GREEN

Safety Assessment of Modified Terephthalate Polymers as Used in Cosmetics

CIR EXPERT PANEL MEETING

DECEMBER 10-11, 2012

Cosmetic Ingredient Review
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November 16, 2012

MEMORANDUM

To: CIR Expert Panel and Liaisons

From: Lillian C. Becker, M.S.
Scientific Analyst and Writer

Subject: Draft Report for Modified Terephthalate Polymers as used in cosmetics

The Cosmetic Ingredient Review (CIR) announced the Scientific Literature Review (SLR) for modified terephthalate polymers in June, 2012. Comments and unpublished data have been received from industry and incorporated into the report.

We are using the same evaluation process that was employed for PMMA, i.e., relying on the extensive evaluation of the FDA's Center for Devices and Radiological Health of these polymers as used in medical implants. FDA has approved the use of terephthalate polymers in surgical sutures, esophageal dilators, and surgical mesh.

It was brought to our attention that there is concern about glitter made of modified terephthalate polymers causing eye damage. The glitter is created by the chopping of sheets of the polymers is a manner that leaves jagged edges that will adhere to skin and hair better. If these become imbedded in the eye, the jagged edges make removal by a doctor necessary to avoid further injury. A literature search only revealed one paper on the subject in Japanese that could not be retrieved. A search of the internet found several eye makeup products that contain glitter as well as products that are containers of loose glitter that are meant to be used on the eye lids as well as elsewhere on the body. I interviewed Dr. Stephen Glasser, an ophthalmologist who has experience with this problem. The notes from this interview as well as a copy of the email are included in the data packet.

The Panel should review the Draft Report and decide whether any additional data are needed in order to reach a safety conclusion for modified terephthalate polymers. If there are additional data needed, then an insufficient data announcement should be issued. If no additional data are required, then the Panel should read a tentative conclusion, with the discussion forming the basis for the tentative report discussion.

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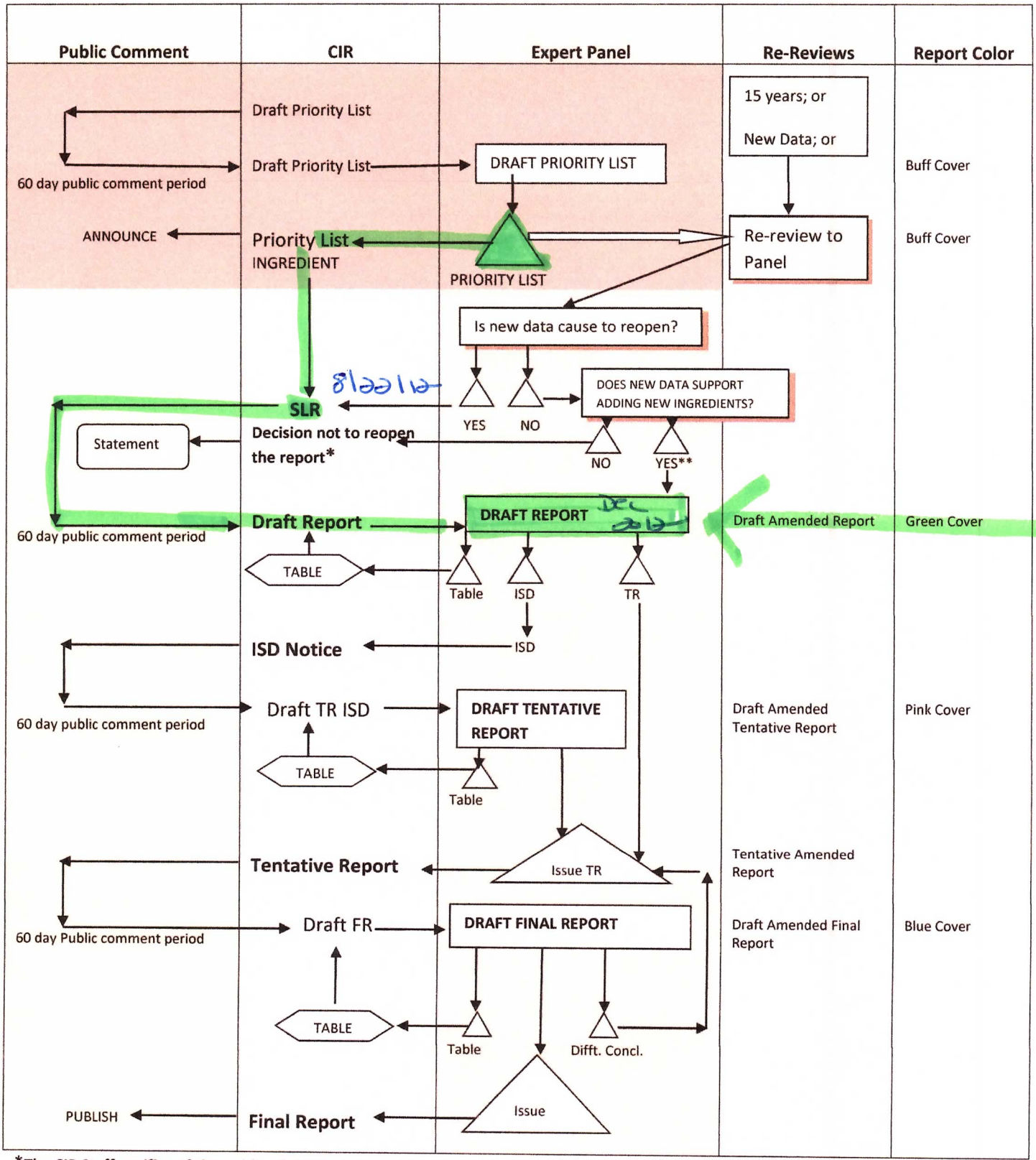
Modified Terephthalate Polymers Dec 2012

SAFETY ASSESSMENT FLOW CHART



*The CIR Staff notifies of the public of the decision not to re-open the report and prepares a draft statement for review by the Panel. After Panel review, the statement is issued to the Public.

**If Draft Amended Report (DAR) is available, the Panel may choose to review; if not, CIR staff prepares DAR for Panel Review.

△ Expert Panel Decision

▭ Document for Panel Review

⇨ Option for Re-review

History of
Modified Terephthalate Polymers

June, 2012 – SLR announced.

December, 2012 – Panel reviews Draft Report for first time.

Modified Terephthalate Polymers ingredients Data Profile for December, 2012. Writer - Lillian Becker																		
	ADME			Acute toxicity			Repeated dose toxicity			Irritation			Sensitization					
	Dermal Penetration	Log K_{ow}	Use	Oral	Dermal	Inhale	Oral	Dermal	Inhale	Ocular Irritation	Dermal Irr. Animal	Dermal Irr Human	Sensitization Animal	Sensitization Human	Repro/Develtoxicity	Genotoxicity	Carcinogenicity	Phototoxicity
Adipic acid/1,4-butanediol/ terephthalate copolymer																		
Ethylene/ sodium sulfoiso-phthalate/ terephthalate copolymer																		
Polybutylene terephthalate			X															
Polyethylene isoterephthalate			X															
Polyethylene terephthalate (PET)			X							X			X			X		
Polypenta-erythrityl terephthalate																		
Polypropy-lene terephthalate			X															

Search Strategy for Modified Terephthalate Polymers

SciFinder Search Terms: Substance search; CAS Nos.; Name combinations of the polymers.

When multiple patents for medical devices were in the above results, the FDA Medical Devices was searched for PET/ polyethylene terephthalate and terephthalate.

Report

Safety Assessment of Modified Terephthalate Polymers as Used in Cosmetics

Status: Draft Report for Panel Review
Release Date: November 16, 2012
Panel Meeting Date: December 10-11, 2012

The 2012 Cosmetic Ingredient Review Expert Panel members are: Chairman, Wilma F. Bergfeld, M.D., F.A.C.P.; Donald V. Belsito, M.D.; Curtis D. Klaassen, Ph.D.; Daniel C. Liebler, Ph.D.; Ronald A Hill, Ph.D. James G. Marks, Jr., M.D.; Ronald C. Shank, Ph.D.; Thomas J. Slaga, Ph.D.; and Paul W. Snyder, D.V.M., Ph.D. The CIR Director is F. Alan Andersen, Ph.D. This report was prepared by Lillian C. Becker, Scientific Analyst/Writer.

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INTRODUCTION

This is a draft safety asessment of modified terephthalate polymers as used in cosmetics. The seven ingredients in for review in this safety assessment mostly function as exfoliants, bulking agents, hair fixatives, and viscosity increasing agents-nonaqueous (Table 1). These ingredients are:

- Adipic acid/1,4-butanediol/terephthalate copolymer,
- Ethylene/sodium sulfoisophthalate/terephthalate copolymer,
- Polybutylene terephthalate,
- Polyethylene isoterephthalate,
- Polyethylene terephthalate (PET),
- Polypentaerythrityl terephthalate,
- Polypropylene terephthalate.

CIR believes that the modified terephthalate polymers produced for cosmetics are analogous to the polymers used in surgical sutures and other commercial medical devices made of terephthalate polymers. The safety information for those medical devices has been provided to the Food and Drug Administration (FDA) in medical device applications. The FDA has found those data to be adequate and determined that terephthalate polymers are safe for use in devices when used in soft tissue approximation and/or ligation, including cardiovascular, ophthalmic, and neurological tissue. Systemic exposures of terephthalate polymers from devices used in these settings far exceed that expected for modified terephthalate polymer use in cosmetics. The approach of using FDA decisions on the safety of chemicals contained in medical devices but that are also used in cosmetics was employed in the Panel's assessment of polymethyl methacrylate.

The CIR considers that the assessment of modified terephthalate polymer safety as used in medical devices by the FDA provides the basis to establish the safety of these polymers in cosmetics because the polyethylene terephthalate is substantially the same as that used in approved medical devices and is used in a manner that presents less exposure risk. The CIR also contends that given the chemical similarity of the modified terephthalate polymer used in cosmetics to the same ingredient used in medical devices, data previously submitted to the FDA on that ingredient could be extrapolated to support the safety of adipic acid/ 1,4-butanediol/ terephthalate copolymer, ethylene/sodium sulfoisophthalate/ terephthalate copolymer, polybutylene terephthalate, polyethylene isoterephthalate, polyethylene terephthalate, polypentaerythrityl terephthalate, and polypropylene terephthalate.

The literature does contain information on impurities and possible estrogenic activity related to terephthalate polymers. Whether or not these issues apply to the modified terephthalates in this safety assessment will need to be resolved.

One issue has been raised that appears unique to cosmetic uses of these polymers. Apparently, there is a concern that glitter made of modified terephthalate polymers may cause eye damage. The glitter is created by the chopping of sheets of the polymers in a manner that leaves jagged edges that will adhere to skin and hair better. If these become imbedded in the eye, irritation, etc. may occur and the jagged edges make removal by a doctor necessary to avoid further injury. A literature search only revealed one paper on the subject in Japanese that could not be retrieved. A search of the internet found several eye makeup products that contain glitter as well as products that are containers of loose glitter that are meant to be used on the eye lids as well as elsewhere on the body.

CHEMISTRY

Overview of Chemistry and Manufacture

The modified terephthalate polymer ingredients are related to polyesters, wherein terephthalic acid (or an ester thereof) is a primary monomeric repeat unit. Terephthalic acid is an aromatic, dicarboxylic acid, which does not readily form a homopolymer, but easily copolymerizes with polyols (i.e., multifunctional alcohols). The ingredients in this review are formed from diols (i.e., two alcohol functional groups per molecule), with the exception of polypentaerythrityl terephthalate which is prepared from a tetrol (i.e., four alcohol functional groups per molecule; pentaerythritol). Accordingly, with the exception of polypentaerythrityl terephthalate, these polymers are essentially linear. Polymerization of these polymers proceeds first through the esterification of terephthalic acid (or transesterification of a simple terephthtalate ester, such as dimethyl terephthalate), with a diol (Figure 1).[1,2] This results in a new, di-functional monomeric unit with alcohols at the both ends (e.g., bis(hydroxyethyl)terephthalate). The exception, again, is with polypentaerythrityl terephthalate wherein the synthesis results in a mixture of tetrafunctional monomers. These new monomers then undergo polycondensation to produce the modified terephthalate polymer ingredients. Idealized structures of the modified terephthalate polymer ingredients in this safety assessment are provided in Figure 2.

When terephthalic acid is used as the starting material, water is released from the initial condensation reactions.[1,2] When a simple terephthalate ester is used (e.g., dimethyl terephthalate), the corresponding alcohol (e.g., methanol) is the byproduct. Early manufacturing methods of modified terephthalate polymers proceeded primarily from terephthalate esters, because the ester was easier to purify. However, since the mid-1960's when significant progress was made in high-yielding,

high purity acid syntheses, terephthalic acid has become the primary starting material for these polymers, because of the lack of alcohol (e.g., methanol) produced.

The polycondensation step, however, is essentially independent of whether an acid or ester was used to synthesize the ester intermediate.[1,2] The polycondensation step proceeds via a metal oxide (e.g., antimony(III) glycolate) catalyzed transesterification and results in the release of some of the starting material alcohol, and dimers thereof (some of which may be incorporated into the backbone of the polymer).

Depending on processing methodologies (e.g., product cooling rates), most of these ingredients can range from an amorphous glass to having a high degree of crystallinity.[3]

It is common for manufacturers to market copolymers for purposes previously filled by homopolymer PET. Copolymer blends, such as polybutylene terephthalate/PET, have certain advantages over homopolymer PET with regard to mechanical properties and resistance to degradation.[4,5] In the United States, clear plastic bottles made with copolymers may still be legally marketed as PET (21CFR177.1630).

The size and shape of PET particles are defined by precision cutting PET sheets and there is not a typical size distribution. One reported particle size is 0.004" (0.1016 mm).[6] The shapes of these particles may be hexagonal or square.

Physical and Chemical Properties

The limited physical and chemical properties discovered for PET are in Table 2. There were no data discovered for the rest of the ingredients in this safety assessment.

Impurities

The available data on impurities of PET are from studies using bottles and food containers made up of PET and PET copolymers. A representative sample is provided in Table 3. Under different experimental conditions, ethylene glycol and other monomers/processing aids have been detected. In most cases, the amount of impurities detected was greatest in cases of exposures shortly after manufacture and the level decreased with time. Whether the impurities broke down or were reabsorbed was not addressed. Heat increases the amount of antimony (catalyst) that leaches into the contents of bottles and food packages. In all cases, the amount is small.[7-15]

Phthalates were also detected in PET-based containers and estrogenic activity was demonstrated in substances stored in these containers.[15-23] Representative studies are presented in Table 4. However, Enneking stated that "It is important to note that modified terephthalate copolymers do not contain phthalates nor leach them with use. PET is not considered an orthophthalate, nor does PET require the use of phthalates or other softening additives."[24]

It has been noted that not all PET materials are of the same chemical quality.[21] Therefore, it is important to confirm that the PET-related ingredients in this safety assessment are analogous to those used in medical devices and that there are no concerns about impurities and estrogenic activity.

An analysis of the impurities of a sample of PET glitter was showed the following: arsenic, < 0.05; antimony, 169.0; lead, 2.0; cadmium, <0.5; mercury, <0.1; nickel, <1; and chromium, <1 mg/kg.[25] The analysis of the migration of heavy metals showed that arsenic, antimony, lead, cadmium, mercury, chromium, barium, and selenium were below the levels of detection. Free formaldehyde was below the limits of detection.

Diisononylphthalate, diethethylhexylphthalate, dibutylphthalate, diisodecylphthalate, di-n-octylphthalate, butylbenzylphthalate, diisobutylphthalte, dimethylphthalte, diethylphthaltate, dibutylsebacate, di(2-ethylthexyl)adipate, and tributylacetaylcitrate were below the levels of detection for this sample.[25] The same was found for nonylphenoles, organo tin compounds, organic solvents, primary aromatic amines, polycyclic aromatic hydrocarbons, and monomeric plasticisers. The color was fast in the presence of perspiration and saliva.

USE

Cosmetic

Data on ingredient usage are provided to the Food and Drug Administration (FDA) Voluntary Cosmetic Registration Program (VCRP; Table 5).[26] A survey is being conducted by the Personal Care Products Council (Council) of the maximum use concentrations for ingredients in this group.

Polyethylene terephthalate was reported to be used in 394 leave-on products (173 lipsticks and 98 used in the eye area), 1 rinse-off product, and 1 diluted for bath. Polybutylene terephthalate was reported to be used in 21 leave-on products and 2 rinse-off products. Polyethylene isoterephthalate was reported to be used in two lipsticks. Polypropylene terephthalate was reported to be used in 13 leave-on products and 10 rinse-off products (7 in bath soaps and detergents).

There were no reported uses for: adipic acid/1,4-butanediol/terephthalate copolymer, ethylene/sodium sulfoisophthalate/terephthalate copolymer, polypentaerythrityl terephthalate, and polypropylene terephthalate.

As noted above in the Introduction, these polymers may be used in cosmetic formulations as glitter.

Non-Cosmetic

PET is used for high-impact resistant containers.[27] It is used for packaging of soda, mouthwash, pourable dressings, edible oils, and peanut butter. It is used for cereal box liners, soda bottles, boil-in-the-bag pouches and microwave food trays. Modified PETs can be heated in a microwave or in a conventional oven at 180°C for 30 minutes.

Regulation

Regulations regarding the ingredients in this safety assessment are provided in Table 6. These regulations allow for contact with food substances.

With regard to phthalate contamination in water, the US Environmental Protection Agency set the maximum contaminant level goal of di(2-ethylhexyl) phthalate to be zero.[28] The aceptable maximum contaminant level is 0.006 mg/L.

