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Determination of Pesticide Residues in Tomato using Dispersive Solid-Phase Extraction and Gas Chromatography/Ion Trap Mass Spectrometry

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Abstract

Agriculture is a crucial industry for providing food security to the world's growing population. However, the extensive use of pesticides can lead to environmental pollution and health risks. The present study aimed to evaluate the pesticide residues in tomato samples collected from local market retailers. The QuEChERS method was used for the extraction of pesticides, followed by dispersive solid-phase extraction using primary secondary amine sorbent and gas chromatography/mass spectrometry with an ion trap device. The results showed that no pesticide residues were detected in the samples. This study highlights the importance of monitoring pesticide residues in agricultural products to ensure food safety and health protection.

Keywords: QuEChERS, gas chromatography, pesticides, tomato

Introduction

Tomato (Lycopersicon esculentum Mill) is one of the most important vegetables in global agriculture and is consumed in natura, cooked, or processed. It is known for its high nutritional value and vitamin C content, which is essential for human health. However, the extensive use of pesticides in tomato cultivation can lead to environmental pollution and health risks. Therefore, monitoring pesticide residues in agricultural products is crucial to ensure food safety and health protection. The present study aimed to evaluate the pesticide residues in tomato samples collected from local market retailers using the QuEChERS method and gas chromatography/mass spectrometry with an ion trap device. The results showed that no pesticide residues were detected in the samples. This study highlights the importance of monitoring pesticide residues in agricultural products to ensure food safety and health protection.

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2003, the world’s average tomato consumption increased from 11 kg to 16 kg per capita per year, what means a 45.5% increase, according to data from Food and Agriculture Organization of the United Nations.4

The tomato production is favored by an adequate climate in several regions and during the whole year in Brazil. But, greater incidence of weeds, pests and diseases occur during the summer season, requiring large use of pesticides.

The correct use of pesticides provides the benefit of high food availability to the people. However, serious negative aspects might rise up from the indiscriminate use of such compounds in agriculture that might end up as the soil, water and crop product contamination.

Due to the pesticide toxicity character, several countries have established maximum residue limits (MRL) for the presence of pesticide residues in crop products. The MRL is established independently in each country as pesticide registrations and is determine through the result of toxicological and agronomic studies. These values may vary depending on the existing environmental conditions in the country, differing pest pressures, differing pesticide use patterns and good agricultural practices.5

The list of permitted compounds in Brazil and establishment of residue limits for different crops are controlled by the national health surveillance agency (Agência Nacional de Vigilância Sanitária - ANVISA). In 2001, ANVISA started the Program of Food Residue Analysis with the objective of evaluating the pesticide residue levels in crop products for a better food security.

In Brazil, 141 active compounds are registered and permitted for application in tomato crops, in individual or combined formulations, totaling 265 pesticide commercial brands.6 However, according to reported data, Brazil is still engaged, in some regions, with the problem of non-permitted compound use in tomatoes and the pesticide presence above the MRL.6 Nevertheless, the analysis of residues in samples considered above the MRL (using different validated methods for analysis of pesticide residue in tomatoes applied to real samples) showed relatively low residue values, meaning that, there is apparently an adequate use of pesticides in tomato crop in the studied regions, according to good agricultural practices.28-25 Besides, a relatively low number of samples above the MRL were found in studies with tomato samples, satisfactory results were found for a wide range of pesticides analyzed.18-20

Some studies were performed comparing the QuEChERS method with other extraction techniques,21 including the determination of 12 organophosphorus and carbamates insecticides in honey samples by liquid chromatography-ion trap-mass spectrometry.22 Four different approaches were studied for the extraction step: QuEChERS, solid-phase extraction (SPE), pressurized liquid extraction (PLE) and solid-phase microextraction (SPME). The comparison showed that all of them recover all the selected pesticides with a good repeatability. Nevertheless, QuEChERS method presented the highest recoveries (mean recovery 91.67%) followed by the SPE (mean recovery 90.25%) and the PLE (mean recovery 90.25%) whereas the SPME showed the lowest recovery (mean recovery of 49.75%). The QuEChERS method was the most adapted method with around 58% of recoveries higher than 90%. Due to sufficient analytical performance and low cost QuEChERS method presents an attractive approach for routine applications.

