42 ft^3 High Pressure Blowdown Data From the CGA 1996 Large-Scale Test Series

A Presentation to The DIERS Users Group

by

Dr. G. A. Melhem

ioMosaic Corporation
93 Stiles Road
Salem, New Hampshire
03079

Presentation Outline

- Background information
- Issues with silane storage/use
- Large scale experiments and results
- A short video

Industrial Uses of Silane

- Numerous uses in semiconductor industry
- Chemical vapor deposition (CVD) of thin dielectric films on substrates using thermal or plasma heating
- LPCVD
- Manufacture of high purity polycrystalline silicon and silicon nitride and epitaxial silicon

Project Outline

- Funding
  - Funded by the compressed gas association (CGA)
- Management
  - Managed by CGA and Air Products
- Data Users
  - CGA member companies
  - Fire code officials
  - SemaTech
  - Companies such as Motorola, Intel, etc.
- Test site
  - EMRTC, New Mexico

Project Outline

- HAZOP study on bulk storage design
- Fault tree analysis on identified scenarios
- Fire and explosion damage contours
- Estimate safe separation distances for fire code officials
- Conduct large scale experiments to validate model predictions
- Publish the data and results with CGA
- Provide training to CGA member companies on silane handling

Silane is "pyrophoric" and can theoretically ignite in bulk

- Pyrophoricity does not guarantee spontaneous ignition
- Bulk autoignition leads to consumption of reactants instantaneously
  - The associated pressure rise rate is infinite
  - No means for explosion relief are possible other than total containment
- Bulk autoignition is possible for uniform, well mixed clouds
- Most likely ignition scenario for non-uniform clouds is deflagration to detonation transition

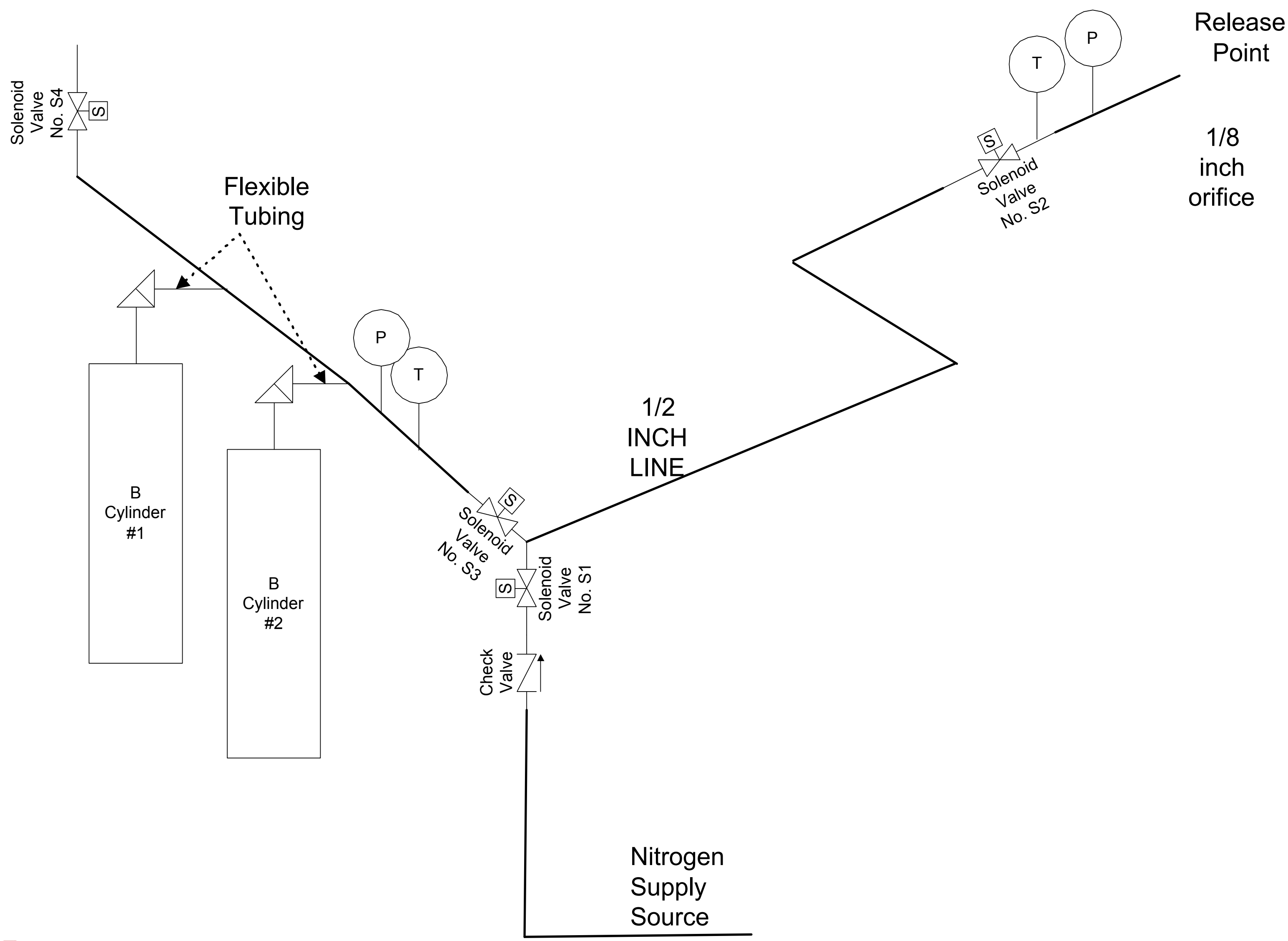
The potential “accumulation” of flammable silane is key to effective hazard management of bulk storage installations

- Silane users would like to move to bulk storage instead of cylinders
- FM Guideline for siting uses 30 seconds of flow to estimate accumulation. For a 1 inch release this translates to 600 kgs of silane.
- Our detailed models estimated 30 kgs
- Actual experiments showed 1.8 kgs !

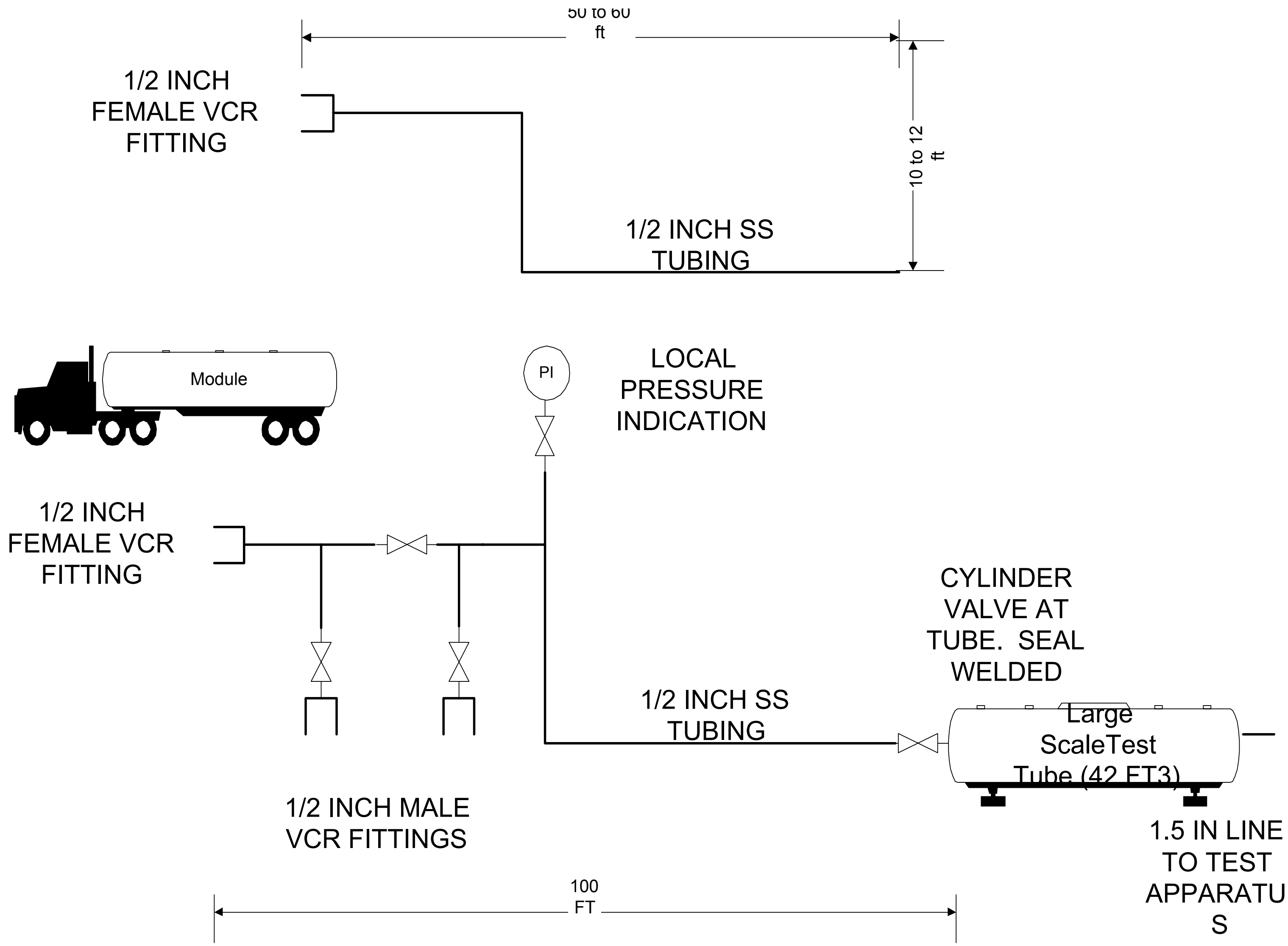
Test Conditions

- 1/8, 1/2 and 1 inch release diameter
- 1100 - 1200 psig vessel pressure
- 56 - 78 F vessel temperature
- 20 % relative humidity
- Variable wind speed, 4 to 11 mph
- Test fluids include Silane and Nitrogen