PET IN MEDICAL DEVICES

The FDA considered the safety of polyethylene terephthalate when approving the following medical devices that include this material:

- Surgical sutures (i.e., PremiCron® Nonabsorbable PET Surgical Suture, TRUBOND® Nonabsorbable Surgical Suture, U.S.P[29,30]),
- Esophageal dialators (i.e., Bard® ELIMINATOR® PET Balloon Dialator[31]), and
- Surgical mesh (i.e., Peri-Strips® Staple Line Reinforcement[32]).

The use of polyethylene terephthalate to make surgical sutures was approved by the FDA as a Class II (special controls) medical device that requires premarket notification and adherence to standards (21CFR878.5000). Required testing includes acute and long-term (>30 day) biocompatibility testing for cytotoxicity, irritation or intracutaneous reactivity, sensitization, systemic toxicity, implantation effects, and hemocompatibility.[33] The sutures may be provided uncoated, coated, undyed, or dyed with appropriate approved colors. The FDA found the data on the safety of PET to be adequate and determined that medical devices containing PETs are safe and effective when used for soft tissue approximation and/or ligation, including cardiovascular, ophthalmic, and neurological tissue.[34]

Esophageal and gastrointestinal dialators are Class II medical devices (21CFR876.5365) that must adhere to the same standards listed above for the surgical sutures. An esophageal dilator, used to dilate a stricture of the esophagus, may consist of a hollow cylindrical instrument (bougie), a weighted bougie with a mercury or metal olive-shaped weight that slides on a guide, such as a string or wire, or may consist of a bougie with a deflated ballon attached to a guidewire. The balloon is made of polyethylene terephthalate.

Patches, pledgets, and intracardiac devices (surgical mesh) are made of polypropylene, polyethylene terephthalate, or polytetrafluoroethylene. They are fabric devices placed in the heart to repair septal defects, for patch grafting, to repair tissue, and to buttress sutures (21CFR870.3470). These devices are also Class II devices and adhere to the same standards listed above for the surgical sutures.

IRRITATION AND SENSITIZATION

Ocular Irritation

In a use test of an eye shadow containing PET (46.272%; cut into flakes) was found to be nonirritating.[35] Subjects (n = 15), who were considered to have sensitive eyes, applied the test material to the upper eye lid once or twice daily for 8 days. The subjects were examined before and after the test period and at 10 min after the first and last application. There was one reported adverse event of palpebral stinging/burning of short duration (6 min). The authors stated that there is a very slight ocular irritant potential, which is normal for this type of product.

In a use test of three mascaras containing PET (4.2% or 4.8%) were found to not have the potential to be irritating or sensitizing.[36] The subjects (n = 29) applied the test material at least once/day for 4 weeks.

Sensitization

In a repeated insult patch test of an eyeliner containing PET (1.5%), there were no signs of irritation nor sensitization.[37] The test material (0.2 g) were administered to the backs of subjects (n = 107) under occlusion three times/week for three weeks. After a 2-week rest, the test material was applied to a naïve sight.

GENOTOXICITY

In Vitro

POLYETHYLENE TEREPHTHALATE LEACHATE

In several tests of water stored in PET for up to 6 months, the water was not mutagenic to *Salmonella* (strains TA98, and TA100) with or without metabolic activation except for one test where the water was mutagenic after storage for 1 month but not at 3 and 6 months (Table 7).[8,14]

CLINICAL USE

POLYETHYLENE TEREPHTHALATE

PET sutures were used in two studies of penetrating keratoplasty surgery (n = 20 and 45) comparing PET and nylon sutures and style of suture using these materials.[38] All complications were mechanical or technique related and not toxicological.

PET sutures were used in penetrating keratoplasty for keratoconus surgery (n = 14).[39] The subjects were followed for 22 – 48 months. There was no vascularization of the stitches, cheese-wiring, or graft rejections. There were four cases of stitch removal, three for mechanical reasons and one for an ulceration (which resolved when the stitch was removed). The author concluded that the problems had to do with technique and not toxicity.

In an evaluation of the use of a mesh made of PET for use in brow suspension ptosis surgery, a survey of five case reports and other cases in the literature were reviewed.[40] There were reported cases of extrusion and granuloma formation one month to one year after implantation characterized as foreign body reactions. There were also cases followed for up to 45 months with no complications. The authors concluded that technique (for example, knot size) and placement were the reasons for the problems and not toxicity.

SUMMARY

This is a draft report of modified terephthalate polymers as used in cosmetics. The seven ingredients in this safety assessment mostly function as exfoliants, bulking agents, hair fixatives, and viscosity increasing agents-nonaqueous.

CIR believes that the modified terephthalate polymers produced for cosmetics is analogous to the polymers in surgical sutures and other commercial medical devices made of terephthalate polymers. The safety information for those medical devices was provided to the FDA in medical device applications including: acute and long-term biocompatibility testing for cytotoxicity, irritation or intracutaneous reactivity, sensitization, systemic toxicity, implantation effects, and hemocompatibility. The FDA found those data to be adequate and determined that PETs were safe and effective for use in surgical sutures, esophageal dilators and surgical mesh.

One issue has been raised that appears unique to cosmetics uses of these polymers. Gliter made of modified terephthalate polymers is used in eye cosmetics and may cause eye irritation, etc. because of the jagged edges of the glitter material.

The available data on impurities of PET are from studies using bottles and food containers made up of PET and PET copolymers. Under different experimental conditions, ethylene glycol and other monomers/processing aids have been detected. In most cases, the amount of impurities detected was greatest in short time exposures and decreased with time, but at all times were low. In some studies, phthalates were also detected in PET containers as well as estrogenic activity demonstrated in substances stored in these containers.

Polyethylene terephthalate was reported to be used in 396 cosmetic products, polybutylene terephthalate in 23 products, polyethylene isoterephthalate in 2 products, and polypropylene terephthalate in 23 products. There were no reported uses for: adipic acid/1,4-butanediol/terephthalate copolymer, ethylene/sodium sulfoisophthalate/terephthalate copolymer, polypentaerythrityl terephthalate, and polypropylene terephthalate.

PET is safe for use in food packaging that may be stored, heated, or microwaved.

In use tests of two different eye products containing PET p to 46.272%, the products were found to be non-irritating and non-sensitizing.

An eyeliner containing PET (1.5%) was not irritating or sensitizing in a repeated insult patch test.

In several tests of water stored in PET, the water was not mutagenic to *Salmonella* except for one test where the water was mutagenic after storage for 1 month but not at 3 and 6 months.

Complications from the use of PET sutures were attributed to mechanical or technique issues and not toxicological issues.

TABLES AND FIGURES

Table 1. Definitions and functions of the ingredients in this safety assessment.[41]
(The italicized text below represents additions made by CIR staff.)

Ingredient CAS No.	Definition	Function
Adipic Acid/ 1,4-Butanediol/ Terephthalate Copolymer 55231-08-8	Adipic Acid/1,4-Butanediol/Terephthalate Copolymer is a copolymer of Adipic Acid, 1,4-Butanediol and dimethyl terephthalate monomers.	Exfoliant
Ethylene/Sodium Sulfoisophthalate/ Terephthalate Copolymer	Ethylene/Sodium Sulfoisophthalate/Terephthalate Copolymer is a copolymer of Glycol, sodium dimethyl sulfoisophthalate and dimethyl terephthalate.	Bulking agent
Polybutylene Terephthalate 24968-12-5 26062-94-2	Polybutylene Terephthalate is the polymer that *is as shown in the structure in Figure 2. Polybutylene Terephthalate is a copolymer of 1,4-butanediol and dimethyl terephthalate or terephthalic acid.*	Film former; hair fixative; viscosity increasing agent-nonaqueous
Polyethylene Isoterephthalate	Polyethylene Isoterephthalate is the polymer that *is as shown in the structure in Figure 2. Polyethylene Terephthalate is a copolymer of ethylene glycol and one or more dimethyl terephthalates or terephthalic acids (i.e. not exclusive to 1,4-dicarboxylic acid monomers, but may include 1,2- and/or 1,3-dicarboxylic acid monomers).*	Bulking agent
Polyethylene Terephthalate 25038-59-9	Polyethylene Terephthalate is the organic compound that *is as shown in the structure in Figure 2. Polyethylene Terephthalate is a copolymer of propylene glycol and dimethyl terephthalate or terephthalic acid.*	Adhesive; film former; hair fixative; viscosity increasing agent-nonaqueous
Polypentaerythrityl Terephthalate	Polypentaerythrityl Terephthalate is the polyester of Pentaerythritol and Terephthalic Acid.	Film former; hair fixative
Polypropylene Terephthalate	Polypropylene Terephthalate is the homopolymer that *is as shown in the structure in Figure 2. Polypropylene Terephthalate is a copolymer of propylene glycol and dimethyl terephthalate or terephthalic acid.*	Emulsion stabilizer; skin-conditioning agent-miscellaneous

Table 2. Chemical and physical properties of modified terephthalate polymers.

Property	Value	Reference
Polyethylene terephthalate		
Density/Specific Gravity @ ºC	1332	27
Melting Point ºC	270	3
	255-265	27
Water Solubility g/L @ ºC & pH	Insoluble	27

Table 3. Studies on chemicals leaching from PET.

Study	Results	References
Potential migrants were isolated from commercial amber PET bottles by Soxhlet extraction using absolute ethanol, concentrated by distillation and nitrogen flushing, and analyzed.	A total of 19 migrants identified. Most were intermediate reaction products or residual monomers of their dehydration and transesterification products. Processing aids (i.e., fatty acids, plasticizers) also identified. The 7 most common compounds were: ethylene glycol (14.4 µg/g), terephthalic acid (19.7 µg/g), bis-2-ethylhexyl) phthalate (820 µg/g), bis-(2-ethylhexyl) adipate (560 µg/g), dibutyl phthalate (220 µg/g) diethyl phthalate (120 µg/g), pyrogallol (0.6 µg/g).	12
PET packaging materials (laminates, bottles, and roasting bags) were tested for volatile content after exposure to high temperatures (120, 150, or 230°C) for 50 min, according to sample type.	Few volatiles were found for samples composed only of PET. Volatiles from laminates varied according to the sample structure, but the main substances identified were not related to PET (probably from printing inks and adhesives). The authors concluded that the migration potential of PET in high temperature applications is very low.	10
Migration of ethylene glycol from PET bottles into a food simulate that was 3% acetic acid was measured. The bottles were stored at 32°C for up to 6 months.	1 month - trace ethylene glycol in food simulant; 6 months - ~100 ppb (~ 94 µg/bottle).	11
3 PET bottles were crush to < 0.7 mm particles and exhaustively extracted with methylene chloride for 3 d	Ethylene glycol was extracted at ~ 15 ppm.	11
The migration of benzene, butyric acid, dodecane, octadecane, tetracosane, diazinon, lindane, and copper (II) ethyl hexonate from PET sheets into the food simulants, 8% ethanol/water, and n-heptane. The contaminated PET sheets were extruded from PET chips that had been previously contaminated but were washed, dried, and remelted.	Contaminants levels ranged from benzene at 0.6 mg/kg - copper salt at 24 mg/kg. Migration of the residual contaminants from the extruded PETE sheets resulted in concentrations < 10 µg/kg in the food simulants.. The crystallinity of PET sheets in this study ranged from 5% to 15%, which is lower than that of most commercial PET (30%). The authors concluded that the samples represent the most severe conditions for conservative exposure evaluations.	13
Two food contact grade PET samples (in pellet form) were analyzed for content of elements. Fresh samples were exposed to food simulants (olive oil of a suitable grade for overall migration testing, acetic acid, or ethanol) for 10 d @ 40°C or 2 h @ 100°C according to European Economic Community directives.[42]	**Sample content (mg/kg)** / **Content in 3% acetic acid (µg/kg)** / **Content in 15% ethanol (µg/kg)** / **Content in olive oil (mg/kg)** **40°C/10 d** / **100°C/ 2 h** / **40°C/10 d** / **40°C/10 d** / **100°C/ 2 h** **Mg** < 1 / 0.51 / <0.1 / < 210 / < 13 / < 10 5.9 / 2.8 / <0.1 / <220 / <0.9 / <8 **Al** 0.66 / 0.78 / <0.1 / <160 / <200 / <140 620 / 0.75 / <0.1 / <170 / <1 / <220 **Co** 58 / 0.08 / 0.05 / 0.13 / <0.01 / <0.01 33 / 0.24 / 0.15 / 0.24 / <0.01 / <0.01 **Ge** 0.95 / <0.07 / <0.05 / <0.2 / <0.1 / <0.09 14 / 0.25 / <0.05 / <0.2 / <0.1 / <0.07 **Sb** 160 / 2.7 / 3.9 / 23 / <0.01 / <0.01 230 / 1.2 / 2.6 / 1.1 / <0.01 / <0.01	9
1.5 liter green PET bottles of naturally carbonated mineral water were stored up to 6 months.	The total organic content of the mineral water was similar to that stored in glass bottles except for week 2 of storage. Acetaldehyde, dimethyl terephthalate, and terephthalic acid were detected.	14
1.5 liter green PET bottles tested according to EEC and FDA tests with and without the modification of freeze-drying the distilled water.	EEC standard and modified: 16 ± 1.2 and 121 ± 4.0 ppm total migrants (60 ppm limit). FDA standard and modified: 38 ± 1.4 and 171 ± 2.5 ppm total migrants (50 ppm limit).	14
Distilled water was stored in PET bottles for 10 d at 40°C and 2 h at 120°. The water was tested for total organic carbon content.	Total organic carbon content was 1.5 ppm.	8
Distilled water was stored in PET bottles in the dark and sun light. The water was tested for total organic carbon content every 15 d for 6 months.	There was more organic carbon content detected in the light- than the dark-stored bottles. The peaks were approximately at 1, 3, and 5 months (in daylight: ~1, ~1, and ~3.5 mg/L; in dark ~1, ~0.6, and ~1.5 mg/L, respectively).	8
7 Brands of PET bottles were washed with Milli-Q water or used as received. Ultrapure water (20 ml) was poured into the bottles at room temperature or boiling. The original caps were used when the temperature reached room temperature. The bottles were stored in the dark for 24 h.	Boiling water increased the amount of antimony (2.077 vs 8.145 ppb) for all 7 brands of PET bottles. The authors concluded that this was a minor effect on leaching. There was more antimony in unwashed bottles than in washed bottles. No significant leaching was detected for Al, V, Cr, Mn, Co, Ni, Cu, As, Se, Mo, Ag, Cd, Sb, Ba, Tl, or Pb.	7

Table 3. Studies on chemicals leaching from PET.

Study	Results	References
The above experiment was repeated with room temperature and ice-cold water	There was no difference in the amount of leaching of antimony from the PET bottles between the temperatures. There was more antimony in unwashed bottles than in washed bottles. No significant leaching was detected for Al, V, Cr, Mn, Co, Ni, Cu, As, Se, Mo, Ag, Cd, Sb, Ba, Tl, or Pb.	7
The above experiment was repeated with room temperature water and placement in a microwave oven (1200 W) for 3 min in cook mode.	Microwaving the water in the bottles increased antimony content (0.381 - 10.51 µg/L) relative to controls. The authors concluded that this was a minor effect on leaching. There was more antimony in unwashed bottles than in washed bottles. No significant leaching was detected for Al, V, Cr, Mn, Co, Ni, Cu, As, Se, Mo, Ag, Cd, Sb, Ba, Tl, or Pb.	7
The above experiment was repeated with 20 ml acidic water (pH = 4.0). These bottles were stored for 7 days.	Acidic water increased the antimony content (0.459 – 4.611 µg/L) relative to controls. The authors concluded that this was a minor effect on leaching. There was more antimony in unwashed bottles than in washed bottles. No significant leaching was detected for Al, V, Cr, Mn, Co, Ni, Cu, As, Se, Mo, Ag, Cd, Sb, Ba, Tl, or Pb.	7
The above experiment was repeated with the bottles left directly in natural sunlight for 7 days with or without a foil cover.	Direct sunlight increased the antimony content (0.049 – 2.428 µg/L) relative to controls. The authors concluded that this was a minor effect on leaching. There was more antimony in unwashed bottles than in washed bottles. No significant leaching was detected for Al, V, Cr, Mn, Co, Ni, Cu, As, Se, Mo, Ag, Cd, Sb, Ba, Tl, or Pb.	7
The above experiment was repeated with the bottles left in an unairconditioned car, window closed and parked in an open parking lot during the day for 7 days.	Environmental conditions in a car (20°C - 45°C) increased the antimony content (0.482 – 3.08 µg/L) relative to controls. The authors concluded that this was a minor effect on leaching. There was more antimony in unwashed bottles than in washed bottles. No significant leaching was detected for Al, V, Cr, Mn, Co, Ni, Cu, As, Se, Mo, Ag, Cd, Sb, Ba, Tl, or Pb.	7
Commercially packaged carbonated mineral water and lemon/orange/citrus drinks were analyzed for acetaldehyde.	Acetaldehyde was detected in 2/4 mineral waters at 30 and 31 ng/ml. The amount of acetaldehyde in the bottles ranged from 1.7 – 3.8 µg/g and did not correlate with the results in the mineral waters. In the citrus drinks, the amount of acetaldehyde ranged from 11 – 7447 ng/ml. The amount of acetaldehyde in the bottles ranged from 1.1 – 3.8 µg/g and did not correlate with the results in the citrus drinks.	15

Table 4. Studies on leaching and estrogenic activity of PET containers.

