

TRACE ELEMENT ANALYSIS OF SOUND AND DECAYED NORWAY SPRUCE (*PICEA ABIES*) BY XRF AND NAA METHODST. RAUNEMAA, P. JARTTI, A. HAUTOJÄRVI, V. LINDFORS, J. LAURÉN
and J. RÄISÄNEN

SELOSTE:

TERVEEN JA LAHON KUUSEN HIVENAINENALYYSEJÄ RÖNTGENFLUORESENSSI- JA NEUTRONIAKTIVOINTIMENETELMILLÄ

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Ashed tree samples from sound and decayed Norway spruce were studied by means of fast neutron activation analysis (neutron energy 14 MeV) and, for comparison, also by X-ray fluorescence analysis. In fast neutron activation analysis the following elements were detected: (Na), Mg, Al, Si, K, Ca, Mn, Rb, Sr and Ba, and according to the results of the X-ray fluorescence method the elements present in the wood samples were: K, Ca, Mn, Rb, Sr and Ba. A general diminishing was revealed by both methods in most elemental concentrations, with the exceptions of K and Rb, when going from a sound tree to a decayed one. The use of the ratio of the amounts of potassium to calcium as an indicator of the degree of decayedness is therefore proposed.

1. INTRODUCTION

Internal decay in growing trees, at its worst, can result in about one third of the trees in spruce forests being discarded in the timber industry. As this obviously has unfavourable economic consequences, an early indication of the presence and extent of decay in growing trees is of importance.

Of the devices which exist for measuring the internal decay, only few are completely nondestructive. Examples of the non-destructive methods are those using ultrasonic equipment or X-ray photography. These methods, however, have serious disadvantages, as they are for instance not

reliable and their sensitivity depends on the thickness of the tree's bark.

Other methods are therefore being looked into for measuring the condition of trees. A large selection of nuclear methods has been utilized during recent years in the investigation of various environmental problems. Amongst the methods using radiation is activation analysis by neutrons, which is worth examining in the context of tree inspection. In this method neutrons are used to excite characteristic radioactivity in the sample nuclei by nuclear reactions and the radioactivity is measured by radiation detectors. This radioactivity reveals

the elements present in the sample. If the chemical composition correlates with the state of the tree, activation analysis by neutrons could serve as a rapid inspection method. Fast or thermal neutrons can be employed for the activation; in this work fast neutrons were employed.

In order to compare the effectiveness of neutron activation in elemental analysis another nuclear method was also checked. Thus tree samples were studied using the X-ray fluorescence method, whereby characteristic X-rays of the elements in the sample are excited by gamma radiation or by energetic, charged particles. In the excitation process vacancies in the inner orbitals of the sample atoms are first created, and as these vacancies are immediately filled by a series of electron transitions, emission of X-ray photons follows.

These X-rays can be identified by suitable radiation detectors. In this work gamma and X-ray radiation from long lived radioisotopes were employed for to create the excitation.

Technical details of these two independent investigations and the results obtained for elemental concentrations in sound and decayed tree samples are presented herein. The spruce samples for the examinations were cut from spruce forests in Virrat, Central Finland.

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2. EXPERIMENTAL TECHNIQUES

2.1. Sample characteristics

Samples of decayed and sound spruce were burned to ash at low temperature (about 200-300 C) and the cooled ash strained with a 0.5 mm sieve.¹⁾ For use in the neutron activation approximately half-gram samples of ash were weighed accurately and put in small polyethylene irradiation boxes. The area of the prepared samples was 3.61 cm² with a thickness of about 1 mm. As this sample thickness (~1 mm) does not affect much the penetration of energetic neutrons (in our case 14 MeV energy) in the samples, no serious absorption problems arise. This makes the use of neutrons in the decay studies favourable.

Almost all γ -rays used in identification

of elements in neutron activation analysis appear at energies above 200 keV (in this study all of them), which also means that γ -ray absorption is negligible. The maximum attenuation of γ -rays of 100 keV and 500 keV energy would in 1 mm of carbon ($Z = 6$) sample be 3 per cent and 2 per cent, respectively. In a sample consisting predominantly of potassium ($Z = 19$) the maximum attenuation would be 2 and 0.7 per cent, respectively. In our case then, about 97 per cent of the γ -radiation is left unabsorbed in the sample material.

