

Committee of Experts on the Transport of Dangerous Goods and on the Globally Harmonized System of Classification and Labelling of Chemicals

Sub-Committee of Experts on the Transport of Dangerous Goods

14 November 2016

Fiftieth session

Geneva, 28 November-6 December 2016

Item 2 (c) of the provisional agenda

**Recommendations made by the Sub-Committee on
its forty-seventh, forty-eighth and
forty-ninth sessions and pending issues:
listing, classification and packing**

Addendum to ST/SG/AC.10/C.3/2016/82: Special Provision 308 for Fish Meal (Fish Scrap), Stabilised (UN 2216): Class 9

Transmitted by IFFO

Additional results

1. IFFO submitted a report to the Sub-Committee of Experts on the Transport of Dangerous Goods in September 2016 on the 12-month results of the fishmeal stability trial. Unfortunately, the report was only partially complete as the results of the relevant and important self-heating test on all the samples were not available at that time. We have now received those results and would like to submit them to the sub-committee for review and to take into account as additional background to a request for a decision regarding the proposed changes to Special Provision 308 for Fish Meal (Fish Scrap), Stabilised (UN 2216): Class 9.
2. The table, updated with the additional self-heating results, can be seen below (Table 2.).

Table 2: Results of the treatments at Day 0, 6 months and 12 months storage

| Treatments | | Sampling intervals | | | | | |
|--|-----------|-------------------------|-------------------------|-------------------------------|------------------------------------|-------------------------|-------------------------------|
| | | Day 0 | 6 months | | | 12 months | |
| | | Antioxidant level (ppm) | Antioxidant level (ppm) | Self heating test (Pass/fail) | Oxygen Bomb induction period (hrs) | Antioxidant level (ppm) | Self heating test (Pass/fail) |
| EQ: 300 ppm | 50 kg bag | 318 | 259 | pass | 9.5 | 151 | pass |
| | 1 ton bag | 297 | | | 9.5 | 213 | pass |
| EQ: 30-50 ppm* | 50 kg bag | 28 | <15 | | 1.2 | <15 | pass |
| | 1 ton bag | 48 | | | 2.7 | 36 | pass |
| BHT solution: 2,000 ppm | 50 kg bag | 438 | 368 | pass | 2.4 | 290 | pass |
| | 1 ton bag | 438 | | | 2.6 | 342 | pass |
| BHT solution: 4,000 ppm | 50 kg bag | 858 | 751 | | 2.8 | 659 | pass |
| | 1 ton bag | 866 | | | 2.9 | 775 | pass |
| Tocopherol/rosemary extract blend: 2,000 ppm | 50 kg bag | 385 | 243 | pass | 2.5 | 209 | pass |
| | 1 ton bag | 400 | | | 3.8 | 280 | pass |
| Tocopherol/rosemary extract blend: 4,000 ppm | 50 kg bag | 628 | 488 | | 3.4 | 277 | pass |
| | 1 ton bag | 752 | | | 3.5 | 598 | pass |

* Originally included as a 600ppm ethoxyquin treatment, but an error in dosing the fishmeal during the trial resulted in a much lower concentration.

3. The 12-month self-heating test was performed on all the treatments. All the treatments passed the test (including the mistakenly low ethoxyquin dosage levels of 30ppm (50 kg bag) and 50 ppm (1 ton bag)).

4. The maximum temperature reached during the 24h self-heating test for all 12 treatments can be seen in Figure 3. It is clear that the maximum temperature for each treatment was well below the maximum temperature of 200°C above which the sample would have failed the test and would therefore be deemed to have self-heating properties. The maximum temperatures reached for the treatments are between 30 - 55°C below 200°C well below the 200°C maximum temperature defined as the upper limit in the methodology.

