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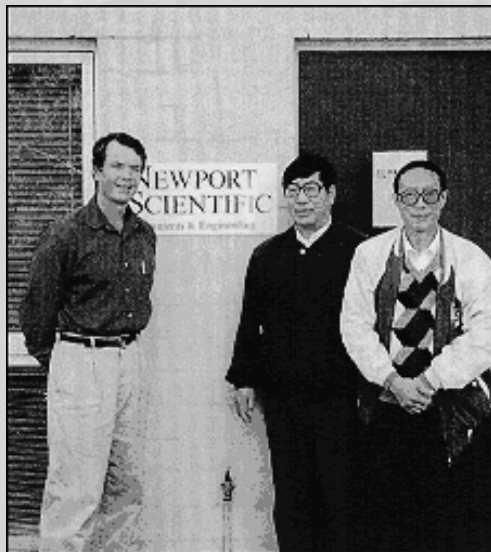
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## **Chinese Research Professors**

The Cereal, Oil and Food Research Centre in Beijing has been using an RVA-3D+ for almost a year now, researching the Chinese cereals industry. In July, two of their research professors, Fan Tie and Ling Jiayu, visited Australia on a study tour.

The Professors' visit included site tours at the University of Sydney's Wheat Research Institute at Narrabri, the CSIRO Department of Entomology's Stored Grain Research Laboratory in Canberra, and the Bread Research Institute in Sydney, as well as work in Newport Scientific's RVA laboratory.



Visitors from Beijing, Fan Tie and Ling Jiayu with Rodney Booth and Newport Scientific.

## **Paris Launch**

In June, Newport Scientific's Managing Director, Rod Booth, Senior Research Scientist, Mark Bason, and International Product Manager, Bronwyn Elliott, were the guests of our French agents Foss Electric France, major suppliers of testing equipment to the cereals and dairy industries, for the European launch of extrusion applications using the RVA. During the two-day session, our agents representing seven countries joined us to hear guest speaker Dr Paul Whalen of Whalen Consulting, Minneapolis USA, introduce extrusion techniques and explain how to use the RVA to obtain process and product information. Then it was the customers' turn to share the experience of the new RVA applications.

## **ICC Congress 1996**

The 10th ICC International Cereal and Bread Congress was held at Porto Carras (Chalkidiki) in Greece in June - the first time in the history of the ICC that such an event has been held at a holiday resort.

The theme of the conference was "Cereals '96 - The Source and Future of Civilisation" and it received the

patronage of the Hellenic Minister for Agriculture, Mr Stefanos Tzoumakas, and the Minister for Culture, Mr Stavros Benos.

Almost 500 participants from 40 countries presented 300 scientific lectures and more than 180 posters. Newport Scientific's Managing Director, Rod Booth, presented the paper, "Solving Cereal Processing Problems Using the Rapid Visco Analyser" and two poster papers, "Correlation of the Japanese Brabender Method for Wheat Flour Viscosity to a Rapid Visco Analyser Method" and "Controlling Extrusion Cooking Processes Using the Rapid Visco Analyser". Newport Scientific also had a stand in the trade exhibition with an RVA on display.

The next ICC Conference in 2000 is a little closer to home for us - the beautiful holiday region of Surfer's Paradise at the Gold Coast in Queensland, Australia. Book early - it's only a week before the 2000 Olympics!