For the analysis by X-ray fluorescence, thin pellets of 1 mm · 3.14 cm² size were prepared from the ash by using a pressure of 4500 kg/cm². In X-ray fluorescence analysis the sample thickness (~1 mm) has a more pronounced effect, the extent of which depends on the energy utilised for excitation as well as on the energy of the radiation excited. The attenuation of 5.9 keV X-rays (i.e. the exciting radiation from a Fe-55 source) is 91 per cent and that of 59.6 keV γ -rays (the exciting radiation from an Am-241 source) is 3 per cent in 1 mm of carbon sample. Respectively, ~ 100 and 5.5 per cent attenuation would result in 1 mm thick samples

¹⁾ As it was considered possible that quantities of chemical compounds of some elements might have been evaporated during the ashing process, which could lead to erroneous conclusion as to the mineral contents, the melting and boiling points of the most probable mineral compounds were checked. Those were found to be too high to allow vaporisation at the low burning temperatures used.

of potassium. Absorption effects must therefore be carefully taken into account when X-rays are used for identification. Only in the case of thin samples, such as air particulate matter with a few tenths of milligrams per square centimeter on thin filters, can the absorption be almost negligible. Even then other factors such as particle size effects might be seriously involved (RÄISÄNEN et al. 1978), and the sample composition should be equally accurately known.

2.2. Sample analysis

2.2.1. Neutron activation

In neutron activation analysis the samples were irradiated for 15 min with 14–15 MeV neutrons from a SAMES T 400 neutron generator at the Department of Physics, University of Helsinki. The average neutron flux used was $5 \cdot 10^9$ n/cm²·s. Shorter irradiations were performed in identifying short lived activities. After irradiation the samples were removed from the irradiation position and transported manually to the measuring apparatus. The transport time from irradiation to measurement was about one minute. According to the present results a 5 minute irradiation should be enough to enable the same identification procedure as employed here. Four to five samples could be irradiated simultaneously, but usually limitations in detecting capacity make this impracticable.

The gamma spectra were measured by using a coaxial 43 cm³ Ge(Li) semiconductor detector and a 4 K ADC connected to the PDP 9/L computer of the Department of Physics, University of Helsinki. An experimentally determined response curve for the efficiency to detect γ -rays of different energy was used. The experimental detector resolution was 3 keV for 1333 keV ⁶⁰Co γ -rays. Standard IAEA sources and a ¹⁵²Eu source were used for energy calibration. Measuring times were 5 and 15 minutes, the former starting about one minute after irradiation, and the latter one minute after the first measurement.

In routine work only one measurement per sample is needed and can be performed during the first five minutes. Sample

measurements can thus be performed in a 7–10 minute sequence, which means that approximately 50 samples can be investigated in one day.

2.2.2. X-ray fluorescence

In the X-ray fluorescence method the sample measurements each took 4 minutes in the XRF spectrometer facility at the Department of Physics, University of Helsinki. Characteristically for this method, excitation and detection were made simultaneously. As no separate irradiation and detection periods are needed, noticeable reduction in the investigation time results.

Each sample pellet to be analysed was placed on thin mylar foil supported by a metal ring. X-ray spectra of the samples were measured using a 5 mm thick intrinsic germanium X-ray detector with 25 mm² sensitive area. The energy resolution of this detector for Mn KX-rays was 150 eV. A module based pulse processing system and a 4096 channel pulse height analyser with paper and magnetic tape registration devices were used in data collection. These instruments are routinely employed in nuclear spectroscopy. When using the Fe-55 excitation, the low energy X-radiation can best be detected in vacuo. A brief description of the spectrometer characteristics is given below with a more detailed account reported elsewhere (RAUNEMAA et al. 1978).

The XRF spectrometer employed in this study had been constructed specially for measuring environmental samples. The radiations used for the excitation of X-rays

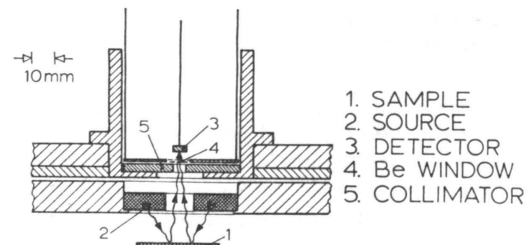


Figure 1. An overview of the spectrometer.
Kuva 1. Spektrometrin toimintaperiaate.
1. Näyte 2. Lähde 3. Ilmaisim 4. Be-ikkuna
5. Rajoitin.

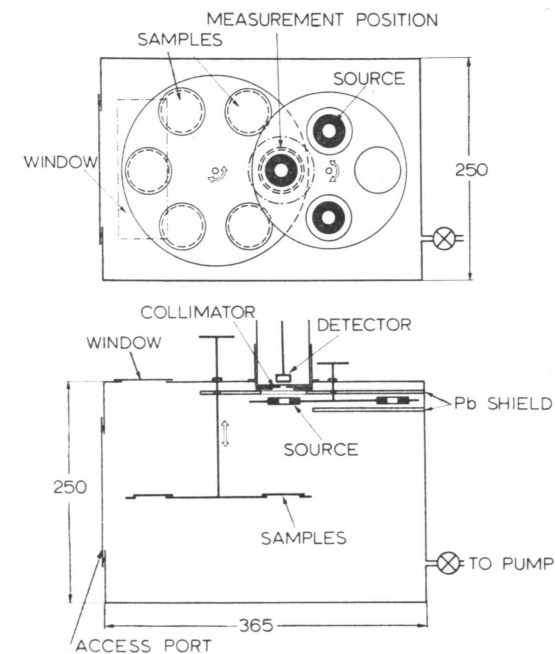


Figure 2. The fluorescence chamber characteristics. The dimensions are given in mm.
Kuva 2. Mittauskammion rakenne ylhäältä ja sivulta.

originate from radioisotopes of Fe-55, Cd-109 and Am-241. In this study only the Fe-55 and Am-241 sources were used. The

sources, which have annular shape, have an activity level of 1–4 GBq, i.e. they show about 10^9 decays/s.

