Chemical Composition of the Surface Layer of Atmospheric Aerosol of the Baikal Region Studied by Means of Secondary Ion Mass Spectrometry

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Abstract

Elemental and phase composition of the surface layers of two kinds of atmospheric aerosol sampled in Irkutsk, Listvyanka settlement and at the mountainous station Mondy was investigated by means of secondary ion mass spectrometry. The first kind includes the aerosol collected by pumping air through the filters, the second one includes aerosol particles captured while snow was falling, and afterwards collected on filters when pumping the melted snow. It was established that the action of water causes washing out Na, Ca and Ti from the surface layers and the formation of hydroaluminosilicates. Such an effect of water allows assuming that large range of scattering of the element concentrations in the surface layers of airborne aerosol particles is due to the action of atmospheric humidity, which depends on the residence time of aerosol in the atmosphere.

INTRODUCTION

Extensive attention which is being paid at present to the investigation of chemical composition of atmospheric aerosol (AA) particles is due to their participation in a number of the most important processes taking place in the atmosphere. These processes may include scattering and absorption, both of the direct sunlight and of the ascending heat radiation; absorption of many other atmospheric components as adsorption and condensation of water vapour, various gases, metal compounds, *etc.* [1, 2]. The surface of AA can serve as a catalyst of various photo chemically stimulated reactions. It is assumed in a number of recent papers that many photocatalytic reactions can proceed on the surface of solid aerosol particles in the troposphere under the action of light, thus providing substantial impact on global chemical processes in the atmosphere [3–5]. This stimulated our desire to investigate chemical composition of the surface layers of AA by means of secondary ion mass spectrometry (SIMS), because this procedure allows investigating the distribution of composition along the depth from the surface into the volume [6, 7]. Unlike other methods used for the analysis of aerosol [8], SIMS is distinguished by the fact that it is able to register all the major elements of atmospheric aerosol brought about by soil erosion: Si, Al, Ca, Fe, Ti, Mg, Na, K, *etc.*

It is well known that the chemical composition of tropospheric aerosol is determined by its origin and various transformations under the action of atmospheric factors during transport: temperature, humidity, sunrays, *etc.* The

formation of continental aerosol, with the particle size of several micrometers, occurs mainly due to weathering of the soil and rocks [1, 2]. The interaction of aerosol with clouds can strongly affect the composition of water-soluble compounds in aerosol because aerosol survives 10 cycles of condensation/evaporation as a mean, the lifetime per each cycle being 0.5−1 h [9]. During these cycles, the aerosol particles are subjected to the action of the atmosphere with increases humidity. It is natural that investigation of water-soluble admixtures of atmospheric aerosol attracts much attention [9]. At the same time, we are unaware of any studies of the composition of the surface layers of aerosol after the action of water on them. For instance, investigation of water-soluble part of AA of Siberia indicated that the cation

composition includes Na^+, K^+, Mg^{2+} and Ca^{2+} [8]. So, it may be assumed that the content of the indicated elements in the surface layer of AA can be substantially dependent on humidity, temperature and atmospheric residence time of the particles.

In the present work we study the composition of the surface layer of atmospheric aerosol of the Baikal region sampled at the stations of Limnological Institute, SB RAS, in Irkutsk, Listvyanka settlement and in the mountainous region Mondy. Two kinds of AA were investigated: the aerosol samples collected in winter 1998 by pumping the air through a filter, and the aerosol particles captured by falling snow in winter 2001. These AA particles were later on collected on filters by pumping the melted snow through them.

