Monitoring Volatiles in Food Contact Packaging by Purge & Trap GC/MS

Rebecca E. Wittrig
Restek Corporation
110 Benner Circle
Bellefonte, PA 16823

Restek Corporation
www.restekcorp.com

Volatiles in Food Contact Packaging

- Reasons for testing
- FDA regulations
- Method development
- Linearity data
- Case studies
- Summary

Why Test Food Contact Packaging?

- Ability of compounds to migrate into or out of packaging materials
  - Styrene at trace levels in food can give a “plastic” taste
- Constant stream of new packaging choices
- Popularity of convenience foods!
  - food preparation often takes place within the package
- Changes in the food or ingredients used
  - e.g. change in the fat content
- Changes by the packaging manufacturer

Why Test Food Contact Packaging?

"Anyone manufacturing food contact articles for use in the home or in food service establishments should make sure that nothing from the articles imparts flavor, color, odor, toxicity, or other undesirable characteristics to food, thereby rendering the food adulterated."

Source: Requirements of Laws and Regulations Enforced by the US FDA (1997).

Regulation of Food Contact Articles

- Food & Drug Administration
  - 21 CFR 170.39
  - Outlines data needed in requests for exemption as food additives (use conditions, time/temperature, food type, and single or repeat usage)
- Migration Studies
  - Worst-case (time/temperature) intended use conditions
  - Food simulating solvents are used
- Residual Studies
  - Level of the substance in the finished food-contact article
  - Used to estimate a worst-case dietary concentration level (assume 100% migration)

Testing Methodologies

- FDA Guidelines under 21 CFR 170.39
  - Detailed description of analytical method used
  - Validation data (including the detection limit)
  - Conditions of use for the substance
- ASTM Methods F1308-98 and F1519-98
  - Qualitative and quantitative procedures for microwave susceptors
  - Microwave headspace sampling vs. purge & trap sampling
  - Volatile extractables are defined as substances released from the susceptor and detected in the headspace
  - **Extractability does not necessarily mean migration to the food**

Method Development - Modeling the Separation

- A target list of volatiles was developed
- The column chemistry was selected
- Initial separation parameters were entered into ezGC™
- The separation was optimized:
  - Column geometry
  - Oven program
  - Linear velocity
- A trial run was performed using the calculated parameters

Optimized Run Conditions

GC Parameters

- Column: Rtx-5MS, 30m x 0.25mm x 1.0um
- Injector: 250°C, 20:1 split
- Carrier gas: Helium at 1 mL/min, constant flow
- Oven: 50 °C to 92 °C at 3 °C/min, to 220°C/min. at 20°C/min. (1 min. hold)

