ORGANIC
PEROXIDE
PRODUCERS
SAFETY
DIVISION

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Mr. Ed Mazzullo
Director, Office of Hazardous Material Standards
U. S. Department of Transportation
RSPA (DHM-10)
400 7th Street S.W.
Washington, DC 20590-001

Dear Mr. Mazzullo:

The Organic Peroxide Producers Safety Division (OPPSD) of the Society of the Plastics Industry, Inc. (SPI) has developed a proposed Methodology for Vent Sizing. The OPPSD is forwarding the attached proposed Methodology to the Department of Transportation/RSPA for consideration as a new regulation for determining the emergency vent capacity required to be fitted to a specific IBC or portable tank for a particular organic peroxide, Type F or self-reactive substance Type F, or formulation thereof to insure safe depressurization without fragmentation during an emergency situation. The OPPSD's objective is to achieve harmonization with respect to the existing U.N. Method, Appendix 5. In that regard, the OPPSD is continuing to work with the International Group of Experts on the Explosion Risks of Unstable Substances (IGUS), Dr. P. Schuurman.

The OPPSD respectfully requests your consideration of the attached proposed methodology for a new regulation.

Sincerely,

Lynne R. Harris
Executive Director, Organic Peroxide Producers Safety Division

Attachements

Cc: C. Ke, DOT
R. Tarr, DOT

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EXAMPLE OF A TEST METHOD FOR VENT SIZING

1. Introduction

This example of a method for vent sizing is used to determine the required emergency vent capacity to be fitted to a specific IBC or portable tank for a particular organic peroxide, Type F or self-reactive substance Type F, or formulation thereof. The method is based on experimental data which indicates that, for organic peroxides or self-reactive substance formulations, the ratio of the minimum emergency vent area to the capacity of the IBC or tank is constant and can be determined using a reduced scale tank with a 10 liter capacity. In the tests, the reduced scale tank is heated at rates equivalent to that given by complete fire engulfment or, in the case of insulated IBCs or portable tanks, the heat transfer through the insulation with the assumption that 1% of the insulation is missing (see 4.2.1.13.8 and 4.2.1.13.9 of "Recommendations on the Transport of Dangerous Goods, Model Regulations", United Nations, Eleventh Revised Edition). Other methods may be used provided that they adequately size the emergency relief device(s) on an IBC or portable tank to vent all the material evolved during self-accelerating decomposition or a period of not less than one hour of complete fire-engulfment.

Warning: The method does not take into account the possibility of initiation of deflagration. If this is a possibility, particularly if initiation in the vapor phase can propagate to the liquid phase, then tests should be performed which take this into account.

2. Apparatus and materials

The reduced scale tank consists of a stainless steel test vessel with a gross volume of 10 liter. The top of the vessel optionally is provided with either a 1 mm opening which simulates the pressure relief valve (PRV) of the IBC or portable tank or a real PRV of a diameter which is scaled using the vent area to vessel volume ratio. A second opening simulates the emergency vent opening and is closed by a bursting disk. The diameter of this vent opening can be varied by using orifice plates with different apertures. The bursting pressure of the disk fixed to the 10 liter vessel should be equal to the maximum rupture pressure of the bursting disks to be fitted to the IBC or portable tank. This pressure should be lower than the test pressure of the IBC or portable tank involved. Usually, the bursting pressure is set at a level that can cope with the pressures encountered during normal transport conditions such as hydrostatic pressure from the liquid due to turn over of the IBC or portable tank, slopping of the contents, etc. The 10 liter vessel should be provided with a bursting disk with a set pressure in the range of the disk(s) fitted on the tank or IBC as to be used in transport. For safety, it is recommended to provide the test vessel with an extra bursting disk (bursting pressure approximately 80% of the design pressure of the 10 liter test vessel) with a large opening for additional emergency venting of the test vessel in the event that the chosen orifice diameter is too small.

The outer surface of the test vessel, below the liquid level, is provided with an electrical heating coil or cartridge heaters connected to a power supply. The 10 liter test vessel contents should be heated at a constant rate independent of the heat being generated by the organic peroxide. The resistance of the heating coil or cartridge heaters should be such that, with the power available, the calculated heating rate (see section 3) can be achieved. The whole vessel is insulated with rock wool, cellular glass or ceramic fiber.