TABLE 1

Characterization of aerosol samples

| Atmospheric aerosol		Fresh snow		
Sample No.	Sampling date	Sample No.	Sampling date	Water volume, ml
		Irkutsk		
1	21−22.01.98			
2	22−23.01.98	231	28−29.10.01	400
3	28−29.01.98	238	1.12.01	245
4	17−18.01.98			
5	26−28.02.98	239	2.12.01	195
6	25−26.02.98	248	3.01.02	350
7	24−25.02.98	252	28.01.02	255
8	22−23.02.98	259	16−17.03.02	400
		Listvyanka		
16	21−22.01.98			
17	24−25.01.98	115	23.11.01 г.	200
18	20−21.01.98	119	2.12.01 г.	410
19	10−11.02.98	125	30.12.01 г.	350
20	9−10.02.98	129	10−11.01.02 г.	250
21	14−15.02.98	132	28−29.01.02 г.	215
22	18−19.02.98			
		Mondy		
12	9−12.10.98	57	29.10.01	400
13	16−19.10.98	58	11.11.01	315
14	20−30.11.98	59	21.11.01	400
15	1−5.10.98	60	11.12.01	400
16	19−23.10.98			
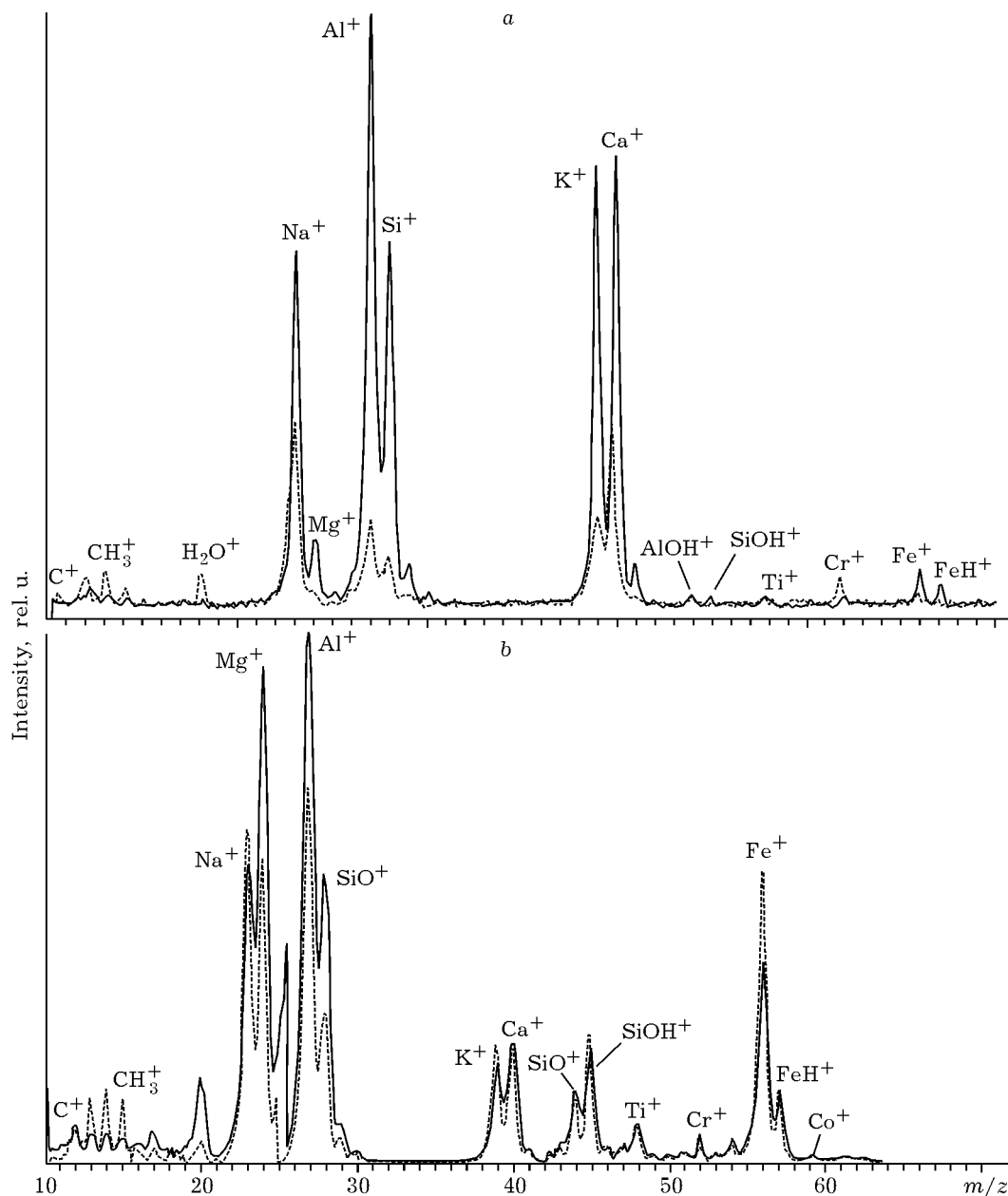
Fig. 1. Mass spectra of secondary ion emission of AA: a – sample No. 3, b – AAFS sample No. 252 (intensity of peaks with m/z = 10–25 is increased by a factor of 15, with m/z = 42–65 – by a factor of 5); *broken line* – spectrum of the 1st cycle (surface), *continuous line* – spectrum of the 5th cycle.

