in focus

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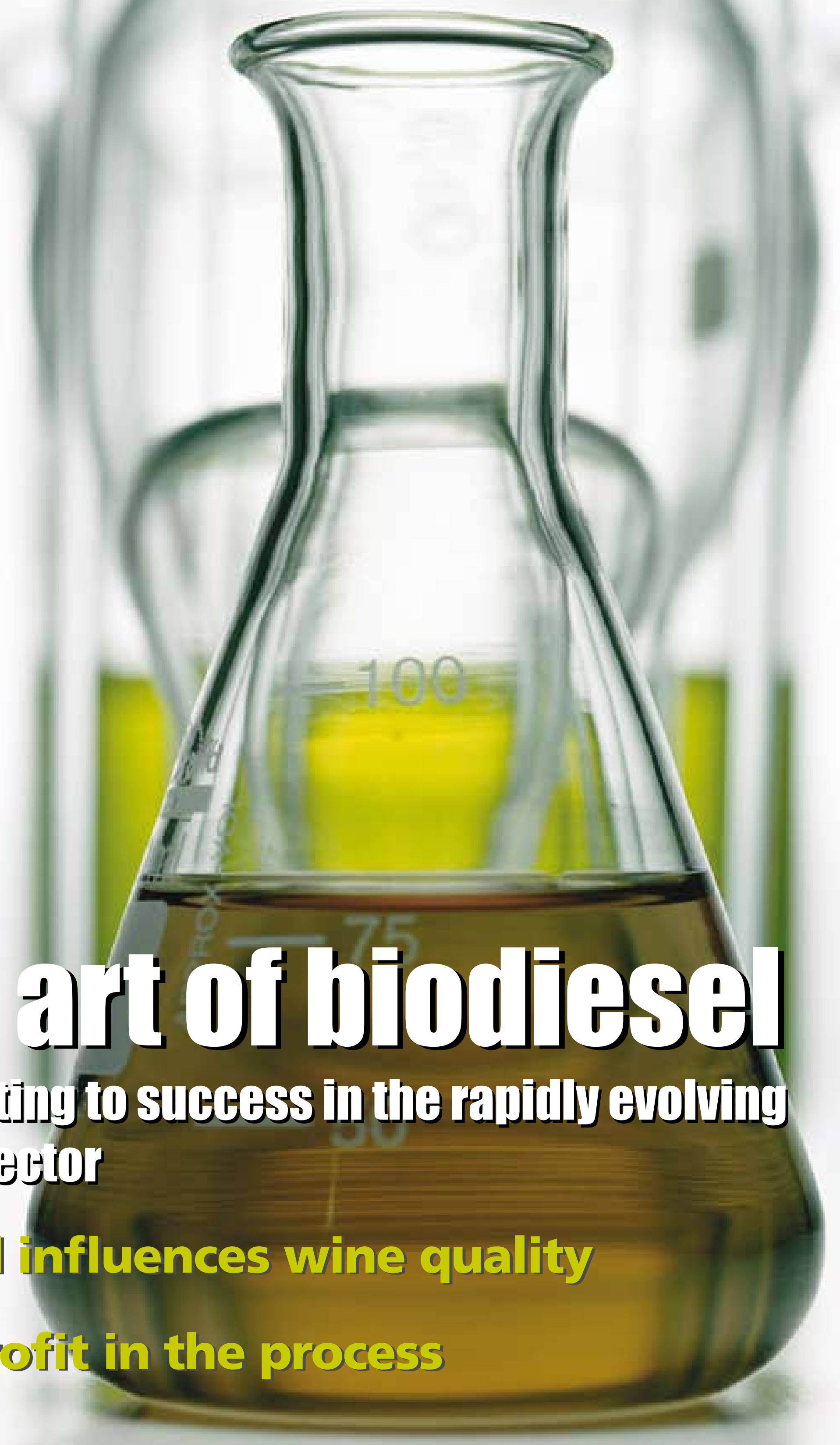


The art of biodiesel

Contributing to success in the rapidly evolving biofuel sector

Infrared influences wine quality

More profit in the process

Raw material costs highlight demand for FOSS solutions

As I write this, oil prices have just surged to record highs above $97 a barrel. In fact, prices have more than quadrupled since 2002 and oil is now 40% more expensive than at the beginning of the year.

As if that was not enough, there is little comfort to be found in The International Energy Agency's (IEA) 2006 World Energy Outlook (WEO) report, which warns of declining oil output and growing consumption. The report also projects that biofuels will play an increasing role in road transport, providing up to 7% of total consumption in 2030 although with the caveat that the growing demand for food will limit the potential.

What's more, the food or fuel dilemma is now being compounded by factors such as drought and poor harvests. Grain stocks are falling and grain carryover stock levels (the amount in the bin when the next harvest begins) are at their lowest for thirty years. In the EU, the wheat carryover stock is currently 32 days.

Before I start to sound like one of those end-of-the world prophets I should perhaps move on quickly to the more positive news and, I am pleased to say, how FOSS is playing an ever active part in handling change.

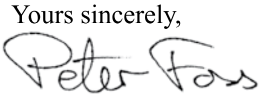
One story that comes to mind is from the Netherlands where a FOSS XDS analyser is facilitating the efficient production of biodiesel produced from a variety of sources, including waste cooking oil. Another is the example of how FOSS process analysis solutions are helping to ensure standardization of products and thereby effective use of raw material, not to mention the benefits of production economy, energy usage and consistent quality.

Attacking on another flank in the resource battle is grain analysis with instruments like the new model of Infratec 1241 – helping to maintain quality regardless of market or supply conditions. In the United States for instance, the well-known Infratec is helping to ensure the right levels of fatty acids in soy beans for more healthy food products. Furthermore, premiums for grain are sky-rocketing due to shortages and are making quality testing increasingly relevant. The premium for malting barley over feed barley is currently €110 per ton!

All of which convinces me that we are right on track with our solution strategy. As we approach another successful year of growth we will be maintaining our year-in, year-out investment in research and development with a view to the growing challenges in food and agriculture.

I am pleased that you will soon see the fruits of such investment in the form of new dedicated analytical solutions – innovations that will extend our product portfolio to exciting new applications in food and agriculture. So please keep an eye out for announcements and news from FOSS about new ways to improve efficiency in production and quality control. All the signs are that we are going to need them even more than we do today.

Yours sincerely,



Peter Foss,
President, FOSS A/S

In Focus

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Front picture:
Biodiesel can be made from a variety of vegetable oils, stock picture

Notes for contributors

Proposals for technical articles and papers are welcome and can be sent to RIM@foss.dk stating that the material is available for publication and whether it has been previously published or is being considered for publication elsewhere. An honorarium is paid for original material upon publication.

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Analytical procedures in the dairy are shifting from the laboratory towards at-line and continuous on-line process control, page 18.

Soxtec™ supports analysis of poisonous Ragwort in hay

The Netherlands: There is increasing alarm about a poisonous plant called Ragwort (Senecio jacobaea) which is finding its way into hay and killing livestock. A FOSS Soxtec™ instrument is supporting analysis requirements.

More than one per cent of Ragwort in hay is considered dangerous. Yet the distinctive yellow-flowered plant can often be seen growing along the roadside and what is particularly alarming for livestock owners is that it is also infiltrating fields used for natural hay production. Entire livestock herds have died – a situation that has prompted an Animal Health Service in Deventer in the Netherlands, to take action by analyzing samples of hay thought to contain the poisonous plant.

Mr. Harry Kolk, Analytical Chemist at the Health Service, said: "The problem started when the trend towards 'nature hay' appeared." Natural hay is grown without the use of fertilizer that would normally kill the Ragwort. When it is then dried and mixed up in the hay, Ragwort loses its bitter taste and yellow colour, but not its poison!

Suppliers now sell Ragwort-free hay but even so, it is a difficult claim to prove and contaminated hay can also become blended with other hay at the market – for example with hay produced from regular grass fields. The problem is particularly worrying for horse owners who often have to rely on bought-in feed of unknown origin and quality.

The Health Service has developed a method to analyse suspect hay based on Mass Spectrometer analysis with the FOSS Soxtec 2050 supporting the sample preparation process.

Finding the right sample

Finding the right sample in the first place is a challenge, however, because the Ragwort plants often grow randomly across a field. Out of 120 bales, perhaps only one will contain the poisonous plant. Kolk said that livestock owners should use common sense when feeding animals by looking closely at the hay to spot any suspicious-looking plants, but he also stressed that this can be difficult to the untrained eye. "Nowadays many cattle breeders lack this 'feeling'. In the past they knew exactly which plants were poisonous," he said.

Once a sample is found the Soxtec instrument is used to dissolve the alkaloids (species with a poisonous character) from the hay samples.

The Animal Health Service in Deventer, Netherlands (GD) provides knowledge about animals and their characteristics regarding animal health and welfare and the production of safe food. A website can be found at www.gddeventer.com.

■

by Marc Vendrig, FOSS, The Netherlands(mvg@foss.dk)

Approvals for new Infratec™ 1241

A new model of the well-know Infratec™ 1241 grain analyser was released earlier this year as reported in In Focus, no.1, 2007. Approval procedures are well underway and so far, approvals have been received from The Physikalisch-Technische Bundesanstalt (PTB) in Germany and The Laboratoire National de Métrologie et d'Essais (LNE) France.

Approval has also been received from the National Type Evaluation Program (NTEP) in the U.S. for the determination of test weight in all classes of wheat and barley as well as for soybeans, corn and milo with the Infratec test weight module. This makes the Infratec 1241 the only U.S.-approved grain analyzer for moisture, oil, protein, and test weight.

Updates on further approvals will be posted on the FOSS web site.

■

FoodScan™ receives AOAC approval

The FOSS FoodScan™ analyser has received AOAC approval for the analysis of moisture, fat, and protein in meat and meat products using FOSS artificial neural network (ANN) prediction models. The method number is 2007-04.

The approval allows food producers to exploit the leading meat analytical solution with full confidence in an officially approved method.

Established method

FoodScan is a popular analytical solution widely used in the food industry. It is supplied with comprehensive ANN calibrations that guarantee consistent analysis results across different instruments and locations. A common use is in routine production control. Here it helps to optimise the ratio between cheaper fatty, and more expensive lean meat, providing associated cost savings and improved product consistency.

The AOAC approval provides credibility for the extended use of FoodScan in support of quality control operations. FoodScan users can now refer to a documented and tested method, for example, when dealing with product labelling issues.

Food producers using FoodScan can also enjoy greater confidence in providing their customers with reliable information based on an approved method.

Collaborative study

The AOAC approval is based on a collaborate study involving 15 laboratories in the United States, all of which are existing FoodScan users. A total of 17 instruments were involved, two of the laboratories had two instruments and ran the sample set on both instruments. Ten different samples representing a wide range in composition were used, covering beef, pork, poultry, finishing products and in-process emulsions. They were sent to the collaborators as blind sample pairs making a total of 20 test samples.

About AOAC

The AOAC is a non-profit scientific association committed to worldwide confidence in analytical results and is a key authority for food production in the USA.