Recently microextraction methods have been employed for pesticide analysis which usually requires both smaller sample size and organic solvent volumes when compared with the conventional methods. The main advantages of these procedures are the high degree of enrichment for the analytes in complex matrices, which enable limits of detection23,24 down to the levels required by the regulatory bodies to the analysis of pesticide residues in water and food. On other hand, despite their high-throughput, requires long extraction times, which is perhaps the major disadvantage of the technique and its automation seems to be very difficult and has not yet been achieved, thus new developments in this area are required.25

Other extraction techniques meant for pesticide analyses also have been employed for environmental and food matrices, including the supercritical fluid extraction (SFE),26 solid-phase extraction (SPE),27,28 and matrix solid phase dispersion (MSPD),29-32 but these techniques are more costly than the QuEChERS and require more skilled technicians.

Therefore, the objective of this study was to use the QuEChERS sample extraction method for the quantitative determination of pesticide residues in tomato samples, considering the possible matrix effects. The pesticides analyzed were buprofezin and carbophuran, largely used in Brazilian tomato crops to control pests and diseases, and also, endosulfan-α, endosulfan-β, endosulfan sulfate and monocrotophos, which are non-permitted pesticides.
detected in samples analyzed by the monitoring program coordinated by ANVISA.

**Experimental**

**Standards and reagents**

Pesticide standards (buprofezin, carbofuran, monocrotophos, endosulfan-alpha, endosulfan-beta and endosulfan-sulphate) were purchased either from AccuStandard (New Haven, USA) or from Riedel-de-Haën (Seelze) with a minimum of 99% purity.

Stock solutions of individual standards (10 mg mL\(^{-1}\)) were prepared in toluene, considering standard purity, and stored in dark flasks at -20 °C. The calibration standard solutions were prepared in toluene containing the six pesticides in concentrations ranging from 0.250 to 4.00 μg mL\(^{-1}\). Standard solutions prepared in acetonitrile were used for spiking tomatoes samples at 0.0625, 0.250 and 1.00 mg kg\(^{-1}\) levels.

Toluene and acetonitrile of HPLC or spectra grade were obtained from Tedia, and formic acid from JT Baker. High purity and anhydrous MgSO\(_4\) was purchased from Sigma-Aldrich, and NaCl from Mallinckrodt. Primary secondary amine (PSA) sorbent (40 μm particle size) was obtained from Varian®.

**Samples**

Samples of tomatoes (1 kg) were monthly collected from different retailers in the City of Piracicaba, State of São Paulo, Brazil. Sampling was carried out during the period between August 2007 and October 2008. A total of 33 samples were analyzed in duplicate for the presence of six pesticides. The analytical method was validated using pesticide-free organically produced tomatoes.

**Analytical instrumentation and conditions**

The gas chromatographic analysis was performed on a Finnigan MAT GCQ gas chromatography-ion trap mass spectrometer (Thermo, USA). A fused silica capillary column, 5%-phenyl-methylpolysiloxane as stationary phase (30 m × 0.25 mm i.d.) and 0.25 μm film thickness (Quadrex, Woodbridge, U.S.A) was used with helium as carrier gas at a constant flow (1 mL min\(^{-1}\)).

The system was equipped with split-splitless injection inlet and 2 μL aliquot of sample or standard was injected in splitless mode at 250 °C. The GC oven was operated with the following temperature program: initial temperature 100 °C held for 3 min, ramped at 25 °C min\(^{-1}\) to 175 °C not held, followed by a ramp of 8 °C min\(^{-1}\) to 290 °C and held for 5 min. The total run time was 25 min and XCalibur1.2 chromatography data system software was used for instrument control and data analysis.