### **IN THIS ISSUE**

- Degree of cook analysis
- Rice pasting quality method lift-out
- Starch based custard powders application
- Meet the people

**If you're serious about starch . . . there is only the Rapid Visco Analyser**



## DEGREE OF COOK ANALYSIS BY RVA

**Paul J. Whalen, Ph.D.**  
**Whalen Consulting Inc.,**  
**200 Fifth Street NW,**  
**Suite E, Elk River MN 55330, USA.**  
**Phone: (612) 241 8871**  
**Fax: (612) 241 8897**

### Introduction

The ability to directly measure product conversion in cook processes has always been attractive because of the inherent advantages afforded in both routine production and research work. It is well recognised that starch is the key biopolymer involved in the transformation of raw ingredients to final products (1). However, the challenge of following changes in the status of starch in the numerous types of cook processes is factored by the interaction of ingredients as well as the interpretation of the results of the particular method employed. Overviews of the numerous methods are replete in the literature (2, 3, 4, 5, 6). The need for some descriptor of degree of cook is frequently addressed in the context of the method employed in the immediate work, but no method is universally useful, applied or accepted in the broad categories of cereal technologies. It follows that the relative conversion of starch in the different products can be categorised in terms of extremes - breads and crackers undergo milder treatments when compared to extrusion puffed cereals and snacks.

The extrusion industry is unlike the baking industry where performing miniature cooks to assess product performance is a common practice. In many cases, variation in a final product made by extrusion may go undiagnosed as being attributed to the process or caused by the ingredients. Further, the transformation pathways for starch conversion are multiples of the numerous processes used at each of the production steps. One needs only to survey

the grocer's shelf to discover the various levels of conversion in the snack and cereal products using cursory sensory criteria. The challenge is greater to specifically identify levels of conversion via analysis. This is because many procedures are specific for structures that may not exist in highly cooked products of extrusion or are confounded by the procedure itself making interpretation difficult.

Generally, thermogravimetric methods require some crystalline or substantial structure in the product or intermediate to make comparisons based on energy changes (7). Molecular starch degradation is certainly occurring and is unarguably the fundamental basis for texture as well as many flavour aspects. However, it is the interaction of the various fragments comprising the structures that is important. Thus, sample preparation which dissolves these structures in various solvents to measure the individual units as in size exclusion chromatography (4, 5) can only allude to the key factors that can be applied back to the ingredients and process. At least these methods are quantitative and reproducible applications of the scientific method. Reasonable sample size and replication is made and useful information generally revealed. Less reliable are the subjective methods of microscopic examination based on starch granule integrity simply because no functional aspects of material transformed or intact can be related. In this sense, the information is as general as that obtained from a well-trained sensory panel.

Viscosity measurement of extrusion products is useful both by in-line methods (slit rheometry (81) or on-line analysis (6, 7, 9). However, single point viscosity such as cold and hot paste viscosity do not show much detail. In-line rheometry also can show only certain aspects of the product

prior to expansion or downstream processing. A method for evaluating all phases of the product forms during production is needed wherein raw material, dough, intermediates, dried half-product, and, final product can be assessed.

Paton and Spratt reported their work on extrudates using the Ottawa Starch Viscometer (6, 10, 11) and proposed that such starch viscosity profiles more fully met the criteria for degree of cook analysis. Indeed they made suggestions for instrument improvements (6) that are now fully met by the new Rapid Visco Analyser (RVA). These authors also suggested the application of viscosity profiles as a standard procedure for food processors using extrusion. Based on this work and general references to employing the Brabender Amylograph in a similar manner, the RVA has been suggested as an industry tool in extrusion, however, since its introduction in 1987 to the US, the RVA has been used primarily in grain quality applications and starch (12). This paper reports application of the RVA in extruded products. The scope of this work is intended to discuss applications, methods, and general interpretations that can be made from RVA analysis in extruded dough and direct expanded products with a brief comparison to some commercial products and applications.

### Methods

This section discusses one of the most common issues in RVA analysis sample preparation. Because one of the most useful applications for RVA is on-line analysis of high moisture dough and half-products, this topic merits more in-depth discussion. It is meant as a guide or starting point in assessing any multi-unit operation. The methods are applications of those of Paton and Spratt (6, 10, 11) and/or observations of the work of Whalen et al. (13).



