

TABLE I  
Conversion of Commercial Stearic Acid to the Acid Chloride

Chlorinating agent	Solvent	% Free acid in product
PCl <sub>5</sub> .....	Skellysolve F	1.7
PCl <sub>5</sub> .....	Benzene	1.4
PCl <sub>5</sub> .....	Carbon tetrachloride	0.7
PCl <sub>5</sub> .....	Skellysolve F	4.8
PCl <sub>5</sub> .....	Benzene	0.8
PCl <sub>5</sub> .....	Carbon tetrachloride	1.1

the product containing less than 1.5% free acid based on infrared analysis. In the case of tetrabromostearic acid 4.0 g. of phosphorus pentachloride was used with 10 g. of acid, and the solution was washed with water at room temperature to avoid crystallization of the tetrabromo-stearoyl chloride.

Since the method could be readily adapted to the commercial preparation of the acid chlorides, a number of runs was also made, using a sample of commercial stearic acid<sup>3</sup> with both phosphorus pentachloride and trichloride in three different solvents. Ten g. of acid were placed in 100 ml. of solvent, and 7.5 g. of the pentachloride or 5.0 g. of the trichloride were added, and the mixture was refluxed for 1 hr. The results of these runs are given in Table I. A layer of phosphoric acid formed on the sides of the reaction

<sup>3</sup> Obtained from W. C. Hardesty Company of Canada Limited.

flask with the trichloride, and there was a slight absorption at 1,740 cm<sup>-1</sup> in the infrared spectra, which was believed to be caused by a small amount of anhydride formation.

### Summary

A rapid method has been found for preparing the long-chain fatty acid chlorides, which eliminates purification by distillation. It gave a quantitative yield of product containing less than 1.5% free acid. The method involves treating the free acid with phosphorus pentachloride or trichloride in an inert organic solvent and removing the excess chlorinating agent by washing the solvent phase with water. Phosphorus pentachloride and Skellysolve "F" were preferred for laboratory preparations. For commercial purposes however either chlorinating agent could be used in a variety of inert organic solvents.

Infrared analysis was found to give a rapid measure of the free acid content of the prepared acid chlorides.

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## The Work of the Technical Safety Committee

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### Minutes of the Technical Safety Committee Meeting, September 25, 1956

A. Ernest MacGee, chairman, welcomed to the meeting T. H. Hopper, president of the Society, and Howard Black, vice president. Both expressed pleasure at seeing the safety work successfully getting under way and commended those present, particularly the members of the Technical Safety Committee, for their participation for the general good of the cause. MacGee then pointed out that this meeting would serve the two-fold purpose of conducting a safety symposium of formal papers and effecting a meeting for the purpose of conducting current business of the committee. The group attending the session was composed of 15 members of the committee and about 70 other members of the Society interested in the session's activities. Committee members were:

- Rex Wingard, Blaw-Knox Company, Chicago, Ill.
- Fred K. Bieri for R. W. Cornell, Pittsburgh Plate Glass, Red Wing, Minn.
- Walter F. Bollens, Swift and Company, Chicago, Ill.
- Wm. T. Coleman, Western Cottonoil Company, Abilene, Tex.
- H. D. Fincher, Anderson, Clayton and Company, Houston, Tex.
- Ralph P. Hutchins, French Oil Mill Machinery Company, Piqua, O.
- George J. Hutzler, Spencer Kellogg and Sons, Buffalo, N. Y.
- Henry James, Ralston-Purina Company, St. Louis, Mo.
- A. Ernest MacGee, Skelly Oil Company, Kansas City, Mo.
- Paul R. Sheffer, Corn Products Refining Company, Argo, Ill.
- Louis M. Smith, A. E. Staley Mfg. Company, Decatur, Ill.
- Norman H. Witte, Central Soya Company, Inc., Decatur, Ind.
- Harvey E. Marxhausen, Cargill Inc., Minneapolis, Minn.
- W. J. Miller for Robert Stokes, Buckeye Cellulose, Cincinnati, O.

Don F. Starr for J. W. Dunning, V. D. Anderson Company, Cleveland, O.

Those indicated below, although unable to attend, took the time to write the chairman, explaining why and offering a number of suggestions in connection with the committee's work. Portions of these letters were read to the group by MacGee.

- Robert Stokes, Buckeye Cellulose, Cincinnati, O.
- John W. Dunning, The V. D. Anderson Company, Cleveland, O.
- James H. Brawner, Southern Cotton Oil Company, New Orleans, La.
- Reider Arneson, Archer-Daniels-Midland Company, Minneapolis, Minn.
- Odell J. Jones, Western Cottonoil Company, Abilene, Tex.
- F. P. Parkin, Borden's Soy Processing, Waterloo, Ia.
- Reuben W. Cornell, Pittsburgh Plate Glass, Red Wing, Minn.
- James K. Sikes, Plains Cooperative Oil Mill, Lubbock, Tex.

