1. At the end of the hearings held on 29 September 2009 Judge Bennouna asked the following question:

What process and products are used by the Botnia plant for its cleaning? Uruguay was asked to provide a reply by 9 October 2009, and Argentina was provided with an opportunity to respond by 19 October 2009.

- 2. Uruguay provided an oral response during the hearings on 2 October 2009 (CR 2009/23, pp. 14-15, paras. 6-8 (Mr. Reichler), and made available a copy of an affidavit dated 30 September 2009, as written by Mr Gervasio Gonzalez Simeonoff. By letter dated 8 October 2009 Uruguay merely reaffirmed the response it had given on 2 October 2009, without adding anything new.
- 3. Argentina is pleased to have an opportunity to respond to the new evidence provided by Uruguay. As set out below, Argentina notes that Uruguay has provided an incomplete response to the question posed by Judge Bennouna, and submits that the evidence before the Court points decisively to the fact that the Botnia plant has used nonylphenols that are banned in the pulp and paper industry in the European Union. Before addressing the statement of Mr. Gonzalez, it is appropriate to place it in the context in which it was prepared.
- 4. Argentina raised the issue of nonylphenols in its new written documents submitted on 30 June 2009 after monitoring the Uruguay River for more than a year.¹ Specifically, this monitoring identified the presence of elevated levels of nonylphenols in the waters, clams, in the algal bloom of February 2009 and in sediments around the Botnia plant, as well as the presence of nonylphenols in pulp samples from the plant which matched the nonylphenols identified elsewhere.² This monitoring and analysis points directly and unambiguously to the use of nonylphenols at the Botnia plant. Uruguay has had ample opportunity

¹ New Documents Submitted by Argentina on 30 June 2009, Chapter 3: Uruguay River Environmental Surveillance Program Biogeochemical Studies, in particular Executive Summary at p. 4 and sections 3.4.1 and 3.5.

² Ibidem

to respond to the evidence submitted by Argentina. It could have introduced its own evidence but it has not done so. Consequently, the unchallenged evidence before the Court shows that (1) pulp produced at the Botnia plant is contaminated by the presence of significant levels of nonylphenols, and (2) the waters, clams, the algal bloom and the sediments in the immediate vicinity of the plant's effluent discharge pipe show significantly elevated levels of nonylphenols as compared with samples taken in other places on the river. Both factors point conclusively to the use of nonylphenols ethoxylates (NPEs) at the plant, the presence of which is reflected in degraded nonylphenols in pulp, waters, clams, algal blooms and sediments.

5. Against this background, instead of addressing the issue by way of testing or monitoring by its competent authorities, Uruguay has served two affidavits that are part of the evidence before the Court and made inconsistent arguments by way of submission that go beyond what its own evidence will support. This has been fully addressed by Argentina during the hearings,³ and need not here be repeated. On 15 July 2009 Uruguay served a first affidavit by Ing. Alicia Torres.⁴ Ing. Torres has no apparent expertise on the subject of the production of pulp, including issues of cleaning, or on nonylphenols. Her affidavit raised more questions than it answered, and was conspicuous by its failure to address the cleaning of the eucalyptus wood chips or the cleaning of the plant itself, the process in which nonylphenols could be expected to be used. Despite Ing. Torres affidavit, and an affidavit dated 20 September 2009 by Mr Gonzalez (which was referred to in part in a letter submitted by Uruguay to the Court on 28 September 2009 but which Argentina has not seen in full), on 22 September 2009 counsel for Uruguay told the Court that although Uruguay was "convinced" that there "is" no use of nonylphenols - he did not say there "has never been" any use of nonylphenols - if the plant was using nonylphenols then Uruguay would put a stop to it (CR 2009/17, p. 24, para. 38 (Mr. Reichler). It was readily apparent

³ See CR 2009/12 14 September 2009, p. 59, para. 24 (Prof Wheater); CR 2009/14 16 September 2009, pp. 45-47, paras. 15-17 and 18-23, and p. 51, paras. 26-28 (Prof Colombo); CR 2009/15 17 September 2009, pp. 16-18, paras. 12-16 (Prof Sands) and p. 24, para. 7 (Prof Wheater); CR 2009/20 28 September 2009, pp. 50-51, paras. 21-23 (Prof Colombo);, CR 2009/21 29 September 2009, p. 21, para. 32 (Prof Sands).

⁴ Affidavit of Agr. Eng. Alicia Torres, Director of DINAMA, 13 July 2009, Uruguay's Comments on Argentina's New Documents, 15 July 2009, Annex C24.

that the state of knowledge and certainty of Uruguay as to what was being discharged into the river was less than complete.⁵

- 6. On 2 October 2009, nearly three months after this issue was first raised by Argentina to the Court in its 30th June 2009 Report, Uruguay revisited the issue for a third time, submitting a further affidavit dated 30 September 2009 and written also by Mr Gonzalez. Mr Gonzalez works for Botnia and therefore lacks independence. Once again, the affidavit is incomplete, in failing to address all aspects of the cleaning of the eucalyptus wood chips. It is ambiguous and contains errors of fact that suggest that its author lacks technical expertise.
- 7. Argentina has obtained a report on Mr Gonzalez's affidavit, by providing a copy to Dr Bruce Sithole, an expert on the use of nonylphenols in the pulp and paper industry. As can be seen from his curriculum vitae, which is attached to his report, Dr Sithole worked for 22 years at FPInnovations Paprican (the Pulp and Paper Research Institute of Canada), where he was a Principal Scientist and Group Leader. He has prepared a detailed report dated 16 October 2009 on the use of nonylphenols in pulp mills in general and at the Botnia Mill, a copy of which is attached. His report sets out *inter alia* the context and processes by which it is necessary to remove lipophilic extractives from the hardwood eucalyptus chips in order to produce high quality and commercially valuable pulp. Those processes constitute a cleaning process, and relate to matters that are addressed by Judge Bennouna's question. Dr Sithole states at paragraph 31 that

the mere addition of defoamer (even one incorrectly described as a 'surfactant' by Mr. Gonzalez in his affidavit) in the washing process will not be sufficient to achieve pulp of the quality which is produced at the

