

A reliable and routine LC/MS/MS
**Quantitation of Multiclass PGRs in Grapes Using
Agilent Triple Quadrupole LC/MS coupled with an
Agilent 1290 Infinity LC**

FOOD SAFETY



The Agilent 6460 Triple Quadrupole LC/MS System provides high sensitivity and simple, fast, and economic sample cleanup for simultaneous quantitation of multiclass PGRs in grapes



Plant hormones, or phytohormones, are chemicals that regulate plant growth. Manmade compounds called PGRs are used to regulate growth of cultivated plants, weeds and in vitro grown plants and plant cells. There are five major classes: abscisic acid, auxins, cytokinins, ethylene, and gibberellins. PGRs activate cellular response including cell death. Some can adversely affect human cancer cells. European Union testing labs found chlormequat in imported grapes from India, which triggered additional testing for PGRs.

Multiresidue monitoring of PGRs is challenging due to the diversity of their chemical properties. These compounds are thermally unstable and therefore not amenable to gas chromatographic analysis without derivatization. Matrix co-extractives interfere with the target HPLC-UV signals, lowering the sensitivity of PGR analysis by high performance liquid chromatography (HPLC). Moderate to high aqueous solubility restricts multiresidue recovery by phase separation with organic solvents and makes

the sample cleanup difficult due to variable affinity of different PGRs towards different cleanup agents or adsorbents.

This note describes the simple, fast, and economic sample cleanup process and time segmented MRM scan for simultaneous quantitation of multiclass PGRs in grape samples.

The Approach

An Agilent 1290 Infinity LC with an Agilent ZORBAX Rapid Resolution High Definition (RRHD) and an Agilent ZORBAX Extended C18 column interfaced to the Agilent 6460 Triple Quadrupole LC/MS system provided excellent retention time reproducibility and high peak capacity that is required for the quantitation of multiclass PGRs in grapes.

Compounds

- Chlormequat, daminozide, zeatin, kinetin, 6-benzyldehyde, IAA, IBA, forchlorfenuron, paclobutrazo, GA3, abscisic acid, 2-4-D and d4-chlormequat as ISTD

Key Benefits

- Results shows very good sensitivity and linearity for quantitation of PGRs such as chlormequat in grape samples using Agilent 6460 Triple Quadrupole LC/MS with Jetstream ESI ion source and Agilent 1290 Infinity LC
- Simple, economic, and rugged sample preparation using dispersive C18 ODS bulk without any derivatization process
- Simultaneous detection of multi-class PGRs without sacrificing sensitivity



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Figure 1 shows good chromatographic separation of nine PGRs in positive mode and three PGRs in negative mode.

Linearity and repeatability for all standards is within acceptable limits. Figure 2 shows the linearity graph for chlormequat. Precision across all levels was within 2.0%, and the linearity value was $r^2 = 0.996$.

Dispersive C18 sample cleanup techniques show good recoveries of 80 to 120% for standards spiked and extracted from grapes (Figure 3).

Summary

The Agilent 6460 Triple Quadrupole LC/MS system with Jetstream ESI ion source coupled with an Agilent 1290 Infinity LC and Dispersive C18 ODS sample cleanup shows excellent sensitivity, linearity, and recoveries for the quantitation of multiclass PGRs in grapes.

Acknowledgment

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Reference

1. P. Stone, Y. Chen, J. Cappozo "Determination of Aflatoxins (B1, B2, G1, G2) in food matrices using Triple Quadrupole LC/MS system," Agilent Technologies Santa Clara, CA, NCFST- USA.

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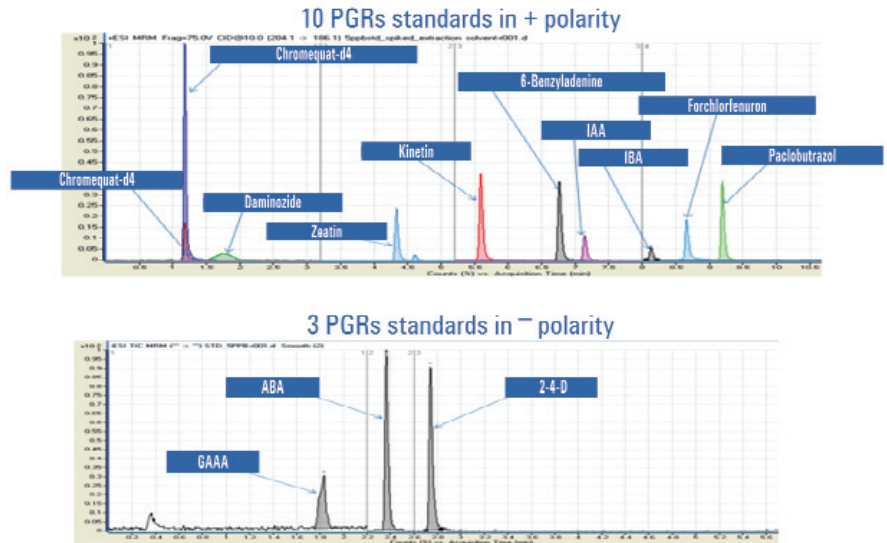


Figure 1. Good chromatographic separation of all PGRs at 5 ppb concentration level.

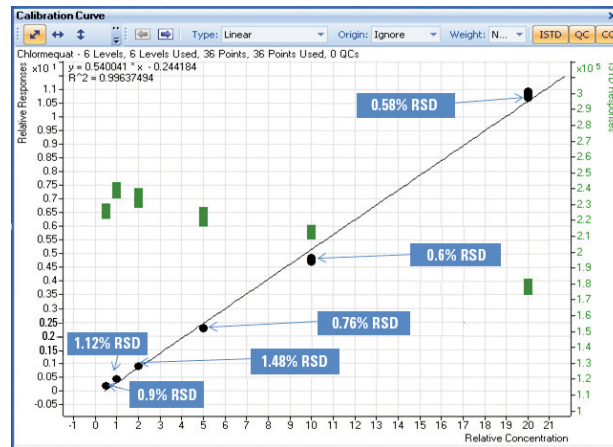


Figure 2. RSD values calculated for $n = 6$.

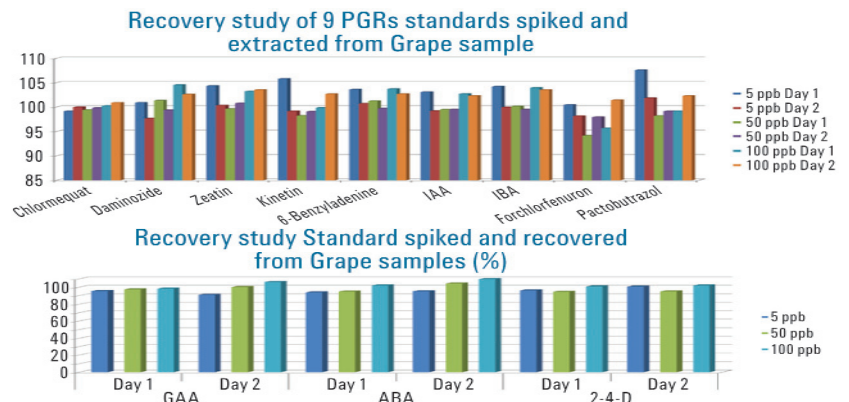


Figure 3. Recoveries for all PGRs within range of 90-100%.

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