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Monitoring of ionic concentrations in airborne particles and rain water in an urban area of central Germany

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ABSTRACT

The primary objective of this investigation was to document fluctuations of levels of ions in airborne particles and in rain water with the help of ion chromatography. The environmental emission situation in the investigated geographic area is characterized by a widespread use of domestic coal-burning furnaces. The use of a parallel impactor for particulate sampling permits the results to be interpreted additionally in terms of particle size. The anions and cations were extracted from the filters in the sampling devices into ethanol–water mixtures by sonication. Rain water samples were analysed after a simple dilution step. The data obtained on ionic fluctuations are compared with similar data sets published by other laboratories.

INTRODUCTION

Frequent analyses of air contaminants in rain water and airborne and settled dust particles are essential for any reliable estimates of damage caused by air pollution. After polyaromatic and halogenated hydrocarbons, inorganic anions and cations represent the most important information that needs to be provided by any air monitoring programme. Especially a knowledge of the levels of anionic components in aerosols is desirable in view of the increasing acidity of natural precipitation. It is well known that the main causes of this effect are the high emission levels of sulphur and nitrogen oxides, which are converted under atmospheric conditions into detectable amounts of sulphuric and nitric acid. Additional anionic contributions to atmospheric pollution in the form of fluoride and chloride come from power-generating processes. Determined cation levels are used in mass balance calculations to verify the completeness of the analytical information obtained with regard to total ion contamination.

Apart from these chemical properties, the physical properties, in particular the particle size distribution, are important for the toxicity of airborne dust. Airborne dust particles from aerosols and are taken up by humans chiefly by inhalation. Coarse dust particles (> 3.5 μm) are retained largely in the rhynopharynx and discharged relatively quickly via the gastro-intestinal tract. In contrast, fine dust particles (< 3.5 μm) may enter the alveolar system. Therefore, the resorption rate and hence the toxicity are significantly higher. Irrespective of the toxicity aspect, published investigations on acid aerosols include, in most instances, determinations of pollutants without regard to particle size.

The results presented in this paper were obtained in a highly industrialized area characterized by the widespread use of relatively obsolete, emission-prone processes. The problem of high levels of air pollution has been ongoing, despite the large number of industrial stoppages and the drastic decrease in production output that have occurred in the last 2 years. Even with such decreased levels of industrial production, the Leipzig–Halle–Bitterfeld industrial area is currently (1990–91) still showing levels of air pollution that are distinctively higher than those found elsewhere in Germany [1,2]. Our lab-

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oratory participates in air monitoring as a member of an extensive network, which on completion will provide a complete coverage of the local geographical area. Some of the recent results indicate a clear trend towards a general improvement in the environmental situation.

EXPERIMENTAL

Sampling techniques

The sampling of dust was carried out at a location in Halle characterized by a high traffic volume and a high density of housing utilizing predominantly brown coal for heating. Two different designs of dust sampling devices were utilized during the air monitoring experiments.

The first investigations carried out from February to June 1991 dealt with the time dependence of the pollutants absorbed by the airborne dust particles on their surface and its distribution according to the particle size. The sampling device, an ICP 5 parallel impactor developed by the Frauenhofer Institute for Environmental Studies in Hannover (Germany), was a prototype. The function of this sampler is that a defined volume of aerosol sucked in is split into six equal flows and fed via prefilters in such a manner that only defined particle fractions can reach the analytical filter arranged beneath. The fractions obtained are as follows: (I) 0–3.5 μm; (II) 0–6 μm; (III) 0–10 μm; (IV) 0–17 μm; (V) 0–25 μm; (VI) total dust. According to Puxbaum [3], such fractionation allows one to discriminate reliably between very fine particles (accumulation maximum at 0.7 μm) and coarser particles (accumulation maximum at 10 μm). The sampling period was 48 h and the air volume was *ca.* 28 m^3.

The filtration material tested was compressed glass-wool beds owing to the limited pump capacity of the device. This filter material makes it possible only to determine anions because it has a high sodium blank level. On the other hand, it is sufficient to determine the anion values for assessment of pollutants.

For ion balance studies a second experimental series was started in June 1992, using another sampling device, a GS 050/3, obtained from Derenta (Berlin, Germany). Here we used PTFE filters (diameter 50 mm, pore size 1.2 μm, from Sartorius, (Göttingen, Germany). The sampling period was usually 24 h and the collected dust volume was 35–40 m^3.

