Date: 7 April 2020

Report: 11296/002

Test Report:

- **Elemental Content Analysis:**
  - Determination of Prodegradant Catalyst
- **Accelerated Ageing Studies:**
  - Thermal Stability
  - Thermal Degradation, Following Initial UV Exposure

Test Reference: **11296 Vaneco (Polczynski Jakub)**

Product Tested: **LDPE Toothbrush**

Prepared for: **Vaneco**

Prepared by: **Cameron Yule**, Scientist

Symphony Environmental Ltd, Product Testing Laboratory
6 Elstree Gate, Elstree Way, Borehamwood WD6 1JD

Telephone: +44 (0)20 8207 5900
technical@d2w.net
www.symphonyenvironmental.com

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TEST REPORT

1.0 AIMS

To evaluate the presence of prodegradant catalysts in the LDPE toothbrush samples by means of determination of the prodegradant catalyst metal cation by x-ray fluorescence (XRF) spectroscopy; and to evaluate the stability and degradation behaviour of the LDPE toothbrush samples by means of accelerated laboratory ageing techniques, while monitoring extent of polymer oxidation by infrared (IR) spectroscopy as a function of time.

2.0 SAMPLE DETAILS

Samples Provided by: Vaneco

Product Type: LDPE Toothbrush

Date Received: 18/02/2020

Table 1: Sample Details

SEPTL Sample ID	Sample Reference	Polymer Type	Prodegradant Additive
11296 A	1	LDPE	*None*
11296 B	2	LDPE	1% d_2w 93390

Table 1 is based solely on information provided by the customer.

3.0 RESULTS & CONCLUSIONS

3.1 Determination of Prodegradant Catalyst

The presence of the prodegradant catalyst in each sample is confirmed by determination of the prodegradant catalyst metal cation by energy-dispersive X-ray fluorescence (XRF) spectroscopy.

The prodegradant catalyst content of the LDPE toothbrush samples, as determined by x-ray fluorescence (XRF) spectroscopy, is shown in Table 2.

Table 2: Determination of Prodegradant Catalyst

Sample ID	Spectrum Ref.	Prodegradant Content
11296 A	2710/51093	Not Detected
11296 B	2710/51095	Prodegradant Present, Effective Concentration

3.2 Accelerated Ageing - Thermal Stability

The real-time stability of polymer products stored at ambient temperatures and protected from extended exposure to sunlight is evaluated over shorter period of time in the laboratory by monitoring degradation during thermal ageing at elevated temperatures according to ASTM D5510.

Polymer degradation is evaluated by determination of polymer oxidation by infrared spectroscopy. The increase in magnitude of features of the infra-red spectra corresponding to the carbonyl products of polymer oxidation, is recorded as carbonyl optical density. The period of accelerated thermal ageing where no significant increase in carbonyl optical density (not exceeding 0.0010) is observed is considered to be representative of the real-time period of product stability in storage conditions.