⁵ Argentina notes that the subject of nonylphenols is not the only one on which Uruguay's submissions and evidence are incomplete. Uruguay has challenged Argentina's interpretation of the word 'oxidabilidad' as used in OSE monitoring data for the period from 19 April 2007 to 13 May 2009 (CR 2009/23, pp. 17-19, paras. 12-17, referring to Argentina's arguments made at CR 2009/21, p. 29, para. 33). Uruguay's preferred interpretation is that 'oxidabilidad' means 'oxides' (CR 2009/23, p. 18, para. 15), yet the term 'oxides' is not one that has meaning in this context and is not usually associated with the measurement of water quality. In any event, the evidence before the Court indicates serious problems with biochemical oxygen demand (BOD) and shows that "continued recordings of the buoys confirmed recurrent oxygen deficits in the Uruguay River relative to the Bay which presented more oxic conditions": New Documents submitted by Argentina on 30 June 2009, Volume 1, Scientific and Technical Report, Chapter 3, Executive Summary, p. 2, second paragraph, and Section 3.2.3. This evidence has not been challenged by Uruguay.

mill. The very fact that a defoamer is required, indicates to me that a surfactant is indeed used in the washing process.

Dr Sithole concludes at paragraph 23 of his report that, contrary to the claim made by Uruguay during the hearings (CR 2009/23, p. 14), a 2008 AMEC report (included at Annex 48 of Uruguay's Reply) does not specifically describe the principal cleaning chemicals used by the Fray Bentos mill. At paragraph 34 of his report, Dr Sithole concludes that Mr Gonzalez's affidavit of 30 September 2009

does not provide a complete answer to the question and does not fully describe the "process and products ... used by the Botnia plant for its cleaning".

His report further concludes at paragraph 33 that a number of factors point strongly to the use of nonylphenols at the plant:

I am not able to express an absolutely definitive view. However, there are a number of factors that are significant: the eucalyptus hardwood used to produce the pulp at the plant; the absence of easily available alternatives to clean the eucalyptus or its wood chips to remove lipophilic material; the issues of cost, taking into account the type of pulp produced at the mill; the repeated failure on the part of Uruguay and Botnia to provide detailed information on the cleaning processes used at the Botnia plant or the compounds that are used; the strong evidence of NPEs in higher concentrations in the waters into which the plant's effluent discharges are made; the evidence of NPEs in sample of sediments, clams, algal blooms; and the evidence of NPEs in samples of pulp produced by the mill. These factors point strongly towards the clear conclusion that NPEs have been used at the Botnia plant in one or more cleaning processes associated with the production of wood pulp. In the light of my professional experience, I would be very surprised if NPEs had not been used. If I were asked to quantify my level of certainty that NPEs had been used, I would put it at 95%.

8. Argentina adopts Dr Sithole's conclusions. These conclusions will not have come as a surprise to Uruguay. Uruguay was familiar with the issues he has raised, and had ample opportunity to address them by way of detailed evidence, including by way of complete affidavit. Argentina submits that the fact that Uruguay has not addressed the issues raised by Dr Sithole, has not provided full information as to the surfactants that are used to remove the lipophilic extractives, and has not provided any evidence as to its own testing for the presence of nonylphenols, points conclusively to the use of nonylphenols ethoxylates (NPEs) by the Botnia plant.

9. Uruguay accepts the dangers posed by nonylphenols, including the irreversible damage that they may cause to plant, animal and human life. In the circumstances, this is exactly the kind of issue for which the principles of prevention and precaution play an important role. Argentina submits that the evidence before the Court indicates that the burden is on Uruguay to establish conclusively that it does not use, and has not used, nonylphenols. It has not met that burden. Specifically, in response to Judge Bennouna's question, by failing to provide a complete account of the "process and products ... used by the Botnia plant for its cleaning" Uruguay's actions require the Court to conclude that the evidence before the Court points compellingly to the use of nonylphenols ethoxylates (NPEs) at the plant, in particular for the purpose of removing lipophilic extractives from the eucalyptus hardwood chips that are used at the plant.

Buenos Aires, 19 October 2009

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Ambassador Susana Ruiz Cerutti Agent of the Argentine Republic

Report into: (1) the Use of Nonylphenols in Pulp Mills in General and (2) at the Botnia Mill, Uruguay

Dr B Sithole, Quebec, 16 October 2009

Personal background and professional experience

- 1. I am a research chemist and I hold a Ph.D. in Environmental Analytical Chemistry from the Trace Analysis Research Center, Department of Chemistry, Dalhousie University, Halifax, Nova Scotia, Canada, 1983. As indicated in my Curriculum Vitae, which is attached as Annex 1, my research experience includes extensive analysis of *nonylphenol ethoxylate surfactants* ('NPEs') as used in pulp and paper samples and for the removal of lipophilic extractives in pulp and paper making processes.
- 2. From 1987 to 2009, I was employed at FPInnovations –Paprican (formerly the Pulp and Paper Research Institute of Canada), Quebec, where I was a Principal Scientist and Group Leader. My work at Paprican involved methods development and analysis of phytosterols, wood extractives, deposits and additives in pulp and paper matrices. The developed methods are used to solve production problems, assess environmental pollution, improve mill production, and for reverse-engineering studies to help in new product development. The analytical techniques used include LC-MS, GC/MS, pyrolysis-GC/MS, FTIR, HPLC, GPC, SPE and ion chromatography. I was also involved in troubleshooting environmental and wet-end issues in pulp and paper mills.
- 3. My publications on NPEs include the following (see further the full list as set out at Annex 1 to this report):

B.B. Sitholé and L.H. Allen, 1989. Determination of nonionic nonylphenol ethoxylate surfactants in pulp and paper mill process samples by spectrophotometry and HPLC, *J. ASSOC. OFF. ANAL. CHEM.*, 72, 273-276.