### **Samples and Sample Collection**

Samples must be obtained from unit operations which have achieved steady state. Extrusion samples and collection should be made immediately exiting the unit operation and rapidly cooled or frozen. Dry pellet product need only be air cooled. These procedures stem from their logical treatment at the unit operations. Cooked product should be snap frozen with liquid nitrogen or frozen with dry ice (6, 11). The first requirement in handling dough or extruded ropes is to preserve the structure of the product exiting the cooking unit to assess that unit's effect on the product. Rapid freezing drops the energy level and, for all practical purposes, locks the structure of the material as it results from the cooker. Secondly, freezing allows handling and milling of the samples in preparation for analysis. All high moisture product must be stored frozen (0°C or lower).

Pellets resulting from a dryer are likely to condense if snap frozen and are usually in a rubbery state when hot. If assessing a dryer effect via RVA then the product should be snap frozen. If assessing the end product of a dryer, then air cooling is more appropriate to avoid artefacts as a result of condensation. Obviously, simple comparisons can be made within any system to observe the effects and select the method.

Direct expanded products and low moisture extrudates (<10%) are generally stable in the short-term sample interval (minutes to 1 hour). They rapidly set their structure off the end of the extruder (or puffing device) and are representative of the unit operation without snap freezing. The product should still be stored frozen after cooling.

Products off the grocery shelf should be considered stable but depend on the category (i.e. snacks with high fat have a shorter shelf-life). Representative sampling can be done by replication, purchasing regionally,

nationally or internationally as desired. Storage at freezing is best if a product cannot be analysed within a reasonable time after purchase.

### **Sample Preparation**

Raw materials can be assessed as received and therefore be representative of how the cook process sees the material, or milled through a standard lab mill to obtain a comparative sample and eliminate variation between suppliers. All materials which are the product of the cook system need to be reduced in size for RVA analysis. Product should be first reduced to < 1 x 1 cm by placing in liquid nitrogen, holding for several minutes and shattering by striking with a suitable pestle (i.e. steel). Snap frozen dough and high moisture extrudate product will readily mill in devices such as a Waring pulverizer (model SS 110). Paton and Spratt used a coffee grinder (6, 11). Any high speed blade-type milling device is suitable for high moisture products and pellets. It is also desirable that the mill be constructed out of steel or otherwise be capable of holding a small quantity of liquid nitrogen or dry ice during milling. This serves two purposes, the first of which is to maintain the frozen state of the sample and fracturability. The second is to insure that no thermal degradation of the starch occurs during milling which may introduce artefacts in the RVA analysis.

Some dry pellets and other half-products samples (< 10% moisture) may be milled in standard lab mills such as the Cyclotech and Udy type. However, impeller/abrasion mills like these should not have screens that are too small (i.e. 0.5 mm) due to increased residence time which may cause further thermal degradation. Otherwise, pellet products can be milled in the blade-type pulverizers as above.

All samples should be sieved to obtain a uniform distribution of material for RVA analysis. Size is relative to

homogeneity in that it is desirable to mill a representative amount of material to pass a screen size of at least 35 US mesh or 500 micron (10). Generally, the majority of sample (50 to 75%) should pass the screen. Practical screen sizes for high moisture samples are from 35 down to 60 US mesh (250 micron). A screen size should be selected which meets the needs of the samples to be processed. It should not affect the sample composition (i.e. separate out fiber or other components). Frozen samples should be processed using a cold frozen sieve to avoid condensation. Preparation procedures should be flexible and explore different sieve sizes for different products as needed. However, the same sized sieve should be used for any comparative analyses between samples. The primary effect of particle size in the RVA is a lower profile for the coarser sieve.

High fat samples should be crushed and defatted via hexane extraction. The resulting material may then be ground, sieved and analysed via RVA. Some high fat (>15%) samples can be sufficiently ground but will blind the screen in the sieve step.

