

# Acidification of freshwaters – a large scale titration\*

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## Abstract

Poorly buffered freshwaters in large areas of northern Europe and eastern North America that lie in areas receiving acidic precipitation with  $\text{pH} < 4.7$  are vulnerable to acidification. Based on the hypothesis that acidification of freshwaters can be envisaged as a titration of a bicarbonate solution with strong mineral acids, water-chemistry data for 719 lakes in south Norway provide the basis for a «predictor» nomograph by which the pH-levels in lakes can be predicted given only the calcium concentration in the lake water and the pH of precipitation. This nomograph can be used to predict the effects of future changes in the acidity of precipitation on the acidity of sensitive aquatic ecosystems.

## Introduction

Acidification of lakes and rivers accompanied by the loss in fish populations is a problem in Scandinavia,<sup>1-4</sup> northeastern USA,<sup>5,6</sup> southeastern Ontario, Canada,<sup>7,8</sup> and southwestern Scotland.<sup>9</sup> Common to these areas are granitic or other siliceous bedrock types, thin and patchy podzolic soils, soft and poorly-buffered surface waters and decidedly acid precipitation (volume-weighted average pH 4.0–4.6). Recently, also in small areas on quartz sands in Belgium,<sup>10</sup> the Netherlands,<sup>11</sup> Denmark<sup>12</sup> and Italy<sup>13</sup> acidified lakes have been reported. Sulphate is usually the major anion in these acidified waters. In similar geologic areas, where the precipitation is not acidic ( $\text{pH} > 5.0$ ), surface waters generally have pH levels  $> 5.5$  and bicarbonate is the major anion.<sup>14-16</sup> Plots of pH and calcium values of such waters will fall below an empirically drawn curve,<sup>17</sup> while corresponding plots for waters in areas receiving acidic precipitation will fall above the curve.

Quantitatively, acidification can be operationally defined as the difference between preacidification alkalinity (original alkalinity) and the present day alkalinity.<sup>17</sup>

Sillén<sup>18</sup> has suggested that the ocean is the result of a gigantic acid-base titration; acids that have leaked out of the interior of the earth are titrated with bases that have been set free by the weathering of primary rock.

In this paper I will test the hypothesis that *acidified waters are the result of a large scale acid base titration; bases that have been set free by the weathering of primary rock are titrated with acids that are deposited from the atmosphere.*

This will be done by means of precipitation and water chemistry data from Norway.

## Chemical composition of surface waters

Rodhe<sup>19</sup> has suggested that the composition of freshwaters is, like the water of the ocean, governed by geochemical processes. In spite of large fluctuations in concentration, lakes tend to attain a «standard composition». He used data from lakes in Sweden collected by Lohammar<sup>20</sup> in 1935 and compared those with the world average found by Clarke<sup>21</sup> in 1924 (Table 1a). Also compared with Livingstone's<sup>22</sup> world average

\*Dedicated to the late Einar Snekvik of the Norwegian Directorate for Wildlife and Freshwater Fish whose intuition and foresight made this paper possible.

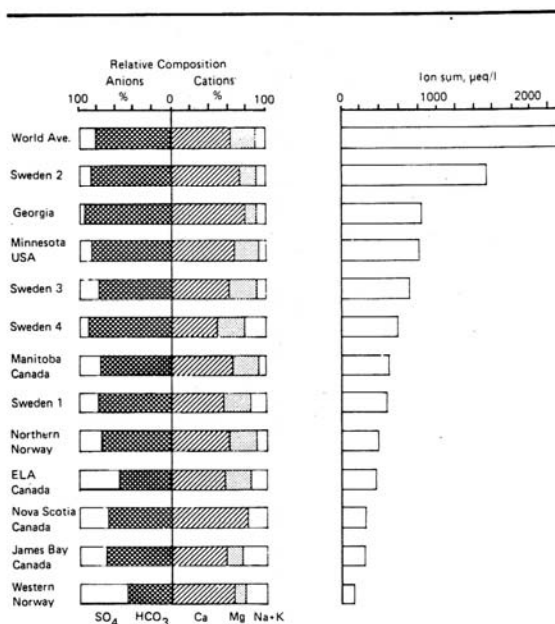


Fig. 1. Relative ionic composition of freshwaters in Scandinavia and North America situated in areas relatively unaffected by acid precipitation at the time of sampling.

Table 1a. Rodhe's standard composition of freshwaters compared with the world averages of Clarke and Livingstone.