Transfer line temperature was set at 275 °C and the source temperature at 175 °C. The mass spectrometer was operated in selected ion monitoring mode (SIM) with an electron impact (EI) ionization with an ionizing energy of 70 eV.

Analysis was performed in the selected ion monitoring mode (SIM) according to the parameters shown in Table 1, based on the use of one target and two qualifier ions and according to the retention times. Target and qualifier ions were determined by injection of individual pesticide standards under the same chromatographic conditions in full-scan mode.

<table>
<thead>
<tr>
<th>Pesticides</th>
<th>Retention time / min</th>
<th>Ions monitored in SIM(^a)</th>
<th>M / (g mol(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monocrotophos</td>
<td>10.93</td>
<td>127, 67, 97</td>
<td>223</td>
</tr>
<tr>
<td>Carbofuran</td>
<td>11.62</td>
<td>164, 149, 122</td>
<td>221</td>
</tr>
<tr>
<td>Endosulfan-alpha</td>
<td>16.29</td>
<td>195, 241, 277</td>
<td>407</td>
</tr>
<tr>
<td>Buprofezin</td>
<td>16.78</td>
<td>175, 172, 105</td>
<td>305</td>
</tr>
<tr>
<td>Endosulfan-beta</td>
<td>17.95</td>
<td>195, 241, 339</td>
<td>407</td>
</tr>
<tr>
<td>Endosulfan-sulfate</td>
<td>18.85</td>
<td>272, 229, 387</td>
<td>423</td>
</tr>
</tbody>
</table>

\(^a\)Target ions are printed in bold.

Spiked blank samples were used as standards to counteract possible matrix effects.

**Sample preparation**

The tomato samples were rinsed in water and ground to complete liquefying and homogenized for 5 min using a domestic blender and stored in glass flasks and kept frozen at -20 °C until analysis.

The samples were prepared according to the QuEChERS method,\(^3\) as follows: 10 g aliquot sample was transferred into a teflon centrifuge tube, and 10 mL acetonitrile were added and the solution was homogenized using an Ultra-Turrax homogenizer at 12.000 rpm for 2 min; then, 4 g anhydrous MgSO\(_4\) and 1 g NaCl were added and the solution was shaken again for 1 min; the tube was centrifuged for 5 min at 5.000 rpm; 6 mL aliquot of the upper layer were transferred to a teflon centrifuge tube containing 150 mg PSA and 900 mg anhydrous MgSO\(_4\). The extract (pH 8) was vigorously shaken for 1 min and then centrifuged for 5 min at 5.000 rpm.

The upper layer was filtered through Teflon filter 0.45 μm and 2 mL of extract were transferred into a glass flask and 20 μL of 5% formic acid in acetonitrile...
solution were added (pH 5-5.5, where most acid and base labile pesticides are sufficiently stabilized); the extract was then evaporated to dryness (about 30 min). Care was taken to remove the tube immediately to prevent over-drying; following, 500 µL of toluene and 0.107 g of anhydrous MgSO4 were added to the dry extract, which was centrifuged for 5 min at 5,000 rpm and transferred to GC vial for GC analysis. The samples were analyzed as soon as prepared or stored in a freezer to avoid any adverse affect on the analytes evaluated.

Method validation

The parameters used to validate the method were linearity, matrix effect, precision and accuracy, sensitivity (limits of detection and quantification) and repeatability. All the analyses were carried out using the pesticide-free organically produced tomatoes.

Linearity was studied by constructing analytical curves using standard solutions in toluene and in the matrix extract for comparison purposes. The range was from 0.250 to 4.00 ng µL⁻¹ and three injections were made at each of the six concentration levels.

