



Julius HYMAN & Company

DENVER 1, COLORADO

2 December 1954

SHELL CHEMICAL CORPORATION
50 West 50th Street
New York 20, New York

Attention: Mr. R. L. Kittle

Gentlemen:

EPON ADHESIVE VI

We have received your letter of November 22 and the samples of Adhesive VI obtained from shipments to Fairchild Guided Missiles Division, Fairchild Engine and Airplane Corporation.

The sample of Lot 5-C, which appears to be undyed, is Adhesive VI from our production. It is believed the dye had been added to the adhesive after the asbestos was added rather than to the Vinylite-AGE. This procedure has been found to considerably reduce the intensity of the adhesive color. The dye is added only for identification purposes and has no affect on the performance of the adhesive. This material conforms to the MIL-A-8331 specification tensile shear strength requirements. Obviously it is high viscosity (> 2 MM CP @ 77°F) material. You are aware of this characteristic and our program to reduce the viscosity.

We do not have specific information as to the nature of the performance objections; therefore, are not in a position to further evaluate the material effectively.

With reference to the remaining dyed sample (Emeryville production), the separation of liquid upon prolonged standing appears to be characteristic of Adhesive VI. This should not affect its performance or application. We are forwarding this sample to Emeryville and by copy of this letter requesting their examination or comments. A copy of your letter will also be attached to advise them of Fairchild's complaint.

PLAINTIFF'S EXHIBIT

SH-1037

REC'D DEC 6 1954
18709

TFB *Sam*
EEG *7C*
DYE *✓*

TO HANDLE:

Notify P&S
Sample to TFB

all memo
1-7-55
SC Denver to SC NY

ANS'D 12/24/54 BY: *TFB/DYE*

LAM 029026

ABS-018232

SHELL CHEMICAL CORPORATION
50 West 50th Street
New York 20, New York

Attention: Mr. R. L. Kittle

2 December 1954

Page 2

We feel no further work on this problem is practical until detailed information about Fairchild's application and difficulty is available.

Very truly yours,

JULIUS HYMAN AND COMPANY

ORIGINAL SIGNED BY J. G. BEJARANO
J. G. Bejarano,
Executive Vice President

cc: (with attachment)

Att: T. M. Bradley - Shell Development - Emeryville

← Copy for →

LAM 029027

ABS-018233



SHELL CHEMICAL CORPORATION

RCA BUILDING
50 WEST 50TH STREET
NEW YORK 20

November 22, 1954

Julius Hyman & Company
P. O. Box 2171
Denver 1, Colorado

Attention: Mr. J. G. Dejarano
Executive Vice President

Gentlemen:

EPON ADHESIVE VI

Under separate cover we are forwarding to you two samples of the Epon Adhesive VI which was shipped to Fairchild Guided Missiles Division, Fairchild Engine & Airplane Corporation.

We understand that Fairchild have had variable results using these two batches, and as you will see on examination, one appears to be dyed and the other undyed. There appears additionally to have been a separation of liquid from the adhesive to form an upper layer. We presume that this material has been manufactured at Emeryville; however, on the chance that it may have been produced at Denver, we are sending it to you first for your examination. If it is determined that this material was not manufactured at Denver, we would appreciate your forwarding these samples to Emeryville and advising them of Fairchild's complaint. Because of the difference in the appearance of the two samples, there is some question as to the identity of the adhesive, that is whether it is Adhesive VI or Adhesive VIII.

We would appreciate your advising us of what action you take in order that we may report our findings back to Marketing.