Components	World average		«Standard composition» Rodhe 1949
	Clarke 1924	Livingstone 1963	
Ca + Mg	80.9	76.5	84.2
Na + K	19.1	23.5	15.8
Cl	10.1	15.6	9.5
HCO <sub>3</sub>	73.9	67.9	74.3
SO <sub>4</sub>	16.0	16.5	16.2

Table 1b. Corrected for sea-salts

Components	World average		«Standard composition» Rodhe 1949
	Clarke 1924	Livingstone 1963	
Ca + Mg	88.2	87.9	88.6
Na + K	11.8	12.1	11.4
HCO <sub>3</sub>	83.0	82.0	83.0
SO <sub>4</sub>	17.0	18.0	17.0

of 1963 there is a clear similarity in relative composition. Gorham<sup>23</sup> and Malmer<sup>24</sup> both point out that in areas near the coast a large part of the salts found in dilute lakes come from the atmosphere as sea-salt spray carried down by precipitation. To examine the chemical changes occurring in freshwaters resulting from atmospheric components from sources other than seawater, it is necessary to subtract off the sea-salt component.<sup>25</sup> Chloride is usually chosen as the indicator. Thus, correcting for sea-salt influence the similarity in composition of the world averages and Rodhes standard composition is striking (Table 1b), calcium and magnesium being the main cations and bicarbonate the main anion.

The relative ionic composition of waters from relatively unaffected areas in Scandinavia and North America is, when corrected for sea-salt influence, also remarkably similar to the world average, especially considering the 20-fold range in ionic strength (Figure 1).

Further these data show that in natural oligotrophic surface waters the sum of non-marine calcium and magnesium is approximately equivalent to the alkalinity (Table 2, Figure 2). Also, non-marine calcium and alkalinity are closely related. These relationships suggest that we can estimate preacidification alkalinity from the present day sum of non-marine calcium and magnesium, or alternatively, non-marine calcium only.

A consistent characteristic of acidified waters is the replacement of bicarbonate by sulphate, while little if any change in the relative concentrations of cations has occurred.<sup>25</sup> This is illustrated in Figure 3. Lakes in Sweden analyzed by Lohammar<sup>20</sup> in 1935 were reanalyzed in 1974 by Andersson;<sup>32</sup> while the relative composition of the cations has not changed significantly, the bicarbonate lost has been replaced by a corresponding amount of sulphate which has increased by a factor of 3 to 4.

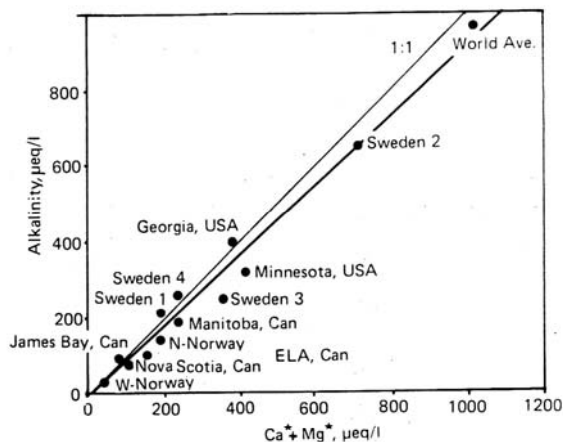


Fig. 2. Relation between alkalinity and the sum of non-marine calcium and magnesium ( $Ca^* + Mg^*$ ) based on the 13 sets of data given in Table 2. The least-squares regression lines are:

$$Alk = -14 + 0.93(Ca^* + Mg^*) \quad r = 0.99$$

$$Alk = -10 + 1.23 Ca^* \quad r = 0.99$$

The least-squares lines through the origin are:

$$Alk = 0.91(Ca^* + Mg^*) \quad Alk = 1.21 Ca^*$$

We may therefore postulate that the acidification of freshwaters is a continuous process analogous to the titration of a bicarbonate solution with sulphuric acid. Weathering of primary rock provides the bicarbonate, the atmosphere provides the acid through precipitation and dry deposition.

Table 2. Chemical composition of freshwaters in Scandinavia and North America situated in areas relatively unaffected by acid precipitation at the time of sampling.

