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CGA G-13 large-scale silane release tests — Part I. Silane jet flame impingement tests and thermal radiation measurement

Eugene Y. Ngai ^a, Ron Fuhrhop ^b, Jenq-Renn Chen ^{c, *}, Jenny Chao ^d, C. Regis Bauwens ^d, Crystal Mjelde ^e, Gary Miller ^f, Jerry Sameth ^{g, 1}, John Borzio ^h, Michael Telgenhoff ⁱ, Bruce Wilson ^j

- ^a Chemically Speaking LLC, Whitehouse Station, USA
- ^b Praxair Inc., 175 East Park Dr., Tonawanda, NY 14150, USA
- ^c National Kaohsiung First University of Science and Technology, Kaohsiung, Taiwan
- ^d FM Global, 1151 Boston-Providence Turnpike, Norwood, MA 02062, USA
- e REC Silicon, 119140 Rick Jones Way, Silverbow, MT 59750, USA
- f Air Products & Chemicals Inc., 7201 Hamilton Blvd., Allentown, PA 18195, USA
- g MATHESON, 150 Allen Road, Basking Ridge, NI 07920, USA
- ^h Air Liquide, 19 Steel Road West, Morrisville, PA 19067, USA
- ⁱ Dow Corning Corporation, 2200 W. Salzburg Road, Midland, MI 48686-0994, USA
- ^j Linde North America LLC, 1 Greenwich Street, Stewardville, NJ 08886, USA

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ABSTRACT

In early 2011, the G-13 Silane Modeling Task Force of the Compressed Gas Association (CGA) proposed a series of tests to better define pyrophoric behavior during unintentional, large-scale releases of silane. The tests were conducted in two phases under the direction of CGA and its guidelines. Phase I took place from June 27 to June 30, 2011, focusing primarily on thermal radiation and the heat transfer from flame impingement due to silane release from a fully open pressure relief device (PRD) on a tonner. Phase II took place on June 19 and 20, 2012, focusing on thermal radiation and explosion overpressure. The results were subsequently utilized to revise CGA G-13 guidelines on the safe handling of silane. In the present two-part papers, the results from the tests are summarized in order to highlight the key findings. The first part of summary described the results of the flame impingement and thermal radiation tests. Three different test series were conducted, including shakedown tests using nitrogen instead of silane, silane flame-impingement tests onto an adjacent target tonner, and heat-flux tests. For comparison with known values in the published literature, thermal radiation of ethylene flame jets was also measured. In addition, metallurgical analyses of the target tonner indicated that the metallurgical properties of the cylinder material were not altered by the flame impingement. The steel surface temperature at the point of impingement was estimated to be below 853.15 K and definitely did not exceed 950.15 K. Thus, the combination of internal pressure and vessel metal temperature was unlikely to exceed the rupture pressure of the ton cylinder.

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1. Introduction

Silane is one of the most important specialty gases and is widely used in semiconductor, flat panel, and photovoltaic fabrication

E-mail address: jrc@nkfust.edu.tw (J.-R. Chen).

http://dx.doi.org/10.1016/j.jlp.2014.12.010 0950-4230/© 2014 Published by Elsevier Ltd. industries. Silane is a well-known pyrophoric gas that typically ignites upon contact with air. However, a silane release from a pressurized source may not always lead to prompt ignition (Koda, 1992). Delayed ignition allows formation of an explosive silane-air mixture that may ignite explosively causing significant damage (Ngai et al., 2007; Tamanini and Chaffee, 1996a,b). In response to reported silane incidents involving fire or explosion, CGA published a safety standard for the storage and handling of silane titled ANSI/Compressed Gas Associations (CGA) G-13 "Storage and Handling of Silane and Silane Mixtures" 2nd Edition, 2006. G-13 is

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^{*} Corresponding author.

¹ Present address: Compressed Gas Association, 14501 George Carter Way, Chantilly, VA 20151, USA.

recognized worldwide and establishes safety setback distances based upon worst-case scenarios developed using data from testing that was conducted in 1996 by CGA (Chowdhury, 1997). Setback distances listed in G-13 for bulk systems have been questioned since its initial publication. Consequently, and as part of the CGA standards revision process, a Task Force was established to conduct a detailed review of distance-to-exposure setback distances in G-13

As part of the review of setback distances in G-13, the task force decided that testing was required to better define silane behavior during a release. A Working Group was subsequently formed in 2010 to develop a test plan, conduct testing, and analyze data in order to establish appropriate setback distances in the revision of G-13.