The sample preparation for both types of filter merely involved the extraction of inorganic ions from the particles accumulated on the filters during the sampling. To achieve this, each of the filters was placed in a polyethylene beaker containing 10 ml of ethanol–water (1:9). The extraction of water-soluble sample components with ethanol–water mixtures is a widely used standard procedure [4]. The filters were kept immersed in the extraction liquid overnight and then subjected to ultrasonication. After filtration through a 0.45-μm filter (Type HV; Millipore, Bedford, MA, USA), the extraction liquid containing water-soluble ions from the collected dust particles was subjected to ion chromatographic analysis. A check on this dissolution step by performing a second one under identical conditions confirmed a better than 95% efficiency for the single-step extraction procedure.

The sampling of rain water was carried out in an industrial park located in the eastern part of Halle. During the period poor in rain between February and July 1992 (only sixteen "rain events" occurred), the samples were collected with the help of a "wet-only collector".

The collector obtained from the Institute of Energetics in Leipzig (Germany) possesses an electrode sensor that opens a polyethylene funnel after coming into contact with wet precipitation. The rain water runs into a 2-l polyethylene flask that is positioned in a sampling carousel. At programmable intervals (standard condition = 24 h) another polyethylene bottle will be positioned below the reception funnel. When the heatable sensor at the end of deposition becomes dry, the reception funnel is closed. A temperature sensor controls a three-level heating system, the function of which is to prevent the pyramid setting sensor becoming moistened by dew or hoar frost. This device also guarantees the melting of snow and the opening of the collector to collect water from snow. Each of the sixteen samples was divided into a number of aliquots that were utilized for pH and conductivity measurements and finally also for ion chromatographic (IC) analyses for anions and cations.

The IC runs were carried out immediately, if possible. When an immediate analysis of samples was not possible, the samples were preserved by storage at freezing temperature until the time of their injection into the IC system.

Ion chromatography

Non-suppressed IC was utilized exclusively. The IC system consisted of a Model 510 pump, a U6K injector and a Model 431 conductivity detector. All hardware components and the data acquisition and data reduction system were obtained from Waters Chromatography Division of Millipore (Milford, MA, USA).

The separations of anions (fluoride, chloride, nitrate, sulphate) were performed on an IC Pak A HC column (Waters) with a gluoconic acid–boric acid (1.3 m*M* each, pH = 8.5) aqueous solution as the eluent. The flow-rate was maintained at 2 ml/min. An IC Pak C M/D column (Waters) was used for all cation separations (sodium, ammonium, potassium, magnesium, calcium) with 3.0 m*M* nitric acid–EDTA 0.1 m*M* solution as the eluent. The flow-rate was 1.0 ml/min. Quantitative analysis was based on calibration graphs obtained with five different standard concentrations in suitable ranges for each application. Peak areas rather than heights were plotted against standard concentrations to construct the calibration graphs.

To determine the blank levels, five unexposed PTFE filters were extracted with ethanol–water as described above. The extracts were filtered and injected into the IC system for the measurement of anions and cations. The background levels in the analysis of rain water were evaluated by rinsing the "wet-only collector" with a volume of deionized water corresponding to the collected sample volume of 400 ml (median value). The blank rinse was collected in presoaked polyethylene containers and analysed for anions and cations by IC.

The detection limits listed in Table I were estimated according to VDI 2449/1 [5] from the blank levels and three times the standard deviation for deionized water in eight determinations:

$$x = x_0 + 3\,s_{x0}$$

where x = detection limit, x_0 = middle background level and s_{x_0} = standard deviation from n determinations. For the airborne dust particles from an average sampling volume of 40 m^3 the relative detection limit obtained are also given in Table I. The accuracy of the quantitative results for anions and cations was verified by analysis of artificial rain samples prepared according to procedures specified by VDI [6] and also with standard reference material (SRM 2694, NIST, Gaithersburg, MD, USA). The ranges of analyte recoveries were 88–125% and 88–94% for anions and cations, respectively.

