DEPARTMENT OF HEALTH & HUMAN SERVICES

Public Health Service

Memorandum

AD

Date August 1, 2000

From Division of Product Manufacture and Use, Chemistry and Exposure Assessment Team, HFS-246

Subject FCN 000059 - Ciba Specialty Chemicals, Co. Submissions dated 3-13-00 and 4-14-00. Use of perfluoroalkyl substituted carboxylic acid as an oil and water repellant for paper and paperboard.

To Division of Petition Control, HFS-215
Attn: E. Machuga, Ph. D.

Ciba Specialty Chemicals Co. is submitting this notification for use of N,N-bis[2-hydroxy-3-(2-propenyloxy)propyl]glycine, monosodium salt reaction products with ammonium hydroxide and pentafluoroiodoethane-tetrafluoroethylene telomer (common name: perfluoroalkyl substituted carboxylic acid, PFASCA) as an oil and water repellant in paper and paperboard complying with 21 CFR 176.170 and 176.180. The level of PFASCA shall not exceed 15 lbs of actives (7.8 lbs of fluorine) per ton of treated paper of a sheet basis weight of up to 300 lbs per 3000 ft^2 as determined by analysis for total fluorine in the treated paper and paperboard without correction for any fluorine that might be present in the untreated paper or paperboard, when such paper is used in contact with nonalcoholic food under conditions of use B through H.

Identity and Manufacture

CAS Name: glycine, N,N-bis[2-hydroxy-3-(2-propenyloxy)]-, monosodium salt, reaction products with ammonium hydroxide and pentafluoroiodoethane-tetrafluoroethylene telomer

Common Name: perfluoroalkyl substituted carboxylic acid (PFASCA, active components of (b) (4) [redacted])

Trade Name: (b) (4) [redacted] for the aqueous mixture containing PFASCA and other nonactive components)

CAS Registry No.: 220459-70-1

Structure (of PFASCA, the active components of (b) (4) [redacted]:

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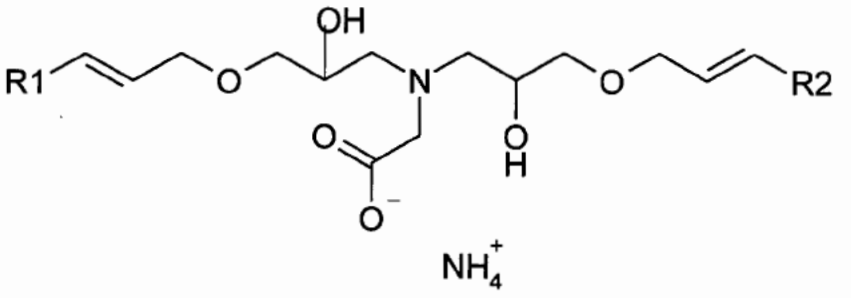

where R1 = C_nF_{2n+1} and R2 = $C_{n'}F_{2n'+1}$ and where n or n'= 6, 8, 10, 12, 14

The average molecular weight of PFASCA is 1215 g/mole. The molecular weight distribution of PFASCA is a follows:

n + n'	Molecular Weight	Wt.-%
12	908	0.5
14	1008	7.3
16	1108	31.1
18	1208	30.5
20	1308	18.5
22	1408	9.0
24	1508	2.8
26	1608	0.7
28	1708	0.1

The composition of (b) (4) E [redacted], determined using either ^{1}H NMR, gas chromatography, or silver nitrate titration (see Attachment 1 and 3), is listed on page 3(b) and tabulated below. Infrared and ^{1}H NMR spectra (b) (4) E [redacted], which support the structures of the additive, are provided in Attachment 1.

Compound	Typical Composition
PFASCA	(b) (4)
(b) (4)	
(b) (4)	
(b) (4)	

to decrease viscosity.