EXPERIMENTAL

Investigations of the elemental composition of the surface layers were carried out with MS-7201 instrument equipped with monopole mass analyzer with automated control, collection and processing system based on DVK-3 computer with CAMAC interface. The set-up and procedure of investigation were described in detail in [6]. It should be noted that the diameter of the primary ion beam falling at the angle of 45° to the holder surface normal was 2 mm; the area of bombarded surface was 5 mm². Residual gas pressure did not exceed $2\ 10^{-6}$ Pa. Bombardment was carried out with argon ions of 3 keV and current density $J = 6\ mA/cm^2$.

The AA samples were collected in different sites by pumping the air through Whatman filters for 1 to 3 days (Table 1). In Irkutsk, the

TABLE 2

Peaks observed in the mass spectra of secondary ion emission of AA and their identification

m/z	Ion	Ionization potential, eV	Designa- tion	m/z	Ion	Ionization potential, eV	Designa- tion
12	C^+	11.26	1				
13	CH^+	10.6	1				
14	CH_2^+	10.396	1				
	N^+	14.53		36	C_3^+	11.9	
15	CH_3^+	9.8	1		$^{36}Ar^+$		
	NH^+	13.1					
16	CH_4^+	12.98	1				
	NH_2^+	11.4	2	38	$C_3H_2^+$		
	O^+	13.61	*	39	K^+	4.34	*
17	NH_3^+	10.15	2		$C_3H_3^+$ cyclo	5.8	
	OH^+	13.18	**		AlC^+		
18	H_2O^+	12.61	**	40	Ar^+	15.76	
19	H_3O^+		**		$C_3H_4^+$	9.83. 10.2.	
	F^+	17.4			Ca^+	6.11	*
20	HF^+	15.77			SiC^+	9.2	
	Ca^{++}	11.87	*	41	$^{41}K^+$	4.34	*
	Ar^{++}	27.63		42	$^{42}Ca^+$	6.11	
23	Na^+	5.14	*	43	AlO^+	9.5	**
24	Mg^+	7.64	*		$^{43}Ca^+$		
	C_2^+	11.9		44	SiO^+	10.51.	**
	NaH^+				$AlOH^+$		**
25	C_2H^+	12.2	1		CO_2^+	13.79	
	$^{25}Mg^+$	7.64	*		$^{44}Ca^+$		
	$^{24}MgH^+$			45	$SiOH$		
26	$C_2H_2^+$	11.4	1		Sc		
	Mg^+	7.64	*	48	Ti^+	6.82	*
	CN^+	14.2			C_4		
27	Al^+	5.98	*	51	V^+	6.74	
	$C_2H_3^+$	9.5	1	52	Cr	6.76	*
	HCN^+	13.73		53	^{53}Cr		
28	Si^+	8.15	*	54	^{54}Fe		*
	$C_2H_4^+$	10.5	1		^{54}Cr		
	CO^+	14.01			Al_2		**
	N_2^+	15.58		55	Mn	7.43	
29	$^{29}Si^+$	8.15	*		$AlSi$		
	$C_2H_5^+$	8.38	1	56	Fe	7.89	*
	CHO^+	9.8			Si_2	7.4	**
	N_2H^+				CaO	6.5	**
	$^{28}SiH^+$			57	^{57}Fe		*
30	^{30}Si	8.15	*		FeH		**
	NO^+	9.27	2		$CaOH$		**
	$N_2H_2^+$	9.8			Si_2H		
	COH_2^+	10.86		58	Ni	7.63	*
	C_2H_6	11.5		59	Co	7.87	*
31	$N_2H_3^+$	7.9	2	60	Ni	7.63	
	SiH_3^+	8.0	*		SiO_2	11.7	**
	P^+	10.49		61	Ni		
	COH_3^+	10.72					
32	$N_2H_4^+$	8.74	2	63	Cu	7.72	*
	S	10.36		64	TiO	6.4	**
	CH_3OH^+	10.8		65	Cu	7.72	*
	O_2^+	12.075					

Note. Designations: see text.

