil.

catalyst concentrations, which had k values as great as 0.0342 min.$^{-1}$

The selectivity (as indicated by the linoleic acid content) and isomerization characteristics of Runs 9, 10, 13, and 14 are indicated by Figures 3 and 4, respectively. Relatively nonselective hydrogenation occurred in all runs. All runs at 135°C. had approximately the same selectivity, and the run at 145°C. was only slightly more selective. Eldib and Albright (2) had also previously shown that selectivity is only slightly affected by temperature or catalyst concentration when high rates of agitation are provided. Figure 4 indicates that higher temperatures and lower pressures for hydrogenation resulted in slightly higher amounts of *trans* isomers.

The rates of hydrogenation of the runs shown in Figure 2 were compared to similar runs of Eldib and Albright (2). The k values for their runs are somewhat lower than those of Figure 2. Although the same catalyst was used, the cottonseed oil of this investigation was possibly sufficiently different from that of the previous study to account for the differences. Figure 5 indicates that the above reasoning may be correct. A refined and bleached cottonseed oil sample was obtained from each of three American companies that hydrogenate cottonseed oil industrially. These three oils were hydrogenated at 135°C., with an hydrogen pressure of 44 p.s.i.a. and 0.05% nickel catalyst, employing an agitator speed of 1,100 r.p.m. The agitator used for this series of runs was probably less efficient though than that of the runs of Table II; as a result, the present hydrogenation rates are lower. Oils B and C hydrogenated, within experimental accuracy, at approximately same rate; however Oil A hydrogenated significantly slower. The selectivity and isomer formation for the three oils were essentially identical. The soap and peroxide analyses for the oils indicated no significant difference, but the free fatty acid content of Oil A is appreciably

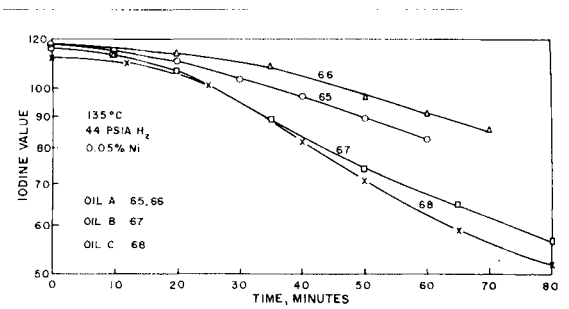

FIG. 5. Comparison of hydrogenation rates of three refined and bleached cottonseed oils.

TABLE II

Reaction-Rate Constants for Nonsolvent Runs

Run number	Temperature, °C.	H_2 pressure, p.s.i.g.	Catalyst concentration, % Ni	Agitation, r.p.m.	k, $min.^{-1}$
1	145	60	0.07	1000	0.0310
3	145	60	0.07	1700	0.0352
8	135	60	0.05	1000	0.0197
9	135	60	0.05	1700	0.0205
10	135	30	0.05	1000	0.0158
11	135	30	0.05	1700	0.0150
12	135	30	0.05	1100	0.0140
13	145	30	0.05	1100	0.0196
14	135	30	0.15	1100	0.0342

higher and may account for the reduced hydrogenation rate.

Hydrogenation of Oil Dissolved in Hexane. Figure 6 shows the relationship of the iodine values as a function of time for hydrogenation runs, using hexane as a solvent. The rates of hydrogenation for these runs are slightly lower than those of the nonsolvent runs. Table III indicates k values of the runs.

