

[News story: CEN updates affecting chemical measurements April 2018](#)

[EN 17049:2018](#) – Animal feeding stuffs: Methods of sampling and analysis – Identification of tylosin, spiramycin, virginiamycin, carbadox and olaquinox at sub-additive levels in compound feed – Confirmatory analysis by LC-MS

Tylosin, spiramycin and virginiamycin are macrolide compounds that have been widely used in feed additives to prevent infectious disease and improve animal growth but which are now prohibited in the EU.

A screening method using thin-layer chromatography (TLC) to detect spiramycin, tylosin and virginiamycin in animal feed originating from plants is described in [EN 16939:2017](#) where for confirmatory purposes liquid chromatography coupled to a mass spectrometer (LC-MS) is required.

[EN 17049:2018](#) describes a method for confirmatory analysis using high performance liquid chromatography coupled to mass spectrometers in tandem (LC-MS/MS) identifying tylosin, spiramycin, virginiamycin, carbadox and olaquinox in animal feeds.

The method involves dissolving the feed sample in a methanol/water solution. An aliquot of the methanol/water extract solution is taken and added to a pre-conditioned solid phase extraction (SPE) column where the macrolide compounds are separated from other interfering compounds. The macrolide compounds are washed off the SPE column using methanol which is collected and evaporated with the residue containing macrolide compounds re-dissolved in dilute formic acid for analysis using LC-MS/MS.

Identification is based on the most abundant constituents because tylosin, spiramycin and virginiamycin are fermentation products consisting of several closely related compounds. The limits for identification are stated as 1 mg/kg for tylosin, spiramycin and virginiamycin, 4 mg/kg for carbadox and 3 mg/kg for olaquinox.

EN 17049 has been developed in accordance with European Commission Mandate [M/521](#) to prepare standards for the determination of food contaminants implementing the framework of [Regulation \(EC\) No 882/2004](#) on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules.

Further information on food legislation can be found on the Government Chemist website:

[Food and feed law: Compendium of UK food and feed legislation with associated context and changes during October to December 2017 – Government Chemist Programme Report](#)