The temperature inside the vessel is measured by means of three thermocouples, two located in the liquid phase (near the top and bottom) and one in the gas phase. Two thermocouples are used in the liquid phase in order to check the homogeneity of the heating. The pressure is recorded by a pressure transducer(s) capable of recording slow and fast (at least 1000 point/sec.) changes in pressure. Examples of test vessels are illustrated in Figure 1. Additional information may be obtained if the vessel is mounted in a tray designed to collect any solids or liquids ejected and/or on a scale to measure weight loss vs. time.

The tests should be performed at a test site with suitable safety distances. Alternatively, the test can be performed in a bunker provided with sufficient ventilation and vent openings to prevent pressure build-up in it. Explosion-proof electrical equipment should be used in such a bunker to minimize the risk of ignition. *However, the tests should be performed on the assumption that the decomposition products will ignite.*

3. Calculation of the heating rate to be used in the test

If an IBC or portable tank is non-insulated, the heat load to the shell as given in 4.2.1.13.8 of the Model Regulations is required. For an insulated IBC or tank, the Model Regulations require that the heat load to the shell he equivalent to the heat transfer through the insulation plus the heat load to the shell on the assumption that 1 % of the insulation is missing.

The following information on the IBC or portable tank and organic peroxide or self-reactive substance is needed for the heating rate calculation:

F_r	=	fraction of tank directly heated (1 if non-insulated, 0.01 if insulated)	[-]
M	=	total mass of organic peroxide and diluent	[kg]
K	=	heat conductivity of the insulation layer	$[W.m^{-1}.K^{-1}]$
L	=	thickness of insulation layer	[m]
U	=	K/L= heat transfer coefficient	$[W.m^{-2}.K^{-1}]$
A	=	wetted area of IBC or portable tank	$[m^2]$
C_p	=	specific heat of the organic peroxide or self-reactive substance formulation	$[J.kg^{-1}.K^{-1}]$
T_{po}	=	temperature of organic peroxide or self-reactive substance formulation at relieving conditions	[K]

Heat input, q_i (W), via indirectly exposed surface (insulated part) is calculated by equations (1) and (2):

$$q_i = 70961 * F * (1 - F_r) * A^{0.82} \qquad (1)$$

where:

F = insulation factor;

F = 1 for non-insulated shells, or

$$F = 2 * \frac{U * (923 - T_{po})}{47032} \quad \text{for insulated shells.} \qquad (2)$$

In the calculation of F a multiplication factor of 2 is introduced to take into account a 50% loss in insulation efficiency in an incident.

Heat input, q_d (W), via the directly exposed surface (non-insulated part) is calculated by equation (3):

$$q_d = 70961 * F * F_r * A^{0.82} \qquad (3)$$

where:

F = insulation factor = 1 (non-insulated)

The overall heating rate, dT/dt (K/min), due to fire engulfment is calculated by equation (4):

$$dT/dt = \frac{(q_i + q_d)}{M_t * C_p} * 60 \qquad (4)$$

Example 1: insulated portable tank

For a typical 20 m^3 insulated portable tank:

F_r	=	fraction of tank directly heated	=	0.01
M_t	=	total mass of organic peroxide and diluent	=	16268 kg
K	=	heat conductivity of the insulation layer	=	0.031 $W.m^{-1}.K^{-1}$
L	=	thickness of the insulation layer	=	0.075 m
U	=	heat transfer coefficient	=	0.4 $W.m^{-2}.K^{-1}$
A	=	wetted area of portable tank	=	40 m^2
C_p	=	specific heat of the organic peroxide form.	=	2000 $J.kg^{-1}.K^{-1}$
T_m	=	temperature of peroxide at relieving conditions	=	100 °C

and

$$q_i = 70961 * 2 * \frac{0.4 \times (923-373)}{47032} * (1-0.01) * 40^{0.82} = 13533 \text{ W}$$

$$q_d = 70961 * 1 * 0.01 * 40^{0.82} = 14611 \text{ W}$$

$$dT/dt = \frac{(13533 + 14611)}{16268 * 2000} * 60 = 0.052 \text{ K.min}^{-1}$$

Example 2: non-insulated IBC

For a typical 1.2 m^3 non-insulated stainless steel IBC (only direct heat input, q_d):