		No. of samples	Concentrations, µeq/l							Corrected for sea-salts						
			Ca	Mg	Na	K	Cl	HCO <sub>3</sub>	SO <sub>4</sub>	Ca	Mg	Na	K	Cl	HCO <sub>3</sub>	SO <sub>4</sub>
World average	(22)	-	749	337	274	59	220	957	233	741	291	87	55	0	957	210
Sweden 1	(20)	47	126	77	49	11	26	208	30	125	70	27	11	0	208	29
Sweden 2	(20)	21	581	162	126	40	102	645	92	577	136	39	38	0	645	87
Sweden 3	(20)	21	250	121	86	18	64	247	80	248	108	32	17	0	247	73
Sweden 4	(24)	65	150	90	70	14	30	250	40	149	84	60	14	0	250	37
Northern Norway	(26)	21	131	78	87	8	85	138	54	128	60	15	6	0	138	45
Western Norway	(26)	25	38	14	44	4	40	23	33	37	6	10	3	0	28	29
James Bay, Canada	(27)	-	65	23	38	9	21	84	39	65	19	20	9	0	84	37
Nova Scotia, Canada	(28)	29	100	25	135	5	135	80	54	95	-3	20	2	0	80	40
Manitoba, Canada	(29)	17	170	82	44	13	42	182	62	169	73	8	12	0	182	58
ELA, Canada	(16)	103	99	53	39	9	21	99	78	98	49	21	9	0	99	76
Georgia, U.S.A.	(30)	5	345	75	149	26	149	398	32	340	44	22	23	0	398	17
Minnesota, U.S.A.	(31)	58	299	118	41	12	21	313	60	290	114	23	12	0	313	60

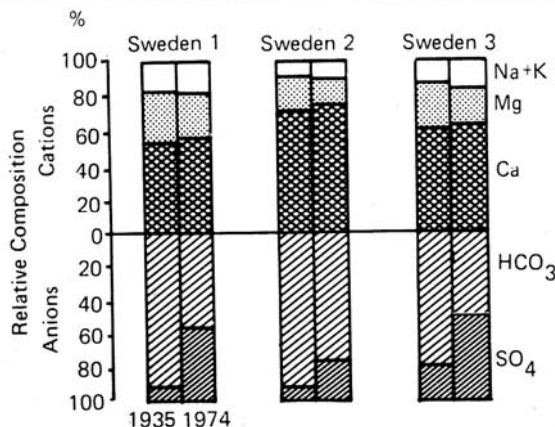


Fig. 3. Relative ionic composition of 3 sets of lakes in Sweden sampled in 1935<sup>20</sup> and in 1974<sup>32</sup>, corrected for sea salts.

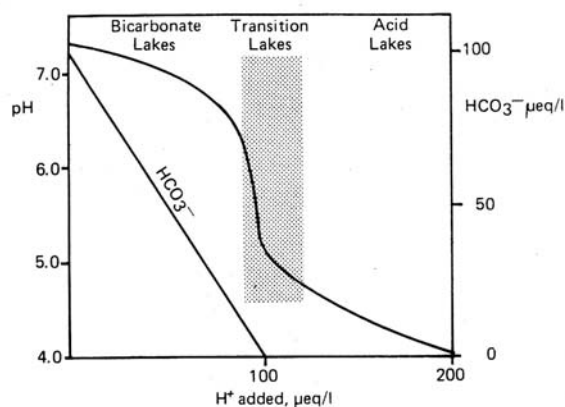


Fig. 4. Titration curve for bicarbonate solution at a given concentration (100 µeq/l) that illustrates the acidification process.

#### Acidification of lakes

The «titration curve» describing the acidification process of a lake can be segmented into three stages (Figure 4) which define both the extent and nature of the water quality change and the biological impacts. The first stage is characterized by decreased alkalinity, but maintenance of the bicarbonate buffer system and pH levels stay above 5.5–6.0 (*bicarbonate lakes*). Fish populations are usually normal at this level of acidification. In the second stage the bicarbonate buffer is lost during longer periods and severe pH fluctuations occur resulting in stress, reproductive inhibition and episodic mortalities in fish populations (*transition lakes*). The final stage of lake acidification is characterized by chronically depressed pH-levels (well below 5.0) and elevated metal concentrations, in particular aluminium. In these *acid lakes* fish are generally absent.