B.B. Sitholé, B. Zvilichovsky, C. Lapointe and L.H. Allen, 1990. Adsorption of nonylphenol ethoxylate surfactants on metal surfaces: effect on quantitation by liquid chromatography, *J. ASSOC. OFF. ANAL. CHEM.*, *73(2), 322-324*.

B.B. Sitholé and E.J. Pimentel, 2009. Determination of nonylphenol and nonylphenol ethoxylates in pulp samples by Py-GC/MS, *J. ANAL. APPL. PYROLYSIS*, 85(1-2): 465-469.

Consultation report: Deresination of aspen in sulphite dissolving pulps: comparison of NPE and non-NPE surfactants, *FPInnovations Confidential Internal Report*, 2008.

4. My professional memberships and related activities are set out in full in my Curriculum Vitae at Annex 1. In 1997 I was elected Fellow of the Chemical Institute of Canada. I was the Chair of the Fine and Coated Papers Committee, Pulp & Paper Technical Association of Canada, from 1998-2002. In 2002, I was awarded a Certificate of Appreciation by the Pulp & Paper Technical Association of Canada, 2002, Such certificates are given "To recognise individuals who have provided exceptional services to PAPTAC and/or the Canadian pulp and paper industry".

Terms of Reference

- 5. I have been asked by the Government of Argentina to prepare an independent report, on the basis of materials provided to me or which I obtained myself, on the following issues:
 - 1. The use of NPEs in pulp mills;

2. Whether it is possible or likely that NPEs are being used at the Botnia plant;

3. The information that would be needed to reach a definitive conclusion on the use of NPEs at the Botnia plant.

In addressing these issues, I have been asked to respond to the question posed to Uruguay by Judge Bennouna during the hearing, taking into account also the response given by Uruguay to the question, during the hearing and on 9 October 2009. The question asked by Judge Bennouna was:

"What process and products are used by the Botnia plant for its cleaning?"

I understand Judge Bennouna's question to address the cleaning of the plant itself and the eucalyptus wood chips, which process would include the removal of lipophilic extracts.

Materials Considered in the Preparation of this Report

- 6. In preparing this report I have drawn on my professional experience and expertise in the field. In addition, the materials which I have used in preparing this report include:
 - (i) A copy of the 'Rio Uruguay Environmental Surveillance Program (Chapter III, Biogeochemical Studies - November/08-April/09)' report, pages 1 through 42 provided by Argentina".
 - (ii) An English translation of the Affidavit of Agr. Eng. Alicia Torres, director of the National Environmental agency, provided by Argentina.
 - (iii) A technical report attached to the Affidavit of Ing. Torres: Nonylphenol and its Ethoxylates in Environmental matrices. Case study Uruguay. Soledad Andrade B.S., Sandra Castro Scarone, B.S., Natalia Barboza, B.S., May 2008, produced by DINAMA, provided by Argentina ("DINAMA Report").

- (iv)A copy of Tables 1-5 indicating individual Nonylphenol (NP1 to NP6) and total concentrations in settling particles, water samples, pulp and technical product, entitled 'Data_Table_Nonylphenols [1], provided by Argentina.
- (v) A case study from the USA entitled 'Pulp and Paper industry Voluntarily Reduces Use of Nonylphenol Ethoxylates', available at <u>http://www.deq.state.mi.us/documents/deq-ead-p2-p5-npe.pdf</u>
- (vi)A fact sheet on the pulp mill project published by Botnia available at

http://w3.upmkymmene.com/upm/internet/cms/upmmma.nsf/lupgraphics/Botnia%20Uruguay%20fact%20sheet_ ENG.pdf/\$file/Botnia%20Uruguay%20fact%20sheet_ENG.pdf

- (vii) A press release available at <u>http://w3.upm-</u> kymmene.com/upm/internet/cms/upmcms.nsf/pkv/UPM_and_Botnia's_Fray_Bent os_pulp_mill_in_Uruguay?OpenDocument
- (viii) The current published price for Botnia's FRB EUCA (effective 1 October 2009) source: <u>http://www.botnia.com/en/default.asp?path=204,210,211,2672,3058</u>
- (ix) Arguments made by Counsel for Uruguay in the course of the hearing held at the International Court of Justice on Tuesday 22 September 2009 (CR 2009/17) (in particular at paragraphs 22 to 28 on pages 8-9).
- (x) A September 2007 AMEC Report, included as Annex 48 to Uruguay's Reply in the court proceedings.
- (xi) A copy of the affidavit of Mr. Gervasio Gonzalez, Environmental Manager at the Fray Bentos pulp mill, dated 30 September 2009, together with annexed attachments.

Other material which I would have liked to have seen

7. It would have been helpful in preparing this report to have had access to other material. Specifically, it would be very useful to be provided with a document setting out in detail the list of all additives used at the Botnia plant, together with their MSDS information. This information would allow me to understand the composition of all

the chemical formulations used at the plant. For example, a case study conducted in Michigan, USA, in 2000, found that of the 780 process chemicals used by 17 mills in the state,

- 604 products were NPE-free;
- 60 products were identified by their vendors as containing NPE (at 1-6% levels);
- 31 products were no longer sold by vendors;
- 85 products were identified as having unknown NPE content.

