Patent Owner's Demonstrative Exhibits

June 10, 2015 Case IPR2014-00606 Patent No. 6,833,404

Henkel Corporation,
Petitioner
v.
H.B. Fuller Company,

Patent Owner

FÆGRE BAKER DANIFLS

Anticipation



"Hot Melt Adhesive"



'404 Patent

"This invention relates to improved **hot melt**adhesive compositions utilizing a hydrogenated substantially aliphatic tackifying resin having a high glass transition temperature. The adhesive compositions have surprisingly high heat resistance combined with good cold temperature flexibility, light color and excellent color stability."



Claim 22

A hot melt adhesive composition comprising:

- a) about 10% by weight to about 50% by weight of at least one hydrocarbon tackifying resin derived, at least in part, from dicyclopentadiene and having a glass transition temperature of greater than about 65° C;
- b) about 10% by weight to about 80% by weight of at least one thermoplastic base polymer selected from the group consisting of copolymers and terpolymers of ethylene; amorphous polyalphaolefins, homogeneous ethylene/α-olefin interpolymers, and mixtures thereof.



Claim 28

A hot melt adhesive comprising:

- a) from about 10% by weight to about 80% by weight of at least one aliphatic tackifying resin having a glass transition temperature (T_g) of greater than about 65° C; and
- b) from about 10% by weight to about 80% by weight of at least one thermoplastic base polymer selected from the group consisting of copolymers and terpolymers of ethylene; amorphous polyalphaolefins, homogeneous ethylene/α-olefin interpolymers, and mixtures thereof.



Henkel's Petition

Inter Partes Review of U.S. Patent No. 6,833,404
Case IPR: Unassigned
Petition under 35 U.S.C. § 311

(*Id.* at ¶79 (citing '393 publication (Ex. 1013) at 15, Il. 12-16).) "Therefore, the composition disclosed in the Exxon Reference is a hot melt adhesive composition comprising: a) 40% by weight of an aliphatic tackifying resin having a glass transition temperature (Tg) of greater than about 65 °C; and b) 20% of ethylene vinyl acetate as a thermoplastic base polymer." (*Id.* at ¶80.) The Exxon Reference thus anticipates claim 28.

4. Claim Chart for Ground 2

The table below shows how each limitation of claims 22-25 and 28 is taught by the Exxon Reference.

Claim 22 of the '404 Patent	Exxon Reference
A hot melt adhesive composition comprising:	A hot melt adhesive composition comprising (Exxon Reference at 2, FN 1):
a) about 10% by weight to about 50% by weight of at least one hydrocarbon tackifying resin derived, at least in part, from dicyclopentadiene and having a glass transition temperature of greater than about 65 °C;	a) 40% Escorez® 5340 hydrocarbon tackifying resin, which is derived, at least in part, from dicyclopentadiene and has a glass transition temperature of 76 °C (Exxon Reference at 2, FN 1; '404 patent, col. 6, ll. 14-29; col. 9, ll. 6-9);
b) about 10% by weight to about 80% by weight of at least one thermoplastic base polymer selected from the group consisting of copolymers and terpolymers of ethylene; amorphous polyalphaolefins, homogenous ethylene/a-olefin interpolymer, and mixtures thereof.	b) 20% by weight of Escorene UL 7750, which is a copolymer of ethylene, as a thermoplastic base polymer (Exxon Reference at 2, FN 1; '404 patent, col. 3, ll. 6-8; col. 6, l. 61 to col. 7, l. 2, 15-16, 30-32).

36



(Paper 2 at 36; Paper 4 at 36; see also Paper 23 at 31 (citing Petition at 29-38); Paper 11 at 9.)

Henkel's Petition

 "Hot melt adhesives 'are 100[%] solid thermoplastic materials that flow when heated' and 'can bond wide possible ranges of paper stock."

(Paper 2 at 6 (quoting declaration of Dr. Hsu ¶15); Paper 4 at 6.)

 "As compared to other adhesives, hot melt adhesives 'have the distinct advantages of being capable of extremely high tack with medium to very fast setting speeds."

(Paper 2 at 6 (quoting declaration of Dr. Hsu ¶15); Paper 4 at 6.)



