

ing studied or because of the nature of the study itself. For example, one company may request that a raw material be extracted but not desolventized. This necessitates the disjoining of the desolventizing drier from the extraction system and the collecting of the extracted material wet with solvent. Another company may request that the miscella from an extraction be collected after distilling to a rich miscella of 50% solvent or not distilled at all. Another customer may request that an extracted, solid material be mechanically desolventized instead of thermally desolventizing.

These conditions inform the design engineer and the operator that the solvent pilot plant must be shut down and started up perhaps one or two times a day. And yet the solvent pilot plant must be capable of 24 hours a day, seven days a week operation when required.

Other papers in this symposium have emphasized the extreme importance of continuous, smooth operation of a commercial-scale, solvent plant in relationship to safety in that plant. They have stressed the importance of plant design, safety engineering, and safety equipment in the reduction of hazards in the operation of commercial-scale, solvent plants. The relative merits of steam purging *versus* inert gas-purging, the importance of ventilation and many other aspects of safety have been likewise thoroughly discussed. All of these factors are important to the safe operation of a pilot plant.

ONE FACTOR however that cannot be systematized, recorded, or scientifically detected is the human factor. One can provide an immense fan to ventilate

the pilot-plant area, but to do any good the fan must be turned on.

As far as specific safety items are concerned, the solvent pilot plant is designed in accordance with the same safety specification of commercial-scale equipment. Double fire doors and a fire wall separate the plant from the rest of the building. The floor of our pilot plant is elevated approximately 15 feet above the ground and three feet below the floor elevation of the adjoining Expeller pilot plant. Sufficient floor ventilation is provided to change the air in the plant area once every three minutes. In addition, the length of one wall is provided with windows. At shut-down all solvent is pumped or drained into vapor-tight containers. Sparge steam is utilized for purging prior to equipment alterations. If the pilot plant is to be left idle, all solvent is pumped to storage and the entire unit is purged.

The above and other safety features and procedures are employed. The one greatest hazard however is the possible lack of attention and alertness on the part of operators through repeated start-ups and shut-downs. To counteract the possibility of this apathy a constant program of safety consciousness is followed. Operators are constantly alerted by the head of the pilot plant, and he in turn is alerted by his superior. Many men can check the design and safety features of a plant, but only one man and one nervous system controls the hand that operates. This operator then becomes the last link separating damage from safety. He is worth attention at least equal to that given the solvent-detection system.

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Report of the Technical Safety Committee, 1956-1957

THANKS to the Local Committee, which supplied a stenographer for the purpose, the minutes of the Technical Safety Committee meeting in Houston on April 4, 1956, were recorded, and copies were supplied to those in attendance and to those who subsequently were appointed to the Technical Safety Committee of the American Oil Chemists' Society. Likewise minutes of the Technical Safety Committee meeting in Chicago on September 25, 1956, were recorded in detail, thanks to the Local Committee which provided the services of a stenographer, and copies were supplied to the members of the Technical Safety Committee. Also, the minutes of both of these meetings were published in appropriate issues of the Journal so they will be a matter of record in the years to come.

At Chicago the committee conducted a safety symposium during which six articles on various phases of technical safety were presented and subsequently published in the March 1957 issue of the Journal. Authors were H. D. Fincher, N. H. Witte, and P. Kane; A. E. MacGee; W. F. Bollens; R. P. Hutchins; and O. J. Jones, H. R. Belew, and O. L. Williams.

In Chicago it was decided that the Technical Safety Committee would be composed of three subcommittees, Solvent Extraction, Laboratory, and General Plant. Paul R. Sheffer was named vice chairman of the Solvent Extraction subcommittee and Harold H. Schultz vice chairman of the Laboratory subcommittee.

The Solvent Extraction subcommittee established three task groups: Safe Operations and Rules under N. H. Witte; Accidents Review under H. D. Fincher; and Corrosions and Erosions under F. P. Parkin. And sufficient work has been done on these subjects for a progress report at the New Orleans meeting.

Likewise the Laboratory subcommittee has been exploring the possibility of developing more satisfactory methods of determining residual solvent in both extracted oils and meals. A preliminary report is expected at the present New Orleans meeting. The Technical Safety Committee is composed of 33 members, of whom 18 are assigned to the Solvent Extraction subcommittee, nine to the Laboratory subcommittee, and five to the General Plant subcommittee.

As a result of the cooperation of various members of the committee during past months there have been prepared some eight articles for presentation at another Technical Safety symposium at the New Orleans meeting, and it is of course planned that these articles subsequently will be published in the Journal.

During the past months there were obtained and sent to members of the committee and officials of the Society the following publications as a matter of general information and help along safety lines: a) Office Safety, b) Safe Practices and Information for Employees of E. I. DuPont de Nemours and Company, c) Safety—Task Assignment, d) Safety Information and Instructions for Contractors, e) Lightning, Its Behavior and What to Do About It, f) Occupancy

Fire Record of Oil-Extraction Plants, and g) How to Get the Facts on Accidents. The foregoing were sent to "All Members of the A.O.C.S. Technical Safety Committee" in letters from the chairman

dated May 16, September 11, and October 19, 1956, and January 2, January 3, March 8, and April 5, 1957.