TABLE I

DETECTION LIMITS FOR IONS MONITORED IN AIRBORNE DUST AND RAIN WATER

Ion	Dust ($\mu g/m^3$)	Rain water ($\mu g/l$)
Fluoride	0.005	200
Chloride	0.01	50
Nitrate	0.01	400
Bromide	0.01	400
Phosphate	0.02	750
Sulphate	0.02	750
Sodium	0.025	50
Ammonium	0.008	30
Potassium	0.02	40
Magnesium	0.005	20
Calcium	0.02	50

RESULTS AND DISCUSSION

Analysis of airborne dust particles

A typical chromatogram from anions, obtained after ethanol–water extraction of dust particles collected on filters inside the dust samplers, is shown in Fig. 1. The chromatogram illustrates a satisfactory separation of all analytes of interest within 18 min. The separation results for cations are shown in Fig. 2. The peaks for the two main components, ammonium and sodium, were not completely resolved. We found, however, that this did not affect the precision of peak area determination. The precision of the results for these two cations was the same as for the other cations. The separation of ammonium, alkali metal and alkaline earth metal cations was accomplished in only 12 min.

The results of anion monitoring during 1991 will be discussed first. Fig. 3 illustrates the time dependence of the sulphate concentrations found in total dust particles during the winter months. During the summer only a few measurements were carried out

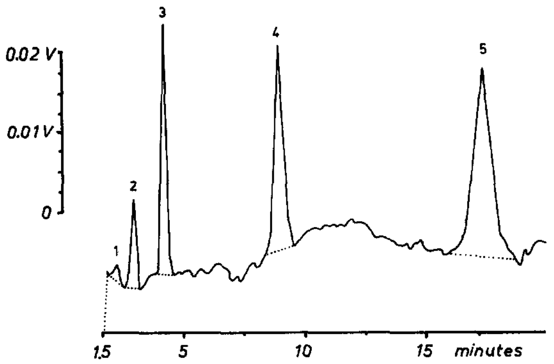

Fig. 1. Ion chromatographic separation obtained from a dust sample. Column, IC Pak A HC (10 μm); eluent, 1.3 m*M* gluconate–borate; pH, 8.5; flow-rate, 2.0 ml/min; detection, non-suppressed conductivity; sample, 50 μl dust ethanol–water extract. Peak identities and concentrations: 1 = fluoride, 0.33 ppm; 2 = hydrogencarbonate; 3 = chloride, 3.03 ppm; 4 = nitrate, 7.93 ppm; 5 = sulphate, 12.42 ppm.

for comparison with the winter period. The summer levels of sulphate are not shown in Fig. 3. One bar represents the sulphate content absorbed by non-fractionated airborne dust particles during 48 h (during February 20–28th the sampling device was faulty).

The range of fluctuations of sulphate concentrations illustrated in Fig. 3 is similar to those of the total mass content of dust particles in the air and to the magnitude of changes in heavy metal cations found in the same dust particles. The uneven distributions of levels of dust particles and of the corresponding ion concentrations are caused by meteorological factors on the one hand and by changes in the industrial emission on the other.

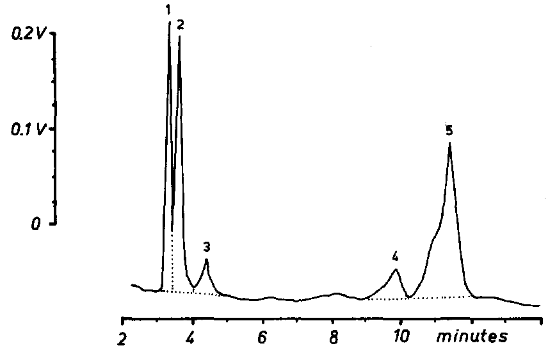

Fig. 2. Typical separation of cations extracted from a dust sample. Column, IC Pak C M/D (5 μm); eluent, 3.0 m*M* HNO_3–0.1 m*M* EDTA; flow-rate, 1.0 ml/min; detection, non-suppressed conductivity; injection volume, 10 μl. Peak identities and concentrations: 1 = sodium, 4.5 ppm; 2 = ammonium, 3.87 ppm; 3 = potassium, 0.47 ppm; 4 = magnesium, 0.58 ppm; 5 = calcium, 8.8 ppm.

