# Test Burn in Holcim Cement Kiln, Hon Chong, Kien Giang Province, Vietnam

# **15 February 2004**

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#### **1** Introduction

Sound hazardous waste management requires the availability of adequate treatment options to destroy or render hazardous substances harmless for health and environment. Vietnam is currently lacking sufficient treatment options for large quantities of hazardous waste

In the Southern Focus Economic Zone (SFEZ) of Vietnam more than 30 000 industrial enterprises produces yearly more than 80 000 tonnes of hazardous wastes, but no environmentally sound treatment and disposal options is currently available. The development of a proper hazardous waste management infrastructure is not only required to protect human health and the environment but it is also necessary to sustain further development of the economy in the region.

Safe and sound disposal of organic hazardous wastes requires high temperature incineration. High temperature incineration involves thermal destruction of the organic molecules, converting these into carbon dioxide and water. To achieve a complete thermal destruction, sufficient temperature, oxygen supply, residence time and mixing conditions are needed. Both dedicated hazardous waste incinerators and cement kilns can fulfil these requirements but normally cement kilns have higher temperature and longer residence times than dedicated hazardous waste incinerators. These advantages are widely utilised and cement kiln co-processing is regarded as one of the most cost-effective and viable waste treatment technologies available and represents the Best Environmental Practice for many industrial liquids and solid wastes today. Research and studies has confirmed this in many countries and also shown that the cement industry will emit considerably less  $CO_2$  by substitution of non renewable fossil fuel.

The Master Plan for Hazardous Waste Management in SFEZ recommended developing sound hazardous waste treatment facilities through private investments and one of the options identified was the Holcim cement kiln in Kien Giang Province.

Holcim Cement Plant in Hon Chong is a modern plant built in 1998 with the latest technology and with good access for waste through a sea harbour. Based on the recommendations in the Kåre Helge Karstensen / khk@sintef.no Master Plan and their leading experience in alternative fuel and raw material utilisation Holcim initiated a phased project, which aims to offer a cement kiln co-processing treatment solution for selected organic hazardous wastes in South Vietnam.

The main regulation on Hazardous Waste Management in Vietnam today is given in the Decision 155 which requires an environmental impact assessment as a basis for granting a treatment permit. Since the current regulation is not properly enforced in the region, the availability of wastes and the willingness to pay for disposal is uncertain. This phased project therefore aims to develop the necessary infrastructure in Hon Chong in a step-by-step approach, by increasing the investments as the wastes become available. Since it's not possible to perform a complete EIA without assessing the final and permanent facility, the first phase of the project aims to verify the performance of the cement kiln. The results of the test burn and the subsequent compliance with the TCVN's current emission limit values is the most comprehensive input to an environmental impact assessment and should be sufficient for issuing an interim permit (this is common practice in most countries) to co-process organic hazardous wastes for a limited period of three years. During this period, the climate for investment has hopefully improved and the permanent technical installations and infrastructure should be in place for a final EIA. During this interim period, Holcim will also assist the environmental agencies to develop recommendations for regulation of Alternative Fuels and Raw Materials management that allow facilities reasonable operating flexibility while protecting human health and the environment. There is no permit requirement for co-processing non-hazardous wastes or fuel.

The objective of the first phase of this project was to perform a test burn to demonstrate that the kiln is able to co-process hazardous wastes in an irreversible and environmental sound manner, i.e. to compare the emissions under normal baseline conditions when only fossil fuel is used with the emissions when fossil fuel is partly replaced by hazardous waste. Test burns with hazardous organic chemicals have shown to be the most comprehensive and accurate way to assess a kiln performance and are currently the main requirement in the stringent US regulation.

An application for permit to carry out the test burn plan was sent to the Department of Natural Resources and Environment in Ho Chi Minh City and Kien Giang Province in late August 2003.

A permit endorsed by the Ministry of Natural Resources and Environment, Department of Environmental Impact Assessment and Appraisal, was given in October 2003.

A solvent-based insecticide was identified to be a suitable candidate for the test burn, i.e. available in sufficient quantity, homogenous, with low viscosity and containing aromatic molecules that are difficult to incinerate.

An Australian independent test company carried out the stack gas sampling and the subsequent analysis in compliance with the most stringent international standards and the Holcim code of conduct.

A working group with leading experts and managers in Ministries and Provincial Departments has been supervising the project from the beginning. The test burn was carried out in October 2003 and was inspected by the leading scientists of the most prominent universities and research institutes in Vietnam. Information about the test burn was given in advance to all relevant stakeholders, provincial departments and responsible authorities.

#### 2 Cement production in brief

In short, cement is made by heating a mixture of calcareous and argillaceous materials to a temperature of about 1450°C. In this process, partial fusion occurs and nodules of so-called clinker are formed. The cooled clinker is mixed with a few percent of gypsum, and sometimes other cementitious materials, and ground into a fine meal - cement.



Figure 1 Processes identification and system boundaries of cement production

The main components of clinker are lime (CaO), silica (SiO<sub>2</sub>), alumina (Al<sub>2</sub>O<sub>3</sub>) and iron oxide (Fe<sub>2</sub>O<sub>3</sub>). The first stage in the industrial process of cement manufacture is the quarrying of raw materials. To obtain the proper composition of the raw mix, corrective ingredients normally have to be added to the quarried raw materials. Examples of corrective materials are sand, bauxite and iron ore; which compensate for deficiencies of silica, alumina and iron oxide, respectively. Fly ash, iron slag and other types of process residues can also be used as partial replacements for the natural raw materials.

The raw materials are ground in a raw mill into a fine powder – raw meal or raw mix – that is suitable for burning in a cement kiln. The produced clinker would typically contain 66% CaO, 21% SiO<sub>2</sub>, 5% Al<sub>2</sub>O<sub>3</sub>, 3% Fe<sub>2</sub>O<sub>3</sub> and 5% of other components. The four main oxides make up four major clinker phases, called alite, belite, aluminate and ferrite. To produce these phases, and to obtain them in right proportions, the raw meal is pyroprocessed, i.e. "burned".



#### Figure 2 Clinker production

The cooled clinker is mixed with cementitious materials such as gypsum, slag and flyash. Different clinker types and different types of additives are used, depending on what type of cement that is to be produced. The mix is ground simultaneously in cement mills and intermediately stored in cement silos. Finally the cement is packed in bags and dispatched, or in bulk.

Short kiln systems with 5 cyclone multi stage preheating and precalcination are considered best available technology for ordinary new plants and such a configuration will use 2900-3300 MJ/ton clinker. The application of the latest generation of clinker coolers and recovering waste

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heat as far as possible, utilising it for drying and preheating processes, will cut primary energy consumption even further.



Figure 3 Rotary kiln with cyclone preheater and gas dust collection

In the clinker burning process it is essential to maintain kiln charge temperatures of between 1400 to 1 500 °C and gas temperatures of about 2 000 °C. Also, the clinker needs to be burned under oxidising conditions. Therefore an excess of air is required in the sintering zone of a cement kiln.

The rotary kiln consists of a steel tube with a length to diameter ratio of between 10:1 and 38:1. The tube is supported by two to seven (or more) support stations, has an inclination of 2.5 to 4.5% and a drive rotates the kiln about its axis at 0.5 to 4.5 revolutions per minute. The combination of the tube's slope and rotation causes material to be transported slowly along it. In order to withstand the very high peak temperatures the entire rotary kiln is lined with heat resistant bricks (refractories).

The fuel introduced via the main burner produces the main flame with flame temperatures around 2 000°C. For process-optimisation reasons the flame has to be adjustable within certain limits. In a modern indirectly fired burner, the flame is shaped and adjusted by the primary air (10-15% of total combustion air). Flame shaping is mainly accomplished via multi-primary air channel burners.

Potential feed points for supplying conventional and alternative fuel to the kiln system are:

- via the main burner at the rotary kiln outlet end;

- via a feed chute at the transition chamber at the rotary kiln inlet end (for lump fuel);

- via secondary burners to the riser duct;

- via precalciner burners to the precalciner;

- via a feed chute to the precalciner/preheater (for lump fuel);

- via a mid kiln valve in the case of long wet and dry kilns (for lump fuel).

Preparation of different types of waste for use as fuel is usually performed outside the cement plant by the supplier or by waste-treatment specialist's organisations. This means they only need to be stored at the cement plant and then proportioned for feeding to the cement kiln. Wastes that are fed through the main burner will be decomposed in the primary burning zone at temperatures up to 2 000 °C. Waste fed to a secondary burner, preheater or precalciner will be burnt at temperatures up to 1 200°C.

Types of waste most frequently used as fuels in the cement industry today are used tyres, waste oils, sewage sludge, rubber, waste woods, plastics, paper waste, paper sludge, spent solvents and hazardous wastes.

#### **3** Outline of the test burn

The purpose of the test burn was to demonstrate that the kiln is able to co-process hazardous wastes in an irreversible and environmental sound manner, i.e. to compare the emissions under normal baseline conditions when only fossil fuel is used with the emissions when fossil fuel is partly replaced by a hazardous waste.

#### 3.1 Plant, kiln and control system description

Holcim Vietnam Ltd. was established pursuant the investment license No 808/GP issued 25/02/1994 by the State Committee of Investment and Cooperation. It is a Joint Venture between Holcim Ltd. (65%) and Ha Tien 1 (35%) for 50 years. The Hon Chong cement plant is located on the west coast of Vietnam, about 300 km west of Ho Chi Minh City, in Binh An ward, Kien Luong district, Kien Giang Province.

