Safety Procedures

Laboratory reactors and pressure vessels


Overview

- Consider the chemistry
- Barricades and ventilation
- Loading limits
- Pressure and temperature limits
- Materials of construction

Consider the chemistry

Basic safety questions

- Is the reaction exothermic?
- What by-products what will be their behavior?
- What maximum temperature and pressure will be observed?
- Under what circumstances (temperature, pressure and catalyzing agent) might the reaction run out of control?

Consider the chemistry

Violent chemical behavior

- Cautious handling of reactions which might release sudden surges of heat or pressure.
- Close attention must be given to any by-products or end-products suspected to have explosive or detonating properties.


I didn't know it would explode...

Explosions results reactions with sudden liberation of heat or volumes of gas or both.

With new or unfamiliar materials always advisable to run preliminary experiments with small volumes of reactants.


I didn't know it would explode…

NAME	STRUCTURE
Acetylide	$-C \equiv C-\text{Metal}$
Amine oxide	$\geq \overset{\oplus}{N}-\overset{\ominus}{O}$
Azide	$-N=\overset{\oplus}{N}=\overset{\ominus}{N}$
Chlorate	$-ClO_3$
Diazo	$-N=N-$
Diazonium	$(-N \equiv N)^{\oplus}X^{\ominus}$
Fulminate	$-O=N=C$
N-Haloamine	$-N(Cl)(X)$
Hydroperoxide	$-O-O-H$
Hypohalite	$-O-X$
Nitrate	$-O-NO_2$
Nitrite	$-O-NO$
Nitro	$-NO_2$
Nitroso	$-NO$
Ozonide	$-O-O-$ (bridged by O)
Peracid	$-C(=O)-O-O-H$
Perchlorate	$-ClO_4$
Peroxide	$-O-O-$

Barricades and ventilation

- Pressure reactors should be located close to a laboratory hood or exhaust fan. (safe discharge of released gases)
- Over-pressurization can be prevented by safety rapture disc. (bursting rapture disc can damage the hearing)
- If the shock wave of the explosion fast enough, it can damage the bomb before the rapture disc can release → Barricade!


Loading limits

- M … l
  ha
  av
- D
  su … in a
  cl
- **M**

LIQUID VOLUMES AND VAPOR PRESSURES FOR WATER IN A CLOSED VESSEL AT ELEVATED TEMPERATURES

Temperature, T °F	°C	Specific Volume of the Liquid cu.ft./lb.	Vapor Pressure psig (gage)	Volume Multiplier $Sp.V_T/Sp.V_{77F}$	Volume Increase %
77	25	.01607	—	1.00	0
212	100	.01672	0	1.04	4
392	200	.01853	211	1.15	15
482	250	.0201	562	1.25	25
540	282	.0215	948	1.34	34
572	300	.0225	1230	1.40	40
610	321	.0241	1650	1.50	50
660	349	.0278	2350	1.73	73
685	363	.0315	2780	1.96	96
700	371	.0369	3070	2.30	130
702	372	.0385	3120	2.40	140
704	373	.0410	3160	2.55	155
705 (Critical Point)	374	.0503	3190	3.13	213

Data from Keenan & Keyes, "Thermodynamic Properties of Steam", John Wiley & Sons, Inc., New York

MAWL=(0.9*Bomb Volume/Volume Multiplier at Max. Temp.)

Pressure and temperature limits

- Maximum temperature and pressure will depend on the design and the materials used to build the vessel.
- All materials loose strength at elevated temperatures → Ratings must be stated in terms of the temperature at which it applies.
- **P**ressure **R**ating **F**actors can be used to convert pressure ratings to higher or lower temperatures.

