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and its Impact on Environment

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# Norwegian Journal of Agricultural Sciences

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Sulphur – Plant Nutrient and its Impact on Environment

Proceeding from a NJF seminar at Moss, Norway  
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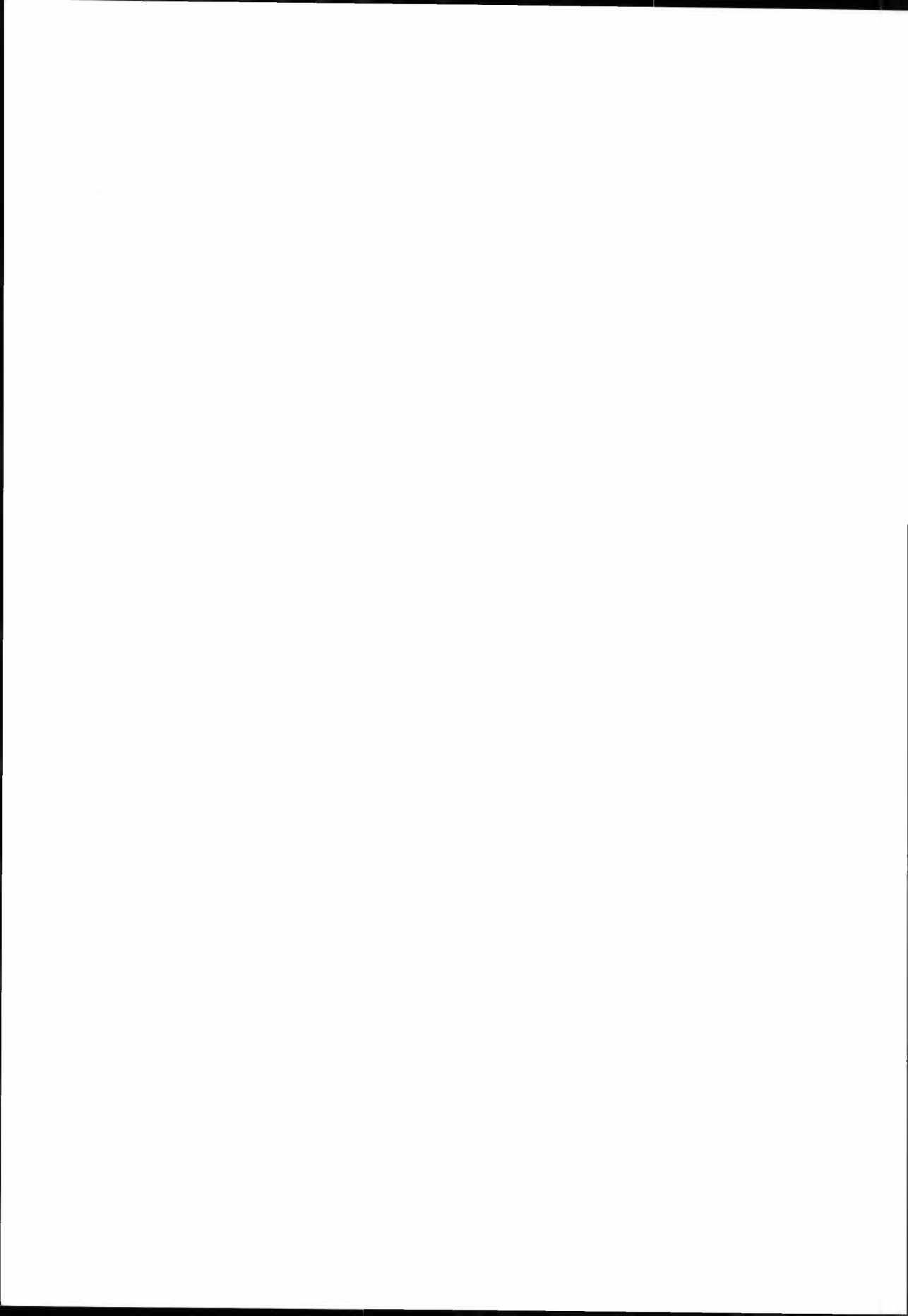
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## Preface

Sulphur has been known as an essential plant nutrient for more than 100 years but greater attention to it was paid only in the early part of this century. Sulphur plays many important roles in plant's biochemistry and is essential for a number of enzymes, vitamins and other important compounds in the plant. Therefore not only the crop yield but also the crop quality is declined when plant suffers with sulphur deficiency.

In the last two decades, sulphur received greater attention as an environmental pollutant to terrestrial and aquatic ecosystems but its role as a plant nutrient was generally ignored. However, the increasing number of cases of sulphur deficiency in field crops in the last few years in several Scandinavian countries have again demanded our attention towards its role as a plant nutrient. Therefore the theme chosen for this seminar "Sulphur – Plant Nutrient and its Impact on Environment" was considered very appropriate and timely under the prevailing conditions in northern Europe and especially in Scandinavia. In northern Europe, the atmospheric deposition of sulphur increased consistently over the years up to late 1970's when stringent regulations for sulphur emission from industry were enforced in many countries. This resulted in reduced atmospheric deposition of sulphur and the amounts deposited were not sufficient to fulfil the requirements of sulphur for plant growth. In many countries increasing crop production, greater use of chloride based commercial fertilizers and greater losses of sulphur from anaerobically stored animal manure slurry have also resulted in reduced input of sulphur to agricultural crops.

The main objectives of this seminar were three fold:

1. To gather the existing knowledge on various aspects of sulphur nutrition of crop plants and on its ecological effects.
2. To identify gaps in our knowledge and prioritize needs to initiate new research programs on sulphur.
3. To come out with new ideas for future research on different aspects of sulphur either related to crop nutrition and its quality or related to environmental pollution.

Although the seminar was originally intended to be only for Nordic countries but the interest and willingness of the scientists from other countries to participate in the seminar made it more of an international seminar. Our guest speakers from Germany, Scotland, USA and Australia generously contributed to the success of this seminar and they deserve our appreciation and thanks for their contribution and for sharing their experience with us.

Dr. Donald Messick of The Sulphur Institute in Washington, USA kindly agreed to support financially the publication of the proceeding of this seminar in the Norwegian Journal of Agricultural Sciences and the support is gratefully acknowledged.

The continuous assistance from the members of the scientific committee and the staff of the NJF Secretariat during the planning and execution of the seminar made my task easier and I would like to convey my sincere thanks to them. The Department of Soil and Water Sciences

of the Agricultural University of Norway allowed me to freely use their physical and secretariat facilities and deserves our appreciation. My thanks also go to Mrs. Liv Grønlund and Mrs. Torild Grønneflåta for their immense support during the execution of the seminar.

April 1994

Bal Ram Singh, Editor

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# Plant nutrient sulphur: A global review of crop requirements, supply, and environmental impact on nutrient balance

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Agriculture worldwide faces several great challenges. In some regions, production must be expanded to provide food for growing populations, while in others current production levels have to be maintained while striving for the right balance between intensive agriculture and environmental concerns. Plant nutrient sulphur (PNS) will be of growing worldwide importance as food production continues to increase while overall sulphur inputs tend to diminish. Sulphur (S) is one of at least 16 elements essential for plant growth. Its functions within the plant are closely related to those of nitrogen and the two nutrients are synergistic. Sulphur is required for plant growth in quantities equal to and sometimes exceeding those of phosphorus. Sulphur has a variety of vital functions within the plant's biochemistry. It is a major constituent of amino acids, such as cysteine and methionine, the building blocks of proteins. It is also essential in the formation of enzymes, vitamins, such as biotin and thiamine, and a variety of other important compounds in the plant, including chlorophyll. When S is deficient, both yield quality and quantity suffer. Plants that are S-deficient are characteristically small and spindly. The younger leaves are light green to yellowish and in the case of legumes, nodulation of the roots is reduced. The oil content of seeds is diminished and the maturity of fruits is delayed in the absence of adequate S (Buckman & Brady 1969). Increasing crop production, reduced sulphur dioxide (SO<sub>2</sub>) emissions, and shifts in major fertilizer sources have led to worldwide increases of documented S deficiencies. Despite the vital role of S, most of the growth in fertilizer consumption has been essentially in S-free nitrogen and phosphorus fertilizers, even though higher yields of crops per unit area are removing greater quantities of S from the soil. Obviously, much of this S must be replaced or S deficiencies will severely limit agricultural production (Morris 1987).

Key words: Crop S-requirements, nutrient balance, sulphur, S-supply

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## CHANGES IN AGRICULTURE AND INCREASING SULPHUR DEFICIENCIES

The agronomic factors which make PNS important have held true for many years. However, the world's dramatic political, economic, and environmental changes over the last few decades have significantly impacted today's modern agriculture. In recent years, interest in S as a plant

nutrient has increased considerably. S nutritional deficiencies are occurring with greater frequency and in more locations throughout the world. About 20 years ago, only 36 countries reported S deficiencies. In the 1980s, this number exceeded 70 countries and this trend is expected to continue through the 1990s (Morris 1988). The increased occurrence of S deficiencies worldwide can be attributed primarily to: (1) Increased use of high analysis fertilizers containing little or no S; (2) Multiple cropping and higher yielding crops, which remove greater amounts of S from the soil; (3) Greater control of industrial emissions of S combined with the decreased use of high S fuels; (4) Decreased use of S in pesticides; (5) Declining reserves of soil S; and (6) Use of crop residues for feed and fuel.

### Fertilizer use

As shown in Fig. 1, over the past 20 years, the amount of S consumed in all fertilizers worldwide has remained about 10 million tons per year. However, nitrogen consumption has doubled during this period approximately from 40 million tons in 1971 to about 80 million tons in 1990. The increased consumption of S-free, high-analysis fertilizers is one of the most significant causes of S deficiency. When single superphosphate and ammonium sulphate were more prominent in the phosphorus and nitrogen fertilizer markets, farmers were applying a significant amount of S, in many cases without realizing it. In fact, single superphosphate contains 12% S and ammonium sulphate contains 24% S. However, with increased popularity of materials such as urea, triple superphosphate, and ammoniated phosphates, world agriculture has witnessed a drastic reduction in S additions as a percentage of the total fertilizer applied (Fig. 2).

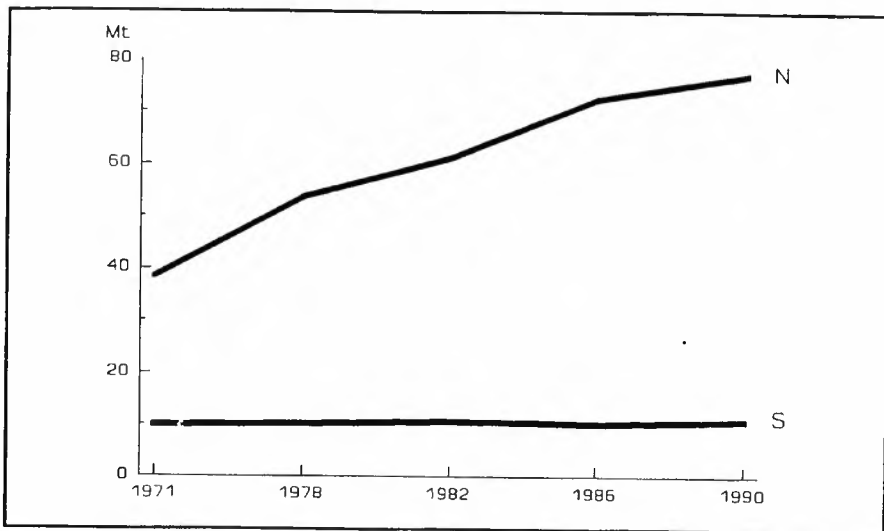


Fig 1. World N and S in fertilizer consumption

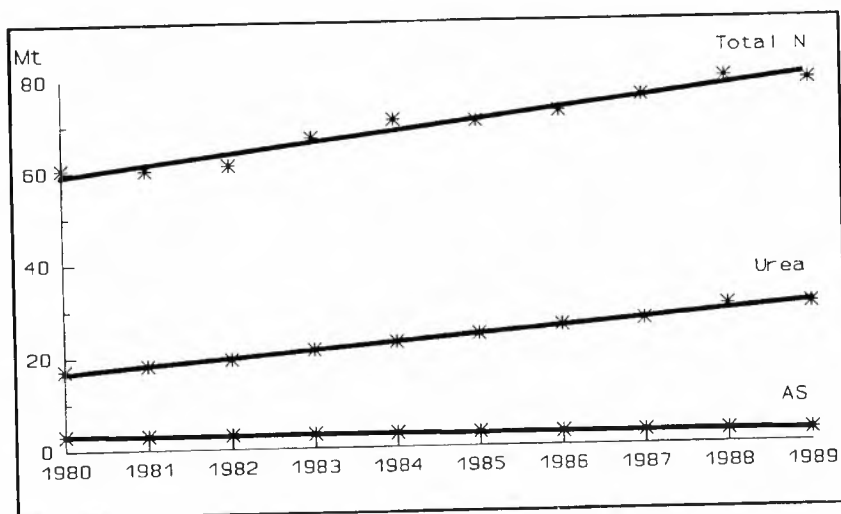


Fig. 2. World N Fertilizer consumption

In Western Europe total nitrogen consumption in 1973 was equal to 8.3 million tons. That same year, ammonium sulphate contributed 0.6 million tons of nitrogen or 7.2% of the total, while urea with 0.4 million tons contributed, represented 4.8% of the market. In 1989, total nitrogen consumption was 11 million tons, ammonium sulphate contribution of 0.4 million tons dropped to 3.6% of the market as urea's market share increased to 14% with 1.5 million tons of nitrogen contributed.

### Increased crop production

Increased cropping intensity and higher crop yields removing greater quantities of S from the soil contribute to greater incidence of S deficiencies (Table 1). Food security is essential for the elimination of hunger; thus it is vital for the economic strength and the stability of any country in the world. To fulfil this requirement, food production in developed countries has benefitted from high input farming since the end of World War II with the implementation of technological and chemical breakthroughs. The developing world has lagged behind for many years, but is now also benefiting from the same technological inputs and, particularly over the past decade, food production in this region has increased dramatically.

Compared to the recent past, the agricultural scenario in most Asian countries has improved and is still progressing steadily. In contrast to 10 years ago, when most of the countries in the region had to import much of their cereal needs, China, India, and Indonesia are now self-sufficient in rice, and other countries are nearing self-sufficiency (Morris 1987). In the 1950s, two of the most agriculturally important Asian countries, India and China, were dependent on imports of food grains. Increasing population growth necessitated the adoption of a policy aimed at self-sufficiency in food grain production. The mid-1960s represented the turning-point in agricultural production toward self-sufficiency in India and China when a "New Agricultural Policy" based on high yielding variety crops and higher inputs was established (Biswas & Chanda 1992).

## 10 Plant nutrient sulphur: A global review

Table 1. Sample crop production in selected nations

Year	Brazil – Soybeans	India – Rice	China – Rice	France – Cereals	Germany – Cereals	UK – Cereals
			kg ha <sup>-1</sup>			
1961	1124	1542	2082	2276	2530	3176
1971	1210	1711	3318	3876	3990	3926
1981	1765	1962	4334	4729	4450	4933
1990	1732	2628	5704	6066	5814	6163

These food production gains have come about primarily as a result of intensification of agriculture. In Asia, excluding the Middle East and former Soviet Asia, about 90% of the land suitable for agriculture is already under cultivation, in contrast to 18% in Latin America (Morris 1987). Although per capita grain production has increased in both Latin America and Asia over the past 15 years, the growth rate in grain production in Asia has been greater and can be attributed to an increase in production per unit area (von Uexkull 1984). In 1990, there was about 0.52 ha of arable land per person in developed countries but only 0.17 ha per person in developing countries. From 1970 to 1990 arable land per capita decreased by only 15% in developed countries, but 29% in developing countries (Roy & Harris 1993).

The average annual cereal production increase in Asia from 1961 to 1990 was 3.3% while the harvested area increased at only 0.4%. During this period, in China and India, rice production has shown an exponential growth of 3.6% and 2.7%, compared with 0.5% and 0.6% in the harvested area. Wheat during the same period followed a similar trend. Chinese wheat production increased at 6.6% while the area increased at 0.8%. In India, wheat production growth was similar at 6.2% whereas the area grew at 2.5%. From 1961 to 1990, cereal production in France increased at an annual average rate of 3.2% versus 2.2% in Germany. However, the area harvested in France grew at an average rate of only 0.2%, and in Germany it even decreased by 0.2% annually. This intensification of land use, which is likely to continue in the future, has commanded a proportionate increase in S demand.

World S uptake has increased from 3 million tons in 1961 to 6 million tons in 1991. Therefore, during the past 30 years, S removal by crops has doubled worldwide. Total crop removal of S in Asia is about 2.6 million tons and represents about 43% of the world's total. Within the region, China and India remove the highest portion of S in crops and together they comprise 66% of total removal in Asia. Western European crops removed 0.6 million tons of S in 1991 or 11% of total world removal. Within West Europe, France alone removed 26% of the total (Fig. 3).

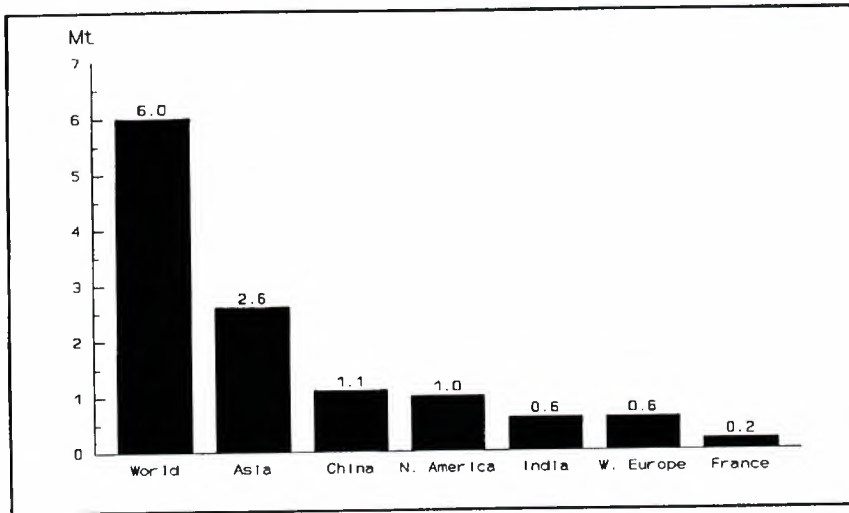


Fig. 3. Regional crop removal of S in 1991

### Decreasing sulphur dioxide depositions

Another factor has affected worldwide demand for S fertilizer consumption and will become of even greater importance in the future. That is, increased global environmental concern and compliance has prompted reductions in  $\text{SO}_2$  emissions which, in the past, have benefitted crop growth in many countries. Sulphur dioxide from the atmosphere finds its way into the plant through the soil, whether through wet precipitation or dry deposition. The Environmental Law Institute (Washington, DC, 1990, personal communication) reports that many industrialized countries have made dramatic reductions in their  $\text{SO}_2$  emissions over the past several years (Table 2). France, the former West Germany, the United Kingdom, and Japan have reduced their contribution of  $\text{SO}_2$  by more than 30% from 1970 to 1985. Sweden has reduced its contribution of  $\text{SO}_2$  emissions by 70% in the same period.

Table 2. Sulphur dioxide emissions and trends

Country	Total $\text{SO}_2$ Emissions-1985 million metric tons	% Change $\text{SO}_2$ Emissions 1980-85	% Change $\text{SO}_2$ Emissions 1970-85
USA	20.7	-11	-27
France	1.8	-48	-38
Federal Republic of Germany	2.4	-25	-34
UK	3.6	-23	-42 (1975-85)
Japan	1.1 (1983)	-12	-36 (1970-83)
Canada	3.6 (1983)	-22	-46 (1970-83)
Sweden	0.3	-44	-70

In the coming years we can expect this trend to continue. In fact, the Clean Air Act passed in The United States in 1990 calls for a national reduction in SO<sub>2</sub> emissions to 10 million tons by 2000 from a current level of 20 million tons. Currently, the high levels of SO<sub>2</sub> discharged by factories in Eastern Europe are under discussion, and the German government is requiring factories in the former German Democratic Republic to decrease the SO<sub>2</sub> emissions to levels comparable with those of factories in the west. Emissions have been reduced radically in Poland also as a result of major investments in the area of air protection. The amount of SO<sub>2</sub> released into the atmosphere dropped by one million ton, or almost 25% from 1989 to 1992. By 2000, it has been estimated that there will be a total reduction of SO<sub>2</sub> emissions equal to 30% of the 1989 level (Pudlis 1993).

Although in the past this matter has received little attention in Latin America, the North American Free Trade Agreement has brought this issue to the forefront in Mexico. Furthermore, the Earth Summit in Brazil in 1992 ensured that the subject will be addressed in the coming years and SO<sub>2</sub> levels will decrease. In the developing world, China and Korea have begun to address their air quality as a result of pressures from the Japanese government and China's interest in joining the GATT nations. Other developing countries have now reduced or stopped altogether the practice of burning sugar cane and various crop residues which release SO<sub>2</sub> into the atmosphere. One study has shown that 40% to 60% of the S contained in the rice straw is lost to the atmosphere through burning (Blair et al. 1991). As levels of SO<sub>2</sub> decrease throughout the world, more farmers will discover that they must include S in their fertilizer programmes to maintain profitable and healthy crop yields. They will no longer benefit from the S in the atmosphere feeding their crops. This is already a reality in northern European agriculture.

## ENVIRONMENTAL IMPACT OF SULPHUR DEFICIENCY

In this paper, the intention is not to discuss in detail the crop responses to S fertilization and the impact of diminishing S supplies on natural ecosystems. However, some of the main issues will be touched on briefly. During the 1980s, the S applications on European crops decreased dramatically (Schnug 1991). The situation in the northern cropping areas of Europe has become so serious that Schnug (1992) reported S deficiency as the major nutritional problem in arable crops. Today in the United Kingdom, Sweden, Denmark, Germany, and France, S deficiencies are the most widespread of all nutrient deficiencies in oilseed rape. Sulphur deficiencies also seriously affect wheat and other cereals. The average S uptake since 1980 for oilseed rape, and since 1990 for wheat, is no longer satisfied by atmospheric depositions (Schnug 1991, 1992). The reduction of the S supply became dramatic during the early 1980s after the enactment of maximum thresholds of S concentrations in various sources of fuel in 1979, and the output of power plants in 1984 (Sendner 1985). Sulphur dioxide emissions in the United Kingdom peaked in the late 1960s and have since decreased by approximately 40% to the current level of 1.8 million tons per year (United Kingdom Review Group on Acid Rain 1987, 1990). On the west coast region of the German Federal State, Schleswig-Holstein, Kurmies (1957) estimated that the atmospheric S input from 1955 to 1956 was 80 kg ha<sup>-1</sup> per year. Thirty years later in 1984–85, Schnug & Holz (1987) determined an annual S deposition of only 20 kg ha<sup>-1</sup>.

The diminishing supply of S to crops has far-reaching implications and consequences.

Sulphur plays an important role in the primary and secondary plant metabolism as a component of proteins, glucosinolates, and other compounds, which relate to several parameters determining the nutritive quality of crops. The deterioration of the S supply will have several consequences on natural ecosystems that have to be considered in future developments for improved soil fertility and environmental quality.

Particularly noteworthy, are recent crop responses to S fertilizers in soils surrounding industrial areas, which were thought to be very unlikely to respond only a few years ago. In Germany, S deficiencies, which were first reported during the early 1980s, began spreading rapidly southward and now S deficiencies in oilseed rape have also been reported in southern Germany (Ofenhitzer 1990; Wimmer 1990). Similarly, field experiments in England prior to 1980 indicated no yield response of oilseed rape to S. Subsequently, researchers at the Agricultural Development and Advisory Service conducted field trials in England and Wales between 1981 and 1987 and responses were restricted to sites on chalk soils (Syers et al. 1987). More recently, S deficiency in oilseed rape has become increasingly widespread particularly in the north of England and Scotland (Zhao et al. 1991). In 1991 and 1992, research plots at Woburn, United Kingdom, showed yield responses of rapeseed to S fertilization despite SO<sub>2</sub> depositions of about 20–30 kg/ha which today are among the highest in the United Kingdom (AFRC 1992). These results are especially important because they occurred in an area between London and Birmingham with a history of high SO<sub>2</sub> depositions.

The increasing production of oilseed rape in Germany, Denmark, France, and the United Kingdom has forced many farmers to apply S fertilizers for optimal plant nutrition. Because of substantial increases in S deficiency in oilseed rape, the North of England Arable Centre issued an advisory to farmers in 1991, instructing them to watch for S deficiency and advising them on the use of S fertilizers to correct the deficiencies (Murphy 1991). In Scotland, as early as 1985, a general recommendation for S application was issued (Anon. 1985). Similarly, in Denmark a blanket recommendation for S has been introduced (Knudsen & Pedersen 1992). Farmers in Ireland and the United Kingdom have also seen benefits from S additions to their forages. As a result of this and other evidence, the Agricultural Development and Advisory Service in the United Kingdom recently developed a recommendation for farmers to use S fertilizers to improve forage production (Agricultural Development and Advisory Service 1988). In Ireland, research conducted in the late 1970s also documented grass production gains. This research and industry's promotion have increased the consumption of S fertilizers three-fold (Murphy 1991). Many Asian regions have also dramatically shown the agronomic value of PNS. In 1970, only four countries had reported S nutritional deficiencies; in 1991, 12 countries on the Asian continent and two countries in the Pacific had identified S deficiencies. Especially noteworthy is the vast amount of research conducted throughout Asia in the last two decades clearly documenting the negative implications of S deficiency in agriculture (Blair et al. 1991; Ceccotti & Messick 1993).

In addition to reducing plant yield, S deficiency is now also widely recognized as adversely affecting the quality of crops grown for both human and animal consumption. For example, in breadmaking wheat, the S nutritional status is positively correlated with yield and baking and milling quality (Schnug 1992). Furthermore, S fertilization has been shown to influence forage quality through increased vitamin A content of alfalfa, increased chlorophyll content of red clover, increased protein content and amino acid composition of forages, decreased nitrogen to S ratios and non-protein nitrogen and nitrate, and reduced hydrogen

cyanide content (Tisdale 1977). The nitrate concentration in vegetables and forages has become an important criterion for food quality (Schnug 1990). Nitrogen and S are main constituents of proteins; therefore, a shortage in the S supply of crops also affects the utilization of nitrogen within plants for the synthesis of proteins. Thus, S deficiency may cause an enrichment of non-protein nitrogen compounds including nitrate in the plant tissue (Murphy 1991). Therefore, it is of utmost importance to maintain an optimal S nutritional status in order to prevent nitrate enrichments within plant tissue.

The effect of S on forage crop quality is important because of its ultimate impact on ruminant nutrition and performance. Increased dietary S levels in a number of studies with ruminant animals have shown increased feed uptake, dry matter digestibility, and improved nitrogen balance, all of which may result in increased meat, milk, and wool production (Morris 1987). While both S fertilization of forages and direct dietary supplements are beneficial, several studies have shown that dry matter intake was greater where forage was fertilized with S than where the lower-quality forage was supplemented with this element (Tisdale 1977). This accentuates the importance of supplying the right amount of S to agricultural ecosystems since where a S deficient condition exists, dietary supplements cannot completely make up for lost production and reduced nutritional efficiency.

Sulphur availability contributes to the overall health of a plant. The content of S-containing secondary compounds in plants is not only of importance for nutritive value or flavor, but also for resistance against pests and diseases. This is of great importance for natural resistance of plants in both agricultural and non-agricultural systems and for alternative (organic) agricultural systems where the use of pesticides is prohibited. Here, S fertilization is a valuable method to enhance the natural resistance of plants against diseases and insect damage (Schnug 1990).

Recently, fertilizing practices have come under criticism as an important factor contributing to pollution. The contamination of ground water with nitrates is one of the most serious problems. Nitrogen and S are both involved in protein biosynthesis; thus, a shortage in the S supply of crops also lowers the utilization of applied fertilizer nitrogen. Therefore, besides poor efficiency for nitrogen fertilization, S deficiencies may increase the loss of nitrogen from agricultural soils through volatilization and leaching (Schnug et al. 1993).

## ECONOMIC ASPECTS OF PLANT NUTRIENT SULPHUR

Sulphur fertilization on deficient soils is not only necessary to sustain healthy crops, but is economical and profitable. Some countries are aware that deficiencies exist, but are still in the process of quantifying the incidence. Other countries have seen remarkable production increases from S and have pursued S research more aggressively. This research has produced valuable information, which is bringing about changes in fertilization practices and increasing the demand for S fertilizers worldwide. In certain parts of the world, applications of S are as common as nitrogen applications and may even be more profitable on a per unit basis. Research conducted in the last decade in areas of North America indicated that where a US\$3 return on every dollar invested in fertilizer is expected, with S fertilizer an investment of US\$1 commonly resulted in a US\$9 to \$10 return on S-deficient soils (Segars 1990). Similarly, Takkar (1987) reported that in India, an application of S from 20 to 40 kg ha<sup>-1</sup> in all cropping



systems is economical. This is likely to hold true in other developing countries.

The financial losses caused by S deficiency to farmers is well-represented by some practical examples. In northern Germany, an application of 100 kg ha<sup>-1</sup> of S on deficient oilseed rape crops normally yields an extra 2 t ha<sup>-1</sup>. In economic terms, with oilseed rape selling at DM750 per ton (US\$441 per ton), and the price of S at DM0.4 per kg, the application costs DM40 (US\$24) while returning DM1500 (US\$885) on the investment, roughly a 38:1 value-to-cost ratio (Schnug, Institute for Plant Nutrition and Soil Science, April 1992, personal communication). Similarly, Richards (1990) calculated that in the main grass-growing areas of the United Kingdom, an application for second- or third-cut silage of 25 kg S ha<sup>-1</sup> resulted in an additional yield of approximately 1 ton dry matter ha<sup>-1</sup> per year. With S costing around £7 ha<sup>-1</sup> (US\$10) and the extra yield worth about £80 (US\$118), it resulted in a value-to-cost ratio of 11:1. Further, Murphy (1990) estimated that annual S fertilization on deficient areas of Ireland, could result in an additional 1.5 million tons of herbage dry matter with a value of I£75 million (US\$110 million). Once the farmers realize the economic implications suggested by this example, S fertilization is more likely to become an integral part of the agronomic practices.

As farmers realize the benefits of S fertilization in a balanced plant nutrition scheme, they are willing to pay a premium for it. In many parts of the world, they pay handsomely for a material that they received free a few decades ago. While S in fertilizers is a value-added product for the fertilizer industry, this success would not be realized if the farmers did not recognize the benefits resulting from increased yield and profits. In the developed world, farmers are now often paying between US\$0.25 and \$0.45 per kg for S in their fertilizers. This translates to a S equivalent of US\$255 to \$440 per metric ton. Many manufacturers of ammonium sulphate have started marketing this product as a multinutrient fertilizer, rather than just a nitrogen fertilizer. This product promotion has led to granular ammonium sulphate prices reaching historically high levels in the US during the spring 1993 fertilizer season. Compared to the same period in 1992, granular ammonium sulphate FOB prices increased by 10% in the Corn Belt, while urea prices have shown only a 5% gain, and phosphate and potassium prices declined. Also in developing countries, farmers are requesting and paying a premium for ammonium sulphate versus urea. In India, based on equivalent nitrogen values, urea sold for US\$285 per ton of nitrogen, while ammonium sulphate sold at US\$438. This translates into US\$0.15 per kg of S or US\$148 per metric ton. This confirms that where farmers acknowledge the advantage of using a S carrier, they will ask and pay for it. In Thailand, urea sold for US\$441 per ton of nitrogen and ammonium sulphate sold for US\$614 per ton of nitrogen. This translates into US\$0.17 per kg of S or US\$168 per metric ton (Fertilizer Trade Information 1991).

## AGRICULTURAL SULPHUR BALANCE

The need for PNS for crop growth is a function of two main parameters: the balance between the addition and removal of S from the soil system, and the ability of the soil to supply S. This relationship is dependent upon the soil type and the local environmental conditions. Crop removal and leaching represent the major subtractions from the system, while S fertilization, the soil supply potential, atmospheric depositions, irrigation water, decomposing crop residues, and manure applications represent the major additions to the system (Messick et al. 1991). To predict world and regional requirements for PNS, The Sulphur Institute (TSI) has developed

a model based on International Fertilizer Industry Association (IFA) and TSI fertilizer statistics, Food and Agriculture Organization of the United Nations crop production statistics, average crop S contents, and S fertilizer efficiency factors. The Sulphur Institute estimates that the total unrealized world market potential for PNS in 1990 was 6.6 million tons. The S requirement has shown a steady growth since the early 1960s and is expected to continue with this trend into the 21st century. The world PNS deficit has been projected to reach 8.1 million tons annually by the turn of the century and increase to 11.1 million tons by 2010.

Asia has shown an increase in consumption of S-containing fertilizers since 1973, but the S requirement is increasing at an even higher rate (Fig. 4). Assuming that food production trends continue at the same pace and S fertilizer consumption remains at levels comparable to those of the last five years, Asia will have an annual deficit of 5.5 million tons in 2000. This will increase to 7.3 million tons by 2010. Developing economies within Asia will continue to be an important market-place for S fertilizers. China and India will be particularly important to the fertilizer industry because, together, they account for about 30% of the worldwide fertilizer consumption and over 60% of the total S fertilizer applied in the region. China has the

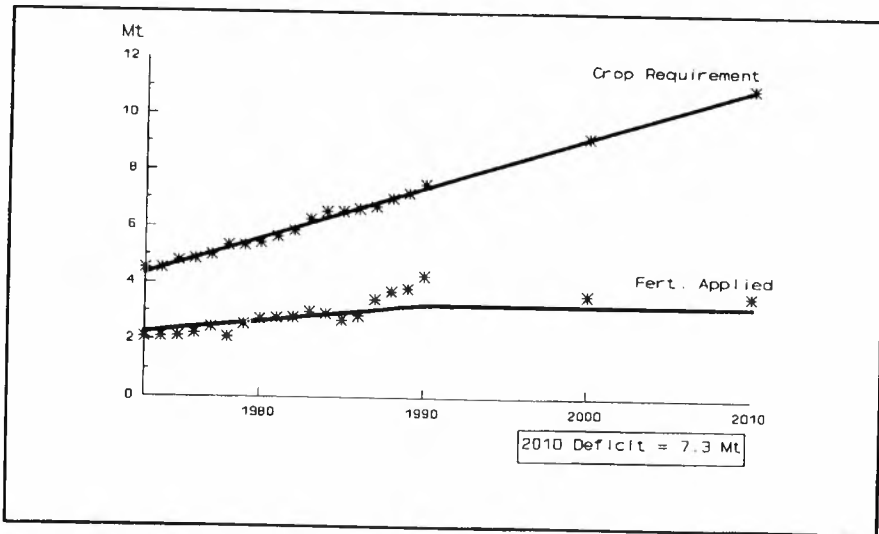


Fig. 4. PNS balance in Asia

largest potential market within Asia and levels of S fertilizer consumption varied between 1.1 million tons in 1973 and 2.4 million tons in 1990 (Fig. 5). However, China has not shown a steady increase in consumption, while the need for S fertilizer has continued to increase. As a result, China's deficit in S fertilizers has increased and will be equal to 2.1 million tons per year in 2000 and 2.9 in 2010, unless S consumption increases above its current level. The level in 2010 represents a 190% increase over the 1990 deficit and 39.7% of the entire estimated deficiency of S fertilizers for Asia. These statistics illustrate the importance of S fertilizer

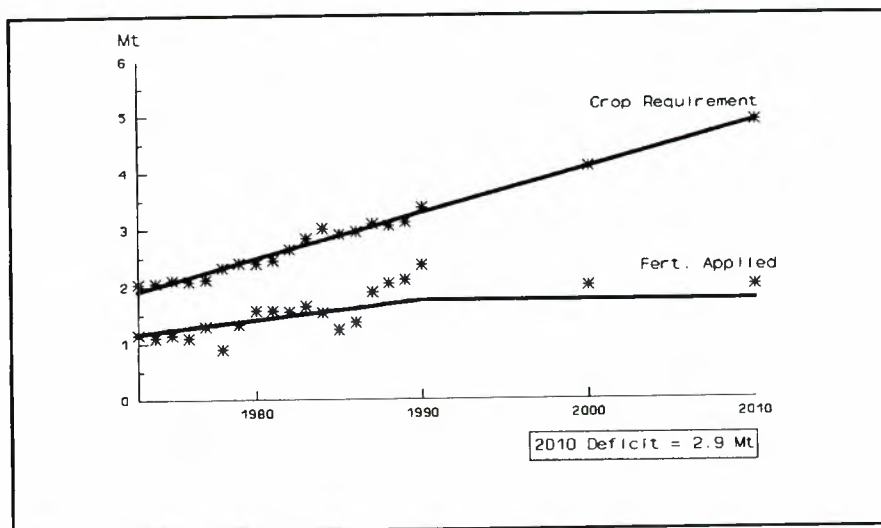


Fig. 5. PNS balance in China

research currently being conducted in China, along with the government policies to be implemented, upon the future of the country's improvement in agricultural production. India has shown a rapid and consistent growth in consumption of S fertilizers (primarily as Single super phosphate) which doubled from 0.3 million tons in 1973 to 0.6 million tons in 1990 (Fig. 6). However, India has shown an even more rapid and steady increase in crop production

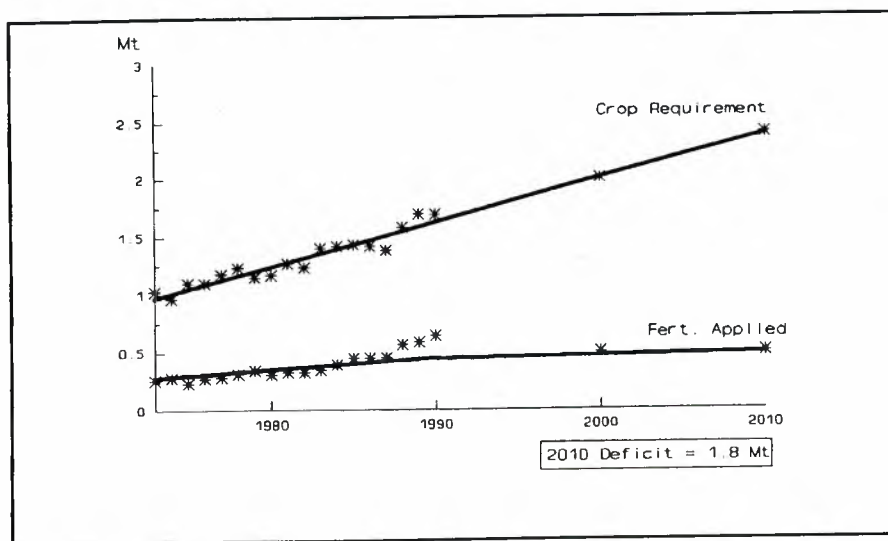


Fig. 6. PNS balance in India

growth rate, and thus S requirement. The S fertilizer deficit in India has fluctuated from 0.8 million tons in 1973 to 1.0 million tons in 1990. With crop production increasing, the consumption of S fertilizers will also need to increase above the current level of 0.6 million tons, or the deficit will climb considerably. If the increase in consumption of S fertilizers does not occur, the country will show an annual deficit of 1.8 million tons in 2010, an 80% increase compared to the 1990 level.

North America has shown a relatively constant level of S consumption since the early 1970s and it is expected to remain constant continuing the current trend at about 1 million tons consumed each year. Within North America the United States represents, by far, the greatest market potential, which is projected to reach 1.4 million tons by 2010 or 87% of the total in North America. Despite the expected limited increases in food production levels for North America, the deficit for S fertilizer in this region will be approximately 1.3 million tons at the turn of the century. This will increase to 1.6 million tons by 2010 (Fig. 7). In the same year, Latin America will show a deficit of 1.3 million tons of S fertilizers, while Africa's deficit is projected to equal 1.2 million tons annually (Fig. 8).