(b) (4)

[redacted]

Use/Technical Effect

PFASCA be used as an oil and water repellant in paper and paperboard complying with 21 CFR 176.170 and 176.180. The level of PFASCA will not exceed 15 lbs of actives (7.8 lbs of fluorine) per ton of treated paper of a sheet basis weight of up to 300 lbs per 3000 ft^2 as determined by analysis for total fluorine in the treated paper and paperboard without correction for any fluorine that might be present in the untreated paper or paperboard, when such paper is used in contact with nonalcoholic food under conditions of use B through H.

The incorporation of PFASCA into paper and paperboard products provides repellancy properties that effectively prevent the absorption of aqueous and fatty foods into the paper and paperboard. To support this claim, the notifier has provided data on the oil and water repellant properties of paper containing PFASCA as an external and internal paper sizing. Data on page 6(b), where the additive is used as an external paper sizing in two different paper formulations, demonstrate that oil holdout and turpentine penetration increases with increasing weight-% of the additive and that % staining are decreases with increasing weight-% of the additive. Also, data on page 6(c), where PFASCA is used an internal paper sizing in a paper formulation, demonstrate that oil holdout increases with increasing weight-% of the additive and that water penetration decreases with increasing weight-% of the additive. We concur that the intended technical effect of PFASCA has been adequately demonstrated.

Migration

[redacted] was applied to 39# Waterleaf paper together with 2% (based on dry weight of

paper) Ethylex-2065 (ethoxylated starch) using a vertical size-press and with wet pick-up adjusted to 100%. The 8 inch by 18 inch paper sheets were dried on a photographic dryer on both sides for 30 seconds at 170° C. The dried paper was analyzed for fluorine and the active ingredient (PFASCA). The average active ingredient solids in paper is 0.755% (or 0.395% fluorine).[2] Control paper containing 2% Ethylex-2065 was found to contain 0.09% fluorine.

Protocol

A sample plaque (40 in^2, including both sides) was placed in a two-sided extraction cell (capable of withstanding high pressures) along with 160 mL of food simulant. This corresponds to a food simulant-to-surface area ratio of 4 mL/in^2. Extraction conditions were as follows:

10% ethanol: 212°F, 2 h/104° F, 238 h

n-heptane: 212°F, 2 h/104° F, 238 h

A total of twelve extractions were carried out for each sample in each food simulant. Triplicate extracts were analyzed after 2 hours at the initial high temperature extraction, and after 24, 96, and 240 hours at 104° F. Each extract was analyzed by gas chromatography (GC). Solvent blanks were run for each food simulant.

Analytical Methods and Migration Results

For the 10% ethanol extracts, a 50-mL portion was placed in a 55-mL headspace jar containing 0.20 g of sodium chloride. The jar was placed in an 80° C oven for 25 minutes prior to injection. The solution was analyzed for PFASCA using GC with an electron capture detector (ECD), as outlined in Appendix A on page 86.

Four standard solutions of PFASCA (0.125, 0.800, 2.00, and 5.00 μg/mL) were prepared in 10% ethanol. A standard calibration for the four concentrations of the PFASCA versus instrument response was determined (in Appendix B on page 89). A calibration curve was constructed and determined to be linear with a correlation coefficient of 0.992. The limit of detection was determined to be < 50 ppb (or < 0.5 $\mu g/in^2$).

For the *n*-heptane extracts, a 32-mL portion was placed in a 125-mL round bottom flask and evaporated to dryness using a rotary evaporator. Four milliliters of tetrahydrofuran (THF) was added to the dry residue which was then sonicated for 10 minutes. The solution was analyzed for PFASCA using GC with ECD, as outlined in Appendix A on page 87.

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[2]This is equivalent to the maximum level of fluorine in paper proposed by the notifier (7.8 lbs fluorine/ton paper x ton paper/2000 lbs paper x 100 = 0.39% or 0.39 g fluorine/100 g paper).