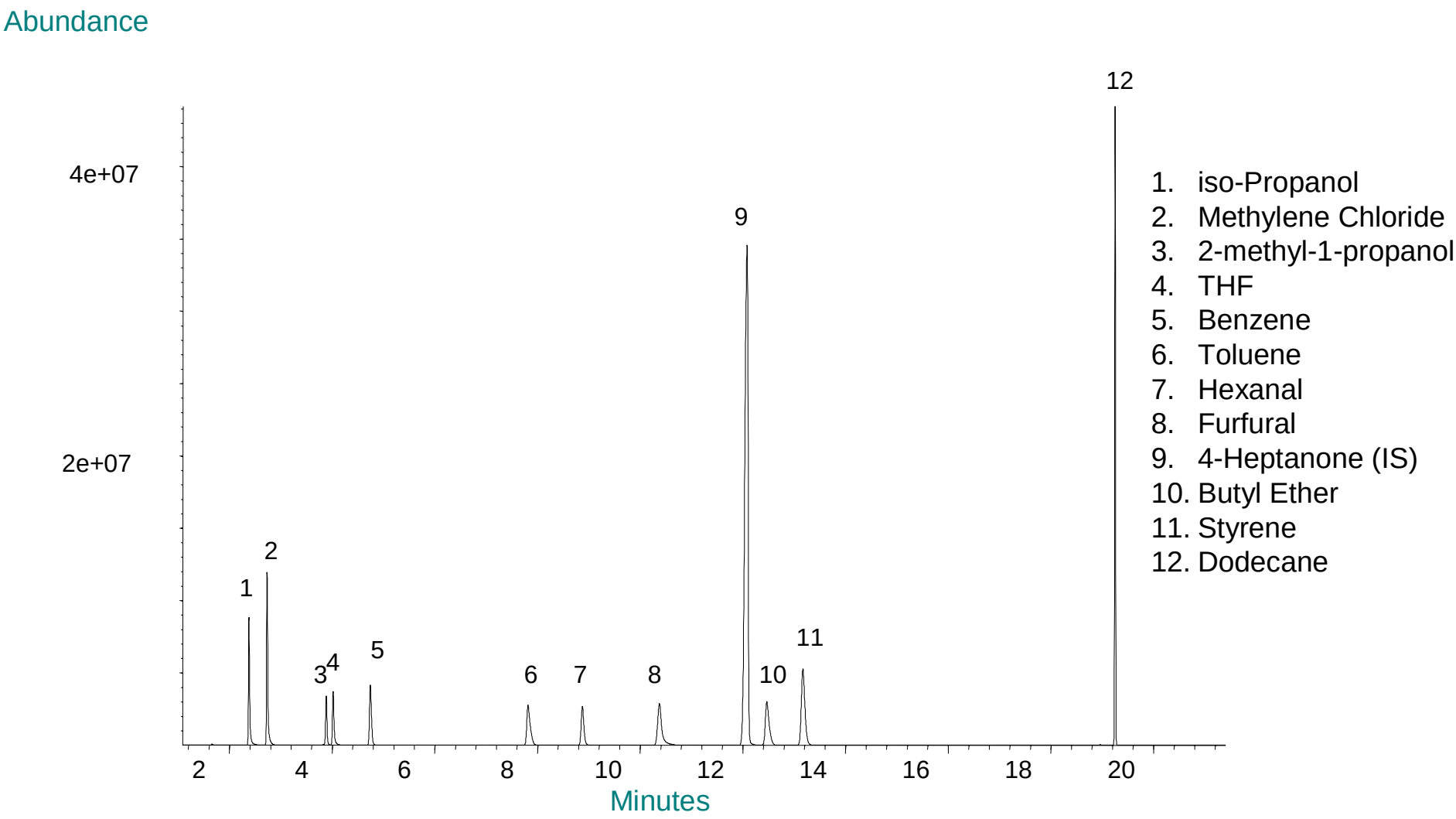
MSD Parameters

- Temperature: 280°C
- Scan Range: 35-260, 1 min. solvent delay
- Ionization: EI @ 70eV

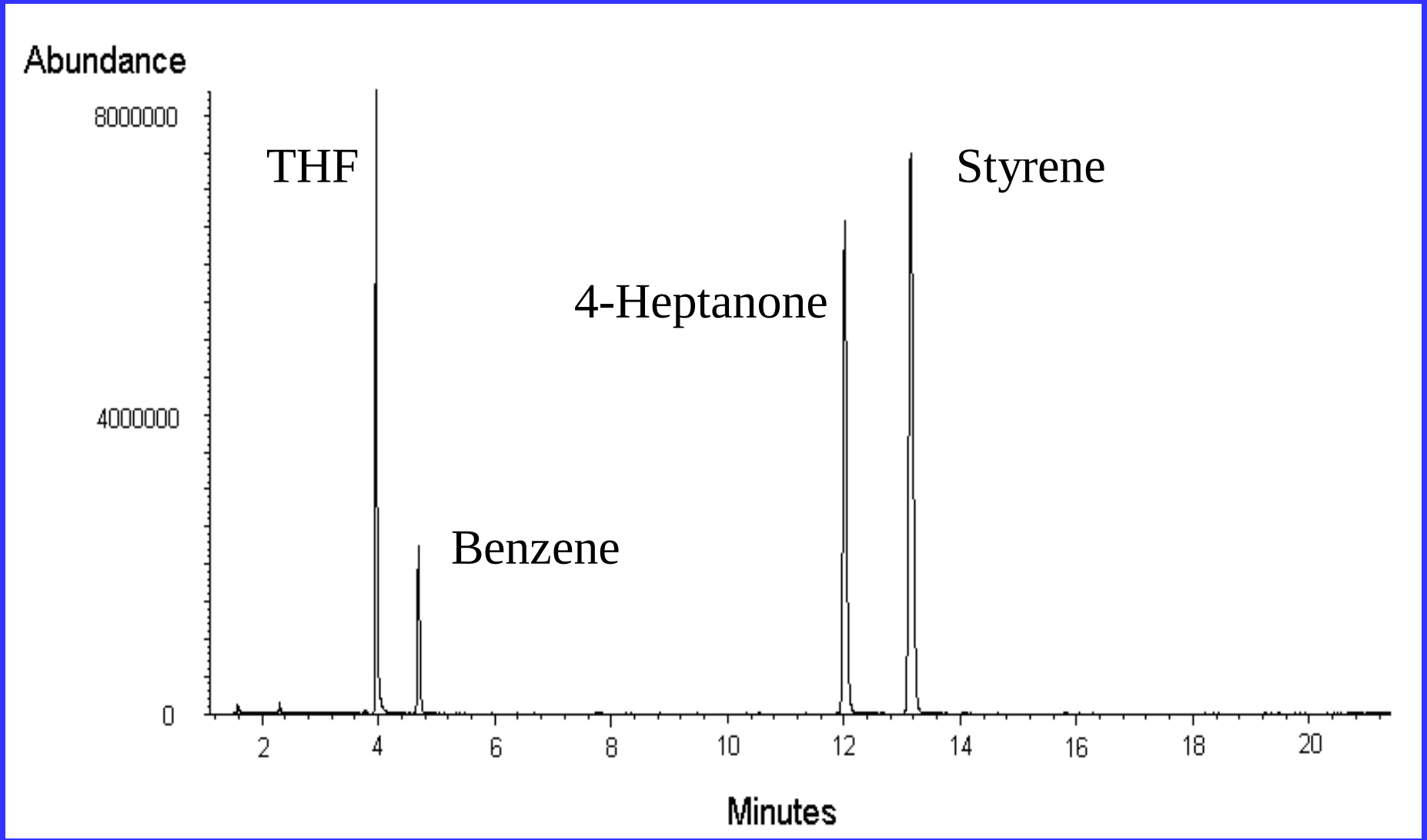
Purge & Trap Parameters

- Concentrator: Tekmar LSC-3100 with Vocarb 3000 (type K) trap
- Purge: 10 min. at 40 mL/min, 60°C
- Dry purge: 3 min. at 40 mL/min.
- Desorb: 2 min. at 40 mL/min, 245°C

