

HUMOR

HUMEX

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HUMIC LAKE  
ACIDIFICATION  
EXPERIMENT

Status September 1990,  
one week before start  
of the treatment

Report NO 1/91  
from  
HUMEX

# HUMOR

Humic substances, modifiers of the response  
of aquatic of ecosystems to acidification

## Sponsors

- Commission of European Communities (Directorate General XII, Science, Research Development.
- Various national institutions in Finland, Germany The Netherlands, Norway, Spain, and United Kingdom.

## Background and goal

It is now firmly established that in many parts of Europe acidification of surface waters is proceeding due to atmospheric deposition of sulphur and nitrogen compounds. Despite a considerable research effort aimed at understanding the impact of surface water acidification on aquatic biota, many of the published results have been inconsistent or even contradictory.

There are strong reasons to believe that part of this inconsistency is attributable to the varying amounts and functional properties of brown dissolved organic matter (humic substances, HS) in surface waters undergoing acidification. Evidence is becoming available that HS can;

- contribute directly to acidification
- interact with other dissolved materials
- influence microbial enzyme processes
- directly influence aquatic biota.

The purpose of the "HUMOR" project is to strengthen the scientific knowledge of humic substances influencing the acidification of surface waters and its reversibility, and altering the impact of acidification on aquatic organisms.

The work is carried out in both lakes and rivers in Finland, The Netherlands, Norway, Spain, Sweden, United Kingdom and West Germany by 7 contractors. The main body of the research will be carried out in the home country of each participant with a cooperative effort by all members at the HUMEX lake in western Norway. Each participant provides one part of an overall holistic approach which can be listed in hierarchical order;

- (1) chemical characterization including lipid and fatty acid composition of acidified and naturally acidic humic lakes,
- (2) effect of pH and HS on iron and phosphorus complex formation and nutrient availability,
- (3) effect of pH and HS on microbial enzymatic processes,
- (4) effect of pH and HS on lipid solubility and invertebrate membrane transport,
- (5) effect of artificial acidification on geochemistry and biological processes in an experimental watershed in western Norway.

## Participants

- Finland: University of Helsinki. Lammi Biological Station, Lammi. Responsible: Dr. Kalevi Salonen.
- Germany: Max-Planck-Institute fur Limnologie, Abt. Mikrobiologie, Plön. Responsible: Dr. Uwe Munster.
- The Netherlands: Limnological Institute, Tjeukemeer Lab., Oosterzee. Responsible: Dr. Henk De Haan.
- Norway: Norwegian Institute for Water Research, Oslo. Responsible: Dr. Egil Gjessing.
- Spain: Consejo Superior de Investigaciones Cientificas, Barcelona. Responsible: Dr. Juan Grimalt.
- Sweden: University of Lund, Limnology Institute, Lund. Responsible: Dr. Robert C. Petersen Jr.
- United Kingdom: University of Lancaster. Department of Biological Sciences, Lancaster. Responsible: Dr. Roger I. Jones.



# NIVA - REPORT

Norwegian Institute for Water Research



NIVA

**Main Office**  
P.O.Box 33, Blindern  
N-0313 Oslo 3  
Norway  
Phone (47 2) 23 52 80  
Telefax (47 2) 39 41 29

**Regional Office, Sørlandet**  
Grooseveien 36  
N-4890 Grimstad  
Norway  
Phone (47 41) 43 033  
Telefax (47 41) 42 709

**Regional Office, Østlandet**  
Rute 866  
N-2312 Ottestad  
Norway  
Phone (47 65) 76 752

**Regional Office, Vestlandet**  
Breiviken 5  
N-5035 Bergen - Sandviken  
Norway  
Phone (47 5) 95 17 00  
Telefax (47 5) 25 78 90

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Abstract:
<p>An important conclusion from the acid rain research during the late 1970's and the early 1980's, was that humic substances (HS) in the water affected the response of acid rain. HS acts as a modifier on both the chemical composition and on the biological activity of acidified surface water. The Humex project will, by artificial acidification of a whole catchment, study the role of HS on the acidification and the role of acidification on the biological properties of HS. In fall 1988, a dystrophic lake was divided in two halves by a plastic curtain, from the middle of the natural outlet to the opposite side. During the following two years, through September 1990, the water chemistry of the two lake halves has been monitored and a number of scientists from Europe and North America have been studying the organic matter and the biota in the water, and in the catchment area prior to the artificial acidification, which started October 6th 1990.</p>

4 keywords, Norwegian

1. Humus
2. Forsuring
3. Nedbørfelt manipulasjon
4. Status

4 keywords, English

1. Humic Substances
2. Acidification
3. Catchment Manipulation
4. Status

Project leader

*Egil Gjessing*  
Egil Gjessing

For the Administration

*Bjørn Olav Rosseland*  
Bjørn Olav Rosseland

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## INTRODUCTION

The lake Skjervatjern was divided into two halves already during fall 1988, initiated and sponsored by Norwegian Institute of Water Research. The lake was divided by a plastic curtain from the middle of the natural outlet to the opposite shore side. The outflowing water from these two outlets has been monitored by weekly water samples, and analyzed for major cat- and anions. During 1989 the planning and preparation for gaging the water flow from the two outlets took place, and in 1990 necessary funds were available from The Ministry of Environment and from NIVA, to start preparing for the artificial acidification of the one lake half and the corresponding catchment.

As a base for future studies, the participating scientists have been collecting as much information as possible about differences and similarities between the half to be acidified and the control catchment, during this pre-acidification period.

As this project is the first of its kind, and as expensive installations are necessary, we have emphasised on information about the project and invited scientists to participate with their expertise. There are a number of unanswered questions of ecological importance concerned with acidification of surface water containing humic substances. The HUMEX project is dealing with an environmental problem that needs an interdisciplinary approach.

In connection with the invitation from EC to participate in the STEP program, we contacted colleagues in England, Finland, Germany, Spain, Sweden and The Netherlands and suggested a joint application to Brussels. Today there are all together 10 institutions connected to the HUMEX activities and there are reasons to believe that additional scientists will join in the near future.

## THE SITE

Skjervatjern is situated on the west coast of Norway, about 10 km east of Førde. In this part of Norway the deposition of acid rain is low. Mean concentration of major ions in precipitation and wet deposition at Nausta is given in Table 1. The sampling station Nausta is situated only 20 km north-west of the Humex site.

Table 1 Weight yearly mean concentrations of ions in precipitation and the total yearly wet deposition at Nausta, about 20 km north-west of Skjervatjern.

Mean concentration (mg/l)						Year	Year	Wet deposition			
Year	SO <sub>4</sub> -S	NO <sub>3</sub> -N	NH <sub>4</sub> -N	Ca	Mg	mean pH	precip. mm	SO <sub>4</sub> -S	NO <sub>3</sub> -N	NH <sub>4</sub> -N	H <sup>+</sup>
								mg/m <sup>2</sup>	mg/m <sup>2</sup>	mg/m <sup>2</sup>	meq/m <sup>2</sup>
1985	0.29	0.13	0.09	0.09	0.12	4,70	1943	561	246	177	39
1986	0.27	0.10	0.08	0.09	0.16	4,74	2314	614	227	176	42
1987	0.27	0.12	0.11	0.09	0.11	4,72	1969	523	236	213	37
1988	0.21	0.13	0.09	0.14	0.23	4,68	2253	476	302	193	47
1989	0.21	0.12	0.07	0.10	0.23	4,80	3330	708	407	227	53

### Lake Skjervatjern- morphology

Fig.1 illustrates the depth profile and the surface area/volume relationship of the two basins. Some physical and morphological data are given in Table 2.

