# Nitrogen oxide emissions from pulp mills and the factors affecting them – a summary of the current knowledge

Report for the Finnish Recovery Boiler Committee

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## 1. Background and introduction

The control of emissions from European pulp mills and the development of emissions regulations are guided by the following EU document:

> *Integrated pollution prevention and control (IPPC), reference document on best available techniques in the pulp and paper industry, 2001, European Commission, European Integrated Pollution prevention and Control Bureau, December 2001, 475p*

The document is available on the Internet, at: http://eippcb.jrc.es/pages/FActivities.htm.

The document, known as the IPPC document, specifies, among others, the best available techniques ('BAT guidelines'), for limiting nitrogen oxide emissions from pulp mills.

Table 2.43 (p. 107) of the document sets the range for nitrogen oxide emissions at 1.0-1.5 kg NOx/ADt when a pulp mill is using a 'suitable combination of best available techniques'. The figures include emissions from recovery boilers, lime kilns and malodorous gas boilers, but not emissions from auxiliary boilers or bark boilers.

The following summary of BAT guidelines for NOx emissions is presented on page 108 of the document:

> *NOx emissions: The emission of nitrogen oxides can be controlled by burner design (low NOx burners) and modified combustion conditions (primary methods).*
>
> *- The design of the recovery boiler (staged air feed systems) can result in relatively low NOx concentrations. The recovery boiler is however responsible for the largest contribution to the total NOx emission because of the large amount of liquors that are being burnt. Increased black liquor dry solids can lead to a slight increase in NOx emissions. Achievable emission levels are in the range of 80 – 120 mg NOx/Nm3 (40-60 mg NOx/MJ) or 0.7 – 1.1 kg NOx/ADt respectively. Process steam production is between about 13 and 18 GJ/ADt of pulp.*
>
> *- The lime kiln is also a source of NOx emissions in kraft mills because of the high combustion temperature. In lime kilns the type of fuel also influences the NOx formation. Higher NOx levels are measured with gas firing than with oil firing. In oil firing the NOx emissions of the lime kiln are in the range of 100 – 200 mg/Nm3 or 0.1 – 0.2 kg NOx/ADt whereas gas fired kilns achieve 380 – 600 mg/Nm3 or 0.4 – 0.6 kg NOx/ADt. Combustion of malodorous gases in the lime kiln can also increase NOx emissions. The possibilities to decrease the*

> *NOx emissions by adjusting the kiln running parameters, the flame shape, the air distribution and the excess oxygen is limited but can lead to a slight reduction of NOx formation (about 10-20%).*
>
> *-A separate furnace for TRS burning adds about 0.1 –0.2 kg NOx/ADt.*
>
> *Secondary methods as selective non-catalytic reduction (SNCR) are not in operation.*

The document was published in 2001 and its background information, such as Table 2.42 (p.106) on emissions to air from selected mills, dates from the 1990s. Thus, it is quite clear that at the time the document was written, not enough essential research data was available, particularly on nitrogen oxides.

The nitrogen cycle and the formation of nitrogen oxide emissions in the recovery process of a pulp mill are more complex than previously thought. Detailed studies on the formation of nitrogen oxides in the burning of black liquor began in the 1990s. However, a complete picture of the cycle of nitrogen compounds at pulp mills has only been clarified relatively recently; most of the key studies focusing on the complete picture have been published in the 2000s. A major part of this research has been carried out in Finland (see references) and in fact Finland has played a leading role in examining the issue.

This report contains a short review of the current knowledge on nitrogen oxide emissions from pulp mills. The aim is to provide an overview of the factors that are important when determining the total amounts of nitrogen oxide emissions at pulp mills, and especially to highlight the new research results that were not available when the IPPC document was drawn up.

The unit kg NOx/ADt of emission used in the IPPC document is always calculated per air-dry tonne of pulp. Total nitrogen oxide is given as NOx, but calculated as nitrogen dioxide i.e. $NO_2$. Thus, the unit of emission in the IPPC document corresponds to the unit kg $NO_2$/ADt used in this report.

This report was commissioned by the Finnish Recovery Boiler Committee.

