LC/MS Application Notes:
EI/APCI Identification and Quantitation of Polymer Additives

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Instrumentation

Chromatogram

MS Spectrum

Calibration Curve

Quantitation

Key Words:

Polypropylene

Additives

EI/APCI

The analysis of polymer additives has always been important to some industries (polymer, food packaging, medical device, chemical, inks/coatings).   However, the identification and quantitation of polymer additives has been rather elusive. Reports of polymer additive analysis by single quadrupole LC/MS are scarce. This work demonstrate how quantitative and qualitative analysis of polymer additives can be accomplished by the complementary LC/MS techniques: Electron Ionization (EI) and API (API).

Instrumental Conditions

HPLC

- Waters Alliance Solvent Delivery System
- Symmetry® C_8 (3.9 x 150 mm) with Acetonitrile/Water gradient
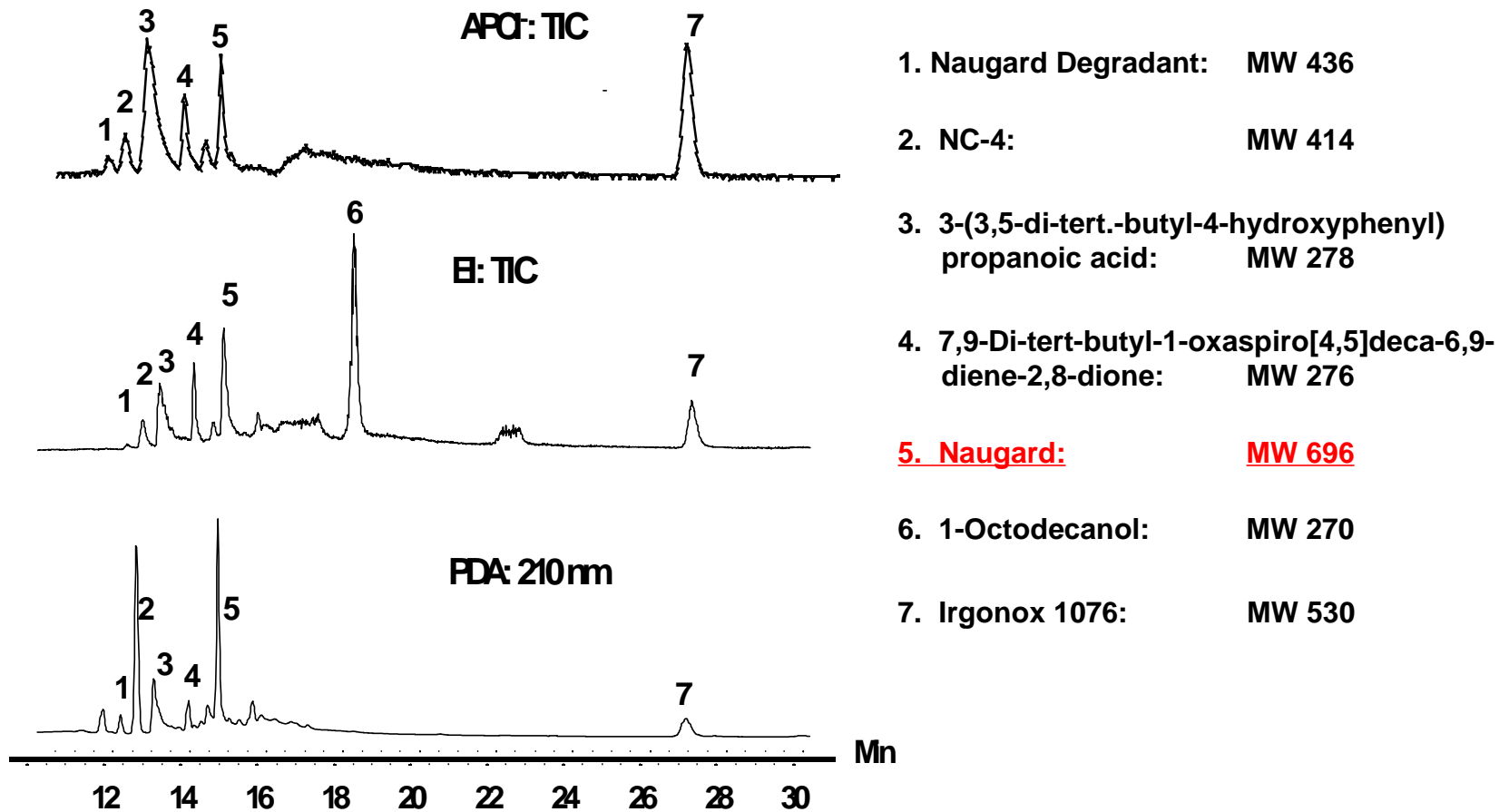
- 10 μL injection 0.4 mL/min. flow rate