This «titration curve» illustrates the pH changes occurring in a lake with a given alkalinity (bicarbonate concentration) when subjected to increasing acid inputs. Lakes with different original alkalinity levels will follow other, but similar «titration curves». The pH level of a lake should therefore be a function of both the original alkalinity of the lakewater and the acidity of the precipitation falling in the watershed. The changes in pH in solutions at all levels of alkalinities when

subjected to increasing inputs of acid can be illustrated in a pH-contour nomograph (Figure 5). The 1:1 line depicts solutions exactly at the equivalence point, at which  $\text{pH} = 5.65$  assuming equilibrium with atmospheric  $\text{CO}_2$  at 25 °C. To the left of this line the alkalinity is positive (bicarbonate solutions), to the right the content of strong acids is positive (acid solutions). Thus, this nomograph, which is based on a titration of pure bicarbonate solutions at all concentrations with strong acid, tells us the theoretical pH value of a solution with a given original alkalinity when a given amount of strong acid is added.

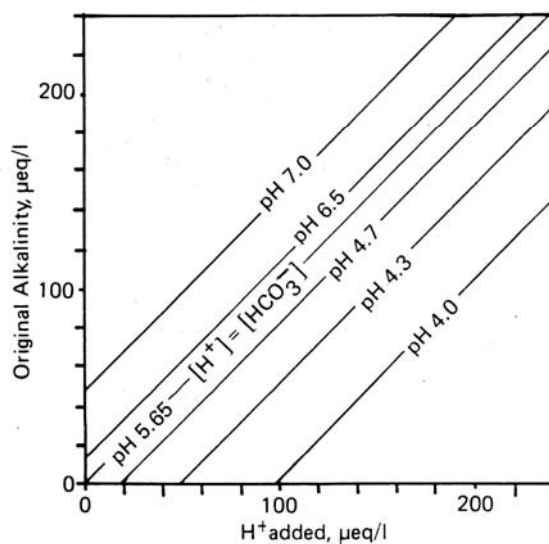


Fig. 5. pH-contour nomograph depicting expected pH levels as the result of titration of bicarbonate solutions by strong acid in equilibrium with atmospheric  $\text{CO}_2$ .

To test our hypothesis we need sets of data from series of lakes with similar levels of original alkalinities (calcium levels), but at different stages of acidification on the one hand, and from series of lakes with varying original alkalinities (calcium levels) but at same levels of acidification on the other hand. Such data are available from the lake survey conducted in the ice-free season of 1974 and -75 in southernmost Norway (comprising 719 lakes) and organized by the late Einar Snekvik of the Directorate for Wildlife and Freshwater Fish.<sup>33</sup>

The regression lines for the sum of non-marine calcium and magnesium on the non-marine sulphate concentrations for lakes in the pH-range 5.2–5.4 and for lakes in the pH-range 4.6–4.8 are highly significant (Figure 6). When these regression lines are transposed onto the titration nomograph shown in Figure 5, where the theoretical 5.3 and 4.7 lines are depicted (Figure 7), the regression line for lakes in the pH-range 5.2–5.4 coincides with the theoretical 5.3 line indicating that lakes behave as pure bicarbonate solutions when  $\text{H}^+$  is added until pH falls to about 5.3. The corresponding regression line for lakes in the pH-range 4.6–4.8, however, falls below the theoretical 4.7 line. These lakes have apparently received more strong acids than their pH levels would suggest. The position of this regression line in the diagram indicates that the pH of these lakes are higher than expected. This will be the case if the lakes contain a buffer which is active in this pH-range and which partly neutralizes the  $\text{H}^+$ -ions added. Aluminium is such a buffer. Al concentrations are higher in acid waters,<sup>34, 35</sup> apparently due to leaching by inputs of strong

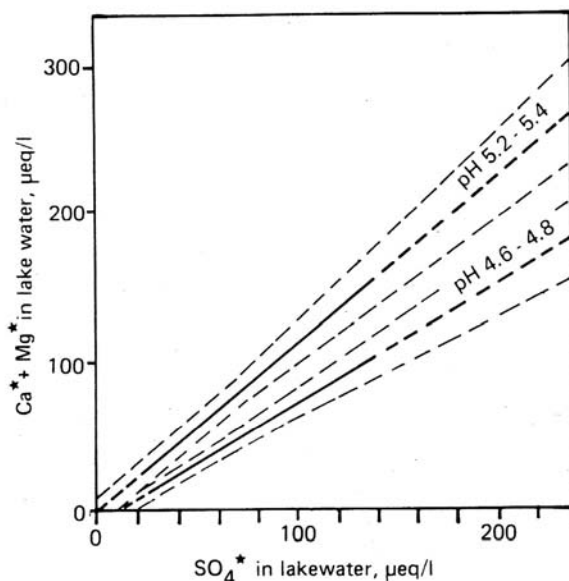


Fig. 6. Regression lines for the sum of non-marine calcium and magnesium on non-marine sulphate for lakes in southern Norway in the pH-range 5.2–5.4 and in the pH-range 4.6–4.8. Thin broken lines depict the 95% confidence limits for the regression lines.