The three nearby mountains Cay Xoai, Bai Voi, and Khoe La serves the limestone source for the plant. The other major raw material component, clay, is excavated from a deep clay pit on site. After drilling using Atlas Copco drills, the limestone is blasted from the quarry face, recovered by excavators and continuously transported to the primary crusher house by 40 t Caterpillar trucks. The clay is exploited by means of a bucket excavator, delivering 200 tons per hour. The other raw materials, which are brought in by ship, are 70 000 tons per annum of Laterite from Dong Nai, 70 000 tons per annum of gypsum from Thailand and 150 000 tons per annum of coal from the North of Vietnam. Primary crushing of the coal to less than 120 mm is carried out by 750 tons per day impact crusher with a Hazemag rotor.

The limestone and clay pass through the transfer and analysing station which houses a Gamma Metrics PGNA analyser. For pre-blending, the main raw materials are stacked in 2 x 31 000 ton stockpiles within the covered area; high grade limestone is held in a 3000 t open store and the 10000 ton Laterite storage area lies close to the main pre-blending storage hall. After pre-

blending, the chemically balanced raw mix passes through the mill feed system, to the 350 tons per hour roller mill power by a 2800 KW motor. The mill is fed with hot gases from the kiln to dry the raw mix to less than 1% humidity. The dried material is then conveyed to 8000 t capacity blending silo IBAU Hamburg, where continuous homogenisation ensures the correct raw meal composition.

The clinker production is performed in a dry suspension preheater cement kiln equipped with a precalciner. The kiln rotates with a speed of 3.5 rounds per minute, is 4.6 meter in diameter, 72 meter long with a 110 meter high double string 5-stage preheater tower and produces approximately 4 400 tonnes of the intermediate product clinker per day. The clinker is cooled in a grate cooler, 21 meter long and 4.2 meter wide. The maximum output of clinker is 187 tons per hour with free-lime range of 1-1.5 %. The maximum feeding capacity is 305 tons of raw meal per hour to the second stage cyclone from the top. The precalciner is 29.1 meter high with an inside diameter of 6.6 meter.

The kiln and precalciner is fired with coal through two burner systems, the main burner and the precalciner. The main burner is a Pillard Rotaflam three channel burner, usually fired with 7 tons coal per hour; the precalciner takes 13 tons per hour. The normal fuel consists of anthracite coal with an average calorific value of 29 792 joule/kg.

Steel storage and feeding tank of 16m<sup>3</sup> was built near the preheater tower for the insecticide test burn candidate. The tank was connected to the light fuel oil pumping system with automatic dosage and switch off-on through the main control system. The tank was equipped with a diaphragm pumping system to empty the drums and was placed in a bunded concrete construction for spill recovery. The insecticide was pumped from the tank through stainless steel pipes to the burner platform, through a calibrated flow meter and into the main flame together with coal through the three channel burner.

The storage tank and introduction system was purpose made for handling liquid hazardous waste and was designed with a feeding capacity of up to 3000 l/h.



Picture 1 Storage and feeding tank for insecticide

The gas flows in the system provides combustion air to the main burner and the precalciner, and is primarily taken from cooling air in the clinker cooler and ensures maximum heat recovery. The production of normal clinker requires oxidative conditions, which means surplus oxygen. Normal oxygen concentration in the kiln inlet is 2-5% and the gas flow out of the stack is approximately 350 000  $\text{m}^3$ /hour (not corrected for humidity and oxygen).

Under normal operation, the exit gas from the preheater is directed through a conditioning tower to the raw material mill and the coal mill for drying purpose. A small portion of the gas (8%) can be directed to a by-pass system to reduce build-up of chlorine and alkalies. After drying, the gas is de-dusted in high efficiency electro static precipitator (ESP) before entering the main stack.

The normal operation mode is when the hot exit gas passes through the raw mill. This is called compound mode and produces approximately 11 tons of dust per hour. When the raw mill is of, usually less than 10% of the time, the gas flow is by-passed the raw mill before it is de-dusted in the ESP; this mode of operation is called direct mode and produces approximately 16.8 tons of dust per hour. All the dust produced are recovered and utilised in the process, either fed back to the blending silo in compound mode or fed to the kiln system in direct mode.

The process is monitored and controlled through an ABB Advant OCS process control system with continuous monitoring of the following gases: the kiln inlet gas is analysed by online monitors for  $O_2$ , CO and  $NO_x$ , the preheater outlet gas for  $O_2$ , CO and  $NO_x$  and the stack outlet gas for  $O_2$ , CO, CO<sub>2</sub>, NO, NO<sub>2</sub>, SO<sub>2</sub>, HCL, NH<sub>3</sub>, H<sub>2</sub>O and VOC.

The exit gas is sampled in the main stack, 122 meter high and a diameter of approximately 4 meter. The sampling platform is located at the top of the preheater tower and equipped with water and electricity.



Figure 4 Outline of the kiln, preheater, precalciner, raw mill, electro precipitator and stack

#### 3.2 Test schedule

The entire test exercise was conducted over three days, between 16 and 18 October 2003. The test burn constituted two days, first a baseline study and then a test burn, both days in compound mode operation. The third day, a regular internal baseline study was carried out in direct mode operation. Since these two modes of operation are not comparable, only the emissions the first two days are compared and discussed in this report.

A few days before the start of the test campaign the pumps and flow meters were calibrated by using light fuel oil and all monitors were checked. The AirLab team from Australia arrived the day before the baseline test and began to set up the sampling and monitoring equipment.



Picture 2 Stack gas sampling at the top of the preheater tower

The trucks with the insecticide arrived on-site the day before the test burn and the emptying of the drums were done manually with a steel lance, chemical resistant hose and a diaphragm pump connected directly to the storage tank with. The emptying operation was performed by persons equipped with personal protective gear and supervised by fire department staff and emergency staff. Procedures for preventive measures, potential spill abatement and emergency back up was drilled with dedicated personal well in advance of the exercise.



#### Picture 3 Emptying of insecticide drums into storage and feeding tank

The first day of the project, a baseline test was performed, i.e. stack gas sampling under normal conditions with coal firing only and with the exit gas through the raw mill (compound mode). The stack sampling started 10:00 and was finished 17:15. There was one raw mill stoppage during the sampling, from 14:02 to 14:07 due to error in temperature signal.

The test burn was performed the second day, i.e. with insecticide introduction through the main burner substituting parts of the coal and with the exit gas through the raw mill (compound mode). The stack sampling started 11:09 and was finished 18:13. There was one raw mill stoppage during the sampling, from 03:16 to 03:41 due to a hydraulic problem. The insecticide was introduced to the kiln at the following rate: from 06:05 to 07:00 approximately 1 000 litre per hour; from 07:00 to 18:45 approximately 2 000 litre per hour; from 18:45 to 00:12 approximately 2 500 litre per hour. The feeding pump which delivered the insecticide to the kiln was calibrated in advance of the test burn and showed a precision of  $\pm 2\%$ . In total, 39,497 litre insecticide was fed to the kiln. The insecticide tank and pipes was cleaned with the introduction of 2 500 litre light fuel oil per hour between 00:51 and 03:08.

#### Text Box 1 Outline of test burn

Day 1	Day 2
Baseline study 1	Test Burn
Compound operation and coal feed only.	Compound operation with insecticide and coal feed.

#### 3.3 Hazardous Waste Candidate for the Test Burn

A solvent-based insecticide called Access with two active insecticide ingredients, 18.8% Fenobucarb and 2.4% Fipronil, was used as the test burn candidate. The insecticide is solved in a mix of flammable solvents; consisting of 10.7% Cyclohexanone and 65% of heavy aromatic solvents produced by Exxon and called Solvesso.



#### Figure 5 Fenobucarb and Fipronil

The IUPAC name of Fenobucarb is 2-sec-butylphenyl methylcarbamate, Chemical Abstracts name is 2-(1-methylpropyl)phenyl methylcarbamate and the CAS RN [3766-81-2]. The IUPAC name of Fipronil is ( $\pm$ )-5-amino -1- (2,6-dichloro- $\alpha$ , $\alpha$ , $\alpha$ ,-trifluoro-p-tolyl) -4-trifluoromethylsulfinylpyrazole-3-carbonitrile, Chemical Abstracts name is 5-amino-[2,6-

dichloro -4- (trifluoromethyl)phenyl] -4- [(1R,S)-(trifluoromethyl)sulfinyl] -1H- pyrazole-3- carbonitrile and the CAS RN [120068-37-3]. Quantitative and qualitative analysis of Fenobucarb and Fipronil as an active ingredient is usually done by HPLC with UV detection, residues by GC with ECD.

Fenobucarb has a molecular weight of 207.3 with the molecular formula  $C_{12}H_{17}NO_2$ . Fipronil has a molecular weight of 437.2 with the molecular formula  $C_{12}H_4Cl_2F_6N_4OS$ , which means that the molecule contains 16.2% chlorine and 26.06% fluorine. Fenobucarb and Fipronil contain 6.7% and 12.8% nitrogen respectively.

