Acid Gas, Acid Aerosol and Chlorine Emissions from Trichlorosilane Burning Processes

Jhy-Charm Soo1, Siou-Rong Li1, Jenq-Renn Chen2, Cheng-Ping Chang3, Yu-Fang Ho3, Trong-Neng Wu4*, Perng-Jy Tsai1,4*

1 Department of Environmental and Occupational Health, Medical College, National Cheng Kung University, 138, Sheng-Li Road, Tainan 70428, Taiwan
2 Department of Safety, Health and Environmental Engineering, National Kaohsiung First University of Science & Technology, University Road, Yenchau, Kaohsiung, 824, Taiwan
3 Institute of Occupational Safety and Health, Council of Labor Affairs, 99, Lane 407, Hengke Road, Sijhih City, Taipei County 22143, Taiwan
4 Department of Occupational Safety and Health, College of Public Health, China Medical University and Hospital, 91, Hsueh-Shih Road, Taichung 40402, Taiwan

ABSTRACT

This study was set out to investigate the emission characteristics of HCl (in both particle (HCl_p) and gaseous (HCl_g) forms), and Cl_2 during the trichlorosilane (TCS) burning process under various relative humidity conditions (RH; range = 55%–90%) which might exist at its storage area. All experiments were conducted in a test chamber. We found that HCl_p was consistently as the most dominant contaminant (= 1.30 × 10^5–1.46 × 10^5 mg/m^3), followed by the HCl_g (= 9.03 × 10^3–11.4 × 10^3 mg/m^3) and Cl_2 (= 1.91 × 10^3–2.18 × 10^3), emitted from the TCS burning process for the all selected RH conditions. The particle sizes of HCl_p fell to the range of the accumulation mode (MMADs = 0.808–1.04 \mu m; GSDs = 2.13–3.50). Fractions of emitted HCl_p reaching to the alveolar region (= 85.8–88.8%) were much higher than that of the tracheobronchial region (= 6.53–8.80%) and head region (= 4.67–5.40%). It is concluded that more ill-health effects on the deep lung region can be expected than other regions as workers exposed to the contaminants emitted from TCS burning processes.

Keywords: Trichlorosilane; Burning process; Acid gas, Acid aerosol; Chlorine.

INTRODUCTION

According to statistical data, the annual world production of trichlorosilane (TCS) is ~350,000–380,000 tons and ~60–65% of TCS is consumed by the semiconductor industry as an alternative silicon source gas (Howe-Grant, 1997; Williams, 2000). TCS is also used as the basic ingredient in the production of solar cells, optical fibers, and the manufacturing of PV grade polysilicon and silane gas. For the above industries, many study have been conducted to address their fugitive emissions at process areas (Hu et al., 2010; Lin et al., 2010; Shih et al., 2010) or pollutant removal efficiencies of various air pollution control devices (Lin et al., 2010; Shiue et al., 2011). Though many studies have been conducted to address the emissions arising from combustion related activities (Choosong et al., 2010; Li et al., 2010; Ning et al., 2010), very limited have been conducted to characterize the emissions of raw materials during the accidental fire or explosion cases.

TCS is known with a boiling point of 31.85°C, flash point of –28°C, and flammability range of 7%–82% (Laurence, 1990). Apparently, its major hazard might simply lie on its flammability. Therefore, TCS emergency response guidelines proposed by different organizations, such as the Silicones Environmental, Health and Safety Council of North America (SEHSC), Centre Européen des Silicones (CES), Silicone Industry Association of Japan (SIAJ), concern still mostly on its fire and explosion effects, rather on the impact caused by its resultant combustion by-products (e.g., hydrogen chloride (HCl) and chlorine (Cl_2)) (Higgins et al., 1999; CES, 2003). However, the impact of its combustion by-products has never been investigated.
In principle, the generation of HCl from the TCS burning process can be described as (Mores, 1984):

$$5\text{SiHCl}_3 + 6\text{O}_2 \rightarrow 5\text{SiO}_2 + \text{HCl} + 7\text{Cl}_2 + 2\text{H}_2\text{O}$$

(1)

In addition, part of emitted Cl₂ could further react with H₂O to form HCl:

$$\text{Cl}_2 + \text{H}_2\text{O} \rightarrow \text{HClO} + \text{HCl}$$

(2)

From the Eq. (1), the generated HCl could be presented simultaneously in both gaseous (i.e., HCl_g) and aerosol (i.e., HCl_p) forms. Here, HCl_g might be formed directly via the condensation of HCl_g on particles (such as SiO₂) via the heterogeneous condensation process, or by directly dissolving HCl_g into H₂O coated on the surface of SiO₂ particle or water droplets. From Eq. (2), the generated HCl might directly dissolve water droplets to form HCl_p.

