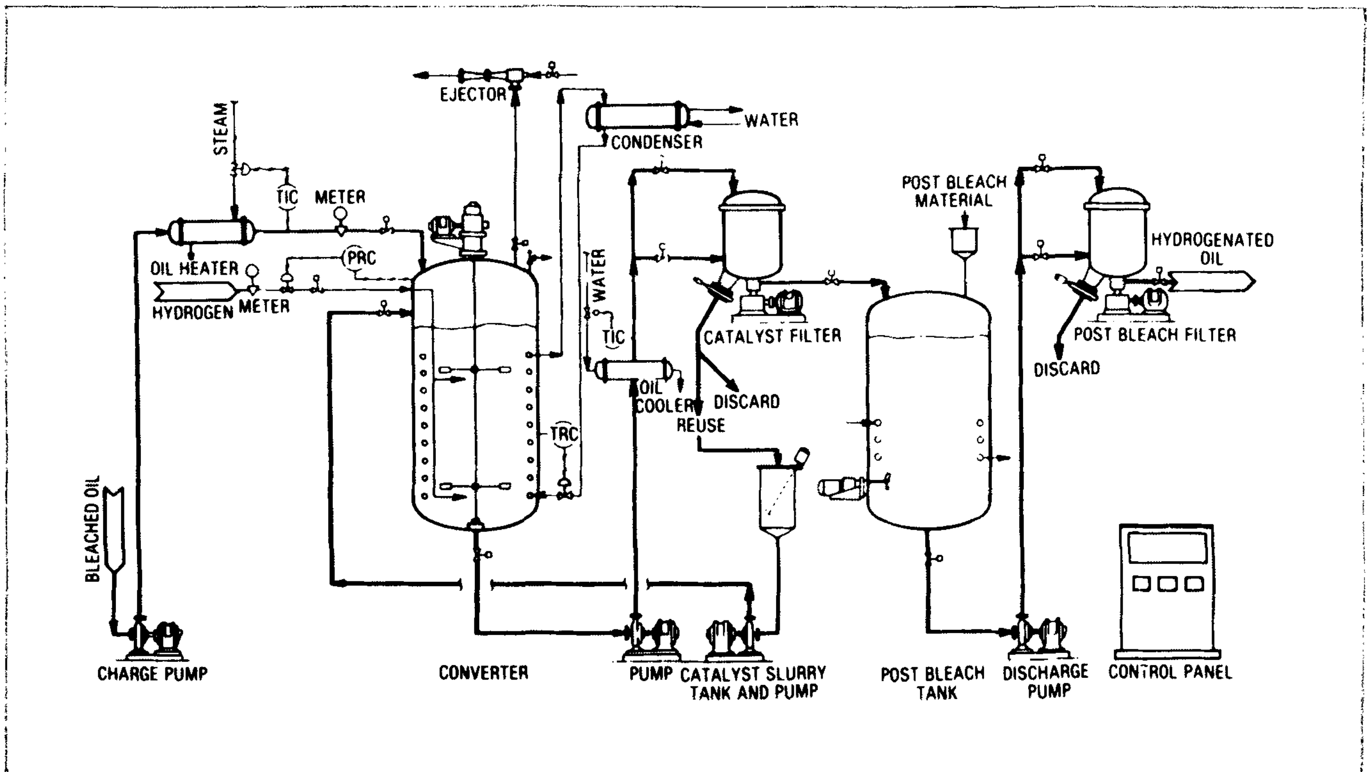
Industry News

The Hardening Plant



The following is an edited transcript of the question-and-answer session entitled "The Hardening Plant" held during the 1983 AOCS Annual Meeting in Toronto. Robert C. Hastert of Harshaw Chemicals served as organizer and mediator for the session, which attracted a crowd of several hundred persons on the afternoon it was presented. Panel members and their affiliations at the time of the session were: Hector Gil A. of Gamesa S.A., Cuidad Obregon, Mexico; E.G. Latondress, Davy McKee Corp., Chicago, Illinois; Jack Thompson, Durkee Foods, Louisville, Kentucky; James B. Yeates, Archer Daniels Midland, Lincoln, Nebraska; Giles S. Farmer, Anderson Clayton Foods, Dallas, Texas; J. Raymond Carroll, A.E. Staley, Decatur, Illinois; W.H. (Billie) Walker, Humko Products, Memphis, Tennessee; James O. Wheeler, Hunt-Wesson, Savannah, Georgia; and Jim H. Ritchie, Canada Packers Inc., Montreal, Canada.

Hastert: I wish to make a disclaimer before we start that I do not expect to agree with everything that is said this afternoon, and I am sure the panelists will not always agree with each other. What we are going to hear are the personal opinions, which the panelists are willing to share with us, of some very knowledgeable and experienced "old boys" concerning the operation of an edible oil hardening plant.

HYDROGEN

Jack Thompson: *How do you know when the catalysts in a steam-methane reforming plant should be replaced?*
Thompson: The first thing to do is take two gas samples out of the hydrogen producing system. The first should be taken after purification before the methanation step. It will indicate the performance of the reforming catalyst. Methane content should be less than 0.1% with new catalyst. Over a period of time, the percentage increases to a point where it becomes excessive – about 2.5%. The life of reforming catalyst can be prolonged by increasing the temperature a few degrees while steaming the catalyst. Steaming will remove carbon and/or sulfur deposits. If you are getting sulfur through the catalyst, it will not work properly. CO content of this first sample also indicates the performance of the gas purification system. As the shift catalyst loses activity, CO content will increase to where a change is required. We usually think of this as being 0.5%.

Life of shift catalyst also may be prolonged by increasing the temperature. This may be accomplished by decreasing the steam to gas ratio in the reforming tubes, thereby converting more CO to CO_2 in the reforming tubes and lessening the load on the shift converters. Another indicator is the temperature profile across the bed. Watch the progress of the hot spot. As it approaches the exit of the converter bed, a catalyst change is imminent. The second gas sample should be taken at the exit of the methanator. This will indicate the performance of the methanation catalyst. Here again, analyze the methane content. It should be roughly the sum of the methane and the CO of the previous sample. The CO_2 content and the CO content both should be nil.

In our plant, it has been approximately seven years between catalyst changes. The hydrogenation converter is in itself also a good tool for evaluating the performance of the catalysts in the H_2 gas plant. If you have hydrogenation problems, look at your gas streams.
Hastert: Hector Gil, what is your experience in Mexico?
Gil: We consider replacing catalyst when we can't get the throughput, or the hydrogen purity, or both. However, before proceeding with the replacement, we first check that the indicating and controlling instruments are working properly. We also check for any deviation from the operating limits such as pressure, flow, temperature, sulfur content in the feed, pressure and flow of the steam, reformer temperature and effluent temperature from the reformer tubes, pressure drop through the tube, regeneration cycle, etc. If there is no evident deviation and the output is below expectations, there is always the possibility of temporary poisoning of the reformer catalyst. The procedure then would be to reduce the feed, maintaining the flow of steam, and keeping the tubes at the specified temperature for six to eight hours. If the throughput and/or purity do not then bounce back, the catalyst should be replaced.