TABLE 3

Coefficients of relative sensitivity of elements determined form the analysis of reference soil samples

Standard sample	Composition	Na	Mg	Al	K	Ca	Ti	Fe
ST-1a	Effusive rock	6.9	4.3	4.1	28.7	4.7	1.5	1.9
SSK-2	Soils polluted with industry-related elements	4.6	7.0	9.7	46.4	6.3	2.3	3.7
SKR-3	The same	6.1	5.5	11.3	20.3	7.8	1.4	2.0
CGKhM-1	Carbonate-silicate mellow deposits	3.2	2.5	8.1	20.9	3.0	1.4	2.4
CGKhM-2	The same, aluminosilicates	3.6	5.8	8.7	42.6	10.0	1.5	2.6
CGKhM-3	The same, carbonate-silicate	4.1	1.8	8.1	25.7	2.9	2.5	1.4
CGKhM-4	The same, aluminosilicates	3.9	8.5	10.8	38.4	12.8	1.7	2.6
	Mean	4.6	5.0	8.7	31.8	6.8	1.76	2.4
	Standard deviation	1.3	2.1	2.1	10.2	3.7	0.45	0.06

samples of fresh snow were collected in plastic basins mounted on the roof of Limnological Institute, in Mondy on the roof on container, in Listvyanka on the surface of tamped ground. Snow was collected during each snowfall. The snow was melted in enamel saucepans closed with caps, usually from evening till morning. After that, the volume of melted sample was measured. The melted water was pumped through filters. The solid particles were deposited on filters. For convenience, we will call these samples AAFS (atmospheric aerosol of fresh snow). The data on these samples are listed in Table 1.

The preparation of AA samples for analysis involved the following stages: the part of aerosol deposit was removed from the filters by scraping off with a scalpel, and then rubbed in the substrate made of high-purity indium (99.99999 %). No fibres of the filter remained on the substrate. The results of analyses of the substrate and filter samples without aerosol indicated that this procedure of the investigation of dielectric samples allows eliminating their charging under the ion beam and does not bring about any impurities. The particles of aerosol samples collected with fresh snow were deposited on indium holder as imprints by pressing the filter tightly to the indium surface.

RESULTS AND DISCUSSION

Investigation of the surface layers of aerosol was carried out by means of cycling registration of the spectra within the range $m/z = 10-120$. In each experiment, six consequent spectra were recorded within the indicated mass range; one spectrum was recorded for 170 s. Typical mass spectra of secondary ion emission (SIE) of AA and AAFS samples are shown in Fig. 1, *a, b*. Dotted line represents the first spectrum, and the continuous line represents the fifth spectrum observed after the ion current values reach the steady level. The last spectrum corresponds to the etching depth of ~40 nm. Since ion currents of the elements come over to the steady level at this depth, it may be assumed that these values correspond to the bulk concentrations of elements.

Identification of the peaks observed in mass spectra is explained in Table 2 where possible ions and sources of their formation are indicated for each m/z value.

In spite of simplicity of SIMS procedure, scattering and emission of charged particles,

which is the basis of this procedure, are too complicated for theoretical description, which creates definite difficulties for quantitative analysis, especially for multiphase and multicomponent materials. Thermodynamic model for the determination of concentration of an element in a multicomponent matrix (it is this model that allows carrying out the best semi-empirical description) shows that the logarithm of the ratio of secondary ion current to the concentration of an element is proportional to ionization potential [10]. Therefore, ionization potential values shown in Table 3 allow one to carry out sampling of the most probable ions for the major part of mass peaks. For example, the peak with $m/z = 27$ corresponds to Al^+; however, at the initial stage of ion etching $C_2H_3^+$ ion as a fragment of adsorbed hydrocarbons can make contribution into this peak. A similar conclusion can also be made about the peak with $m/z = 28$; its intensity is due mainly to the contribution from silicon, but at the initial stage of bombardment the $C_2H_3^+$ ion can be noticeable. Mass peaks are rather easily identified almost for all the main and extrinsic elements. Nevertheless, there are peaks, which can hardly be interpreted unambiguously, for example peak 56 in the case of low iron concentration.