TABLE II

MEAN MONTHLY TOTAL DUST LOAD AND ITS WATER-SOLUBLE ANION CONTENT COLLECTED IN HALLE IN 1991, USING THE ICP 5

Month (1991)	*n*	Total dust (μg/m^3)	Water-soluble ion content (μg/m^3)		
			Chloride	Nitrate	Sulphate
January	6	243.5	8.1	10.8	47.6
February	10	272.2	7.8	13.8	42.9
March	15	214.9	4.7	14.4	29.9
April	15	168	3.6	8.5	33.1
June	8	54.7	1.3	4.1	9.9

A comparison of montly average concentrations of anions is presented in Table II. As expected, the anion concentrations and dust load are higher during the winter months than in summer. This represents clear evidence of the contributing role of domestic coal-burning furnaces to the pollution of air by dust and inorganic contaminants.

In the following we discuss the dust fractions collected using the ICP 5. Fig. 4 and Table III illustrate the distribution of three anions among the collected

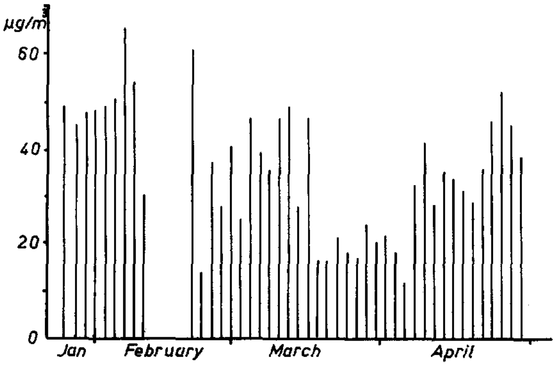

Fig. 3. Sulphate content in unfractionated dust samples collected using the ICP 5 in 1991 in Halle.

TABLE III

TWO TYPICAL EXAMPLES (WINTER AND SUMMER) OF ANION DISTRIBUTIONS (mg/l) IN DIFFERENT PARTICLE SIZE FRACTIONS OF AIRBORNE DUST

Period	Ion	Particle size (μm)					
		0–3.5	0–6	0–10	0–17	0–25	Total dust
Winter (2–3 Feb. 1991)	Chloride	9.1	10.2	10.3	10.2	10.4	10.4
	Nitrate	8.4	9.3	9.5	10.9	10.9	11.2
	Sulfate	46.9	49.0	49.2	49.0	49.0	49.2
Summer (18–20 June 1991)	Chloride	0.3	0.4	0.5	0.4	0.4	0.4
	Nitrate	2.7	2.9	2.9	3.2	3.5	3.6
	Sulfate	4.0	4.6	4.6	4.7	4.7	6.2

particle size fractions. It is shown that in winter 95% of sulphate, 87% of chloride and 75% of nitrate are found in particles having diameters smaller than 3.5 μm. This is in a good agreement with the results of monitoring heavy metal levels in dust [7]. The very small size of the dust particles carrying the largest portion of ionic contaminants facilitates deep deposition of toxic substances in the alveolic system of the lungs.

The simultaneous assays of anions and cations were performed in June 1992. These values were utilized for, among other things, calculations of the ionic mass balance in the dust samples obtained. The montly averages (n = 29) and the minimum and maximum values found for each of the ions measured are presented in Table IV. The ion ratio $\varepsilon_{\text{cations}}:\varepsilon_{\text{anions}}$, calculated from the equivalent concentrations listed in Table IV, is 1.17. This imbalance is not surprising, as the method employed for anions does not provide a complete value for silicates, carbonates and organic acids. Also not included in our mass balance calculations is the concentration of hydronium ions.

Table IV shows sulphate and ammonium ions to exhibit the highest concentration in airborne particles at the sampling sites. In view of the additional results presented in Fig. 5 and in Table V, it is possible to conclude that ammonium sulphate is followed by calcium sulphate as the main components

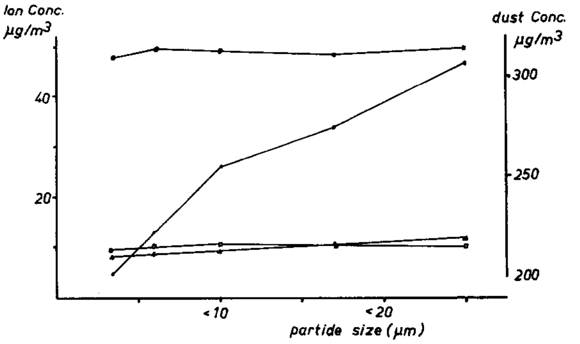

Fig. 4. Distribution of anion concentrations in different particle size fractions of dust. ● = Dust; ■ = sulphate; △ = nitrate; □ = chloride.