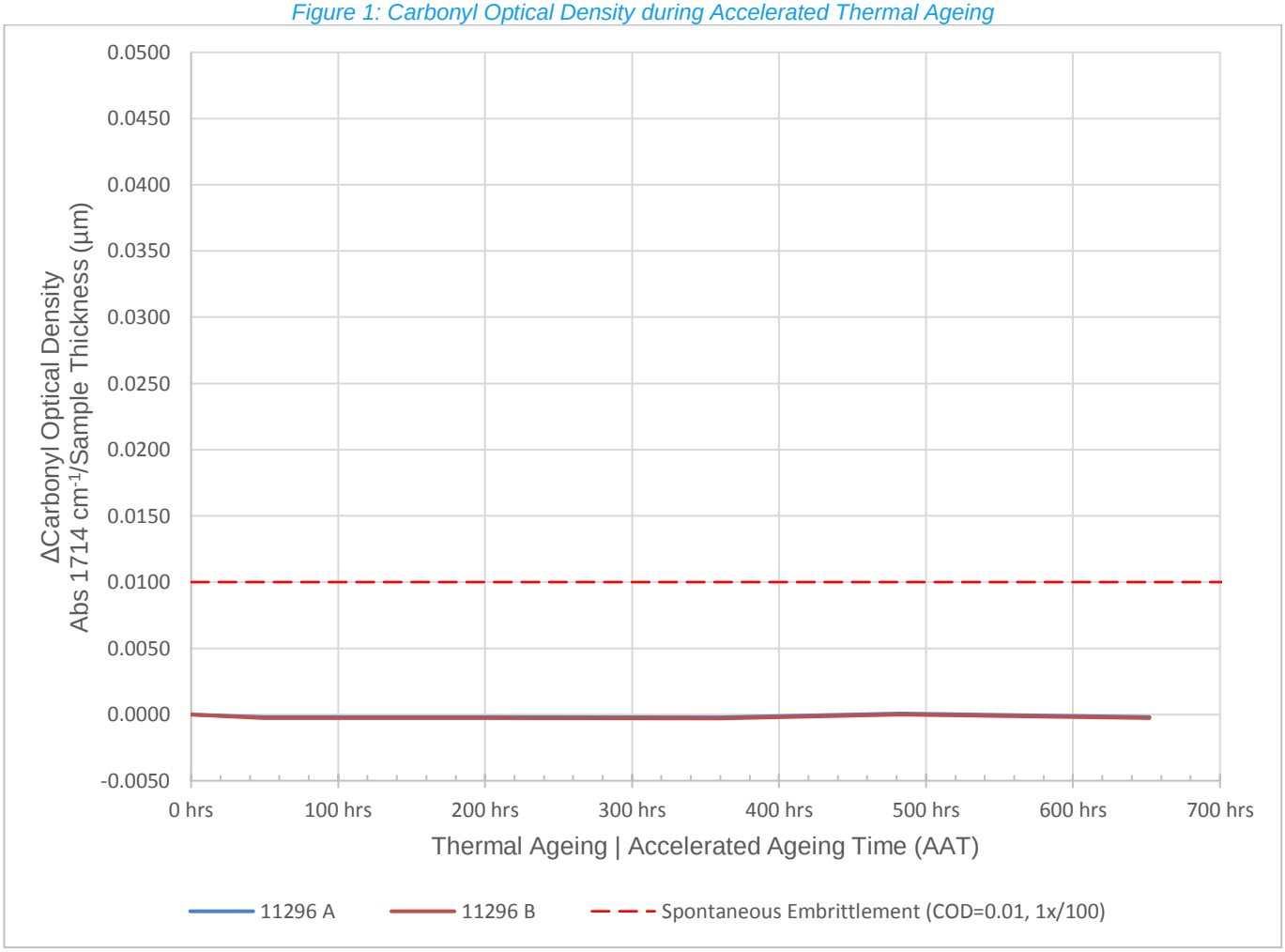
Samples 11296 A & B demonstrated no significant oxidation during the accelerated stability test. The samples demonstrated an average carbonyl optical density value which did not exceed 0.0001 up to 652 hours accelerated ageing (Table 3). This result is consistent with the samples having undergone no significant degradation.

The absence of degradation in the samples for the duration of this test confirms that the products are stable in dark conditions, at ambient temperatures for an initial life period corresponding to the product life. Based on these results a product life of 12 months is recommended for these products, provided storage at an average ambient temperature which does not exceed 30°C in indoor storage conditions and is protected from extended sunlight exposure.

Table 3: Carbonyl Optical Density during Accelerated Thermal Ageing

Accelerated Ageing Time (AAT)	ΔCarbonyl Optical Density (IR Abs 1714 cm^{-1}/Thickness)	
	11296 A	11296 B
0 hrs	0.0000	0.0000
49 hrs	-0.0002	-0.0003
195 hrs	-0.0002	-0.0003
360 hrs	-0.0002	-0.0003
483 hrs	0.0001	0.0000
652 hrs	-0.0002	-0.0003

Figure 1: Carbonyl Optical Density during Accelerated Thermal Ageing



3.3 Accelerated Ageing - Thermal Degradation, Following Initial UV Exposure

Polymer degradation in dark conditions, following initial exposure to sunlight is evaluated in a shorter period of time in the laboratory by monitoring degradation during accelerated fluorescent UV ageing in accordance with ASTM D5208 followed by thermal ageing at elevated temperatures according to ASTM D5510.

Polymer degradation is evaluated by determination of polymer oxidation by infrared spectroscopy. The increase in magnitude of features of the infra-red spectra corresponding to the carbonyl products of polymer oxidation, is recorded as carbonyl optical density. A carbonyl optical density value of 0.0100 is considered indicative of advanced degradation as such to bring about spontaneous embrittlement. The rate of degradation is evaluated and compared by monitoring carbonyl optical density as a function of ageing time.

The samples were initially exposed to constant fluorescent UV ageing for a period of 48 hours. The samples were then exposed to accelerated thermal ageing in dark conditions. During accelerated ageing, test sample 11296 B demonstrated a significant increase in carbonyl optical density (Table 4).

