RAPID ANALYSIS OF POLYMER ADDITIVES USING UPLC® WITH PDA AND ELS DETECTION

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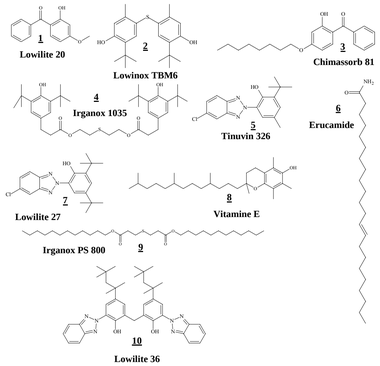
Solution for Polymer Additive Analysis

- *Resolution*
- *Sensitivity*
- *Speed*
- *Innovation*

Experiment

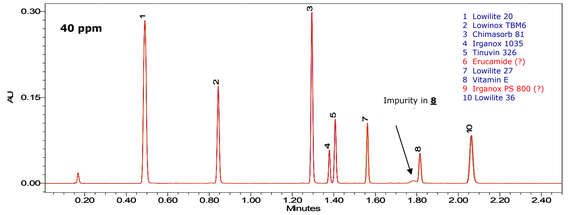
2.5 minute analysis of a blend of 10 UV & non-UV absorbing polymer additives



Instrument and Operation Conditions

LC:	ACQUITY UPLC®/PDA and ELS detectors		
Software:	Empower™		
Column:	ACQUITY UPLC BEH C_{18} 2.1 mm x 50 mm		
Weak & Strong Wash Solvent:	CH_3CN (600 µL)		
Mobile Phase	A: 0.05 v % of TFA in H_2O		
Mobile Phase	B: 0.05 v % of TFA in CH_3CN		
Gradient:	50% B to 100% B in 1.4 minutes, then hold for 1.1 minutes at 100% B		
Flow Rate:	1 mL/min		
Sampling Rate:	20 pt/s		
Injection Volume:	2 µL (full loop)		
Column Temperature:	50 °C		
PDA:	210 to 500 nm		
PDA Filter Response:	0.1 sec		
ELSD Parameters:			
Gain:	500	Nebulizer:	Cooler
N_2 Gas pressure:	40 psi	Date Rate:	20 pt/s
Drift tube temperature:	57 °C	Time Constant:	0.1

Overlay 7 Replicate Injections: PDA Timed Chromatograms



UPLC® RT Reproducibility

Table 1.
Component Summary For Retention Time

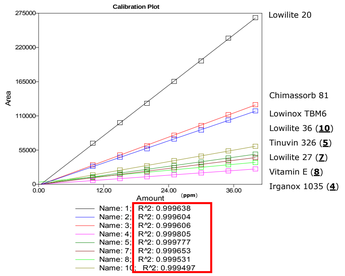
	1 (min)	2 (min)	3 (min)	4 (min)	5 (min)	7 (min)	8 (min)	10 (min)
1	0.490	0.843	1.295	1.379	1.408	1.561	1.815	2.063
2	0.490	0.843	1.294	1.378	1.406	1.562	1.814	2.061
3	0.490	0.844	1.295	1.380	1.408	1.561	1.815	2.062
4	0.490	0.843	1.294	1.379	1.407	1.562	1.815	2.062
5	0.490	0.844	1.295	1.379	1.408	1.561	1.815	2.062
6	0.490	0.844	1.295	1.379	1.407	1.561	1.815	2.062
7	0.490	0.844	1.295	1.380	1.408	1.563	1.816	2.064
Mean	0.490	0.843	1.295	1.379	1.407	1.562	1.815	2.062
Std. Dev.	0.000	0.000	0.001	0.001	0.001	0.001	0.001	0.001
% RSD	0.07	0.05	0.04	0.04	0.04	0.05	0.04	0.05

UPLC® Area Reproducibility

Table 2.
Component Summary For Area

	1	2	3	4	5	7	8	10
1	267836	115147	176434	31584	68465	60975	39276	89901
2	268385	115152	176419	31765	68629	60937	38960	90615
3	268179	115335	176468	31478	68294	61050	39159	90031
4	267946	115169	176208	31460	68318	61092	38741	89953
5	268062	115090	176213	31482	68239	60838	38936	89671
6	268178	115058	176268	30975	67542	60974	38958	89741
7	268383	115191	176518	31568	68415	61157	38747	90225
Mean	268139	115163	176361	31473	68272	61003	38968	90020
Std. Dev.	207	88	128	243	347	106	197	320
% RSD	0.1	0.1	0.1	0.8	0.5	0.2	0.5	0.4

UV/PDA Calibration Curves



What Do All These Products Contain?