Access was a product of Aventis Cropscience, now Bayer Cropscience, produced in 2000 and expired January 2003. Approximately 40 000 litres was stored in Bayer Cropscience warehouse in Dong Nai Province in 200 steel drums. The pesticide product was screened through 0.25 mm after production and blending and was in true solution. No settlements, particles or polymerization or degradation of the active ingredient was observed. Analysis of the active insecticide ingredient from different drums performed in several laboratories came up with marginal different results, most probably due to contamination or dilution of the sub samples. The result established by the Plant Protection Department in Ho Chi Minh City was 18.8% Fenobucarb and 2.4% Fipronil and are used as the average composition in this study as it is believed to be the most reliable result.

Both Fenobucarb and Fipronil are sold as active ingredients in separate pesticide formulations and they are usually potent insect killers, with different mechanisms and reaction time. However, the pesticide product was not suitable and saleable in Vietnam as it did not have the intended function on Vietnamese insects. Both active insecticide ingredients are classified by the World Health Organisation to be moderately hazardous on their scale from extremely and highly hazardous to slightly hazardous.

This material was chosen because it was considered to be a representative candidate of the hazardous waste streams which are available in Vietnam to day and which needs a sound treatment option. We were not been able to establish the thermal stability of the two molecules

but general knowledge indicate that the insecticide is quite stable and "not-easy" to destruct, and as such, a good candidate for verification of the cement kiln destruction capability.

The other requirement to the test burn candidate which was decided beforehand was the need of sufficient amounts of a homogeneous and fluid hazardous compound. The insecticide chosen was a free flowing liquid with a viscosity similar to water and possible to pump through the main burner. The calorific value of the insecticide was measured to 36.6–38.1 mega Joule per kilogram (after the test burn), fine coal is by comparison 29.5–30.5 mega Joule per kilogram.

Safety during transportation, handling and transfer had the highest priority and due care was demonstrated during the course of the trial. The transport of the 200 drums of insecticide was carried out by 10 trucks and organised by Bayer. The emptying of the insecticide from the drums to the 16m<sup>3</sup> storage/transfer tank was carried out by trained personnel from Holcim Vietnam. The personnel were equipped with suitable personnel protective equipment including organic vapour cartridge face masks. Preventive measures were in place in case of exposure, spillage and fire. All installations and drums were earthed during the transfer of the pesticide to the transfer tank.

All empty drums were taken back to Bayer in Dong Nai by the same trucks.

#### 4 Results and discussion

In this chapter, the process conditions and the results of the entire test burn project are discussed.

#### 4.1 Process conditions during testing

Feeding of raw meal was approximately 292 tons per hour and the subsequent production of clinker was 179 tons per hour during the entire test. Feeding of coal to the precalciner was stable at 13 tons per hour; the coal feed to the main primary burner was reduced by 1.5 tons from 7 to 5.5 tons when the insecticide was introduced to compensate for the heat input, see figure 6.

#### Text Box 2 Outline of entire test campaign; test burn project constituted day 1 and 2.

Day 1 – 16 October	Day 2 – 17 October	Day 3 – 18 October
Baseline study 1	Test Burn	Baseline study 2
Compound operation and coal feed only.	Compound operation with insecticide and coal feed.	Direct operation and coal feed only.



Figure 6 Feeding of raw meal and coal (tons/hour)

Due to erroneous knowledge about heat content of the solvent of the insecticide the coal to the main burner was not reduced sufficiently during the test burn. Measurements prior to the test burn had shown a calorific value of approximately 22.5 mega Joule; after the test burn the calorific value of the insecticide was measured to 36.6–38.1 mega Joule per kilogram; fine coal is by comparison 29.5–30.5 mega Joule per kilogram.

When introducing 2 tons of insecticide per hour the coal feed should have been reduced by approximately 2.5 tons to balance the heat requirement; it was reduced with 1.5 tons only. The insecticide also added 31.34 kg of nitrogen per hour as fuel  $NO_x$ . The consequence of this inadequate compensation was higher temperature in the kiln and subsequently higher  $NO_x$  levels.



Figure 7 NO<sub>x</sub> on-line measurements in kiln inlet gas (ppm) and in main stack (mg/m<sup>3</sup>)

There are mainly three types of  $NO_x$  generated in cement production, thermal  $NO_x$  which is due to the high temperature, and fuel and raw material  $NO_x$ , which is due to release of  $NO_x$  from the two inputs. The  $NO_x$  generation in cement kilns consists mainly of NO which is formed at temperatures above 1200°C and increases markedly above 1400°C.

Thermal NO<sub>x</sub> dominates over fuel NO<sub>x</sub> in the main burner flame in the kiln. In the secondary firing of the precalciner with a flame temperature below 1 200°C, the formation of thermal NO<sub>x</sub> is much lower compared to the main burner flame; hence fuel NO<sub>x</sub> is generated primarily in the precalciner.

 $NO_x$  formation increases with the amount of excess air in the flame and decreases in reducing atmospheres with low oxygen. When operating near to stoichiometric conditions in the kiln, localized generation of carbon monoxide will convert any NO to nitrogen.



#### Figure 8 Burning zone temperature (°C)

The fluctuations in the  $NO_x$  level caused minor process control disturbances. The process operator tried to achieve control by adjusting primary parameters like oxygen. The operator tried to control the increasing  $NO_x$  level on the 17 October by reducing the oxygen supply to the kiln. The operator was at this stage not aware that the elevated  $NO_x$  level was caused by to much heat input into the kiln. The coal to the main burner was reduced with 1.5 tons but should have been reduced with 2.5 tons per hour or slightly more taking into consideration the additional nitrogen load and the easy burnability (causes a more intense flame) of the solvent of the insecticide compared to coal.

The  $NO_x$  level was higher than normal even the third day, mainly due to high oxygen content. Many contributing factors are interrelated in different ways and with non-linear response. Without adequate a-priory knowledge about the right heat value and burnability, good  $NO_x$  control is difficult.



Figure 9 Kiln inlet NO<sub>x</sub> (ppm)



Figure 10 Kiln inlet O<sub>2</sub>(%)

The formation of  $NO_x$  in cement kilns is complex and as yet incompletely understood. Most studies have concluded that more than 90% of the  $NO_x$  emissions from cement kilns are NO, the rest  $NO_2$ ; almost no nitrous oxide ( $N_2O$ ) is formed in cement kilns. Thermal  $NO_x$  usually makes up 70% or more of the total  $NO_x$  and is formed by direct oxidation of atmospheric nitrogen through the dissociation of  $O_2$  and  $N_2$ . Thermal  $NO_x$  begins to form at temperatures as low as 1200°C and increases rapidly with even small temperature increases when within the range of 1370°C to 1870°C; the high-end temperature approximates that of the gas temperatures in the kiln's sintering zone. Given the high kiln temperatures, even small shifts in the amount of combustion oxygen can have a pronounced effect on the amount of thermal  $NO_x$  formed.



Figure 11 Kiln in-let CO

There are many ways to abate and control  $NO_x$  emissions by primary measures and optimization, including process control, improved firing techniques, optimized cooler connections, fuel selection, air optimization regimes (control ambient air infiltration), raw material proportioning and blending, use of waste material to achieve flame cooling in the sintering zone, staged combustion and reducing conditions.

The on-line monitoring of the  $NO_x$  levels measured in process control monitors shows slightly higher values than the daily averages measured by the AirLabs, see chapter 4.2.9.

The next two figures show the effect of running the process in direct mode operation, i.e. with the raw mill off as was done the third day. Because there is no material absorption or heat exchange in the mill the temperature in the electro static precipitator increases as the emissions of dust and hydrochloric acid. These modes of operation are very different and can not be compared with regards to performance etc. The plant is run in direct mode in less than 10% of the time.

There is no way of differentiating between chloride species coming from HCl or NH<sub>4</sub>Cl, the analytical method (ion-chromatography) is only detecting chloride species. Based on the NH<sub>3</sub> results it seems reasonable that a significant amount of the HCl is in fact NH<sub>4</sub>Cl.





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Figure 13 Effect of running the process with raw-mill off (18/10); increased temperature and emissions.

#### 4.2 Emissions to air

The results of stack gas sampling and analysis for the two test burn days are presented and compared with the current emission limit values (ELV) in the Vietnamese standard TCVN 5939-1995 and 5940-1995. The Vietnamese standards were tightened in 2001 and the ELVs were lowered. For the purpose of comparison in this study, only the most stringent ELVs are used.

#### 4.2.1 Sampling conditions during testing

The following testing conditions and stack values were recorded during the two test burn days and are valid for all subsequent analysis results.

#### Table 1Testing conditions and stack values

Testing conditions and stack values	Baseline	Test Burn
Sampling date	16 October 03	17 October 03
Duration of ampling	10:00 - 17.15	11:09 - 18:13
Stack dimensions at sampling plane (m)	4.00	4.00
Average stack gas temperature (°C)	110	110
Average velocity at sampling plane (m/s)	11.2	11.9
Actual gas flow rate (Am <sup>3</sup> /min)	8 440	8 970
Moisture content (% v/v)	12.2	12.3
Gas flow rate at STP (Nm <sup>3</sup> /min)	5 280	5 610
Gas flow rate at STP & 10% O <sub>2</sub> (Sm <sup>3</sup> /min)	7 590	8 080
Gas flow rate at STP & 10% O <sub>2</sub> (Nm <sup>3</sup> /hour)	456 000	485 000
Average oxygen concentration (%v/v)	5.24	5.21
Concentration of carbon dioxide (%v/v)	24.1	24.3

#### 4.2.2 Destruction and Removal Efficiency (DRE) of Fenobucarb and Fipronil

The solvent-based insecticide with 18.8% Fenobucarb and 2.4% Fipronil was fed through the main burner at an average rate of 2 027 litre per hour from 07:00 to 18:45 on the 17 October. The stack gas sampling was done between 11:09 and 18:13. From 18:45 to 00:12 the introduction rate was increased to approximately 2 500 litre per hour. The feeding pump which

delivered the insecticide to the kiln was calibrated in advance of the test burn and showed a precision of  $\pm 2\%$ . All together 39 497 litre was fed to the kiln during the 17 October.