Figure 1 presents an overview of the experimental arrangement of the spectrometer with the sample, radioisotopic source and radiation detector.

The samples, the sources and the detector are enclosed in a «fluorescence» chamber shown in Figure 2. The chamber is vacuum-tight and can be evacuated or filled with a chosen gas. The chamber material is aluminum, as this material does not cause interferences in the measurements.

An efficiency calibration of the detector, which is usually necessary, can be avoided by using single element standards (GIAUQUE et al. 1975).

2.2.3. Data manipulation

Data manipulation was carried out using the computer program VIPUNEN (PAATERO 1977) in a Burroughs 6700 computer. A least-squares fit of a polynomial to calibration points yields in this program the energy-channel response, which is used to calculate the needed information i.e. the energies and intensities. The program is constructed useroriented and can very well be adapted to analysis of any spectroscopic data.

3. RESULTS

3.1. Neutron activation analysis

Characteristic gamma spectra resulting from neutron activation of sand and decayed tree ash samples, respectively, prepared as described above, are reproduced in Figure 3. Reaction channels, reaction cross sections and characteristics of the reaction products used in elemental identification are presented in Table 1.

To determine the relative abundances of different elements in the samples one has to calculate normalised intensities for peaks in the spectra. The number of atoms N_j of element j in the sample is then

$$N_j = \frac{\lambda \Delta N e^{\lambda t_w}}{\sigma \phi} (1 - e^{-\lambda t_a})^{-1} (1 - e^{-\lambda t_m})^{-1}$$

λ = radioactive decay constant = $\ln 2/t_{1/2}$
 ΔN = intensity of the gamma peak
 ϕ = neutron flux
 σ = reaction cross section
 $t_{1/2}$ = radioactive half-life of the reaction product
 t_w = waiting time
 t_a = activation time
 t_m = measuring time

The photopeak efficiency of the gamma detector, the variation of the neutron flux and the differences in dry weights of the sam-

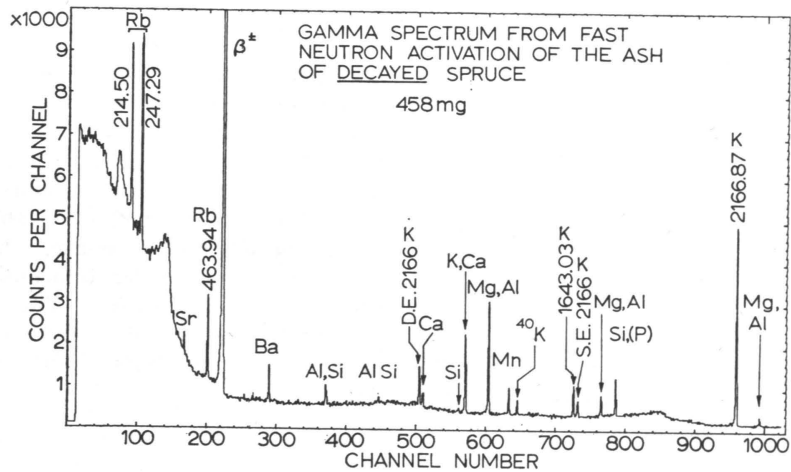
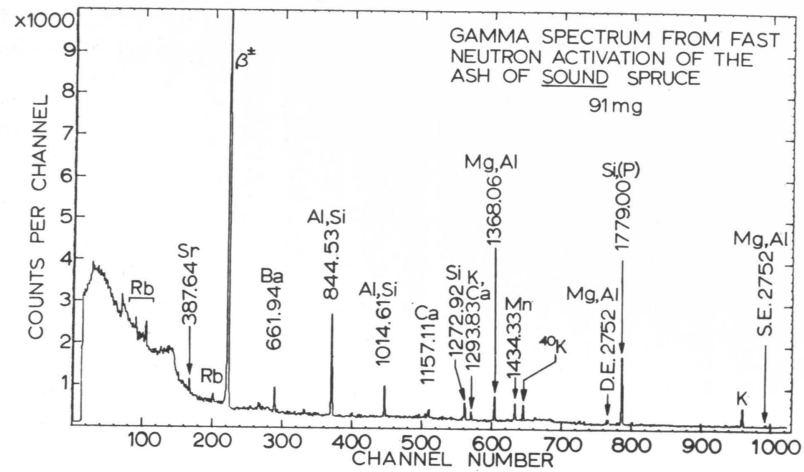


Figure 3. Gamma spectra from fast neutron activation of the ash of sound and decayed spruce samples. Gamma-ray energy and/or the element in question are shown by the keV value and/or the chemical symbol, respectively, in each relevant case.