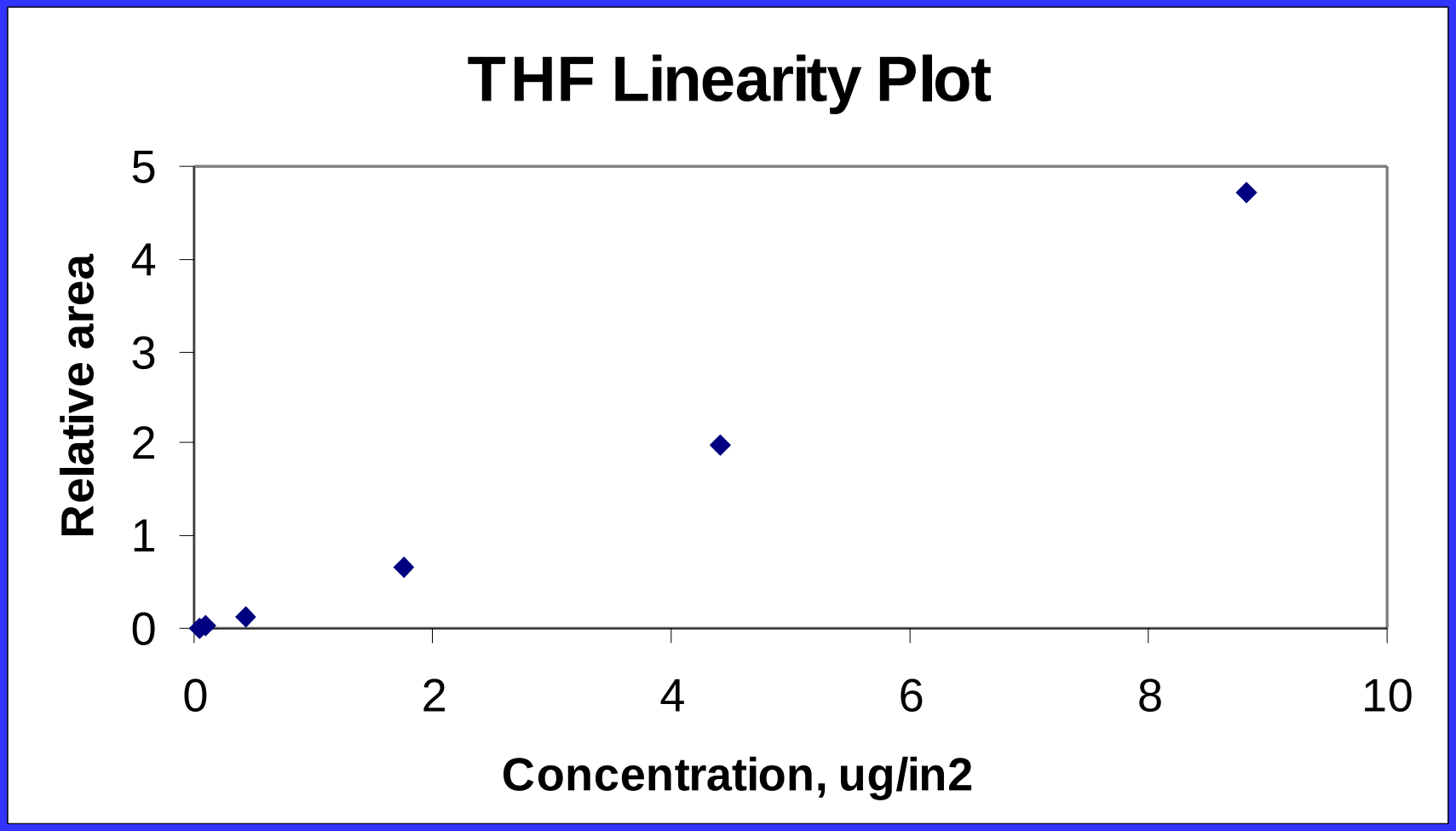
Volatiles Standard on Rtx-5MS



Volatiles Linearity Standard on Rtx-5MS



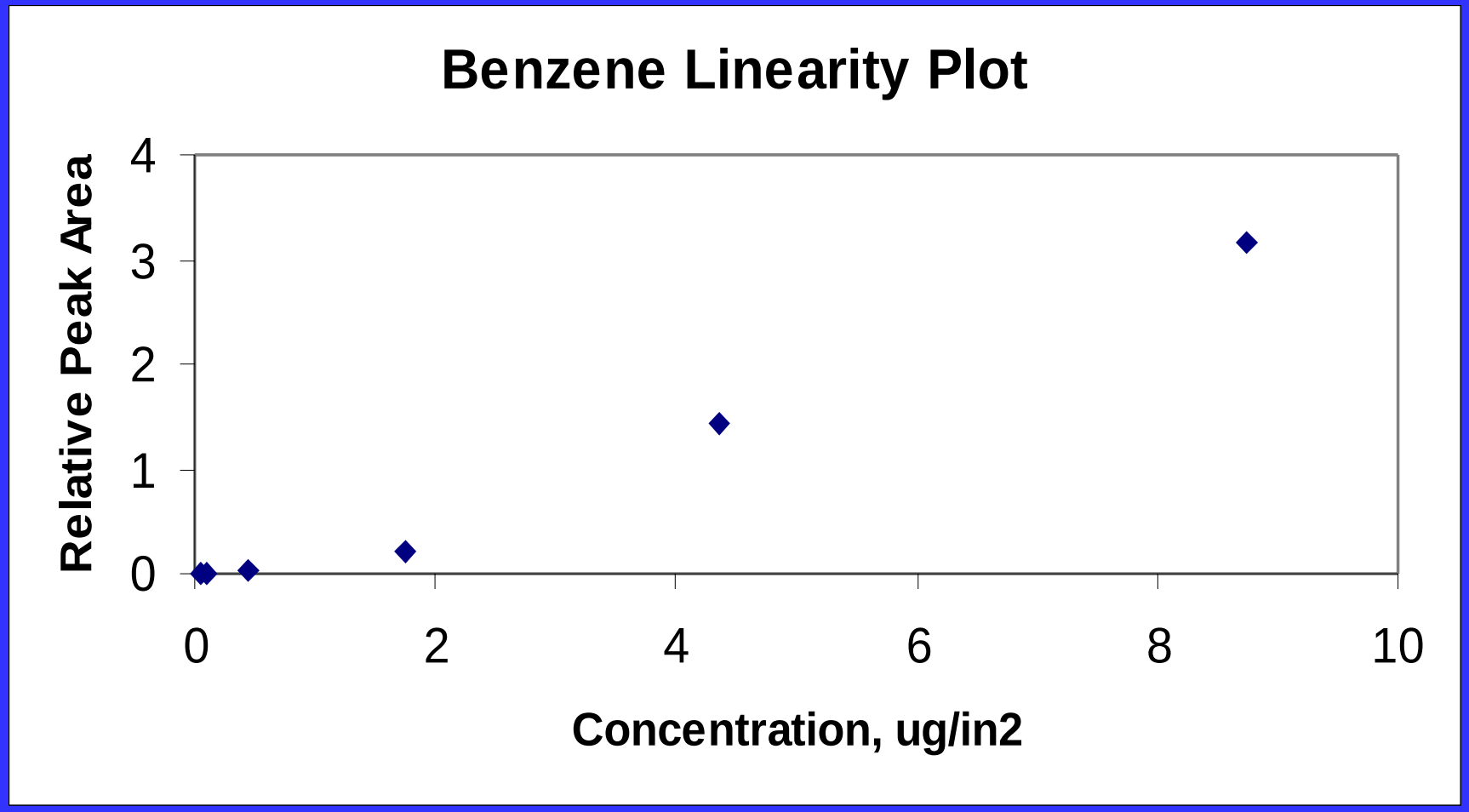
THF Linearity