Study	Results	Reference
	Estrogenic activity	
20 brands of mineral water, 9 of which are available both in glass and in PET bottles were tested with a yeast estrogen screen, employing a strain transfected with the human estrogen receptor α. Negative control- borosilicate Erlenmeyer flasks with culturing water.	3/9 brands in glass and 7/9 brands from PET bottles demonstrated estrogenic activity in this bioassay. Estrogenic contamination was detected in 60% of all samples with a maximum activity equivalent to 75.2 ng/L of the natural sex hormone 17β-estradiol. It is not certain that the estrogenic substance or substances leached from the bottles; the contamination may have been prior to bottling.	23
PET water bottles and glass bottles (same as above) were emptied of water and filled with a defined culture medium (pH 8.0 ± 0.5) and incubated New Zealand mudsnails, *Potamopyrgus antipodarum*, for 56 days.	Production of embryos increased among snails incubated in PET bottles compared with snails incubated in glass bottles across all brands ($p < 0.001$). For example, production of embryos incubated in PET bottles of brand D was roughly double the production of embryos incubated in glass bottles of brand D. However, in the yeast estrogen screen, this same brand showed no difference in estrogenic activity between PET bottle and glass bottle. The authors suggest that the *in vivo* snail bioassay might be more sensitive than the *in vitro* yeast estrogen screen. The authors concluded that the PET material were potent enough to trigger estrogenic effects *in vivo* similar to 25 ng/L 17α ethinylestradiol . The maximum estrogen activity detected in any brand of water was equivalent to 75 ng/L of ethinylestradiol.	23
30 samples of commercial brands (n = 9) of Italian mineral water packaged PET were analyzed for estrogenic activity using the Yeast Estrogen Screen (S. cerevisiae RMY326 (His3 Leu2-3,112trp1-1ura3-52/hER-TRP1-2 µ [pG/ER(G)], ERE-CYC-LacZ-URA3-2 µ [pUCΔSS-ERE],HIS-3CEN/ ARS[pRS423]) containing the human estrogen receptor a (hERa) and an estrogen-responsive element (ERE) bound	90% of samples exhibited estrogenic activity lower than 10% of the activity induced by 10nM 17β-estradiol (E2). The highest estrogenic activity measured was 11.32% of E2, corresponding to 23.1 ng/L estradiol equivalents.	21

Table 4. Studies on leaching and estrogenic activity of PET containers.

Study	Results	Reference
to the reporter gene lacZ encoding for the enzyme b-galactosidase.		
	Leachates	
71 commercial brands of bottled water, available both in glass and PET, were analyzed for: PhA, DEHP, DMP, DEP, DiisoBP, and DBP[1].	The concentration of all phthalates combined was > 20x higher in PET (3.52 µg/l) than in glass (0.19 µg/l) bottled water in all brands. The concentration of phthalates in water from glass bottles was below the limits of detection in most cases. The most abundant phthalates observed in PET-bottled water were DBP, DiisoBP, and DEP. There were slightly higher concentrations of phthalates observed for the PET bottled still water samples than for sparkling water samples. There was no correlation between the phthalate concentrations and other physicochemical properties of the different water samples. The concentration of phthalates was always below 0.1% of the limit set by the EPA in 2006.	20
Water from PET and glass bottles were analyzed at purchase and after 10 weeks of storage for DMP, DEP, di-n-butylphthalate, butylbenzylphthalate, DEHP, and bisphenol A diglycyleter. Source waters from aquifers were also analyzed.	At purchase, the concentration of phthalates was at or below detection limits in almost every case. At 10 weeks the concentration of phthalates in glass-bottled water was similar. 3/5 brands with PET bottles showed measurable levels of DEHP after 10 weeks (ave 0.134 µg/L). All 5 brands had measurable levels of DEP at 10 weeks (ave 0.214 µg/L). Total phthalate were up to 1.7 µg/ L. Water from aquifers measured 0.005 – 0.331 µg/L.	18
PET bottles filled with water were incubated in direct sunlight. for 17 hr	The maximum concentration of DEHP was 0.71 µg/L, respectively, similar to those reported in studies on commercial bottled water. Only food flavor constituents of previous bottle contents identified above a detection limit of 1 µg/L. The country of origin was the only consistent variable.	22
Mineral water (still and carbonated) collected from a bottling plant was used to fill PET and glass bottles. All bottles were stored at room temperature. Each month, for 12 months, samples of water were lyophilized, the powders then shaken with acetone, and the acetone extracts analyzed using GC/MS.	No phthalates were observed for the first 8 months in any sample. Beginning at month 9 for PET-bottled noncarbonated water, and month 10 for PET-bottled carbonated water, the phthalate content increased from 0.4 to > 3.0 mg/L. DEHP was detected.	16
The interaction of incubation time with storage temperature on the leaching of DEHP from PET bottles was studied by using a solution of 3% acetic acid as a food simulant. Bottles were incubated up to 120 days, at 25°C or 45°C.	On day 0, DEHP in PET bottles was below detection limits. On day 25, the amount of DEHP at 25°C was 1.2 mg/L; at 45°C was 2.1 mg/L. On day 66, the amount of DEHP at 25°C peaked at 1.4 mg/L; 45°C at 2.5 mg/L.	19
45 samples of products packed in PET containers were incubated for 30 days. Group 1 (n = 9), soft drinks preserved with orthophosphoric acid; group 2 (n = 14), soft drinks preserved with Na-benzoate; group 3 (n = 5), soft drinks preserved with K-sorbate; group 4 (n = 8), soft drinks preserved with a combination of Na-benzoate and K-sorbate; and group 5 (n = 9), mineral water without preservatives. The amounts of DMP, DBP, DOP, DEP, BBP, and DEHP were measured.	Group 1- mean pool phthalate levels were 91.67 µg/L at a pH of 2.82 ± 0.30; Group 2 - 116.93 µg/L at 2.75 ± 0.32; Group 3 - 819.40 µg/L at 2.88 ± 0.15; Group 4 - 542.63 µg/L at 2.82 ± 0.54; Group 5 - 20.22 µg/L at 5.82 ± 1.26. There were large variations in the concentrations of phthalates both across beverages and across manufacturers (i.e., no DMP in any brand of mineral water after 30 days was detected, whereas DMP was the most abundant phthalate detected in the soft drinks). Among soft drinks preserved with both sodium benzoate and potassium sorbate incubated for 30 days, the concentration of DMP ranged from 18 - 2,666 µg/L, mean 501 µg/L. DMP in mineral water was below detection limits. DEHP (unlike DMP) did not differ between soda beverages and mineral water (ave < 100 µg/L in all their specimens, with no difference between soda and mineral water. The authors suggested that the lower pH of the soft drinks might account for differences.	17
Commercially packaged carbonated mineral water and lemon/orange/citrus drinks were analyzed for acetaldehyde.	Acetaldehyde was detected in 2/4 mineral waters at 30 and 31 ng/ml. The amount of acetaldehyde in the bottles ranged from 1.7 – 3.8 µg/g and did not correlate with the results in the mineral waters. In the citrus drinks, the amount of acetaldehyde ranged from 11 – 7447 ng/ml. The amount of acetaldehyde in the bottles ranged from 1.1 – 3.8 µg/g and did not correlate with the results in the citrus drinks.	15

[1] BBP – benzylbutyl phthalate; DBP - dibutyl phthalate; DEHP - bis(2-ethylhexyl) phthalate; DEP - diethyl phthalate; DiisoBP - diisobutyl phthalate; DMP - dimethyl phthalate; DOP – dioctyl phthalate; PhA - phthalic acid.

Table 5. Frequency of use according to duration and exposure of modified terephthalate polymers. The Council is conducting a survey for the concentrations of use.[26]

Use type	Uses	Maximum Concentration (%)	Uses	Maximum Concentration (%)	Uses	Maximum Concentration (%)	Uses	Maximum Concentration (%)
	Polyethylene terephthalate		Polybutylene Terephthalate		Polyethylene Isoterephthalate		Polypropylene Terephthalate	
Total/range	**396**		**23**		**2**		**23**	
Duration of use								
Leave-on	394		21		2		13	
Rinse-off	1		2				10	
Diluted for (bath) use	1		NR		NR		NR	
Exposure type								
Eye area	98		3		NR		NR	
Incidental ingestion	173		1		2		NR	
Incidental Inhalation-sprays	16		2		NR		NR	
Incidental inhalation-powders	7		NR		NR		NR	
Dermal contact	167		10		NR		23	
Deodorant (underarm)	NR		NR		NR		NR	
Hair-noncoloring	NR		NR		NR		NR	
Hair-coloring	1		NR		NR		NR	
Nail	47		12		NR		NR	
Mucous Membrane	175		1		2		8	
Baby	NR		NR		NR		NR	

NR = Not Reported; Totals = Rinse-off + Leave-on Product Uses.
Note: Because each ingredient may be used in cosmetics with multiple exposure types, the sum of all exposure type uses may not equal the sum total uses.

Table 6. Code of Federal Regulations regarding the polybutylene terephthalate and PET.

Ingredient	Rule	Reference
Polybutylene terephthalate	Approved as a substance that may come in contact with food in packaging. Poly (tetramethylene terephthalate) is the reaction product of dimethyl terephthalate with 1,4-butanediol to which may have been added certain optional substances to impart desired technological properties to the polymer. Poly(tetramethylene terephthalate) may contain optional adjuvant substances. The quantity of any optional adjuvant substance employed in the production of the polymer does not exceed the amount reasonably required to accomplish the intended technical or physical effect. Such adjuvants may include substances generally recognized as safe in food, substances used in accordance with prior sanction, and substances permitted under applicable regulations in this part.	21CFR177.1660
Polyethylene terephthalate (PET)	May be safely used as, or components of plastics (films, articles, or fabric) intended for use in contact with food. Polyethylene phthalate films consist of a base sheet of ethylene terephthalate polymer, ethylene terephthalate-isophthalate copolymer, or ethylene-1,4-cyclohexylene dimethylene terephthalate copolyesters, to which have been added optional substances, either as constituents of the base sheet or as constituents of coatings applied to the base sheet. Polyethylene phthalate articles consist of a base polymer of ethylene terephthalate polymer, or ethylene-1,4-cyclohexylene dimethylene terephthalate copolyesters to which have been added optional substances, either as constituents of the base polymer or as constituents of coatings applied to the base polymer. Polyethylene phthalate spunbonded nonwoven fabric consist of continuous filaments of ethylene terephthalate polymer and ethylene terephthalate-isophthalate copolymer to which may have been added optional adjuvant substances required in their preparation and finishing. The ethylene terephthalateisophthalate copolymer component of the fabric shall not exceed 25 percent by weight. The filaments may be blended with other fibers regulated for	21CFR177.1630

	the specific use and the spunbonded fabric may be further bonded by application of heat and/or pressure.	
	May be safely used as articles or components of articles, intended for use in producing, manufacturing, packing, processing, preparing, treating, packaging, transporting or holding food.	21CFR177.1850
	May be safely used in producing, manufacturing, processing, and preparing food.	21CFR177.2260
	May safely be used as articles or components of articles intended for use in producing, manufacturing, packing, processing, preparing, treating, packaging, transporting, or holding food.	21CFR177.2800
	Hydrogen peroxide solution identified in and complying with the specifications in this section may be used by itself or in combination with other processes to treat food-contact surfaces to attain commercial sterility at least equivalent to that attainable by thermal processing for metal containers as provided for in part 113 of this chapter. The packaging materials identified in paragraph (e)(1) of this section may be used for packaging all commercially sterile foods.	21CFR178.1008
	May be safely subjected to irradiation incidental to the radiation treatment and processing of prepackaged foods.	21CFR179.45
	Cardiovascular Prosthetic Devices Intracardiac patch or pledget made of polypropylene, polyethylene terephthalate, or polytetrafluoroethylene. Identification: An intracardiac patch or pledget made of polypropylene, polyethylene terephthalate, or polytetrafluoroethylene is a fabric device placed in the heart that is used to repair septal defects, for patch grafting, to repair tissue, and to buttress sutures.	21CFR870.3470
	Nonabsorbable poly(ethylene terephthalate) surgical suture. Identification: Nonabsorbable poly(ethylene terephthalate) surgical suture is a multifilament, nonabsorbable, sterile, flexible thread prepared from fibers of high molecular weight, long-chain, linear polyesters having recurrent aromatic rings as an integral component and is indicated for use in soft tissue approximation. The poly(ethylene terephthalate) surgical suture meets U.S.P. requirements as described in the U.S.P. Monograph for Nonabsorbable Surgical Sutures; it may be provided uncoated or coated; and it may be undyed or dyed with an appropriate FDA listed color additive. Also, the suture may be provided with or without a standard needle attached.	21CFR878.5000
	Esophageal dilator. Identification:. An esophageal dilator is a device that consists of a cylindrical instrument that may be hollow and weighted with mercury or a metal olive-shaped weight that slides on a guide, such as a string or wire and is used to dilate a stricture of the esophagus. This generic type of device includes esophageal or gastrointestinal bougies and the esophageal dilator (metal olive)	21CFR876.5365

Table 7. Genotoxicity tests of contents of PET bottles after storage.

Study	Results	Reference
Ames test performed on water stored in PET bottles (500 – 4000 ml/plate) for up to 6 months	Not mutagenic to *Salmonella* (strains TA98, TA100) with or without metabolic activation.	[14]
Ames test performed on concentrated mineral water after storage in shaken PET bottles for 24 or 48 h at 40°C.	Not mutagenic to *Salmonella* (strains TA98, TA100) with or without metabolic activation. The 48-h sample was toxic to the bacteria.	[8]
Ames test performed on mineral water stored in PET bottles[1] in the dark and in sunlight for 1, 3, or 6 months. Glass bottles served as controls.	The samples stored for 1 month (both dark and day light) were mutagenic to *Salmonella* (strain TA98) with metabolic activation. At 3 and 6 months, the samples were not mutagenic.	[8]
Salmonella (strains TA98, TA100) were incubated in PET bottles containing mineral water. *Salmonella* (strains TA98, TA100) were incubated in glass flasks containing mineral water that was stored in PET bottles for up to 6 months.	Not mutagenic to *Salmonella* (strains TA98, TA100) with or without metabolic activation.	[8]
Salmonella (strains TA98, TA100) was inoculated into sterilized PET bottles. Glass flasks served as + controls; flasks with known mutagens added served as - controls	Not mutagenic to *Salmonella* (strains TA98, TA100) with or without metabolic activation.	[8]
Distilled water (100%, 95%, 90%, 75%, 50%) stored in PET bottles at 10 d at 40°C and for 1 month at room temperature in sunlight was used as the water phase for preparation of Vogel-Bonner stock. Stock was placed in glass flasks and inoculated with *Salmonella*. Flasks were shaken for 24 h at 37°C. The bacteria were then tested for reversion.	Not mutagenic to *Salmonella* (strains TA98, TA100) with or without metabolic activation.	[8]

[1] PET made from the polycondensation of dimethyl terephthalte and ethyleneglycol.

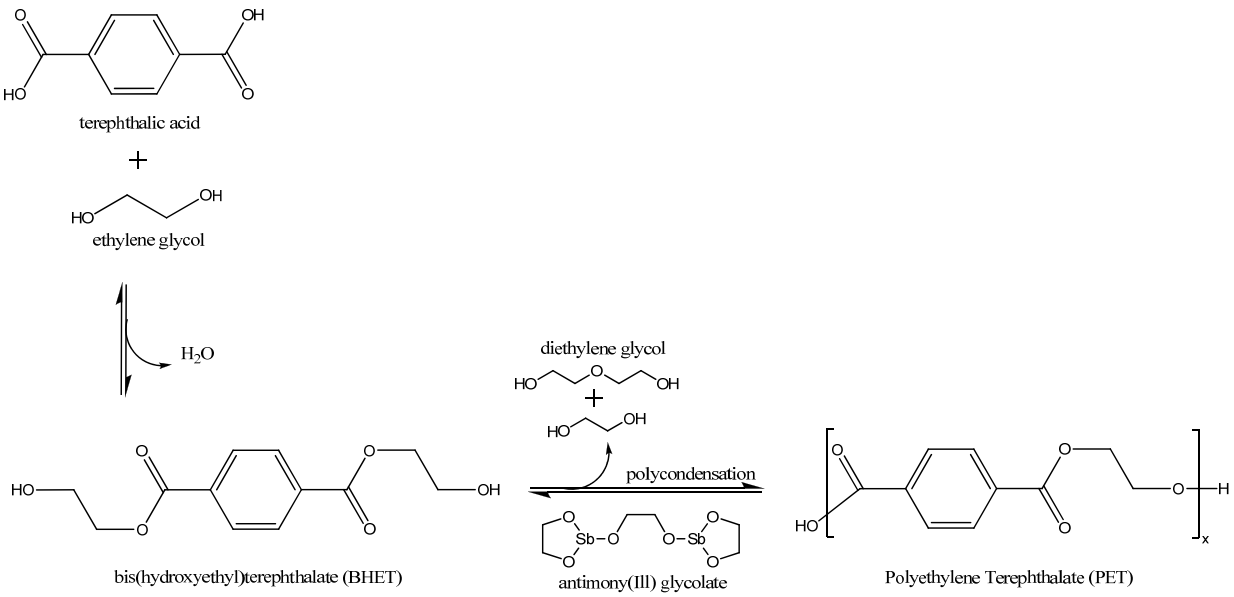
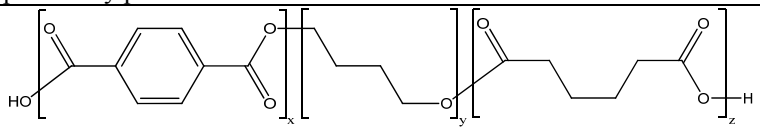
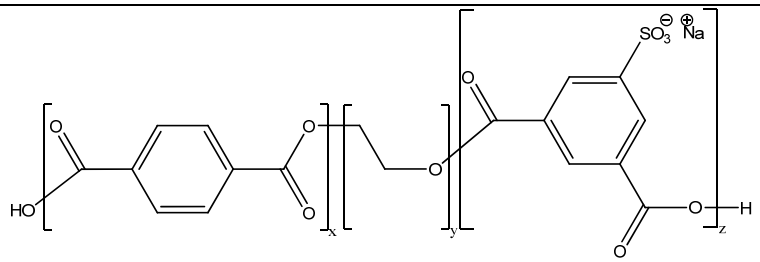
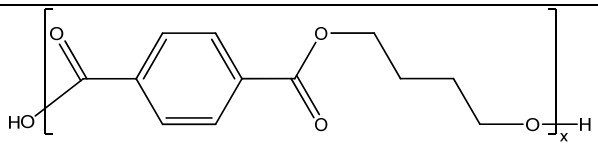
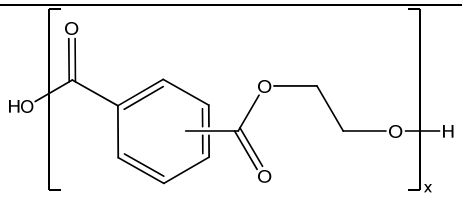
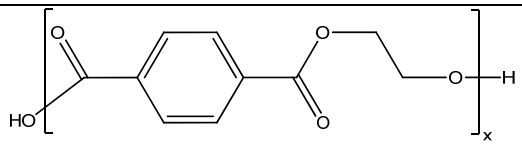
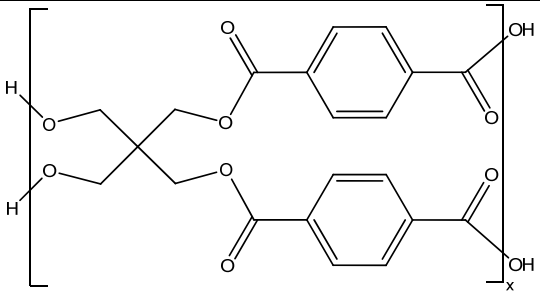
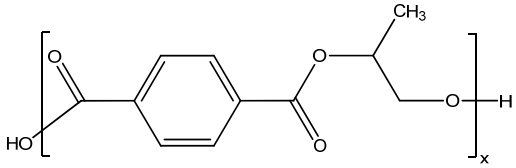


Figure 1. Most common manufacturing method for PET.