Hastert: Thank you, Hector. There is obviously a difference in outlook between companies and between locations. If you are in Louisville, Kentucky, you have a perspective quite different than if you are in Cuidad Obregon, Mexico, and you proceed accordingly. *Can catalyst changes be done in-house or do they require an outside specialist?*
Yeates: At ADM we use outside consultants for catalyst changeouts who have the knowledge and technology for handling catalyst. We feel it is not worth taking a chance of damaging it.
Hastert: Ed Latondress, do you have any comments?
Latondress: If you have had experience in changing catalysts, I think your plant personnel are capable of doing it. Catalyst manufacturers will help with instructions and procedures.
Hastert: We have asked Jim Wheeler to *describe briefly a catalyst turnaround.*
Wheeler: We like to shut down on a Friday night, open up the furnace doors on Sunday morning, then be ready on Monday morning. When talking of catalyst turnaround, I am principally thinking of the reformer catalyst. However, we do sometimes also change shift catalysts and methanator catalyst. The desulfurizing catalysts or activated carbon traps usually can be changed while the plant is on stream. The reformer catalyst is changed after cool down and nitrogen purge. We unbolt the flanged top and bottom and drop the catalyst into drums for rescreening. I like to inspect for carbonization. A white color is created by bad steam. Some of this can be prevented by burn off – holding the reformer furnace at 100-150 degrees above the 1800-1900 F operating temperature for 10-12 hours. To recharge, we use a drop-door cylinder. With the trap-door cylinder you can prevent dropping and breaking the catalyst which causes restrictive flow. For this whole operation, removal of the old catalyst and addition of the new, we figure 4-6 hours per tube. When we have to change high temperature shift and low temperature shift, we cool down with steam, purge and cool with nitrogen and then flood with water to keep from igniting. We then remove into barrels. The entire process takes about eight hours. Normally, it takes about 72 hours to desulfurize the high temperature shift. However, it can take longer, depending on the shape and size of the vessel. In a gas plant utilizing the MEA systems for CO_2 removal, there is normally a carbon filter which is used in a slipstream on a portion of the MEA being circulated for removal of glycine. This keeps the MEA in an unoxidized condition. This filter, which contains a granular carbon, can be changed without shutting the system down – three to four hours of work, normally.

In the newer gas plants which use the absorption columns instead of the MEA absorption and reactivation, we find a different problem. These columns generally have three different materials in them – activated alumina

Panel members (from left, first photo): Giles Farmer, Jack Thompson, Ed Latondress, Jim Ritchie and chairman Robert Hastert

Panel members (from left, second photo): James B. Yeates, Hector Gil A., Billie Walker, James Wheeler and J. Raymond Carroll

in the bottom, a main portion filled with activated carbon, then a sector in the top containing a molecular sieve.

I have no personal experience with this type of column, but have been informed that four men can change out a column in about 12 hours. However, due to the compacting required, I would suggest using a technical representative. We are fortunate to have in our industry very knowledgeable people in the field of hydrogen production and I have never been refused service by these individuals and companies whenever I have called for assistance.

Hastert: *What mechanical repairs should be considered during the shutdown for catalyst turnaround?*

Latondress: You can estimate quite well how long it is going to take to do the catalyst turnaround. It will take 12 hours to cool the plant down and even longer starting up, so there is a considerable period of time when there is no hydrogen in the plant and it is perfectly safe to weld. When planning a shutdown for a catalyst turnaround, your maintenance people should advise you what needs to be done and provide a schedule of how long it is going to take to accomplish what is needed. If you are really rushed for production, you may not finish everything you want, and you have to make the business decision of how many repairs must be completed before starting up.

Hastert: *What are the most common problems to be on the look-out for when a reforming plant is running?*

Walker: Aside from the problems inherent in any chemical process, such as leaking mechanical seals and instruments out of calibration, the main problem is the loss of catalyst activity. If the catalyst starts to go, you can compensate to some extent, but sooner or later you are going to have to get that catalyst out. When the CO conversion catalyst starts to go, the temperature on the methanator will begin to go up. Even when it is working perfectly, you still get some CO in the stream to the methanator, and it will show a temperature rise. When this is no longer observed, the methanator catalyst should be checked and replaced if necessary. MEA temperature should also be watched closely. If it gets too high, some MEA will decompose and form catalyst poisons and you will have lots of problems with hydrogenation.

Yeates: In my opinion, one of the biggest concerns in running a hydrogen plant is to take care of the reformer and reformer catalyst. If you let mercaptans slip past the initial purification, or if the steam to C ratio gets out of whack and you start carboning up the reformer catalyst, you are going to be in trouble in a big hurry. Steaming the catalyst sometimes helps, but it is very wise to closely monitor the reformer itself. If you lose the reformer, you lose the whole plant.

Hastert: Hector, because of your somewhat unique experience when your supply of propane was cut off in 1973, *Can gases other than methane, such as propane or butane, be reformed? If so, can they be used interchangeably with methane or each other, or are expensive modifications necessary?*

Gil: Yes, gases other than methane can be reformed. We are presently reforming 90% propane with the balance being ethane and traces of iso-butane and N-butane. But we could reform methane as well. We would need to substitute the propane vaporizer system with a natural gas compressor.

Latondress: When I was with Swift, we had a methane reforming plant and, with no problem at all, equipped it to reform propane. Really, there is no problem in reforming any kind of hydrocarbon, provided you purify it before running it across the catalyst.

Hastert: Giles Farmer, *How do you know how much nitrogen (or other inerts) are in hydrogen and at what level will they affect the activity and/or selectivity of the reaction?*

Farmer: Certainly you would use the purity meter which is part of the hydrogen plant system. Laboratory analysis will tell what impurities and the percent of each that are present. I assume by inerts you mean nitrogen and carbon dioxide. They don't affect the reaction per se. The problem is getting rid of them from the head space of the converter. Many times we blame slow reaction rates on poor catalyst activity when, in reality, it is because we are not purging the impurities to allow hydrogen to get into the converter.

Gil: I am not familiar with the analysis of gases. This doesn't mean we are not in a position to know the hydrogen purity produced in the reforming plant or the electrolytic plant. The first is furnished with a thermal conductivity type hydrogen analyzer which records continuously the hydrogen purity. The instrument doesn't tell us how much each of CO or CO_2 is present, but the sum of both gases. In the event there would be some nitrogen present, it would be totalled as CO and CO_2. This is because the thermal conductivity of three gases is very similar. We know that if the hydrogen is less than 99.4% purity the reaction will be retarded.