Due to problems with the sampling equipment the feeding rate was decreased slightly between 10.30 and 11.00 to make sure that we had enough insecticide to carry out an adequate test burn. However, the problem with the sampling equipment was solved in a relatively short time and the stack gas sampling started at 11.09 and was finished 18:13; see figure below.



Figure 14 Feeding of insecticide (litre/hour)

To make sure that Fenobucarb and Fipronil was not a product of incomplete combustion normally found in the emissions, Fenobucarb and Fipronil were also analysed in the samples taken on the 16 October, i.e. when they were not introduced. This analysis represents the blank value. The analysis results are given in table 2 and shows that Fenobucarb and Fipronil were not

detected in any sample. The detection limit of the method was very low, at nanogram per cubic meter.

#### Table 2Analysis results Fenobucarb and Fipronil

	Baseline	Test Burn
Sampling date	16 October 03	17 October 03
Sampling time	10:00 - 17.15	11:09 - 18:13
Fenobucarb (ng/m <sup>3</sup> ) (dry, 273K, 101.3kPa, 10% O <sub>2</sub> )	< 18	< 21
Fipronil (ng/m <sup>3</sup> ) (dry gas, 273K, 101.3kPa, 10% O <sub>2</sub> )	< 12	< 14
Gas flow rate at STP & 10% O <sub>2</sub> (Nm <sup>3</sup> /hour)	456 000	484 800

Except for the  $NO_x$  fluctuations discussed earlier, no process upsets or disturbances occurred during the test burn with the insecticide. No changes in the controllable process conditions were observed during the pesticide introduction.

The destruction and removal efficiency DRE of Fenobucarb and Fipronil in the process is calculated according to the following equation:

 $DRE = [(W_{in} - O_{out}) / W_{in}] \ge 100$ 

The introduction of 2 027 litre Access per hour amounts to 362 kg pure Fenobucarb and 46.2 kg pure Fipronil per hour when corrected for the density of the Access, 0.95 (kg/l). No Fenobucarb

or Fipronil were detected in the exit gas and the subsequent detection limit was 21 ng/m<sup>3</sup> for Fenobucarb and 14 ng/m<sup>3</sup> for Fipronil. With a exit gas volume of 484 800 normal cubic metre per hour (corrected to 10% oxygen and normal conditions), this amounts to less than 0.0101808 gram of Fenobucarb and less than 0.0067872 gram of Fipronil per hour, which constitute a destruction and removal efficiency DRE of at least 99.999997% and 99.999985% for Fenobucarb and Fipronil respectively.

#### Table 3Destruction and removal efficiency DRE of Fenobucarb and Fipronil

	Fenobucarb	Fipronil
Destruction and removal		
efficiency - DRE	> 99.999997%	> 99.999985%

There is no requirement to demonstrate the combustion performance and subsequent destruction and removal efficiency DRE by test burn in Vietnam or elsewhere except in the US. The US Environmental Protection Agency (EPA) regulations require operators of US cement kilns that use hazardous waste as fuel to demonstrate that the kilns achieve a DRE of 99.99%. The most common principal organic hazardous constituents (POHCs) used as test burn candidates in the US are tetra-chloro-ethylene, tri-chloro-benzene, 1,2-di-chloro-benzene, and tri-chloro-ethylene. These chlorinated organic compounds are not very different from the insecticide used in this test burn.

Reports from the US that indicated cement kiln DRE results below 99.99% are either from outdated sources or improperly designed tests, or both. In the early years of development of this technology and the sampling and analytical techniques to evaluate its environmental performance, there were several instances where POHCs were selected that did not meet the criteria needed by a test burn candidate. For example, a major problem with many early tests was that the POHCs selected for DRE evaluation were organic species that are typically found at trace levels in the stack emissions from cement kilns that burn solely fossil fuel. While these

products of incomplete combustion (PICs) were emitted at very low levels, they nonetheless greatly interfered with the measurement of POHC destruction, i.e. DRE could not be properly measured if POHCs used in testing were chemically the same or closely related to the type of PICs routinely emitted from cement kiln stacks.

The most stringent regulation proposed in the world today is the requirements proposed in the newly signed Stockholm or POP's Convention. The Convention states that the "….necessary levels of destruction and irreversible transformation of POP's should be in the order of 99.9999%…..".

The insecticide used in this test burn is not a POP (persistent organic pollutant) and a DRE of 99.99% would have been sufficient to comply with the most stringent regulation currently available. The test burn clearly demonstrates the suitability of the Holcim cement kiln to co-process hazardous wastes in an irreversible and environmental sound manner and that a controlled substitution of fossil fuel with hazardous waste doesn't affect the emissions.

#### 4.2.3 PCDDs and PCDFs

According to our knowledge, this is the first time dioxins and furans (PCDD/Fs) have been measured in an industrial facility in Vietnam. The sampling and analysis of PCDD/F is complicated and expensive and requires outstanding experience and equipment. There is no emission limit value (ELV) for PCDD/F emissions from cement kilns in Vietnam, but TCVN 6560-1999 gives an ELV of 1 ng I-TEQ/m<sup>3</sup> for hospital incinerators.

The results of the dioxins and furans (PCDD/Fs) sampling and analysis are given in table 4. The results uncover very low dioxins and furans (PCDD/Fs) levels, comparable with the best performing cement kilns in the world. No international toxicity equivalents (I-TEQ) congeners were identified or quantified in the Baseline or during the test burn.

	Baseline 1	Test Burn	ELV in Vietnam
			(hospital)
Sampling date	16 October 03	17 October 03	
Sampling time	10:00 - 17.15	11:09 - 18:13	
ng PCDD/F I-TEQ/m <sup>3</sup> including half LOD values (dry gas, 273K, 101.3kPa, 10% O <sub>2</sub> )	0.0095	0.014	1

#### Table 4PCDD/F results

It is important to note that no I-TEQ congeners were positively identified or quantified in the baseline test or during the test burn, all congeners were below the detection limit. The result given in table 4 is the sum of 50% of the limit of detection for the different TEQ congeners; see table 5.

The result shows that the kiln is well in compliance with the Vietnamese emission limit value for hospital incinerators, 1 ng PCDD/F I-TEQ/m<sup>3</sup>.

The current PCDD/F emission standard for existing and new cement kilns co-processing hazardous waste in the US is either 0.20 ng I-TEQ/m<sup>3</sup> or 0.40 ng I-TEQ/m<sup>3</sup> and control of flue gas temperature not to exceed 200°C at the inlet to the air pollution control device APCD.

In the European Union, cement kilns co-processing of hazardous waste and secondary fuel are dealt with in the Directive 200/76/EC on the incineration of waste, having an emission standard for dioxins and furans of 0.1 ng I-TEQ/m<sup>3</sup>.

Dioxins and furans can be formed in all kinds of incineration processes under right conditions, i.e. with the presence of chlorine, carbon or hydrocarbon precursors, sufficient time and the right temperature window.

PCDD/F can be new-synthesized in cooler zones of a cement production facility, especially in the ESP region. PCDD/F formation (and subsequent emission) requires the simultaneous presence of the following factors of influence:

- Particulate surfaces/catalytic sites;
- Hydrocarbons and chlorides;
- Appropriate temperature window: between 200°C and 450°C;
- Appropriate residence time > 2 seconds.

In addition, molecular  $O_2$  has to be present in the gas stream.

It is a fact that the best available technique for the production of cement clinker, from an energy and emission point of view, is considered to be a dry process kiln with multi-stage preheating and precalcination, as the Holcim plant. Primary measures have shown to be sufficient to control an emission level below 0.1 ng I-TEQ/Nm<sup>3</sup> in such plants. The following primary measures have proven to be most critical:

- Quick cooling of kiln exhaust gases to lower than 200°C.
- No waste or alternative raw material feed as part of raw-mix if it includes organics.
- No waste feed during start-up and shut down.
- Feeding of hazardous waste only through the main burner or precalciner.
- Homogenous raw material mix and fuel feed;
- Regularity in fuel characteristics (both alternative and fossil);
- Excess oxygen.

All these requirements are fulfilled in the Holcim kiln and the test burn confirms that the plant is well operated and that the co-processing of hazardous waste doesn't influence the emissions of Kåre Helge Karstensen / khk@sintef.no

PCDD/Fs. The I-TEQ congeners and detection limits in the different samples are given in table 5.

