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Researchers Develop a 36-Minute Test for 107 Pesticides in White Wine

It distinguished mirror-image chemical forms that routine wine tests miss, including a fipronil case masked by total-residue reporting

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Researchers have reported a faster way to measure pesticide residues in white wine while also distinguishing between mirror-image forms of some chemicals that standard tests usually lump together.

In a study discussed Monday, November 9 by LCGC International and published in Analytica Chimica Acta, the team said it used low-pressure gas chromatography coupled with tandem mass spectrometry, or LPGC-MS/MS, to screen white wine for 107 pesticides in a 36-minute cycle. Analytical data collection was completed in 27 minutes, and the method achieved acceptable separation for seven pairs of chiral pesticides, the researchers said.

The work addresses a long-standing problem in pesticide testing. Many pesticides are chiral, meaning they exist as two mirror-image forms, or enantiomers. Those forms can behave differently in living organisms and in the environment. One enantiomer may provide most of the intended pest control, while the other may be less useful or may carry different toxicological risks. Even so, residue rules generally focus on the total amount of a pesticide rather than the amount of each enantiomer.

That creates a gap in routine food monitoring, especially in a product such as wine, where residues can be present at very low concentrations in a chemically complex matrix that includes alcohol, sugars, pigments and many natural compounds. Earlier pesticide methods used for wine did not separate those mirror-image forms, even when the target compounds were chiral.

The researchers said the new approach was designed to overcome the usual trade-off in chiral gas chromatography between speed, resolution and sample capacity. Conventional chiral separations often rely on long, narrow columns that can provide good resolution but take longer to run and accept only limited sample amounts. The team instead combined a short, wide-bore chiral column with low-pressure outlet conditions. According to Mariosimone Zoccali, the corresponding author, that setup allowed higher gas velocities and faster separations while maintaining enough resolving power for targeted pesticide analysis.

Under the selected conditions, the method used a 5-meter wide-bore γ-cyclodextrin column and a temperature program of 5 °C per minute from 50 °C to 180 °C. Zoccali said temperature control had the biggest effect on enantiomeric resolution because the interactions that separate the mirror-image forms are weak and sensitive to heat. Steeper temperature ramps reduced performance, while a slower 2 °C per minute

program improved resolution but pushed the run time to about 75 minutes. The final method was a compromise between speed, separation and signal quality.

The study said the wide-bore column also helped with sample loading and routine robustness. The system allowed a 5 µL splitless injection of concentrated extract, which improved detectability for trace residues without overloading the column. The researchers said the setup remained usable over about 450 injections.

For sample preparation, the team used a modified QuEChERS procedure with ethyl acetate extraction. Ten grams of wine were extracted with 10 mL of ethyl acetate in the presence of magnesium sulfate, sodium chloride and citrate salts, then concentrated by about 100 times under a gentle nitrogen stream. An added clean-up step using dispersive solid-phase extraction was tested but ultimately omitted because it did not improve analytical performance and could introduce interfering compounds, the researchers said.

Detection was carried out with triple-quadrupole mass spectrometry in multiple reaction monitoring mode, with two monitored transitions for each pesticide. Because low-pressure gas chromatography produces faster elution and narrower peaks, the method used scheduled compound-specific acquisition at 14 Hz to collect enough data across the peaks while preserving sensitivity.

The researchers said the method generally met the performance expectations used in European routine pesticide monitoring. Reported limits of quantification were at or below the relevant maximum residue limits, ranging from 2.1 to 74.4 µg kg^{-1}. Intra-day precision was below 15% for the tested compounds, with an average coefficient of variation of about 6%. Accuracy ranged from 73 to 125% at 0.01 mg kg^{-1}, from 85 to 118% at 0.05 mg kg^{-1} and from 89 to 107% at 0.1 mg kg^{-1}. Recoveries were lower and more variable, averaging about 56% at 0.01 mg kg^{-1} and 42% at 0.1 mg kg^{-1}, though the associated precision remained within commonly applied criteria.

The study also pointed to a practical reason for separating enantiomers before regulators require it. Zoccali said one commercial wine sample showed a case in which one fipronil enantiomer exceeded the applicable maximum residue limit while the other remained below it. A single total-residue result, he said, could therefore give a less

informative picture of actual contamination and exposure.

The researchers did not present the method as an immediate replacement for current residue rules. Instead, they described it as a tool that could help build the evidence needed for a more detailed regulatory framework by generating occurrence data and supporting future risk assessment on individual enantiomers.

For the beverage industry, the method could have broader implications if it proves reliable outside the initial study. Faster screening of residues in wine may help testing laboratories handle more samples in less time, while giving wineries and food safety authorities a more detailed view of contamination profiles than conventional multiresidue methods provide. That could be especially relevant in routine surveillance programs, where adding chiral information has traditionally meant a significant time penalty.

The authors said the method appears more compatible with high-throughput monitoring than traditional chiral gas chromatography workflows, but they also stressed its limits. They said broader interlaboratory validation is still needed before the approach could be considered for full routine regulatory use. They also said additional testing across other wine types and food matrices would be required.

The paper was authored by Giorgia Rinaldi, Zoccali, Peter Q. Tranchida and Luigi Mondello. In the interview, Zoccali said the team’s goal was not only to shorten run times, but also to make enantioselective pesticide analysis more practical in a real-world monitoring setting where laboratories need both speed and selectivity.