Full details of the collaborative study behind the AOAC approval are available in an article published in the Journal of AOAC entitled, *Determination of Fat, Moisture, and Protein in Meat and Meat Products by Using the FOSS FoodScan™ Near-Infrared Spectrophotometer with FOSS Artificial Neural Network Calibration Model and Associated Database: Collaborative Study*, Print ISSN: 1060-3271, Volume: 90 | Issue: 4, July 2007.

A free copy can be downloaded from the FOSS website using this link: www.foss.dk/aoac-approval

■

by Richard Mills, FOSS (rim@foss.dk)

Snow guarantee: winners from last year's FOSS 50 jubilee customer competition enjoy a dog sledging session as part of their prize trip to Greenland

The group, which included FOSS customers from around the world, also sighted musk ox and reindeer and fished for cod through holes in the ice.

■

FOSS showcase for South American business relations

The Danish prime minister Anders Fogh Rasmussens made an official visit to Argentina and Brasil earlier this year as part of a drive to consolidate existing business relations with Denmark.

FOSS president, Peter Foss was among the invited delegates. During a visit to a 'state-of-the-art' dairy production facility owned by Mastellone Hermanos S.A. he was pleased to explain how FOSS dedicated analytical solutions add value in quality and production control. Mastellone is Argentina's leading processor and manufacturer of fresh consumption dairy products.

FOSS has been active in South America for many years and on May 1, 2007 established a new sales subsidiary in Argentina and Chile.

■

The Danish prime minister Anders Fogh Rasmussen visiting Mastellone's Gral Rodríguez dairy processing plant

The value of quality control: Peter Foss explains the use of FOSS solutions in the dairy process

International Dairy Federation visits FOSS

FOSS was pleased to receive a visit from the International Dairy Federation (IDF) earlier this year in connection with the appointment of Steen Kold-Christensen from the FOSS Dairy group to the Danish National IDF Committee.

The visit provided the opportunity to reaffirm collaboration on projects and events.

Steen represents Denmark in the Standing Committee on "Quality Assurance, Statistics of Analytical Data & Sampling" (QASADS) and is also a member of the Joint Action Team (JAT) on Automated Methods of the Standing Committee QASADS as well as member of the Danish Working Group on Methods of Analysis.

■

How it all started: IDF committee member Steen Kold-Christensen (middle), conducts a tour of the FOSS museum for IDF director general, Christian Robert (right) and, IDF Technical Director Jörg Seifert (left)

Fast, clean and quiet

New Automatic Sample Analyser integrates Infratec™ 1241 with the help of FOSS Data Link

The process of preparing grain samples for analysis can create dust and noise. It is also time consuming, especially when a silo foreman is waiting to allocate loads according to quality.

The BITZER Automatic Sample Analyser improves the process. It automates the sample preparation and then determines all relevant qualities, including impurities, clean grain, small seeds, hectolitre weight, humidity, protein, sedimentation value, gluten and oil. A label is printed with the results.

The new system integrates the FOSS Infratec™ 1241 for the grain analysis part of the process, making use of the FOSS data link software to communicate bidirectionally between the sample analyser and the Infratec 1241. In one direction, the BITZER informs Infratec about the material to be analysed and in the other the Infratec returns the results.

The system can be integrated with data from the weighbridge using BITZER AGRAR weighing software. In which case, a complete delivery receipt can be produced in one reception operation.

How it works

The BITZER Automatic Sample Analyser itself looks like a drinks vending machine, but delivers a purified grain sample instead of a cup of coffee. The operator first scans an identity label and pours the sample in at the top of the analyser. The grain then goes through the sample cleaner, which also includes automatic weighing systems. The purified sample is automatically put into the FOSS Infratec 1241. The results from the Infratec are transmitted to the sample analyser and displayed on an integrated touch-screen. The sample cleaner and Infratec 1241 can operate simultaneously. Finally, the Automatic Sample Analyser prints a label. The sample is retained for reference.

The simple handling enabled by the new system allows analysis to be performed by non-laboratory personnel and leads to a faster, cleaner and quieter grain analysis procedure compared with conventional methods.

by Mikael Persson, FOSS (mpn@foss.dk)

FOSS DataLink™

- Provides a data link between Infratec™ analysers and Windows® software applications
- Simplifies integration of analysers to SCADA (Supervisory Control And Data Acquisition) and other applications
- Remote control of analyser and data collection through the link
- Easy to install
- Software developers kit included

The BITZER Automatic Sample Analyser

- Dust protection and sound insulation through enclosed construction
- Enclosed process helps to avoid operator errors
- Time saving and improvement of efficiency in the laboratory
- Simultaneous operation of sample cleaner and grain analyser is possible
- Print-out of the retained sample's label
- Quality and weighing data are merged in one database (optional)
- Generation of complete delivery notes (optional)

The right mix New ice cream test promises improved operations

When you are producing around 100 million gallons of ice cream a year, it is worth investigating any new ways to improve the production process. That's why Wells' Dairy, Inc., USA were keen to test the InfraXact™ analyzer and its ready-made ice cream calibration as an alternative to existing routine analysis methods. The results of their investigation promise easier and faster analysis of ice cream mixes with a number of potential spin-off advantages.

Wells' Dairy, Inc. is the world's largest family-owned dairy processor and a market leader in the United States. The company is based in the Midwest town of Le Mars, Iowa – the self-proclaimed Ice Cream Capital of the World®.

Wells' Dairy is based in the midwest town of Le Mars, Iowa – the self-proclaimed Ice Cream Capital of the World® and home to the "BLUE BUNNY®" brand ice cream.

Ice Cream Capital of the World® is a licensed trademark of the LeMars Area Chamber of Commerce Corporation.

Making ice cream on a grand scale demands forward thinking and the company is regarded as one of the most technically advanced in the industry. As part of this approach, Wells' Dairy, Inc. runs a research centre where new technologies are monitored and investigated for potential use in producing the many varieties of the famous "BLUE BUNNY®" brand ice cream.

Working at the centre is Mayur Acharya Ph.D., a Research Scientist who has recently conducted a study to evaluate an InfraXact™ analyzer for the routine analysis of ice cream mix using near infrared (NIR) against existing methods based on Microwave (MW)/Nuclear magnetic resonance (NMR) spectroscopy.

"I wanted to look at NIR as a routine method alternative to the existing solution currently in use in production," says Mayur. "FOSS seemed very keen to help and I was particularly interested in the availability of a ready-to-use calibration for the InfraXact based on a huge database for ice cream mix."

Comparative study

The study used samples of ice cream mix taken just before freezing. The samples were split into three portions, one for the existing analysis method, one for InfraXact and the third for wet chemistry analysis at a reputable reference laboratory.

The InfraXact calibration is based on a database consisting of samples from both North America and Europe. Samples include both butterfat and vegetable oil based mixes and many different flavours. The fat content of samples varies from 4-16 per cent.

A total of thirty samples from a diverse product range were measured as an independent validation set. They were tested for fat and total solids content.

"Our primary goal was to find out how close the numbers from InfraXact would be compared to the existing method and wet chemistry," says Mayur. Other factors such as the use of consumables, ease of use and speed were also examined.

Improved accuracy and repeatability

According to the results from the study, the InfraXact compares well to the existing analysis method for both fat and total solids parameters. "The general conclusion was that the accuracy of the FOSS InfraXact is at least as good, if not better than the existing method in terms of how close it is to wet chemistry numbers," says Mayur. "The repeatability was also better."

Significant savings in sight

The potential improvements in accuracy promised by the study are of great interest due to the high production volume at Wells' Dairy, Inc.

Mayur explains how the existing method showed an underestimation of around 0.3%, whereas InfraXact was right on target. "If you are targeting 10% fat in a mix, but you are consistently using 10.3% then that's a loss," says Mayur. He is keen to stress that the concept is yet to be tested in real life, but the potential is considerable.

Collaboration and the calibration issue

A common concern about NIR analytical instruments is the need for the data-gathering involved in making a reliable calibration. In the past, this may have proved a barrier to use of NIR, but in recent years, collaboration between the ice cream industry and FOSS has greatly improved the availability of calibration data to everyone's benefit – the new ice cream calibration for InfraXact being a case in point. The use of sophisticated calibration techniques allows a single calibration to cover a large number of recipies, minimizing calibration costs for instrument users.

With calibration concerns eased, the versatility of the InfraXact compared to other methods is a particular advantage that Mayur has firmly in mind. "With MW/NMR equipment you can only do fat and total solids, but with InfraXact you can do practically anything you can calibrate it for," he says. "We may need to do sugars or protein and obviously there is calibration work involved, but once it's done, it's done and the calibration is transferable – avoiding a lot of work in setting up individual instruments."

The internet networking capability provided through the FOSS RINA™ software can also prove highly relevant by allowing the management and monitoring of multiple instruments from a central location, regardless of distance.

Other benefits

Unlike the NMR method, InfraXact does not use any consumables, saving around 60 cents per test. But more importantly, it is very simple to operate which helps to avoid any manual procedures that can lead to inconsistent test results.

The InfraXact is also much faster. "The NMR method takes six to seven minutes to do a test whereas with FOSS it is just 35 seconds," says Mayur. Time savings can be made in production, especially where plants are running hundreds or even thousands of samples a month. Not that an operator is likely to stand around idly while waiting for results, but a lot of 'in-between results' time can be effectively avoided.

The next planned step at Wells' Dairy is to run another study with InfraXact units running live at production sites.

More about the company can be found on www.wellsdairy.com

■

by Richard Mills, FOSS (rim@foss.dk)

InfraXact™ for analysis of ice cream mix

- Takes less than one minute compared to six to seven minutes with existing methods
- No consumable costs are involved
- According to the Wells' Dairy study, accuracy is consistently closer to wet chemistry figures, allowing tighter production control and savings in raw materials
- Fat, total solids and other parameters can be measured with the highest confidence
- Calibration transferability ensures consistency of results across multiple units and sites
- One calibration covers a huge range of ice cream mixes
- Simplicity of use helps to avoid potential operator error

Ice cream mix

Ice cream mix is made according to set formulas defined by consumer demands and legal standards from organisations such as the FDA. The formulas cover various ingredients such as raw milk cream, concentrates, sweeteners stabilizers and so on. These are homogenised and pasturised to make the ice cream mix.

The study at Wells' Dairy involved both raw and unpasteurised mix. The parameters tested were fat and total solids. InfraXact™ can test many other parameters subject to available calibrations. For instance, it is ideal for the analysis of milk powder, delivering results for fat, moisture, protein, ash, acidity and lactose within a minute.

InfraXact is a relatively new analytical solution combining proven technology with ease of use to make routine NIR analysis simple for virtually anyone to perform.

FIAstar™ in Germany:

Determination of sulphur dioxide

Many commercially available red and white wines have too low a content of free sulphur dioxide (SO_2), leading to insufficient protection against oxidation. On the other hand, too high a content of free SO_2 is not desirable either. What's needed is a fast and precise method for the determination of free SO_2 to give optimum protection of wine against oxidative effects.