Precision and accuracy data were obtained with recovery tests carried out by spiking samples of organic tomatoes with pesticide standards at levels of 0.0625, 0.250 and 1.00 mg kg⁻¹. The spiked samples as well as the unspiked controls were analyzed in three replicates. The method repeatability was evaluated through the relative standard deviation (RSD%) associated to pesticide measurements performed during the recovery procedures.

The limits of detection (LOD) and quantification (LOQ) were calculated as 3.3 and 10 times respectively, the ratio between the standard deviation of the response (σ) and the slope of the calibration curve (S) and was estimated based on the specific calibration curve in the range of LOD. The estimate of σ expressed as the standard deviation of y-intercepts of regression lines was used as the standard deviation.

Results and Discussion

Validation study

The mean recoveries for spiked sample and RSD ranged from 71 to 111% and 8 to 15%, respectively (Table 2). In the lowest spike level (0.0625 mg kg⁻¹) six pesticides were recovered in the range of 77 to 107% as recommended by SANCO Guidelines which prove mean recovery values within the range 70-120%.

Figure 1 shows a chromatogram blank tomato (A), standard solution pesticides 1.00 ng µL⁻¹ in toluene (B) and spiked tomato 1.00 ng µL⁻¹ (C) whose peaks have their characteristic ions presented in Table 1.

All pesticides showed linearity in the concentration range of 0.250 to 4.00 mg L⁻¹, with determination coefficients r² higher than 0.990 (Table 2). Relative standard deviations (RSD%) of the three replicate injections ranged from 4.00 to 15.0% meaning good precision. LOQs of the method were in the range of 0.0127-0.0501 mg kg⁻¹, which are below the MRLs established for these compounds by ANVISA.

Matrix effect

A significant better linearity was observed for all pesticides (r² > 0.990) when analyzed in the matrix extract, evidencing a positive matrix influence on parameters of linear interval and r², making more stable the chromatographic system responses and providing better analytical sensibility and precision (Figure 2).

Figure 1. Chromatograms of (A) blank tomato sample, (B) standard solution in toluene (1.00 ng µL⁻¹) and (C) spiked tomato sample (1.00 ng µL⁻¹). Chromatographic conditions are described in the Experimental section. 1: Monocrotophos, 2: Carbofuran, 3: Endosulfan-alpha, 4: Buprofezin, 5: Endosulfan-beta and 6: Endosulfan-sulfate.
Table 2. Calibration data (equation, determination coefficient $r^2$), mean percent recovery ($R_*$), relative standard deviation (RSD), limit of quantification (LOQ) of pesticides in tomato samples and maximum residue limit (MRL)

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Calibration Curve Equations ($r^2$)</th>
<th>Spiked level / (mg kg$^{-1}$)</th>
<th>$R^*$</th>
<th>RSD / (%)</th>
<th>$R_*$ / (%)</th>
<th>RSD$_m$ / (%)</th>
<th>LOQ / (mg Kg$^{-1}$)</th>
<th>MRL / (mg Kg$^{-1}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Buprofezin</td>
<td>$y = 153443x - 33397$ (0.999)</td>
<td>0.0625</td>
<td>77</td>
<td>8</td>
<td>71</td>
<td>9</td>
<td>0.0501</td>
<td>0.500</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.250</td>
<td>72</td>
<td>8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.00</td>
<td>63</td>
<td>12</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Carbofuran</td>
<td>$y = 439148x - 34238$ (0.999)</td>
<td>0.0625</td>
<td>107</td>
<td>11</td>
<td>111</td>
<td>8</td>
<td>0.0264</td>
<td>0.100</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.250</td>
<td>101</td>
<td>6</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.00</td>
<td>125</td>
<td>8</td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Endosulfan alpha</td>
<td>$y = 202705x - 426$ (0.999)</td>
<td>0.0625</td>
<td>94</td>
<td>6</td>
<td>83</td>
<td>9</td>
<td>0.0127</td>
<td>0.500</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.250</td>
<td>80</td>
<td>8</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.00</td>
<td>75</td>
<td>13</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Endosulfan beta</td>
<td>$y = 211004x - 6259$ (0.999)</td>
<td>0.0625</td>
<td>83</td>
<td>9</td>
<td>75</td>
<td>11</td>
<td>0.0362</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.250</td>
<td>70</td>
<td>15</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.00</td>
<td>72</td>
<td>9</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Endosulfan sulfate</td>
<td>$y = 247295x - 234$ (0.999)</td>
<td>0.0625</td>
<td>99</td>
<td>7</td>
<td>79</td>
<td>11</td>
<td>0.0244</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.250</td>
<td>72</td>
<td>14</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.00</td>
<td>66</td>
<td>12</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Monocrotophos</td>
<td>$y = 142194x - 29623$ (0.998)</td>
<td>0.0625</td>
<td>103</td>
<td>17</td>
<td>80</td>
<td>15</td>
<td>0.0229</td>
<td>–</td>
</tr>
<tr>
<td></td>
<td></td>
<td>0.250</td>
<td>75</td>
<td>13</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>1.00</td>
<td>62</td>
<td>14</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