### **Sample Size**

The RVA-4 offers more options that affect the measurable viscosity range (motor size and variable rpm). However, sample size for an extruded product is usually between 3 to 5 g on a dry weight basis. The size is dependent upon sensitivity. Depending upon the product, certain features of the RVA profile will be more revealing and therefore the focus of analysis. An example is the cold viscosity region of a direct expanded product. One may only be interested in the cold viscosity and paste region of the temperature ramp up to 95°C for such a product. As one experiments with sample size it will become apparent that some profile features will be highlighted or masked based on sample size. Initially, the RVA motor size determined what the



effective operating range was based simply on maximum viscosity.

**RVA Profile**

The temperature program or Thermocline parameters can be manipulated to fit the type of sample being analysed. The most useful or all-purpose program is the long standing 25°C to 95°C to 25°C profile historically used in the Brabender and Ottawa machines. For the RVA the following is a comparable profile: a 2 min. hold at 25°C followed by a target temperature of 95°C at 7 min., holding at 95°C to 10 min. and cooling back to 25°C at 15 min. and holding to complete the profile in 18 to 22 min. as desired. The hold time at 95°C may be increased based on observation of the sample trough relative to the setback. The setback value is useful in evaluating certain samples and requires full cook out at the 95°C hold or results will vary.

If a sample is likely to have a cold viscosity as a result of its cook history then a 25°C hold time is desired. If the sample is known to be of a 'raw' or uncooked nature such as traditional cracker, pretzel, tortillas, etc. then a 50°C initial hold temperature may be employed. The setback may also be increased to 50°C for such raw samples if it is apparent that it will be quite high.

**Other**

Virtually all of the sample manipulations that are routinely used in viscosity measurements can be used with samples for RVA (14). For example, some direct expanded products are highly hygroscopic and cause lumping in the canister. Wetting the sample with ethyl or isopropyl alcohol can be done, simply be aware that the profile will change due to the solvent and you may have to lower the peak paste temperature setting below 95°C. Likewise, hydration of modified starches may be assisted by adding a small amount of fine granulated sucrose to the starch sample to avoid 'fish-eyes'.

Another means of aiding dispersion in cooked samples is shaking the canister vigorously prior to mounting in the tower. The order of addition to the canister is water first, followed by the sample, capping with an inverted No. 8 rubber stopper or any suitable equivalent (rubber mat) and shaking vigorously for 5 to 10 seconds. The cap is scraped clean in a sliding motion upon removal. The walls of the canister are then scraped down using the spindle blade. The procedure can be repeated if material fails to be incorporated in the mix. A routine should be established and comparisons made between operators since this can account for minor variations in the cold viscosity. We have noted no significant differences due to shaking.

**Examples**

Generally, three different pathways of starch degradation are readily obvious from simple experiments with grain cooking pasting, mechanical, and thermal degradation. The properties of pasting are apparent by repeating an RVA on a raw grainlike rice or oats and comparing the cooked sample to the uncooked sample (Figure 1). The effects of mechanical degradation are most apparent in low moisture, direct expanded products. In the RVA profile, high shear direct expansion shows the characteristic cold viscosity and low setback. There is no question that the energy input in such situations is due to the mechanical effect of the screw and/or die. Thermal degradation can be shown in a high moisture extrusion cook producing a dough and then raising or lowering the barrel temperature (Figure 2). Thermal degradation lowers the entire profile in this situation.

Production of key elements in a cook can be shown by following the progressive cook pattern formed in products spanning the cook conditions between that for a high moisture dough to a direct expanded puff product. This condition was created by controlling the water addition rate from 29 to 15% in a twin screw extruder on the same formulation. Figure 3 shows the

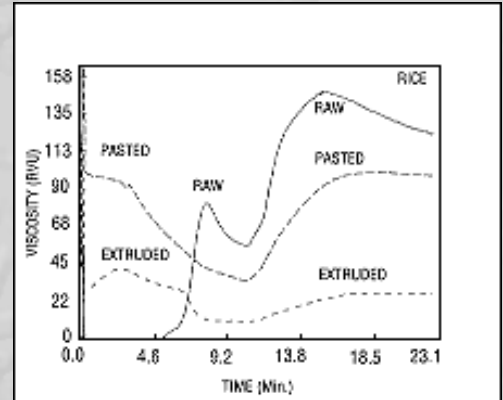


Figure 1.