Figure 2. Idealized structures of the modified terephthalate polymer ingredients in this safety assessment. These idealized structures are merely generalized, two-dimensional estimations of the true three-dimensional frameworks that comprise theses polymers. Though monomer units are in some instances drawn sequentially, by necessity, this by no means implies that these are block-type polymers. Instead, these structures are meant to represent only one example of the multitude of potentially produced connectivities found within these macromolecules.

Adipic Acid/1,4-Butanediol/ Terephthalate Copolymer	
Ethylene/Sodium Sulfoisophthalate/ Terephthalate Copolymer	
Polybutylene Terephthalate	
Polyethylene Isoterephthalate	
Polyethylene Terephthalate	
Polypentaerythrityl Terephthalate	
Polypropylene Terephthalate	

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Data

Cosmetic Ingredient Review
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C I R

November 16, 2012

MEMORANDUM

To: CIR Expert Panel and Liaisons

From: Lillian C. Becker, M.S.
Scientific Analyst and Writer

Subject: Unpublished Data for the Draft Report for Modified Terephthalate Polymers as used in cosmetics

The Personal Care Products Council submitted unpublished data for modified terephthalate polymers. This data has been incorporated into the report.

The data submitted include:

1) Product/production data on polyethylene terephthalate (PET) glitter and an analysis of impurities and leachates.

2) A product use study of two eye shadows containing PET (46.272%).

3) A product use test of mascaras containing PET (4.2% and 4.8%; RIPT of an eyeliner containing PET (1.5%).

4) Email from David Steinberg about his concern of eye injury due to PET glitter.

5) Notes from an interview with Dr. Glasser about his experience with glitter and eye injuries.

6) VCRP data.

1101 17th St., NW, Suite 412 Washington, DC 20036-4702
Phone 202.331.0651 FAX 202.331.0088 http://www.cir-safety.org

Personal Care Products Council
Committed to Safety, Quality & Innovation

Memorandum

TO: F. Alan Andersen, Ph.D.
Director - COSMETIC INGREDIENT REVIEW (CIR)

FROM: Halyna Breslawec, Ph.D.
Industry Liaison to the CIR Expert Panel

DATE: August 31, 2012

SUBJECT: Information Regarding Polyethylene Terephthalate

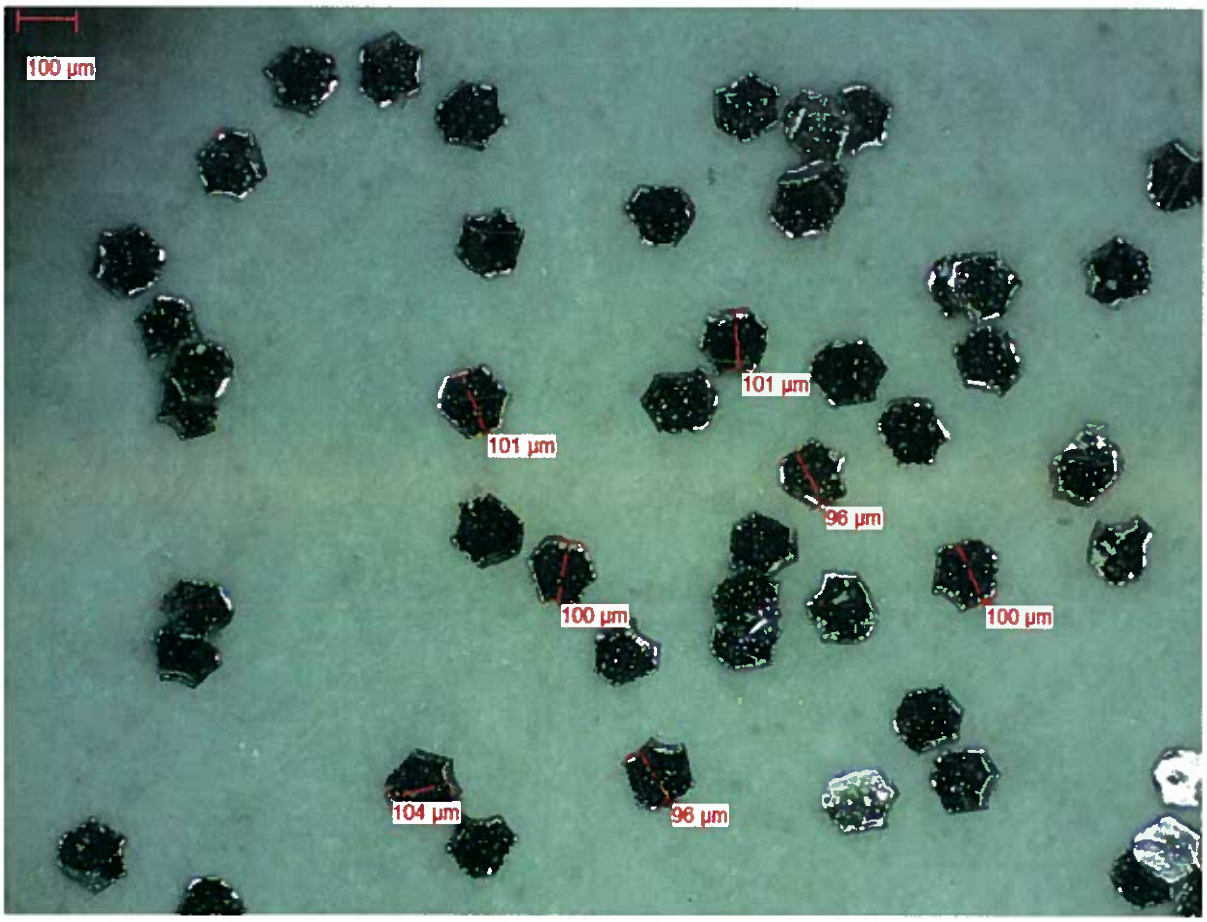
Method of Manufacture:

coating of Polyethylene Terephthalate film (metallized with aluminum)
roll slitting
precision cutting (hexagonal or square shape particles)
sifting
quality control and packaging

Hexagonal and square shapes are available. The size and shape is defined by a precision cutting process. Because of the cutting process Polyethylene Terephthalate used in cosmetics does not have a typical particle size distribution as other products (for example, mica) normally have. An example of the measurement of particle size of a 0.004" hexagonal cut product is attached.

SGS Institut Fresenius. 2011. Analytical report for polyester glitter (Polyethylene Terephthalate). Test Report No. 2052901-02.

June 2012



0.004" hexagonal cut Polyethylene Terephthalate

SGS INSTITUT FRESENIUS

SGS INSTITUT FRESENIUS GmbH · Postfach 1261 · 65220 Taunusstein

[redacted]

Order No. : 2052901
Client No. : 10006307

Dr. Michael Kofink / AC
Tel. +49 (0)6128/744-383, Fax-201
michael.kofink@sgs.com

Karin Wanke
Tel. +49 (0)6128/ 744-383, Fax -201
karin.wanke@sgs.com

Consumer Testing Services Germany

SGS INSTITUT FRESENIUS GmbH
Im Maisel 14

Taunusstein, 19.10.2011

Subject	:	Analytic1est
Your reference	:	[redacted]
Order Date	:	01.09.2011
Sample No.	:	110560530
Testing period	:	05.09.-19.10.2011

Test Report no. 2052901-02
Reference test for article group / colour: [redacted] Polyester Glitter [redacted]

Dear [redacted]

Regarding your order we have received the samples that have been investigated.

We received the samples on September 5th 2011 and they have been documented as below:

The product was analysed referring to EN 71-3 and EN 71-9, European Cosmetic Directive and Oecotex-Standard. Please note that if a parameter is mentioned in another standard it was only be analysed one-time and evaluated according to the more strict limit / requirement.

The test results can be considered as a reference for the following article numbers:

[redacted]

Sample No.	Sample Description
110560530	[redacted] Polyester Glitter [redacted]

[redacted]

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Geschäftsführer: Vorsitz Gustav Fenzau, Aufsichtsratsvorsitzender: Dirk Hoffmann, Sitz der Gesellschaft: Taunusstein, HRB 31581 Amtsgericht Wiesbaden

SGS INSTITUT FRESENIUS

Order No 2052901
Test Report No. 2052901-02
Sample No. 110560530
Client [redacted]

19.10.2010

Results

Parameter	Limits/ Requirements	Results 110560530 2510-50-3hex Silgit Polyester Glitter Grade I .008 hex:silver LOT No:1161611
1. Heavy metals using ICP-OES/ICP-MS/AAS limits according to BGA requirements for cosmetic products		
arsenic	max. 5 mg/kg	< 0,5 mg/kg (1)
antimony	max. 10 mg/kg	169,0 mg/kg (2)
lead	max. 20 mg/kg	2,0 mg/kg (3)
cadmium	max. 5 mg/kg	< 0,5 mg/kg (1)
mercury	max. 1 mg/kg	< 0,1 mg/kg (1)
nickel (total amount) *	not regulated	< 1 mg/kg (1)
chromium *	not regulated	< 1 mg/kg (1)
2. Migration of heavy metals using ICP-OES/ICP-MS/AAS limits according to EN 71 - 3		
arsenic	max. 25 mg/kg	< 1,0 mg/kg (1)
antimony	max. 60 mg/kg	< 1,0 mg/kg (1)
lead	max. 90 mg/kg	1,5 mg/kg (3)
cadmium	max. 75 mg/kg	< 1,0 mg/kg (1)
mercury	max. 60 mg/kg	< 0,05 mg/kg (1)
chromium	max. 60 mg/kg	< 1,0 mg/kg (1)
barium	max. 1000 mg/kg	< 1,0 mg/kg (1)
selenium	max. 500 mg/kg	< 1,0 mg/kg (1)

* Nickel soluble in sweat solution and hexavalent chromium was only determined if there was a positive measurement of nickel (total amount) and chromium

(1) Measurement below the limit of quantification
(2) Measurement exceeds the limit of 150 mg/kg (technical avoidable amount for antimony in glitter raw material); Opinion of the working group "Personal Care" of the German Chemical Society (Lebensmittelchemie 64, 49-80 (2010))
(3) Measurement below the limit

[redacted]

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SGS INSTITUT FRESENIUS

Order No 2052901
Test Report No. 2052901-02
Sample No. 110560530
Client [redacted]

19.10.2010

Results

Parameter	Limits/ Requirements	Results 110560530 2510-50-3hex SILIgit Polyester Glitter Grade I .008 hex silver LOT No:1161611
3. Formaldehyde (free) using HPLC-DAD limit according to EU Cosmetic Directive	max. 0,2 %	< 5 mg/kg (1)
4. Phthalates using GC-MS according to EU Cosmetic Directive		
Diisononylphthalate (DINP)	not regulated	< 10 mg/kg (1)
Diethylhexylphthalate (DEHP)	not detectable	< 10 mg/kg (1)
Dibutylphthalate (DBP)	not detectable	< 10 mg/kg (1)
Diisodecylphthalate (DIDP)	not regulated	< 10 mg/kg (1)
Di-n-octylphthalate (D-n-OP)	not regulated	< 10 mg/kg (1)
Butylbenzylphthalate (BBP)	not detectable	< 10 mg/kg (1)
Diisobutylphthalate (DIBP)	not regulated	< 10 mg/kg (1)
Dimethylphthalate (DMP)	not regulated	< 10 mg/kg (1)
Diethylphthalate (DEP)	not regulated	< 10 mg/kg (1)
Dibutylsebacate (DBS)	not regulated	< 10 mg/kg (1)
Di(2-ethylhexyl)adipate (DEHA)	not regulated	< 10 mg/kg (1)
Tributylacetylcitrate (TBAC)	not regulated	< 10 mg/kg (1)
5. Nonylphenole using GC-MS according to EU Cosmetic Directive		
4-Nonylphenole	not detectable	< 10 mg/kg (1)
4-n-Nonylphenole	not detectable	< 10 mg/kg (1)
4-n-Octylphenole	not detectable	< 10 mg/kg (1)

(1) Measurement below the limit of quantification

[redacted]

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SGS INSTITUT FRESENIUS

Order No 2052901
Test Report No. 2052901-02
Sample No. 110560530
Client [redacted]

19.10.2010

Results

Parameter	Limits/ Requirements	Results 110560530 2510-50-3hex SiLigit Polyester Glitter Grade I .008 hex silver LOT No:1161611
6. organo tin compounds using GC-MS (ISO 17353) limits according to Oecotex-Standard		
Monobutyltin-	not regulated	< 0,01 mg/kg (1)
Dibutyltin-	1,0 mg/kg	0,19 mg/kg (2)
Tributyltin-	0,5 mg/kg	< 0,01 mg/kg (1)
Tetrabutyltin-	not regulated	< 0,01 mg/kg (1)
Monooctyltin-	not regulated	< 0,01 mg/kg (1)
Dioctyltin-	1,0 mg/kg	< 0,01 mg/kg (1)
Tricyclohexyltin-	not regulated	< 0,01 mg/kg (1)
7. Organic solvents using GC-MS limits according to EN 71-9 Section 2e)		
2-Ethoxyethanol	technical avoidable amount	< 0,02 mg/kg (1)
2-Methoxypropylacetate		< 0,02 mg/kg (1)
2-Methoxyethylacetate		< 0,02 mg/kg (1)
Styrene		< 0,03 mg/kg (1)
2-Ethoxyethylacetate		< 0,02 mg/kg (1)
Bis-(2-methoxyethyl)-ether		< 0,02 mg/kg (1)
Nitrobenzene		< 0,01 mg/kg (1)
Isophorone		< 0,1 mg/kg (1)
Methanol		< 2,0 mg/kg (1)
Dichlormethane		< 0,01 mg/kg (1)
Trichlorethene		< 0,01 mg/kg (1)
Toluene		< 0,10 mg/kg (1)
Ethylbenzene		< 0,10 mg/kg (1)
p-/m-Xylene		< 0,10 mg/kg (1)
o-Xylene		< 0,10 mg/kg (1)
Cyclohexanone		< 0,10 mg/kg (1)
Diglym		< 0,02 mg/kg (1)

(1) Measurement below the limit of quantification
(2) Measurement below the limit

[redacted]

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SGS INSTITUT FRESENIUS

Order No 2052901
Test Report No. 2052901-02
Sample No. 110560530
Client [redacted]

19.10.2010

Results

Parameter	Limits/ Requirements	Results 110560530 2510-50-3hex SiLight Polyester Glitter Grade I .008 hex silver LOT No:1161611
8. Primary aromatic amines using GC-MS after reductive splitting limits according to EU Cosmetic Directive		
Biphenyl-4-ylamine	not detectable	< 10 mg/kg (1)
Benzidine		< 10 mg/kg (1)
4-Chlor-o-toluidine		< 10 mg/kg (1)
2-Naphtylamine		< 10 mg/kg (1)
o-Aminoazotoluene		< 10 mg/kg (1)
5-Nitro-o-toluidine		< 10 mg/kg (1)
4-Chloraniline		< 10 mg/kg (1)
4-Methoxy-m-phenylendiamine		< 10 mg/kg (1)
4,4-Methylendianiline		< 10 mg/kg (1)
3,3-Dichlorbenzidine		< 10 mg/kg (1)
3,3-Dimethoxybenzidine		< 10 mg/kg (1)
3,3-Dimethylbenzidine		< 10 mg/kg (1)
4,4-Methylendi-o-toluidine		< 10 mg/kg (1)
6-Methoxy-m-toluidine		< 10 mg/kg (1)
4,4-Methylen-bis-(2-chlor-aniline)		< 10 mg/kg (1)
4,4-Oxydianiline		< 10 mg/kg (1)
4,4-Thiodianiline		< 10 mg/kg (1)
o-Toluidine (conversion product von o-Aminouotoluoene)		< 10 mg/kg (1)
4-Methyl-m-phenylendiamine (conversion product von 5-Nitro-o-toluidine)		< 10 mg/kg (1)
2,4,5-Trimethylaniline		< 10 mg/kg (1)
o-Anisidine		< 10 mg/kg (1)
4-Aminoazobenzene		< 10 mg/kg (1)
2,4-Xylidine		< 10 mg/kg (1)
2,6-Xylidine		< 10 mg/kg (1)

(1) Measurement below the limit of quantification

[redacted]

