

# Pine needles (*Pinus sylvestris* L.) as a bioindicator of sodium and calcium deposition in the area around pulp and paper mills at Kemi, Northern Finland

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This study presents the most recent data on Na and Ca concentrations in Scots pine (*Pinus sylvestris* L.) needles sampled in bioindicator surveys carried out in 2009 at Kemi (Northern Finland), as well as at Pietarsaari (Western Finland) in 2007, at Enso (Eastern Finland) in 1994–1996 and in the Jyväskylä region (Central Finland) in 2006, which are areas with operating pulp and paper mills. Na and Ca concentrations in Scots pine needles at Kemi varied within 7–280 mg/kg (d. w.) and 1 410–5 450 mg/kg (d. w.), respectively. Na concentrations in both C and C + 1 needles at Kemi in 2009 were in good agreement with the values of 31–105 mg/kg (d. w.) and 54–151 mg/kg (d. w.), respectively, observed at Enso. Ca concentrations in C + 1 needles at Kemi were in good agreement with the values of 2 432–5 598 mg/kg (d. w.) observed at Pietarsaari in 2007 and with the values of 2 700–7 900 mg/kg (d. w.) observed in the Jyväskylä region in 2006, although the highest Ca concentrations – 7 900 mg/kg (d. w.) – in the C + 1 needles in the latter area was obviously higher than the corresponding value of 5 450 mg/kg (d. w.) observed in our study at Kemi.

**Key words:** calcium, particulate emissions, *Pinus sylvestris*, pulp mill, sodium

## INTRODUCTION

The gaseous and particulate matter emissions from industrial plants impact the environment in the form of dry and wet deposition. The distance and area over which pollutants spread are dependent, inter alia, on the height they reach in the atmosphere and on climatic factors. Compared to gaseous compounds, the distance to which particulates are transported is generally relatively short. The transport distance depends on factors connected with the production plant, such as the height of the stack and emission levels, as well as on the size and density of the particles. Large particles are deposited relatively close to the emission sources, while smaller particles, aerosols and gaseous metallic compounds can be transported hundreds of kilometres through the atmosphere [1]. Scots pine (*Pinus sylvestris* L.) is a tree species widely distributed in Northern and Eastern Europe, and its needles have proved to be suitable air quality indicators of the deposition of pollutants [2, 3]. The use of pine needles as emission

indicators around pulp and paper mills in Finland was first reported in the late 1960s [4].

The aims of this study were: (i) to determine sodium (Na) and calcium (Ca) concentrations in Scots pine (*Pinus sylvestris* L.) needles in the area around industrial plants (i. e. pulp mills, chromium mine, municipal district heating plants and port operations) at Kemi, Northern Finland, because sodium (Na) salts are released from the recovery boiler and calcium (Ca) salts from the lime kiln of the pulp mills, and (ii) to compare the Na and Ca concentrations observed in this study with the results of recent studies carried out in the parts of Finland where pulp and paper mills operate.

This study is part of a major project focussing on the environmental effects of the forest industry in the Kemi-Tornio region, Northern Finland. Our previous studies have dealt with the use of Scots pine (*Pinus sylvestris* L.) needles as bioindicators for determining the distribution pattern of aerial emissions from the pulp and paper mills [5, 6] and the use of peach as a bioindicator of trichloroguaiacol accumulation in fish [7].

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## EXPERIMENTAL

### Study area and pollution sources

The study was carried out in the town of Kemi (65°44'N, 24°35'E) on the Gulf of Bothnia, Northern Finland (Fig. 1). In 2008, Kemi had a population of about 22 400. Sulphur and particle emissions in the study area originate from the pulp and paper mills of Stora Enso Veitsiluoto Mill (Fig. 1; abbreviation SE), from the pulp mill of Oy Metsä-Botnia Ab Kemi Mill (Fig. 1; abbr. MB), which is integrated with the lineboard mill registered under the name of M-Real Oyj, from the chromium mine of Outokumpu Chrome Kemi Mine Oy (Fig. 1; abbr. OC), from the municipal district heating plants of Kemin Energia Oy (Fig. 1; abbr. KE) and Keminmaan Energia Oy (Fig. 1; abbr. KME), and from the port operations (Fig. 1; abbr. PORT) located on Ajos peninsula.

