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Urinary Perchlorate Exposure and Risk in Women of Reproductive Age in a Fireworks Production Area of China

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Abstract Perchlorate is used widely in fireworks, and, if ingested, it has the potential to disrupt thyroid function. The concentrations of perchlorate in water and soil samples and in urine samples of women of reproductive age from Liuyang, the largest fireworks production area in China, were investigated. The results showed that the average perchlorate concentrations in groundwater, surface water, farmland soil, and urine samples of women from the fireworks production area were significantly greater than those from the control area. The health risk of perchlorate ingested through drinking water was assessed based on the mode recommended by the United States Environmental Protection Agency. The values of hazard quotient of river water and groundwater in the fireworks production area were much greater than the safe level (=1), which indicates that adverse health effects may result from perchlorate when these sources of water are used as drinking water. These results indicated that the environment of the fireworks production area has been polluted by perchlorate and that residents were and are facing greater exposure doses of perchlorate. Fireworks production enterprises may be a major source of perchlorate contamination.

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Perchlorate is an oxidizer that has been used in many applications, including rockets, missiles, fireworks, matches, air bags, and road flares. As a competitive inhibitor of iodide, perchlorate can alter endocrine function of animals by inhibiting iodide uptake into thyroid tissue at the site of sodium-iodide symporter, thereby decreasing the production of thyroid hormones (Wolff 1998; Smith et al. 2004). For example, the United States Government Accountability Office (USGAO) reviewed 90 studies about the health risks of perchlorate published from 1998 to 2005 and concluded that one quarter of them indicated that perchlorate had an adverse effect on human health, especially on thyroid function (USGAO 2007). Moreover, perchlorate affects not only thyroid function but also that of other systems, such as the nervous, reproductive, and immune systems (Bernhardt et al. 2006; Gilbert and Sui 2008; USATSDR 2008).

Perchlorate was added to the drinking water contaminant candidate list by the USEPA in 1998 (USEPA 1998a). According to a report of United States government in 2005, 65 % of perchlorate contamination found in groundwater and surface water in the United States has been related to defense and aerospace activities (such as rocket testing, missile testing, or blasting treatment), which are the greatest known source of perchlorate contamination (USGAO 2005).

Perchlorate has also been widely using in the production of fireworks. Some studies have shown that fireworks displays resulted in perchlorate contamination of surface water, groundwater, and soil (Massachusetts Department of Environmental Protection [MDEP] 2007; Wilkin et al. 2007; Leung et al. 2013). Wilkin et al. found that perchlorate concentrations in surface water adjacent to a site of fireworks displays spiked to values ranging from 24 to 1,028 times the mean baseline value within 14 h after the fireworks (Wilkin et al. 2007).

China produces 90 % of the fireworks in the world. More than two thirds of fireworks factories are located in four places: Liuyang, Liling, Shangli, and Wanzai, the junction area of Hunan Province and Jiangxi Province. To investigate the pollution status of perchlorate in Liuyang, the largest fireworks production area in China, we measured concentrations of perchlorate in groundwater, spring water, surface water, and farmland soil. To understand the level of human exposure to perchlorate, we measured the concentrations of perchlorate in the urine of local women of childbearing age due to the facts that (1) perchlorate released into the environment could enter into the human body; and (2) 70 to 95 % of a perchlorate dose is excreted unchanged in the urine with a half-life of approximately 8 h (Blount et al. 2007). The potential health risk of perchlorate from drinking water for adults was also evaluated in this study. The method we used here was established by the National Academy of Sciences and was recommended by the USEPA and consists of four steps: hazard identification, dose–response assessment, exposure assessment, and risk characterization (National Research Council 1983). This is the first study to investigate the impacts of firework production areas on human health. Findings from the present study will inform local fireworks enterprises and women in the area about perchlorate contamination and associated health risks.

Materials and Methods

Study Site

The study was performed in Liuyang, which is known as the "Capital of Fireworks." It lies within Changsha, a city in Northeast Hunan Province in China, between 113°10′24″ and 114°14′58″ east longitude and 27°51′20″ and 28°34′06″ north latitude. Fireworks and firecrackers made in Liuyang are sold in >150 countries and regions, accounting for two thirds of the country's total export volume and 70 % of the global market share. In 2006, there were 1,058 fireworks enterprises in Liuyang, among which were 102 down-lead plants, 69 black powder plants, 150 fireworks operating companies, 10 group companies, 128 fireworks display enterprises, and 542 enterprises of fireworks raw materials. Four regions with different specialties have been formed: the Southern District is the main production area of small fireworks; the Northern District produces display shells; the Western District produces flower fireworks; and the Eastern District concentrates its production in cold light fireworks. Two towns in the fireworks production center in the Southern District were selected as the research area. Another agriculture and tourism-oriented town located at the northeast of Liuyang was selected as the control area.