Among these emitted contaminants, Cl₂ is known for its acute health effect on the respiratory system (Agabiti et al., 2001; Rabinowitz and Siegel, 2002; Uyan et al., 2009) and acute ischemic stroke (Kose et al., 2009). HCl_g is an upper respiratory tract irritant because of its high water-solubility (Salem, 2005). HCl_p, on the other hand, might penetrate into the deep lung of the respiratory tract and cause more serious ill-health effects depending on its particle size (ICRP, 1994). Therefore, simultaneously measuring Cl₂, HCl_g and HCl_p are considered a better approach to characterize the release of toxic chemicals from the TCS burning process.

In this study, a chamber study was conducted to investigate the emission characteristics of Cl₂, HCl_g and HCl_p during the TCS combustion process. In order to simulate the real situation for TCS storage in the indoor gas yard, four relative humidity (RH) conditions were used based on RH records provided by semiconductor industries in Taiwan. The results obtained from this research will provide information for the government, semiconductor and optoelectronic industries to generate appropriate strategies for TCS emergency response process.

**MATERIAL AND METHODS**

**Test Chamber and its Apparatus**

Fig. 1 shows the stainless steel 316 test chamber used in the present study. The chamber comprised three parts,
including an air inlet section (i.e., the lower pyramid part), a TCS burning section, and an air outlet section (i.e., the upper pyramid part). Before the supply air entering into the lower pyramid, it was treated by an air cleaning and conditioning system (Fig. 2). The system consisted of a compressor, a dryer, a surge tank, an activated charcoal, a high-efficiency particulate air (HEPA) filter, a home-made heating tank, and a humidifier (model FC-125, Perma Pure Inc., NJ, USA). The mass flow controller (MFC, Side-Trak® Model 840, Sierra Instruments Inc., CA, USA) was also installed in the system to ensure to provide a designated air flow with a preset humidity condition. The flat top of the air inlet section was installed with a honeycomb to provide a uniformly distributed laminar flow for the TCS burning section. This burning section was a 0.42 m³ cube installed with one glass door, and three stainless steel side-walls. On the opposite wall of the glass door was installed with a TCS feeding tube (diameter = 0.64 cm) to transport a fixed amount of TCS (ACS grade, ~99.9% purity) from the TCS storage cylinder to the test pan (diameter = 5.0 cm, height = 3.0 cm). In order to know the consumption rate of TCS, the test pan was placed on a digital balance (accuracy = ± 0.02 g, model SKY-600, Javeder Scale Co., Taiwan) to continuously send weight signals to the laptop per 1/30,000 second during each combustion test run. All air samples were collected at the converging part of the air outlet section.

Selected Test Conditions

Four RH conditions (= 55%, 65%, 80% and 90%) were selected for conducting TCS combustion experiments to simulate possible humidity conditions of the storage area year round. After being pretreated by the air cleaning and conditioning system, the RH conditions used in the present study were 57.3%, 65.5%, 79.8% and 89.8%, respectively (with relative standard deviations (RSDs) < 1.01%). The air flow feeding rate was specified at 0.042 m³/min by a mass flow controller in the present study to simulate the air change per hour (ACH = 6) commonly found in the indoor gas yard. The resultant chamber air velocity (0.0017 m/s) was also comparable to the indoor work environment. To assess the uniformity of air velocities occurred at the TCS burning section, four cross-sections were chosen from the length of the burning section. For each cross-section, it was divided into 16 equal areas (~0.035 m² for each area) and air velocities measurements were conducted at the center of each area. The total measured air velocities were ranging from 0.0017 to 0.0019 m/s (RSD = 1.20%) indicating the uniformity of the test chamber was quite acceptable.