In Western Europe we are witnessing a dramatic decrease in S applications while S requirements have continued to increase (Fig. 9). This is coinciding with rapidly growing S deficiency problems throughout Western Europe. If the current trend levels off at present values, we can expect the PNS deficit in Western Europe to reach 0.3 million tons in 2010. Whereas if the current trend persists, the deficit could increase by a greater margin. Although the model indicates the market potential for S fertilizer, one must understand the agronomic and market conditions affecting fertilizer. For example, the model predicts that Western Europe will only have a S fertilizer deficit of 0.1 million tons in 2000 and 0.3 million tons by 2010 (Fig. 9). However, cropping patterns are changing in this region. Cultivation of oilseed rape, which

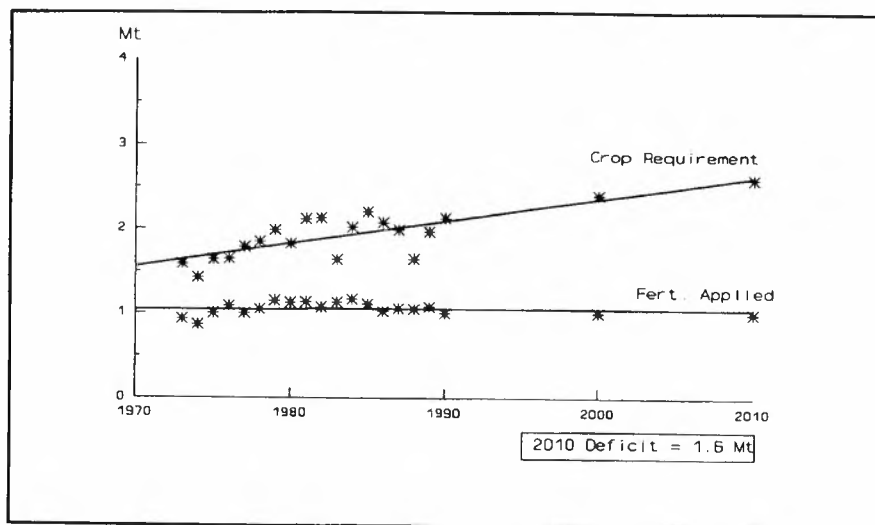


Fig. 7. PNS balance in North America

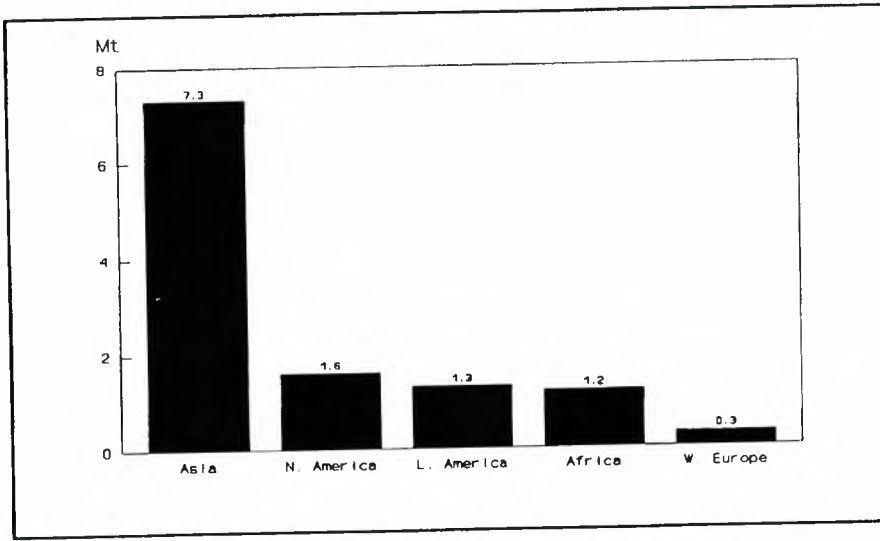


Fig. 8. Regional PNS deficit in the year 2010

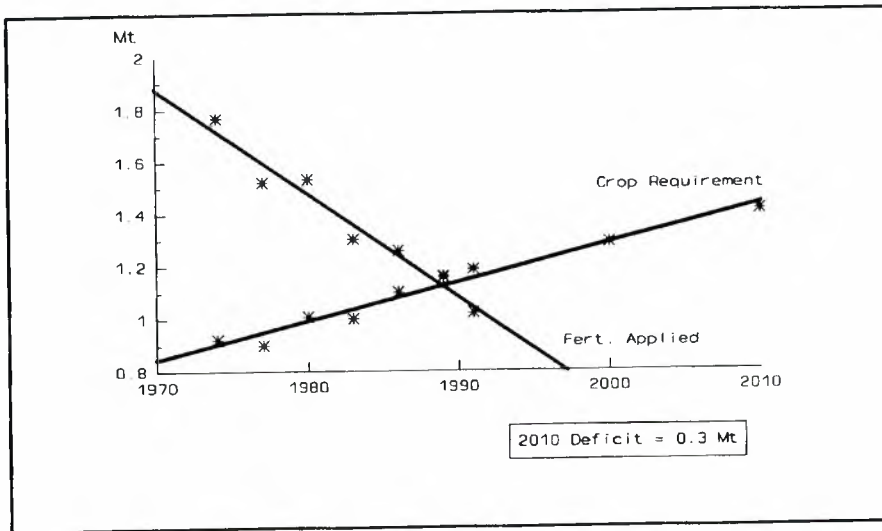


Fig. 9. PNS balance in Western Europe

requires two to three times the amount of S per unit area for optimal growth as compared to wheat, has become more prevalent (Walker & Booth 1992). In fact, rapeseed is one of the most important crops in northern Europe. In certain areas of Denmark and northern Germany, it shares more than one-third of the cropping area (Schnug 1989), making this region the most intensively cropped rapeseed area in the world. As mentioned previously,  $\text{SO}_2$  emissions are expected to decline even further, exacerbating the occurrence of S deficiencies. Moreover,

fertilizer consumption in Western Europe has been declining since 1986–87 falling by 24%. The fall in consumption is expected to continue during the next ten years, but more gradually. The reduction in 1992–93 in Western Europe is estimated at 12% (Fettah & Maene 1993); therefore, the need for S fertilizers may increase by a greater margin than the model predicts. Ultimately, changes in the EEC Common Agricultural Policy subsidy and set aside policies, along with further developments of alternative uses such as rapeseed oil for biodiesel production, will affect future production levels and S requirements.

## FERTILIZERS TO SUPPLY THE GROWING SULPHUR NEEDS

In order to maximize and reconcile economic and environmental demands in agricultural practices, there is a need to reconsider the vital role of S for agroecosystems and especially the importance of S fertilization in optimizing crop exploitation of other nutrients, in particular nitrogen.

Sulphur can be applied to crops in fertilizer or as an elemental spray, each method having its advantages and the choice depending on farm practices. Sulphur is taken up mainly through the roots so fertilizers are the most immediately available source. On the other hand, elemental S sprays have a fungicidal effect on the leaves and become available to the crop roots once washed onto the soil and oxidized to the sulphate form. A variety of S fertilizers are available to suit various methods of application. The more common sources include ammonium sulphate, single superphosphate, potassium magnesium sulphate, and potassium sulphate. Mixed grades and directly applied materials, such as ammonium thiosulphate and S-bentonite, also contain S in varying amounts.

Ammonium sulphate and single superphosphate are now recognized as multiple nutrient fertilizers rather than solely nitrogen or phosphorus sources, and many farmers are using these to correct multiple nutrient deficiencies. Over 15 million tons of ammonium sulphate is produced annually and much of this production is a co-product of other industries, mainly the caprolactam industry. Since ammonium sulphate is a co-product and traded internationally, this material will influence the market for all S fertilizers. Single superphosphate is a significant fertilizer in China, India, Australia, and New Zealand, but of little importance in other countries, such as those in North America and Europe. Furthermore, most of the new phosphate fertilizer facilities under consideration worldwide include ammoniated phosphates and compound fertilizers rather than single superphosphate.

During 1990, single superphosphate and Ammonium Sulphate fertilizers contributed most of the S consumed worldwide, followed by compound fertilizers (Table 3). Single superphosphate contributed 40% of the total, while Ammonium Sulphate contributed 30% of the total. Therefore, these two sources alone contributed 70% of the total worldwide S consumption in fertilizers. More frequently, the popularity of these fertilizers is a result of their S content. In 1990, total S consumption in fertilizers was 10.1 million tons. In that same year, total raw material S consumption was 58.8 million tons. Therefore, almost 20% of the total raw material S consumed was directly applied in the form of fertilizers.

As the market for S fertilizers expands, S producers are introducing new products to meet the diversified requirements. Fertilizer technology experts in New Zealand have developed a process to add elemental S during fertilizer manufacturing. This process avoids grinding

Table 3. Sulphur contributions from various fertilizer sources

Fertilizer source	%S	1990 million tons
Ammonium sulphate	24	3.0
Single superphosphate	12	4.1
Triple superphosphate	1	0.1
Ammonium phosphate	2	0.4
Compound fertilizers	VAR	1.9
Potassium sulphate	18	0.3
Others	VAR	0.3
Total S Consumption		10.1

elemental S and facilitates the incorporation of elemental S into fertilizers. Hi-Fert, an Australian firm, introduced a S-coated triple superphosphate that is available in two grades, Gold-Phos 10 (0-41-0-10S) and Gold-Phos 20 (0-36-0-20S). The company has expanded its product line to include S-coated monoammonium phosphate and single superphosphate, S-coated partially acidulated rock phosphate and single superphosphate, and S-coated partially acidulated rock phosphate and triple superphosphate.

Solterra Minerals, Inc. of Canada has developed the technology for two products. Sulchem SUPER SULF-8 (7-0-0-68S) is a granular product suitable for use in a suspension, or in a liquid fertilizer form, or as the S component of a dry bulk blend. The product contains both sulphate S and elemental S and is designed to give the farmer both immediate and reserve availability of S. Eight percent of the S in the material is in the immediately available sulphate form. The other portion is in the elemental form, but, due to its small particle size, will oxidize rapidly and become available during the growing season after the initially available sulphate portion is depleted. The other product is named SULCHEM 95 (0-0-0-95S). This product is also granular, but contains only an elemental source. It can be used in suspension, clear liquid, or dry blends. Because the product rapidly disperses into fine particle sizes regardless of its application method, it converts to sulphate rapidly to become plant-available. More recently, Solterra has introduced a newer elemental S-based material. This 44-micron material can be used in fertilizers. The company has also developed the technology to granulate the material for easier handling and application.

In the United States, Kerley Ag. Inc., developed a unique and versatile product. Kerley is currently the only manufacturer of potassium thiosulphate, which is marketed under the trade name KTS. It is a clear liquid fertilizer containing 25% potassium and 17% S, which offers additional versatility to farmers and fertilizer retailers.

The European fertilizer industry is also responding with new products to capture a part of this rapidly growing market. La Grande Paroisse in France and ICI in the United Kingdom have introduced new fertilizers. ICI's Fertilizer product, Sulphur-Gold, contains 30% nitrogen and 19% S and is particularly designed for oilseed rape and winter cereal cropping systems. Kemira and Norsk Hydro, the two largest fertilizer companies in Europe, along with BASF and DSM have introduced and are marketing S-containing fertilizers. Sulphur Ten (20-4-14-7S), developed for silage crops, has been marketed by Kemira for several years. More recently, Kemira Fertilizers in the United Kingdom, has released DoubleTop, a new granular product,

which contains 27% nitrogen as well as 12% S and is specially formulated for oilseed rape and cereals. All this renewed activity on the market, reflects the commitment of the fertilizer industry to pursue this relatively new market after recognizing its potential.

Agriculture is the largest sulphur-consuming industry. Approximately 60% of yearly S production is consumed in the production of fertilizers, mainly as superphosphates, ammonium phosphate, and ammonium sulphate. Favorable research findings, mainly in countries with advanced agricultural production systems, have established S as a valuable fertilizer in its own right. These newer materials are expected to meet a large percentage of the growing S requirement. The future appears favorable for the consumption of S-containing fertilizers. By 2010, there will be an estimated global unrealized market potential equal to 11.1 million tons of S. Effective research advancements, product development, marketing, and promotion will determine what proportion of this relatively new market will be translated into worldwide consumption.

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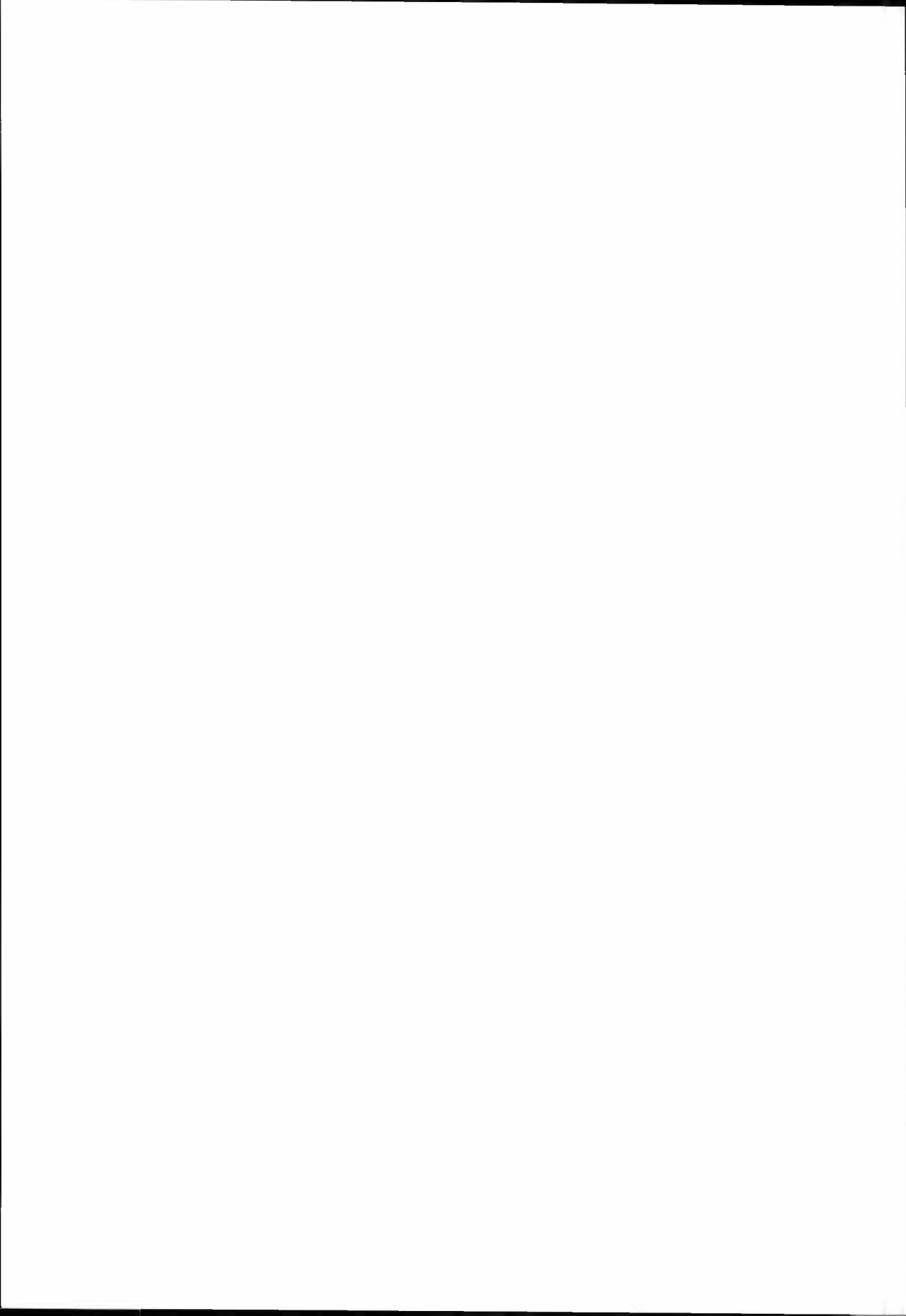
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# Sulphur status of soils – a global study

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Soil samples from a total of 3571 sites in 29 countries were analyzed using the AAAC-EDTA method. The sulphur content of the soil is presented and the correlations between soil sulphur and six soil factors are discussed. Generally, developing countries with low fertilizer use and low industrialization was found to have the highest need for fertilization with sulphur.

Key words: Extractable soil sulphur, Europe, Oceania, Latin America, Far East, Near East and Africa

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In Finland the only soil extraction test method for macro-nutrients in routine use is the AAAC method (Vuorinen & Mäkitie 1955). In recent years this method has been used also for testing sulphur (Sillanpää & Jansson 1991). When testing soils for most micronutrients the method in use is the AAAC-EDTA extraction (Lakanen & Erviö 1971) but in the case of boron the hot water method has been used. It has been demonstrated however, that also soil boron could successfully be tested using AAAC-EDTA method (Lakanen & Erviö 1971; Sippola and Jansson 1993). Sulphur has only recently been recognized as an element that could be tested using AAAC-EDTA as extractant (Urvas 1993).

In international surveys the AAAC-EDTA method has been used for testing soils for micronutrients (Sillanpää 1982; Sillanpää 1990) but also for other elements such as cadmium, lead, cobalt and selenium (Sillanpää & Jansson 1992). AAAC-EDTA has proved to be an excellent extractant but often a pH correction was needed for many elements. The need for a pH correction coefficient for microelements with acid multiextractants like AAAC-EDTA has been pointed out by Lindsey & Cox (1985).

In this paper some preliminary sulphur results of a FAO/Finland project are presented.

## MATERIAL AND METHODS

Sulphur was determined from the same soil samples collected from 29 countries that were used in earlier studies for other elements (Sillanpää 1982, Sillanpää and Jansson 1992). The soil samples represents wheat and/or maize growing areas. General soil properties like pH, organic C, CEC, electric conductivity, texture and CaCO<sub>3</sub> equivalent were also analyzed earlier and this background data could also be used in this study. The main participants were developing countries but some developed countries also participated (see Table 1).

Table 1. Average sulphur contents (mg/l) of soils (AAAc-EDTA extractable) of different countries

	No. of sites	S in soil			
		Range		mean	S.D.
<b>Europe and Oceania</b>					
Belgium	40	21 -	55	34	9
Finland	91	8 -	145	43	25
Hungary	245	15 -	438	57	49
Italy	176	4.3 -	8143	163	804
Malta	25	78 -	211	152	39
New Zealand	38	10 -	174	52	37
<b>Latin America</b>					
Argentina	199	7.9 -	35	17	5
Brazil	64	7.3 -	74	34	16
Mexico	243	8.4 -	4486	135	475
Peru	70	3.4 -	393	46	60
<b>Far East</b>					
India	281	5.1 -	851	59	86
Korea Rep.	94	12 -	177	42	28
Nepal	49	5.6 -	174	28	30
Pakistan	235	8.7 -	4226	224	502
Philippines	195	1.6 -	396	32	49
Sri Lanka	21	8.1 -	102	31	28
Thailand	150	7.6 -	436	49	59
<b>Near East</b>					
Egypt	198	29 -	829	147	118
Iraq	147	8.1 -	5536	676	1137
Lebanon	16	15 -	606	191	171
Syria	37	5.1 -	6300	392	1257
Turkey	296	3.3 -	872	103	98
<b>Africa</b>					
Ethiopia	121	7 -	53	21	8
Ghana	93	5.7 -	72	17	12
Malawi	100	2.8 -	617	120	138
Nigeria	137	3.9 -	657	47	99
Sierra Leone	47	13 -	105	29	17
Tanzania	118	2.5 -	135	16	17
Zambia	45	4.3 -	85	18	16
<b>Whole internat. material</b>					
	3571	1.6 -	8143	108	393

Acid ammonium acetate-EDTA (Lakanen & Erviö 1971) was used to extract sulphur from the soils. For determining sulphur from the extracts a plasma emission spectrometer was used.

## RESULTS AND DISCUSSION

As can be seen in Table 1, the highest average contents of AAAC-EDTA extractable sulphur in the different countries are found in the Near East region. In this region the soil pH is high (Sillanpää 1982; Sillanpää & Jansson 1983) and even the lowest average pH(CaCl<sub>2</sub>), 7.4 in Turkey, is higher than that in most other countries. Usually high concentrations of soil sulphur are found in countries with high pH such as Italy with an average pH of 7.1, Malta with 7.5, Mexico 7.2 and Pakistan 7.8. Malawi, however, is an exception as the maximum pH content in this country is as low as 6.8 and the average pH is only 5.0 and yet the mean extractable sulphur concentration is 120 mg/l soil. This is higher than the mean of the whole international material. Finland has about 45000 hectares cultivated acid sulphate soils where the pH is very low and the sulphate content high (Erviö & Palko 1984). In the countries with low sulphur contents, however pH is low: in Argentina the soil pH was 5.5, in Ethiopia 5.7, in Ghana 5.8, in Tanzania 6.3 and in Zambia 5.0.

There is a strong correlation between soil electric conductivity and soil extractable sulphur (Table 2). Thus, in countries the highest el. conductivity figures (Sillanpää 1982) i.e. Italy, Malta, Mexico, Pakistan, Egypt, Iraq, Lebanon and Syria the extractable soil sulphur content is also high (Table 1).

Table 2. Equations and correlation coefficients for regressions of AAAC-EDTA extractable soil S on various soil factors

Soil factor	n	Regression	Correl. coeff.
pH	3571	logy = 8.67-2.521x + 0.213 x <sup>2</sup>	0.595***
Texture index	3568	logy = 0.698+0.0351x-0.000294x <sup>2</sup>	0.292***
Organic C	3571	y = 111.8-115.4 logx	0.077***
CEC	3571	logy = 1.316+0.0183x-0.000223x <sup>2</sup>	0.155***
El. cond	3571	logy = 1.361+0.984 logx	0.707***
CaCO <sub>3</sub> eq	3571	logy = 1.406+0.0598 logx-0.000842(logx) <sup>2</sup>	0.606***

The strongest relationships between AAAC-EDTA extractable soil S and the soil factors studied (pH, texture, CEC, CaCO<sub>3</sub>-equiv., el. cond and organic carbon) were found between soil S and el. conductivity, pH and CaCO<sub>3</sub>-equivalent. The weakest relationship was found between soil S and soil organic carbon.

Within the countries the variation in extractable soil sulphur is often very large. According to the classification established by Urvas (1993), even in the highest sulphur status country, Iraq there are non-irrigated sites where the soil sulphur contents is low enough to

indicate deficiency. Generally, developing countries with low fertilizer use and low industrialization had the highest need for fertilization with sulphur.

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# Sulphur in Swedish agriculture

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The reasons for sulphur deficiency in Swedish agriculture are discussed. Sulphur (S) deficiencies have increased because of the more widespread use of S-free and low S-content fertilizers, reduced deposition of atmospheric S and increasing crop yields. Soil tests and plant analyses as diagnostic methods are briefly considered and the necessity of planning for the annual supply of S, particularly when S-demanding crops are grown, is pointed out.

Key words: S deficiency, S deposition, S infertilizers, sulphur.

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In recent years sulphur deficiency has increasingly been reported in Swedish agriculture (Olsson 1988; Gruvaeus 1993; Persson 1993; Wallenhammar 1993). In the spring of 1993 in particular there was a more widespread deficiency of sulphur (S) in many areas. The spring began with a very warm period that was conducive to rapid above-ground growth, while the soil temperature was too cold for extensive root development. Under normal conditions these symptoms disappear later in the spring when the roots penetrate to deeper soil volumes.

The objectives of this paper were to found credible explanation to the resent years more and more fequently occurrence of sulphur deficiencies.

## SULPHUR BALANCE SHEET IN SWEDISH AGRICULTURE

The reason why S deficiencies have been occurring more frequently during recent years can perhaps be explained by the reduced supply of S in fertilizers and in the reduced deposits of atmospheric S on agricultural land. Figure 1 presents the changes in the level of emissions of sulphur dioxide (SO<sub>2</sub>), mostly from fossil fuels, into the atmosphere from 1950 to the present in Sweden. The emissions increased until the early 1970s to about 900 10<sup>3</sup> t SO<sub>2</sub> as a maximum. Thereafter, they continued to decrease to about 200 10<sup>3</sup> t SO<sub>2</sub> in 1990 as a result of air pollution regulations (SNV, 1987).

Most of the S deposits over Sweden originate in other countries, where the reduction in S emissions has proceeded somewhat more slowly. Sulphur deposits (wet and dry) during the last decade decreased by about 20%. At present, the total deposition of atmospheric S on soil

can vary from about 32 kg ha<sup>-1</sup> yr<sup>-1</sup> in southern regions to 16 kg ha<sup>-1</sup> yr<sup>-1</sup> in mid-Sweden and to less than 10 kg ha<sup>-1</sup> yr<sup>-1</sup> in the northern part of Sweden.

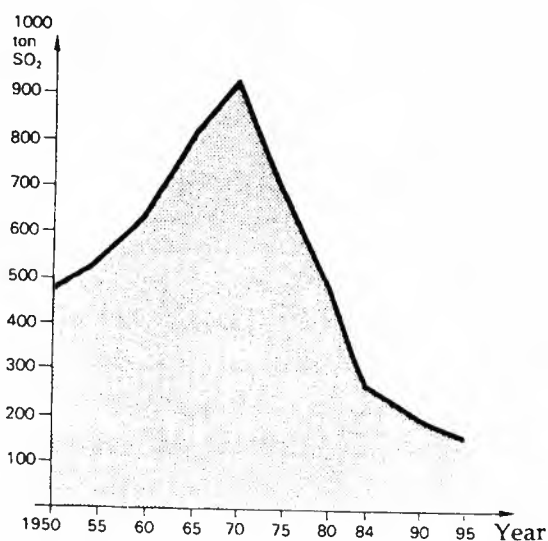


Fig. 1. Emission of sulphur dioxide in Sweden, (SNV Report 3409)

The changes in S supplied to agriculture with different fertilizers between 1981/82 and 1991/92 are shown in Table 1. Addition of S decreased from 39,907 t in 1981/82 to 5,928 t in 1991/92. Expressed in kilograms per hectare, the supply of S in commercial fertilizers corresponds to 13.8 in 1981/82 and 2.1 in 1991/92. The various sources of supply of S to the soil and the losses by crop removal and by leaching can be seen in Table 2.

Table 1. Supply of sulphur in fertilizers, S g 10<sup>6</sup>

Fertilizers	1981/82	1991/92
P fertilizers	2 960	219
K fertilizers	228	144
PK fertilizers	20 506	1 448
NPK fertilizers	16 213	4 117
Total	39 907	5 928

The total S supply from atmospheric deposits and fertilizers has decreased during the 10-year period from 20 kg ha<sup>-1</sup> yr<sup>-1</sup> to 16 kg ha<sup>-1</sup> yr<sup>-1</sup> in mid-Sweden. This amount of S, plus the S mineralization, would be more than enough for the crops. However, in humid regions like Sweden the soluble sulphates are subject to leaching. Therefore, there is little residual plant-available sulphate left in the soil after a wet winter. Under such circumstances when the sulphate content in the soil in spring is low and the mineralization rate of organic sulphur

compounds is not sufficiently rapid, the soil is unable to supply enough S to meet plants' demand without the addition of S-containing fertilizers. In the future, I believe we will have to include sulphur among the macronutrients N, P and K when we plan the annual supply of fertilizers. This will be particularly important when sulphur-demanding crops are grown.

Table 2. Sulphur balance sheet, S kg ha<sup>-1</sup>

	1981/82	1991/92
<b>Gains from</b>		
Atmosphere*	20	16
Fertilizers**	13.8	2.1
	33.8	18.1
<b>Losses by</b>		
Crops	6–20	6–20
Leaching	28–14	12– -2

\* Valid for the mid-Sweden. There is a great difference in deposition of S between southern, middle and northern Sweden. S deposition in 1981: 40, 20 and 10 kg ha<sup>-1</sup> yr<sup>-1</sup>, respectively. And in 1991: 32, 16 and 10 kg ha<sup>-1</sup> yr<sup>-1</sup>, respectively. The supply of S from the atmosphere has decreased by ca. 20% over a period of 10 years

\*\* Average for arable land (1982, 2.9 · 10<sup>6</sup> ha; 1992, 2.77 · 10<sup>6</sup> ha).

Losses by leaching of S in field experiments measured in middle Sweden during the 1980s were between 14 and 37 kg ha<sup>-1</sup> yr<sup>-1</sup> or ca. 28 kg ha<sup>-1</sup> yr<sup>-1</sup> according to Barbro Ulén, Division of Water Management, Swedish University of Agricultural Sciences

## SOIL TESTS AND PLANT ANALYSIS

In order to be able to estimate the mineralization capacity of the soils, determinations have been made of total S, extractable sulphate-S and the mineralization of organically bound S. The content of total S in soils varies between 100 and 800 mg kg<sup>-1</sup>. The extractable sulphate-S is 1–10 mg kg<sup>-1</sup> and the net mineralization rate at room temperature is 0.005–0.1 mg kg<sup>-1</sup> day<sup>-1</sup> (Simán, 1969). Because of the wide variation in the rate of S net mineralization and the mobility of sulphate in the soil, none of these methods is sufficiently reliable in predicting the capacity of the soil to supply S. More knowledge about various other soil and climatic factors is necessary in order to be able to interpret the results of soil tests.

In discussing the soil supply of plant available sulphur, the amount of sulphate at different depths and the depth to which the roots penetrate the soil should also be taken into account. As a rule, the concentration of sulphate-S increases with depth, as shown in Table 3.

In order to make a better evaluation of the mineral availability of the plants, several different crops have been subjected to plant analyses in Sweden. Sulphur deficiency has often been registered during the growing period. Fertilization with S in cases where sulphur deficiency has been identified has sometimes resulted in yield increases in oilseed crops and improvements in baking quality in cereals (Bertilsson 1987; Jonsson 1992).

Table 3. Available soil S (0.01 M CaCl<sub>2</sub> extractable sulphate sulphur, turbidimetric determination), mg S kg<sup>-1</sup> soil

Soil depth cm	Location		
	Ugerup	Lanna	Kamstorp
0-20	1.8	0.1	1.1
20-40	2.7	3.5	0.7
40-60	2.6	5.0	1.6

## CONCLUSIONS

The decreased atmospheric deposition of sulphur and the smaller amounts of S used in fertilizer as ballast will lead to an increased risk of sulphur deficiency, particularly in sulphur-demanding crops such as winter oilseed rape and pulses. Visible sulphur deficiency can occur, particularly in the spring and early summer under warm conditions when the growth of above-ground plant parts is vigorous while the roots are unable to penetrate to deeper depths in the soil profile. In order to counteract the yield loss or quality deterioration it is important to assess the S requirements of the crops and the S availability when planning the cropping programme.

Where there is a sulphur deficiency in the crop, the use of foliar applications can be appropriate. Results from foliar applications show that the crop can take up relatively large amounts of sulphur via the leaves and use it very rapidly in the plant's metabolic processes.

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# Sulphur requirements for crop production in Norway

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Through a brief review of the work carried out in Norway, the factors controlling the Sulphur (S) supply to crops are described and the role of the major factors, soil, atmosphere and fertilizers, in S supply to plants is discussed. Almost all of the plant available S in soils is present as an organic fraction but probably < 10% of this fraction is mineralized during the growing season. Inorganic S generally constitutes < 25% of the total S content of most agricultural soils. In cultivated temperate soils, sulphate is adsorbed only lightly and can easily be desorbed and leached out. About 90% of the applied S in a loam soil in southern Norway was recovered in leaching water within a couple of years after S application. The concentrations of S in the air and in precipitation in southern and central Norway which increased during the 1960s and 1970s, generally declined in 1991 by 33–43% as compared to those measured in 1979. In the past 10–12 years in Norway, S supply through NPK fertilizers has decreased by more than 50% and as a result the ratio between N and S has increased manifold. The total addition of S through animal manure to cultivated land in Norway is probably of the same order as that added through atmospheric deposition. In field experiments, cereals removed the lowest and the cruciferous plants the highest amounts of S. Future research areas are suggested.

Key words: Atmospheric deposition, crop requirements, sulphate leaching, sulphur in soils, sulphur fertilizers.

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That sulphur (S) is an essential plant nutrient has been known for more than 100 years but it first began to receive increasing attention as a plant nutrient in the early part of this century (Lipman et al. 1918; Reimer & Tartar 1919). In Norway, attention was focused on this element in the late 1950s when sporadic cases of S deficiency in field crops were observed because of either greater use of relatively S-free commercial fertilizers or a more intensive use of nitrogenous fertilizers (Ødelien 1967). The more intensive use of N fertilizers resulted in a narrowing down of S and N ratios, thereby causing imbalances in S supply to plants. A series of field experiments with S fertilization were initiated in the 1960s which showed positive and significant crop responses to S application on several sites (Ødelien 1966) and consequently the amount of S in commercial fertilizers was increased.

In the past two decades, sulphur again received greater attention as an environmental pollutant to terrestrial and aquatic ecosystems but its role in plant production in northern

Europe, including Norway, did not receive much attention. The reasons for decreased attention to sulphur as a plant nutrient in the past 20 years in Norway are the increased atmospheric deposition of sulphur and the use of NPK fertilizers containing sulphur. It has, however, been observed lately that the S component of acid precipitation has gone down by more than 40% and the amounts deposited are not sufficient to fulfil the requirements of S for plant growth. Furthermore, reduced use of sulphate-based commercial fertilizers and greater losses of sulphur from anaerobically stored farmyard manure slurry have also resulted in a lower input of sulphur to agricultural crops. There is therefore a renewed interest in investigating the role of sulphur in plant production and in improving the nutritive quality of the product.

This paper presents an overview of the factors affecting the sulphur supply for plant production in Norway and identifies some topics for future research.

### SULPHUR IN SOILS

The total S concentration in humid temperate soils like those in Norway can range from 100 to 15000 mg kg<sup>-1</sup> but only a very small fraction, 5–500 mg kg<sup>-1</sup>, is generally soluble in dilute acid or water (Wainwright 1984). Inorganic S generally constitutes < 25% of the total S content of most agricultural soils. In some forest soils of southern Norway, SO<sub>4</sub>-S in the B horizon (5–20 cm) of podzol soils (Typic udipsamment) constituted >50% of the total S but the corresponding SO<sub>4</sub>-S fraction in the brown-earth soil (Aquic Haploboroll) was <1% (Singh 1980). Almost all of the plant available S in soils is present as an organic fraction but probably <10% of this fraction is mineralized during the growing season. This corresponds to 3–5 kg S ha<sup>-1</sup> y<sup>-1</sup> in an intensive arable soil (Chaney & Kershaw 1986) and about 8–10 ha<sup>-1</sup> y<sup>-1</sup> in a grassland soil (Keer et al. 1986). Sulphate is the most common form of inorganic S and can exist as easily soluble SO<sub>4</sub><sup>2-</sup>, adsorbed SO<sub>4</sub><sup>2-</sup>, insoluble SO<sub>4</sub><sup>2-</sup> or insoluble SO<sub>4</sub><sup>2-</sup> co precipitated with Ca CO<sub>3</sub>. Under humid climatic conditions sulphate concentration in the soil solution is generally very low. In the cultivated peat soils in Norway, S may also be released as H<sub>2</sub>S.

### SULPHUR ADSORPTION AND LEACHING

Sulphate adsorption by soils is an important property affecting both the availability of S to plants and the leaching losses of sulphate. In cultivated temperate soils sulphate is adsorbed only lightly and can easily be desorbed and leached out. Amounts of sulphate adsorbed by soils vary widely and depend upon soil properties and especially on the content of Al and Mn oxides, content and nature of clay mineral, and soil pH (Couto et al. 1979, Singh, 1980, 1984). Singh (1980) reported that only podzol soils under forest vegetation containing relatively high amounts of Al oxides were able to adsorb appreciable amounts of applied sulphate under laboratory conditions and that more than 50% of the adsorbed sulphate was easily desorbed by calcium nitrate.

Ødelien (1965) in lysimeter studies with a loam soil in Norway found that with a moderate rate of S application (equivalent to 40–50 kg ha<sup>-1</sup>) about 90% of the applied S was recovered in leaching water and crops within a couple of years after application.

The cumulative losses of sulphate from the lysimeter increased consistently with the amount of drainage water (Fig. 1). The figure shows that sulphate leaching from potassium sulphate is somewhat faster than from sulphate from gypsum from superphosphate (Ødelien, 1965). In a lysimeter experiment with  $^{35}\text{S}$  in gypsum it was found that > 50% of the S application corresponding to 40–50 kg ha<sup>-1</sup> will easily be leached out from the top 20 cm layer by 100–200 mm water (Ødelien, 1965). He further stated that S concentrations in the drainage water from cultivated areas varied mainly according to the S content of the soil and the precipitation and that the losses of sulphate from the soil by leaching are certainly an important factor causing S deficiency in cultivated plants in Norway. By the same rate of fertilization and for one and the same plant species, lack of S may or may not occur at one and the same locality as a consequence of varying intensity of leaching in the preceding year.

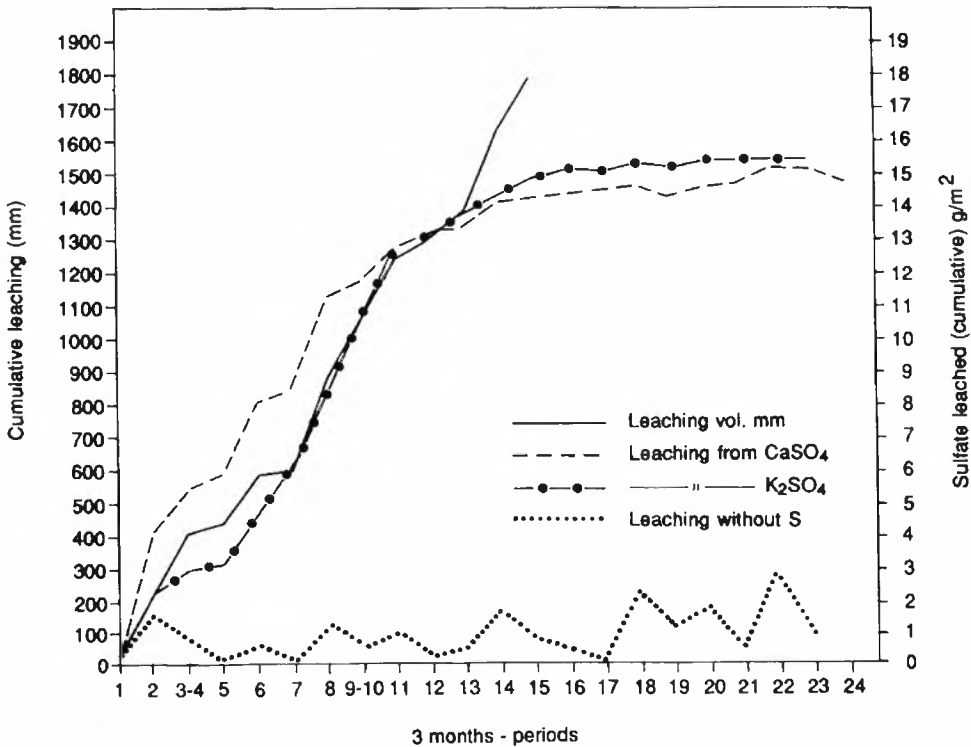


Fig. 1. Leaching losses of sulphur in a lysimeter study (Ødelien 1965)

## SUPPLY OF SULPHUR

The amount of available S in the soil is of prime importance for plant nutrition in certain parts of the world, whereas in other areas the quantity supplied by the atmosphere or by fertilizer application renders the native soil supply relatively insignificant. The latter situation seems to be applicable in most light-textured cultivated soils of Norway.

## ATMOSPHERIC SULPHUR

The concentrations of S in the air and in precipitation in southern and central Norway which increased during 1960 and the 1970s, generally declined in 1991 by 33–43% as compared to those measured in 1979 (SFT, Norwegian State Pollution Control Authority, 1992). No such changes were observed in northern Norway however. The annual mean concentrations of sulphur dioxide have decreased by 47–77% in southern Norway and by 45% in Finmark (northern Norway). It was reported that during the 1970s relatively high amounts of sulphate (30–40 kg S ha<sup>-1</sup>) were added annually to the soils of southern Norway through dry and wet deposition (Doveland et al. 1976). This amount has decreased to less than 10 kg ha<sup>-1</sup> in 1991 (SFT 1992). Recent studies in Norway (Singh 1991) have revealed significantly high crop response to S application in the central parts of the country.

## SULPHUR IN FERTILIZERS

For a number of years in Norway relatively S-free fertilizers were used and even in late 1950s or early 1960s the total concentration of S in the NPK fertilizers used was only 10–12 kg ha<sup>-1</sup> y<sup>-1</sup>. The corresponding values for Denmark, Sweden, Finland, Iceland were 23, 21, 11 and 3 kg ha<sup>-1</sup>, respectively. In the past 10–12 years in Norway S supply through NPK fertilizers has gone down by more than 50% (Fig. 2) and as a result the ratio between N and S has increased many-fold (Fig. 3). Similarly, the total consumption of S in Norway has dropped from 13,696 in 1987/88 to 11,308 in 1991/1992 (Bjørn Svoldal, Norsk Hydro, personal communication).

The total S concentration in most of the NPK fertilizers used in Norway varies from 1.3 to 2.8% but some chloride-free NPK fertilizers, used for crops like potato, contain higher levels of S (up to 9%). Use of superphosphate for agricultural crops is almost negligible. Considering the increasing demand for S for several crops in different parts of the country it is anticipated that the S levels in a few NPK fertilizers such as 17-5-13 and 18-3-15 may be increased by 2.8–3.6%. Similarly, S levels may also be raised in other NPK fertilizers or nitrogenous fertilizers such as calcium nitrate.

Organic manures can provide a further source of S but information on their S levels and efficiency of utilization by crops is rather limited. Based on a large number of animal manure samples from different parts of Norway Uhlen (1968) reported 0.08% as a mean value of S in the manure analysed. The total additional S through animal manure to the cultivated land in Norway is probably of the same order as that added through atmospheric deposition. However, the release of S in animal manure is slower but at the same time S in this form is less susceptible to leaching (Ødelien 1970). If one presumes that about 25% of the S in organic



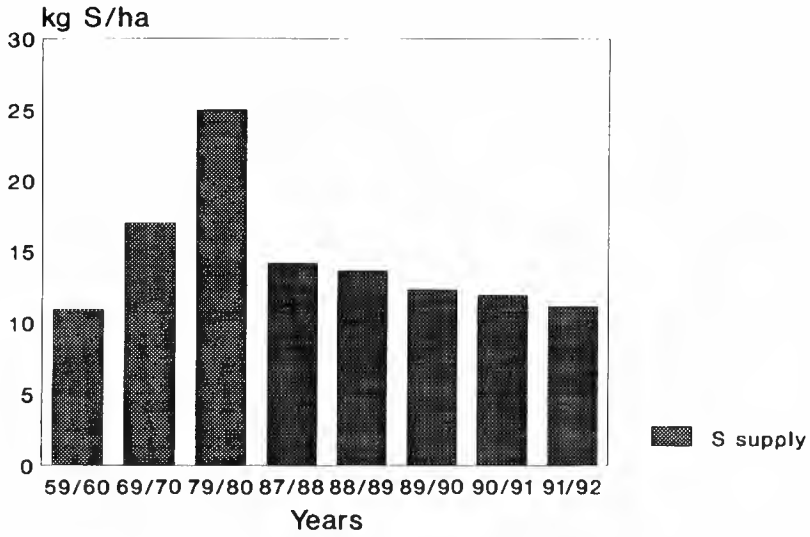


Fig. 2. Variations in S supply through commercial fertilizers in Norway. (Bjørn Svoldal, Norsk Hydro, personal communication)

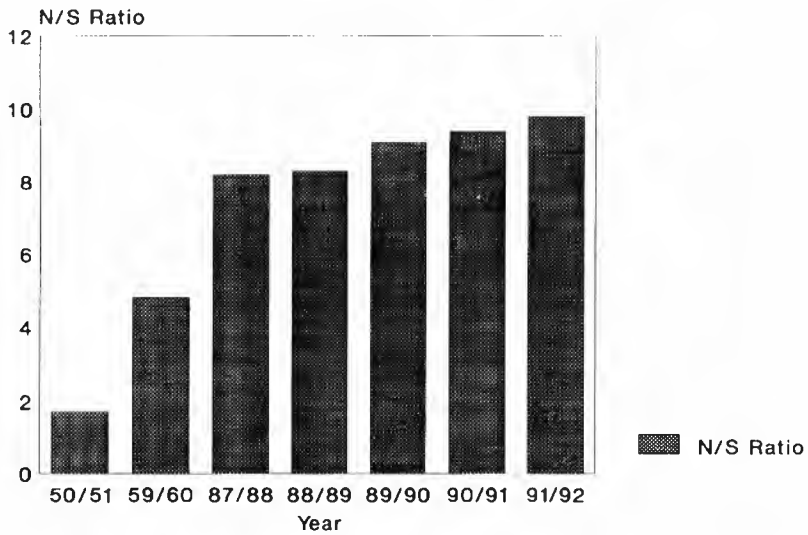


Fig. 3. Changes in N and S ratios in Norway (Bjørn Svoldal, Norsk Hydro, personal communication)

manure is available to crops during the first year of its application, it can be estimated that 20t of farmyard manure will supply crops with about 4 kg S. However, if animal manure is applied in autumn it is likely that a greater proportion of the available S may become lost through leaching.