Sample Collection

Samples of groundwater, spring water, surface water, and farmland soil were collected from seven villages (named R1, R2, R3, R4, R5, R6, and R7) in the research area and three villages (named C1, C2, and C3) in the control area (Fig. 1). Groundwater samples were taken from groundwater wells (at depth from 5 to 10 m); samples of spring water were collected from the springs used as sources of drinking water; and samples of surface water were collected from local rivers and lakes. There was no wastewater discharge near these sampling sites. All samples of water were kept in Teflon containers in a freezer at −20 °C until analysis was performed. Soil samples were collected below the surface of the ground located in farmland at least 300 m from rail lines or highways. All soil samples represented a well-proportioned mixture of the sites and were put into plastic bags and stored in a freezer at −20 °C.

Urine samples were collected from local women of reproductive age (age 20–45 in this study). All of the subjects are Han Chinese. They signed an informed consent before participation and were healthy and not pregnant. Urine samples were put into Teflon containers and stored in a freezer at −20 °C before perchlorate analysis.

Sample Preparation

Water samples were filtered through a 0.22-μm filter before analysis. Soil samples were freeze-dried and homogenized by sifting through a stainless steel 40-mesh (0.35-mm) sieve. Then 2.0 g of homogenized samples were placed in individual 50-mL flasks, and 20 mL of deionized water was added to each flask. The flasks containing soil and water were shaken at a speed of 200 rpm for 3 h at room temperature. Then the turbid soil–water mixture was centrifuged for 25 min at 6,000 rpm. The supernatant solution was decanted and filtered through a 0.22-μm nylon membrane, an OnGuard H pretreatment cartridge (Dionex, US) and, last, an OnGuard RP pretreatment cartridge (Dionex, US) before perchlorate anion (ClO_4^-) determination.