Concentration range tested: 0.04 – 8.8 ug/in^2

$R^2 = 0.992$

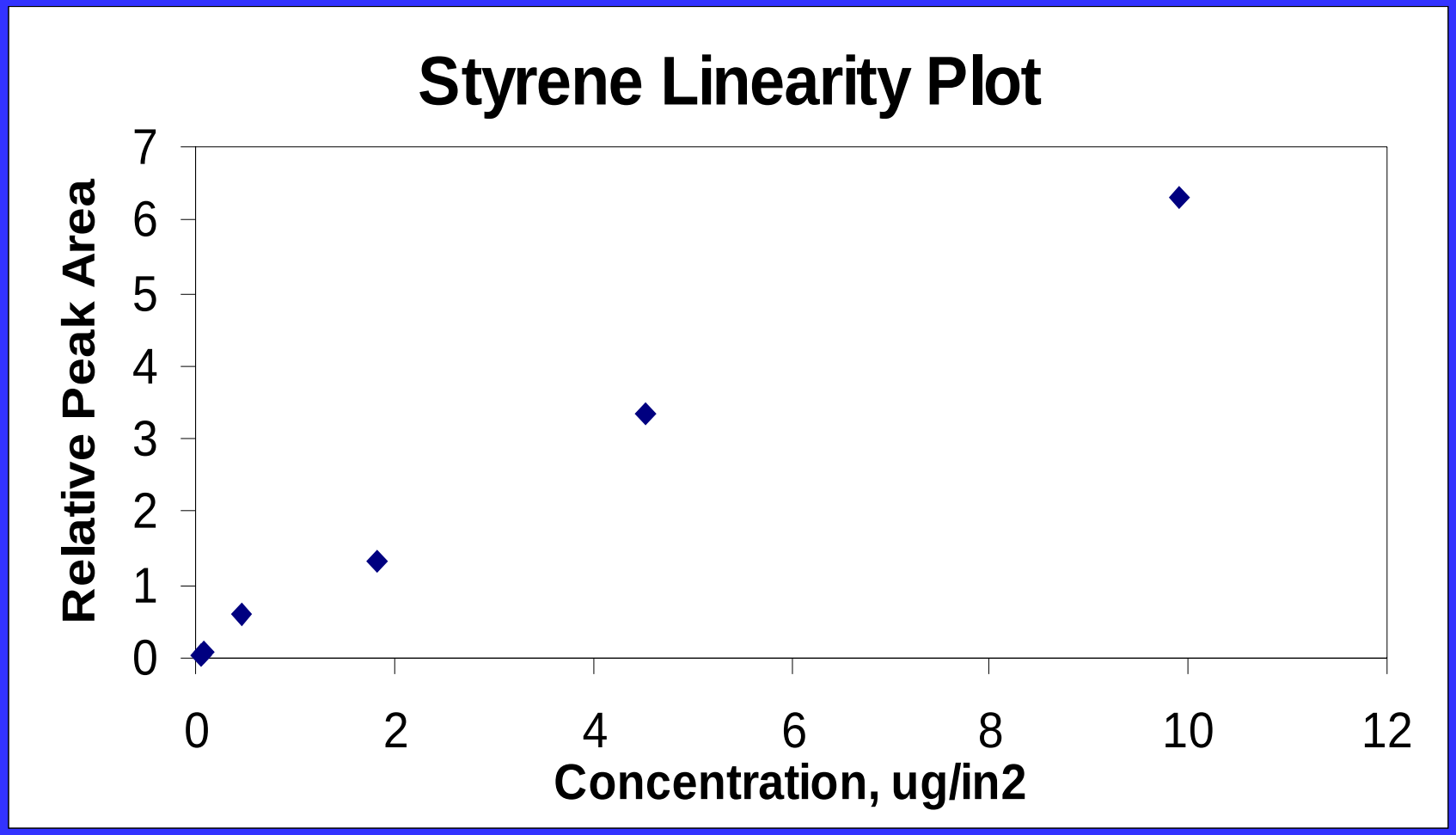
Benzene Linearity



Concentration range tested: 0.04 – 8.7 ug/in^2

$R^2 = 0.985$