TABLE IV

AVERAGE MASS AND EQUIVALENT CONCENTRATIONS OF IONS IN DUST COLLECTED IN HALLE IN JUNE 1992 USING THE GS 50/3

Ion	Monthly average		Minimum (μg/m³)	Maximum (μg/m³)
	(μg/m³)	(nmol/m³)		
Fluoride	<0.05			
Chloride	0.33	9.3	0.10	0.97
Nitrate	2.72	43.6	0.74	9.83
Sulphate	9.37	195.1	3.14	19.80
$\varepsilon_{\text{anions}}$		248.0		
Sodium	0.32	13.9	0.03	1.27
Ammonium	2.67	146.7	0.69	5.17
Potassium	0.33	8.4	0.02	1.11
Calcium	2.17	108.2	0.88	4.43
Magnesium	0.13	10.7	0.01	0.33
$\varepsilon_{\text{cations}}$		287.9		

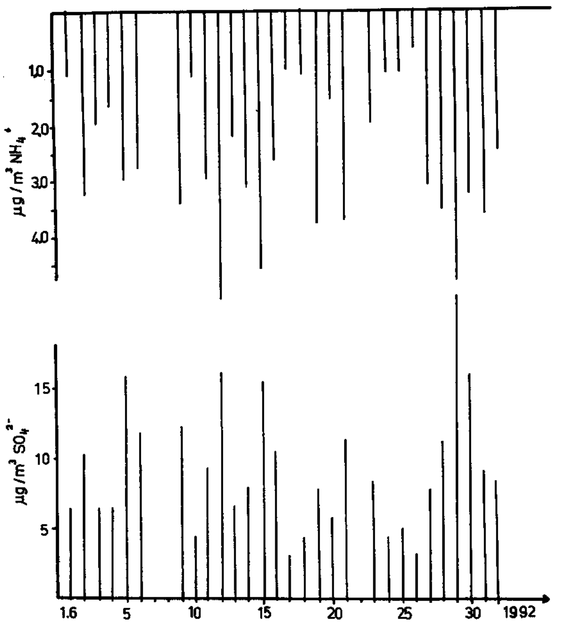

Fig. 5. Mutual dependence of sulphate and ammonium concentrations in airborne dust particles collected in 1991 in Halle.

of the airborne dust in this area. These conclusions agree also with some previously published data [9,10].

Comparisons enable us to identify the source of ammonium sulphate-containing dust. During the winter, there is a clear dependence between the mass of total dust load on the one hand and the ammonium, sulphate and chloride concentrations on the other (see also Table V). These observations help to identify the individual household furnaces, using salt- and sulphur-rich coal for fuel, as the source of inorganic ions in dust particles.

TABLE V

CORRELATIONS BETWEEN COMPONENTS IN WINTER AEROSOLS

Correlation factor		Parameters		
X	*Y*	*f*	*r*	*P*
Dust	Sulphate	42	0.53	0.99
	Ammonium	27	0.42	0.95
	Chloride	40	0.34	0.95
	Calcium	27	0.24	
	Sodium	27	0.10	
Sulphate	Ammonium	27	0.89	0.99
	Calcium	27	0.58	0.99
Chloride	Sodium	27	0.64	0.99

Table VI shows a comparison of ion concentrations in airborne dust determined in several German cities. A comparison of the ion concentrations that we determined with values published by other workers is difficult because the sampling periods were rarely identical. The concentration levels determined by us in Halle can probably be considered similar to those obtained in Berlin. In comparison with cities in southern Germany, Halle is burdened by air pollution having clearly higher levels of anthropogenic air components, such as total dust, chloride, nitrate and sulphate. The same is also true for cations in dust, *i.e.*, ammonium and calcium.