Different methods for the determination of free and total SO_2 have therefore been evaluated here at the Institute for Wine Analysis and Beverage Research. Emphasis has been on comparison of different methods with the EU reference method. This is a distillation method that is too time-consuming for routine analysis, which means that in Germany fast alternative methods are used instead. This article takes a look at a new system for determination of SO_2 using Flow Injection Analysis (FIA).

The FIAstar™ 5000 from FOSS is a well-proven system with components that are also used in the analysis of water, soil and foodstuffs. The system comprises two special modules for the determination of SO_2 *[Möller, 2005]*, allowing simultaneous analysis of free and total sulphur dioxide in about 60 wine samples per hour. The method was developed at ETS Laboratories *[G.Burns, I.Herve 2004]* in cooperation with FOSS *[S.Anderson 2004]*.

FIA method for determining free SO_2

Please see Figure 1. The samples are injected into an aquatic carrier (C) and merged with a diluted mineral acid (R1). The liberated SO_2 diffuses over a teflon membrane in a gas diffusion cell into a recipient stream (R2), which is reacted with the DNTB reagent (R3) to form a yellow dye that is measured in the detector (D).

Gas diffusion in FIA is a very elegant technique for separation of interferants, allowing sensitive and reliable determnation of small amounts of free SO_2.

FIA method for determining total SO_2

Please see Figure 2. The samples are injected directly into a phosphate buffer (C, R1) and merged with the DTNB reagent (R2). After reaction at 50°C, the resulting yellow dye is diluted and separated from interferants by dialysis. An acceptor stream (R3) carries the product to the flow cell of the detector.

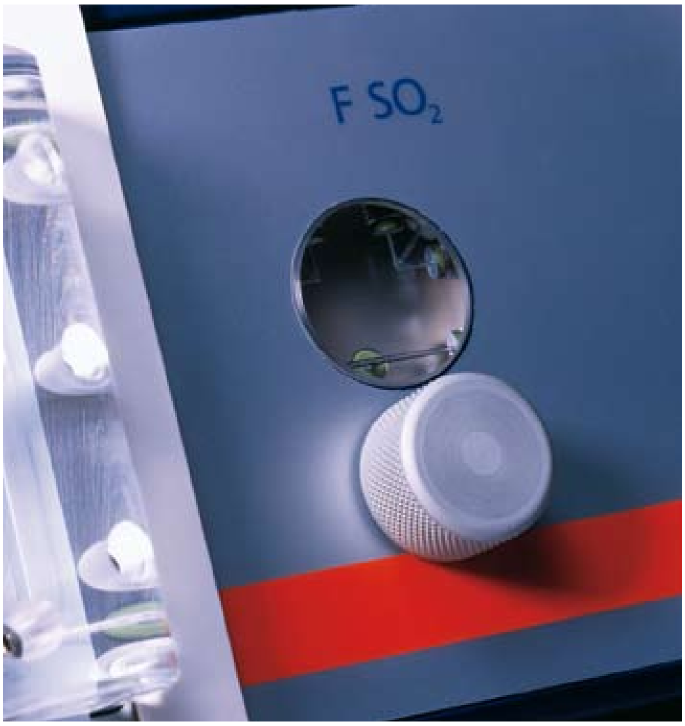
Calibration is carried out using ethanolic solutions of sodium disulphite in the range of 1-50 mg/l for free SO_2 and 5-250 mg/l for total SO_2. In this concentration range a linear calibration function can be obtained. Samples with higher concentrations can be diluted. The ethanol in the FOSS Application Note can be replaced with methanol. The use of denatured ethanol is to be avoided.

Reference and other methods

For reference and comparison purposes, results for free SO_2 have been compared with the method described in EU Directive EEC 2676/90, and for total SO_2 with the distillation method according to IFU 7a. IFU 7a is a modified version of the EEC 2676/90 method, using stronger acid, which leads to slightly higher values for total SO_2. In both methods phosphoric acid is used to expel the SO_2 into a receiver solution containing peroxide, for oxidation to sulphate. Subsequent titration with sodium hydroxide allows the calculation of SO_2 content.

Numerous alternative methods have also been compared with the reference methods. These have included direct colorization with DTNB *[Berger, 2002]*, enzymatic and different titrimetric methods, direct iodometric titration using starch for free and total SO_2, and titration using a double platinum electrode. Reducing substances that can interfere when using the titration methods have been determined separately and taken into account. The double platinum electrode is frequently used for titra-

using Flow Injection Analysis



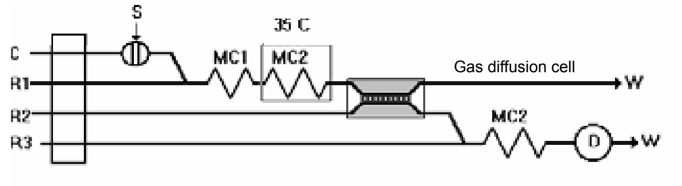


Fig 1: Flow scheme for FIA determination of free SO_2. C: Carrier (distilled water), R1: 1N HCl, R2: Phosphate buffer, R3: DNTB; MC: Mixing coil, D: Detector, W: Waste, S: Sample [Möller, 2005]

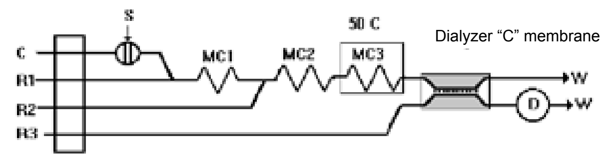


Fig 2: Flow scheme for FIA determination of total SO_2. C: Carrier (phosphate buffer), MC: Mixing coil, D: Detector, W: Waste, R1: Phosphate buffer, R2: DNTB, R3: Distilled water, S: Sample [Möller, 2005]

tion of red wines, where a visual endpoint is difficult to detect.

Figure 3 gives an overview of the different analytical techniques used in determining SO_2. Within each group, numerous variations of analytical conditions (temperature, acid, gas, oxidation agent) are possible. Most variegated is determination of total SO_2 where different direct methods as well as chromatographic methods may be applied.

For determining free SO_2, iodometric titration is the routine method most frequently used, although the influences of interfering substances like ascorbic acid and phenols may be substantial. Even after correction for separately determined reducing substances, a significant difference compared to the reference method may remain.

To avoid artefacts from other volatile substances when using the reference method, the interference effect of different substances, including acetic acid (up to 20 g/l), acetaldehyde and sugar, was investigated. No interference reaction in the receiver solution could be found for any of these substances. Due to the nitrogen carrier (gas), only easily released gases like CO_2 and SO_2 are transferred to the receiver solution, and not acetic acid and other substances that might be released by hot water steam distillation. More selective determination of the sulphate in the receiver solution, for example by gravimetry, nephelometry or ion chromatography, is also possible.

Generally, the reference method does not define 'true' values for sulphur dioxide but is a consensus method that is capable of producing comparable values if instructions are strictly followed. Legal limits for SO_2 are based on values obtained using the reference method and not on 'true' content. Investigations using a hyphenated HPLC – Biosensor system have shown that the actual content of total SO_2 can be as much as 33 per cent higher than that determined by the reference method *[Patz and Galensa et al., 1997]*. In any case, in terms of legal limits, values obtained using the EEC 2676/90 method are binding.

FIA v EEC 2676/90

In the following, results obtained using the reference method are compared with those obtained using the FIA method. Different international red and white wines available in German retail outlets were analyzed.

Free sulphur dioxide

Figures 4 and 5 show the results for determination of free SO_2 in red and white wines using the FOSS FIAstar 5000 and the EEC 2676/90 method. The white wines covered a range of 0-37 mg/l free SO_2 and the red wines 5-41 mg/l. All samples were measured directly and without any dilution. The FIA method proved to be very suitable for these determinations.

The intercept of the regression lines for both methods is a little higher for red wines (+ 2,6 mg/l) than for white wines (+ 0,7 mg/l). This means that, on average, the FIA method produces slightly lower values for red wines than the EEC

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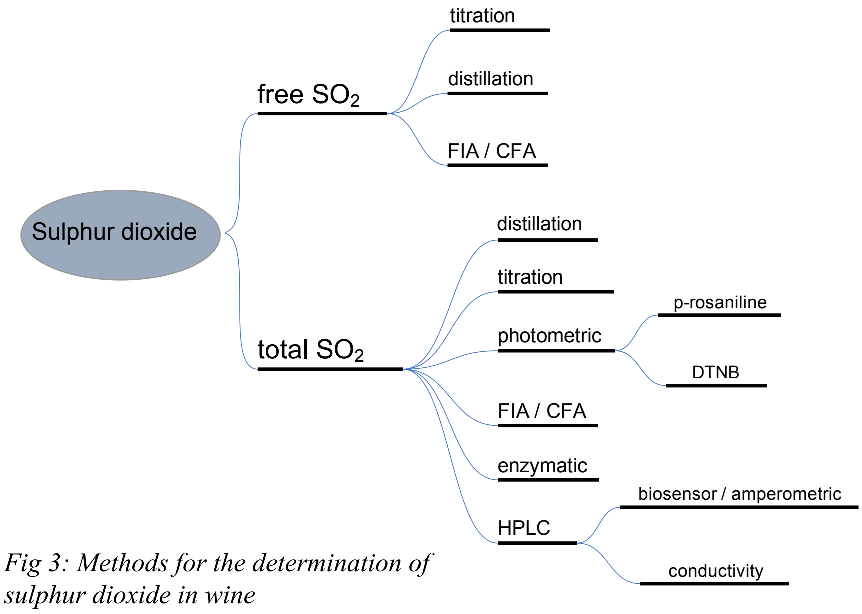

Fig 3: Methods for the determination of sulphur dioxide in wine

		Bias [mg/l] SO_2	SD of differences [mg/l] SO_2	R^2
Free SO_2	White wine	0,7	2,6	0,915
	Red wine	1,4	2,7	0,898
Total SO_2	White wine	3,2	4,4	0,986
	Red wine	6,2	9,9	0,909

Table 1: Bias and standard deviation (SD) of difference between reference method and FIA method for determination of free and total SO_2 in red and white wines

Cont'd from page 11

2676/90 method. When compared with the titrimetric methods used in routine analysis, the FIA method generates intrinsically more reliable and more precise results. The number of deviating results due to the composition of the wine (phenols and ascorbic acid) is highest for the titration methods.

The FIA method is excellent for fast and reliable determination of free SO_2. The reference method demands an analysis time of at least 15 min for a single determination, and the difference between duplicates is substantially higher than with the FIA method. With FIA, duplicate determinations can be performed within 2 min, generating simultaneous results for both free and total SO_2. Exact and reliable determination of free SO_2 is absolutely necessary, particularly for process control and during bottling.

During the Third Workshop on Wine Analysis, held in Germany in 2005, it was assessed that many white wines distributed in Germany had levels of free SO_2 that were too low to provide sufficient protection against oxidation. However, increased use of L-ascorbic acid in UTA prevention makes a minimum of SO_2 necessary, as the degradation of the ascorbic acid results in peroxide, a strong oxidation reagent. Reliable and correct determination of free SO_2 is therefore a necessity.