## 2. The nitrogen cycle in the recovery process

Most nitrogen oxides escaping from pulp mills are contained in flue gases discharged from the recovery boiler or the lime kiln. Some mills have a separate combustion boiler for malodorous gases, from which some nitrogen oxides also escape into the atmosphere. Treatment of malodorous gases also generates nitrogen emissions. Ammonia forms the main component of these emissions.

Diagram 1 presents a summary of the nitrogen cycle in the recovery process. The diagram is based on recent research at Åbo Akademi University, and for the first time provides an overall picture of the behaviour of what is called reactive nitrogen (i.e. everything except molecular nitrogen in the atmosphere) at a pulp mill (Kymäläinen et al., see references).

Raw wood material contains organic nitrogen compounds as natural constituents, typically 0.05-0.20% (nitrogen) of dry matter (Section A, Figure 1). In normal cooking, nitrogen compounds in the wood dissolve more or less completely in the alkaline cooking liquor and are thus transferred as part of the black liquor to the evaporation plant and further to the recovery boiler. Typically, around a third of the nitrogen in the black liquor is oxidized into nitrogen oxide in the recovery boiler forming the NOx emission contained in the flue gases (Section B). Thus, the nitrogen content of raw wood material has a direct influence on nitrogen oxide emissions from the recovery boiler.

Recent research has shown that what is called thermal nitrogen oxide, i.e. the NO originating in nitrogen from the air, does not play any role in normal recovery boiler burning. (On the other hand, thermal NO is formed in a lime kiln, especially in the burning of natural gas).

An interesting and completely new observation was that another significant fraction of the organic nitrogen in the black liquor is transferred into the recovery boiler smelt as a new, previously unknown compound: sodium cyanate NaOCN (Figure 1, Section C). The cyanate nitrogen in the smelt passes into the green liquor through the dissolving tank  along with the smelt’s other salts. In the green liquor, alkaline hydrolysis gradually transforms cyanate nitrogen into ammonia.

The ammonia formed in the green liquor and in the white liquor partially evaporates into dilute malodorous gases (D). However, most of the ammonia continues as part of the white liquor back to the cooking (E). From the cooking, the ammonia passes into the black liquor together with the organic nitrogen compounds of the wood. The ammonia is finally quantitatively separated from the liquor in the evaporation plant, passing into concentrated malodorous gases and methanol (F).

The total emissions of nitrogen compounds from a pulp mill are thus dependent on the NOx emissions from the recovery boiler (B), and on how the ammonia flows generated in the chemical recovery cycle through the smelt are treated. At old mills where the treatment of dilute malodorous gases is deficient, most of the ammonia vapour (D) from the production of white liquor has escaped into the atmosphere with other dilute malodorous gases as ammonia emissions, which could not previously be monitored.