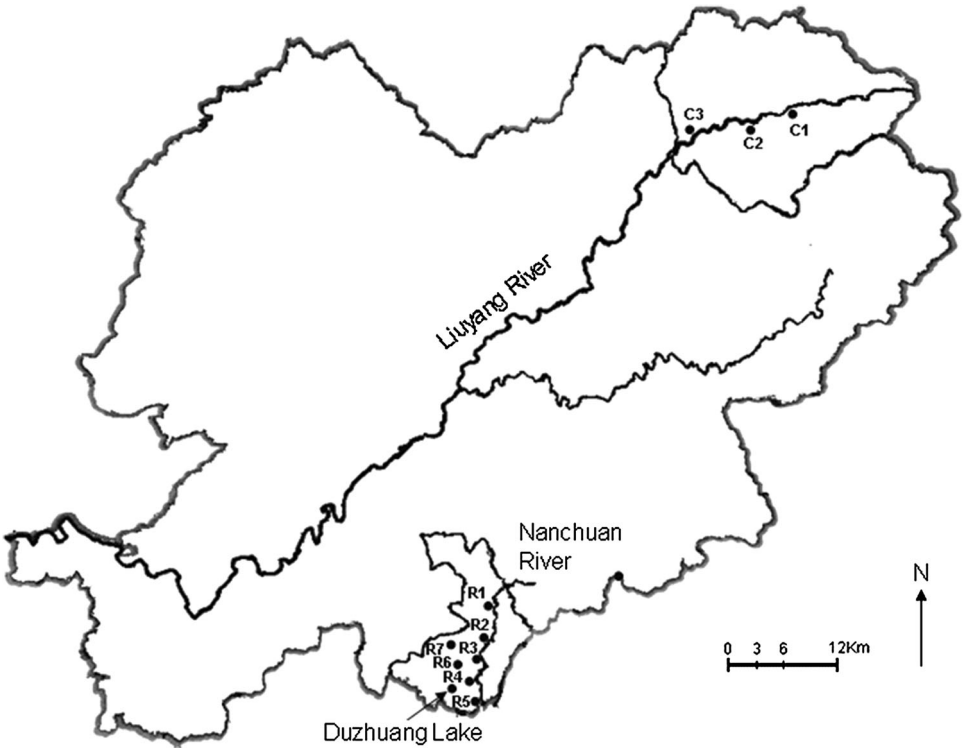

Fig. 1 Sampling locations in Liuyang, Hunan Province, China. *R1–R7* were the sampling villages in the research area, and *C1–C3* were the sampling villages in the control area. Nanchuan River and Duzhuang Lake are the main surface waters in the research area. The sampling sites of the former are located at *R1*, *R2*, *R4*, and *R5*, and those of the latter are located at *R5* and Duzhuang Lake. Liuyang River is the main surface water in the control area, the sampling sites of which located at *C1–C3*

Urine samples were diluted 10 times by deionized water. The diluents were filtered through a 0.22-μm nylon membrane and an OnGuard RP pretreatment cartridge (Dionex, US) before measurement of ClO_4^-.

Instrumental Analysis

A Dionex ICS-3000 ion chromatograph coupled with an electrospray tandem mass spectrometer (ESI-mass spectrometry [MS]-MS) (ABI US) was used to determine ClO_4^- concentration in accordance with the method reported by Shi et al. (2007). The concentration of urinary creatinine was detected by the spectrophotometric method recommended by the National Health and Family Planning Commission of the People's Republic of China (NHFPC PRC) (NHFPC PRC 1997).

Health Risk Assessment Method

Inhalation, ingestion, and dermal absorption are the major routes by which a chemical can cross the boundary from outside to inside the body. Perchlorate is easily absorbed from the gastrointestinal tract. It does not pass readily through the skin. Uptake of inorganic ions such as perchlorate is frequently <1 % through the skin. Because perchlorate is less volatile at room temperatures, exposure by way of inhalation to fumes or vapors is expected to be negligible and that by particles would depend on the particle size (USEPA 1998b). In this study, the health risk of perchlorate entering the human body by drinking water was calculated. The potential noncarcinogenic effect was evaluated by comparing an exposure level during a specified time period (e.g., lifetime) with a reference dose (RfD) derived for a similar exposure period (USEPA 1989) (e.g., lifetime). This evaluation is based on the assumption that there is a level of exposure (i.e., RfD) below which it is unlikely for even sensitive populations to experience adverse health effects. The ratio of exposure to toxicity is called the "hazard quotient" (HQ) (Eq. 1). A value >1 indicates there may be adverse health effects. The RfD of perchlorate given by Integrated Risk Information System of the USEPA is 0.0007 mg/kg/day. The exposure dose of perchlorate ingested through drinking water can be estimated using Eq. 2 as recommended by the USEPA (USEPA 1989).

$$\mathrm{HQ} = \frac{\mathrm{CDI}}{\mathrm{RfD}} \tag{1}$$

$$\mathrm{CDI} = \frac{\mathrm{CW} \times \mathrm{IR} \times \mathrm{EF} \times \mathrm{ED}}{\mathrm{BW} \times \mathrm{AT}} \tag{2}$$

where CDI is the chronic daily intake (mg/kg/day), *i.e.*, the average daily dose; CW is the chemical concentration in water (mg/L); IR is the ingestion rate of water (L/day); EF is the exposure frequency (days/years); ED is the exposure duration (years); BW is the body weight (kg); and AT is the averaging time (period during which exposure is averaged—number of days).

Statistical Methods

Perchlorate concentrations falling below analytical detection limits were assigned a value of zero for statistical analysis. Before comparison of means, all data were checked for normality and heteroscedasticity. Nonparametric statistics were used for comparison of means when heterogeneity of variance appeared. Statistical tests were considered significant for $p < 0.05$.