The designations used in Table 2 are described below:

* − Ions, which should be attributed to scattering of the main and extrinsic elements of AA, because they are observed at any bombardment time: Si^+ ($m/z = 28, 29, 30$), Ca^+(40, 42), Al^+(27), Na^+(23), Mg^+(24, 25), K^+(39, 41), Fe^+(54, 56), Ti^+(48, 46, 47), Cr^+(52, 50, 53) and Co^+ (59).

** − Molecular ions, which are formed due to emission of fragments from lattice: AlO^+(43), $[AlOH^+ + SiO^+]$ (44), $SiOH^+$(45), $[FeH^+ + CaOH^+]$ (57).

1 − Ions formed during argon ion scattering of adsorbed hydrocarbons or organic species. A characteristic feature of the behaviour of these ions is a rapid decay of intensity (ion currents) because of rapid decrease in the concentration in the adsorbed layer [7]. Here it should be added that the intensities of $C_nH_m^+$ ions decrease with an increase in n. Indeed,

one can see in Fig. 1 that the intensity of $C_2H_2^+$ ion is much lower than that for CH^+ ion. Because of this, the contribution from these ions into the intensity of the ions of main elements is small, especially after ion bombardment for several seconds. For example, the contribution from $C_2H_3^+$ ions into the peak of ions with $m/z = 27$ can be neglected. A similar consideration is true for the peaks with $m/z = 28, 39, 40$ and so on.

2 − Ions, which are formed during bombardment of nitrogen-containing compounds. It should be noted that the mass spectrometer does not possess sufficient resolution to distinguish between the ions formed during scattering of hydrocarbon and nitrogen-containing compounds, for example CH_3^+ and NH^+, *etc.*, but the presence of N^+, $N_nH_m^+$, NO^+ ions in the spectra cannot be excluded, because trace amounts of NO, N_2O, NH_3 gases are present in the atmosphere [11].

3 − Emission of COH^+, COH_2^+, NO^+ ions is also due to adsorbed compounds. This conclusion follows from rapid decay of ion currents during bombardment (within the time of recording one spectrum). The intensity of these molecular ions decreases rapidly during bombardment. Comparing the SIE spectra shown in Fig. 1, *a* and *b*, one can see that the intensities of Na, K, Ca ions and, to a less extent, Ti and Mg for AA samples are substantially higher than for AAFS samples (with respect to the intensities of Al and Si ions). Quantitative analysis will further show that the relative concentration of the indicated elements in AAFS samples is much lower than in AA. This is undoubtedly due to the action of water on AAFS during sampling, that is, water washes out the compounds with these elements.

The intensity of molecular ions formed due to emission of lattice fragments, such as AlO, SiO, SiOH, CaOH, increases while etching proceeds; the very fact of emission of the oxide fragments allows assuming that aerosol is represented by silicate and aluminosilicate compounds. For AAFS samples, the peak of SiOH is more intensive than the peak of SiO. For AA samples, except some separate ones, the peak 44 is, quite contrary, larger than peak 45. We assume that the action of water

