

## EMPIR HIT

# Inline sampling at VPG

14IND11 HIT.



#### **Danish Technological Institute**

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# 1. Introduction

Inline sampling conducted directly at a production line potentially has significant value for production companies and society. Optimising drying processes may reduce energy consumption and increase production quality. The best way to the data required for this purpose is to conduct inline sampling directly at the production line. The present investigation aims at enhancing such efforts by comparing inline sampling with more elaborate and traceable laboratory analysis.

In the current activity, inline sampling was conducted right at the output of the dryer at the production line. This setting is ideally suited for adjusting and optimising the dryer. Subsequently, additional experimental tests on selected samples were conducted at DTI to validate and further investigate inline sampling conduction directly at a production line.

The investigation was conducted as part of the EMPIR 14IND11 HIT project in collaboration between Source Technology<sup>1</sup> and Danish Technological Institute (DTI), with assistance of Vital Petfood Group<sup>2</sup>.

## 2. Inline sampling method

The sampling was conducted at the facility of Vital Petfood Group (VPG) in Ølgod, Denmark. VPG is one of the leading companies in the pet food industry in Scandinavia. It has been specialised in the private label concept for all pet categories since 1997.

The sampling site is shown in Figure 1. Source Technology has developed a device for inline sampling, which allows for determination of water activity and moisture content (as well as bulk density). A photo of this device taken at the same facility (VPG) during a previous test is shown in Figure 2.



Figure 1. The sampling site. The dryer is located behind the wall and samples are extracted through a small hatch.

<sup>1</sup> <u>https://sourcetechnology.dk/</u>

<sup>2</sup> http://www.vpg.dk/

A mock-up of this device was employed in the present investigation for the inline sample characterisation. That is, the water activity was measured using equivalent chamber and RH sensor (Rotronic), see Figure 3, and the moisture content was determined by a Kern DBS60-3 Moisture Analyser (Figure 4). The calibration of the RH sensor showed that it was accurate within about 0.5% RH (95% confidence). Samples were collected manually at the falling stream of product from the dryer. A small grinder was employed to pulverise the sample, to speed up the measuring process.



Figure 2. The inline sampling and test device developed by source technology, see text. The device automatically performs inline sampling and measures moisture content as well as water activity.

The mock-up inline sampling process was as follows.

- 1) A sample of about 500 g was extracted.
- 2) The temperature of the sample was determined.
- 3) The sample was divided into two parts.
  - a. The majority of this was stored in an air-tight container for further analysis at DTI. See Figure 5.
  - b. A small fraction was analysed immediately at the production facility
- 4) The inline sample material was pulverised in the grinder (Figure 3).



Figure 3. The chamber for measuring water activity and the Rotronic RH sensor (Source: Source Technology).



Figure 4. The KERN DBS 60-3 moisture analyser.

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Figure 5. Left: A sample of cat food from VPG in the DTI sample chamber (compare to the pulverised sample in Figure 3). Right: The same sample chamber mounted in the setup at DTI.

- A small fraction of 2 3 g was added to the moisture analyser, where the moisture content was measured utilizing the Loss-on-Drying technique (LoD) at 120 °C (Figure 4).
- b. The water activity was measured using a slightly larger fraction ( $\sim 120 \text{ cm}^3$ ).

The measurement of the moisture content could be performed straightforwardly and required about 7 minutes. On the other hand, the determination of the water activity required more consideration. The extracted samples could be quite hot (T > 70 °C was observed) whereas the sample chamber was about 20 °C. The resulting practical complications are



Figure 6. The determination of the water activity for the inline samples VPG-1 (left) and VPG-2 (right). The graphs show the relative humidity in % versus the time in sec. Black dots: measured data; Red curve: fitted exponential decay. Only the ranges indicated by yellow have been used for the curve fitting. The initial increase of the RH is due to the cooling of the hot samples. In the case of the right graph (VPG-2), the fitted curve follows the experimental data also outside of the fitting range, thus giving confidence in the accuracy of the curve fitting and consequently in the obtained water activity (= equilibrium RH). The contrary is the case for the sample at the left (VPG-1).

evident from the data in Figure 6 and discussed further below. However, it is essential to remark that the cooling of the sample material in the chamber and resulting condensation on chamber wall and sensor could cause the required measurement time to be very long (> 30 min) and/or the results to be inaccurate, see Figure 6.

A predictive algorithm base exponential fitting allowed for the determination of water activity in close-equilibrium conditions. This procedure could reduce the measurement time and increase the accuracy, see (Nielsen et al. 2015) and Figure 6.