### CROP REQUIREMENTS FOR SULPHUR

The need for S fertilization to field crops in Norway was first recognized in the early 1960s when S deficiency in a number of crops was observed as a result of intensive use of fertilizers containing little or no sulphur. It was also observed that S deficiency not only reduced the crop yield but also reduced the content and quality of protein and resulted in accumulation of nitrate in plants (Ødelien 1963). A series of field experiments were conducted in the 1960s and a yield increase with S application was observed in a number of cases. Some examples of crop response to S application in a multisite field experiment conducted in the early 1960s by Ødelien (1966) are presented in Table 1. In these experiments the rates of S through gypsum were 0, 8, 24, 48, and 96 kg ha<sup>-1</sup>. Although all crops responded to S fertilization at each site, significant yield differences were obtained only for barley at the Rakkestad and Krødsherad sites (Table 1). At these sites, the plants displayed visual S deficiency symptoms. The significant yield increases as a result of S fertilization varied from 200 to 700 kg grain ha<sup>-1</sup>. The increases in straw yields were of the same magnitude. In cruciferous root crops increases of 1000–1500 kg ha<sup>-1</sup> were obtained in a number of cases (Ødelien 1966). In more recent studies (Singh 1991; Ruud & Singh 1992) S fertilization was again found to have a positive effect on crop yields at several sites but the most significant yield increase was obtained in grasses at Vågønes (Table 2). At this site S fertilization resulted in a yield increase of as much as 25%, but in this study the S concentration in grasses was scarcely affected by S application.

Table 1. Effects of S application on grain yields(kg ha<sup>-1</sup>) of different crops (Ødelien 1966)

Site	Crop	S applied(kg ha <sup>-1</sup> )					LSD
		0	8	24	48	96	
Ullensaker	Barley	3820	4000	3950	4130	4090	240
Spydeberg	Barley	3230	3420	3410	3400	3330	220
	Oat	3220	3220	3220	3170	3400	2240
Rakkestad	Oat	4690	4880	4790	4560	4850	560
	Barley	2480	2820	2870	2810	2780	250
Krødsherad	Barley	1740	2220	2570	2190	2300	380
Vågønes	Grass	8790	9530	9240	9530	9280	1010

Table 2. Crop yields (kg ha<sup>-1</sup>) as affected by S application at different sites (Singh 1991)

Sites	Crop	W/out S	With S(8 kg ha <sup>-1</sup> )
Vågønes	Grass	5750a	7790b
Ås	Wheat	3350a	3520a
Buskerud	Barley	3900a	4050a

Means with the same letter in the same row are not significantly different at  $p=0.05$

Table 3. Sulphur content(%) in barley grain and turnip as affected by S fertilization (Ødelien 1966)

Crop	S applied kg ha <sup>-1</sup>				
	0	8	24	48	96
Barley	0.13	0.13	0.16	0.15	0.15
Turnip	0.16	0.26	0.21	0.30	0.23

Table 4. Sulphur requirements of different crops in southern Norway

Crop	Plant part	Yield level (kg ha <sup>-1</sup> )	Sulphur removed (kg ha <sup>-1</sup> )
Wheat	Grain	4000	6.4
Oat	Grain	3750	6.4
Barley	Grain	3500	5.2
	Straw	3000	3.0
Grass	Herbage	6500	11.1
Swedes	Roots	10000*	30
	Tops	1600*	14
Turnip	Roots	7000*	17.5
	Tops	1800*	6.7
Marrow-stem kale	Whole crop	7500*	30

\* Yields are expressed on dry matter basis

The S fertilization had little or no appreciable effect on the content of S in grain crops but it showed a considerable increase in S content in the dry matter of turnip and swede crops (Table 3). In one case of grain crops where S deficiency was observed in young barley plants, S fertilization resulted in greater S content and a higher ratio of S to N. The S and N contents of normal plants were 0.34 and 4.86% and the corresponding values for deficient plants were 0.11 and 6.55%, resulting in a N and S ratio for normal plants of 14.3 and that for deficient plants of 59.3 (Ødelien 1966).

The amounts of S removed by different crops in southern Norway are presented in Table 4. From the table we can see that cereals removed the lowest amounts and the cruciferous plants (swedes, turnips, and kale) removed the highest amounts of S. This is in agreement with earlier observations by others (Ødelien 1970; Hewgill 1987). Ødelien (1970) reported that the S concentration in plants is not always a good indicator of the S requirements of the crop, although a general relationship between these parameters does exist. He further stated that S deficiency is more common in cruciferous plants and that in Norway cereals seldom display deficiency symptoms. In grasses under intensive treatment with commercial fertilizers, reduced yields in subsequent cuts or years have often been observed (Ødelien 1967).

## CONCLUSIONS AND FUTURE PERSPECTIVES

The results presented in this paper from the experiments conducted in the past or those from recent years suggest a significant response to S application by a number of crops grown in Norway. As sulphur deposition in Norway has decreased substantially (>40%) in the past decade, cases of S deficiency or crop response to S application are on the increase in different parts of the country. This "scenario" demands that we now pay greater attention to this nutrient than it has received in the past few decades. It is essential that the S requirements of crops at risk under different climatic and soil conditions and under different management and fertilizer practices are investigated. We need to understand the process involved in S cycling and to quantify it in different soil types under different agroecological conditions. Effective ways of utilizing S from animal manure also require to be investigated. There is also a need to develop or validate soil test methods which make it possible to predict S availability to crops under varied soil and climatic conditions. Understanding the process involved in S adsorption/desorption by soils and quantification of the leaching losses of S under varied soil and climatic conditions are essential for devising reliable fertilization schemes for different crops or cropping systems.

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# Sulphur Fertilization in Danish Agriculture

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Knudsen, L. & C.A. Pedersen 1994. Sulphur Fertilization in Danish Agriculture. Norwegian Journal of Agricultural Sciences. Supplement No. 15: 45-50. ISSN 0802-1600.

The need for fertilization with sulphur have been examined in about 150 field trials all over the contry. The main results from these trials were, that in most of the trials in oilseed rape was a significant increase of yield due to application of sulphur. In cereals and peas there were no response for sulphur application while there in few of the trials in cut grass showed positive response. The responses were greatest on sandy soils. There were no influence of organic manure on the response for sulphur in fertilizer. The effect of different S fertilizer were similar. The need for sulphur could be predicted by plant analysis.

Key Words: Responses to application of S, Sulphur fertilization.

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In 1987, Sulphur (S) deficiency was recognized as a prominent nutritional problem in oilseed rape production. Therefore in 1988 the Danish Agricultural Advisory Centre began a series of field trials to investigate the need for S. The local advisory service conducted the field trials on commercial farms, meaning that the trials were treated in the same way as the other crops in the field, except for application of S. This also meant that the need for S was tested under a wide range of conditions.

In the period 1988-92 we conducted about 150 trials on different crops. The purpose of these trials was to address the following questions:

- Do crops in general require S application?
- What differences, if any, exist between the soil types?
- Is organic manure capable of supplying sufficient S?
- Are soil and plant diagnostic procedures available to predict the need for S in specific fields?
- What type of S application is most profitable for the farmer?

In this article we will report on our conclusions from these trials, most of which were carried out on oilseed rape crops and this report is based mainly upon these results.

## OILSEED RAPE RESPONSES TO S

**Responses of different soil types**

The result of 38 trials in winter rape crops are presented in Table 1, where the trials are grouped according to the content of clay in the soil. The highest response to S application was measured in soils with a low clay content. The increase in yield on these soils averaged 40%.

Responses were measured even on the soils with the highest content of clay. In Fig. 1, the responses of each trial are plotted as a function of the clay content. The variations between the responses are high.

In Table 2, the responses to S application are given for summer oilseed rape. The trials are grouped according to a clay content above or below 10%. In the summer oilseed rape response is higher on the lighter soils.

Table 1. Response to 40 kg ha<sup>-1</sup> S from K<sub>2</sub>SO<sub>4</sub> in 38 winter rape trials in Denmark, 1990-92

Soil type	No. of trials	% clay	Yield kg ha <sup>-1</sup>	Response to S kg ha <sup>-1</sup>
Less than 5% clay	6	4	1609	640
Between 5 and 10% clay	11	8	2201	414
Between 10 and 15% clay	11	13	3250	136
More than 15% clay	10	18	3329	110
Total/Average	38	12	2708	289

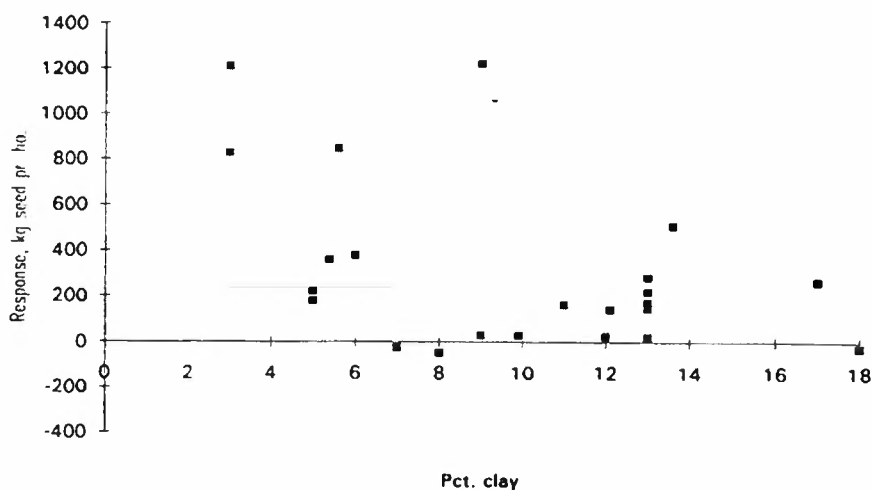


Fig. 1. Responses to S in winter oilseed rape and the clay content in the soil, 38 trials, 1988-92



Table 2. Relative yield of summer rape with application of 40 kg ha<sup>-1</sup> S from K<sub>2</sub>SO<sub>4</sub> in 46 trials, 1988–91 (100 = yield without S application (lowest, highest) in the individual trials)

Year	Sandy soils (<10% clay)	Clay soils (>10% clay)
1991	102 (101–104) (2 trials)	103 (1 trial)
1990	107 (97–134) (11 trials)	
1989	105 (82–185) (17 trials)	103 (102–104) (2 trials)
1988	105 (90–152) (11 trials)	103 (101–106) (2 trials)

### RESPONSES OF TRIALS WITH AND WITHOUT THE APPLICATION OF ORGANIC MANURE

In Denmark, applications of organic manure, especially slurry to oilseed rape, are common. Normally, slurry has a concentration of 0.3 kg t<sup>-1</sup> S. Knowing how much S the slurry can supply to the plants is important.

In Table 3, the results stated in Table 1 are grouped according to application of organic manure. Normally, the trials involving an application of organic manure have received organic manure in previous years.

Table 3. Response to 40 kg ha<sup>-1</sup> inorganic S in winter oilseed rape trials with and without applications of organic manure

Soil type	Without organic manure			With organic manure		
	No. of trials	Yield kg ha <sup>-1</sup>	Response kg ha <sup>-1</sup>	No. of trials	Yield kg ha <sup>-1</sup>	Response kg ha <sup>-1</sup>
Less than 5% clay	3	1750	812	3	1468	468
Between 10 and 15% clay	9	2357	321	2	1500	831
More than 15% clay	7	3297	111	3	3403	107

The response to S is lower on the lightest soil in trials with applications of organic manure. However, responses to additional applications of inorganic S remained high. On the soils with a high clay content, we did not discover any relationship between an application of organic manure and the responses to inorganic S. We conclude that an application of organic manure does not prevent a S deficiency.

## AMOUNT AND TYPE OF S

The trials on winter oilseed rape included application rates of 0, 20, 40 and 60 kg ha<sup>-1</sup> S. The response curves for four trials on light soils and nine trials on clay soils can be seen in Fig. 2. The response curves show that full response is obtained at only 20 kg ha<sup>-1</sup> S. Although the S uptake of high yielding oilseed rape crops is 50–70 kg in the trials, the recommendation for light soils in Denmark is 40 kg ha<sup>-1</sup> S to secure a positive S balance. On clay soils and soils with applications of organic manure, a rate of 10–30 kg ha<sup>-1</sup> S is recommended.

In the trials, S was applied in the form of K<sub>2</sub>SO<sub>4</sub>, but other types of S fertilizers have been tested as well. A good effect was measured for water soluble and non-water soluble SO<sub>4</sub> and also for elemental S.

In the trials, S was applied to winter oilseed rape in March and to summer oilseed rape before drilling. Applying S to winter oilseed rape in the autumn did not result in any additional response; therefore, we assume that applications of S in the autumn are unnecessary.

## PREDICTION OF S NEED

Methods that predict the requirements level for all nutrients are important, therefore many researchers have developed a great interest in plant analysis determination assessing the need for S. In the trials, and in some test fields, analyses of total S in the youngest leaf at bud stage have been conducted.

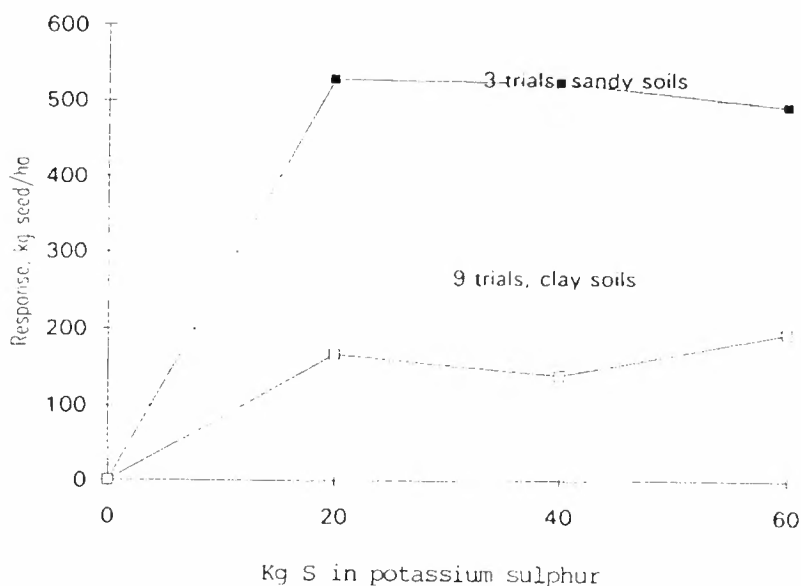


Fig. 2. Responses to application of inorganic S in winter rape, 1991

In Fig. 3 the yields of the rape crops are plotted against the S content (as a percentage of dry matter) in winter and summer oilseed rape. This relationship permits the extrapolation of the value of S required in the plant tissue to sustain a targeted yield level. If the plant analyses show a lower concentration, the crop needs S.

### RESPONSE TO APPLICATION OF S TO OTHER CROPS

In the period 1988–92 we conducted trials in other crops as well (Table 4). With the exception of a few trials conducted on grass, the trials showed no response to S. During 1992, winter cereal crops did not show a response either, despite visible symptoms of S deficiency in many winter wheat and winter barley crops. Because of serious deficiency problems in winter cereals during 1992, about 35 trials were conducted on winter wheat and winter barley crops during 1993.

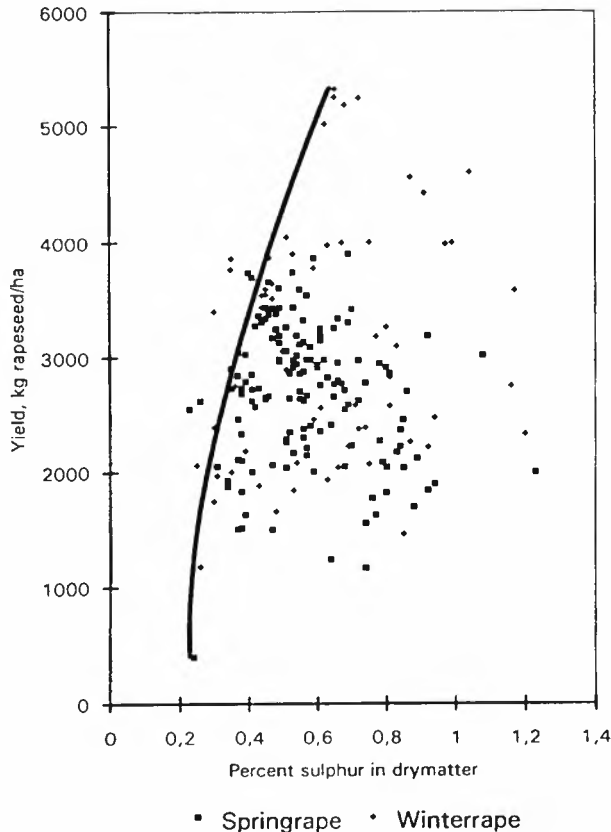


Fig. 3. Obtained yield of oilseed rape as a function of the S content in the leaves. Results from trials and test fields 1988–92

Table 4. Number of trials with application of S in different crops, 1988-92

	No. of trials
Winter wheat	13
Spring barley	16
Peas	13
Grass (for silage)	16

## CONCLUSIONS

Through this research, we were able to conclude that soils throughout Denmark are generally S deficient. Because oilseed rape is a prominent Danish crop, growers should pay close attention to S levels. We discovered that S applications to oilseed rape are most efficient from the beginning of March through the beginning of flowering. Because we found the effects of all types of S fertilizers to be beneficial, S applications are most economical when made as multinutrient fertilizers.

A method for determining a crop's need for S is through a plant analysis test. However, the Danish Agricultural Advisory Centre recommends the following applications:

- on sandy soils, oilseed rape crops require 40 kg ha<sup>-1</sup> S
- on clay soils, oilseed rape crops require 20-30 kg ha<sup>-1</sup> S
- in cereals, peas and grasses, in order to maintain the S balance, we recommend 20 kg ha<sup>-1</sup> S.

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# Sulphur Deficiency in Iceland

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Responses to sulphur applications have been observed widely in Icelandic soils under permanent grass. The soils are mostly mineral, freely or excessively drained, of tephra-loess origin. The first signs of deficiency date back to 1959 although they were not recognized at the time. As a preventive measure, NPK fertilizers sold in Iceland since 1985 contain 2% S on an elemental basis. Sulphur in precipitation in the southern part of Iceland can vary from 3 kg S/ha annually to 12 kg S/ha of which almost 50% can be seaborne. The critical S level in grasses when sulphur deficiency occurs is 0.095% S on a dry matter (DM) basis while the N/S ratio equals 16 when maximum yields are obtained.

Key words: Deficiency, fertilizers, grass, Iceland, sulphur.

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In the last decade sulphur has been recognized as an essential fertilizer nutrient in Iceland in addition to the three main nutrients N P and K. Yield responses that could be attributed to fertilizer-S first appeared in fertilizer trials on grassland in 1959 and 1960 at Hólar in the central north and at Sámsstaðir in the south of Iceland (Jónsson & Björnsson 1964) although not recognized as such at the time. A few years later sulphur deficiency was confirmed on some mineral soils under low precipitation in the northeastern districts. Farmers in the locality had been complaining about declining yields (Sigvaldason 1966) in spite of heavy dressings with ammonium nitrate, which since 1953 had been the nitrogen fertilizer used in Iceland. Subsequent fertilizer trials and observations in the north and northeast of the country showed widespread sulphur deficiencies (Sigvaldason 1966, 1967).

After the start of production of high analysis fertiliser compounds Iceland in 1972 the Fertilizer Monopoly attempted in 1974 to meet this shortage of S as a plant nutrient by introducing a specially designed compound primarily intended for use in the north and northeast. As plant responses to sulphur applications on grassland had also been confirmed in other parts of the country through trials (Helgadóttir et al. 1977) and various observations made in the southern part of the country (Fig. 1), it was decided that from 1985 sulphur would be incorporated in all NPK fertilizers with the exception of one.

The sulphur quantity in those compounds intended for grassland use amounts to 2% elemental S in the form of sulphate. In the beginning this sulphur came from potassium sulphate but lately from sulphate of ammonia. This percentage for sulphur is arrived at, on the one hand from experimental results indicating that 8-10 kg S/ha would suffice in most instances, and on



Fig. 1. Localities where yield responses to sulphur have been observed in Iceland

the other from the general fertilizer recommendation to farmers to use 400–500 kg of compound/ha. Consequently many soils or sites that may not be in immediate need of fertilizer-S will receive unnecessary amounts of S thus causing further leaching and increased soil acidity. Such a practice can perhaps be disputed when bearing in mind that sulphur deficiency does not always show up every year. On the other hand, as means of forecasting this kind of deficiency are of a very uncertain nature, this preventive measure seems justified.

So far sulphur deficiency has only been confirmed on grassland reflecting the fact that permanent grass for haymaking accounts for more than 95% of agricultural land in Iceland. Mainly areas of low precipitation have been affected, particularly areas of low spring and early summer rainfall. It is to be expected that sulphur in precipitation falling on frozen ground will be of little or no value as a potential plant nutrient.

Previously, sufficient sulphur supplies were received through the use of single superphosphate, farmyard manure plus the supply received through the atmosphere. The atmospheric contribution, however, is variable as can be seen from Figs. 2 & 3 for Reykjavík (Rjúpnahæð).

Measurements of sulphur in precipitation in Iceland were first undertaken in 1958 for Reykjavík and in 1960 for Vegatunga, an inland agricultural site 45 km from the coast, in the southern part of the country. Measurements of the latter site were discontinued in 1973. A new observation site was established at Írafoss near the lake Thingvallavatn in 1980 when the

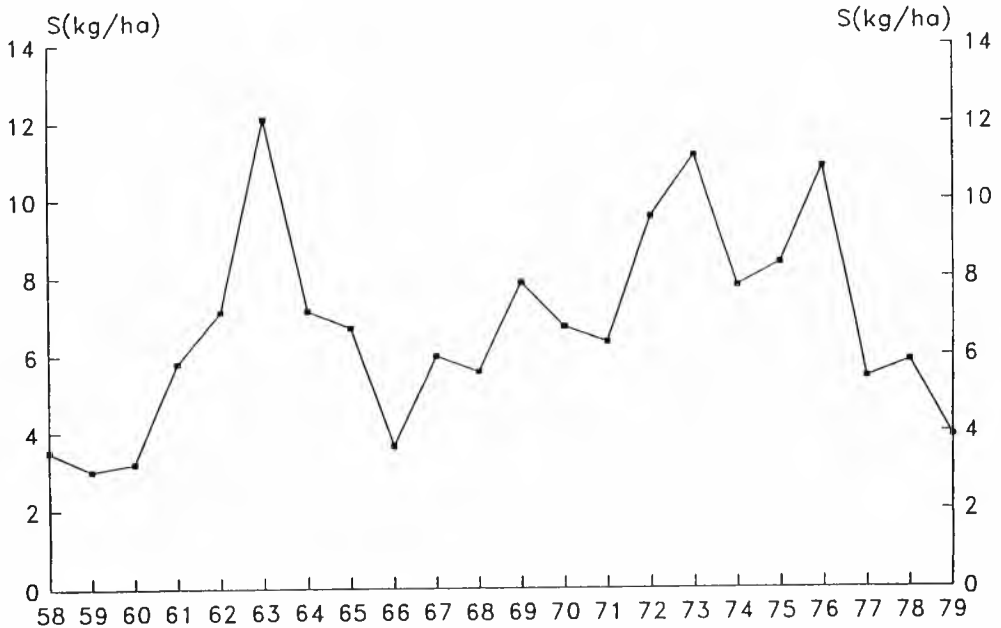


Fig. 2. Annual variations in precipitation-S in Reykjavik 1958-79

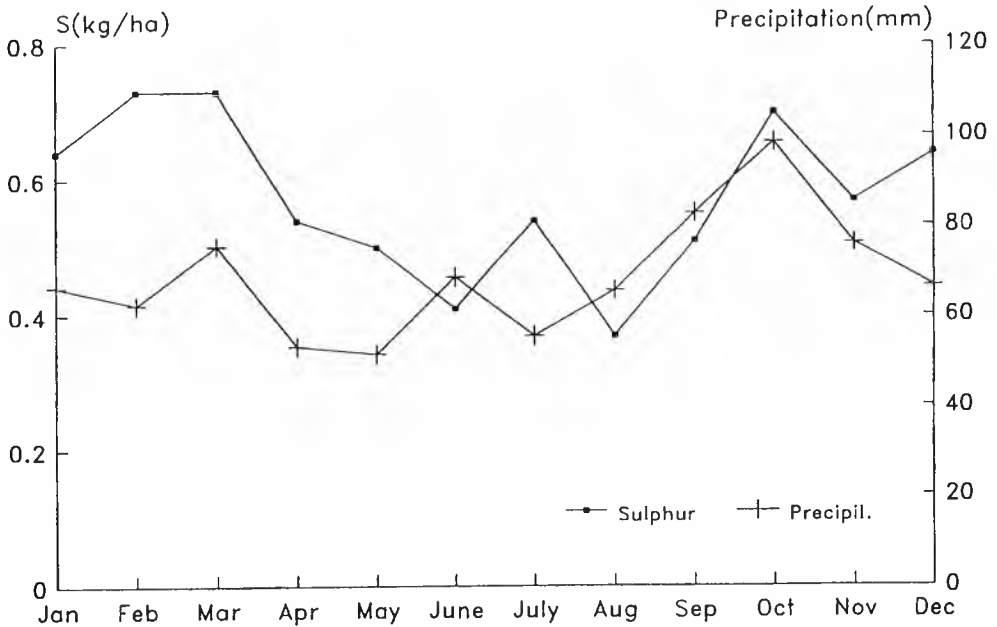


Fig. 3. Monthly variations in precipitation-S in Reykjavik 1958-79 (mean values)

Reykjavík measurements were discontinued. These measurements show clearly the magnitude in annual variations of atmospheric-S from a low value of 4.3 kg S/ha to a high one of 10.9 kg S/ha for the period 1961–72 at Vegatunga, while the corresponding figures for Reykjavík are 3.6 kg S/ha and 12 kg S/ha (Fig. 2). For the longer period 1958–79 in Reykjavík the lowest value is 3 kg S/ha while the higher value remains unchanged. On the other hand, at Vegatunga there seems to be slightly more variation in the monthly mean values as compared to those of Reykjavík both when comparing the same length of periods and also the longer one for Reykjavík. In general these figures follow quite closely the trend for the monthly mean values in precipitation (Veðráttan 1958–79).

In May–July, which is normally regarded as the period of least precipitation but is also the most important for growth of grass and eventual haymaking, sulphur in rainfall has varied from as low as 0.6 kg S/ha to about 2.3 kg S/ha at the agricultural site of Vegatunga. The corresponding figures for Reykjavík are 0.6 kg S/ha and 7.2 kg S/ha (Fig. 3). The latter figure for Reykjavík may seem doubtful as the nearest value to it reaches only 2.3 kg S/ha, indicating more or less the same situation for the two stations.

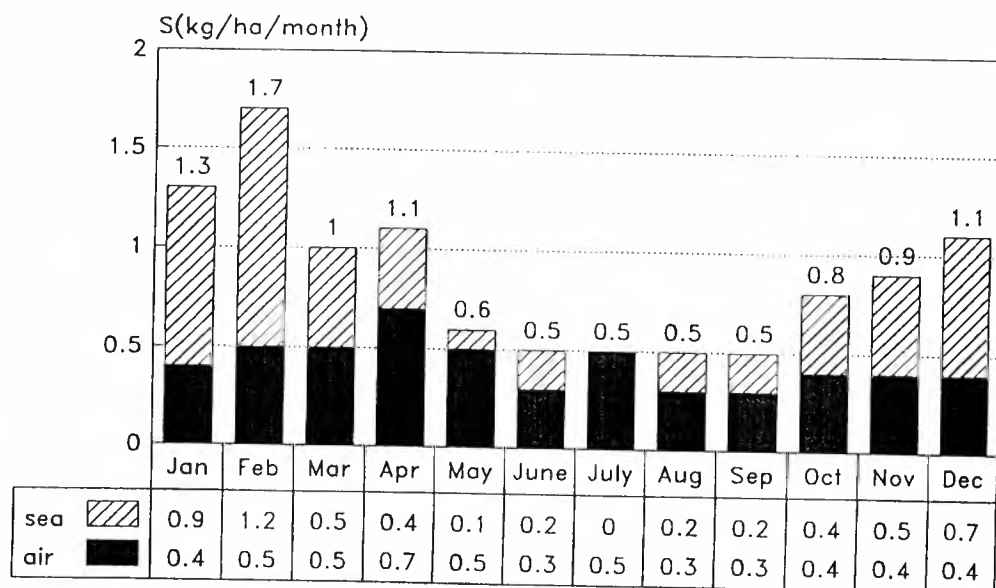


Fig. 4. Sulphur in precipitation 1980–91 air- and sea-borne in a noncoastal area (Írafoss) S-Icel. Monthly mean values

On the other hand how much of this precipitation-S is directly airborne from industrial Europe is not known, nor is the contributing role of sea-spray. For example, the monthly average contribution of such sea-borne S at Írafoss (Veðurstofa Íslands 1993) is estimated to be as high as 70% during the winter months 1980–91, although it can drop down to zero in midsummer (Fig. 4). Írafoss is an inland site some 25 km from the Atlantic coast. It seems logical that distance from the sea is an important factor agriculturally, especially distance from the south coast. In Reykjavík one might thus expect the contribution of seaspray to be even higher than this estimate.



In Iceland a common feature of most soils liable to sulphur shortages is their mineral character of tephra-loess origin, being freely to excessively drained and relatively low in organic matter when bearing in mind other Icelandic soils. On one occasion, however, a response to sulphur application was observed on a peaty soil. This observation, which was attributed to exceptionally low summer temperatures and consequently poor growing conditions, has yet to be followed up with further trials. Another general feature is that these soils have usually received no or little farmyard manure although present day agricultural practice presumably will lead to its better utilization and thus a more effective contribution of manure-S.

In previously mentioned trials Helgadóttir et al. (1977) observed yield responses to sulphur in 15 trials carried out in the period 1959–76, although results in only eight of these were statistically significant ( $p=0.05$ ). In three of these trials testing different levels of sulphur, yield responses were not obtained beyond 5.8 kg S/ha of fertilizer applied S.

These authors attempted to estimate the critical level of S in grasses where deficiency might occur by using the relationship between grass yield and its sulphur content. Accordingly, the critical sulphur level corresponded to 0.095% S on a dry matter (DM) basis with a maximum yield of 6300 kg/ha DM. This critical level however, is considerably lower than observed elsewhere (Jones et al. 1972). On the other hand, when looking only at the ratio of total nitrogen (N) to total sulphur (S) in these Icelandic trials the ratio has varied between 10 and 20. When maximum yield was reached the N/S ratio equalled 16. As a contrast to the critical value this N/S value is in good agreement with published values for grasses receiving an adequate sulphur supply (Dijkshoorn et al. 1960, Jones et al. 1972). These apparent differences between low critical S levels on the one hand and the presumably normal N/S values on the other have been taken to indicate a relatively late maturity of the grasses at the time of sampling. This, however, requires further study.

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56 *Sulphur Deficiency in Iceland*

Veðráttan 1958–62, 1967, 1970, 1974, 1979. Ársyfirlit. Veðurstofa Íslands.

Veðurstofa Íslands 1993. Unpublished data (by courtesy of Veðurstofa Íslands).

# Mineralization of organic sulphur compounds in soil

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In this paper the contents and distribution of soil organic sulphur are reviewed. The fundamental processes of sulphur mineralization and methods to predict the net mineralization of soil organic sulphur are also discussed. Some suggestions for future research in this field are presented.

Key words: Mineralization, soil, sulphates activity, sulphur.

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## POOLS OF ORGANIC SULPHUR

### Contents of organic sulphur

Organic sulphur is the predominant reservoir of sulfur in most agricultural soils. In a study of Australian soils, Freney (1967) found that, on average, 93 percent of total sulphur was in organic forms. These values have been supported by a number of later studies (Germida et al. 1992). As expected, the organic sulphur fraction is generally positively correlated with the soil organic matter content, but exceptions may occur in saline, calcareous or waterlogged soils, where high levels of inorganic forms of sulphur can exist (Germida et al. 1992).

The most reliable methods for estimating the contents of organic sulphur in soils are based on determination of the difference between total sulphur and inorganic sulphur contents. Several methods for total sulphur determination (dry ashing, alkaline oxidation, acid digestion) have been described by Tabatabai (1982 a).

Stevensson (1986) summarized data on relationships between C,N and S in soil organic matter. On average, the reported values of C:N:S ratios in agricultural soils were 107:7.7:1. The variations between different soils are high, with native grass soils, woodland soils and peats having higher C:S ratio than agricultural soils. The N:S ratio of soils generally shows less variation than the C:S ratio (Freney 1986). The relationships may also differ between different soil organic fractions. Bettany et al.(1979) reported higher C:N:S ratios in humic acids (113:10.6:1 to 270:23.3:1) than in the total soil organic matter (61:6.5:1 to 112:9.7:1). In fulvic acids, on the other hand, the C:N:S ratios were lower than in the total soil organic matter (48:3.1:1 to 85:3.9:1).

### **Sulphur in biomass**

Soil organic sulphur occurs in plants, microbial biomass and humic matter, with humic matter as the largest pool. According to Saggiar et al. (1981) and Banerjee et al. (1993), microbial biomass sulphur constitutes only 1–4% of soil organic sulphur. Even though only a small fraction of soil organic sulphur is bound in the microbial biomass, this fraction is very labile and is probably the most active pool in the transformations of sulphur in soil (Germida et al. 1992).

Reliable methods for determination of biomass sulphur are therefore of great importance but are difficult to carry out because of the low content of biomass sulphur and the complicated chemistry of sulphur in living organisms. The published values of biomass sulphur are based on the chloroform treatment technique. Chloroform treatment causes lysis of the microbial cells, resulting in release of cellbound sulphur. The method implies chemical extraction of sulphur from the soil before and after treatment with liquid or gaseous chloroform (Saggiar et al. 1981). Modifications of the method were investigated by Bajerneet et al. (1993). They found that period of chloroform treatment, type of extractant and time of extraction affected the biomass sulphur values. Studies in this field must be regarded as very important as the turnover of microbial biomass sulphur is a key process in soil sulphur transformations.

### **Sulphur in humic matter**

Rather little is known about the exact chemical nature of sulphur in humic matter. Most fractionation studies of soil organic sulphur are based on the binding forms of sulphur. Sulphur in sulphate esters (C-O-S), sulphamates (C-N-S) and sulphated thioglycosides (N-O-S) is reduced to  $H_2S$  after treatment with hydroiodic acid (HI) in an acid solution.  $H_2S$  can thereafter be converted to methylene blue and determined colorimetrically (Tabatabai 1982). The dominant fraction of sulphur reduced to  $H_2S$  by HI is supposed to exist as sulphate esters (Freney 1967). Many authors describe HI-reducible sulphur as organic  $SO_4-S$ . Sulphate is also reduced to  $H_2S$  by HI (Johnson & Nishita 1952). The values of HI-reducible sulphur must therefore be corrected for the content of sulphate in the soil. On average, about 50 percent of soil organic sulphur is reported to exist in HI-reducible forms. The variations are great, however, and values from about 30 to 60% are reported (Stevenson 1986). Bettany et al. (1973) suggested that the main part of HI-reducible sulphur is associated to side chains of humin, humic acids and fulvic acids. Most of the remaining soil organic sulphur in soil, determined as the difference between total organic and HI-reducible sulphur, is thought to be bound to carbon (Freney 1967). Amino acid sulphur constitutes the main part of carbon-bound sulphur in soils but it is believed that also a large number of more complex sulphur compounds are included in this fraction. The distribution of sulphur in a number of Australian soils is illustrated in Fig. 1.

### **Organic sulphur in soil solution**

In studies of camp and non-camp soils, Nguyen & Goh (1992a,b) found that organic sulphur in soil leachates was about 0.5–1.5% of total soil organic sulphur. Of total sulphur in the leachates, about 15–40% was in organic form.

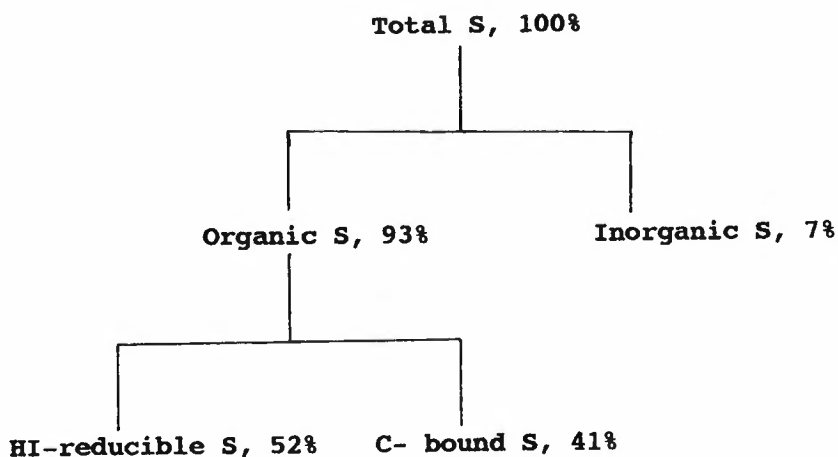


Fig. 1. Distribution of sulphur in 24 Australian soils (from Freney 1967)

### Forms of sulphur in living cells compared to that of humic matter

Carbon-bound sulphur in the amino acids cysteine and methionine are the dominant forms of organic sulphur in living cells. Only a small proportion of sulphur in cells is bound to oxygen, mainly occurring in structural components of plant and animal tissues. In humic matter, on the other hand, C-S and C-O-S compounds occur in about the same amounts.

The question arises why the relations between C-S and C-O-S differ between living matter and soil humic matter, and a number of possible explanations may be suggested. One is that the C-O-S compounds are comparably stable and therefore accumulate to high levels in soil until there is an equilibrium between synthesis and decomposition. However, there is no support in the literature for this idea.

McLaren & Swift (1977) suggested that HI-reducible sulphur of fulvic acids could be a result of oxidative breakdown of C-bonded sulphur, and thereby a result of enzyme-catalyzed processes. Another possible explanation is that the high levels of sulphur esters in soil humic matter are a consequence of incorporation of sulphate into organic matter. The formation of sulphate esters from sulphate in soil is reported by several authors and was demonstrated by Fitzgerald & Strickland (1982,1987) in hardwood forest soils. These authors found that  $^{35}\text{SO}_4^{2-}$  was rapidly converted to ester sulphate and carbon-bonded sulphur. In other studies, the involvement of ATP-sulphurylase in the reaction has been suggested (Strickland & Fitzgerald 1985; Swank & Fitzgerald 1984). This enzyme catalyses the first step in the formation of organic sulphur in living cells.

The possibility of non-biologic incorporation of sulphate into organic matter does not appear to have been discussed in the literature.

## MINERALIZATION OF SULPHUR IN SOILS

### **Biological and biochemical mineralization**

In their discussions on sulphur mineralization processes, Germida et al. (1992) use the terms biological and biochemical mineralization. This approach gives a good insight into the fundamentals of the processes.

Biological mineralization is directly coupled to microbial activity and is affected by the microbial need for carbon and energy. When the micro-organisms oxidize carbon to obtain energy, other elements associated with carbon are mineralized or incorporated into the microbial biomass. In these processes, water-soluble organic sulphur compounds may probably occur as intermediates. There will be a net mineralization of sulphur if the content of sulphur in the substrate is higher than the requirement of the growing micro-organisms. Carbon-bound sulphur will be released as  $H_2S$  or sulphate, whereas esterbound sulphur will be released only as sulphate.

In biochemical mineralization, the release of sulphur bound as sulphate esters is catalyzed by extracellular enzymes. The production of extracellular enzymes is supposed to be controlled by the need for sulphate by both microbes and plants.

In mineralization studies, incubation techniques and type of system greatly affect the amount of sulphur released. In an open system, where sulphur is lost from soil by leaching and plant uptake, the mineralization rate is higher than in a closed system, where the sulphate concentration increases during incubation. These effects have been well demonstrated by Maynard et al. (1983,1985). The lower mineralization rate in a closed system is probably a consequence of retarded biochemical mineralization affected by an increasing sulphate concentration.

### **Methods to predict the net release of soil organic sulphur**

Chemical procedures to predict the potential of a soil to release plant available sulphur can be based on the following approaches:

- Estimation of the C:S relations of soil organic matter or added plant residues
- Chemical fractionation of soil organic sulphur
- Estimation of biomass sulphur
- The use of mild extractants.

The C:S relationships of total soil organic matter offer little information about the sulphur mineralization potential, a reason probably being the great variation in binding forms of sulphur in soil organic matter (Bettany 1979, Ghani et al. 1991). The C:S relationships of added plant residues, on the other hand, may have great effects on the amount of sulphur released after soil incorporation. In a study by Wu et al. (1993), the amendment of rape leaves (1% w/w) with C:S ratio 64:1 resulted in a significant increase of sulphate in the soil. The addition of the same amount of barley straw with C:S ratio 206:1 gave about the same levels of sulphur as the unamended soil. Biomass sulphur increased after both amendments but the increase in the rape-amended soil was about three times higher than in the straw-amended soil. The results show that the sulphur content of the recently added organic matter is of great importance for the net mineralization of sulphur and the formation of labile sulphur fractions. The fractionation of sulphur into C-O-S and C-S compounds, as well as fractionation into

humic acid and fulvic acid sulphur, has been described by several authors (Bettany et al. 1979, Freney 1986). There are results indicating that sulphate esters in side chains of humic- and fulvic acids are the most labile sulphur compounds in humic matter (Bettany et al. 1973, 1979). Contradictory results were reported by Ghany et al. (1991). They found that C-bonded sulphur, probably in the form of amino acid sulphur, best predicted the mineralization of sulphur and that C-O-S sulphur contributed only little to the mineralized sulphur. They suggested that it may be possible to use C-bonded sulphur for predicting sulphur mineralization from soils. In their excellent discussion they pointed out, however, that the complexity of the mineralization processes complicated the identification of a homogeneous, potentially mineralizable, sulphur pool. The reason why some authors have suggested C-O-S sulphur to be the most labile fraction, while others have suggested C-S, may be that the balance between synthesis and degradation of organic sulphur compounds in soil is difficult to predict by means of chemical fractionation techniques. It is obvious that there are still no reliable methods for fractionation of sulphur into biologically meaningful fractions.