Kuva 3. Gammaspektrit terveestä (yllä) ja lahosta (alla) kuusinäytteestä neutroniaktiivointimenetelmällä. Gammasäteilyn energia-arvot ja piikkien alkuperä on merkitty kuvioon.

ples were taken into account via correction factors. By using the reaction cross sections (LEPPÄMÄKI and KANTELE 1970) and half-lives of the reaction products (TANNILA and KANTELE 1969), the relative abundances of the elements were finally found. The results presented in Table 2 correspond to the data collected in the 15 min measurement periods. The values obtained are

compatible as between sound and decayed trees and within the two groupings.

In calculating the errors of relative abundances, two main error sources should be mentioned: the errors in peak areas, as given by the error analysis of the computer program VIPUNEN, and the uncertainties in the timing. For the waiting time a rather large uncertainty of ± 0.5 min

Table 1. Identification data used in neutron activation analysis.
Taulukko 1. Tunnistukseen käytetty aineisto neutroniaktiivointianalyysissä.

Product nucleus Tuoteydin	Half-life Puoliintumisaika	Main γ -ray Energies Intensities γ -säteilyn Energiat Intensiteetit		Reaction Ydinreaktio	Abundance Runsaus %	Cross Section Vaikutusala mb
		keV				
^{23}Ne	37.6 s	440	0.33	$^{23}\text{Na}(n,p)$	100	40
^{24}Na	15.03 h	1 368.5	1.00	$^{24}\text{Mg}(n,p)$	79	170
		2 753.9	1.00	$^{27}\text{Al}(n,\alpha)$	100	114
^{27}Mg	9.45 m	843.8	0.72	$^{27}\text{Al}(n,p)$	100	83
		1 014.4	0.28	$^{30}\text{Si}(n,\alpha)$	3	86
^{28}Al	2.24 m	1 778.9	1.00	$^{28}\text{Si}(n,p)$	92	250
				$^{31}\text{P}(n,\alpha)$	100	125
^{29}Al	6.52 m	1 273.3	0.94	$^{29}\text{Si}(n,p)$	5	92
^{38}Cl	37.18 m	2 167.5	0.55	$^{41}\text{K}(n,\alpha)$	7	35
		1 642.4	0.40			
^{41}Ar	1.82 h	1 293.6	0.99	$^{44}\text{Ca}(n,\alpha)$	2	35
				$^{41}\text{K}(n,p)$	7	74
^{38}K	7.71 m	2 166.8	1.00	$^{39}\text{K}(n,2n)$	93	5.1
^{44}K	22.15 m	1 156.9	0.58	$^{44}\text{Ca}(n,p)$	2	42
		2 151.3	0.23			
^{52}V	3.76 m	1 433.9	1.00	$^{55}\text{Mn}(n,\alpha)$	100	39
				$^{52}\text{Cr}(n,p)$	84	110
$^{84\text{m}}\text{Rb}$	20.40 m	247.9	0.65	$^{85}\text{Rb}(n,2n)$	72	640
		215.4	0.37			
		463.7	0.32			
$^{86\text{m}}\text{Rb}$	1.02 m	556.1	0.98	$^{87}\text{Rb}(n,2n)$	28	930
$^{87\text{m}}\text{Sr}$	2.81 h	388.4	0.89	$^{88}\text{Sr}(n,2n)$	83	200
$^{137\text{m}}\text{Ba}$	2.55 m	661.6	0.85	$^{138}\text{Ba}(n,2n)$	72	1250

(i.e. 8 per cent) was taken, as this time was not regularly checked. Fairly large errors therefore result in the abundances when short lived reaction products (eg. ^{137}Ba) are considered. In the analysis based on very long lived activities (eg. $^{87\text{m}}\text{Sr}$) rather large errors are due to the short irradiation-measurement sequence.

The following elements were detected in fast neutron activation analysis (NAA): (Na), Mg, Al, Si, K, Ca, Mn, Rb, Sr and Ba. Furthermore the presence of P is probable, but due to interfering activities was difficult to resolve unambiguously.

3.2. X-ray fluorescence analysis

X-ray spectra resulting from excitation using Fe-55 and Am-241 sources are presented in Figures 4 and 5.

In calculating the abundances of different elements in the samples the KX-line intensity was used. This intensity depends on the flux of the exciting radiation (I_0), the cross section for production of photoelectrons (τ), the probability for de-excitation by X-rays ($\omega = \omega_K$) and on the weight fraction (W) of the fluorescent elements in the sample. The exciting as

Table 2. Relative number of atoms of elements and their ratios in sound tree ash sample (1) and decayed tree ash sample (5) normalised to equivalent dry weight.