Most of the energy needed by the mills is generated by burning black liquor, wood residues (i. e. wood chip, sawdust and bark) in the bubbling fluidized bed boilers of the power plants, although heavy fuel oil is also needed in the boiler. In addition, Stora Enso Oyj Veitsiluoto Mill incinerates peat. Outokumpu Chrome Kemi Mine Oy is a large, underground mine. Underground mining started in 2003 and at the beginning of 2006 replaced the open cast mining. The Outokumpu Chrome Kemi Mine Oy is heated using propane gas which has no sulphur dioxide emissions. However, sulphur dioxide emissions are derived from the heavy fuel oil

burners (1.4 MW + 1.5 MW) and from the light oil burners (1.1 MW).

The municipal district heating plants of Kemin Energia Oy and of Kemin Energia Oy produce energy by bubbling fluidized bed boilers of 32 MW and 6 MW, respectively. The energy is produced by incinerating forest residues (i. e. wood chips, sawdust and bark) and peat. The main sulphur and particle emission sources in the port operations on Ajos peninsula originate from cargo vessels and vehicles, as well as from the heavy oil boiler of Neste Oil Oyj (5.1 MW). The sulphur dioxide emissions from road traffic in Kemi are very low; in 2008 they were only about 0.7 t [8]. For a more comprehensive review of the fuels used and the incineration techniques at those industrial plants, see our previous publications [5, 9, 10].

### Development of gaseous sulphur and particulate matter emissions at Kemi

During the period 1980–2008, gaseous sulphur (SO<sub>2</sub> + TRS) emissions at Kemi fell ten-fold – from 4 502 tonnes to 485 tonnes per year [11]. Furthermore, particulate matter emissions during 1992–2008 decreased from 2 044 tonnes to 124 tonnes per year (Fig. 2). No information is available about particulate matter emissions before the year 1992 because the operators of industrial plants were not obliged by their environmental permits to regularly monitor the release of particulate matter (i. e. emission monitoring) before 1992.

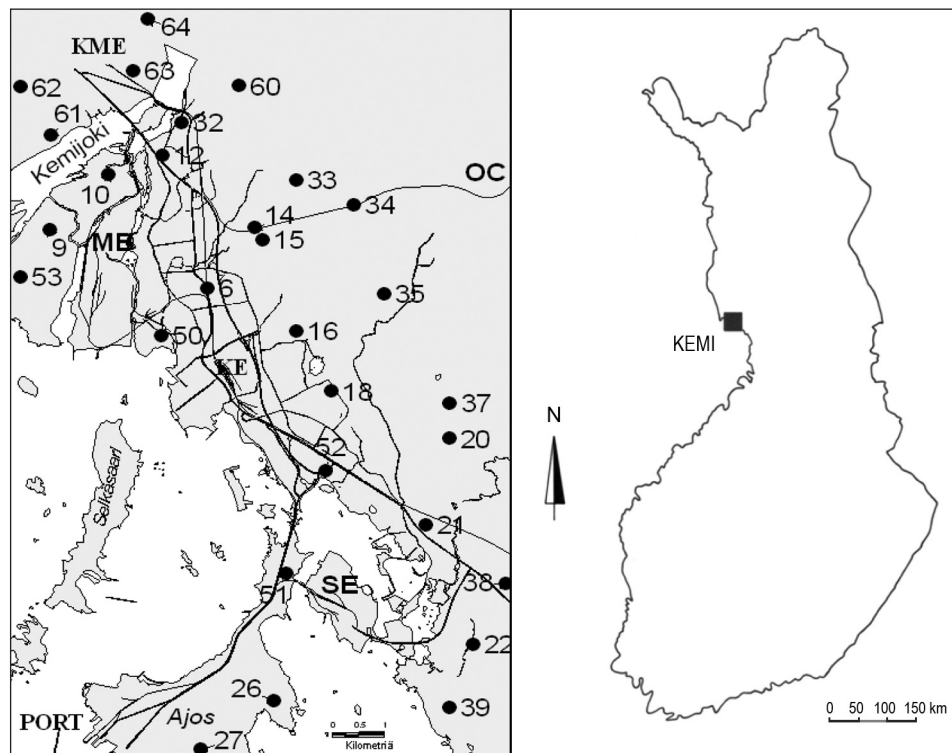


Fig. 1. Map of Finland showing the location of the sampling sites and industrial plants at Kemi in 2009 [5].