The problems with estimation of biomass sulphur were discussed earlier in this paper. The use of mild extractants as salt solutions or diluted acids has been suggested by some authors for estimation of labile fractions of soil sulphur. Blair et al. (1991) treated the soil with 0.25M KCl at 40°C and found good correlation between the sulphur released during extraction and the uptake by plants.

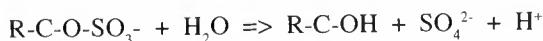
In a study of soil organic matter as a source of plant available sulphur, Freney et al. (1975) concluded that it will not be a simple matter to develop a test method for plant-available sulphur because several chemical fractions of soil organic sulphur contribute to the available sulphur supply. Their conclusion still describes the situation today, nearly twenty years later, although a large number of mineralization studies have been reported.

### **Sulphatase activity and mineralization of sulphate esters**

#### *Methods*

Sulphatases are enzymes that catalyse the hydrolysis of sulphate esters, giving sulphate as one of the products. Reviews of sulphatases in soil have been published by Skujins (1976), Speir & Ross (1978) and Germida et al. (1992).

The method used in most sulphatase studies in soils was suggested by Tabatabai and Bremner (1970a). They used p-nitrophenyl sulphate as substrate. In the enzymatic reaction there is formation of p-nitrophenol which is readily extracted from the soil. Estimation of the p-nitrophenol content of the extract is easily performed by a colorometric method. A modification of the method suitable for organic soils has been suggested by Sarathchandra & Perrott (1981). Analysis of the released sulphate is also possible but gives lower precision because the values must be corrected for the original sulphate contents of the soil. The enzymatic reaction proceeds as follows:



In studies of soil sulphatase activity, the substrate is added to the soil in excess, giving a zero order enzymatic reaction. According to the Michaelis-Menten kinetics, the reaction rate under these conditions is a function of the content of active enzymes in the system. It has been

shown that p-nitrophenyl sulphate is not hydrolysed in autoclaved soils. As the substrate is an aromatic sulphate ester the enzymatic activity is often named arylsulphatase activity.

#### *Occurrence and substrate specificity of sulphatases*

Extracellular sulphatases are thought to be necessary for the release of sulphate from sulphate esters in soil. Sulphatases have been detected in plants, animals and micro-organisms (Speir & Ross 1978, Dodgson et al. 1982) and it is supposed that the extracellular sulphatases in soil have been released mainly by plant roots and microorganisms. Fitzgerald & Strickland (1987) demonstrated that the hydrolysis occurred in the presence of an antiseptic agent, which indicates that the reaction was catalyzed by preformed sulphatases.

Sulphatases can hydrolyse a large number of organic sulphate esters but we do not know much about the specificity of the enzymes. Houghton & Rose (1976) investigated the release of sulphate from five different sulphate esters in neutral, acid and alkaline soils. There was a release of sulphate from all substrates and in all soils. For some esters there was an immediate release and for others there was a lag phase. These results indicate that there is a variety of sulphatases in the soil. The use of arylsulphatase activity as a measure of the capability of a soil to hydrolyse naturally occurring sulphate esters may therefore be discussed (Lee & Speir 1979).

Factors affecting arylsulphatase activity in soils have been studied by many authors (e.g. Tabatabai & Bremner 1970b, Speir et al. 1980; Speir 1984; Baligar & Wright 1991 and Neal 1982). The pH-optimum of the reaction lies between 5.5 and 6.5. The activity is generally positively correlated with organic matter content and the seasonal variations are rather low compared with the variations in microbiological activity. As many soil factors affect enzyme activities, results from different studies must be compared with great care.

#### FUTURE RESEARCH

Organic sulphur is the dominant pool of sulphur in most agricultural soils. Reliable methods for prediction of the potential mineralization and a good understanding of the factors that influence mineralization and immobilization of sulphur are therefore of great importance for possibilities to apply balanced sulphur fertilization to crops. Projects with the following aims are therefore suggested:

- More reliable methods to estimate microbial biomass sulphur.
- Better understanding of the enzymatic processes that catalyze the immobilization and mobilization of sulphate in soil.
- More precise methods to predict the sulphur mineralization potential of organic fertilizers (e.g. manure, sludge, compost) and crop residues.
- Better methods for fractionation of soil sulphur into fractions of biological importance.



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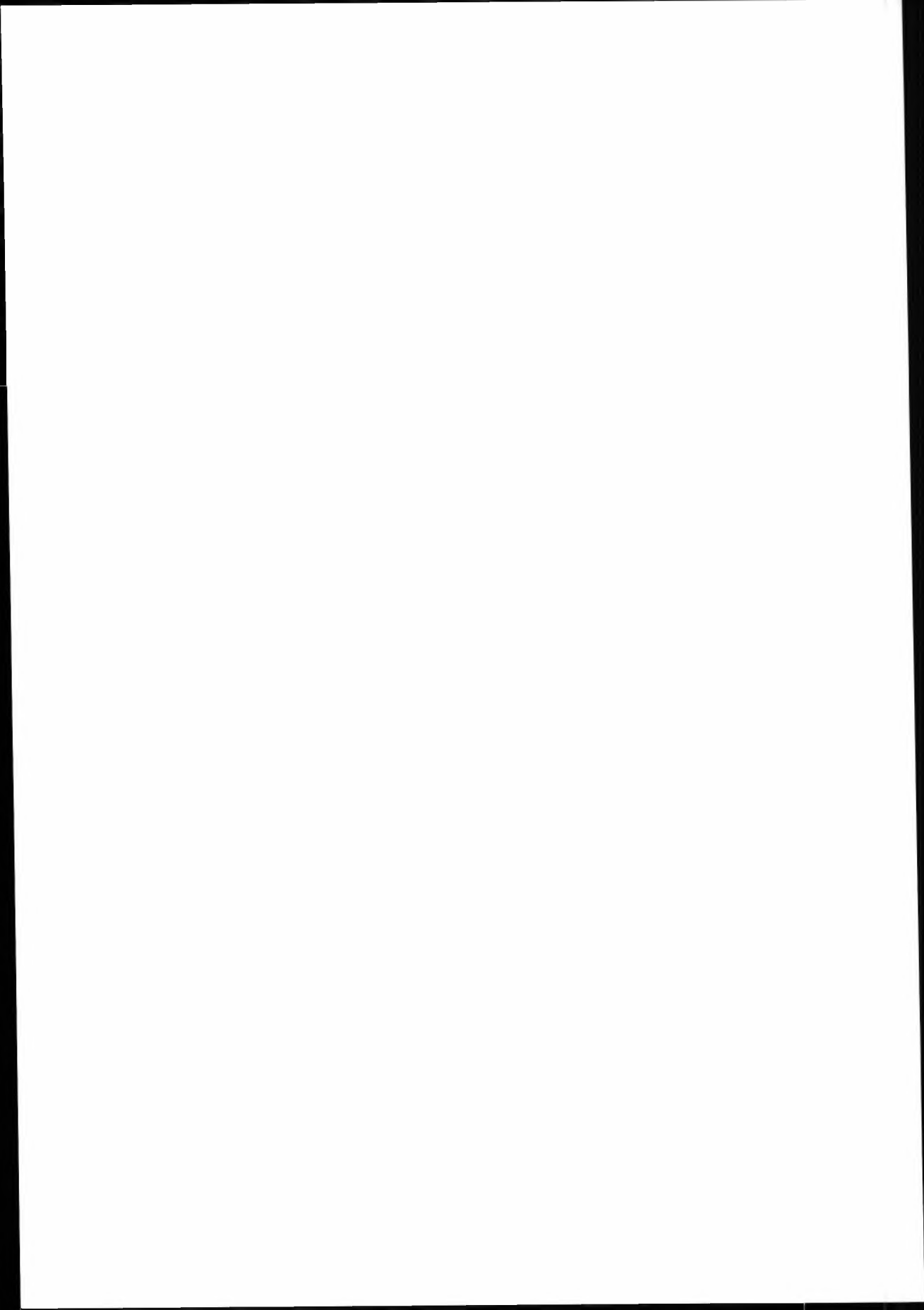
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# The AAAC and AAAC-EDTA extractable sulphur in Finnish soils

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The acid ammonium acetate-EDTA (AAAC-EDTA) extraction method used in Finland in soil testing for microelements was compared for testing soil sulphur with the acid ammonium acetate (AAAC) method which is used for macronutrients. The AAAC-EDTA method had stronger extraction power than AAAC. The ratios between the methods were 1:1.5 for clay soils, 1:1.7 for coarse mineral soils and 1:1.9 for organic soils. None of these methods can be preferred. When analysing macronutrients in soil testing for fertilizer recommendation, the S content can be analysed economically from the same AAAC extractant. When the AAAC-EDTA method is used for trace element extraction in environmental monitoring the determination of S from the same extract provides useful additional information.

Key words: AAAC method, AAAC-EDTA method, soil sulphur, soil testing

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In Finland problems of both excess and deficiency of sulphur have occurred. In the 1950s an excess of sulphur was found as a local problem in some coastal areas of the Gulf of Bothnia (Purokoski 1958). The sulphur-rich sediments became too acid after reclamation and draining when they were dried and oxidized. In order to solve these problems the sulphur content of soil was also analysed. The method used by Purokoski & Lakanen (1959) was complexometric titration and sulphur was extracted from the soil with acid ammonium acetate (AAAC-method, Vuorinen & Mäkitie 1955).

The problem of sulphur deficiency has been rare (Salonen et al. 1965). Therefore the determination of soil sulphur and the development of methods to analyse it have received only a modicum of attention.

When agriculture became more intensive and compound fertilizers without sulphur came into general use, the need to analyse sulphur increased. Salonen et al. (1965) suggested a 0.01 M CaCl<sub>2</sub> solution for extracting soil sulphur (Nemeth 1963). This method was used in a soil testing laboratory in Finland (Viljavuuspalvelu OY) up until 1990.

New methods for extracting sulphur in soils have been studied continuously (Korkman 1973; Yli-Halla 1987) and the AAAC method has also been included in tests, while at the same time the comparisons with earlier methods have been carried out (Chesnin & Yien 1951,;

Bardsley & Lancaster 1960; Saalbach et al. 1962; Öien 1979).

Differences between the extractants become apparent, but also differences between the analytical methods, e.g. plasma emission spectrometry and gravimetry gave dissimilar results for the same extract (Yli-Halla 1987).

For testing the macronutrients of farmers' soils, plasma emission spectrometry (ICP) has been used since 1986 and the AAAC extractant method since 1955 (Vuorinen & Mäkitie 1955). In order to facilitate the analysis work, from 1991 the Finnish soil testing survey (Viljavuuspalvelu OY) also began to determine sulphur with ICP from AAAC extractants. The Institute of Soils and Environment at the Agricultural Research Centre has used this method in earlier work (Erviö et al. 1990; Sillanpää & Jansson 1991).

Micronutrients have been analysed in Finland by the AAAC-EDTA-method as early as 20 years ago (Lakanen & Erviö 1971). Sillanpää (1982) used this method for a large global material and sulphur determinations have also been made (Jansson 1994). In order to compare the results of Erviö et al. (1990) and Jansson (1994) and also to widen the application of this universal extractant, there was a need to evaluate the potential of AAAC-EDTA multielement extractant for assessing soil sulphur status, which is the aim of this study.

## MATERIALS AND METHODS

The soil samples used in the study were those collected for an investigation of mineral elements in timothy (*Phleum pratense* L.) fields throughout Finland (Erviö et al. 1990). A total of 1340 samples consisting of 179 clay, 892 coarse mineral and 269 organic soils were collected. For the analyses, soil samples were air-dried and crushed to pass through a 2-mm mesh sieve. Sulphur (S) was extracted using two methods. One was the acid ammonium acetate method (0.5 M  $\text{CH}_3\text{COONH}_4$ , 0.5 M  $\text{CH}_3\text{COOH}$ , pH 4.65), abbreviated here to the AAAC method (Vuorinen & Mäkitie 1955). The other one was the method using an acid ammonium acetate-EDTA solution (0.5 M  $\text{CH}_3\text{COONH}_4$ , 0.5 M  $\text{CH}_3\text{COOH}$ , 0.02  $\text{Na}_2\text{EDTA}$ , pH 4.65) (Lakanen

Table 1. Means, medians, minimum and maximum values for pH and humus content of the sample material

Soil type	pH( $\text{H}_2\text{O}$ )				Humus %			
	Mean	Med.	Min.	Max.	Mean	Med.	Min.	Max.
Clay soils, n=179	5.9	5.9	4.7	7.5	6.8	5.7	2.0	13.9
Coarse mineral soils, n=892	5.9	5.9	4.4	7.7	6.4	5.8	1.5	18.9
Organic soils, n=269	5.3	5.3	4.4	6.4	51.2	48.2	8.9	89.3
All soils, n=1340	5.8	5.8	4.4	7.7	15.4	6.5	1.5	89.3

& Erviö 1971). In both cases the extracting ratio was 1:10 by volume and the extracting time one hour. The concentrations were determined from the solution by plasma emission spectrometry.

The content of organic matter varied in mineral soils from 1.5% to 18.9% and in organic soils, which consisted of 11 gyttja, 94 mould and 164 peat soils, from 8.9% to 89.9% (Table 1). pH(H<sub>2</sub>O)-values ranged from 4.4 to 7.7.

Data were subjected to statistical regression analyses in order to relate the results obtained with the two extraction methods with each other and to modify the interpretation of the AAAC method used in Finland for the AAAC-EDTA method.

In Finland a system of seven classifications is used for interpretation of soil test data. The limit values of this classification for S are 3, 6, 10, 15, 50 and 150 mg/l when extracted by the AAAC method. The extraction values were classified as poor, rather poor, fair, satisfactory, good, very good and excessive.

## RESULTS AND DISCUSSION

Less sulphur was extracted by the AAAC method than by the AAAC-EDTA method. There were also differences between the S content of soil groups (Table 2). On average coarse mineral soils had less sulphur than the others when S was extracted by the AAAC method and organic soils had the greatest content, on average 25.0 mg/l (ranging from 4.3 to 269.9 mg/l). Using the AAAC-EDTA method, the clay soils were found to have the smallest content of sulphur, 27.5 mg/l (4.8–215.6 mg/l). The highest levels were extracted from organic soils. The ratios between results analysed by these two methods were 1:1.5 for clay soils, 1:1.7 for coarse mineral soils and 1:1.9 for organic soils.

Table 2. Means, medians, minimum and maximum values AAAC and AAAC-EDTA extractable soil sulphur

Methods		Clay soils n=179	Coarse mineral soils n=892	Organic soils n=269	All soils n=1340
AAAC extr. sulphur mg/l	Mean	18.1	17.4	25.0	19.0
	Median	14.8	15.0	19.6	15.6
	Min.	6.8	4.0	4.3	4.0
	Max.	150.8	151.0	270.0	270.0
AAAC-EDTA extract. sulphur mg/l	Mean	27.5	30.2	47.5	33.3
	Median	21.8	26.3	37.0	26.9
	Min.	4.8	5.7	2.2	2.2
	Max.	215.6	194.5	301.0	301.0

## S in clay soils

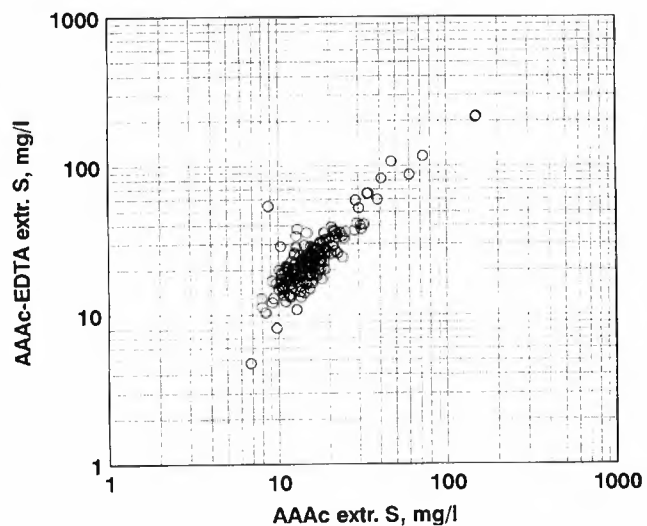


Fig. 1. Relationship between acid ammonium acetate (AAAC) and acid ammonium acetate-EDTA (AAAC-EDTA) extractable in clay soils

## S in coarse mineral soils

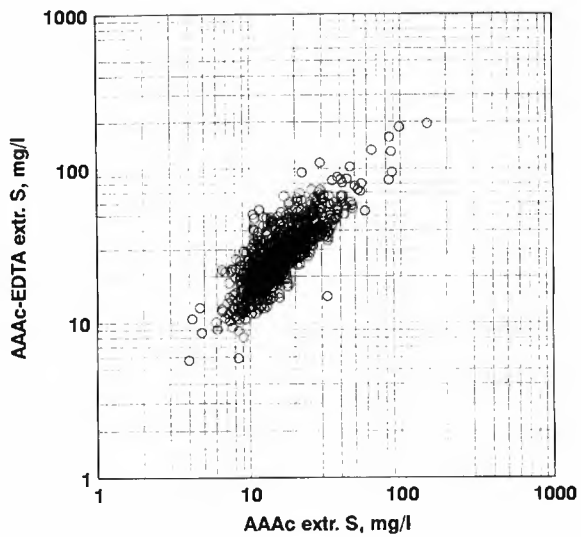


Fig. 2. Relationship between AAAC and AAAC-EDTA extractable sulphur in coarse mineral soils



## S in organic soils

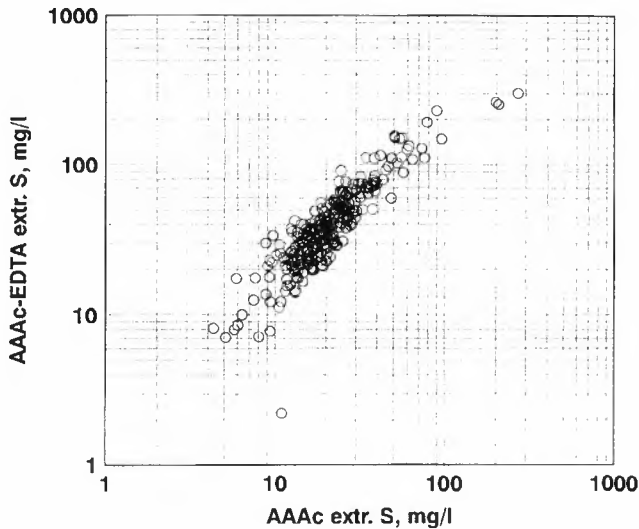


Fig. 3. Relationship between AAAC and AAAC-EDTA extractable sulphur in organic soils

When Lakanen & Erviö (1971) selected the method for analysing soil micronutrients they already found that the addition of a suitable chelating agent (EDTA) made it possible to extract trace elements bound by soil organic matter. The AAAC-EDTA and neutral AAAC-EDTA methods were the most powerful extractants. Even at that time the AAAC-EDTA method was suggested as being suitable for the simultaneous extraction of both macro- and micronutrients even though the amount of phosphorus increased considerably. Furthermore, in this study the AAAC-EDTA method extracted more sulphur than the AAAC method and proportionally more from organic soils than mineral ones. On the other hand, according to Korkman (1973) the amount of sulphur extracted by the AAAC method was quite independent of the content of organic matter.

Yli-Halla (1987) has reported earlier, that AAAC was marginally the most accurate extractant in predicting the sulphur uptake of plants, because the amounts of sulphur extracted by this method were in close correlation with the yields and sulphur uptake. Here the values for the amounts of sulphur extracted by the AAAC and AAAC-EDTA methods were so similar that the suggestion to transform the results reached by AAAC-EDTA according to the fertility classes assessed by the AAAC method seems to be justified.

To obtain the limit values for interpretation of the results made by the AAAC-EDTA method a regression equation was calculated ( $y = -1.9 + 1.95x - 0.00318x^2$ ,  $r = 0.91^{***}$ ). It was used to transform the AAAC limit values to those of the AAAC-EDTA method (Fig. 4).

According to the classification used in Finland, in the lowest class there were no samples (Fig. 5). In the classes ranked as rather poor and fair, where the S deficiency appears, there was

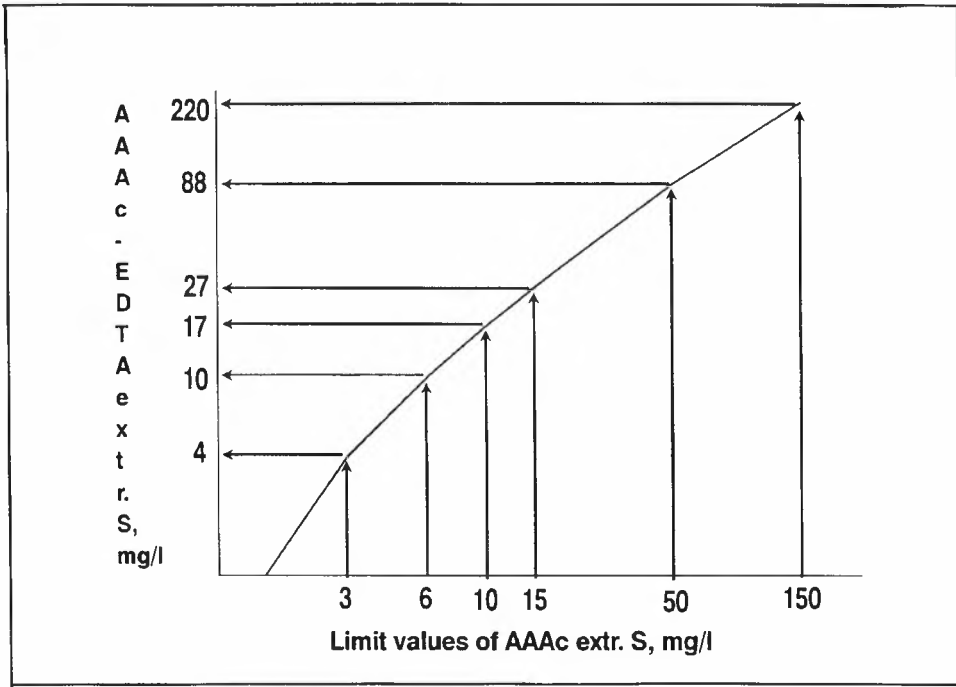


Fig. 4. Transformation of the AAAC extractable sulphur limit values used in Finland to those of the AAAC-EDTA method

Table 3. Limit values (mg/l soil) for AAAC-EDTA extractable soil sulphur derived from AAAC method limit values

Fertility class	AAAC method <sup>1)</sup>	AAAC-EDTA method <sup>2)</sup>
Poor	below 3	below 4
Rather poor	3 - 6	4 - 10
Fair	6 - 10	10 - 17
Satisfactory	10 - 15	17 - 27
Good	15 - 50	27 - 88
Very good	50 - 150	88 - 220
Excessive	over 150	over 220

1) Acid ammonium acetate method

2) Acid ammonium acetate-EDTA

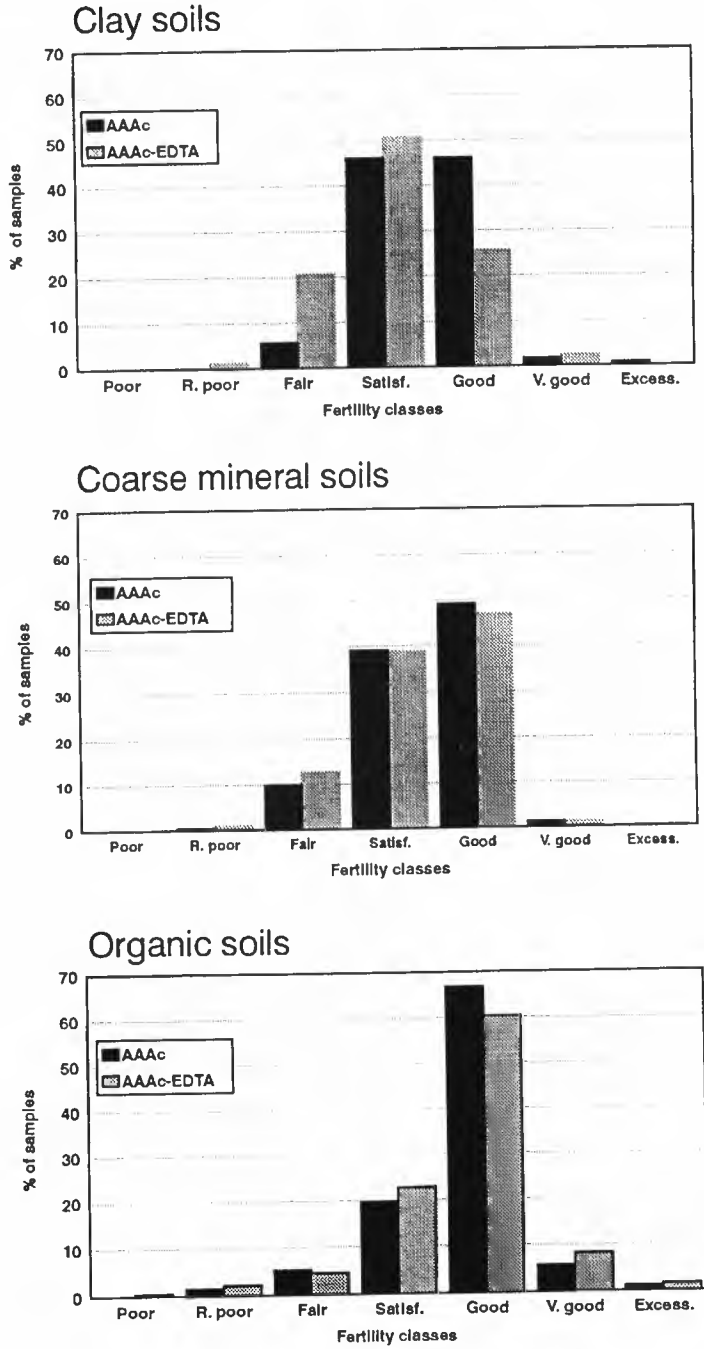


Fig. 5. The distribution of samples into fertility classes used in Finland and according to counted limit values for the AAAC-EDTA method in three soil type groups

less than 10% of organic and clay soils. On the other hand, in the two highest classes there were also only a few samples.

The reason for the distribution of this material being so concentrated in the best classes is that samples were taken from farmers' fields that were in good condition. When preparing the classification for sulphur the material also included soils with sulphur deficiency (Viljavuustutkimuksen 1991).

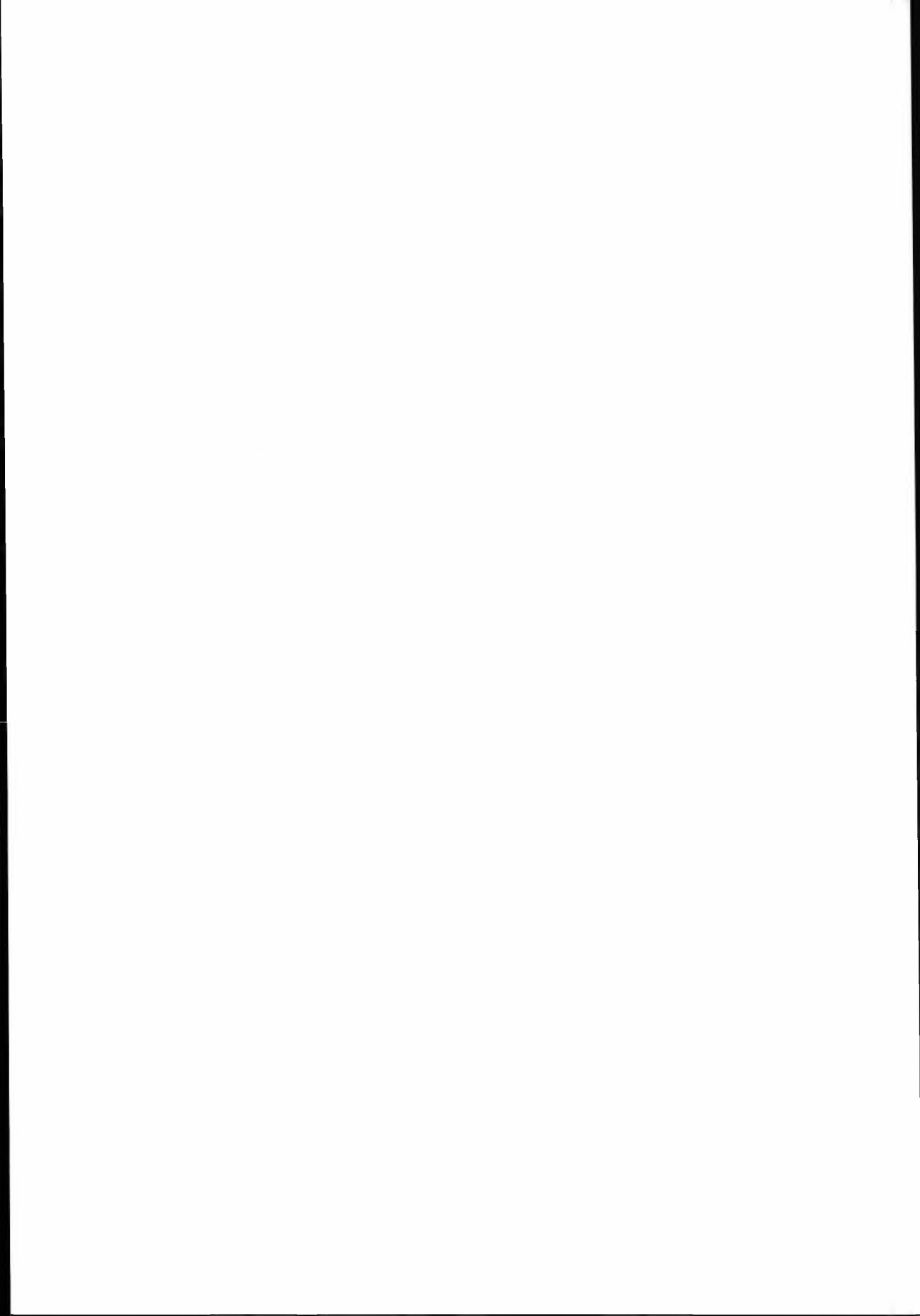
The distribution of samples into fertility classes according to the AAAC-EDTA method was quite similar to earlier distribution of organic and coarse mineral soils. In the group of clay soils the amount of samples extracted by the AAAC-EDTA method increased in the class ranked as fair and decreased in the class ranked as good. This probably means that the clay fraction plays some role in sulphur fixation in the soil.

At the Institute of Soils and Environment the AAAC-EDTA method has been found appropriate for analysing calcium, potassium and magnesium (Lakanen & Erviö 1971) and, in addition to the other micronutrients, boron, which up until now has been extracted by hot water (Sippola & Jansson 1993). The AAAC-EDTA method has been recommended and used as a reference method in European networks on trace elements (FAO Report 1977) and there seem to be no restrictions to extracting sulphur also by this method.

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# Soil organic matter as a source of plant-available sulphur

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A low sulphur input from external sources means that the release of sulphur (S) from soil organic matter is an important source for the sulphur supply of plants. The sulphur supplying capacity of five Danish soils to plants grown in pots was investigated. Net mineralization of S differed between soils (3.2 to 6.7  $\mu\text{g S g soil}^{-1} \text{ year}^{-1}$ ) independently of cropping with the exception of one soil. Physical protection of organic sulphur was studied by extracting soil organic sulphur from soils incubated with  $^{35}\text{S}$ . The extraction was carried out with no dispersion or full dispersion using an ultrasonic probe (1800 J mL $^{-1}$ ). It was found that more  $^{35}\text{S}$  was incorporated in the easily extractable sulphur (without dispersion) than in the more protected organic matter released by dispersion.

Key words: Mineralization, pot experiment, soil organic sulphur, sulphur.

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In recent years an increase in the frequency of sulphur (S) deficiency has been observed in Denmark, mainly as a result of decreasing atmospheric inputs of S and the use of low S-containing fertilizers. This means that the release of S from soil organic matter is of even greater importance for the supply of sulphur to plants.

Soil organic S is present as C-bonded S (C-S) or as sulphate esters (C-O-S). McGill & Cole (1981) suggested that mineralization of organic S involves two types of processes: biological and biochemical mineralization. Biological mineralization is considered to be driven by the microbial need for organic C to provide energy, and sulphur released as sulphate is a by-product of the oxidation of C to carbondioxide. Biochemical mineralization is the release of sulphate from C-O-S through enzymatic hydrolysis. Since S in the form of sulphate esters can be readily hydrolysed, the C-O-S fraction is considered to be the most labile fraction of soil organic S (Biederbeck 1978).

The aim of this paper is to present the major results from a pot experiment designed to determine the sulphur supplying capacity of the organic fraction of different Danish soils by growing plants in the pots. In addition, some results from a study of the physical protection of soil organic sulphur will be discussed briefly.

## MATERIALS AND METHODS

The experiment presented will later be published elsewhere in more detail, so only the outline will be given here.

**Pot experiment**

Samples from the plough layer of five Danish experimental stations were collected (Table 1), mixed with quartz sand (1:1 w/w) and placed in polyethylene pots outdoors, under a transparent roof. In spring, inorganic S was removed by leaching with water, and half of the pots were planted with Italian rye grass (*Lolium multiflorum* Lam.) and the rest with spring rape (*Brassica napus* L.). Nutrients N, P, K, Mg, Cu and Mn were applied before planting. After harvesting of the year 1 crop, the pots were left standing until spring the following year, leached with water and planted with either Italian rye grass again or spring barley (*Hordeum vulgare* L.).

Table 1. Some properties of the experimental soils

pH(CaCl <sub>2</sub> )	St Jydevad 5.8	Lundgaard 6.0	Foulum 5.4	Askov 5.7	Roskilde 6.4
	Percent				
Clay	3.6	4.6	6.8	9.7	11.4
Silt	3.7	4.8	12.0	12.4	15.5
Sand <0.2mm	10.1	19.2	48.9	37.3	45.5
Sand >0.2mm	80	69.1	28.6	36.3	25.2
Total C	1.52	1.41	2.25	2.59	1.39
Total N	0.11	0.11	0.18	0.19	0.13
	µg S g soil <sup>-1</sup>				
Total S	169	178	260	317	187
Organic S					
C-S	115	122	161	227	101
C-O-S	52	52	92	85	82
Inorganic S	2	3	7	5	4

Total S was determined in the harvested crops and roots by turbidimetry after wet ashing, and in soils and leachates by a reduction-distillation method.

**Study of physical protection of soil organic sulphur**

The two sandy soils were incubated for 2, 4 and 8 weeks at 10 and 25°C. Carrier-free <sup>35</sup>S was added prior to incubation, so its incorporation into different organic S pools could be followed. The organic matter from the soils was extracted as described by Keer et al. (1990), with either no dispersion or full dispersion with an ultrasonic probe (1800 J mL<sup>-1</sup>). The concentration of S and the recovery of <sup>35</sup>S were determined in the extracts.



## RESULTS AND DISCUSSION

The content of sulphur in the different fractions is presented in Table 1. The total S content varies between 169 and 317  $\mu\text{g S g soil}^{-1}$  of which 97–99% is organic. Sulphate esters account for 27–45% of the organic S and there is a good correlation between total C and carbon-bonded sulphur, as would be expected. The inorganic S in Table 1 corresponds to the removal of S in leachate before year 1 planting and it is therefore thought that S taken up by plants originates from mineralization of organic S.

The total removal of S in above-ground plant material is displayed in Fig. 1, as well as the amount of S in leachates and S in root material just after the last harvest for the different combinations of soil type and crop. The amount of sulphur removed in Italian rye grass in year 1 was significantly higher than that removed in spring rape for all soils. A difference in harvest time (two cuts of rye grass were taken after the harvesting of spring rape) explains only 60–70% of this difference, which must also be ascribed to inhibition of the growth because of sulphur deficiency. Apparently, the spring rape needs a certain amount of sulphur to grow at all, so because of growth inhibition the rape may not have taken up all the sulphur released from the soil. The leachates collected after the spring rape, in turn, contained considerably more S than those collected after the ryegrass. Thus the total removal of S in year 1 (in plant material plus leachates) was approximately of the same size for both crops with the exception of the Foulum soil, where the total removal of S in ryegrass plus leachate was significantly highest ( $P=0.001$ ).

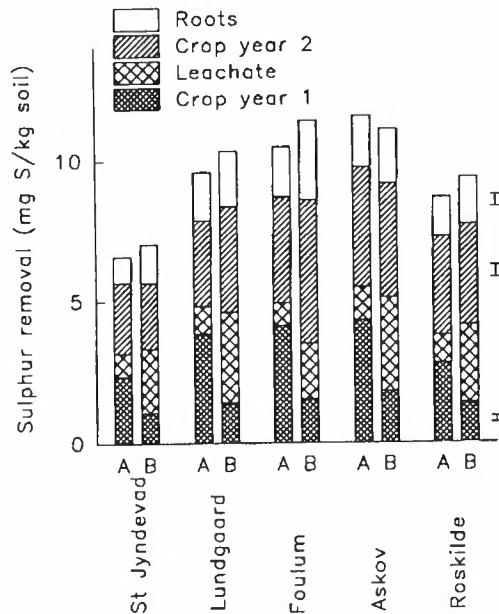


Fig. 1. Total removal of sulphur throughout the experiment per g dry soil in the pot. Cropping system: A) Italian rye grass both years, B) spring rape followed by spring barley. Error bars:  $\text{LSD}_{0.95}$ . No LSD values for leachates because of sample pooling

Also in year 2 the total removal of S was similar for both crops with the exception of the Foulum soil, where most S was removed by grain, straw and roots of barley. Both above-ground materials and roots contributed to this.

An analysis of variance on the total S removal throughout the experimental period (two years) showed a significant effect of crop type ( $P=0.05$ ), even though differences between the two cropping systems were only significant for the Foulum soil. There was a significant effect of soil type ( $P=0.01$ ) on the total S removal.

The net mineralization, as measured by removal of sulphur in the different fractions, constituted only 1.7 to 3.1% per year of the organic S pool in the soils and was always too small to correct S deficiency. The net mineralization over both years was unaffected (except for the Foulum soil) by the cropping system used (spring rape-spring barley or rye grass-rye grass). Several workers have found that the presence of growing plants increases the net mineralization of sulphur as opposed to absence of plants, where often immobilization occurs (Frenay & Spencer 1960; Nicolson 1970; Maynard et al. 1985). The results from this experiment suggest, that these differences are caused by a build-up of sulphate which inhibits mineralization in the absence of plants. In the presence of plants the solution sulphate will be kept at low levels because of plant uptake.

The content of S in the soil organic sulphur fractions does not explain the observed differences in sulphur supplying capacity. This is especially pronounced for the difference between the two sandy soils, St Jyndeved and Lundgaard, that have very similar soil properties (Table 1), but quite different sulphur supplying capacities. This indicates that there is a difference in the nature of the organic sulphur that causes the St Jyndeved soil to be less able to supply sulphur to plants.

To investigate this difference in the nature of the organic matter, the two soils were incubated with  $^{35}\text{S}$  as described above. The results of organic matter extraction after eight weeks of incubation at  $25^\circ\text{C}$  are presented in Table 2. For both soils it was found that full dispersion of the soils considerably increased the amount of extractable S. However, the activity of  $^{35}\text{S}$  incorporated into the organic matter did not increase significantly, indicating that the  $^{35}\text{S}$  had not been incorporated into the more protected organic matter released by dispersion. It can also be seen that twice as much  $^{35}\text{S}$  was incorporated in the organic S pool in the Lundgaard soil as that in the St Jyndeved soil, suggesting a more rapid turnover of sulphur in the former soil type. This might also partly explain the observed differences in the pot experiment.

Table 2. Content of S and  $^{35}\text{S}$  in organic matter of two sandy soils extracted with acetylacetone at full or no dispersion ( $1800\text{ J mL}^{-1}$ ), after eight weeks of incubation at  $25^\circ\text{C}$

		$\mu\text{g S g soil}^{-1}$		% of added $^{35}\text{S}$	
		No disp.	Disp.	No disp.	Disp.
St Jyndeved	av.	64.4	94.3	33.1	34.2
	SE	2.3	10.4	1.0	3.0
Lundgaard	av.	64.4	103.3	63.8	66.8
	SE	1.9	4.8	2.2	3.2

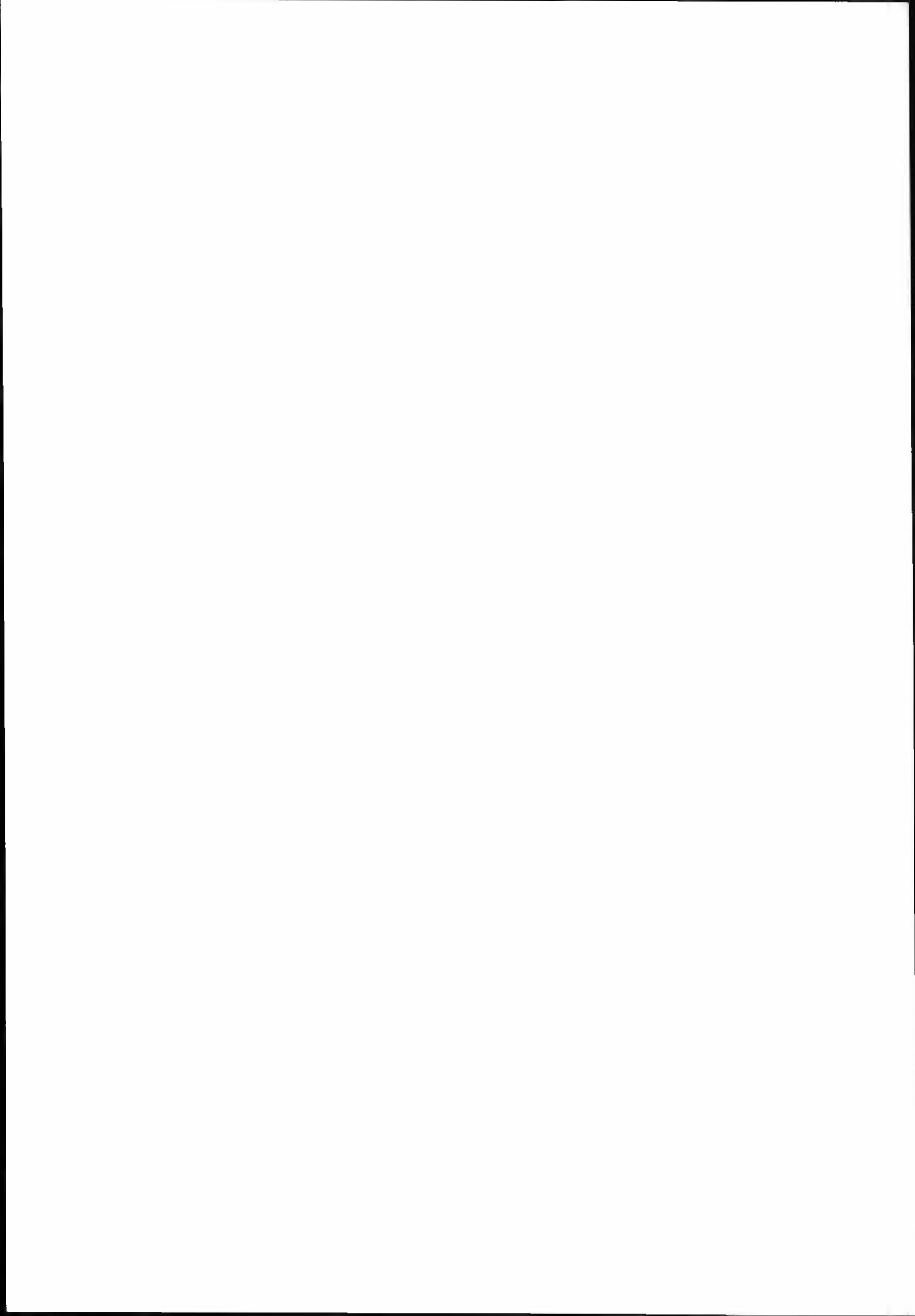
## CONCLUSION

The net mineralization does not seem dependent on type of crop grown, and it is stressed that the primary role of plants in soil sulphur cycling is as a sink. Mineralization of organic sulphur contributes little to the supply of sulphur to plants and cannot alone correct sulphur deficiency. There are great differences between the investigated soils in their ability to supply sulphur to plants. Differences that cannot be explained by the content of ester-bonded or carbon-bonded sulphur in the soil.