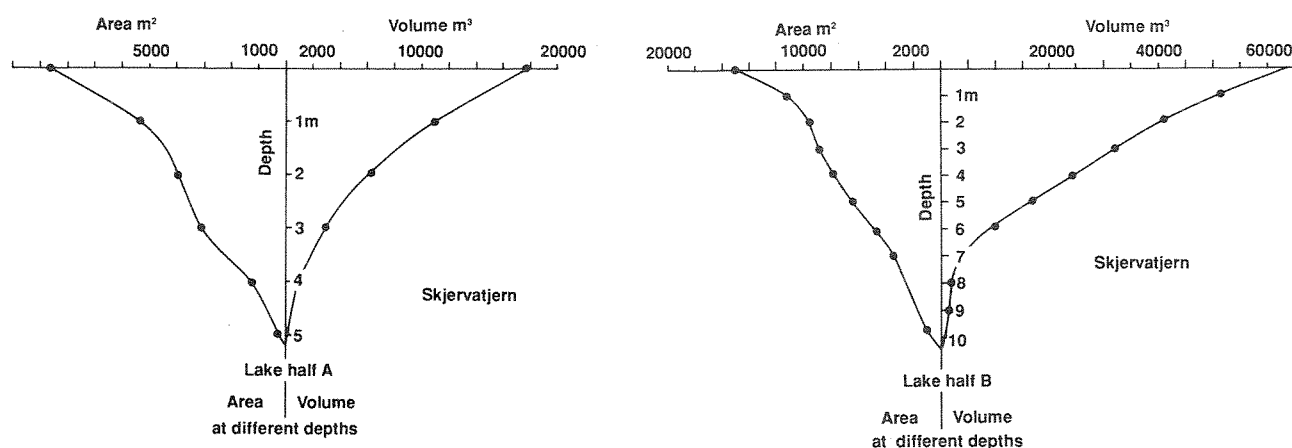


Fig. 1. Area and volumes at different depths in the two lake halves.

Table 2. Lake Skjervatjern; physical and morphological data.

	Experiment. half (A)	% of total	Refer half (B)	% of total
Lake area (ha)	0.9	38	1.5	62
Catchment (ha)	2.5	19	10.9	81
Maximum depth (m)	5.2	-	10.4	-
Mean depth (m)	2.0		4.2	
Volume (m <sup>3</sup> )	17800.0	22	63109.0	78
Theoretical retention time;	0.4	-	0.3	-

## TECHNICAL INSTALLATIONS DURING 1990

### The two lake halves

The lake was divided into two halves in November 1988 by a plastic curtain from the middle of the natural outlet to the opposite side. The length of this crossing is 105 m and maximum depth 4.5 m. The profile of the curtain was adjusted more or less according to the profile of the cross section, however, 3 to 4 m wider. This extra plastic curtain was pressed into the sediments by large numbers of sand bags. As the depth of the "soft" sediments generally are 0.5 - 1 m, these bags will secure a good physical separation of the water-masses. The position of the dividing curtain is shown on Figure 2.



Fig.2. A plastic curtain divides the Lake into two separate basins

### **Facilities for biological studies downstream the lake**

The project will as much as possible relate changes in chemical composition of the water from the two basins with change in biological activity. As will be seen from Figure 3, the two "artificial" outlets are piped into two separate tanks from where experimental water can be led into the field station which is shown on Figure 4. This is planned to house set-ups for fish experiments. The water-flows from the two lake halves are recorded. The device for flow-measurement is installed outside on the tubes (see Figure 5). This is based on acoustic measurement of water flow.

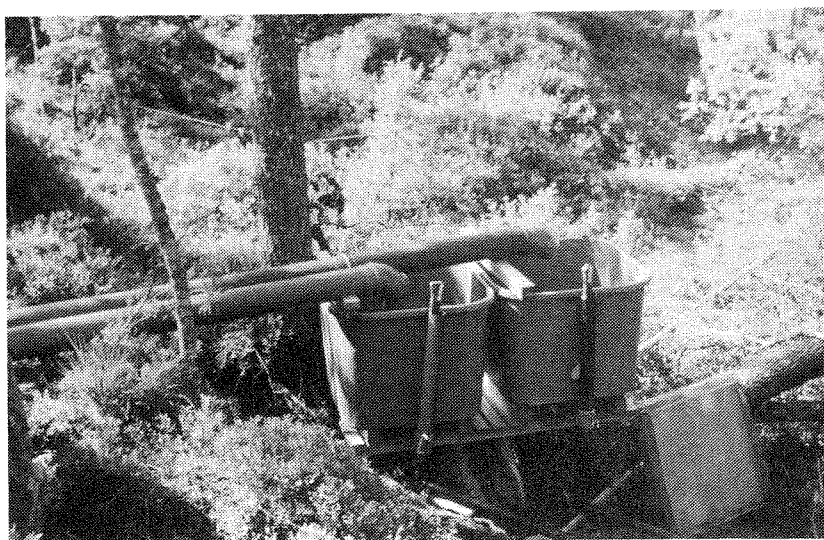


Fig.3. The outlets are piped into two separate "streams"

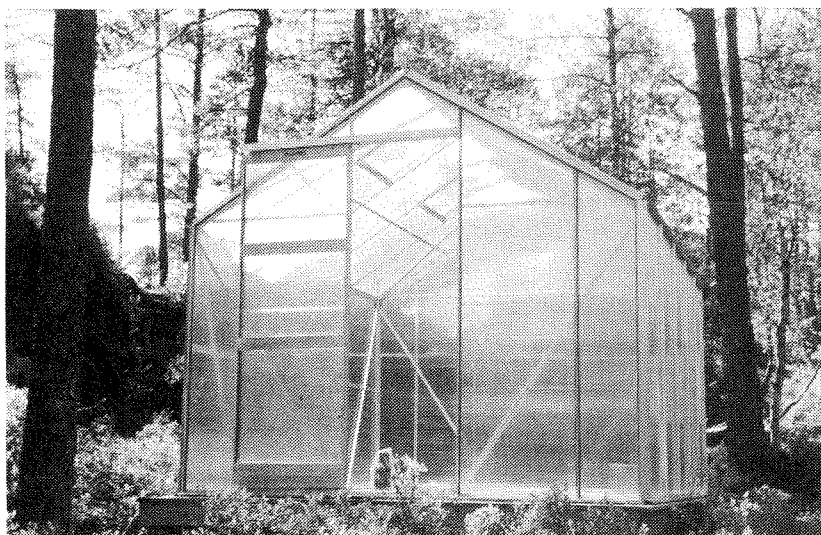


Fig. 4. The field station for biological experiments

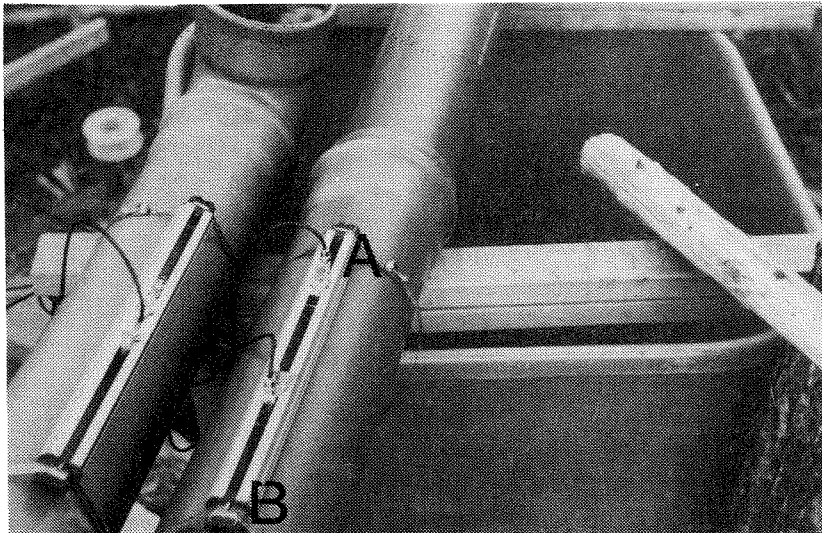


Fig.5. Devices for recording water flow from the two outlets. The pipe must be filled with water. An acoustic signal, sent down into the flowing water at B, is received at A after a certain time  $t_1$ . Similarly a signal sent from A, is received at B, after a time  $t_2$ . Depending on the flow rate,  $t_1 - t_2$  will differ, and this time-difference is proportional to the water flow.