Note: Abbreviations in Figure 1: KE = Kemin Energia Oy, KME = Keminmaan Energia Oy, MB = Oy Metsä-Botnia Ab Kemi Mill (including M-Real Oyj), OC = Outokumpu Chrome Kemi Mine Oy, PORT = port operations on the Ajos peninsula, SE = Stora Enso Oyj Veitsiluoto Mill

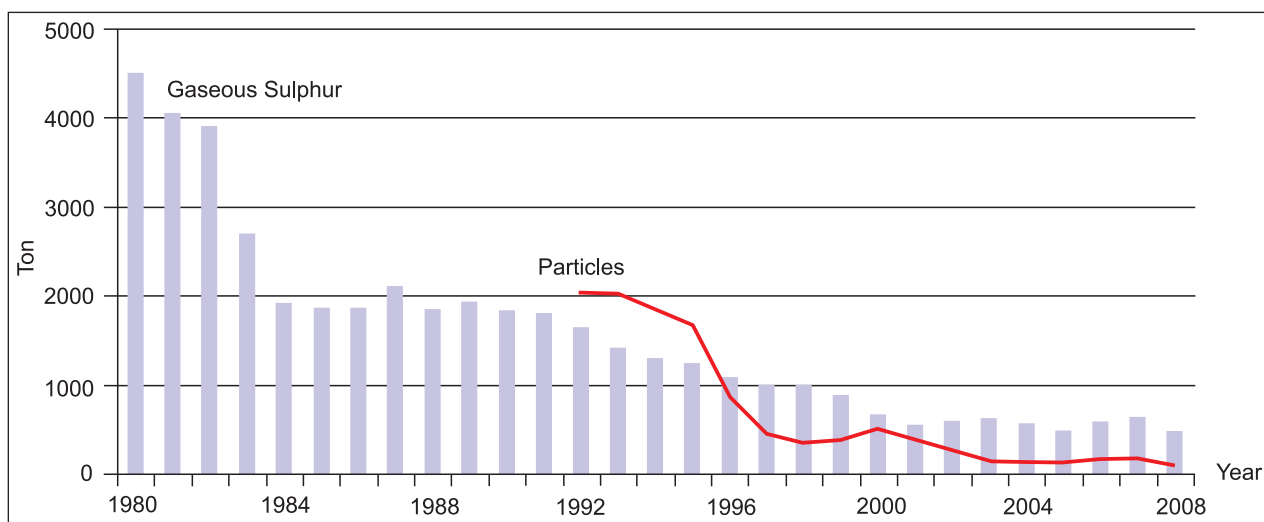


Fig. 2. Gaseous sulphur ( $\text{SO}_2$  + TRS expressed as S; ton per year) and particulate matter (ton per year) emissions in Kemi during 1980–2008 [11; partly in 5]

The decrease in gaseous and particle emissions is due to the fact that the industrial plants have upgraded their processes and, in addition, the burning of heavy fuel oil is nowadays minimal. The decreasing trend in gaseous sulphur and particle emissions is a result of the growing awareness of environmental issues, environmental training and investments in modern, cleaner, resource-saving technology and the introduction of environmental management systems at the industrial plants in the study area. In addition, the falling trend in gaseous sulphur emissions is also partly attributable to the extension of district heating. Stora Enso Oyj Veitsiluoto Mill and Oy Metsä-Botnia Ab Kemi Mill sell energy to the municipal energy company of Kemin Energia Oy, which is used for district heating of the residential areas located in the immediate vicinity of the mills. Furthermore, the use of propane gas instead of heavy fuel oil in heating at the underground mine of Outokumpu Chrome Kemi Mine Oy has decreased sulphur emissions from the mining complex. The particle emissions in Fig. 2 are mainly derived from the pulp mills of Stora Enso Oyj Veitsiluoto Mill and Oy Metsä-Botnia Ab Kemi Mill, and primarily consist of sodium (e. g.  $\text{Na}_2\text{SO}_4$ ) and calcium salts (e. g.  $\text{CaO}$ ) emitted from the soda recovery boilers and from the lime kilns, respectively [12, 13].

