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June 3, 1954

MEMORANDUM OF DISCUSSIONS

Shell Chemical Corporation
Denver, Colorado

Denver, Colorado
May 10 and 11, 1954

Denver
Mr. R. H. Bellin
Mr. P. R. Bolenbaker
Mr. J. H. Knaus
Mr. E. M. Lando
Mr. C. C. McKenna (part time)
Mr. G. McMasters (part time)
Mr. R. E. Scheidt
Mr. R. M. Stager (part time)
Mr. M. Smith
Mr. S. Zevin

New York
Mr. B. H. Cummings
Mr. W. C. Walker

EPON ADHESIVES VI AND VIII

A meeting was held between Denver and New York personnel to discuss the nature of the shortcomings of the Epon Adhesives VI and VIII manufactured at Denver and to develop information to carry to Emeryville regarding procedures and equipment for discussion with Emeryville personnel. Excessively high viscosity for the Adhesives and the reported short "pot life" for one batch of Adhesive VIII were the main factors necessitating such a meeting. The viscosity of a number of batches of Adhesives from Denver has exceeded the sales requirements of 1 and 2 MM cp. for Adhesives VI and VIII, respectively. In addition, recent information was received from Western Division Marketing of a complaint from North American Aviation that the "pot life" of a batch of Adhesive VIII was only 20 minutes compared with the minimum of one hour which they consider satisfactory.

Discussions were initiated with a review of the Denver and Emeryville procedures for preparation of Adhesives VI and VIII. A description of each of the manufacturing processes for both locations is appended. The principal difference between the Emeryville and Denver procedures is the much longer batch times at Denver. Preparation of the Adhesives is essentially a mixing operation, with all of the ingredients blending together relatively easily excepting the Vinylite AYAF which is added to the mixture as 1/8"-1/4" pellets. The Vinylite dissolves very slowly at temperatures less than 100°C and must actually be melted to obtain ready solution. At Emeryville the Vinylite is predissolved in the Epon 828 for a batch of Adhesive at 160°C in a stirred, heated kettle. At Denver no separate kettle is used to predissolve the Vinylite -- all the ingredients for a batch being added to a Read mixer and mixed at 90°C. At this lower temperature the time required to dissolve the Vinylite is not definitely known, but the mixture is not homogeneous after six hours mixing. For operating convenience the mixing is allowed to continue overnight at this temperature, with a total of twenty hours elapsing from the beginning of the batch until the Adhesive is ready for packaging.

A brief discussion of the conditions which might possibly lead to loss of allyl glycidyl ethyl (AGE) -- loss of which would increase the viscosity of the Adhesive -- indicated that the Denver apparatus is not completely vapor tight but that a metal cover on the mixer prevents stray air currents from contacting the batch. The joints between the cover and the mixer appeared to be effectively sealed by the Adhesives. Preliminary laboratory tests have shown that substantially all the AGE is retained in the batch based upon a volatiles recovery test. However, the volatiles removed were not analyzed and may contain materials other than AGE. This volatiles test presently consists of measuring the weight loss from a uniform film of Adhesive 1/16" thick on a glass slide heated for one hour at 180°C at 10 mm. mercury absolute pressure.

Denver has found that a batch of Adhesive VIII prepared in the laboratory under plant conditions had a lower viscosity than the plant material, 1.3 MM cp. versus 2.0 MM cp. However, the Epon 828 used in the laboratory batch was not identical with the plant batch and, therefore, results are not strictly comparable. For instance, the viscosity of Epon 834 varies widely. Viscosity determinations of 1.1 MM cp. and 2.0 MM cp. have been made at Denver on separate batches of Epon 834.

A discussion of the possible effect of raw material properties on viscosity of the finished products indicated that there is little likelihood that variations in the Vinylite AYAF have any significant effect. The proportion of Vinylite to that of 828 is low (4.3%w), and the variations of viscosity of 828 are undoubtedly fully as large as those of Vinylite.