Hastert: Jack Thompson, *What catalyst poisons are found in hydrogen made from reformed methane?*

Thompson: The most common poison is carbon monoxide. It causes a very serious problem in hydrogenation. The only thing to do is remove it from the gas plant proper.

Hastert: Jim Yeates has offered to respond to *Which type hydrogen purification is the edible oil industry presently purchasing, amines absorption or pressure swing absorption? Which looks the better for the long-term future?*

Yeates: Presently, the PSA type plant appears to have the edge in the U.S. We have had one in Lincoln the past year and a half. We have the dubious honor of having 5-6% nitrogen in our methane. It is 1,000 BTU gas and the ultrahigh purity plant takes out all the nitrogen. This results in a much easier hydrogenation reaction. There are a lot more inherent problems with the old amines type hydrogen plant, such as amines vapor carryover. Even if you have to use HD-5 propane, you can hydrogenate the propylene unsaturates. You can run a little hydrogen in the front and hydrogenate your unsaturates. It's easier on the oil as far as stability – I don't think you abuse the oil as much. The PSA plant appears to be the plant – for the near future anyway.

Hastert: We've given Ray Carroll a nice, easy question to begin: *Can good quality hydrogen be made by electrolysis? What are the potential impurities in electrolytic hydrogen?*

Carroll: Yes, you can make high quality hydrogen by electrolysis. Typically the purity range is 99.8%. In most applications, electrolytic hydrogen has a cost penalty in comparison with natural gas reforming, but there are advantages as well. The process requires very little operator attention and maintenance costs are much lower. Possible impurities in hydrogen that has been electrically reduced would be trace amounts of oxygen and inert gases that have been dissolved in the electrolyte solution that you separate, and trace amounts of moisture carryover. In practice, very few problems exist with impurities and little postpurification is required.

Hastert: Jim Wheeler is going to tell us, *Is liquid hydrogen available and, if so, from where?*

Wheeler: Thanks to the space program we have two major suppliers of readily available liquid hydrogen. With the escalation of natural gas prices, I think many of us will closely watch the pricing of liquid hydrogen. Presently you can buy liquid hydrogen at $1.18-$1.30 per 100 cu ft, provided you have the tank. Truck and driver charges this past week (April 1982) at our plant without the tank were $200/truck and $395 for the truck operator. On a large contract basis, I think you'll find that they are quite competitive. I know of one plant with a long term contract that is paying 80¢ per 100 cubic feet.

Hastert: *Is there any special equipment necessary to use it*, Jim Ritchie?

Ritchie: Liquid H_2 is a noncorrosive substance, so special materials of construction are not required. However, because of its extremely cold temperature, equipment must be of materials suitable for cold temperature operation.

Liquid hydrogen is normally stored in horizontal cylindrical tanks of double wall construction, vacuum insulated and equipped with safety relief valves to protect tanks designed to ASME specifications for the pressures and temperatures involved.

Transfer of liquid H_2 requires the use of a closed system with proper reliefs, which can be purged to eliminate the possibility of creating a flammable atmosphere.

Purging must be done with helium, since liquid H_2 can solidify other gases and cause plugging and possible rupture of transfer lines or storage vessels.

Transfer lines must be vacuum-insulated to minimize product loss through vaporization or the formation of liquid air on the lines with the subsequent hazards of oxygen enrichment.

To convert liquid H_2 to the gaseous state, a steam vaporizer, designed to handle peak volumes and pressure, is required. The liquid is vaporized by flowing through a coil immersed in a water bath equipped with temperature-regulating controls. The total vaporizer system is usually designed to operate in automatic mode requiring little operator attention.

Hastert: *Is liquid hydrogen more or less dangerous to handle?*

Wheeler: Let me answer that by saying that in Claxton, Georgia, every year there's a rattlesnake roundup. On the final day in a big cotton warehouse, there will be hundreds of rattlesnakes in cages. Any hydrogen–liquid or gaseous– is "liken unto" those rattlesnakes. You can live with it as long as it's controlled or caged. I mean it's high purity and is normally stored in an outside facility. No problems from a leaking hydrogen compressor as when using a low pressure holder. Higher pressure also makes leaks easier to find and, in the event of a fire, you get a blue flame instead of an unpleasant explosion.

Overall, if we weigh liquid versus the old-style plants, I'd say yes, but comparing with the new high purity gas plant utilizing the pressure swing absorption systems with modern safety devices, about the same.

Hastert: Jim, do you have the same experience?

Ritchie: I feel more precautions are necessary in handling liquid H_2 because of the cryogenics.

Besides the normal hazards of H_2, we must also be concerned with cold burns and the possibility of liquid hydrogen contacting other flammable materials. Also, due to the economics of delivery and storage, much larger quantities are usually on site at any given time.

Hastert: But you don't have the rattlesnake problem in Quebec?

Ritchie: We have plenty of problems in Quebec, but that's not one of them.

Hastert: Giles, you're in a management function with Anderson-Clayton, *When you're going to need additional hydrogen, what are your considerations in making a decision about which way to go?*

Farmer: The decision is economic. Even with today's natural gas prices, methane reforming is still usually the most economical. However, in some locations where electricity is cheap, electrolytic hydrogen production is the way to go. The equipment to produce hydrogen by either method costs in excess of a million dollars, so you must consider the cost of the plant in the formula to determine the most economical source. If your usage is small you might consider liquid hydrogen, even though it costs several times the direct cost of hydrocarbon reformed hydrogen.

Moderator Robert Hastert

CATALYST

Hastert: Now that we have our hydrogen made, we will now move on to catalyst. Jim Ritchie, *When feedstock is dried in the converter, should catalyst be added before or after drying?*

Ritchie: Our procedure is to add catalyst after drying for the simple reason that we avoid the possibility of poisoning the catalyst with moisture.

Hastert: Giles Farmer, *What are the advantages (technical, operational and economic) of a single-use catalyst policy?*

Farmer: From a technical standpoint, if you use fresh catalyst, endpoint control and finished product control are much easier. From an operational standpoint, if you use it only once you don't have to have bins or tanks to store used catalyst. From an economic standpoint, the question gets a little tougher. It really boils down to how much nickel do you use for a pound of oil hydrogenated. If you start out with 0.1% nickel and use it ten times, you come up with a usage of .01%. If you start out with .01% and use it once, you come up with the same usage. You need to compare the total cost, handling as well as catalyst cost and the capital investment necessary for storing the catalyst to get the total cost per pound of oil hardened for the two methods. When catalyst was $4-$5 per pound of nickel, the single-use policy was attractive. Now that catalyst is $14 per pound of nickel, consideration should be given to reusing catalyst.

Hastert: What do you do in Lincoln, Jim?

Yeates: We have a single-use catalyst policy. Our opinion is that for our product mix that's the best way to go.