MacGee discussed the fact that safety permeated not only solvent-extraction plant activities but also extended throughout all plant manufacturing operations. In many of these safety matters there are things which are dependent upon scientific or technical features of both a fire hazard and health hazard nature, namely, things which properly, and in many cases with advantage, could be included in any broad activity of a technical safety committee. Although a number of things in this connection are generally accepted by all, many safety measures and practices are somewhat controversial and obviously can best be resolved by turning upon them the spotlight of knowledge of those chemists and engineers in the oil and fat industry whose daily work brings them into

more or less direct contact with the problems. Therefore it behooves the Technical Safety Committee, which now is composed, with two exceptions, of members whose interest centers around vegetable and animal oil solvent-extraction plant operations, to broaden its membership so as to include members whose interests center around laboratory activities within the industry and those whose interests center around other general manufacturing activities of the industry, such as hydrogenation, soap manufacturing, and refrigeration.

In this connection E. A. Gastrock of the Southern Regional Research Laboratories in New Orleans, said that he considered this to be a fine step, and he commented further that the safety work being undertaken by the Society was a big job and one that, with advantage, probably could be coordinated with a number of other organizations, such as the National Safety Council and Underwriters' Laboratories. It was his thought that those interested in safety matters would be missing a good chance to expedite the forward movement of the program if we did not take advantage of the opportunity to collaborate with other organizations with generally similar objectives.

Harold H. Schultz of S. C. Johnson and Son, Racine, Wis., pointed out that the National Safety Council planned to devote a day to the topic of safety in the laboratory at their forthcoming convention in Chicago, and it was his thought that a number of the companies in the oil and fat industry should find it worthwhile to send representatives to attend this session.

After further discussion it was moved by Paul R. Sheffer and seconded by H. D. Fincher that the Technical Safety Committee be composed of three subsections, namely a Solvent Extraction Subcommittee, a Laboratory Subcommittee, and a General Subcommittee. The motion was carried unanimously, and MacGee appointed Sheffer as chairman of the Solvent Extraction Subcommittee, subject of course to formal approval by President Hopper.

In accepting, Sheffer said it was his understanding that the safety committee was to be composed primarily of those who would be willing to expend some work for the good of the cause and that he was glad to do his part in behalf of his company, the industry, and the Society as a whole. And he went on to say that the membership of the committee represented several hundred years of extraction-plant operations and that, for this reason as well as the fact that all were of a general chemical or engineering background, he could not think of a better source of recommendations on safety matters than was represented by this membership. He said that he would appreciate receiving notes from anyone who desired to volunteer for various phases of the work since each of the members, as well as other members who will be appointed later, has had experience which might be unique in some particular operation. It would be better for them to volunteer for some particular phase of the committee's work rather than wait to be assigned a task by him. By volunteering, each could pick a gem out of his experience which could be incorporated in final recommendations to be passed around to other members of the group.

MacGee urged various members to volunteer for some particular phase which they thought they might contribute to the best advantage, and he went on to urge others who were interested in safety matters generally and who were not now members of the Technical Safety Committee to volunteer as the field was plenty broad enough to take care of all. And he went on to say that the tendency in nearly all societies was for a few of the old-timers to appear to monopolize the various committees and other assignments, but he pointed out that this apparent monopolizing was not primarily a matter of choice but hinged also on the fact that those responsible for heading up the activities and getting out the work of the organizations simply did not know a lot of the younger members and, more importantly, did not know what activity would have the most appeal for them. Therefore, it would certainly help the chairmen of these safety groups, as well no doubt as other officials and chairmen in the Society, if the younger members would come forward and let them know the things in which they were interested and the things about which they thought they could be of help to the Society. In the case of

the Technical Safety Committee, workers were needed and the more, the better as far as the Society is concerned.

By way of an introduction, MacGee pointed out that the first formal paper of the meeting had to do with the personal element as it affected safety in solvent extraction plants, and he went on to say that "accidents don't just happen; they usually are caused by someone's ignorance or carelessness." At this time H. D. Fincher presented his paper entitled "The Role of Personnel in Safety of Solvent-Extraction Operations."

