



# United Kingdom Accreditation Service

Commercial in Confidence

## DESKTOP ASSESSMENT

|                                      |  |   |  |
|--------------------------------------|--|---|--|
| <b>Organisation</b>                  | Alex Stewart Agriculture Limited, trading as | <b>Project Reference</b>                        | 225280-00-01                               |
| <b>UKAS Reference Number</b>         | 4352<br>6686                                 | <b>Assessment Standard / Criteria</b>           | ISO/IEC 17025:2005<br>- Testing Laboratory |
| <b>Technical Assessor</b>            | Derek Finnegan                               | <b>Date of Review</b>                           | 15/10/2017                                 |
| <b>Report Issued By</b>              | Jay Bhatta                                   | <b>No. of Findings Require Mandatory Action</b> | 0  |
| <b>Report Issued Date</b>            | 18/10/2017                                   | <b>No. of Findings with Recommended Action</b>  | 0  |
| <b>Method of Reporting</b>           | Email  | <b>No. of Close Out Required by UKAS</b>        | 0  |
| <b>Agreed Action Completion Date</b> | N/A  | <b>Close Out Time</b>                           | N/A  |

### Scope of Assessment - 2017 ETS Pet food Liverpool Assessment (W68309)

ETS to extend matrix scope from feed to include new matrix types in the moisture, fat, fibre, ash and fatty acid methyl ester accredited test methods.

## Documentation / Data Reviewed

Electronic AC4 application, validation packs, test method SOPs, key reagent quality and traceability, uncertainty of measurement estimates, evidence of satisfactory performance in proficiency test schemes, staff training records, for

- 07L.1.002 – moisture content
- 07L.1.011 – ash (crude)
- 07L.1.004 – oils and fats
- 07L.1.007 – crude fibre
- 07L.1.006 – fatty acid methyl esters

## Recommendation

The recommendation is that accreditation to ISO 17025:2005 for the application for extension to scope to cover the new pet food matrices for ash, fats/oil, crude fibre and moisture content and to extend the matrix scope to food, fats, oils and pet food for fatty acid methyl esters is granted. No findings or recommendations were raised. This recommendation is subject to independent approval within UKAS.

An example presentation for the schedule is given below.

| Animal feed, dry pet foods and wet pet foods            | <u>Chemical Tests</u>        | Documented in-house methods identified by method number |
|---|------------------------------|---|
|   | Ash (Crude)                  | 07L.1.011 based on ISO 5984:2002                        |
|   | Fibre (Crude)                | 07L.1.007 based on ISO 6865:2001                        |
|   | Moisture and Volatile Matter | 07L.1.002 based on ISO 6496:1999                        |
|   | Oils and Fats (Crude)        | 07L.1.004 based on Commission Regulation (EC) 152/2009  |
| Food, feed, fats, oils, dry pet foods and wet pet foods | Fatty Acid Methyl Esters     | 07L.1.006 based on ISO 12966-2:2011                     |

## Assessment Summary (including details of compliance/non-compliance)

The laboratory has accredited methods for the 5 determinands but limited generally to feed. The extensions for ash, crude fibre, fat and moisture contents (i.e. composition) are to capture new matrices (pet foods – both wet and dry) while the fatty acid methyl ester (FAME) method (used to calculate the content of saturated, monounsaturated and polyunsaturated) is to extend the matrix scope to cover food, oils, fats, pet foods (wet and dry). The calculations used to derive these are appropriate and in a controlled spread sheet. A desktop assessment was suitable as the laboratory is familiar with the methods and there is no change in the basis or detection principles.

Approximately 50 files were sent to support the validation of the new test/matrix combinations, with appropriate summary of validation for the compositional methods (combined summary) and a separate one for FAME methods.

The validation reports followed a standard procedure and covered detection limit, repeatability, reproducibility, linearity, and statistical analysis to derive internal quality control (IQC) limits for the range of new food/pet food matrices examined. The standard operating procedures describe the basis of the method, reagents and calibration materials, methodology and instrumentation conditions (e.g. GC conditions for FAMEs). Staff training records indicate that experienced analytical chemists undertook the validation and training files were updated. Test method basis is suitable for reporting composition in conformity to EU 152/2009. Validation work for composition methods covered a suitable range of dry and wet pet food while that for FAMEs captured all of the required food matrix extensions. In both cases there was a sufficient range (high and low content) of target analyte levels. Measurement uncertainty has been estimated and appears of appropriate magnitude. There has been successful participation in different (blind) accredited proficiency test (PT) schemes for all test methods. The laboratory has also made thorough use of PT material during validation. Equipment suitability is checked as part of the method and routine equipment calibrated and verified for on-going performance. The standard operating procedures have been updated and are controlled documents. As existing accredited methods, they are subject to local policies and procedures for internal audit and test reporting. Validation work has been reviewed and signed off as fit for purpose.

