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## CHANGES IN THE OUTPUT OF IONS FROM A WATERSHED AS A RESULT OF THE ACIDIFICATION OF PRECIPITATION<sup>1</sup>

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**Abstract.** The average hydrogen ion input to a mountain watershed in Colorado increased by a total of  $0.142 \text{ meq} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$  over a 150-wk study period. Over the same period, the output of bicarbonate ions from the watershed, as determined by weekly stream chemistry measurements, decreased by  $0.162 \text{ meq} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$  after correction for a slight decrease in bicarbonate input. The output of sulfate, nitrate, ammonia, and dissolved organic matter all increased significantly. These increases are not enough to constitute a major change in watershed mass balance, but the nitrate and ammonia changes in particular could be important to the stream biota because of the low background levels of inorganic nitrogen. Output of calcium, magnesium, sodium, potassium, phosphate, and hydrogen ions showed no significant individual trends, but there is some evidence of a decrease in total cation output. It is postulated that essentially all of the hydrogen ions entering in the form of acid precipitation combine with bicarbonate ions in the soil, and this accounts for the reduction in bicarbonate output of the watershed. This hypothesis is well supported by the match between rates of change in hydrogen ion input and bicarbonate output. Since carbonates are not present in the soil, a combination of acid with bicarbonate would lead to the production and release of free  $\text{CO}_2$  in amounts equivalent to the acid which is neutralized. Acidification of precipitation thus appears to cause a stoichiometric reduction in bicarbonate output and smaller but potentially important shifts in output of other ions as well.

**Key words:** acid precipitation; Rocky Mountains; stream chemistry; watershed mass balance.

### INTRODUCTION

Recent investigations have shown that the acidification of precipitation by anthropogenic mechanisms has occurred at a rapid pace in northern Europe (Oden 1976), parts of the eastern United States (Cogbill and Likens 1974, Likens and Bormann 1974) and, according to some very recent information, parts of the western United States as well (Lewis and Grant 1980, Table 1). The environmental effects of acid precipitation are only now being investigated and are thus at the present time extremely uncertain (Gorham 1976). Serious effects which seem to be relatively certain are connected with acidification of lakes (Almer 1974, Hendry and Wright 1975, Beamish 1976, Schofield 1976). Alteration of growth rates of vegetation may also occur but still require documentation (Abrahamsen et al. 1976, Cogbill 1976, Tamm 1976, Tamm and Cowling 1976). The effects of acid precipitation on soils are also uncertain. Acidic soils with low exchange capacity and low buffer capacity are likely to be most vulnerable (Wiklander 1974), but several authors have pointed out that the total buffering capacity of soils is high by comparison with the rate of acidification of rainfall (e.g., Frink and Voigt 1976, McFee et al. 1976, Cole and Johnson 1977). The exact fate of hydrogen ions added to soil is difficult to judge owing to the great complexity of soil chemistry, but it would appear that the acid-absorbing capacity of most soils

in a strictly chemical sense is quite great. Nevertheless, the addition of acid to soils could easily result in the release of other materials such as ions deriving from ion exchange reactions or acid-base reactions (Oden 1976, Wright et al. 1976, Johnson et al. 1977). Similarly, addition of acids might well reduce the amounts of certain substances liberated from soils.

We have previously shown that the watershed of Como Creek, near the Continental Divide in Colorado, has experienced steady and marked increase in the input of hydrogen ions associated with increasing amounts of nitric acid in precipitation over a 3-yr period, June 1975–May 1978 (Lewis and Grant 1980, Table 1). This increased acid input, which is of unknown but probably anthropogenic origin, has resulted in a decline in pH of bulk precipitation of 0.8 pH units in the 3-yr interval (from pH 5.4 to pH 4.6, adjusted annual mean). Since the output rates of major chemical substances were documented over the same period, we are able to examine the output data in search of trends which might be related to the acidification of precipitation and thus deduce some of the mechanisms by which incoming acid is processed in the terrestrial system as well as the effect of acid precipitation on the mass balance of the watershed and the chemistry of the stream draining the watershed.