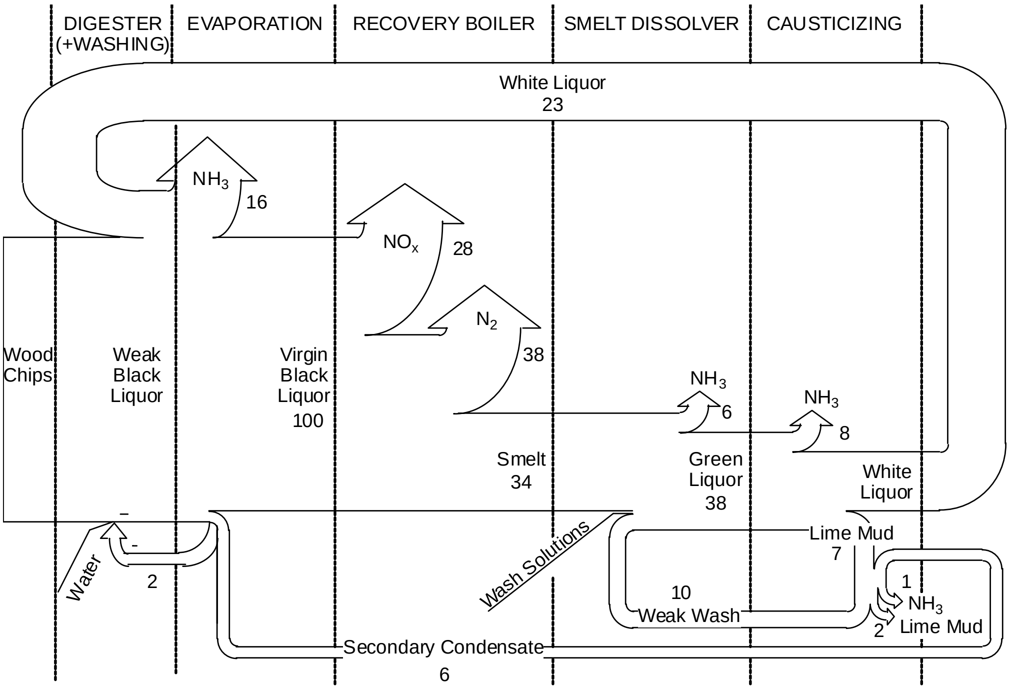

**Diagram 1.** The nitrogen cycle in a pulp mill recovery process. The diagram is based on research at Åbo Akademi University (Kymäläinen et al.).

In the latest processes, dilute malodorous gases are collected efficiently and led together with the combustion air, to the recovery boiler, thus allowing, for example, the ammonia emissions from the white liquor production to be recovered. On the other hand, further processing of these ammonia emissions may increase NOx emissions, from the recovery boiler, for example.

The ammonia in concentrated malodorous gases (F) ends up – depending on the mill’s processing operations – in the recovery boiler, lime kiln, or in a separate combustion boiler for malodorous gases. In all these operations ammonia in the concentrated malodorous gases has a strong impact on the NO emissions from the installations.

It is worth noting that when the EU’s IPPC document was drawn up, the cyanate in the recovery boiler smelt and the ammonia formation in the green and white liquor was not known at all. . At the time, the collection of dilute malodorous gases and exhaust gases was still deficient and no attention was paid to nitrogen emissions (ammonia emissions) in the malodorous gases as little was known about the types of nitrogen species they contained. Therefore, the document does not mention the significant influence of ammonia contained in malodorous gases on NOx emissions when the collection of malodorous gases is made more effective and gases are led to combustion. The potential NOx emissions resulting from the further treatment of both dilute and concentrated malodorous gases, and the factors influencing them are still being intensively studied.

Research is also being conducted on optimizing nitrogen chemistry in recovery boilers and lime kilns. To what extent can the NOx emissions of flue gases from the recovery boiler be reduced, while the amount of cyanate being transferred to the chemicals recovery cycle through the smelt is still under control? Would it be possible to control the amount of cyanate – and, consequently, the amount of ammonia transferring to the green liquor – with suitable changes in the operation of the recovery boiler? Can the principles of low NOx burning be applied to lime reburning without the quality of the lime sludge suffering?

## 3. Nitrogen balances and nitrogen oxide emissions at today's pulp mills

Figure 2 presents an example of a recent nitrogen balance measurement at a Finnish pulp mill. This is one of the few such measurements that also take into account the fate of ammonia in the green and white liquor circulation, and to date all of them have been carried out in Finland and Sweden. These types of measurements are demanding. Closing the balance of the nitrogen compounds requires reliable information not only on the concentration of nitrogen compounds at the right points of the process, but also accurate estimates of the quantities of the essential mass flows. In fact there is great uncertainty, especially about mass flows. For example, there has been considerable inaccuracy in measurements of flue gas volume flows in recovery boilers, and specifically for this reason some of the information on NOx emissions from recovery boilers is clearly wrong. Up to now, there have been no commonly agreed standards for determining flue gas flows for these purposes. A calculated estimate of the flue gas volumes based on fuel flow, fuel analyses and the composition of flue gases has proved the most reliable method. This method has been used in calculating the balances for the mill in Figure 2.

In spite of the problems involved, reliable comparisons and conclusions on the total emissions from different processes can only be made with careful mass flow balances.

When the balance was being calculated, the pulp mill used as the example manufactured about 56 air-dry tonnes of short-fibred pulp an hour (ADt/h) for the production of fine paper and folding paper. The raw materials used were birch, aspen and sawdust, containing 0.13%, 0.09% and 0.17% of nitrogen, respectively. The nitrogen flows in the diagrams are shown as kg of elemental nitrogen per air-dry tonne of pulp produced, i.e. kg N/ADt.