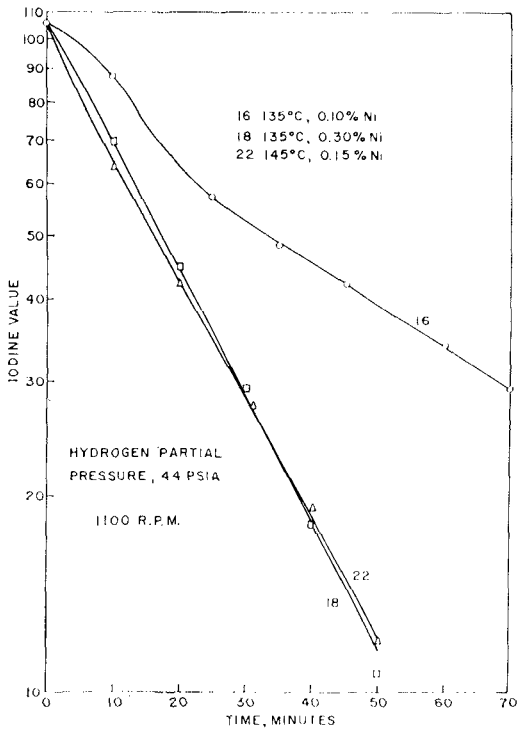

FIG. 6. Hydrogenation rate of cottonseed oil dissolved in hexane.

Since commercial hexane was used, such catalyst poisons as sulfur compounds could possibly have been present. An effort was therefore made to remove any poisons from the hexane. Some hexane was pretreated with fresh nickel catalyst at 135°C. for an hour, then the catalyst was filtered from the oil. This technique did not alter the hydrogenation rate, selectivity, or formation of *trans* isomers. If catalyst poisons were present moreover, part of the catalyst would become ineffective because of the poison. Doubling the catalyst concentration, for example, should presumably then more than double the rate of hydrogenation. None of the results of this investigation however indicate that catalyst poisons were significant in the solvents.

The reproducibility of the runs, using hexane (or other solvents), was poorer than similar nonsolvent

TABLE III

Reaction-Rate Constants for N-Hexane Runs

Run number	Temperature, °C.	Total pressure, p.s.i.g.,	Catalyst concentration, % Ni	k, $min.^{-1}$
15	135	86	0.05	0.0130
16	135	86	0.10	0.0150
17	135	86	0.20	0.0198
18	135	86	0.30	0.0447
19	135	86	0.30	0.0441
20	135	86	0.30	0.0486
21	135	86	0.225	0.0382
22	145	98	0.15	0.0402

Operating Conditions: oil, 1 liter; solvent, 500 ml.; hydrogen partial pressure, 45 p.s.i.a.; agitation, 1,100 r.p.m.

runs as plots of the k value *versus* the catalyst concentration indicate. The poorer reproducibility of the solvent runs is probably caused in part at least by variable solvent losses during the runs. The formation of *trans* isomers (Figure 7) and the selectivity of the hexane runs were essentially identical to comparable nonsolvent runs.

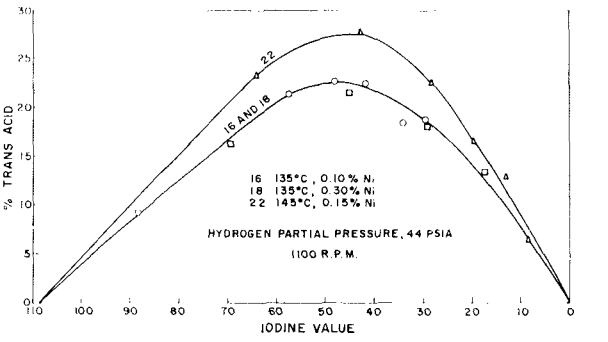

FIG. 7. *trans*-Isomerization during hydrogenation of cottonseed oil dissolved in hexane.

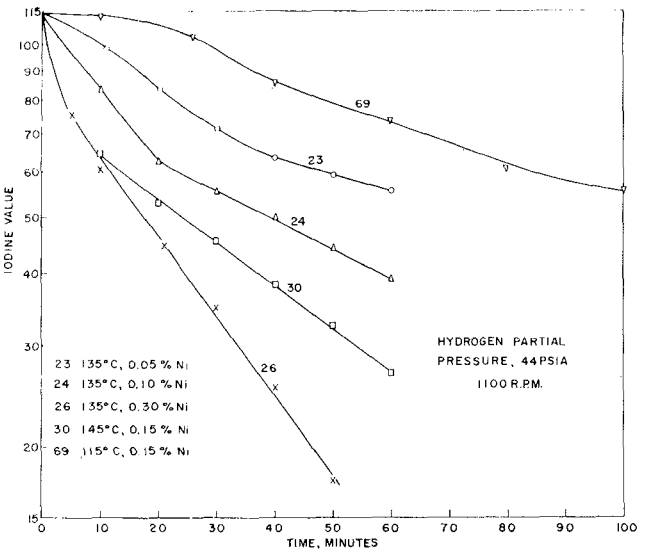

FIG. 8. Hydrogenation rate of cottonseed oil dissolved in isopropyl alcohol.