Taulukko 2. Samaan kuivapainoon normitetut alkuaineiden suhteelliset määrät ja niiden suhde terveessä ja lahossa puussa.

Stable isotope Alkuaine- isotooppi	Reaction used in identification Ydinreaktio	Relative number of atoms of elements Alkuaineatomien määrä		Ratio Suhde N(D)/N(S)
		Sound tree Terve puu N(S)	Decayed tree Laho puu N(D)	
²⁴ Mg	(n,p)	18.3 ± 0.9	16.0 ± 0.4	0.87 ± 0.07
²⁷ Al	(n,α)	3.61 ± 0.16	0.155 ± 0.003	0.043 ± 0.003
²⁷ Al	(n,p)	3.5 ± 0.2	0.16 ± 0.02	1.146 ± 0.008
²⁸ Si, ³¹ P	(n,p), (n,α)	21 ± 4	1.8 ± 0.4	0.09 ± 0.04
²⁹ Si	(n,p)	1.8 ± 0.2	0.05 ± 0.02	0.030 ± 0.016
³⁰ Si	(n,α)	1.20 ± 0.07	0.032 ± 0.005	0.027 ± 0.006
³⁹ K	(n,2n)	11.1 ± 1.0	21.9 ± 1.3	2.0 ± 0.3
⁴¹ K	(n,α)	0.84 ± 0.20	1.75 ± 0.09	2.1 ± 0.6
⁴⁴ Ca	(n,p)	1.25 ± 0.16	0.35 ± 0.04	0.28 ± 0.07
⁵⁵ Mn	(n,α)	7.8 ± 1.3	1.8 ± 0.3	0.23 ± 0.08
⁸⁵ Rb	(n,2n)	0.022 ± 0.007	0.040 ± 0.006	1.8 ± 0.5
⁸⁸ Sr	(n,2n)	0.18 ± 0.05	0.076 ± 0.012	0.42 ± 0.20
¹³⁸ Ba	(n,2n)	0.27 ± 0.06	0.08 ± 0.02	0.29 ± 0.13

Note: Reaction ²³Na(n,p)²³Ne is not included here because of the short half-life of the reaction product. Error values shown are due to statistical and systematic errors in the measurements of only one sample, the sound tree sample (1) and the decayed tree sample (5), respectively.

Huomautus: Natriumin tunnistamiseen on käytetty reaktiota ²³Na(n,p)²³Ne. Virhearviot sisältävät statistiset ja systemaattiset mittausvirheet.

well as the fluorescent radiation are influenced by the sample matter, due to absorptive processes. The X-ray intensity is therefore decreased by a factor F, the attenuation factor, given by

$$F = \frac{1 - e^{-(\mu + \mu')m}}{(\mu + \mu')m}$$

where μ and μ' are the mass attenuation coefficients for exciting and fluorescent radiation, respectively, and m is the mass of the sample (g/cm²).

Fluorescent intensity I_j for a homogeneous sample can now be expressed in the form (RÄISÄNEN et al. 1978).

$$I_j = \frac{I_0 G \tau \omega W}{\mu + \mu'} (1 - e^{-(\mu + \mu')m}) \mu$$

where the geometry factor G takes into account the differences in experimental arrangements. From this equation the amount of an element j , $m_j = W \cdot m$, in the sample can be determined. The values of ω , τ for different elements can be found in the literature (BAMBYNEK et al. 1972, VEIGELE 1973, FREUND 1975).

If the factor $(\mu + \mu')m$ is ≥ 4 in the intensity equation, the sample is said to be thick, and total absorption is at least 75 per cent. If, on the other hand, total absorption is less than 5 per cent, the sample is said to be thin with respect to that radiation. The thin sample criterion is in the present work true only for BaKX-rays when using Am-241 excitation. The thick sample criterion is correspondingly fulfilled only for KX-rays of Mn or of any lighter element. The elements in between Mn and