pH-range	$\overline{\text{pH}}$	Regression equation	$r$	$n$
5.2–5.4	5.28	$\text{Ca}^* + \text{Mg}^* = -5 + 1.13 \cdot \text{SO}_4^*$	.96	58
4.6–4.8	4.69	$\text{Ca}^* + \text{Mg}^* = -11 + .75 \cdot \text{SO}_4^*$	.85	207

acids to soils. Aluminium acts as a buffer in the pH 4–5 range and relative to a pure acid solution will lower the slope of the regression line for lakes in the pH-range 4.6–4.8. The effect of aluminium can be tested using  $\text{pH}^* + k \cdot \text{Al}^{3+}$  (where  $0 \leq k \leq 1$ ) instead of pH in the regression for the 4.6–4.8 groups of lakes. The line of best fit is found when  $k = 0.4$  and the calculated line is approximately equal to the theoretical line (Figure 8). This indicates the valence of the aluminium ion at pH 4.7 to be about 1.2<sup>+</sup>, which fits reasonably well with the known equilibrium constants for aluminium in the presence of organic aluminium complexes.

These results confirm our hypothesis that the acidification of freshwaters can be described as a large-scale titration. The mineral acids in precipitation titrate the bicarbonate until neutralized. Additional inputs of acid leach aluminium out of the catchment and the product is a strong acid, aluminium buffered system.

### Predicting acidification

A nomograph can now be constructed that provides a model for lake acidification, and which relates the stages of acidification to levels of precipitation pH in sensitive regions (Figure 9).

The three types of lakes – bicarbonate dominated (pH > 5.3), transition lakes (pH 4.7–5.3), and strong acid lakes (pH < 4.7) – correspond to groupings with respect to fish population in 684 of the 719 lakes in southernmost Norway – Snekvik's lakes (Figure 10). Most of the acid lakes are barren of fish, the transition lakes contain mostly sparse populations, and the bicarbonate lakes have good fish populations.

The validity of this «predictor» nomograph can be tested with a second independent set of data. For Norway such data are available from the October 1974 regional survey of 155 lakes.<sup>14</sup> If the nomograph is used to predict into which pH group each of the lakes falls, the prediction agrees with the measured pH for more than 85% of the lakes (Figure 11).

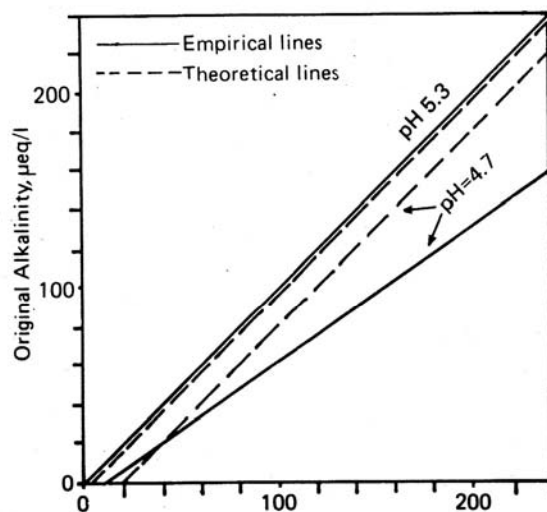


Fig. 7. The regression lines in Fig. 6 depicted in the pH-contour nomograph (Fig. 5). The non-marine sulphate concentration in the lakes are assumed equal to the amount of acid added and the sum of non-marine calcium and magnesium concentrations are assumed proportional to the original alkalinity. ( $\text{Alk} = 0.91 \cdot (\text{Ca}^* + \text{Mg}^*)$ ).