## 3. Additional offline investigation at DTI

Two relatively large samples (about 400 g) were collected during the inline sampling and transported to DTI for further analysis. The sample chamber (Figure 5) was sealed from right after the sampling and until the analysis began at DTI.

Two techniques were employed at DIT.



Dew Point Generator

Figure 7. Sketch of the experimental setup at DTI. The sample chamber is located in an oven and weighted using a load cell. A chilled-mirror hygrometer determines the humidity of the air exiting the sample chamber.

Top: single-pass mode; Bottom: recycle mode.

In the single-pass mode, the humidity of the air input is controlled by the dewpoint generator while the moisture content is measured using the load cell. The moisture content can be determined during transient conditions (ad- or desorption of water).

The recycle mode is employed to measure the data points for the sorption isotherms. In this mode air is circulated through sample chamber and hygrometer until equilibrium between air and sample material is reached. At that point the water activity is established by  $a_w = RH_{equilibrium}/100\%$ , and the weight yields moisture content of the sample.

- To determine the dry mass and the initial moisture content, LoD analysis was performed at 105 °C using an accurate comparator and a calibrated mass of 5 kg (± 8 mg).
- Sorption isotherms were measured using equipment constructed for the HIT project. The method is explained in Figure 7.

Both methods above are traceable, i.e. the measured data can be traced back to the definition of the SI units.

Sample 1 ('VPG-1') was initially dried at 105 °C for 24 h and analysed by LoD. Subsequently, ad- and de-sorption isotherms were measured at 50 °C.

Sample 2 ('VPG-2') was initially analysed for water activity. Subsequently, ad- and desorption isotherms were measured at 50 °C, and finally a supplementary LoD analysis was carried out to determine the dry mass.

## 4. Results

Table 1 presents key parameters for the two analysed samples of cat food collected at the facility of VPG. In the case of VPG-2, relatively good agreement is observed between the water activity measurements at VPG and DTI<sup>3</sup>. It should be remarked that the inline analysis is for 20 °C, whereas the DTI value is for 30, 40, and 50 °C. The DTI data indicates that the water activity of the original sample decreased about 2.6% for each 10 °C lower temperature<sup>4</sup>.

Table 1. Key parameters for the samples measured in the inline test. 'wb' = wet basis, i.e.
water weight divided by original (= wet) sample weight. The water activity of sample may
have been influenced by the difficulties illustrated in Figure 6.

	Inline at VPG		Offline at DTI	
	a <sub>w</sub> (at 20 °C)	Moisture con- tent (wb)	a <sub>w</sub> (±0.007)	Moisture con- tent (wb, ±0.01%)
VPG-1	0.40 - 0.43*	6.17%		7.665%
VPG-2	0.402	6.38%	0.428 @ 30 °C 0.453 @ 40 °C 0.468 @ 50 °C	7.173%

The difference between sample temperature and chamber temperature was significant at the inline sampling: 50 - 70 °C for the sample vs. 20 °C for the chamber. The resulting effect was significant practical problems during the measurement of the water activity. This is because sample (and surrounding air) is cooled to the chamber temperature, and as result condensation will occur at the chamber walls initially during the cooling process. To avoid condensation the requirement is that the dew point of the air around the sample must be smaller than the chamber temperature. There are two possible solutions to this.

<sup>&</sup>lt;sup>3</sup> The initial water activity of VPG-1 sample was not measured at DTI.

<sup>&</sup>lt;sup>4</sup> The dependence is approximately linear due to the narrow temperature range.



Figure 8. Ad-sorption isotherm measured at 50 °C for the sample VPG-2 (cat food, type 2). The graph shows datapoints with uncertainties (k = 2) and model fit based on the Hailwood and Horrobin equation.

- The sample can be allowed to cool down before the chamber is closed and measurements starts. For 70% relative humidity (RH) and a chamber temperature of 20 °C this translates to a sample temperature of 25 °C or less.
- 2) Alternatively, the chamber must be heated. For 70% relative humidity (RH) and a sample temperature of 70 °C the chamber temperature of 62 °C or more is required.

The first of these procedures was employed in the present investigation. It has the advantage that it provides the water activity at room temperature. The second method would be much faster on the other hand. In addition, a temperature controlled sample chamber would allow for the measurement of water activity as a function of temperature.

Measurements of the water activity as a function of temperature has been conducted for the sample VPG-2 near  $a_w = 0.4$ , and showed change of  $\Delta a_w \approx 0.026$  for a temperature change of  $\Delta T = 10$  °C. This result illustrates that it is possible to correct the water activity for the influence of temperature changes, yet it should be remarked that such corrections to some extent will be sample specific.

