

HOW BTEX ARE TESTED IN FOOD

Testing for benzene, toluene, ethylbenzene and xylenes (BTEX) calls for particular precautions: these substances tend to move from food into the surrounding air and may be lost before reaching the instrument. Reliability depends not only on laboratory sensitivity but on the entire sequence from sampling to interpretation.

WHY VOLATILE COMPOUNDS REQUIRE A SPECIFIC METHOD

BTEX are relatively small, volatile molecules. Opening packaging, mixing or heating a product may cause some of any compounds present to enter the gas phase. The rate depends on temperature, duration of exposure to air, product surface area and composition. Water, fats, proteins and other components can alter distribution between food and air.

A reliable analysis is therefore not simply a matter of 'putting a sample into a machine'. A method validated for the matrix and the individual analytes is required. A method suitable for water is not automatically equivalent when applied to oil, milk, flour or high-fat products.

SAMPLING: THE FIRST CRITICAL STAGE

Samples may change when repeatedly transferred, left in open containers or stored at inappropriate temperatures. In general, airtight containers, materials compatible with the analytes and procedures limiting losses are used. Minimising headspace in containers may be important for aqueous samples tested for volatile compounds; other matrices and methods may require different arrangements. There is no single rule for all foods.

External contamination must also be avoided: fuel vapours, solvents used during sampling, unsuitable environments, gloves and packaging materials may introduce substances not originally present. At very low target concentrations, sampling and laboratory blanks are especially important.

The laboratory needs information on sample origin, food type, packaging, collection date, transport conditions and storage times. Chain-of-custody documentation records these stages. Transport temperature and maximum holding time should be determined by the adopted method and its validation, not set arbitrarily.

THE HEADSPACE TECHNIQUE

Headspace analysis is widely used: a defined quantity of sample is placed in a sealed vial under controlled conditions. Some volatile compounds partition into the gas above

the sample, and that fraction is introduced into the analytical system. Secure closure and reproducible times and temperatures are essential.

Static headspace involves sampling part of the gas phase after an equilibration period. Alternatives include purge-and-trap, which uses a gas stream to carry volatiles to an adsorbent for concentration, and solid-phase microextraction (SPME), which collects them on a fibre. Selection depends on analytes, matrix and required performance; no technique is universally best for every food.

GAS CHROMATOGRAPHY AND MASS SPECTROMETRY

After extraction, substances are often separated by gas chromatography (GC). As they pass through a capillary column, different molecules emerge at different times. Mass spectrometry (MS) provides characteristic signals for identification and quantification. HS-GC-MS denotes the combination of headspace, gas chromatography and mass spectrometry.

A chromatographic peak alone does not prove benzene is present: the laboratory must apply suitable identification criteria, investigate interferences and calibrate appropriately. Isotopically labelled internal standards can compensate for some variability in preparation and instrumental response. For example, an FDA method for benzene in beverages uses deuterium-labelled benzene and HS-GC-MS.



THE FOOD MATRIX CAN CHANGE THE RESULT

Two products containing the same amount of a volatile compound do not necessarily release the same amount into the gas phase during testing. Salinity, lipid content, sugars and physical structure influence phase equilibrium. This 'matrix effect' must be addressed through appropriate calibration and recovery experiments.

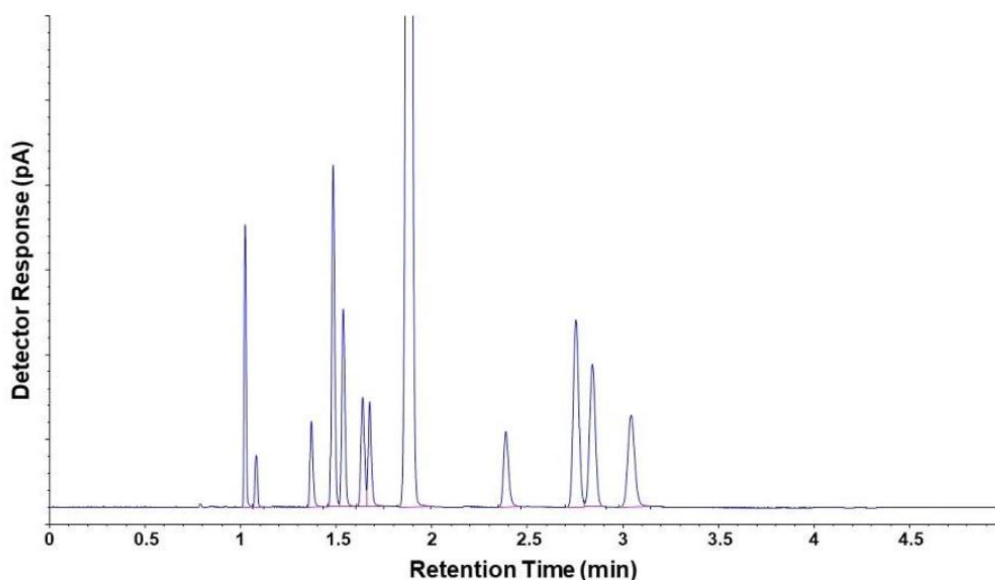
Laboratories assess selectivity, linearity, repeatability, recovery, precision and robustness. Accuracy for a clear beverage does not automatically demonstrate equivalent performance for cream or oil. Results must therefore be linked to the method's actual validated scope.