### **Test method**

Test method standard operating procedures (SOPs) are written in a clear flowing format and contain suitable detail. They are controlled documents and have been updated to capture the new matrices and some changes to gas chromatography and extraction conditions for FAMEs. Moisture and ash are gravimetric, fat and crude fibre 'wet chemical' and FAMEs is gas chromatography based. Overall, there is no change in the methodology. Test method SOPs present principle of the method, scope, reagents preparation and shelf life, extraction, equipment settings, IQC requirements (including acceptance criteria), calculations and reporting. Cross reference is made to equipment SOPs and other laboratory procedures where required. Overall, the test method appears suitably detailed to instruct a trained analyst to perform the test method in a consistent manner.

### **Method Validation**

A comprehensive validation pack (50 files) was sent to cover all of the ETS methods. Two separate reports summarised the performance and fitness for purpose of the 4 composition and the single FAMEs methods. Files covered both technical requirements and quality system integration. Reports describe linearity, limit of detection (LOD), repeatability, reproducibility, evaluation of bias, measurement uncertainty estimation, reagent quality and traceability and equipment and IQC acceptance criteria in appropriate detail. Validation was undertaken by experienced analytical chemists over several days. The test methods continue to meet the requirements of EU 152/2009 (for feed analysis). The 4 methods for composition analysis utilised a suitable range of pet food types covering dry and wet materials. Wet dog food (Bipea 05-1367) and dry cat food (Bipea 04-167) covered the extremes of the range (fat ca 2-20%, moisture ca 5-80%, ash ca 1-8% and fibre ca LOD-3%). Fat, fibre, moisture and ash contents were determined on these 5 times over various days by 2 analysts. The data was analysed statistically to set method performance criteria (e.g. LOD, reproducibility) to derive data that was used to set method performance acceptance criteria. Statistical analysis of the laboratory results with those of Bipea indicated no statistically significant bias.

The FAMEs method has been validated using a longer capillary column and a new gas chromatograph. The longer column has improved resolution of FAME peaks. Method validation for FAMEs was similar and covered a suitable range of matrices for the scope claimed (soya, infant cereal, meat, fish, oils, dairy spreads, yoghurt, seeds). Note that a large proportion of these foods used as validation samples were ex PT materials and all results were within scheme organisers acceptance limits. The ability to identify the target FAMEs used a standard reference materials.

Validation followed the same approach as for composition and limits of detection, quantification, repeatability, reproducibility were derived. Statistical analysis of the laboratory results with those of Bipea indicated good concordance and no statistically significant bias. Cross comparison of a standard oil sample with an accredited laboratory (Dr A Verwey BV) showed agreement of results (individual FAMES and saturated, monounsaturated and polyunsaturated).

The laboratory procedure for estimation of uncertainty of measurement has been applied and this captured reproducibility, bias (even though insignificant) and equipment. The magnitude of the values appear reasonable for these food tests. The validation report summarises test method performance and has been reviewed and signed off as fit for purpose.

### **Internal Quality Assurance**

System suitability checks (chromatography peak shape, retention time) are documented and acceptance criteria written into the FAMES method. Blanks and in-house reference materials are used with every batch of analysis and there are appropriate acceptance criteria for these. The laboratory has existing internal quality control procedures in place for the test methods and these are recorded in statistical control charts. The current control chart for polyunsaturates (FAMES) method shows a very slight positive bias but this has not been raised as a finding as when sufficient data are generated the limits can be re-set.

### **External Quality Assurance**

The laboratory has made good and thorough use of ex-proficiency test material (Bipea, LGC, FAPAS, AOCS) during validation. It continues to take part in Bipea (approximately one sample per month) and results since February 2017 have been within scheme organiser acceptance limits. Results from a recent blind LGC proficiency round for composition showed that fat, moisture, fibre and ash were all within 2 z-scores. The organisation will continue to participate in the Bipea scheme.

### **Uncertainty of measurement**

Data from analysis of the Bipea materials (reproducibility and bias) derived during validation were used to estimate for measurement uncertainty for all determinands. Bias appeared insignificant. The uncertainty of measurement estimates appear of reasonable magnitude.

### **Equipment and traceability of reagents**

Equipment settings and system suitability checks (e.g. FAMES) are documented as part of the SOPs. Certificates of analysis for reagents (e.g. FAME mix for retention time conformation) were sent and the purity and stability of reagents is noted in the SOPs.

A spread sheet used to calculate saturated, monounsaturated and polyunsaturated content from FAMES was sent and key calculation cells were seen to be locked.

### **Staff and technical competence**

Training records (training evidence form that appears to be used to record activities during training and the individual's training record with sign-off authorisation) were sent and showed that the 4 members of staff involved in the validation had been signed off. On-going performance will be captured as part of the local quality procedures.

### **Quality system integration**

As these are accredited methods, they are subject to internal audit and existing reporting procedures.

## **Conclusion**

Based on the data and other documents sent and assessed, there is satisfactory evidence that the extension to scope for new matrices in the 5 test methods has been validated appropriately and aligned with ISO 17025:2005. There is satisfactory evidence that the test method and equipment have been integrated to a suitable level of conformity to the Standard.