Next MacGee pointed out that the Central Soya Company had, over the years, exhibited a very good safety record in their solvent-extraction plant operations and that their representative no doubt would have some worthwhile remarks for the group. At this time N. H. Witte presented his paper entitled "Special Equipment and Operating Features which Contribute to Safety in Extraction-Plant Operations," supplemented by slides showing various things of interest about a solvent-extraction plant, among which might be mentioned a relatively simple and inexpensive inert gas producer.

Robert M. Starr, Honeymead Products, Mankato, Minn., asked if one of the slides pictured a Davenport cooler, and Witte replied that it was a Linfield Steam Tube Drier. Then Fincher asked if two doors are used to form an air lock in the case of a pressurized room. Witte replied that only one door was used. Keator McCubbin, Blaw-Knox, Chicago, Ill., commented that there really was no need for two doors since, in the case of a pressurized room, a person could be inside the room and the door closed before the pressure was reduced appreciably.

In introducing the next formal paper MacGee pointed out that packing houses had the general reputation of close margin operations, extending to a point of utilizing everything but the squeal in an animal, but that, from his own observation, he felt sure that Swift and Company had not spared any reasonable expenditure in the construction and operation of their solvent-extraction plants in order to build safety into them and their operation. This seems to have been money and effort well spent since they, too, had shown a very good safety record over the years of operating a number of plants. Then Walter F. Bollens presented his article entitled "Safety in Solvent-Extraction Plant Operation." MacGee asked three questions:

1. "In your plants how do you go about bonding the equipment to the ground in order to obtain an effective 'ground'?"
2. "Has your experience been that there is considerable difficulty in attaining the 0.1-ohm resistance mentioned in your paper?"
3. "How often is the grounding checked in the various plants?"

Bollens answered that bonding by electric cable should not be necessary except where there is no other way to effect a ground. He indicated that every bond was checked after the plant is completed and, if any big resistance is found anywhere, the workmen go over the installation again. He explained that the equipment is all set in the steel frame of the building and that a typical installation consists of a one-piece copper cable which runs continuously around the outside of the plant, going to each corner, at which a triangle bond is formed by driving three rods at each corner into the ground. He said the effectiveness of the ground is actually tested with electrical equipment and it is so constructed as to be well below the 0.1-ohm resistance and that a check of this grounding is made whenever the plant is shut down or at any time a major change is made.

MacGee asked if in effecting grounding in the plants attention is given to the moisture content of the soil. To this Bollens answered that the grounding rods ordinarily go down eight feet and that, with the moisture in the ground in the areas where their extraction plants are located, an adequate

ground is effected without the necessity of adding additional moisture to the soil.

McCubbin pointed out that the use of a continual cable separately grounded is not generally used in solvent-extraction plants. In cases where he participates in the design of solvent-extraction plants, he does not use a grounding cable unless requested to do so but instead prefers to depend upon individual grounding. His observation had been that people ordinarily do not take into account the ground moisture but that this is an important safety feature and should not be overlooked.

Ralph P. Hutchins said that he knew of cases where people had dumped salt into the soil in order to corrode the grounding rods. And he went on to say that in the case of plants in which he cooperated in designing about nine grounding rods in an extraction building should be used and that ordinarily he ground the extractor with a cable. He said that all major building columns should be grounded as well as all pieces of equipment and that, in plants which his firm constructed, the plan was to check with an ohmmeter in order to be sure that the "ground" resistance is substantially under 1.0 ohm.

Harvey E. Marxhausen stated that he assumed the precautions mentioned were checked by a special plant-safety man apart from the personnel ordinarily doing the bonding, and he asked if the safety man had authority to shut down a plant if he considered there was something wrong. Bollens answered that, in his firm, there was a special safety committee and that the members of it made a monthly check of each plant and followed a definite safety outline. With this outline the various items deemed important are checked to be certain that everything is functioning properly and within safety tolerances allowed, and then each man on the committee signs the report. Although the safety committee made recommendations, the only person who had the authority to shut down the plant was the superintendent. And he said that they had trained their operating personnel very thoroughly and that it was his observation that they did not take any chances and that they called upon members of the safety committee for advice whenever there was any doubt.

MacGee displayed four booklets by E. I. du Pont de Nemours and Company, pertaining to safe practices followed in their plants, which he said probably could be obtained by anyone writing on company letterhead to the du Pont Company. These booklets covered quite a wide area: "Safety Information and Instructions for Contractors;" "Safe Practices and Information for Employees;" "Safety Task Assignment as a Prime Safety Requirement for Foremen and Supervisors;" and "Office Safety." He said that he would attempt to get extra copies of these booklets for each member of the committee and considered that they were well worthwhile studying in detail because of the extra fine safety record that has been established by various plants of the du Pont Company, a record which, during 1955 and as reported by the National Safety Council and recorded in the September 10, 1956 issue of Chemical and Engineering News, showed that du Pont's synthetic fiber plant at Old Hickory, Tenn., paint and varnish plant at Parlin, N. J., industrial gases plant at Belle, W. Va., photographic film plant at Parlin, N. J., and chlorine-alkali plant at Niagara Falls, N. Y., respectively, were the top five plants in the chemical industry for the United States from an injury-free, man-hours standpoint. The Old Hickory plant ran up 28,700,000 man-hours without an accident. Reprints of the articles, "Extraction Solvents and Safety" and "Safety, the Lodestar to Industrial Happiness and Profits," were distributed to those present.

