

Pesticide Analysis in Tomatoes by AOAC 2007.1 QuEChERS Methodology

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Introduction

The global agriculture industry uses over a thousand different pesticides in the production of food and foodstuffs. Producers require pesticides to meet the increasing demand for reasonably priced food. This growing demand has increased the use of pesticides and expanded poor agricultural practices, elevating risks in the food supply and the environment. The QuEChERS method is the industry standard for pesticide extraction and cleanup for a wide variety of food samples. QuEChERS kits are easy to use and enable fast sample preparation, resulting in excellent pesticide recoveries.

Experimental

The QuEChERS AOAC 2007.01 method used in this work combines a salting out liquid-liquid extraction and dispersive solid phase extraction (dSPE).

Sample preparation

A 15 g sample of homogenized organic tomato was weighed into a 50 mL centrifuge tube (part number 5610-2049) with two ceramic homogenizers (part number 5982-9313). Specified samples were spiked with recovery standards (>200 pesticides). Fifteen milliliters of acidified acetonitrile (ACN + 0.1% acetic acid) was added, and the sample was vortexed to mix. The QuEChERS salt extraction packet for AOAC 2007.1 method (part number 5982-6755) was added, and the capped tubes were placed on a GenoGrinder vertical shaker for three minutes, then centrifuged at 5,000 rpm for five minutes. Six milliliters of the extract were transferred to the QuEChERS dispersive kit for general fruit and vegetables (part number 5982-5058). Next, the samples were vortexed for two minutes and centrifuged at 5,000 rpm for five minutes. Extracts were transferred to 2 mL analysis vials (part numbers 5183-2072 and 5182-0717) for analysis with the Agilent 7000D triple quadrupole GC/MS (GC/TQ). An outline of this sample preparation procedure is shown in Figure 1.

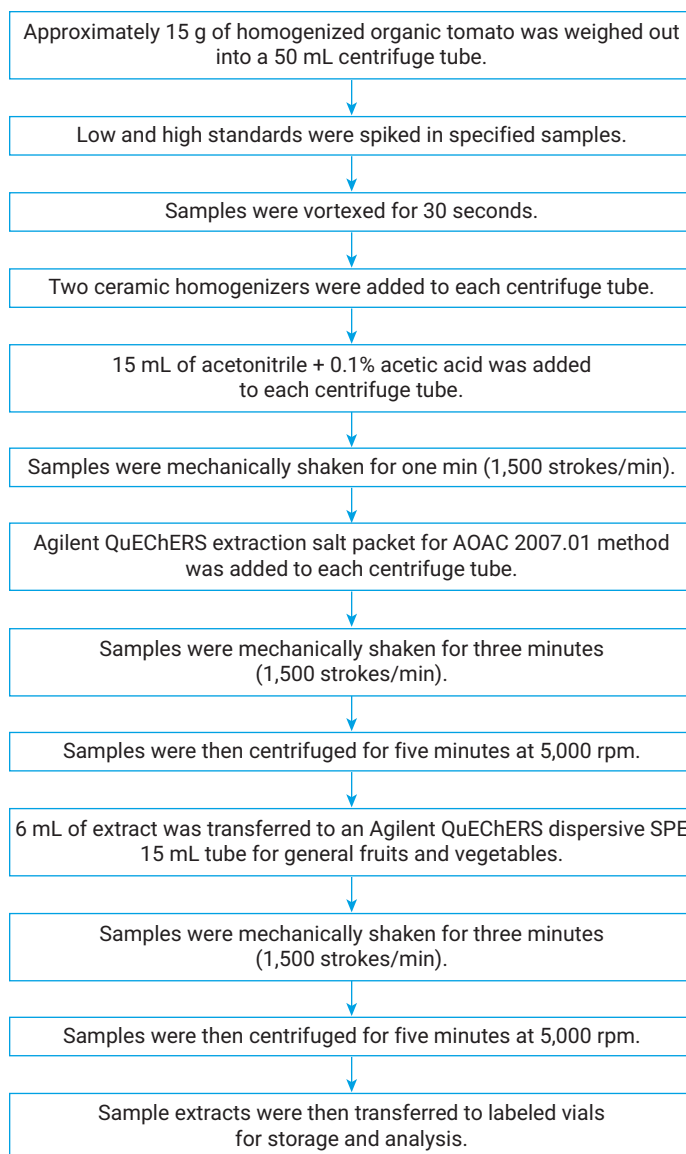


Figure 1. QuEChERS sample preparation method.