Small Scale Test Setup

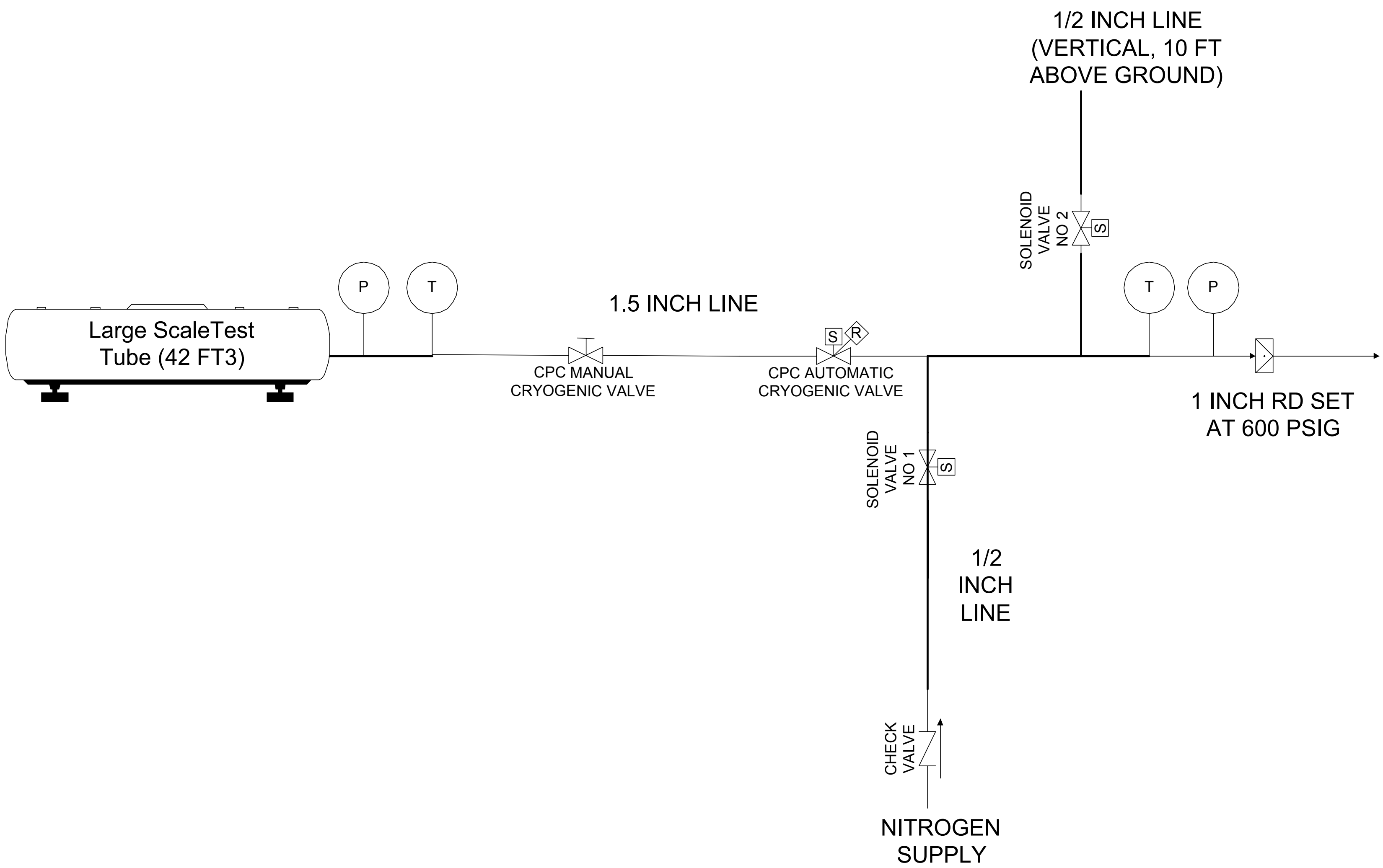


Large scale test setup. Transfill connections to silane module

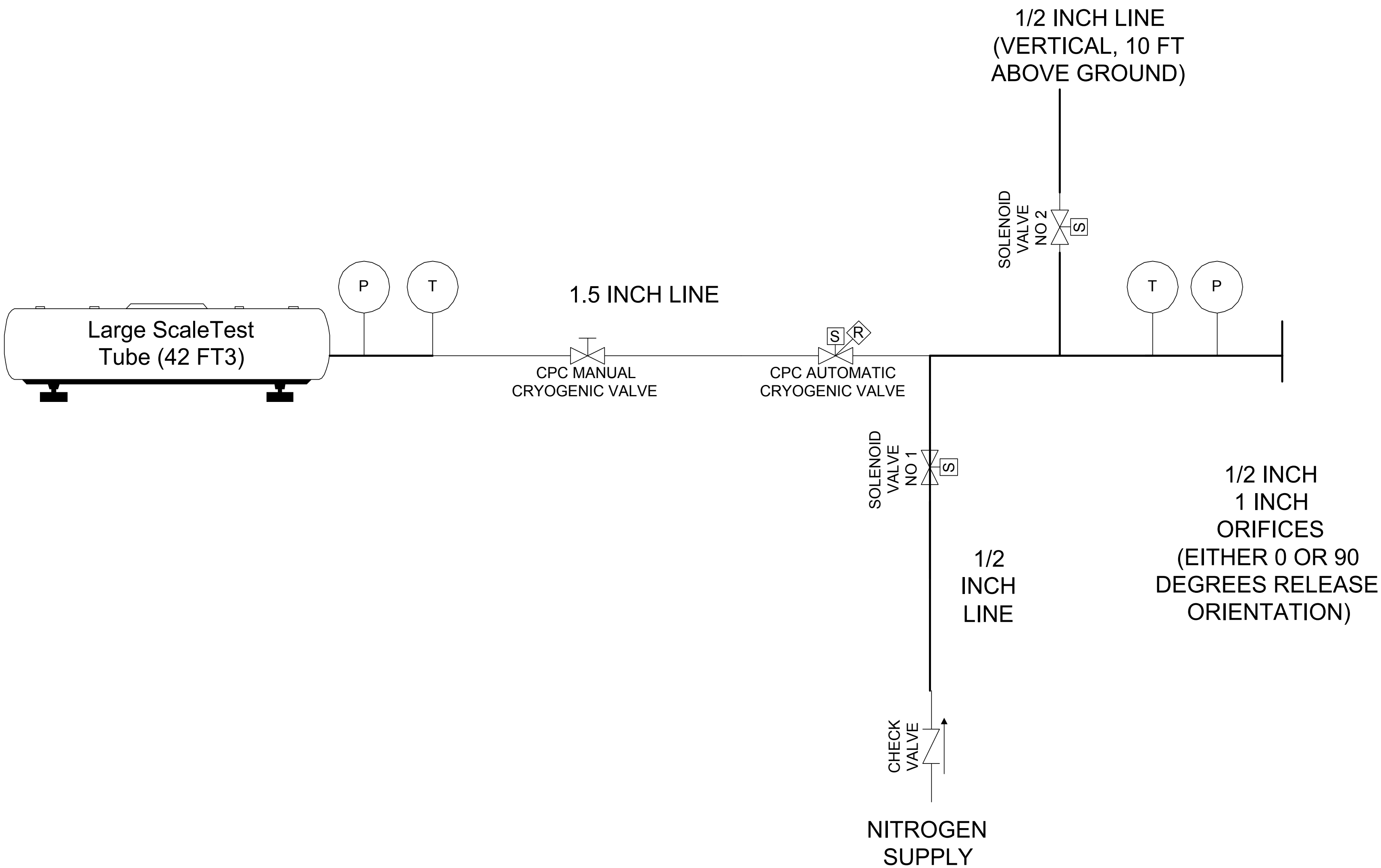


ioMosaic
discovering solutions

Large Scale Test Setup: Rupture Disk Configuration



Large Scale Test Setup: 1/2 inch and 1 inch orifice release configuration



ioMosaic
discovering solutions

Large Scale Test Matrix Summary

	Test #2	Test #3	Test #4	Test #5	Test #6	Test #7	Test #8	Test #9
Test Date	9/26/96	9/26/96	9/27/96	9/27/96	9/28/96	9/28/96	9/30/96	9/30/96
Test Time	7:26 am	4:05 pm	2:50 pm	7:10 pm	1:15 pm	3:15 pm	12:20 pm	4:48 pm
Ambient Temperature (F)			62.00	60.00	82.40	81.00	76.00	74.00
Relative Humidity (%)			17.00	21.00	20.00	14.00	26.00	29.00
Wind Speed (mph)			3.50	1.00	1.80	1.00 - 4.5	10.00	3.00 - 5.00
Test Fluid	N2	N2	SiH4	SiH4	SiH4	SiH4	SiH4	SiH4
Storage Volume (ft3)	42.00	42.00	42.00	42.00	42.00	42.00	42.00	42.00
Storage Pressure (psig)	840.00	1430.00	980.00	920.00	980.00	1000.00	940.00	1000.00
Storage Temperature (F)	49.40	69.20	65.60	60.20	80.00	80.00	75.00	73.00
Storage Mass (kg)	88.00	138.00	230.00	245.00	180.00	205.00	190.00	217.00
Orifice Diameter (in)	1.00	0.50	1.00	0.50	1.00	1.00	0.50	0.50
Release Orientation	Vertical	Vertical	Vertical	Vertical	Horizontal	Vertical	Horizontal	Horizontal
Release Blockage	No	No	No	No	No	No	Flat Plate	Flat Plate
RD Set Presure (psig)			600.00			600.00		