One run, using hexane, was made at 115°C. but with a less efficient agitator. The rate of hydrogenation was quite low, and the selectivity and isomerization that occurred during this run were appreciably higher than other hexane runs.

Hydrogenation of Oil Dissolved in Isopropyl Alcohol. The results for the isopropyl alcohol runs are shown in Table III and in Figures 8 and 9. The rates

TABLE IV

Reaction-Rate Constants for Isopropyl Alcohol Runs

Run number	Temperature, °C.	Total pressure, p.s.i.g.	Catalyst concentration, % Ni	k, min.$^{-1}$
23	135	93	0.05	0.0089
24	135	93	0.10	0.0112
25	135	93	0.20	0.0260
26	135	93	0.30	0.0281
27	125	80	0.30	0.0140
28	135	93	0.30	0.0260
29	135	93	0.20	0.0230
30	145	106	0.15	0.0169
31	145	106	0.15	0.0146
32	135	93	0.40	0.0302

Operating Conditions: oil, 1 liter; solvent, 300 ml. (237 g.); hydrogen partial pressure, 45 p.s.i.a.; agitation, 1,100 r.p.m.

of hydrogenation were somewhat less than those of the hexane and nonsolvent series of runs. As expected, rate of hydrogenation increased with higher catalyst concentrations. Since the relation was not linear, mass-transfer resistances were presumably significant at the higher catalyst concentrations. The formation of *trans* isomers, as shown in Figure 9,

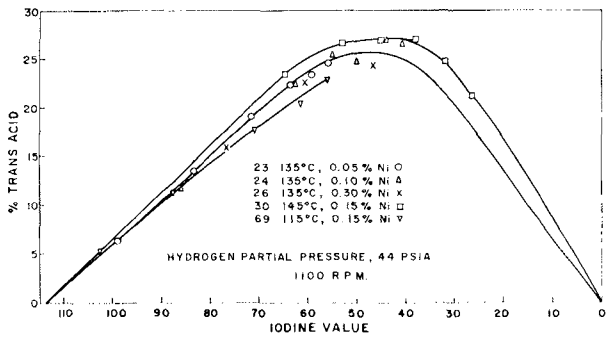

FIG. 9. *trans*-Isomerization during hydrogenation of cottonseed oil dissolved in isopropyl alcohol.

and selectivity did not vary significantly with catalyst concentration, and they increased to only a small extent with higher temperatures. Both the selectivity and isomerization of the isopropyl alcohol runs were slightly higher than those of the nonsolvent and hexane runs.

The average k value for two runs (Runs 30 and 31) at 145°C. is 0.0158 min.$^{-1}$. A comparable k value for 135°C. is 0.0191 min.$^{-1}$, a value determined by interpolating the results of Runs 23–29 and 32 to 0.15% of nickel catalyst concentration. The difference in k values between 135 and 145°C. may not be real; at best the k value at 145°C. is slightly higher than the one at 135°C. however. A significant decrease of k values occurs as the temperature decreases from 135 to 124°C. (Runs 26, 27, and 28). Apparently the maximum rate of hydrogenation occurs in the range of about 135 to 145°C.

Hydrogenation of Oil Dissolved in Di-isopropyl Ether: The results of hydrogenation runs in which cottonseed oil was dissolved in di-isopropyl ether are shown in Figures 9 and 10. Runs were made for 1 hr. and the iodine value dropped only to about 50 to 64. Plots of the logarithm of the iodine value *versus* time (Figure 9) do not approach straight lines adequately to determine the k values. The average rates of reaction were obviously less for these runs than those of the other runs. The rates did increase with both increased catalyst concentration and temperature in the range investigated. The increase of the rate with