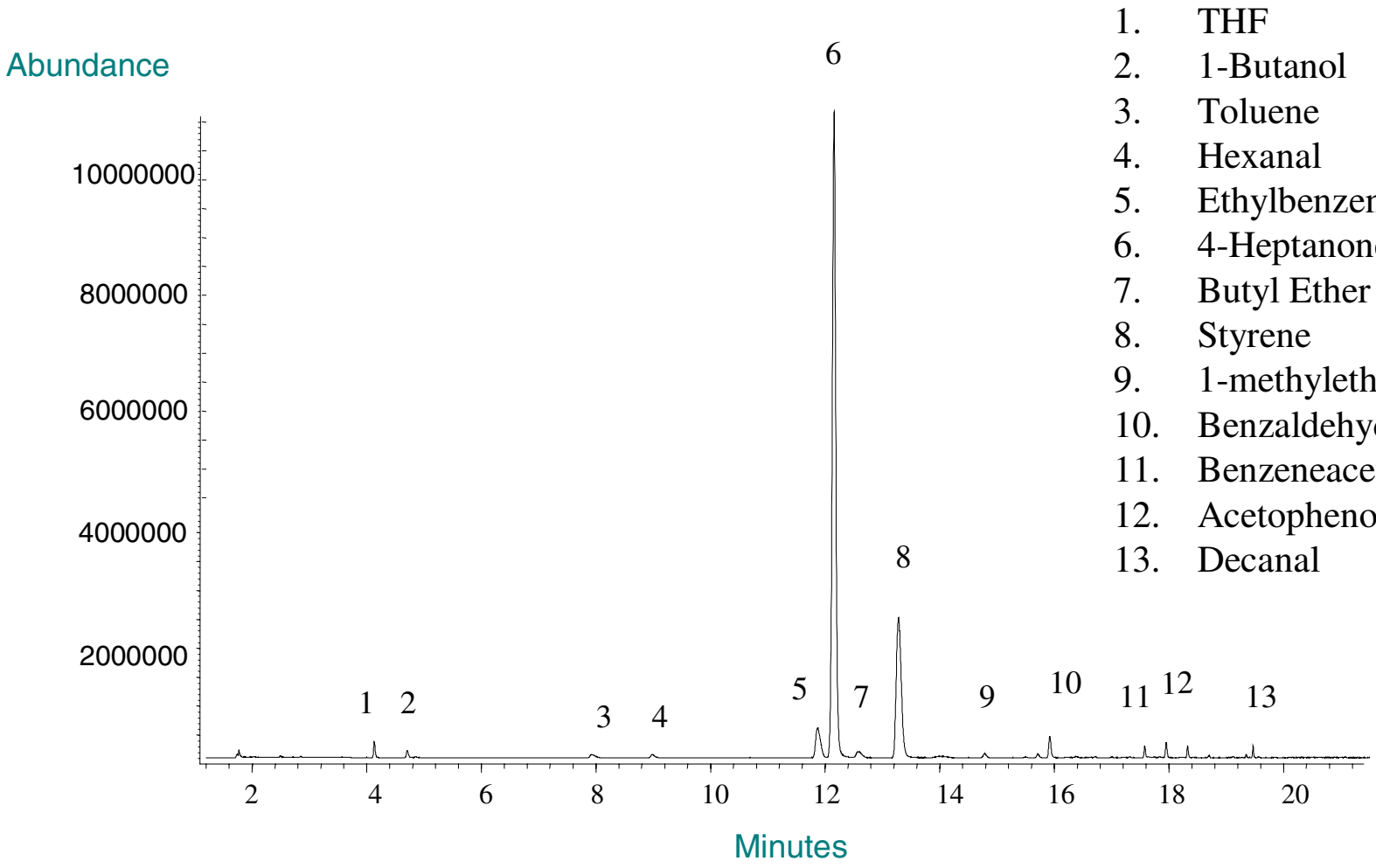
Styrene Linearity



Concentration range tested: 0.05 – 9.9 ug/in^2

$R^2 = 0.994$

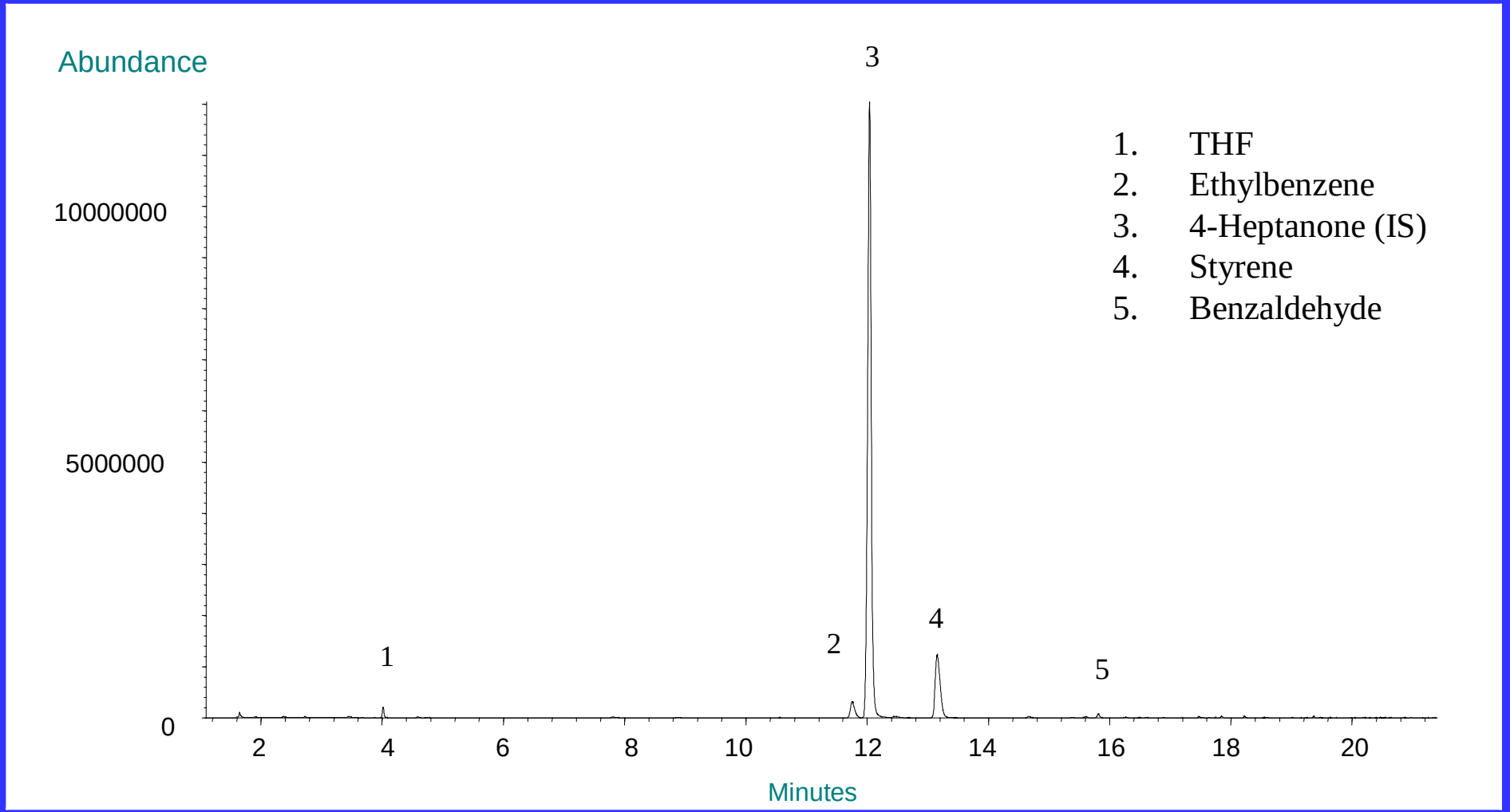
Packaging Label Overwrap at Seam



1. THF
2. 1-Butanol
3. Toluene
4. Hexanal
5. Ethylbenzene
6. 4-Heptanone (IS)
7. Butyl Ether
8. Styrene
9. 1-methylethylbenzene
10. Benzaldehyde
11. Benzeneacetaldehyde
12. Acetophenone
13. Decanal