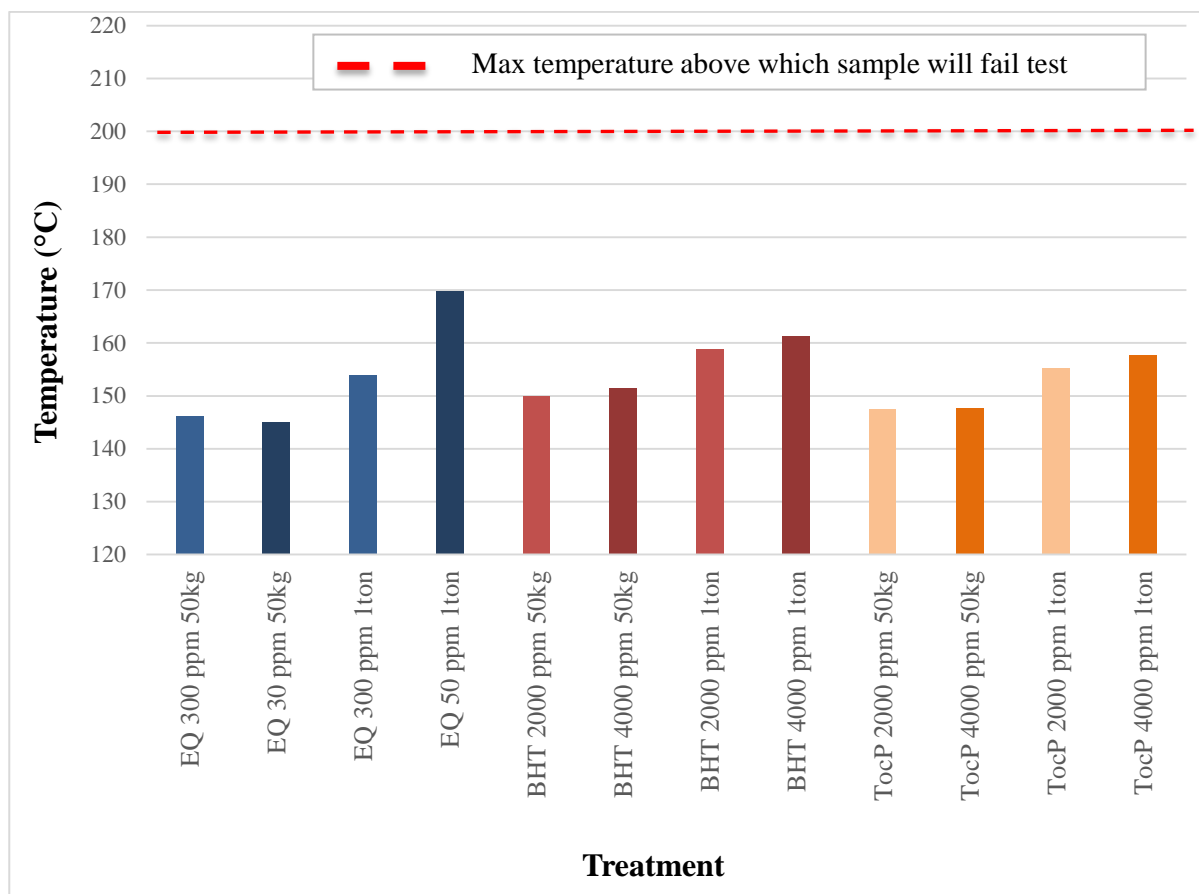


Figure 3: Maximum temperatures reached during self-heating test on all treatments performed at 12 months.

Conclusions

5. The conclusions on the test results from the laboratory that had performed the analyses are copied below:

Based on the above test results, the following conclusions were made:

(a) The material represented by samples #16121-1 -16121-12 did not appear to be Division 4.2 Self-Heating Substances, as defined by UN/DOT criteria. This is because negative results were obtained in testing of all samples using a 100 mm sample cube at 140 °C, when the *UN Test N.4, Test Method for Self-Heating Substances* was performed on the material.

6. The results therefore indicate that the alternative antioxidants, BHT and the natural tocopherols and rosemary extract blend, provide effective protection to fishmeal at the doses specified and over the trial period of 12 months. The natural tocopherols and rosemary extract therefore provide another option for the stabilisation of fishmeal during shipping.