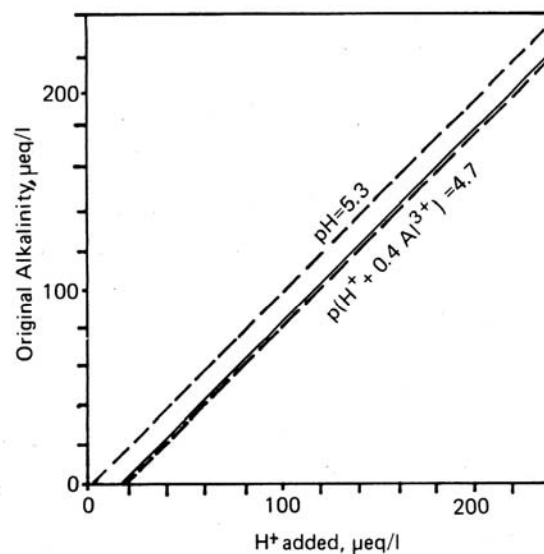


Fig. 8. Regression lines for original alkalinity on acid added ( $\text{SO}_4^*$ -concentrations) for lakes in the pH-range 4.6–4.8. ( $\text{pHAl} = \text{pH}^* + 0.4 \text{Al}$ ).

Regression equation:

$$\text{Ca}^* + \text{Mg}^* = -20 + 1.10 \cdot \text{SO}_4^* \quad (r = .92, n = 165)$$

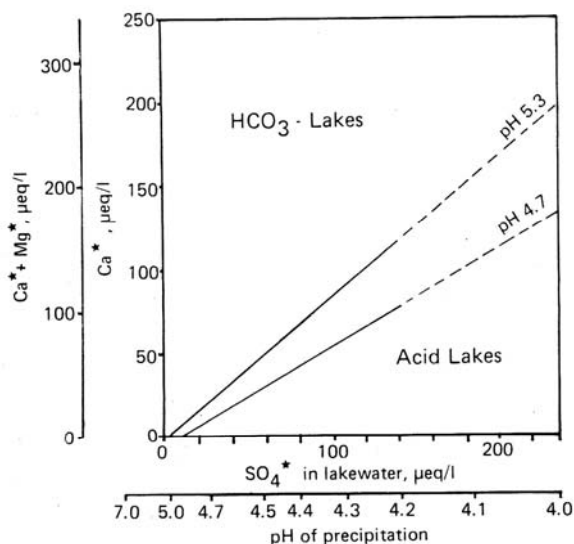


Fig. 9. A nomograph to predict the pH of lakes given the sum of non-marine calcium and magnesium concentrations or non-marine calcium concentration only and the non-marine sulphate concentration in lake water or the weighted-average hydrogen ion concentration in precipitation. The non-marine sulphate concentrations in lakes are linearly related to weighted-average hydrogen ion concentration of precipitation at each lake. Data for 59 lakes in granitic basins in southern Norway sampled as part of a regional survey in 1974 showed that non-marine sulphate in lakewater ( $[SO_4^{2-}]_w$ ) is highly correlated to non-marine sulphate concentrations ( $\mu\text{eq/l}$ ) in precipitation ( $[SO_4^{2-}]_p$ ).  $[SO_4^{2-}]_w = -19 + 1.9 [SO_4^{2-}]_p$ ,  $r = 0.84$ . In turn non-marine sulphate and  $H^+$  concentrations in precipitation are linearly related as shown by data obtained for precipitation collected July 1974 – June 1975 at 33 stations in southern Norway.<sup>14</sup>  $[SO_4^{2-}]_p = -2.7 + 1.37 [H^+]_p$ ,  $r = 0.89$ , where  $[H^+]_p$  is the weighted-average  $H^+$  concentration ( $\mu\text{eq/l}$ ) in precipitation. Combining these regression lines gives  $[H^+]_p = 9 + 0.38 SO_4^{2-}$ , which provides the pH scale for the abscissa.

Estimates of pre-acidification alkalinity are based on the assumption that calcium and magnesium neither decrease nor increase significantly in response to acidification. Such an increase would result in an overestimate of the original alkalinity and thereby affect the calculated acidification. Almer et al.<sup>15</sup> and Dillon<sup>36</sup> both have concluded that increased calcium concentrations are found in acidified areas. Schofield,<sup>37</sup> Malmer<sup>38</sup> and Watt,<sup>39</sup> however, found that no significant changes have occurred in lakes in the Adirondack Mountains, New York, U.S.A., south-central Sweden and the Halifax, Nova Scotia, area in Canada. It could be that the effect depends on the calcium levels of the lake involved. This question certainly deserves further attention.

