

TEST REPORT

Result: PASS Report Date: 27.NOV.2013

Customer Name: Enviplas (ENVITM)

Tested To: ASTM D6954-04

Description: Enviplas (ENVITM) added PE Films 13 mm thickness

Test Type: Degradation, Material Test, Carbon Dioxide Evolution

Job Number: 2013/09/306

Project Number: 2013/207

Project Manager: Johnny WILL

Thank you for having your product tested by Waters Agricultural Laboratories.

Report Authorization: Keith GREENE Date: 27.NOV.2013

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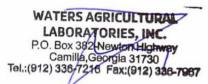
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	Sampling	Control Mean	Test Substance, Rep. 1 Net		Test Substance, Rep. 2 Net		Reference Substance Net	
Sampling Interval	Time (Days)	Accumulated CO ₂ (mg)	Accumulated CO ₂ (mg)	Percent ThCO ₂	Accumulated CO ₂ (mg)	Percent ThCO ₂	Accumulated CO ₂ (mg)	Percent ThCO ₂
33	11.04	-1.59	2.72	3.8%	3.51	4.9%	66.73	46.7%
34	11.38	-1.60	3.17	4.5%	4.00	5.6%	68.81	48.1%
35	11.71	-1.63	3.33	4.7%	4.18	5.9%	69.52	48.6%
36	12.05	-1.73	3.75	5.3%	4.70	6.6%	71.35	49.9%
37	12.38	-1.75	3.90	5.5%	4.86	6.8%	71.96	50.3%
38	12.71	-1.75	4.32	6.1%	5.33	7.5%	73.66	51.5%
39	13.05	-1.76	4.45	6.3%	5.48	7.7%	74.22	51.9%
40	13.38	-1.77	4.86	6.8%	5.94	8.4%	75.84	53.0%
41	13.72	-1.78	4.99	7.0%	6.09	8.6%	76.38	53.4%
42	14.05	-1.85	5.40	7.6%	6.53	9.2%	77.90	54.5%
43	14.38	-1.86	5.53	7.8%	6.67	9.4%	78.42	54.8%
44	14.72	-1.85	5.92	8.3%	7.11	10.0%	79.86	55.8%
45	15.05	-1.86	6.06	8.5%	7.25	10.2%	80.36	56.2%
46	15.39	-1.86	6.46	9.1%	7.69	10.8%	81.74	57.2%
47	15.72	-1.86	6.60	9.3%	7.84	11.0%	82.20	57.5%
48	16.06	-1.86	7.00	9.8%	8.26	11.6%	83.51	58.4%
49	16.39	-1.87	7.14	10.0%	8.42	11.8%	83.94	58.7%
50	16.73	-1.88	7.53	10.6%	8.83	12.4%	85.19	59.6%
51	17.06	-1.88	7.66	10.8%	8.97	12.6%	85.58	59.8%
52	17.39	-1.85	8.05	11.3%	9.38	13.2%	86.74	60.7%
53	17.73	-1.85	8.16	11.5%	9.50	13.4%	87.13	60.9%
54	18.06	-1.85	8.54	12.0%	9.90	13.9%	88.27	61.7%
55	18.39	-1.85	8.65	12.2%	10.03	14.1%	88.65	62.0%
56	18.73	-1.86	9.00	12.7%	10.41	14.6%	89.72	62.7%
57	19.97	-1.89	9.12	12.8%	10.55	14.8%	90.09	63.0%
58	20.31	-1.95	9.49	13.3%	10.94	15.4%	91.14	63.7%
59	22.02	-1.96	9.62	13.5%	11.08	15.6%	91.49	64.0%
60	22.35	-1.93	9.97	14.0%	11.46	16.1%	92.49	64.7%
61	22.69	-1.92	10.10	14.2%	11.57	16.3%	92.83	64.9%
62	23.02	-1.88	10.45	14.7%	11.93	16.8%	93.78	65.6%
63	23.35	-1.88	10.56	14.9%	12.06	17.0%	94.09	65.8%
64	23.69	-1.83	10.88	15.3%	12.40	17.4%	94.99	66.4%