Hastert: And I know that Hunt-Wesson has a multiuse policy, Jim Wheeler. *What do you see as the advantages of a multiuse policy?*

Wheeler: I think we lean in this direction for the basic reason that in an all-around business like we have, you can utilize a reuse program. We normally look at five runs on 80-105 IV shortening and up to eight runs of soy winter stock or frying oils. We feel some products perform better on rerun catalyst.

Operationally, I feel that it's easier to break up or split one big slurry. We have four converters, so we make up five catalyst charges. It's easier to make them up once and use them continuously 12 or 13 times. We normally start off with about 30,000 lb oil, 25 lb nickel and 200 lb filter aid. The economics are simple here, using 20 lb of nickel at .06% for 12 times.

Hastert: So the long and the short of it is that there is no "correct" answer to this and many of these questions. Companies have different product mixes and are also comfortable with what they have always done. But, *Supposing you are going to a reuse program,* Ed Latondress, *how would you go about doing it?*

Latondress: The first thing is make up your mind that you're going to clean up the oil just beautifully all the time. If you don't, you are going to have tremendous differences in performance of catalyst from run to run. You should have filter presses which lend themselves to not reducing the percentage of nickel substantially from one run to another. For example, if you have presses in which you must precoat each time you filter a batch, you will put 15 lb of nickel into the first converter, plus 15 lb of precoat and 15 lb of body; then if you keep adding precoat and body, after 6 runs the used catalyst contains 480 lb of filter aid and 15 lb of nickel. It doesn't get to be very economical because you can't sell the spent catalyst. In addition, you have to do a good job of record-keeping. You have to set up standards for how many times you are going to reuse catalyst and in what concentration for the various oils you are hydrogenating and absolutely adhere to these standards.

Hastert: Ray Carroll, what's the Staley attitude on this? Do you have a reuse program?

Carroll: At Staley, we use the catalyst once on some products and reuse on others. However, some of the things that you have to key on with setting a catalyst reuse program are a strong consideration to the product mix that you're going to end up using. I certainly agree that it is very important in a reuse program to have clean feedstock to minimize some of the inconsistency that we see in hydrogenation when reusing catalyst. There are going to be some inconsistencies there anyway and you have to adjust your hydrogenation conditions and/or your blending conditions to compensate.

Hastert: Another aspect of catalyst is how you get it into the converter. Hector Gil, *Can converter vacuum be used to draw in catalyst?*

Gil: Yes, and that is the way we have always done it. When the converter is being loaded, some oil is directed to a suspension tank into which the weighed catalyst is added. All is kept at 100 C. When the oil in the converter has reached a temperature 10 C below hydrogenation temperature, the catalyst suspension is sucked into the converter.

Hastert: Jim, how do you do it in Montreal?

Ritchie: We draw our catalyst into the converter in a dry flaked form, using a flexible hose and vacuum.

The advantages of this procedure are: mix tanks, lines and pumps are not required; less space is required; lower capital cost; few operator steps and, it is easier to add catalyst after the reaction is started.

Hastert: *What is the best way to monitor catalyst usage both overall and on an individual converter basis,* Jack Thompson?

Thompson: Weigh each catalyst charge and keep very accurate records.

Hastert: How do you prevent operators from adding more than they should?

Thompson: We give the operator on each shift the catalyst charges required and if he needs additional, he has to get it from the supervisor.

Hastert: The next question is, *Does catalyst lose activity in storage?* I volunteered to answer this one myself. My opinion used to be that catalyst did not lose any activity in storage. I was certain of this, as I was a lot of things at a younger age, but more recently I have become somewhat dubious. We had an experience of a customer who found catalyst in their warehouse that had been misplaced several years before. We tested samples under laboratory conditions and found out that it had lost activity. In the face of that, I have had to alter my opinion, and now believe that air oxidation probably does penetrate the protecting oil over a long period of time and catalyst will very slowly lose activity. It is certainly good practice to always use the oldest first.

Hastert: Giles, I know you're on a committee of the Shortening Institute dealing with this next matter, *What are the present federal regulations and guidelines for disposal of spent nickel catalyst?*

Farmer: Bob, I think there has been some misunderstanding about what those regulations are. About two years ago, EPA published regulations for hazardous substances in the Federal Register. It gave a list of materials which were considered to be hazardous. It also gave a list of materials in Table VIII which might at some time be classified as hazardous. Nickel was in that second list. It is my understanding that EPA will give us time to comment if they ever propose including nickel as hazardous. At the present time, there is no prohibition of disposing of used nickel in a landfill.

Walker: I might mention that waterborne catalyst waste which contains nickel is considered hazardous. We go to a public water treatment works and they consider it to be a toxic heavy metal and have imposed a limit of 5 ppm.

Hastert: Jim, *Is nickel recovery a viable economic alternative to landfill dumping?*
Yeates: It depends on the nickel content of the spent and your plant's distance from a reclaimer. Transportation costs are the key.

FEEDSTOCK PREPARATION

Hastert: *Since soybean oil heat bleaches during hydrogenation, why is it desirable to bleach it before hardening?*
Latondress: Oil is bleached before hydrogenation, but not to reduce the color. It is to remove oxidation products and soaps. If you do not properly prepare oil before hydrogenation, the most immediate result will be inconsistent SFI curves out of the converter. The second result, which takes longer to discern, is that the oil will not have as good keeping quality.

Hastert: *How important is it to have the oil dry before beginning hydrogenation?*
Farmer: It's very important for two reasons: one, moisture promotes hydrolysis and increases free fatty acid. Also, if oil isn't dry you form aldehydes and ketones, the precursors of color reversion and peroxide development.

Hastert: *Is drying under vacuum the best? What equipment is necessary to do it?*
Gil: Drying under vacuum is the best, and that is the way we do it. We want to have feedstock as completely deaerated, dried and free of volatile impurities as possible in order to have a reproducible hydrogenation reaction. In any case, a vacuum of about 28" is necessary before admitting hydrogen to the converter to prevent an explosive mixture. The vacuum equipment used may be a compressor, a vacuum pump or steam ejectors.
Hastert: Ray, how are you doing this?
Carroll: The only thing I would add is that in my opinion, drying under vacuum is not only the best way to do it, it is probably the only way to do it. In designing the vacuum vessel and the vacuum-producing equipment, a spray-type arrangement helps increase the contact area, flashing off the moisture. We use a combination system of ejectors and pumps to produce the vacuum.

Hastert: *What are the most common impurities in feedstocks and at what levels are they usually considered acceptable?*
Yeates: As far as soybean oil is concerned, phosphatides and soap are the common impurities. Anything above zero soap will cause problems, i.e., hydrogenation, filtration, and oil stability are all affected by the impurities in the oil.
Ritchie: In meat fats, proteinaceous material is a catalyst poison so this must be at a zero level. We also try to avoid sulfur compounds, which frequently are high in canola oil. Levels above 3 ppm affect the catalyst substantially.

