Summary of OECD 111 Tier 1 Testing: Hydrolysis of ALFOTERRA® Surfactants as a Function of pH

RESEARCH REPORT

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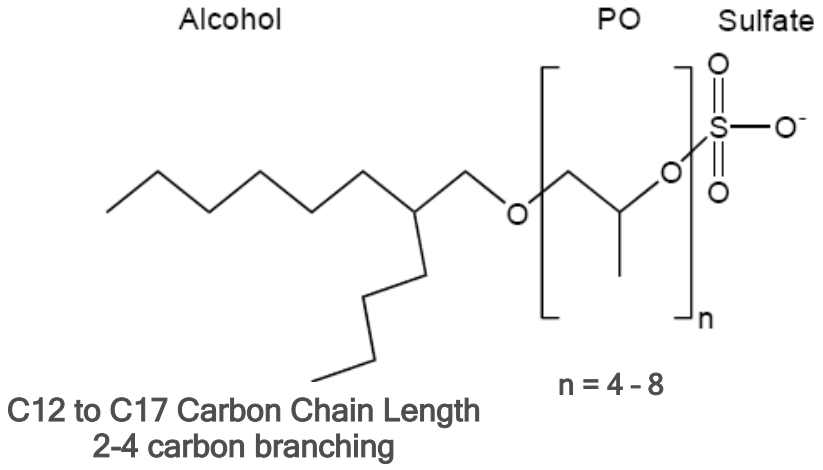
Test Substances

The following materials were tested for hydrolytic stability under *OECD 111: OECD Guidelines for the Testing of Chemicals: Hydrolysis as a Function of pH*. These alcohol propoxy sulfates are manufactured to have an active component of 30 ± 5%. This translates to the product sold containing 25 - 35% alcohol propoxy sulfate. Additionally, there is up to 10% unsulfated starting material, the propoxy alcohol. The target value for unsulfated propoxy alcohol is <5%. The balance of 55 - 65% is water.

Table I. Sasol Italy Substances Tested Under Tier I Guidelines of OECD 111

Trade Name	Common Name	Chemical Name	CAS Number	Carbon chain Length	PO Units (n)
ALFOTERRA® 123-4S	C12C13 branched propoxy sulfate, sodium salt	Poly[oxy(methyl-1,2-ethanediyl)], α-sulfo-ω-hydroxy-, C12-13-branched and linear alkyl ethers, sodium salts	958238-81-8	12-13	4
ALFOTERRA® 123-8S	C12C13 branched propoxy sulfate, sodium salt	Poly[oxy(methyl-1,2-ethanediyl)], α-sulfo-ω-hydroxy-, C12-13-branched and linear alkyl ethers, sodium salts	958238-81-8	12-13	8
ALFOTERRA® 145-4S	C14C15 branched propoxy sulfate, sodium salt	Poly[oxy(methyl-1,2-ethanediyl)], α-sulfo-ω-hydroxy-, C14-15-branched and linear alkyl ethers, sodium salts	958238-82-9	14-15	4
ALFOTERRA® 145-8S	C14C15 branched propoxy sulfate, sodium salt	Poly[oxy(methyl-1,2-ethanediyl)], α-sulfo-ω-hydroxy-, C14-15-branched and linear alkyl ethers, sodium salts	958238-82-9	14-15	8
ALFOTERRA® L167-4S	C16C17 branched and linear propoxy sulfate, sodium salt	Poly[oxy(methyl-1,2-ethanediyl)], α-sulfo-ω-hydroxy-, C16-17-branched and linear alkyl ethers, sodium salts	958238-83-0	16-17	4
C16C17 branched and linear propoxy sulfate, sodium salt		Poly[oxy(methyl-1,2-ethanediyl)], α-sulfo-ω-hydroxy-, C16-17-branched and linear alkyl ethers, sodium salts	958238-83-0	16-17	7

Figure 1. Structure of ALFOTERRA® Substances Studied



Buffer Solutions

For Tier I testing, buffers were prepared on 6-22-10 according to "Annex 3" of OECD 111. Each pH buffer prepared was tested with a calibrated pH meter, and the actual pH values for the buffers were found to be 4.0, 7.0, and 9.0 as intended. For details on the preparation of buffer solutions, refer to the procedure followed in Appendix I.