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	Sampling	Control Mean	Test Substance, Rep. 1 Net		Test Substance, Rep. 2 Net		Reference Substance Net	
Sampling Interval	Time (Days)	Accumulated CO ₂ (mg)	Accumulated CO ₂ (mg)	Percent ThCO ₂	Accumulated CO ₂ (mg)	Percent ThCO ₂	Accumulated CO ₂ (mg)	Percent ThCO ₂
65	24.02	-1.83	11.00	15.5%	12.52	17.6%	95.30	66.6%
66	24.36	-1.78	11.33	15.9%	12.87	18.1%	96.17	67.3%
67	24.69	-1.78	11.45	16.1%	12.99	18.3%	96.46	67.5%
68	25.03	-1.78	11.77	16.6%	13.31	18.7%	97.30	68.0%
69	25.36	-1.79	11.88	16.7%	13.42	18.9%	97.59	68.2%
70	25.70	-1.76	12.18	17.1%	13.75	19.3%	98.39	68.8%
71	26.03	-1.75	12.31	17.3%	13.86	19.5%	98.66	69.0%
72	26.37	-1.72	12.62	17.8%	14.18	19.9%	99.44	69.5%
73	26.70	-1.70	12.74	17.9%	14.30	20.1%	99.72	69.7%
74	27.03	-1.76	13.03	18.3%	14.58	20.5%	100.39	70.2%
75	27.37	-1.77	13.13	18.5%	14.67	20.6%	100.64	70.4%
76	27.70	-1.75	13.38	18.8%	14.91	21.0%	101.24	70.8%
77	28.03	-1.74	13.48	19.0%	15.02	21.1%	101.50	71.0%
78	28.37	-1.73	13.76	19.3%	15.30	21.5%	102.13	71.4%
79	28.70	-1.72	13.84	19.5%	15.39	21.6%	102.34	71.6%
80	29.04	-1.70	14.10	19.8%	15.66	22.0%	102.95	72.0%
81	29.37	-1.69	14.19	20.0%	15.75	22.1%	103.16	72.1%
82	29.71	-1.66	14.46	20.3%	16.02	22.5%	103.76	72.6%
83	30.04	-1.66	14.55	20.5%	16.11	22.7%	103.96	72.7%
84	30.38	-1.61	14.80	20.8%	16.38	23.0%	104.53	73.1%
85	30.71	-1.60	14.88	20.9%	16.46	23.2%	104.73	73.2%
86	31.05	-1.56	15.12	21.3%	16.71	23.5%	105.28	73.6%
87	31.38	-1.55	15.19	21.4%	16.79	23.6%	105.45	73.7%
88	31.71	-1.50	15.42	21.7%	17.04	24.0%	105.97	74.1%
89	32.05	-1.50	15.51	21.8%	17.13	24.1%	106.14	74.2%
90	32.38	-1.48	15.73	22.1%	17.35	24.4%	106.66	74.6%
91	32.71	-1.48	15.80	22.2%	17.44	24.5%	106.82	74.7%
92	33.05	-1.51	16.02	22.5%	17.63	24.8%	107.32	75.0%
93	33.38	-1.51	16.11	22.7%	17.71	24.9%	107.49	75.2%
94	33.72	-1.45	16.33	23.0%	17.93	25.2%	107.98	75.5%
95	34.05	-1.44	16.40	23.1%	18.00	25.3%	108.13	75.6%
96	34.39	-1.38	16.61	23.4%	18.23	25.6%	108.61	76.0%
97	34.72	-1.36	16.68	23.5%	18.30	25.7%	108.75	76.1%

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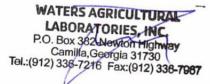
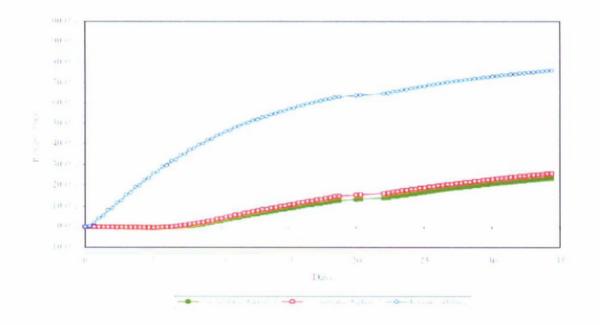




FIGURE 1 Cumulative Percent Theoretical CO: versus Time for the Test and Reference Substances



Conclusion

PE Film samples from Enviplas (ENVITM) were tested to select requirements of ASTM D6954 including Degradation, Material Test, Carbon Dioxide Evolution. Based on the results of testing, the PE film samples provided by Enviplas (ENVITM) passes the select requirements of ASTM D6954 for Degradation, Material Test, Carbon Dioxide Evolution.