Comparison of RVA profiles for different types of cook processes (rice).

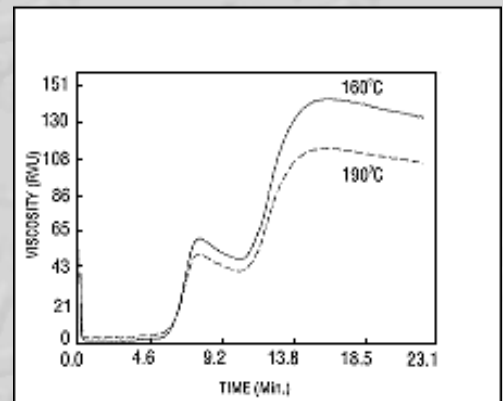


Figure 2.

RVA profile of thermal degradation pattern in corn-based extrudates.

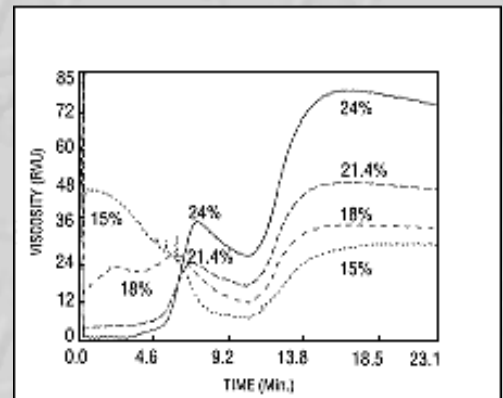


Figure 3.

Progressive cook via decreasing water addition in a twin screw extruder at 24 - 15% total moisture in a corn-based formulation.



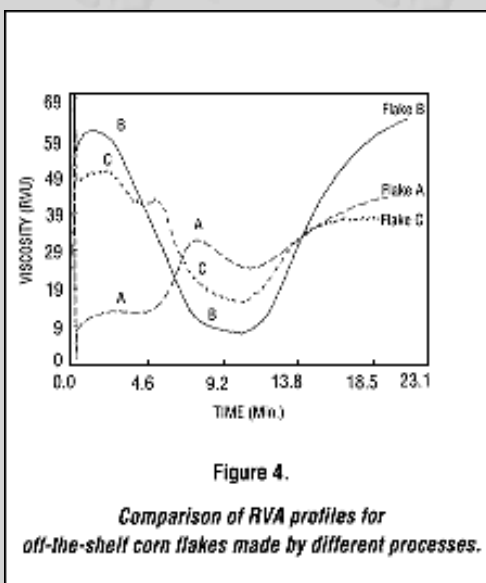


Figure 4.

Comparison of RVA profiles for off-the-shelf corn flakes made by different processes.

progressive shift as a linear decrease in setback, accompanied by an increase in cold viscosity. Intermediate peaks are apparent in the ramping region between 2 and 10 min. on the profile. The RVA captured the degradation pattern and was far more sensitive to changes in energy input than system responses such as torque and amperage.

Water plays a key role in the cook and production of cereal products. Add to this the interaction of all three types of pathways occurring to some extent, depending upon the process, and an extremely interesting combination results. These are evident in the products preferred on the grocer's shelf. RVA profiles reflect the main cook process since this fundamentally affects the starch pattern. Thus, products that are cooked in a manner which preserves raw structure show this by reflecting raw features in the profile. These are usually evident by peaks that occur in the temperature ramping area of the profile from 25°C to 95°C. This holds true for direct expanded products as well which simply need more focused detail in the early part of the profile associated with cold viscosity. A classic example of this is a comparison of corn flakes made by three different processes. Figure 4 compares the leading US brand, Product A, a batch cook/corn grit process with two products made by extrusion dough cook/pellet

process. Both Products B and C show that they were produced by high shear extrusion cooks due to the high cold viscosity but differ in the downstream treatment. Product C was a poor quality product while Product B was a close match to the traditional Product A.