HYDROGENATION PRACTICES

Hastert: *What is the best way to check for external leaks in a converter?*
Ritchie: We use a portable battery-powered H_2 detector. It is an explosimeter combustible gas indicator model 2A. It is FM approved for use in Class I, Division II Groups A, B, C, and D. Our day shift operator checks the agitator packing gland daily as this is the most frequent problem area. Others have used sound leak detectors with some success.

If all else fails or no instruments are available, an old method is to pressurize the vessel and lines with a 10% mixture of ammonia and air and test by passing a cotton swab saturated with acetic acid near all connection. A leak will cause a puff of white smoke.

Hastert: *What is the best way to check for leaks in the heating/cooling coils of a converter, Hector?*
Gil: One way is to check for moisture in the batch being unloaded from the converter. If there is any moisture present, some problems will occur in respect of the filtration rate through the catalyst press. If the leak is very serious, the hydrogenation reaction will be retarded substantially.

Hastert: *How often do the heating/cooling coils usually need to be replaced in your experience*, Jim Yeates?
Yeates: Our cooling coils in Lincoln have been in use for 12 years and we haven't changed them yet. I'm not saying they haven't had a few leaks from time to time, but I think the key here is having good cooling water treatment program. Some refiners use tempered water for cooling and there are different ways to take stress off the cooling coil life. I think two keys are water hammer and cooling treatment. As far as the steam coils go, the same thing is true.

Hastert: Giles Farmer, *Would it be a good idea to increase the RPM of the agitator in the converter?*
Farmer: I think first you would have to know what the RPM of the agitator was – more correctly, what the mass transfer characteristics are. If you increase the RPM, you will get poorer selectivity, but you will decrease the *trans*-isomer formation.

Hastert: *Does it make any difference how much head space there is in a converter,* Jack Thompson?
Thompson: Yes, it does. The converter is designed for the agitation, head space, etc., and if you start messing around with head space, you're going to change the conditions.

Hastert: *Is fresh or used nickel catalyst the best for promoting* trans *isomers? What hydrogenation conditions promote high* trans?
Wheeler: Used catalyst is best and conditions are high temperature and low pressure.

Hastert: *What is necessary to produce 100% soybean margarine oils so that they will not develop beta crystals in packaged product on subsequent temperature cycling?*
Latondress: Twenty-five years ago, it was common to have 10% hydrogenated cottonseed oil in soybean oil margarines. Cottonseed oil is a beta-prime former. We don't want to get too technical, but the point is, there were enough beta-prime forming crystals in that mixture to keep the margarine in the beta-prime form. But if you're going to make a 100% soybean oil margarine and want to store it and keep it from becoming sandy (getting the beta crystals), then you have to keep it cold.
Hastert: Giles, what is Anderson-Clayton's feeling on this?

Farmer: Most margarine oil formulations today are blends of two base oils. The blended oils are more resistant to beta-crystal formation and that is why we don't have the problems we used to. If you abuse soybean oil margarine (heat/cold/heat/cold) you can get sandy margarine, but I haven't seen any for a long time.

Hastert: *Is the winterization of a partially hydrogenated soybean oil a process to improve the functional quality of the oil, or is it a process just to keep the oil nice-looking when stored in the refrigerator?*

Walker: You can answer that "yes." The housewife often puts salad oil in the refrigerator. If you're the supplier and it crystallizes, you'll hear about it. In making mayonnaise, if you put it in the refrigerator, the cold may break the emulsion. The proper answer also depends on end use. Certainly winterization is functional when the oil is used in frying.

Hastert: *How does brush hydrogenation affect the cloud point of soybean oil?*

Ritchie: Brush hardening is performed to reduce the linolenic in soy and thereby improve the stability. Unfortunately, even though selective conditions are used, some linoleic is also hydrogenated, forming higher melting components. Some *trans* isomers also are formed. These cause oil to cloud at a relatively high temperature and make it necessary to winterize "brush-hardened" soy.

Hastert: *What tests can be run by the hardening plant operator and what tests should only be performed in the control laboratory?*

Gil: From our point of view, the plant operator should run only refractive index. All other tests – IV, congeal, melting point, SFI – are performed in the control laboratory. This is because we think the operator should be concerned with the operation of the hardening plant rather than with analysis.

Hastert: *In brush-hydrogenating soybean oil to make salad oil, should the most preferentially selective conditions be used so as to obtain the most stable end-product?*

Farmer: As Jim Ritchie said, the idea behind hydrogenating soybean oil to make winter oil is to lower the linolenic content, so in theory you should use the most selective conditions, but those selective conditions promote *trans* isomer formation and cause problems in winterization yield. Since you don't make many saturates at 100 IV, you should use conditions which keep the *trans* isomer content low and not worry about selectivity. The catalyst, of course, is one variable that can affect both selectivity and *trans* so it should be carefully selected.

Hastert: *Does AOM stability of soybean oil increase in direct proportion to the decrease in iodine value?*

Carroll: The purists among us will say that the resistance to oxidation is actually determined by the concentration of active methylene groups. However, the practical ones will say, "yes, the AOM stability does increase almost directly in proportion to decrease in iodine value."

Hastert: *What oils should be hydrogenated for use as hardstocks for liquid shortenings? Should these also be used for solid shortenings?*

Latondress: For liquid shortenings, hard stock should be made from an oil which will convert quickly to the beta crystal, and soybean oil is the most readily available. For plastic shortenings, where you are interested in the beta-prime phase, use something which promotes that phase, like palm oil or cottonseed.

Hastert: *What are some of the hydrogen safety precautions that should be taken in a hardening plant?*

Walker: Avoid pockets of hydrogen – the improper mixing of oxygen with hydrogen. Converters should be under positive pressure. We use centrifugal pumps – drafts are needed to sweep the hydrogen to the atmosphere. All electrical equipment must be explosion-proof. Open flames and smoking must be prohibited. There should be a barometric seal on the hydrogen purge line.

Hastert: Hector, any additional comments?

Gil: The converter should be under a vacuum of no less than 27" Hg before admitting hydrogen.

If repairs have to be done in the area, close the valves at the high pressure tank, evacuate the hydrogen until a vacuum of 28" is reached and then disconnect the hydrogen line at the supply tank.

No man should go into a converter until it has been tested safe with a reliable exposimeter.

Hastert: There seems to be a difference of opinion among insurance companies concerning electrical motors and wiring in a hydrogenation plant from the standpoint of compliance with Class I Division 2 National Electrical Code and other regulatory agencies. *What type of motors, controls and wiring should be used?*

Thompson: The whole thing should be explosion-proof. All drawings and specifications should be cleared with your insurance company. If you were not able to be insured, it would be a sad situation. Whatever the local codes are, and whatever the insurance company tells you to do, that's the way you have to do it.

Hastert: Jim Ritchie, you recently had an expansion and I am sure you were thinking about these things.

