

Additives Analysis for Polymers and Plastics in Toray Research Center

Additives contained in polymers, plastics, which is one of the items analysis is required widely. Based on the accumulated preparation know-how or analytical skills in , Toray Research Center we shall provide advanced services for identifying unknown additives.

Choice of Analytical Method

Sample information

Polymer Species

- Sample amount
- Sampling position

Solvent solubility

Additives
(Stabilizer, Function imparting agent, Reactant, e.g.)

Objective (Analytical Approach)

Additive composition (Qualification)

Specification of unknown Compounds , Analysis of degradation products

Additive content (Quantitation)

Correlation analysis among physicality, performance Investigation of manufacturing loss

Choice of Preparation method

Solvent-soluble

Separation of additives from Polymers With Dissolution and re-precipitation

Solvent-insoluble

Chromatographic Fractionation, If necessary

Solvent extraction

※Quantitative values should be handled with care, because it is difficult to extract the entire amount in sample.

Choice of Measurement and evaluation

Additive Mw

High
10⁴
2000
500
Low

MALDI-MS
Py GC/MS, SEC, HPLC
HPLC(UV,ELSD,CAD)
LC/MS
GC, GC/MS

NMR

small ← → large Content

Other measurements

Detection of Characteristic elements, which is included in additives

Quantitation of specific elements

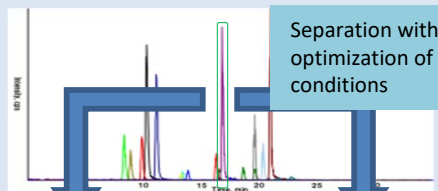
Detection of active HALS (Hindered amine stabilizer)

ESR

Suggested Analysis Flow Examples and Results

Qualification of anti-oxidant, UV-absorbent

- LC/PDA, HR-LC/MS, GC/MS



UV spectrum

High-Resolution MS spectrum
MS/MS spectrum

skeletal structure

Formula, Partial structure

Identification with additive spectra database accumulated in Toray Research Center

Quantitation of Surfactants , HALS, which have a molecular weight distribution
● LC/CAD, LC/ELSD, LC/MS/MS

Sample

Preparation

(Separation of additives from polymers)
Crushing, centrifugal separation, drying, solvent extraction etc.

Step1

Qualification

Spectroscopic Analysis

Database Search

Identification

Step2

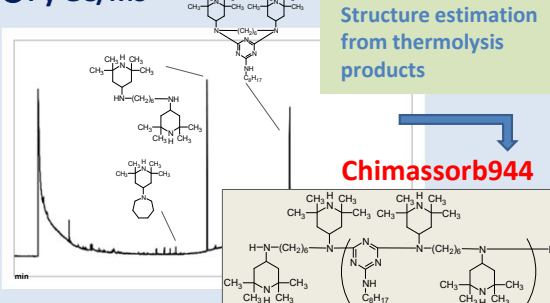
Quantitation

High-sensitive detection

It is important to apply multiple measurements for complementary analysis

Qualification of HALS including high-molecular types

- Py GC/MS



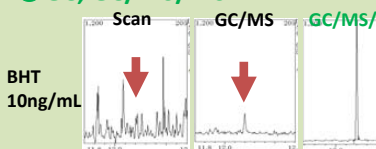
Reaction Py GC/MS result (TICC)

- MALDI-MS of GPC fractions, HR-LC/MS/MS

Possible to identify high-molecular types , polymer-binding additives

Quantitative analysis of anti-oxidant

- GC, GC/MS/MS



High-sensitive detection with MS/MS optimization

- LC/UV, LC/CAD, LC/ELSD, LC/MS/MS

Examples of Analytical Results

Species	Classification of Additives		Examples of analytical results
PP, PE, PS, ABS PC, PET, PBT, PA Gum (SBR,EPDM) , e.g.	Stabilizer	Primary antioxidant	Phenol-based
		Secondary antioxidant	phosphoric acid-based , Sulfur-based
	Function imparting agent	UV absorbent	Benzotriazole-based, Benzophenone-based, Polymer-binding type
		HALS(Hindered amine light stabilizer)	N-H type, N-CH ₃ type, NO-alkyl type, High-molecular type
Others	Surfactants, Polymerization initiator or inhibitor, Flame retardants, Antibacterial agent, e.g.		PEG 0.1%, MEHQ 0.02%