Sample Collection and Analysis

For each selected test conditions, three repeated particle size segregating samplings were conducted at the converging part of the air outlet section of the test chamber using a Micro-Orifice Uniform Deposit Impactor (MOUDITM model 110, MSP Corp., Minneapolis, MN, USA), and followed by Nano Micro-Orifice Uniform Deposit Impactor (Nano-MOUDITM model 115, MSP Corp., Minneapolis, MN, USA) for collecting the generated HClp. The whole sampling train consisted of an inlet stage (with a 50% cut-off aerodynamic diameter \(d_{50\%}=18 \mu m\)), thirteen impaction stages (with \(d_{50\%}\)s of 10.0, 5.6, 3.2, 1.8, 1.00, 0.56, 0.32, 0.18, 0.10, 0.056, 0.032, 0.018, and 0.010 \(\mu m\), respectively), and a 37 mm PTFE back-up filter (pore size = 2.0 \(\mu m\), ZeflourTM Pall Corp., Ann Arbor, MI, USA). The summation of HClp concentrations of all impaction stage and the back-up filter was regarded as the total HClp concentration. The sampling flow rates of MOUDITM and Nano-MOUDITM were set at 30.0 and 10.0 L/min, respectively and were checked periodically throughout the entire sampling period. Yet, it is true that many instruments can be used to conduct aerosol samplings with particle sizes covering both micron and submicron ranges (Li et al., 2009; Kim et al., 2010; Liu et al., 2010; Intra et al., 2011), the use of MOUDI and Nano-MOUDI in the present study has the advantage in their continuous collected particle size ranges.

Beside HClp, three repeated HClp and Cl₂ samples were simultaneously collected using a silica gel tube (Cat. No. 226-10-03, SKC Inc., Eighty Four, PA, USA) and a 25 mm sliver membrane filter (pore size = 0.45 \(\mu m\), Cat. No. 225-
RESULTS AND DISCUSSION

C$_{\text{HClp}}$, C$_{\text{HClg}}$, and C$_{\text{Cl2}}$ Emitted from TCS Burning Processes

Table 1 shows means and their corresponding RSDs of C$_{\text{HClp}}$, C$_{\text{HClg}}$, and C$_{\text{Cl2}}$ emitted from TCS burning processes under the four selected RH conditions. For all test conditions, we found that the C$_{\text{HClp}}$ (mean = 1.30 × 10$^5$–1.46 × 10$^5$ mg/m$^3$; RSD = 6.90–22.0%) was the dominant by-product, followed by the C$_{\text{HClg}}$ (mean = 9.03 × 10$^3$–1.14 × 10$^4$ mg/m$^3$; RSD = 8.90–37.1%) and C$_{\text{Cl2}}$ (mean = 1.91 × 10$^1$–2.18 × 10$^2$ mg/m$^3$; RSD = 4.00–11.0%). Here, it should be noted that no significant difference was found among C$_{\text{HClp}}$ obtained from the four selected RH conditions (Kruskal-Wallis test, $p$ > 0.05). The same trend can also be seen in C$_{\text{HClg}}$ and C$_{\text{Cl2}}$. The above results indicate that RH didn’t have a significant effect on the composition of the by-products emitted from the TCS burning process.

The emitted HCl concentrations (i.e., C$_{\text{HClp}}$ + C$_{\text{HClg}}$) were much higher than Cl$_2$ concentrations (i.e., C$_{\text{Cl2}}$). Apparently, it was contradictory to the theoretical predictions based on eq. (1) (i.e., C$_{\text{Cl2}}$ should be ~14 times in magnitude higher than that of the summation of C$_{\text{HClp}}$ and C$_{\text{HClg}}$). The above inconsistency might be explained by Eq. (2) (i.e., most of the generated Cl$_2$ might further react with H$_2$O to form HCl). The above inference is consistent with results found by Chow et al. (1994). They found that ~90% of chloride containing in the coal was converted to HCl during the coal combustion process. Therefore, it could be concluded that the existence of water vapor during TCS burning process would result in less acute inhalatory effect because of converting the emitted Cl$_2$ into HCl. However, more serious deep lung irritation and inflammation might occur due to higher resultant concentrations in HCl$_p$.

