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Original Article

Quantitative assessment of occupational exposure to total indium dust in Japanese indium plants

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Abstract: This study quantitatively assessed personal exposure of 86 workers to indium compounds as total dust at 11 Japanese indium plants. The personal exposures to indium concentrations in the breathing zone during an 8 h work-shift were determined by ICP-MS. The arithmetic mean indium concentration of all the workers was 0.098 mg Indium (In)/m^3, with individual values ranging from 0.0001 to 1.421 mg In/m^3. There were 11 workers whose exposure to indium concentrations exceeded the American Conference of Governmental Industrial Hygienists' Threshold Limit Value-Time Weighted Average (TLV-TWA) of 0.1 mg In/m^3. Based on the condition TLV-TWA<X_{95} (upper 95th percentile of log-normal distribution), five indium plants were judged as "control measures required", while 3 other plants were evaluated as "control measures not required". Five workers belonging to the worst group were exposed to far higher indium concentrations than the TLV-TWA. Another group of 5 workers belonging to the best group was exposed to far lower indium concentrations than the TLV-TWA, and this was attributed to the stringent engineering control measures used at their workplaces. The quantitative assessment of occupational exposure to indium dust was influenced by different occupational exposure limit values without carcinogenicity and particle size-selectivity of indium particulates or "total" dust.

Key words: Indium dust, Total dust, Indium-Tin-Oxide, Personal sampling, Particle size-selectivity, Upper 95th percentile

Introduction

Exposure of workers to hardly soluble indium dust was reported to have caused 7 cases of interstitial pneumonia in Japan, 2 cases of pulmonary alveolar proteinosis (PAP) in the USA, and 1 case of PAP in China[1]. It is of prime importance to protect workers from health outcomes due to excessive exposure to indium dust with reference to the occupational exposure limit (OEL). Different OEL values for indium dust have been recommended by several international organizations of occupational health. The Threshold Limit Value-Time Weighted Average (TLV-TWA) of 0.1 mg/m^3 of indium for indium and indium compounds was recommended by the American Conference of Governmental Industrial Hygienists (ACGIH)[2]. The same value of 0.1 mg indium (In)/m^3 was designated as the Recommended Exposure Limit (REL) for indium compounds by the National Institute for Occupational Safety and Health (NIOSH)[3]. The Japanese Ministry of Health, Labour and Welfare (MHLW) has issued the

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Technical Guideline[4] for Preventing Health Impairment of Workers Engaged in the Indium Tin Oxide (ITO) Handling Processes, which defines two occupational standards: an Acceptable Exposure Concentration Limit of 3×10^{-4} mg In/m^3, and a Target Concentration of 0.01 mg In/m^3 in the respirable fraction of indium dust. The Technical Guideline[4] designates the particle size-selective capturing of indium particles of cut-size 4 μm (50% efficiency) as respirable dust, which is hazardous when deposited in the deep gas-exchange region of the lungs. On the other hand, ACGIH's TLV and NIOSH's REL for indium dust are expressed in terms of total dust, since indium dust is not designated an "inhalable, thoracic or respirable fraction of particulate matter depending on particle size-selective sampling criteria", but as "particulates not otherwise regulated, total"[2, 3, 5].

Our previous study[6], a generic control banding assessment of workers' health risks in 13 Japanese indium plants using the COSHH Essentials[7], noted that since indium dust is highly hazardous to workers' health, the majority of indium-handling operations in the 13 plants were subject to "Control Approaches 3 and 4 according to the COSHH Essentials"[7], and that highly efficient engineering control measures such as enclosed-type local exhaust ventilation (LEV) and respiratory protective equipment (RPE) with high Assigned Protection Factor (APF) should be provided. However, there were some limitations regarding the generic control banding assessment, since the hazard levels, the amount in daily use and the "dustiness" characteristics of indium dust in the workplace were broadly categorized using simple scales. The present study was designed to quantitatively assess the personal exposures of 86 indium-handling workers to indium compounds in terms of total dust at 11 Japanese indium plants with reference to the TLV-TWA. We also examined the industrial hygiene control measures used in the 11 plants for an efficiency evaluation in comparison to the OEL using the statistical exposure indices of the exposure concentrations obtained.