ioMosaic discovering solutions

Small Scale Test Matrix Summary

	Test #12	Test #13	Test #14	Test #15	Test #16	Test #17	Test #18	Test #19	Test #20
Test Date	10/1/96	10/1/96	10/2/96	10/2/96	10/2/96	10/2/96	10/2/96	10/3/96	10/3/96
Test Time	2:43 pm	3:48 pm	9:37 am	10:45 am	11:45 am	2:00 pm	3:00 pm	10:00 am	11:30 am
Ambient Temperature (F)	83.00	81.00	68.00	71.00	75.00	81.00	82.00	56.00	62.00
Relative Humidity (%)	23.00	22.00	37.00	32.00	31.00	22.00	22.00	67.00	57.00
Wind Speed (mph)	6.00-12.0(	5.00-10.0(	5.00-7.00	3.00-6.00	3.00-7.00	5.00-6.00	5.00-6.00	7.00-11.00	2.00-7.00
Test Fluid	N2	SiH4	SiH4	SiH4	SiH4	SiH4	SiH4	SiH4	SiH4
Storage Volume (ft3)	1.73	3.46	3.46	3.46	3.46	3.46	3.46	3.46	1.73
Storage Pressure (psig)		1385.00	1260.00	1300.00	1460.00	1350.00	1450.00	1260.00	1300.00
Storage Temperature (F)									
Storage Mass (kg)	6.12	31.98	28.58	27.22	29.03	26.54	See (1)	27.90	15.88
Orifice diameter (in)	1/8	1/8	1/8	1/8	1/8	1/8	1/8	1/8	1/8
Release Orientation	Horizontal	Horizontal	Vertical	Vertical	Vertical	Horizontal	Horizontal	Horizontal	Horizontal
Release Blockage	No	No	No	No	No	No	Flat Plate	Flat Plate	Flate Plate, (2)
RD Set Pressure (psig)									
Comments/Observations/Notes	1- Release orifice pulgged due to silica formation								
	2- Overall confinement of release location								

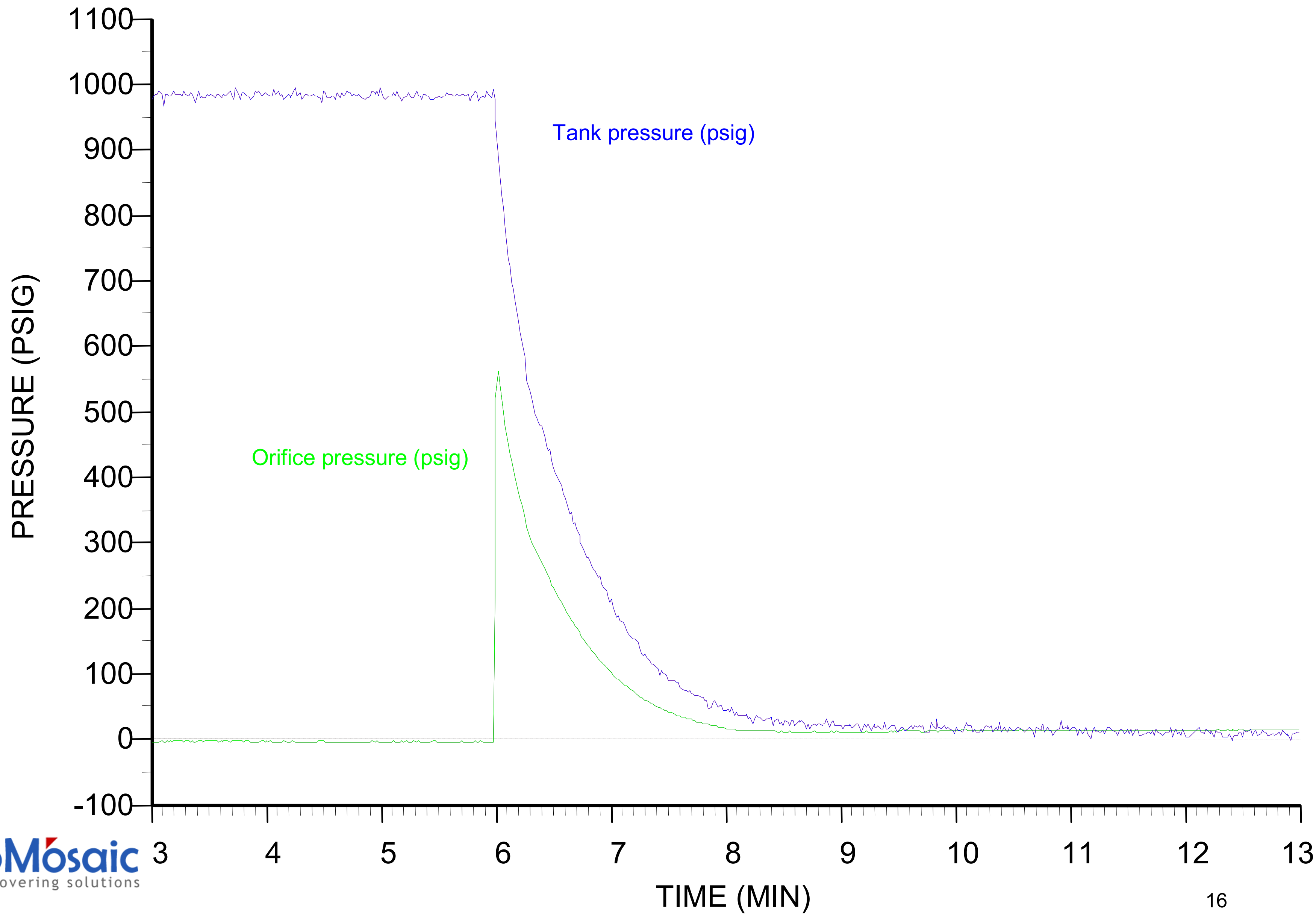
The tests were designed such that the impact of the following variables on flammable mass accumulation can be quantified:

- Release orifice diameter
- Presence of blockage
- Release orientation
- Source pressure

While conducting the tests, the following data was collected:

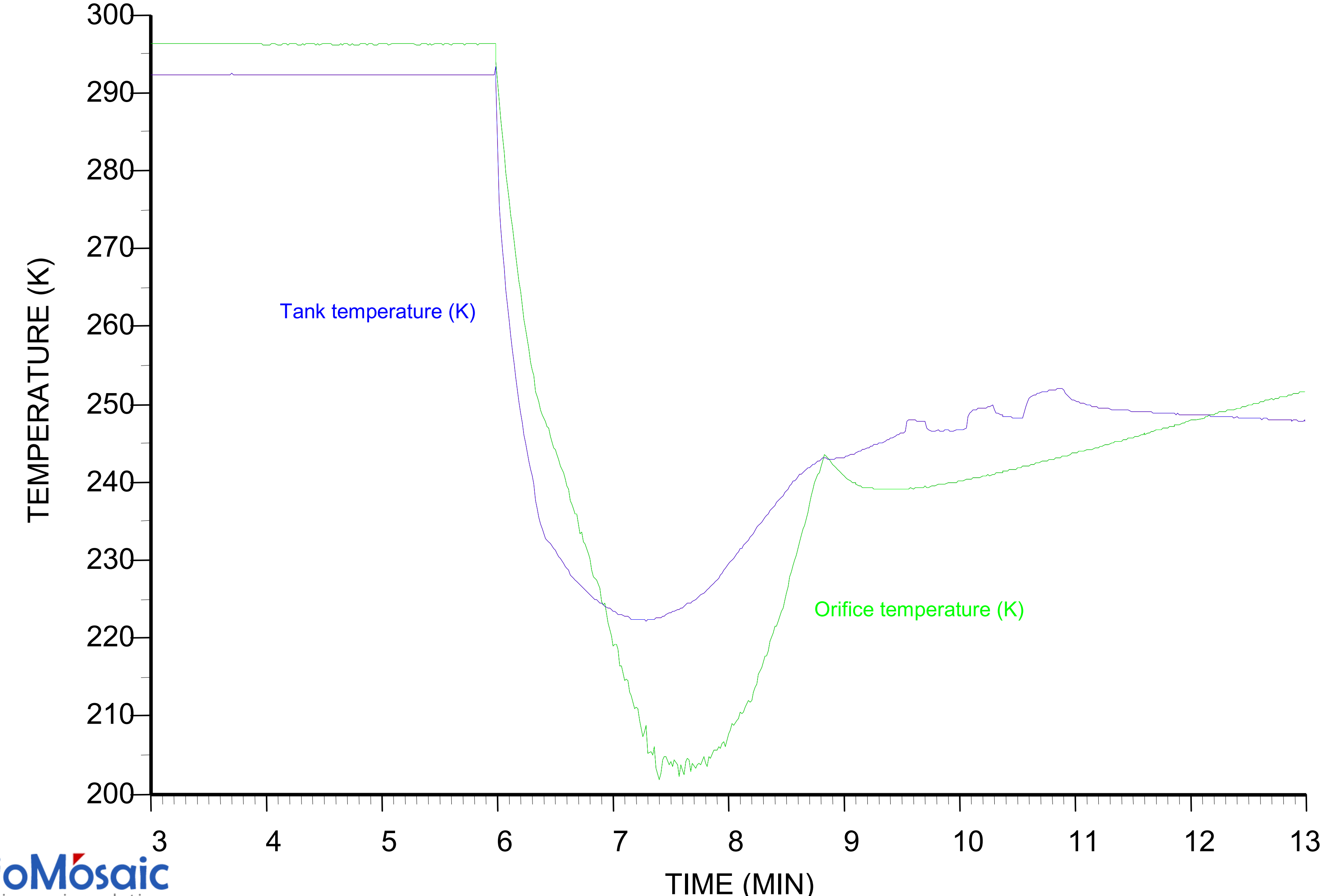
- Visual recording of all tests
- Wind speed, ambient temperature, pressure, and relative humidity
- Eight overpressure measurements
- Silane supply and flow data including temperature and pressure measurement at source and upstream of the flow orifice