1.88 kg N/ADt of nitrogen compounds are carried with the black liquor to the evaporation unit. An additional 0.10 kg N/ADt enters the recovery boiler with the dilute malodorous gases mixed with the combustion air. Of this total nitrogen flow entering the boiler, 0.51 kg N/ADt forms nitrogen oxide in the flue gases, which corresponds to nitrogen oxide emissions of 1.66 kg $NO_2$/ADt.

0.49 kg/ADt of nitrogen in cyanate form is transferred into the smelt, which through the green liquor (complicated by the recirculation of white liquor) partly ends up in the dilute malodorous gases, but mostly in the cooking and, consequently the black liquor. This ammonia is released from the black liquor in the evaporation plant into concentrated malodorous gases, which at this mill are incinerated in the lime kiln.

The lime kiln mostly burns heavy fuel oil which also introduces organic nitrogen to the kiln. The flue gas emissions from the lime kiln amount to 0.04 kg N/ADt, i.e. 0.14 kg $NO_2$/ADt. Thus, the total nitrogen oxide emissions generated by this process are 1.80 kg $NO_2$/ADt.

Table 1 presents a number of recent nitrogen measurements at up-to-date Finnish mills. The figures are shown courtesy of the participant mills. The processes at the mills differ significantly from each other. There are differences between the wood raw materials, the products manufactured, the internal circulations of the processes, and especially the treatment of malodorous gases.

Emissions from the recovery boilers vary between 1.07 and 1.66 kg $NO_2$/ADt. The nitrogen content of the wood raw material explains most of this variation, although there are also other factors at play. However, it is essential to note that these figures are much higher than those in the BAT description of the EU document (see the start of this report):

> *"Achievable emission levels are in the range of 80-120 mg NOx/Nm3 (40-60 mg NOx/MJ) or 0.7-1.1 kg NOx/ADt respectively."*)*[1]

When the lime kiln emissions in table 1 are taken into account at those mills for which reliable measurement data is available, total emissions rise to between 1.55-1.84 kg $NO_2$/ADt, which is also clearly higher than the 1.0-1.5 kg $NO_2$/ADt given in the BAT description.

The emission figures given for the selected pulp mills in table 2.42 (p. 106) of the IPPC document seem extremely low in the light of the latest information. It is difficult to specify what the reasons are for the substantial difference between the current situation and the pulp mills in the IPPC document. Nevertheless, it is clear that when the IPPC document was drawn up, information on total nitrogen oxide emissions from pulp mills was still insufficient, and thus the reliability of the individual measurement results provided should be viewed with considerable caution. Only at the start of the 2000s did it become possible to produce reliable results for total emissions by combining nitrogen oxide emission measurements with the total nitrogen balance of the pulp mill.

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T[1] There is reason to suspect that the unit given in this sentence of the IPPC document is wrong; 80-120 mg NOx/Nm3 does not correspond with the range of 40-60 mg NOx/MJ. Presumably the unit of the first pair of figures was thought of as parts per million by volume, ppmv. In other words, the text should probably read: "Achievable emission levels are in the range of 80-120 ppm (40-60 mg NOx/MJ) or 0.7-1.1 kg NOx/ADt respectively." – This error does not affect the numerical values of the total emissions.