Materials and Methods

Subjects of study

Thirteen plants enlisted for the preliminary investigation were chosen from among the 38 companies in Japan whose annual production of indium exceeded 500 kg in 2009. Since the generic control banding assessment of occupational exposure to indium and its compounds in the 13 indium plants has been carried out in a previous study[6], 11 out of the 13 indium plants were investigated for the comprehensive assessment of personal exposure of workers to indium dust in the present study. These 11 companies consisted of 3 plants having 300 employees or more, 4 plants having 100 to 299 employees, 1 plant having 50 to 99 employees and 3 plants having less than 50 employees. We examined the personal exposures of 86 workers to indium compounds in terms of total dust, as a sample of the 279 workers assigned to various indium-handling operations in the 11 plants.

Analysis of indium

The analysis of indium was carried out with a slight modification to the method described in the MLHW's Technical Guideline[4] and the NIOSH Manual of Analytical Methods No.7301[8] for indium, using an Inductively Coupled Plasma-Mass Spectrometer (ICP-MS) (Agilent7500i, Agilent Technology, Japan). The analytical conditions of ICP-MS are summarized as follows: 1,400 Watts of RF power at 1.7 V, flow rates of 15 l/min for the plasma gas and 1.0 l/min for the carrier gas, and measured m/z values of 115 and 118. The membrane filter on which indium-containing particulate matter was collected was placed into a flask containing 30 ml of mixed acid [nitric acid (Wako Pure Chemicals, Ltd., Tokyo, Japan), hydrochloric acid (Wako Pure Chemicals, Ltd.), ultra-pure water (18 MΩcm, Toraypure LV-10T, Japan) in the ratio of 1:3:4] and subjected to ultrasonic agitation for 60 min, before being digested on a hot plate at 160°C for 60 min. A standard solution of indium for atomic absorption spectrometric analysis (Kanto Chemicals, Japan) was used in the preparation of the calibration curve for the quantitation. The calibration curve of peak counts of m/z (Y) in ICP-MS versus indium concentrations (X) in the mixed acid (ng/ml) used in the present study was found to be highly linear in the range of 0.5 ng/ml to 100 ng/ml and highly correlated; the regression equation was $Y=2270X + 15310$ with a correlation coefficient of 0.9997. The quantitation limit of indium was found to be 0.07 mg/ml. The quantitation limit of airborne indium was estimated to be 0.003 μg/m^3, based on assumptions of the final volume of the solution used in the analysis being 40 ml per filter, and the volume of collected workplace air being 960 l at a flow rate of 2 l/min.

Determination of personal exposure concentrations of indium

Indium compounds in the total dust suspended in the workplace air collected in the workers' breathing zones were analyzed for personal exposure concentrations of indium which were expressed as the 8-h time-weighted average (8h-TWA). The total dust containing indium compounds was collected on a mixed cellulose ester membrane filter (0.8-μm pore size, 37-mm diameter, Japan Millipore, Co.) in a cassette filter holder (SKC, Inc.). The workplace air was drawn at a flow rate of 1.0 l/min for a sampling period of 6 h or longer, except during lunchtime, using a portable suction pump (Aircheck 2000, SKC, Inc.). We used the NIOSH sampling method (NMAM0500)[8] for the collection of indium dust as "particulates not otherwise regulated, total". The sampling period for the collection of total dust was also in accordance with the directions of the NIOSH Occupational Exposure Sampling Strategy Manual[9] that the measurement results are valid only for 6 out of 8 h, or the sampling portion of the period should cover at least 70% to 80% of the full exposure period. The indium concentration of personal exposure to total indium dust expressed as 8h-TWA was calculated using the following equation:

$$8\text{h-TWA} = (C_{pi} \times T_{pi})/8\ \text{h},$$

where C_{pi} (mg/m^3) and T_{pi} (h) indicate observed indium concentration of exposure to the total dust during the sampling period, and the period of time used for sampling the indium dust, respectively.