* $n = 3$; $R$ = Recovery; **Total endosulfan (sum of alpha, beta and sulfate). Source: “ANVISA”$^{36}$, “CODEX ALIMENTARIUS”$^{37}$; No MRLs established or prior MRLs revoked in Codex and/or ANVISA.

Figure 2. Comparison between the slopes of standard curves prepared in toluene and tomato matrix.
The relationship between the curve slopes obtained in solvent and matrix provide information about the matrix effect. Another way of evaluating the matrix effect is the use of F test and Student t test. In this study, a 20% increase or decrease on the slope was considered matrix effect (Table 3).

Table 3. Matrix effects estimated by the angular coefficient variation of the curves in solvent and matrix (ME%), and by the F and t tests (p < 0.05)

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>ME (%)</th>
<th>Statistics</th>
<th>Conclusion from the two evaluations</th>
</tr>
</thead>
<tbody>
<tr>
<td>Buprofezin</td>
<td>71</td>
<td>F test: F_{ab} &gt; F_{tb} t test: t_{ab} &gt; t_{tb}</td>
<td>Significant matrix effect</td>
</tr>
<tr>
<td>Carbofuran</td>
<td>74</td>
<td>F test: F_{ab} &gt; F_{tb} t test: t_{ab} &gt; t_{tb}</td>
<td>Significant matrix effect</td>
</tr>
<tr>
<td>Endosulfan-alpha</td>
<td>29</td>
<td>F test: F_{ab} &gt; F_{tb} t test: t_{ab} &gt; t_{tb}</td>
<td>Significant matrix effect</td>
</tr>
<tr>
<td>Endosulfan-beta</td>
<td>16</td>
<td>F test: F_{ab} &gt; F_{tb} t test: t_{ab} &gt; t_{tb}</td>
<td>Significant matrix effect</td>
</tr>
<tr>
<td>Endosulfan-sulfate</td>
<td>42</td>
<td>F test: F_{ab} &gt; F_{tb} t test: t_{ab} &gt; t_{tb}</td>
<td>Significant matrix effect</td>
</tr>
<tr>
<td>Monocrotophos</td>
<td>−5</td>
<td>F test: F_{ab} &lt; F_{tb} t test: t_{ab} &lt; t_{tb}</td>
<td>Non-significant matrix effect</td>
</tr>
</tbody>
</table>

According to the literature, several compounds might be extracted together with the pesticides from the matrix, introducing spectral interferences. The interpretation of a positive or negative matrix effect over the method selectivity and the magnitude of such effect will depend on the matrix/pesticide interaction. The matrix effect is compound-dependent, probable due to the different components co-extracted with pesticides from the matrix.

Nowadays, the best procedure to minimize this influence on the pesticide quantification is to correct the matrix effect by means of matrix-matched calibrations.