NOTE: UTA is an unwelcome aging off-flavour that is predominantly found in young white wines. This off-flavour stems mainly from 4-amino-aceto-phenone, which is formed during storage. Wines smell 'like acacia flowers'. This off-flavour is a major problem because a lot of white wines are no longer drinkable after storage of no more than one or two years.

Total sulphur dioxide

Figures 6 and 7 show results for white wines (range 70-250 mg/l) and for reds (range 38-195 mg/l). Bias and standard deviations for red wines are clearly higher than for whites.

The mean bias between the FIA method and the reference method is 3 mg/l for white wines, and the standard deviation of difference (sD) is 4,4 mg/l. For red wines, the mean bias is 6 mg/l and the standard deviation of difference (sD) is 10 mg/l. When compared with the direct titration method, FIA shows clearly better agreement with the reference method and significantly better repeatability.

The FIA method is therefore more reliable than the direct titration method, especially for higher sample throughputs. This should be emphasized here, as the titration method is approved for AP analysis.

NOTE: All German quality wines are awarded an official Approval Number (AP Number). For an AP Number to be awarded, the wine has to be examined by chemical and sensory analysis.

Table 1 gives a compilation of the results. When compared with the reference method, the FIA method shows systematically lower values for red wines, especially for total SO_2 (see Fig. 7).

When compared with the titration method, the FIA method is not sensitive to interferences e.g. from ascorbic acid, acetaldehyde, phenols or other reducing compounds. Interferences from residual sugars are very low, and can be disregarded up to a sugar concentration of about 100g/l.

For routine control of red wines, the negative bias of the FIA method for total SO_2 can be accounted for by applying the reference method before reaching legal limits for total SO_2 content.

Discussion

FIA is a fast and reliable method for determination of free and total sulphur dioxide in wine. The advantage of the method lies in the relatively high sample throughput of about 60 samples per hour, which makes it the method of choice if 30 or more determinations are made per day. Thanks to the speed of analysis, it makes sense to analyze several samples consecutively as against, say, one sample every 30 min. Results are comparable with those obtained using the reference method, especially in relation to results obtained using other approved methods. For total SO_2 in red wines, the FIA results are systematically about 10-15 mg/l lower than those obtained using the reference method. Compared with other fast methods, FIA produces results closest to the reference method. For single samples, the titration methods in use always produce outliers when compared with the reference method.

Reliable determination of free SO_2 content is only possible with FIA or the reference method. For fast and precise determination of free SO_2 before and after bottling, FIA is the most reliable method in terms of

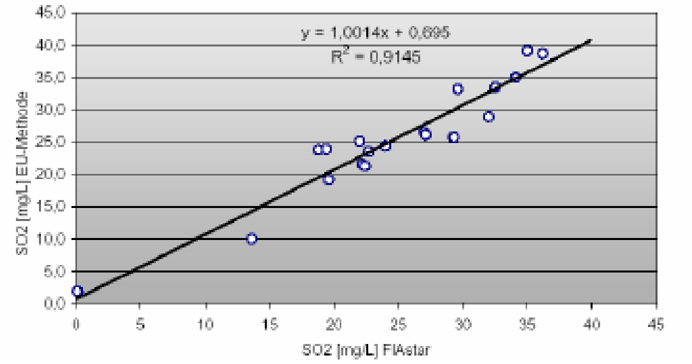

Fig 4: Free sulphur dioxide in white wines: FIAstar™ v EEC 2676/90

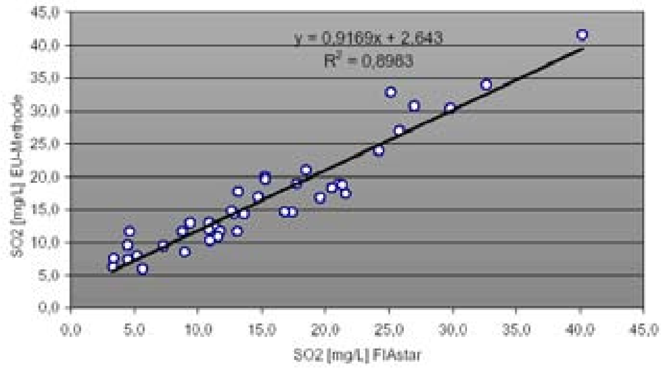

Fig 5: Free sulphur dioxide in red wines: FIAstar™ v EEC 2676/90

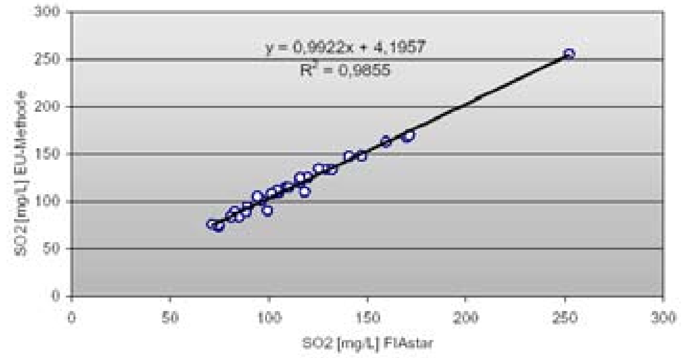

Fig 6: Total sulphur dioxide in white wines: FIAstar™ v EEC 2676/90

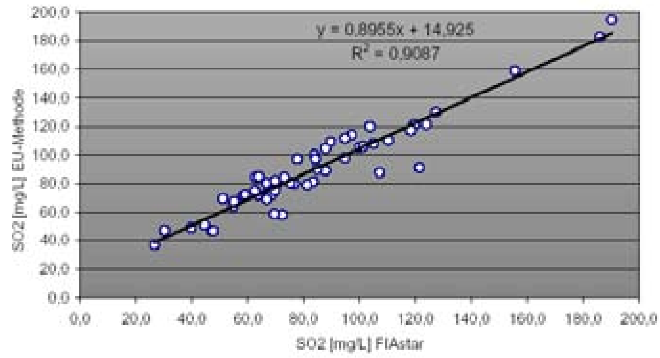

Fig 7: Total sulphur dioxide in red wines: FIAstar™ v EEC 2676/90

accuracy as well as repeatability.

For total sulfur dioxide, the same is valid as for any other fast method: they are all suitable for fast screening, and should be validated by the reference method when close to limit values. When compared with titration methods, FIA is not, or is much less, prone to interferences from ascorbic acid, phenols, sugars and acetaldehyde.

Conclusions

FIA is a fast and reliable method that permits high sample throughputs. Daily calibration using freshly prepared standards, and validation using a quality control sample, are part of GLP and should be applied to all analyses.

For production control purposes, the method delivers very reliable results for free SO_2, which is of high practical value. For determination of total SO_2, especially in red wines, the method may show lower results than those obtained using the reference method. However, this bias can be corrected.

The FIA method is considerably less prone to interferences and errors than titration methods. Accurate control of actual content of free SO_2 is a necessity, and here FIA is the method of choice.

During 2006 the FIA method was compared with the official reference method in a major ring test in Germany. Satisfying results were obtained. Approval for the purposes of German AP analysis is therefore only a question of time.

FIAstar™ method allows simultaneous analysis of free and total sulphur dioxide in wine

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IFU methods can be found under: http://www.ifu-fruitjuice.com/

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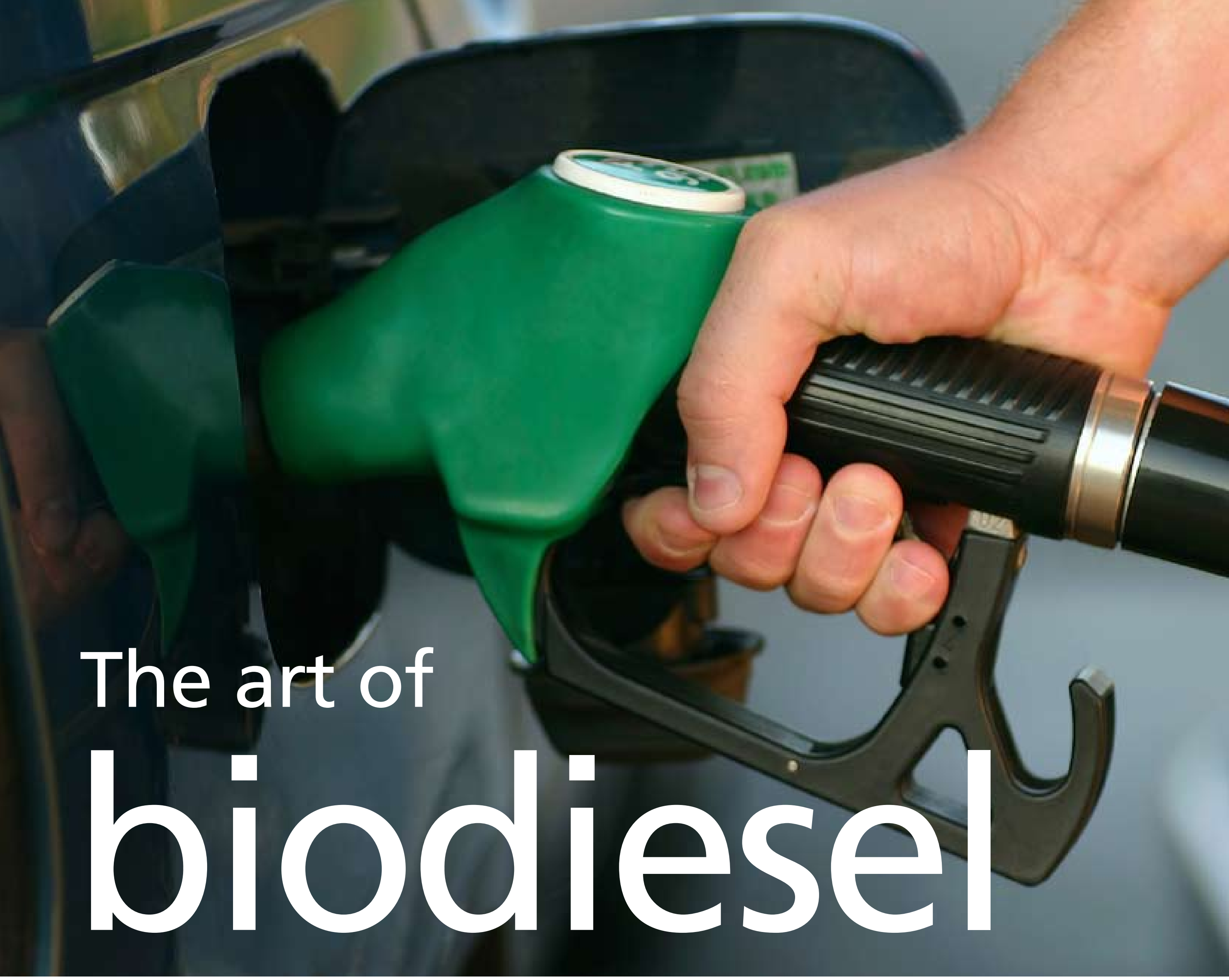

The art of biodiesel

The first biodiesel plant to start up in the Netherlands is using a FOSS XDS analyser to achieve its high quality standards. In Focus went to find out more about how routine analysis is contributing to success in the rapidly evolving biofuel sector.