Calculation of statistical indices

In addition to conventional statistical analyses, the upper 95th percentile of the log-normal distribution (X_{95}) was calculated using to the following equation:

$$\log(X_{95}) = \log(GM) + 1.65 \times \log(GSD),$$

where GM and GSD are the geometric mean and geometric standard deviation, respectively. The condition "TLV<X_{95}" was used to define "control measures required" in the workplace, an unacceptable exposure in accordance with the concept of exposure risk defined by the NIOSH Occupational Exposure Sampling Strategy Manual[9], Bullock and Ignacio[10], and Hashimoto *et al*[11]. We also compared the indium concentrations of individual workers with the Action Level (AL)[9], which is defined by NIOSH as one-half of the REL (0.05 mg In/m^3). When the indium plants had less than 5 workers, we made our judgment of "control measures required" based on either AL<Max or TLV<Arithmetic mean (AM).

Results

Indium-exposed workers examined

Table 1 presents the basic demographic data of the 86 indium-exposed workers employed in various indium-handling operations at the 11 indium plants. The indium plants were grouped into the two different sectors of the indium industry: 4 plants performing recycling/reclamation, and 7 plants manufacturing electronic devices. Various forms of indium materials such as metallic indium, ITO, indium oxide, indium hydroxide and indium phosphide are handled in these operations. The indium-handling processes characterized by weighing, bagging, crushing, casting and sintering were found to be dusty, while other processes, dehydration and electrophoresis, were deemed to be clean. Grinding and cutting of indium crystals were performed within either a glove box or an enclosed-type LEV to prevent excessive exposure to indium materials.

In Fig. 1, individual 8h-TWA indium concentrations of the 86 workers are plotted on a logarithmic scale. The individual workers are labelled with numbers and uncapitalized letters, a–k, which represent each of the 11 indium plants. The logarithmic concentrations of personal exposure to indium compounds in the total dust were found to follow a sigmoidal curve, indicating a log-normal distribution.

Table 2 shows the indium concentrations in total dust as determined by the personal exposures of the workers in the breathing zone at the 11 indium plants. The AM indium concentration of the 86 workers was 0.098 mg In/m^3, with individual values ranging from 0.0001 mg In/m^3 to 1.421 mg In/m^3. The exposure concentrations could be regarded as following the log-normal distributions according to the Kolmogorov-Smirnov test (Excel-Toukei, Social Survey Research Information Co., Ltd.). We used two different methods for defining and judging exposure profiles to determine the acceptability of occupational exposure to total indium dust. First, we compared the personal exposure data of individual workers with ACGIH's TLV-TWA as the OEL, and AL as one-half of NIOSH's REL. There were 11 (13%) workers whose exposures to indium concentrations exceeded the TLV-TWA. Seven workers were from 3 recycling/reclamation plants, and four workers were from 3 plants manufacturing electronic devices. Nineteen (22%) of the 86 indium-exposed workers had indium concentrations exceeding the AL. We also used the condition TLV<X_{95} to determine unacceptable exposures which required control measures in the workplace. Five indium plants (a, d, e, g, and i) were evaluated as requiring