Pending discussions with Emeryville personnel, a further program of process improvement was discussed. It was generally agreed that all process improvement studies and the corresponding new Adhesive preparations would be made at Denver. In any event, Denver's program will include laboratory test runs to establish laboratory repeatability and reproducibility of viscosity measurements using a new 1-30 MM cp.-range Brookfield Viscometer just received at Denver May 11.

During the time remaining from the discussions at Denver, laboratory and plant facilities were inspected keeping in mind the requirements which might be imposed should it be decided to provide product applications testing facilities at Denver. In general, Denver has adequate laboratory and plant space for any foreseeable developments in Adhesive manufacture.

Shell Development Company
Emeryville, California

Shell Development
Mr. R. M. Cole
Mr. H. DeJong
Mr. D. W. Elam
Mr. H. C. Klute
Mr. F. M. McMillan
Miss M. Naps
Mr. C. Rapean

Emeryville, California
May 12, 13, and 14, 1954

Denver
Mr. P. R. Bolenbaker
Mr. J. H. Knaus

New York
Mr. B. H. Cummings
Mr. W. C. Walker

Western Division
Mr. A. P. Howe
Mr. J. Morrill

Discussions were held at Emeryville on May 12, 13, and 14, 1954, to review the current situation on Adhesives and to determine upon a program for future operation in this field. The present problem of high viscosity in the Denver production of both Adhesives VI and VIII was reviewed from the standpoint of manufacturing technique, customer requirements, and immediate supply.

An exhaustive discussion of the process variables which might influence the properties of Adhesives VI and VIII indicated that the major point of departure of Denver from Emeryville's procedure was the long mixing time at Denver for the complete formula at 90°C. Miss Naps reported that the pH of asbestos floats is on the alkaline side (8-11 pH) and that there was the possibility of further hydrolysis at 90°C to yield alkaline materials. Since Epons are known to react with alkali at high temperatures with consequent increases in melting point and weight per epoxide equivalent, there is a strong possibility that excessive mixing of the Adhesives at elevated temperatures may lead to a viscosity increase. The possibility of reaction between AGE and Epon also exists. Denver's laboratory experiment, reported in discussions at Denver, does not substantiate this theory, and further control experiments will be required to establish the point.

Denver will proceed immediately with the necessary experiments in the laboratory. Meanwhile, the plant will initiate equipment changes to permit following Emeryville's procedure in all details. It will be necessary that promising laboratory samples be evaluated for performance by both Union and Emeryville before Denver will proceed to make plant-scale batches.

Close visual inspection of samples of Adhesive VIII from Emeryville and Denver reveals discrete particles of Titanox in the Denver sample. Here again, a difference in manufacturing procedure is probably responsible for the difference. At Emeryville a 60%w concentrate of Titanox in Epon 828 or Epon 834 is made by mixing the two materials and passing them four times through a Morehouse Hy-R-Speed mill. This procedure was adopted at

Emeryville to eliminate large particles of Titanox which were otherwise obtained in the Adhesives. It is felt that a comparable method of Titanox dispersal should be adopted for Denver. Since a grinding mill is not available at Denver, means will be investigated for having this operation performed for Denver by some firm specializing in this work.

Some time was devoted to discussing North American's report of excessively short "pot life" for Denver Batch 11 which had shown a good viscosity (1.67 MM cp.). From the scanty evidence at hand it would appear that "pot life", as indicated by North American's test, is not necessarily measurable as viscosity, and further study of consistency will probably be required. Various types of apparatus for determining consistency were described by Mr. McMillan. Among those mentioned were the Gardner Mobilometer and the Stormer Viscosimeter as being perhaps more suited to Epon Adhesives than the Brookfield Viscometer.

A group of tests made with Adhesive VIII uncatalyzed, using a Brookfield Viscometer operated at different rpm. at room temperature, showed that apparent viscosity drops with rate of shear on Adhesive VIII but did not explain the short "pot life".