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TEST REPORT

1. Objective

The objective of this test is to evaluate PE Film samples to all requirements of ASTM 6954-04. The sample was tested for degradation by UVCON and in the Oven (Thermal degradation at 70 C) were composed and exposed as given in the Table below.

Composition and Testing

Sample	Recipe (%)	ENVI TM		Sample Des	Degradation Analyses			
Number		% added	Туре	Sample characteristic	Color	UV	Thermal	
201309306-01		N		Film	Transparent	0-3-5-7- 11	10-20-30	
201309306-02		Y	ENVI TM	Film	transparent	0-3-5-7- 11	10-20-30	

The sample had a thickness of 47-55 μ m.

2. Sample Description

Testing was performed on plastic PE Film Samples provided by Enviplas (ENVITM). Samples for accelerated weathering followed by mechanical testing were prepared by using a Wallace Specimen Cutting Press.

The samples cut had a band width of 13 mm and a length of 165 mm. The samples were prepared parallel to the direction of polymer orientation.

Accelerated Ageing

3. Methods used:

The samples were fixed to suitable specimen holders and exposed to accelerated weathering in a QUV Accelerated Weathering Tester - Spray. Exposed area is 67 mm. The accelerated weathering device is equipped with 340 nm UVA Fluorescent lamps and is suitable for testing procedures according to ISO 4892-3 (ASTM G154), part of step 1 in ASTM D6954-04.

The weathering cycle is provided in the Appendix.

For Oven testing (Thermal Degradation) samples of film 165 mm (millimeters) long, 13 mm wide and with thickness as provided were subject to dark heated degradation at 70 C and 3 samples were tested at each sample point.

4. Mechanical Testing

Tensile properties of non-aged and aged samples were determined according to ISO 527-2 (ASTM D882).

Samples of each quality were exposed in each period of ageing. If a sample was broken after ageing or too brittle to be subjected to tensile testing, the tensile properties could not be determined. In such cases strain at break and elongation at break for these particular samples was set to zero. In case all three samples are broken after ageing or too brittle to be subjected to tensile testing strain at break and elongation at break are set to zero and the number of samples tested is three.

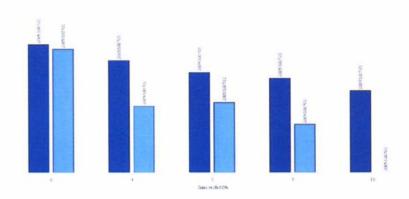


5. Results

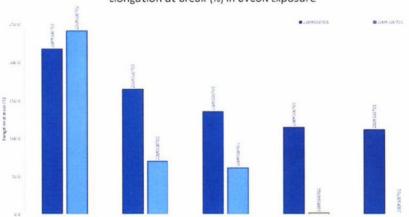
Samples, average and standard deviation of residual Elongation at Break (%) and Tensile Strength (mPa) during testing in UVCON Weather-O-Meter and testing in air circulation oven at 70 °C.

UVCON weather-o-meter exposure

Tensile Strength (Mpa) in UVCON Exposure

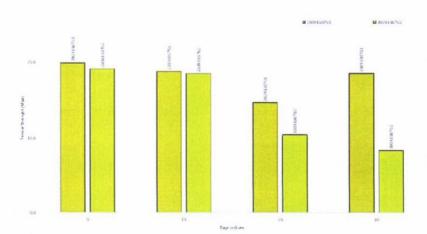


Elongation at break (%) in UVCON Exposure



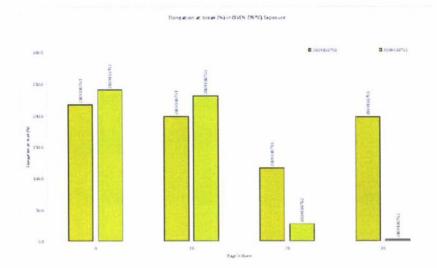
Thermal Degradation Testing

Tensile Strength (Mpa) in OVEN (70°C) Exposure





Elongation at break (%) in Oven (70°C) exposure.