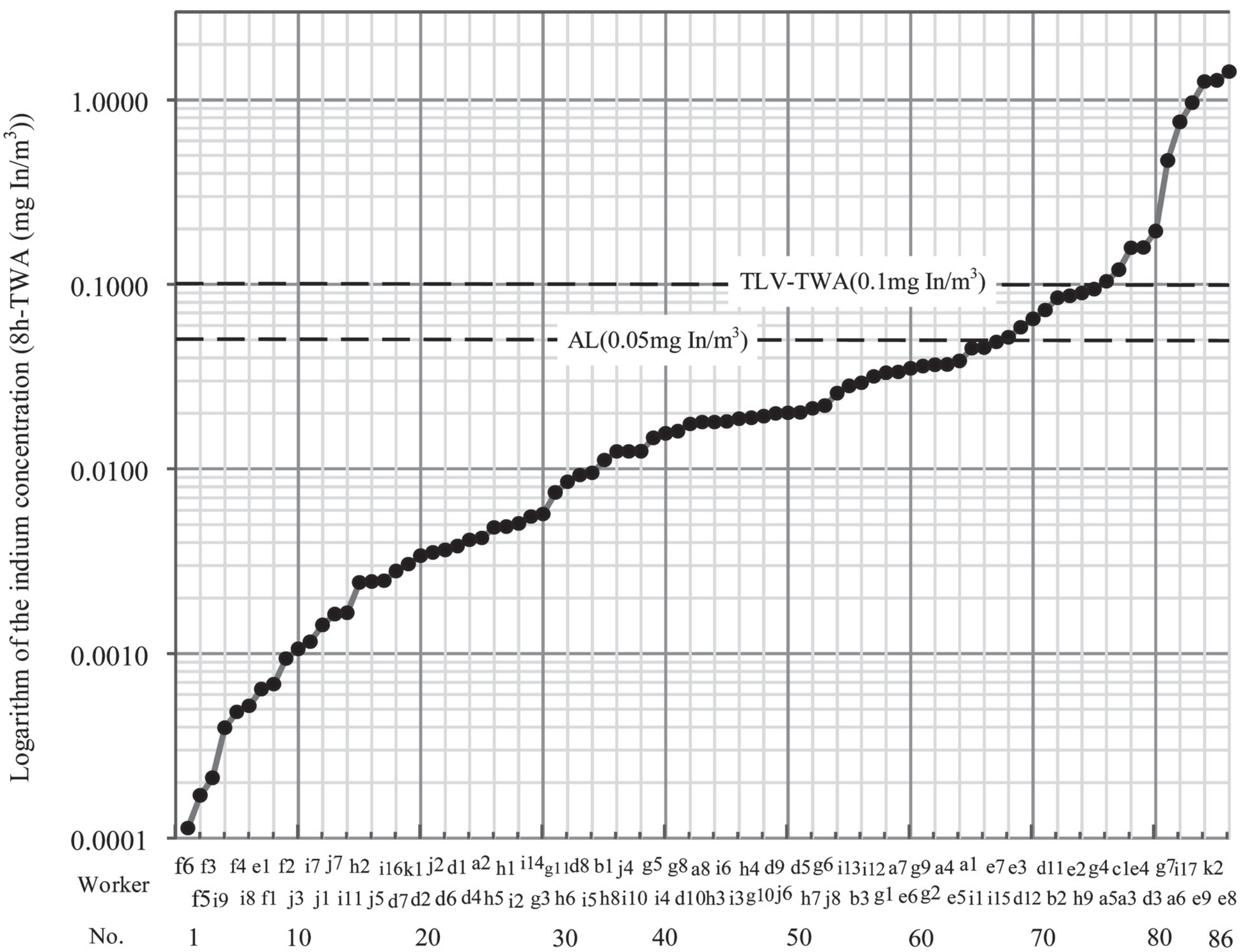

Fig. 1. Individual 8h-TWA indium total dust exposure concentrations of the 86 workers. The individuals are identified by a number and a letter, which indicates the plant where they were employed. The two dashed lines indicate the TLV-TWA (0.1 mg/m^3) and the AL (0.05 mg/m^3).

control measures. Three plants (f, h, and j) were evaluated as not requiring control measures. The remaining 3 plants (b, c, and k) which employed less than 50 workers were evaluated as requiring control measures using the conditions of AL<Max for Plant b, and TLV<AM for Plants c and k.

The worst and best personal exposures to indium dust

Table 3 presents the worst and best the 86 of 5 cases of indium concentrations from among the personal exposures of workers employed in various indium-handling operations at the 11 plants. In the worst group of 5 cases, all the workers were exposed to almost 10-fold higher indium concentrations than the TLV-TWA. They wore reusable half-face masks with particle filters with an assigned protection factor (APF) of 10. However, the engineering control measures of LEV were inadequate.