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Order No 2052901
Test Report No. 2052901-02
Sample No. 110560530
Client [redacted]

19.10.2010

Results

Parameter	Limits/ Requirements	Results 110560530 2510-50-3hex SILigit Polyester Glitter Grade I .008 hex silver LOT No:1161611
9. Polycyclic aromatic hydrocarbons (PAH) using GC-MS limits according to the EU Cosmetic Directive		
Phenanthren	not regulated	< 0,2 mg/kg (1)
Anthracen	not regulated	< 0,2 mg/kg (1)
Fluoranthen	not regulated	< 0,2 mg/kg (1)
Pyren	not regulated	< 0,2 mg/kg (1)
Benzo(a)anthracen	not regulated	< 0,2 mg/kg (1)
Crysen	not detectable	< 0,2 mg/kg (1)
Naphthalin	not regulated	< 0,2 mg/kg (1)
Acenaphthylen	not regulated	< 0,2 mg/kg (1)
Acenaphthen	not regulated	< 0,2 mg/kg (1)
Fluoren	not regulated	< 0,2 mg/kg (1)
High volatile PAH (total amount)		< 0,2 mg/kg (1)
Benzo(b)fluoranthen	not regulated	< 0,2 mg/kg (1)
Benzo(k)fluoranthen	not detectable	< 0,2 mg/kg (1)
Benzo(a)pyren	not detectable	< 0,2 mg/kg (1)
Indeno(1,2,3-cd)pyren	not regulated	< 0,2 mg/kg (1)
Dibenzo(ah)anthracen	not detectable	< 0,2 mg/kg (1)
Benzo(ghi)perylen	not regulated	< 0,2 mg/kg (1)
Low volatile PAH (total amount)		< 0,2 mg/kg (1)
Total PAH		< 0,2 mg/kg (1)

(1) Measurement below the limit of quantification

[redacted]

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SGS

INSTITUT FRESENIUS

Order No 2052901
Test Report No. 2052901-02
Sample No. 110560530
Client [redacted]

19.10.2010

Results

Parameter	Limits/ Requirements	Results 110560530 2510-50-3hex SiLiglit Polyester Glitter Grade I .008 hex silver LOT No:1161611
<u>10. monomeric plasticiser</u> using GC-MS according to EN 71-9 Section 2e)		
o-Trikresylphosphate	technical avoidable amounts	< 5 mg/kg (1)
m-Trikresylphosphate		< 5 mg/kg (1)
p-Trikresylphosphate		< 5 mg/kg (1)
Triphenylphosphate		< 5 mg/kg (1)

(1) Measurement below the limit of quantification

Parameter	Evaluation 110560530 2510-50-3hex SiLiglit Polyester Glitter Grade I .008 hex silver LOT No:1161611
<u>11. Colour fastness (fastness to perspiration and salvia)</u> method § 35 B 82.10-1; according to EN 71	Pass

Thank you for your order. We hope we could help you on this matter.

Yours faithfully

SGS INSTITUT FRESENIUS GmbH

I.V. Dr. Michael Kofink
(project leader cosmetics)

I.V. Karin Wanke
(project leader cosmetics)

[redacted]

Personal Care Products Council
Committed to Safety, Quality & Innovation

Memorandum

TO: F. Alan Andersen, Ph.D.
Director - COSMETIC INGREDIENT REVIEW (CIR)

FROM: Halyna Breslawec, Ph.D.
Industry Liaison to the CIR Expert Panel

DATE: October 17, 2012

SUBJECT: Information on a Product Containing Polyethylene Terephalate

Peritesesco. 2009. Ocular acceptability study of two eye shadows during 8 days under ophthalmological supervision (product 701102 contains 46.272% Polyethylene Terephthalate as precision cut flakes).

PERITESCO
Centre de Recherche Biomédicale

STUDY REPORT
Version 1 (30 December 2009)

OCULAR ACCEPTABILITY STUDY OF TWO EYE SHADOWS DURING 8 DAYS UNDER OPHTHALMOLOGICAL SUPERVISION

	INVESTIGATOR
ADDRESSES	
	Investigating Laboratory: PERITESCO 4, rue Villedo 75001 PARIS
STAFF IN CHARGE OF THIS STUDY	
	Director and Principal Investigator: Dr M. PERICOI Ophthalmologist Co-Investigator Dr T. GAMAR Ophthalmologist
REFERENCES	
FARD A PAUPIERES REF. 701100 LOT 20091013-1 FARD A PAUPIERES REF. 701102 LOT 20091013-2	results removed from report 5894-43-01 - contains 46.272% Polyethylene Terephthalate as precision cut flakes

PERITESCO
4, rue Villedo
75001 PARIS

☎ 08 25 05 15 65
Fax: 01 53 29 97 25
R.C.S. Paris B 389 377 672

STUDY ADMINISTRATIVE STRUCTURE AND TECHNICAL STAFF

Investigating laboratory

1. Address

PERITESCO
4, rue Villedo
75001 PARIS

2. Staff

Principal Investigator	Dr. M. PERICOI Ophthalmologist D.E.A of Dermopharmacy Clinical Toxicology and Pharmacology Degree Clinical Expert (Decree of the 29th of June 1985)
Co-Investigator	Dr. T. GAMAR Ophthalmologist
Study Manager	Miss M. BERNARD
Report Writing	Mrs. K. LOCH Assistant

TABLE OF CONTENTS

CERTIFICATE OF QUALITY POLICY

TITLE: OCULAR ACCEPTABILITY STUDY OF TWO EYE SHADOWS DURING 8 DAYS UNDER OPHTHALMOLOGICAL SUPERVISION

REFERENCES: 5894-43-01

INVESTIGATIONAL **PRODUCTS :**
- FARD A PAUPIERES REF. 701100 LOT 20091013-1
- FARD A PAUPIERES REF. 701102 LOT 20091013-2 = 46.272% Polyethylene Terephtalat precision cut flakes

PRINCIPAL INVESTIGATOR: Dr. M. PERICOI, Ophthalmologist

To my knowledge, the study described in this report was carried out according to the protocol, to PERITESCO Standard Operating Procedures (SOP) and in the spirit of the Good Clinical Practice guidelines of the International Conference on Harmonization (ICH Topic E6 « Note for guidance on Good Clinical Practice CPMP/ ICW 135/ 95 »), the FDA (62 FR 25692 of May 9, 1997), and the EEC (Directive n° 2001/20/CE of April 4, 2001). Audits were performed in compliance with the general procedures of Quality Assurance department and in the spirit of Good Clinical Practices guidelines of the International Conference on Harmonization (ICH Topic E6 « Note for guidance on Good Clinical Practice CPMP/ ICH/ 135195 »).

The following table mentions the audits that were carried out during the study, and the date of transmission of the audits reports to the investigator and the study manager:

Type **of audit**	**Date of audit**	**Date of report to the investigator**
Protocol audit	29/10/2009	29/10/2009
Preliminary results audit	30/11/2009	30/11/2009
Report audit ..	30/12/2009 . .	30/12/2009
Last in-life audit performed for the same type of study	22/06/2009 22/07/2009	24/08/2009

This report constitutes a true and faithful record of the original raw data of the investigating laboratory, generated during the performance of the study.

Miss M. HERING
Quality Policy
PERITESCO

31/12/09
date

CERTIFICATE OF QUALITY CONTROL

TITLE: OCULAR ACCEPTABILITY STUDY OF TWO EYE SHADOWS DURING 8 DAYS UNDER OPHTHALMOLOGICAL SUPERVISION

REFERENCES: 5894-43-01

INVESTIGATIONAL PRODUCTS:
- FARD A PAUPIERES REF. 701100 LOT 20091013-1
- FARD A PAUPIERES REF. 701102 LOT 20091013-2

PRINCIPAL INVESTIGATOR: Dr. M. PERICOI, Ophthalmologist.

A quality control program checked that the report presents the raw data of the study.

All the controls on the data veracity and conformity with the protocol were performed at each step of the study:

Verifications	Date	Controlled by
Protocol conformity	2711012009	MB
Case report form	0411112009	GC
Clinical data	2011112009	MB
Preliminary results	2511112009	GC
Report	1011212009	MB

BERNARD

Miss. M. BERNARD
Quality Control
PERITESCO

31.12.2009
date

METHODOLOGY

I. STUDY DESCRIPTION

I.1. Investigational products

The investigational products were supplied

These investigational products had the following characteristics:

Investigational products' denominations and references	FARD A PAUPIERES REF. 701100 FARD A PAUPIERES REF. 701102
Batch numbers	LOT 20091013-1 LOT 20091013-2
Presentation	Ochre glittered compact powder Brow glittered compact powder
Packaging	Plastic transparent case
Supplied quantity	17 samples of each investigational product
Date of receipt	23 October 2009
Storage	Between 15°C and 25°C

The name and reference of the investigational products as labeled on the samples were the following:
- Fard a Paupieres F1A# 701100 Batch# 20091013-1
- Fard a Paupieres F1A# 701102 Batch# 20091013-2

All the investigational products (including the investigational products given back by the subjects at the end of the study and the possible investigational products in surplus) were destroyed within the deadline foreseen in the study protocol.

A sample of each investigational product is stored at the clinical unit where the study took place, for 6 months after sending the study report. The products will then be destroyed, according to the PERITESCO SOP, unless otherwise instructed by the sponsor.

I.2. Clinical methods

I.2.1. Aim of the study

This study, carried out on cosmetic products which security was previously assessed by a toxicologist, was performed in order to confirm the safety of these products which will be used by a large amount of users under normal and reasonably foreseeable conditions of use.

The aim of the study was to confirm the ocular and peri-ocular acceptability of the investigational products under normal conditions of use, as foreseen by the sponsor, under ophthalmological control.

I.2.2. Relevancy

Cosmetic products can induce ocular, cutaneous or mucous discomfort and/or irritation signs.

The medical ophthalmologic and/or dermatological supervision of a group of subjects using a cosmetic product under normal conditions of use, the collection of functional signs and the observation of physical signs that appeared during the study period, as well as the analysis of the investigational products' imputability, enable the evaluation of the investigational products' safety, their tolerance, and the classification of their irritant potential.

The analysis of the standard deviation of each studied sign enabled the determination of the number of subjects to include in the study. The occurrence frequency standard deviations as well as physiological criteria enabled the determination of the duration of the study.

The number of subjects, the frequency of use and the duration of this study are satisfactory to study the tolerance of the investigational products.

Each investigational product was applied on the upper eyelid of both eyes. This is a normal use for this kind of product.

I.2.3. Study design

* Location: The study was carried out at the clinical unit of Paris.

* Design: The study was open and non-randomized.

15 subjects tested the investigational product "FARD A PAUPIERES REF. 701100 LOT 20091013-1" and 15 subjects tested the investigational product "FARD A PAUPIERES REF. 701102 LOT 20091013-2" according to the attribution table established by PERITESCO.

The attribution table is presented in Appendix III

* Timetable: - Clinical study start date: 6 November 2009
- Clinical study end date: 17 November 2009

I.2.4. Statistics

The statistical analysis was carried out in accordance with the statistical protocol mentioned in the study protocol (Procedures in Appendix I).

I.2.5. Subjects participating in the study

The following table gives information about the participation in the study of all the retained subjects:

	Number of subjects	Reason and day of occurrence
Recruited	30	
Non-Included	0	
Study premature withdrawals	0	
Not analyzed data	0	
Valid cases	**30**	

The average age of the subjects was:
Age: 50.3 ± 11.9 years old (extreme values: 24 - 64 years old).

These subjects were selected according to the following criteria:

I.2.5.1. Inclusion criteria

The voluntary subjects satisfied the following criteria:
- **Female,**
- **Aged from 18 to 65 years old,**
- **Presenting all types of skin,**
- **Presenting sensitive eyes** according to the following criteria:
  - tear film break-up time inferior to 10 seconds
  - and/or lid margin examination revealing a seborrheic hypersecretion with at least a slight intensity and/or a dysfunction or an atrophy of meibomius glands,
  - and/or subjects fulfilling the subjective criteria of organic ocular sensitivity without optical ocular sensitivity (photophobia, astigmatism, heterophoria, cataract or corneal scars),
- Able and willing to give their written participation consent,
- Affiliated to the social security in accordance to the recommendations of the French Act ("Loi Huriet": n° 88.1138 - 20.12.88) about the biomedical research.

I.2.5.2. Non-inclusion criteria

The voluntary subjects did not satisfy the following criteria:
- Taking part or willing to take part in another study during the study period,
- Who could not commit themselves on the absence of pregnancy or breaetfeeding during the study period,
- Presenting a medical history of medical or psychiatric illness or major surgery, suffering from acute or chronic or progressive illnesses, or presenting a dermatological or ophthalmological pathology likely to interfere with the data of the study,
- Unwilling to give their written informed consent,
- Who could not be contacted in case of emergency by phone,
- PERITESCO's employees,
- Who did not fulfill the above inclusion criteria.

I.2.5.3.Study premature withdrawal criteria

A subject included in the study was excluded if:

- The investigator judged that an adverse event was directly attributable to the investigational products,
- A hypersensitive reaction or an allergic reaction directly linked to the investigational products occurred,
- The subjects wanted to withdraw from the study, for whatever reason,
- The subject did not respect the study constraints,
- No adequacy during the study with the initially checked inclusion criteria,
- Lost to follow-up subject.

I.3. Protocol amendment

No protocol amendment was requested.

I.4. Protocol deviation

The study was conducted according to the protocol specifications.

However, the following deviation was observed:

Type of deviation / Subject n°	Number of subjects	Age	Type of subjects	Study duration	Number of applications (7 to 17)	Missing examination
Subject n°18*					6 applications	

* tested the investigational product "FARD A PAUPIERES REF. 701100 LOT 20091013-

This deviation had no consequence on the validity of the observed results.

II. STUDY SCHEDULE

II.1. Initial Time

The subjects received a daily-log where they recorded daily the use of the attributed investigational product and the possible signs felt or observed during the use, or any other information.

At T0: before any application of the attributed investigational product.

The ophthalmologist practiced:
* an ocular and peri-ocular functional signs investigation on both eyes,
* a biomicroscopic examination of ocular and peri-ocular structures on both eyes,
* a colorimetric examination of the cornea and the conjunctiva on right eye,
* a tear film break-up time measurement on right eye.

At T10 min: at least 10 minutes after the first application of the attributed investigational product.

The ophthalmologist practiced:
* an ocular and peri-ocular functional signs investigation on both eyes,
* a biomicroscopic examination of ocular and peri-ocular structures on both eyes,
* a colorimetric examination of the cornea and the conjunctiva on right eye,
* a tear film break-up time measurement on right eye.

Every observation was reported in the case report form.

II.2. Final Time

At T10 min: at least 10 minutes after the last application of the attributed investigational product, performed at the clinical unit.

The ophthalmologist practiced:
* an ocular and peri-ocular functional signs investigation on both eyes, analyzing the observations recorded in the daily-log,
* a biomicroscopic examination of ocular and peri-ocular structures on both eyes,
* a colorimetric examination of the cornea and the conjunctiva on right eye.

Every observation was reported in the case report form.

III. INVESTIGATIONAL PRODUCTS' APPLICATION

During the study, the investigational products were used as follows:

Application area	The upper eyelid of both eyes
Applied quantity	Necessary and sufficient for this kind of product
Frequency	Once to twice a day
Duration	8 days
Total number of applications on the whole panel	151 applications of the investigational product "FARD A PAUPIERES REF. 701100 LOT 20091013-1" 152 applications of the investigational product "FARD A PAUPIERES REF. 701102 LOT 20091013-2"
Conditions of use	The attributed investigational product was applied on the upper eyelid of both eyes, once to twice a day, during 8 days.
Constraints of the study	Allowed: (except when coming to the clinical unit for the control visits) - The usual cleansers and make-up removers, - The usual care products, - The light face make-up (powder and blusher), - The lip make-up with usual products. Not allowed: - Any other eye make-up, - Any modification of other cosmetic habits.

IV. ETHICAL AND JURIDICAL CONSIDERATIONS

IV.1. Ethics

The study, without any direct therapeutic aim, was carried out according to the most recent recommendations of the World Medical Association (Helsinki Declaration of 1964, and its successive updates). As this study is not concerned with the French Act n° 88-1138 of 20 December 1988, about the protection of subjects involved in biomedical research, and its successive updates, the most recent one being Act n° 2004-806 of 9 August 2004, the CPP (Comité de Protection des Personnes) authorization was not required. However, the principles of this Act were respected, with the exception of the recording of the subjects in a national file and the submission of the documents related to the study to an Independent Ethics Committee.

The investigational products did not contain and were not derived from material with specific risks as defined by the Commission decision n° 2000/418/CE, modified by the Commission decision n°2001/2/CE.

IV.2. Conditions to obtain the subjects' informed consents

On the inclusion day, the investigator or the person delegated by the investigator provided to the subject detailed information about the study modalities so that the subject could decide whether or not she wanted to participate in the study. The investigator or the person delegated by the investigator ensured that the subject obtained answers to any question she asked concerning her participation in the study.
Two copies of the informed consent were signed and dated by the subject and then checked, countersigned and dated by the investigator or the person delegated by the investigator.

IV.3. Insurance

The study is covered by the insurance contract subscribed by the sponsor, civil liability contract covering the studies on healthy subjects, by AIG Europe, contract n° 7.109.393.

Additionally, the investigating laboratory subscribed a civil liability insurance by AGF, contract n° 41 263 662.

IV.4. Study filing

The investigating laboratory retains a copy of the signed protocol and its possible amendments, of the signed financial agreement, of every version of the reports, the original case report forms and everything that was used to fill them in, the informed consent forms, the source documents and all the documents related to the study for a period of ten years after completion of the study. The whole of the computerized data of the study will be stored on an internal network for 10 years as well.