## Table 5I-TEQ PCDD/F congeners

PCDD/PCDF	Baseline 1	Test Burn	I-TEF
Congeners	ng/Nm <sup>3</sup>	Ng/Nm <sup>3</sup>	
2378 TCDF	< 0.007	<0.010	0.1
2378 TCDD	<0.0034	<0.0051	1
12378 PeCDF	<0.0068	<0.010	0.05
23478 PeCDF	<0.0068	<0.012	0.5
12378 PeCDD	<0.0068	<0.010	0.5
123478 HxCDF	<0.0089	<0.013	0.1
123678 HxCDF	<0.0089	<0.013	0.1
234678 HxCDF	<0.0089	<0.013	0.1
123789 HxCDF	<0.0089	<0.013	0.1
123478 HxCDD	<0.0089	<0.013	0.1
123678 HxCDD	<0.0089	<0.013	0.1
123789 HxCDD	<0.0089	<0.013	0.1
1234678 HpCDF	<0.034	<0.051	0.01
1234789 HpCDF	<0.034	<0.051	0.01
1234678 HpCDD	<0.034	<0.051	0.01
OCDF	<0.17	<0.25	0.001
OCDD	<0.17	<0.25	0.001

#### 4.2.4 PAHs and HCBs

In table 6, the results for PAH and HCB are given. Hexachlorobenzene is currently not subject to regulatory monitoring in any cement plants but HCB monitoring of industrial facilities will be a requirement under the Stockholm Convention in the future. HCB was below the detection limit both under the baseline and the test burn.

The results in table 6 and 7 show that the emissions of PAH is very low and independent of coprocessing of hazardous waste. There is no ELVs for PAH or HCB in Vietnam.

Of the US and EU priority PAH measured, only Fluorene, Phenanthrene and Fluoranthene was identified in very low concentrations in the Baseline 1 test and only Phenanthrene was identified in very low concentration during the test burn. Naphthalene could not be quantified in any of the samples as it was found to be contaminant in the XAD-2 resin.

#### Table 6Summary of results for PAHs and HCB

	Baseline	Test Burn	ELV in
			Vietnam
μg PAHs/m <sup>3</sup> (US EPA priority)	1.8	0.49	
(dry, 273K, 101.3kPa, 10% O <sub>2</sub> )			
ng HCB/m <sup>3</sup>			
(dry, 273K, 101.3kPa, 10% O <sub>2</sub> )	< 31	< 35	

The congeners of the US and EU Priority PAH's and detection limits in the different samples are given in table 7.

	Baseline	Test Burn
PAH Congeners	μg/Nm <sup>3</sup>	μg/Nm <sup>3</sup>
Acenaphthylene	<0.17	<0.25
Acenaphthene	<0.17	<0.25
Fluorene	0.26	<0.25
Phenanthrene	1.36	0.49
Anthracene	<0.17	<0.25
Fluoranthene	0.20	<0.25
Pyrene	<0.17	<0.25
Benzo(a)anthracene	<0.17	<0.25
Chrysene	<0.17	<0.25
Benzo(b)fluoranthene	<0.17	<0.25
Benzo(k)fluoranthene	<0.17	<0.25
Benzo(e)pyrene	<0.17	<0.25
Benzo(a)pyrene	<0.17	<0.25
Perylene	<0.17	<0.25
Dibenz(ah)anthracene	<0.17	<0.25
Indeno(123cd)pyrene	<0.17	<0.25
Benzo(ghi)perylene	<0.17	<0.25
Sum US EPA PAHs	1.8	0.49

# Table 7Results of US EPA and EU priority PAH congeners measured

#### 4.2.5 PCBs

Polychlorinated biphenyls (PCB's) was sampled and analysed during the baseline and the test burn with the insecticide. All the PCBs were below the detection limit, i.e.  $ng/m^3$  and clearly shows that the plant has an excellent performance. Measurements done in 13 German cement kilns in 2001 revealed a maximum concentration of 0.4 µg PCB/Nm3. There is currently no emission limit value for PCB's in Vietnam. PCBs are not subject to regulatory monitoring in any cement plants today but will be a requirement under the Stockholm Convention in the future.

#### Table 8US EPA PCB priority pollutants results

Polychlorinated Biphenyl (ng/Nm <sup>3</sup> )	Baseline	Test burn
Non–ortho PCB's – Dioxin like PCBs		
3,3',4,4'-Tetrachlorobiphenyl (PCB 77)	< 0.034	< 0.051
3,4,4',5-Tetrachlorobiphenyl (PCB 81)	< 0.017	< 0.025
3,3',4,4',5-Pentachlorobiphenyl (PCB 126)	< 0.009	< 0.013
3,3',4,4',5,5'-Hexabiphenyl (PCB 169)	< 0.009	< 0.013
Mono–ortho PCB's – Dioxin like PCB's		
2,3,3',4,4'-Pentachlorobiphenyl (PCB105)	< 0.170	< 0.254
2,3,4,4',5-Pentachlorobiphenyl (PCB114)	< 0.034	< 0.051
2,3',4,4',5-Pentachlorobiphenyl (PCB118)	< 0.341	< 0.507
2',3,4,4',5-Pentachlorobiphenyl (PCB123)	< 0.034	< 0.051
2,3,3',4,4',5-Hexabiphenyl (PCB 156)	< 0.170	< 0.254
2,3,3',4,4',5'-Hexabiphenyl (PCB 157)	< 0.034	< 0.051
2,3',4,4',5,5'-Hexabiphenyl (PCB 167)	< 0.170	< 0.254
2,3,3',4,4',5,5'-Heptabiphenyl (PCB 189)	< 0.034	< 0.051

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#### 4.2.6 Volatile Organic Compounds & Benzene

VOC and benzene was measured all three days. Emissions of VOC and benzene are usually due to volatilisation of hydrocarbons in the raw materials when heated in the preheater and is normal in cement production.

The analysis results in table 9 show very low and stable levels, well in compliance with the current emission limit value in Vietnam. The emission of VOC or benzene are not be influenced by co-processing of hazardous waste when feeded to the main burner or the precalciner.

#### Table 9Benzene and VOC results

Concentration mg/Nm <sup>3</sup>	Concentration Baseline mg/Nm <sup>3</sup>		ELV Vietnam		
Benzene	2.2	3.2	80		
VOCs	17	26	200		

#### 4.2.7 Hydrogen Chloride and Hydrogen Fluorides

Hydrogen chloride, hydrogen fluoride and ammonia was measured all test days and table 10 reveals concentrations well below the current emission limit value in Vietnam. Even if the insecticide used in the test burn contains both chlorine and fluorine, the results show that the emissions of hydrogen chloride and hydrogen fluoride are not influenced by the co-processing of this hazardous waste. This result was expected.

Concentration mg/Nm <sup>3</sup>	Baseline	Test Burn	ELV Vietnam
Hydrogen			
chloride HCl	2.1	2.4	90
Hydrogen			
fluoride HF	<0.21	<0.23	4.5
Ammonia NH <sub>3</sub>	<1.0	<0.44	45

#### Table 10 Hydrogen chloride, hydrogen fluoride and ammonia

#### 4.2.8 Carbon Monoxide and oxygen

Table 11 shows the result of the CO and oxygen measurement. The result for CO is well below the current emission limit value in Vietnam and independent of the insecticide co-processing.

Carbon monoxide can arise from any organic content in the raw materials and, occasionally, due to the incomplete combustion of fuel. The contribution from the raw materials, due to preheating, will be exhausted with the kiln gases.

Control of CO levels is critical in cement kilns when ESPs are used for particulate abatement, to ensure concentrations are kept well below the lower explosive limit. If the level of CO in the ESP rises (typically to 0.5% by volume) then the electrical system is switched off to eliminate the risk of explosion.

The oxygen content measured during the test is within the normal range in cement kilns.

Concentration	Baseline	Test Burn	ELV Vietnam	
CO mg/Nm <sup>3</sup>	99	131	225	
O <sub>2</sub> %	5.24	5.21	-	

#### Table 11Carbon monoxide and oxygen

#### 4.2.9 Nitrogen and Sulphur Oxides

Table 12 shows the result of the nitrogen and sulphur oxides measurement. The result for  $SO_2$  is well below the current emission limit value in Vietnam and independent of the insecticide coprocessing. However, the mode of operation will have an effect; when the exit gas passes the raw mill under normal compound operation, the raw meal absorbs  $SO_2$ . 99% of the sulphur oxides emitted from cement kilns is in the form of  $SO_2$ .  $SO_2$  mainly originates from sulphides and organically bound sulphur in the raw materials.

The reason for the high  $NO_x$  levels during the test burn was due to high heat input through the main flame due to wrong information about heat content of the solvent of the insecticide prior to the test. The coal feed was approximately 1 ton higher than required, and the insecticide also contained nitrogen. The consequence of this inadequate compensation was higher temperature in the kiln and higher  $NO_x$  levels.

The result in table 12 confirms what most studies have concluded with earlier, that more than 90% of the  $NO_x$  emissions from cement kilns are NO, the rest is  $NO_2$ .

Concentration mg/Nm <sup>3</sup>	Baseline	Test Burn	ELV in
			Vietnam
SO <sub>2</sub>	1.8	2.0	225
NO <sub>2</sub>	21	40	-
NO	760	1220	-
NO <sub>x</sub> expressed as NO <sub>2</sub>	1180	1910	450

#### Table 12Nitrogen and sulphur oxides

The reference emission limit value for  $NO_x$  for the cement plant given in TCVN 5939 is 1000 mg/Nm<sup>3</sup>, but this emission limit value has recently been tightened to 450 mg/Nm<sup>3</sup>, which makes it one of the most stringent in the world. The NO<sub>x</sub> emissions are clearly in non-compliance.

Table 13 presents the  $NO_x$  normally found in cement plants. Preheater and precalciner kilns usually have the lowest emissions.