### Sprinkler system

The artificial acidification is carried out by a sprinkling system that is mounted at the top of the highest trees in the catchment. Each of the sprinklers (Fig. 6) has a distribution radius of 15 m. In order to be able to acidify the 2.5 hektar experimental area properly, there is a need for 50 of these sprinklers. For the lake half itself, which is 0.9 ha, two bigger sized sprinklers are used, placed at the shore side. The sprinkling system will be activated manually when a certain amount of precipitation has been accumulated. The amount of artificial acid rain will be 5 to 10 percent of this natural precipitation, with a pH of 3.0. Some ammonium nitrate is also added to this acidified precipitations ( $0.8 \text{ mg NH}_4\text{NO}_3 / \text{mg SO}_4$ ).





Fig.6. Fifty sprinklers are mounted at the top of the highest trees in the area to be acidified.

The annual precipitation in this region is in the range of 2000 mm. This means that about 5000 m<sup>3</sup> of acidified water is to be sprinkled out during a year (see Table 3). The water used for the acidification is taken from Åsvatn, which is a part of the Jølster watercourse. Some chemical characteristics of this water are shown in Table 4.

Table 3. Precipitation at Nausta (mm).

	Jan	Feb	Mars	April	May	June	July	Aug	Sept	Oct	Nov	Dec	Year	% of Norm
1985	122	137	130	75	41	55	106	209	289	261	197	302	1943	
1986	100	47	289	9	210	35	66	81	230	345	587	316	2314	112
1987	119	284	69	93	59	89	139	156	386	199	106	269	1969	88
1988	189	149	45	173	71	21	200	163	337	183	271	451	2253	111
1989	788	398	341	20	287	208	116	274	275	263	145	218	3330	151

Table 4. Chemical composition of Åsvatn during an study in 1972 - 73. (Mean of 13 samples).

pH	Cond mS/m	Turb NTU	TOT-N mg N/l	COD Perm mg O/l	Ca mg/l
5.76	1.63	0.49	146	1.46	0.78

The water is pumped from a point 3-4 m depth, 50 m from the shore of Åsvatn. After addition of acid, the water is pumped through a distribution device (see Fig. 7 ), to the different sub-areas. The artificial acidification system is operating as follows: Depending on the amount of natural precipitation, the time needed to distribute 10 percent of this amount of acidified water, is read from a table. When activating the electronic device (see Figure 10) "pure" lakewater is pumped to sub-area No I, corresponding to 1.5 mm precipitation. When vegetation and soil are soaked with this water, x mm of acidified water at pH 3 (containing  $\text{NH}_4 \text{NO}_3$ ) are added. With for example 50 mm precipitation accumulated, 5 mm of acidified water are added. Before the electronic device allows to switch to sub-area No II, 1.5 mm of "pure" lakewater is added in order to rinse off the acid water from the vegetation.

The time needed to distribute 1.5 mm artificial rain, is 25 minutes; consequently the time needed to distribute 5 mm acid of pH 3, is 108 minutes for each of the 4 sub-areas. Regarding the fifth sub-area, which is the lake-half, the initial "pure"-water addition, is omitted. During winter there are freezing temperatures in this part of the country, and some of the precipitation will be in the form of snow. As the climate changes rapidly during the winter season, with mild periods between frost, we expect technical problems regarding proper acidification. Normally there will be some snow accumulated during winter; in these cases sufficient amount of acid will be added manually (see Figure 8).

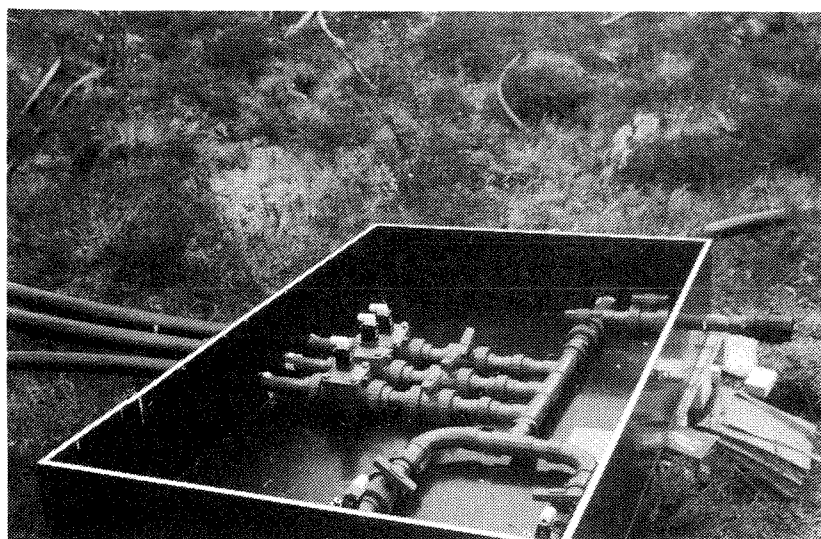


Fig.7. The water is pumped to a set of electronically operated magnetic valves, and distributed to the different sub-areas.



Fig.8. Manual acidification of a snow covered catchment.

### **Field laboratory**

Presently there are about two dozens scientists involved in the project, and we expect that there will be extensive fieldwork, particularly during the period of May through September. For practical reasons, therefore, we have put up two barracks for laboratory work and with cooking and sleeping facilities. The barracks were transported to the lot by helicopter (see Figure 9). Between these barracks the water pump, the acidification device and other electronic devices are installed (Figure 10).



Fig. 9. The barracks were transported from a nearby road by helicopter

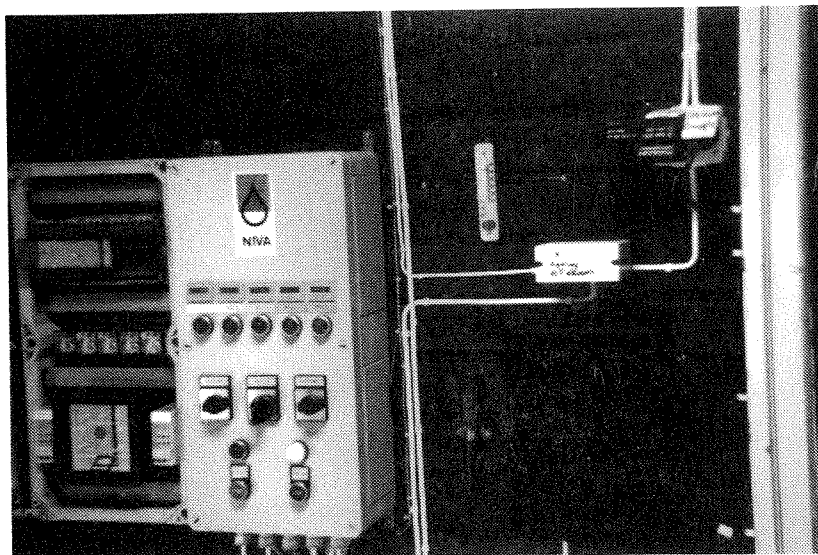


Fig.10. The operating panel for the water acidification and for the sprinkling system.