TABLE VI

COMPARISON OF ION CONCENTRATIONS IN DUST FROM SEVERAL GERMAN CITIES

City	Year	Sampling period	Ref.	Dust (μg/m³)	Mean ion concentration (μg/m³)							
					Na^+	NH_4^+	K^+	Ca^{2+}	Mg^{2+}	Cl^-	NO_3^-	SO_4^{2-}
Berlin	1987	Jan.–Dec.	8	98	1.0	4.7	0.6	3.1	0.3	2.1	7.5	16.5
Berlin	1988	Jan.–Dec.	9	79	0.8	3.8	0.4	2.3	0.2	1.4	5.5	11.7
Mannheim	1988	Jan.–Dec.	10	36	0.5	2.0	0.3	0.4	0.1	<0.2	1.3	6.7
Stuttgart	1988	Jan.–Dec.	10	33	0.4	1.4	0.2	0.8	0.1	<0.2	1.0	4.8
Halle	1991	Jan.–July	This work	211						5.3	11.7	35.5
Halle	1991	June–July	This work	55						1.3	4.1	9.9
Halle	1992	June–July	This work	66	0.3	2.6	0.3	2.2	0.1	0.3	2.7	9.4

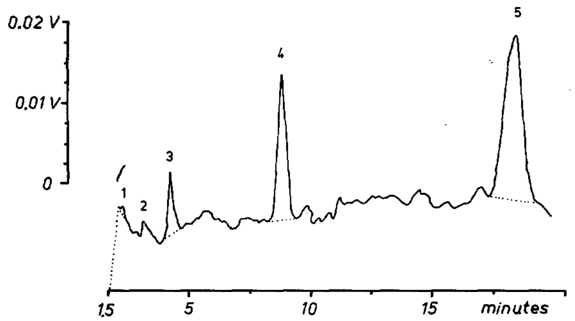

Fig. 6. Anion chromatogram of rain water sample. Conditions as in Fig. 1. Peak identities and concentrations: 1 = fluoride; 2 = hydrogencarbonate; 3 = chloride, 0.42 ppm; 4 = nitrate, 3.09 ppm; 5 = sulphate, 6.65 ppm.

TABLE VII

SUMMARY OF RESULS FROM MONITORING RAIN WATER SAMPLES COLLECTED BETWEEN FEBRUARY AND JUNE 1992

Parameter	Average	Minimum	Maximum
Rain volume (ml)	715	184	2100
Conductivity (μS/cm)	44.1	25	105
pH	4.7	4.2	6.5
Ion concntrations (mg/l):			
Chloride	0.8	<0.2	3.4
Nitrate	3.8	1.7	9.3
Sulphate	9.2	3.4	29.7
Sodium	0.4	0.2	2.4
Ammonium	1.4	0.6	3.3
Potassium	0.14	0.02	0.53
Magnesium	0.24	0.11	0.74
Calcium	2.38	0.74	7.30

Analysis of rain water

The chromatograms in Figs. 6 and 7 are typical for our results obtained with rain samples. They were obtained by injecting a sample of rain from June 1992 into the ion chromatograph. Both chromatograms show sufficient sensitivity and separation for all rain water samples, including the only lightly contaminated samples collected during the summer months. The most important results from the rain water monitoring are summarized in Table VII.

In a similar fashion to the dust samples, the rain water samples show differences in the levels of ions during the water and summer periods. The time dependence of ion levels in rain water is presented in Table VIII.

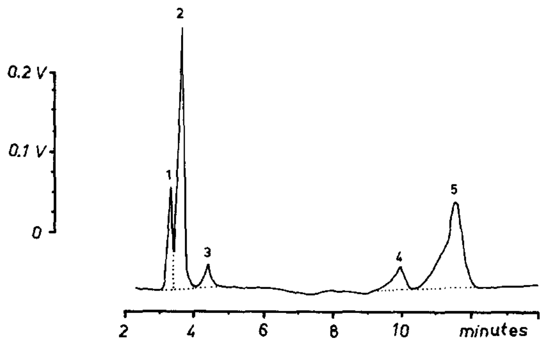

Fig. 7. Typical separation of cations found in rain water samples. Conditions as in Fig. 2. Peak identities and concentrations: 1 = sodium, 0.27 ppm; 2 = ammonium, 0.97 ppm; 3 = potassium, 0.08 ppm; 4 = magnesium, 0.13 ppm; 5 = calcium, 0.83 ppm.

Another similarity between the dust and rain water samples is the predominance of ammonium, calcium and sulphate ions. As with dust, ammonium sulphate and calcium sulphate seem to be the main components also in rain water (see Fig. 8). For comparison, Fig. 8 includes not only the values obtained from rain water, but also the data from the dust monitoring discussed in the preceding section. The similarity of the dust and rain water results is not surprising and represents a strong indication of the contribution of individual coal-burning furnaces not only to dust pollution but also to ionic contamination of airborne aerosols.