In accordance with PERITESCO SOP, the paper documents archiving is outsourced in Société Générale d'Archives (Archiving site address: Entrepôt GARONOR, E.P 405 Bât.1 Porte 3, 93617 Aulnay-sous-Bois Cedex). The traceability of the documents will be the responsibility of PERITESCO, who will keep updated records of their filing address. These documents will be available for inspection within a reasonable period by an authorized sponsor representative or by the regulatory authorities.

The investigating laboratory can destroy the study archiving file only if the sponsor has given a formal signed agreement.

IV.5. Confidentiality

The investigating laboratory grants the study sponsor for data monitoring or audit, the regulation authorities, as well as the ethical committee if need be, permission to have direct access to the study source documents without violation of data confidentiality.

The confidentiality of the personal data of the subjects participating in the study was maintained throughout the study, in accordance with the Act "Informatique et liberté" of 6 January 1978, and with the Helsinki Declaration. To ensure their anonymity, a five-letter code was attributed to each subject. This code was composed of the first three letters of their surname followed by the first two letters of their first name.

The results of this study are considered as confidential information. This information, or any part of it, will not be disclosed, submitted for common publication or be the object of industrial property, unless the sponsor agreement is obtained.

The results gained through this study will remain the property of the sponsor.

Neither publication nor communication could be made without the foremost agreement of the sponsor of the study.

IV.6. Quality policy

The investigating laboratory developed a quality audit program in order to guarantee that the in-life study and the study report comply with the protocol, the PERITESCO standard operating procedures and are in the spirit of the standards of Good Clinical Practices guidelines according to the International Conference on Harmonization (ICH Topic E6 « Note for guidance on Good Clinical Practice CPMP/ ICH/ 135/ 95 »), the FDA (62 FR 25692 of May 9, 1997), and the EEC (Directive n° 2001/20/CE of April 4, 2001).

A quality policy statement mentioning the audits carried out during the study is included in this report see "Certificate of Quality Policy".

IV.7. Quality control

The protocol, the case report forms, the preliminary results and the reports are systematically submitted to quality control in order to verify that they are correctly written, complete and coherent. These controls are performed and registered throughout the study and are the object of a statement in this report.

The raw data of the study (whether on hard or electronic copy) was submitted to quality control. The date and identification of the person who performed the control of the data are also registered.

RESULTS

V. SUBJECTS' CHARACTERISTICS

The following table presents the subjects' characteristics:

Characteristics		Percentage of subjects
Ocular criteria	Sensitive eyes	100.0
Skin type	Sensitive skin	53.3
	Non-sensitive skin	46.7
Skin nature	Oily skin	3.3
	Combination skin	73.3
	Normal skin	16.7
	Dry skin	6.7

The subjects' individual characteristics are presented in Appendix IV.

VI. FARD A PAUPIERES REF. 701100 LOT 20091013-1

VI.1. Clinical results

VI.1.1. Functional signs

No subject showed any functional sign at Initial Time T0 before the first application of the investigational product.

The following table presents the functional signs which appeared during the study

	Nb of occ	% of subjects	Freq %	Average intensity	Gr 1	Gr 2	Gr 3	Delay min	Duration min	Irritant Freq.%	Total irritation frequency
Ocular stinging	0	0.00	0.00	0.00	0	0	0	0.00	0.00	0.00	0.00
Ocular burning	0	0.00	0.00	0.00	0	0	0	0.00	0.00	0.00	
Foreign body feeling	0	0.00	0.00	0.00	0	0	0	0.00	0.00		
Blur	0	0.00	0.00	0.00	0	0	0	0.00	0.00		
Palpebral stinging or burning	1	6.67	0.66	1.00	1	0	0	3.00	0.50		
Other sign	0	0.00	0.00	0.00	0	0	0	0.00	0.00		

Key: Nb of occ Number of occurrences
Freq Frequency
Gr Grade

VII. FARD A PAUPIERES REF. 701102 LOT 20091013-2

VII.1. Clinical results

VII.1.1. Functional signs

No subject showed any functional sign at Initial Time T0 before the first application of the investigational product.

The following table presents the functional signs which appeared during the study:

	Nb of occ	% of subjects	Freq %	Average intensity	Gr 1	Gr 2	Gr 3	Delay min	Duration min	Irritant Freq.%	Total irritation frequency
Ocular stinging	0	0.00	0.00	0.00	0	0	0	0.00	0.00	0.00	0.00
Ocular burning	0	0.00	0.00	0.00	0	0	0	0.00	0.00	0.00	
Foreign body feeling	0	0.00	0.00	0.00	0	0	0	0.00	0.00		
Blur	0	0.00	0.00	0.00	0	0	0	0.00	0.00		
Palpebral stinging or burning	1	6.67	1.32	1.00	1	0	0	0.08	6.00		
Other sign	0	0.00	0.00	0.00	0	0	0	0.00	0.00		

Key: Nb of occ — Number of occurrences
Freq — Frequency
Gr — Grade

The ocular irritation occurrences regroup the following functional signs: ocular stinging and burning with an intensity and/or duration meaning an irritation.

The objective contact rate noticed by the investigator is 36.67% (of the applications).

The following table presents the subject who showed functional signs:

		Palpebral stinging or burning	
N°	CODE	Grade	Number
24	LAVSY	1	2

The individual results are presented in Appendix VI.

VII.1.2. Biomicroscopic examination

The following tables present the physical signs which appeared during the study.

		INITIAL TIME T0		INITIAL TIME T10 min					
		Average grade	Number of occurrences	Average grade	Number of occurrences	Grade 1	Grade 2	Grade 3	Bilateral occurrences
Seborrheic hypersecretion	right	1.00	15						
	left	1.00	15						
Dysfunction of meibomius glands	right	0.00	0						
	left	0.00	0						
Eyelids	right	0.00	0	0.00	0	0	0	0	0
	left	0.00	0	0.00	0	0	0	0	
Bulbar conjunctival redness	right	0.00	0	0.00	0	0	0	0	0
	left	0.00	0	0.00	0	0	0	0	
Conjunctival pallor	right	0.00	0						
	left	0.00	0						
Papillae	right	0.00	0						
	left	0.00	0						
Folliculosis	right	0.00	0						
	left	0.00	0						

		FINAL TIME T10 min					
		Average grade	Number of occurrences	Grade 1	Grade 2	Grade 3	Bilateral occurrences
Seborrheic hypersecretion	right	1.00	15	15	0	0	15
	left	1.00	15	15	0	0	
Dysfunction of meibomius glands	right	0.00	0	0	0	0	0
	left	0.00	0	0	0	0	
Eyelids	right	0.00	0	0	0	0	0
	left	0.00	0	0	0	0	
Bulbar conjunctival redness	right	0.00	0	0	0	0	0
	left	0.00	0	0	0	0	
Conjunctival pallor	right	0.00	0	0	0	0	0
	left	0.00	0	0	0	0	
Papillae	right	0.00	0	0	0	0	0
	left	0.00	0	0	0	0	
Folliculosis	right	0.00	0	0	0	0	0
	left	0.00	0	0	0	0	

The following table presents the number of subjects who showed corneal cells loss.

	Total number of corneal cells loss	Number	Surface (Grade from 0 to 10)	Depth (Grade from 0 to 3)
Initial Time T0	0			
Initial Time T10 min	0			
Final Time T10 min	0			

The following tables present the physical signs observed during the study.

*** Initial Time T0.**

N°	CODE	Seborrheic hypersecretion (Grade)
2	TOUFA	1
3	PAISA	1
5	PARNA	1
6	PUYDA	1
10	BELMA	1
12	SOULA	1
13	FERMA	1
14	AMESA	1
15	SACVI	1
17	RASCA	1
20	RIVSN	1
23	POLBR	1
24	LAVSY	1
27	MADFR	1
29	KOEIS	1

*** Final Time T10 min.**

N°	CODE	Seborrheic hypersecretion (Grade)
2	TOUFA	1
3	PAISA	1
5	PARNA	1
6	PUYDA	1
10	BELMA	1
12	SOULA	1
13	FERMA	1
14	AMESA	1
15	SACVI	1
17	RASCA	1
20	RIVSN	1
23	POLBR	1
24	LAVSY	1
27	MADFR	1
29	KOEIS	1

The individual results are presented in Appendix VI.

VII.2. Tear film break-up time measurement.

The comparison between individual levels of break-up times before and after application was carried out using a match paired Wilcoxon test (significativity threshold= 5%).

The following table presents the results:

	B.U.T. values (seconds)		Statistics	
	Initial Time T0	Initial Time T10 min	Initial Time T0 / Initial Time T10 min	Difference
Results	6.27 ± 1.62	6.00 ± 1.89	P = 0.575	Not significant

The results of the tear film break-up time measurements are presented in Appendix VI.

VII.3. Colorimetric examination of cornea and conjunctiva

The following tables present the colorimetric indexes obtained at different times:

* Initial Time T0.

. Surface

	Superior conjunctiva	Inferior conjunctiva	Superior cornea	Inferior cornea
Number of subjects with a grade different from 0	0	0	0	0
Rate (%)	0.00%	0.00%	0.00%	0.00%
Average conjunctival rate (%)	0.00%			
Average corneal rate (%)			0.00%	

. Depth

	Superior cornea	Inferior cornea
Number of subjects with a grade different from 0	0	0
Depth	0.00	0.00
Average depth	0.00	

* Initial Time T10 min.

. Surface

	Superior conjunctiva	Inferior conjunctiva	Superior cornea	Inferior cornea
Number of subjects with a grade different from 0	0	0	1 Grade 1	1 Grade 1 1 Grade 2
Rate (%)	0.00%	0.00%	0.67%	2.00%
Average conjunctival rate (%)	0.00%			
Average corneal rate (%)			1.33%	

. Depth

	Superior cornea	Inferior cornea
Number of subjects with a grade different from 0	1 Grade 1	1 Grade 1 1 Grade 2
Depth	1.00	1.50
Average depth	1.25	

* Final Time T10 min.

. Surface

	Superior conjunctiva	Inferior conjunctiva	Superior cornea	Inferior cornea
Number of subjects with a grade different from 0	0	0	0	2 Grade 1
Rate (%)	0.00%	0.00%	0.00%	1.33%
Average conjunctival rate (%)	0.00%			
Average corneal rate (%)			0.67%	

. Depth

	Superior cornea	Inferior cornea
Number of subjects with a grade different from 0	0	2 Grade 1
Depth	0.00	1.00
Average depth	1.00	

* Results of colorimetric global indexes of the investigational product (maximal values between the difference between the colorimetric index Initial Time T10 min and Initial Time T0 and between the difference between the colorimetric index Final Time T10 min and Initial Time T0):

	Results
Maximal conjunctival index	0.00%
Maximal corneal index	1.33%

The individual results are presented in Appendix VI.

VIII. ADVERSE EVENTS AND SERIOUS ADVERSE EVENTS

VIII.1.Definitions

VIII.1.1.Adverse event

The Good Clinical Practices (ICH Topic E6 « Note for guidance on Good Clinical Practice CPMP/ ICH/ 135/ 95 ») define an adverse event as any unfavourable and unintended sign, symptom or disease temporarily associated with the use of the investigational product, whether or not related to the investigational product.

VIII.1.2.Adverse product reaction

The Good Clinical Practices (ICH Topic E6 « Note for guidance on Good Clinical Practice CPMP/ ICH/ 135/ 95 ») define an adverse product reaction as any noxious and unintended response which relationship with the investigational product cannot be ruled out.

VIII.1.3.Serious adverse event or serious adverse product reaction

The Good Clinical Practices (ICH Topic E6 « Note for guidance on Good Clinical Practice CPMP/ ICH/ 135/ 95 ») define a serious adverse event or serious adverse product reaction as any adverse event or adverse product reaction which:

- Results in death
- Is life-threatening
- Requires inpatient hospitalization or prolongation of existing hospitalization
- Results in persistent or significant disability
- Is a congenital anomaly defect

VIII.2.In case of adverse event

In case of adverse event, an adverse event form is filled in by the investigator and transmitted to the study monitor, immediately in case of a serious adverse event, or within the following 48 hours.
The adverse event form mentions the date of beginning of the event, its severity, the follow-up modalities and the conclusion of the investigator concerning the link between the event and the investigational product.

If a further examination test has to be carried out following the occurrence of an adverse event, a protocol amendment is written. This amendment indicates the type of test, its principle, the scheduled inclusion date, the duration of the test, the number and the identification of the subjects. The subjects keep the same identification code as for the original study.

VIII.3.Description of Adverse Events occurring during the study

In this study, no adverse event or serious adverse event was observed by the investigating ophthalmologists.

DISCUSSION - CONCLUSION

IX. SUBJECTS' CHARACTERISTICS

The following table presents the subjects' characteristics:

Characteristics		Percentage of subjects
Ocular criteria	Sensitive eyes	100.0
Skin type	Sensitive skin	53.3
	Non-sensitive skin	46.7
Skin nature	Oily skin	3.3
	Combination skin	73.3
	Normal skin	16.7
	Dry skin	6.7

X. DISCUSSION

X.1. Investigational products ocular acceptability

X.1.1. FARD A PAUPIERES REF. 701100 LOT 20091013-1)

The results are carried out in 15 subjects.

(a) Functional signs and contact rate:

The functional signs are classified in analytical categories. This classification is made according to the opinion and experience of the investigator, and according to the nature of the signs, their intensity, duration and their physiopathology.

The following table presents the frequency of functional signs.

Functional signs	Frequency
Ocular irritation functional signs	0.00%
Mechanical ocular functional signs	0.00%
Physico-chemical ocular functional signs	0.00%
Palpebral irritation functional signs	0.00%
Palpebral discomfort functional signs	0.66%

The investigational product did not induce the appearance of any ocular functional signs.

The investigational product induced the appearance of palpebral discomfort functional signs (palpebral stinging or burning), of slight intensity, of short duration (0.50 minute on average), occurring in 1 subject with sensitive eyes, combination and non-sensitive skin, with a frequency of 0.66%, low for this kind of product.

The contact rate of the investigational product with the eyes is of 26.67%, which is normal for this kind of product.

The analysis of the impact the investigational product has on the ocular structures enables to determine the following characteristics:

Investigational product kinetics	Normal contact
Remanence on tear film	Unknown
Corneal epithelium	Non toxic
Bulbar conjunctiva	Non toxic
Conjunctival epithelium	Non toxic
Lid margin	Non toxic
Functional tolerance	Good
Investigational product repeated use	No foreseeable risk

The ocular functional and physical signs analysis enables to consider that the investigational product shows a practically nil ocular irritant potential, which is very good for this type of product.

The palpebral functional and physical signs analysis enables to consider that the investigational product shows a practically nil palpebral irritant potential, which is very good for this type of product.

X.1.2. FARD A PAUPIERES REF. 701102 LOT 20091013-2

The results are carried out in 15 subjects.

(a) Functional signs and contact rate:

The functional signs are classified in analytical categories. This classification is made according to the opinion and experience of the investigator, and according to the nature of the signs, their intensity, duration and their physiopathology.

The following table presents the frequency of functional signs.

Functional signs	Frequency
Ocular irritation functional signs	0.00%
Mechanical ocular functional signs	0.00%
Physico-chemical ocular functional signs	0.00%
Palpebral irritation functional signs	0.00%
Palpebral discomfort functional signs	**1.32%**

The investigational product did not induce the appearance of any ocular functional signs.

The investigational product induced the appearance of palpebral discomfort functional signs (palpebral stinging or burning), of slight intensity, of short duration (6.00 minutes on average), occurring in 1 subject with sensitive eyes, combination and sensitive skin, with a frequency of 1.32%, normal for this kind of product.

The contact rate of the investigational product with the eyes is of 36.67%, which is normal for this kind of product.

The following table regroups the subject who presented functional signs:

		Palpebral stinging or burning	
N°	CODE	Grade	Number
24	LAVSY	1	2

(b) Physical signs:

The investigational product did not induce the appearance of any ocular physical sign, which shows an **absence of conjunctival and corneal irritant potential.**

The investigational product did not induce the appearance of any palpebral physical sign, which shows an **absence of palpebral irritant potential.**

(c) Additional ocular examinations:

(i) Tear film break-up time measurement.

The investigational product does not significantly modify the tear film stability (the B.U.T. value decreases from 6.27 to 6.00).
It therefore respects the ocular surface defense abilities.

(ii) Colorimetric examination of cornea and conjunctiva.

The colorimetric examinations revealed a maximal corneal index of 1.33% and a maximal conjunctival index of 0.00%, which shows an **absence of toxicity to the conjunctiva and a very slight toxicity to the cornea.**

(d) Results summary:

The clinical examinations revealed an ocular irritation rate of 1.33/2,000 i.e. 0.07% and an ocular comfort rate of 498.68/500 i.e. 99.74%.

The following table presents the results:

Items		Results
Functional signs	Irritant ocular	0.00%
	Mechanical ocular	0.00%
	Physico-chemical ocular	0.00%
	Palpebral irritation	0.00%
	Palpebral discomfort	**1.32%**
Physical signs	Bulbar conjunctival redness	No occurrence
	Corneal cells loss	No occurrence
B.U.T.		Initial Time T0: 6.27 seconds Initial Time T10 min: 6.00 seconds Not significant modification
Colorimetry	Maximal conjunctival index	0.00%
	Maximal corneal index	**1.33%**
Ocular irritation rate		0.07%
Ocular comfort rate		99.74%

The analysis of the impact the investigational product has on the ocular structures enables to determine the following characteristics:

Investigational product kinetics	Normal contact
Remanence on tear film	Unknown
Corneal epithelium	Very slightly toxic
Bulbar conjunctiva	Non toxic
Conjunctival epithelium	Non toxic
Lid margin	Non toxic
Functional tolerance	Good
Investigational product repeated use	No foreseeable risk

The ocular functional and physical signs analysis enables to consider that the investigational product shows a very slight ocular irritant potential, which is normal for this type of product.

The palpebral functional and physical signs analysis enables to consider that the investigational product shows a practically nil palpebral irritant potential, which is very good for this type of product.