The predictor nomograph is based on the assumption that in oligotrophic pristine freshwaters the sum of non-marine calcium and magnesium is accompanied by an equivalent amount of bicarbonate. In such areas one also finds waters in which this is not the case, for example waters containing high concentrations of natural organic acids (highly coloured waters). In such lakes a major part of the analytically-determined Ca and Mg is probably bound to organics and thus not in the cation state. Ionic Ca + Mg are thus overestimated. Also the presence of organic anions should be considered. Such lakes will not fit into the nomograph. A modified approach should be worked out for this type of waters.

The nomograph can be used to predict changes in lake pH in response to changes in precipitation pH (Figure 12). For ex-

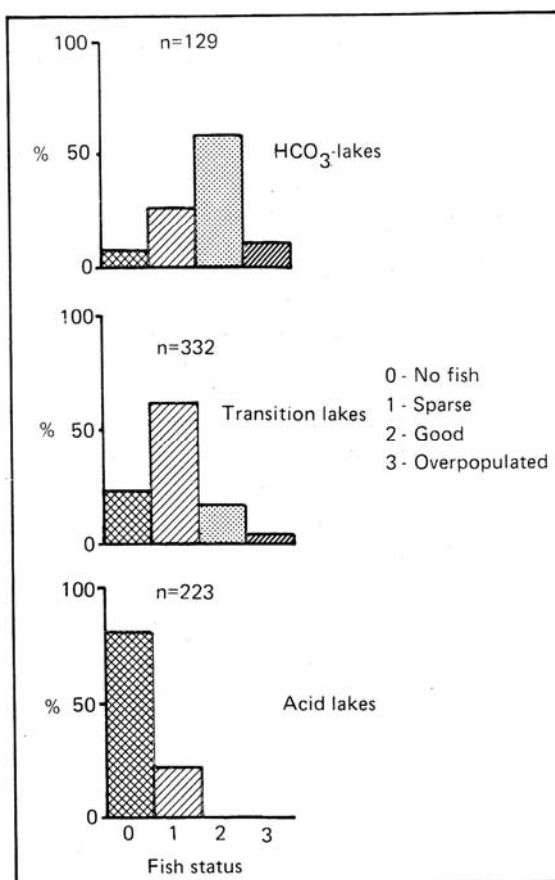


Fig. 10. Frequency histograms for fish status for 684 of Snekvik's lakes<sup>33</sup> separated according to their position in the nomograph shown in Fig. 9.

ample a lake with a calcium concentration of 50  $\mu\text{eq/l}$  situated in an area receiving precipitation with a yearly-average pH of 4.7 should still be a bicarbonate lake. This lake will enter the transition zone when the pH of precipitation decreases to about 4.4 and will turn acid at precipitation pH of about 4.3. If calcium increases in response to acidification then it will take a lower pH of precipitation to bring the lake into the acid lake category.

The nomograph has been constructed using data from small lakes in south Norway. It is, however, also valid for the large lakes in south Norway (Figure 13). Their pH values are as predicted from the nomograph. If the acidity of precipitation increases, apparently many of the larger lakes in Norway will turn acid, while others will not be significantly affected because of their higher calcium (and hence bicarbonate) concentrations.

We have seen that the acidification process can be envisaged as a large-scale titration of a bicarbonate solution with a strong mineral acid. The strength of the bicarbonate depends on the geologic nature of the watershed. If this process is reversible, the model also predicts to which pH level the precipitation must be reduced to restore a now-acid lake to a bicarbonate lake. For example, in southernmost Norway the yearly weighted-average pH of precipitation is today 4.2–4.3. To bring lakes with Ca-levels  $> 25 \mu\text{eq/l}$  back to bicarbonate lakes, the yearly weighted-average pH of precipitation should be raised to 4.7, that is, the hydrogen-ion concentration