Very truly yours,

ORIGINAL SIGNED BY R. L. KITTLE

R. L. Kittle, Manager
Manufacturing Operations

BHC:mev

bc - Shell Development - Mr. R. M. Cole ~~THIS COPY FOR~~
H.O. - Resins & Plastics - Mr. F. S. Swackhamer

SHELL DEVELOPMENT CO.	
RECD NOV 24 1954	
FOR C	FOR G
for 24	for 24
for 24	for 24
TO HANDLE:	
copy TFB, FB, BWE	
11/24/54	
ANSO: 12-54 BY J. G. Dejarano	

Epon Adhesive Lot LR 432-103 9.4" shipped 3/28/51
" " 777-189-2 16.0" " 1/25/52

ABS-018234

MESSAGE

270.21
☐ INCOMING ☒ OUTGOING
☐ EMERGENCY ☒ REGULAR

SEPTEMBER 29, 1954

NO. 83

SHELL CHEMICAL CORPORATION
50 WEST 50TH STREET
NEW YORK, NEW YORK
ATTENTION: DR. J. P. CUNNINGHAM, PRODUCT DEVELOPMENT DEPARTMENT

REURTEL 616 OF 9/28/54. CONFIRM THAT EPON ADHESIVE LOTS
777-144 AND 777-153-1, SUPPLIED BENDIX LATE 1951 WERE ADHESIVE VI.

SIGNIFICANTLY ORANGE DYE WAS NOT ADDED TO THESE BATCHES
ALTHOUGH CURRENT PRACTICE IS TO ADD DYE TO ADHESIVE VI AND NOT
ADHESIVE VIII. PRIOR TO THIS PRACTICE BOTH ADHESIVES WERE GRAY.

F. B. HILMER MARKET DEVELOPMENT DEPARTMENT EMERYVILLE

GAA:gil

LAM 029029

ABS-018235

WORK WITH 14-10-21-54
J. H. H. JO. SE. NY
C. H. H. JO. SE. NY

771-050

SHELL DEVELOPMENT CO.			
REC'D OCT 25 1954			
10622			
TFB	S. H.	FF	Car
FEL	S. H.		
DVE	S. H.		
CHK	S. H.		
TO HANDLE:			
Julius Hyma and Company			
C. R. L. M. P. X. R. N. H. H. S.			
ANS'D:		BY:	

MEMORANDUM OF DISCUSSION - EPON ADHESIVES

Time and Place: September 14 and 15, 1954 - Shell Development Co., Emeryville, Calif.

Participants: Shell Development

Julius Hyma and Company

T. F. Bradley ← Copy for →
D. W. Elam
C. H. Klute
J. C. Rapean
H. DeJong

S. Zevin

see well
#268, 9-8-54
S. H. H. to S. D. H. H.

A visit was made to Emeryville for the purpose of watching them prepare a batch of Adhesive VI in their equipment using Denver EPON 834 stocks. This project was designed to answer the question of whether a specification viscosity adhesive could possibly be made from the EPON 834 used at Denver.

PREPARATION OF ADHESIVE VI

A batch of EPON Adhesive VI was made with EPON 834, lot 3B1, from Denver stock on September 13, 1954 so that it would be ready for testing by September 15. A second drum of EPON 834, lot 3B1, shipped was found to have the gasket dried out and crumbled. Before this material could be used, it would be necessary to strain out the rubber shreds. Therefore a demonstration run with Emeryville EPON 834, lot 43156, was made by their usual process as follows: The drum of EPON to be used was placed in a hot-box at 70°C for about 8 hours. Before dumping into the blender, it was allowed to stand about for a while so that the resin actually entering the blender was at 34°C. Gross and tare weights were taken on the drum showing 492 lb charged. Steam was then turned on to the blender coils, slowly heating the charge. With the agitator running, 176.5 lb asbestos floats, grade 7TF2, were slowly sprinkled on the EPON and mixed in. Very little dry asbestos was ever visible. Asbestos addition took about an hour, after which 5520 grams of a 33.3% TiO₂ dispersion in EPON 834 was added. This dispersion had been prepared in a colloid type mill using coarse carborundum stones as grinding surfaces. Blending was then continued for 2-1/2 hours, reversing the blades occasionally. At this time the blender contents has reached 70°C. An AYAF solution had been prepared by heating 855 lb allyl glycidyl ether in a 300 gallon stainless steel autoclave to 60°C, adding 456 lb Vinylite AYAF and mixing for six hours at 60°C. Then 714 grams acetanilide orange dye were added and mixing continued for another hour. The solution was then stored in drums for future use. Of this solution, 135.5 lb were weighed out in 5 gallon pails and poured into the running blender. When all was in, a loose fitting cover was clamped on and agitation continued 30 minutes. The finished adhesive was then dumped by tilting the blender after fastening a weir plate across the top. During the dumping operation an exhaustor was turned on to prevent allyl glycidyl ether vapor escaping into the room. Packaging was into 5 gallon open head pails to a gross weight of 46 lb, net about 40 lb.