### SITE CHARACTERISTICS

The Como Creek Watershed is located at 2900 m altitude 6 km east of the Continental Divide in north-central Colorado (Fig. 1). The stream above the sam-

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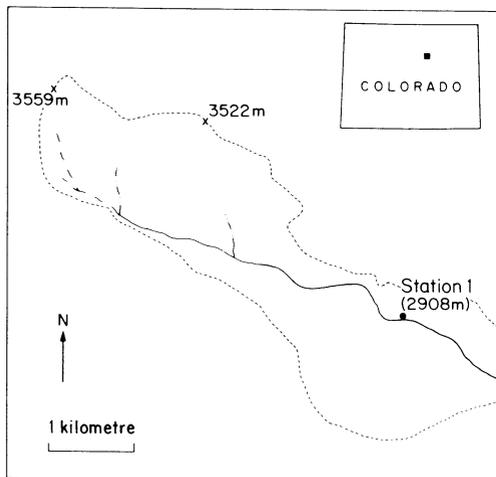


FIG. 1. Map of the Como Creek Watershed showing the location of the stream sampling station.

pling site drains an area of 664 ha that has been subjected to minimal human activity over the past 40 yr. The area is covered primarily with conifers (*Abies lasiocarpa*, *Picea engelmannii*, *Pinus contorta*, *Pinus flexilis*), but also has some aspen (*Populus tremuloides*) and *Salix*. Approximately 20% of the watershed is not forested because it is above tree line, too boggy, or was cut some 60–75 yr ago and never reseeded. These areas are covered with alpine herbs, grasses, sedges (*Carex*), and willows (*Salix*).

The discharge of Como Creek varies from 3.5 l/s in late summer to a maximum of  $\approx 360$  l/s during the runoff period (June) of a wet year. Total wet precipitation reaching the watershed averages  $\approx 770$  mm/yr, and there is a slight increase in the amount of precipitation from the bottom to the top of the watershed. Weather comes principally over the Continental Divide from the west, although upslope storms from the east are not unusual in spring or early summer (Barry 1973).

The watershed soils, which are presently under intensive study (S. Burns, *personal communication*), are typically quite thin (mean  $\approx 60$  cm; maximum  $\approx 200$  cm) and overlie granitic parent material. The cation exchange capacities range between 5 and 30 meq/100 g and average  $\approx 20$  meq/100 g. Surface pH values range mostly between 4.5 and 6.0. No carbonates are present. The soils are of the podzol type, but according to revised taxonomy would be classed as spodosols with intermixed alfisols undergoing spodosolization (Soil Survey Staff 1975; Burns, *personal communication*).

Como Creek has a very low dissolved-solids content (average specific conductance, 32  $\mu$ s/cm at 25°C) and very low dissolved-nutrient levels. Although the pH of the stream changes seasonally, the median pH is slightly below 7.0. Substantial amounts of dissolved organic carbon derived from the terrestrial system (2.7 mg C/l, weighted mean) are typical of the stream.

## METHODS

Stream water and bulk precipitation were sampled on a uniform schedule for 150 wk. Stream samples were taken weekly with a hard plastic bottle over a period of  $\approx 2$  min at a single location. Bulk precipitation was collected continuously at two stations 1 km apart along the stream. The precipitation collector was emptied and rinsed at weekly intervals. The methods of collection and analysis for bulk precipitation samples have been given elsewhere (Lewis and Grant 1978) and need only be outlined here.

Analysis of all samples was initiated within 3 h of the time of collection. The samples were filtered through Whatman GF/C glass fiber paper (effective pore size  $\approx 2 \mu$ m) so that the particulate and dissolved fractions could be analyzed separately. For present purposes, only the analyses of pH and the major dissolved cations and anions are relevant.

The amount of hydrogen ion was determined from the pH of a fresh sample. Major cations (calcium, magnesium, sodium, potassium) were determined by atomic absorption on acidified samples (1% of 1.0 normal HNO<sub>3</sub>). Alkalinity was determined by titration of a 100-ml aliquot with standard acid to a pH of 4.4. All of the alkalinity in the sample is assumed to be due to the CO<sub>2</sub>-HCO<sub>3</sub><sup>-</sup> system, although in fact  $\approx 5\%$  was due to the presence of other bases. Sulfate was determined by a barium precipitation technique (Golterman 1969) and phosphate by an ascorbic acid method (Golterman 1969). The ammonia concentrations were determined by a phenylhypochlorite method (Solorzano 1969) and the nitrate concentrations by cadmium-copper reduction followed by formation of an azo dye (Bendschneider and Robinson 1952, Wood et al. 1967).