TABLE 4

Elemental composition of AA, %

Sample No.	Na	Mg	Al	Si	K	Ca	Ti	Fe	Al/Si
				Listvyanka					
17	10.0	1.7	12.2	54.2	1.2	13.1	1.8	5.2	1 : 4.4
18	3.1	2.4	12.4	46.0	1.8	21.0	7.2	5.5	1 : 3.7
19	33.7	1.8	8.0	28.9	10.0	14.5	1.0	2.2	1 : 3.6
20	1.9	2.9	14.5	63.2	1.1	11.4	1.6	3.3	1 : 4.3
21	7.9	2.9	13.1	39.1	2.8	26.4	2.8	4.2	1 : 3
22	10.1	2.7	40.3	31.5	2.0	6.1	0	6.3	1 : 0.8
23	5.7	2.1	19.3	54.8	1.8	13.4	0.6	2.0	1 : 2.8
				Irkutsk					
1	14.9	2.5	12.5	28.9	6.5	24.7	2.7	7.1	1 : 2.3
2	3.5	2.3	9.4	61.0	2.0	15.5	1.5	4.6	1 : 6.4
3	6.6	3.8	9.6	54.5	1.3	23.2	1.1	1.9	1 : 5.6
4	8.3	2.9	10.5	48.1	3.3	20.5	1.8	4.5	1 : 3.3
5	18.8	3.7	31.1	35.2	2.6	6.9	0.4	1.1	1 : 1.1
6	11.1	1.9	10.8	59.4	2.2	11.0	0.8	2.7	1 : 5.5
7	6.4	3.8	21.2	62.0	0.1	2.5	0.6	3.6	1 : 2.9
8	10.1	2.8	14.6	49.3	4.1	14.1	0.5	4.4	1 : 3.4
				Mondy					
12	5.1	3.7	13.4	55.3	2.3	15.5	2.5	2.1	1 : 4.1
13	19.1	2.4	7.6	19.5	10.8	29.3	2.0	9.3	1 : 2.6
14	9.8	3.2	23.7	26.6	4.9	23.4	4.3	4.0	1 : 1.1
15	4.7	2.0	7.7	28.8	1.9	36.5	0.1	18.4	1 : 3.7
16	1.5	3.8	14	70.6	1.8	2.3	1.1	4.8	1 : 5

on aluminosilicates can cause the formation of hydroaluminosilicates.

The procedure used for quantitative analysis in the present work is based on the fact that the current I_i of secondary ions of element i is proportional to concentration C_i and its coefficient of relative sensitivity γ_i:

$$I_i = k\gamma_i C_i \qquad (1)$$

Coefficients γ_i were determined using the same formula but basing on the results of analysis of reference materials with known concentrations. Relative concentration of an element with mass i was determined using equation

$$C_i = \frac{C_i^* \cdot 100}{1 + \sum C_i^*} \, (\%) \qquad (2)$$

where $C_i^* = (I_i / I_{Si}) / \gamma_i$, I_i and I_{Si} are ion currents of element with mass i and with the mass of silicon ($m/z = 28$), respectively, because γ_i for silicon is accepted to be equal to 1 in the calculations of relative concentrations. Relative concentration of Fe was determined from the intensity of peak $m/z = 56$ taking into account contributions from Si_2^+ and CaO^+ ions: $I(Fe^+) = I(m/z = 56) - 0.007I(28) - 0.002I(40)$, where $I(28)$, $I(40)$ are currents of the corresponding masses and the coefficients of them were determined on the basis of measurements of secondary ion emission of SiO_2 and CaO. It should be stressed that the correction is usually within the error limit.

Standard State Samples of soils CGKhM-1, CGKhM-2, CGKhM-3, CGKhM-4, SSK-2, SKR-3, ST-1A were used as reference samples [12]. Coefficients of relative sensitivity for the indicated soil standards are listed in Table 3. A brief characterization of the soils used is presented there, too. For instance, standards

CGKhM-1 and CGKhM-3 are mellow carbonate-silicate deposits, while CGKhM-2 and CGKhM-4 are mellow aluminosilicates deposits. It is natural that one cannot choose a reference material ideally corresponding in composition to the aerosol samples under investigation. Because of this, as a first approximation for quantitative analysis, we used the coefficients of relative sensitivity averaged over all the reference samples. Such an approximation allows one to carry out qualitative comparison of the chemical composition of surface layers (Tables 4 and 5). One can see that the AA samples collected on different days exhibit large scattering of relative concentrations of Na, K, Ca and Ti, while such a scattering is absent for the AAFS samples. Moreover, concentrations of these elements in AAFS samples are noticeably lower than those in atmospheric aerosol samples. It is natural to assume that these effects are caused by the influence of

water, which washes out the elements. It follows that large scattering of Na, K, Ca concentrations in AA is likely to be due to different action of atmospheric water on these samples, and therefore depends on atmospheric residence time of AA. Indeed, if we compare samples No. 1 and 2 or 13 and 15, or 18 and 19, we see that a decrease or an increase in relative concentration of sodium or potassium occur synchronously.