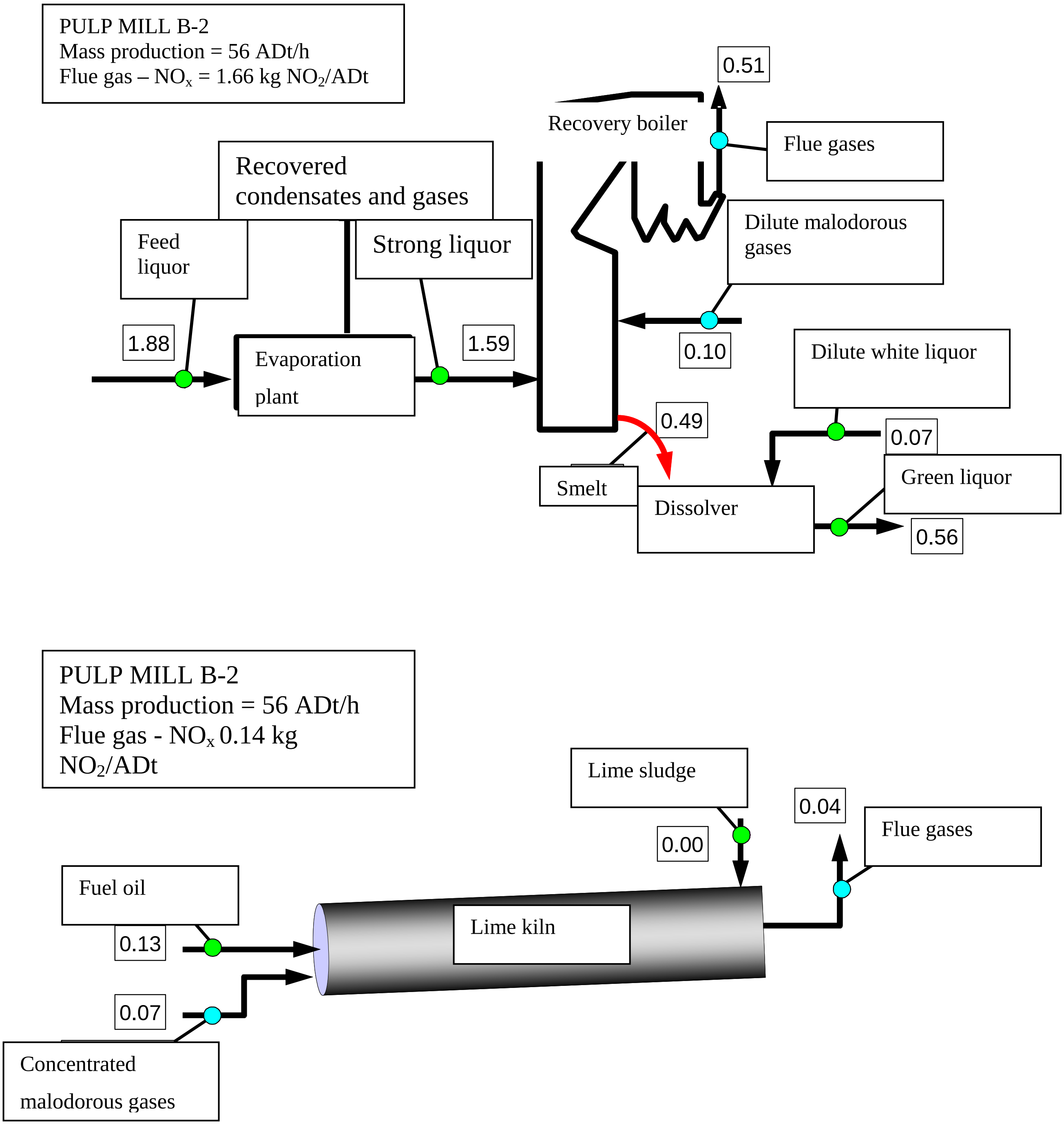

**Figure 2**. The flows of nitrogen compounds around a recovery boiler (top diagram) and a lime kiln (bottom diagram), pulp mill B-2. The nitrogen flows in the diagram boxes are presented as kilograms of elemental nitrogen per air-dry tonne of pulp, i.e. as kg N/ADt.