## **PREACIDIFICATION STUDIES**

### Hydrological studies

In the summer of 1990 the spatial pattern of subsurface water flow into each side of Skjervatjern was mapped, and samples for chemical analyses were taken at the land/water interface. Two different source areas and types of discrete source entry points were identified and found to supply water of different chemistry to the lake. One source is from the ombrotrophic bogs and the other from the shallow podzolic soil areas. The effect of catchment acidification on these two different sources will be followed during the project (Lena Petersen, Lund).

### Chemical studies

During the period from November 1988 , when the lake Skjervatjern was divided, till early October 1990, when the acidification part of the experimental part started, water samples have been collected weekly from the outlets. In addition, 8 series of

water samples have been taken from various depths in the two basins. Figure 11 illustrates the variation of pH. Figures 12 and 13 illustrates the corresponding variation of sulphate( $\text{SO}_4$ ), UV-absorption and colour. Figure 14 illustrates oxygen and temperature in vertical profiles in the two lake halves during 3 different seasons. All chemical data are given in Appendix 1.

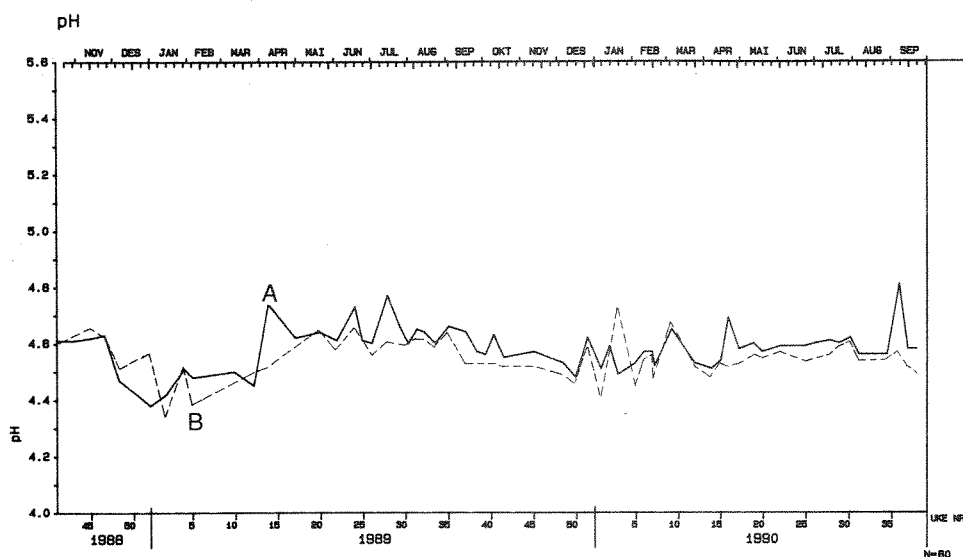


Fig.11. pH as a function of time in the outflowing water from the two basins.

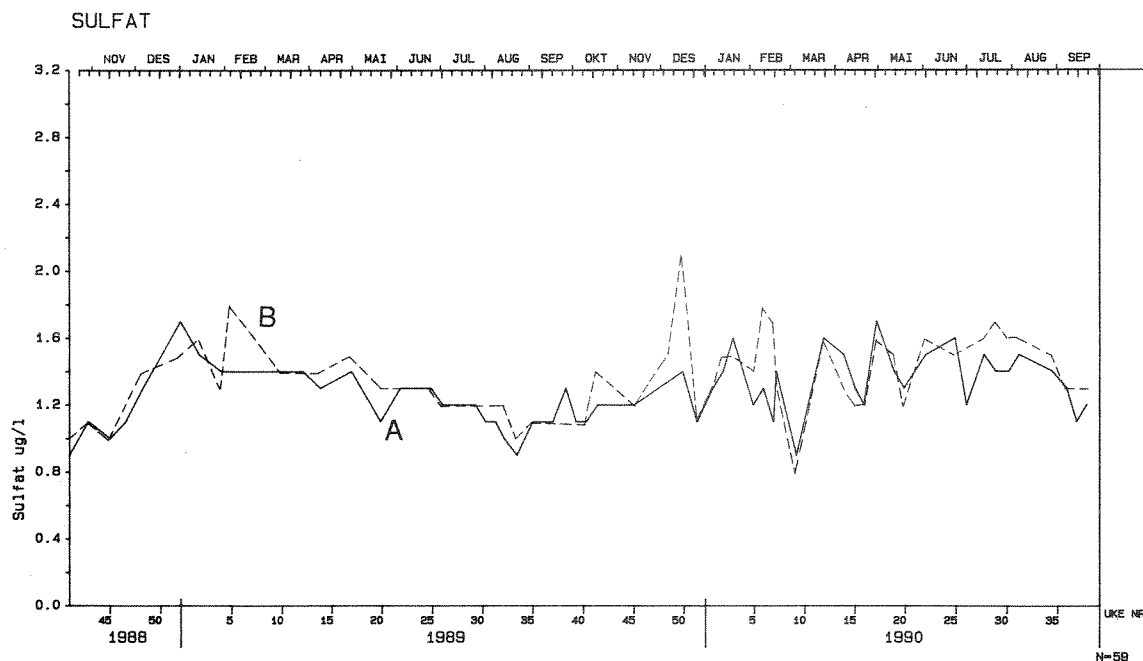


Fig.12. Change of sulphate concentration with time at two outlets.