RPM.	Shell Dev. VIII*	Denver VIII Batch 11**
10	800,000 cp.	805,000 cp.
5	960,000	930,000
2	1,540,000	1,120,000
1	2,170,000	1,280,000

* Old batch supposedly OK.
** Insufficient sample. Brookfield spindle not at prescribed depth.

The effect of the presence of catalyst on stiffening was examined for three batches of Adhesive VIII: Batch 11, Batch 8, and a sample of Emeryville production. The Adhesive was catalyzed with 6 parts by weight of Catalyst A per 100 parts of VIII and spread with glue spreaders on sheets of 20 in. x 20 in. masonite panel. Immediately after adding catalyst and 20 minutes later there was little noticeable difference in spreadability. However, to some observers the 20-minute spread for Batch 11 seemed slightly more stiff and tacky. All three batches were still spreadable after one hour. After standing four hours the catalyzed batches had all stiffened noticeably, but Batch 11 was much stiffer and more rubbery than either Batch 8 or Emeryville material. From this it would appear that the cure may have been advanced in Batch 11 before the catalyst was added but that viscosity of the uncatalyzed resin is not necessarily an index of cure.

On the basis of these findings, it was decided that an accurate evaluation of North American Aviation's problem could be achieved only by first-hand inspection of their operations. Accordingly, arrangements were made to visit their assembly plant on Monday, May 17. On reaching this

decision, it was agreed that Emeryville would not be asked to make any Adhesive VIII until after a visit to North American.*

Stocks of Adhesive VIII presently at Denver are as follows:

Batch	Lot	Pounds on Hand	Viscosity MM cp.	Tensile Shear		
				77(2500)	180(1250)	-70(2500)
2	A	495#	2.43	3110	3144	2834
4	A	360#	2.56	3183	2966	2962
5	A	720#	2.11	3453	3266	2792
6		1350#	2.43	3013	3242	-
10	A	720#	1.67	3150	3200	2840
	B		"	3126	3125	2610
	C		"	3090	3212	2470
12	A	1395#	1.73	3330	3412	2470
	B		"	2920	3055	2570
	C		"	2838	3139	2440
13		1300#	1.92	2820	3000	2550
14		1400#	2.05	2910	3120	2360

Batches 16, 17, and 18 (not shown above) were made by blending existing stocks to reduce the Vinylite AYAF to 6 phr. These are undergoing evaluation tests. These three batches amount to 4,200 lb. The situation, therefore, is one where about 2,000 lb. of marginally acceptable material is on hand at Denver, with the possibility that more may pass specifications but have yet to be evaluated. Whether it will be necessary to have Emeryville make more Adhesive VIII will depend upon the outcome of discussions to be held at the North American Aviation assembly plant in Torrance on May 17 and upon Denver's ability to develop an acceptable Adhesive on short notice.

Some time was taken to discuss testing equipment for possible installation at Denver in order to expedite application evaluation of Adhesive products. Mr. H. C. Klute of Emeryville advised that the Tinius-Olson Tensile Tester, costing in the neighborhood of $6,000, was the cheapest apparatus that would be capable of giving satisfactory results. To this would need to be added a high temperature and a low temperature enclosure for samples at about $1,000 and $1,600, respectively. With all auxiliary equipment he "guesstimated" that the complete testing apparatus might cost about $10,000. Other manufacturers of such equipment were listed and will be investigated.

There was considerable discussion on coordination and close liaison between Shell Development, Marketing, and Manufacturing on both new processes and on any desired changes in specifications for products

* Emeryville has subsequently been requested to make 3,000 lb. of Adhesive VIII and to maintain minimum stocks of 2,000 lb. each of Adhesives VI and VIII until further notice.

being manufactured. A case in point with regard to changes in specifications on an existing product was the recent change in the formula for Adhesive VIII, reducing the Vinylite content from 8 to 6 phr. Epon 828. Denver was not advised of this change, and several batches of Adhesive VIII were made to the old recipe before the change was discovered.