#### Sampling and analysis – statistical analyses

Scots pine (*Pinus sylvestris* L.) needles were collected at 29 sampling sites around industrial plants in the Kemi area (see Fig. 1). Two background samples were collected at Kuivaniemi, about 25 km to the south from Kemi (not shown in Fig. 1). The coordinates of the sampling sites were determined in the field by GPS (Garmin eTrex Venture CX). In this survey (2009), the sampling sites were the same as those used in our previous surveys carried out in 1979, 1989 and 1999 [5].

The study area is located in the northern part of the middle boreal vegetation zone. Sampling was carried out between 19

and 20 January 2009 according to the SFS 5669 standard [14]. Needle samples were taken at heights of 4 to 7 metres on pines of ages ranging between 50 to 100 years at each sampling site, and 5 to 10 sample trees were randomly selected per sampling site. Current (C) and previous-year (C + 1) needles were sampled at each site. After sampling, C needles were combined to give one composite sample per site. C + 1 needles were also combined to give one composite sample per site. The samples were stored in plastic bags in a freezer ( $-20\text{ }^\circ\text{C}$ ) before analysis. Unwashed needles were dried at  $40\text{ }^\circ\text{C}$  until constant weight and then milled to pass through a 0.2 mm sieve. The samples (0.5 g) were digested with 10 mL of 65% nitric acid ( $\text{HNO}_3$ ) in a CEM Mars 5 micro-process controlled microwave-oven, using CEM HP 500 Teflon vessels (CEM Corp., Matthews, USA) in accordance with the USEPA method 3051 [15]. The cooled solutions were transferred into 100 mL volumetric flasks and diluted to the mark with ultrapure water. The ultrapure water was generated by an Elgastat Maxima ion exchange water purification system (Elga Ltd., Bucks, England). All reagents and acids were suprapure or pro analysis quality. The concentrations of Na and Ca were measured with an inductively coupled plasma optical emission spectrometer (ICP-OES, Thermo Electron IRIS Intrepid II XDL, Franklin, USA).

The descriptive statistics (i. e. mean value, standard deviation, minimum-, maximum- and the 25, 50 and 75 percentile as well as the kurtosis-values) for Na and Ca concentrations (mg/kg; d. w.) in the current- (C) and previous-year (C + 1) Scots pine needles at Kemi in 2009 were made employing the Microsoft Office Excel 2007. Microsoft Office Excel 2007 was applied to study the linear regression relationship between the element concentration in C and C + 1 needles. In addition, statistical differences (i. e. a paired t-test) between Na and Ca concentrations in the current- (C) and previous-year (C + 1) Scots pine needles at Kemi in 2009 were calculated using Origin 7.5 SR6 programme.

## RESULTS AND DISCUSSION

### Na and Ca concentrations in pine needles at Kemi in 2009 and comparison with results of other studies

Na and Ca concentrations (mg/kg; dry weight; abbr., d. w.) in C and C + 1 pine needles at Kemi and in the background area at Kuivaniemi in 2009, as well as in C + 1 needles at Pietarsaari (Western Finland) in 2007 [16], at Eno (Eastern Finland) in 1994–1996 [17] and in the Jyväskylä region (Central Finland) in 2006 [18], which are areas with pulp and paper mills, are given in Table 1.