Table 3 also shows that all 5 workers of the best 5 cases were exposed to far lower indium concentrations than the TLV-TWA. Three workers wore no masks, or disposable, unwoven mask in the dusty operation of grinding and cutting InP crystals. However, at their workplaces a glove box or enclosed type LEV was installed and this type of installation can further reduce the exposure concentration to 1/10 of that of a hood type LEV[7]. The other two workers were employed in an indium residue-handling operation and they wore half-face dust masks without any LEV in the workplace.

Discussion

The present results indicate that 11 out of 86 indium-exposed workers were exposed to higher indium concentrations than the TLV-TWA. There were also 19 (22%)

Table 1. Summary of 11 Japanese indium plants examined for indium material handled, operations in manufacturing processes, number of employees and number of workers exposed to indium compounds in total dust

Plants	Sectors of indium industry	Number of employees	Indium compounds handled	Operations in indium-manufacturing processes	Number of examined workers exposed to indium/Total number of possibly exposed workers
a	Recycling/Reclamation	21	ITO, Metallic indium, Indium hydroxide	Crushing raw materials, Filling and bagging, Solubilizing indium materials	8/14
b	Manufacturing electronic devices including ITO	118	Indium hydroxide, ITO	Ingot filling, Solubilizing, Drying and crushing	3/18
c	Manufacturing electronic devices including ITO	135	ITO	Pellett and powder filling, Crushing	1/2
d	Manufacturing electronic devices including transparent electrodes and panels	486	Indium hydroxide, ITO	Weighing and powder filling, Formulation, Bonding, Grinding and cleaning	12/33
e	Manufacturing electronic devices including transparent electrodes and panels	150	Indium hydroxide, ITO	Deposition by beam evaporation, Filling into a 18-l container, Maintenance (powder)	9/14
f	Manufacturing electronic devices including sputtering target	70	ITO	Shot-blasting, Dust cleaning	6/10
g	Recycling/Reclamation	579	Indium oxide, Indium hydroxide	Filtering and drying, Crushing, Powder filling	11/32
h	Manufacturing electronic devices including sputtering target	192	ITO	Sintering (Ingot, solubilized metal), Crushing and bonding (clumpted metal), Grinding and Powder fillling	9/14
i	Manufacturing electronic devices including sputtering target, Surface materials and semiconductors	318	Metallic indium, ITO, Indium hydroxide, Indium phosphide	Pressing (cake-like), Bonding (clumped metal), Drying, Filling,Grinding and dust cleaning	17/114
j	Recycling/Reclamation	45	Metallic indium, ITO	Ingot melting and solubilizing, Electrophoresis, Sintering	8/11
k	Recycling/Reclamation	25	Metallic indium, ITO	Crushing, Solubilizing, Sintering ingot	2/3

workers out of the 86 whose indium concentrations exceeded the AL, suggesting that certain provisions must be initiated at their workplaces, such as consecutive exposure measurements, training of employees, and medical surveillance, according to the NIOSH Occupational Exposure Sampling Strategy Manual[9]. It has been reported that the condition TLV<X_{95} can be used as a definition of "control measures required"[9–11]. By this definition, eight of the eleven indium plants required control measures, while the other 3 plants were evaluated as not requiring control measures. Notably, all 5 workers of the worst exposure group were employed in various indium-handling operations, such as collection of indium oxide dust, crushing and sintering and cutting of ITO targets, were exposed to much higher indium concentrations of total dust than the TLV-TWA. The present results are consistent with the findings of Cummings *et al.*[12] in a NIOSH survey of personal breathing zone analysis of indium total dust concentrations in US companies. They reported that various activities with increased potential for indium exposure included the manufacturing of inorganic indium compounds, ITO sputter target resurfacing, and abrasive blasting to remove residues. They also reported the pneumatic sanding of ITO targets resulted in high worker exposure to indium of 3.1 mg In/m^3 of total dust[12]. The present results are consistent with the findings of Miyauchi *et al.*[13] that breathing zone indium concentrations of total dust are higher in the processes of ITO milling (0.67 mg In/m^3 as TWA), and pulverization and heating (7.1 mg In/m^3 as TWA) than in the electrolyte refining process (0.043 mg In/m^3 as TWA)