Sample Data From Test 4



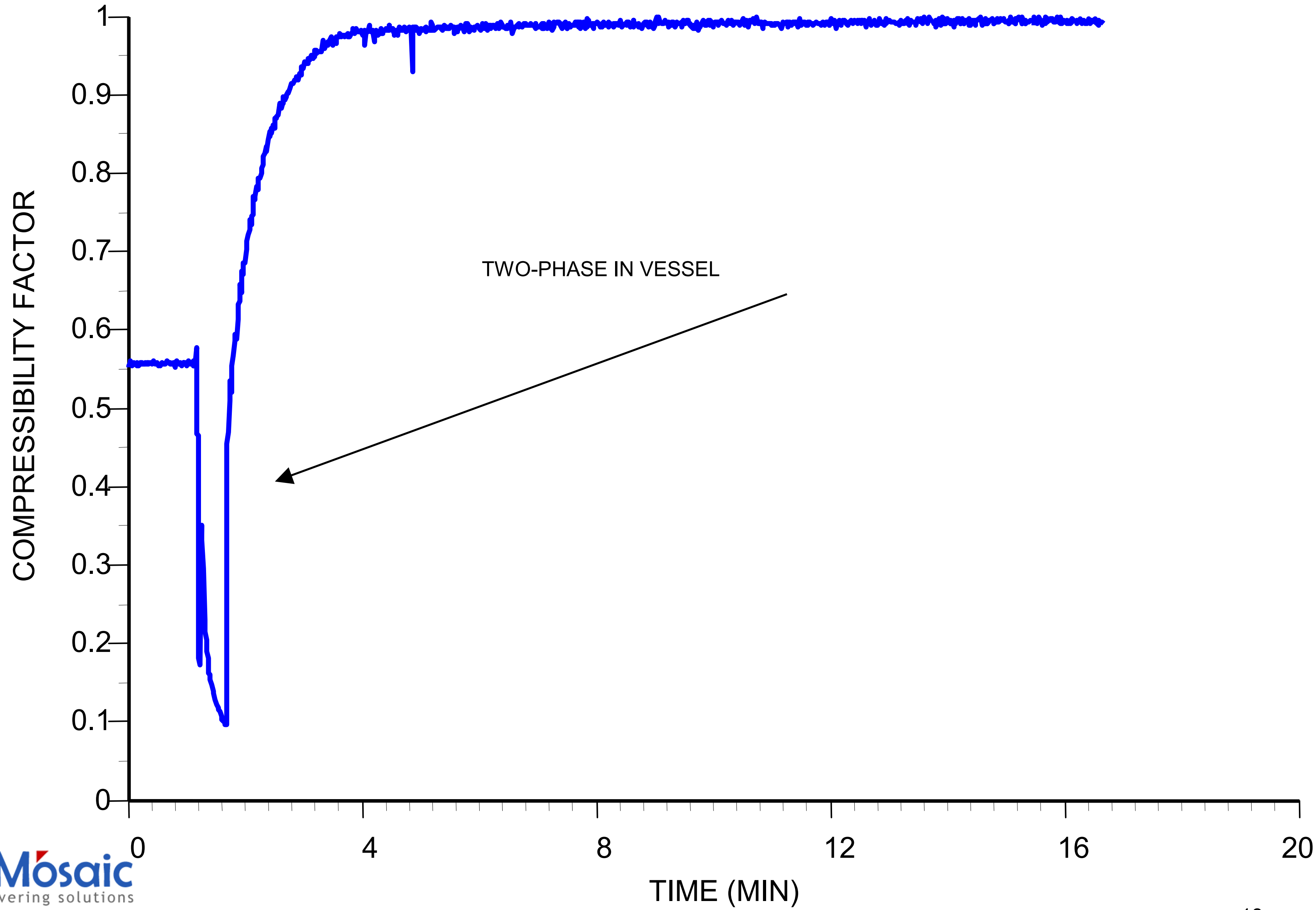
ioMosaic discovering solutions

Sample Data From Test 4



ioMosaic
discovering solutions

Sample Data From Test 6

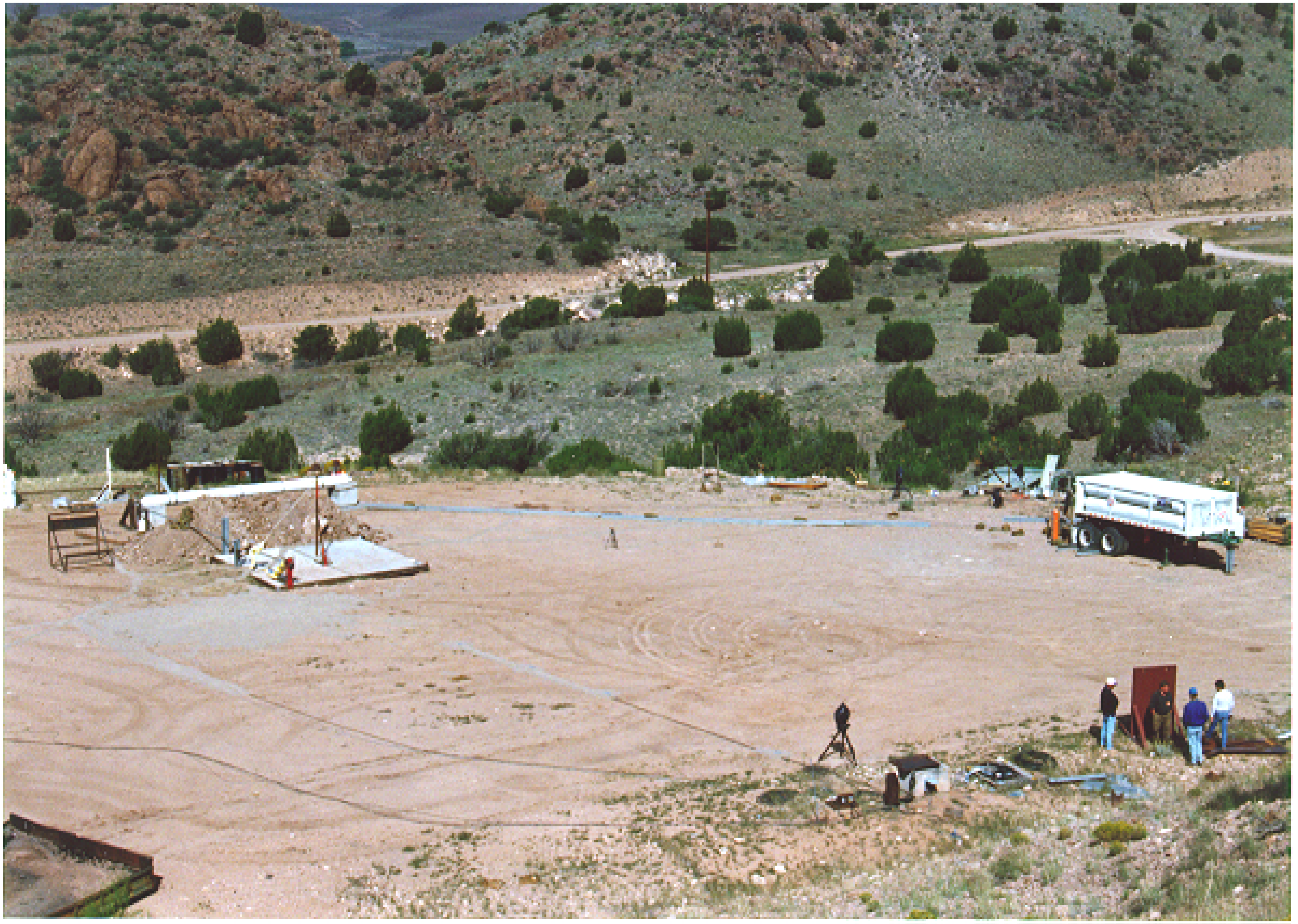


Analysis of Flow Data

- For all the 1 inch orifice/rupture disk releases, where rapid depressurization of the tube contents occurred, the tube contents became two-phase
- Two-phase flow was not observed for the 1/2 inch or the 1/8 inch releases
- Rapid depressurization for tests with 1 inch release orifice resulted in all gas flow followed by two-phase flow followed by all gas flow
- The silane flow was regulated by the orifice
- This is evident from the pressure profiles at the source and just upstream of the orifice
- The pressure drop in the 1.5 inch line was minimal
- The estimated peak flow rate for the 1 inch, 1/2 and 1/8 inch orifice releases agreed well with the experimental values

Conclusions and Recommendations

- All observed silane explosions can be classified as bulk autoignitions or detonations
- Peak accumulation of flammable silane is independent of release duration
- Peak accumulation of flammable silane is a strong function of storage temperature, pressure and release orifice diameter
- Rapid depressurization for tests with 1” release orifice resulted in all gas flow followed by 2-phase flow followed by all gas flow
- Release orifice diameter effect is evident from the 1/8, 1/2 inch, and 1 inch tests
- Tests 4 and 7 (RD tests) require more analysis of flow data. Reducing the velocity upon rupture disk actuation by “swedging up” may lead to immediate ignition