Dr. Hsu's Declaration

"15. Hot melt adhesives (interchangeably referred to as hot melts) 'are 100[%] solid thermoplastic materials that flow when heated; the final bond is gained when the temperature has fallen to the ambient one, i.e., that of the material to be glued.' Hot melts can bond wide possible ranges of paper stock and 'consist of polymers, tackifying resins, and waxes.' Hot melts 'have the distinct advantages of being capable of extremely high tack with medium to very fast setting speeds."



Henkel's Reply Brief

"In this case, both parties agree that a hot melt adhesive is broadly defined as a solid material that flows when heated and forms a bond when cooled to ambient temperature, and it was accepted in the art that a hot melt adhesive generally comprised a tackifier, polymer and optionally a wax."



'404 Patent & Wax

"At higher wax levels, bondability of the adhesive system may be affected. At lower levels, the adhesive may set too slowly or heat resistance may adversely be affected. In a preferred embodiment, all synthetic high melting point wax is utilized. When the thermoplastic polymer is an **ethylene copolymer**, it is preferable to use from about 10% by weight to about 30% by weight of the composition. When the thermoplastic polymer is a **homopolymer or copolymer of propylene** it may be preferable to use less wax, for example, less than about 10 wt-%, or no wax at all, as it changes the compatibility of the system."

(Ex. 1001 at 9:52-64; see also Paper 23 at 8-9, 35-36; Ex. 1039 at 240, 252.)



Dr. Hsu's Testimony Is Not Entitled to Any Weight



Dr. Hsu's Testimony Is Not Entitled To Any Weight

- 1. Dr. Hsu did not require a person of ordinary skill in the art to have experience working with hot melt adhesives.
- 2. Dr. Hsu does not have any experience working with hot melt adhesives.
- Dr. Hsu has an established relationship with Henkel.



Dr. Hsu's Testimony

Q: And in forming your opinions in this case, you did not require the person of ordinary skill – the person of ordinary skill in the art to have any experience working with hot melt adhesives. Correct?

A: That is correct.

Q: And, sir, as of today, is it fair to say that you do not have any experience working with an actual hot melt adhesive?

A: Correct.

(Ex. 1039 at 48:14-22; see also Paper 23 at 20-21.)



Dr. Hsu's Testimony

Q: So have you ever **made** a hot melt adhesive?

A: No.

Q: Have you ever **personally formulated** a hot melt adhesive?

A: No.

Q: Have you ever – at the University of Massachusetts Amherst, have you ever analyzed a hot melt adhesive? Have you **personally analyzed** a hot melt adhesive?

A: No.

Q: During the course of your career, have you ever done any testing on a hot melt adhesive?

A: No.

Q: Have you ever **done any experiments of any kind** on an actual hot melt adhesive?

A: No. (Ex. 1039 at 34:9-21, 36:1-3 (emphasis a

(Ex. 1039 at 34:9-21, 36:1-3 (emphasis added); see also Paper 23 at 21.)



No Anticipation By *Eastotac* Brochure



No Anticipation By *Eastotac* Brochure

- 1. Eastotac Brochure does not disclose a specific hot melt adhesive with H-142R resin.
- 2. Henkel's "picking, choosing, and combining" argument is improper and insufficient to prove anticipation.
- 3. Henkel's own evidence undermines its anticipation argument.



Eastotac Brochure (Ex. 1002)

Introduction

Eastotae resins are hydrogenated, C₅ aliphatic hydrocarbon tackifying resins. They are characterized by low odor, good solubility, excellent heat stability, and low color. These products also exhibit good tackifying characteristics and broad compatibility with numerous elastomers, polymers, and other resins.

Eastotac hydrocarbon resins are produced from petroleum feedstock by polymerization followed by hydrogenation. Production of the resin with the desired softening point is obtained by controlling the polymerization process. These hydrocarbon resins are essentially fully saturated. As a result, they have good color, heat stability, and aging properties that can earry through to the final product in which they are used.

Eastotac resins are available in four color levels with varying softening points which allow the selection of the resin most suitable for a particular application. Typical physical properties and color grades of these resins are listed in Table 1. Solubility characteristics are shown in Table 2 and

Eastotac resins are available in four color levels with varying softening points which allow the selection of the resin most suitable for a particular application. Typical physical

Page 3 of 8



(Ex. 1002 at 3; see also Paper 23 at 16, 24, 28.)