Wilfred Hadders, General Manager of Sunoil Biodiesel has little doubt about the key to success in the biodiesel business. “Quality is the most important consideration,” he says, holding a bottle of raw material in one hand and a bottle of finished biodiesel in the other to indicate how the quality principle applies throughout the process.

Biodiesel is an increasingly popular fuel with studies indicating rapid growth in consumption. Europe currently represents 90 per cent of consumption, with other countries catching up quickly. In the U.S. the number of retail outlets has risen from 300 in 2005 to more than 950 in 2006. One survey by Emerging Markets Online predicts that the fuel could represent as much as 20 per cent of all road diesel consumption in Brazil, Europe, China and India by 2020.

Add to this the European Union targets for biofuel consumption and biodiesel appears to be a good business to be in. But challenges remain, such as competition from bigger and more established plants in Germany. International standards must be met and cheaper imports from outside Europe are a constant threat. The quality that Hadders refers to is therefore essential to the future of Sunoil.

A young company

The company started just two years ago with production operating for about a year. The plant is neat, clean and efficient and to the unfamiliar eye surprisingly small for a plant currently producing around 80 million litres of biodiesel per year. If it were moved a few hundred kilometres further south it could easily be mistaken for a large winery.

There is an air of a successful start-up company and obvious pride in the whole undertaking of producing quality biodiesel. The laboratory manager Marc Arends describes how he used to make his own biodiesel at home until he discovered the Sunoil plant was starting up in his home town. He was quick to make a career move and his hobby became a full time job.

The process and need for analysis

Sunoil employ the most common method of making biodiesel. This involves taking raw oil and separating out the glycerin using a catalyst – a process called transesterification, see figure 1.

First the oil is put into a process tank and stirred. A catalyst consisting of potassium hydroxide and methanol is added. After a time the lighter diesel separates leaving a heavier residue of glycerine. The process is repeated to ensure that the conversion is as complete as possible, typically 99.5%. The diesel is cleaned to remove any leftover catalyst and methanol which is then re-used for subsequent processing. The glycerine is used for biogas production or for animal feed with the methanol removed. The biodiesel is used as is (B100) or mixed with regular diesel, for instance B10 or B20.

The XDS Rapid Liquid™ Analyzer is located in the plant laboratory and is used at different stages of the production process to check: 1. all incoming raw material, 2. the conversion process and 3. final product quality.

Hadders explains the importance of having a good laboratory to check incoming raw material. "Every batch is different and it is important to check everything to decide how best to process it," he says.

This flexibility is important. One day the plant could be using rapeseed oil, the next day soya oil or waste cooking fat. Sunoil is also investigating new potential sources including Jatropha, an inedible plant that grows well in arid conditions in countries such as Africa and India that could provide an effective non-food source for biodiesel.

Measurements throughout the process

Whatever the source, all incoming raw material is measured for moisture and free fatty acids.

A too high concentration of free fatty acids creates problems in production, for example with soap being produced – not exactly a desired biofuel. Generally, rape seed provides the best raw material source with a typical free fatty acid content of 0.7%. Refined soya oil is about 0.01 and used cooking oil is up to 5.0%. A high concentration of saturated fat makes it difficult to produce fuel that performs well at low temperatures, so waste cooking oil is exploited mostly for summer fuel.

Moisture content is important for usage of the fuel at low temperatures and is part of an international standard EN 14214 with an upper limit of 500mg/kg.

In addition to the raw material meas-

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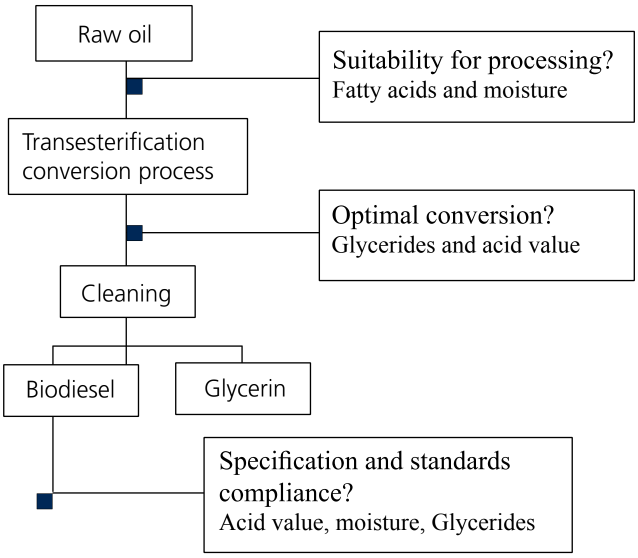

Fig.1 Schematic description of the biodiesel production process and control points. The transesterification method is a popular way to make biodiesel because it requires only low temperature and pressure, the conversion rate is high and there is a direct conversion with no intermediate compounds

The near infrared advantage: measurements with the XDS analyser shown here are fast, accurate and simple to perform by anyone

Sunoil Biodiesel currently produces 80 million litres of biodiesel per year

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urements, analysis is done right after the conversion process to check the effectiveness of the conversion process. The final biodiesel product is tested for compliance with standards including the EN 14214 standard.

The near infrared advantage

The plant originally used a Gas Chromatography instrument for analysing glycerides.

Measurements take about an hour with this method compared to about two minutes with the XDS near infrared (NIR) instrument. But for Marc Arends, the main advantage with the XDS is in its simplicity of use.

Anyone in the plant can now make a reliable measurement allowing more frequent analysis without the risk of human error that can occur with operators unfamiliar with Gas Chromatography technique. With the XDS, a liquid sample is simply placed in the XDS analyser and the results are displayed on a computer screen. "In the past there was some doubt about some of the results because obviously not everyone working in the plant is a trained laboratory technician," says Arends. "But now, with the XDS, it is so simple to make a measurement that the human error is removed and I can get on with other things instead of checking the validity of measurements."

The simplicity-of-use angle has similar advantages for checking the conversion process and spotting potential problems such as reduced yield due to poor conversion rate. "If there is a problem we can react more quickly," says Arends.

Besides the control parameters provided by XDS there are of course many more to be measured, "We dream of being able to measure everything as quickly and easily as with the XDS, remarks Hadders.

The specific parameters measured with the XDS NIR analyser during the production process are: Mono, Di and Triglycerides, Free Glycerin, water and Acid value.

NIR analysis is not just for big plants

While some may say that NIR analysis is only worthwhile for bigger companies performing a lot of tests every day, Sunoil have a different angle. This is to do with their special approach that involves knowledge, experience and a certain amount of instinct. "It helps that we are quite a small company," says Hadders. There is a feeling in the whole plant – a common goal to achieve the best quality- you loose that with a bigger plant."

The bottle of finished diesel that Hadders has used to explain the process may look ordinary to the untrained eye, but to everyone working at Sunoil it is proof of their success in achieving quality in a profitable way. The XDS is performing a vital role in achieving that success, providing a straightforward way for any of the staff to track production and maintain the company's competitive edge against bigger players. "We can do more analysis, we know what is going on, we know that what we are producing is of a high standard and that is everything for us," says Hadders.

■

by Richard Mills, FOSS (rim@foss.dk)

Biodiesel

Biodiesel is made by converting raw vegetable oil or animal fats into diesel with the use of a catalyst. It can be used directly in existing diesel motors as is or mixed with regular diesel.

The process creates glycerine mixed with methanol as a by-product. This can be used for biogas production or for animal feed with the methanol removed. It is also used in the pharmaceutical industry.

A variety of sources can be used according to price, availability and environmental conscious. Sources include rape seed oil, soya oil, palm oil, sunflower oil or even waste cooking oil. Potential new sources include Jatropha, an inedible plant that grows well in arid and poor soil conditions.

The oil to diesel conversion is efficient – one litre of rapeseed oil can make as much as one litre biodiesel.

Touch of quality

US dairy producers Oakhurst Dairy give their reaction to the new touch screen software available with the MilkoScan™ FT2.

Wendy Donovan Landry, Director of Quality Control at Oakhurst Dairy is directly responsible for ensuring consistent quality of Oakhurst brand name products supplied to their New England customers. If there is an issue with quality control, it is Wendy that gets the call, day or night! Hardly surprising then that she has a keen interest in the effectiveness of analysis operations.

She describes how analysis is a constant daily demand, from checking the supplied milk to testing finished products. The laboratory is certified for official testing of all raw milk entering the plant. "People pay extra for the Oakhurst name and package and we want to protect it," says Wendy.

Oakhurst Dairy acquired a MilkoScan™ FT2 in April this year to replace a MilkoScan FT120 that had seen many years of constant service. The aim with the new FT2 was to continue where they left off with a former FT120, using it to test all the raw milk as well as the finished product. The dairy then took up the offer from FOSS to try out the new touch screen concept for three months.

Touch and walk away

The touch screen software was tested in the QC laboratory for analysis of fat and total solids on finished products and analysis of protein, lactose and total solids on raw material. It was also tested by production operators, typically to check butterfat and total solids at the start of a production run and then butterfat in the middle and end.

The new software was a particular hit with the production operators as Wendy explains: "It is very friendly for the daily operators to use and they are the ones using it around the clock. They can just touch and go and we save a lot of time that way," she says. The ease of use was crucial in this respect. "It stopped the phone calls in the middle of the night," says Wendy.

To do an analysis with the touch screen software the user simply selects the product from an icon on the screen. If the required product is not displayed it can be selected from a complete list of products. Then the test is started and that's it.

The instrument at Oakhurst has been set-up to automatically clean one minute after the analysis. "We made it as easy as possible for them to select the product and get the result and then they could just walk away," says Wendy. She also explains how the simplicity of use worked well with the dairy's monitoring policy. "We always track the results and operators fill out a hardcopy report. So if I have a customer complaint about a gallon of whole milk with low butterfat content I can see if it has been tested properly. It holds the filler operators very accountable."

Point and select: The touch screen software makes it simple to select the required product

From FT120 to FT2

The MilkoScan FT2 has proved to be familiar and modern at the same time. Calibrations from the FT120 were transferred and new calibrations were also supplied, ready-to-go. Installation of the new FT2 took around a day and a half. The FT120 was run in parallel to check results with duplicate samples with very consistent results.