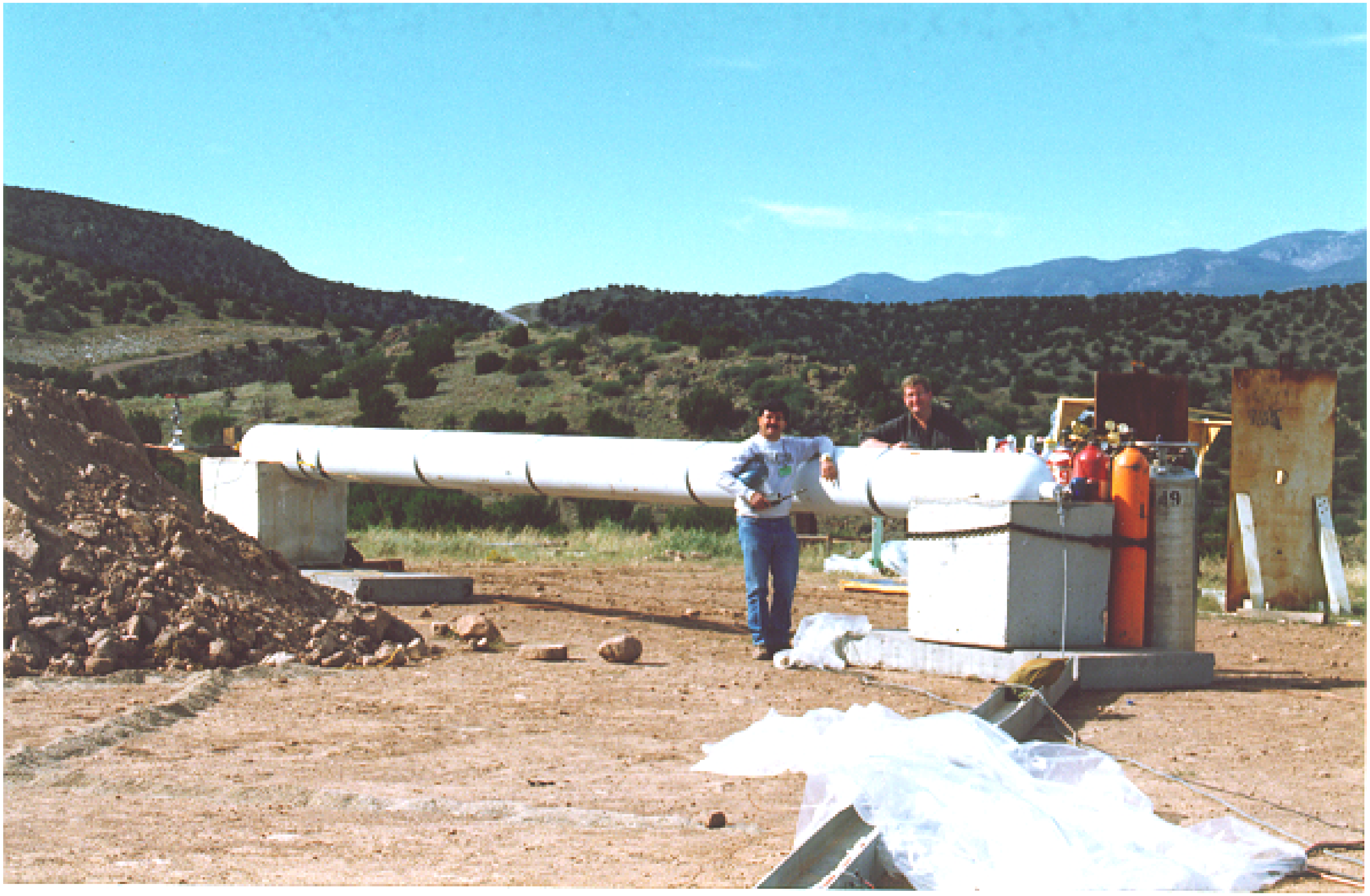


ioMosaic
discovering solutions

ioMosaic
discovering solutions

ioMosaic
discovering solutions

ioMosaic
discovering solutions



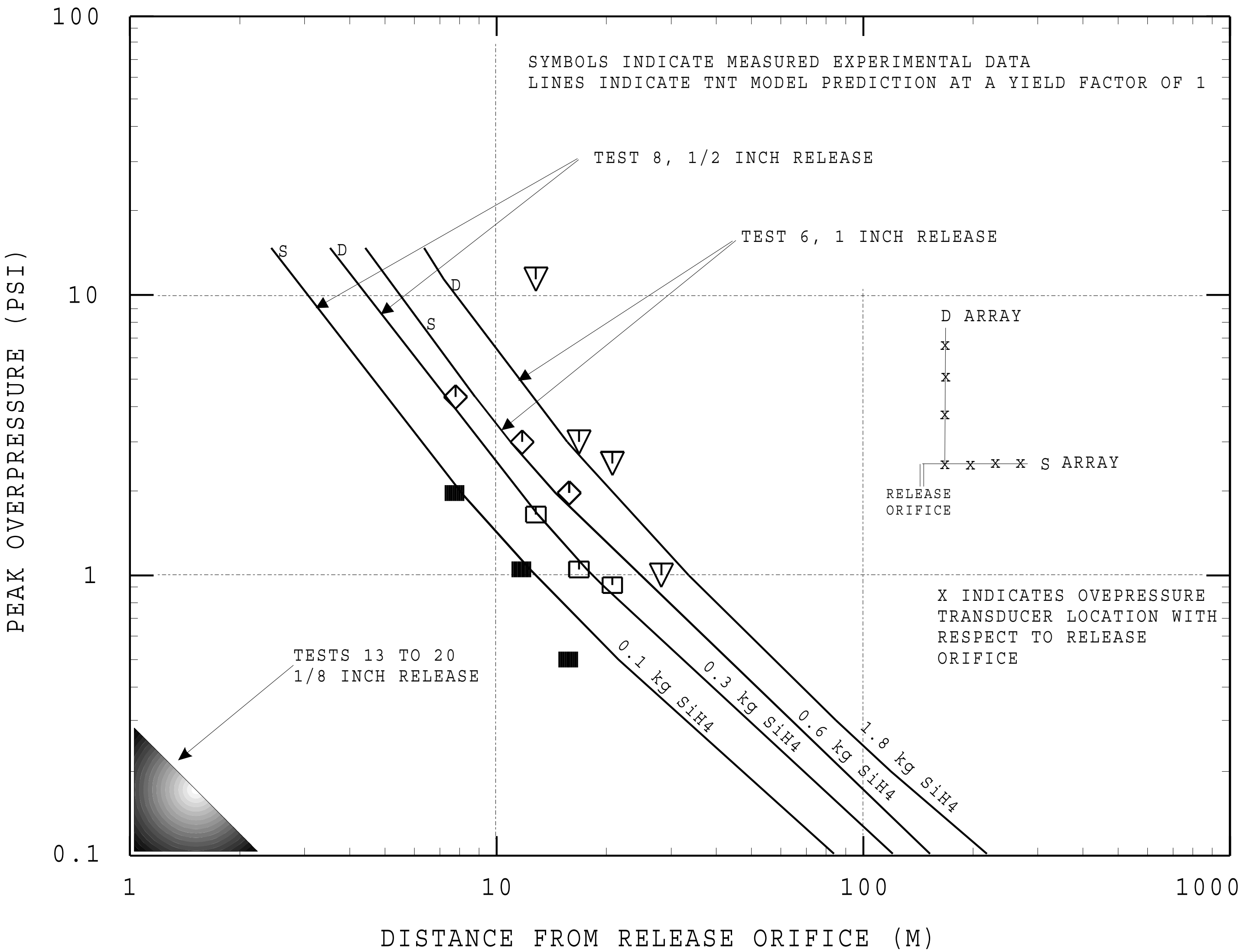
ioMosaic
discovering solutions


ASiMI
ADVANCED SILICON MATERIALS INC.
2203


EMRTC


1
2
3
4
5
6
7
8
9

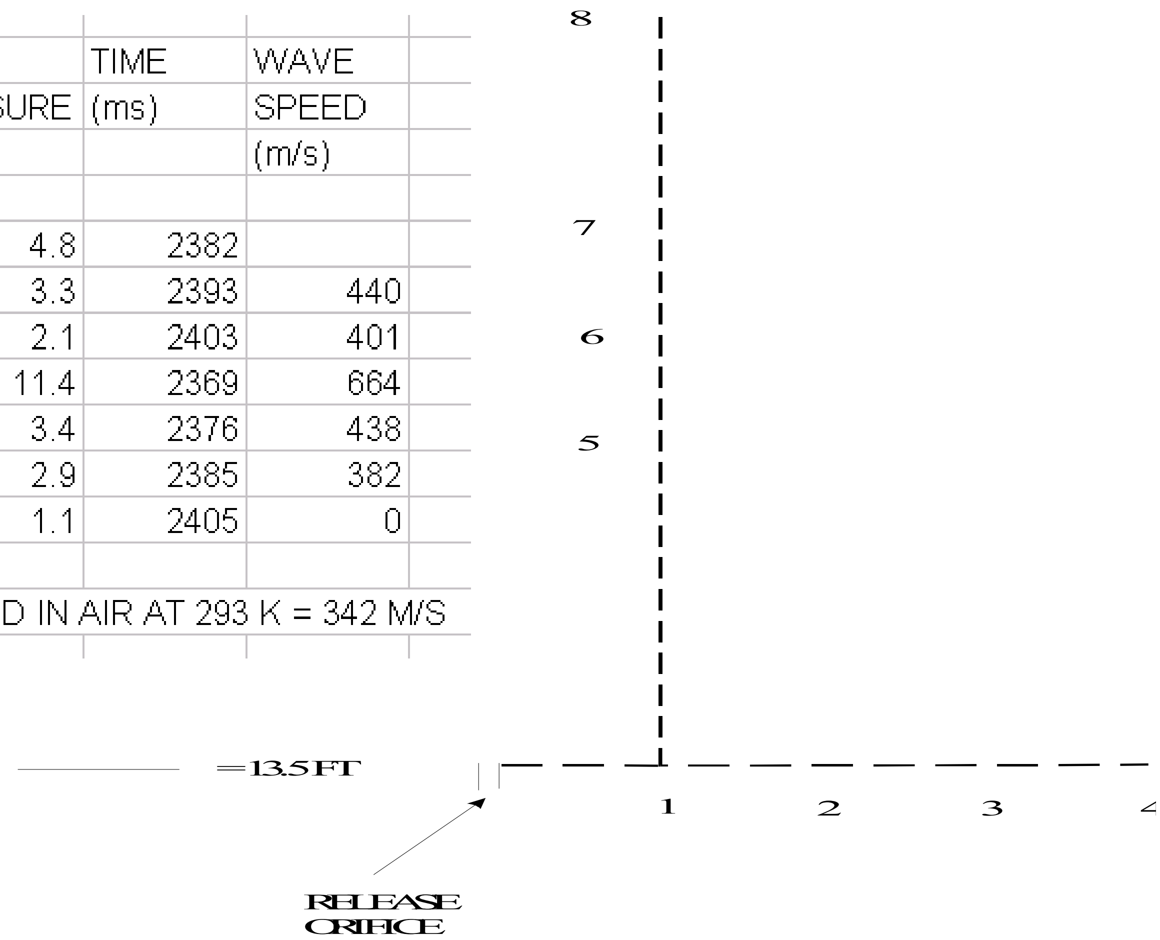
Measured Peak Overpressure Data From Large Scale Tests 6 and 8