Ritchie: We attempted to acquire explosion-proof motors that would be acceptable for Group B, but we found they were extremely difficult, if not impossible, to obtain. We presently use Class I, Group D explosion-proof motors.

I understand that open drip-proof induction type motors have also been accepted in some installations.

In the case of controls, switches, etc., we use only those approved for Class I, Group B. The wiring method in our plant is threaded metal conduit with termination fittings fabricated for Class I locations.

Hastert: *What factors in the hydrogenation process can contribute to oil degradation and to what extent?*

Farmer: Anything which prolongs the reaction is detrimental to the quality of the oil. One thing that is sometimes overlooked is to have enough hydrogen available at the time you start so as to sustain the reaction at the desired rate.

Hastert: Jim Yeates had a session on computerization so he can surely answer a little brainstorming question here of, *Just how computerized can you envision hydrogenation becoming in the next 10-20 years?*

Yeates: I have been in the business for 20 years and we've come a long way. 20 years ago we had very simple single-loop pneumatic controllers and a large number of direct sensing pressure and temperature gauges. Through the 60s and early 70s, we got into the more total analog control pneumatics. In the late 70s, we started getting into electronic analog controllers. And now we're starting to get into microprocessors, digital control, data highway systems, total computer operated refinery processes. So it's not going to take another 10-20 years to see a complete revolution. It's already begun.

Hastert: *Is it true that heat exchange systems built into converters designed today are so energy efficient that they require no preheating of the oil? If so, is there a throughput penalty for this energy efficiency?*

Carroll: Sufficient exothermic heat is generated by the heat of hydrogenation so that preheating from outside sources is not required to sustain an ongoing hydrogenation operation. In the past, the problem has been one of making that heat available for subsequent use while still maintaining control of the reaction at the desired conditions. The use of heat sink type systems and efficient heat exchange arrangements has for the most part solved this problem. There doesn't have to be a throughput penalty.

Hastert: *What are the limiting factors to batch converter size?*

Latondress: The only limiting factors are how much concrete you want to pour in the foundations, how big a motor you want to put on the agitator drive, and how big a shaft you want to put on the agitator. It's a matter of, is it economically feasible to make it as big as you possibly can? You could hydrogenate 500 thousand pounds in a batch. However, it would take a pretty big motor to turn the agitator.

Hastert: Billie Walker, I think you're doing some looking in this direction at present.

Walker: Giles brought up a good point when he said that any time you start a batch you want to have enough hydrogen to finish it. There are a number of factors to consider — not only how efficient it would be and the size of your hydrogen plant, but also your product mix. Of course if you make a 500,000 pound batch and it's out of spec, you're going to have a lot of fun figuring out how to dispose of it.

Hastert: *What would be typical processing costs for edible oil hydrogenation, breaking it down into catalyst, hydrogen, labor, overhead, etc.?*

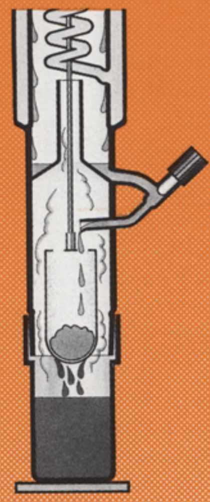
Farmer: First let's define what we mean by processing costs. I don't think I should comment on overhead costs, these vary with the operation and accounting practice. The direct costs, though they vary with the price you must pay for the raw materials and labor, can be calculated and the methodology is pretty straightforward.

For example, if you have natural gas at $3.50 per MCF, electricity at 4¢ per kWh and labor at $10 per hour and are hydrogenating SBO to a 70 IV, then the direct cost can be calculated using these usage requirements.

In a modern hydrocarbon reforming hydrogen plant, it takes 540 BTU to make 100 cubic feet of hydrogen or 19¢. If you add the cost for maintenance, catalyst, electricity, etc., you get a direct cost of about 30¢ per 100 cubic feet. Since you make more steam than is required, if you give credit for that steam you reduce the cost to about 25¢ per 100.

Using a 10% excess of hydrogen over the stoichiometric requirements, or .0165 cubic feet per IV drop, then a 70 IV oil will require 107 cubic feet per 100 lb of oil. At 25¢, that's 27¢/cwt.

You must heat the oil before starting hydrogenation. Assuming heating from 100F and using the $3.50 per MCF gas and 75% boiler efficiency, the cost is about 12¢ per cwt. Catalyst at $14 per pound nickel and use of 0.02% nickel concentration, adds 28¢/cwt. So far we have 27¢ for hydrogenation, 12¢ for steam and 28¢ for catalyst: a total of 67¢. Labor costs vary depending on the size of the operation, but 5¢ per hundred might be typical. That gives a total of 72¢, and if you add another 5¢ for maintenance and miscellaneous ex-



pense and 2¢ for electricity you are up to 79¢ per 100 lb.

Hastert: Jack Thompson, do you have anything to add to that?

Thompson: I think Giles has given us good methodology. However, if anyone here would like to sell me some hydrogen at about 67¢, please see me after the meeting.

Hastert: *What is the present state of the art and future possibilities of continuous hydrogenation systems?* I'll take this one.

Procter and Gamble have had continuous slurry hydrogenation for many years, so it obviously can be done. Fixed bed hydrogenation is another possibility. A lot depends on having good quality feedstock and good quality hydrogen, or catalyst cost for a fixed bed will be prohibitive. There has been much improvement in the quality of feedstock and we are making very good hydrogen these days. While the present state of the fixed bed art is almost nowhere, I am aware of several people working on it seriously. I predict it will be common practice in 10 years.

Hastert: *Describe the equipment and techniques to monitor the endpoint of partial hydrogenation by measuring the hydrogen added?*

Wheeler: The one I have been familiar with for about 12 years is a very simple totalizing meter in the line. It is based on 14.95 standard cubic feet of hydrogen for 1 IV drop per 1,000 lb of oil. We run 30,000 lb batches and one "count" is equivalent to 1 IV of hydrogen. The operator gets an IV on the feedstock, calculates the requirement and presets his meter accordingly.

Ritchie: We also have hydrogen meters in our plant. The flow is measured by 2 differential pressure transmitters across a single orifice plate. This configuration provides a wide range measurement system with a usable low range signal down to approximately 4% of maximum flow rate.

Although the calibration and accuracy of these meters are relatively stable, we have yet to reach the confidence level to use them for final end point determination. Our operators set the meters at 95% of anticipated H_2 requirements, then sample and fine tune the end-point using both meter and refractometer.

Hastert: I've given you an easy one this time, Giles — *Has anyone ever invented a satisfactory continuous refractometer? What are the problems in devising such an instrument?*

Farmer: If they have, I wish they would hold up their hand. What are the problems in devising such an instrument? Well, one is getting the catalyst out of a continuous stream. A small filter plugs up before the batch is complete. If you use a larger filter the residence time is too long and you aren't reading a sample representative of the oil in the converter.