Table 1. Sodium (Na) and calcium (Ca) concentrations (mg/kg; dry weight) in current- (C) and previous-year (C + 1) Scots pine (*Pinus sylvestris* L.) needles at Kemi and in the background area at Kuivaniemi in 2009, as well as in C + 1 needles at Pietarsaari (Western Finland) in 2007 [16], at Eno (Eastern Finland) in 1994–1996 [17] and in the Jyväskylä region (Central Finland) in 2006 [18], which are areas with operating pulp and paper mills

Sampling site in Fig. 1	Na (mg/kg; d. w.)		Ca (mg/kg; d. W.)	
	C needles	C + 1 needles	C needles	C + 1 needles
6	67	160	2 080	3 510
9	22	53	2 380	3 290
10	19	51	2 400	3 600
12	50	140	2 390	3 980
14	33	62	1 730	3 100
15	13	46	2 090	3 730
16	19	40	2 450	4 110
18	7	36	2 190	3 940
20	22	36	2 300	3 140
21	32	81	2 080	3 190
22	22	45	1 890	3 230
26	46	70	2 240	3 430
27	42	45	1 980	3 840
32	18	66	2 260	3 380
33	22	51	2 360	3 650
34	74	280	1 750	3 550
35	31	95	2 020	2 960
37	23	81	2 430	3 480
38	63	110	1 410	2 120
39	23	59	2 390	4 010
50	15	72	1 680	2 770
51	78	190	3 060	5 450
52	51	100	2 430	2 830
53	43	130	2 130	3 500
60	75	83	2 570	4 180
61	67	110	1 870	3 180
62	78	91	2 110	2 790
63	80	120	1 620	2 570
64	76	110	2 290	3 400
Background	51	71	1 400	2 695
Pietarsaari (2007)			2 432–5 598	
Eno (1994, 1995, 1996)	31–105	54–151		
Jyväskylä (2006)			2 700–7 900	

In 2009, Na concentrations in pine needles in the study area varied between 7–80 mg/kg (d. w.) in C needles and 36–280 mg/kg (d. w.) in C + 1 needles, with average values of 42 mg/kg (d. w.) and 90 mg/kg (d. w.), respectively. Na and Ca concentrations at each sampling site were higher in C than in C + 1 needles.

The background area is rather distant from Kemi (about 25 km to the south), and it is therefore unlikely that emissions containing Na and Ca particles from the industrial plants located at Kemi reach this area. Although Na and Ca are also marine-derived ions (i. e. sea salt aerosols) and are present in soil dust [19], Na and Ca concentrations in the pine needles collected at Kemi are primarily attributable to emissions from the pulp mills because particulate emissions from both the recovery boilers and the lime kilns contain sodium and calcium salts [12, 13]. The Na- and Ca- containing particles emitted by the industrial plants, i. e. pulp mills, are deposited either on the needle surfaces or on the soil, from where trees take them up as nutrients [19].

We compared the concentrations of Na and Ca in pine needles from the study area to those collected in the background area. This enabled us to eliminate the insignificant effect of marine-derived ions from the Gulf of Bothnia, which in fact is only a slightly brackish water body. Furthermore, according to the bioindicator survey carried out by Viskari and Kärenlampi [20], the accumulation of sodium and calcium in needles may also be partly due to the use of deicing salts (i. e. NaCl and CaCl<sub>2</sub>) during winter. However, according to Viskari and Kärenlampi [20], Na and Ca concentrations in needles decrease rapidly on moving away from roads, and already at distances of 20 and 30 metres from a road Na and Ca concentrations in pine needles are at the background levels. The effect of wintertime deicing salt is most probably negligible because, in our study, the sampling sites were located at a distance of more than 30 metres away from roads.

The highest sodium concentrations (63 ≤ Na < 80 mg/kg; d. w.) in C needles occurred at sampling sites 63, 62, 64, 60, 34, 6 and 61 located in the northern part of the study area, as well as at sampling sites 51 and 38 located in the southern part of the study area. Sodium concentrations in C needles at these sites were ca. 24–57% higher than the corresponding background value (51 mg/kg; d. w.). The highest (100 ≤ a < 280 mg/kg; d. w.) sodium concentrations in C + 1 needles occurred at sampling sites 34, 51, 6, 12, 53, 63, 61, 64, 38 and 52. The sodium concentrations in C + 1 needles at these sampling sites were by ca. 41–294% higher than the corresponding background value (71 mg/kg; d. w.).