Results and Discussion

Perchlorate Concentrations in Waters and Soils

Nineteen groundwater samples, 7 spring water samples, and 21 soil samples were collected from 7 villages in the research area, and 9 groundwater samples, 3 spring water samples, and 9 soil samples were collected from 3 villages in control area. Surface water samples collected from the main rivers and lakes in the research and control areas numbered 6 and 3, respectively. Average perchlorate concentration in groundwater at the research area ranged from 12.2 to 182.3 μg/L with a mean value 49.1 ± 64.8 μg/L, which is much greater than that at the control area, which ranged from 0.6 to 4.7 μg/L with a mean value 2.7 ± 4.1 μg/L ($p < 0.05$) (Table 1). The highest perchlorate concentration (>182 μg/L) in groundwater was detected at R6, most probably due to its proximity to a large fireworks production enterprise. The average concentration of perchlorate in spring water from the research area was 3.1 ± 2.3 μg/L, which is approximately 22 times greater than that from the control area; however, the difference is not statistically significant ($p > 0.05$). The average perchlorate concentration of surface water at the research area was 115.6 ± 58.7 μg/L, which is 46.2 times greater than that of the control area ($p < 0.05$) (Table 2). Moreover, perchlorate concentrations at the downstream parts of Nanchuan River and Duzhuang Lake were 211.0 and 163.0 μg/L, respectively; both were greater than that of the upper reaches (Table 2). The average concentration of perchlorate in the soil samples from the research area was 9.1 ± 12.4 μg/kg dry weight, which was approximately 26 times greater than that from the control area ($p < 0.05$) (Table 1).

Concentrations of perchlorate in groundwater, surface water, and soil at the research area were all greater than that at the control area. This indicated that there was contamination of perchlorate in the research area. Perchlorate is an ingredient used in fireworks manufacturing. During the manufacturing process of fireworks and firecrackers, oxidants are the main fireworks pharmaceutical ingredients, accounting for approximately 50–75 % of the pollution. Chlorate, perchlorate, nitrates, and metal oxides are oxidants often used in fireworks agents. Because potassium chlorate was banned for use in the production of

Table 1 Perchlorate concentrations in groundwater and soil samples

Site	Groundwater (μg/L)		Soil (μg/kg)	
	n	Mean ± SD	*n*	Mean ± SD
Research area				
R1	2	41.0 ± 21.1	3	2.5 ± 3.7
R2	3	14.9 ± 16.2	3	0.5 ± 0.6
R3	3	17.8 ± 8.3	3	14.2 ± 11.2
R4	3	24.5 ± 24.6	3	4.7 ± 5.1
R5	3	35.7 ± 24.7	3	20.3 ± 19.3
R6	3	182.3 ± 59.6	3	14.6 ± 21.9
R7	2	12.2 ± 0.3	3	7.1 ± 6.4
Total	19	49.1 ± 64.8[a]	21	9.1 ± 12.4[d]
Control area				
C1	3	2.6 ± 4.2	3 (1)[c]	0.6 ± 0.6
C2	3	4.7 ± 6.0	3	0.4 ± 0.4
C3	3	0.6 ± 0.4	3 (2)[c]	0.1 ± 0.1
Total	9	2.7 ± 4.1[b]	9	0.4 ± 0.4[e]

[a, b] Significant differences between areas as determined by ANOVA ($p < 0.05$)

[c] Number of nondetects

[d, e] Significant differences between areas as determined by ANOVA ($p < 0.05$)

Table 2 Perchlorate concentrations in surface water samples

Site	*n*	Mean (± SD)
Research area		
R1[a]	1	73.9
R2[a]	1	70.2
R4[a]	1	71.3
R5[a]	1	104.0
Duzhuang Lake[b]	1	163.0
R5[b]	1	211.0
Total	6	115.6 (±58.7)[d]
Control area		
C1[c]	1	4.4
C2[c]	1	1.0
C3[c]	1	2.1
Total	3	2.5 (± 1.7)[e]

[a] Sampling sites located along Nanchuan River

[b] Samples collected from Duzhuang Lake. Site R5[b] was located at the branch of Duzhuang Lake

[c] Sampling sites located along Liuyang River

[d,e] Significant differences between areas as determined by ANOVA ($p < 0.05$)

fireworks by the Chinese government in 2002, potassium perchlorate has become the preferred alternative and has been used with greater frequency. Along with the discharges of scrap materials and wastewater generated during the process of fireworks production, perchlorate can be released into the environment. In addition, perchlorate can also be released during the process of developing new products, testing, and product-quality inspections. The research area and the control area are both in Liuyang. The main difference between them is that fireworks production is the economic pillar industry in the former, whereas the economic pillar of the latter is agriculture and tourism. Therefore, manufacturers of fireworks and firecrackers may be the major source of perchlorate pollution in the research area. Similarly, an investigation performed by Isobe et al. in South India found also that concentrations of perchlorate in groundwater were significantly greater in a fireworks factory area than in other locations, which indicated that the fireworks and safety match industries are the principal sources of perchlorate pollution (Isobe et al. 2013).