#### Table 13 Specific NOx emission rates for different cement kiln types

Kiln type	Typical heat input	Range of NO <sub>x</sub> emissions
	requirement (MJ/kg clinker)	(g/kg clinker)
Long wet kiln	5.0-6.0	1.6-8.8
Long dry kiln	4.7	2.8-4.8
Preheater kiln	3.1-4.0	1.1-5.3
Precalciner kiln	3.0	0.4-3.2

There are many ways to abate and control  $NO_x$  emissions by primary measures and optimization, including process control, improved firing techniques, optimized cooler connections, fuel selection, air optimization regimes (control ambient air infiltration), raw material proportioning and blending, use of waste material to achieve flame cooling in the sintering zone, staged combustion and reducing conditions.

Wastes used in cement kiln co-processing normally have lower heat content than normal coal and can be used for NOx control, but as clearly seen in this test; some wastes have higher heat content, which can cause minor problems if not compensated for.

#### 4.2.10 Total Solid Particles

Table 14 shows the result of the dust measurements. The result is well below the current emission limit value in Vietnam and independent of the insecticide co-processing.

The dust emissions from the plant are very low, one of the lowest in Vietnam.

#### Table 14Total suspended particles

Concentration mg/Nm <sup>3</sup>	Baseline	Test Burn	ELV Vietnam
Dust	33	20	100

#### 4.2.11 Metals

The measurements of Arsenic, Cadmium, Cobalt, Chromium, Copper, Mercury, Manganese, Nickel, Lead, Antimony, Tin, Thallium, Vanadium and Zinc are given in table 15. Vietnamese ELV's are given for Arsenic, Cadmium, Copper, Lead, Antimony and Zinc and the results are in compliance if our ELV information is right. The source of heavy metals to a cement kiln is raw materials and fuels and will be very site specific. The emission levels uncovered in this test is low and not influenced by insecticide co-processing.

Heavy Metal Concentration (µg/Sm3)	Baseline	Test Burn	Emission Limit
			Value in Vietnam
Arsenic	<5.4	<2.7	4 500
Cadmium	0.71	0.74	450
Cobalt	<0.54	<0.27	
Chromium	1.7	4.3	
Copper	<1.1	0.71	9 000
Mercury	4.7	0.33	
Manganese	12	14	
Nickel	1.6	1.8	
Lead	<4.3	<2.2	4 500
Antimony	<3.3	<1.6	11 250
Tin	71	38	
Thallium	<2.7	<1.4	
Vanadium	<0.65	0.82	
Zinc	13	2.7	13 500

### Table 15Heavy Metal Concentration in µg/Sm³

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#### 4.3 Solid samples

In the tables 16-17 the major inorganic components are given for raw meal, clinker, fine coal, ESP dust and BP dust sampled during the baseline and the test burn respectively. The results clearly show that certain elements are enriched in the dust fractions, which is normal, but no effect of hazardous waste co-processing is observed. All the dusts are recovered in the process.

Analyte mg/kg	Raw meal	Clinker	Fine coal	ESP dust	BP dust
Cl	480	18	94	1 500	25 000
F	2.7	< 0.40	1.7	3.7	< 8.0
К	660	6 000	390	1 000	42 000
Na	470	3 100	60	830	2 700
NH <sub>4</sub>	NR	NR	NR	< 2.0	< 2.0

#### Table 16Major inorganic components Baseline 1

Table 17Major inorganic components test burn

Analyte mg/kg	Raw meal	Clinker	Fine coal	EP dust	BP dust
Cl	470	19	140	1 300	24 000
F	2.7	< 0.40	1.6	3.8	< 8.0
К	720	5 400	400	990	43 000
Na	470	2 200	62	810	2 400
NH <sub>4</sub>	NR	NR	NR	< 2.0	< 2.0

In the tables 18-19 the trace inorganic components are given for raw meal, clinker, fine coal, ESP dust and BP dust sampled during the baseline and the test burn respectively. The results show that certain elements are enriched in the dust fractions, which is normal, but no effect of hazardous waste co-processing is observed. All the dusts are recovered in the process.

#### Table 18Trace inorganic components Baseline 1

Analyte	Raw meal	neal Clinker Fine coal EP dust		EP dust	BP dust	
mg/kg						
Arsenic	< 3.0	7.0	5.0	4.0	5.0	
Cadmium	0.6	0.5	0.5	0.8	40	
Cobalt	2.0	5.0	6.0	3.0	4.0	
Chromium	17	40	3.0	17	32	
Copper	2.0	5.0	14	2.0	8.0	
Mercury	1.1	< 0.05	0.73	7.0	< 0.05	
Manganese	140	210	38	160	170	
Nickel	6.0	11	13	6.0	8.0	
Lead	7.0	5.0	11	9.0	320	
Antimony	< 2.0	3.0	< 2.0	< 2.0	< 2.0	
Tin	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5	
Thallium	< 5.0	< 5.0	< 5.0	13	< 5.0	
Vanadium	12	< 0.5	8.0	15	28	
Zinc	17	31	27	22	39	

Analyte mg/kg	Raw meal	Clinker	Fine coal	EP dust	BP dust
Arsenic	< 3.0	4.0	5.0	4.0	7.0
Cadmium	0.7	< 0.5	< 0.5	0.9	45
Cobalt	2.0	5.0	6.0	3.0	4.0
Chromium	15	41	3.0	17	33
Copper	0.9	5.0	5.0 14 2.0		7.0
Mercury	1.0	< 0.05	0.57	7.2	< 0.05
Manganese	140	220	37	180	170
Nickel	4.0	11	12	6.0	8.0
Lead	7.0	< 2.0	11	9.0	390
Antimony	< 2.0	3.0	< 2.0	< 2.0	< 2.0
Tin	< 0.5	< 0.5	< 0.5	< 0.5	< 0.5
Thallium	< 5.0	< 5.0	< 5.0	10	< 5.0
Vanadium	12	38	8.0	15	27
Zinc	17	31	27	23	49

# Table 19Trace inorganic components test burn

#### 4.3.1 Product quality

Ordinary quality testing was performed on clinker, cement and concrete produced the day of the test burn. Because the clinker samples taken the two baseline days was not sufficient for both quality testing and external analysis, clinker produced under normal conditions on the 13 and 20 October was used as a baseline and reference.

Sample	Blaine cm <sup>2</sup> /g	R45 %	MgO %	Cl %	SO <sub>3</sub> %	LOI %	WD %	Sini min	Sfin min	1d MPa	3d MPa	7d MPa	28d MPa
13.10.03	4 870	5.2	1.53	0.02	2.20	1.40	25.4	115	145	24.5	46.2	54.7	62.0
Test burn	4 120	7.8	1.48	0.01	2.43	1.55	25.2	120	150	21.1	42.3	53	61.6
20.10.03	4 220	7.7	1.47	0.02	2.78	1.45	25.0	120	150	23.9	44.1	52.7	59.7

#### Table 20Normal quality testing of product

Blaine is a test of the fineness of the cement, unit in square centimetre per gram; R45 is also a fineness and residue analysis and gives the fraction that doesn't pass a 45 micron (45/1000 mm) screen. LOI is a measure of loss of ignition and WD of the water demand. Sini gives the initial setting time in minutes, i.e. how long time it take for the mixture of cement-water comes to harden; Sfin gives the final setting time in minutes, i.e. how long time it take for the mixture of cement-water to be hard and stable. 1d, 3d, 7d, 28d gives the strengths of concrete after 1 day, 3 days, 7 days and 28 days in mega-Pascal or N/mm<sup>2</sup>.

The results are within normal ranges of variation and shows clearly that the product quality is unaffected by the introduction of the insecticide.

#### 5 Conclusion

The objective of this project was to perform a test burn to demonstrate that the Holcim kiln in Hon Chong Kien Giang Province is able to co-process hazardous wastes in an irreversible and environmental sound manner, i.e. with no influence on the emissions when fossil fuel is partly replaced by hazardous waste.

Test burns with hazardous organic chemicals have shown to be the most comprehensive and accurate way to assess a kiln performance and are currently the main requirement in the stringent US regulation.

A solvent-based insecticide was identified to be a suitable candidate for the test burn, i.e. available in sufficient quantity and concentration of the active ingredient, homogenous, with low viscosity and containing aromatic molecules that are difficult to incinerate. The insecticide had two active insecticide ingredients, 18.8% Fenobucarb and 2.4% Fipronil, and was solved in a mix of flammable solvents. Fenobucarb has the molecular formula  $C_{12}H_{17}NO_2$  and Fipronil the molecular formula  $C_{12}H_4Cl_2F_6N_4OS$ , with 16.2% chlorine and 26.06% fluorine.

The insecticide was an unusable and expired product belonging to Bayer Cropscience stored in Dong Nai Province waiting for an appropriate disposal solution. This material was chosen because it was considered to be a representative candidate of the hazardous waste streams which are available in Vietnam to day and which needs a sound treatment option.

Safety during transportation, handling and transfer had the highest priority and due care was demonstrated during the course of the test. The transport of the 200 drums of insecticide was carried out by 10 trucks and managed by Bayer. The emptying of the insecticide from the drums to the 16m<sup>3</sup> purpose built storage/transfer tank was carried out by trained personnel, equipped with suitable personnel protective equipment including organic vapour cartridge face masks. Preventive measures were in place in case of exposure, spillage and fire. All installations and drums were earthed during the transfer of the pesticide to the transfer tank. All empty drums were taken back to the Bayer facility in Dong Nai by the same trucks.