Pressure and temperature limits

Pressure Rating Factors

					Temperature — °C					
MATERIAL	**25**	**100**	**200**	**300**	**350**	**400**	**450**	**500**	**550**	**600**
T316 Stainless Steel	1.13	1.13	1.09	1.04	1.00	0.97	0.95	0.93	0.90	0.75
Alloy 400	1.20*	1.20*	1.20*	1.20*	1.19	1.11	0.54	0.24 at 472°C Maximum		
Alloy 600	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	0.75	0.42
Alloy B-2	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20* at 427°C Maximum			
Alloy C-276	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	1.20*	0.88
Nickel 200	0.60	0.60	0.60	0.60	0.60 at 316°C Maximum					
Alloy 20CB	1.20*	1.20*	1.17	1.16	1.16	1.16	1.15 at 427°C Maximum			
Titanium Grade 2	0.75	0.64	0.51	0.36	0.34 at 316°C Maximum					
Titanium Grade 4	1.20*	1.20*	0.81	0.63	0.47 at 316°C Maximum					
Zirconium Grade 705	1.20*	0.98	0.76	0.65	0.61	0.60 at 371°C Maximum				

*Ratings higher than 1.20 cannot be applied without checking all aspects of the design.


Materials of construction

- Each alloy has its own physical strength, temperature characteristic and unique resistance to certain corrosive agents.

Nominal Chemical Composition of Pressure Vessel Materials

	Major Elements — Percent					
Material	**Fe**	**Ni**	**Cr**	**Mo**	**Mn**	**Other**
T316 Stainless Steel	65	12	17	2.5	2.0	Si 1.0
Alloy 20CB	35	34	20	2.5	2.0	Cu 3.5, Cb 1.0 max
Alloy 400	1.2	66				Cu 31.5
Alloy 600	8	76	15.5			
Alloy B-2	2	66	1	28	1	Co 1.0
Alloy C-276	6.5	53	15.5	16	1	W 4.0, Co 2.5
Nickel 200		99				
Titanium Grade 4		Commercially pure titanium				Ti 99 min
Zirconium Grade 705		Zr 95.5 min, Hf 4.5 max, Co 2.5				

Materials of construction

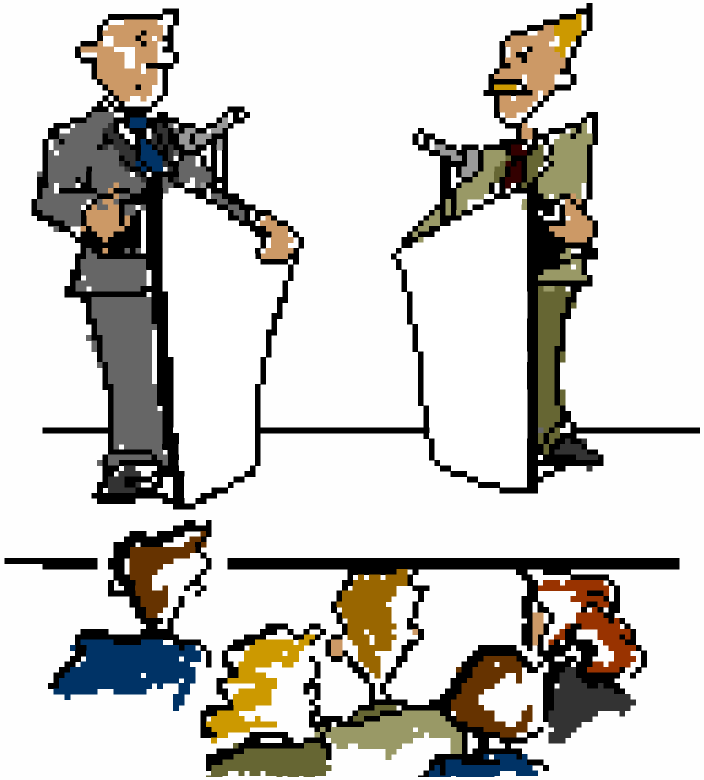
<u>T316 Stainless Steel:</u>

- Excellent material for organic systems.
- Not normally the material of choice for inorganic systems:
  - At elevated temperature sulfuric phosphoric and nitric acids readily attack T316.
- Excellent resistance to surface corrosion with caustics, but above 100°C stress corrosion cracking can be problematic.
- Halogen salts can cause severe pitting in all stainless steels.
- Halogen acids attack all forms of stainless steels even at low temperatures and in dilute solutions.
  - A few organic acids and organic halides, under certain conditions can hydrolyze to form inorganic halogen acids.