The touch screen and software took about three hours to install and operators were up and running with it within about ten minutes. As far as Oakhurst Dairy's quality control manager is concerned the verdict on the touch screen software is clear. "I loved using the touch screen within about five minutes of trying it," she says. ■

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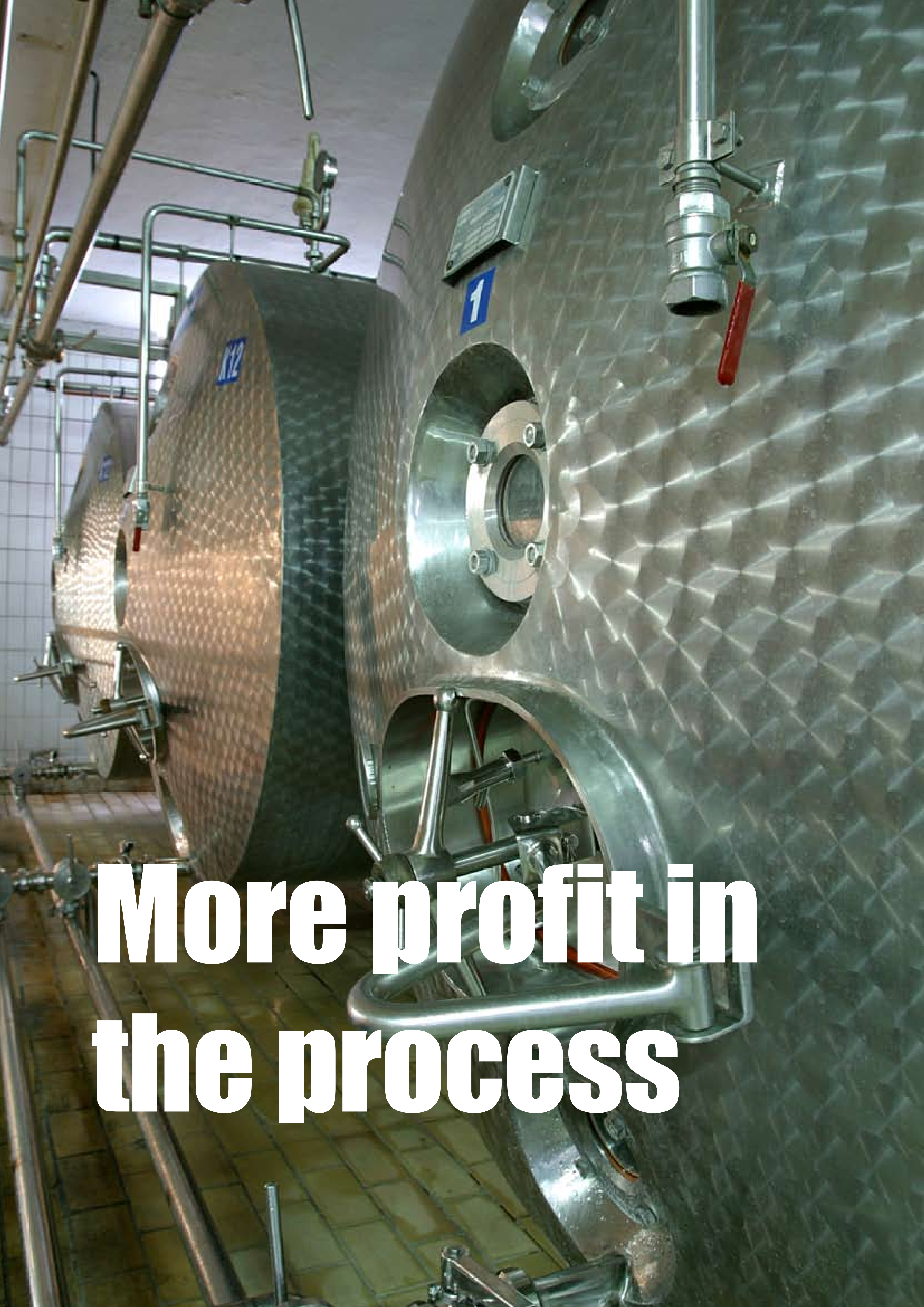
Background

Oakhurst Dairy from Maine is Northern New England's largest family-owned, independent dairy company. Quality control is paramount for brand protection.

The dairy had used a MilkoScan™ FT120 for nine years before upgrading to a MilkoScan FT2 earlier this year. The touch screen software was tested for three months.

www.oakhurstdairy.com



More profit in the process

Analytical procedures in the dairy industry are changing as the focus shifts from the laboratory towards at-line and continuous on-line process control. But what sort of business can really benefit and is it worth the time and investment in implementing a process control solution?
Henrik Boisen from FOSS explains the available options.

Process control – a great concept, but is it really for me?

That's a common question that I always answer with another one in return. Do you need to find ways to improve profit and reduce investments in your dairy process?

Assuming the answer is yes, then it is worth looking at a number of options for improving process efficiency through process analysis – options that are happily increasing in step with advances in process technology in general.

Automation has been the key word in recent decades in the drive to improve production efficiency, for instance, in standardisation with 'indirect' control (the interpretation of "indirect" is when signals other than the direct measurements provided by a component analyser form the basis for the process control/standardisation). Density and various flow-blending solutions are the commonly used indirect methods of controlling the milk composition in final or intermediate dairy products.

Protein components

In milk standardisation, the focus has been on adjusting to the right protein level, and even different sources of protein are now widely used within a single batch.

The complexity in standardising two or more components on-the-fly is a significant challenge. This is leading to a growing interest in process solutions from forward-looking dairy producers who are seeking ways to get the standardisation results right in real time. Such solutions free up storage capacity and increase throughput and efficiency as well as ensuring top quality. Many producers want to cash-in on such advances in technology and achieve a higher profit while protecting their brands.

Analysis technology has been developed to enter the process floor and now offers options for dedicated or more versatile applications. Among the technologies we can list: light scattering, infrared (IR), near infrared (NIR) and Fourier Transformation IR (FTIR) as well as other new technologies that are targeting new interesting applications in the dairy industry. And many producers are already reaping the benefits from advances in analysis technology.

Examples in action today include the FOSS ProcesScan™ FT (Figure 1 – FTIR) for liquid applications and XDS Process Analytics™ (Figure 2 – NIR) for viscous or powder applications and controller solutions referred to as ProcessTouch™ (Figure 3).

Process control in action today

Powdered products

Production of powdered dairy products is one area where process has taken off in recent years. On-line process analysis and control is the key to constantly surveying and recording what is running in the process pipe and potentially feeding the evaporator directly.

At the same time, the process controller takes advantage of the compositional input and automatically maintains the required target levels for the recipe in process. A dedicated controller solution will additionally compensate for process disturbances, providing a more sophisticated and flexible solution. With protein standardisation as the second component or even with more complicated special recipes, the compositional variations in the final product are narrowed significantly. The payback in saved components will pay for the installation of the on-line process control.

Cheese

In cheese production the twin issues of control and profitability have been a driving force for investment in on-line control and, in particular, during the last decade, protein standardisation has made it even more attractive to go on-line.

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Fig. 2

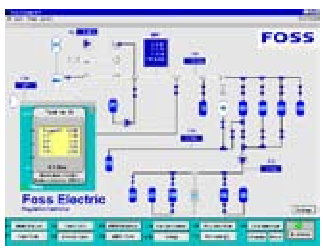

Fig. 3

Fig. 1

Examples in action today include the FOSS ProcesScan™ FT (Fig. 1 - FTIR) for liquid applications and XDS Process Analytics™ Microbundle Multiplexer (Fig. 2 – NIR) for viscous or powder applications and controller solutions referred to as ProcessTouch™ (Fig. 3)

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One thing is fat and protein standardisation which is profitable on its own. When different sources of protein and other products must be administrated on-the-fly and in real time, a process solution is the answer. By narrowing the variation in the cheese-yielding protein and the fat between vats and thus the fat in dry matter variation, the cheese production manager is given more freedom to optimise fat and moisture level resulting in stable quality, constant weight and profitable revenue.

Whey protein concentrate

Once considered a by-product of cheese production, Whey Protein Concentrate (WPC) is now an important revenue generator too and protein content of the finished WPC products are focused on as a major contribution in an optimised production loop. Here also, several manufacturers are now taking advantage of the on-line process solution with payback periods calculated in months.

Fluid milk

Fluid milk standardisation typically involves fat standardisation and the process performance of a process control solution will form the platform for the tangible calculation and the pay back period. Additionally, process considerations such as flexibility, storage capacity, labour requirements and standardisation on-the-fly will also contribute to the intangible value of such an installation. In some cases, solids-standardisation and, in the future, protein standardisation or standardisation with vegetable oil, are areas where a process solution can handle the complexity with an improved performance. In most cases, payback can be calculated in months.

Butter

Butter is the most common application handled by Near Infrared process solutions in the dairy industry. The real time analysis particularly controls, moisture at the highest possible level without exceeding legal limits. Start up or restart of the continuous butter churn is eased and the target is reached quickly. Standardisation of other parameters like salt and solids and control of the fat level will also contribute to the tangible value of such an installation. These installations have, in most cases, a manual interface for adjustment of the control flow based on a visualisation system and additionally, a regulation controller will increase the efficiency of the overall solution.

Other NIR applications include cream cheese, quark, processed cheese, mozzarella, powder among others.

Process control considerations

The technologies used for process analysis have their pros and cons within the applications for which they are marketed

The different technologies provide various prediction performances and calibration stabilities on the different components measured. The choice of technology and the associated technology provider is therefore an important issue to consider. What you may save at the time of installation can easily be lost shortly afterwards if, for example, the calibration stability or performance is missing.

Secondly, some process sensors are primarily used for trending analyses. Here, the process control needs to be manually adjusted according to the compositional results of manual samples measured on an accurate bench analyser. Process noise and switches in raw material supply etc. can easily change the picture and thus the final process performance achieved.

A common factor that can be seen across the different application possibilities available is that process control works best when it is implemented in the big picture. It does not really help if just some of the things are in place.

So an analytical process solution means thinking big to include a dedicated analyser/sensor, the optimal control solution, PLC communication and the engineering involved in installation.

With the right solution in place, continuous process improvements then follow.

ProcessScan™ FT

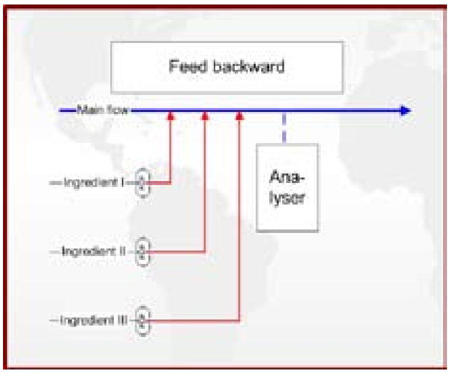

Fig. 4

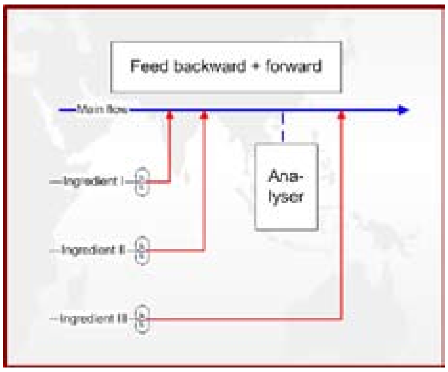

Fig. 5

The future of process control – continuous optimisation

The major advantage of accurate measurements from a process analyser is that it will automatically and continuously provide the platform to optimise the process. You don't need to be concerned about the results after filling a batch and you avoid post standardisation, thus saving time and storage capacity. Additionally, you can avoid intermediate storage tanks in many processes.