Eastotac Brochure: "Applications"

- pressure sensitive adhesives
- hot melt adhesives
- caulks
- sealants
- rubber systems
- inks
- paints
- varnishes
- dry-cleaning solutions
- investment casting and highway marking



Eastotac Brochure: Table 1

 $\begin{array}{c} {\rm Tal} {\scriptstyle :} \ 1 \\ \\ {\rm Typical\ Properties\ of}\ Easitac\ Hydrocarbon\ Resins*} \end{array}$

PROPERTY	H-100E	H-100R	H-100L	H-100W	H-115E	H-115R	H-115L	H-115W	H-130E	H-130R	H-130L	H-130W	H-142R
Ring & Ball Softening Point, °C (°F)	100 (212)	100 (212)	100 (212)	100 (212)	115 (239)	115	115	115	130	130	130	130	142
Physical Form	Flake												
Gardner Color (Molten)	8	4	2	<1	8	4	2	<1	8	4	2	<1	4
Gardner Color (in 50% Toluene)	6	2	<1	<1	6	2	<1	<1	6	2	<1	<1	2
Acid Number	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Density	1.04	1.04	1.04	1.04	1.04	1.04	1.04	1.04	1.04	1.04	1.04	1.04	1.04
Viscosity, Brookfield Thermosel, 190°C (374°F), cP	200	200	200	200	400	400	400	400	1200	1200	1200	1200	3000
Bromine Number	15	5	3	1	15	5	3	1	15	5	3	1	5
Chlorides, total, ppm	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Ash, %	<0.002	<0.002	< 0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.001
Flash Point, Cleveland Open Cup, °C (°F)	242 (468)	242 (468)	242 (468)	242 (468)	257 (495)	257 (495)	257 (495)	257 (495)	299 (570)	299 (570)	299 (570)	299 (570)	321 (610)
Fire Point, Cleveland Open Cup, °C (°F)	266 (510)	266 (510)	266 (510)	266 (510)	283 (542)	283 (542)	283 (542)	283 (542)	325 (617)	325 (617)	325 (617)	325 (617)	335 (635)

^{*}Reported for information only. Eastman makes no representation that the material in any particular shipment will conform exactly to the values given.



Eastotac Brochure: Table 3

Table 3
General Composition Ranges for *Eastotac* Resins in Hot Melt Adhesives

	Cor	mpositions, % by Wei	ght	
Materials	PE Base	APP Base	EVA Base	
Eastotac resin	10-40	30-50	30-60	
Low-molecular-weight polyethylene wax	60-90	0-20	_	
Amorphous polyolefin	-	50-70	. –	
Ethylene-vinyl acetate copolymer			30-50	
Petroleum wax or Fischer-Tropsch wax	-		0-20	
Antioxidant	0.25-0.5	0.25-0.5	0.25-0.5	



(Ex. 1002 at Table 3; see also Paper 23 at 24-29.)

The '033 Patent (Ex. 1019)

United States Patent [19]

Stauffer et al.

[75] Inventors: Daniel Stauffer, Kane, Pa.; Paul P.

Puletti, Pittstown, N.J.; Thomas F. Kauffman, Easton, Pa.

[73] Assignee: National Starch and Chemical Investment Holding Corporation, Wilmington, Del.

[21] Appl. No.: 21,148

[22] Filed: Feb. 23, 1993

[54] HOT MELT ADHESIVE

C08L 91/06 [52] U.S. Cl. 524/275; 524/277;

References Cited

U.S. PATENT DOCUMENTS

3,884,786	5/1975	Domine et al	204/159.14
4,146,521	3/1979	Godfrey	260/27 R
4,167,433	9/1979	Lakshmanan	156/322
4,404,299	9/1983	Decroix	524/77
4,434,261	2/1984	Brugel et al	524/109
4,460,728	7/1984	Schmidt et al	
4.471.086	9/1984	Foster	
4.500.661	2/1985	Lakshamanan	
4.567.223	1/1986	Ames	
4.631.308	12/1986	Graham et al	
4,752,634	6/1988	Goss	
4,816,306			

5,331,033 [11] Patent Number:

[45] Date of Patent: Jul. 19, 19

4,874,804 10/1989 Brady et al. 524/ OTHER PUBLICATIONS

"Ethylene-n-butyl Acrylate Resins for Adhesive F mulating", Preliminary Information Report, U.

"Hot melts in recycled packaging", E. M. Wise, Ed rial, Tappi Journal, Sep. 1990.