In the present study, we also found that C$_{\text{HClp}}$ was much higher than that of C$_{\text{HClg}}$. The above result was not so surprising because (1) the generated HCl$_p$ might condensate on existing SiO$_2$ particles to form HCl$_p$ via the heterogeneous condensation process (Friedlander, 2000), (2) the generated HCl$_p$ might dissolve into H$_2$O coated on the surface of SiO$_2$ particle, and (3) the generated Cl$_2$ might further react with H$_2$O to form HCl$_p$.

Finally, our study also indicated that humidity didn’t have a significant effect on the compositions of TCS burning products. By examining all test conditions, ~0.655–0.962 g/min humidity was originally containing in the inlet air (RH = 57.3–89.8 %). In addition, there was ~0.440–0.498 g/min water vapor emitted from TCS burning processes based on the TCS consumption rates (CR = 8.28–9.38 g/min) obtained from this study.

Table 1. Mean emitted concentrations of C$_{\text{HClp}}$, C$_{\text{HClg}}$, and C$_{\text{Cl2}}$ and their corresponding relative standard deviations (RSDs) from TCS burning processes under the four selected RH conditions

| RH (%) | C$_{\text{HClp}}$ | RSD (%) | C$_{\text{HClg}}$ | RSD (%) | C$_{\text{Cl2}}$ | RSD (%) |
|--------|-----------------|---------|-----------------|---------|---------------|---------|
| 57.3   | 1.35 × 10$^5$   | 22.0    | 1.06 × 10$^4$   | 8.90    | 2.05 × 10$^3$ | 11.0    |
| 65.5   | 1.46 × 10$^5$   | 6.90    | 1.13 × 10$^4$   | 20.2    | 1.91 × 10$^3$ | 10.1    |
| 79.8   | 1.43 × 10$^5$   | 11.7    | 9.52 × 10$^3$   | 9.70    | 2.18 × 10$^3$ | 7.20    |
| 89.8   | 1.30 × 10$^5$   | 10.2    | 9.03 × 10$^3$   | 37.1    | 2.11 × 10$^3$ | 4.00    |
Theoretically, the required water vapor importing rate to maintain the saturation condition of the test chamber was 1.07 g/min (at 25°C). Considering the total water vapor importing rates (i.e., water vapor containing in the inlet air + water vapor emitted from the TCS burning process) (= 1.12–1.46 g/min) were consistently higher than 1.07 g/min indicating that the test chamber was always on a water saturated condition for all TCS burning experiments. As a result, the effect of humidity on the compositions of TCS burning products becomes insignificant.

### Particle Size Distributions of HCl$_p$

Table 2 shows particle size distributions of HCl$_p$ obtained from the four selected RH conditions. It can be seen that the emitted particles (MMADs = 0.808–1.04 μm; GSDs = 2.13–3.50) fell to the range of the accumulation mode (0.1–2.5 μm). For illustration, Fig. 3 shows the particle size distribution obtained from the test condition of RH = 65.5%. As described in the previous section, HCl$_p$ could be generated by condensation of HCl$_g$ on the emitted SiO$_2$ particles via the heterogeneous condensation process, or via the absorption of HCl$_g$ onto H$_2$O coated on SiO$_2$ particle surface. Based on the above description, the emitted HCl$_p$ particles might fell to the range of the nuclei mode (i.e., 0.1 μm). However, it should be noted that the particle sizes of these primary HCl$_p$ particles might further increase to the accumulation mode because of the effects caused by both the Brownian and turbulent coagulation (Friedlander, 2000). In addition, our results were also consistent with the results obtained by Schenkel and Schaber (1995).

Yet, it is true that high moisture content in the air might lead to an increase in the particle size according to the Kelvin effect theorem (Friedlander, 2000; Hu et al., 2010). In this study, the four selected RH conditions did not affect the emitted particle sizes of HCl$_p$ which might because the test chamber was always on a water saturated condition for all TCS burning experiments (as described earlier).