The ionic mass calculations for rain water yielded well balanced cation to anion ratios ($\varepsilon_{cations}$:$\varepsilon_{anions} \approx$ 1) over the entire monitoring period. The relatively small number of measurements, however, do not allow further interpretation. We also feel that a comparative interpretation of the analytical results for rain water and dust obtained in this study is still problematic owing to differences in sampling techniques and atmospheric conditions.

The air pollution situation can be further illustrated by comparing the results of our monitoring study with those obtained by other workers in different regions of Germany (see Table IX). As already mentioned in the discussion about dust, the

TABLE VIII

TIME-DEPENDENT CHANGES IN ION CONCENTRATIONS IN RAIN WATER COLLECTED IN HALLE BETWEEN FEBRUARY AND JUNE 1992

Month	n	Cl^-	NO_3^-	SO_4^{2-}	Na^+	NH_4^+	K^+	Mg^{2+}	Ca^{2+}
February	2	2.4	7.3	17.4	1.35	2.12	0.36	3.95	0.5
March	1	2.5	7.1	11.3	0.67	1.83	0.36	2.66	0.34
April	5	0.3	3.2	9.2	0.23	1.35	0.11	3.18	0.22
May	5	0.3	3.1	6.4	0.28	1.11	0.06	1.72	0.2
June	3	0.3	2.7	7.1	0.32	1.25	0.12	1.02	0.16

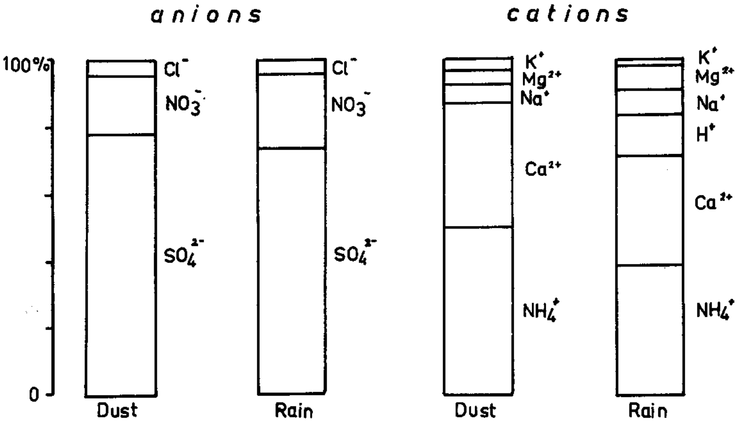

Fig. 8. Distributions of the most important anions and cations in dust and rain water.

TABLE IX

COMPARISON OF ION CONCENTRATIONS IN PRECIPITATIONS IN DIFFERENT GERMAN REGIONS

Region	Sampling period	Ref.	Ion concentration (mg/l)					
			Cl^-	NO_3^-	SO_4^{2-}	NH_4^+	Na^+	K^+
Germany (ranges)	Aug. 79–July 81	11	0.6–5.0	1.8–3.5	3.0–6.6			
Stollberg (Jülich)	Jan.–Dec. 83	12	1.5–1.7	3.1	5.1–6.0			
Kolmberg/Odenwald	May 88	13	1.2	9.0	9.5	2.7	1.0	1.17
Halle	Feb.–June 92	This work	0.8	3.8	9.2	0.4	1.4	0.14
Winter levels								
Jülich	Jan. 89	14	2.1	4.4	6.6	2.6	1.0	0.16
Essen	Jan. 89	14	1.9	4.1	5.2	1.2	0.7	0.2
Dortmund	Dec. 88	14	3.1	2.1	3.3	0.7	1.3	0.12
Hamburg	Jan. 89	14	6.8	4.3	7.1	1.0	2.9	2.09
Halle	Febr. 92	This work	2.4	7.3	17.4	2.1	1.4	0.36

values obtained in this work cannot be compared in detail with those published elsewhere. The 6-month averages obtained in Halle appear to be in general agreement with 12-month ionic averages obtained in other parts of Germany. A more differentiating comparison, however, shows distinctly higher levels of anthropogenic pollution in Halle, especially in February and other winter sampling periods. The anthropogenic contamination is especially recognizable by elevated levels of SO_x and NO_x emissions during the household heating periods. This leads to an acidic pH (*ca.* 4.2) and to an increased sulphate and nitrate load in natural precipitation.

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