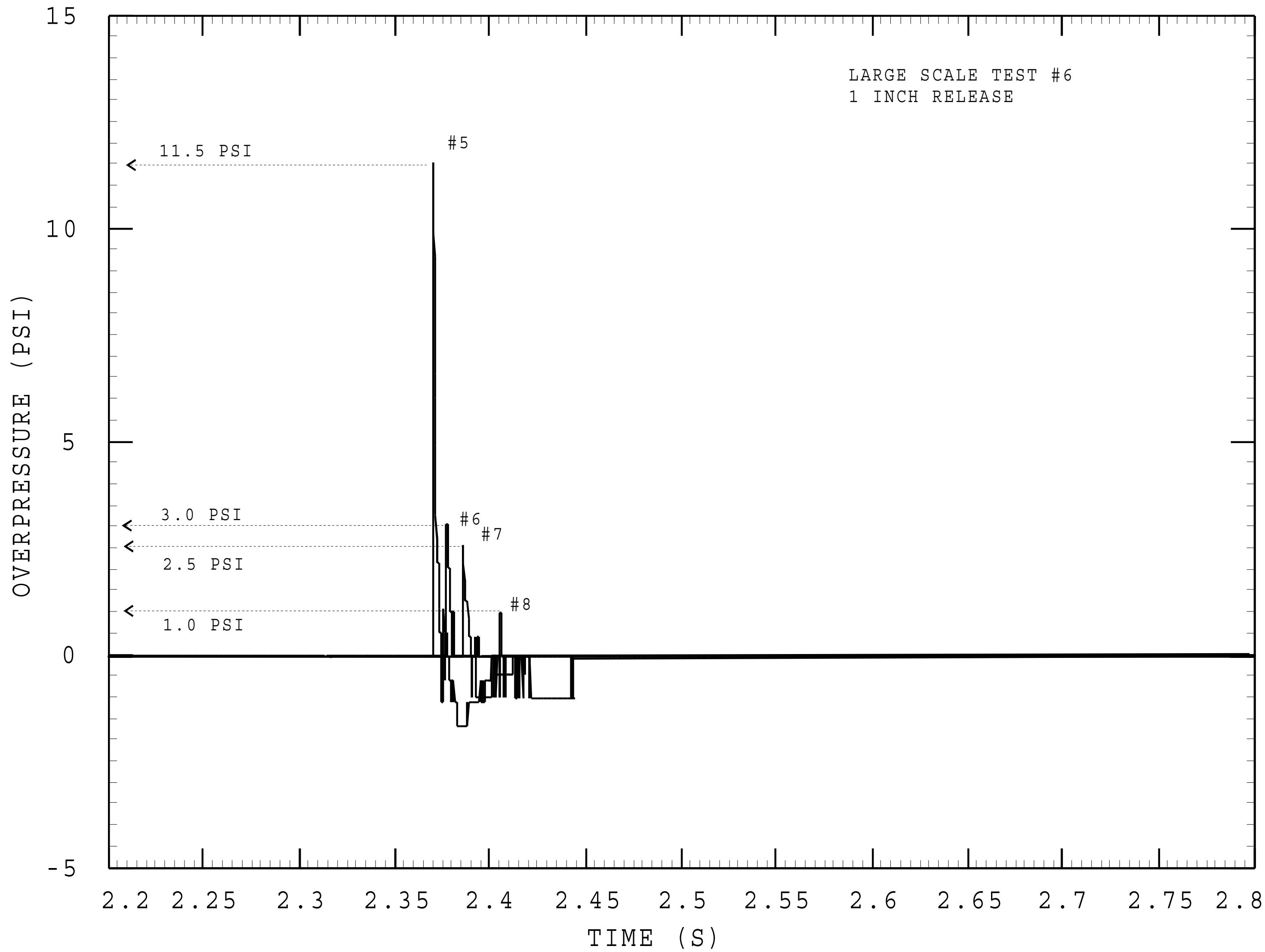
ioMosaic discovering solutions

Overpressure transducers placements and measured time of arrival data for large

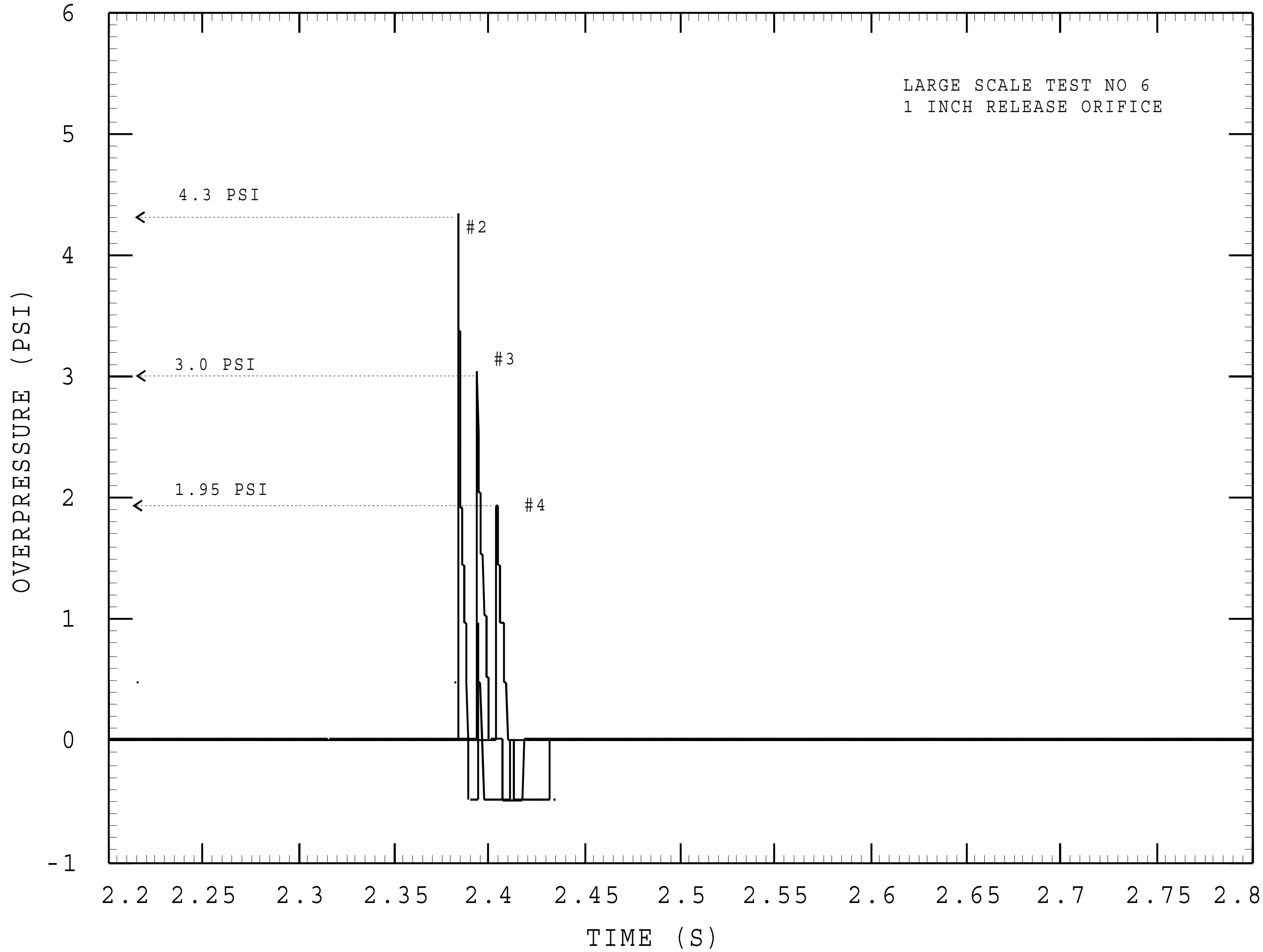
TRANSD NO	PEAK PRESSURE (PSI)	TIME (ms)	WAVE SPEED (m/s)
2	4.8	2382	
3	3.3	2393	440
4	2.1	2403	401
5	11.4	2369	664
6	3.4	2376	438
7	2.9	2385	382
8	1.1	2405	0
SPEED OF SOUND IN AIR AT 293 K = 342 M/S			