### Fractions of Inhaled HCl$_p$ Reaching Different Regions of the Respiratory Tract

Fig. 4 shows the fractions of inhaled HCl$_p$ reaching the alveolar region ($F_{%alv}$), tracheobronchial region ($F_{%tb}$), and head region ($F_{%head}$) under the four selected RH conditions during the TCS burning process. For all test conditions, a consistent trend of $F_{%alv}$ (mean = 85.8–88.8 %; RSD = 1.50–7.45 %) > $F_{%tb}$ (mean = 6.53–8.80 %; RSD = 0.50–4.90 %) > $F_{%head}$ (mean = 4.67–5.40 %; RSD = 0.60–2.62 %) was found in the present study. The highest fraction found in the alveolar region indicates that most HCl$_p$ emitted from

### Table 2. Particle size distribution of HCl$_p$ emitted from TCS burning processes under the four selected RH conditions.

| RH (%) | MMAD (μm) | GSD          |
|--------|-----------|--------------|
| 57.3   | 0.939     | 2.13         |
| 65.5   | 1.04      | 3.43         |
| 79.8   | 0.861     | 3.50         |
| 89.8   | 0.808     | 3.49         |

Fig. 3. The particle size distribution of HCl$_p$ emitted from the during the TCS burning process under the test condition of RH = 65.5 %
TCS burning processes could penetrate into the deep lung of the respiratory tract. It is known that HCl g could only reach the upper respiratory tract due to its high water solubility. In particular, as described in the previous section, the emitted $C_{HCG} (= 1.30 \times 10^5–1.46 \times 10^5 \text{ mg/m}^3$) were consistently much higher than $C_{HClg} (= 9.03 \times 10^3–11.4 \times 10^3 \text{ mg/m}^3$) in all experimental campaigns. Our results suggest more serious damage might occur at the deep lung region than other regions of the respiratory tract as workers exposed to contaminants emitted from TCS burning processes.

Theoretical and Measured Total Chloride Emitted Concentrations

Table 3 shows the consumption rates of TCS and the measured total chloride emitted concentrations and their corresponding theoretical values under the four selected RH conditions. No significant difference can be found in consumption rates of TCS among the four selected RH conditions (mean = 8.28–9.39 g/min; RSD = 6.56–19.0 %) (Kruskal-Wallis test, $p > 0.05$). Therefore, it could be expected that no significant difference can be found in the theoretical total chloride emitted concentrations (mean = $1.55 \times 10^5–1.75 \times 10^5 \text{ g/min}$; RSD = 6.56–19.0 %) (Kruskal-Wallis test, $p > 0.05$). On the other hand, we found that there were slight differences between theoretical and their corresponding measured total chloride emitted concentrations (mean = $1.40 \times 10^5–1.60 \times 10^5 \text{ mg/m}^3$; RSD = 8.19–26.1%). Based on the above values, the resultant recoveries (= (measured values/theoretical values) $\times 100\%$) fell to the range of 90.1–91.0 %. The above discrepancies were because the parts of HCl were not sampled in the present study, including those were deposited on the chamber walls, or being absorbed by the H$_2$O coated on the chamber wall. However, the above results also suggest that our results can fairly characterize the emissions of contaminants from the TCS burning processes.

CONCLUSIONS

We found that the HCl p was consistently as the most dominant contaminant, followed by the HCl g and Cl$_2$, emitted from the TCS burning processes. We also found that humidity didn’t have a significant effect on the composition of the above three emitted contaminants under the four RH test conditions. Therefore, lower RH test
conditions are suggested for further research in the future, in particular for TCS could be stored in areas with much lower RH conditions. The emitted HClp fell to the range of particular for TCS could be stored in areas with much lower RH conditions, and the fraction of HClp deposited on the alveolar region was consistently higher than those on tracheobronchial region and head region for the selected RH test range. Therefore, more serious ill-health effect on the deep lung region of the respiratory tract can be expect as workers exposed to contaminants emitted from TCS burning processes. The results obtained from this study will provide information for semiconductor and optoelectronic industries to initiate an appropriate TCS emergency response plan to prevent workers from the ill-health effects associated with toxics emitted from TCS burning processes.

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