Taking into account the location of the emission point sources in Kemi (Fig. 1), we can conclude that sodium concentrations in both C and C + 1 needles at the northern-most sampling sites 6, 12, 53, 60, 61, 62, 63 and 64 are mostly due to emissions of particulate matter (e. g. Na<sub>2</sub>SO<sub>4</sub>) from Oy Metsä-Botnia Ab Kemi Mill. Sampling site 34 is located at the side of a road leading to the Outokumpu Chrome Kemi Mine Oy. Therefore, it is very likely that Na concentrations in both C

Table 2. Descriptive statistics and the linear correlation analysis (the Pearson's correlation coefficients) for Na and Ca concentrations (mg/kg; dry weight) in current- (C) and previous-year (C + 1) Scots pine (*Pinus sylvestris* L.) needles at Kemi in 2009 (note: the data of the background samples are not included)

Descriptive statistics	Na (mg/kg; d. w.)		Ca (mg/kg; d. w.)	
	C needles	C + 1 needles	C needles	C + 1 needles
N	29	29	29	29
Mean	42	90	2 158	3 445
Standard deviation	24	53	337	616
Min	7	36	1 410	2 120
Max	80	280	3 060	5 450
Percentile 25	22	51	1 980	3 140
Percentile 50	33	81	2 190	3 430
Percentile 75	67	110	2 390	3 730
Kurtosis	-1.41	4.80	0.93	3.18
Linear correlation analysis (The Pearson's coefficients for the correlation)				
Metal/needle	Na (C needles)	Na (C + 1 needles)	Ca (C needles)	Ca (C + 1 needles)
Na (C needles)	1	0.704*	-0.067	-0.020
Na (C + 1 needles)		1	-0.072	0.127
Ca (C needles)			1	0.766*
Ca (C + 1 needles)				1

\* Correlation is significant at the 0.01 level (2-tailed).

and C + 1 needles at this site primarily originate from road dust rather than from the mining area, even though the quarry, roads and stores at the mining area are potential emission sources [21]. Sodium concentration in C and C + 1 needles at sampling sites 38, 51 and 52 was primarily due to Na<sub>2</sub>SO<sub>4</sub> emissions from Stora Enso Oyj.

In 2009, Ca concentrations in pine needles in the study area varied between 1 410–3 060 mg/kg (d. w.) in C needles and between 2 120–5 450 mg/kg (d. w.) in C + 1 needles, the average values being 2 158 and 3 445 mg/kg (d. w.), respectively (Table 1). The average Ca concentration in C needles in the study area was by 54.1% higher than that in the background area (1 400 mg/kg; d. w.). In C + 1 needles collected from the study area, the average concentration of Na was 27.8% higher than in the background area (2 695 mg/kg; d. w.). The highest Ca concentrations in C needles (2 300 ≤ Ca < 3 060 mg/kg; d. w.) and in C + 1 needles (3 500 ≤ Ca < 5 450 mg/kg; d. w.) occurred at sampling sites close to the pulp mills of Stora Enso Oyj and Oy Metsä-Botnia Ab. Elevated Ca concentrations around the pulp mills are expected because the lime kilns of the pulp mills emit calcium in the form of, e. g., CaO.

According to the results presented in Table 1, Na concentrations in both C and C + 1 needles at Kemi in 2009 were in good agreement with the values of 31–105 mg/kg (d. w.) and 54–151 mg/kg (d. w.), respectively, observed in 1994–1996 at Eno (Eastern Finland), in an area where a pulp mill is operating [17]. According to a literature survey, there is no recent data currently available on sodium concentrations in Scots pine needles around Finnish pulp mills. This is due to the fact that the operators of pulp and paper mills are not obliged by their environmental permits to determine sodium concentrations in bioindicators. Furthermore, Ca concentrations in C + 1 needles at Kemi in 2009 were in good agreement with the values of 2 432–5 598 mg/kg (d. w.) observed at Pietar-

saari (Western Finland) in 2007 [16] and with the values of 2 700–7 900 mg/kg (d. w.) observed in the Jyväskylä region (Central Finland) in 2006 [18]. However, the highest Ca concentrations of 7 900 mg/kg (d. w.) in C + 1 needles in the latter study area was clearly higher than the corresponding value of 5 450 mg/kg (d. w.) observed in our study in 2009.