Sample 11296 B demonstrated a carbonyl optical density value of 0.0239 in 499 hours exposure (inclusive of 48 hours initial fluorescent UV exposure). This result is consistent with this sample having undergone significant degradation.

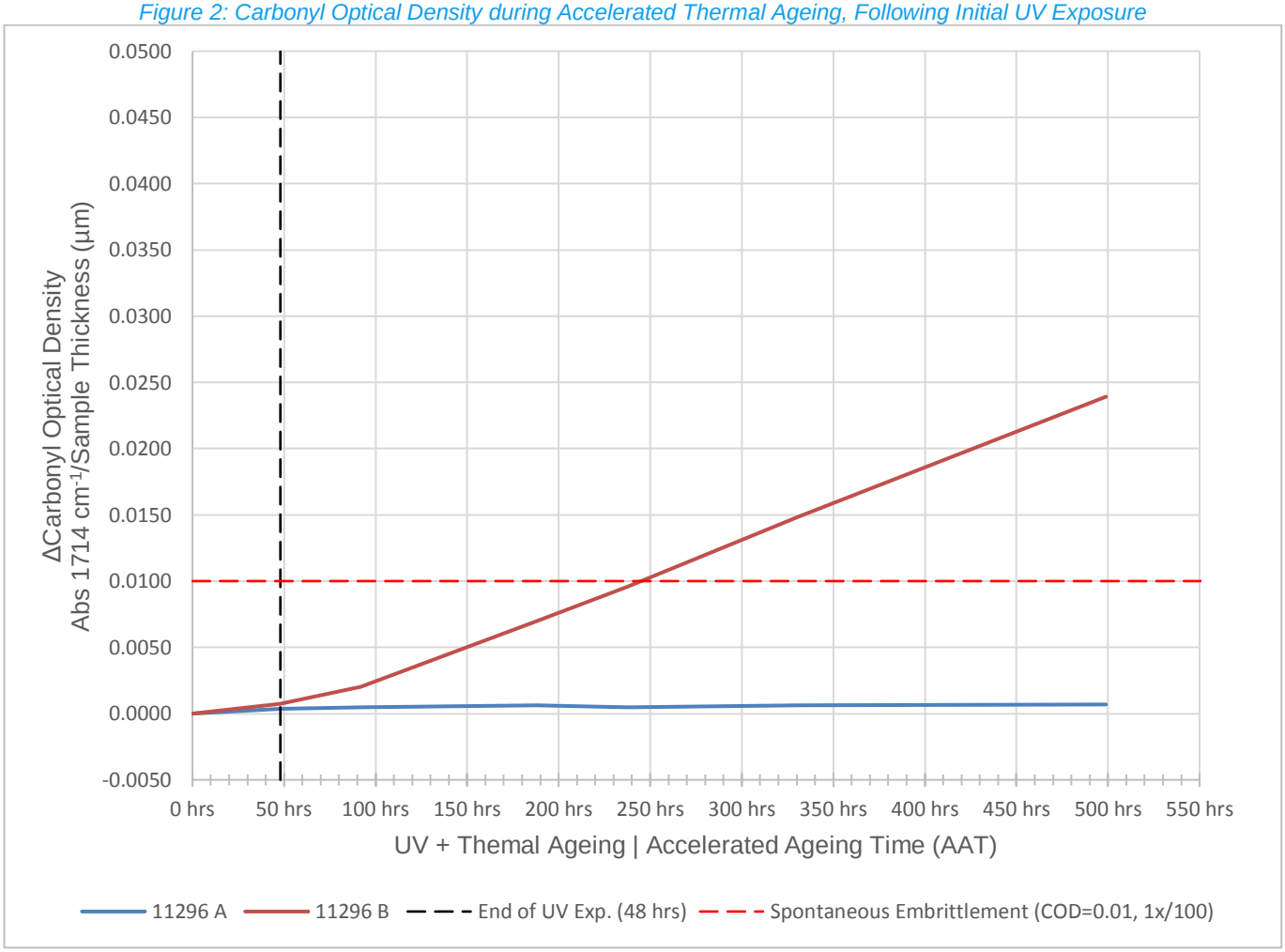
Sample 11296 A demonstrated no significant increase in carbonyl optical density throughout the test (Table 4). This result is consistent with this sample having undergone no significant degradation.

The observation of degradation exclusively in the sample which contains the prodegradant additive is consistent with the additive promoting degradation of the product in dark conditions, following initial exposure to sunlight in the environment as litter.