Following a recess, Mr. Hutchins presented his paper entitled "Safety and Design in Solvent-Extraction Plants." Afterward Louis M. Smith said that, in some cases which had come to his attention, the dump tanks for solvent had an automatic dump valve but did not have an automatic overflow valve

for use in cases of emergency shut-down and that it was possible for an operator pumping hexane or other solvent into the tank to keep the liquid running until it had overflowed into the water-stripper, from which it finally could enter the sewer. However, he went on to say that, in his opinion, a dump tank was a very important thing to have in a solvent extraction plant.

Mr. Gastrock pointed out that non-sparking tools, although an undoubtedly fine thing to use from a safety standpoint, were not so good as working tools because of inferior quality material, and he suggested that companies manufacturing safety tools could with advantage carry on research with a view toward bettering their tools and making them more trustworthy. He also suggested that those responsible for designing solvent-extraction plant equipment could design much of the auxiliary equipment with opening and closing locks so that use of wrenches would not be necessary. Bruce P. Neil of the Ralston-Purina Company pointed out that if one would limit himself to keeping impact tools spark-proof and then use Masonite or similar strips on the floor around the machines being worked upon, the men could then use the more sturdy non-sparking wrenches or other similar work tools without undue hazard since the Masonite or similar strips would not permit a spark to be caused in case the tools should be dropped.

In introducing the paper by Odell J. Jones, MacGee said that the paper was quite timely inasmuch as it was discussing insurance rates and safety code requirements for solvent-extraction plant construction and operation from the viewpoint of one with experience in plant-insurance work as well as practical solvent-plant operations. As Jones was unable to be present, his paper was read by W. T. Coleman, "Safety in Solvent Extraction from the Viewpoint of Insurance and Practical Operation."

MacGee reported to the committee about the working session that was held September 18 and 19 in Chicago by the Sectional Committee on Solvent Extraction of the National Fire Protection Association about developing standards for solvent extraction plants. The N.F.P.A. undertook the development of standards for solvent-extraction plants at the request of the Soybean Processors' Association and, as was mentioned on the occasion of the Technical Safety Committee's meeting in Houston last spring, this Sectional Committee is composed of the following members:

Paul C. Lamb, chairman, Lever Brothers  
 S. L. ("Steve") Halak, The Glidden Company  
 Louis F. Langhurst, extraction plant consultant  
 George H. Steele, Ralston-Purina Company  
 G. G. Fleming, Celanese Corporation of America  
 Hugh V. Keepers, Fire Prevention and Engineering Bureau of Texas  
 E. J. Sestak, Factory Insurance Association  
 W. H. VanArnum, National Board of Fire Underwriters  
 Theron H. Wright, Ohio Inspection Bureau  
 George A. Quandee, Swift and Company

Also, of course, the committee has a worthwhile guide in the person of Miles E. Woodworth, the flammable liquids engineer of the N.F.P.A. Committee on Flammable Liquids. MacGee pointed out that Woodworth had indicated that the Sectional Committee considered it proper for him to be a member of the committee representing the Technical Safety Committee of the A.O.C.S., both being of the opinion that, since the two committees had many parallel objectives in mind, such an arrangement would facilitate liaison and exchange of ideas between the two groups for the general good of those concerned.

In reporting on the work of the above-mentioned Sectional Committee, MacGee said that it was his thought that this

N.F.P.A. group consisted of personnel well qualified from practical training and experience in fire protection and insurance matters. The only apparent deficiency was that its solvent-extraction plant personnel represented essentially the soybean industry and that it would also have been desirable to have had representatives more directly connected with other phases of the solvent-extraction industry, such as those pertaining to flax seed, corn germ, meat scraps, peanuts, and cotton seed. However this deficiency is now overcome to some extent by having as a member a representative of the Society's Technical Safety Committee, which covers the entire field of the oil and fat industry.