Primary Examiner-Paul R. Michl Assistant Examiner—LaVonda R. DeWitt Attorney, Agent, or Firm—Ellen T. Dec

Hot melt packaging adhesive compositions consist essentially of 20 to 50% by weight of an ethylene butyl acrylate copolymer containing 25-45% weight, preferably 30 to 40% n-butyl acrylate and h ing a melt index of at least 10; 30 to 60% of an aliph or cycloaliphatic hydrocarbon resin or hydrogena derivative thereof or a hydrogenated aromatic hydrogenated carbon resin; and 10 to 30% by weight of a high mel synthetic wax provide adhesives characterized by excellent balance of high and low temperature pe mance without sacrifice to machinability or the stability. Further, the adhesive is characterized by a density and consequently is readily filterable in recycling systems.

18 Claims, No Drawings

HOT MELT ADHESIVE

Filed:

Feb. 23, 1993

National Starch and Chemical Investment Holding Corporation,

Page 1 of 6

HENKEL Exhibit 1019



(See Paper 23 at 29-30.)

The '033 Patent (Ex. 1019)

5,331,033

Jul. 19, 1994

United States Patent [19]

Stauffer et al.

4,874,804 10/1989 Brady et al. 524/1

[54] HOT MELT ADHESIVE
[75] Inventors: Daniel Stauffer, Kane, Pa.; Paul P. Puletti, Pittstown, N.J.; Thomas F. Kauffman, Easton, Pa.

 Assignee: National Starch and Chemical Investment Holding Corporation, Wilmington, Del.
 Appl. No: 21,148

[22] Filed: Feb. 23, 1993
[51] Int. Cl.: COBL 91/06
[52] U.S. Cl. 524/275; 524/277;
524/481; 524/485; 524/487; 524/489
[58] Field of Search 524/270, 272, 275, 277,

[56] References Cited

O.S. TATEM DOCUMENTS						
	3,884,786	5/1975	Domine et al 204/159.			
	4,146,521	3/1979	Godfrey 260/27			
	4,167,433	9/1979	Lakshmanan 156/32			
	4,404,299	9/1983	Decroix 524/7			
	4,434,261	2/1984	Brugel et al 524/10			
	4,460,728	7/1984	Schmidt et al 524/48			
	4,471,086	9/1984	Foster 524/48			
	4,500,661	2/1985	Lakshamanan 524/			
	4,567,223	1/1986	Ames			
	4,631,308	12/1986	Graham et al 524/2"			
	4.752.634		Goss 524/2			
	4,816,306	3/1989	Brady et al 428/36.			

OTHER PUBLICATIONS
"Ethylene-n-butyl Acrylate Resins for Adhesive Formulating", Preliminary Information Report, U.S.I. Chemicals.

Chemicals.

"Hot melts in recycled packaging", E. M. Wise, Editorial, Tappi Journal, Sep. 1990.

Primary Examiner—Paul R. Michl Assistant Examiner—LaVonda R. DeWitt Attorney, Agent, or Firm—Ellen T. Dec

] ABSTRACT

[45] Date of Patent:

Hot melt packaging adhesive compositions consisting essentially of 20 to 50% by weight of an ethylene n-butyl acrylate copolymer containing 22-45% by weight, preferably 30 to 40% n-butyl acrylate and having a melt index of at least 10, 30 to 60% of an aliphatic or cycloaliphatic hydrocarbon resin or hydrogenated derivative thereof or a hydrogenated aromatic hydrocarbon resin and 10 to 30% by weight of a high melting synthetic wax provide adhesives characterized by an excellent balance of high and low temperature performance without sacrifice to machinability or thermal stability. Further, the adhesive is characterized by a low density and consequently is readily filterable in recycling systems.

18 Claims, No Drawings

Page 1 of 6

HENKEL Exhibit 1019

FAEGRE BAKER DANIELS

Dr. Hsu's Testimony

"For example, U.S. Patent 5,331,033 (issued Jul. 19, 1994) ('the '033 patent') (Ex. 1019) is directed to **high heat** resistant hot melt adhesives."

(Ex. 1015 ¶ 23 (emphasis added); see also Paper 23 at 29-30.)

The '033 Patent (Ex. 1019)

5,331,033

an aliphatic, aromatic hydrocarbon resin from Goodyear Chemicals; Escorez 5300, a partially hydrogenated cycloaliphatic petroleum hydrocarbon resin from Exxon Chemical Company; and Eastman Resin H 100 and/or H 130, partially hydrogenated cycloaliphatic 5 petroleum hydrocarbon resins. These resins are utilized in amounts of 30-60% by weight of the adhesive com-

position.