A. Ernest MacGee, chairman

Cyclization During Heat Bodying of Safflower Oil at 300°C.

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IT WAS CONCLUDED in previous work (1) on heat-bodying of linseed oil that cyclization of linolenic acid took place to an extent of 17% during the initial stages of polymerization because the isolated cyclic product was found to contain an average of two double bonds. Wells and Common (2) also affirmed that the cyclic products obtained from the polymerization of linseed oil were derived from linolenic acid. Therefore it is presumed that oils containing only linoleic acid (but not linolenic) may not result in cyclic products during the polymerization.

However it was pointed out by Paschke and Wheeler (3) that methyl linoleate also cyclized during heat-bodying. They observed that after prolonged polymerization 60% of the monomer was monoethenoid and did not hydrogenate to methyl stearate. The product was thought to be the cyclic monoethenoid isomer of methyl linoleate although it was found to contain slightly higher unsaturation than corresponded to one double bond per mole of esters.

Indian safflower oil was chosen to study the cyclization of linoleic acid during heat-bodying at 300°C. as the oil contains mainly the di-ethenoid acid with very little or no linolenic acid. The methyl esters of the bodied oil samples were distilled, and the distillates were fractionated with urea to separate the cyclic products present, if any. The esters of the acetone-soluble oil were also subjected to distillation, followed by urea fractionation to find out the extent of intradimerization and the cyclization in the monomeric fraction of the oils.

Experimental

I. Safflower oil (I.V. 144.1) was heat-bodied at 300°C. under an atmosphere of CO₂ for 3 hrs. Samples were taken at intervals of 1 hr. after the oil had attained temperature. These were analyzed for viscosity, refractive index, iodine value, and molecular weights, and the data are given in Table I.

Urea Fractionation. Samples were hydrolyzed and then esterified according to the method recommended by Bradley *et al.* (4). The esters were distilled from a liquid metal bath at 250°C. with 10-mm. of mercury

pressure to estimate the amount of monomer in each sample. The total mixed esters as well as the distilled esters were fractionated with urea, using 1:4:4 proportion of esters:urea:methanol. The data of both the experiments are given in Tables II and III, respectively.

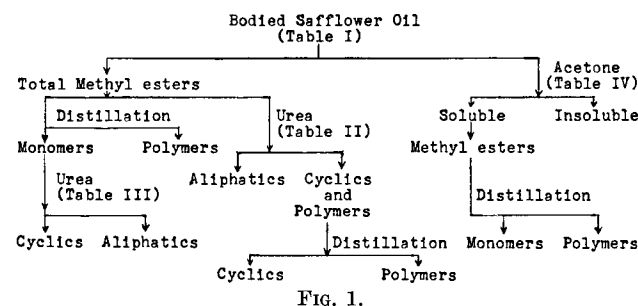


FIG. 1.

Acetone Fractionation. The samples were extracted with 5 vol. of cold acetone, and soluble and insoluble portions were separated. The acetone-soluble portions from samples of bodied oil were converted to methyl esters and subsequently subjected to distillation.

II. Safflower oil was heat-bodied under the atmosphere of CO₂ at 300°C. for 6 hrs. Methyl esters of the bodied oil were prepared as above, and the cyclic esters were concentrated by fractionation with urea.

The total mixed methyl esters were first treated with urea in this case to remove all the straight chain monomeric esters as urea adducts. From the non-adduct esters the cyclic material was concentrated by distillation at 10 mm. of mercury pressure to separate it from polymers, followed by urea fractionation of the distillate (1:4:4) to ensure complete removal of non-cyclic material. The monomeric urea non-adduct concentrate was analyzed for iodine value (Wijs and Benham and Klee methods), saponification value, and refractive index. The results are given in the discussion section.

The experimental procedure has been shown in Figure 1.

TABLE I
Safflower Oil Heat-Bodied Under the Atmosphere of CO₂ for 3 hrs. and 6 hrs. at 300°C.

Sample No.	1	2	3	4	5
Heating time in hours after attaining temperature.....	0	1	2	3	6
Viscosity in poises at 29°C.....	0.59	1.15	2.74	5.7	80.0
Refractive index at 20°C.....	1.4802	1.4828	1.4848	1.4868	1.4906
Iodine value (Wijs) 1 hr.	136.1	126.7	116.7	110.2	85.2
Iodine value drop					
Theoretical ^a	1.8	3.1	4.75	6.9
Observed.....	8.1	17.4	27.4	33.9
Molecular weight (uncorrected)M _n	908.1	928.0	959.0	1000.0

^a I.V. drop calculated from the equation: $Y = 57.8 \times 875(1/880 - 1/M_n)$.
M_n: Molecular weight of the bodied glyceride.