The vendors were pressured to change the 60 NPE-laden products and the results were:

- 28 products were re-formulated to NPE-free products
- 2 products did not have the potential for waste water discharge, and so were not converted
- 6 products were no longer sold by vendors
- 24 products could not be re-formulated

If such a case study had been conducted in Uruguay I am certain that the results would demonstrate that many additives used in Uruguay also contain NPEs. Thus it is, in my view, very likely that a significant number of products sold by vendors in Uruguay to the pulp and paper industry will contain NPEs, considering that the sale of such products is not banned in the country.

8. The DINAMA Report to which I have been referred (paragraph 6 above, document 3), shows that significant amounts of products having NPE concentrations are used in Uruguay and would be likely to result in industrial discharges. It is striking, however, that the industries referred to in the report do not include the pulp and paper industry, and that no reference is made to data on pulp and paper mill effluents, which I understand to be a major industry in Uruguay. This raises the question, to my mind, as to why this industry was not included in the study?

9. The DINAMA Report shows that NPEs are widely used in Uruguay, including in the Raw wool contains lipophilic materials that impart undesirable wool industry. properties to the final products and thus need to be removed. Lipophilic materials are natural fats and fatty compounds that are present in animals and plants and are soluble in organic solvents. These materials are also present in wood and need to be removed as they interfere with the pulp and papermaking process, since they impart poor quality to the final product. The more lipophilic material there is in the pulp and paper, the lower is its quality and hence its commercial value. There are therefore good financial reasons for removing lipophilic materials from pulp. Estimates are that the North American industry spends \$750 million annually to tackle problems caused by lipophilic extractives. NPE cleaning agents are cheap and very effective in the removal of these lipophilic materials, and that is why they have been widely used in the pulp and paper industry throughout the world. In my opinion it is very probable that Uruguayan vendors of NPE-containing cleaning agents to the wool industry in Uruguay would also be in a position to sell the same products to the pulp and paper industry, since they could be used in that industry. They would be relatively cheap and easily available.

Description of the process, and the point at which, NPEs would be introduced

- 10. The terminologies used with respect to NPEs may be confusing and warrant a brief explanation as some people incorrectly use them interchangeably. Nonylphenol ethoxylates (NPEs) are ethoxylated products of nonylphenol (NP) that is, they are manufactured from NP. They are the major components in the NPE formulations sold for industrial use. However, NPEs degrade in the environment to form NP which is very toxic to aquatic biota.
- 11. There are many unit operations or stages in the manufacture of pulp. From the materials to which I have been referred, and in particular the Affidavit of Ing Torres, she seems to focus on just two of the unit operations, namely production (which would be the cooking process) and bleaching. Her affidavit does not refer expressly

to pulp washing operations (where the pulp has already been produced) or to cleaning operations (where dirt and contaminants are removed from the pulp), which are the processes in which NPE are typically used. Also, in many instances, high amounts of extractives and lipophilic extractives lead to what we call deposition problems on mill machinery. Fully bleached kraft pulp like the one produced at the Botnia plant is sold on the basis of kappa number and lipophilic extractives content. Kappa number is an indication of the residual lignin in the pulp that affects the whiteness of the pulp. The lipophilic extractives threshold values are around 0.03% using dichloromethane as the extraction solvent. Final pulps that have high extractives values will cause severe problems for customers when they convert the pulp to final products such as photocopy paper. The problems include sticky deposits on paper machines, unacceptable odours when the paper is used on photocopiers (the fats in the extractives become rancid and emit objectionable smells), slippage on the paper on copier machines (paper sticking in and jamming copier machines). The deposits that are caused by the extractives require cleaning with alkali and surfactants, and NPEs may also be present in these industrial cleaners. NPEs may be present in products used to clean the plant itself. Some mills add surfactants in the digester - during the cooking stage to solubilise the problematic lipophilic extractives but not where eucalyptus is used for pulp production because of cost issue.

- 12. An extensive series of unit operations are utilised in the production of fully bleached pulp, e.g., debarking, chipping, cooking, brownstock washing, pressing, bleaching, etc. These unit operations function to produce/convert wood into pulp. Cleaning operations, such as boil outs, serve to clean the system and ensure the production of good quality pulp. They are not directly involved in the conversion of wood into pulp.
- 13. In Kraft pulping, the stage of the process at which NPEs are introduced is in the washing of the pulp after cooking (high amounts will impair the bleaching process and cause sticky deposits on mill equipment) and after bleaching to remove extractives that have been affected by the bleaching process. I would have expected Ing. Torres to have mentioned this in her Affidavit. They can also be introduced in

pulp storage tanks where surfactants (dispersing agents) are added to disperse the lipophilic extractives thereby preventing them from agglomerating to form the sticky deposits. Ing. Torres does mention the addition of dispersing agents but gives no indication on the chemistry of the surfactants used. NPEs can be effective dispersing agents at dosages similar to those mentioned by Ing. Torres.

14. In Kraft pulping, as practiced at the Botnia plant, the produced brown pulp is washed in brownstock operations to remove lignin and the lipophilic materials mentioned earlier Surfactants are used at this stage as they solubilise the lignin and lipophilic extractives. The surfactants function just like soap that is used to wash out dirt in laundry. Alkali washing alone can be effective in softwood pulps because the components in their lipophilic are easily solubilised and washed out. However, the components in eucalyptus and other hardwoods are not easily solubilised by alkali and thus remain with the pulp. Addition of NPEs to the wash water is effective in solubilising and removing these components. Extensive studies have shown that surfactants that do not contain NPEs are not effective in removing the problematic components in hardwood extractives. The washed brown pulp is then bleached in the bleaching operations to remove residual lignin and turn the fibres white. A series of washing operations are used including some that operate in alkaline conditions, where surfactants are also used to effect removal of residual lipophilic extractives. The lipophilic materials are effectively removed under alkaline conditions and in the presence of surfactants. If the lipophilic materials are not removed, they can build up and accumulate on mill equipments, e.g., even blocking pipes in serious cases. Mills that use hardwoods – such as eucalyptus - need to periodically shut down and clean the whole process to remove the lipophilic materials, a process termed "boil-out". This entails filling the lines with a series of cleaning agents that include alkaline and surfactants at elevated temperatures. Here again NPEs have been the traditional choice, due to their effectiveness and low cost. However, when the cleaning chemicals are discharged into the mill effluents, there is a dramatic rise in the concentration of NPEs in the receiving waters. Surfactants containing NPEs are also sometimes added as lubricants on the pulp machine.