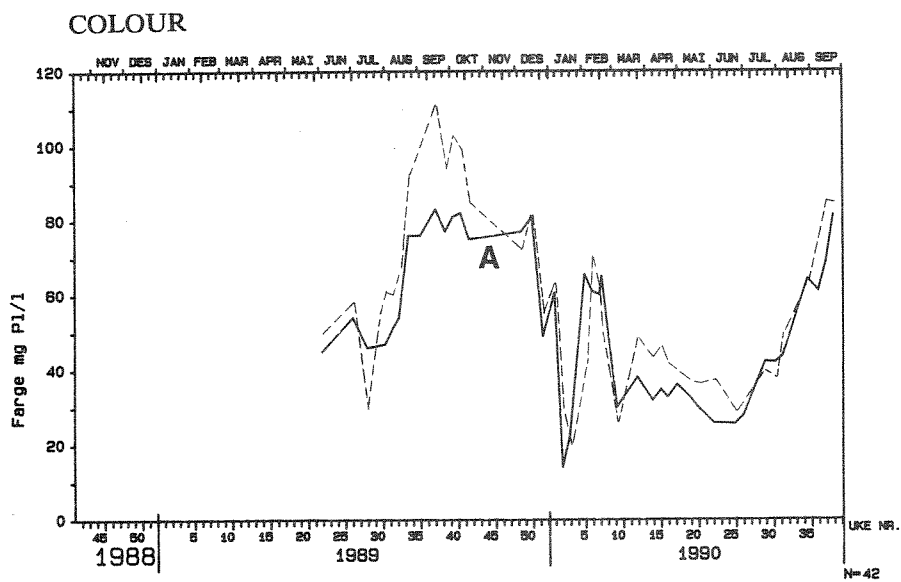
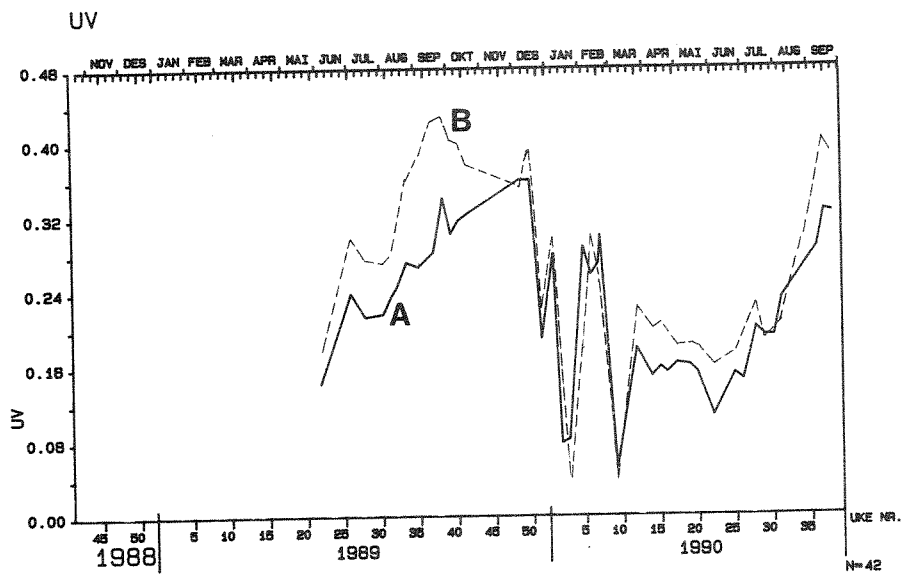


Fig. 13. Variation in the absorption of UV (254 nm./1 cm) and in colour with time in the water from the two outlets.

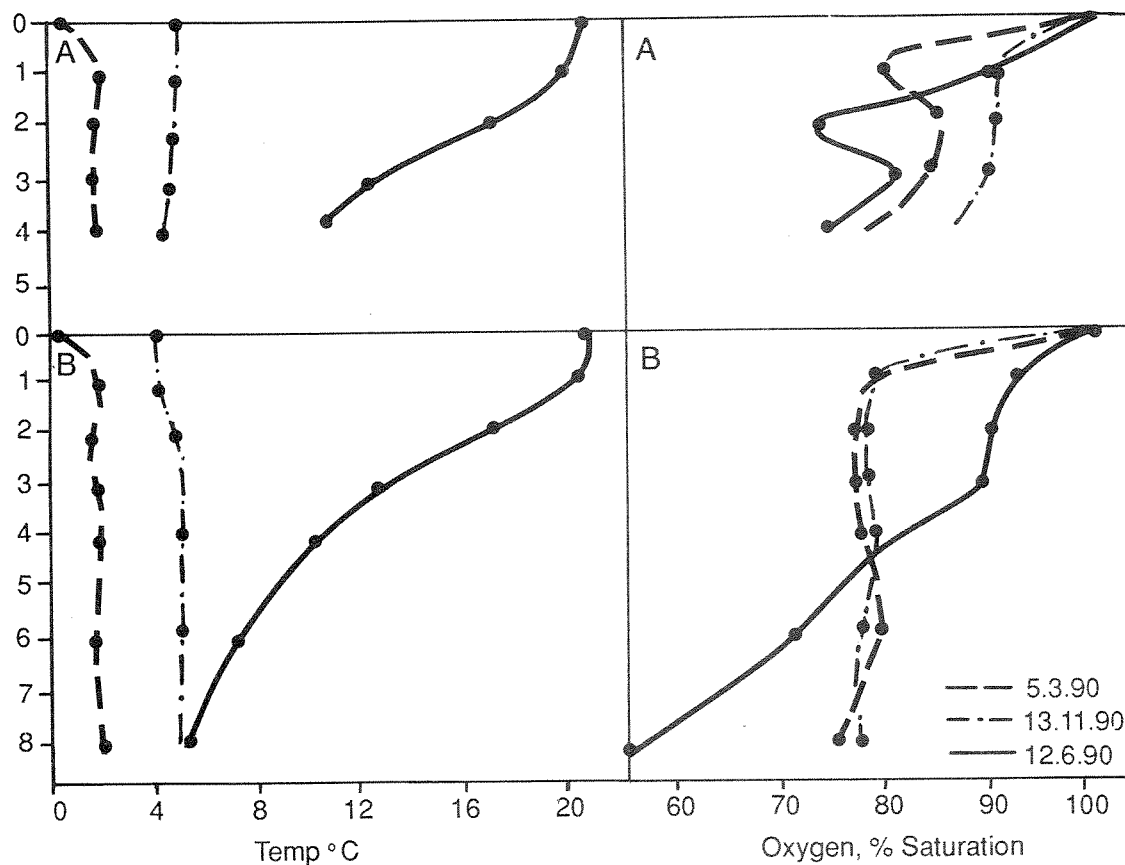


Fig.14. Temperature and % O<sub>2</sub>-saturation in the two basins. Changes with depth, during winter, summer and fall.

Table 5 gives the mean concentration of all chemical characterization data that have been collected prior to acidification, and Table 4 shows the Secchi-depth in the two basins at seven different dates during this period.

Table. 4. Secchi-depth in Lake Skjervatjern prior to acidification.

Date	A (m)	B (m)
18/6-89	2,2	2,3
13/7-89	2,8	2,8
06/5-90	2,3	2,5
12/6-90	2,2	2,2
08/7-90	2,6	2,6
21/8-90	2,6	2,6
30/9-90	2,3	2,3
Mean	2,43	2,47

Table 5. Composition of water from Lake Skjervatjern's basin A (to be acidified), and basin B. Mean values from the period 1/1-1989 to 30/9-1990. Vertical profile data are included.

	A 1)				B 1)			
	Mean Value	St. Div.	Min	Max	Mean Value	St. Div.	Min.	Max.
pH	4.60	0.08	4.38	4.81	4.55	0.07	4.34	4.76
Cond mS/m	3.15	0.48	2.10	4.66	3.33	0.40	2.15	4.44
Ca	0.20	0.03	0.10	0.33	0.21	0.03	0.11	0.33
Mg	0.30	0.07	0.16	0.63	0.30	0.05	0.20	0.54
Na	2.58	0.50	1.26	4.11	2.71	0.42	1.31	4.13
K	0.13	0.03	0.06	0.20	0.15	0.04	0.08	0.48
Cl	4.88	1.31	1.30	8.70	4.94	1.36	1.20	8.20
NO <sub>3</sub> -N	7.20	9.44	1.00	63.00	9.50	12.90	1.00	89.00
SO <sub>4</sub>	1.34	0.44	0.80	5.10	1.57	0.83	0.80	5.60
TOC	5.54	1.94	1.33	12.00	5.91	2.18	0.90	13.10
RAL	65.70	17.70	10.00	127.00	76.40	19.30	12.00	138.00
ILAL	35.70	14.70	10.00	85.00	42.90	16.40	10.00	84.00
LAL	30.00	9.70	0.00	61.00	33.50	9.60	2.00	85.00
NH <sub>4</sub> -N	15.50	9.00	6.00	67.00	16.20	8.20	5.00	42.00
TOT-N	223.00	64.30	87.00	363.00	208.00	64.20	90.00	407.00
Colour	50.8 <sup>3)</sup>	18.30	14.00	87.00	57.6 <sup>4)</sup>	20.70	20.00	112.00
UV abs	0.22 <sup>5)</sup>	0.068	0.051	0.360	0.257 <sup>6)</sup>	0.079	0.037	0.429

1) member of observations (n) = 91

2) n = 111 (including vertical profile)

3) n = 74

4) n = 94

5) n = 69

6) n = 87

#### Octanol/water partition coefficient

In the summer of 1990 the octanol/water partition coefficient of HS was determined in soil water, at the soil/water interface and in the lake. Differences in the coefficient were found to be related to hydrological source. It is expected that, following acidification, there will be additional changes in the lipid solubility of HS (Kullberg, Lund). The technique is also being used in a Master of Science degree programme at the University of Oslo to study the changes in lipid solubility of HS in different kinds of pretreated samples.



