

### 1 愛玩動物用飼料中の無機ヒ素の液体クロマトグラフィー誘導結合プラズマ質量分析計による定量法

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We have developed a quantitative determination method of the concentration of inorganic arsenic in pet foods using a liquid chromatograph-inductively coupled plasma-mass spectrometer (LC-ICP-MS). After adding 2 w/v% TMAH solution to a sample, inorganic arsenic was extracted by heating and the extract was collected by water. The pH of the solution was adjusted, and injected into a LC-ICP-MS to determine the concentration of inorganic arsenic. LC separation was carried out on an ODS column with 10 mmol/L sodium 1-butanefulfonate, 4 mmol/L malonic acid, 4 mmol/L TMAH and 0.05 % methanol solution as a mobile phase. A collaborative study was conducted by nine laboratories using dry and wet-type pet foods, formed jerky, dried jerky and biscuit. Dry-type pet food and dried jerky was added with 2 mg/kg of As (III). Wet-type pet food was added with 0.5 mg/kg of As (III). Formed jerky was added with 1 mg/kg of As (III). Biscuit was added with 0.2 mg/kg of As (III). The mean recoveries, repeatabilities and reproducibilities in the form of relative standard deviation ( $RSD_f$  and  $RSD_R$ ), and HorRat, were 95.4 % to 98.3 %, less than 2.9 %, less than 9.1 %, and 0.22 to 0.51, respectively.

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### 2 飼料中のゼアラレノン, ゼアララノン, ゼアラレノールおよびゼアララノールの分析法妥当性確認および日本における汚染実態

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The occurrence of zearalenone and the metabolites (zearalanone, zearalenols and zearalanols) in feeds distributed in Japan from fiscal years 2014 to 2019 was investigated using the official analytical method validated by the interlaboratory study. As a result of investigating on 121 samples of corn, 62 samples of soybean meal and 205 samples of formula feed, zearalenone was detected in over 90 % of the samples.

Zearalanols were not detected in any samples. Among the zearalenone metabolites,  $\beta$ -zearalenol was detected at the highest rate. Furthermore, it was suggested that the proportion of the zearalenone metabolites differed depending on the type of raw material.

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### 3 LC-MS/MSによる飼料中のクロルプロファム定量法の試験室間共同試験

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A collaborative study for validating the determination method of chlorpropham in feeds by LC-MS/MS was conducted in 13 laboratories using 2 kinds of formula feeds, oats, barley, wheat, and corn. The resulting trueness ranged from 75.3 to 87.0 %, repeatability and reproducibility in terms of relative standard deviation ( $RSD_f$  and  $RSD_R$ ) were within 7.3 % and 33 % respectively, and the HorRat values ranged from 0.39 to 1.5. The limit of detection and limit of quantitation of chlorpropham in feed were 0.008 mg/kg and 0.003 mg/kg, respectively. This method was thus validated as useful for inspections of chlorpropham in feed.

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