Preliminary investigations indicate that physical protection of soil organic matter plays an important role in the extractability of organic sulphur and that the fractions extracted without dispersion have higher turnover rates. This needs to be further evaluated in future research.

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# The development of a soil test for sulphur

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Sulphur (S) exists in the soil in a number of inorganic and organic forms. Some S is directly available to plants, but some requires transformation to plant-available forms. These S pools vary within soils, over time, and between soils, in terms of their size, immediate availability and longer term availability for plant growth. It appears that soil S tests have met with limited success since the extractants do not access the different S pools in the same proportion as plants. Eight techniques for extracting soil S were evaluated in 18 pasture soils by comparing the relationship between the S test value and the relative response of pasture to applied S. Extraction in 0.25M KCl at 40°C (KCl-40) was found to correlate well with pasture response ( $r^2 = 0.73$ ) and to result in a significant improvement over the more widely used extraction with monocalcic phosphate ( $r^2 = 0.47$ ). Comparison of these two extractants in a further 30 pasture soils confirmed the superiority of the KCl-40 test. Extraction of soil S from a pot trial in which rice was grown, under non-flooded conditions, with an application of  $^{35}\text{S}$ -labelled gypsum showed that the specific activity of the KCl-40 extract was closest to the specific activity of the plant, compared to five other soil S extractants. This indicates that the plant and the KCl-40 test are accessing S from the same, or very similar, soil S pools. The KCl-40 S tests appear to extract S from the soil solution, the adsorbed sulphate pool and a readily available portion of the organic S pool.

Key words: Estersulphate, soiltest, sulphur

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## Soil S and S dynamics

Sulphur (S) occurs in soils in both organic and inorganic form but the proportion of organic to inorganic S can vary widely according to the soil type (pH, drainage status, organic matter content, mineralogical composition) and depth in the profile (Williams 1975; Freney 1986a).

The C:N:S ratio of soils varies widely from 147:10:14 for soils from northern Scotland, 121:10:1.17 and 155:10:1.4 for pasture and podzolic soils of Australia, 130:10:1.3 for grassland soils of New Zealand, 114:10:1.5 and 145:10:1.01 for Minnesota and Oregon soils. There is a closer correlation between total N and organic S in soils than between organic C and organic S (Tabatabai & Bremner, 1972b; Bettany et al. 1973; Neptune et al. 1975). This close N:S ratio suggests similarities in the cycling of N and S within the soil-plant system (Biederbeck 1978).

In most surface soils, inorganic S accounts for only a small fraction of the total S (Bohn et al. 1986). In general, tropical soils have lower levels of sulphate in the soil solution compared to temperate soils (Blair 1979) and the subsurface layers usually contain more sulphate and are capable of adsorbing more than the surface layers (Ensminger 1954). Sulphate may occur in soils as water soluble salts, adsorbed to soil colloids or as insoluble forms (e.g.  $\text{CaSO}_4$ ; Fe and Al-sulphate) (Williams 1975). The concentration of sulphate in the soil has been observed to vary throughout the year (Barrow 1966; Ghani et al. 1990).

Considerable fluctuation in the concentration of water-soluble sulphate in surface soils is observed due to the effect of seasonal conditions on mineralization of organic S, leaching of soluble sulphate and sulphate uptake by plants (Tabatabai 1982). Tabatabai (1982) also reported that the application of fertilizers and the sulphate content of rain and irrigation waters may affect the concentration of watersoluble sulphate in soils. The amount of S accessible to plants is determined by the soil-soluble sulphate concentration, and the supply of S to this pool from either organic matter via the microbial pool or directly from fertilizer, animal residues, or atmospheric inputs (Blair 1988).

Sulphate is adsorbed by hydrous oxides of Fe and Al and by the edges of aluminosilicate clay particles (Chao et al. 1962; Harward et al. 1962). Soils vary widely in their capacity to adsorb sulphate. In some soils, sulphate adsorption plays an important part in the retention of sulphate against leaching, but others possess little or no sulphate adsorption capacity. Bloom (1981) found that sulphate was also adsorbed onto organic matter, probably onto (Al, Fe) – humus complexes.

Organic S accounts for >95% of the total S in the most soils from humid and semi-humid regions (Tabatabai 1982; Freney 1986a). Factors that affect the organic matter content of soil also influence the amount of total as well as organic S in these soils (Stevenson 1986). Freney et al. (1972) reported that amino acid S accounted for 21 and 30% of the total organic S in two podzolic soils in Australia, and approximately 60% of the amino acid sulphate was cystine sulphur. Scott et al. (1981) found that the S-containing amino acids accounted for 11 to 15% of the total S in several Scottish soils.

Characterization of the soil organic S pools has been achieved by the use of chemical extraction techniques. Freney (1986a) used a sequential process to characterize the organic S complexes in soils. A grouping of organic S compounds based on the reactivity of organic S with certain reducing agents has been made (Williams 1975; Tabatabai 1982; Freney 1986b). These are:

- (a) Organic S that is reduced to hydrogen sulphide ( $\text{H}_2\text{S}$ ) by hydriodic acid. This sulphur is not bonded directly to carbon and is believed to be largely in the form of ester sulphate (e.g. phenolic sulphate).
- (b) Organic S that is reduced to inorganic sulphide by Raney nickel and that seems to consist almost entirely of amino acid S (e.g. cysteine and methionine).
- (c) Organic S that is not reduced by either hydriodic acid or Raney nickel. This fraction is assumed to consist of sulphur bonded directly to carbon, but not recoverable by current methods used for estimation of carbon-bonded S.

### **Sulphur transformations in soils**

The transformation of sulphur in soils results from both microbial activity (Freney et al. 1971;

Stevenson 1986) and chemical processes (Bloomfield & Coulter 1973). Mineralization and immobilization occur simultaneously in soils wherever organic debris is undergoing microbiological decomposition (Maynard et al. 1983, 1985). Saggart et al. (1981) found that inorganic sulphate was mostly immobilized into the fulvic acid fraction after 64 days' incubation, and that the transformation of immobilized S into the more resistant humic acids and humin fractions occurred in long-term incubation studies. Freney (1961) suggested that ester sulphates readily became available to plants and Shedley (1982) obtained direct evidence of this from  $^{35}\text{S}$  studies.

The oxidation process is of considerable importance, because the ultimate source of S in soil is sulphate, and many S fertilizers are based on the reduced form of S. In general, elemental S, sulphide and several other inorganic S compounds can be oxidized slowly in soils by a purely chemical process (Weir 1975), whilst microbiological oxidation by various groups of photosynthetic, autotrophic and heterotrophic bacteria, fungi and actinomycete is much more rapid under favourable conditions (Bettany & Stewart 1983).

### **Soil testing for S status**

The S cycle in soil includes differently sized pools, with different flow rates between the pools (Till 1979), as discussed earlier. Determination of the S needs by soil testing is complicated by the various forms by which S is held in the soil (Blair 1979).

Soil testing to determine S status has met with variable success (Blair 1979; Jones 1986; Blair et al. 1992) and the results are often inconsistent (Mahler et al. 1993). Numerous methods of soil S testing have been reported by many investigators and there is no general agreement as to which estimate best defines a soil's S supply (Probert 1976; Tsuji & Goh 1979). These analytical methods for soil S testing have been reviewed by Freney (1986a), Blanchar (1986) and Anderson et al. (1992).

The poor performance of soil S testing is related to the inability of the extractants to estimate organic S forms that can be mineralized, and hence they underestimate the soil sulphate supplying capacity. On the other hand, some extractants may extract more organic S than that which may become available to the plant, and hence they overestimate the size of the available S pool.

It was hypothesized that the ideal soil test can measure the sulphate in solution, estimate the adsorbed sulphate which is available for plant uptake and also estimate that portion of the actively turning-over organic S component in the soil that will become available to the plant. Gianello & Bremner (1986a, b) used a heated KCl extract to determine available N in soils; this appeared an appropriate method to evaluate S.

## **MATERIAL AND METHODS**

### **Field calibration and verification studies**

Soil samples (0–7.5 cm) and yield data were obtained from 18 field-based pasture experiments in northern New South Wales, Australia. The sites had a broad range of parent materials and fertilizer histories, ranging from no fertilizer in the three years prior to experimentation up to 157 kg S ha<sup>-1</sup> over a 10-year period. The samples were airdried in a glasshouse and ground to pass through a 2 mm sieve. They were stored in a dry condition for up to two years prior to

their use. Soil samples from an additional 30 pasture trials throughout southern Australia (some 0–7.5 cm and some 0–10 cm) were used in a verification study.

Soil S was measured using the following extraction techniques:

- (i) Measurement of S in soil solution by extraction with water (1:5, w:v) (Freney 1958).
- (ii) Measurement of S in soil solution plus adsorbed S by 0.01 M  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  (1:5, w:v) (Barrow 1967).
- (iii) Measurement of S in soil solution, adsorbed S and a portion of organic S by either heated 0.25 M KCl (modified, Gianello & Bremner 1986a) or 0.5 M  $\text{NaHCO}_3$  (Kilmer & Neary 1960).

For the heated KCl extracts, 3 g soil was weighed into glass screw-top vials and 20 ml 0.25M KCl added (compared with 2M KCl in Gianello & Bremner 1986a, b). The vial was tightly capped and heated in an oven at temperatures of 100, 80, 40 or 25°C for three hours (instead of the four hours used by Gianello & Bremner 1986a, b). Reducing the concentration to 0.25M KCl and time to three hours was found, in a preliminary experiment, to be more appropriate. An additional treatment, where the soil was shaken at 25°C for 16 h was also included. The extracts were filtered and S analysed by ICP-AES.

All extractants were used in the calibration study but only the methods which used  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  (MCP) and KCl at 40°C (KCl-40) were used in the verification study.

### **Glasshouse study**

Soil samples were collected from an experiment in which  $^{35}\text{S}$ -labelled sources and different fertilizer placement S were compared over two consecutive rice crops grown in the same pot under flooded or non-flooded conditions (Chaitep 1990). The soil used was an Aquic Haplustalf which was responsive to S. Data from the gypsum fertilized, non-flooded soil are presented here.  $^{35}\text{S}$ -labelled gypsum (0.03 M Bq  $^{35}\text{S}$  mg S<sup>-1</sup>) was added to the soil at transplanting at a rate equivalent to 18 kg S ha<sup>-1</sup>.

When a labelled sulphate fertilizer is added to a soil the  $^{35}\text{S}$  enters the inorganic and organic S pools. Initially, there is a rapid increase in the specific activity (SA = ratio of labelled to unlabelled S) of the soil solution, which declines over time as the labelled sulphate enters a number of pools, as unlabelled sulphate is released from these pools and as sulphate is taken up by the plant. Similarly, the SA in the plant increases and then decreases as the SA of the soil pool decreases. When the SA of the plant is initially high, only a small amount of S and dry matter has accumulated. The SA of the plant declines as more S and dry matter accumulate.

The ratio of the SA of the plant at harvest to SA of the extracted soil pools, termed the specific activity ratio (SAR = SA of the plant / SA of the soil extract), gives an indication as to whether the plant has been using sulphur from the same pool/s as removed by the extractant. An SAR value of 1 indicates that the soil S pool extracted by the plant and that removed by the extractant are similar or the extractant and the plant are drawing sulphur from the same soil pool/s.



Soil samples were first extracted by the same techniques listed above and the extracts analysed for S by ICP-AES, and for  $^{35}\text{S}$ , by liquid scintillation counting. The soil remaining after extraction was centrifuged at  $5200 \times g$  for 20 minutes and a 0.6 g subsample of the extracted soil sample transferred to a round-bottomed Johnson & Nishita (1952) flask. HI reducible S (HI-S) remaining in the soil was determined by the Dean method (1966). The HI-S in the soil prior to extraction was determined using a 0.2 g sample. The percentage of HI-reducible S removed by the different extractants was determined as:  $[(\text{HI-S before extraction} - \text{HI-S after extraction})/\text{HI-S before extraction}] \times 100$ .

## RESULTS

### Field calibration study

In the 18 pasture soils, the mean concentration of S in the various extractants increased in the order:  $\text{KCl-}25^\circ\text{C} < \text{MCP} = \text{KCl-}40 = \text{KCl-}16 \text{ hr} < \text{H}_2\text{O} < \text{KCl-}80^\circ\text{C} < \text{KCl-}100^\circ\text{C} < \text{NaHCO}_3$  (Table 1). As the extraction temperature with KCl was increased from  $25^\circ\text{C}$  to  $100^\circ\text{C}$  the amount of S extracted significantly increased from a mean of  $5.4 \text{ g S g}^{-1}$  soil to  $15.6 \text{ g S g}^{-1}$  soil. The coefficient of determination ( $r^2$ ) for the relationship between the percentage of maximum dry matter yield from the 18 pasture soils and the critical soil test values for the different methods are presented in Table 1. The KCl-40 extraction method gave the best relationship between extractable sulphur and the percentage of maximum dry matter yield, with an  $r^2$  value of 0.73. Increasing the temperature of the KCl extractant to  $100^\circ\text{C}$  resulted in a decrease in the  $r^2$  value to 0.51. An  $r^2$  value of only 0.47 was recorded for the MCP extract and the relationship was not significant with water or KCl- $25^\circ\text{C}$ . It was not possible to fit a Mitscherlich curve to the  $\text{NaHCO}_3$  data, so a linear relationship with a very low  $r^2$  (0.04) was fitted (Table 1).

Table 1. Coefficient of determination ( $r^2$ ) for the relationship between extractable S and the percentage of maximum yield and the critical soil test values for different S soil tests on 18 pasture soils from northern New South Wales

Sulphur extraction method	Coefficient of determination <sup>1</sup>	Critical level <sup>2</sup>
Water	0.45	8.4
0.01M $\text{Ca}(\text{H}_2\text{PO}_4)_2$ (MCP)	0.47*	7.1
0.25M KCl heated at $100^\circ\text{C}$	0.51*	19.1
0.25M KCl heated at $80^\circ\text{C}$	0.54*	12.4
0.25M KCl heated at $40^\circ\text{C}$	0.73**	6.5
0.25M KCl heated at $25^\circ\text{C}$	0.30	6.6
0.25M KCl shaking for 16 h at $25^\circ\text{C}$	0.48	6.7
0.5M $\text{NaHCO}_3$	0.04 <sup>3</sup>	-

<sup>1</sup>  $p < 0.05$  and \*\*  $p < 0.01$ , with  $n = 18$ .

<sup>2</sup> Soil S test level at  $Y = 90\%$  maximum yield.

<sup>3</sup> Linear regression

### Verification study

The soil S test values and relative yield data from a further 30 pasture trials are presented, along with the original data, in Fig. 1. The relationship between the percentage of maximum

yield and KCl-40 extractable S did not change significantly, but the relationship with the MCP extractable S was altered. A higher correlation between soil S test level and the percentage of maximum yield was found with the KCl-40 test than with the MCP test. The  $r^2$  value of the verification study was less than that in the calibration study in both the KCl-40 and MCP methods. The  $r^2$  value decreased from 0.73 to 0.40 for the KCl-40 and from 0.47 to 0.30 for MCP (Table 2). The data from these sites also changed the soil S test value at 90% maximum yield from 6.5 to 6.4 g S g<sup>-1</sup> for the KCl-40 method and from 7.1 to 7.8 g S g<sup>-1</sup> for the MCP method (Table 2).

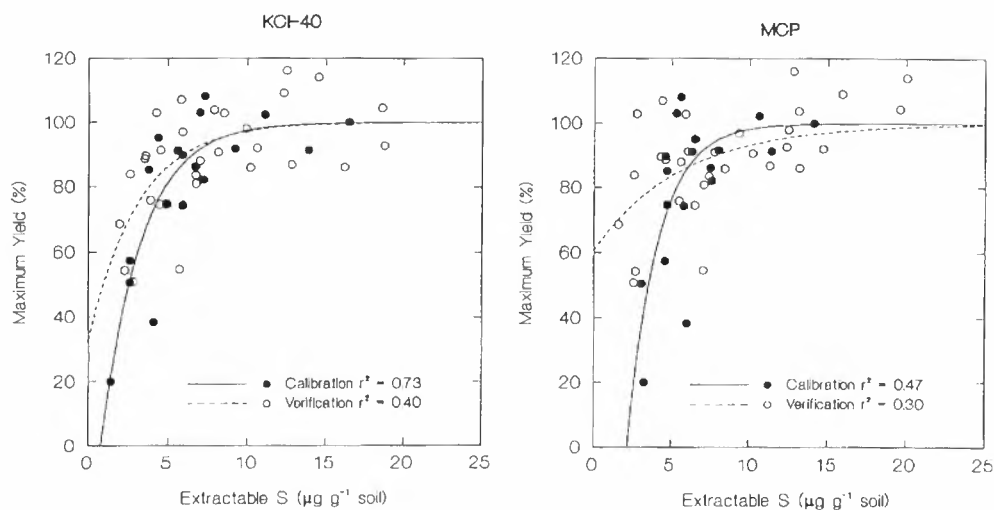


Fig. 1. The relationship between the percentage of maximum dry matter yield and S extracted by the KCl-40 and MCP methods for the 18 original and 30 new pasture sites

Table 2. Coefficient of determination ( $r^2$ ) for the relationship between S extracted by the KCl-40 and MCP methods and the percentage of maximum yield and the critical soil test value (soil S test value at 90% maximum yield) from the calibration (18 pasture soils) and verification (30 pasture soils) studies

Extractant	Coefficient of determination ( $r^2$ )		Critical soil test value (g S g <sup>-1</sup> )	
	Calibration	Verification	Calibration	Verification
MCP	0.47*	0.30*	7.1	7.8
KCl-40	0.73**	0.40*	6.5	6.4

### Glasshouse study

The lowest concentration of extractable S was in the H<sub>2</sub>O and MCP extracts, whilst the NaHCO<sub>3</sub> extractant had the highest concentration of S (Table 3). The KCl-100 extract also had a high S concentration. As the temperature of extraction with KCl was reduced from 100°C to 40°C, the concentration of S in the extract decreased.

Table 3. Extractable S ( $\mu\text{g S g}^{-1}$  soil), percentage of S removed from the HI fraction and specific activity ratio (SAR) for a range of soil extractants

Extractant	Extractable S ( $\mu\text{g S g}^{-1}$ soil)	Percentage of S removed from HI-S pool	SAR
H <sub>2</sub> O	3.3 e	6.0 c	1.21 e
MCP	2.9 f	5.9 c	1.14 hd
KCl-40	3.6 d	5.9 c	1.07 ie
KCl-80	6.3 c	11.5 c	1.23 c
KCl-100	18.8 b	32.6 b	2.12 b
NaHCO <sub>3</sub>	24.8 a	38.8 a	3.66 a

Numbers within a column followed by the same letter within each letter set are not significantly different according to DMRT

NaHCO<sub>3</sub> removed the highest amount of S from the HI-reducible S fraction. Extraction with KCl 100°C also removed a significant amount of the HI-reducible S fraction. This amount declined as the extraction temperature was reduced to 40°C. The amount of S removed from the HI-reducible S did not significantly differ between the water, KCl-40 and MCP methods.

The lowest SAR value was recorded in the KCl-40 treatment and this was close to 1.00. The SAR with the water extract was not significantly different from the KCl-80 treatment, and higher than MCP. The highest SAR value was found in the NaHCO<sub>3</sub> treatment followed by the KCl-100.

## DISCUSSION

### Comparison of extractants

Increasing of the temperature of the KCl extraction from 25°C to 100°C resulted in a higher amount of extracted S. Williams & Steinbergs (1959) suggested the use of "heat soluble sulphur" and indicated that heat is the main factor leading to the release of additional sulphur.

This study found a poor correlation between MCP or water extractable S and pasture response to S. Similar or poorer correlations between MCP extractable S and pasture response have been found in several field studies: Rayment (1983) ( $r^2 = 0.47$ ), Reneau & Hawkins (1980, cited in Jones 1986) ( $r^2 = 0.43$ ), Vaughn et al. (1987) ( $r^2 = 0.40$ ), Probert & Jones (1977) ( $r^2 = 0.34$ ), Hoefl et al. (1973) ( $r^2 = 0.32$ ) and Jones et al. (1983, cited in Jones 1986) ( $r^2 = 0.11$ ).

In this study the total amount of S removed by the various extractants was measured by ICP-AES, except in the NaHCO<sub>3</sub> which underwent digestion to oxidize extracted organic S prior to turbidimetric determination. On the basis of previous studies, ICP-AES analysis would include soluble organic S (Maynard et al. 1987; Boswell 1991).

Anderson (Unpubl.) adapted the KCl-40 method to oxidize the organic S in the extract to sulphate so it could be determined turbidimetrically. This involved transferring 5 ml of the KCl-40 solution to a 50 ml Schott bottle, adding 1 ml of 30% hydrogen peroxide and allowing

predigestion for two hours at room temperature. The bottles were then placed in an oven at 80°C for 16 h (overnight), removed from the oven, allowed to cool and analysed for sulphate using the BaSO<sub>4</sub> turbidmetric method of Till et al. (1984). The relationship between auto-analyser S (Y) and ICP S (X) was  $Y = 1.03X$  with  $r^2 = 0.98$  and  $n = 40$ , over a range of 2 to 25 g S g<sup>-1</sup> soil.

#### **Source of S removed by extractants and plants**

The pot study, in which <sup>35</sup>S was used to trace S dynamics, revealed that the KCl-100 and NaHCO<sub>3</sub> extractants, removed a considerable amount of HI-reducible S from the soil (Table 3). The high amount of S extracted by the KCl-100 and NaHCO<sub>3</sub> methods was related to the amount of S removed from the HI-reducible S pool by these extractants.

The data on specific activity ratio (SAR) showed that the SAR of the KCl-40 extract was close to 1.00 (Table 3). This means that the KCl-40 extract had the closest specific activity to that of the plants, which indicates that the KCl-40 extract and the plants were drawing S from similar pools. The KCl-40 extract removed S from the soil solution and adsorbed sulphate, plus a portion of the HI-reducible S. The HI-reducible S is believed to be composed mainly of ester sulphates (Freney 1986a, b) which are rapidly turning over in the soil system (Blair et al. 1992). The other extractants had higher SAR (greater than 1.00) indicating that these extractants are removing S from soil pools not taken up by the plant.

The poor correlation with pasture response in the KCl-100 and NaHCO<sub>3</sub> methods in the calibration study (Table 1) suggests that these extractants correlate poorly with plant response to S because they overestimate the contribution of S from this labile S pool. This is supported by the SAR data. Similarly, Anderson (1992) found that the KCl-100 and NaHCO<sub>3</sub> methods tended to overestimate the size of the available S pool with only 6 to 55% of the extracted S being taken up by pasture. In a previous study, Probert (1976) reported that NaHCO<sub>3</sub> removed some soil S that was not available to plants.

#### **CONCLUSION**

In the calibration and verification studies, the KCl-40 method was found to correlate well with pasture response when analysed in 0–7.5 cm and 0–10 cm soil samples. Although the KCl-40 method performed well in this study, it is unlikely to be as predictive in soils where the S leaches deeper in the profile, such as that reported by Probert & Jones (1977) and Rayment (1983), unless deeper sampling is practised. However, it is suggested that the KCl-40 method will still perform well on these deeper samples due to its ability to extract both sulphate and labile organic S.

KCl-40 soil S also appears to be correlated with the yield of canola (Lefroy et al. 1993). To obtain a more predictive soil S test for annual crops it may be necessary to increase the depth of sampling, especially on light-textured soils. On these soils, with low S sorption capacity, the intensity and frequency of drainage is a major factor governing sulphate movement (Ghani et al. 1990) and thus the efficacy of the soil test. Losses of sulphate could be relatively high and yet these losses are not allowed for in the soil S test.

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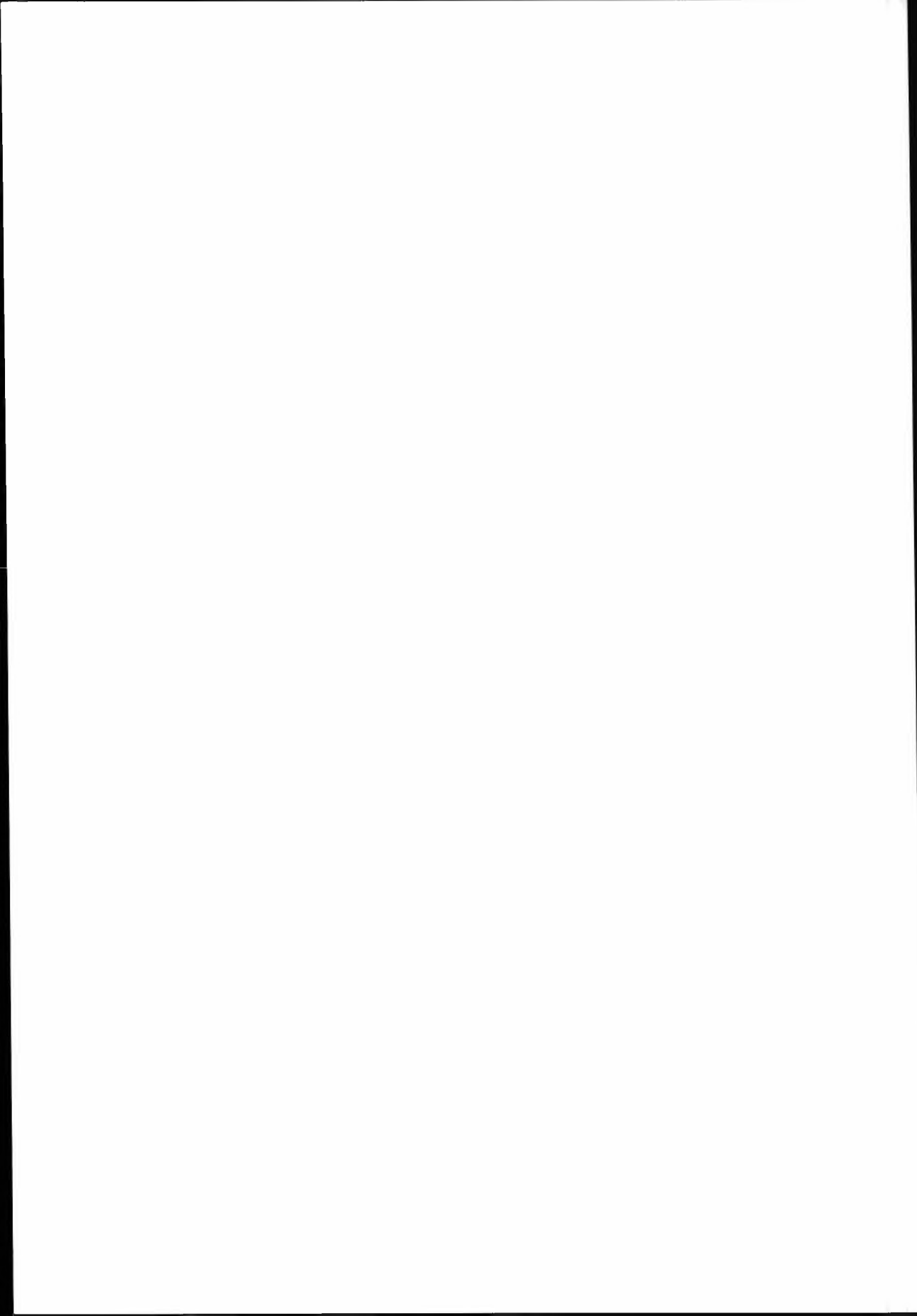


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# Sulphur deficiency in Scotland and the effects of sulphur supplementation on yield and quality of oilseed rape

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Oilseed rape yields were raised several-fold by the application of up to 64 kg S ha<sup>-1</sup> on a sulphur-deficient site. Those varieties with a low glucosinolate level were most prone to sulphur deficiency. Additional nitrogen to sulphur-deficient plants reduced yield. Applied sulphur raised glucosinolate levels but the influence of site and season was far greater. Increased sulphur availability enhanced glucosinolates in the vegetation, which reduced and delayed disease development. Adjustment of the glucosinolate profile to maximize natural resistance is more likely through plant breeding than agronomic means. Where sulphur is limited, low glucosinolate varieties are less able to respond to disease control than high glucosinolate varieties, possibly because of the more impaired sulphur metabolism of the former.

Keywords: Disease, glucosinolates, nitrogen, oilseed rape, season, site sulphur yield

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The sulphur requirement of oilseed rape is high compared to other crops. Oilseed rape removes 20–30 kg S ha<sup>-1</sup> in the seed, whereas a cereal crop removes only 5–15 kg S ha<sup>-1</sup> (Klessa & Sinclair 1989). Consequently the oilseed rape crop is at high risk from sulphur deficiency where sulphur supply is restricted.

The amount of sulphur received from atmospheric deposition declined overall by 34% in the United Kingdom (UK) between 1970 and 1985 (Unsworth & Fowler 1985). Since then the rate of decline has accelerated and the amount of sulphur deposited per year in north east Scotland is the lowest in the UK at less than 10 kg S ha<sup>-1</sup> with less than half of this received during the growing season (Anon 1990). In addition, as with much of northern Europe, the move to high nitrogen analysis fertilizers containing little or no sulphur has progressively replaced high S-containing fertilizers. The naturally low sulphur content of many of the soils in the north of Scotland compounds this problem. A survey of 125 farms in the area revealed that 46% of soils tested were low in sulphur (Scott & Munro 1979). As a result of these factors, sulphur supply to crops has become progressively restricted and sulphur deficiency has been observed at increasingly widespread levels (Walker & Booth 1992).

The following review will describe studies conducted in Scotland over the last five years investigating the complex relationships between soil, climate, sulphur and nitrogen on yield

and quality of oilseed rape. All the trials described were conducted within the area of north east Scotland where S deposition is lowest ( $< 10 \text{ kg ha}^{-1} \text{ year}^{-1}$ ). Where sulphur responsive and nonresponsive sites were compared, trial sites were selected on the basis of the levels of extractable  $\text{SO}_4$  in the respective soils coupled with previous observations of whether sulphur deficiency had been noted. Sites were selected as close together as possible in order to minimize agroclimatic differences and to minimize differences in S deposition.

#### YIELD RESPONSE TO SULPHUR AND NITROGEN

On the low sulphur site in 1989/90 the yield response to elemental sulphur varied from 40% to over 300% (Table 1) (Booth et al. 1991). Irrespective of nitrogen treatment, Tapidor – a very low glucosinolate variety – gave the largest response to sulphur followed by Cobra – a low glucosinolate variety and then Rafal, – a high glucosinolate variety. This indicates that correct sulphur nutrition is more critical for the new generation of low glucosinolate varieties and adds weight to the hypothesis that such varieties provide less capacity for sulphur storage in the form of glucosinolates which can be broken down in time of sulphur deficiency (Schnug 1989). The overall results indicate the substantial loss that may be incurred where sulphur is limiting growth.

Table 1. The effect of sulphur and nitrogen on yield of oilseed rape on a low sulphur site 1989/90

Nitrogen ( $\text{kg ha}^{-1}$ ) Variety Sulphur ( $\text{kg ha}^{-1}$ )	Yield ( $\text{t ha}^{-1}$ )					
	150 Rafal	Cobra	Tapidor	250 Rafal	Cobra	Tapidor
0	2.36	2.27	1.18	1.05	1.12	0.82
8	3.41	2.76	2.23	1.90	2.41	1.69
16	3.53	3.13	2.83	2.56	2.56	2.04
32	3.95	3.79	3.50	3.17	3.19	3.13
64	3.77	3.78	3.40	3.40	3.93	3.50

SE (internal mean) 0.370

SE (S mean) 0.320

SE (N mean) 0.261

Where no foliar sulphur was applied, the yield from plots of all varieties which had  $250 \text{ kg ha}^{-1}$  nitrogen was considerably lower than that from plots with  $150 \text{ kg ha}^{-1}$  nitrogen. A larger response to sulphur application was associated with the higher nitrogen application. For the  $150 \text{ kg ha}^{-1}$  nitrogen level there was a yield response to additional sulphur up to the  $32 \text{ kg ha}^{-1}$  treatment compared to a yield response up to  $64 \text{ kg ha}^{-1}$  treatment for the higher nitrogen level. Aulakh et al. (1980) and Janzen & Bettany (1984) have also shown that there was a significant interaction between nitrogen and sulphur and that the maximum yield was only obtained when sulphur and nitrogen applications were balanced. These results underline the importance of correct diagnosis of sulphur deficiency symptom in the field because if it were mistaken for nitrogen deficiency and additional nitrogen was applied, a greater yield penalty would result

than if the deficiency remained undiagnosed.

Where an identical trial to that described was conducted on a high sulphur site the mean total sulphur level in the vegetation across all varieties was  $4.64 \text{ mg}^{-1} \text{ g}$  compared with  $2.39 \text{ mg}^{-1} \text{ g}$  for the low sulphur site. At this high sulphur site in the same season and only 100 m distant from the low site, there was no response to sulphur, and overall site mean yield was  $4.8 \text{ t ha}^{-1}$  compared with  $2.7 \text{ t ha}^{-1}$  for the low sulphur site. In addition, there was a significant response to nitrogen at the high sulphur site.

#### EFFECT OF SULPHUR AND NITROGEN ON GLUCOSINOLATES

The application of sulphur increased glucosinolate levels, particularly for the high glucosinolate variety Rafal (Tables 2, 3). However, at the low sulphur site there was the same pattern of response of glucosinolates to sulphur and nitrogen as there was for yield (Table 2). Glucosinolate levels were lower in Rafal at the high nitrogen level indicating that the nitrogen sulphur imbalance affected glucosinolates as well as yield. For Tapidor, the glucosinolate levels were too low for such a trend to be detected. However, site had a far more influential effect on seed glucosinolate levels than either sulphur or nitrogen, particularly for the high glucosinolate variety Rafal. At the high sulphur site the mean glucosinolate levels for Rafal, Cobra and Tapidor were  $60$ ,  $15$ , and  $8 \mu \text{ Mg}^{-1}$  seed compared with  $23$ ,  $6$  and  $5 \mu \text{ Mg}^{-1}$  seed for the same varieties, respectively, at the low sulphur site.

Table 2. The effect of sulphur and nitrogen on glucosinolate content of oilseed rape on a low sulphur site 1989/90

Nitrogen (kg ha <sup>-1</sup> ) Variety Sulphur (kg ha <sup>-1</sup> )	Total glucosinolate concentration ( $\mu \text{ Mg}^{-1}$ seed)						
	150 Rafal	Cobra	Tapidor	250 Rafal	Cobra	Tapidor	
0	25.4	6.6	4.0	7.3	4.1	3.9	
8	27.0	6.2	3.9	5.5	3.4	4.2	
16	27.9	6.8	4.1	7.1	4.5	3.5	
32	38.0	7.9	4.9	18.2	7.6	5.2	
64	46.0	9.6	4.9	29.8	7.9	7.6	
SE (internal mean)	7.22	SE (S mean)	1.32	SE (N mean)	5.10		

Table 3. The effect of sulphur and nitrogen on glucosinolate content of oilseed rape on a high sulphur site 1989/90

Nitrogen (kg ha <sup>-1</sup> ) Variety Sulphur (kg ha <sup>-1</sup> )	Total glucosinolate concentration ( $\mu$ Mg <sup>-1</sup> seed)						
	150			250			
	Rafal	Cobra	Tapidor	Rafal	Cobra	Tapidor	
0	66.2	14.2	5.2	69.0	14.3	6.9	
8	69.3	14.8	5.5	62.1	19.6	6.9	
16	71.6	11.0	9.4	70.8	16.3	7.0	
32	68.4	14.8	8.0	69.6	16.6	7.6	
64	75.0	13.4	8.7	69.5	18.6	10.5	
SE (internal mean)	4.10	SE (S mean)	2.98	SE (N mean)	2.90		

## EFFECT OF SEASON ON RESPONSE TO SULPHUR

In 1988/89, a trial on a low sulphur site examining the response of Cobra, a low glucosinolate variety, and Rafal, a high glucosinolate variety, to varying levels of sulphur showed no response (Table 4) (Booth & Walker 1992). However, the following year severe sulphur deficiency symptoms were noted and this was reflected in a yield response to applied sulphur. At the corresponding high sulphur site no response to S was detected in either season. Any doubts about the classification of the low site were dispelled on examination of the comparison of the glucosinolate levels which showed far lower levels at the low sulphur site for both varieties in both years. This variation in response to sulphur supports the findings of Wetter et al. (1970) who have also reported the seasonal variability of the extent of sulphur deficiency. The lack of response in 1988/89 compared with 1989/90 was reflected in the number of sulphur-deficient samples submitted to the Scottish Agricultural College Crop Clinic for diagnosis (Walker & Booth 1992). A study of weather patterns in the two years showed that throughout December, January and February soil temperatures were 1°–2°C higher in the 1988–89 growing season than the following year. Soil temperature has a considerable effect

Table 4. The effect of sulphur application on yield and glucosinolate content at a low sulphur site over two seasons

Sulphur application kg ha <sup>-1</sup>	Yield (t ha <sup>-1</sup> )				Glucosinolate content ( $\mu$ mol g <sup>-1</sup> seed)			
	Rafal		Cobra		Rafal		Cobra	
	1989	1990	1989	1990	1989	1990	1989	1990
0	3.99	1.70	3.79	1.69	39.2	16.3	11.5	5.4
8	4.20	2.65	3.72	2.59	35.8	16.2	11.5	4.8
16	3.93	3.04	3.83	2.84	48.5	17.5	13.8	5.6
32	4.13	3.56	3.83	3.49	55.1	28.1	13.4	7.8
64	-	3.58	-	3.86	-	37.9	-	8.8
SE	0.174	0.261	0.174	0.261	1.88	5.10	1.88	5.10

on root growth (Payne & Gregory 1988) and it has been demonstrated that a 1°C change in soil temperature can significantly affect rooting depth and nutrient uptake (Walker 1969). However, it is apparent that more detailed work is required to relate root development to sulphur uptake with the aim of improving the predictability of a crop's requirement for sulphur supplementation.

## SULPHUR, GLUCOSINOLATES AND DISEASE RESISTANCE

It has been shown that sulphur availability affects the glucosinolate content of the vegetative parts of the plant and that *in vitro* certain glucosinolates have an anti-fungal effect (Mithen et al. 1986). In the following pot experiment, the low glucosinolate highly disease susceptible cultivar Cobra was grown in a high sulphur and low sulphur environment (Luong et al. 1993). Half of both sets of pots were inoculated with *Alternaria brassicicola* and then isolated from the remaining uninfected pots. Leaf samples were removed one month before inoculation, 10 days and 17 days after disease inoculation for analysis by High Performance Liquid Chromatography (HPLC) for individual glucosinolates.

High sulphur status was associated with significantly higher levels of total glucosinolate (Table 5). Although significant increases in the levels of the glucosinolates 2-hydroxy-pent-4-enyl, but-3-enyl and pent-4-enyl were observed, the proportion that each glucosinolate contributed to the total glucosinolate content was very similar irrespective of sulphur treatment (Table 6). *Alternaria* symptoms were observed on both the high and low sulphur status plants at the same time – five days after inoculation. However, when assessed on 24 July, 10 days after inoculation, the high sulphur status plants had less than half the disease level of the low sulphur plants (Table 7). However, within a week these differences in *Alternaria* level could no longer be observed. A natural infection of *Erysiphe cruciferarum* reinforced the benefits of enhanced sulphur nutrition, and consequently raised glucosinolate levels, when assessed on 13 September (Table 7). However, as differences in sulphur availability did not adjust the proportion of individual glucosinolates substantially it would appear that plant breeding is the best method of manipulating individual glucosinolate levels to increase their fungitoxic effect.