Table 4: Carbonyl Optical Density during Accelerated Thermal Ageing, Following Initial UV Exposure

Accelerated Ageing Time (AAT)	Exposure	ΔCarbonyl Optical Density (IR Abs 1714 cm^{-1}/Thickness)	
		11296 A	11296 B
0 hrs	UV	0.0000	0.0000
48 hrs		0.0004	0.0007
92 hrs	Thermal	0.0005	0.0020
188 hrs		0.0006	0.0070
238 hrs		0.0005	0.0096
331 hrs		0.0006	0.0149
499 hrs		0.0007	0.0239

Figure 2: Carbonyl Optical Density during Accelerated Thermal Ageing, Following Initial UV Exposure



1.0 TEST METHODOLOGY

1.1 Preparation of Film Pressings

The samples were pressed into sheets using an Atlas series laboratory hydraulic press fitted with Specac heated plate accessory and constant film thickness maker, at a temperature of 140°C under a pressure of ~2 tons.

1.2 XRF Spectroscopy

1.2.1 Determination of Prodegradant Catalyst

The XRF spectrum of each unaged sample is recorded using a Bruker S2 Ranger A20-X10 bench top spectrometer in air over 120 s with 40.00 kV, 250 mA X-ray source and a 500.0 µm aluminium filter. Samples were prepared and the total thickness made up to ~200 µm with discs using pressed material.

The concentration of the prodegradant catalyst metal cation is quantified by correlation with a calibration of validated reference samples.

1.3 Accelerated Ageing Exposure

1.3.1 Sample Preparation/Holders

35 x 90 mm film samples were cut using a scalpel and secured in a sample holder with four exposure windows.

1.3.2 Thickness Determination

The thickness of the sample material is determined before ageing using a digital electronic micrometer, in not less than four random locations across the test sample and the average value recorded.

1.3.3 Accelerated Thermal Ageing

Thermal ageing of the samples was carried out in a Memmert UFE 600 fan assisted oven at a temperature of 70ºC in accordance with ASTM D5510 Procedure B: Forced Ventilation Oven.

1.3.3.1 Thermal Stability Real Time Exposure Prediction

The real time period (RT) at ambient temperature (T_{RT}) represented by accelerated ageing time (AAT) at elevated accelerated ageing temperature (T_{AA}) is predicted in accordance with ASTM F1980 using the relationship outlined in Equation 1.

The calculation is performed using an anticipated average storage exposure temperature (T_{RT}) of 30°C and a conservative ageing factor (Q_{10}) of 2.

Equation 1: $$AAT = \frac{RT}{Q_{10}^{[(T_{AA}-T_{RT})/10]}}$$

1.3.4 Accelerated Fluorescent UV Ageing

Samples were exposed to ultraviolet radiation in accordance with ASTM D5208 in a Q Panel QUV/se test apparatus fitted with UVA 340 lamps, set to a black panel temperature of 50ºC and irradiance of 0.78 W/m^2/nm @ 340 nm.

1.4 FT-IR Spectroscopy

Infrared (IR) spectra is recorded in accordance with ISO 10640 using a Nicolet iS10 fourier transform infrared (FT-IR) Spectrometer and the absorbance at 1714 cm^{-1} was recorded.

1.4.1 Carbonyl Optical Density Calculation

The extent of oxidation is reported as the carbonyl optical density, a function of the net increase in infrared absorbance (ΔIR Abs) at 1714cm^{-1} during ageing, per unit of the path length (sample thickness in µm):

Equation 2: $$\textbf{Carbonyl Optical Density} \quad \frac{\Delta\text{IR Abs}_{1714\ \text{cm}^{-1}}}{\text{Thickness } (\mu\text{m})}$$

2.0 REPORT HISTORY

Date	Report Ref.	Comments
Previous Versions		
11/03/2020	11296/001	Prodegradant Content Report
Current Version - This report supersedes all previous versions		
07/04/2020	11296/002	Added Accelerated Ageing Data

3.0 REPORT AUTHORISATION

Author:

Cameron Yule
BSc (Hons)
Scientist

C. Yule

Digitally signed by Cameron Yule
DN: cn=Cameron Yule, o=Symphony Environmental Ltd, ou=Scientist, email=cameron.yule@d2w.net, c=GB
Date: 2020.04.07 14:43:47 +01'00'

"The information presented in this report is based on the material actually tested. Performance of finished product made with $d_2w^®$ additive depends on the conditions under which and length of time for which the additive is stored and on the method of manufacture of the finished product and the heat, light, stress and other conditions to which the finished product is exposed. Nothing in this report constitutes or implies a license to use Symphony's intellectual property".