MacGee went on to say that his observation of the work of this committee was that the members were dedicated to the cause and were striving hard to produce a proposed standard that would best serve the interests of all directly concerned and also the general public. He said that rapid strides were being made in compiling the various items and arranging the wording to conform with customary standards and that the committee had another meeting planned for the latter part of November in St. Louis to complete their preliminary work at that time. After that the proposed standards would be printed in bulletin or booklet form for distribution to industry for comments; and then, in all probability, copies would be sent to each member of the Technical Safety Committee for study and comments for additions, deletions, or other changes.

MacGee said that since Bollens and Fincher had exhibited the booklets entitled "Safety for Solvent Plants" and "Rules for Solvent-Plant Safety," respectively, as followed within their company extraction plant operations, and since Sheffer at the Houston meeting of the committee had exhibited the safety schedule followed in extraction plants of his firm, it appeared that many other plants in the oil and fat industry would have similar bulletins or booklets pertaining to safety ideas or procedures which they followed. A working group of the extraction plant subcommittee could make a worthwhile contribution to general safety by obtaining as many of these safety bulletins or booklets as practical and, after a careful analysis and study of them, combine the best features of all into one bulletin, which could then be made available to the various plants of the industry for consideration by their safety and insurance people with a view toward utilizing any features of it for their particular plant which they might consider desirable and which they might not already have. Sheffer said that he would appoint a working group to go into this matter, and he emphasized the point that, in his opinion, all firms in the industry could cooperate in this work since there would not be any obligation on the part of any of them to use any of the recommendations that might arise from the study. In other words, the committee would simply be acting as advisors or consultants with the understanding that any technical advice developed in such a booklet would be useful in some cases and employed if the respective company desired so to use it.

Witte, in discussing the matter of fires or other accidents in solvent-extraction plants, pointed out that, although he had seen several reports on fires in extraction plants, these reports were treated as extremely confidential and he had the general feeling of an outsider looking in as far as major accidents were concerned. Past experience in major fires or explosions undoubtedly would be very valuable if a study of the cases could be made and the reasons behind the accidents found. Of course, such a study could not be made unless the details of the various accidents were made available to the committee, and he said that he considered that the past histories of

accidents would be a valuable guide for all in the industry as they would highlight some of the things to watch for and be on guard against.

Sheffer said that he would appoint a working subgroup to see what could be developed, and both he and MacGee said that they could see no reason why practically all of the companies which had had accidents should hesitate to make data available for the committee inasmuch as everything pertaining thereto, such as the company's name, dates, persons involved or injured, and dollars' damage would be treated as a matter of commercial confidence by the few of the committee who worked with and analyzed the data. Thus an analysis of these accidents might prevent some other company from experiencing a similar accident and, for that matter, might enable the same company to prevent further accidents because of the committee's ability to dig out somewhat obscure facts and analyze them in relation to other accidents and thereby shed light on them which otherwise would not exist.

MacGee read a letter from F. P. Parkin which, in part, suggested that some problems which the extraction plant subcommittee or other working groups could delve into during coming months were a) handling of solvents, b) condenser problems, c) vent problems, d) solvent loss problems, e) dust problems, f) mechanical equipment as related to designed capacity and operating capacity, g) failure of operating equipment, h) distillation problems including cleaners used, i) corrosion problems. Sheffer said that he would keep these in mind and, as time went on, probably would appoint a working group to look into some of these as well as perhaps other phases of extraction-plant operation.

As the Technical Safety Committee previously had voted to establish a laboratory subcommittee, Coleman expressed the view that such activity could be worthwhile help to the industry and the Society, but he emphasized that such a subcommittee should not do work that had already been done. He emphasized that a lot of information had been accumulated regarding laboratory safety but that, in many cases, it had not been stressed or had not been compiled in readily usable form with the result that it simply had been "lost in the literature." Therefore he suggested the need for a working subgroup to review literature pertaining to laboratory safety and suggested that it would be worthwhile to put in the Journal abstracts on safety instructions or articles. Coleman went on to say that he thought a review of the literature pertaining to laboratory safety should be summarized in the form of a paper which could be presented at some subsequent meeting of the Technical Safety Committee. It was his thought that one of the prime efforts of the committee would be to stimulate an interest in safety, especially since the tendency for everyday chemical workmen is to simply tolerate safety instructions and to look upon much of it as "propaganda." Thus there is a real field for contributions to safety and the good of the cause if more real interest in safety can be stimulated on the part of various laboratory personnel. MacGee said that these things would be borne in mind and probably activated as soon as a laboratory subcommittee could be established.

MacGee announced that there would be a similar meeting of the Technical Safety Committee at the spring convention of the Society in New Orleans and urged each member to make a special effort to attend.

A. ERNEST MACGEE, chairman.