The other problem, of course, is temperature. Most instruments won't function at converter operating temperature so the oil must be cooled. You must compensate for temperature and this is a problem when you are dealing with a variety of oils. The industry needs this instrument — I hope someone develops it.

Hastert: *Does feedstock purity or hydrogen purity affect hydrogenated oil SFI?*

Ritchie: Yes. Catalysts that have become partially inactivated by poisons produce hydrogenated fats that are higher in *trans* acids at a given iodine value. This, of course, changes the shape of the SFI curve. If hydrogen is carrying inert impurities, then the relationship of hydrogen, catalyst and oil are altered and this again affects SFI curves.

Hastert: That, of course, is the basis of commercial sulfur promoted catalysts which do that very thing in a predictable way.

FILTRATION AND BLEACHING

Hastert: *What are the factors in deciding which filter aid to use in the black press?*

Gil: There should be a compromise among filtration rate, Ni retentivity and oil retention in the cake.

Hastert: *How much filter aid should be used in the black press?*

Thompson: Typically we use 0.1%. A lot of it is determined by the quality of refined oil you put into the converter. If you can use less, do so.

Carroll: One other point to keep in mind is that there are various grades of filter aid available and you can experiment to find the one that gives you acceptable flow rates and acceptable clarity.

Hastert: *How much residual nickel should be in oil coming from the black press?*

Ritchie: Oil should be recirculated until nickel levels are below 10 ppm. This level is easily achieved with good catalyst. Approximately 0.2 percent activated bleaching earth will reduce nickel to less than 1.5 ppm in the postbleach.

Hastert: *What is the best chelating agent, phosphoric acid, citric acid or something else?*

Latondress: We use both phosphoric and citric when we have very good agitation and citric when we do not.

Hastert: *Where should the chelating agent be added?*

Latondress: It depends on how you are postbleaching. It's quite common to add the chelating agent as the oil is being pumped into the vacuum bleacher and then to filter the oil using just diatomaceous earth. If that practice is being followed, the chelating agent should be added as the oil is being fed into the vacuum bleacher. I personally am not terribly fond of that system. Activated bleaching clay has the advantage of effectively adsorbing nickel soaps formed during hydrogenation and removing them from the oil. If you use an activated clay for postbleaching, I recommend adding the chelating agent after the oil comes out of the postbleaching press.

Hastert: *How much chelating agent should be used?*

Carroll: Usually .01% chelating/sequestering agent is sufficient to remove the trace amounts of nickel that are not retained in the primary catalyst press. With a very good black filter operation, you may be able to use less. However, many processors prefer to use the .01 level and not take a chance there will be a problem.

Hastert: *Is it necessary to go through a* *postbleaching step?*

Ritchie: If you have bleached soybean oil before you hydrogenate it, you're not removing any color. So you really don't have to go through a postbleaching step. But, as Ed pointed out, a minimal amount of activated clay will remove some nickel and other impurities and result in a more stable oil.

Hastert: What are your comments, Jim?

Yeates: At this stage, what you did before really counts. This is the position in the process where you are going to pay the price, one way or the other, for how you have done your homework in refining and each subsequent step.

Hastert: *What practices will ensure having as little free oil in the spent catalyst as is possible?*

Wheeler: I think first and most important is to make sure the operator is aware of how much money he is dealing with here. In our experience, we blow for about 10-15 minutes with hydrogen and then with steam. Just recently we found that if we heat the hydrogen up and put it through a little exchanger, we get slightly better results. Why I don't know, but it works.

Hastert: Hector, how do you handle that in Mexico?

Gil: Our practice is to blow the black press with air until practically no oil is seen through the sight glass. In our case this will take about 15 minutes. The press is then steamed for about 30 minutes. Finally 5 minutes air blowing. The spent cake will not have more than 35% oil.

Hastert: *What safety practices should be followed when handling spent catalyst?*

Yeates: Here again you can get the runaround, depending on which authority you talk to. The current NIOSH standard for metallic nickel is one milligram of nickel per cubic meter air averaged over a 9-hour period. The final conclusion is, their only real concern seems to be exposure from the air, especially in the summer time. When it is warm, there is more perspiration and NIOSH doesn't like to see exposure of nickel dust to the outside of the body.

Hastert: I would like to say in conclusion what a pleasure it has been for me to put this session together and to work with these great fellows who have been here on the griddle for 3 hours. I believe they deserve a standing ovation. (Which they received).

(Tape recordings for transcription and photographs provided courtesy of Joe R. Crafton, Humko Products, Memphis, Tennessee.)

1982 oilseed crops

U.S. production of five major oilseeds during 1982 totaled 70.7 million metric tons, the second largest harvest ever, led by record soybean production of 61.96 million metric tons.

The U.S. Department of Agriculture's final summary of 1982 crop production, released during January, showed production of 295.5 thousand metric tons of flaxseed, 1.96 million tons of peanuts, 2.58 million tons of sunflower and 4.33 million tons of cottonseed (Table I). The 1981 oilseed production for the five crops totaled 64.3 million metric tons; the all-time oilseed harvest was in 1979 when production totaled 72.43 million tons.

The soybean crop, 2.28 billion bushels, slightly surpassed the previous record of 2.27 billion bushels set in 1979. The increase was a result of record soybean acreage plus a record national yield of 32.3 bushels per acre.

Flaxseed production surpassed 10 million bushels for the first time in three years. Annual production in 1980 and 1981 was below 8 million bushels. The 1982 production was the highest since 1979's 13.5 million bushels.

Sunflower production was the second largest in U.S. history to 1979's 7.3 billion pound harvest. Production of oil-type sunflower was 5.4 billion pounds, up 31% from 1981, while non-oil production was 293 million pounds, down 21% from 1981.

Peanut acreage dropped 14% in 1982 as a result in changes in acreage and price support programs for peanuts. Production was about 3.44 billion pounds, down about 14%. The drop in cottonseed production similarly reflected reduced acreage.

Intriguing soy oil uses

The American Soybean Association's Dave Erickson shared some of the more intriguing potential inedible uses of soy oil with Illinois farmers attending a January clinic on use of vegetable oils as carriers for herbicides and pesticides.

Soy oil or lecithin can be used in grain elevators to knock down potential explosive dust, Erickson said. And with increased use of kerosene-burning home space heaters, a vegetable oil fuel may soon be competitive with nonodorous kerosene that costs about $5 a gallon in metropolitan areas, Erickson said.

In the U.S. Gulf Coast area, one entrepreneur reports he is using soybean oil to extract the red colorant from discarded lobster shells, Erickson said. The extract is used by processors of salmon and other fish who want to add some coloring to their product, according to Erickson, who is ASA's director of soy oil programs.