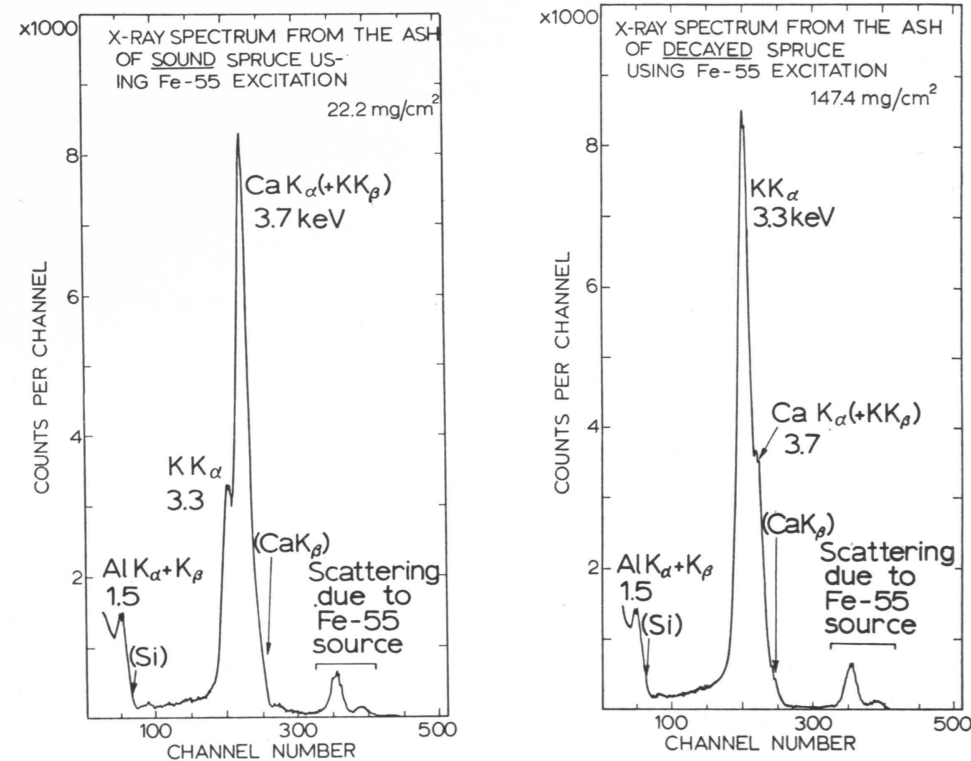


Figure 4. X-ray spectra from the ash of sound and decayed spruce samples obtained by XRF method. Fe-55 excitation.

Kuva 4. Röntgenspektrit terveestä (vasemmalla) ja lahosta (oikealla) kuusinäytteestä röntgenfluoresenssimenetelmällä. Fe-55 viritys.

Table 3. Attenuation factor F using Fe-55 excitation. Taulukko 3. Vaimennuskertoimen F arvot Fe-55 virityksessä.

Sample - Näyte Origin - Alkuperä	Mass Massa (mg/cm ²)	Fluorescent radiation Röntgensäteily	
		Ca E _{KX} = 3.7 keV	V E _{KX} = 4.8 keV
Sound (1) - Terve	22.2	F = 0.278	F = 0.402
Decayed (5) - Laho	147.4	F = 0.043	F = 0.068

Ba fall within these extreme cases, and must be considered with more care.

The attenuation factors F for Am-241 excitation, as applied to the samples studied in this work, are illustrated in Figure 6 by the solid curves drawn through the points calculated for some elements. These calcu-

lations are based on the mass absorption coefficients for the most probable tree ash sample compositions.

When one uses the Fe-55 excitation, the theoretical «thick sample» criterion is always fulfilled. The values of the attenuation factors for the fluorescent radiations of Ca

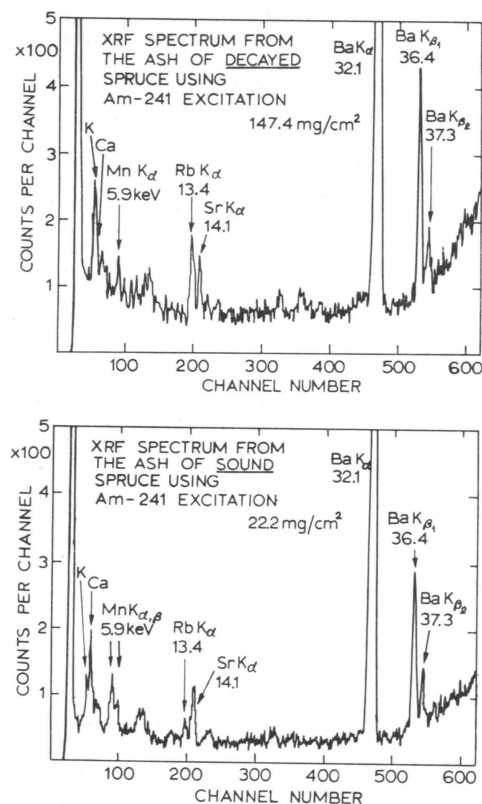


Figure 5. X-ray spectra from the ash of sound and decayed spruce samples obtained by XRF method. Am-241 excitation.

Kuva 5. Röntgenspektrit terveestä (alla) ja lahosta (yllä) kuusinäytteestä röntgenfluoresenssimenetelmällä. Am-241 viritys.

Table 4. The amounts of different elements in sound (1) and decayed (5) tree ash samples.

Taulukko 4. Alkuaineiden pitoisuudet terveessä ja lahossa kuusinäytteessä.