The descriptive statistics (i. e. mean value, standard deviation, minimum-, maximum-, and the 25, 50 and 75 percentile, as well as the kurtosis-values), and the linear correlation analyses (i. e. the Pearson's coefficients for the correlations) for Na and Ca concentrations (mg/kg; d. w.) in current- (C) and previous-year (C + 1) Scots pine needles at Kemi in 2009 are given in Table 2. The linear correlation between Na concentrations in C and C + 1 needles was significant ( $r = 0.704$ ;  $p < 0.01$ ; 2-tailed). In addition, the linear correlation between Ca concentrations in C and C + 1 needles was also significant ( $r = 0.766$ ;  $p < 0.01$ ; 2-tailed).

The results for sodium and calcium concentrations in the current- (C) and previous-year (C + 1) Scots pine needles were compared with a paired t-test which showed that the concentrations of Na ( $t = -6.49$ ;  $df = 28$ ;  $p = 4.9 \times 10^{-7}$ ) and Ca ( $t = -16.6$ ;  $df = 28$ ;  $p = 5.2 \times 10^{-16}$ ) were significantly different in C and C + 1 needles at Kemi in 2009. The statistical differences in the concentrations of Na and Ca in the current- (C) and previous-year (C + 1) Scots pine needles may be caused, inter alia, by the variation in the length of accumulation period and the principle of accumulation [19, 22].

## CONCLUSIONS

Due to the fact that nowadays the burning of heavy fuel oil is minimal, and as a result of the modernisation and modification of processes at the industrial plants located at Kemi, Northern Finland, the gaseous sulphur emissions at Kemi

fell almost 10-fold – from 4 502 to 485 tonnes per year in 1980–2008. Furthermore, during the period 1992–2008, particulate matter emissions decreased from 2 044 to 124 tonnes per year.

In 2009, Na concentrations in pine needles at Kemi varied between 7–80 mg/kg (d. w.) in C needles and between 36–280 mg/kg (d. w.) in C + 1 needles, whereas the corresponding background values were 51 and 71 mg/kg (d. w.), respectively. The highest Na concentrations in pine needles at Kemi were observed in the vicinity of the pulp mills. Therefore, we can conclude that Na concentrations in the pine needles are mainly due to particulate emissions of Na<sub>2</sub>SO<sub>4</sub> from the soda recovery boiler of the pulp mills. There was a significant difference between Na concentrations in the current- (C) and previous-year (C + 1) Scots pine needles at Kemi in 2009 ( $t = -6.49$ ;  $df = 28$ ;  $p = 4.9 \times 10^{-7}$ ; 2-tailed).

In 2009, Ca concentrations in pine needles at Kemi varied between 1 410–3 060 mg/kg (d. w.) in C needles and between 2 120–5 450 mg/kg (d. w.) in C + 1 needles, whereas the corresponding background values were 1 400 mg/kg (d. w.) and 2 695 mg/kg (d. w.), respectively. The highest Ca concentrations in needles were observed in the vicinity of the pulp mills. Therefore, we can conclude that Ca concentrations in pine needles are mainly due to particulate emissions of CaO from the lime kiln of the pulp mills. There was a significant difference between Ca concentrations in the current- (C) and previous-year (C + 1) Scots pine needles at Kemi in 2009 ( $t = -16.6$ ;  $df = 28$ ;  $p = 5.2 \times 10^{-16}$ ; 2-tailed).

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## PUŠIES SPYGLIAI (*PINUS SYLVESTRIS* L.) KAIP NATRIO IR KALCIO NUSĖDIMO BIOINDIKATORIUS ŠIAURĖS SUOMIJOS KEMI POPIERIAUS GAMYKLŲ APYLINKĖSE

### Santrauka

Pateikti ir aptarti palyginamieji natrio ir kalcio koncentracijų pušies (*Pinus sylvestris* L.) spygliuose keliose Suomijos vietovėse šalia popieriaus gamyklų – Kemi (Šiaurės Suomijoje), Pietarsaari (Vakarų Suomijoje), Eno (Rytų Suomijoje) ir Jyväskylä (Vidurio Suomijoje) tyrimų rezultatai.