LAM 029030

ABS-018236

This blender, while similar to the one at Denver, differs in several respects. It is somewhat smaller, so that an 816 lb batch almost covers the blades. These blades run in the same direction and at the same speed of 30 RPM. The Denver blender operates at a 1632 lb charge which just covers the blades. The rear blade runs at half the speed of the front blade and in an opposite direction. Speeds are 25 and 50 RPM. The Denver blender is steam jacketed, permitting quicker heating and higher temperatures.

Viscosity of the batch made on September 13 from EPON 834, lot 3DL, was 960 M CP thus showing that the resin used can make a low viscosity adhesive.

TESTING PROCEDURE AND EQUIPMENT

Panels to be used in testing the adhesives are sheared from 0.64" sheets of 24S-T3 Alclad aluminum to a size that will allow sawing out 12 strips 1" wide. They are notched on each end 1/2" from an edge to provide gage marks when cementing. The panels were then washed in trichloroethylene, dried, etched 20 minutes in a 160°F dichromate solution, (10 parts $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ + 50 parts 90% H_2SO_4 + 340 parts distilled water) washed, dried and stored in a dust-free place. Adhesive was weighed out and 5.8 parts Curing Agent A added per hundred parts adhesive, mixed, and applied to the etched edge of the strips. It was observed that the time between mixing and application was allowed to range from about 1/2 to ten minutes. The adhesive was then spread out to a film thickness of .006" by a doctor blade. Two such panels were then matched by the edge notches, backed by bakelite bars 12" x 1" x 1/4" which were protected by cellophane strips and held by six clamps. These are spring loaded "C" clamps, 3" to 5" jaw depth, with an opening of about 3/4". Clamping pressure is estimated at 30 lb, but it will vary with the condition of the spring and the set of the jaws. The source of these steel body clamps was not known, but a similar die-cast clamp is available at Aircraft Tools, Inc., Los Angeles, California. The latter are not desirable as they take on a permanent set during the curing cycle. Curing was stated to be at 200°F for 45 minutes. (Union Laboratories Report SP-10-90 of May 10, 1954 states that curing time should be 90 minutes @ 200°F.) Strips are cut to a 1" width, as measured by a steel rule, on a small band saw. Edges are left rough. No objection was seen to our using steel backing bars, circular saw, or screw "C" clamps if they were not turned to tight.

During discussions with Mr. C. H. Klute it was learned that while a little difficulty may be experienced at -70 and 180°F in obtaining uniform temperatures when testing adhesive cemented strips, at 500°F fluctuation (temperature differences with time) and variation (temperature difference with location) both became quite large. Sub-zero temperatures were not especially difficult to attain or control. A report (Emeryville No. P-366) describes the hot and cold boxes in use and their operating characteristics. In brief, constancy of 2°C or less may be expected from -70 to 200°F, with fluctuations of 5° and variations of 13°C at 500°F. Recovery time of two to four minutes were observed at -70 and 180°F. No tests were made at this time at 500°F. It was recommended that all heated fixtures be especially normalized to prevent distortion at 600°F. For high temperature work it is imperative that rapid changes of samples be possible. Prolonged manipulation is quite difficult in a hot box. Grips similar to those detailed in the above report were advocated.

LAM 029031

ABS-018237

Other tests have been run on the same material and have shown that the curing rate, viscosity, and their effects on properties of Adhesive VIII suggested that an effort be made to tighten the specifications under which Adhesive VIII is manufactured and to relate the specifications to those properties important to adhesives. It was suggested that these variations may be a function of minor differences in EPA:epi-allylhydrazine ratios or differences in washing or drying procedures. Another possibility for variations in EPON properties (as pointed out by the Vinylite group) may lie in the molecular weight distribution. That is, while the average molecular weight of two samples may be the same, in one case most of the individual molecules may lie close to the average; in the other case the average is the result of a mixture of both very high and very low weight molecules.