Table 5. The effect of sulphur on total glucosinolate content ( $\mu$  Mg<sup>-1</sup> DM)

Date sampled Sulphur	19 June	24 July	31 July	Mean
High S	5.2	5.9	4.6	5.2
Low S	4.3	4.3	3.0	3.9
SE	0.72	0.72	0.72	0.41

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Table 6. Effect of sulphur on proportion of individual glucosinolates (percentate of total glucosinolates)

Glucosinolate	High Sulphur	Low Sulphur
2-hydroxy-but-3-enyl	11.0	14.2
2-hydroxy-pent-4-enyl	2.2	1.9
But-3-enyl	4.9	4.3
Pent-4-enyl	4.5	4.0
4-hydroxy-3-indole-methyl	tr	tr
3-indole-methyl	28.9	24.4
1-methoxy-3-indole-methyl	41.3	43.1
2-phenylethyl	7.2	8.1
	100	100

Table 7. Effect of sulphur on *A. brassicicola* infection (24, 31 July) and *Eriysphe cruciferarum* infection (13 September) expressed as a percentage of leaf area infected

Date of Assessment Sulphur	24 July	31 July	13 September
High S	1.5	2.4	2.5
Low S	3.2	2.6	25.4
SE	0.55	0.55	6.33

In 1992/93 a field trial was conducted on a low sulphur site to assess the relative importance of soil applied sulphur and fungicides on yield of the high glucosinolate variety Bienvenu and the low glucosinolate variety Samourai (Table 8). In the absence of sulphur, there was only a small improvement in yield from fungicide application in the low glucosinolate variety Samourai, whereas the high glucosinolate variety responded far more to disease control. Where sulphur supply was unlimited, both varieties responded similarly to sulphur. These data also suggest that the greater degree of imbalance of metabolites within a sulphur-deficient low glucosinolate plant results in a greater inability to respond to inputs than a high glucosinolate plant. In many ways this echoes the inability of low glucosinolate varieties to respond to nitrogen where sulphur is limiting as described in Table 1, and reinforces the need for satisfactory sulphur nutrition for low glucosinolate varieties if they are to respond to inputs and achieve their full yield potential.

Table 8. The effect of fungicide application on yield under different sulphur application rates 1992/93

Sulphur application kg ha <sup>-1</sup>	0		160	
Fungicide application	-	+	-	+
Variety				
Bienvenu	0.87	1.22	1.57	1.68
Samourai	0.70	0.82	1.41	1.67
SE	0.125			



## SUMMARY

Studies have shown that the yield of oilseed rape can be raised several-fold by sulphur application on a deficient site. The new generation of low glucosinolate varieties is far more susceptible to sulphur deficiency than high glucosinolate varieties, and where sulphur is deficient, additional nitrogen can reduce yields. However, soil sulphur supply and season have a far greater effect on seed glucosinolate levels than applied sulphur or nitrogen. Glucosinolates do have a role to play in the protection of the plant, and by influencing glucosinolate levels in the vegetation, sulphur availability affects disease infection levels and disease development. However, manipulation of leaf glucosinolates to a profile conferring greater plant protection is more likely via plant breeding than by agronomic means.

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# Effect of sulphur fertilization on grain crops in central and eastern Norway

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The response to sulphur(S) fertilization was determined on 12 sites, all located on mineral soils in central and eastern Norway. Yields of barley grain are presented for 1991 and 1992. In the cold and wet season of 1991, significant yield responses were registered on seven of 12 trials. In the warm and dry season of 1992 only small and insignificant yield responses for S application were recorded. Three explanations are suggested for S deficiency in Norwegian crops at the present time: 1) Reduced S contribution from precipitation. 2) Low soil temperature during the growing season, and rainy winters may reduce mineralization and increase leaching. 3) Animal manure is commonly handled as slurry, and in this anaerobic condition some S will be reduced chemically and volatilized as hydrogen sulphide to the atmosphere. We would like to develop a method of S soil testing, analogous to that for phosphorus, potassium, etc. The level of S fertilization needed will depend on the crop, the nutritional status and soil type. Our experiments are designed to address these questions.

Key words: Fertilizer application, grain crops, mineralization, sulphur

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During the past four years it has become apparent that there is an increasing frequency of sulphur(S) deficiency in Norwegian crop production. Central Norway in particular seems to be affected, but in eastern Norway, too, these problems have to be dealt with. Symptoms of sulphur deficiency are observed in small grains, cruciferous crops, perennial grasses and clovers. Crops grown on sandy soils are the most vulnerable, but plants on morainic soil types may also suffer deficiencies. In areas with high precipitation, the sulphur supply can be a problem even on organic soils.

Ødelien (1966, 1970) studied sulphur deficiency and sulphur nutrition in Norwegian crops. Based on his work the compound NPK fertilizer "Fullgjødsel", manufactured by Norsk Hydro, for sale in Norway, has since then been supplemented with sulphur.

Here, we present a preliminary study on sulphur fertilization. The investigation includes both cereals and fodder crops and will continue on various soil types for several years. In this preliminary report results from the barley experiments will be presented.

### **Background for deficiency**

The increasing incidence of sulphur deficiency in crops in Norway since 1990 can be explained in several ways:

- \* The content of plant available sulphur in animal manure has decreased from approximately 500 g per ton in 1960 to about 300 g in 1990.
- \* During the last five years the soil has scarcely been frozen in central Norway. Most of the precipitation during the winters has been rain. This may have led to leaching of sulphate from the soil.
- \* The amount of sulphur in the precipitation has decreased from approximately 6.0 to 3.0 kg S/ha per year in central Norway during the period from 1970 to 1990.
- \* According to soil sample analyses the P content in topsoil seems to be relatively high on many Norwegian farms. In order to exploit this high P content, some farmers have applied only NK or N fertilizers containing no sulphur at all.
- \* Increased yields remove more sulphur from the soil. Relatively favourable climate, use of high yielding varieties, better farming management, more efficient utilization of nitrogen in animal manure, etc., are elements in this consideration.
- \* Use of imported NPK fertilizer, containing no sulphur.

### **Practical approach**

To avoid pollution of lacustrine waters by nutrients from arable land, the farmers were advised to minimize application of phosphate fertilizers. When, after some seasons they again applied superphosphate, the plants became healthier and more vigorous. The advisory groups now had a new question to address: Were the positive effects on the crops attributable to phosphorus or to sulphur, or to both elements?

## **MATERIAL AND METHODS**

A series of field experiments with barley was designed with four replicates and with the following six treatments for annual fertilization:

1. NK 22-11
2. NK 22-11 + 15 kg S as gypsum per hectare
3. NK 22-11 + 45 kg S as gypsum per hectare
4. NP 20-9 + KCl
5. NP 20-9 + KCl + 15 kg S as gypsum per hectare
6. NP 20-9 + KCl + 45 kg S as gypsum per hectare.

The rate of N was 100 kg/ha in central Norway and 110 kg/ha in eastern Norway.

In this preliminary report the results from 12 experiments will be presented. Eight of the experiments were started in 1991 and four in 1992. All trials were fertilized and harvested for three years in succession. Results only from the first two years are available now. Most of the fields are located on sandy soils which are considered to be low in plant available sulphur, but other soil types are also included in the study.

Plant samples for chemical analyses were taken just prior to heading. Grain samples were taken after harvest.

The chemical analyses are performed at Chemical Analysis Laboratory, Holt Research Station, Tromsø, Norway. For S only the total content is determined. Total-N is analysed according to the Kjeldahl method. Nitrat-N is determined, in this way: Plant tissue is extracted in a 0,02 N  $\text{CuSO}_4$ -solution on a boiling water bath for 60 minutes, and then read on a selective electrode.

## RESULTS AND DISCUSSION

### Grain yields

The grain yields in the first and second years are presented in Table 1.

Table 1. Effect of sulphur fertilization on grain yields. Kilograms of grain with 15% moisture per hectare

Treatment Kg S/ha	Means of all 12 trials		1st year		2nd year Means of 7 trials	
	Grain	Rel.	Grain	Rel.	Grain	Rel.
NK	4470	100	4480	100	4240	100
NK + 15 kg S	4520	101	4680	104	4080	96
NK + 45 kg S	4530	101	4730	105	4080	96
NPK	4750	106	4740	106	4350	103
NPK + 15 kg S	4700	105	4820	108	4320	102
NPK + 45 kg S	4840	108	4930	110	4460	105
LSD (p=0.05)		270		210		420

\*) Trials with positive response to S

In the first year (1991) a significant, positive response to sulphur fertilization was registered for 7 of 12 trials. In the second year (1992) several experimental sites were exposed to drought in the early summer. The vegetative growth was halted temporarily and the yield on these sites originated to a large extent from lateral shoots.

### Chemical analyses

Chemical composition of plant tissue harvested prior to heading, and of grains are given in Table 2. In Table 3 we quote results with permission from the Lokal Experimental Groups in Nord-Trøndelag County, central Norway.

Table 2. Chemical composition of barley plant tissue prior to heading, and of grains. Results based on dry matter (DM)

Treatment S/ha	Plant tissue *)			Grain **)	
	% P	% S	NO <sub>3</sub> -N mg/kg	% P	% S
NK	0.32	0.181	980	0.37	0.127
NK + 15 kg S	0.31	0.212	900	0.36	0.140
NK + 45 kg S	0.29	0.243	940	0.36	0.146
NPK	0.34	0.184	680	0.39	0.123
NPK + 15 kg S	0.33	0.212	630	0.37	0.132
NPK + 45 kg S	0.33	0.225	640	0.37	0.136

Means of 8 trials \*) Means of 6 trials

Table 3. Chemical composition of barley plant tissue prior to heading. Results from a single experiment in central Norway in 1991 (Fiskum 1991), based on dry matter (DM)

Treatment S/ha	DM %	Total N %	Total S %	NO <sub>3</sub> -N mg/kg
No sulphur	13.3	2.65	0.137	1630
15 kg S	14.8	2.37	0.193	278
45 kg S	15.4	2.33	0.232	98

The sulphur content in barley plants increased significantly after fertilization. To obtain normal growth and optimal protein quality a sulphur content between 0.2 and 0.5% of dry matter is considered to be adequate (Aasen 1986). Sulphur deficiency caused an accumulation of nitrate-N in the plants (Table 3). As sulphur is an essential constituent of proteins, S deficiency results in an inhibition in protein synthesis. The S-containing amino acids (cysteine and methionine) which are essential building blocks of protein are deficient and thus proteins cannot be synthesized. For this reason non-S- containing amino acids accumulate in sulphur-deficient plant tissue.

We only have relatively few chemical analyses, and until all the experiments have been analysed these preliminary results should be treated with caution.

### Sulphur balance

From a field lysimeter experiment at Kvithamar Research Station, Stjørdal, in central Norway we present some preliminary results for the sulphur balance in barley. The soil is a silty clay loam with 10% organic matter in the upper 0–20 cm layer. The field has been under cultivation for 90 years, and has been used in conventional rotation cropping of cereals, vegetables and perennial fodder crops (Myhr 1993). At present we have results covering three years (1991–1993). Temperatures during all three winter seasons were considerably higher than normal, and almost all precipitation was registered as rain. The soil was frozen for only a few weeks each winter and drainpipe discharge was a common occurrence. Surface runoff and erosion were insignificant.

A sulphur balance sheet for grain production under the described conditions in central Norway is estimated as follows:

Removed by the grain crop .....	10 kg	S/ha
Leaching in drain water .....	40 kg	S/ha
Total loss from soil .....	50 kg	S/ha
<hr/>		
Supplied by fertilizer .....	7 kg	S/ha
Precipitation .....	3 kg	S/ha
<hr/>		
Totally supplied .....	10 kg	S/ha
<hr/>		
Deficit per year .....	40 kg	S/ha

The preliminary results from Kvithamar indicate a leaching of 40 kg S/ha and per year (Oskarsen & Myhr 1993). From a single site on a cultivated organic soil in central Norway an annual leaching of 270 kg S/ha and per year has been monitored (Bærug & Uhlen 1982). From an industrial area under central European conditions a leaching of 130 kg S/ha per year is reported (Pfaff 1963, referred to by Mengel & Kirkby 1987). From these results a preliminary conclusion would be that the leaching of sulphur in Norwegian soils is underestimated.

### Visual deficiency symptoms

In 1991 and 1993 obvious sulphur deficiency symptoms were registered in many fields in central Norway. Both cereals and fodder crops were affected. In grain crops sulphur deficiency and nitrogen deficiency are sometimes difficult to distinguish. In plants suffering from S deficiency the rate of plant growth is reduced, but generally the growth of shoots is affected more than root growth. Frequently, the plants are rigid and brittle and the stems remain thin. Chloroplast formation is affected and decomposition of chloroplast may occur in severe cases. In contrast to N deficiency, the chlorotic symptoms occur first in the younger, most recently formed leaves. This indicates that older plant tissues cannot contribute significantly to the S supply of the younger leaves. A relevant question is: Will the grain yield be reduced in quantity, and in protein quality, even without visible symptoms of sulphur deficiency? We think that our future experiments will address this question.

### Mineralization

Sulphur occurs in the soil in inorganic and organic form. In most soils organic sulphur compounds provide the major reservoir. The organic S fraction of the soil is rendered available to plants by microbial activity. In this process of mineralization  $H_2S$  is formed which, under aerobic conditions, readily undergoes auto-oxidation to  $SO_4^{2-}$ . A review of the S cycle in relation to soil and plant nutrition has been presented by Whitehead (1964).

The sulphur cycle combined with meteorological data may help in explaining the fluctuations in deficiency from season to season. In 1991 and 1993 we had a wet and cold start to the growing season. Only a small amount of sulphur was released in the soil, and some of it might have leached through the drainpipes. In 1992 we had a warm and dry early summer,

with relatively high soil temperature. The mineralization of organic matter was probably greater than usual, and substantial amounts of sulphur were released from soil organic matter.

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# Effect of sulphur fertilization on oilseed crops and cereals in Sweden

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Field experiments with sulphur carried out in Sweden during the 1980s revealed that the addition of sulphur in fertilizers is sometimes necessary. It was found that wheat quality was negatively affected by sulphur deficiency in roughly 10% of the experimental sites. In 1992–93 more widespread sulphur(s) deficiency was found in oilseed rape, both in practical agriculture and in experiments. In summer rape 10 kg S/ha sufficed to prevent sulphur deficiency but 20–30 kg was needed in winter rape. The most severe deficiency was observed in sandy soils, but response to sulphur was often found also in clay soils. Oilseed rape showed responses in 30–50% of the experimental sites. The role of diminished atmospheric deposition and less sulphur in fertilizers is discussed generally.

Keywords: Field experiments, plant analysis, sulphur.

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In recent years two trends have contributed to the change in the sulphur situation of crops. One is a reduction in air pollution, diminishing the sulphur flow from the atmosphere to soils and vegetation. The other is a change toward use of more concentrated fertilizer products containing less sulphur.

These developments have initiated a renewed interest in sulphur as a nutrient. Sulphur responses have been detected in most parts of western Europe.

In Sweden the policy until the end of the 1980s was to supply sulphur as a safeguard. PKfertilizers containing substantial amounts of sulphate were important. Most NPK fertilizers contained some sulphate.

It was shown in the beginning of the 1970s that the atmospheric supply was not a guarantee against sulphur deficiency (Bertilsson 1974). On sandy soils in free air symptoms of sulphur deficiency regularly appeared on sites more than 10 km distant from major population centra.

At Bulstofta, the Hydro Supra experimental station, in long-term frame experiments in the open air, visible signs of sulphur deficiency regularly appear on rape crops on sandy soils.

However, at the end of the 1980s, the sulphur in fertilizers was considerably reduced. The market preferred more concentrated products to sulphur as a safeguard.

The sulphur in soils resides in both the organic and the inorganic pools. There is a delicate and critical balance between the requirement of the crop and the combined effect of

the supply from the pools in topsoil as well as the subsoil and the direct supply from the atmosphere. The sulphur situation, therefore, is difficult to assess by means of soil analysis. Several attempts have been made, but no generally reliable method has yet been found.

Field experiments or plant analysis surveys are needed.

## FIELD EXPERIMENTS IN SWEDEN

In this report mainly the experimental work conducted by Supra/Hydro Supra is described. The field work and analyses have been carried out by the regional experimental organizations and laboratories in Sweden.

Normally, three or four blocks are used in the field experiments.

### Winter wheat 1982-85

Concerning sulphur, this work was carried out in two parts: one, a survey of sulphur content in the grain and the other an experimental series where sulphur was added to some main nitrogen treatments. During the period around 20 experiments were performed annually, each with six main nitrogen treatments in the range 0-200 kg N/ha. Each nitrogen level had two fungicide intensities. In 1984 243 samples, representing different areas, soils, and yield levels as well as a wide range of nitrogen intensities, were analysed. The results are presented in Fig. 1. A typical result from one of the experiments is given in Table 1.

Table 1. Protein and sulphur content in the grain of winter wheat. A typical example from one site (1982, site 94129). Average of two fungicide levels. No sulphur added to any treatment

Treatment	Protein %	S %	N/S
No nitrogen	9.1	0.135	11.8
80 kg N/ha	10.4	0.140	13.0
120 "	11.3	0.148	13.4
160 "	12.4	0.153	14.2
200 "	12.7	0.153	14.5

The experiments with sulphur were an extension of the nitrogen experiments mentioned above. The layout was extended to include three N+S treatments. Sulphur was supplied as gypsum in Spring. A total of 15 experiments were harvested in 1982-85. Baking quality determinations were made on all treatments.

### Summer rape, 1989-91

In 1989 an experimental series with nitrogen and sulphur to summer rape was initiated. Sulphur was supplied as calcium sulphate before sowing.

The experiments were placed in middle Sweden in areas not close to major centra. A total of 12 reliable experiments were harvested. The layout and results are presented in Table 2.

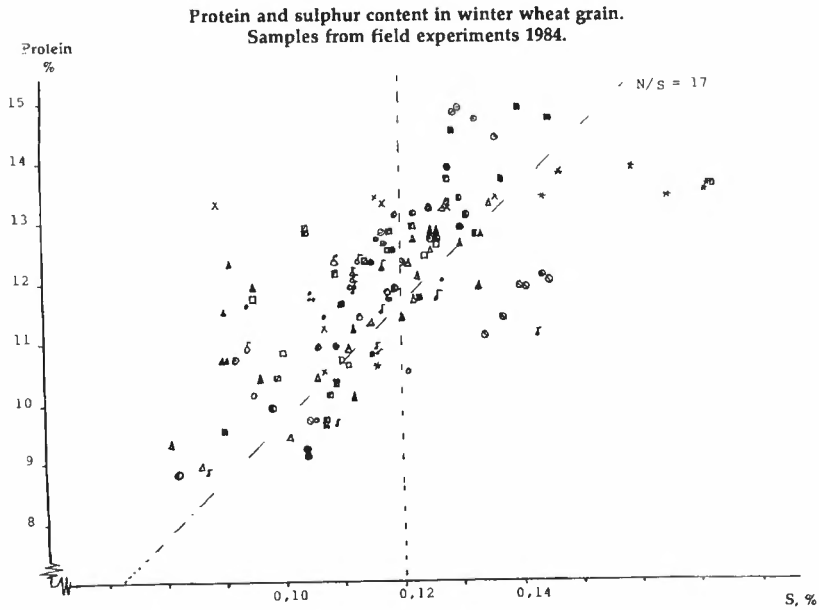


Figure 1.

Table 2. Field experiments with nitrogen and sulphur to summer rape. Average results of 12 experiments 1989-91 (S-8934). Expressed as kg/ha and as a percentage respectively. Glycosinolates (aliphatic) expressed as  $\mu\text{m/g}$

N	S	Yield	Oil	Protein	Glycosinolates
0	0	1380	49	38	
50	0	1780	48	40	22
50	10	1850	48	40	22
50	30	1840	48	39	24
100	0	2040	46	43	26
100	10	2130	47	41	27
100	30	2140	47	41	24
150	0	2230	46	44	26
150	10	2340	46	43	31
150	30	2300	46	43	31

**Summer rape 1992**

Sulphur was one factor in this combined layout, along with phosphate and combine drilling.

Four experiments were conducted in 1992 in middle Sweden on clayey soils. Constant nitrogen level: 140 kg N/ha. Sulphur as calcium sulphate incorporated by harrowing before sowing. Combine drilling with special machine. Layout and results can be seen in Table 3.

Table 3. Sulphur and phosphate to summer rape. Average of four experiments in 1992-93. S comparison 1992: treatments 4-5. S comparison 1993: treatments 6-7

Treatment	1992 Yield	Oil	Protein	1993 Yield (preliminary)
1. CAN, broadcast	2104	46	47	2883
2. NP 26-6, broadcast	2097	46	47	3004
3. CAN, combine	2321	45	47	2955
4. NP 26-6, combine (0 S)	2443	46	46	2987
5. NP 26-6, combine + 20 S	2428	46	46	
6. NPK, combine (0 S)				3050
7. NPKS, combine (28 S)				3195

**Barley 1992**

Seven experiments concentrated to middle Sweden were conducted on clayey soils. Here too, sulphur was a factor, along with N, P and placement studies. N level: 120 kg N/ha. Other factors as for summer rape above. Layout and results are presented in Table 4.

Table 4. Sulphur and phosphate to spring barley. Average of seven experiments 1992 (S-9216). Expressed as kg/ha and as a percentage respectively. Comparison for sulphur effects: treatments 4 and 6

Treatment	Yield	Protein
1. CAN, broadcast	3710	13.2
2. NP 26-6, broadcast	3750	12.9
3. CAN, combine	4010	13.2
4. NP 26-6, combine (0 S)	4270	13.2
5. NP 26-6, combine + CN	4020	12.9
6. NP 26-6, combine + 20 S	4260	13.2
7. CN top-dressed	3870	12.7

**Winter rape 1993**

Four experiments were conducted, three of which took place in the southernmost part of Sweden, and one in middle Sweden (Östergötland). Sulphur forms just one part of the experiment, but comparisons can be made at three levels: S early, early+late, late. N level: 80 kg N/ha at each application, in total 160. First application in early Spring, application 2 3-4 weeks later. The NPK-fertilizer contained soluble sulphate, kieserite in the two sulphur treatments at application 2.

Layout and results are set out in Table 5.

Table 5. NPK and sulphur to winter rape. Average of four experiments 1993 (S-9331). Evident sulphur effects in two sites

Treatment	Appl. 1	Appl. 2	Kg S/ha	Yield	Oil	Protein	S %
0		0	0	1388	48	37	0.55
CAN		CN	0	2313	46	42	0.56
NPK		CN	0	2427	45	43	0.56
NPK + S + B		CN	16	2831	46	43	0.61
NPK + S + B		CN + S	32	2930	46	43	0.63
CAN		CN + S	16	2573	46	43	0.58
CAN		CN + B	0	2344	45	42	0.56

### Summer rape 1993

Four experiments have been harvested. At this stage only preliminary data uncorrected for water content are available. (Table 3).

### Summer barley and wheat 1993

Six and five experiments respectively were conducted in 1993. There were no significant yield responses for sulphur on either crop. The wheat has still to be analysed for baking quality.

### Soil characteristics and sulphur response

Soluble sulphur (extractable with 1 M KCl in connection with soil mineral N analysis) was analysed on most experimental sites. These values, together with location in Sweden, soil type, pH and the response to sulphur are presented in Table 6.

Table 6. Site characteristics in relation to response for sulphur. Experiments with rape. Location: middle, east, west or south. Response in kg/ha and indication of regularity. Sulphate-S extracted by 1 M KCl, soil/solution ratio 1:1, mg/kg

Year	Crop	Location and soil	pH	S, soil	Response
1989	S rape	W. Heavy clay	7.2	6.1	+ 213 reg.
1989	"	W. Loam	6.1	6.5	+ 74 reg.
1989	"	E. S. loam	6.8	6.6	+ 98 irreg.
1989	"	E. L. sand	7.7	7.0	+ 106 reg.
1989	"	E. Heavy clay	6.6	10.0	+ 18 irreg.
1990	"	W. Heavy clay	7.0	4.4	+ 64 reg.
1990	"	E. Medium clay	6.6	5.2	+ 72 reg.
1990	"	E. Medium clay	6.6	5.4	0
1990	"	E. Loam	7.9	8.7	+ 92 reg.
1991	"	E. Medium clay	6.6	1.8	+ 72 reg.
1991	"	M. Heavy clay	6.5	2.8	0
1991	"	E. Heavy clay	7.1	4.6	0
1991	"	E. Loam	7.9	9.9	0
1991	"	W. Medium clay	7.2	n.d.	+ 350 reg.
1992	"	M. Heavy clay	6.1	n.d.	0
1992	"	E. Medium clay	6.8	2.7	0

tabell 6 forts.

1992	"	W. Silty loam	6.3	3.8	+ 82
1992	"	W. Medium clay	6.4	8.7	0
1993	"	W. Medium clay	7.1	1.9	+ 192
1993	"	M. Medium clay	6.2	2.1	0
1993	"	E. Heavy clay	6.7	2.4	0
1993	"	M. Medium clay	6.5	4.5	+ 220
1993	W. rape	E. Loamy sand	7.1	1.6	+ 1300 reg.
1993	"	W. Medium clay	6.9	2.3	+ 114 irreg.
1993	"	S. Sandy loam	6.9	3.1	+ 160 irreg.
1993	"	S. Loamy sand	6.9	5.4	+ 114 irreg.

## DISCUSSION

### Sulphur situation and responses

Results are given for field experiments from the beginning of the 1980s. Until 1988 there was a considerable sulphur flow through fertilization of agricultural soils. Although not always efficient (mostly autumn application of PK fertilizer) and not often directly needed, this flow added to the sulphur supply capacity of agricultural soils. In 1988/89 this source of sulphur was considerably diminished.

Naturally, the sites of sulphur experiments were always kept free from background sulphur-containing fertilizers. However, more long-term indirect effects cannot be excluded. The fertilizer sulphur helped keep up the levels in both the organic and inorganic sulphur pools.

According to the experiments described and observations in practical agriculture, sulphur deficiencies were more widespread in 1992 and 1993 than earlier. One reason could be that the more available sulphur pools gradually diminished because of less input by fertilizers. Another reason could be that the switch to rape low in glycosinolates rendered the sulphur situation of the rape more critical.

The experiments before 1989 were aimed at cereal quality (sulphur to oilseeds was considered natural). The principle was confirmed: sulphur deficiency is detrimental to protein quality and baking characteristics. The effects could be striking, but they were found in only one experiment out of 15.

Some critical values indicated in the literature are S content in grain of 0.12% and an N/S ratio of 17 (Randall et al. 1981; Haneklaus et al. 1992). The results of the field experiments 1982–85 is that of 243 analysed wheat samples 58% had a sulphur content lower than 0.12%, while 71% of samples had an N/S ratio higher than 17. Only 22% satisfied both criteria (Sturesson 1987). However, the baking quality was good or acceptable in most cases even at an S content of around 0.10% and at N/S ratios of 19–20. Furthermore, S supplements only rarely improved the values. It seems that the critical values mentioned above did not fully apply to the varieties and conditions prevailing in Sweden during the 1980s (Fig. 1).

The figure also indicates one important relation: the sulphur content increases with the nitrogen content. If the sulphur supply is not restricted the plant has a certain regulatory ability. An increased nitrogen uptake also increases the sulphur uptake although no extra

sulphur has been applied. Typical details from one of the experiments contributing to Fig.1 is presented in Table 1. The experiments with sulphur were an extension of the nitrogen experiments mentioned above. The layout was extended to include three N+S treatments. A total of 15 experiments were harvested in 1982–85. Baking quality determinations were made on all treatments (Sturesson 1987). Summary of these experimental results:

\* No yield effects of sulphur were found.

\* Sulphur improved baking quality only in 1982, in the form of dramatic effects on one site only. The effects were confirmed with baking tests with striking results. On the other sites no sulphur effects were found.

In 1989 new experiments with spring rape were started. The aim was to discover to what extent there was an economical sulphur response. Sites were chosen where the geographical location would be conducive to sulphur response. There was some response, on average (Table 2). However, a statistically significant response was found only at about one out of the 4–5 sites tested each year. Expressed otherwise: 20–25% of the sites responded significantly to sulphur.

Two rates were tested, 10 and 30 kg S/ha. In these experiments it was found that a full effect was achieved with 10 kg sulphur.

In 1992 sulphur was tested in both summer rape and barley. The yield formation was very greatly affected by severe summer drought. In neither of these crops could any response for extra sulphur be detected (Tables 3 and 4). But in practical agriculture sulphur deficiencies in oilseeds were observed.

The year 1993 turned out to be an extraordinary one for sulphur deficiencies. In many oilseed fields in middle Sweden sulphur deficiency was very evident. Sulphur became the focus of attention in practical agriculture and the experiments also showed more frequent responses to sulphur that year. A new series in winter rape produced very large responses, on average, and this was characteristic for two experiments out of four (Table 5). The results also indicate that sulphur is needed early in the year. The later dressing produces some response, but much less than that with the earlier application.

In summer rape the sulphur effects were smaller but very consistent. All four experiments showed tendencies to positive response, and on average the sulphur effect was statistically significant ( $p < 0.01$ ).

### **Soil characteristics and sulphur response**

From Table 6 it can be seen that responses occur over almost the whole range of sulphate sulphur levels, from 1.6 to about 8.0. Quantitatively, 8 mg S/kg means about 20 kg S in the topsoil, so it is clear that these levels do not in themselves guarantee a safe supply for rape. These results only confirm the generally known difficulty of finding a reliable diagnostic soil test for sulphur.

The most evident deficiency was found on a sandy soil, but it should be noted that responses occurred on several clayey soils.

## SUMMARY

There has been increasing awareness of sulphur deficiencies in Swedish agriculture during 1992–93. In field experiments with rape responses were found that, on average, were high enough to motivate the use of sulphur-containing fertilizers. For rape, sulphur responses have been recorded on 20–50% of the experimental sites depending on year.

For summer rape 10 kg S/ha as calcium sulphate gave the same response as 30 kg S/ha.

In the experiments with winter rape 16 kg S/ha was not sufficient, but a supplementary later dose produced an additional response. However, for winter rape it was essential that sulphur was applied early in the spring.

Similar experiments with barley and summer wheat did not produce yield responses for sulphur. For wheat, the quality analysis has still to be carried out.

Field experiments with winter wheat in 1982–84 in general did not reveal any sulphur effects. However, in individual cases there were dramatic effects on baking quality.

It is quite evident that sulphur needs to be considered both as a plant nutrient and as a fertilizer constituent in Sweden.

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# Sulphur nutrition of field crops in Finland

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In earlier studies sulphur fertilization was shown to be important in intensively cropped grass leys in northern Finland, but in recent experiments at six sites sulphur (S) application did not significantly increase the dry matter (DM) and protein yields of grasses. In a clay soil in southern Finland plant S content was decreased by potassium chloride application and increased by phosphorus fertilizers which contained a small amount of S. The residual effects of sulphate were small. Close dependence was found between DM yields and plant S content of pot-grown grasses.

Key words: Grasses, plant analysis, sulphate and other anions, sulphur fertilization, turnip rape

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The importance of sulphur fertilization of field crops in Finland has been demonstrated most distinctly in intensively cropped grass leys (Korkman 1973; Tähtinen 1977; see also Table 1). The availability of this nutrient in the soil was poorest in the northern parts of the country, where the atmospheric deposition of sulphur is smallest and the decomposition of soil organic matter slowest. A reasonable supply of sulphur to all field crops has been ensured by a small addition of this nutrient to all compound fertilizers since the early 1970s.

Table 1. Effects of sulphur fertilization on yield and S content of timothy hay on a peat soil in northern Finland (Tähtinen 1977)

S appl. kg/ha	N appl. kg/ha	Hay yield, kg/ha		S content, g/kg DM	
		1970	1971	1970	1971
0	48	1700	1280	1.2	0.8
34	48	4090	2850	1.9	1.1
0	96	3050	2140	1.0	0.9
68	96	4920	5350	1.9	1.3

During the 1980s the study of sulphur nutrition of field crops was again undertaken. Although no actual problems were found in Finland, the sulphur balance was estimated to be negative in many fields as a result of the declining atmospheric deposition (Tuovinen et al. 1990) and the increasing removal in yields. Because sulphur deficiency is detrimental to forage quality (Ettala et al. 1978), the studies were aimed at assessing a sufficient rate of supply of sulphur to ley grasses. The sulphur requirement of turnip rape and the content of extractable sulphur in some soil profiles were also investigated.

## MATERIALS AND METHODS

Field experiments with five rates of sulphur (S) fertilization were carried out in grass leys at six research stations of the ARC in central and northern Finland during 1987–90 (Hahtonen & Saarela 1993). The five S treatments comprised two NPK fertilizers that differed in their S content, a normal commercial fertilizer (2–4% S) and a low-S (0.3%) equivalent, and complementary sodium sulphate applications. The S application were split for every harvests and their annual rates ranged from 3 to 38–68 kg S/ha. The rate of annual N application was 180–240 kg/ha and the S/N ratio 2–28%.

The S nutrition of a cocksfoot-clover ley was studied in a clay field in Jokioinen by splitting the blocks of long-term phosphorus and potassium fertilization experiments with two rates of S (0 and 50 kg/ha). The residual effects of repeated application of S in the field as well as the S requirements of turnip rape and grasses and red clover were studied in pot experiments.

The availability of sulphur in soils was assessed by the acid ammonium acetate extraction method (Yli-Halla 1987a; Sillanpää & Jansson 1991). Plant material was digested in nitric acid and the contents of S as well as other nutrients were determined by plasma emission spectrometry (Sillanpää & Jansson 1991). Total nitrogen was determined by the Kjeldahl method.

## RESULTS AND DISCUSSION

### **Dry matter yield and S content of grasses**

The annual dry matter (DM) yields obtained by three cuts at four of the six research stations varied from 8100 to 10,050 kg/ha between the sites. The two cuts taken in the northernmost two locations yielded 5100–6300 kg/ha annually. No significant yield increases with sulphur fertilization treatments were found. However, the response tended to be positive when the total content of S in plant DM by the control fertilization was lowest (1.4–1.6 g/kg) and the N/S ratio of plant highest (13–14).

In one peat soil the highest sulphur rate of 50 kg/ha caused a statistically significant decrease in DM yield. The mechanism of this negative effect is not known, but one possibility is the reduction of sulphate to toxic hydrogen sulphide during wet periods (Bloomfield 1969).

Sulphur fertilization of grasses can be useful even though no yield increases are obtained, because the sulphur requirement of ruminants is higher than the content that suffices for maximum growth of grasses (Mannerkorpi & Kossila 1993). The average increase in the content of sulphur in plant DM at the six sites was 16% with the highest rate of S (52 kg/ha). The

apparent utilization percentage of fertilizer S was no higher than 6% of this amount, but as much as 15% appeared to be used from the smallest rate (11 kg/ha).

The content of extractable S in these soils was close to the average level of the cultivated soils in Finland (range 9–30 mg/l). A detailed presentation of these experiments is under preparation (Hahtonen & Saarela 1993).

### Interaction of S with K and P fertilization

The long-term experiments in Jokioinen had received only traces of fertilizer sulphur during the years 1988–89, but 20–80 kg S/ha was applied in single superphosphate during 1980–87 with the exception of the P0 control fertilization which had received no S. The content of extractable S in this soil was about 10 mg/l in the topsoil layer and 15 mg/l at a depth of 25–50 cm. No differences were found between the S rates which were applied earlier. The clover percentage was 25–35% in the first year (1991) and less than 10% in the second year.

The content of total sulphur in plants varied between the treatments in both experiments. Potassium fertilization, as the chloride that was applied by single dressing in the spring (60 kg/ha K and 55 kg/ha Cl), decreased the content of S in plants significantly (Table 2). This effect was probably a result of an antagonistic mechanism or competition between the major anions sulphate and chloride in the nutrient absorption by roots.

Table 2. Dry matter (DM) yield and sulphur content of a cocksfoot-red clover ley in a long-term field experiment with different rates of freshly applied and residual potassium fertilization on a clay soil in Jokioinen. Differences between treatments are significant if not marked with a common letter (Duncan 0.05)

K applied kg/ha yearly	Dry matter yield, kg/ha				S content, g/kg DM		
	Cut 1	Cut 2	Cut 3	Total	Cut 1	Cut 2	Cut 3
<b>1990 (about 3 kg/ha of S applied in P fertilizer in the spring)</b>							
0 1980-	3070c	4020a	1610b	8700b	2.28a	2.14ab	2.79ab
20 1980-	2960b	4120a	1600b	8680b	2.34a	2.22b	2.73ab
60 1980-	2740a	3980a	1460a	8180a	2.19a	2.01a	2.59a
80 1980-88	3180b	4060a	1660b	8900b	2.21a	2.08ab	2.97b
<b>1991 (about 3 kg/ha of S applied in P fertilizer in the spring)</b>							
0 1980-	2390a	3390a	3430a	9200a	3.24a	2.42a	2.12a
20 1980-	2500a	3330a	3700a	9540a	3.19a	2.48a	2.13a
60 1980-	2440a	3520a	3790a	9750a	2.88a	2.50a	2.02a
80 1980-88	2450a	3220a	3470a	9140a	3.14a	2.50a	2.10a
<b>1991 S application 50 kg/ha as magnesium sulphate in the spring</b>							
0 1980-	2340a	3420a	3550a	9310a	4.29c	3.66ab	3.84b
20 1980-	2510a	3470a	3740a	9720a	3.88b	3.90b	3.63ab
60 1980-	2430a	3480a	3660a	9600a	3.22a	3.35a	3.54a
80 1980-88	2560a	3400a	3860a	9820a	4.27c	3.97b	3.62ab

The decrease in plant S content by potassium chloride application was largest at the higher level of applied S in the second year, up to 25% from the control that received no KCl. In the first year the decrease in plant S content occurred together with a significant decrease in the DM yields in the first and third cuts. The cocksfoot stand appeared paler green with K application even in the second year.

These findings suggest that the S contents in grasses could be increased and the requirement for S application possibly decreased by using low-chloride K fertilizers instead of application of the customary compound fertilizers that contain KCl. Restricted relative application of Cl would be particularly effective in the low-K organic soils, where high rates of K are required for maximum yields (Saarela 1983), but additional studies are needed in order to evaluate the significance of these findings and their possible utilization.

The significant increases in plant S content by P application evidently resulted from the small amount of S in the triple superphosphate (Table 3). Similar amounts of S impurities were also applied by the "zero" control treatment in the six experiments in grassland presented above. This is a possible explanation for the inefficiency of the S application. Soon after the first cut in the year 1991, the cocksfoot stand in the clay soil in Jokioinen was also very distinctly darker green with the higher rate of P that was applied in the spring, but no significant differences in DM yield were found.

Table 3. Effects of freshly applied and residual phosphorus fertilization on a cocksfoot-red clover ley on a clay soil in Jokioinen (see Table 2 for details)

P(S) kg/ha	applied	Dry matter yield, kg/ha				S content, g/kg DM		
		Cut 1	Cut 2	Cut 3	Total	Cut 1	Cut 2	Cut 3
<b>1990</b>								
0 (0)	1980-	2840a	4070a	1550a	8470a	1.93a	1.77a	2.33a
15 (1)	1980-	2810a	3910a	1570a	8280a	2.03ab	1.79a	2.38ab
45 (3)	1980-	2890a	3890a	1590a	8540a	2.11b	2.04b	2.55b
60 (0)	1980-88	2940a	4010a	1460a	8420a	1.94a	1.85a	2.55a
<b>1991 No additional S application</b>								
0 (0)	1980-	2540a	3230a	4050a	9830a	2.11a	1.98a	1.75ab
15 (1)	1980-	2570a	3350a	4030a	9950a	2.36b	1.94a	1.73a
45 (3)	1980-	2760a	3340a	4220a	10310a	2.74c	2.35b	1.96b
60 (0)	1980-88	2670a	3350a	4050a	10070a	2.11a	2.07a	1.78ab
<b>1991 S application 50 kg/ha as magnesium sulphate in the spring</b>								
0 (0)	1980-	2460a	3420a	3690a	9520a	3.12a	2.90a	2.70ab
15 (1)	1980-	2630a	3400a	4100a	10080a	3.10a	2.81a	2.65ab
45 (3)	1980-	2570a	3570a	3980a	10090a	3.20a	2.97a	2.88b
60 (0)	1980-88	2600a	3410a	3860a	9880a	2.93a	2.89a	2.59a

### Residual effect of sulphate application

No residual effects of the large rates of S (up to 640 kg S/ha) applied in single superphosphate during 1980–87 were found. In this field the sorption of sulphate in the surface soil was evidently very weak because of the high pH (6.8–7.1) and the high content of extractable phosphorus (Yli-Halla 1987b). Similar results were also obtained in a pot experiment with turnip rape, in which this clay and two soils from other P fertilization experiments were studied (Table 4).

Table 4. Residual effects of field-applied sulphur on DM yield of pot-grown turnip rape on three soils from Jokioinen in 1985 (g/pot). Field applications of 250 and 1000 mg S/pot represent 20 and 80 kg S/ha annually during 1980–84. 500 mg S/pot added directly to the 5-l pots

S application mg/pot	Mull soil		Loamy clay		Fine sand	
	Seed	Stalk	Seed	Stalk	Seed	Stalk
0	0	14.8	0	7.0	0	9.8
250 in field	0	14.5	0	7.8	-	-
1000 in field	0	16.4	0	9.6	0	22.8
500 to pot	37.7	66.9	34.5	63.0	34.0	61.2

The low availability of S in the plough layer together with the high amounts of S taken up from the soil in the field suggest that the deeper layers would be important sources of this nutrient. The increase in the content of extractable S with depth was detected in several profiles in clay soils, but not in some silt and fine sand soils. The distribution of available S in the soil profiles should evidently be taken into account in developing the methodology of chemical soil testing for sulphur.

### Sulphur requirements of plants

According to Tähtinen (1977) the optimal S content in the DM of timothy hay was 1.3 g/kg or higher and the critical ratio of total N to total S was 14. In the field experiment at Jokioinen with three cuttings at the silage stage the critical S content appeared higher, about 2.0 g/kg, but the critical N/S ratio was quite similar.

In the pot experiment with rye grass, timothy, meadow fescue, cocksfoot and red clover, the absolute S requirement of the grasses, which also were cut at the silage stage, was much lower, about 1.0 g/kg (Fig. 1), but the critical N/S ratio (Fig. 2) was again the same as earlier, about 14. In a study by Jones et al. (1972) the sufficient S content of pot-grown rye grass was assessed as 1.7 g/kg and the critical N/S ratio 15.

Optimal S content of pot-grown turnip rape was about 3.5 g/kg in the seeds and 1.2 g/kg in the stalks (Fig. 3). Total prevention of seed formation in the absence of applied S showed the poor availability of this nutrient in the soil as well as the indispensability of S for the oilseed rapes. The declining atmospheric deposition of S has greatly increased the requirement for sulphur fertilization of oilseed rapes in other European countries in the recent years (Schnug 1991; Walker & Booth 1992). Additional research on sulphur nutrition of oilseed rapes would also be needed in Finland.