Type of NPEs used: the difficulties of using hardwood (eucalyptus) and why operators of such mills are more likely to need NPEs.

15. Pulps made from softwoods are easy to clean with alkali and surfactants, in general due to the composition of the lipophilic materials that is easy to solubilise. The lipophilic components in hardwoods, on the other hand, are recalcitrant and difficult to remove. It has been ascertained that NPEs are very effective in the removal of these lipophilic materials. I have done extensive studies over many years on NPEfree surfactants and, so far, have not found any that are as good and effective as NPEs. Indeed, I have worked with a North American mill that had planned to use aspen hardwood in the manufacture of a certain grade of chemical pulp, but abandoned the idea as good quality pulp could not be achieved without the use of washing surfactants containing NPE. The removal of NPEs in cleaning agents has necessitated the implementation of long and expensive procedures such as extra washing at higher alkali charges and higher operating temperatures. These procedures have drawbacks in terms of cost of using extra chemicals, cost of energy to operate at higher temperatures, and possible damage to the fibres exposed to more alkali. Since the Botnia plant uses hardwood, and claims not to use NPEs, it would be useful to get information about the procedures used to achieve low and acceptable levels of extractives in the final products made at the mill. I understand that such information has not been made publicly available, and have not been able to find such information on the basis of web-based research.

The dangers posed by NPEs and why NPEs are banned in many places across the world.

16. NPEs are highly toxic to fish and other water dwelling organisms and are considered to be hormone disrupting substances, mimicking oestrogen. They degrade relatively readily in the environment to form the even more harmful nonylphenol (NP). For example, concentrations of nonylphenol as low as 0.017 mg/L (17 μ g/L) have been shown to be lethal to winter flounder in 96 hour exposures. Nonylphenol is not

readily biodegradable and takes months or even longer to degrade in surface waters or in soils and sediments, (where it tends to be easily immobilised). Non-biological degradation is negligible. Bioconcentration and bioaccumulation is significant in water dwelling organisms and birds, where it has been found in internal organs at between 10 and 1000 times greater than the surrounding environment. Nonylphenols are not broken down effectively in sewage treatment plants. Because of the bioaccumulation and persistence of nonylphenol (the primary degradation product of NPE), it is possible that it could be transported significant distances, and so have a potentially global reach in its effects.

- 17. Due to these concerns, the use of NPE-laden products has been banned in pulp and papermaking operations in the European Community. As far as I am aware, no such restrictions exist in Uruguay.
- 18. Current commercial products used as surfactants contain concentrations of more than 0.1% of NPE by mass. They therefore cannot be used for the manufacturing of pulp and paper, in accordance with EU Directive 2003/53. The levels vary from 1 to 6%. Some NPE surfactants are sold as 100% actives and are added to the wash water at 1-5 kg/tonne.
- 19. Possible alternatives to not using NPEs include: pulping softwoods instead of hardwoods (supply limitations for the Botnia mill); wood seasoning to reduce the amount of lipophilic extractives in the wood by natural means by leaving cut logs for 3-4 months (or in chip form for 3-6 weeks) before processing them for pulping; and good debarking (bark contains 6 times more extractives than wood). I believe that the Botnia mill is not implementing these alternatives because there is no ready availability of softwoods in Uruguay. Wood seasoning is expensive as it ties up capital and requires a lot land. Also, wood seasoning causes wood darkening and fungal growths that then would result in extra costs for the mill due to higher consumption of cooking and bleaching chemicals. Non NPE surfactants such as alcohol ethoxylates (AEs) have been developed to replace NPEs in North America in

the past 5 years, but they are not effective for hardwood extractives. I am not aware of their use in South American mills.

20. NPEs are commodity products and therefore relatively cheap. The newly-developed AEs are specialty products that are 3-4 times more expensive than NPEs.

Overall assessment and conclusions as to use of NPEs in some way at or on the Botnia plant on the basis of the material to which I have been referred.

- 21. I have reviewed the data submitted by the Argentine Government. Assuming it to be accurate, it provides convincing evidence of the presence of NPEs and their degradation product, NP, in the matrices collected from the Uruguay River and an analyzed pulp sample. The analytical protocols used are sound and have been carefully carried out according to international standards. The data is consistent with the conclusion that the Botnia plant is the source of the NPEs that have been detected. This conclusion is considerably strengthened by the observation that the amounts of NPEs in the analysed samples are highest at the mill effluent discharge points, and there is no evidence of discharges in material quantities from other sources (other than the mill) that may have contributed to the measured NPEs at such levels.
- 22. The prevalence of NPE-using industries that have similar problems as the pulp mill and the lack of formal NPE prohibition in Uruguay lead me to believe that the high quality hardwood pulp at the Botnia mill is most likely manufactured with aid of additives that contain NPEs.
- 23. From data published by Botnia, it seems that the plant produces fully bleached eucalyptus Kraft pulp. This type of pulp is well known to require extensive removal of extractives and lipophilic matter, and eucalyptus is notorious for difficulty in achieving very low levels of the extractives in the final pulp. Cost considerations