Element Alkuaine	Fe-55 Excitation Fe-55 viritys		Am-241 Excitation Am-241 viritys			
	K (%)	Ca (%)	Mn (%)	Rb (ppm)	Sr (ppm)	Ba (ppm)
Sound (1) Terve	6.6 ± 0.7	7.5 ± 0.8	0.9 ± 0.3	310 ± 50	970 ± 120	3700 ± 100
Decayed (5) Laho	13.2 ± 1.3	2.7 ± 0.3	0.3 ± 0.1	350 ± 110	190 ± 75	790 ± 65
Decayed — sound Laho — Terve	2.0 ± 0.3	0.36 ± 0.05	0.33 ± 0.15	1.1 ± 0.4	0.20 ± 0.08	0.21 ± 0.02

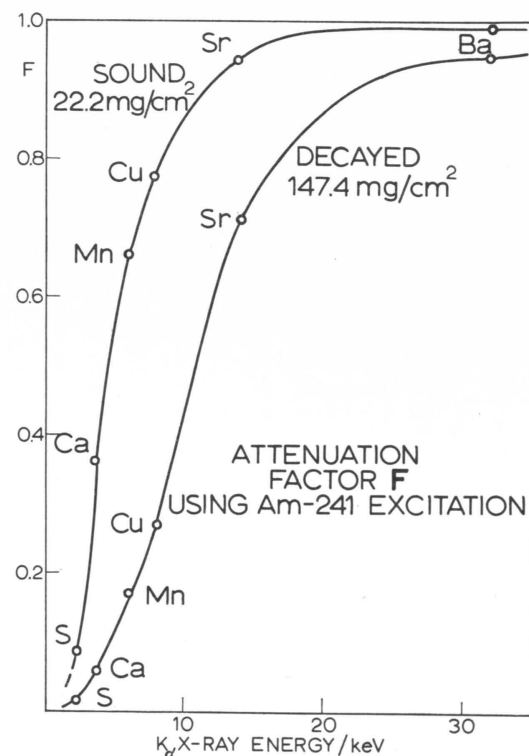


Figure 6. Attenuation factor using Am-241 excitation.

Kuva 6. Vaimennuskertoimen Am-241 viritystä käytettäessä.

and V excited by the Fe-55 radiation are given in Table 3.

The quantitative results of the X-ray fluorescence analysis for the samples (1), sound tree, and (5), decayed tree, are presented in Table 4. The error limits were calculated by taking into account uncertainties in standardisation, in geometry and in absorption correction. Statistical errors of the peak areas, as given by the computer program VIPUNEN, are included. Differences in absorption correction as various thicknesses and compositions of sample (due to developments in the ashing technique) were also considered.

The quantitative values are based on single element standards which are normally used in our laboratory for thin sample determinations. Systematic errors may thus exist, but on the relative abundances (at least of neighbouring elements) and on the decayed to sound ratio this will have only a minor effect.

The elements K, Ca, Mn, Rb, Sr, and Ba were found to be present in the ashed tree samples, according to the results of the X-ray fluorescence analysis (XRF). In addition, trace amounts of the elements Al, Ti and probably Cr were seen. Short investigation periods (4 min) and relatively thick samples (tenths of mg/cm²) prevented clear detection of other possible elements.

3.3. Comparison of the results obtained with NAA and XRF methods

The results obtained by NAA and XRF are relatively consistent with each other in that which concerns the number of elements found and their relative amounts. Matrix effects which are of importance in XRF applied on thick sample studies can affect systematic differences between the NAA and XRF results. Both methods reveal a general diminishing in most elemental concentrations in going from a sound tree to a decayed one. The striking exceptions, the elements K (potassium) and Rb (rubidium), whose concentrations increase, were also similarly obtained by both methods.

In both investigations roughly equal time sequences were employed in the analysis. Experimentally, routine analysis is much faster and easier to accomplish with XRF than with NAA. Other factors, however, limit the usefulness of XRF.

Of the main elemental components in Norway spruce (JALAVA 1952), i.e. calcium, manganese, potassium, magnesium, silicon, sulphur, phosphorus, and sodium, the presence of all elements other than sulphur and phosphorus was distinctly revealed in fast neutron activation analysis. In X-ray fluorescence analysis, the lightest elements

Table 5. Relative amounts of potassium and calcium in sound (1) and decayed (5) tree ash samples by different methods.

Taulukko 5. Kaliumin ja kalsiumin suhteelliset määrät terveessä ja lahossa kuusinäytteessä eri menetelmillä.

		NAA	
Sound (1) — Terve	K	12.0 ± 1.4	$\frac{(K)}{(Ca)} = 0.19 \pm 0.04$
	Ca	63 ± 8	
Decayed (5) — Laho	K	24.3 ± 1.0	$\frac{(K)}{(Ca)} = 1.4 \pm 0.2$
	Ca	17 ± 2	
		XRF	
Sound (1) — Terve	K	6.6 ± 0.7	$\frac{(K)}{(Ca)} = 0.9 \pm 0.2$
	Ca	7.5 ± 0.8	
Decayed (5) — Laho	K	13.2 ± 1.3	$\frac{(K)}{(Ca)} = 4.9 \pm 1.0$
	Ca	2.7 ± 0.3	

TANNILA, O. & KANTELE, J. 1969. Table of Principal Gamma-Rays from Radioactive Species ($T_{1/2} > 1$ ms) Arranged According to Increasing Energy. Research Report No. 3/1969. Department of Physics. University of Jyväskylä.