The entire test campaign was carried out over three days, between 16 and 18 October 2003. The first day a baseline test was performed, i.e. stack gas sampling under normal conditions with coal firing only and with the exit gas through the raw mill (compound mode). Another baseline test was carried out the third day for internal investigative reasons. The results of the second baseline were in compliance with the emission limit values but are not used for further comparison in this report because the modes of operation is not comparable.

The test burn with the insecticide was performed the second day, i.e. with insecticide introduction through the main burner substituting parts of the coal and with the exit gas through the raw mill (compound mode). 2 027 litre per hour of insecticide was introduced to the kiln over a period of approximately 12 hours while exit gas was sampled from the stack. In total, 39497 litre insecticides was fed to the kiln and disposed of in less than 20 hours. The insecticide tank and pipes was cleaned with light fuel oil.

An Australian independent and accredited test company carried out the stack gas sampling and subcontracted accredited analysis laboratories. The entire testing and analysis complies with the most stringent international standards and the Holcim code of conduct.

Except for temperature and  $NO_x$  fluctuations, no process upsets or disturbances occurred during the test burn with the insecticide. The destruction and removal efficiency DRE was measured to be at least 99.999997% for Fenobucarb and at least 99.999985% for Fipronil. A destruction and removal efficiency of 100% will never be possible to establish or demonstrate due to detection limits in the analytical instruments. This means that the demonstrated DRE is higher in reality. These results are excellent and can be compared with the most stringent regulatory requirements in the world today, namely the USA, were industrial facilities and cement kilns combusting hazardous wastes must perform a similar test burn and demonstrate 99.99% destruction and removal efficiency (DRE). This result was 100-1000 times better.

With the exception of the  $NO_x$  levels, which was due to wrong information about the heat content of the solvent of the insecticide, all other measured parameters were low compared with international standards and in full compliance with the Vietnamese emission limit values. No influence on any emissions was observed when the insecticide was co-processed. The

fluctuations in temperature and  $NO_x$  levels was not caused by the insecticide itself, but the heat content and the easy burnability of the solvent used in Access.

This was the first time dioxins and furans (PCDD/Fs) were measured in an industrial facility in Vietnam and all the results were below the detection limit. According to international conventions 50% of all the detection limit values of the 17 individual I-TEQ congeners are summed to give a figure instead of a "below-the-detection-limit-value". A summary of half the LODs of the 17 I-TEQ congeners gives a figure of 0.0095 ng PCDD/F I-TEQ/m<sup>3</sup> for the baseline tests and 0.014 ng PCDD/F I-TEQ/m<sup>3</sup> for the test burn; but no TEQ congeners were detected.

This test burn has clearly demonstrated that the Holcim kiln in Hon Chong Kien Giang Province is able to co-process hazardous wastes in an irreversible and environmental sound manner and that there are no changes or influences in emissions when fossil fuel is partly replaced by hazardous waste.

Vietnam is currently lacking treatment options for large quantities of hazardous waste. In the Southern Focus Economic Zone of Vietnam more than 30 000 industrial enterprises produces approximately 80 000 tonnes of hazardous wastes yearly, without having any environmentally sound treatment or disposal options available.

The advantages of high temperature cement kilns are widely utilised around the world and coprocessing is regarded to be one of the most cost-effective and viable waste treatment technologies available and represents the best environmental practice for many industrial liquids and solid wastes today. Research and studies has confirmed this in many countries and also shown that the cement industry will emit considerably less  $CO_2$  by substitution of non renewable fossil fuel.

The Master Plan for Hazardous Waste Management in the SFEZ recommended development of sound hazardous waste treatment facilities through private investments and one of the options identified was the Holcim cement kiln in Kien Giang Province. This kiln has now shown to have an outstanding performance complying with the most stringent international regulations.

Holcim has a leading international experience in alternative fuel and raw material utilisation, and are offering a cement kiln co-processing treatment solution for selected organic hazardous wastes in South Vietnam. Holcim aims to develop the necessary infrastructure in Hon Chong in a step-by-step approach, by increasing the investments as the wastes become available. Based on the results of the test burn Holcim is applying for an interim permit to co-process organic hazardous wastes for a period of three years.

A working group with leading experts and managers in Ministries and Provincial Departments has been supervising the project from the beginning and the test burn was inspected by the leading scientists from the most prominent universities and research institutes in Vietnam.

Information about the test burn was disseminated well in advance to all relevant stakeholders, departments and responsible authorities. The findings of the test burn will be published to share the experiences with other developing countries.

#### Annex 1 Sampling and analysis methods

The Australian Testing Company AirLabs was responsible for the stack sampling and for all subsequent analysis. The sampling of process samples was carried out by the plant staff.

AirLabs followed stringent QA/QC procedures throughout the monitoring program. These procedures address such facets as sampling equipment, maintenance of sample conditions and concealment, storage and freight of collected samples. The subcontracted analysis laboratories follow the same stringent set of QA/QC procedures.

#### **Temperature, Gas Velocity and Volume Flow Rate**

Gas velocity and volume flow rate were determined in accordance with USEPA Method 2 – Determination of stack gas velocity and volumetric flow rate (type s pitot tube). Gas velocity profiles were obtained across the sampling plane using an S-type pitot/ K-type thermocouple probe linked to a TCR Tecora Flowtest calculator. Positions for velocity pressure and temperature measurement are determined in accordance with USEPA Method 1. Gas velocity and volume flow rate are then calculated in accordance with USEPA Method 2.

The estimated accuracy is  $\pm 1^{\circ}$ C for temperature and  $\pm 10\%$  for gas velocity and volume flow rate.

#### **Moisture Content**

Stack gas moisture content was determined in accordance with USEPA Method 4 – Determination of moisture content in stack gases. A measured volume of stack gas was bubbled

through a series of chilled impingers each containing 100mls of water and then drawn through a silica gel tube.

The total change in volume of water in the impingers and the weight change of the silica gel tube were used to calculate the percentage moisture in the stack.

The estimated accuracy is  $\pm$  5%.

#### **Oxygen and Carbon Dioxide**

Oxygen and carbon dioxide concentrations were monitored in accordance with USEPA Method 3A 'Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyser Procedure)'. In accordance with this method the sample gas was withdrawn continuously from the gas stream and portions wer conveyed to a paramagnetic O2 analyser and a Non Dispersive Infrared (NDIR) CO2 analyser. AirLabs uses a Horiba PG250 portable gas analyser.

The estimated accuracy is  $\pm 2\%$ .

#### **Carbon Monoxide**

Carbon monoxide concentrations were monitored in accordance with USEPA Method 10 'Determination of Carbon Monoxide Emissions from Stationary Sources'. In accordance with this method the sample gas was withdrawn continuously from the gas stream and conveyed to a Non Dispersive Infrared (NDIR) CO analyser. AirLabs uses a Horiba PG250 portable gas analyser.

The estimated accuracy is  $\pm$  5%.

#### Nitrogen Oxides

NO and NO2 concentrations were monitored in accordance with USEPA Method 7E 'Determination of Nitrogen Oxides Emissions from Stationary Sources (Instrumental Analyser Procedure)'. In accordance with this method the sample gas was withdrawn continuously from the gas stream and a portion was conveyed to a NO/NO2/NOx Chemiluminescent analyser. Total nitrogen oxides are expressed as nitrogen dioxide (NO2). AirLabs uses a Horiba PG250 portable gas analyser.

The estimated accuracy is  $\pm$  5%.

#### **Sulphur Dioxide**

Sulphur dioxide concentrations were monitored in accordance with USEPA Method 6C 'Determination of Sulphur Dioxide Emissions from Stationary Sources (Instrumental Analyser Procedure)'. In accordance with this method the sample gas is withdrawn from the gas stream and conveyed to a Non Dispersive Infrared (NDIR) SO2 analyser. AirLabs uses a Horiba PG250 portable gas analyser.

The estimated accuracy is  $\pm$  5%.

#### **Total Solid Particles**

Sampling for solid particles is conducted in accordance with US EPA Method 5 'Determination of Particulate Emissions from Stationary Sources'. Stack gas was withdrawn isokinetically

through a nozzle, which had a sharp and tapered leading edge. Particulate matter was collected downstream on a pre-weighed glass fibre filter located in a holder in a heated box.

Gases are then conditioned (for removal of moisture) and finally metered for flow rate and sample volume. The weight change of the filter is determined using an electronic balance and this figure used to calculate the concentration of particulate matter.

The estimated accuracy is  $\pm 15\%$ .

#### Ammonia

Monitoring for ammonia was conducted in accordance with NIOSH Method 6015. A measured volume of stack gas was drawn through a solid sorbent tube (SKC code 226-10-06). Gas phase ammonia was trapped in the sorbent tube which is extracted, and the extract was analysed using Visible Absorption Spectrophotometry.

The accuracy of this technique is estimated to be  $\pm 15\%$ .

#### PCDDs, PCDFs, PAHs, HCBs, PCBs and Insecticide

Sampling for Polychlorinated Dibenzo-p-Dioxins (PCDDs), Polychlorinated Dibenzofurans (PCDFs), Polychlorinated Biphenyls (PCBs), Polycyclic Aromatic Hydrocarbons (PAHs), Hexachlorobenzene (HCBS) and Pesticides (Fenobucarb and Fipronil) was performed in accordance with USEPA Method 23 - "Determination of Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans from Stationary Sources", using a USEPA Modified Method 5 sampling train. This method has been proven to be effective for the sampling of a wide range of semivolatile organic compounds from combustion systems, including PCBs, PAHs, HCBs and Pesticides.