For the moisture content, the data from the inline moisture analyser yields lower values than the offline DTI analysis for both samples. There are several possible reasons for this difference. First, the analysis was much faster at the inline sampling, 7 min. inline vs. 24 h. at DTI respectively. Second, the hot samples were grinded prior to the inline analysis: it is possible that this procedure resulted in water loss prior to the analysis. Finally, the result recorded by the moisture analyser may have been inaccurate, either because the small sample was contaminated or because the analyser required adjustment (it was not calibrated). The sample temperature cannot explain the difference, because the temperature set at the inline analysis was larger than the one used at DTI: 120 °C vs. 105 °C.

The main effort of the DTI analysis was devoted to measurements of sorption isotherms. Sorption isotherms for the two samples are shown Figure 8 and Figure 9. In the case of



Figure 9. Sorption isotherm measured at 50 °C for the sample VPG-1 (cat food, type 1). The graph shows datapoints with uncertainties (k = 2). Hysterisis is evident, see text: The data for adsorption are present in the lower curve, while desorption gives rise to the upper curve.

VPG-2 (Figure 8) curve fitting was conducted using the equation of Hailwood and Horrobin equation (Hailwood and Horrobin 1946); this equation is mathematical identical to the GAB equation (see e.g. Bell and Labuza 2000 for different equations). The equation takes the form

$$Moisture \ content = \frac{a_w}{C_1 + C_2 \cdot a_w - C_3 \cdot a_w^2}.$$

Here  $C_1, C_2$  and  $C_3$  are constants which control the shape of the sorption-isotherm curve.

The conducted curve fitting allowed to determine the effect for overdrying. For example, it may be assumed that the goal in the case of VPG-2 was a water activity of 0.60, yet the initial water activity was about 0.402. Using the value from the fit,

$$Moisture_{VGP-2} = \frac{a_w}{0.078 + 0.2120 \cdot a_w - 0.2282 \cdot a_w^2}$$

the moisture content can be determined to be 11.3% and 7.2%, respectively (dry basis). Consequently, the overdrying has resulted in an additional weight loss of more than 4% in the case of VPG-2.

Another aspect that must be considered is the fact that the measured sorption isotherm of the samples exhibits hysteresis. This can be observed in Figure 9 for VPG-1, with the upper part of the graph showing desorption (drying) and the lower adsorption (humidification). Hysteresis occurs when a sample for some reason do not (i.e. cannot) reach equilibrium (see discussion and references in Bell and Labuza 2000 p 22 - 25). It seems probable that the adsorption curve (lower part of graph) best represent the equilibrium state.

The observed hysteresis raises important practical questions: Which part of the curve will the sample follow at the production line? Actually, the inline samples appear to be following

the adsorption curve. In the case of the sample VPG-2, the water activity measured inline at 20 °C (if corrected by  $\Delta T = 30$  °C, see page 8) and the initial DTI data at 50 °C exhibit excellent agreement with the adsorption curve displayed in Figure 8<sup>5</sup>. The results for VPG-1 support the same conclusion, yet the evidence is less strong due to the issues (see Figure 6) with the initial water activity. It should be recalled that the physical conditions at the dryer at the production line are significantly different compared to those of the test chamber. Therefore, it is likely that hysteresis effect may be different in the two cases.

## 5. Conclusion and Outline

Inline sampling at the production line of VPG has been conducted in combination with more detailed subsequent laboratory investigations at DTI.

Good agreement was observed in the case of the water-activity measurements. The practical problems related to measuring the water activity of a hot sample has been discussed, and it was suggested either to cool the sample or heat the analysis chamber prior to the measurements. The accuracy was typically  $\leq 0.01$ .

The laboratory measurements of sorption isotherm allowed to quantify the weight loss related to overdrying of the samples. As an example, drying one of the samples to a water activity of 0.402 rather than 0.6 resulted in an additional weight loss of more than 4%. Furthermore, the influence of the temperature on the water activity could be measured, and in one case a change  $\Delta a_w \approx 0.026$  would occur for a temperature change of  $\Delta T = 10$  °C (near  $a_w = 0.45$ ). Such result make it possible to correct water activities measured at elevated temperatures.

Hysteresis was observed for the sorption isotherms during the laboratory analysis. Comparison with the inline measurements indicated that the inline samples at the production line follows the lower graph. Accurate knowledge of the sorption isotherm allows for the water activity to be determined from moisture analysis.

The inline measurements of the moisture content yielded smaller values than the subsequent laboratory analysis. Some possible explanations have been stated, however further investigations are required to firmly establish the cause.

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<sup>&</sup>lt;sup>5</sup> Not shown in this report: The mentioned water activities do not agree with the VPG-2 sorption isotherm measured for desorption at DTI.

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