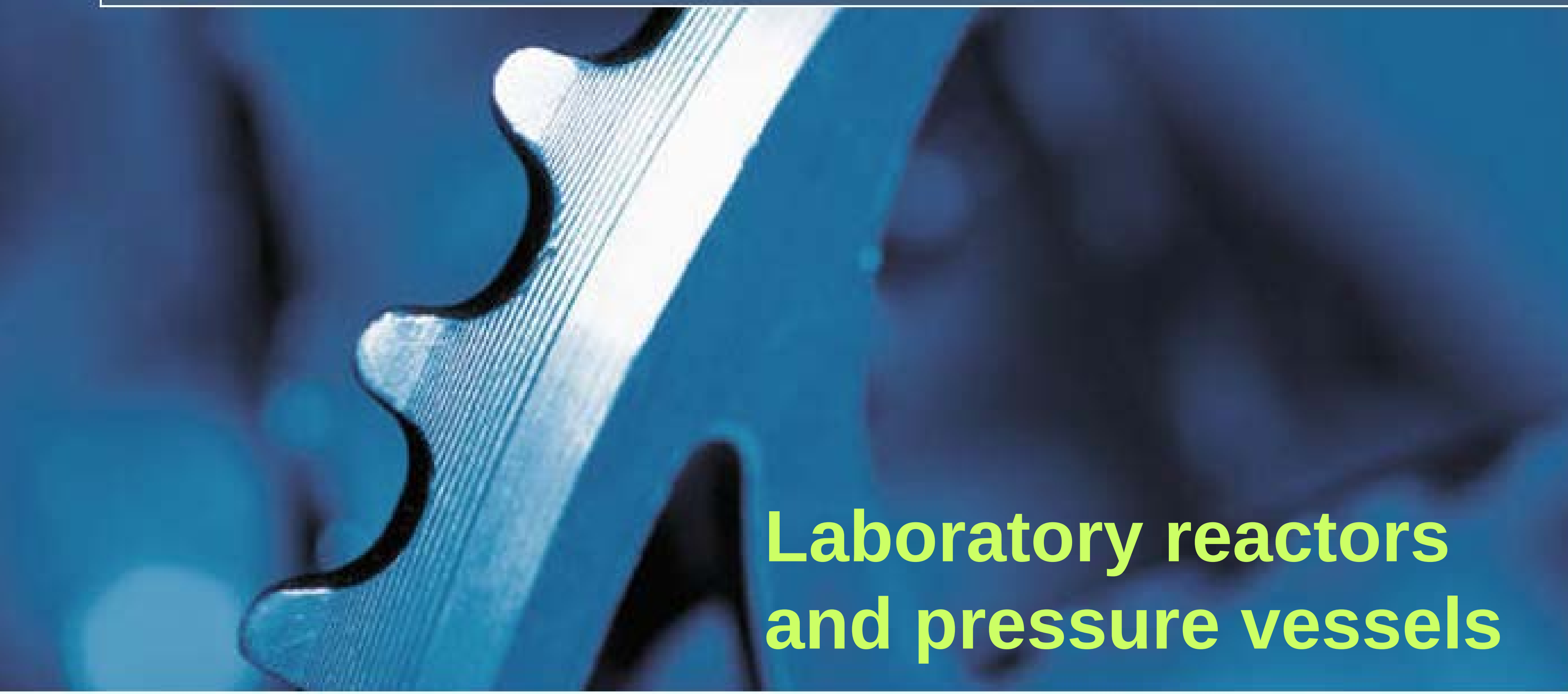
Take home message

- Know the chemistry involved in your process!
- The room in which a pressure reactor is to be operated must be well ventilated!
- A vessel must never be filled to more than three-fourths of its available space, calculate MAWL!
- All materials lose strength at elevated temperatures → know the pressure limits to your own process!

Questions



Safety Procedures



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