6. Discussion and conclusion

The purpose of this test was to evaluate adding Enviplas (ENVITM) to degrade the samples of white film with a thickness of 45-77 μm .

Residual Elongation at Break (%) at or below 1% is taken to show the polymer sample has degraded to a molecular weight (MW) of less than 10.000 amu. This limit is used as a measure of molecular weight at which samples can be bio-mineralized (Micro-organisms can digest the short chain oxygenated plastic polymers).

UVCON exposure is made to simulate environmental exposure (outdoor) and Thermal exposure at 70 °C is used to determine thermal (indoor) degradation/stability (use-life).

UVCON exposure

The sample containing Enviplas (ENVITM) was totally degraded and brittle after 11 days of accelerated UV exposure. The unloaded film had still good properties left after same grade of exposure. 11 days of exposure in UVCON collaborate with 4-6 months outdoor in American summertime.

THERMAL exposure

The sample containing Enviplas (ENVITM) was totally degraded and brittle after 30 days of heat exposure (70 C). The unloaded film had still very good properties left after same grade of exposure. Estimated shelf life time for film containing Enviplas (ENVITM) will be approximately 1 year stored unexposed for sun and in temperatures below 30 C.





APPENDIX ISO

4892-3 Test Cycle

Test cycle for weathering according to ISO 4892-3 was:

4 hours UV340 at Temperature 60 $^{\circ}$ C. 5 minutes water spray (Water Temperature 8-10 $^{\circ}$ C. 3 hours and 55 minutes with Condensation and Temperature at 40 $^{\circ}$ C. The Cycle is repeated every 8 hours. Samples are pulled (3 replicates at each time) at intervals as described.

ISO 527-3 Brief Description

For film and sheet materials with a thickness up to 1 mm. For materials with a thickness greater than 1 mm ISO 527-1,-2 should be used.

Property	Definition
Tensile Modulus	Slope of the stress-strain curve between two specified strains ($\epsilon 1$ =0.0005 and $\epsilon 2$ =0.0025)
Tensile Strain at Break	Tensile strain corresponding to the point of rupture.
Tensile Strength	Maximum tensile stress sustained by the specimen during a test.
Tensile Stress at Yiel	d Tensile stress at which an increase in strain occurs without an increase in stress (yield point).

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Descriptions

Elongation at Break: Elongation recorded here is at the moment of rupture of the specimen, expressed as a percentage of the original length. It corresponds to the breaking or maximum load.

Tensile Strength: Tensile Strength measured here is the maximum stress the sample can withstand.

RAPRA Approximate Table of Degradation

In most cases the acceleration factor between the UVCON weather-o-meter conditions described in chapter 3 and natural weathering in southern countries is 8-12 (see next page). The Smithers RAPRA Table of 'Practical Guide to the Useful life of Plastics'.

T	Days to 75 % reduction in tensile strength			
Exposure conditions	PP (unstabilised)	HDPE (unstabilised)		
QUV 4 hours on 4 hours off 60/50 °C QUVA 340 lamps	60	65		
QUV 4 hours on 4 hours off 70/60 °C QUVA 340 lamps	25	40		
QUV continuous 60 ^o C QUVA 340 lamps	25	40		
QUV 4 hours on 4 hours off 60/50 °C QUVA 313 lamps	50	45		
Xenon lamp 102 min. light at 73 °C / 18 min water spray	70	120		
Xenon lamp 102 min. light at 63 °C / 18 min water spray	45	75		
Natural weathering, the UK	940*	3900*		
Natural weathering, Australia	360	640		

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Biodegradability of Enviplas (ENVITM) Using the CO₂ Evolution Method

A biodegradation test by the CO₂ evolution method was conducted at Waters Agricultural Laboratories, Inc. to determine the aerobic biodegradability of Enviplas (ENVITM). The test was conducted for 35 days beginning on August 11, 2013.