Test Conditions

The tests performed in the Tier 1 hydrolysis study were done in two separate batches of single replicate runs. ALFOTERRA® 123-4S and ALFOTERRA® 123-8S solutions were incubated for five days from 6/23/10 to 6/28/10. ALFOTERRA® 145-4S, ALFOTERRA® 145-8S, ALFOTERRA® L167-4S, and ALFOTERRA® L167-7S solutions were incubated for five days from 7/1/10 to 7/6/10. Test solutions in 1 oz. vials were incubated in the dark, in a forced air oven, which held an internal temperature of 50°C. 100 mL samples were prepared for each test material at each pH required yielding buffered solutions with active concentrations between 0.25 and 0.35%. Two 1 oz vials were filled with approximately 30mL of each solution to make duplicate samples. One of the duplicates was incubated for five days and then analyzed, while the other duplicate was analyzed immediately to obtain a time "0" analysis. Further details of the experimental method can be found in Appendix I of this report.

Table II. Amount of Test Substance diluted in Buffer Solutions

Test Material	Mass diluted to 100mL with pH 4 buffer (g)	Mass diluted to 100mL with pH 7 buffer (g)	Mass diluted to 100mL with pH 9 buffer (g)
ALFOTERRA® 123-4S	0.7434	0.7660	0.7332
ALFOTERRA® 123-8S	1.1844	1.1722	1.1633
ALFOTERRA® 145-4S	0.7342	0.7398	0.7483

ALFOTERRA® 145-8S	1.1196	1.1075	1.1019
ALFOTERRA® L167-4S	0.8947	0.9236	0.9051
ALFOTERRA® L167-7S	1.1305	1.1457	1.1224

Results

ASTM D3049, *Synthetic Anionic Ingredient by Cationic Titration,* was the analysis used to quantify the amount of material in each test sample. Sasol control charts show the repeatability of this test to have a standard deviation of 0.16% when expressed as % active anionic surfactant.

The amount of hydrolysis that occurred was determined on a molar basis by comparing the meq/100mL of the time "0" sample to the that of the time "5 day" sample. The following equation gives the % reduction of active surfactant test material after incubation for 5 days at 50°C.

$$\% \text{ reduction} = \frac{\text{"meq/100mL of time 0"- "meq/100mL of time 5 days"}}{\text{"meq/100mL of time 0"}} \times 100\%$$

Table III. Reduction of Test Materials after 5 days at 50°C in pH 4 buffer

Test Material	Time "0" Analysis (meq active/100 mL)	Time "5 Day" Analysis (meq active/100 mL)	% reduction
ALFOTERRA® 123-4S	0.4560	0.4373	4.10
ALFOTERRA® 123-8S	0.4670	0.4502	3.60
ALFOTERRA® 145-4S	0.4401	0.4248	3.48
ALFOTERRA® 145-8S	0.4578	0.4285	6.40
ALFOTERRA® L167-4S	0.4679	0.4437	5.17
ALFOTERRA® L167-7S	0.4682	0.4489	4.12

Table IV. Reduction of Test Materials after 5 days at 50°C in pH 7 buffer

Test Material	Time "0" Analysis (meq active/100 mL)	Time "5 Day" Analysis (meq active/100 mL)	% reduction
ALFOTERRA® 123-4S	0.4660	0.4581	1.70
ALFOTERRA® 123-8S	0.4550	0.4373	3.90
ALFOTERRA® 145-4S	0.4428	0.4358	1.58
ALFOTERRA® 145-8S	0.4505	0.4269	5.24
ALFOTERRA® L167-4S	0.4767	0.4758	0.19

ALFOTERRA® L167-7S	0.4767	0.4599	3.52

Table V. Reduction of Test Materials after 5 days at 50°C in pH 9 buffer

Test Material	Time "0" Analysis (meq active/100 mL)	Time "5 Day" Analysis (meq active/100 mL)	% reduction
ALFOTERRA® 123-4S	0.4239	0.4074	3.90
ALFOTERRA® 123-8S	0.4563	0.4428	3.00
ALFOTERRA® 145-4S	0.4529	0.4517	0.26
ALFOTERRA® 145-8S	0.4462	0.4395	1.50
ALFOTERRA® L167-4S	0.4703	0.4642	1.30
ALFOTERRA® L167-7S	0.4694	0.4673	0.45

According to the above data and OECD 111, the six materials tested above are considered to be hydrolytically stable since in all cases less than 10% hydrolysis was observed after 5 days of incubation at 50°C. No additional testing was performed under OECD 111 Tier 2 or Tier 3 guidelines.