The most accurate control set up is feed backward (fig. 4) but in some special cases you can take full advantage of a combination of feed backward and feed forward (fig. 5). Feed forward is occasionally established in the liquid process stream, but can also be administrated for dry ingredients in the powder sections of the drying process.

The ultimate advantage to be taken from a real time, process analyser is an advanced regulation control which is not programmable in a PLC. Such programming will do simple set point adjustment whereas a dedicated and customised controller solution covers the most complicated standardisation challenges you can ever imagine.

Achieving this scenario though demands dedicated process analysers combined with controller solutions to improve quality, flexibility and not least the profitability in your production.

■

by Henrik Boisen, FOSS (hb@foss.dk)

At-line, On-line and In-line process control definitions

At-line process control is where a stand-alone analyser is used, close to, or directly in the process environment for making fast, manual measurements.

On-line process control (e.g. FTIR) includes a sensor/analyser which automatically takes a little sample from the process line for analyses. The compositional results are then made available for a controller system.

In-line process control (e.g. NIR) includes a sensor/analyser which automatically makes non-destructive measurements of the component concentration of a product in the process stream.

Infrared influences wine quality

Leading wine producer in Alsace, the Wolfberger group demonstrates its ambition to optimise the quality of grape content.

The Eguisheim site handles an average of 400 tonnes of grapes per day. With an estimated 2 tonne content per cooperative member, the GrapeScan™ being handled by Stephan Grappe, oenologist at Wolfberger, carries out nearly 200 maturity analyses

With an annual production of 100,000 hl of wine divided among five production sites, the Wolfberger group is the leading market representative of Alsace wines. For several years, the group's research and development centre which is managed by Stephan Grappe, has been examining the relevance of including quality parameters for grape yields. "We started this research as the result of a request made by some of our members", explained the oenologist.

Cooperative members estimate that for grape contents with an equivalent sugar content, the state of health and concentration of organic acids must contribute to establishing a "fairer" price. The GrapeScan™ analyser, built and commercialized by FOSS, uses Fourier Transform infra-red technology (FTIR). Used in other sectors of the food-industry (dairy, fruit juice, etc.), it is also suitable for the analysis of wine and grape must. "The FTIR analyses back up our observations, and the speed of execution – about two minutes between the moment the sample is taken and the result – makes it possible to carry out real-time harvest orientations, particularly with regard to soundness related quality criteria", explains Stephan Grappe.

This is a particularly noticeable advantage this year, due to the many cases of rot registered in Alsace vineyards. The device is used on an advanced station beside the weighbridge to manage content during harvest. The rest of the year, it is used in the laboratory for routine analyses and the monitoring of bottling lines. During the weighing, a sampling is drawn from the skip or casks. An analysis can then be carried out.

Grappe says: "The device is connected to our own computer system, through which we can edit the content ticket as soon as the analysis is completed and indicate its qualitative destination from among several unloading platforms." At Eguisheim (Upper-Rhine), the cooperative's head office, the fermenting room is equipped with eight wine presses by means of which four different selections can be made, without interrupting the pressing process. Apart from the potential ABV (alcohol by volume), acidity, pH and other standard parameters, the GrapeScan provides indexes which allow us to estimate the microbiological activity associated with flavour. To evaluate the grape's degree of soundness, the machine detects the presence of metabolic compounds (gluconic acid and glycerol) synthesised by Botrytis cinerea, the agent of rot.

This measurement is then refined. "We calibrate three references from musts coming respectively from healthy, rotten, and a mixture of half-healthy and half-rotten grapes," says Grappe. This grey rot index is used to attribute a health note showing the microbiological activities of the must (grey and acid rot, fermentative and lactic activities). It mainly depends on the potential ABV and the concentration in malic and tartaric acid, two physiological tracers of grape maturity. These two notes balanced on a daily average established on each site, in line with all the harvest contents, contribute to calculating the price of the grape.

"Thanks to GrapeScan, we have been optimizing our harvest schedule for two years," confirms the oenologist. The vintages reputed to be sensitive, such as the Pinot Noir and the Pinot Gris, are systematically analysed, as well as the 19 grand cru appellations converted into wine by the group. Knowing that the quality of the wine is directly correlated to the quality of the grape, there is good reason to wish for a better understanding of the nature of the raw material, and to advance in the quality control of elaborated wine.

The only drawback is the machine's lack of flexibility. Describing how to operate the GrapeScan, Grappe says: "You must strictly observe the handling and maintenance procedures of the machine. The database provided by the manufacturer and set up from 12 harvest campaigns allows you to make extremely reliable calibrations. And thanks to the 'maturity network' set up by the cooperative members, we are refining these calibrations with our own analyses in order to take into account the vintage wine effect."

The company has agreed to an investment of nearly €300,000 to equip its five sites. "Every year, we organize plenary sessions to compare the results observed with the management methods adopted by wine growers". For Stephan Grappe, this carefully planned investment contributes to making all the cooperative members more aware and ratifies all the efforts agreed to with regard to vine management, before entering the cellars.

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RÉGINE SÉRANGE,
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Handling non-linear data with Artificial Neural Network calibrations

Artificial Neural Network, ANN, plays an important role in the world of chemometrics. It is a tool that has been used by FOSS for more than a decade and is a tool important for both NIR and image based instruments. Martin Lagerholm from FOSS elaborates on the subject.

An Artificial Neural Network is a construction inspired by some aspect on how the mammal brain works. One could claim that Biological Neural Network is the foundation of intelligent life. Now it would be spelling to state that Artificial Neural Networks is the foundation of intelligent calibrations, but that would be a slight exaggeration.

By itself, an artificial neuron is a very simple construction inspired by the real biological neurons. A single neuron can only perform very simple calculation, typically by responding to an input in a non-linear fashion, for example, by producing just a single number as an output. But a highly connected network of neurons can, in principle, make any calculation possible by a computer. An appealing characteristic with ANN is that they are strong (just like the brain) where traditional computing is weak e.g. forecasting, recognition tasks, learning by examples and optimization tasks.

Within the NIR field, a standard calibration method is PLS (Partial Least Square), a fundamentally linear method that in many cases works very well at least as long as the problem to a good approximation is linear. Non-linearities enter into the problem from many sources; for example, scatter, instrument differences, temperature effects, the reference method. In addition, the fundamental law in this field, the Beer-Lamberts law, that expresses a linear relationship between the absorbance of light and concentration of material is an approximation of complex underlying physics.

Even with clever pre-processing that aims to remove non-linear effects, one often finds that linear methods are outperformed by ANN. This is especially the case when large databases are available and when the calibration is evaluated over a large population of instruments. Examples from the FOSS product portfolio include meat with FoodScan™ and wheat with Infratec™. In some cases, a linear approximation is especially poor and ANN could be called for even with smaller databases and for single instruments, for example, moisture in whole grain over a wide range or a wide range of fat in meat.

ANN is also used extensively for image analysis. From the FOSS product portfolio the best example is the Cervitec™ product range used for classifying kernel damage in rice and wheat.

With such complex phenomena as kernel damage (Figures 1 and 2) one cannot assume that the image response is linear. ANN is therefore the obvious choice of calibration technique.

And even if some of the phenomena are of a linear nature, ANN is excellent in handling those as well. Although the parameters to be measured are complex, the calibration development is straightforward: All you need is a database with the kernels sorted out in their final classes; this will allow you to train the ANN 'intuitively'.

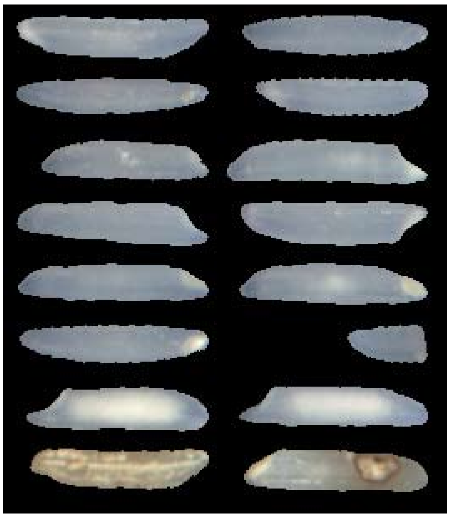

Figure 1. An example of long grain rice captured by a Cervitec™ 1625 where ANN quickly picks up the differences between the classes and learns how to predict unknown examples accurately. There are 13 sound kernels, one broken, two chalky and two discolored kernels (in that order). It is also very easy for us to spot the unsound kernels with our human eyes and our biological neural network, but when it is necessary to analyse tens of thousands of images every hour it becomes very hard and tedious work.

Figure 2. Example of images from a Cervitec™ 1642 showing Australian durum wheat. An ANN calibration predicted the six first kernels as vitreous (hard kernels that form good semolina for pasta making), the next one as non-vitreous (opaque kernel) and the last one bleached (the surface makes it hard to see through the kernel).

by Martin Lagerholm, Ph.D., FOSS (mlm@foss.dk)

Vis-NIR tools for quality assessment of button mushroom compost

The mushroom industry representing composters, spawn and casing producers, producer groups (growers), marketing groups and other associated sectors is estimated to be worth 2 Billion Euro to the European economy and employs 120,000 people largely in rural areas. In recent years, studies have been conducted to identify key parameters of raw materials such as wheat straw and poultry litter at pre-wet, important changes in the substrate during phase I, II and III (spawn-run) stages and to assess NIR technology as a rapid analysis option. The results reveal how Vis-NIR analysis can lead to a rapid monitoring of changes in substrate parameters during production.

The industry in the European Union

Button mushrooms *(Agaricus bisporus)* are widely grown in Poland, Holland, Belgium, Ireland, the UK & other EU member states. However, the production technologies used are different, ranging from indoor composting in Holland to phase I, II and III production protocols adopted in Ireland or Poland.

The preparation of compost is a balance between allowing the synthesis of nutrients for *Agaricus bisporus* that confers selectivity to the substrate and minimising the loss of carbon fraction. Moisture control is critical in optimising the decomposition process, as it not only affects the microbial processes but also aeration and gaseous exchange within the compost. In phase I, raw materials are first wetted, followed by blending and left as a loose pile for 1–3 days before forming a windrow or transferring into a bunker for a duration of 7–11 days. This is followed by a phase II stage, consisting of pasteurisation and conditioning periods for 5 to 7 days in a bulk tunnel. European researchers have investigated compositional changes in compost for nitrogen dry matter (NDM), dry matter, pH, carbon, ash, lignin, phenolic acids, thermophilic population of micro-organisms and fibre fractions of substrates. During phase III/spawn-run and cropping stages, changes in the concentration of NDM, the dormant biomass, fibre fractions and certain enzymes have also been monitored. The factors affecting the productivity of substrate are many, including differences in the availability of key nutrients listed above, the type of spawn used, the management of crop and environmental conditions in the mushroom house and watering regime adopted during case-run and cropping.

The European industry is a highly competitive marketplace and is influenced by financial and political directives of the European Union. The supply chain is flexible enough to import raw materials, such as wheat straw from neighbouring member states based on cost and availability of suitable material. Another example is the supply of phase I compost produced in Poland to composters in the UK or other countries for phase II and III stages of production and cropping, as it is cheaper to prepare the substrate in Poland compared to higher labour costs in Western Europe. In addition, grower groups in Hungary, Poland, Ireland and Holland supply mushrooms to the UK whole sale market.