VEIGELE, WM. J. 1973. Photon Cross Sections from 0.1 keV to 1 MeV for Elements $Z = 1$ to $Z = 94$. Atomic Data. Vol. 5 No. 1: 51-111.

SELOSTE:

TERVEEN JA LAHON KUUSEN HIVENAINENANALYYSEJÄ RÖNTGEN- FLUORESENSSI- JA NEUTRONIAKTIVOINTIMENETELMILLÄ

Tutkimuksessa on selvitetty neutroniaktivointianalyysin (NAA) ja röntgenfluoresenssianalyysin (XRF) käyttöä kuusipuun lahoisuuden mittaamiseen. Pääosa tutkituista puunäytteistä oli peräisin kuusimetsiköstä Virroilta, Keski-Suomesta. Näytteet poltettiin tuhkaksi ja tuhka jauhettiin.

NAA-analyysissä säteilytettiin n. 0,5 g:n tuhkanäytteitä nopeilla 14 MeV:n neutroneilla 15 minuutin ajan, minkä jälkeen syntynyt gamma-säteily mitattiin koaksiaalisella 43 cm³:n (GeLi) puolijohdeilmäsimellä. Säteilytykset suoritettiin Helsingin yliopiston fysiikan laitoksen neutronigenaattorilaboratoriossa. Mittausajat vaihtelivat 5 ja 15 min välillä. Tyypilliset mittauksissa havaitut spektrit terveestä ja lahosta puunäytteestä on esitetty kuvassa 3. Alkuaineiden tunnistamisessa käytetyt tiedot on luetteloitu taulukossa I. Neutroniaktivointianalyysissä löydettiin tutkittujen puiden tuhkasta seuraavia alkuaineita: (Na), Mg, Al, Si, K, Ca, Mn, Rb, Sr ja Ba. Näiden alkuaineiden suhteelliset osuudet terveessä ja lahossa puussa on esitetty taulukossa II.

Röntgenfluoresenssianalyysiä varten puristettiin jauhetusta tuhkasta n. 1 mm:n paksuisia tabletteja. Analyysissä käytetty energiadiispersiivinen laiteisto, joka on rakennettu Helsingin yliopiston fysiikan laitoksella, on esitetty kuvassa 2. Röntgensäteilyn virittämiseen käytettiin radioaktiivisia isotooppeja Fe-55, Cd-109 ja Am-241. Säteilyn ilmaisu suoritettiin Ge-röntgenilmaisimella, jonka energian erotuskyky oli noin 150 eV mangaanin röntgenenergialle. Röntgenspektrit Fe-55 ja Am-

241 virityksillä on esitetty kuvissa 4 ja 5. Röntgenfluoresenssianalyysin tulosten perusteella puun tuhkassa on seuraavia alkuaineita: K, Ca, Mn, Rb, Sr ja Ba. Näiden alkuaineiden prosentuaaliset osuudet terveessä ja lahossa puussa on esitetty taulukossa IV.

Tuloksia vertailtaessa havaitaan eri menetelmillä saatujen tulosten olevan sopusoinnussa keskenään. Molemmilla menetelmillä voitiin selvästi todeta samansuuntaiset alkuainekonsentraatioiden muutokset siirryttäessä terveestä puusta lahoon puuhun. Kun pitoisuudet yleensä pienenevät muille alkuaineille, muodostavat jaksollisen järjestelmän ryhmän I alkuaineet K ja Rb poikkeuksen niiden määrien lisääntyessä. Pitoisuuksien suhteellista muutosta on pyritty selvittämään vertaamalla kalium/kalsium -suhdetta sekä terveessä että lahossa puussa (taulukko V). K/Ca -suhteen voidaan todeta voimakkaasti suurenevan puun lahoessa. Näin ollen saattaisi olla mahdollista käyttää K/Ca-suhdetta puun lahoisuusasteen määrittämiseen.

Kun esimerkiksi tyvilahon eteneminen halutaan määrittää kasvavasta puusta, on NAA-menetelmä suositeltavampi kuin XRF-menetelmä, sillä röntgensäteily vaimenee jo puun kuorikerroksessa. NAA-menetelmästä on mahdollista kehittää kenttäkäyttöön soveltuva versio; laitteiston muodostaisivat pienikokoinen kannettava neutronilähde ja tuikeilmäisin lisälaitteineen. Ilmaisu olisi mahdollista välittömästi mittaushetkellä.