Stock solutions of the test and reference substances were prepared and used for dosing the test system. A 1.00 mg/mL stock solution of the test substance was prepared by diluting 500.1 mg of Enviplas (ENVI $^{\text{TM}}$) and 60.0 mg of the Fortifier in 500 mL of reagent water. A 1.00 mg/mL stock solution of the reference substance was prepared by diluting 1000.1 mg of sodium benzoate in 1000 mL of reagent water.

Activated sludge served as the microbial inoculum. Sludge was collected from aeration basin #1 at the Municipal Solid Waste facility located on Camilla, Georgia. The activated sludge sample was homogenized in a blender for two minutes, solids in the sample were allowed to settle for 30 minutes, and the supernatant was filtered through glass wool.

Each test system consisted of a 5-liter glass carboy filled with a mixture of mineral salts media, activated sludge, reagent water, and the appropriate volume of test or reference substance solution. The final volume in each carboy was 2 liters. Each carboy was placed in a waterbath over a magnetic stir plate and solutions were stirred with a Teflon-coated magnetic stir bar. The samples were connected to a Columbus Instruments Micro-Oxymax respirometer, which measured the CO2 in the sealed headspace above each sample. The Micro-Oxymax respirometer measured the mass of accumulated CO2 (mg) at each 8-hour sampling interval. After odd numbered sampling intervals, the respirometer refreshed the headspace of each carboy with atmospheric air.

The test systems were prepared as summarized in Table 1. The control system contained activated sludge, mineral salts media, and reagent water but no test or reference substance. Duplicate test substance carboys were prepared and contained activated sludge, mineral salts media, reagent water, and Enviplas (ENVITM) at a nominal concentration of 9.90 mg C/L. The reference substance carboy contained activated sludge, mineral salts media, reagent water, and benzoic acid dosed at a nominal concentration of 19.9 mg C/L.

Percent theoretical CO₂ (% ThCO₂) production from each test and reference system was calculated and determined as follows:

Net Accumulated CO_2 (mg) $-100 = \% ThCO_2$

Theoretical CO₂

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The net accumulated CO_2 was calculated as the difference in the accumulated CO_2 from the control systems (mean of the two replicates) and the accumulated CO_2 from the test or reference substance systems. The theoretical CO_2 (Th CO_2) for the test substance, 71.1 mg CO_2 , was calculated from the nominal test substance concentration (9.90 mg C/L) the volume of test solution after initiation (1960 mL), and the carbon to carbon dioxide conversion factor. The carbon to carbon dioxide conversion factor used was 3.664 [(44.01 g CO_2 /mole)/(12.01 g C/mole)]. Similarly, the theoretical CO_2 for the reference substance was calculated to be 143 mg CO_2 .

The test substance, Enviplas (ENVITM), exhibited % ThCO₂ values of 23.5% and 25.7% for replicates 1 and 2, respectively, by day 35 of the study. Enviplas (ENVITM) may be classified as inherently biodegradable (>20% ThCO₂).

The reference substance, sodium benzoate, exhibited a % ThCO $_2$ value of 76.1% by day 35 of the study. The results from the reference substance treatment indicate that the inoculum was viable.

TABLE 1. Preparation of the Test Systems

Carboy ID	Reference Substance Solution (mL)	Test Substance Solution (mL)	Mineral Salts Medium (mL)	Activated Sludge Inoculum (mL)	Reagent Water (mL)
Control, Replicate 1	-	-2	1600	20	400
Control, Replicate 2	-	-	1600	20	400
Reference Substance	69 ¹	-	1600	20	331
Test Substance, Replicate 1		229 2	1600	20	171
Test Substance, Replicate 2	2	229 2	1600	20	171

Note: The activated sludge inoculum and mineral salts medium were combined in the test carboys on day (-1) and were aerated until test initiation when all other solutions were added.

The nominal test concentrations for the test substance treatment, 9.90 mg C/L, and reference substance treatment, 19.9 mg C/L, were based on the percent carbon in the test and reference substances. The percent carbon in the reference substance was calculated to be 58.3% based on the molecular formula (C7H5O2Na). The percent carbon in the test substance, 8.73%, was supplied by the sponsor.

