# AOAC Developing Standards for Analysis of Ethylene Oxide Residues

n March 6, 2024, during the AOAC Midyear Meeting, the new Working Group on Ethylene Oxide Residues, co-chaired by Luis Georges Quintelas (SQUALI) and Lukas Vaclavik (Eurofins Scientific), provided a look at standards development activities for the analysis of ethylene oxide residues (ethylene oxide and 2-chloroethanol) in oil seeds and nuts; dried herbs and spices; food additives; dietary supplement products and ingredients; dried fruits and vegetables; and spice, flavor, and herbal extracts.

"Ethylene oxide is an extremely volatile and reactive molecule that immediately interacts with the sample matrix upon sample fortification," said the working group chairs. "It is difficult to fully demonstrate method performance for ethylene oxide. Certified reference materials or proficiency testing schemes are not available for direct analysis. The presence of ethylene oxide in real samples is very rare. A pragmatic approach is needed when considering method performance requirements for ethylene oxide."

Introduced through the AOAC Analytical Science Forum, ethylene oxide is an emerging topic that was identified as a priority, resulting in a new AOAC project supported by an advisory panel representing the food, dietary supplement, technology provider, and testing lab industries (*see* sidebar). The working group is developing AOAC *Standard Method Performance Requirements* (SMPRs<sup>®</sup>) based on objectives and priorities established by the advisory panel.

### Analytical Challenges and Testing Landscape

Ethylene oxide is a colorless, flammable, and highly reactive gaseous substance historically used in the food industry as a fumigant because of its capability to disinfect without the use



of heat treatment; high diffusivity and strong penetrating properties; control of bacteria, fungi, and viruses, and effectiveness in dry food commodities. Main commodities include spices, nuts, oil seeds, dried fruits and vegetables, and dried herbs and botanicals. However, use of ethylene oxide has been discontinued in most parts of the world due to its highly toxic nature. The International Agency for Research on Cancer (IARC) classifies it as a group I carcinogen (carcinogenic to humans).

Maximum residue levels (MRLs) vary for different commodities, and regulations differ among countries. Use of ethylene oxide as a fumigant has been banned in the European Union since 1991.

Some methods are available, such as direct, conversion, and chromatographic separation and detection, but are limited in scope. Analytical challenges include analyte volatility, matrix complexity, sensitivity, and selectivity, among others.

#### **Toward Consensus**

Prior to launching the new project, AOAC conducted an interest survey focused on target matrix categories and main challenges related to analysis.

In developing draft SMPRs, the working group reached consensus that methods are needed for determination of ethylene oxide and its marker residue 2-chloroethanol (analyzed and reported as a sum expressed as ethylene oxide) with limits of quantitation (LOQs) meeting EU and other global regulations in the following targeted matrices: oil seeds and nuts; dried herbs and spices; spice, flavor, and herbal extracts; food additives; dietary supplement products and ingredients; and dried fruits and vegetables. Methods are intended to be used as global reference methods for surveillance and monitoring by trained technicians.

As recommended by the working group, methods using chromatographic separation with mass spectrometric detection are preferred but, ultimately, it is open to the method developer(s).

#### **Next Steps**

The working group is continuing to finalize the draft SMPR. After working group consensus, the draft standard will be posted on the AOAC website for public comment. All comments will be reviewed and reconciled, if necessary, by working group chairs and AOAC.

It is anticipated that the draft SMPR will be posted for public comment in summer 2024 and reviewed for possible approval in the fall. Once approved, AOAC will publish the standard in the *Official Methods of Analysis<sup>SM</sup>* compendium and issue a call for methods.

Standards developed and adopted by the working group will drive support of regulatory compliance and provide the analytical basis for development of new testing methods.

## **Thank You**

AOAC thanks the following Ethylene Oxide Working Group contributors: Agilent Technologies Eurofins Europe Herbalife Medallion Labs/General Mills Nestlé NOW Foods Shimadzu Analytical (India) Pvt Ltd