LOD AND LOQ: WHAT DOES 'NOT DETECTED' MEAN?

The limit of detection (LOD) is the level at which a signal can be distinguished from background variation according to method-defined criteria. The limit of quantification (LOQ) is the lowest level at which the laboratory can provide a quantitative measurement with suitable performance. They are not interchangeable and may vary by substance, matrix, preparation and instrument.

A report stating 'below the LOQ' does not mean the compound is absent: it cannot be quantified reliably below that level by the method used. 'Not detected' also requires caution and must be understood in relation to the stated detection limit. Without the LOQ, two 'not quantifiable' results cannot be meaningfully compared.

For instance, if two laboratories report 'benzene < LOQ' but one has an LOQ of 1 µg/kg and the other 10 µg/kg, the results convey different analytical information. These figures are illustrative only; they are not statutory thresholds for food.



QUALITY CONTROLS AND MEASUREMENT UNCERTAINTY

Each analytical batch should include controls appropriate to the purpose: blanks, calibration standards, fortified samples and quality-control materials. Blanks identify contamination from reagents, containers and the environment. Recovery trials show what proportion of a known amount added to the matrix is actually measured. Repeat measurements help estimate variability.

Reported concentrations must have correct units and, depending on purpose, an accompanying measurement uncertainty. Liquids may be expressed in µg/L and solids or semi-solids in µg/kg. Conversion requires consideration of density; merely changing the unit symbol is insufficient. Uncertainty does not mean a gross mistake, but the plausible spread of values attributable to the measurand.

PUBLISHED METHODS, STANDARDS AND VALIDATION

Authoritative technical references exist. ISO 20595:2018 describes static headspace GC-MS analysis of highly volatile organic compounds in water. It is relevant to the aqueous matrices within its scope, not a general validation for every food. US EPA Method 8260D describes GC-MS analysis of volatile organic compounds in several environmental matrices and includes different sample-introduction techniques; it does not replace food-specific validation.

For beverages, the FDA published a specific benzene method using HS-GC-MS and isotope dilution. The existence of a published method does not mean that every laboratory achieves the same performance: actual implementation, technical competence and, where relevant, test accreditation for the specified scope are decisive.

HOW TO READ A TEST REPORT CORRECTLY

An informative BTEX report should state at least: which substances were tested (and which xylene isomers or sum); the matrix; technique and method; units; applicable LOD and LOQ; measurement uncertainty where relevant; and whether the procedure falls within the laboratory's accredited scope.

Detection does not automatically demonstrate a health risk, just as failure to quantify does not certify total absence. Health interpretation requires data on actual concentrations, consumption and toxicology; regulatory compliance instead requires identification of a provision applicable to that specific substance and matrix. These are distinct from analytical quality alone.

CONCLUSION

Instrument sensitivity is only one determinant of a reliable BTEX result. Sampling, transport, preparation, prevention of losses, calibration, validation and transparent reporting together determine data quality. A good analytical result is not necessarily the lowest one, but one obtained by an appropriate method and reported in an understandable, verifiable way.

SCIENTIFIC AND TECHNICAL REFERENCES

1. U.S. FDA (2006). Determination of Benzene in Soft Drinks and Other Beverages. [Source](#)
2. ISO (2018). ISO 20595:2018 – Water quality: determination of selected highly volatile organic compounds in water by HS-GC-MS. [Source](#)
3. U.S. EPA (2017). SW-846 Method 8260D: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry. [Source](#)
4. Regulation (EU) 2017/625 on official controls in the agri-food chain (general context; it does not prescribe one BTEX method for all foods). [Source](#)