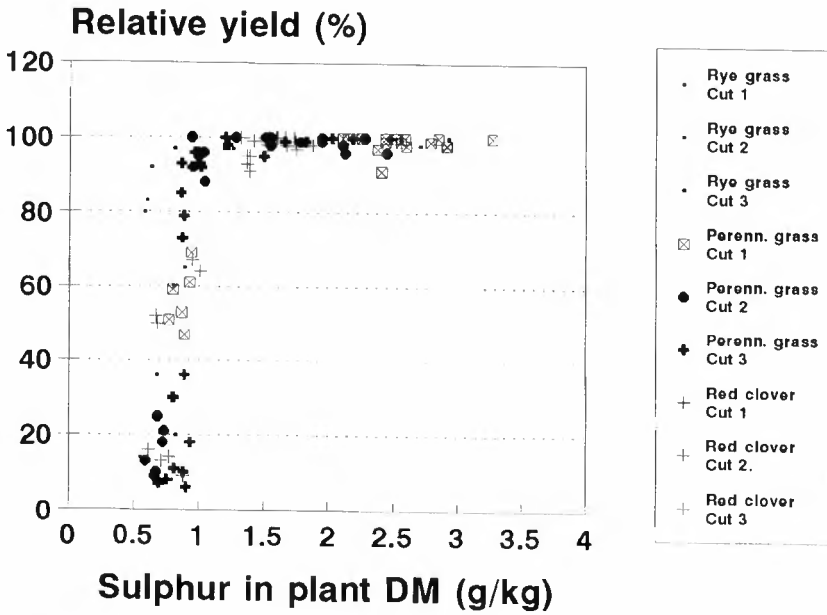


Fig. 1. Relationships between relative DM yield and content of total sulphur in pot-grown grasses and clover

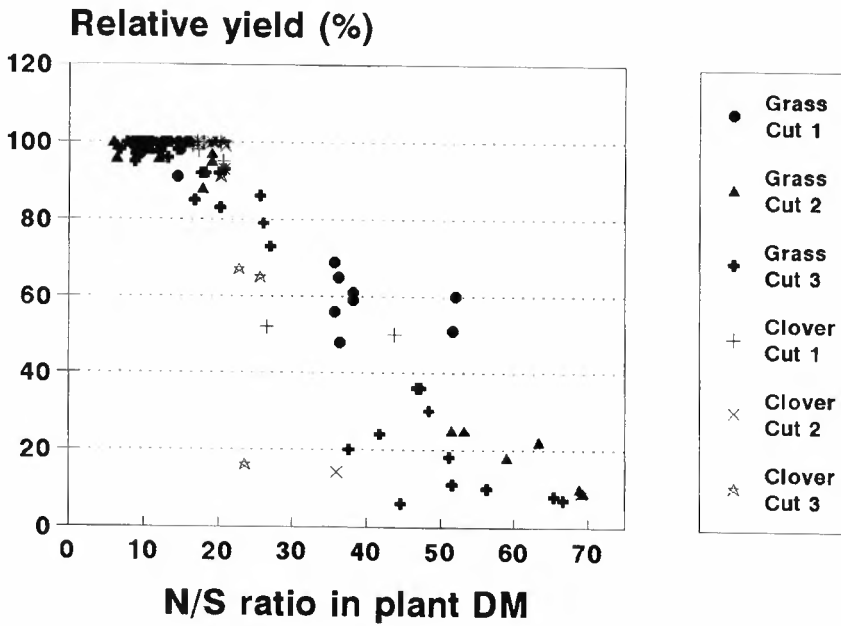


Fig. 2. Relationships between relative DM yield and the ratio of total N to total S in pot-grown grasses and clover

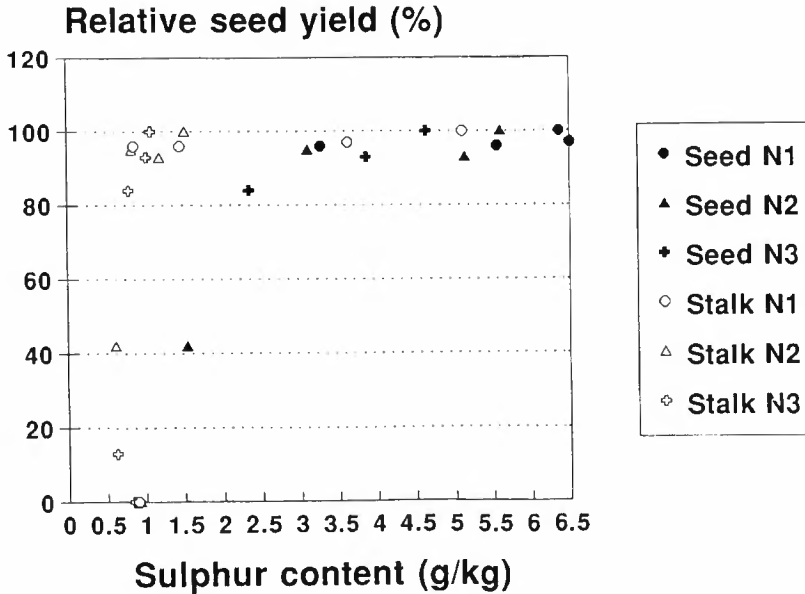


Fig. 3. Relationships between seed yield and content of total sulphur in seeds and stalks of potgrown turnip rape

## CONCLUSIONS

Significant increases in plant S content with small amounts of applied S showed that small rates of sulphate are utilized efficiently by grasses when the initial availability of S in the soil is poor. The decreased content of S in grass after KCl application implies that plant S content could be increased by using low-chloride fertilizers.

The insignificant and weak residual effects of prior S fertilization suggest that sulphates should be applied annually. Subsoil sampling seem to be essential for reliable soil testing for sulphur. The ratio of total N to total S in plants appears to be a useful indicator of S nutrition of grasses.

The high sulphur requirement of oilseed turnip rape and the poor reserves of available sulphur in Finnish soils were demonstrated in a pot experiment. The declining atmospheric deposition of S has greatly increased the requirement of S fertilization in other European countries. Sulphur nutrition of the oilseed rapes in Finland would thus be a relevant topic for additional studies.

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# Sulphur deficiency of potatoes as reflected in chemical composition and in some measures of nutritive value

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In pot experiments, S deficiency reduced tuber dry matter (DM) yield and the concentration of DM, starch, dietary fibre and total-S (S<sub>t</sub>). As concentrations of total-N (N<sub>t</sub>) and of most mineral constituents increased, the ratio N<sub>t</sub>/S<sub>t</sub> increased from about 11 to 33 with S deficiency. Lack of S also reduced the content of essential amino acids in crude protein, particularly cystine and leucine, but increased glutamic acid (glutamine) and arginine. The chemical score of crude protein decreased from 77 to 41 with S deficiency. The cystine- + methionine-S content of total-S increased from about 45% to about 80% without S application. In N-balance trials with rats, S deficiency reduced the biological value of crude protein from 81 to 55. Thus, close agreement was established between chemical and biological protein quality evaluation.

Key words: Amino acids, biological value, dietary fibre, micronutrients, nitrate, N/S ratios, starch.

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In recent years, an increasing number of cases of S deficiency have been reported in several European countries. In Denmark, so far, S deficiency has been found mainly in Brassica crops, especially rape (*Brassica napus* L.) (Skriver 1991). Changes in fertilizer practice and in levels of atmospheric S are mainly responsible for the increasing necessity, under certain soil and climatic conditions, to apply fertilizer-S to crops with a high S demand. However, gradually, even less vulnerable crops may be affected by S deficiency too. Recent estimates have indicated that Danish agricultural soils on average have a negative S balance of about 16 kg S ha<sup>-1</sup> year<sup>-1</sup> (Eriksen 1990). As S deficiency not only affects crop yields but also the chemical composition and quality of crops, the aim of the present experiment, which is part of a more comprehensive investigation of nutrient effects on vegetable quality, was to study the effects of S deficiency on chemical composition and the nutritive value of potatoes.

## MATERIALS AND METHODS

The plants were grown outdoors, in PVC pots with a cross-section of 500 cm<sup>2</sup> and a height of 40 cm. A sandy soil from Sdr. Omme, Jutland, mixed with 25% by volume of sphagnum, was used. The pH (H<sub>2</sub>O) of the soil mixture was 4.0. Each pot received a basal dressing of 50 g CaCO<sub>3</sub>, 1.5 g MgO, 0.25 g Mn as MnCl<sub>2</sub> · 4H<sub>2</sub>O, 0.25 g Cu as CuCl<sub>2</sub> · H<sub>2</sub>O, 8 mg B as Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub> · 10 H<sub>2</sub>O, 5 mg Mo as Na<sub>2</sub>MoO<sub>4</sub> · 2 H<sub>2</sub>O, 9 g N as NH<sub>4</sub>NO<sub>3</sub>, 4 g P as Ca (H<sub>2</sub>PO<sub>4</sub>)<sub>2</sub> · H<sub>2</sub>O and KH<sub>2</sub>PO<sub>4</sub>, and 8 g K, mainly as KCl. Sulphur was applied as CaSO<sub>4</sub> · 2H<sub>2</sub>O. Depending on treatment, there were 4 or 6 replicates. One chitted potato tuber (*solanum tuberosum* L. cv. Bintje) was planted per pot on April 18, 1991. At harvest, 15. August, 1991, the tubers were washed, photographed, weighed and boiled in 200–300 ml water before storage, including peels, at -20°C and subsequent freeze-drying.

**Analytical methods**

Various chemical analyses and biological tests were carried out as described in earlier studies (Eppendorfer & Eggum 1994).

## RESULTS AND DISCUSSION

Without S application dry matter (DM) yield was reduced by about 30% compared with yields after application of medium levels of S, while 3.0 g S also caused a reduction in yield (Table 1). S deficiency also resulted in a lower percentage of tuber DM.

Table 1. Effects of sulphur applications on dry matter (DM) yield and percentage and on content of starch, total dietary fibre (TDF) and energy of freeze-dried, boiled potato tubers

g S/pot	g DM/pot	Tuber DM, %	Starch %	TDF %	Energy kJ/g
0	269 ± 25	19.9	75.1	9.48	17.0
0.15	340 ± 27	20.5	78.7	8.51	16.8
0.50	385 ± 33	21.2	78.0	8.33	16.9
1.50	376 ± 29	22.0	77.4	7.98	17.0
3.00	312 ± 49	21.7	77.7	7.68	16.3

**Starch and total dietary fibre content**

In Table 1 it can be seen that lack of S application reduced the starch content of freeze-dried, boiled potatoes, whereas the total dietary fibre content was increased by S deficiency. The energy content of DM, on the other hand, was hardly affected by various treatments. The present results are in agreement with those obtained a year earlier (Eppendorfer & Eggum 1994) when S deficiency was more pronounced than in 1991.

### Mineral composition

The effects of increased application of S on the mineral composition of DM are presented in Table 2. It can be seen that S deficiency caused an increase in total-N, and, in particular, in nitrate-N. This is in agreement with earlier findings (Eppendorfer & Eggum 1994) and reflects the fact that S deficiency has a negative effect on the N metabolism of plants, resulting in the accumulation of a number of N compounds, but reducing the synthesis of proteins (Coic et al. 1963; Eppendorfer 1968; Marschner 1986). Total S content of tubers was reduced by about 50% under S deficiency conditions which, together with the corresponding rise in N content, caused an increase in the ratio of total-N/total-S ( $N_t/S_t$ ) from about 11 to almost 33. These results point to a possibility of using either the total-S content or the  $N_t/S_t$  ratio or both in judging and determining the S nutritional status of potatoes and, possibly, obtaining an expression of their nutritive value. The N/S ratio has received considerable attention as a tool in the identification of S deficiency in a number of plants (Freney et al. 1982; Scaife & Burns 1986).

Table 2. Effects of sulphur applications on mineral composition of DM of freeze-dried, boiled potato tubers

g S/pot	Total-N %	NO <sub>3</sub> -N ppm	Total-S %	$N_t/S_t$	P ‰	K ‰	Na ppm	Ca ppm	Mg ppm	Fe ppm	Zn ppm	Mn ppm	Cu ppm
0	2.01	61	0.06	33.5	3.94	18.7	99	313	817	26	15	11	7
0.15	1.68	26	0.08	21.0	3.50	14.9	48	227	733	20	15	9	8
0.50	1.57	18	0.13	12.1	3.28	14.3	44	286	685	21	15	9	8
1.50	1.51	14	0.14	10.8	3.26	15.3	45	261	711	22	15	8	7
3.00	1.62	18	0.15	10.8	-	-	-	-	-	-	-	-	-

It is also evident from Table 2 that S deficiency resulted in increased concentrations of phosphorus, potassium, sodium, calcium, magnesium, iron and, possibly, manganese in DM, whereas zinc and copper concentrations seemed unaffected. The increases in mineral concentrations are probably mainly due to a corresponding yield decrease (Table 1), causing the opposite of a "dilution effect", which is often found when plants or crops are feeding on a limited amount of plant nutrients other than the one that is deficient. Generally, the range of mineral concentrations found in the present investigation agrees quite well with values obtained by True et al. (1979), who also observed that cooking had a negligible effect on the mineral content of raw potato flesh.

### Amino acid composition

S deficiency markedly affected the amino acid composition of crude protein by reducing the concentration (g/16 g N) of all essential amino acids (Table 3). This is not surprising since S, like N, is an integral part of S amino acids, the concentration in crude protein of methionine and, in particular, of cystine, decreased by about 30 and 60%, respectively. Decreases in other essential amino acids, due to S deficiency, amounted to approximately 45, 35, 30, 30, 25 and 20% for leucine, threonine, isoleucine, tryptophan, valine and lysine, respectively. Of none essential amino acids, glutamic acid (glutamine) and arginine concentrations in crude protein were considerably increased by S deficiency, which could be a characteristic effect of S

deficiency. Similar and even more pronounced results were obtained in a corresponding experiment with potatoes a year earlier (Eppendorfer & Eggum 1994) when S deficiency was stronger than in 1991. Concentrations of crude protein are also given in Table 3, as they often have been found largely to determine the amino acid composition of crude protein of a number of crops, including potatoes. Thus, an increase in crude protein content, whether due to N applications or deficiencies in P and K, normally causes decreasing concentrations (g/16 g N) of a number of essential amino acids. In the present investigation, although crude protein content was increased by S deficiency, the corresponding decrease in essential amino acid concentrations was considerably larger than might have been expected from the modest rise in crude protein.

Table 3. Effects of sulphur applications on amino acid composition of freeze-dried, boiled potato tubers

g S/pot	0	0.15	0.50	1.50	3.00	0	0.15	0.50	1.50	3.00	Ref. prot. <sup>4</sup>
% CP <sup>5</sup>	12.56	10.50	9.81	9.44	10.13	12.56	10.50	9.81	9.44	10.13	
	g amino acid / kg DM					g amino acid / 16 g N					
Cys	0.706	0.974	1.26	1.21	1.37	0.56	0.93	1.28	1.28	1.36	} 3.5
Met	1.38	1.25	1.45	1.32	1.62	1.10 <sup>2</sup>	1.19 <sup>1</sup>	1.48 <sup>2</sup>	1.40 <sup>1</sup>	1.60 <sup>2</sup>	
Lys	4.88	4.68	4.90	4.59	4.99	3.88	4.46	4.99	4.86	4.93	5.5
Thr	2.81	3.01	3.30	3.19	3.42	2.24 <sup>3</sup>	2.87 <sup>3</sup>	3.37 <sup>3</sup>	3.38 <sup>3</sup>	3.38 <sup>3</sup>	4.0
Trp	1.04	1.02	1.17	1.05	1.18	0.83	0.97	1.19	1.11	1.17	1.0
Ile	3.20	3.33	3.57	3.34	3.74	2.55	3.17	3.63	3.54	3.69	4.0
Leu	3.60	4.66	5.21	5.11	5.46	2.86 <sup>1</sup>	4.43 <sup>2</sup>	5.31 <sup>1</sup>	5.41 <sup>2</sup>	5.39 <sup>1</sup>	7.0
Val	5.03	4.82	4.93	4.65	5.27	4.00	4.59	5.03	4.93	5.20	5.0
Phe	3.56	3.54	3.76	3.68	3.97	3.56	3.37	3.83	3.90	3.92	} 6.0
Tyr	3.35	2.92	2.83	2.79	2.82	2.67	2.78	2.88	2.96	2.78	
Asp	24.4	21.5	18.1	17.3	18.3	19.5	20.5	18.5	18.3	18.1	
Glu	25.9	15.9	13.8	13.1	15.4	20.6	15.1	14.1	13.9	15.3	
Arg	8.02	4.54	3.74	3.69	3.89	6.38	4.33	3.81	3.91	3.85	
His	1.54	1.52	1.64	1.53	1.72	1.23	1.45	1.67	1.62	1.70	
Ser	3.67	3.32	3.45	3.41	3.68	2.92	3.16	3.52	3.61	3.63	
Pro	2.01	2.46	2.75	2.73	2.98	1.60	2.35	2.81	2.89	2.94	
Gly	1.95	2.54	2.84	2.81	3.01	1.55	2.42	2.89	2.97	2.97	
Ala	3.11	2.95	3.05	3.18	3.42	2.48	2.81	3.12	3.37	3.38	
Chemical Score						41	61	76	77	77	
% amino acid N of total N						65.7	65.0	67.1	67.1	68.4	
% cystine + methionine-S of total-S						80.8	66.0	49.8	43.3	47.6	

<sup>1</sup>), <sup>2</sup>), <sup>3</sup>) first, second and third most limiting amino acid

<sup>4</sup>) WHO/FAO reference protein

<sup>5</sup>) Crude protein

In Table 3 values of a FAO/WHO reference protein are also given, which allows a ranking according to the most limiting amino acid. Thus, leucine was the first limiting amino acid in three treatments and cystine + methionine in two. A comparison with the reference protein values also shows that the concentrations of most essential amino acids were less than recommended, even at the lowest concentration of crude protein. The effects of S deficiency resulting in changes in amino acid composition are also reflected in the corresponding decrease

in chemical score, which expresses the percentage value of the first limiting essential amino acid in relation to the corresponding recommended value of the reference protein. Table 3 also contains values of the percentage of cystine + methionine-S of total-S. Naturally, these values are highest under S deficiency conditions.

In Table 3 the amino acid composition is also expressed as g/kg DM. Normally, an increase in crude protein percentages will result in increased concentrations of all amino acids when expressed as g/kg DM, although to varying degrees. However, under S deficiency conditions a number of essential amino acids deviate from this general rule, as their concentration is lowest at the highest crude protein concentration (treatments 0 and 0.15 g S/pot).

#### Biological evaluation (N-balance trials with rats)

In order to compare and supplement the above results of chemical protein quality evaluation with results from a biological assessment, N-balance trials with rats were conducted with material from four of the five treatments. The results in Table 4 show that the true digestibility (TD, the percentage of the N intake absorbed in the body) of potato crude protein increased with increasing crude protein percentages (Table 3). Such increases are often found in various foods and feeds irrespective of the cause of increase in crude protein content, whether they were due to N applications or a deficiency of other nutrients.

Table 4. Effects of sulphur applications on percentage of true digestibility (TD), biological value (BV), net protein utilization (NPU), and digestible energy (DE) in DM of freeze-dried, boiled potato tubers

g S/pot	TD	BV Mean $\pm$ s.e.	NPU	DE
0	84.9 $\pm$ 0.38	54.6 $\pm$ 0.37	46.4 $\pm$ 0.40	90.2 $\pm$ 0.45
0.15	84.1 $\pm$ 0.44	67.2 $\pm$ 0.26	56.6 $\pm$ 0.43	90.0 $\pm$ 0.33
0.50	83.5 $\pm$ 0.32	78.2 $\pm$ 0.35	65.3 $\pm$ 0.15	90.1 $\pm$ 0.36
1.50	81.2 $\pm$ 0.43	81.3 $\pm$ 0.39	66.0 $\pm$ 0.60	90.6 $\pm$ 0.33

As Table 4 shows, the biological value of the crude protein (BV, the percentage of N absorbed that is retained in the body) was greatly reduced by S deficiency. This decrease was much more pronounced than that normally found with higher crude protein content. This is clearly evident from Fig. 1, which shows the relationship between crude protein content of DM and the biological value of both S treatments and of other treatments from experiments conducted in 1990 and 1991 (Eppendorfer & Eggum 1994). There is a close negative relationship that appears to be independent of deficiency or excess of phosphorus and potassium. Generally, the concentration of crude protein seems to determine the size of the BV. However, as Fig. 1 clearly indicates, there is an exception, S deficiency, which causes the values of BV to deviate negatively from the general regression line. The reason for this deviation is to be found in the low levels of essential amino acids of treatments 0 and 0.15 g S/pot, which are also revealed in the corresponding low values of the chemical score in Table 3. Present results thus indicate close agreement between the chemical and biological protein quality evaluation. The net protein utilization (Table 4) was also negatively affected by S deficiency, but less so than the

BV as NPU = TD  $\times$  BV  $\times 10^{-2}$ . The digestible energy of potato DM on the other hand was not affected by S deficiency.

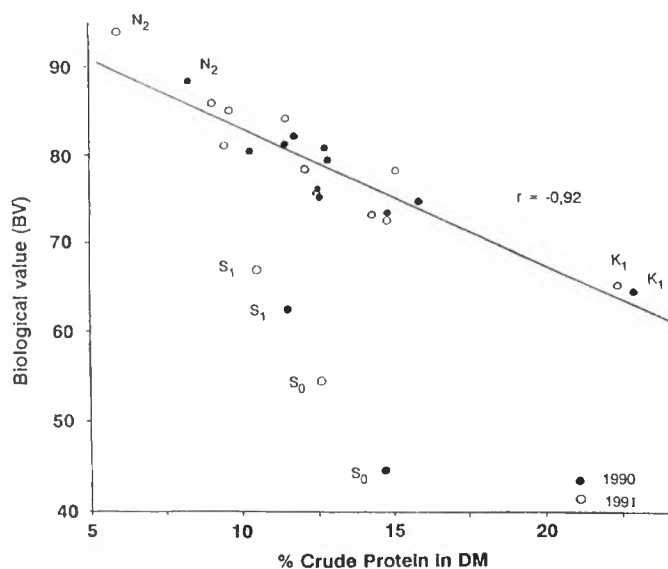


Fig. 1. Relationship between crude protein content and biological value of crude protein of freeze-dried, boiled potato tubers, as influenced by various degrees of S deficiency in two growing seasons ( $S_0$  = without S applied and  $S_1$  = 0.15 g S/pot). Treatments  $N_2$  and  $K_1$  indicate N and K deficiency. Excluding treatments  $S_0$  and  $S_1$  the linear regression for  $BV = 98.78 - 1.55 \times \% \text{ crude protein}$ ,  $r = -0.92^{***}$

A clearer picture of the effects of S deficiency on some of the more important constituents of potato tuber DM and on some measures of their nutritive value can be seen in Fig. 2 which gives the relationships between increasing S applications and DM yield, chemical composition and  $N_t/S_t$ , chemical score and biological value. This figure indicates that, by and large, a S application of 0.5 g/pot was about sufficient for maximum DM yield and for an optimal nutritive value of potato crude protein. The relationships in Fig. 2 also make it appear likely that an optimal yield, composition and nutritive value will be obtained with an S concentration in DM of about 0.13% and a  $N_t/S_t$  ratio of about 12–13.

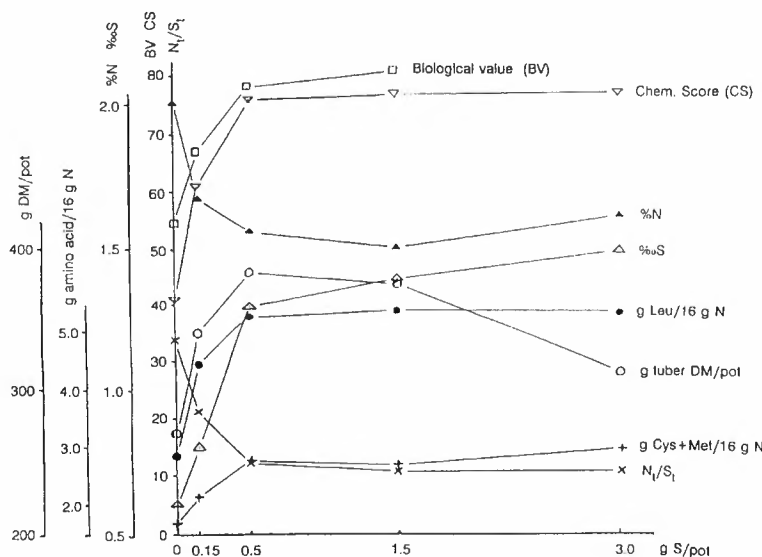


Fig. 2. Relationships between increasing S-applications and dry matter yield, chemical composition,  $N_{total}/S_{total}$  ratio and nutritive value of freeze-dried, boiled potato tubers

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# Effect of sulphur deficiency on amino acid composition

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In a series of analyses it was found that sulphur deficiency caused a decrease in the concentration (based on nitrogen content) of the sulphur-containing amino acids, methionine and cysteine, in seeds of rape, grain of barley and wheat, and in the vegetative parts of Italian ryegrass and white cabbage. It was also found that the concentration of other amino acids was affected by sulphur deficiency, e.g. a decrease in lysine was observed. For pea, the effect of sulphur deficiency on the amino acid content was much less pronounced. In leaves of white cabbage sulphur deficiency caused an increase in the content of glutamic acid (+ glutamine) in particular, while in Italian ryegrass severe deficiency caused a dramatic increase in the content of aspartic acid (+ asparagine), most of which was present in the free state in the form of asparagine.

Key words: Barley grain, cysteine, lysine, methionine, rape seed, ryegrass, wheat grain, white cabbage.

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Sulphur is taken up by plants in form of sulphate and for the most part has to be reduced before incorporation in the organic compounds in the plants. Only a few organic compounds containing sulphur in an unreduced form are known in plants, such as the glucosinolates, especially occurring in the Cruciferae and the sulpholipides. In its reduced state, sulphur is predominantly present in the amino acids methionine and cystine/cysteine, which probably can be considered as the most important sulphur-containing functional compounds in plants, even though sulphur also plays a role by its presence in some of the coenzymes such as thiamine, biotine and coenzyme A (Marschner 1986). The amino acid methionine is used in initiation of the synthesis of the protein chains, and also plays a role as a methyl donator in different metabolic processes. Cysteine residues in the protein have the capability to form S-S bridges between the protein chains, which is of importance for the tertiary structure and function of the enzymes.

As a constituent of the amino acids and proteins, sulphur must be considered as the most essential nutrient next to nitrogen for the synthesis of amino acids and proteins. Sulphur deficiency is known to decrease the fraction of sulphur amino acids in the seed proteins such as in barley (Eppendorfer 1968), field beans (Eppendorfer 1971) and wheat (Randall &

Wrigley 1986).

The present investigations comprised amino acid analyses of seed and grain and the vegetative material of different plant species in order to investigate the effect of sulphur deficiency on the amino acid content.

## MATERIALS AND METHODS

The plant material, rape, barley, wheat, pea and white cabbage, used for the analyses was taken from pot experiments (pot volume 30 l, surface area 0.1 m<sup>2</sup>) where the plants were grown on media poor in available sulphur or where sulphate was removed beforehand by leaching. Different degrees of sulphur deficiency were obtained by supplying the pots with different amounts of sulphate-S. Samples of Italian ryegrass were selected according to the content of sulphur from plants grown in pots on different soils (sandy to loamy) in a depletion experiment.

After harvest, the plant material was dried and analysed as follows: Total nitrogen was determined by a Kjeldahl method, total sulphur by turbidimetry after wet-ashing with magnesium nitrate and perchloric acid (Nes 1979) or dry-ashing with magnesium nitrate alone.

The content of the sulphur amino acids was determined by ion chromatography after oxidation with performic acid and hydrolysis with hydrochloric acid (Bech-Andersen et al. 1981). In addition to the sulphur amino acids, the programs used for the chromatography also gave the content of the lysine, threonine, aspartic acid and, in some cases, other amino acids.

In the material analysed for free amino acids and amides the extraction was performed with perchloric acid followed by derivatization with phenylisothiocyanate (PITC) (Henrikson & Meredith 1984) and determined by HPLC chromatography on a Hewlett-Packard 1084B instrument.

## RESULTS AND DISCUSSION

### **Amino acids in seeds**

Rape was grown at two levels of nitrogen and an interaction between nitrogen and sulphur was observed for the yield and nutrient content (Fig. 1). At the low level of nitrogen (1.5 g N/pot) nitrogen was the limiting factor for yield and only a small or no effect of sulphur could be observed (seed yield varying from 16 to 19 g DM/pot). At the 4 g N/pot level a marked effect of sulphur was found. When no sulphur was supplied the yield of seed (3 g DM/pot) was even lower than that achieved at the low nitrogen level, whereas the highest yield of seed achieved (48 g DM/pot) by sulphur addition was more than the double of that at the low nitrogen level.

The concentration of sulphur (Table 1) was highest at the low nitrogen level and increased with sulphur supply. The nitrogen content was independent of the sulphur supply at the low nitrogen level, but decreased at the high nitrogen level. The relative concentration (based on nitrogen content) of the sulphur amino acids increased with an increased supply of sulphur, but was more pronounced at 4.0 g N than at 1.5 g N. This was also the case for lysine, while the concentration of aspartic acid (includes hydrolysed asparagine) was independent of the sulphur supply.

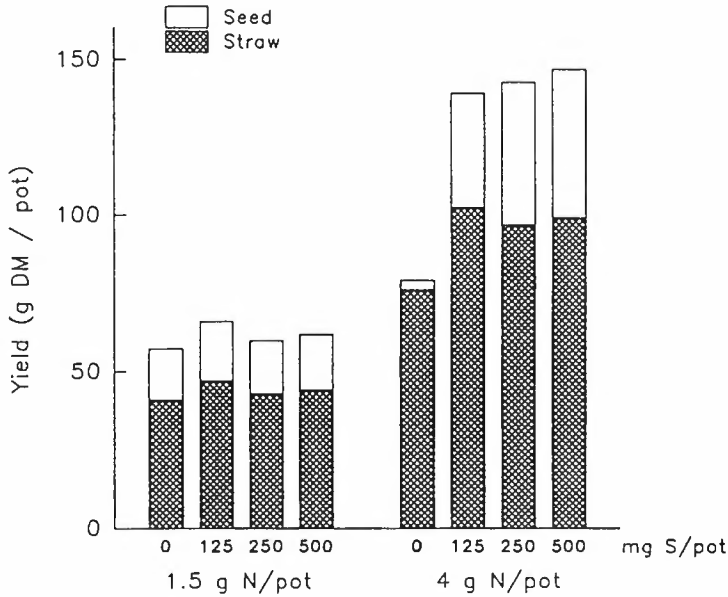


Fig. 1. Effect of sulphur on yield of seed and straw of rape (*Brassica napus* L. ssp *oleifera*) at two levels of nitrogen fertilizer. Pot experiment with growth medium poor in available sulphur

Table 1. Influence of sulphur on amino acid composition in rape seeds at two levels of nitrogen. Pot experiment

Nutrient supply		Content in seeds					
S (g/pot)	N	Nutrients		Amino acids			
		S (% in DM)	N	Met	Cys (amino acid N in % of TN)	Lys	Asp
0	1.5	0.23	2.8	1.13	1.38	7.0	5.4
0.125	1.5	0.37	2.8	1.17	1.49	7.1	5.3
0.250	1.5	0.28	2.8	1.17	1.38	7.1	5.2
0.500	1.5	0.39	2.7	1.22	1.59	7.6	5.4
0	4.0	0.20	4.1	0.81	0.76	5.4	5.3
0.125	4.0	0.20	3.6	0.92	0.99	5.9	5.8
0.250	4.0	0.24	3.2	1.02	1.13	6.4	5.5
0.500	4.0	0.29	3.1	1.19	1.46	7.1	5.5

For barley, wheat and pea the yield was strongly increased by the sulphur supply (Fig. 2), indicating that severe sulphur deficiency occurred when no sulphur was given. For barley and wheat the sulphur content was almost unchanged (Table 2) with increasing sulphur supply, while the nitrogen content was decreased. In pea, the content of both sulphur and nitrogen was increased by the highest application of sulphur. The discrepancy between pea and the cereals is probably partly due to a difference in the mode of nitrogen supply. The peas had to meet their demand for nitrogen by symbiotic nitrogen fixation, while barley and wheat were fertilized with nitrate-nitrogen and possibly sulphur has a greater influence on the fixation than on the

uptake and assimilation of nitrate. An analogous difference has been found between the legume subterranean clover and ryegrass in the effect of sulphur on the nitrogen concentration in young leaves (Gilbert & Robson 1984).

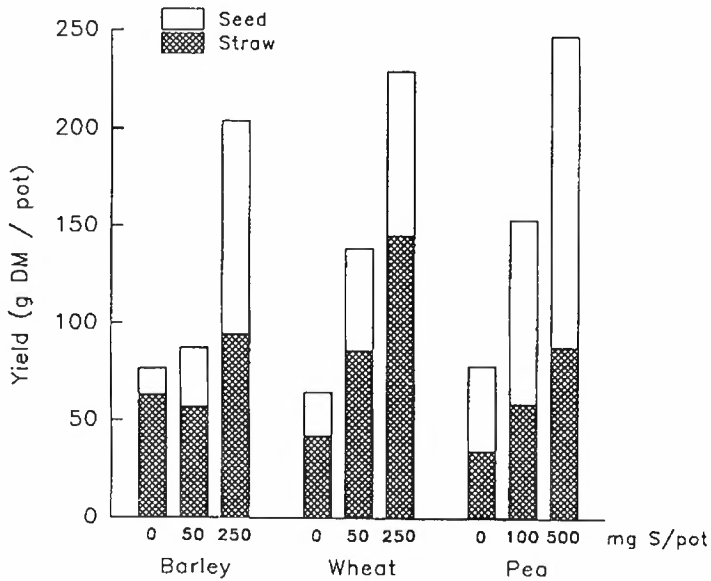


Fig. 2. Effect of sulphur on yield of grain and straw of barley (*Hordeum vulgare* L.), spring wheat (*Triticum aestivum* L.) and pea (*Pisum sativum* L.). Pot experiment with growth medium poor in available sulphur

Table 2. Effect of sulphur on amino acid composition in grain of barley, wheat and pea. Pot experiment on a medium poor in available sulphur

Species	Sulphur supply (g/pot)	Content in grain							
		Nutrients		Amino acids					
		S	N	Met	Cys	Lys	Thr	Asp	
		(% in DM)			(amino acid N in % of TN)				
Barley	0	0.1	3.3	0.65	0.87	3.34	2.12	6.93	
	0.05	0.10	2.7	0.73	0.94	3.74	2.25	4.22	
	0.25	0.10	1.5	1.01	1.50	4.70	2.74	4.26	
Wheat	0	0.12	3.2	0.61	0.99	3.03	1.89	4.01	
	0.05	0.12	2.8	0.53	1.05	2.90	1.93	4.02	
	0.25	0.12	2.2	0.88	1.52	3.30	2.22	3.28	
Pea	0	0.13	3.1	0.61	1.08	9.20	3.12	7.62	
	0.10	0.13	3.1	0.59	1.16	8.94	3.06	7.57	
	0.50	0.19	3.5	0.66	1.22	9.29	3.12	7.87	
LSD (P=0.05)		0.02	0.3	0.20	0.10	0.43	0.12	1.13	

The relative amino acid concentration was affected differently in the different species by the sulphur supply. For barley the concentrations of methionine, cysteine, lysine and threonine were clearly increased, while the concentration of aspartic acid (+ asparagine) was decreased. The same trends were found for wheat, but were less pronounced. For pea only small changes were found even for the sulphur amino acids.

The effect of sulphur deficiency on protein content in cereals, oilseeds and legumes is often found to be variable (for a review, see Randall & Wrigley 1986). Changes in the amino acid composition in seeds caused by sulphur deficiency can, in general, be ascribed to an increase in synthesis of proteins low in sulphur amino acids (Fullington et al. 1987; Spencer et al. 1990).

### Amino acids in vegetative material

The concentration values (based on N) of some selected amino acids in white cabbage are presented in Table 3. Supply of sulphur gives a clear increase in the concentration of sulphur and a decrease in the concentration of reduced N and nitrate-N. As expected, the concentration of the sulphur amino acids was increased, but this was also the case for lysine and aspartic acid. The concentration of glutamic acid (+ glutamine) was much higher in the sulphur-deficient plants than in the sulphur-supplemented plants.

Table 3. Content of amino acids in hydrolysed samples of white cabbage (*Brassica oleracea* L. var. *capitata* L. f. *alba* DC.) without and with supply of sulphur. Pot experiment with two soils

Soil	Sulphur supply (g/pot)	Content in cabbage (whole above-ground part)							
		Nutrients			Amino acid				
		S	RN <sup>1)</sup> (% of DM)	NO <sub>3</sub> -N	Met	Cys (amino acid N in % of RN)	Lys	Asp	Glu
1	0	0.12	3.8	2.0	0.36	0.47	2.39	2.47	10.86
1	0.6	0.46	2.7	0.5	0.87	0.84	4.23	4.62	5.83
2	0	0.08	3.0	1.0	0.59	0.57	3.29	3.55	9.47
2	0.6	0.42	2.0	0.2	0.93	0.89	4.77	5.16	5.66

<sup>1)</sup> RN is reduced N, i.e. total-N minus nitrate-N

The relative concentration of amino acids in the four samples of Italian ryegrass seems to be strongly dependent on the sulphur content (Table 3). Methionine and cysteine concentrations increased with increased content of sulphur, which was also the case for lysine. The concentration of aspartic acid was extremely high at the lowest sulphur level and declined with increasing concentrations of sulphur. A similar increase in aspartic acid caused by sulphur deficiency was reported for ryegrass by Millard et al. (1985) and for green barley by Eppendorfer (1968).

There was no noticeable change in the content of nitrate-N (range 1.37 to 1.70% NO<sub>3</sub>-N of DM), nor change consistent with the change in sulphur content in the four samples, which is in contrast to the strong increase in nitrate content that is normally found with sulphur deficiency (Millard et al. 1985) and as observed here for white cabbage.

**Free amino acids**

It was unlikely that the high level of total aspartic acid (+ asparagine) found in the grass sample with the lowest sulphur content (Table 4) could be present in proteins. An analysis of some of the free amino acids (Table 5) revealed an extremely high content of asparagine at the lowest sulphur level. In fact, it was indicated by means of a calculation that at the lowest sulphur level the amount of asparagine in the free state could account for nearly 100% of the total aspartic acid found in the hydrolysed sample, decreasing to about 12% at the highest sulphur concentration. In contrast the content of free threonine varied from only 3 to 5% of the total threonine, independent of sulphur supply. The concentration of the other amide-amino acid, glutamine, was also high at the zero-supply of S, but not to the extreme of asparagine. The concentrations of aspartic and glutamic acid did not vary to the same degree as those of the corresponding amides. The content of serine varied remarkably, while that of alanine and threonine, only changed a little, and not consistently with changes in the sulphur supply.

Table 4. Amino acid composition in hydrolysates of samples of Italian ryegrass (*Lolium multiflorum*) selected for analysis and arranged according to sulphur content

S	Nutrients RN <sup>1)</sup> (% of DM)	NO <sub>3</sub> -N	Content in grass (whole shoots)				
			Met	Cys	Lys	Thr	Asp
			Amino acids (amino acid N in % of RN)				
0.10	4.0	1.4	0.59	0.42	3.94	2.15	24.4
0.15	3.4	1.7	1.07	0.72	6.40	3.27	12.0
0.19	3.5	1.7	1.21	0.88	7.19	3.64	8.9
0.26	4.1	1.5	1.25	0.92	7.15	3.68	6.6

<sup>1)</sup> RN is reduced N, i.e. total-N minus nitrate-N

Table 5. Effect of sulphur on content of some free amino acids in Italian ryegrass, leaves of rape and in barley grain

Material	Sulphur defic. <sup>1)</sup>	Nutrients		Content in material						
		S	N	Asn	Asp	Gln	Amino acids ( $\mu\text{mol g}^{-1} \text{DM}$ )			Ala
		(% of DM)								
Italian ryegrass	1)	0.10	4.0	790	35	40	15	59	37	4
	1)	0.15	3.4	168	21	19	15	27	45	3
	1)	0.19	3.5	67	15	12	15	19	36	3
	2)	0.26	4.1	7	16	2	10	5	46	3
Rape leaves	1)	0.41	4.5	5	9	49	10	13	32	4
	2)	0.99	5.0	3	8	4	6	6	34	4
Barley grain	1)	0.10	3.2	138	18	19	7	3	4	1
	2)	0.10	1.5	2	1	< 1	1	< 1	< 1	< 1

<sup>1)</sup> Sulphur deficiency: 1) Deficient in sulphur according to visual symptoms on the green plants (pale leaves, poor growth)

2) No visual symptoms of sulphur deficiency (healthy leaves, normal growth)

The high content of asparagine in the free state indicates a severe disturbance of the protein synthesis in the leaves under sulphur-deficient conditions, while the nitrogen uptake and assimilation are very likely still going on. This indicates that the uptake and assimilation of nitrogen are not regulated directly by the sulphur supply.

A few analyses for free amino acids were performed on material of rape leaves and barley grain originating from the same experiments as the material referred to in Figs. 1 and 2. In rape leaves, in contrast to Italian ryegrass, the greatest increase under sulphur deficient conditions was found for the glutamine content, while that for asparagine was only increased twofold (Table 5). Some amino acids, such as alanine and threonine, appeared to be independent of the sulphur content in the leaves.

Grains of barley normally have a low content of free amino acids as can be seen from the values for the sample from barley plants with no symptoms of deficiency (Table 5). For the sample from sulphur-deficient barley, asparagine was the free amino acid found in highest concentration.

## CONCLUSION

Sulphur deficiency decreases the yield of seeds and grain, while the concentration of sulphur is maintained at a certain minimum level below which it does not fall.

In seeds and grain, sulphur deficiency impairs the synthesis of storage proteins that are rich in the sulphur amino acids and therefore more of the sulphur-deficient proteins is produced.

The uptake and assimilation of nitrate nitrogen does not seem to be directly regulated by the sulphur status of the plants. In sulphur deficient-plants a high concentration of nitrate and free amino acids can be found, probably because utilization of the nitrogen is not possible. In particular, the content of the amides asparagine and glutamine is high, resembling the effect of high nitrogen supply. Clearly, there is a difference between plant species with respect to the amides accumulated, possibly with asparagine preferably in monocotyledons and glutamine in dicotyledons.