For the information of the group, the representative from Shell Chemical Manufacturing Operations described the procedure for establishing a specification change in a commercial product as follows:

1. Marketing may initiate a request for a change in specifications by letter to Manager, Manufacturing Operations.

2. Upon written notice of the acceptability and establishment of the revised specifications by Manufacturing Operations, Marketing may place orders for product to meet the new specifications.

3. Conversely, Manufacturing may desire to change a product specification and will write a request for approval to Marketing. Upon written approval by Marketing, the change will be established in the Product Specifications Manual.

4. No shipping orders for products outside authorized specifications will be honored by the manufacturing plants without authorization from Manufacturing Operations.

With respect to new processes, Shell Development brought out the benefits to be gained by a Shell Development representative(s) most familiar with the process following the process to Shell Chemical for initial operation. This applies for small projects as well as major ones since frequently the effects of all process variables have not been completely worked out for a small project where exhaustive research is not justified. In some cases, the feel, smell, or looks of a product may be extremely significant but are often difficult to convey by the written word. For example, manufacturing of Adhesives might be compared with home baking where, as is well understood, two cooks working from the same recipe can turn out entirely different types of cake.

The visit to North American Aviation on May 17, 1954, is covered in a separate memorandum of discussion.

B. H. Cummings

W. C. Walker

BHC/WCW:mev

SC NY

Distribution:

Julius Hyman & Co. - Mr. J. G. Bejarano (4)
Shell Development Co. - Messrs. B. S. Greensfelder ◄THIS COPY FOR►
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Shell Chemical Co. - Messrs. F. S. Swackhamer
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DENVER PLANT

EPON ADHESIVE VI MANUFACTURE

OPERATING PROCEDURE

At least 24 hours before a batch of Adhesive VI is to be mixed, two drums of Epon 834 should be placed in the hot-box to liquefy the resin. The adhesive is prepared by mixing by weight in the blender as follows:

1. Allyl glycidyl ether (AGE), 177 lb.
2. Acetamine orange, 147 grams (in cabinet, M. Smith's office).
3. Vinylite AYAF, 94 lb.
4. Titanox, 8 lb. 2 ox. Mix ten minutes, turn on steam, and add
5. Epon 834, 500 lb. (1 drum).
6. Asbestos floats, 353 lb. This should be added in 100 lb. increments, seeing that each addition is mixed in before adding more.
7. When asbestos has almost all been pulled into the mix, stop the blender, scrape down the walls to the point where the blades reach, mix again for a few minutes to be sure all the asbestos has been worked in, then add
8. Epon 834, 500 lb. Allow to mix overnite, reversing the direction of agitation occasionally. Inspect to see that no unmixed resin remains on top. If there is such, work it in with the scraper by hand.
9. When batch is ready, bolt down the blender lid, set up the scale and pour the adhesive into cans as specified. Containers will be 1-gallon, holding 8 pounds, or 5-gallon, holding 40 pounds net. Label each can with designation "VI" and batch number. Each batch is split into lots "A", "B", and "C"; lots to be 500 lb. About at the middle of each lot, take two 1-pint samples properly labeled, which are delivered to R. Bellin in the Laboratory.
10. Toward the end of the packaging, it will be necessary to run the blender in the proper direction to push out the bulk of the adhesive. The last of the contents will have to be scraped out. If another batch of the same adhesive is to be made, the blender need not be scraped clean. If another type of adhesive will be made next, scrape out as much of this batch as possible.

Elapsed time/batch is 20 hours at the beginning of packaging.

DENVER PLANT

EPON ADHESIVE VIII MANUFACTURE

OPERATING PROCEDURE

At least 24 hours before a batch of Adhesive VIII is to be mixed, check to see whether the Epon 828 to be used is in bung or open-head drums. Open-head drums can be used cold; bung type drums must be heated in the hot box before they will run well.