### Isolation of HS by XAD-8

A representative from U.S. Geological Survey, Denver (Malcolm), has collected 1000 L of water from each of the two outlets (Sept. 19.-90) From these volumes were isolated 8 and 12 grams HS from A and B respectively. Dr. Malcolm used the well established XAD- technique for this isolation. This technique is identical with that used for "producing" the Intentional HS and the Nordic HS. The two isolates are to be characterized at USGS in Denver. The plans are to repeat this sampling and isolation probably next fall (1 year after the start of the acidification).

### Biological studies

- Fish: During the early 1980's some trout was transplanted in lake Skjervatjern. By intense net-fishing the last two summers the 55 individuals that were stocked, have probably been caught or have died out. The planned fish experiments will take place in the "greenhouse" downstream from the lake (see Figure 4), and preparations have been made to test the effects on several different fish-species. (Lien, NIVA).
- Phytoplankton: Quantitative samples have been taken from each of the two basins during the seasons of 1989 and 1990. The samples are stored and will be analyzed during 1991.(Brettum, NIVA).
- Zooplankton: Quantitative and qualitative samples have been analyzed from both halves in 1989 and 1990. Metal content in zooplankton will be analyzed in a representative number of samples.(Hessen, NIVA)
- Zoobenthos: During the summers of 1989 and 1990 the species composition and density of the macroinvertebrate benthic fauna of both sides of Skjervatjern have been measured. The findings show that there is a rich species diversity consisting of mostly dragonflies, damselflies and beetles. Samples have been saved for an analysis of the chemical composition of the major taxa. It is hypothesized that acid stress on the experimental side of the lake will alter their chemical composition (Hargeby, Lund).
- Microbial activity: The microbial respiration associated with the coarse (>5mm) organic debris has been measured at the land/water interface in the summers of 1989 and 1990 (Petersen, Lund).
- Macrovegetation: Photos of the lake bottom have been taken during 1989 and 1990 as a base for studying changes in the macrovegetation. Figure 15 shows Sphagnum at the bottom of basin A (Brandrud, NIVA).

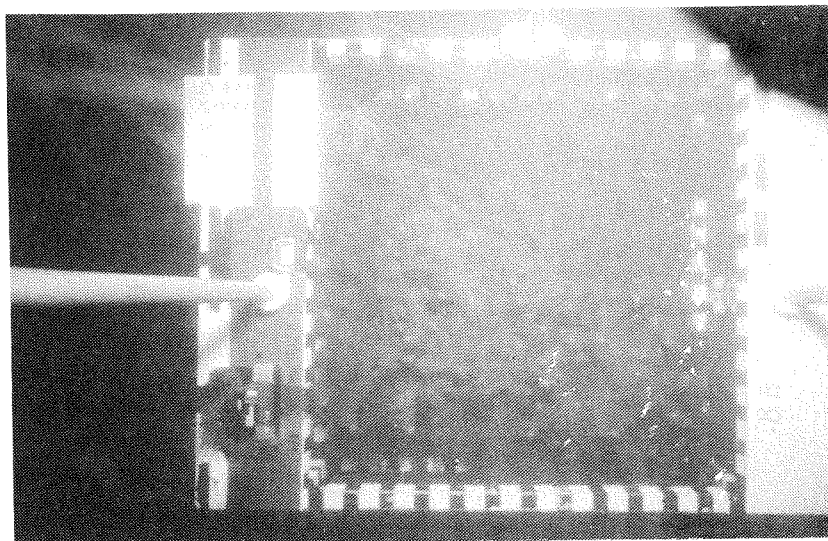


Fig.15. Photos of the Sphagnum - cover in the two basins have been taken.

### **Soil studies**

Soil water sampling devices (ceramic cup lysimeter) have been installed in 3 plots in the catchment to be acidified and in 3 plots in the control-area. These 3 plots are located in the mineral soil in the seepage zone, in bog-area close to the lake, and in bog further up in the catchment. These sites were selected in order to capture the range of different soils in the catchment. The flasks (see Figure 16) have a negative pressure (vacuum) and will suck water up from the different depths in the soil profile. Altogether there are installed 30 "samplers" of this kind. Prior to acidification, 5 series of samples have been collected, all of which have been analyzed on pH, conductivity, inorganic and organic aluminium, and total organic carbon. In addition, a selected number of samples from these and future series will be characterized using XAD-8 separation, membrane dialyzation, HPLC-size fractionation, and determination of octanol/water partition coefficient (see page 13).

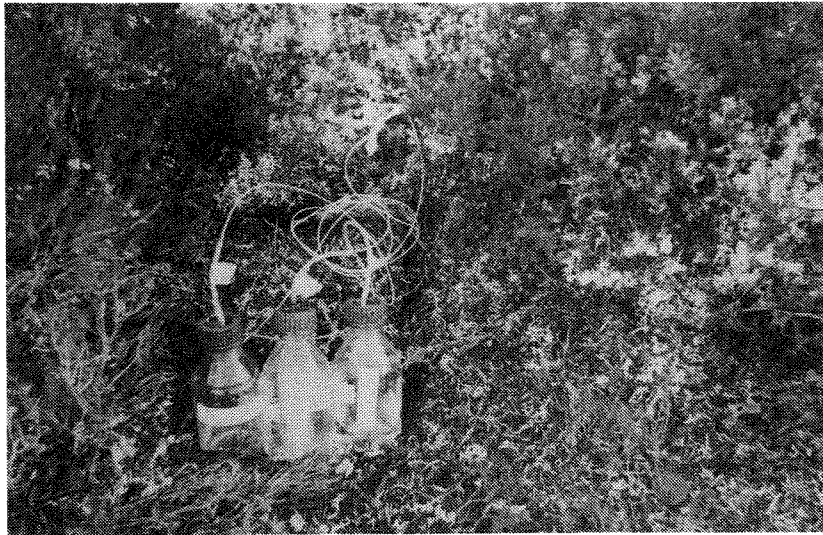


Fig. 16. Vacuumed glass bottles sucks up the soil water from different depths.

### **CLOSING REMARKS**

It is now generally realised and accepted that HS play a major role for the chemical and biological response pollutants in water. The interest that scientists from both Europe and North America have showed the HUMEX project clearly indicate that HS is "discovered" as an element of environmental importance. We strongly believe that there is a demand for more knowledge about HS in general and that catchment manipulation of the kind that is used in the HUMEX project is good approach. We hope therefore that a similar project could be established elsewhere ,for instance in North America.; this will increase the communication between scientists, but first of all it will give us an idea of to which extent one should expect regional differences in the response of acid rain to surface water containing HS.