Conclusions

The results obtained in this study with the evaluation and monitoring of different pesticide classes such as the organochlorine pesticides (endosulfan-α and -β, endosulfan sulfate), organophosphorus pesticides (monocrotophos), thiadiazine (buprofezin) and carbamates (carbofuran) evidenced that the proposed method of sample preparation and pesticide residue analysis involves fast, easy and sensible procedures. Besides, it has the advantage of employing lower amount of organic solvents, reducing the contamination risks for the lab-technicians and environment.

Determination in tomato samples

The thirty three tomato samples collected from local market retailers, prepared and analyzed for the pesticide determinations as described above, presented no detectable residues of pesticides, evidencing that the results were within the actual regulation for tomatoes.

The physicochemical properties (Table 4) of pesticides, as well as the frequency of use, mode of application, biotic and abiotic characteristics of the environment and weather conditions can determine their destination in the environment. With the adsorption coefficient (K_{ow}) it is possible to predict the tendency of the pesticide to be adsorbed onto organic matter in soil (buprofezin). Chemicals with low K_{ow} values (monocrotophos, carbofuran) may be considered relatively hydrophilic; they tend to have high water solubilities, small soil/sediment adsorption coefficients, and small bioconcentration factors for aquatic life. The moderately high Henry’s law constant combined with low water solubility means that endosulfan have a strong tendency to partition from water to air meaning that may evaporate readily from formulations applied.

The results obtained in this study with the evaluation and monitoring of different pesticide classes such as the organochlorine pesticides (endosulfan-α and -β, endosulfan sulfate), organophosphorus pesticides (monocrotophos), thiadiazine (buprofezin) and carbamates (carbofuran) evidenced that the proposed method of sample preparation and pesticide residue analysis involves fast, easy and sensible procedures. Besides, it has the advantage of employing lower amount of organic solvents, reducing the contamination risks for the lab-technicians and environment.

Table 4. Physicochemical properties of the pesticides under study

<table>
<thead>
<tr>
<th>Pesticide</th>
<th>Solubility in water (20 °C) / (mg L⁻¹)</th>
<th>log pK_{ow} (pH 7, 20 °C)</th>
<th>K_{ow} / (mL g⁻¹)</th>
<th>Henry’s Constant (25 °C) / (Pa m² mol⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Buprofezin</td>
<td>0.46</td>
<td>4.93</td>
<td>2722</td>
<td>2.8 × 10⁻²</td>
</tr>
<tr>
<td>Carbofuran</td>
<td>322</td>
<td>1.8</td>
<td>23.3</td>
<td>5.0 × 10⁻⁵</td>
</tr>
<tr>
<td>Endosulfan-alpha</td>
<td>0.32</td>
<td>4.74</td>
<td>11500</td>
<td>1.48</td>
</tr>
<tr>
<td>Monocrotophos</td>
<td>818000</td>
<td>−0.22</td>
<td>19</td>
<td>n.d.</td>
</tr>
</tbody>
</table>

¹IUPAC; n.d.: not determined.
(r² > 0.990) for the pesticides buprofezin, carbofuran, endosulfan-α, endosulfan-β, endosulfan-sulfate and monocrotophos, when evaluated in tomato matrix.

Several and varied interactions may occur among sample-pesticide-chromatographic system, which turn difficult to define a behavior tendency for the matrix effect. Therefore, it is recommended the quantification based on analytical standards prepared in matrix blank extracts to compensate the matrix effects and get more accurate results.

All analyzed samples showed low levels of pesticide residues, below the limits of detection (LOD) for the compounds buprofezin, carbofuran, endosulfan-α, endosulfan-β, endosulfan-sulfate and monocrotophos, indicating that the tomato producers have followed the actual legislation and adopted good agricultural practices in the studied region.

This study evidenced that the QuEChERS method with dispersive solid phase extraction (dSPE) may be used in multiresidue routine analysis of tomatoes, with low LOD and LOQ values and good analytical precision.

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