would point strongly to the use of NPE, since alternative cleaning processes are much more expensive. Having carried out a general review of material that is publicly available on the Botnia plant, I have not been able to find any information on the cleaning process. I have been provided with a copy of Annex 48 to Uruguay's Reply, a report by AMEC from September 2007. In the course of the oral arguments Uruguay has claimed that this includes information on chemicals that are used as cleaning materials at the plant. The AMEC report does not address the use of surfactants and nonylphenol ethoxylates at the mill. The information at page 22 of the AMEC report only refers to the defoamer. It does so by reference to US Environmental Protection Agency (EPA) Cluster Rule Requirements Related to Emissions to Water, which do not regulate NPEs (see http://www.epa.gov/EPA-TOX/2007/September/Day-05/t17542.htm). It appears to me that if the AMEC report had referred to European regulations on this issue then the mill would have had to address the NPE issue. Accordingly, Uruguay was wrong to state that the AMEC report had "specifically described the principal cleaning chemical used by the Fray Bentos mill" (CR 2009/23, p. 14).

- 24. My comments on the information provided in the affidavit of Mr. Gonzalez are set out below.
- 25. I note that at paragraph 25 of the arguments presented to the Court on 22 September (CR 2009/17, p. 23), Uruguay addresses issues of monitoring, but has not challenged the presence of elevated levels of nonylphenols in the vicinity of the plant's discharges and has not provided any satisfactory explanation for such elevated levels. It is important to point out that the reason why European and Canadian mills do not use NPEs is due to pressure from regulatory authorities and/or from customers. Whether or not the mills are 'modern' has no bearing whatsoever on the use of NPEs. In the case of the Uruguay mill therefore, I would ask: is there evidence that the mill is required to demonstrate that no NPEs are used? Canadian mills selling to Europe for example have to demonstrate or certify that no NPEs were used in the mills.
- 26. At paragraph 27 of its submissions presented on 22 September (CR 2009/17, p. 24), Uruguay accepts that there are nonylphenols in the river and addresses the question of where else could they have come from. It is my professional opinion, based on the

evidence obtained from the monitoring programme conducted by Argentina, that it appears that the highest concentrations of NPEs are at the mill effluent discharge point. I would ask how this can be explained, other than by the suspicion that the mill emits NPEs. Such suspicion is strengthened in my view by the fact that NPEs have been detected in the pulp sample. I am surprised that Uruguay has not addressed this point at all, which seems to me to provide the clearest possible evidence confirming the use of nonylphenols.

The statement produced by Ing Torres on 13 July 2009, in particular paragraph 4

27. I have read with interest the affidavit prepared by Ing. Torres on 13 July 2009. She states that "The use of etoxilated nonylphenol in the paper manufacture industry can be identified, but it is not directly identified with the wood pulp industry" (paragraph 4). It is not exactly clear to me what this means. If it is intended to suggest that the use of NPE is not associated with the wood pulp industry then it is inaccurate: for the reasons I describe above, NPEs have been extensively used in and are associated with the production of wood pulp, especially from hardwoods such as eucalyptus. She states that "Nonylphenol and its ethoxylates are not inherent elements in wood pulp manufacture processes by the Kraft method or its bleaching by ECF (elementary chlorine free)" (paragraph 1). It is not clear to me what she means by "inherent": the fact is that NPEs are extensively used in these activities around the world. She states that "the plant of Botnia ... does not use nonylphenol nor its derivative etholixates in any of its production and wood pulp bleaching processes" (paragraph 1). It is not clear to me what is meant by "production and wood pulp bleaching processes". NPEs are not used in the pulp bleaching process or in the production process as such: they are used in the cleaning processes I described above. It would be helpful if Ing. Torres had described by what methods the Botnia plant carries out its cleaning processes, and if she were to provide a list of the commercial products and compounds used in those cleaning processes. This information would be more definitive. She states that the Botnia plant "does not generate nonylphenol nor its etholixate derivatives in any of its processes" (paragraph 2). It is not clear to me what is meant by the word "generate". Typically the cleaning process does not "generate" NPEs, but rather it uses products that contain NPEs, which would typically be discharged into the effluent. In sum, the affidavit is very ambiguous, and uses terms that are strange. The use of NPEs in pulp and papermaking is well documented.

- 28. At paragraph 23 of its submissions made on 22 September (CR 2009/17, p. 23), Uruguay relies on the affidavit of Dr. Alicia Torres to support the claim that the Botnia plant does not use nonylphenols for any process, including the "cleaning of pulp". As I read the statement that is not what she says. Uruguay could have provided concrete evidence on all the chemicals used for the cleaning of the wood chips and the plants itself, but apparently has not done so, with the exception of Mr. Gonzalez's statement which I comment on below. Notwithstanding the limited and in some particulars, incorrect, information provided by Mr. Gonzalez (describing a defoamer as a 'surfactant' for example), the question that arises is this: how does the plant address the problems encountered in producing clean and high quality pulps from eucalyptus hardwood that is high in lipophilic content? Uruguay has provided no explanation.
- 29. I note that at paragraph 28 of the submissions presented on 22 September (CR 2009/17, p. 24), Uruguay states that "it is convinced that there is no use of [NPEs] by Botnia", adding that "At any rate, both Parties agree that Botnia should not use any nonylphenols in its production, cleaning, or any other processes". Uruguay then states that: "If, if, contrary to everything Uruguay believes to be true, Botnia is using nonylphenols, Uruguay will put a stop to it. …" It appears from this statement that Uruguay was, at that date at least, unable to confirm that it was not using nonylphenols. Indeed, it appears from the statement that Uruguay did not know at that date whether NPEs were being used in any processes at the plant. My response to these comments by Uruguay is to ask: Why is there no mill data relating to the use of NPEs in the DINAMA report? Was this information left out on purpose? I would have assumed that DINAMA would have precise data from such an important industry.