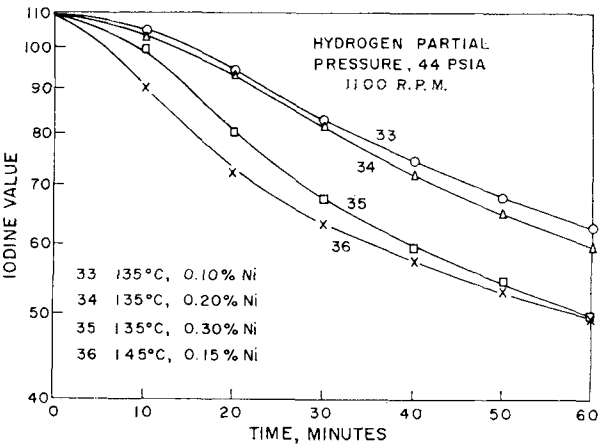

FIG. 10. *Hydrogenation rate of cottonseed oil dissolved in* di-isopropyl ether.

temperature was however less than comparable nonsolvent runs. The *trans*-isomer formation, as shown by Figure 11, and selectivity were approximately the same as those obtained in the isopropyl alcohol runs.

Discussion of Results

The present investigation has demonstrated that small leaks of the hydrogenator are more critical in the case of the solvent type of hydrogenation as compared to the conventional nonsolvent type. Mechanical difficulties, particularly in regard to the packing glands, will tend to be more common, and the glands

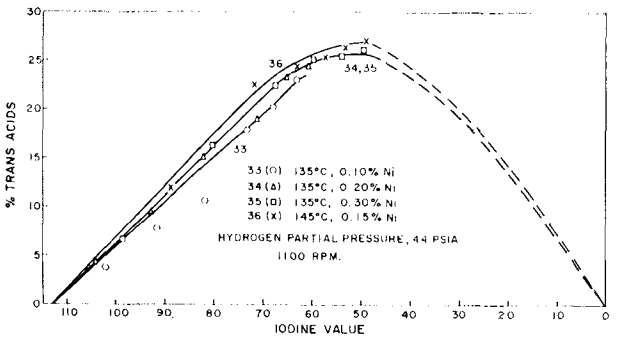

FIG. 11. *trans*-Isomerization during hydrogenation of cottonseed oil dissolved in di-isopropyl ether.

must be designed to operate in solvent vapors. Although the results of this investigation were not as reproducible as desired because of leakage at the packing gland, several characteristics of the solvent type of hydrogenation were determined. The solvent had less effect on the rate of hydrogenation, selectivity, and isomerization than had been anticipated. Hexane was reported by Sanders (7) to decrease the rate of hydrogenation significantly, but the rates of hydrogenation with hexane were slightly higher than those with the two polar solvents, which were reported to improve the rates.

Sanders (7) had also indicated significant changes in selectivity and isomerization because of the solvent. On the contrary, no such differences were found in this investigation. Probably he had operated under conditions relatively different from those used here. First, he may not have had sufficient agitation to eliminate mass-transfer resistances. Further the cata-

lyst that he used was more active than the one employed in this study as tests in our laboratory had shown. Many of his runs were at a temperature lower than 115°C., the lowest temperature of the study. The catalyst used here was not appreciably active at such low temperatures. Investigations in which vigorous agitation is used and in which an effective low-temperature catalyst is employed are therefore still necessary to determine the variables of importance in the solvent type of hydrogenations at relatively low temperatures.

The use of a solvent in the reaction vessel was found to have a significant effect on the relationship between the hydrogenation rate and temperature. Without a solvent the maximum rate occurs well above 200°C. (1), but it appears to be appreciably less with solvents, *e.g.*, about 135–145°C. when isopropyl alcohol was used. The optimum temperature is undoubtedly controlled by the complex relationship of the true reaction-rate constants, the mass-transfer resistances, and the solubility of hydrogen in the liquid phase. Equilibrium solubilities of hydrogen in organic liquids are interesting and relatively unique since in several cases at least the solubility increases with increased temperature, *e.g.*, solubilities in triglycerides (1, 9) and in toluene (3). A better understanding of the solubility phenomenon would probably help explain the effect of temperature and pressure on the hydrogenation reaction. When a solvent is employed, the hydrogen solubility, no doubt, changes radically and may be responsible for the lower optimum temperatures found in this investigation.