The wax used herein is a high melting (above about 100° C.) synthetic wax such as synthetic, high density, 10 which use ethylene vinyl acetate as the copolymer pro-low molecular weight polyethylene waxes or "Fischer-duce a cloudy, poor flowing, thermally less stable prod-Tropsch" wax. Suitable waxes include Bareco C-4040. a low molecular weight polyethylene wax from Petrolite, Escomer H-101 a modified polyethylene from Exxon Chemical and Polywax 1000, or 2000, and Marcus 300 15 low molecular weight polyethylene waxes from Petrolite and Marcus Chemical respectively. Paraflint H-1, a Sasol-SA from Moore & Munger is preferred. The wax component is utilized at levels of 10 to 30% by weight

The adhesives of the invention preferably also contain a stabilizer or antioxidant. Among the applicable stabilizers or antioxidants included herein are high molecular weight hinderedphenols and multifunctional phenols such as sulfur and phosphorous-containing phe- 25 nol. Hindered phenols are well known to those skilled in the art and may be characterized as phenolic compounds which also contain sterically bulky radicals in

close proximity to t In particular, tertia tuted onto the benz positions relative t resence of these s the vicinity of the stretching frequency compound with its (3.5-di-tert-butyl-4-

propionate: n-octagecyi-3(3,3-qi-teri-butyi-4-nyqroxyhenyl)-propionate;4,4'-methylenebis(2,6-tert-butvlphenol);4, 4'-thiobis(6-tert-butyl-o-cresol); 2,6-di-tertbutylphenol: 6-(4-hydroxyphenoxy)-2,4-bis(n-octyl-thihydroxy-benzoate; and sorbitol hexa[3-(3,5-di-tertbutyl-4-hydroxyphenyl)-propionate].

The performance of these antioxidants may be further enhanced by utilizing, in conjunction therewith known synergists such, for example, as thiodipropionate esters 50 and phosphites, particularly useful is distearylthiodipro-pionate. These stabilizers, if used, are generally present in amounts of about 0.1 to 1.5 weight percent, preferaadhesives for various end uses contemplated may also be incorporated in minor amounts into the formulations of the present invention.

The adhesive compositions are prepared by blending the components in the melt at a temperature of about 60 (NFT) were noted. 130°-200° C. until a homogeneous blend is obtained, approximately 2 hours. Various methods of blending are known to the art and any method that produces a homogeneous blend is satisfactory.

ity of less than 8000 cps at 175° C., fiber tearing bonds from Kraft paper within the complete temperature range of -18° C. to +70° C., a Ring and Ball softening

point of 100°-130° C., a cloud point less than 140° C. and clarity at 175° C. Moreover, the adhesives possess excellent heat stability as characterized by the 144 hour 350° F. thermal stability test, with no signs of char, skinning or gel formation, and a density of less than about 0.98. As such the adhesives find particular use as packaging adhesives, for example, for sealing cases and cartons which are to be exposed to extreme temperature conditions. In contrast, similar adhesive compositions uct not suitable for high speed, rapid-fire hot melt appli-

EXAMPLES

In the following examples which are provided for illustrative purposes only, all parts are by weight and all temperatures in degrees Celsius unless otherwise noted. In the examples, all adhesive formulations were prered in single blade mixer heated to 170° C. by blend-

ing the components until homogeneous. The adhesives were then subjected to various tests simulating the properties needed for successful com-

mercial applications.

Melt viscosities of the hot melt adhesives were determined on a Brookfield Model RVT Thermosel viscom-

eter using a number 27 spindle. Test specimens for determining elevated temperature

Eastman Resin H 100 and/or H 130

given temperature for 15 minutes for conditioning. The heating cycle was run until the final bond failed. Each neel and shear specimen was prepared and tested in o)-1,3,5 triazine; di-n-octylthio)ehtyl 3,5-di-tert-butyl-4- 45 duplicate. The elevated peel and shear value shown is the average temperature of failure for the two bonds