Appendix I

Sasol Procedure Followed to determine Hydrolysis as a Function of pH: OECD 111 Tier I Testing

(Derived from *OECD 111 Guidelines for the Testing of Chemicals: Hydrolysis as a Function of pH)*

1. Properly clean glassware
    a. Wash volumetric flasks and other glassware in a laboratory glassware washer with deionized water prior to use.
    b. Rinse new 1 oz. vials for hydrolysis study three times with 18 MOhm NANOpure water, and dry in a vacuum oven overnight at 100°C.

2. Calculate the amount of test material to be used in the hydrolysis study.
    a. Solutions are to be made in 100mL volumetric flasks.
    b. ASTM D3049, *Synthetic Anionic Ingredient by Cationic Titrations,* will be used to characterize samples.
    c. Ideally, a 10mL aliquot of sample will be used to analyze via ASTM D3049, thus the amount of test material to be added to each 100mL volumetric can be calculated by "grams of sample = (0.045 x eq wt of active)/(% active)".

3. Prepare pH 4.0, 7.0, and 9.0 buffers according to "Annex 3" of *OECD 111: OECD Guidelines for the Testing of Chemicals: Hydrolysis as a Function of pH.*
    a. Prepare pH 4.0 buffer by combining 4.0mL of 0.1N NaOH and 500mL of 0.1M potassium biphthalate in a 1000mL volumetric flask. Dilute to volume with 18 MOhm NANOpure water and mix by inverting several times.
    b. Prepare pH 7.0 buffer by combining 296.3mL of 0.1N NaOH and 500mL of 0.1M monopotassium phosphate in a 1000mL volumetric flask. Dilute to volume with 18 MOhm NANOpure water and mix by inverting several times.
    c. Prepare pH 9.0 buffer by combining 213.0mL of 0.1N NaOH and 500mL of 0.1M boric acid in 0.1M KCl in a 1000mL volumetric flask. Dilute to volume with 18 MOhm NANOpure water and mix by inverting several times.

4. Test each pH buffer.
    a. Calibrate a pH meter with purchased pH 4, 7, and 9 standards.
    b. Check each buffer with the calibrated pH meter to ensure it is +/- 0.1 units within the desired pH.
    c. Remake any pH buffers that do not fall with the +/- 0.1 range of the target pH.

5. Sparge each pH buffer with argon for five minutes.

Note: The remaining steps should be repeated for each individual material being tested.

6. Purge and fill glassware with argon.
    a. Three 100mL volumetric flasks are required for each product tested. Purge and fill each with argon.
    b. Six 1 oz. vials are required for each product being tested. Purge and fill each with argon.

7. Charge each 100mL volumetric flask the appropriate amount of material being tested.
    a. Shake or mix the surfactant well before use.
    b. Weigh the appropriate amount of surfactant into three volumetric flasks using an analytical balance.
    c. Record the weight.

8. Dilute the three 100mL volumetric flasks.
    a. Dilute the three 100mL volumetric flasks to volume with pH 4.0, 7.0, and 9.0 buffers.
    b. Invert the flasks several times to thoroughly mix.

9. Dispense the solutions to 1 oz. vials for testing.
    a. Fill two 1 oz. vials to the neck (~30 mL per vial) with the contents of each 100mL volumetric flask. There will be six 1 oz. vials; a duplicate for each pH.
    b. Fill the headspace of the filled 1 oz. vials with argon.
    c. The remainder of the contents of the 100mL volumetric flask can be retained or discarded.

10. Begin the hydrolysis study.
    a. Place one of each pair of 1 oz vials in a equilibrated oven at 50°C.
    b. Submit the duplicate samples to the Analytical Services Department immediately for a time "0" analysis by ASTM D3049.
        i. If samples can not be analyzed immediately, place them in a refrigerator for storage.
        ii. Samples must be at room temperature before performing ASTM D3049 because sampling will be done by volume.

11. Allow the vials to remain in the oven for 5 days.

12. After 5 days, remove the vials from the oven and submit to the Analytical services Department immediately for the final time "5 day" analysis by ASTM D3049.

13. Compare time "0" and time "5 days" results to ascertain the amount of each sample that was hydrolyzed.

a. If more than 10% of hydrolysis is observed after 5 days for any of the three pHs, proceed with Tier 2 testing.