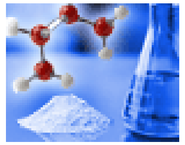
They all contain polymer additives that give desired properties

Types of Polymer Additives:

- Biocides
- UV absorbers & light stabilizers
- Plasticizers
- Lubricants & mold release agents
- Dyes
- Antioxidants & heat stabilizers
- Flame retardants
- Anti-static & conductive agents

Need for Polymer Additive Analysis:

- The correct amount of additives ensures
  - Product physical properties
  - Processing ease
- Competitive product deformulation
- New product development
- Protect health & safety
  - Determine migration into food/medicine



UV Spectra

Extracted from PDA Chromatograms for Library & Peak Purity:

- Confirm peak identity
- Assure non-coelution of spectrally dissimilar components

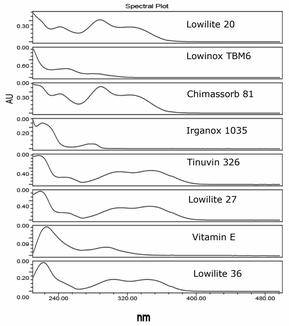
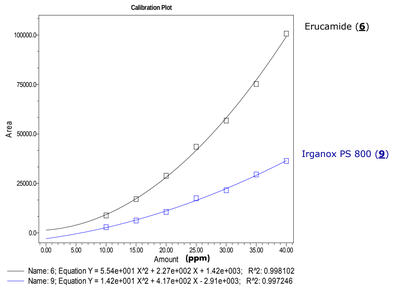


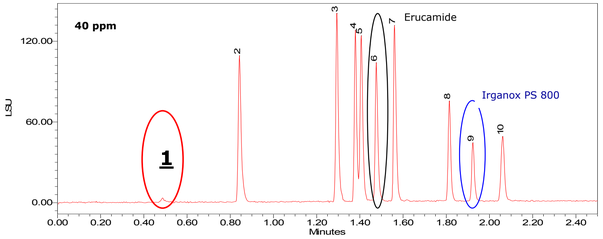
Table 3. Peak identification and quantification

	Component	Match1 Spect. Name	Match1 Angle	< Match1 Threshold	Purity1 Angle	< Purity1 Threshold	Amount (ppm)
1	1	Lowilite 20	0.058	1.084	0.121	0.352	10.10
2	2	Lowinox TBM6	0.268	1.192	0.134	0.416	10.24
3	3	Chimassorb 81	0.054	1.072	0.110	0.349	10.22
4	4	Ingranox 1035	1.215	1.969	0.769	1.104	10.13
5	5	Tinuvin 326	0.054	1.066	0.088	0.344	10.01
6	7	Lowilite 27	0.076	1.067	0.156	0.364	10.22
7	8	Vitamine E	4.054	1.382	0.811	0.768	9.95
8	10	Lowilite 36	0.083	1.113	0.197	0.435	10.07

ELS Quadratic Fit Calibration Curves



Overlay 7 Replicate Injections: ELS Chromatograms



Conclusions

- **ACQUITY UPLC® with PDA and ELS Detectors**
  - 2.5 minutes separation of 10 common polymer additives
  - Excellent reproducibility
    - Peak areas and retention times
  - Great sensitivity
    - Quantify polymer additives
  - Quantify UV & non-UV absorbers in a single run
  - Faster run times
    - System available for more analyses
- **Empower™ 2 Automation from Data Collection to Reporting**
  - Peak identification
  - Peak purity confirmation
  - Calibration plot and quantification

Reference

1. P. J. Lee, A. J. Di Gioia, *ACQUITY UPLC™ WITH PDA AND ELS DETECTION: POLYMER ADDITIVES*, Waters Corporation, Application Note 720001881EN, 2006.
Available for download at www.waters.com

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