Adhesion at 71° C., room temperature (22° C.), 4.4° C., -6° C., -17.8° C. and -28.0° C., where applicable, was determined by applying a ½ inch wide bead of adhesive at 175° C. widthwise to a 2 inch by 3 inch piece of corrugated board and immediately bringing a second piece of board with flutes in the opposite direction into contact. A 200 gram weight was immediately placed on bly 0.25 to 1.0%. Other additives such as plasticizers, pigments, dyestuffs conventionally added to hot melt 55 strength corrugated board. The bond specimens were placed in an oven at 71° C. and freezers at 4.4° C., -6.7° C., -17.8° C., and -28.9° C. The bonds were separated , -17.8° C., and -28.9° C. The bonds were separated by hand and a determination made as to the type of failure. Fiber tearing (FT) and non-fiber tearing bonds

The adhesive set time (open time) was determined in the following manner using 50 pound Kraft paper and an adhesive tester that simulates a case sealing line nomogeneous blend is satisfactory.

The resulting adhesives are characterized by viscos
for the grips of the tester. The bottom specimen was rward at a constant speed under the melt nozzle applicator for the application of the adhesive beau and was stopped directly under the top specimen. The



(Ex. 1019 at 3:1-8; see also Paper 23 at 29-30.)

No Anticipation By Exxon Reference

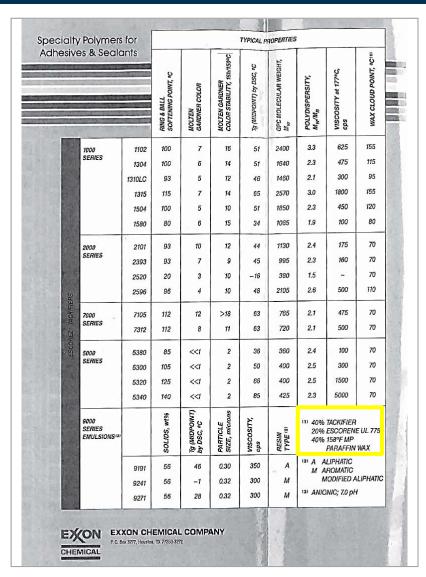


No Anticipation By Exxon Reference

- 1. Exxon reference does not suggest footnote 1 composition is a "hot melt adhesive."
- 2. Henkel offers no evidence showing footnote 1 composition is a "hot melt adhesive."
- 3. 40% wax in footnote 1 composition is inconsistent with teachings of '404 patent.



Exxon Brochure (Ex. 1003)



(1) 40% TACKIFIER
20% ESCORENE UL 7750
40% 158°F MP
PARAFFIN WAX



(Ex. 1003 at 2; see also Paper 23 at 17, 30-38.)

'404 Patent & Wax

"At higher wax levels, bondability of the adhesive system may be affected. At lower levels, the adhesive may set too slowly or heat resistance may adversely be affected. In a preferred embodiment, all synthetic high melting point wax is utilized. When the thermoplastic polymer is an **ethylene copolymer**, it is preferable to use from **about 10% by** weight to about 30% by weight of the composition. When the thermoplastic polymer is a homopolymer or copolymer of propylene it may be preferable to use less wax, for example, less than about 10 wt-%, or no wax at all, as it changes the compatibility of the system."

(Ex. 1001 at 9:52-64; see also Paper 23 at 8-9, 35-36; Ex. 1039 at 240, 252.)



Dr. Hsu's Testimony

Q: So in forming your opinions in this case, you never made a composition that had 40 percent of Escorez 5340, 20 percent Escorene UL 7750, and 40 percent 158 degree F MP paraffin wax. Correct?

A: Correct.

(Ex. 1039 at 220:16-20; see also Paper 23 at 33, 37.)

Q: And in forming your opinions in this case, you didn't do any tests of any kind on the Footnote 1 composition for 5340?

A: That is correct.

(Ex. 1039 at 221:7-10; see also Paper 23 at 33, 37.)

Q: And you did not do any type of testing to see how the bondability of this Footnote 1 composition for 5340 in the Exxon reference, Exhibit 1003, was affected by having 40 percent of the paraffin wax in it. Correct?

A: Correct.

(Ex. 1039 at 255:14-19; see also Paper 23 at 37.)

Q: And similarly, you didn't do any type of experimentation in forming your opinions to show how the adhesive properties of the Footnote 1 composition for 5340 in the Exxon reference would be affected by changing the wax concentration. Correct?

A: Correct.

FAEGRE BAKER

(Ex. 1039 at 256:16-21; see also Paper 23 at 37.)