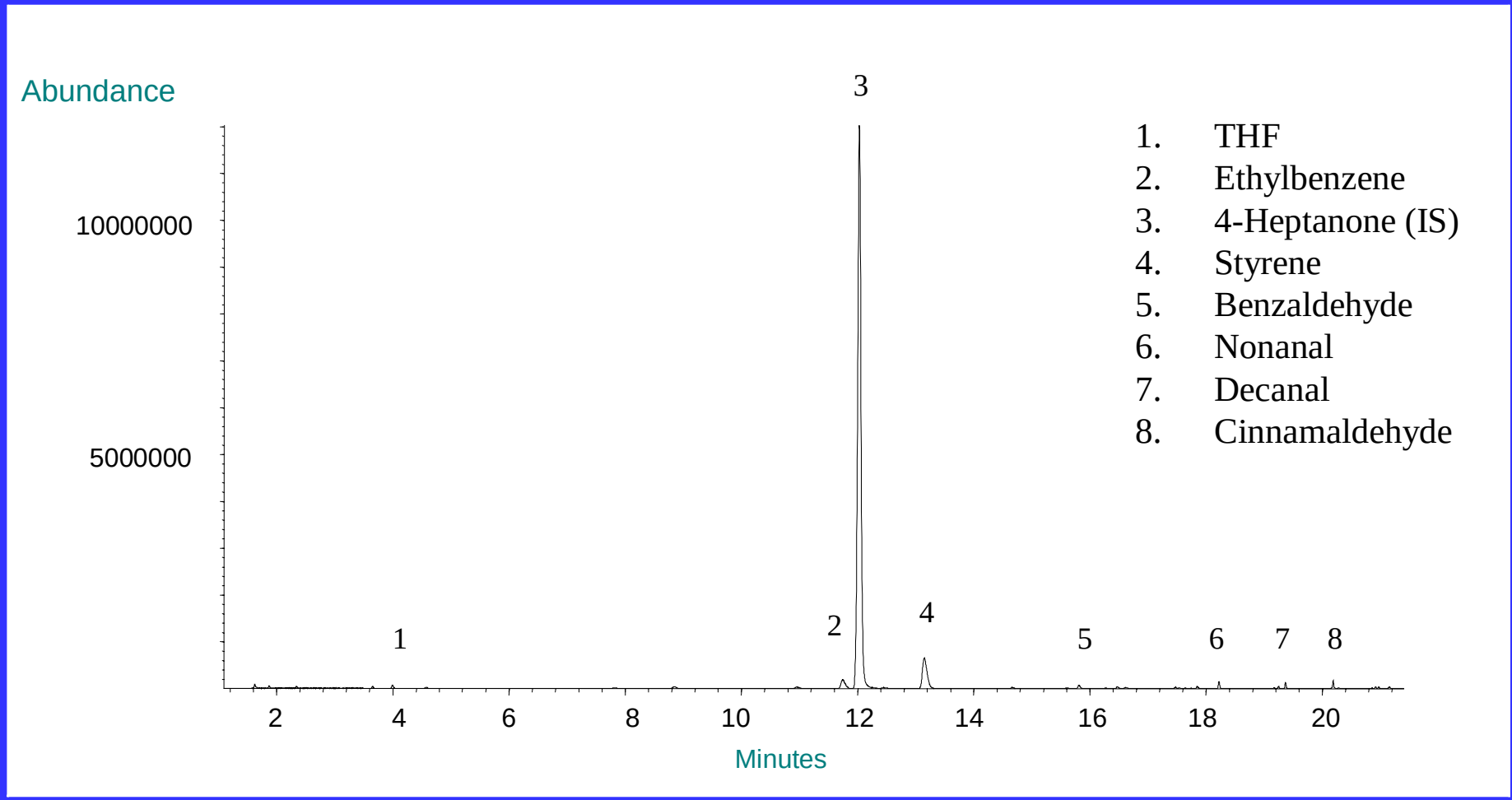
Example: Microwavable bowl with polystyrene label and plastic lid

Packaging Label Overwrap without Seam



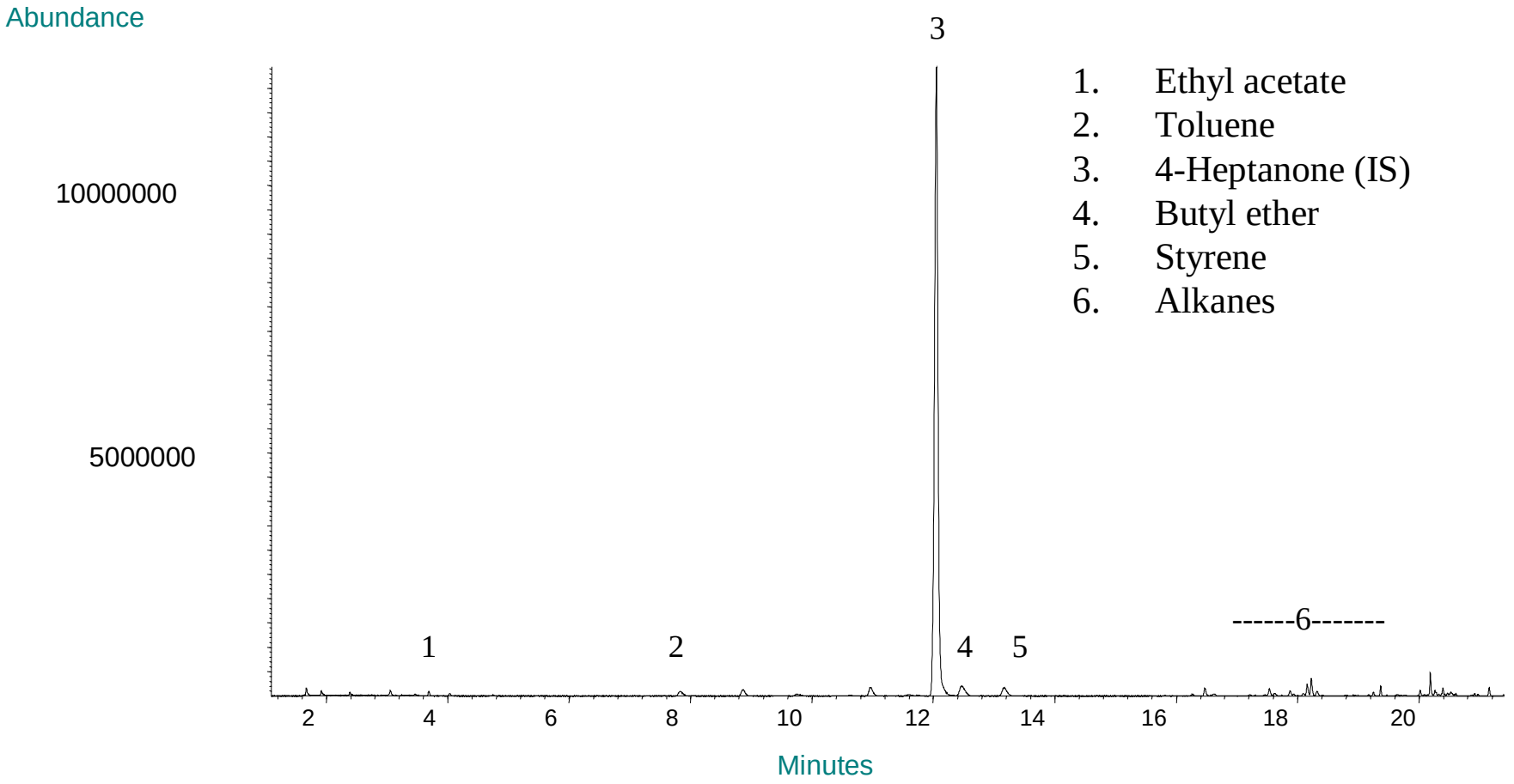
Example: Microwavable bowl with polystyrene label and plastic lid