Discharge from the watershed was estimated weekly by means of a Parshall flume which was empirically calibrated with pumps. In the computation of output rates for the watershed, the amount of each dissolved substance on a particular week was multiplied times the discharge for that week. Output in particulate form was quantitatively unimportant for the present analysis and is therefore not included here. The computation of chemical input was accomplished in a similar manner from the weekly amount of each of the substances in bulk precipitation reaching the watershed. The weekly input and output data were then studied as a function of time by means of regression techniques.

One complicating factor in the determination of trends across years in the output data has to do with the heavy pulsing of output in connection with spring runoff. Variation in amount of discharge can change the output rates because the nutrient pool in the terrestrial system responds to additional flushing with increases in output. This seasonal change might not affect a statistical examination of trends across years if large numbers of years were included in the data base. For a small number of years, however, a fortuitous

TABLE 1. Concentrations and fluxes of major cations and anions in the Como Creek Watershed and the changes in fluxes over a 150-wk period of increasing precipitation acidity. Stream concentrations are weighted averages. Bulk precipitation concentrations are total bulk input for 150 wk divided by total wet precipitation for 150 wk. Trends in input and output are indicated only when they are statistically significant at  $P \leq .05$ .

	Concentrations		Input-output budgets		Change in input-output budgets	
	Bulk precipitation mg/l	Stream mg/l	Mean input meq·m <sup>-2</sup> ·wk <sup>-1</sup>	Mean output meq·m <sup>-2</sup> ·wk <sup>-1</sup>	Change in input meq·m <sup>-2</sup> ·wk <sup>-1</sup> in 150 wk	Change in output meq·m <sup>-2</sup> ·wk <sup>-1</sup> in 150 wk
Ca <sup>++</sup>	0.611	1.85	0.381	0.221	No trend	No trend
Mg <sup>++</sup>	0.070	0.53	0.072	0.104	No trend	No trend
Na <sup>+</sup>	0.304	1.28	0.165	0.133	No trend	No trend
K <sup>+</sup>	0.247	0.26	0.079	0.016	No trend	No trend
NH <sub>4</sub> <sup>+</sup> -N	0.192	0.0101	0.171	0.000170	No trend	+0.00196
H <sup>+</sup>	0.0167*	0.00015*	0.209	0.000350	+0.142	No trend
HCO <sub>3</sub> <sup>-</sup>	2.62	11.1	0.536	0.433	-0.040	-0.202
SO <sub>4</sub> <sup>=</sup>	1.45	0.38	0.376	0.010	No trend	+0.021
NO <sub>3</sub> <sup>-</sup> -N	0.273	0.0038	0.243	0.00065	+0.141	+0.000552
PO <sub>4</sub> <sup>=</sup> -P	0.0083	0.0038	0.010	0.00029	No trend	No trend

\* Expressed as pH, bulk precipitation is 4.77 and stream is 6.83.

downward trend in the amount of spring runoff for different years could cause a decline in output across those years and could thus be confused with other potential causes of declining output, such as acidification of precipitation. To avoid this potentially confounding influence, we determined the changes in output from a multiple regression model (output vs. time and discharge) rather than a simple regression model (output vs. time). The multiple regression model was as follows: weekly output =  $b_1 \cdot (\text{weekly discharge}) + b_2 \cdot (\text{elapsed time in weeks}) + c$ . The regression was always forced to accept discharge as the first variable. We then examined the residual magnitude and significance of the elapsed time in weeks after the influence of discharge had been accounted for. In contrast to the output (stream) data, the input (precipitation) data were treated with simple regression because confounding trends could not be detected.

## RESULTS

Table 1 summarizes the mean input and output of major ions for the Como Creek Watershed over the 150-wk study period. The table shows that the Como Creek Watershed is a strong accumulator of calcium, phosphorus, potassium, sulfate, inorganic nitrogen, and hydrogen ions, and is more nearly balanced with respect to magnesium, sodium, and bicarbonate.