Two previous studies showed that perchlorate is widespread in certain areas of China. Perchlorate was determined to be found in the samples of sewage sludge, rice, bottled drinking water, and milk collected from most regions of China in one study (Shi et al. 2007). In another study, the average concentrations of perchlorate in tap water, groundwater, surface water, and bottled water collected from 15 locations in 13 provinces and municipalities in China were 2.46, 3.04, 2.82, and 0.22 μg/L, respectively (Wu et al. 2010). Compared with results from this study, perchlorate concentrations of groundwater and surface water in the control area in our study were similar to these data, whereas those in the research area were much greater. USEPA has proposed a interim drinking-water health advisory level of 15 μg/L (USEPA 2008), and two states, Massachusetts and California, have also established enforceable standards for perchlorate in drinking water, which is much stricter than the interim drinking-water health advisory level, 2 and 6 μg/L, respectively (Massachusetts Department of Environmental Protection 2006; California Environmental Protection Agency 2004). Average concentrations of perchlorate in groundwater and surface water from the research area were all greater than the interim drinking water health advisory level of USEPA, whereas that of spring water at both areas were below this level.

Concentrations of Urinary Perchlorate

Human exposure to perchlorate most likely occurs after ingestion of contaminated water and food (Borjan et al. 2011). Concentrations of urinary perchlorate can be used to indicate the exposure dose of perchlorate because urine is the principal route by which nonlactating humans excrete perchlorate (Blount et al. 2007). In this study, 52 urine samples were collected from women of reproductive age living in the research area. Half of them were from women worked in fireworks industries and classified as an occupational group. The others were classified as a nonoccupational group. Forty-nine urine samples collected from women living in the control area were used as a control group. None of the women worked in fireworks industries. Concentrations of urinary perchlorate were presented both as micrograms per liter and as micrograms per gram of urinary creatinine to allow for comparisons between different demographic groups and adjustment for differences in urinary dilution (Barr et al. 2005). In fact, women working in fireworks industries have more opportunities for perchlorate exposure, including dermal exposure and inhalation of airborne particulate. However, the average urinary perchlorate concentration of the occupational group was not significantly greater than that of the nonoccupational group ($p > 0.05$): 314.2 ± 411.0 μg/L (442.7 ± 532.4 μg/g creatinine) versus 171.5 ± 202.4 μg/L (278.8 ± 401.3 μg/g creatinine), respectively. One reason for this might be that perchlorate does not enter into the body easily through dermal exposure, which decreases the absorption by dermal contact during working. As listed in Table 3, average concentrations of urinary perchlorate of the groups in the research area were significantly greater than that in the control area, which was 14.6 ± 18.9 μg/L (24.6 ± 31.9 μg/g creatinine) ($p < 0.01$). This indicated that perchlorate released into the environment has entered into bodies of local people and that the exposure dose of perchlorate of people in the research area was much greater than that of people in the control area. The concentrations of urinary perchlorate in this study were greater than those reported by Blount et al. (2007). These investigators found that women of reproductive age had urinary perchlorate levels with a median of 2.9 μg/L [confidence interval (CI) 2.4–3.4 μg/L], 2.97 μg/g creatinine (CI 2.64–3.30 μg/g) based on samples of the 2001–2002 National Health and Nutrition Examination Survey. This variability likely resulted from varied water use, dietary habits, and race/ethnicity.

Health Risk Assessment

The values of parameters used in the exposure assessment model were listed in Table 4. The intake rate of drinking water and the average adult body weight used here were derived from data collected from local residents (Li et al. 2013). The exposure doses of perchlorate ingested through drinking water and the values of HQ are listed in Table 5. The values of HQ of river water and groundwater in the research area were much >1, whereas that of spring water in this district and river water, groundwater, and spring

Table 3 Concentrations of urinary perchlorate

Site	Group	*n*	Concentrations of urinary perchlorate	
			Mean ± SD (μg/L)	Mean ± SD (μg/g creatinine)
Research area	Nonoccupational group	26	171.5 ± 202.4**	278.8 ± 401.3**
	Occupational group	26	314.2 ± 410.9**	442.7 ± 532.4**
	Total	52	242.9 ± 328.7**	360.7 ± 474.1**
Control area	Control group	49	14.6 ± 18.9	24.6 ± 31.9