The statement produced by Mr. Gonzalez, Environmental Manager at the Botnia mill, dated 30 September 2009

- 30. I have reviewed the affidavit of Mr. Gonzalez. In particular, I have noted the comments on chemicals used at the Botnia mill made by Mr. Gonzalez in paragraphs 3, 5 and 6 of his affidavit. In my view, his comments do not explain or shed light on how high quality eucalyptus pulp can be produced at the Botnia mill without the use of NPE surfactants. The pulp produced by the mill is reported to include FRB EUCA which is described in Botnia's handbook on the mill as "a superior quality pulp of even quality and high flexibility" (Sowing the seeds of sustainability, Botnia and the Fray Bentos Pulp Mill", at page 101). The current published price for Botnia's FRB EUCA is USD 590 (effective 1 October 2009) which would confirm that this is not a low grade pulp (source: http://www.botnia.com/en/default.asp?path=204,210,211,2672,3058).
- 31. In my professional opinion, the mere addition of defoamer (even one incorrectly described as a 'surfactant' by Mr. Gonzalez in his affidavit) in the washing process will not be sufficient to achieve pulp of the quality which is produced at the mill. The very fact that a defoamer is required, indicates to me that a surfactant is indeed used in the washing process. I do not know of any surfactants other than NPE that can do the job of effectively cleaning the pulp, unless the mill is implementing other expensive cleaning strategies. It is striking that Uruguay has still provided no information on such strategies in the documents to which I have been referred. Thus, given the strong evidence of NPEs in higher concentrations in the waters into which the mill's effluent discharges are made, and given the failure by Uruguay or Botnia to explain how the mill is able to produce good quality pulp using only the materials referred to in Mr. Gonzalez's statement, I am bound to conclude that the evidence points strongly to the conclusion that NPEs have been used or are being used at the mill.
- 32. My detailed comments on Mr. Gonzalez's affidavit are as follows:

<u>In relation to paragraph 3</u> of his affidavit, I wonder if Mr. Gonzalez is familiar with the pulp making process? He states that "the brown stock" is "then subjected to indigester washing followed by several washing stages…" This statement does not make sense to anyone with familiarity with these processes, because wood chips are converted to pulp in the digester after which they go to a blow tank and then to the washing stages. Thus I do not understand what he means by "in-digester washing".

Even more significantly, paragraph 3 of the affidavit does not give details on how the lipophilic extractives are removed from the eucalyptus chips (as described above, these extractives have to be removed to produce high quality pulp). It would be useful, indeed essential in my view, in order to answer Judge Bennouna's question, for Uruguay to have provided detailed information on this key issue. The fact that it has not done so gives rise to even greater suspicion that it is at this stage in the process that NPEs are used.

In relation to paragraph 5, I note that Uruguay claims to have supplied information on "cleaning" chemicals used at the mill and that Mr. Gonzalez claims that the extractives are removed by a "washing process that uses a surfactant...called BIM AF 4151". However BIM AF 4151 is a defoamer, it is not a surfactant (a cleaner). Chemicals such as BIM AF 4151 are used to counteract the foam that results when a surfactant is used. Thus, the fact the mill uses a defoamer indicates that there are foaming problems at the mill, and the key question is: what is causing the foam? In all probability the foam is caused by the use of a surfactant. This raises the further question as to the identity of the surfactant that is used in the washing process that necessitates the need for a defoamer?

At paragraph 5, Mr. Gonzalez states that "surfactants help to remove air out of the washing water/liquor." This is not correct: surfactants exacerbate foaming problems; defoamers are used to remove foam, and not the other way round! Mr. Gonzalez appears to be confused or lack knowledge about processes and chemical products.

Also at paragraph 5 Mr. Gonzalez states that: "Extractives removed from the pulp during the washing end in the 'black liquor'". This is not entirely true. Some extractives will come out with the black liquor in the early stages of the washing when the concentrated black liquor is removed. After this, the wash liquid is not mixed with the concentrated black liquor to avoid dilution: diluted black liquor is not good for recovery boilers as its burning efficiency is reduced. Such wash liquids are treated to remove the extractives by several means such as floatation or purging into mill effluents. One of the main reasons for an effluent treatment system is to remove the extractives. Since Mr. Gonzalez is in charge of environmental issues at the Botnia mill, it puzzles me why he would say that wash liquids are not discharged to the effluent. Where else can they go? Only mills that are 100% closed can claim this, but the Fray Bentos mill is not one of them, so there must be wash process liquids that are discharged into the effluent system and then into the river.

<u>In relation to paragraph 6</u>, I note that Mr. Gonzalez mentions the use of three chemicals. '211' is an alcohol ethoxylate surfactant that, according to Mr. Gonzalez, is used for hand cleaning equipment at the mill. As mentioned above, alcohol ethoxylates are not as efficient as NPEs in removing hardwood lipophilic extractives, and presumably this is why Mr. Gonzales does not state that this product is used in the washing process at the Fray Bentos mill. The other two chemicals - PROFLOC 1408 and PROFLOC 2903 - are not surfactants at all. They are flocculants or coagulants, having an entirely different purpose.

The upshot of all this is that despite the passage of time since Argentina first raised this issue on 30 June 2009, Uruguay and Botnia have still not explained how the lipophilic extractives are removed from the eucalyptus hardwood chips.