In addition to the mean input and output values, Table 1 shows the amount of change in input and output wherever a statistically significant linear trend exists ( $P \leq .05$ ). The standard error of the slope, wherever significant trends occurred, was always between 22 and 45% of the value of the slope.

The table provides no evidence for trends in the input of any substance except the hydrogen and nitrate ions and, probably as a secondary result of the hydrogen ion trend, the bicarbonate ion. The absence of other trends is advantageous insofar as it simplifies the analysis of output.

Table 1 shows no significant change in the output of calcium, magnesium, sodium, potassium, phosphorus, or hydrogen from the watershed. The amount of bicarbonate leaving the system declined significantly over the 150-wk study period, but the output of ammonium, nitrate, and sulfate ions increased significantly. The magnitudes of change in ammonium, nitrate, and sulfate are trivial in terms of mass balance, although they could be of considerable biological importance in the stream itself. In contrast, the bicarbonate change is major in terms of mass balance. On the whole, the output data indicate a drastic change in the regulation of bicarbonate output and secondary changes in ammonium, nitrate, and sulfate output.

## INTERPRETATION AND DISCUSSION

Since increase in hydrogen ion input is not matched by a significant increase in hydrogen ion output, it is safe to conclude that the increasing amounts of hydrogen ion entering the system are taken up almost entirely by the terrestrial component. If the incoming hydrogen ions were taken up by the mechanism of ion exchange, one would expect to see an increasing trend in some or all of the cations. The data show no such trend.

Another possible fate of incoming hydrogen ions is combination with bases in the soil system. Since the soil in the Como Creek Watershed has a relatively low pH and is of granitic origin, the amount of carbonate present is essentially zero. This means that bicarbonate is the most likely inorganic base to combine with the incoming hydrogen ions and thus account for their failure to appear in the output from the system. This mechanism is consistent with the data, as the bicarbonate output of the watershed shows a significant downward trend corresponding to the upward trend in hydrogen ion input (Table 1). One might expect some decline in bicarbonate solely as a by-product of the declining input of bicarbonate (0.040 meq·m<sup>-2</sup>·wk<sup>-1</sup>),

but the decline in output of bicarbonate is considerably greater than this ( $0.202 \text{ meq} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$ ). Thus a total decline in output of bicarbonate equal to  $0.162 \text{ meq} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$  cannot be accounted for except by means of the increased input of hydrogen ion. The increase in hydrogen ion input is in fact remarkably well matched to the unaccountable decrease in bicarbonate output ( $0.142$  vs.  $0.162 \text{ meq} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$ ). We therefore postulate that hydrogen ions equal to  $0.162 \text{ meq} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$  combine with bicarbonate ions to produce free  $\text{CO}_2$ :



This implies that  $0.162 \text{ meq} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$  of  $\text{CO}_2$  are produced simply as a result of acidification of the soil, and that the full increase in hydrogen ion input can be accounted for by the bicarbonate buffer system. The increase in nitrate input is undoubtedly taken up for the most part by the biota.

An independent check on the proposed soil bicarbonate titration by strong acids in bulk precipitation is possible through a corollary hypothesis. If increasing precipitation acidity does indeed reduce annual bicarbonate yield by titration of soil bicarbonates, a seasonal effect should also be evident, as the flushing of soils by precipitation varies seasonally. The data show that bicarbonate yield increases at a rate significantly less than the increased rate of discharge within any 1 of the 3 yr of the study ( $P < .05$ ). Furthermore, the slope of yield on discharge for bicarbonate is lower than for any major cation, indicating that mere dilution of soil water by precipitation cannot explain the results. We have developed this and more complex aspects of seasonal changes in yield elsewhere (Lewis and Grant 1979). The essential point here is that seasonal changes in yield are as expected if the mechanism we have proposed to account for long-term trends in bicarbonate output is valid.