The present results indicate that a commercial process might be practical in which the cottonseed oil is hydrogenated while still in the solution obtained by the solvent extraction of the oil. The rates of hydrogenation are in the same order of magnitude even though the solvent diluted the oil and hence decreased the concentration of unsaturated bonds.

Acknowledgment

The Procter and Gamble Company donated the refined and bleached cottonseed oil and performed most of the analyses. H. K. Hawley of that company offered valuable advice at frequent intervals during the investigation.

Conclusions

Mixtures of cottonseed oil and hexane, isopropyl alcohol, or di-isopropyl ether were hydrogenated in a dead-end hydrogenator. The rates of hydrogenation of the cottonseed oil for solvent hydrogenation were somewhat less than conventional nonsolvent hydrogenations. The selectivity and *trans*-isomerization characteristics were essentially unchanged by the presence of the solvent.

REFERENCES

1. Bailey, A. E., "Industrial Oil and Fat Products," 2nd ed., Interscience Publishers, New York (1951).
2. Eldib, I. A. and Albright, L. F., Ind. Eng. Chem., *49*, 825 (1957).
3. Littman, H., and Bliss, H., Ind. Eng. Chem., *51*, 659 (1959).
4. Lyubarskii, E. J., J. Applied Chem. U.S.S.R., *5*, 1025 (1932).
5. Markley, K. S., "Fatty Acids: Their Chemistry and Physical Properties," p. 373, Interscience Publishers, New York (1947).
6. Rush, V. A., Dvinyanenkova, I. L., and Lyubarskii, J. Applied Chem. U.S.S.R., *10*, 702 (1937).
7. Sanders, J. H., U. S. Pat. 2,520,440 (1950).
8. Sokol'skii, D. V., Melekhina, L. S., and Perainova, L. I., J. of Applied Chem. U.S.S.R., *30*, 1869 (1957).
9. Tyutyunnikov, B. N., and Novitskaya, I. I., Masloboino-Zhirovaya Prom., *23*, (8), 13 (1957).

[Received September 15, 1959]

Differentiation of Hydrogen Bromide-Reactive Acids in Seed Oils

C. R. SMITH JR., M. C. BURNETT,[1] T. L. WILSON, R. L. LOHMAR, and I. A. WOLFF,
Northern Regional Research Laboratory,[2] Peoria, Illinois

A METHOD is presented for differentiating between epoxy acids and those without epoxy groups that react similarly in the Durbetaki titration of seed oils. Application of the technique to selected oils is discussed. The basis of the procedure is titration with hydrogen bromide before and after reduction of the acid with lithium aluminum hydride. A procedure is also presented for verifying one of the interfering nonepoxide-containing oils, dimorphecolic acid. The latter procedure is based on isolation of the acid by solvent partitioning and reduction to a crystalline derivative.

The discovery that an epoxy acid, vernolic acid, is the principal fatty acid in *Vernonia anthelmintica* seed oil led to a search, both at this laboratory (1, 2) and elsewhere (3, 4, 5), for other natural sources of epoxy acids by examining numerous seed oils. Vernolic acid, shown by Gunstone (6) to be *cis*-12,13-epoxy-*cis*-9-octadecenoic acid, has also been found in seed oils of several other species, including *Vernonia colorata* (3), *Hibiscus esculentus* (3), *Hibiscus cannabinus* (4), and *Clarkia elegans* (7). In addition, three other naturally-occurring epoxy acids have been discovered in seed oils: 9,10-epoxystearic acid, found in *Tragopogon porrifolius* oil (5), coronaric acid, present in *Chrysanthemum coronarium* oil (7, 8) and 15,16-epoxylinoleic acid, present in *Camelina sativa* oil (9).

The Durbetaki method for determination of oxirane oxygen (10, 11) is used routinely at this laboratory as a screening method for detecting epoxy acids in seed oils. It is based on the opening of the oxirane ring to form a bromohydrin:

[1] Present address: Chico State College, Chico, Calif.
[2] This is a laboratory of the Northern Utilization Research and Development Division, Agricultural Research Service, U. S. Department of Agriculture.