**Table 1.** Summary of recent nitrogen measurements at Finnish pulp mills

| | Mill | A-softwood | A-birch | B-1 | B-2 | C | D |
|---|---|---|---|---|---|---|---|
| Production at measurement | ADt/h | 67 | 71 | 55 | 56 | 65 | 77 |
| Pulp[1] | - | Softwood and hardwood pulp | Softwood and hardwood pulp | ECF and TCF bleached hardwood pulp | Short-fibred pulp | TCF bleached softwood pulp | ECF bleached softwood pulp |
| Applicability/ Product [1] | - | Tissue and fine paper and liner | Tissue and fine paper and liner | For manufacturing fine paper and folding box board | For manufacturing fine paper and folding box board | Reinforcement pulp and fully bleached tissue paper pulp | SC and LWC and coated, printing and special papers |
| Raw material[1] | - | Softwood | Birch | Birch, aspen, sawdust | Birch, aspen, sawdust | Softwood | Softwood |
| Nitrogen content | % in dry solids (DS) | 0.07% | 0.10% | Birch=0.13%<br>Aspen=0.09%<br>Sawdust=0.17% | Birch=0.13%<br>Aspen=0.09%<br>Sawdust=0.17% | 0.13%[2] | 0.13%[2] |
| **Recovery boiler** | | | | | | | |
| Black liquor flow | kg DS/s | 31.9 | 29.1 | 20.7 | 27.4 | 30.9 | 38.5 |
| Nitrogen content | % in DS | 0.07% | 0.09% | 0.09% | 0.09% | 0.05% | 0.08% |
| Black liquor nitrogen $N_{in, ML}$ | kg N/ADt | 1.28 | 1.20 | 1.99 | 1.88 | 1.13 | 1.37 |
| **Firing in the recovery boiler** | Y=Yes, -=no | | | | | | |
| Black liquor | | Y | Y | Y | Y | Y | Y |
| Concentrated odour gases | | Y | Y | - | - | Y | Y |
| Dilute odour gases | | Y | Y | Y | Y | Y | Y |
| Methanol | | Y | Y | - | - | Y | Y |
| Biosludge | | Y | Y | - | - | - | Y |
| BCTMP sludge | | - | - | - | - | - | Y |
| **Firing in the lime kiln** | Y=Yes, -=no | | | | | | |
| Natural gas | | | | - | - | - | Y |
| Fuel oil | | | | Y | Y | - | - |
| Heavy oil | | | | - | - | Y | - |
| Concentrated odour gases | | | | Y | Y | Y | - |
| Diluted odour gases | | | | - | - | - | Y |
| Methanol | | | | Y | - | - | - |
| **Emissions** | | | | | | | |
| $NO_x$, recovery boiler | ppm (3% $0_2$) | 95 | 125 | 89 | 148 | 105 | 113 |
| | kg N/ADt | 0.33 | 0.46 | 0.38 | 0.51 | 0.39 | 0.46 |
| | **kg $NO_2$/ADt** | **1.07** | **1.52** | **1.23** | **1.66** | **1.29** | **1.52** |
| $NO_x$, lime kiln | ppm (3% $0_2$) | [3] | [3] | 272 | 148 | 256 | 140 |
| | kg N/ADt | [3] | [3] | 0.10 | 0.04 | 0.17 | 0.08 |
| | **kg $NO_2$/ADt** | [3] | [3] | **0.32** | **0.14** | **0.55** | **0.28** |
| **$NO_x$, total emissions** | **kg $NO_2$/ADt** | - | - | **1.55** | **1.80** | **1.84** | **1.80** |

[1] http://www.metsabotnia.com

[2] Cole, D. and Rapp, M. 1981

[3] not measured

## 4. Methods for reducing emissions

There are a number of methods under development or undergoing testing that could potentially reduce NO emissions at pulp mills. Some of these are also mentioned in the IPPC document. A short overview of the techniques follows and their significance is evaluated in the light of the current information.

Recovery boiler:

Over-fire air (OFA) is a method in which, on top of the conventional air levels (commonly three), an additional air feeding level (fourth air level) positioned at a higher elevation from the boiler floor is incorporated in the boiler. This has been presented as a possible technique for reducing NO emissions by 10-25% depending on the original NO emission level. Additional air levels above the conventional ones can provide more freedom for managing NO emissions with combustion technology, and according to the current data could result in a 10-25% reduction. On the other hand, this technology will require considerable changes to the air feed systems of furnaces, and in several instances the special furnace wall tubes that can withstand corrosion in a reducing atmosphere, i.e. the compound tubes, would have to be extended from the lower part of the boiler to the highest air level, which would be a significant and expensive alteration in a boiler. It has also become apparent that over-fire air or other NO reduction techniques based on optimizing air distributions are not efficient if the boiler load is very high, or if boilers are operating overloaded, as is the case at several pulp mills.

Injecting ammonia into hot (around 900-1000 C) flue gases causes a selective non-catalytic reaction (SNCR), in which nitrogen oxide is converted into molecular nitrogen (Ng) by reacting with ammonia. The process is used in power boilers, although its effectiveness and applicability for recovery boilers is still unclear. One problem is the varying boiler load which moves the temperature window optimal for the SNCR technique to a different location in the flue gas channel. As a result, the efficiency of the NO reduction varies. More data is also needed on the increased corrosion risk of the flue gas channel that this technique may involve. Tests on the method are underway and results are expected in the near future. The potential reduction in recovery boilers (low initial NO level, varying temperature, effect of other components in the gases) is likely to be 30-50%.