EI

- Waters Integrity ThermaBeam™ System

   - Library search yields MW information

     proposed structures

API

Micromass PlatformLC System

APCI positive and negative

- Full scan provides MW

- Selected Ion Recording (SIR) provides quantitation

Figure 1: Sample Identification

1. Naugard Degradant: MW 436

2. NC-4:   MW 414

3. 3-(3,5-di-tert.-butyl-4-hydroxyphenyl)
   propanoic acid: MW 278

4. 7,9-Di-tert-butyl-1-oxaspiro[4,5]deca-6,9-
   diene-2,8-dione: MW 276

5. **Naugard:**   **MW 696**

6. 1-Octodecanol: MW 270

7. Irgonox 1076:   MW 530

Quantitation

- The quantitation was obtained by positive APCI SIR acquisition
- There were four standards commercially available
- Four calibration curves were obtained with a representative curve shown on Figure 3

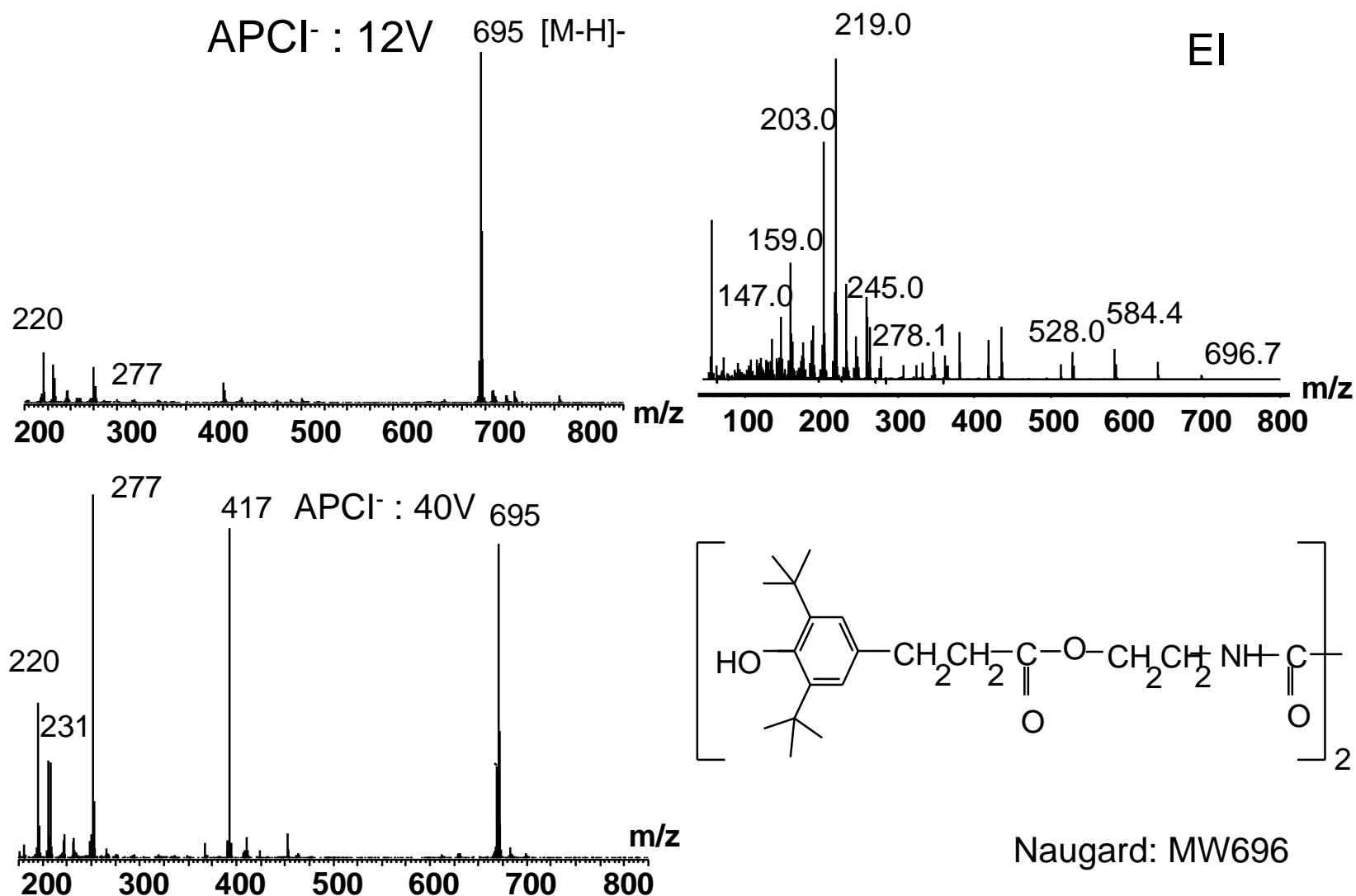|	LOD (ppb)	Linear Range (ppb)	% in Polymer (w/w)
NC-4	30.4	30.4 - 7600	0.07
Naugard-XL	14.4	14.4 - 3600	0.04
Octadecanol	27.2	27.2 - 6800	0.4
Irgonox 1076	12.8	12.8 - 3200	0.06

Figure 2: Identification of Peak #5

Figure 3: Calibration Curve of Naugard-XL

Coefficient of Correlation: 0.997561
Calibration curve: 16531 * x + 135.558
Respons: External Std, Area

m/z 473

14.4 - 3600ppb