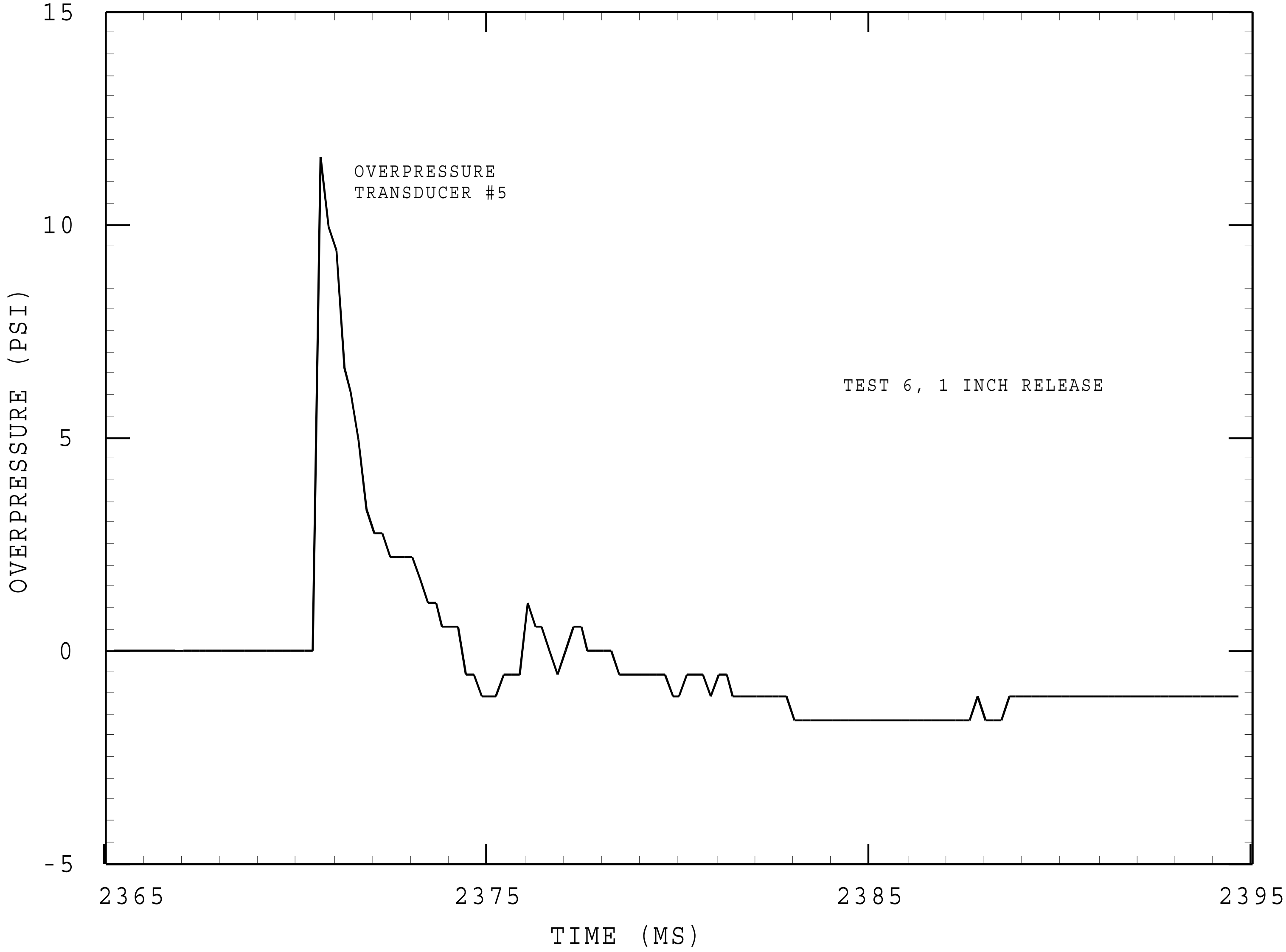
Measured overpressure profiles for large scale test No. 6 at locations 5, 6, 7, and 8.



Measured overpressure profiles for large scale test No. 6 at locations 2, 3, and 4.