The composition of AA and AAFS samples averaged over several days is shown in Table 6 along with the mean terrestrial composition of soil. Analysis of these data shows that the AA contain increased amount of Na, Ca and Ti in comparison with the terrestrial mean values. At the same time, quite contrary, a decreased content of Na, K and Ca is characteristic of AAFS samples. It should be noted that an increased concentration of Ti and a decreased concentration of Mg are observed for all the samples (see Table 6).

TABLE 5

Elemental composition of AA, relative atomic concentration, %

Sample No.	Na	Mg	Al	Si	K	Ca	Ti	Cr	Fe	Co
Listvyanka										
L115	2.38	3.43	18.1	69	1.72	2.62	0.78	0	2.43	
L119	1.02	1.95	20	70	0.94	3.08	1.19	0	2.01	
L125	1.02	1.54	18.5	70	1.21	3.49	1.51	0	2.91	
L129	1.05	1.59	17.4	72	1.05	2.87	1.35	0	2.41	
L132	1.05	1.58	19	72	0.84	2.64	0.82	0	2.16	
Mean	1.3	2.02	18.6	70	1.15	2.94	1.13	0	2.38	
Irkutsk										
231	2.7	5.6	15	64.7	3.1	2.6	0.5	0.01	5.8	
238	2.1	1.6	14.5	76.8	0.8	1.2	0.8	0.02	3.7	
239	2.7	3.5	13.2	70.0	1.8	3.4	0.6	0.02	4.7	0.01
248	0.5	1.6	13.0	78.8	0.8	1.4	0.7	0.01	3.2	
252	1.0	1.4	20.1	67.1	0.7	3.9	0.8	0.04	3.9	0.01
259	2.6	3.6	12.3	71.0	0.5	3.6	0.75	0.03	5.6	0.001
Mean	1.9	2.9	14.7	71.4	1.3	2.7	0.7	0.02	4.4	
Mondy										
57	1.6	3.4	16	67.1	2.3	4.4	0.8	0.07	4.2	0.1
58	9.9	3.0	19.1	45.5	1.9	6.2	7.7	0.3	6.2	
59	1.5	5.2	13.7	64.3	3.1	4.5	1.8	0.02	5.7	0.03
60	1.0	5.9	14	65.6	3.4	3.0	1.9	1.4	3.5	0.15
Mean	1.4	4.8	14.6	65.7	2.9	4.0	1.5	0.5	4.5	0.09

TABLE 6

Elemental composition of AA and AAFS samples obtained by averaging the data presented in Tables 4 and 5

Sample	Na	Mg	Al	Si	K	Ca	Ti	Cr	Fe
				Listvyanka					
AA	10.3	2.6	17.1	45.4	3.0	15.1	2.1		4.1
AAFS	1.3	2.02	18.6	70	1.1	2.90	1.1	0	2.4
				Irkutsk					
AA	9.4	2.8	15.7	50.7	2.6	14.0	1.0		3.7
AAFS	1.9	2.9	14.7	71.4	1.3	2.7	0.7	0.02	4.4
				Mondy					
AA	7.4	3.1	13.5	41.8	4.0	20.3	2.1	0.3	7.4
AAFS	1.4	4.8	14.6	65.7	2.9	4.0	1.5	0.5	4.5
Clarke	6.4	6.0	17.0	56.6	3.7	5.1	0.5		4.6

It is necessary to stress that the chemical compositions of the surface of AA and AAFS samples for all the sampling sites (Itkutsk, Listvyanka and Mondy) are indistinguishable within the error limit, though Irkutsk is an industrial region, while Mondy is a pure mountainous region situated at the height of more than 2000 m. This is likely to be explained by the fact that the samples were taken in winter, in the presence of snow cover, and the source of aerosol is not localized in the vicinity of the indicated sites.

CONCLUSIONS

1. The action of water causes washing out of Na, Ca and Ti from the surface layers of AA and the formation of hydroaluminosilicates. Such a manner of water action allows assuming that large scattering of element concentrations in AA samples is due to the action of atmospheric humidity, which depends on the atmospheric residence time.

2. The composition of the surface layers of the winter atmospheric aerosol of the Baikal region (Irkutsk, Listvyanka, Mondy) averaged over several days is independent of sampling site. The composition differs from the mean terrestrial one by increased Na, Ca and Ti content.

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