XI. CONCLUSION

In the conditions of the study, the investigational product "FARD A PAUPIERES REF. 701100 LOT 20091013-1":

- presents a **very good ocular comfort, a very good ocular safety and a very good global ocular tolerance** in subjects presenting sensitive eyes,
- presents a **good palpebral tolerance.**

In the conditions of the study, the investigational product "FARD A PAUPIERES REF. 701102 LOT 20091013-2":

- presents a **very good ocular comfort, a good ocular safety and a good global ocular tolerance** in subjects presenting sensitive eyes,
- presents a **good palpebral tolerance.**

XII. INVESTIGATORS' STATEMENT AND SIGNATURES

The study described in this report was carried out according to the protocol, to PERITESCO SOP and to ICH Topic E6 « Note for guidance on Good Clinical Practice CPMP/ ICH/ 135/ 95 ».

I declare that this report is an accurate account of the procedures followed during the study, and constitutes a true and faithful record of my own conclusions.

Dr. M. PERICOI
Ophthalmologist
Director and Principal Investigator
PERITESCO
Date: 30.12.09

Dr. T. GAMAR
Ophthalmologist
Co-Investigator
PERITESCO
Date: 31.12.09

Personal Care Products Council
Committed to Safety, Quality & Innovation

Memorandum

TO: F. Alan Andersen, Ph.D.
Director - COSMETIC INGREDIENT REVIEW (CIR)

FROM: Halyna Breslawec, Ph.D.
Industry Liaison to the CIR Expert Panel

DATE: October 17, 2012

SUBJECT: Information on a Products Containing Polybutylene and/or Polyethylene Terephthalate

Consumer Product Testing Co. 2007. Ophthalmological in-use safety evaluation of mascara containing 4.8% Polyethylene Terephthalate and 4.2% Polybutylene Terephthalate. Experiment Reference Number: C06-1021.01-.03.

Consumer Product Testing Co. 2011. Repeated insult patch test of an eyeliner containing 1.5% Polyethylene Terephthalate. Experiment Reference Number: C11-2066.03.

The ingredients in these products are described as solid, particles cut from sheets of plastic.

1101 17th Street, N.W., Suite 300 | Washington, D.C. 20036-4702 | 202.331.1770 | 202.331.1969 (fax) | www.personalcarecouncil.org

Consumer Product Testing Co.

EST. 1975

FINAL REPORT

CLIENT: [redacted]

ATTENTION: [redacted]

TEST: Ophthalmological In-Use Safety Evaluation
Protocol No.: BNIW-138

TEST MATERIALS:
.01 Blue RD#35080
.02 Brown RD#35076
.03 Purple RD#35077

Mascara contains 4.8% Polyethylene Terephthalate and 4.2% Polybutylene Terephthalate

EXPERIMENT REFERENCE NUMBER: C06-1021.01 - .03

Charles J. Kronengold, M.D.
Board Certified Ophthalmologist

Joy Frank, R.N.
Executive Vice President, Clinical Evaluations

Report Date: 1.3.07

70 New Dutch Lane • Fairfield, New Jersey 07004-2514 • (973) 808-7111 • Fax (973) 808-7244

Consumer Product Testing Co.

EST. 1965

QUALITY ASSURANCE UNIT STATEMENT

Study No.: C06-1021.01-.03

The objective of the Quality Assurance Unit (QAU) is to monitor the conduct and reporting of clinical laboratory studies. These studies have been performed with adherence to the applicable ICH Guideline E6 for Good Clinical Practice and requirements provided for in 21 CFR parts 50 and 56 and in accordance to standard operating procedures and applicable protocols. The QAU maintains copies of study protocols and standard operating procedures and has inspected this study. All data pertinent to this study will be stored in the Consumer Product Testing Company archive, unless specified otherwise, in writing by the Sponsor.

Quality Assurance personnel involved:

Quality Assurance — Date: 1.3.07

The representative signature of the Quality Assurance Unit signifies that this study has been performed in accordance with standard operating procedures and the applicable study protocol as well as any government regulations regarding such procedures and protocols.

70 New Dutch Lane • Fairfield, New Jersey 07004-2514 • (973) 808-7111 • Fax (973) 808-7234
Clinical • Toxicology • Analytical Chemistry • Microbiology

Objective: To evaluate the safety and ocular irritation potential of a mascara following repetitive, daily use conditions.

Participants: Thirty (30) female subjects, ranging in age from 18 to 62 years, were selected for this study. Twenty-nine (29) subjects completed the study. Subject #15 discontinued her participation due to personal reasons, unrelated to product use.

Inclusion Criteria:

a. Subjects between the ages of 18 and 65 years, who are free from any disease which may affect the test results.
b. Must be regular users of a mascara.
c. Approximately 50% of subjects must be contact lens wearers, 50% will be non-contact lens wearers and 50% self-perceived sensitive eyes.
d. Pre-screening ophthalmic examination of participants to ensure eye health and if appropriate, the correct fit and condition of their contact lenses.
e. Completion of a Medical History form and the understanding and signing of an Informed Consent form.
f. Considered reliable and Capable of following directions.

Exclusion Criteria:

a. Ill health or taking medication(s), other than birth control, which could influence the purpose, integrity or outcome of the study.
b. Females who are pregnant or lactating.

Test Materials:

.01 Blue RD#35080
.02 Brown RD#35076
.03 Purple RD#35077

Study Schedule:

Initiation Date	Completion Date
November 8, 2006	December 6, 2006

Methodology: Prior to acceptance, each subject received a qualifying ophthalmic examination by a Board Certified Ophthalmologist to ensure eye health and if appropriate, the correct fit of their contact lenses.

Methodology (continued):

The Ophthalmologist evaluated (by gross and slit lamp examination) the subjects' eyelids, corneas, conjunctivae, anterior chambers and pupillary reactions, as well as visual acuity. The parameters for the examination were:

Visual acuity
Pupillary Response
External Slit Lamp:
- Palpebral conjunctivae
- Bulbar conjunctivae
- Fornices
- Cornea abnormalities

The panel was comprised of sixteen (16) soft and one (1) hard contact lens wearers. The remaining thirteen (13) subjects were non-contact lens wearers. The panel was also comprised of eighteen (18) subjects with self-perceived sensitive skin.

After completion of the ophthalmic examination, acceptable candidates received the test product and were instructed to apply the test product daily for four (4) weeks, according to the following directions:

Instructions:

Discontinue the use of your current mascara and use only the product provided for the duration of this study. You are permitted to wear your own makeup products for the duration of this study.

Do not introduce any new cosmetic brands or products during the test period.

Please do not wear any eye makeup to the clinic for eye examinations.

Keep out of the reach of children. Do not let anyone else use the test product.

USAGE DIRECTIONS:

Use in place of your current mascara, at least once (1) a day.

The time of application must be recorded on the daily diary.

Report any adverse reactions or problems immediately to the laboratory staff.

Methodology (continued):

To document compliance, subjects were required to maintain a daily diary to record each use.

A comprehensive ocular examination, as previously described, was repeated for each subject after four (4) weeks of product usage.

Daily diaries were reviewed for completeness, prior to dismissal of the subjects.

Adverse Reactions:

There were no adverse events reported.

Results:

All ophthalmological examinations remained within normal limits throughout the test interval.

All completed ophthalmologic examination records and daily diaries are provided under separate cover.

Subject demographics are listed in Table 1.

Summary:

Under the conditions of this study, Test Materials: Blue RD#35080, Brown RD#35076, and Purple RD# 35077 did not indicate a potential for ophthalmologic irritation or hypersensitivity and can be considered safe for use by both contact lens and non-contact lens wearers as well as individuals with sensitive eyes.

Table 1

Subject Demographics

Subject Number	Initials	Age	Contact Lens	Sensitivity
1	JS	48	Non	Non
2	EG	50	Non	Sensitive
3	MR	46	Soft	Non
4	PL	55	Non	Sensitive
5	ML	48	Soft	Sensitive
6	KL	19	Soft	Sensitive
7	CY	19	Non	Non
8	LR	49	Non	Non
9	CP	44	Soft	Sensitive
10	RC	19	Non	Sensitive
11	NK	49	Soft	Non
12	LG	39	Soft	Sensitive
13	KM	28	Soft	Non
14	RH	53	Soft	Sensitive
15	NS	18	Soft	Non
16	LB	60	Soft	Sensitive
17	CD	41	Soft	Sensitive
18	EF	35	Soft	Sensitive
19	PJ	49	Non	Non
20	JD	46	Non	Non
21	JB	57	Non	Sensitive
22	MQ	39	Soft	Non
23	SC	58	Non	Non
24	MA	47	Non	Non
25	DF	50	Soft	Sensitive
26	MD	48	Non	Sensitive
27	HT	62	Soft	Sensitive
28	JS	60	Non	Sensitive
29	JD	31	Soft	Sensitive
30	LD	56	Hard	Sensitive

Consumer Product Testing Co.

FINAL REPORT

CLIENT:

ATTENTION:

TEST: Repeated Insult Patch Test
Protocol No.: 1.01L

TEST MATERIAL: EYELINER - ENG050918-0.1.1.1, PEN3-48-1

Contains 1.5% Polyethylene Terephthalate

EXPERIMENT REFERENCE NUMBER: C11-2066.03

Reviewed by: Richard R. Eisenberg, M.D.
Medical Director
Board Certified Dermatologist

Approved by: Michael Caswell, Ph.D., C.C.R.C., C.C.R.A.
Director, Clinical Evaluations

Approved by: Joy Frank, R.N.
Executive Vice President, Clinical Evaluations

70 New Dutch Lane • Fairfield, New Jersey 07004-2514 • (973) 808-7111 • Fax (973) 808-7234

Consumer Product Testing Co.

QUALITY ASSURANCE UNIT STATEMENT

Study Number: C11-2066.03

The Consumer Product Testing Company, Incorporated (CPTC) Quality Assurance Unit (QAU) is responsible for monitoring the conduct, content and reporting of all clinical laboratory studies that are conducted at CPTC.

This study has been conducted in accordance with ICH Guideline E6 for *Good Clinical Practice*, the requirements of 21 CFR Parts 50 and 56, other applicable regulations, CPTC Standard Operating Procedures, and the approved Study Protocol.

The CPTC QAU has reviewed all data, records, and documents relating to this study and also this Final Report. The following QAU representative signature certifies that all data, records, and documents relating to this study and also this Final Report have been reviewed and are deemed to be acceptable, and the study conforms to all of the requirements as indicated above.

All records and documents pertaining to the conduct of this study shall be retained in the CPTC archives for a minimum of ten (10) years. At any time prior to the completion of the tenth archival year, a Sponsor may submit a written request to the CPTC QA Department to obtain custody of study records once the CPTC archive period has been completed. This transfer shall be performed at the Sponsor's expense. In the absence of a written request, study-related records shall be destroyed at the end of the CPTC archive period in a manner that renders them useless.

Quality Assurance Representative

7/18/11
Date

70 New Dutch Lane • Fairfield, New Jersey 07004-2514 • (973) 808-7111 • Fax (973) 808-7234
Clinical • Toxicology • Analytical Chemistry • Microbiology

Objective: To determine by repetitive epidermal contact the potential of a test material to induce primary or cumulative irritation and/or allergic contact sensitization.

Participants: One hundred fourteen (114) qualified subjects, male and female, ranging in age from 16 to 78 years, were selected for this evaluation. One hundred seven (107) subjects completed this study. The remaining subjects discontinued their participation for various reasons, none of which were related to the application of the test material.

Inclusion Criteria:

a. Male and female subjects, age 16[a] and over.
b. Absence of any visible skin disease which might be confused with a skin reaction from the test material.
c. Prohibition of use of topical or systemic steroids and/or antihistamines for at least seven days prior to study initiation.
d. Completion of a Medical History form and the understanding and signing of an Informed Consent form.
e. Considered reliable and capable of following directions.

Exclusion Criteria:

a. Ill health.
b. Under a doctor's care or taking medication(s) which could influence the outcome of the study.
c. Females who are pregnant or nursing.
d. A history of adverse reactions to cosmetics or other personal care products.

Test Material: EYELINER - ENG050918-0.1.1.1, PEN3-48-1

Study Schedule:

Panel #	Initiation Date	Completion Date
20110158	May 16, 2011	June 23, 2011
20110162	May 16, 2011	June 24, 2011

[a]With parental or guardian consent

Methodology:

The upper back between the scapulae served as the treatment area. Approximately 0.2 g of the test material, or an amount sufficient to cover the contact surface, was applied to the 1" x 1" absorbent pad portion of a clear adhesive dressing and allowed to volatilize for several minutes. This was then applied to the appropriate treatment site to form a semi-occlusive patch.

Induction Phase:

Patches were applied three (3) times per week (e.g., Monday, Wednesday, and Friday) for a total of nine (9) applications. The site was marked to ensure the continuity of patch application. Following supervised removal and scoring of the first Induction patch, participants were instructed to remove all subsequent Induction patches at home, twenty-four hours after application. The evaluation of this site was made again just prior to re-application. If a participant was unable to report for an assigned test day, one (1) makeup day was permitted. This day was added to the Induction period.

With the exception of the first supervised Induction Patch reading, if any test site exhibited a moderate (2-level) reaction during the Induction Phase, application was moved to an adjacent area. Applications were discontinued for the remainder of this test phase, if a moderate (2-level) reaction was observed on this new test site. Applications would also be discontinued if marked (3-level) or severe (4-level) reactivity was noted.

Rest periods consisted of twenty-four hours following each Tuesday and Thursday removal, and forty-eight hours following each Saturday removal.

Challenge Phase:

Approximately two (2) weeks after the final Induction patch application, a Challenge patch was applied to a virgin test site adjacent to the original Induction patch site, following the same procedure described for Induction. The patch was removed and the site scored at the clinic twenty-four and seventy two hours post-application.

Methodology (continued):

Evaluation Criteria (Erythema and additional Dermal Sequelae):

0	=	No visible skin reaction	E	=	Edema
0.5	=	Barely perceptible	D	=	Dryness
1	=	Mild	S	=	Staining
2	=	Moderate	P	=	Papules
3	=	Marked	V	=	Vesicles
4	=	Severe	B	=	Bullae
			U	=	Ulceration
			Sp	=	Spreading

Erythema was scored numerically according to this key. If present, additional Dermal Sequelae were indicated by the appropriate letter code and a numerical value for severity.

Results:

The results of each participant are appended (Table 1).

Observations remained negative throughout the test interval.

Subject demographics are presented in Table 2.

Summary:

Under the conditions of this study, test material, EYELINER - ENG050918-0.1.1.1, PEN3-48-1, did not indicate a potential for dermal irritation or allergic contact sensitization.

Table 1
Panel #20110158

Individual Results

EYELINER - ENG050918-0.1.1.1, PEN3-48-1

Subject Number	Induction Phase									Virgin Challenge Site	
	24*hr	1	2	3	4	5	6	7	8	9	24*hr	72 hr
1	0	0	0	0	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0	0	0	0	0
3	0	0	0	0	0	0	0	0	0	0	0	0
4	0	0	0	0	0	0	0	0	0	0	0	0
5	0	0	0	0	0	0	0	0	0	0	0	0
6	0	0	0	0	0	0	0	0	0	0	0	0
7	0	0	0	0	0	0	0	0	0	0	0	0
8	0	0	0	0	0	0	0	0	0	0	0	0
9	0	0	0	0	0	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0	0	0	0	0	0
11	0	0	0	0	0	0	0	0	0	0	0	0
12	0	0	0	0	0	0	0	0	0	0	0	0
13	0	0	0	0	0	0	0	0	0	0	0	0
14	0	0	0	0	0	0	0	0	0	0	0	0
15	0	0	0	0	0	0	0	0	0	0	0	0
16	0	0	0	0	0	0	0	0	0	0	0	0
17	0	0	0	0	0	0	0	0	0	0	0	0
18	0	0	0	0	0	0	0	0	0	0	0	0
19	0	0	0	0	0	0	0	0	0	0	0	0
20	0	0	0	0	0	0	0	0	0	0	0	0
21	0	0	0	0	0	0	0	0	0	0	0	0
22	0	0	0	0	0	0	0	0	0	0	0	0
23	0	0	0	0	0	0	0	0	0	0	0	0
24	0	0	0	0	0	0	0	0	0	0	0	0
25	0	0	0	0	0	0	0	0	0	0	0	0
26	0	0	0	0	0	0	0	0	0	0	0	0
27	0	0	0	0	0	0	0	0	0	0	0	0
28	0	0	0	0	0	0	0	0	0	0	0	0
29	0	0	0	0	0	0	0	0	0	0	0	0

24* = Supervised removal of 1st Induction and Challenge Patch

Table 1
(continued)
Panel #20110158

Individual Results

EYELINER - ENG050918-0.1.1.1, PEN3-48-1

Subject Number	Induction Phase									Virgin Challenge Site	
	24*hr	1	2	3	4	5	6	7	8	9	24*hr	72 hr
30	0	0	0	0	0	0	0	0	0	0	0	0
31	0	0	0	0	0	0	0	0	0	0[m]	0	0
32	0	0	0	0	0	0	0	0	0	0	0	DNC
33	0	0	0	0	0	0	0	0	0	0	0	0
34	0	0	0	0	0	0	0	0	0	0	0	0
35	0	0	0	0	0	0	0	0	0	0	0	0
36	0	0	0	0	0	0	0	0	0	0	0	0
37	0	0	0	0	0	0	0	0	0	0	0	0
38	0	0	0	0	0	0	0	0	0	0	0	0
39	0	0	0	0	0	0	0	0	0	0	0	0
40	0	0	0	0	0	0	0	0	0	0	0	0
41	0	0	0	0	0	0	0	0	0	0	0	0
42	0	0	0	0	0	0	0	0	0	0	0	0
43	0	0	0	0	0	0	0	0	0	0	0	0
44	0	0	0	0	0	0	0	0	0	0	0	0
45	0	0	0	0	0	0	0	0	0	0	0	0
46	0	0	0	0	0	0	0	0	0	0	0	0
47	0	0	0	0	0	0	0	0	0	0	0	0
48	0	0	0	0	0	0	0	0	0	0	0	0
49	0	0	0	0	0	0	0	0	0	0	0	0
50	0	0	0	0	0	0	0	0	0	0	0	0
51	0	0	0	0	0	0	0	0	0	0	0	0
52	0	0	0	0	0	0	0	0	0	0	0	0
53	0	0	0	0	0	0	0	0	0	0	0	0
54	0	0	DID NOT COMPLETE STUDY									
55	0	0	0	0	0	0	0	0	0	0	0	0
56	0	0	0	0	0	0	0	0	0	0	0	0