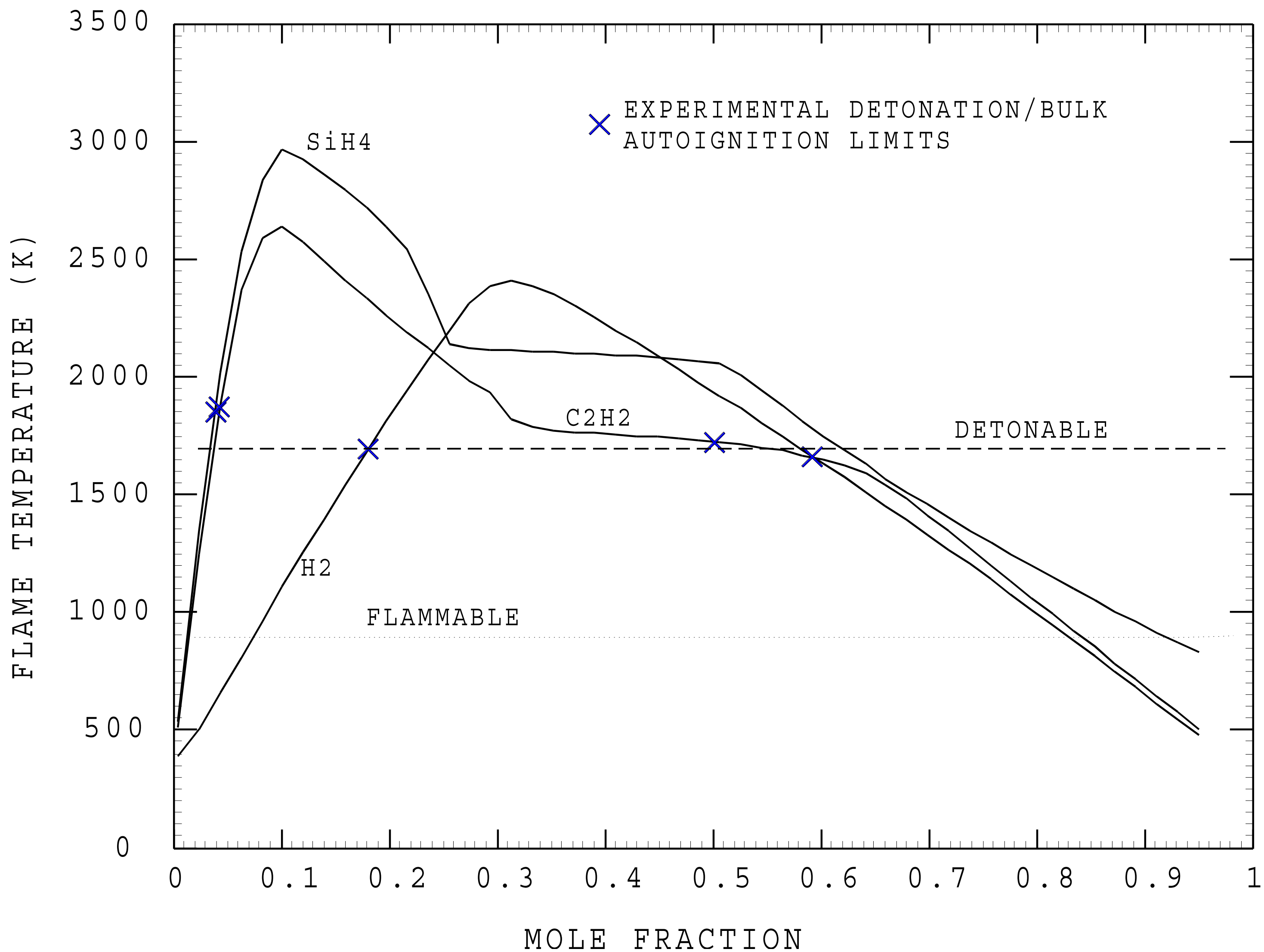


Measured overpressure profile for large scale test No. 6, transducer No. 5



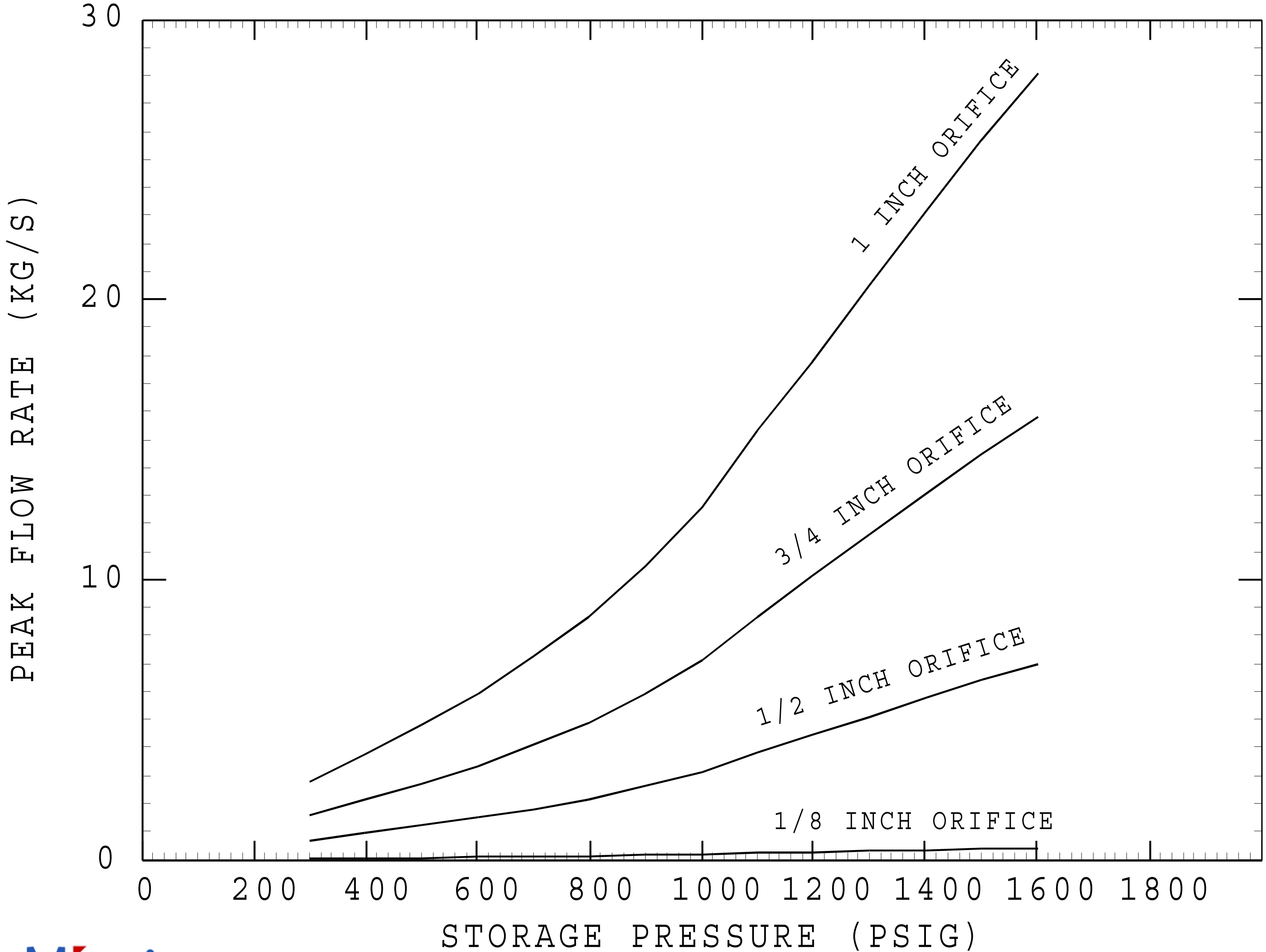
ioMosaic
discovering solutions

Flammability and explosive concentration limits for hydrogen, acetylene and silane

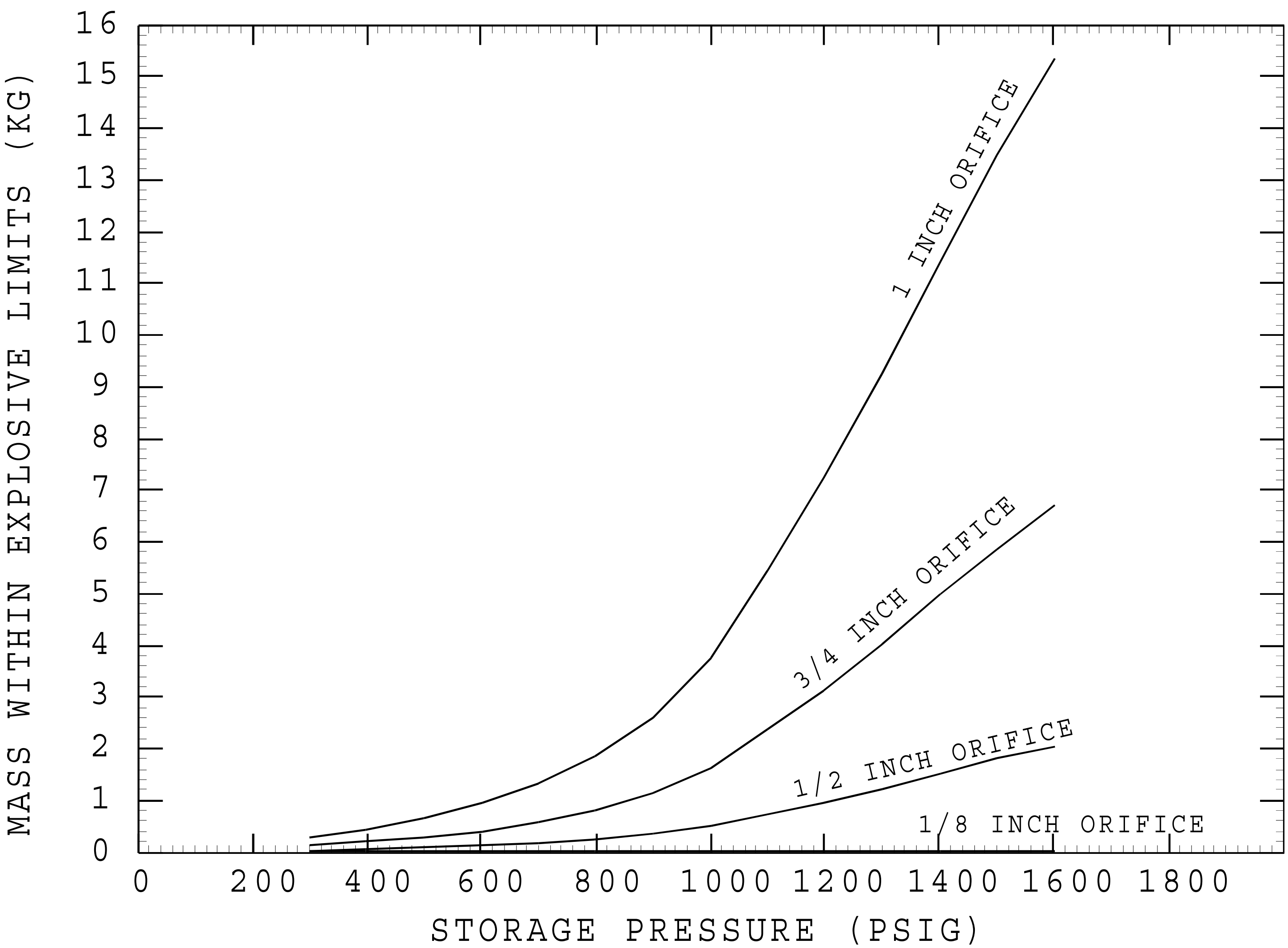


ioMosaic discovering solutions

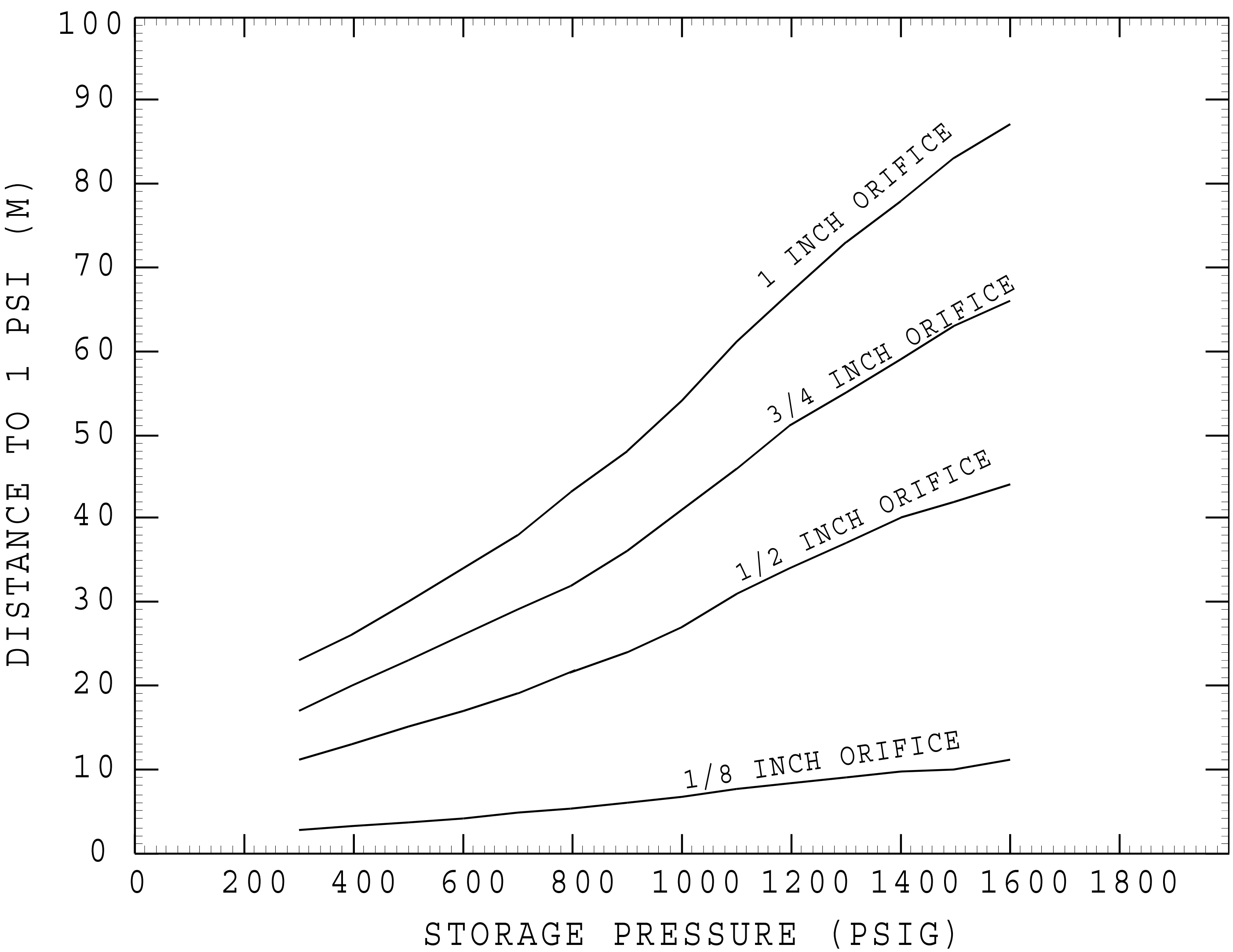
Peak silane release rate as a function of release orifice diameter and storage pressure at 25 C



Silane mass within explosive limits as a function of storage pressure and orifice diameter at 25 C



Distance to 1 psi overpressure as a function of storage pressure and release orifice diameter at 25 C



ioMosaic
discovering solutions