Conclusion

33. I am not able to express an absolutely definitive view. However, there are a number of factors that are significant: the eucalyptus hardwood used to produce the pulp at the plant; the absence of easily available alternatives to clean the eucalyptus or its wood chips to remove lipophilic material; the issues of cost, taking into account the type of pulp produced at the mill; the repeated failure on the part of Uruguay and Botnia to provide detailed information on the cleaning processes used at the Botnia plant or the compounds that are used; the strong evidence of NPEs in higher concentrations in the waters into which the plant's effluent discharges are made; the evidence of NPEs in samples of pulp produced by the mill. These factors point strongly towards the clear conclusion that NPEs have been used at the Botnia plant in one or more cleaning processes associated with the production of wood pulp. In the light of my professional experience, I would be very surprised if NPEs had not been used. If I were asked to quantify my level of certainty that NPEs had been used, I would put it at 95%.

- 34. In conclusion, the arguments of Uruguay presented to the Court at the hearing are contradicted by the evidence I have seen. In relation to the question posed by Judge Bennouna, the affidavit relied upon by Uruguay does not provide a complete answer to the question and does not fully describe the "process and products … used by the Botnia plant for its cleaning". The failure to provide a complete answer reinforces the conclusion that NPEs have been used at the mill.
- 35. I would be pleased to assist the Court in seeking to answer any further questions that it might have.

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Bruce Sitholé, B.Sc.(Hons), M.Sc., Ph.D., FCIC

Annex 1: Curriculum vitae

B. Bruce Sitholé

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Citizenship: Canadian

SUMMARY

- Proven track record in developing innovative analytical methods to help solve production problems and improve productivity
- Application of FDA protocols in the analysis of food packaging
- Analysis of food additives and contaminants in food packaging
- Extensive interactions with both internal and external clients
- Very good communicator to both technical and non-technical audiences
- Well-versed in a variety of analytical techniques including LC-MS, HPLC, GPC, GC, GC/MS, Py-GC/MS, ion chromatography, and FTIR
- Very good at building collaborations with personnel from other departments and groups.
- Experience with washing and process aids
- Experience with use of surfactants in pulping and papermaking
- Consultations to pulp and paper mills in North America, South Africa and Brazil

WORK EXPERIENCE

Principal Scientist and Group Leader, Analytical Chemistry, 1987 - 2009: Analytical Sciences, Paprican, Pointe Claire, Quebec. Methods development and analysis of phytosterols, wood extractives, deposits and additives in pulp and paper matrices. The developed methods are used to solve production problems, assess environmental pollution, improve mill production, and for reverse-engineering studies to help in new product development. The analytical techniques used include LC-MS, GC/MS, pyrolysis-GC/MS, FTIR, HPLC, GPC, SPE and ion chromatography. Troubleshooting environmental and wet-end issues in paper mills.

Research Chemist: ONTARIO WASTE MANAGEMENT CORPORATION, May 1986 - June 1987. Characterization of and methods development for analysis of hazardous industrial wastes.

Research Officer: NATIONAL RESEARCH COUNCIL, Ottawa, October 1984 - April 1986. Research on in-door air pollution by GC/MS.

Post-Doctoral Visiting Fellow: Environmental Health Center, HEALTH AND WELFARE CANADA, Ottawa, December 1983 - September 1985. Analysis of halogenated phenols in drinking water by GC and GC/MS.

Research Chemist: United Kingdom Atomic Energy Authority, Dounreay, Scotland, 1978 - 1979. Characterization of soluble organic matter by GPC, fluorescence spectroscopy and ultrafiltration.

AWARDS AND HONOURS

United Nations Development Programme scholarship at the University of Sierra Leone, 1973-1977.

Third prize in an essay-writing contest held by the British Broadcasting Corporation in 1977.

Commonwealth scholarship at the University of Aberdeen, 1977-1978.

Commonwealth fellowship at Dalhousie University, 1979-1983.

NSERC Visiting Fellowship at Health and Welfare Canada, 1983-1985.

Presidential Citation, Pulp & Paper Research Institute of Canada, "For sustained leadership and innovation in developing analytical chemical procedures for the pulp and paper industry", 1993.

Director, Papricourse (2 week course on the science of pulp and papermaking), Pulp & Paper Research Institute of Canada, 1994-1998.

Elected Fellow of the Chemical Institute of Canada, 1997.

W.A.E. McBryde Award for work in analytical chemistry, The Chemical Institute of Canada, 1999.

Section Editor, Pulp & Paper, Encyclopedia of Analytical Chemistry, John Wiley & Sons, 2000.

Chair, Fine and Coated Papers Committee, Pulp & Paper Technical Association of Canada, 1998-2002.

Certificate of Appreciation, Pulp & Paper Technical Association of Canada, 2002, "To recognise individuals who have provided exceptional services to PAPTAC and/or the Canadian pulp and paper industry".

Member, Editorial Board, TAPPSA Journal (South Africa) since 2008.

International Scientific Committee Member, ISWFPC conferences (since 1997).

PROFESSIONAL MEMBERSHIPS

Chemical Institute of Canada

American Chemical Society

Association of Official Analytical Chemists

Pulp and Paper Technical Association of Canada

Technical Association of the American Pulp and Paper Industry

PUBLICATIONS

- 72 reports and publications in refereed journals
- 4 chapters in books
- 25 oral presentations at national and international conferences

EDUCATION

1) Ph.D. in Environmental Analytical Chemistry

Trace Analysis Research Center, Department of Chemistry, DALHOUSIE UNIVERSITY, Halifax, Nova Scotia, Canada, 1983.

Thesis Title: The Analysis and Chemistry of Antibiotics and Amines in Model Environmental Systems.

2) M.Sc. by course-work and thesis in Analytical Chemistry

Department of Chemistry, UNIVERSITY OF ABERDEEN, Scotland, 1978

Thesis title: Determination of Antimony in Organo-antimony Compounds by AA and Flame Emission Spectroscopy

3) B.Sc. (Hons.) Chemistry, UNIVERSITY OF SIERRA LEONE, Freetown, Sierra Leone, 1977

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