Previous tests have been conducted to characterize the impact from silane releases into air. Britton (1990) was among the first to have carried out silane releases at a large-scale. His testing measured overpressure using a sound meter when electronic grade silane at a pressure of 7.0 MPa or greater was vented (released) from a 10.16 cm diameter vertical pipe. The maximum recorded overpressures were 150 dB (0.63 kPa) at 4.8 m from pipe exit, and 147 dB (0.45 kPa) at 9.6 m from pipe exit for a silane release of 7.8 kg. In two tests to measure thermal radiation, the peak radiation at a distance of 12 m was 4.83 kW/m². In 1996 CGA conducted testing on large-scale silane releases which focused on the determination of the setback distances for bulk silane storage systems (Chowdhury, 1997). Six large-scales tests were performed using orifices having diameters of 2.54 and 1.27 cm. Additionally, there were eight small scale tests performed through an orifice having a diameter of 0.32 cm. Discharge testing resulted in three observed outcomes including delayed ignition/explosion followed by a flame jet, immediate ignition/with a flame jet, and silane jet formation with neither immediate nor delayed ignition. Later tests were conducted by Tamanini and Chaffee (1996a,b) to elucidate the ignition behavior of silane. A later series of tests were conducted by Ngai et al. (2007) on release and explosion from cylinder releases in a gas cabinet, following a tragic silane incident in 2005 (Chen et al., 2006; Chang et al., 2007). Additional detailed silane release tests aimed at better understanding of the delay ignition limits were performed by Chen et al. (2010) and Tsai et al. (2010) using small scale orifices of 6.35 to 3.2 mm diameter tubing.

As the utilization of silane has increased, the silane supply chain has also evolved from a single $49\,L\,(15\,kg)$ cylinder to bulk packages ranging from a $450\,L\,(120\,kg$ of silane) tonner (i.e. ton tank) to an ISO tube module or tube trailer that can store 3000 to $6000\,kg$. While the characteristics of a release from a bulk silane system bear similarity to a release from a cylinder, there are complexities that will not appear in a small-scale release. Among these, the size of the vapor cloud explosion and the quantity of jet fire thermal radiation are the most important and significant issues that must be addressed.

The current ANSI/CGA G-13 standard established the minimum separation or exposure setback distances for an ISO Module or Tube Trailer based upon the risk from overpressure generated during explosive ignition and for cylinders and tonners based upon the risk from thermal radiation generated by a flame jet. To address setback distance questions, the G-13 revision committee reviewed the basis for exposure distances, focusing on thermal radiation and delayed ignition as principal risks. Based on reviewing incidents, industry statistics, fault tree analysis, and risk assessment, thermal radiation from a flame jet was considered appropriate in determining setback distances for all packages and will be proposed in the updated version of G-13. It should be noted that setback distances based on overpressures can also be considered if the user so chooses; guidance will be provided in Appendix D of the proposed revision.

Reflecting the focus on thermal radiation instead of overpressure, the 2011 test program (Phase I) focused on the effects of silane flame jet release and thermal radiation from a tonner with a secondary study of delayed ignition and overpressure. Over 20 releases were performed in Phase I for determining:

- 1. Radiant heat from a full flow PRD release from a tonner;
- 2. The effect of flame impingement on a tonner filled with nitrogen from a PRD discharge of an adjacent silane tonner;
- 3. Radiant heat from vertical and horizontal flame jets;
- 4. Overpressures from delayed ignition of unobstructed silane releases into the open atmosphere;
- 5. Overpressures from delayed ignition of obstructed silane releases into the open atmosphere.

Additional tests were performed in the 2012 test program (Phase II) that was focused on:

- 1. Overpressures from delayed ignition of unobstructed silane releases into the open atmosphere;
- 2. Overpressures from delayed ignition of obstructed silane releases into the open atmosphere;
- 3. Radiant heat of silane flame jets released from a 49 L cylinder.

These results from testing conducted in 2011 and 2012 were subsequently utilized in the revision process of the CGA G-13 standard in order to establish appropriate exposure setback distances. In particular, because there is a drive to eliminate the use of PRDs on silane containers, these results can be used to determine setback distances for containers with and without PRDs in the proposed revision of G-13. In the present paper, only the results from the tests on the heat flux measurement of the silane jet flame and flame impingement are summarized in order to highlight the key findings.

2. Experimental setup

2.1. Flame-impingement tests

The silane test system was fabricated and supplied by REC Silicon. It consisted of a "source" tonner, to provide silane for the release, a "severed" tonner where the silane was released through a PRD, and a "target" tonner where the silane flame impinged upon. A standard tonner is a seamless steel pressure receptacle manufactured to DOT 3AAX2400 cylinder specification with a nominal water capacity of 450 L and is commonly referred to as a tonner, ton

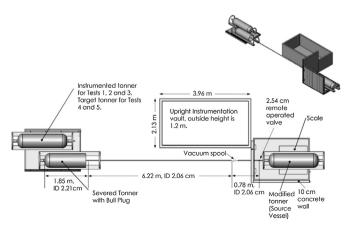


Fig. 1. Configuration for nitrogen shakedown tests.

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