Selective catalytic reduction (SCR), in which ammonia is injected in a special catalytic reactor into cooled flue gases, is another well-known technology that is commercially in use in power boilers, but has only reached the trial stage in recovery boilers. The problem with this technique is ensuring sufficient flue-gas dust removal before the process in the catalytic reactor and the durability of the catalyst. Thus there is no clear consensus on the operational economy of the technique. Preliminary tests on the technique have been conducted in a secondary flow separated from the flue gases in the recovery boiler, but a full-scale demonstration is yet to be reported.

In both techniques mentioned above, unreacted ammonia remaining in the flue gases as ammonia emissions is also a factor about which there is still insufficient practical empirical data and which may restrict the effectiveness of these techniques in reducing NO emissions.

Lime kiln:

Experience has shown that burning oil in a lime kiln produces lower emissions than natural gas, even though oil contains significant quantities of organic nitrogen, that is not present in natural gas. This is because of the higher temperatures of the natural gas flame, which cause the formation of more thermal nitrogen oxide than the burning of oil.

The low NOx burner technology used in power plants can also be aimed at in lime kiln burning. However, the main lime kiln process involves significantly more rigid boundary conditions for burning than at power plants. Applying low NOx technology in a lime kiln requires much more technical testing than in conventional boilers, and the reduction in NO is likely to be smaller compared with the results from burning in boilers.

Malodorous gases:

Burning malodorous gases in a recovery boiler is a potential source of additional NO emissions because of the ammonia contained in malodorous gases. The latest experiments, however, indicate that an increase in NO can more or less be avoided if the malodorous gases are injected in the correct way and in the right location in the boiler’s furnace.

In most cases, burning malodorous gases in a lime kiln results in a significant increase in the NO level. Developing low NOx technology for lime kiln burning, when both malodorous gases and the main fuel are present, will require additional investment, and currently there is no reliable technical solution available.

The ammonia problem of malodorous gases was not known when the IPPC document was drawn up and neither is there any mention of it in the document. This may partly explain the report’s low BAT guidelines for NO emissions.

## 5. Conclusions

On the basis of recent research, the formation of nitrogen oxide emissions from pulp mills has proved significantly more complicated than was perceived when the IPPC document was drawn up.

In particular, the cyanate forming in the recovery boiler smelt and the ammonia generated through it in the chemical recovery cycle are new key factors, the importance of which has only been understood in the last few years. The ammonia formed from the cyanate in the smelt is partially transferred to the malodorous gases released from the recovery cycle and is consequently a significant potential additional source of nitrogen emissions in a pulp mill.

As far as the total nitrogen oxide emissions of a pulp mill are concerned, it is not possible to give ‘typical’ or ‘good’ emission quantities. Many factors particular to each pulp mill play a key role, although these have not been considered in the IPPC document. These factors include the following:

- The nitrogen content of the wood raw material largely determines the NOx level of the recovery boiler. The nitrogen content of hardwood is usually significantly higher than that of softwood.
- The pulp cooking yield affects specific emission figures, when given as kg NOx/ADt.
- The way malodorous gases are treated is crucial. Should concentrated malodorous gases be burned in a recovery boiler, lime kiln or separately? Should dilute malodorous gases be recovered and where should they be led?
- The use of other fuels, such as oil or as a special example biosludge, in the recovery boiler has a fundamental effect on the recovery boiler's NOx emissions.
- The main fuel used for the lime kiln has a fundamental impact on NOx emissions. Oil introduces additional fuel nitrogen through organic nitrogen. It is difficult to control the formation of thermal NO when natural gas is used. There are big differences between individual kilns, and the development and optimization of burner technology for different fuels is incomplete.

Research activity into nitrogen oxide emissions from pulp mills is continuing and several total nitrogen balance measurements are underway. The results will help to produce significantly more reliable information on the total amount of nitrogen oxide emissions and the factors affecting them.

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