** Significant differences between groups in the research area and the control area, respectively ($p < 0.01$, ANOVA). There was no significant difference between the nonoccupational group and the occupational group ($p > 0.05$, ANOVA)

Table 4 Values of parameters used in the exposure assessment

Parameter	Value		Reference
	Male	Female	
CW (mg/L)			This study
IR (L/day)	3.550	2.763	Li et al. (2013)
EF (days/year)	365	365	USEPA (1989)
ED (year)	30	30	USEPA (1989)
BW (kg)	62.45	53.71	Li et al. (2013)
AT (days)	ED × 365	ED × 365	USEPA (1989)

water in the control area were all lower than 1. This indicated that when the river water and groundwater in the research area are used as drinking water, there may be adverse health effects resulting from perchlorate. Although the local government is continuously improving the conditions of drinking water, the surface water (e.g., rivers and lakes) and groundwater were still used as drinking water by some residents (Li et al. 2013). The values of HQ for adult male and female in the research area, by water category, were both in the order of river water > groundwater > spring water. This indicated that more attention should be paid to the drinking river water if this is the case.

Health risk assessment is a useful approach to quantify an index of potentially hazardous contaminants, but there were various uncertainties arising from exposure and toxicity assessment during the processes of risk assessment. Although the use of exposure parameters of local residents can decrease some uncertainties, other uncertainties still exist. For example, during risk assessment, we hypothesized that the concentrations of perchlorate in the river, groundwater, and spring water were stable, ignoring the dynamic variation of the concentrations of perchlorate, which might cause uncertainties in exposure assessment. Moreover, the RfD itself has uncertainty because it is an estimate (with uncertainty spanning perhaps an order of magnitude) of daily exposure to the human population (including sensitive subgroups), which is likely to be without an appreciable risk of deleterious effects during a lifetime (USEPA 2000). This might cause uncertainties in toxicity assessment. When considering other exposure pathways, such as ingestion of food and inhalation of particles, the health risk caused by perchlorate may be increased. Some studies have shown that many plants could take up perchlorate from contaminated soils or perchlorate-contaminated water (Yu et al. 2004; Sanchez et al. 2006; Tan et al. 2004), and food is also an important exposure source for perchlorate (Borjan et al. 2011). In addition, inhalation exposure also contributes to increasing health risk caused by perchlorate if a person is frequently exposed to a high air concentration of perchlorate particles. For example, one study on occupational exposure to perchlorate showed that exposure to the airborne particulate perchlorate resulted in systemic absorption (Lamm et al. 1999).

Table 5 ED and HQ of perchlorate through the pathway of drinking water

Parameter	Type of water	Research area		Control area	
		Male	Female	Male	Female
CDI (mg/kg/day)	Surface water	6.6E−03	5.9E−03	1.4E−04	1.3E−04
	Groundwater	2.8E−03	2.5E−03	1.5E−04	1.4E−04
	Spring water	1.8E−04	1.6E−04	1.1E−05	1.0E−05
HQ (unitless)	Surface water	9.4E+00	8.5E+00	2.0E−01	1.8E−01
	Groundwater	4.0E+00	3.6E+00	2.2E−01	2.0E−01
	Spring water	2.5E−01	2.3E−01	1.6E−02	1.5E−02

Conclusion

The contamination status of perchlorate in the fireworks production area of Liuyang in China was shown in this study. Concentrations of perchlorate in the surface water, groundwater, and soil in the fireworks production area were much greater than those in the control area, and concentrations of urinary perchlorate of women of reproductive age living in the fireworks production area were also significantly greater than those of women living in the control area. All of the above suggested that there was contamination of perchlorate possibly caused by production enterprises. People living in the fireworks production areas face a greater exposure dose of perchlorate. Concentrations of perchlorate in the surface water and groundwater in the fireworks production area were both greater than the interim drinking-water health advisory level of the USEPA, and the values of HQ, both of which were >1, indicated that the surface water and groundwater in the fireworks production area were unfit to be used as a direct source of drinking water. The toxic effects of perchlorate have been confirmed in some animal experiments (Gilbert and Sui 2008; Rainwater et al. 2008). Therefore, detailed epidemiological studies should be performed in this area from the viewpoint of precautionary principle. It is necessary to increase public awareness about the risk of exposure to perchlorate and to take effective measures to decrease perchlorate pollution.

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