Quality assessment of substrate

During the past 15 years, several collaborative projects have been initiated with composters, growers and research institutions in Europe. Main objectives of the work programmes were to identify key parameters of substrates during the production stages and also to develop rapid tools using a FOSS 6500 spectrometer. An additional aim was to establish a range with target values of parameters for monitoring quality at phase I, II & III. The data base could be used for comparing changes in key parameters during production and cropping with spectral results. Extensive sample sets were obtained from commercial composters for analyses followed by randomised cropping trials to monitor differences in key parameters and the productivity of substrates.

Vis-NIR analysis of substrate

The use of the Vis-NIR calibrations as tools could improve the measurement standards of the mushroom industry. Key aspects are as follows:

1. A composter can send raw materials and production batches of compost to a research laboratory equipped with suitable Vis-NIR calibrations and the predicted values of the key parameters can be used for monitoring the substrate recipe formulation and changes taking place at each stage. Key target factors are input raw materials for moisture content, NDM, ammonia and fibre fractions including lignin. During wetting and mechanical blending of straw and chicken litter, the blended materials can also be monitored for the same parameters from a set of Vis-NIR scans of each sample. This will provide the compost yard manager with the opportunity to compare the production efficiency of different batches.

2. The spectra of samples can identify shifts in specific spectral segments, indicating changes in dry matter, nitrogen dry matter, fibre, thermophilic population and lignin-humus content of substrates during production and cropping. Intervention steps for improving quality during production, management of environmental conditions in tunnels and cropping have been highlighted

Spraying of recycled water to a pile of wheat straw and chicken litter

Potential benefits to composters and growers

- Quality assessment of raw materials: e.g. wheat straw/horse manure and chicken litter.
- Improved accuracy in the formulation of raw materials
- Better control of processing during phase I, II and III.
- Reduction in the use of chemicals for crop management.
- Improved quality of harvested.

A quick guide to Mushroom production

Mushroom substrate is prepared by composting wheat straw/horse manure (carbon source) & chicken litter (protein source). The straw bales need to be wetted and opened up using a mechanical device. The straw (1000 kg) is blended with chicken litter (450 kg) along with gypsum (10 kg) and this stage is known as phase I. Thermophilic micro-organisms breakdown the cellulose, hemicellulose and depolymerisation of lignin takes place, resulting in the formation of lignin-humus fractions on the straw. This is followed by phase II stage to pasteurise (58°C for 18-20 hrs) and condition the substrate at near 45°C which takes place inside an insulated tunnel. The last stage of production is phase III which takes place in a controlled environment inside an insulated tunnel. The phase II substrate is inoculated with spawn at a spawning rate of 2% and incubated for ca 15-20 days at 25°C.

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Vis-NIR spectrometer for assessing quality of raw materials and substrate

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Further Information

The important quality parameters for mushroom substrate are NDM, dry matter, pH, fibre fractions including the lignin-humus complex, microbial population, ash and certain minerals (Sharma et al, 2002; Lyons et al, 2006). The spectral calibrations and relationship between the parameters of substrate at each stage of production have been published by our research group (Sharma, 2000, Sharma et al, 2005a; Sharma et al, 2005b). Main conclusions of the publications are that calibrations for assessing the key parameters of fresh raw materials, mushroom compost (phase I and II) and productivity of phase II substrate have been developed using a research instrument (FOSS 6500). However the production systems in Europe, including N. Ireland, have evolved in the past 3-4 years or so for various reasons including the enlargement of the European Union, downsizing of production in the UK and Ireland, strict environmental legislations and the availability of suitable raw materials. Consequently, the existing calibrations will need to be updated with new samples before the calibrations can be transferred to other instruments. In addition, there is a need for developing calibration management protocols to maintain the equations developed from the work programmes. The problems associated with calibration maintenance have been highlighted by Sharma (2004).

Conclusion

The quality of substrate can be variable due to differences in the quality of raw materials used and poor control during phase I, II and III stages of production. Growers regularly incur losses due to disease incidence or reduction in productivity. With the introduction of objective measurement standards using Vis-NIR tools, production and analytical costs of substrate production could be potentially reduced at each of the production stages.

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Patties Foods sets new standards in Food Safety and Quality with MeatMaster™

The FOSS MeatMaster™ is an in-line analyser that measures fat content in meat and spots even the tiniest piece of bone or metal at a rate of up 22 tonnes per hour. MeatMaster is set to revolutionise the meat industry and Patties Foods in Victoria became the first Australian user. John Munro, General Manager of Operations shares his experience.

Patties Foods is one of the largest pie producers in Australia, famous for brands like Patties Pies, Four'n Twenty, Herbert Adams and Nanna's. From humble beginnings – it started in 1966 when Dutch migrants Peter and Annie Rijs bought their local bakery "Patties Cake Shop" – Patties Foods now employs 425 staff members. In spite of a growth rate of 400% over the last four years, Patties Foods was owned and run by the Rijs family until its listing in 2006.

"We don't want one single pie with meat contaminants like bones, gristle or metal anywhere near a consumer."

22,000 pies per hour

General Manager of Operations John Munro explains: "Our meat pie production line runs at a speed of 22,000 pies per hour. Before we bought our MeatMaster we would analyse the minced meat for fat content and for contaminants like bones and metal. However, we mince a tonne of meat at a time so if any contaminants were detected it would cost us a tonne of meat. With the MeatMaster we have an in-line process where the meat is analysed in batches of 25kg. So if we detect a contaminant it will only cost us a 25kg box of meat."

Optimal use of raw materials

"Another advantage of the MeatMaster is better production control. It makes the fat content of our end product consistent to an accuracy of 1%, eliminating out of specification products and optimising our use of raw materials. Some of our products have the 'Heart Tick' (a healthy food indicator) and it is obviously critical that they stay within the limits defined by The Heart Foundation. With our MeatMaster we can be absolutely sure they do".

Food safety

"The reason why the MeatMaster will change the meat industry – and the reason why we chose to be the 'first cab off the rank' – is food safety. It is already a big issue in the community and it will be much bigger in the future. We don't want one single pie with meat contaminants like bones, gristle or metal anywhere near a consumer. One single case could easily cost as much as a MeatMaster in financial terms – and then there's the human aspect on top of it".

Marketing edge

"It is still early days, but when the MeatMaster is 100% up and running I believe it will give us a marketing edge on top of all the other advantages I have just mentioned. Our customers are just as passionate about food safety and correct labelling as we are. We can give them total peace of mind that our products are free from contaminants and contain exactly as much or as little fat as we claim".

Checked alternatives

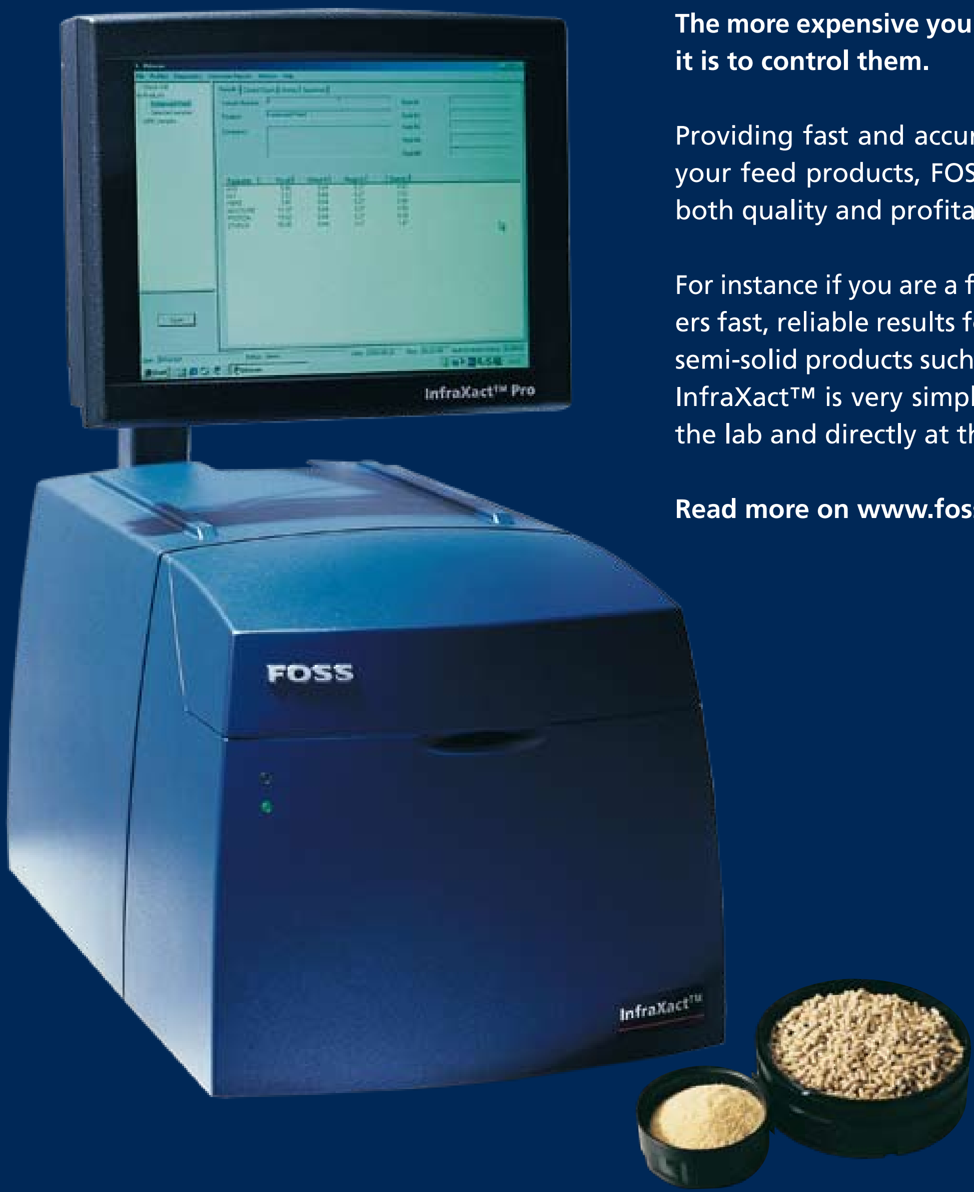
"The FOSS MeatMaster is not the only in-line meat scanner on the market. We checked the market thoroughly before choosing a FOSS. While FOSS is not the cheapest solution, we found their service and back-up to be in a class by its own. The user-friendliness of the FOSS MeatMaster is equally outstanding. We involved the operators in the decision making process and they 'voted' unanimously for the FOSS solution. None of the alternatives were nearly as user-friendly as FOSS".

The future

"We won't have this technology to ourselves for long. A few years down the track any processor serious about quality and food safety will require a MeatMaster...and any processor not serious about quality and food safety won't be able to compete," adds John Munro.

by Casper Reeslev, Ideas Unltd, on behalf of FOSS in the Pacific region

Feed analysis made simple



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