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# Effect of sulphur fertilization on oilseed crops

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Two different experimental programmes with sulphur to oilseed plants are presented. In the first programme, 1986-88, micronized sulphur (Elosan 80% S) was sprayed on spring rape at different stages of development. On average, the yield for all sulphur treatments increased slightly compared with that for the control. The best results were achieved when sulphur was sprayed in the late budding stage (+4%). In 1991 an experiment was carried out with supply of sulphur in phosphatic fertilizer to winter rape. In 1992 this was completed with an experimental treatment in which micronized sulphur was sprayed in the early budding stage. Three double low varieties - Ceres, Libraska and Casino - were sown in the trials every year. On average, the yield for supply of sulphur was no higher than that for the control. There seemed to be a slight tendency for Libraska to react a little more than the other varieties for higher yield and content of glucosinolates. The content of raw fat and chlorophyll was not influenced by supplementary sulphur.

Key words: Oil seed plants, sulphur

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In Sweden about 160 000 ha is used for oilseed plant cultivation. Winter and spring types of rape, (*Brassica napus* L) and turnip rape (*Brassica rape* L. *oleifera* DC) are cultivated but the former types are the more commonly grown ones, whereas winter turnip rape is cultivated only to a small extent. With the exception of winter turnip rape, all oilseed plants are double low varieties. In recent years there has been increasing awareness of and discussion about sulphur deficiency in oil seed plants in both Sweden and other countries (Persson 1993). In the autumn of 1992 several reports were received about the occurrence of symptoms of deficiency in the young winter rape plants, in the northwestern part of south Sweden (Götaland).

The most common explanations for increased symptoms of sulphur deficiency are the change over to fertilizers with a lower content of sulphur and the reduction in atmospheric sulphur deposition. The observations made in autumn of 1992 are undoubtedly also due to the fact that only double low varieties of winter rape were cultivated and these varieties are more sensitive to sulphur deficiency (Schnug 1991). Probably the special weather - conditioned environment for crops in 1992 also have an influence. In some experiments in Sweden one has found good effects with supply of sulphur to oil seed plants especially when grown on light soils. However, today sulphur deficiency is not a common problem in Sweden. The results of two experimental programmes with sulphur fertilizer treatment to oilseed plants are presented.

## MATERIAL AND METHODS

In 1986-88 the University of Agricultural Sciences at Lanna and the Association of Skaraborgs Oilseed growers carried out experimental trials with sulphur to spring rape in the western part of Sweden. Different levels of micronized sulphur (Elosal 80% S) were sprayed at different stages of development. The experimental plan and the results of the fertilizer programme with sulphur are presented in Table 1. Only nine field trials were carried out and the material is therefore limited. In 1991 the Association of Swedish Seed and Oilseed growers initiated a number of field trials with sulphur to winter rape. The sulphur was supplied in early spring along with phosphatic fertilizer (P 9%, S 14%) up to a total of 50 and 100 kg S/ha respectively. As all the experimental plots had the same amount of phosphorus as the experimental treatment at 100 kg S/ha, the other treatments were supplied with the corresponding amounts of phosphatic fertilizer P 20, which contained about 2% S.

Table 1. Effect of micronized sulphur (Elosan) on oilseed yield and raw fat percentage in spring rape. Results of nine trials in 1986-88

S kg/ha	Spraying stages of development	Oilseed yield kg/ha resp. Rel. val.	Raw fat (1) percentage resp. Difference
0		1980	48.0
4	early budding	103	- 0.3
4	late budding	104	- 0.2
4	full bloom	101	+ 0.2
8	late budding	102	- 0.7
4 + 4	early + late budding	103	- 0.2
3 x 4	all stages	101	- 0.6

(1) Six trials 1986-87

Regardless of the fact that the experimental treatment without sulphur received about 5 kg S/ha, this treatment will be termed "without sulphur" ("0" kg S/ha). From 1992 another experimental treatment was conducted, in which spraying was carried out in early budding stage with micronized sulphur containing 80% elemental sulphur up to a total of 4.8 kg S/ha and a further 5 kg S/ha in phosphatic fertilizer. Three double low varieties of winter rape were sown in the trials, Ceres, Libraska and Casino. The trials were set up in the southern and northern parts of south Sweden (Göteborg) and were carried out on fields with soil types ranging from sandy soils to clay loam soils. During the most recent preceding years the fields were not treated with any fertilizers with a sulphur or animal manure content.

## RESULTS

The trials in 1986-88 with sulphur treatment have on average resulted in higher yields than those without sulphur treatment (Table 1). The best result was achieved with 4 kg S/ha in late

budding and with an increase in seed yield of 4%. This experimental treatment gave a higher yield than that in non sulphur treated in all field trials with the exception of one, in which both experimental treatments had equivalent results. The supply with sulphur gave a small decrease in the percentage of raw fat, however.

In Table 2 the results for yield of raw fat from a total of 10 trials conducted in 1992–93 are presented. No difference was found between the treatments with 50 and 100 kg S/ha respectively even though two trials from 1991 were included. On average for all trials, no effects of supply with sulphur were observed. However, a small difference was detected between varieties in that Libraska reacted with a slight increase in yield for sulphur with phosphatic fertilizer. In Table 3 the results from the trials are presented for each year. Supply of sulphur gave a higher yield especially for the varieties Ceres and Libraska in 1991 and 1992. There was no effect of sulphur sprayed in early budding. For the three varieties there was an overall higher yield with supplied sulphur in only a few trials (locations). This occurred for both locations in 1991 and two locations in 1992 for the 100 kg S/ha treatment and in the last year also for the treatment with sprayed sulphur. In two of the same locations – one of each year – even the treatment with 50 kg S/ha produced a higher yield for supplied sulphur.

Table 2. (Experiment no OS-189) Yield of raw fat in winter rape. Results of 10 trials in 1992–93

Variety		Sulphur supplied by phosphatic fertilizer			Micronized (1)
		"0" (2)	S kg/ha 50	100	sulphur 4.8 (2)
Ceres	kg/ha	1550	1480	1510	1480
	Rel. val	100	96	98	96
Libraska	kg/ha	1560	1590	1580	1530
	Rel. val	100	101	101	98
Casino	kg/ha	1480	1440	1430	1400
	Rel. val	100	97	96	95
Mean raw fat kg/ha		1530	1500	1510	1470
Rel. val		100	98	99	96

(1) Sprayed in early stage of budding, (2) Another 5 kg/ha to obtain equal of phosphorus in all treatments

Concerning the percentage of raw fat, on average no difference was found between the different experimental treatments. This is also the case for the content of chlorophyll.

Double low rape has a low content of glucosinolates and therefore it is very important to ascertain whether the application of sulphur gives a higher content of glucosinolates in the seed. The results are presented in Table 4. The content of glucosinolates is measured in micromoles and in Sweden the content in double low rape must not exceed 40 m mol/g fat free dry matter. On average the variety Libraska (20 m mol) had a somewhat higher content of glucosinolates than Ceres (18 m mol) and these two had a higher content than Casino (13 m mol). In the 12 trials the variations in content of glucosinolates in standard treatments were 6–25 m mol for Ceres and 14–32 m mol for Libraska. The supply of sulphur in phosphatic fertilizer had no effect of the glucosinolate content in Casino and only a small effect on the other two varieties, although Libraska seemed to react a little more than Ceres.

Table 3. Effect of sulphur fertilization on yield of raw fat in winter rape in different years

Year Variety	Number of trials	(1) S kg/ha			
		"0"	50	100	4.8
		Raw fat kg/ha = 100		Relative value	
<b>1991</b>	2				
Ceres		1270	102	106	-
Libraska		1230	100	108	-
Casino		1150	105	103	-
<b>1992</b>	5				
Ceres		1280	102	102	99
Libraska		1390	104	102	98
Casino		1250	102	99	98
<b>1993</b>	5				
Ceres		1810	92	95	94
Libraska		1740	99	101	98
Casino		1720	93	95	93

(1) See notes for application of sulphur in Table 2

Table 4. Effect of sulphur fertilization to winter rape on the content of glucosinolates measured in micromoles

Years Variety	Number of trials	(1) S kg/ha			
		"0"	50	100	4.8
<b>1992-93</b>	10	m.mol.		Difference	
Ceres		19	+ 2	+ 3	- 2
Libraska		21	+ 1	+ 4	- 3
Casino		15	- 1	- 1	- 2
<b>1991-93</b>	12				
Ceres		18	+ 1	+ 2	-
Libraska		20	+ 1	+ 4	-
Casino		13	0	0	-

(1) See notes for application of sulphur in Table 2

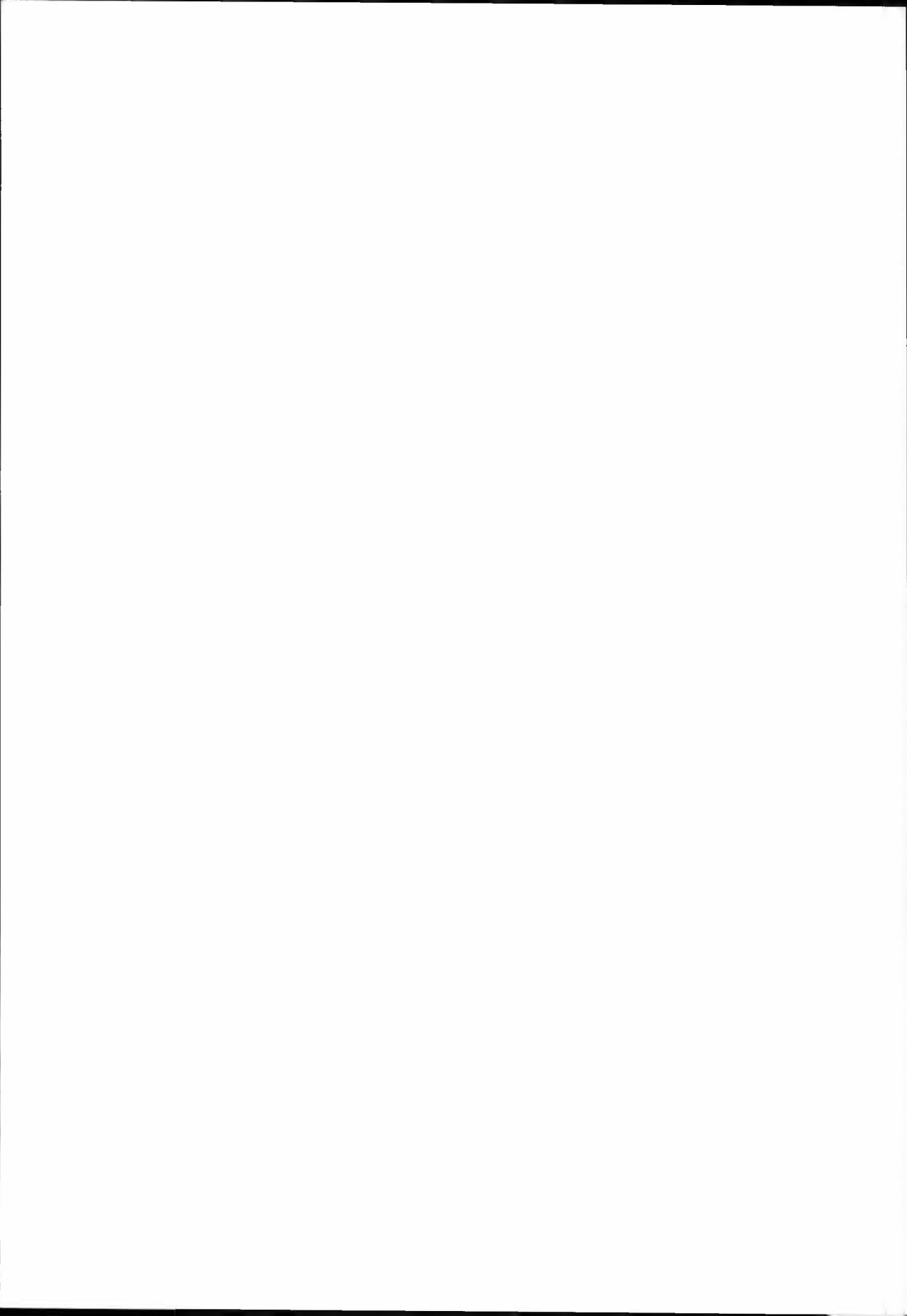
Sulphur sprayed in early budding did not influence the content of glucosinolates. The cultivation location and weather – conditioned environment for crops in a particular year appear to have more impact on the glucosinolate content than supply of sulphur, which has been demonstrated earlier by G. Olsson (1990).

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# The ecological importance of sulphur

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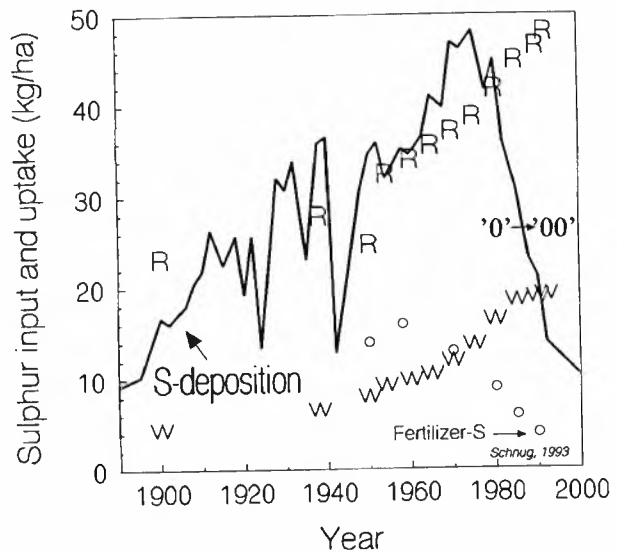
Severe sulphur deficiency in agricultural crops is a major nutrient disorder in northern Europe with strong ecological impacts. An insufficient sulphur supply will not only reduce yields, but also diminish quality of food and feedstuff. Moreover natural defence mechanisms against xenobiotics are linked with the secondary sulphur metabolism of plants which again is influenced by the sulphur nutritional status of the crop. Furthermore under conditions of sulphur deficiency utilization of nitrogen fertilised is reduced causing undesired nitrogen losses to the environment.

Key words: Foodstuff quality, nitrogen utilization, sulphur deficiency, xenobiotics.

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Since the 1980s the sulphur inputs to European soils have been reduced dramatically. The reasons for this are widespread: desulphurization of fumes from natural fuels, changes in fertilizer practices and last but not least the breakdown in major emission capacities in the former east (Schnug 1993a). Sulphur inputs are actually down to the level that existed before the beginning of the industrial revolution in central Europe (Fig. 1).

Fig. 1. Sulphur supply by atmospheric depositions and fertilizers and average sulphur uptake by winter oilseed rape (R) and winter wheat (W) in German agriculture (Schnug 1993a)



Since 1980 the average uptake of oilseed rape crops, and since 1990 the average uptake of wheat crops are no longer covered by atmospheric sulphur depositions (Fig. 1). During the same period sulphur concentrations in plants have been declining continuously (Fig. 2).

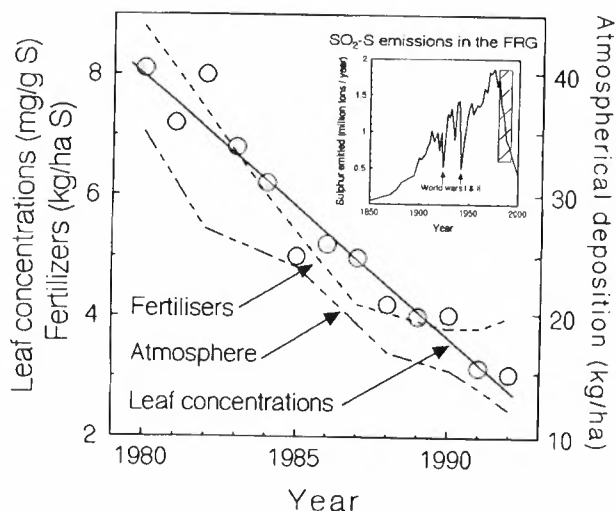


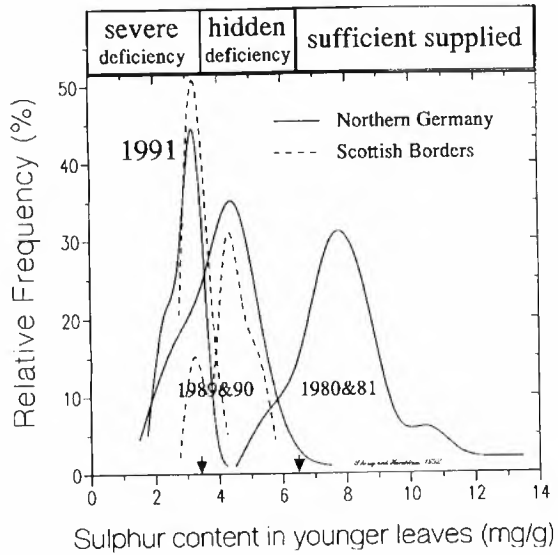
Fig. 2. Sulphur concentrations in younger, fully differentiated leaves of the upper third of sprouting oilseed rape plants in northern Germany, sulphur inputs to agricultural soils and sulphur emissions in Germany (total number of entries = 4273)(Schnug 1993a)

As a consequence, symptoms of severe sulphur deficiency in oilseed rape and wheat are now the most widespread symptoms of any nutrient deficiency in fieldgrown crops in the northern areas of Europe (Schnug 1993a; Schnug & Haneklaus 1994) and yield response to sulphur fertilization is becoming quite common. At the beginning of the 1980s less than a quarter of oilseed rape fields in northern Germany were estimated to respond to sulphur fertilization (Fig. 3; range of "hidden" deficiency) and in no case was the supply down to severe deficiency level. Towards the end of the decade there were only a few crops left in northern Europe which were sufficiently supplied for maximum yield, and at least one-third of the fields were expected to show symptoms of sulphur deficiency (Fig. 3; range of "severe deficiency"). In the beginning of the 1990s the supply decreased rapidly and now there is virtually no unfertilized oilseed rape crop without deficiency symptoms. This dramatic change is attributable to the closing down of many industrial facilities in the east after the German reunification in November 1989. The sulphur dioxide emission per person in the former German Democratic Republic (GDR) was 5 times higher than that in the Federal Republic and the GDR "exported" almost the same amount of sulphur dioxide to West Germany as the combined exports of the UK, France and the Benelux.

Bearing in mind the problems of surplus production in European agriculture, it might be thought that limited yields by reduced sulphur inputs to soils would be a most welcome side-effect of the clean air acts. However, the deterioration in the sulphur supply will have several serious consequences for agricultural as well as non-agricultural ecosystems, which will be discussed in this contribution.



Fig. 3. Relative frequency of total sulphur concentrations in younger, fully differentiated leaves of the upper third of sprouting oilseed rape plants in northern Germany and northern England during 1980-91 (total number of entries 3997) (Schnug 1993a)



### IMPACT OF SULPHUR SUPPLY ON AGRICULTURAL ECOSYSTEMS

The importance of the sulphur supply for agricultural ecosystems can be seen from different viewpoints: sulphur is a major element in biomass production in any crop and thus a shortage in sulphur supply not only decrease yield but also the efficiency of any other input and, with particular regard to energy crops, the amount of captured solar energy. An impressive example is the production of fuel by industrial crops. By fertilizing a severely deficient oilseed rape crop with sulphur an increase in the oil yield of more than five barrels per hectare can be expected under northern European growing conditions (see Fig. 4).

The sulphur status of the plant is positively correlated with nutritional (proteins, flavour and pharmaceutical compounds (Schnug 1988, 1990)) as well as technological parameters (colour, baking quality (Schnug 1990, 1993b)). Thus a generally decreasing sulphur supply will result in loss of nutritive and technological quality of food and feedstuff. This may cause special problems for growers of "organic" food who are particularly concerned about the high nutritional quality of their products.

Phytochelatin (sulphur-containing peptides) play a role in retaining heavy metals in plant roots, which protect the shoots from heavy metal stress (Grill et al. 1990). It seems, however, that the production of phytochelatin is not only governed by the concentration of a particular heavy metal (Grill et al. 1990) but is also dependent on the sulphur status of the plant, because heavy sulphur dressings can decrease the concentrations of, for instance, zinc and copper in the shoots and increase concentrations in the roots (Schnug 1982; Schnug & Schnier 1986). Therefore it is thought that as another consequence of decreasing sulphur loads to agricultural soils, the soil-plant transfer of heavy metals and thus the entrance of heavy metals into the nutritive chain might increase.

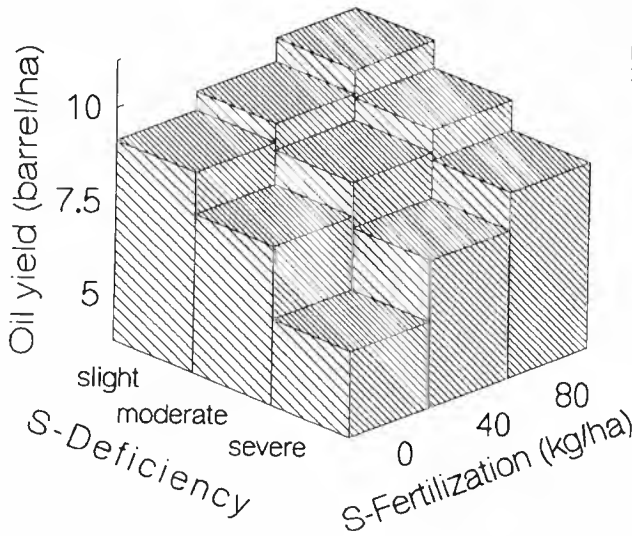
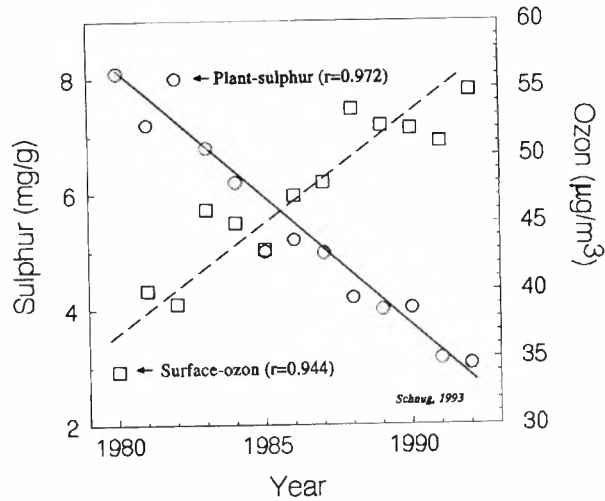


Fig. 4. Influence of sulphur supply on fuel production by oilseed rape (calculated from 3)

Sulphur-containing compounds play a major role in the plant defence mechanisms against biotic and abiotic stress. During sulphate reduction, gaseous losses, mainly in the form of  $H_2S$ , occur (Schröder & Rennenberg 1993). Although directly related to the sulphur status, the amounts lost by this pathway are very small. They do, however, have an important role in plant resistance to diseases, because  $H_2S$  is a very effective fungicide. Other important links between plant resistance and sulphur status are the biosynthesis of secondary plant products (e.g. glucosinolates) or cysteine-rich thionines, both with features of phytopathological interest (Schnug & Ceynowa 1990; Bohlmann 1993). Consequently, in sulphur-deficient environments soil with applied sulphur gives a significant reduction in fungal diseases in agricultural crops (Schnug & Ceynowa 1993; Haneklaus et al. 1994; WALKER 1994). Furthermore, an important consequence of a decreasing sulphur supply in agricultural soils may be increased pesticide inputs into agroecosystems due to a decrease in the natural resistance of plants to pests and diseases.

Among sources of abiotic stress for plants, xenobiotics and increasing surface ozone levels are of great importance. Plants are able to deal with these stress factors mainly by means of the glutathione metabolism (Lamoureux & Rusness 1993; De Kok & Stulen 1993), which again is strongly related to the sulphur supply of the plants (Rennenberg & Lamoureux 1990; Haneklaus et al. 1994a (submitted)). While the plant sulphur supply in northern Europe is continuously decreasing, environmental loads with xenobiotics are still high (Umweltbundesamt 1992) and surface ozone concentrations are steadily increasing (Fig. 5). Therefore it is anticipated that as another result of the changes in sulphur inputs plants will become more vulnerable to the stress factors mentioned above.

Fig. 5. Sulphur concentrations in younger, fully differentiated leaves of the upper third of sprouting oilseed rape plants and atmospheric surface ozone concentrations in northern Germany in 1980-92 (Schnug 1993a)



### IMPACT OF SULPHUR SUPPLY ON NON-AGRICULTURAL ECOSYSTEMS

The importance of sulphur for plant resistance is not just restricted to agricultural crops (s.a.). Abiotic stress caused by xenobiotics (e.g. herbicides) as well as attacks by diseases promoted by simplified rotations in agriculture are also tackled by sulphur-containing compounds in the primary and secondary metabolism of wild plants. Owing to the fact that, obviously, the higher sulphur inputs in the past century enabled plants to adapt to increasing environmental stress, the decline in supply within only one decade might have serious consequences for the stability of recent ecosystems. For instance, sulphur deficiency is thought to be one of the reasons why 50% of all forests are still suffering damage although sulphur emissions have been cut down drastically over the past 10 years (Bundesministerium für Ernährung 1993), because along with the sulphur supply to the trees their resistance has been reduced whilst environmental stress has increased.

Via the metabolism of amino acids nitrogen and sulphur utilisation is interdependent in that an efficient use of a high nitrogen level in agriculture requires a sufficient sulphur supply. Thus, increased ecological problems from agricultural plant production are expected because the utilization of fertilizer nitrogen is diminished in sulphur-deficient crops (Fig. 6). This again may result in increased losses of nitrogen to the environment especially by leaching of nitrate into the hydrosphere or gaseous losses to the atmosphere. On average, each kilogram of sulphur missing from the plant demand means that 15 kg nitrogen is likely to be lost to the environment. For northern European oilseed rape cropping, the actual annual rate of fertilizer nitrogen lost to the environment as a result of declining crop sulphur status is estimated to be 4000–6000 metric tons (Schnug 1993a).

Because of reductive processes, soils and plants are liable to release gaseous sulphur compounds such as  $H_2S$ , dimethylsulphides or mercaptanes into the atmosphere (Schröder & Rennenberg 1993). These losses concern not only living but also decomposing plant residues. With low glucosinolate oilseed rape varieties ("double lows") this was the first time that

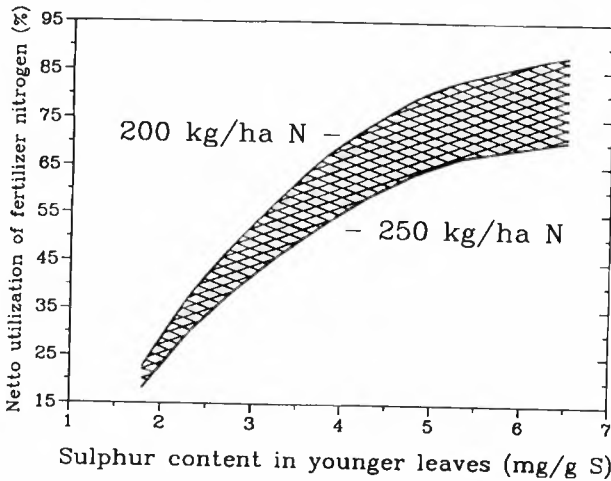


Fig. 6. Utilization of fertilizer nitrogen by oilseed rape crop as influenced by sulphur fertilization (Schnug et al. 1994)

breeds with a genetically modified sulphur metabolism had been released into the environment (Schnug & Haneklaus 1993). Among others, a special characteristic of this type of crop is the higher amounts of sulphur remaining with the residues in the fields. In Germany, the difference from the former varieties amounts to approximately 20 billion kg sulphur (Schnug 1993a), which is bound in easy decomposing low molecular compounds. Gaseous sulphur compounds released from this system significantly contribute to the balance of atmospheric trace gases but are also suspected of interfering with human and animal health (Schnug 1993a).

By means of  $H_2S$  during sulphate reduction and via the glutathione metabolism plants obviously contribute to the degradation of atmospheric oxidants. Thus, a reduction in sulphur inputs to the environment and following up reduced turnover rates in the processes mentioned above in plants in agricultural as well as in non-agricultural ecosystems, could be a new approach toward partly explaining recent increases in surface ozone concentrations.

## CONCLUSIONS

The ecological consequences of a tremendous reduction in the atmospheric sulphur inputs in the past decade are not just restricted to agriculture but will have significant effects on non-agricultural ecosystems too. This is a good example of a poor prediction of the impact of a technological change on non-target ecosystems. The investigation of the impact of global changes in the anthropogenic sulphur cycle on agricultural as well as non-agricultural ecosystems and interference between both types of ecosystems under the strain of sulphur fertilization is an imperative in future research in plant nutrition and soil science.

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## GROUP DISCUSSIONS AND RECOMMENDATIONS

### Compiled by Bal Ram Singh

The main subject of the seminar was divided into three main topics, viz. Sulphur in soils and soil test methods, crop requirements for sulphur and product quality, and the atmospheric deposition of sulphur and environmental risks. Accordingly three groups were formed. These working groups at the start were given some discussion points in order to facilitate their working and were requested to come out with suggestions or recommendations for further work.

### WORKING GROUP I

#### Sulphur in soils and soil test methods

**Coordinator:** Into Saarela

**Rapporteur:** Jørgen Eriksen

#### Discussion points

1. *What are the ongoing, well designed sulphur studies in soils?*

Institution(s):

Objectives:

Date initiated:

2. *What are gaps in our knowledge, identify and prioritize needs to initiate new experiments?*

Institution(s):

Objectives:

3. *Which of the soil test procedures can be employed?*

4. *What are the follow-up needs?*

5. *Is there a scope to initiate joint Nordic projects and how are possibilities of financial support?*

6. *Other points of relevance*

## **Recommendations and suggestions**

After discussion, the group came out with the following suggestions and recommendations.

1. There is a lot of scattered experimental work done in the Nordic countries and all these small observational experiments need to be gathered. This exercise may
  - provide valuable information,
  - make sulphur problems more visible, which is necessary to convince financial supporters of the importance of research,
  - create a platform for further studies.
2. Because of low retention capacity of soils in the Nordic countries, sulphur management in these soils is bit difficult. Basic soil profile S information( e.g.sorption capacity,organic S,inorganic S,HI-S) is needed for a range of "benchmark soils", making comparison of soils under different climatic conditions possible.
3. For organic S and farm yard manure, knowledge is required about composition,turnover,and plant-availability of S-compounds( including analytical methods).
4. The potential of some universal soil tests(e.g. MCP, KCl- 40, H<sub>2</sub>O, AAAC, AAAC-EDTA) and depth of sampling needs to be investigated for nordic countries. It is preferable, but not likely that the same soil test will apply to both organic and inorganic dominated elements. Plant analysis is a valuable tool for estimating soil S status and ideally this should be correlated with soil S tests.
5. There is a good scope to initiate joint Nordic work and the financial support needs to be solicited for such joint projects.

## **WORKING GROUP II**

### **Crop requirements for sulphur and product quality**

**Coordinator:** Gyula Simán

**Rapporteur:** Bjørn Tore Svoldal

### **Discussion points**



*1. What are the ongoing, well designed field experiments?*

Institution(s):

Objectives:

Date initiated:

*2. What are gaps in our knowledge, identify and prioritize needs to initiate new experiments?*

Institution(s):

Objectives:

*3. Which of the crops are at risk under different soil and climatic conditions and under different management and fertilization practices?*

*4. Which aspects of crop quality should be prioritized?*

*5. What are the follow-up needs?*

*6. Is there a scope to initiate joint Nordic projects and how are possibilities of financial support?*

*7. Other points of relevance*

### **Recommendations and suggestions**

This group followed the above mentioned points separately and gave their recommendations accordingly.

1. A number of field experiments being conducted for different crops in the Nordic countries were listed which may provide valuable information on S needs of these crops under different soil, climate and management conditions.
2. In addition to recording of crop yields, the need to know more about the following aspects was felt.
  - soil analysis for S fractions
  - plant analysis for S and amino acids
  - following up historical field trials
  - S-turnover in soil
  - modelling the leaching and movement of S in soil
  - uptake in different oil seed and grass crops
  - plant protection effect
  - seasonal variation in S nutrition of crops

3. All crops of brassica family, legumes, and well fertilized grass crops in light soils are at risk. Wet climate in early spring (April) often result in S deficiency. There is a need to know how much S should be applied under such conditions.
4. Amino acid composition and baking quality of spring wheat are important aspects of crop quality. For oil seed rape, glycosinolate content is important.
5. Diagnostic methods on soil and plant analysis need to be refined. The Australian model should be validated under Nordic conditions.
6. It is possible to get support from "Nordiska Ministerrådet" for some of the projects mentioned below:
  - Diagnostic methods- soils and plants
  - Product quality/ baking quality
  - Animal slurry-S-losses
  - S status of plants at different times (growth stages)
  - use of gypsum in field trials

### WORKING GROUP III

Atmospheric deposition of sulphur and environmental risks

**Coordinator:** G. Abrahamsen

**Rapporteur:** Håken Jansson

#### **Discussion points**

1. *What are the ongoing, well designed experiments on impact of sulphur deposition on terrestrial ecosystems?*

Institution(s):

Objectives:

Date initiated:

2. *What are gaps in our knowledge, identify and prioritize needs to initiate new experiments?*

Institution(s):

Objectives:

3. *Do we need to intensify this type of work? What are the follow-up needs?*

4. *Is there a scope to initiate joint Nordic projects and how are possibilities of financial support?*
5. *Other points of relevance*

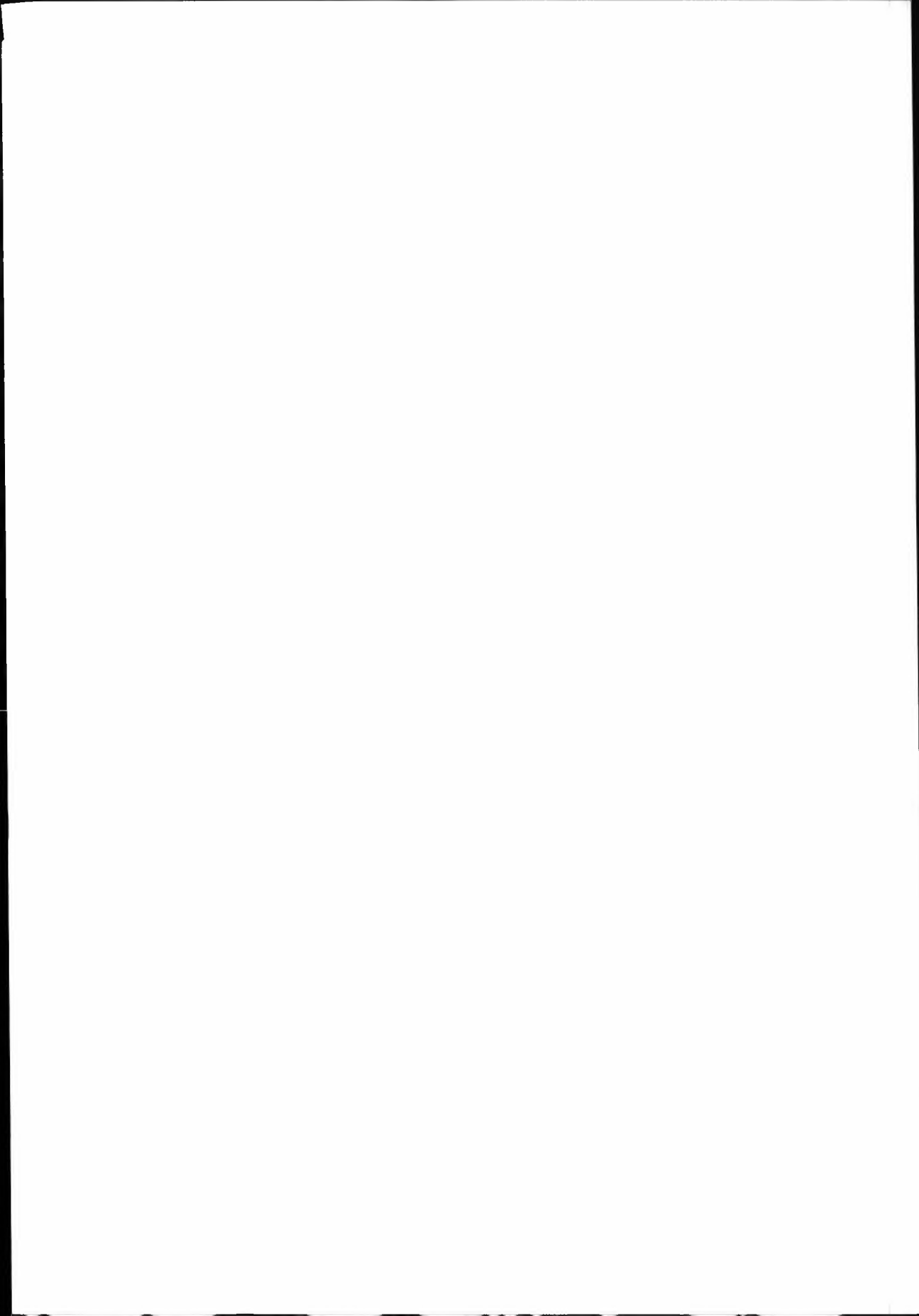
### **Recommendations and suggestions**

Based on these points the group presented the following recommendations.

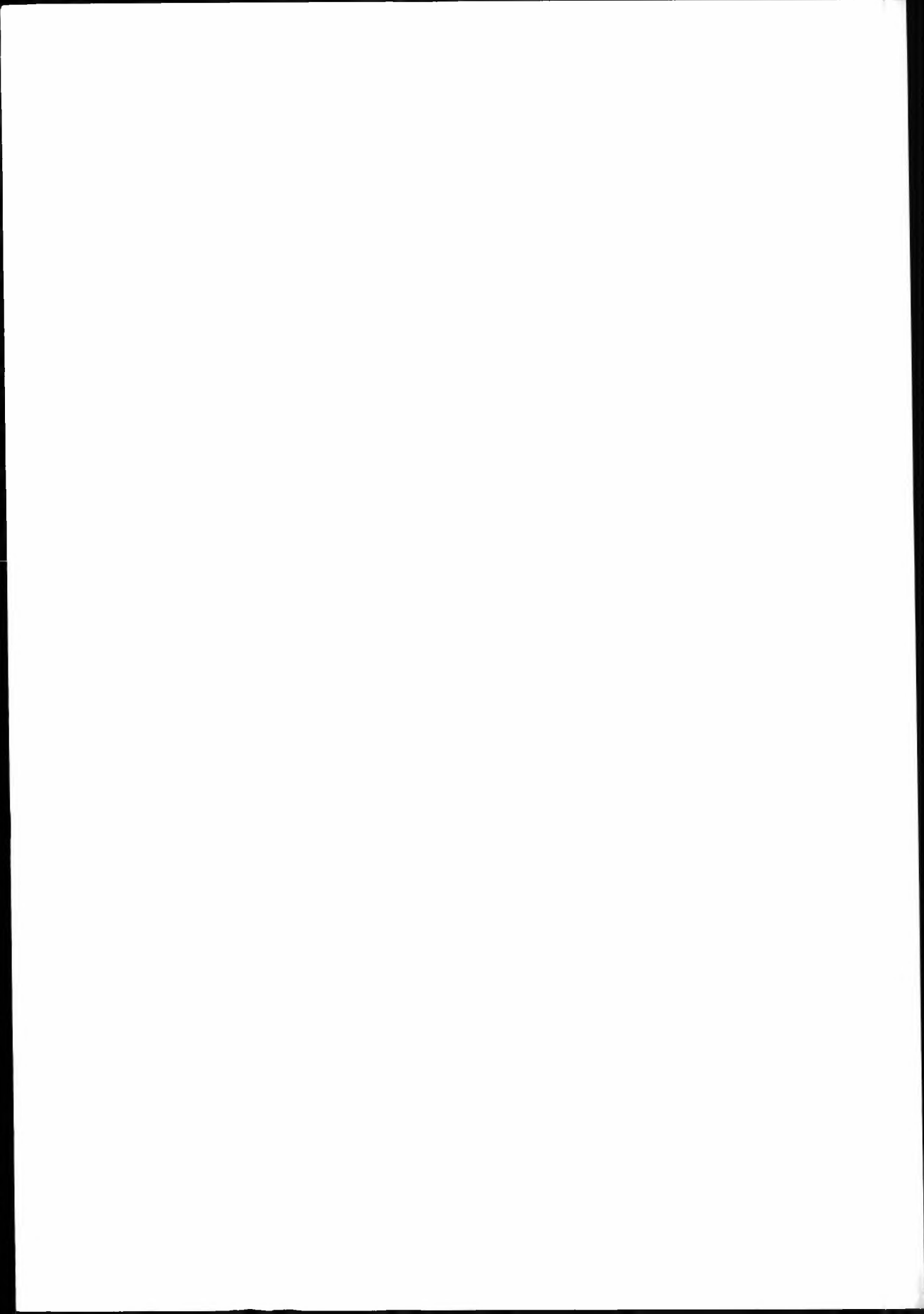
1. There are a number of well designed experiments on this aspect in Sweden.
2. Understanding of the following is required
  - weakening of the barrier against uptake of heavy metals in S deficient plants,
  - volatile S compounds,
  - weathering rates of soils,
  - reduced lime applications
  - (sulphur deficiency in forestry).
3. There is a need to follow up the uptake of heavy metals in S deficient crops. The consequences of the big drop in S deposition and change in deposition N:S ratio need to be taken seriously.
4. There is a need to initiate Nordic projects through support from Nordic countries or through EEC. Involvement of other countries may be solicited.

### **General observations and recommendations**

While summarizing the deliberation of the two days seminar, it was stressed that we have a number of gaps in our knowledge with regards to sulphur nutrition of crops, sulphur reactions in soils, soil processes controlling the supply of S to plants, and product quality. Some of the recommendations made by the three groups, mentioned in the preceding section, were emphasized again and the need to intensify sulphur research was widely felt. Information presented during the seminar may provide baseline data to plan and initiate new research projects. Soil and plant analysis methods were the subjects of an extensive discussion and a closer cooperation between plant and soil scientists was felt necessary to tackle this problem. It was recommended that new joint projects on soil-plant relationships of sulphur in Nordic countries and whenever possible, in cooperation with other countries, especially with Germany and Scotland where extensive sulphur research programs are in progress, should be initiated.







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