Table 2. Indium concentrations (mg In/m^3) as determined by personal exposure to total dust of indium at the breathing zone with reference to ACGIH's TLV-TWA

Items	Plants	Number of workers	Number of workers whose exposure concentrations exceeded the TLV-TWA (0.1 mg In/m^3)	Number of workers whose exposure concentrations exceeded the AL (0.05 mg In/m^3)	Min (mg In/m^3)	Max (mg In/m^3)	AM (mg In/m^3)	GM (mg In/m^3)	GSD	X_{95} (mg In/m^3)	Statistical significance of log-normal distribution at $p \geq 0.10$ by the Kolmogorov-Smirnov test
Indium concentrations in total dust	a	8	3	4	0.004	0.760	0.146	0.052	4.864	0.701	*$p \geq 0.10$
	b	3	0	1	0.011	0.085	0.044	NC	NC	NC	NC
	c	1	1	1	0.120	0.120	0.120	NC	NC	NC	NC
	d	12	1	2	0.002	0.194	0.033	0.013	4.137	0.131	*$p \geq 0.10$
	e	9	3	5	0.001	1.421	0.345	0.073	9.693	3.047	$p=0.0924$
	f	6	0	0	0.0001	0.001	0.000	0.0003	2.321	0.001	*$p \geq 0.10$
	g	11	1	3	0.006	0.469	0.069	0.028	3.403	0.209	*$p \geq 0.10$
	h	9	0	1	0.002	0.090	0.020	0.012	2.894	0.067	*$p \geq 0.10$
	i	17	1	1	0.0004	0.965	0.071	0.010	6.668	0.220	*$p \geq 0.10$
	j	8	0	0	0.001	0.026	0.009	0.005	3.497	0.035	*$p \geq 0.10$
	k	2	1	1	0.002	1.273	0.638	NC	NC	NC	NC
	Total	86	11	19	0.0001	1.421	0.098	0.014	7.610	0.393	*$p \geq 0.10$

AL: Action level; Min: Minmum value; Max: Maximum value; AM: Arithmetic mean; GM: Geometric mean; GSD: Geometric standard deviation; X95:Upper 95th percentile of a log-normal distribution; NC: Not calculated.

Table 3. The worst and best 5 cases of personal exposure to indium compounds in total dust in the breathing zone, and industrial hygiene control measures being used at their workplaces

Personal exposure concentrations of indium in total dust			Control Measures	
The worst or best group of 5 cases	Indium-handling operations (Time spent at the workplace)	Indium exposure concentration as 8h-TWA (mg In/m^3)	Local Exhaust Ventilation (LEV)	Respiratory Protective Equipment (RPE)
Worst group				
Worker e8	Collection of indium oxide dust into 18-l container (44 min)	1.421	None	Half-face dust mask
Worker k2	Crushing (5 min), Sintering (25 min)	1.273	Hood-type LEV	Half-face dust mask
Worker e9	Collection of indium oxide powder into a 18-l container (44 min)	1.256	None	Half-face dust mask
Worker i17	Cutting off and crushing of ITO target (30 min)	0.965	Hood-type LEV	Half-face dust mask
Worker a6	Weighing and bagging (120 min)	0.760	Hood-type LEV	Half-face dust mask
Best group				
Worker f6	Recovery of indium residue in the ditch (16 min)	0.0001	None	Half-face dust mask
Worker f5	Recovery and transfer of indium residue from the ditch (13 min)	0.0002	None	Half-face dust mask
Worker f3	Shot-blasting (380 min)	0.0003	Enclosed-type LEV	Half-face dust mask
Worker i9	Grinding and cutting off of InP crystal (360 min)	0.0004	Glove box	Disposable, unwoven mask
Worker i8	Grinding and cutting off of InP single crystal (360 min)	0.0005	Enclosed-type LEV	None

in an indium-recycling/reclamation plant.