Clearly there are other effects represented in an RVA profile of a product taken off the shelf but many of these are known features of the product. For example, one expects the flake rolls to impact the grit or pellet and change its absorption of water in the profile. Similarly one can anticipate the requirement, of some puffing systems such as fluid bed, gun puffing, or frying and observe these effects within this context. A far more powerful tool is to combine the RVA with known inputs as in the twin screw experiment and compare characteristics of the final product. Extending this work to that of sequentially following effects in a multi-unit operation allows mapping and even predictive use of RVA profiles for product development, formulation, and production. As a result of increased sensitivity to both process and ingredient changes, key sensory characteristics can be associated with degree of cook levels well before the final product. Combined with high reproducibility and quantitative analysis, the RVA offers a unique resource to the extrusion industry.

### References

- Mercier, C., Linko, P., and Harper, J.M. 1989. Extrusion cooking. American Association of Cereal Chemists.
- Colonna, P., Tayeb, J., and Mercier, C. 1989. Extrusion cooking of starch and starchy products. In Extrusion cooking. Eds. C. Mercier, P. Linko, and J.M. Harper. American Association of Cereal Chemists.
- Mason, W.R., and Hosney, R.C., 1986. Factors affecting the viscosity of extrusion-cooked wheat starch. Cereal Chem. 63(5):436.
- Jackson, S.S., Gomez, M. H., Waniska, R. D., and Rooney, L.W. 1990. Effects of single-screw extrusion cooking on starch as measured by aqueous high-performance size-exclusion chromatography. Cereal Chem. 67(6):529.
- Chinnaswamy, R., Hanna, M.A., and Zobel, H.F. 1989. Microstructural, physicochemical, and macromolecular changes in extrusion-cooked and retrograded corn starch. Cereal Foods World. 34(5):415,
- Paton, D., and Spratt, W.A. 1981. Simulated approach to the estimation of degree of cooking of an extruded cereal product. Cereal Chem. 58(3):216.
- Van Lengerich, B. 1989. Influence of extrusion processing on in-line rheological behavior, structure, and function of wheat starch. In Dough rheology and baked product texture, H. Faridi, and J.M. Faubion, Eds. Van Nostrand Reinhold, New York.
- Bhattacharya, M., Padmanabhan, M., and Seethamraju, K. 1994. Uniaxial extensional viscosity during extrusion cooking from entrance pressure drop method. J. Food Sci. 59(1 )
- Guy, R.C.E., and Horne, A.W. 1988. Extrusion and co-extrusion of cereals. In Food Structure: Its Creation and Evaluation. J.M.V. Blanshard and J.R. Mitchell, Eds. Butterworths, London. P. 331-349.
- Paton, D. and Spratt, W.A. 1978. Component interactions in the extrusion cooking process. 1. Processing of chlorinated and untreated soft wheat flour. Cereal Chem. 55(6):973-980.
- Paton, D. and Spratt, W.A. 1984. Component interactions in the extrusion cooking process: Influence of process conditions on functional viscosity of the wheat flour system. J. Food Sci. 49:1380-1385.
- Wrigley, C.W., Booth, R.I., Bason, M.L., and Walker, C.E. 1996. Rapid Visco Analyser: progress from concept to adoption. Cereal Foods World. 41(1):6.
- Whalen, P.J., Bason, M. L., Booth, R. I., Walker, C. E., and Williams, P. J. 1996. Measurement of extrusion effects by viscosity profile using the Rapid Visco Analyser. (Submitted for publication).
- Shuey, W.C. and K.H. Tipple. 1980. The Amyograph Handbook. American Association of Cereal Chemists.