PAANEL DISCUSSION OF ALTERNATIVE VARIABLES

As a general discussion called by Dr. E. F. Bailey, several items regarding adhesives and EPON were brought forth. The wide variations in EPON 828 curing rates and viscosity, and their effects on properties of Adhesive VIII suggested that an effort be made to tighten the specifications under which Adhesive VIII is manufactured and to relate the specifications to those properties important to adhesives. It was suggested that these variations may be a function of minor differences in EPA:epi-allylhydrazine ratios or differences in washing or drying procedures. Another possibility for variations in EPON properties (as pointed out by the Vinylite group) may lie in the molecular weight distribution. That is, while the average molecular weight of two samples may be the same, in one case most of the individual molecules may lie close to the average; in the other case the average is the result of a mixture of both very high and very low weight molecules.

As an example of variations in curing rate, Dr. Bailey cited the experience of an aircraft manufacturer testing Curing Agent 81 (methyl hexamethylene) on a standard lot of Adhesive VIII required four hours to cure while lot 15 cured in one hour. Batch 15 contained equal parts of EPON 828, lots 846-1151 and 7. Batch 81 was made one fourth EPON 828, lot 195-2054, remainder unknown. Dr. Bailey suggested that the question of EPON variations and specifications be discussed at the Machine Meeting October 21 and that it may be helpful to have a representative from Denver attend. No immediate explanation was seen for the high viscosity of EPON Adhesive VIII made at Denver, it being agreed that a laboratory test be made in Denver using solvent and Vinylite shipped in sealed cans from Knoxville. If the high humidity at the Denver location has raised the moisture content of either solvent or Vinylite, it may show up as a difference in resulting adhesive viscosity. It is known that the addition of water to a base while blending will lower the viscosity and cure time. Suggestions that specifications be broadened for possible inclusion of other solvents were objected to on the grounds that users believed that the present solvent was the best, and that a more easily tested and controlled base would have great comparative advantage.

General remarks of the panel were as follows: The panel was told that the curing rate of one can of Adhesive VIII (60 N CP) to 90 N CP was about 10 minutes. The other samples showed curing times of 10 to 30 N CP and from 10 to 30 N CP in 20 days. Lowering curing times later, 30 N CP change at 10 N CP from 60 to 30 in 20 days.

Report that the following data pertaining to this problem are: T.I. 8110, pages 308-310 (Aug. 10, 1954) and 311-312 (Aug. 10, 1954) and 313-314 (Aug. 10, 1954) and 315-316 (Aug. 10, 1954) and 317-318 (Aug. 10, 1954) and 319-320 (Aug. 10, 1954) and 321-322 (Aug. 10, 1954) and 323-324 (Aug. 10, 1954) and 325-326 (Aug. 10, 1954) and 327-328 (Aug. 10, 1954) and 329-330 (Aug. 10, 1954) and 331-332 (Aug. 10, 1954) and 333-334 (Aug. 10, 1954) and 335-336 (Aug. 10, 1954) and 337-338 (Aug. 10, 1954) and 339-340 (Aug. 10, 1954) and 341-342 (Aug. 10, 1954) and 343-344 (Aug. 10, 1954) and 345-346 (Aug. 10, 1954) and 347-348 (Aug. 10, 1954) and 349-350 (Aug. 10, 1954) and 351-352 (Aug. 10, 1954) and 353-354 (Aug. 10, 1954) and 355-356 (Aug. 10, 1954) and 357-358 (Aug. 10, 1954) and 359-360 (Aug. 10, 1954) and 361-362 (Aug. 10, 1954) and 363-364 (Aug. 10, 1954) and 365-366 (Aug. 10, 1954) and 367-368 (Aug. 10, 1954) and 369-370 (Aug. 10, 1954) and 371-372 (Aug. 10, 1954) and 373-374 (Aug. 10, 1954) 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