Four standard solutions of the PFASCA (0.125, 0.800, 2.00, and 5.00 μg/mL) were prepared in THF. A standard calibration for the four concentrations of the PFASCA versus instrument response were determined (in Appendix B on page 95). A calibration curve was constructed and determined to be linear with a correlation coefficient of 0.991. The limit of detection was determined to be < 50 ppb (or < 0.5 $\mu g/in^2$).

Sample chromatograms are shown in Appendix B in Figure B6 (for the 10% ethanol report) and in Figure B14 (for the *n*-heptane report). No PFASCA was detected in the 10% ethanol extracts. The maximum levels of the PFASCA detected in the extracts are summarized below.

Food Simulant	Observed Extractives	
	μg/mL	$\mu g/in^2$
10% ethanol[3]	< 0.125	< 0.500
n-heptane[4]	9.58	4.79

Assuming the ratio of the mass of food contacting the surface area of the paper to the paper area is 10 g/in^2, the maximum concentration in food (<M>) of PFASCA is as follows:

Food Simulant	<M>
10% ethanol	< 50 ppb
95% ethanol	0.48 ppm

Validation

To validate the 10% ethanol extraction studies, spike and recovery studies were performed in triplicate on the 10-day control extracts. The 10% ethanol extracts were spiked (prior to analytical workup) at the limit of detection (50 ppb). Recoveries ranged from 84 - 147 and are acceptable.

To validate the *n*-heptane migration studies, spike and recovery studies were performed in triplicate using 10-day control extracts. The *n*-heptane were spiked (prior to analytical workup)

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[3]The 10% ethanol observed extractives in $\mu g/in^2$ can be calculated using the volume-to-surface area of 160 mL/40 in^2 and the observes extractives in μg/mL.

[4]The *n*-heptane observed extractives in $\mu g/in^2$ can be calculated using the concentration factor of 4 mL/32 mL, the volume-to-surface area of 160 mL/40 in^2, and the observed extractives in μg/mL.

at ½, 1, and 2 times the detected level of PFASCA. Recoveries ranged from 108 - 111 and are acceptable.

Exposure

To Perfluoroalkyl Substituted Carboxylic Acid

Exposure to PFASCA, based on the notifier's migration studies, has been calculated. Using the concentration in food (<M>, listed above), the food-type distribution factors (for uncoated paper) of 0.58 for aqueous and acidic foods and of 0.41 for fatty foods, and a consumption factor of 0.05 for uncoated paper or paperboard treated with greaseproofing agent,[5] the dietary concentration of PFASCA is calculated below.

$$\text{Dietary Concentration} = CF\ [(f_{\text{aqueous+acidic}} \times \langle M_{10\%\ \text{ethanol}} \rangle) + (f_{\text{fatty}} \times \langle M_{95\%\ \text{ethanol}} \rangle)]$$

$$= 0.05\ [(0.58 \times 50\ \text{ppb}) + (0.41 \times 0.48\ \text{ppm})]$$

$$= 0.05\ [29\ \text{ppb} + 197\ \text{ppb}] = 0.05 \times 226\ \text{ppb} = 11\ \text{ppb}$$

The estimated daily intake (EDI), based on a daily diet of 3000 g food/person/day, of PFASCA is:

$$\text{EDI} = 3000\ \text{g food/p/d} \times 11 \times 10^{-9}\ \text{g PFASCA/g food} = 33\ \mu\text{g/p/d}$$

To Components of (b) (4) [redacted]

(b) (4) [redacted]

(b) (4)

[redacted]

To Residual Impurities

(b) (4)

[redacted]

(b) (4)

[redacted]

Notification Language

The acknowledgment letter for this FCN dated 5-18-00 is appropriate as corrected.

Summary

We have no questions.

[redacted]

Roseann M. Costantino, Ph.D.

HFS-225: 245 (Diachenko, Begley): 246 (Kuznesof)
HFS-246:RMCostantino:rmc:418-3000:FCN 000059.wpd:7-19-00
RDInit:MAAdams:7-31-00

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(b) (4)

[redacted]

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