

Determination of antioxidants (Irganox 1076 and Irganox 1010) in polyethylene using thermal desorption and reactive pyrolysis – Part 2

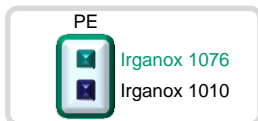
[Background] Irganox 1010 (MW 1178) and Irganox 1076 (MW 530) have been used as an antioxidant in polymeric materials. Determination of each antioxidant in polyethylene (PE) was reported previously in the Technical Note, PYA1-080E, where Irganox 1010 was determined by reactive pyrolysis (RxPy)-GC/MS with tetramethylammonium hydroxide (TMAH) and Irganox 1076, by thermal desorption (TD)-GC/MS. Both of these antioxidants are sometimes co-added in polymeric materials, and it is not easy to determine each antioxidant in such polymeric materials because both antioxidants give the same reaction product by RxPy-GC/MS with TMAH as shown in Fig. 1. Individual concentration of antioxidants in a PE sample containing both antioxidants can be determined according to the analytical protocol given in Protocol 1.

[Experimental] The analytical data was obtained using a Multi-Shot Pyrolyzer (EGA/PY-3030D, Frontier Labs) interfaced directly to the injection port of a GC/MS system. Analytical conditions including amounts of sample and reagent used are identical to those described in the previous technical note (PYA1-080E).

[Results] The concentrations of Irganox 1010 and Irganox 1076 contained in the PE sample was determined to be 1406 ppm (formulated as 1000-1500 ppm) and Irganox 1076 was 476 ppm (formulated as 450-550 ppm), respectively.

Protocol 1: Analytical protocol for the determination of Irganox 1076 and Irganox 1010 co-added in PE.

1. TD-GC/MS is used to determine Irganox 1076 in a PE sample containing both antioxidants.



Determination of Irganox 1076 by TD-GC/MS

2. The methyl derivative of Irganox 1076 produced by RxPy with TMAH is determined by the standard addition of Irganox 1076 to the PE sample containing Irganox 1076 only (Fig. 2), and the determined value is correlated with the concentration of Irganox 1076 obtained in the step 1.
3. By the standard addition of Irganox 1010 to the PE sample containing both antioxidants, concentration of the methyl derivative produced by RxPy with TMAH is determined (Fig. 2). The determined concentration corresponds to the sum of concentrations of Irganox 1010 and Irganox 1076.
4. By subtracting the concentration of Irganox 1076 determined in the step 2 from the sum of concentrations determined in the step 3, the concentration of Irganox 1010 can be determined.

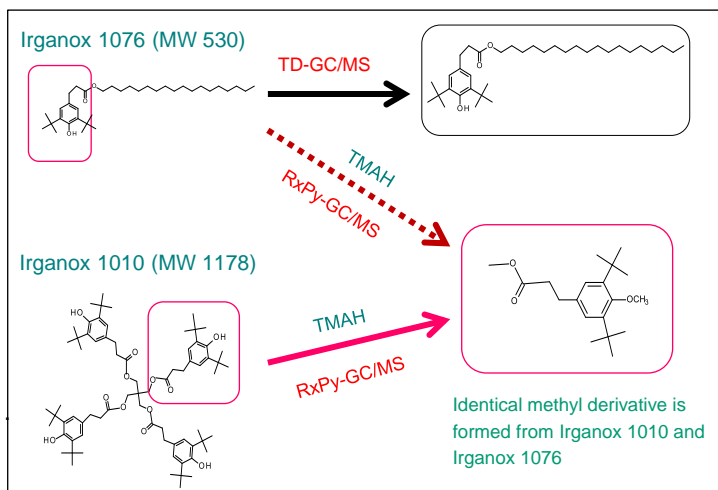


Fig. 1 Methyl derivative of Irganox 1010 and Irganox 1076

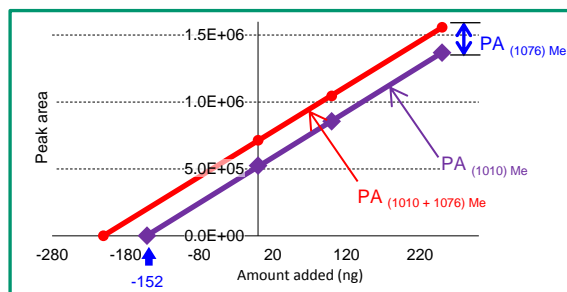


Fig. 2 Calibration curves for the methyl derivative of Irganox 1010

$$PA_{(1010) Me} = PA_{(1010 + 1076) Me} - PA_{(1076) Me}$$

PA: Peak area PA_{Me}: Peak area of the methyl derivative

Keywords : Irganox 1010, Irganox 1076, Additive, Antioxidant, Thermal desorption, Reactive pyrolysis, TMAH

Products used : Multi-functional pyrolyzer, UA⁺-5

Applications : Analysis of additives in polymer

Related technical notes : PYA1-080E

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