The adhesive is prepared by mixing by weight in the blender as follows:

1. Allyl glycidyl ether (AGE), 20 lb.
2. Vinylite AYAF, 60 lb. (recently reduced from 80 lb.).
3. Titanox, 7 lb. Mix a few minutes and add
4. Epon 828, 500 lb. (1 drum). Turn on steam, mix 10 minutes, and add
5. Asbestos floats, 300 lb. This should be added in 100 lb. increments, seeing that each addition is mixed in before more is added.
6. When asbestos has almost all been pulled into the mix, stop the blender, scrape down the walls to the point where the blades reach, mix again for a few minutes to be sure all the asbestos has been worked in, then add
7. Epon 828, 500 lb. Allow to mix overnite, reversing the direction of agitation occasionally. Inspect to see that no unmixed resin remains on top. If there is such, work it in with the scraper by hand.
8. When batch is ready, bolt down the blender lid, set up the scale and pour the adhesive into cans as specified. Containers will be 1-gallon, holding 8 pounds, or 5-gallon, holding 40 pounds net. Label each can with designation "VIII" and batch number. Each batch is split into lots "A", "B", and "C"; lots to be 500 lb. About at the middle of each lot, take two 1-pint samples properly labeled, which are delivered to R. Bellin in the Laboratory.
9. Toward the end of the packaging, it will be necessary to run the blender in the proper direction to push out the bulk of the adhesive. The last of the contents will have to be scraped out. If another batch of the same adhesive is to be made, the blender need not be scraped clean. If another type of adhesive will be made next, scrape out as much of this batch as possible.

Elapsed time/batch is 20 hours at the beginning of packaging.

SHELL DEVELOPMENT COMPANY

EPON ADHESIVE VI MANUFACTURE

1. Dissolve Vinylite AYAF in AGE at 50°C 4 to 10 hours in a stirred, heated kettle. Fifteen parts AGE and eight parts Vinylite are used.
2. Add 0.03 parts acetamine orange.
3. To a Read mixer charge 85 parts Epon 834
4. Heat to 70°C.
5. Add 30 parts asbestos floats and 0.7 parts Titanox as a preground 60% concentrate in Epon 834.
6. Mix for two hours at 70 to 75°C.
7. Add the 23 parts AGE-Vinylite mixture from the stirred kettle.
8. Blend for 30 minutes.
9. Drum at 50-60°C.

SHELL DEVELOPMENT COMPANY

EPON ADHESIVE VIII MANUFACTURE

As a preliminary step in the formulation, a 60%w concentrate of titanium dioxide (Titanox) in Epon 828 is made by premixing the two materials and then passing the mixture through a Morehouse Hy-R-Speed mill using 60 grit carborundum stones. Four passes at about 80-100°C are normally required to obtain a smooth mix and sufficient material is prepared for several adhesive runs.

In a 100-gallon tank, 500 pounds of Epon 828 is heated to 160°C and 30 pounds of Vinylite AYAF is added. The mixture is then stirred at this temperature for one hour to obtain complete solution of the solids in the liquid. As the Epon received at Emeryville occasionally contains foreign particles, the solution obtained after dissolving the Vinylite is filtered hot through an 18-mesh screen. Approximately one-half of the filtered Epon-AYAF solution is transferred to the 150-gallon Read mixer and to this is added 5.8 pounds of Epon-Titanox concentrate. The temperature at this point is about 100-110°C, and after mixing for a few minutes, 150 pounds of asbestos floats is added in portions over a period of 15-30 minutes. Mixing is continued for 2 hours at 90-100°C, and during this time the direction of the blades is reversed at 15-minute intervals to insure thorough blending. With completion of the above period, the remainder of the Epon-AYAF is added to the mix and blending continued for one hour. The charge is completed by the addition of 10 pounds of allyl glycidyl ether, and after a final mixing period of 30 minutes at 80-90°C, the finished adhesive is discharged to suitable containers.

As no corrections are made for the amount of Epon 828 introduced with the addition of the Titanox concentrate, the final formulation has the following composition:

Epon 828	100.5 parts by weight
Vinylite AYAF	6
Asbestos Floats	30
Allyl Glycidyl Ether	2
Titanox	0.7