7. In addition, a proposed dosage level, lower than currently advised in Special Provision 945 in the IMDG Code, of ethoxyquin at 300ppm also provided effective protection. However, it is clear that even lower dosage levels of ethoxyquin of 30 and 50 ppm effectively protected fishmeal even after 12 months of storage.

8. The current stipulated residual antioxidant content of 100 ppm at the time of consignment could therefore be adjusted to allow for ethoxyquin's excellent efficacy. This is supported by the results of the negative self-heating test as well as the slow rate of oxidation seen in the results for the oxidation parameters in the stability test.

9. In view of the latest very positive results IFFO proposes that the **residual ethoxyquin level** of 100ppm should therefore be reduced by 50% to 50ppm (instead of the previous request of 60 ppm), taking into account that a **dosage ethoxyquin level** of only 50ppm effectively protects fishmeal against spontaneous combustion. The residual antioxidant levels after 12 months of 48 – 75% along with the additional antioxidant activity of the ethoxyquin breakdown products assures that by measuring a 50 ppm residual ethoxyquin, the sum of residual antioxidant will be more than sufficient to prevent spontaneous combustion.

10. A proposed reduction in the dosage level of ethoxyquin for stabilisation of fishmeal during shipping is important because it assists in the animal feed and protein production sectors' goals of seeking lower inclusions of feed additives in general. This is especially important at a time when the two named antioxidants in the IMO Code are under a process of reauthorisation in the EU, and where authorisation may likely result in the setting of lower maximum levels in feed as well as maximum residue levels in meat and fish. Both ethoxyquin and BHT are in the reauthorisation process.

Proposal

The following Special Provisions for fishmeal (UN 2216) are written into the current UN Model Regulations (Rev 19):

29 This substance is exempt from labelling, but shall be marked with the appropriate class or division.

117 Subject to these Regulations only when transported by sea.

300 Fish meal, fish scrap and krill meal shall not be transported if the temperature at the time of loading exceeds 35°C or 5°C above ambient temperature whichever is higher.

308 Fish meal or fish scrap shall contain at least 100 ppm (mg/kg) of antioxidant (ethoxyquin) at the time of consignment.

The IMDG Code has, apart from the SPs above, the following additional SPs:

907 The consignment shall be accompanied by a certificate from a recognized authority stating: moisture content; fat content; details of anti-oxidant treatment for meals older than 6 months (for UN 2216 only); anti-oxidant concentration at the time of shipment, which must exceed 100 mg/kg (for UN 2216 only); packing, number of bags and total mass of the consignment; temperature of fishmeal at the time of despatch from the factory; date of production.

No weathering/curing is required prior to loading. Fishmeal under UN 1374 shall have been weathered for not less than 28 days before shipment. When fishmeal is packed into containers, the containers shall be packed in such a way that the free air space has been restricted to the minimum.

928 The provisions of this Code shall not apply to:

fishmeal when acidified and wetted with more than 40% water, by mass, irrespective of other factors; consignments of fishmeal which are accompanied by a certificate issued by a recognized competent authority of the country of shipment or other

recognized authority stating that the product has no self-heating properties when transported in packaged form; or fishmeal manufactured from “white” fish with a moisture content of not more than 12% and a fat content of not more than 5% by mass.

945 Stabilization of fishmeal shall be achieved to prevent spontaneous combustion by effective application of between 400 and 1000 mg/kg (ppm) ethoxyquin, or liquid BHT (butylated hydroxytoluene) or between 1000 and 4000 mg/kg (ppm) BHT in powder form at the time of production. The said application shall occur no longer than twelve months prior to shipment.

IFFO recommends to provisionally modify SP 308 as follows to be in line with SP 945 from the IMDG:

SP 308 Stabilization of fishmeal shall be achieved to prevent spontaneous combustion by effective application of ethoxyquin, BHT (butylated hydroxytoluene) or tocopherols (also used in a blend with rosemary extract) at the time of production. The said application shall occur within twelve months prior to shipment. Fish scrap or fish meal shall contain at least 50 ppm (mg/kg) of ethoxyquin, 100 ppm (mg/kg) of BHT and 250 ppm (mg/kg) of tocopherol based antioxidant at the time of consignment.