Canada's success in using canola oil as a carrier for chemicals sprayed over vast forest areas is leading U.S. Department of Agriculture officials to consider vegetable oil sprays for use by the U.S. Forest Service, Erickson said.

The clinic was sponsored by a local farm equipment dealer near Effingham, Illinois, and attracted more than 100 Central Illinois soybean growers. Erickson listed physical and chemical characteristics of soybean oil that made it suitable for use as a carrier for agricultural chemicals. An equipment representative explained the theory and experience thus far in using vegetable oil as a carrier to permit reduced application rates of herbicides and pesticides. A Southern Illinois University agricultural researcher reported that the reduced volume applications of chemicals performed as well as or better than standard application rates, based on the first year of a proposed multiyear study.

The use of low-volume applications with vegetable oil carriers has become common in many U.S. cotton growing regions. Similar clinics held across the eastern United States this past winter are expected to lead to further usage in 1983. Farmers benefit in reduced cost and time, but need to learn the proper methods to use the system, which also can involve modifying old equipment or buying new equipment.

Oleochemical revival?

A study completed by Experience Incorporated, an agribusiness consulting firm based in Minneapolis, Minnesota, predicts a resurgence in the use of oleochemical raw materials by American industry.

TABLE I

1982 U.S. Oilseed Production

	Area harvested (1,000 acres)			Yield (bushels for flax, soy; pounds for peanuts, sun)			Production (1,000 bushels for flax, soy; 1,000 pounds for peanuts, sun; 1,000 tons for cottonseed)		
	1980	1981	1982	1980	1981	1982	1980	1981	1982
Flaxseed	683	617	815	11.6	12.6	14.3	7,928	7,799	11,635
Peanuts	1,138.8	1,488.7	1,273.0	1,645	2,675	2,703	2,301,282	3,981,850	3,441,435
Soybeans	67,856	66,368	70,783	26.4	30.1	32.3	1,792,062	2,000,145	2,276,976
Sunflower	3,683	3,611	4,924	1,016	1,177	1,156	3,741,640	4,487,410	5,690,660
Cottonseed							4,471	6,397	4,777

Source: USDA 1981 Annual Summary Crop Production (CrPr 2-1 (83)).

"Any unexpected rapid rise in petroleum prices will accelerate the substitution of natural raw materials wherever their use is possible," according to William A. Jarvey, a senior consultant for Experience Inc. Oleochemical raw materials are used in the production of detergents, protective coatings, plastic and food additives, lubricants and intermediates for textile processing, beneficial mining, adhesives and polymers.

Also examining worldwide fats and oils production and demand, the study projected that population growth and rising living standards in developing countries will result in a 3.1% average annual increase in consumption to 1989-90. "Palm, sun and rape oil will all increase their share of the fats and oils market, but soy oil, of which the United States is the dominant producer, will continue to be the principal vegetable oil," William A. Hand, another senior consultant, said.

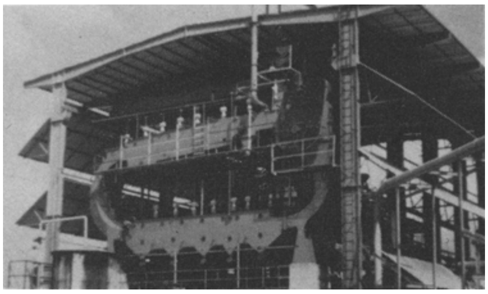
Adhesive forecast

Demand for adhesives and sealants in the U.S. market is forecast to grow at a rate of 6% a year to about 9.5 billion pounds in 1986, according to Strategic Analysis Inc., business consultants in Reading, Pennsylvania. The consulting firm said this represents a 34% increase from the estimated 7 billion pounds consumed in 1981.

In its survey "U.S. Manufacturers of Specialty Chemicals," the firm predicted demand will grow as new applications are developed and as these materials replace conventional fasteners. Copies of the study are available from Strategic Analysis Inc., 2525 Prospect St., Reading, PA 19606.

News briefs

A Crown extractor has been installed at Oleoproteinas Del Suresta S.A., Merida, Mexico, a soybean processing plant with 600 tons a day capacity. Second phase plans are to install a Crown meal dryer-cooler. Raul Tamayo, plant superintendent, is in charge of the construction. . . . California Fats & Oils Inc. has purchased the Richmond, California, oilseed crushing and processing plant that formerly belonged to PVO International Inc. President of the new firm is C.D. Hultberg, the former head of PVO's Philippine subsidiary. The firm has retained PVO's quality control, sales and manufacturing staff. . . . Davy McKee Constructors Inc., a new subsidiary of Davy Inc., began operations as of Jan. 1, 1983. The new company, headquartered in Cleveland, Ohio, will undertake construction projects nationwide for the process industries. . . . Perbadanan Nasional Shipping Line Berhad and Stolt Tankers and Terminals have established a joint venture company, Parcel Tankers Malaysia Snd. Bhd., to serve primarily the Malaysian palm oil trade. . . . Sherex Chemical Company has announced it stopped marketing epoxidized soybean oil as of Dec. 31, 1982. Instead, the company said it is concentrating on marketing and producing its Admex Polyester Plasticizers and unique performance polyester plasticizers. . . . Eschem Inc., parent firm of Swift & Co., has announced plans to construct a 30,000 square foot research and development center in Downers Grove, Illinois. The present R&D laboratory is in the Swift Technical Center in Oak Brook, Illinois. The new facility is expected to be ready by the fall of 1983 for the 50 scientists and support staff who will transfer from Oak Brook. Eschem is a division of Esmark Inc. . . . Escher Wyss Limited has changed its name to Sulzer-Escher Wyss Ltd. following the integration of the Thermal Turbomachinery Division of Sulzer Brothers Limited, Winterthur, into Escher Wyss. Executive management for the new company includes Jakob Kägi, member of the Sulzer Corporate Executive Management, as president; Helmut Pirchl, mechanical equipment for hydraulic power stations; John P. Nägeli, thermal turbomachinery; Emil Lutz, production; and Jakob Wydler, finance. . . . Bauermeister GmbH of Hamburg, West Germany, has merged with Probat-Werke von Gimborn GmbH and Co. of KG. of Emmerich, West Germany. Probat-Werke and Bauermeister GmbH, both family-owned companies, have been suppliers to the food industry for over 100 years.



Crown extractor.

U.S. Soybean Crush—Fourth Quarter 1982

	Total capacity (million bushels)	Total crush (million bushels)	% capacity	Oil produced (thousand lb)	Oil yield	Meal produced (thousand tons)	Meal yield
October	127,481	98,406	77.2	1,075,235	10.93	2,367	48.11
November	121,763	107,552	88.3	1,135,912	10.56	2,551	47.43
December	126,685	111,556	88.1	1,183,203	10.60	2,647	47.45
Total for quarter	375,929	317,514	84.5	3,394,350	10.69	7,565	47.65