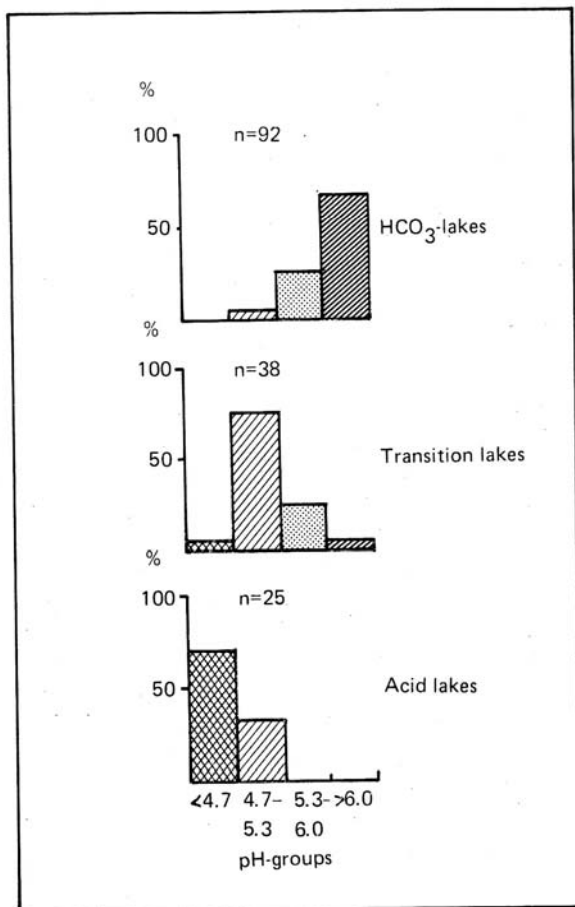


Fig. 11. Frequency histograms for measured pH in the regional lakes surveyed in 1974<sup>14</sup> separated according to their position in the nomograph shown in Fig. 9.

should be reduced by 60 – 70%. If the calcium concentration has increased in response to acidification, it should decrease in a reversed situation and the hydrogen-ion concentration must be reduced even more than 70% in order to restore lakes with Ca-levels > 25 µeq/l.

The question whether the acidification process is reversible will be crucial in any discussion about reduction of acid discharges to the atmosphere. Also, from a scientific point of view the question is important, because the answer will tell us about equilibria conditions in natural ecosystems. Future research should focus on these problems.

Since the calcium concentration of a lake is a function of the geology in its watershed, the ordinate of Figure 9 represents the influence of geology, while the abscissa represents the influence of precipitation. Above the transition zone, the geologic conditions will control the water chemistry, whatever the acidity of the precipitation may be. Below the transition zone, however, precipitation will dominate the water chemistry, as is apparently the case in the regions of Scandinavia, Scotland and North America in which the acidified lakes are found.

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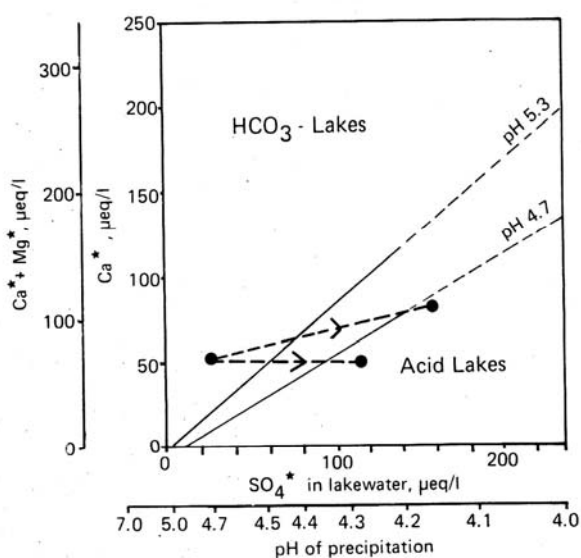


Fig. 12. The nomograph used to predict future changes in lakes when acidity of precipitation increases.

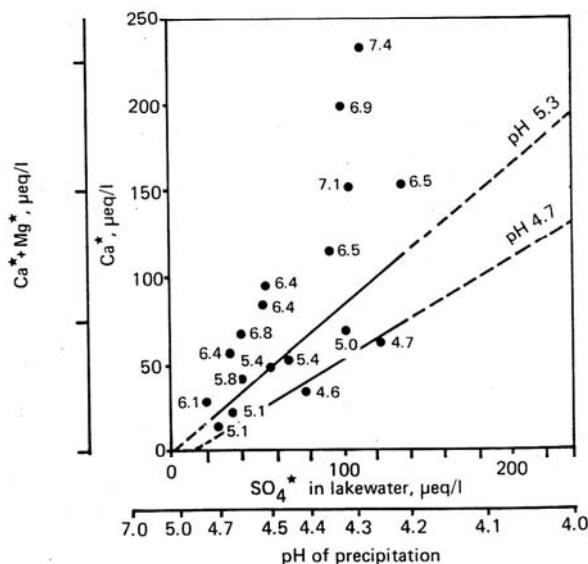


Fig. 13. pH-values of large lakes in south Norway positioned in the nomograph.

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