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¹ Concentration of the reference substance stock solution was 1.00 mg/mL.

 $^{^2}$ Concentration of the test substance stock solution was 1.00 mg/mL.



TABLE 2. Accumulated CO₂ from the Control Systems and Net Accumulated CO₂ and Percent ThCO₂ from the Test and Reference Substance Systems

			est Substance, R	ep. 1 1	est Substance, R	ep. 2	Reference Substa	ince
0 1	Sampling	Mean	Net	D	Net Accumulated	D	Net Accumulated	D
Sampling Interval	Time (Days)	Accumulated CO ₂ (mg)	Accumulated CO ₂ (mg)	Percent ThCO ₂	CO ₂ (mg)	Percent ThCO ₂	CO ₂ (mg)	Percent ThCO ₂
1	0.35	-0.07	-0.01	0.0%	-0.03	0.0%	0.05	0.0%
2	0.68	-0.19	-0.02	0.0%	-0.05	-0.1%	0.52	0.4%
3	1.02	-0.23	-0.03	0.0%	-0.06	-0.1%	2.97	2.1%
4	1.35	-0.35	-0.05	-0.1%	-0.10	-0.1%	6.12	4.3%
5	1.68	-0.37	-0.07	-0.1%	-0.10	-0.1%	7.84	5.5%
6	2.02	-0.45	-0.12	-0.2%	-0.15	-0.2%	11.64	8.1%
7	2.35	-0.48	-0.12	-0.2%	-0.14	-0.2%	13.13	9.2%
8	2.69	-0.58	-0.17	-0.2%	-0.19	-0.3%	16.99	11.9%
9	3.02	-0.61	-0.16	-0.2%	-0.16	-0.2%	18.41	12.9%
10	3.36	-0.65	-0.19	-0.3%	-0.19	-0.3%	22.25	15.6%
11	3.69	-0.68	-0.19	-0.3%	-0.18	-0.3%	23.65	16.5%
12	4.02	-0.75	-0.22	-0.3%	-0.22	-0.3%	27.38	19.1%
13	4.36	-0.79	-0.21	-0.3%	-0.21	-0.3%	28.70	20.1%
14	4.69	-0.88	-0.24	-0.3%	-0.24	-0.3%	32.28	22.6%
15	5.02	-0.93	-0.24	-0.3%	-0.24	-0.3%	33.55	23.5%
16	5.36	-0.97	-0.29	-0.4%	-0.28	-0.4%	36.96	25.8%
17	5.69	-1.02	-0.26	-0.4%	-0.26	-0.4%	38.12	26.7%
18	6.03	-1.08	-0.26	-0.4%	-0.15	-0.2%	41.35	28.9%
19	6.36	-1.10	-0.16	-0.2%	-0.07	-0.1%	42.40	29.7%
20	6.70	-1.16	-0.05	-0.1%	0.07	0.1%	45.44	31.8%
21	7.03	-1.18	0.01	0.0%	0.19	0.3%	46.46	32.5%
22	7.37	-1.24	0.16	0.2%	0.48	0.7%	49.35	34.5%
23	7.70	-1.26	0.26	0.4%	0.60	0.9%	50.30	35.2%
24	8.04	-1.34	0.43	0.6%	0.94	1.3%	52.99	37.1%
25	8.37	-1.39	0.55	0.8%	1.07	1.5%	53.93	37.7%
26	8.70	-1.51	0.86	1.2%	1.41	2.0%	56.53	39.5%
27	9.04	-1.52	1.00	1.4%	1.54	2.2%	57.43	40.2%
28	9.37	-1.52	1.35	1.9%	1.95	2.7%	59.88	41.9%
29	9.70	-1.55	1.50	2.1%	2.15	3.0%	60.70	42.4%
30	10.04	-1.56	1.95	2.7%	2.67	3.8%	63.04	44.1%
31	10.37	-1.57	2.12	3.0%	2.84	4.0%	63.82	44.6%
32	10.71	-1.57	2.56	3.6%	3.34	4.7%	66.01	46.2%

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