**APPENDIX**



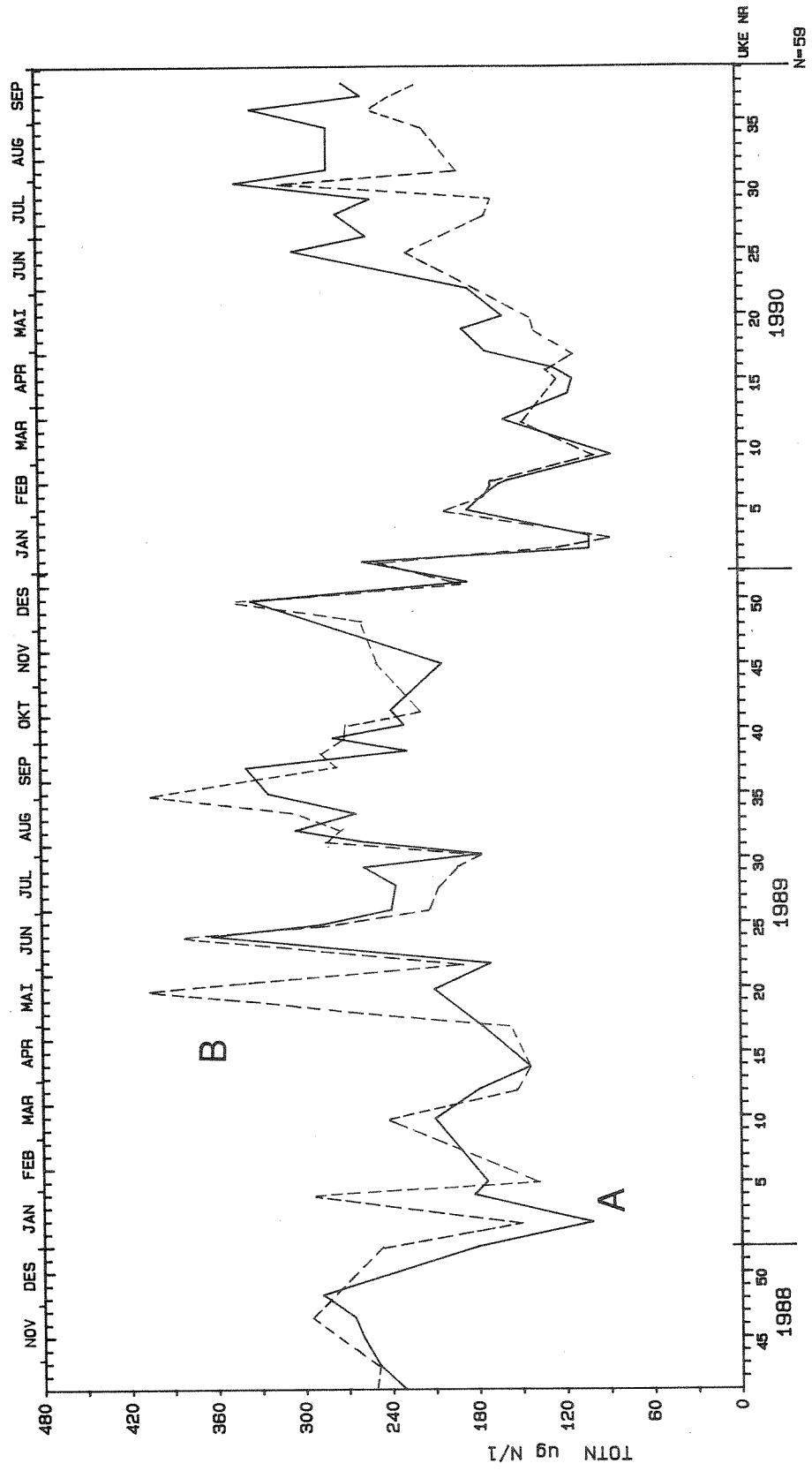






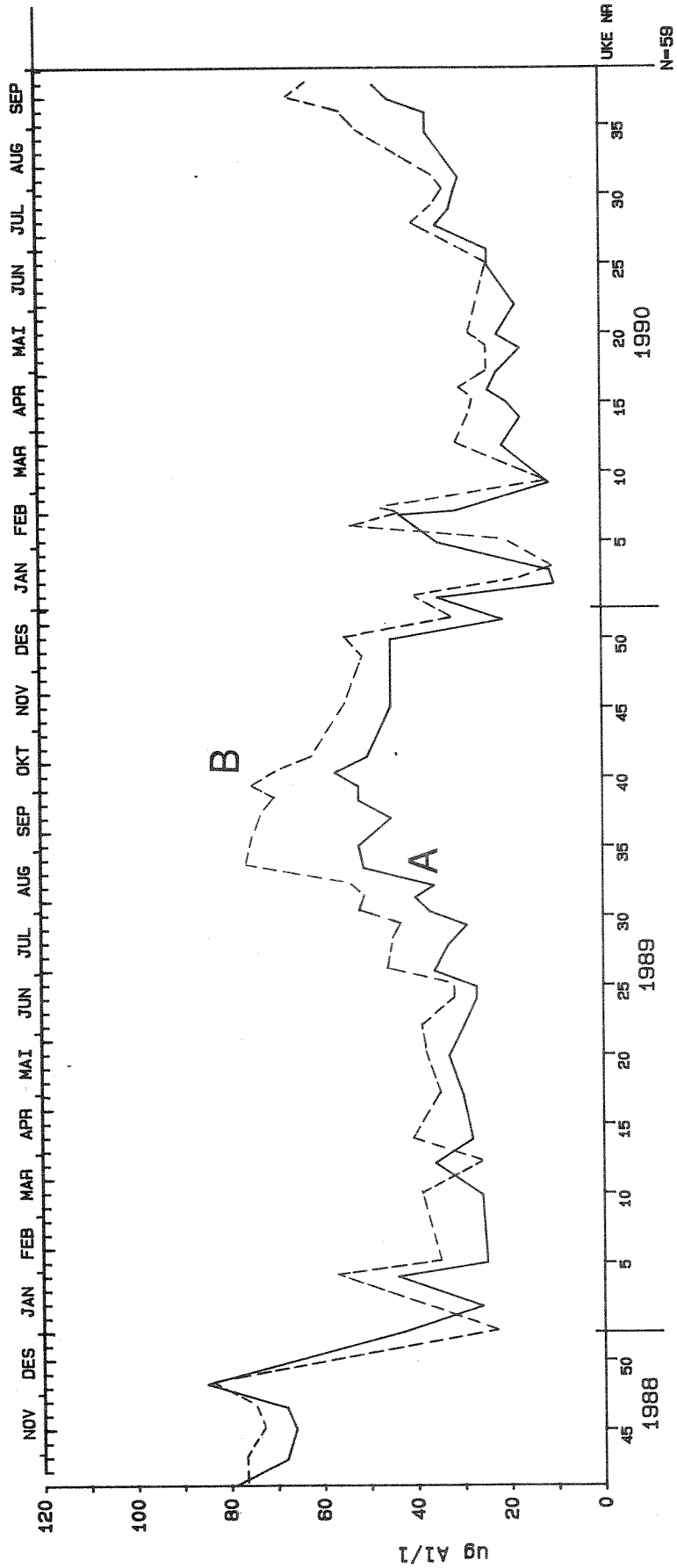


# HUMEX TOTN

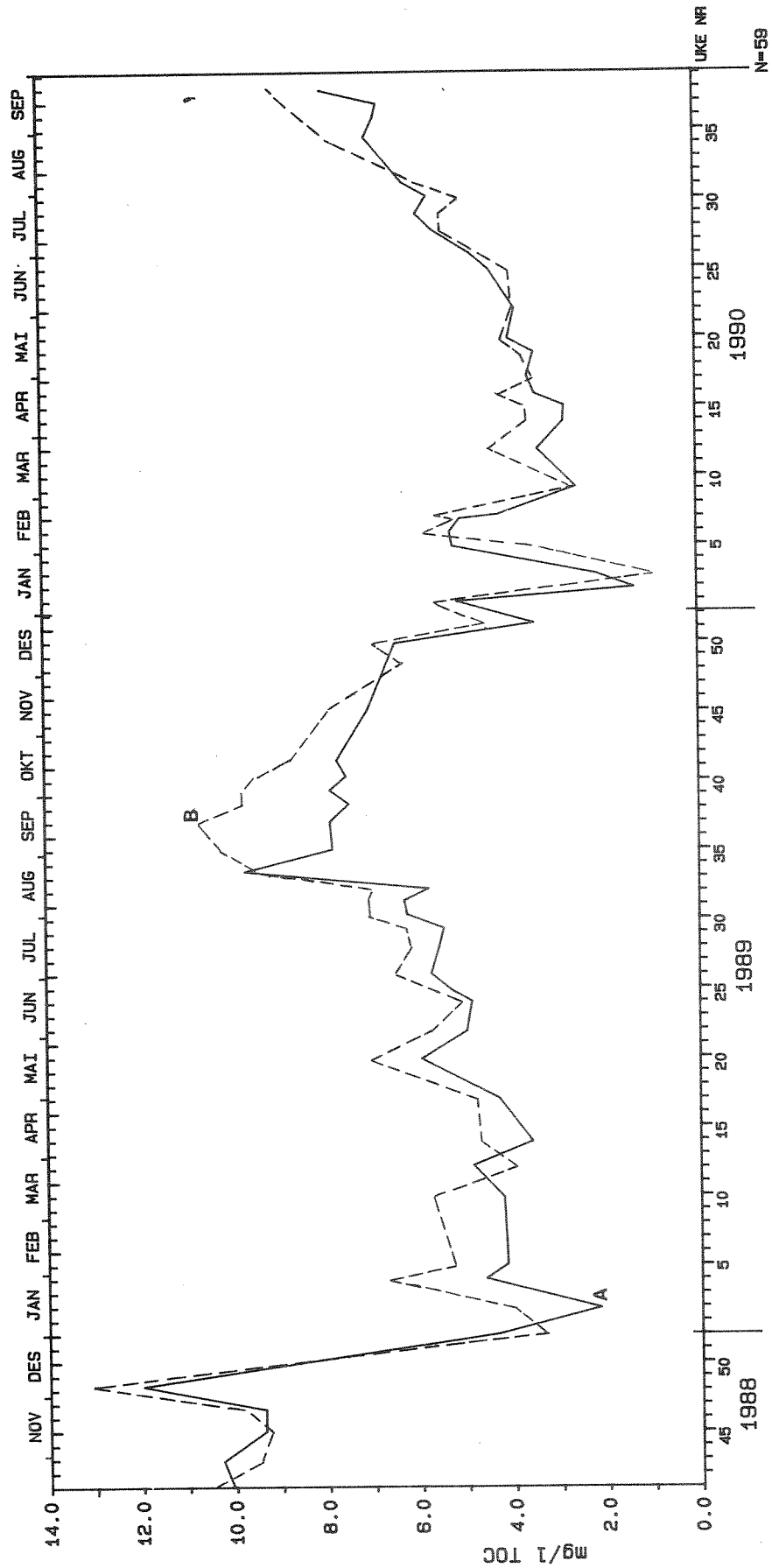


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# HUMEX, ILAL



# HUMEX TOC



# HUMEX

## Humic lake acidification experiment

### Sponsors

- Norwegian Ministry of Environment, Oslo.
- Norwegian Institute for Water Research, Oslo.

### Background and goal

Research on the acidification of lakes and rivers has focused mainly on clearwater systems. Acid deposition also affects colored waters with high concentrations of dissolved organic matter. Here the biological and chemical effects may be substantially influenced by the presence of high levels of humic matter. Humic acids comprise a significant natural buffer system in softwaters, and by complexing aluminium the dissolved organic carbon renders acidified waters less toxic to fish and other aquatic organisms.

Colored waters comprise an important fraction of the surface waters in regions of Europe and north America currently receiving high levels of acid deposition. International negotiations aimed at reducing the emissions of acidifying compounds to the atmosphere are in part based on the premise that such reductions will restore acidified aquatic ecosystems. The interaction of acidic deposition with natural organic acids must be understood before predictions of future acidification of colored lakes can be made. The HUMEX project addresses this question directly by providing experimental information at the ecosystem level.

### The Site

Skjervatjern is a small (2.4 ha), pristine, naturally acidic humic lake located near Førde, western Norway. The lake has a pH value of 4.6 with concentrations of total organic carbon (TOC) of about 7 mg C/l, nonmarine base cations of about 30  $\mu\text{eq/l}$ , and monomeric labile aluminium of less than 50  $\mu\text{g Al/l}$ . The lake supports brown trout when stocked, but no recruitment occurs due to the lack of spawning streams.