Two workers, e8 and e9, were exposed to very high indium concentrations of 1.421 mg In/m^3 and 1.256 mg In/m^3, respectively. These values were 14- and 13-fold higher than the TLV-TWA. Those 2 workers wore half-face dust masks with an APF of 10 and no LEV system was installed at their workplaces. We recommended the employer to implement two kinds of control measures: provide dust masks with an APF of 25 in accordance with the NIOSH Respirator Selection Logic[14], and install a hood-type LEV at their workplaces, since the exposure concentration can be reduced to 1/10 of that of general ventilation by this kind of LEV[7].

Three workers, k2, i17, and a6, were exposed to higher indium dust concentrations than the TLV-TWA. Their control measures were found to be inadequate. They wore half-face masks with an APF of 10 and were employed in indium-handling operations at workplaces having only a hood-type LEV. Since the measured capture velocities were found to fall to approximately 0.1 m/sec, and the canopy hood was positioned distant from the contaminant source of the crushing operation, engineering control measures of increasing the capture velocity of the LEV and replacing the LEV with an enclosed-type LEV near the contaminant source were recommended. Effective mitigation of the exposure concentration to below the TLV-TWA was thought to be achievable through the wearing of full-face dust masks with a higher APF in accordance with the NIOSH Respirator Selection Logic[14].

It was noteworthy that 3 workers, f3, i8, and i9, were exposed to indium concentrations of total dust far below the TLV-TWA, even though 2 of the 3 workers wore no dust mask, or disposable, unwoven masks. The effective mitigation was attributed to more stringent engineering control measures, such as the installation of a glove box and an enclosed-type LEV, both of which have been reported to reduce the exposure concentrations to 1/10 of that of a hood-type LEV[7].

Two clear differences can be recognized between ACGIH's TLV-TWA or NIOSH's REL-TWA and the Technical Guideline's occupational standards. ACGIH[2, 15] recommends a TLV-TWA of 0.1 mg In/m^3 of total dust on the basis of adverse skeletal and gastrointestinal effects and pulmonary toxicity associated with inhaled indium without notations of carcinogenicity. NIOSH recommends a REL-TWA of 0.1 mg In/m^3 total dust for indium compounds without designation of occupational carcinogenicity[3]. On the other hand, the Technical Guideline's occupational standard[4] "Acceptable Exposure Concentration Limit"[4] of 3×10^{-4} mg In/m^3 respirable dust was derived from an empirical dose-response relationship between the inhalation exposure concentrations of respirable ITO dust and incidences of bronchio-alveolar cancer in a 2-yr inhalation carcinogenicity study of rats[16]. Our previous control banding study[6] revealed that the OEL value for indium dust in indium plants should be maintained below 0.01 mg In/m^3, since airborne indium dust was highly hazardous with "high dustiness". Cummings *et al.*[12] suggested that particulate matter containing indium compounds with a mass median aerodynamic diameter less than 4 μm can penetrate into the deep alveolar region when inhaled, and that this would be toxicologically relevant for chronic respiratory diseases and lung cancer. Further study will be needed to quantitatively assess exposures to airborne indium dust with reference to a particle size-selective OEL value expressed in inhalable, thoracic and respirable fractions of particulate matter, in addition to the quantitative risk assessment of indium dust for human carcinogenicity.

Conclusion

The personal exposures of 86 workers to indium and indium compounds in total dust at 11 Japanese indium plants whose annual production exceeded 500 kg, was determined as time-weighted averages by ICP-MS. There were 11 workers whose indium concentrations of personal exposure to total dust exceeded the TLV-TWA. Based on the condition $TLV<X_{95}$, five indium plants judged to require control measures for their indium-handling operations. Several workers of the worst exposure group were exposed to much higher concentrations of indium dust than the TLV-TWA. The control measures of LEV and RPE were found to be inadequate at their workplaces. Another group of 5 workers was exposed to indium concentrations of total dust that were much lower than the TLV-TWA, and this was attributed to the stringent engineering control measures adopted at their workplaces.

Conflict of Interest

The authors declare that there are no conflicts of interest.

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