Lid of Microwavable Package



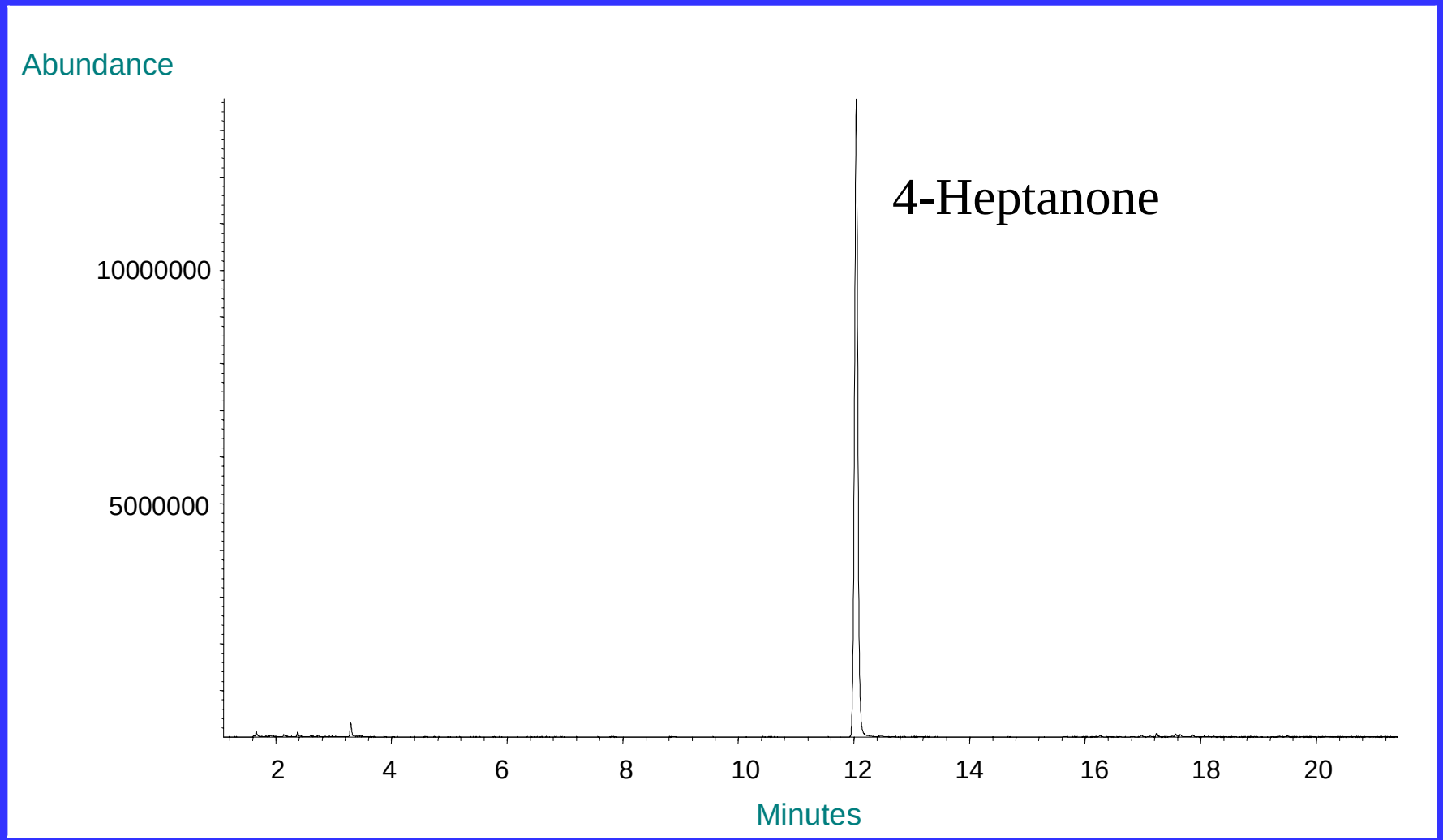
Example: Microwavable bowl with polystyrene label and plastic lid

Inner Bowl



Example: Microwavable bowl with polystyrene label and plastic lid

Microwave Popcorn Bag



Example: Microwavable popcorn bag

Summary

- Reasons for testing food contact packaging
- Optimized separation of common volatiles from packaging using purge & trap GC/MS
- Linearity studies for three target solvents
- Packaging examples
- Information available from the FDA
  - 21 CFR 170.39
  - Information on previous requests for exemption