The terrestrial catchment to Skjervatjern (13.4 ha) is on granitic bedrock covered with thin organic-rich soils. Vegetation is a sparse forest of pine and birch interspersed with patches of *Sphagnum* moss.

Skjervatjern should thus be extremely sensitive to inputs of acid deposition.

### The Lake

In October 1988 Skjervatjern was divided in two halves by means of a 105-m long and 5-m deep plastic curtain.

The outlet is also split into two halves and drainage from each is gauged.

### Project plan

On one half (A) artificial acid precipitation will be applied to the lake and entire terrestrial catchment. The second half will serve as untreated reference. Acid application will be means of a permanent sprinkling system. Water from a nearby lake (cond. 1.4 m S/cm, pH 6, TOC: 1.5 mg/l), acidified to pH 3 by a mixture of sulfuric acid and ammonium nitrate will be sprayed during and immediately following natural rain events. The volume of water added will be equivalent to about 5 % of the natural precipitation. Loadings of pollutant ions  $\text{H}^+$ ,  $\text{SO}_4^{2-}$ ,  $\text{NO}_3^-$  and  $\text{NH}_4^+$  will be similar to those received in the highly impacted areas of southernmost Norway. The experimental design is similar to that used in the large-scale acidification experiments of the RAIN project.

### Participants (in addition to the HUMOR participants)

Norway: Norwegian Institute for Water Research, Oslo. Sigbjørn Andersen, Tor Erik Brandrud, Pål Brettum, Egil Gjessing (project leader), Dag Hessen, Tor Holsen, (Førde site manager), Leif Lien.

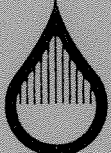
University of Oslo. Dep. of Chemistry, Oslo: Sissel Rannekleiv, Hans Martin Seip, Rolf Vogt.

Finland: National Board of Water and Environment, Helsinki: Pirrko Kortelainen, Irma Mäkinen.

Sweden: University of Lund, Limnology Institute, Lund: Anders Hargeby, Anders Kullberg, Lena Petersen.

United States: The State University of New Jersey, Rutgers, Dept. of Biology: Camden. Mark D. Morgan. U.S. Geological Survey, Denver Colorado: Ronald Malcolm.



Norsk institutt for vannforskning  NIVA

Postboks 69, Korsvoll  
0808 Oslo 8

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