

Qualitative Analysis of Additives in a Recycled Polypropylene Using a New MS Library for Additives (ADD-MS06 Library)

[Background] Commercially available MS libraries are generally used to identify compounds detected by GC/MS; however, the number of additives for polymeric materials stored is not sufficient for practical use. Therefore, a new MS library with commercially available 329 major additives was constructed by use of pyrolysis-GC/MS(Py-GC/MS). The library can be useful estimate additives contained in polymeric materials.

[Experimental] The ADD-MS06 Library was constructed by : (a) directly from analyzed data including pyrograms for the standard additive samples obtained by Py-GC/MS at 600°C, (b) mass spectra for the major peaks observed on each pyrogram, and (c) retention indices (RI) of the major peaks for the additive itself and/or its pyrolyzates. Using this library, additive components of a recycled polypropylene (PP) were estimated from its chromatogram obtained by thermal desorption (TD) from 100 to 300°C at 20°C/min followed by GC/MS separation.

[Results] Fig. 1(a) shows the observed TIC for the recycled PP obtained by TD-GC/MS analysis. The search results of the mass spectra for the peaks A and B are shown in Table 1. The both peaks were estimated by narrowing down their candidates by use of the degree of similarity and RI value comparison. Pyrograms of additives stored in the ADD-MS06 Library are shown in Figs. 1(b), (c), and (d). Comparing these data, additives contained in the recycled PP could be estimated as, (1) tris(2,4-di-*t*-butylphenyl) phosphite, or tetrakis(2,4-di-*t*-butylphenyl)-4,4'-biphenylene diphosphonite, an antioxidant for the peak A component, and (2) decabromodiphenyl ether (DeBDE), a flame retardant for the peak B component respectively.

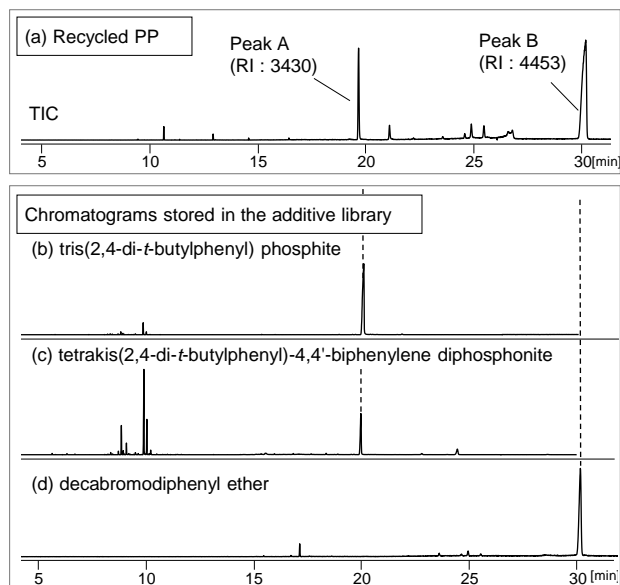


Fig. 1 Chromatograms of PP, and additives stored in MS library
 TD: 100 - 300°C(20°C/min), GC Oven: 40 - 320°C(20°C/min, 4 min) - 340°C(10°C/min),
 He: 1.0 ml/min, Split ratio: 1/50, Amount: 0.3 mg, Col : UA5-30M-0.25F

Table 1 Additive MS library search results

| | Candidates compounds | Degree of similarity[%] | RI |
|-------------------|---|-------------------------|-------------|
| Peak A RI:3430 | 1. tris(2,4-di-<i>t</i>-butylphenyl) phosphite | 62 | 3451 |
| | 2. 2,4,8,10-tetra- <i>t</i> -butyl-5,7-dioxo-6-phospha-dibenzo[a,c]cyclohepten-6-ol | 55 | 2888 |
| | 3. 2- <i>t</i> -butyl-6-methyl-4-ethenylphenol | 1 | 1535 |
| Peak B RI:4453 | 1. decabromodiphenyl ether | 89 | 4412 |
| | 2. isomer of bromophenyl heptabromodiphenyl ether | 13 | 3738 |
| | 3. tetrabromo phthalimide | 5 | 2814 |

Keywords : Additives, MS Library, RoHS, DeBDE, PBDE, Recycled PP

Products used : Multi-functional pyrolyzer, Vent-free GC/MS adapter, F-Search, UA-5

Applications : General Polymer Analysis, Additives

Related technical notes :

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