# RAPID VISCO APPLICATIONS: Starch Based Custard Powders

**Natalie Turner,**  
*Research Scientist, Newport Scientific.*

Viscosity is an important determinant of custard quality. Two commercial samples of starch based custard powders were cooked on a hot plate according to their packet directions. Viscosity of Brand 1 was three times higher than that of Brand 2. The same brands were then tested in the RVA using milk and the following profile at the recommended concentrations (28.0 g total weight). Brand 1 gave a final viscosity about twice that of Brand 2 (Figure 1), reflecting the trend in the hot plate prepared samples.

#### Temperature Profile:

50°C	<b>Idle Temp.</b>
50°C	1:00 min.
95°C	4:45 min.
95°C	9:45 min.
30°C	15:30 min.
<b>End test</b>	20:00 min.

In a series of further tests, custard powder was added to milk and to water at a range of concentrations from one to three times the recommended level. Although custard viscosity in water was lower, good correlations were found between the water and milk viscosities for Brand 1 ( $r=0.98$ ) and for Brand 2 ( $r=0.99$ ). This indicated that water-based tests (at higher concentrations) could be used to predict viscosity results in milk for a given brand.

The RVA method developed reflects differences in custard viscosity between brands prepared by conventional methods. A milk-based test can be used in the RVA, or a water-based test where it is desirable to avoid variation due to milk quality issues. The method could be used in production of custard powders to measure and control cooked viscosity between batches, assuring end user consistency.

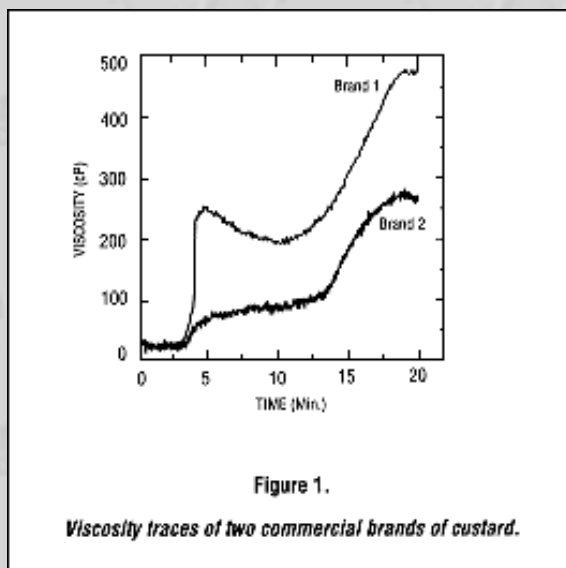
**Meet the People:**  
**Bronwyn Elliott**



Bronwyn is Newport Scientific's newest member of staff, joining the team in June as RVA International Product Manager. Now that the RVA is exported to more than 20 countries and the number of agents continues to grow, there's an increasing need for this role.

With a chemistry degree from the University of Sydney and a Master of Management from Sydney's Macquarie University, Bronwyn also brings 15 years experience in sales - most of which have been involved with scientific equipment. She has spent the last five years working with the RVA's local Australian sales agents, Radiometer Pacific (formerly Foss Electric), and so she has an understanding of both the needs of our RVA agents and our RVA users in research and industry.

Bronwyn's appointment is a recognition that Newport Scientific's reputation depends on the quality and performance of its products as well as the excellence of our customer service. She joins Mark Bason, our Senior Research Scientist, to provide the level of support that both our agents and our customers require.



Newport Scientific P/L  
Unit 1, 2 Apollo Street  
Warriewood NSW 2102 Australia  
Tel: +61 (02) 9979 6992  
Fax: +61 (02) 9979 6993  
Email: [support@newport.com.au](mailto:support@newport.com.au)

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