The XAD-2 resin was spiked prior to sampling with isotopically labelled PCDD/PCDF surrogate standards. In the laboratory, PCDD, PCDF, PAH and PCB recovery standards were added to the sample components.

The filter, resin and impinger solutions were extracted with organic solvents and the extract purified by chemical treatment and solid phase chromatographic techniques.

Measurement of PCDDs and PCDFs was performed using high resolution gas chromatography with high resolution electron impact mass spectrometry in accordance with USEPA Method 1613A. The total toxic equivalent (TEQ) for each PCDD/F congener was calculated using international toxic equivalency factors (TEFs). Results have been expressed using the NATO 1989 TEFs and include half Limit of Detections (LODs) values and reported to 1989 NATO guidelines.

The method of extraction and purification of PAHs is based on USEPA Methods 3540 (soxhlet extraction of solid phase), 3510 (liquid/liquid extraction of aqueous phase), 3630 (SiO2 gel column) and 3640 (GPC). Measurement of PAHs was performed using high-resolution gas chromatography with low-resolution mass spectrometry. The total of 16 USEPA Priority Pollutant PAHs does not include limit of detection values.

The method of extraction and purification of PCBs is based on USEPA Methods 3540 (soxhlet extraction of solid phase), 3510 (liquid/liquid extraction of aqueous phase), 3620 (florisil column) and 3640 (GPC). Measurement of PCBs was performed using high-resolution gas chromatography with high-resolution mass spectrometry. This method provides data for a selection of the most toxicologically significant "dioxin-like" PCB isomers. Toxic equivalents (TEQs) are calculated for each of the targeted congeners using WHO 1998 (TEFs).

HCBS and Pesticides were determined directly from the solid and liquid phase extracts (USEPA Methods 3540 and 3510) using high-resolution gas chromatography with low-resolution mass spectrometry.

All results as presented in the tables are expressed to 2 significant figures for PCDDs, PCDFs and PCBs, PAHs and HCBS. The estimated accuracy for the determination of PCDDs, PCDFs and PCBs in stationary source emissions is  $\pm$  25%. The estimated accuracy for the determination of PAHs and HCBS is  $\pm$  20%. The estimated accuracy for the determination of Pesticides is  $\pm$  15%

#### Hydrogen Chloride, Hydrogen Fluorides and Chlorine

Sampling and analysis was performed in accordance with USEPA Method 26A 'Determination of Hydrogen Halide and Halogen Emissions from Stationary Sources – Isokinetic Method'. Gaseous and particulate pollutants were withdrawn isokinetically from the source and collected on a filter and in absorbing solutions. The filter collects particulate matter including halide salts. Acidic and alkaline absorbing solutions collect the gaseous hydrogen halides and halogens, respectively. Samples are analysed using ion chromatography.

The estimated accuracy is  $\pm 15\%$ .

#### Metals

Monitoring for metals was performed in accordance with USEPA Method 29 - "Determination of Metal Emissions from Stationary Sources". A stack sample was withdrawn isokinetically from the source. Particulate matter was collected on the internal surfaces of the probe and on the heated filter. Metals present in the gas phase and finer than the pore size of the filter were collected downstream in an aqueous acidic solution of hydrogen peroxide and an aqueous acidic solution of potassium permanganate.

Samples were recovered from the probe, filter, and aqueous acidic solution of hydrogen peroxide and analysed for all metals (including Hg). The aqueous acidic solution of potassium permanganate is analysed only for Hg by cold vapour atomic absorption spectroscopy

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(CVAAS). All other metals are determined using inductively coupled argon plasma emission spectroscopy - mass spectrometry (ICAP-MS).

Gaseous (volatile) (including the fine particulate fraction) and particulate fractions were determined separately. The estimated accuracy is  $\pm 15\%$ .

#### Volatile Organic Compounds & Benzene

This method applied to the measurement of volatile organic compounds (VOC) as total gaseous non-methane organics (TGNMO) and benzene in source emissions. Sampling and analysis for VOC was performed in accordance with USEPA Method 18 'Measurement of Gaseous Organic Compound Emissions by Gas Chromatography' – Adsorption Tube Procedure.

A measured volume of conditioned stack gas was drawn through an activated carbon tube. The activated carbon tube trapped any organic compounds present in the sample gas stream minus methane. The activated carbon tube was then extracted using carbon disulphide and the extract was analysed for TVOC and benzene using a gas chromatograph coupled to a flame ionisation detector. TVOC was measured relative to a hexane standard.

The estimated accuracy is  $\pm 15\%$ .

# Annex 2 Definitions

#### General Terms and Abbreviations

AFR	alternative fuel and raw material
APCD	Air pollution control device
BAT	best available technique
DRE	Destruction and removal efficiency
ELV	Emission limit value
ESP	electrostatic precipitator
EU	European Union
LOD	Limit of detection
TEQ	international toxicity equivalents (dioxins and furans)
TEQ/yr	Toxicity Equivalents per Year
THC	total hydrocarbons
TOC	total organic carbon
PCDDs	polychlorinated dibenzo-p-dioxins.
PCDFs	polychlorinated dibenzofurans.
PCDD/Fs	polychlorinated dibenzo-p-dioxins and dibenzofurans.
NATA	National Association of Testing Authorities, Australia
NATO	North Atlantic Treaty Organisation.
TEF	Toxic Equivalency Factor.
TEQ	Toxic Equivalent based on the 2,3,7,8-TCDD congener.
STP	standard temperature and pressure (0°C and 101.3 kPa).
Nm3	gas volume in dry cubic metres at STP.
Sm3	gas volume in dry cubic metres at STP and referenced to 10% oxygen.
<	less than. The value stated is the limit of detection.
g	grams.
mg	milligrams (10-3 grams).

μg	micrograms (10-6 grams).
ng	nanograms (10-9 grams).
pg	picograms (10-12 grams)
min	minute.
LOD	Limit of Dectection
TCDF	tetrachlorodibenzofuran.
TCDD	tetrachlorodibenzo-p-dioxin.
PeCDF	pentachlorodibenzofuran.
PeCDD	pentachlorodibenzo-p-dioxin.
HxCDF	hexachlorodibenzofuran.
HxCDD	hexachlorodibenzo-p-dioxin.
HpCDF	heptachlorodibenzofuran.
HpCDD	heptachlorodibenzo-p-dioxin.
OCDF	octachlorodibenzofuran.
OCDD	octachlorodibenzo-p-dioxin.
TCB	tetrachlorobiphenyl.
PeCB	pentachlorobiphenyl.
HxCDD	hexachlorobiphenyl.
HpCDD	heptachlorobiphenyl.

#### Annex 3 Accreditation – AirLabs statement

Since 1993, AirLabs Pty Ltd (trading as UniLabs Environmental) has been providing quality environmental monitoring services to both private and government organisations. The laboratory has been assessed against ISO/IEC 17025 (1999) requirements to undertake stationary source emissions monitoring. We have been informed that our technical assessment was successful and are awaiting final endorsement from the National Association of Testing Authorities, Australia (NATA).

In reference to analysis, Airlabs contracted SGS Environmental in Sydney who is accredited by the National Association of Testing Laboratories, Australia (NATA). NATA accredits laboratories against criteria based upon the internationally recognised standard ISO/IEC 17025:1999. SGS Environmental, Sydney holds NATA accreditation laboratory No. 262. All test reports are endorsed by NATA.

The analyses of the semi-volatile organic compounds were performed by SGS's Institute for Applied Chromatography, Belgium. SGS Belgium is a member of EA and the EA-MLA (testing) and also a member of ILAC. Accreditation Certificate No. Nr. 003-T.

Quality assurance & quality control (QA/QC)

AirLabs follows stringent QA/QC procedures when conducting all monitoring programs. These procedures were adhered to throughout the monitoring program at the Holcim (Vietnam) Hon Chong Plant. These procedures address such facets as sampling equipment, maintenance of sample conditions and concealment, storage and freight of collected samples.

Similarly, in reference to analysis, SGS Environmental Sydney and Belgium follow a stringent set of QA/QC procedures. The level of adherence to these procedures is reflected through their accreditations.

Système Belge d'Accréditation

Essais et Inspection

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Belgisch Accreditatiesysteem

Testen en Keuring

Member of EA and the EA-MLA (testing) Member of ILAC

#### ACCREDITATION CERTIFICATE

#### Nr. 003-T

In compliance with the provisions of the Royal Decree of December 22nd 1992 concerning the setting up of BELTEST, the Minister of Economy, hereby confirms, on advice of the Accreditation Bureau, that the test laboratory

#### SGS Belgium NV Division Oil, Gas and Chemicals Services Division SGS Environmental Laboratory Services Division IAC Haven 407 Polderdijkweg, 16 2030 ANTWERPEN

has the competence to perform the tests, mentioned in the attached enclosure, in accordance with the requirements of the standard NBN EN ISO 17025. The present accreditation certificate is granted for a period of 3 years starting from 10.05.2003 and is submitted to an intermediate surveillance.

The chair of the Accreditation Bureau,

\$Y\$

Nicole MEURÉE-VANLAETHEM

Original version of this certificate is in Dutch.

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The Minister of Economy,

Fientje MOERMAN

Kåre Helge Karstensen / <u>khk@sintef.no</u>