24* = Supervised removal of 1st Induction and Challenge Patch
DNC = Did not complete study
m = Additional makeup day granted at the discretion of the clinic supervisor

Table 1
(continued)
Panel #20110162

Individual Results

EYELINER - ENG050918-0.1.1.1, PEN3-48-1

Subject Number	Induction Phase										Virgin Challenge Site	
	24*hr	1	2	3	4	5	6	7	8	9	24*hr	72 hr
1	0	0	0	0	0	0	0	0	0	0	0	0
2	0	0	0	0	0	0	0	0	0	0	0	0
3	0	0	0	0	0	0	0	0	0	0	0	0
4	0	0	0	0	0	0	0	0	0	0	0	0
5	0	0	0	0	0	0	0	0	0	0	0	0
6	0	0	0	0	0	0	0	0	0	0	0	0
7	0	0	0	0	0	0	0	0	0	0	0	0
8	0	0	0	0	0	0	0	0	0	0	0	0
9	0	0	0	0	0	0	0	0	0	0	0	0
10	0	0	0	0	0	0	0	0	0	0	0	0
11	0	0	0	0	0	0	0	0	0	0	0	0
12	0	0	0	0	0	0	0	0	0	0	0	0
13	0	0	0	0	0	0	0	0	0	0	0	0
14	0	0	0	0	0	0	0	0	0	0	0	0
15	0	0	0	0	0	0	0	0	0	0	0	0
16	0	0	0	0	0	0	0	0	0	0	0	0
17	0	0	0	0	0	0	0	0	0	0	0	0
18	0	0	0	0	0	0	0	0	0	0	0	0
19	0	0	0	0	0	0	0	0	0	0	0	0
20	0	0	0	0	0	0	0	0	0	0	0	0
21	0	0	0	0	0	0	0	0	0	0	0	0
22	0	0	0	0	0	0	0	0	0	0	0	0
23	0	0	0	0	0	0	0	0	0	0	0	0
24	----------DID NOT COMPLETE STUDY----------											
25	0	0	0	0	0	0	0	0	0	0	0	0
26	0	0	0	----------DID NOT COMPLETE STUDY----------								
27	0	0	0	0	0	0	0	0	0	0	0	0
28	-	0	----------DID NOT COMPLETE STUDY----------									
29	----------DID NOT COMPLETE STUDY----------											

24* = Supervised removal of 1st Induction and Challenge Patch
- = Subject not present for supervised removal

Table I
(continued)
Panel #20110162

Individual Results

EYELINER - ENG050918-0.1.1.1, PEN3-48-1

Subject Number	24*hr	Induction Phase 1	2	3	4	5	6	7	8	9	Virgin Challenge Site 24*hr	72 hr
30	0	0	0	0	0	0	0	0	0	0	0	0
31	0	0	0	0	0	0	0	0	0	0	0	0
32	0	0	0	0	0	0	0	0	0	0	0	0
33	0	0	0	0	0	0	0	0	0	0	0	0
34	0	0	0	0	0	0	0	0	0	0	0	0
35	0	0	0	0	0	0	0	0	0	0	0	0
36	0	0	0	0	0	0	0	0	0	0	0	0
37	0	0	0	0	0	0	0	0	0	0	0	0
38	0	0	0	0	0	0	0	0	0	0	0	0
39	0	0	0	0	0	0	0	0	0	0	0	0
40	0	0	0	0	0	0	0	0	0	0	0	0
41	0	0	0	0	0	0	0	0	0	0	0	0
42	0	0	0	0	0	0	0	0	0	0	0	0
43	0	0	0	0	0	0	0	0	0	0	0	0
44	0	0	0	0	0	0	0	0	0	0	0	0
45	0	0	0	0	0	0	0	0	0	0	0	0
46	0	0	0	0	0	0	0	0	0	0	0	0
47	0	0	0	0	0	0	0	0	0	0	0	0
48	0	0	0	0	0	0	0	0	0	0	0	0
49	0	0	0	0	0	0	0	0	0	0	0	0
50	0	0	0	0	0	0	0	0	0	0	0	0
51	0	0	0	0	0	0	0	0	0	0	0	0
52	0	0	0	0	0	0	0	0	0	0	0	0
53	0	0	0	0	0	0	0	0	0	0	0	0
54	-	0	0	0	0	0	0	0	0	0	0	0
55	0	0	0	0	0	0	0	0	0	----------	DNC	----------
56	0	0	0	0	0	0	0	0	0	0	0	0
57	0	0	0	0	0	0	0	0	0	0	0	0
58	0	0	0	0	0	0	0	0	0	0	0	0

24* = Supervised removal of 1st Induction and Challenge Patch
DNC = Did not complete study
- = Subject not present for supervised removal

Table 2
Panel #20110158

Subject Demographics

Subject Number	Initials	Age	Sex
1	DEF	55	F
2	FMM	54	F
3	RMS	60	F
4	GCL	60	F
5	E-T	52	F
6	PAW	49	F
7	P-G	57	F
8	MDV	29	F
9	FPS	59	F
10	EJK	66	F
11	CLK	65	M
12	MFH	51	M
13	CSE	35	F
14	TEP	45	F
15	HEM	26	F
16	DRL	66	F
17	RWS	64	M
18	G-S	22	F
19	MVM	20	M
20	HLG	35	M
21	A-V	64	F
22	K-P	55	F
23	P-T	50	F
24	K-T	21	M
25	S-T	54	M
26	C-L	59	M
27	S-M	41	F
28	GFM	52	M
29	SAV	48	F

Table 2
(continued)
Panel #20110158

Subject Demographics

Subject Number	Initials	Age	Sex
30	CAS	50	F
31	GMS	68	F
32	MEM	57	M
33	VMP	21	F
34	TAF	62	F
35	SCV	44	M
36	XZZ	67	M
37	DCS	53	M
38	LAV	63	F
39	SMV	55	F
40	B-O	47	F
41	LAT	45	F
42	EBF	39	F
43	MKP	22	F
44	SMP	27	F
45	J-R	37	F
46	MMM	59	F
47	M-J	44	F
48	FAP	43	M
49	ELJ	70	F
50	EAH	47	M
51	LAM	51	F
52	M-B	49	F
53	C-C	43	F
54	MAM	37	F
55	LJD	31	F
56	DJD	52	F

Table 2
(continued)
Panel #20110162

Subject Demographics

Subject Number	Initials	Age	Sex
1	WCD	64	M
2	JEV	50	M
3	N-H	60	F
4	AES	73	F
5	F-B	76	M
6	MMH	72	F
7	MLA	53	F
8	M-M	77	F
9	KMB	68	F
10	A-M	41	F
11	GJM	73	F
12	R-O	78	M
13	NSN	67	F
14	G-P	46	F
15	JJB	22	M
16	DLB	47	F
17	A-N	54	F
18	TMC	38	F
19	ZYS	45	F
20	JTB	24	F
21	BME	77	F
22	A-D	52	F
23	DMP	39	F
24	LMK	71	F
25	GDS	42	F
26	J-J	28	F
27	MHO	22	M
28	J-J	30	F
29	DMC	53	F

Table 2
(continued)
Panel #20110162

Subject Demographics

Subject Number	Initials	Age	Sex
30	D-D	16	M
31	ULH	39	F
32	B-D	38	F
33	MLL	59	F
34	P-S	33	F
35	MPS	16	F
36	SLB	61	F
37	TJM	20	M
38	DWB	61	M
39	NJM	73	M
40	J-D	64	F
41	JWB	34	M
42	DAC	57	F
43	M-A	57	M
44	D-S	74	F
45	T-D	69	F
46	MAE	44	M
47	R-C	40	F
48	JVR	65	F
49	MJL	26	M
50	CEA	34	F
51	JAB	57	F
52	G-S	47	F
53	CAP	45	M
54	SCK	58	F
55	R-H	33	F
56	JLL	59	M
57	FTC	51	M
58	DDR	59	F

From: David Steinberg [mailto:davidpreserve@comcast.net]
Sent: Friday, August 24, 2012 10:51 AM
To: Alan Andersen
Subject: CIR-PET

Dear Alan.

I have been deeply concerned about the safety of PET in cosmetics since the industry started using PET glitter. This should be removed from the market as an unapproved color additive, but that is a different issue.

However, that is not my concern. My issue is the PET is chopped up (using a mechanical knife) to give glitter its jagged edges and allows the PET to adhere to the skin, hair, etc. If this gets in the eye, only a trained doctor should remove it as it will scratch the cornea causing permanent damage. I spoke to both Linda and Stan about this ages ago and suggested all products that contain glitter have this required warning. "If this gets into your eye, do not attempt to remove it yourself, seek medical attention immediately."

Nothing happened!

See you in September.

David

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Notes from Interview with
Dr. Stephen Glasser
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The eye lid has the thinnest skin of the body. There are a high number of blood vessels and is extra sensitive.

The cornea is the most sensitive part of the body.

It is not possible to pass through the epithelium at 0.004 mm. However, it can scratch the surface and open the wound up to secondary infection.

Many of the problems are dermatitis to the eye lid and not in the eye. The eye can clear out most particles with tears (from the edges of the lids) and blinking. Blinking can also push particle onto or into the cornea.

Many problems are due to make up not being removed before bed.

Hairsprays with glitter were a problem. Not due to spraying into the face but moving through the cloud after spraying.

Not seeing many problems here in DC. However, it is suspected that there are more incidents in areas where there are more younger adults or near the theatre districts.

2012 VCRP Data for Modified Terephthalate Polymers

03B - Eyeliner	POLYBUTYLENE TEREPHTHALATE	2
03C - Eye Shadow	POLYBUTYLENE TEREPHTHALATE	1
04E - Other Fragrance Preparation	POLYBUTYLENE TEREPHTHALATE	2
07E - Lipstick	POLYBUTYLENE TEREPHTHALATE	1
07I - Other Makeup Preparations	POLYBUTYLENE TEREPHTHALATE	3
08D - Nail Extenders	POLYBUTYLENE TEREPHTHALATE	2
08E - Nail Polish and Enamel	POLYBUTYLENE TEREPHTHALATE	10
12A - Cleansing	POLYBUTYLENE TEREPHTHALATE	2
		23

07E - Lipstick	POLYETHYLENE ISOTEREPHTHALATE	2

02D - Other Bath Preparations	POLYETHYLENE TEREPHTHALATE	1
03B - Eyeliner	POLYETHYLENE TEREPHTHALATE	25
03C - Eye Shadow	POLYETHYLENE TEREPHTHALATE	62
03F - Mascara	POLYETHYLENE TEREPHTHALATE	8
03G - Other Eye Makeup Preparations	POLYETHYLENE TEREPHTHALATE	3
04B - Perfumes	POLYETHYLENE TEREPHTHALATE	1
04C - Powders (dusting and talcum, excluding aftershave talc)	POLYETHYLENE TEREPHTHALATE	4
04E - Other Fragrance Preparation	POLYETHYLENE TEREPHTHALATE	14
06E - Hair Color Sprays (aerosol)	POLYETHYLENE TEREPHTHALATE	1
07A - Blushers (all types)	POLYETHYLENE TEREPHTHALATE	4
07B - Face Powders	POLYETHYLENE TEREPHTHALATE	3
07C - Foundations	POLYETHYLENE TEREPHTHALATE	3
07D - Leg and Body Paints	POLYETHYLENE TEREPHTHALATE	4
07E - Lipstick	POLYETHYLENE TEREPHTHALATE	173
07G - Rouges	POLYETHYLENE TEREPHTHALATE	2
07I - Other Makeup Preparations	POLYETHYLENE TEREPHTHALATE	27
08D - Nail Extenders	POLYETHYLENE TEREPHTHALATE	8
08E - Nail Polish and Enamel	POLYETHYLENE TEREPHTHALATE	38
08G - Other Manicuring Preparations	POLYETHYLENE TEREPHTHALATE	1
10A - Bath Soaps and Detergents	POLYETHYLENE TEREPHTHALATE	1
12D - Body and Hand (exc shave)	POLYETHYLENE TEREPHTHALATE	6
12F - Moisturizing	POLYETHYLENE TEREPHTHALATE	6
12J - Other Skin Care Preps	POLYETHYLENE TEREPHTHALATE	1
		396

10A - Bath Soaps and Detergents	POLYPROPYLENE TEREPHTHALATE	7
10E - Other Personal Cleanliness Products	POLYPROPYLENE TEREPHTHALATE	1
11A - Aftershave Lotion	POLYPROPYLENE TEREPHTHALATE	1
11F - Shaving Soap	POLYPROPYLENE TEREPHTHALATE	1
12A - Cleansing	POLYPROPYLENE TEREPHTHALATE	1
12C - Face and Neck (exc shave)	POLYPROPYLENE TEREPHTHALATE	7
12D - Body and Hand (exc shave)	POLYPROPYLENE TEREPHTHALATE	5
		23

No uses for:

- Adipic Acid/1,4-Butanediol/Terephthalate Copolymer,
- Ethylene/Sodium Sulfoisophthalate/Terephthalate Copolymer,
- Polypentaerythrityl Terephthalate,
- Polypropylene Terephthalate

Personal Care Products Council
Committed to Safety, Quality & Innovation

Memorandum

TO: F. Alan Andersen, Ph.D.
Director - COSMETIC INGREDIENT REVIEW (CIR)

FROM: Halyna Breslawec, Ph.D.
Industry Liaison to the CIR Expert Panel

DATE: October 17, 2012

SUBJECT: Comments on the Scientific Literature Review on Modified Terephthalate Polymers as Used in Cosmetics

Key Issue

Although there is a request for information to confirm that the PET used in cosmetics is analogous to PET used in medical devices, the report does not contain any information on the specifications of the PET used in medical devices.

Additional Comments

The Council is not aware of any suppliers for the following modified terephthalate polymers:
Adipic Acid/1,4-Butanediol/Terephthalate Copolymer
Polypentaerythrityl Terephthalate

p.1 - In the Dictionary, Polyethylene/Polyethylene Terephthalate Laminated Powder, Polyethylene/Polypentaerythrityl Terephthalate Laminated Powder and Polymethyl Methacrylate/Polypentaerythrityl Terephthalate/Stearate/Palmitate Laminated Powder have JPN after the name. This means that these are names for use exclusively in Japan. These ingredients should not be included in the report because they are Japanese labeling names. The paragraph concerning these ingredients in the Introduction should be deleted from the report.

p.2 - Is there any information regarding impurities in PET medical devices?

p.2 - In the Non-Cosmetic Use section, please provide the appropriate CFR citations for the food contact uses. EPA does not regulate phthalate contamination in food. The maximum contaminant level goal for diethylhexyl phthalate is for drinking water not food.

p.2 - Please correct "surgcal surture"

p.3 - In the description of surgical sutures (21CFR878.500), please indicate that the sutures can be provided uncoated or coated, dyed or undyed with appropriate approved colors.

p.3 - As Polyethylene Terephthalate itself was not tested in the mutagenicity assay, the section heading should be changed to reflect the material actually tested (water stored in PET).

p.3 - Please correct "keratoplasy" and "stich"

p.3 - The Summary states that FDA has safety data on medical devices made from PET "including acute and long-term biocompatibility testing for cytotoxicity, irritation or intracutaneous

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reactivity, sensitization, systemic toxicity, implantation effects, and hemocompatibility." Has this data been requested from FDA? If these data are not going to be included in the report, it would at least be helpful to state how these data could be obtained.

p.6, Table 3 - In the description of reference 12, please indicate that the PET bottles used were amber in color and provide some indication of the conditions, e.g., temperature, at which the Soxhlet extraction was completed.

p.6, Table 3 - The description of reference 9 mentions "food contact grade PET". It would be helpful if the specifications for this grade of PET were described somewhere in the report. The sub-table with reference 9 is not clear. What is the meaning of "Polymer Content" [is this the amount of metal that is in the polymer]? The meaning of the units with the "food simulants" is not clear, perhaps the units really belong with the metals? Are the units mg/kg with olive oil correct (the other units are μg/kg)?

p.7, Table 3 in the study column, row 2, please correct: "was repeated with room temperature water and room temperature water"

p.7-8, Table 4 - This table should be split into two different tables, one table on measurements of estrogenic activity and one table for studies in which leachates were measured.

p.7, Table 4 - In the description of reference 23 (first row) it is not clear what is meant by "significant estrogenic activity". Did they include a positive control in this study?

p.7, Table 4 - The description of reference 7 suggests bottles from different brands of water were tested, not the water itself. The results (row two) says "in any brand of water", which suggests the water itself was tested. Were different brands of water actually tested in this study, or just bottles from different brands of water?

p.8, Table 4 - Di(2-ethylhexyl) adipate is not a phthalate. If measurement of other compounds is included in the table, the title of the table needs to be changed.

p.8, Table 4 - Please revise the following: "Mineral water (still and carbonated) collected from a bottling plant was used to fill PET and glass bottles with mineral water."

p.8, Table 4 - What phthalate compounds were measured in reference 17? It is not clear what "mean pool phthalate levels" represents.

p.8, Table 4 - Acetaldehyde is not a phthalate compound. Therefore, reference 15 does not belong in this table or the title of the table needs to be changed.

p.9, Table 6 - This table should also include information about the composition of polymers permitted for use in food contact materials and medical devices.