The ionic balances of all but the last column in Table 1 are well within analytical limits. The last column, which reports changes in output, is unsatisfactory in this respect, as net change in anions exceeds net change in cations by  $0.180 \text{ meq} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$  over the 150-wk period. Assuming that anion and cation loss should be similar, this degree of imbalance seems unlikely. We therefore considered additional factors which would be likely to account for the difference. (1) Although individual cations do not show a downward trend, it is possible that total cations would. We did the regression analysis for total cations and discovered a negative relation that was not significant statistically but was highly suggestive ( $P = .14$ ). If accepted as valid, this relationship would account for  $\approx 0.050 \text{ meq} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$  as a cation decrease brought about by bicarbonate decrease. (2) Organic anions are not considered in Table 1. A regression study of the DOC (dissolved organic carbon) output shows a highly significant increase ( $P < .001$ ) from  $6.63 \text{ mg}$

$\text{C} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$  at the beginning of the study to  $9.00 \text{ mg C} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$  at the end, or a total change over the 150 wk of  $2.37 \text{ mg C} \cdot \text{m}^{-2} \cdot \text{wk}^{-1}$ . The cause of this change is unknown, but changing rates of decomposition in the terrestrial system may be involved (G. E. Likens, *personal communication*). Although the ionic strength of these organic compounds cannot be estimated reliably, this major change in carbon output also contributes to the resolution of the ionic balance problem in column four of Table 1. (3) Chloride, which is a minor constituent in this system, was not analyzed systematically and may therefore account for part of the apparent imbalance.

The data indicate that the Como Creek Watershed is strongly responsive to increases in the acidity of precipitation as a result of the titration of bicarbonate by strong acids in the soil system. This in turn implies that stream chemistry is responsive to watershed acidification insofar as stream chemistry is regulated by watershed output. Although the total inventory of buffering substances is definitely not exhausted by the increased input of mineral acids in precipitation, the output characteristics of the watershed are changed as a result of the titration of the main buffering compound (bicarbonate) by the hydrogen ions. An effect of this type would be limited to soils containing negligible carbonate, as addition of hydrogen ions to a system containing carbonate would be expected to produce additional bicarbonate rather than reduce the amount of bicarbonate (Stumm and Morgan 1970).

The mechanism we have proposed for the fate of increasing acidity in the Como Creek system requires the production of  $\text{CO}_2$  from bicarbonate as it is titrated with strong acid. If this  $\text{CO}_2$  escapes from the soil, present total release of carbon dioxide attributable specifically to the titration effect would amount to some  $101 \text{ mg C} \cdot \text{m}^{-2} \cdot \text{yr}^{-1}$ , which is small by comparison with fossil fuel sources ( $\approx 10000 \text{ mg C} \cdot \text{m}^{-2} \cdot \text{yr}^{-1}$ , Adams 1977) but could become larger with continued acidification. Any increment in  $\text{CO}_2$  release caused by the titration mechanism must be considered anthropogenic because the increasing acidity itself is anthropogenic.

The increases in output of inorganic nitrogen, although of negligible importance to mass balance, are worthy of note in another context, as fixed inorganic nitrogen is of great potential nutritional significance to the freshwater biota. The increase in stream concentrations of nitrate, after adjustment for variation in discharge by the multiple regression procedure, is 248% over the 150-wk period (change in weighted average of stream concentrations from  $2.0 \mu\text{g/l NO}_3\text{-N}$  to  $4.9 \mu\text{g/l NO}_3\text{-N}$  over the 150-wk period). The comparable figure for ammonia is 375% (change in weighted average stream concentrations from  $3.8$  to  $14.2 \mu\text{g/l NH}_4\text{-N}$ ). The mechanism by which these increases occur is not clear, but if acidification of precipitation is responsible, the trend can be expected to continue and

will thus increase significantly the availability of a potentially limiting nutrient for the biota of the freshwaters draining the watershed.

The conclusions drawn here can only be tentative, as the period of data collection, although long in absolute terms, is insufficient for unqualified conclusions concerning trends. The Hubbard Brook Ecosystem Study has shown that it is highly advantageous to study nutrient cycling in natural ecosystems from the perspective of some 10 yr of data (Likens et al. 1977), but it is excessively restrictive and costly to limit interpretation to such conditions. Moreover, statistical correction for confounding trends greatly reduces the likelihood of erroneous conclusions.

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