Analysis

Pesticide analysis was completed with a 7000D GC/TQ coupled with an Agilent 8890 GC (Figure 2). The acquisition method was retention time locked, and the GC/TQ was operated in dynamic MRM (dMRM) mode, monitoring 729 MRM transitions. The use of the Agilent Pesticide and Environmental Pollutant (P&EP) MRM 4.0 database allowed for seamless and automated development of the acquisition method.¹ Specific method parameters can be seen in Agilent application note 5994-0799EN.^{2,3}

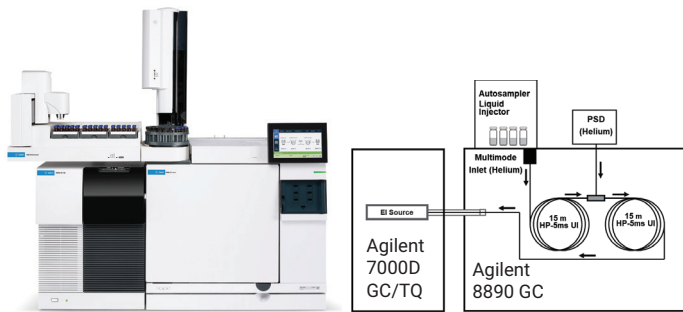


Figure 2. The Agilent 7000D triple quadrupole GC/MS (GC/TQ) coupled with an Agilent 8890 GC.

Results and discussion

Matrix-matched linearity with an $R^2 > 0.99$ was observed over a wide calibration range of 0.5 to 500 ppb (w/v) for all target pesticides (selected compounds shown in Figure 3). Pesticide recoveries were analyzed at both 10 and 100 ppb, and these results can be seen in Figure 4. By comparing absolute responses for pesticides in the prespiked samples to the postspiked blank matrix sample, it was determined that 94% of the tested compounds had recoveries between 80 and 120% at 10 ppb, and 95% of the compounds had recoveries between 80 and 120% at 100 ppb. Example 10 ppb MRM chromatograms are shown in Figure 5.

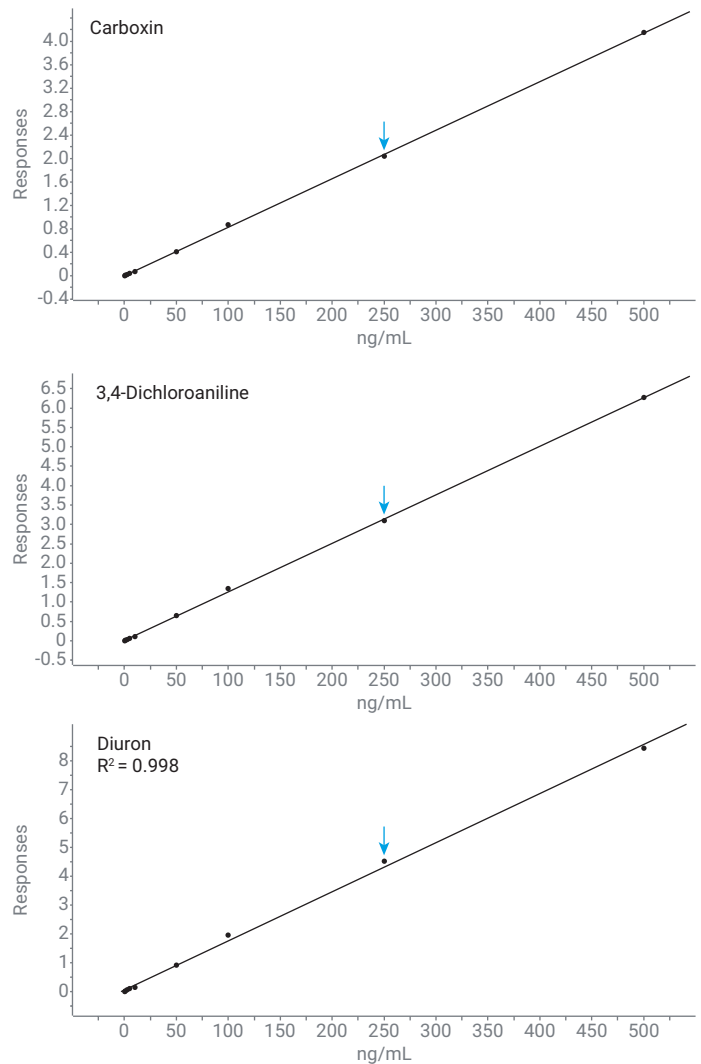


Figure 3. Matrix-matched calibration curves for carboxin, 3,4-dichloroaniline, and diuron on an Agilent 7000D GC/TQ in dMRM mode (0.5 to 500 ppb, w/v).

Conclusion

The QuEChERS method is the industry standard for pesticide cleanup and extraction of a wide variety of food samples. Agilent QuEChERS kits are easy to use and enable fast sample preparation, resulting in excellent pesticide recoveries. Extraction efficiency with high recoveries was achieved for over 200 pesticides in tomatoes with the Agilent 7000D GC/TQ.

References

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