F_r	=	fraction of tank directly heated	=	1
M_t	=	total mass of organic peroxide and diluent	=	1012 kg
A	=	wetted area of IBC	=	5.04 m^2
C_p	=	specific heat of the organic peroxide form.	=	2190 J $.kg^{-1}. K^{-1}$

and

$$q_d = 70961 * 1 * 1 * 5.04^{0.82} = 266 \text{ kW}$$

$$dT/dt = \frac{(0 + 266000)}{1012 * 2190} * 60 = 7.2 \text{ K.min}^{-1}$$

4. Procedure

Fill the test vessel shell with the amount of organic peroxide or self-reactive substance required to give the same degree of fill (by volume of the shell) as to be used in the portable tank (maximum degree of fill 90 %, by volume) and then install the required orifice plate* and bursting disk. For example, it is common practice to fit four 250 mm diameter bursting disks to a 20 ton portable tank. This corresponds to a test vessel orifice diameter of about 11 mm.

** It is recommended that either small-scale vent experiments (100 - 200 ml scale) or experiments using a very strong vessel (>100 bar) be performed prior to the performance of the 10 l vent test in order to obtain information on pressure effects from the test substance and on the required orifice diameter to be used in the first 10 l scale vent test. For safety, start with the largest orifice.*

The vessel is heated at the desired rate by applying power to the heating coil or cartridge heater. A higher than calculated heating rate may be applied initially until a temperature 5 °C above the self-accelerating decomposition temperature (for a 50 kg package) of the organic peroxide or self-reactive substance is reached. The calculated heating rate should be applied once this temperature is reached. The temperature and pressure in the test vessel are recorded during the entire experiment. After rupture of the bursting disk, the temperature should be maintained for approximately 30 minutes more to be sure that all dangerous effects are measured. *After the test, the vessel should not be approached until the contents have cooled.*

The diameter of the orifice should be varied (if necessary) until a suitable opening is determined at which the maximum recorded pressure is:

- for portable tanks tested according to 4.2.1.13.4, not more than 85% of the maximum pressure before failure as determined for the portable tank based on the hydrostatic limit or as determined by Finite Element Analysis, but not to exceed 25 barg.

- for IBCs tested according 6.5.4.8.4, not more than 85% of the of the maximum pressure before failure as determined for the IBC based on the hydrostatic limit or as determined by Finite Element Analysis, but not to exceed 25 barg.

The minimum test pressure of the IBC should be at least as specified in 6.5.4.8.4 of the Model Regulations (e.g. 2.0 bar for metal IBCs) or higher as tested by hydrostatic pressure testing or by Finite Element Analysis (FEA). The minimal test pressure of the portable tank should be 4.0 barg (see 4.2.1.13.4 of the Model Regulations). Since these portable tanks and IBC's are used to transport organic peroxides (and other self-reactive substances) and are not process vessels, permanent deformation is permitted during hydrostatic peak pressure testing so long as there is no fragmentation. A shipping vessel , should there be an incident resulting in permanent deformation, will be discarded without exception (no further use).

The step size used should be related to the options available in practice for the IBC or portable tank, i.e. larger vent sizes or more vents. The test should be performed in duplicate with the minimum total vent area having sufficient capacity. An alternative approach is to perform a series of six runs (6) at varying orifice sizes so as to generate a continuous plot of peak pressure vs. the venting area to working volume ratio. This permits improved working latitudes with respect to the approval of additional peroxides and/or the design of additional containers (See Section 5).

5. Test criteria and method of assessing the results

The minimum or suitable (if it is acceptable to use a vent size larger than the minimum vent size) IBC or portable tank vent area, A_{IBC} or $A_{portable\ tank}$ (m^2), can be calculated using the minimum orifice vent area, $A_{test\ vessel}$ (m^2), determined in the 10 l test at which the maximum pressure is not more than specified in section 4 of this method, and the volumes of the test vessel ($V_{test\ vessel}$, m^3) and IBC or portable tank (V_{IBC} or $V_{portable\ tank}$, m^3). The minimum total IBC or portable tank vent area is given by:

For IBCs: $A_{IBC} = V_{IBC} * (A_{test\ vessel} / V_{test\ vessel})$

For portable tanks: $A_{portable\ tank} = V_{portable\ tank} * (A_{test\ vessel} / V_{test\ vessel})$

Example:

For a typical organic peroxide in a 20 m^3 insulated portable tank:

$A_{test\ vessel}$	=	Minimum suitable orifice area found in test	=	$9.5 \cdot 10^{-5}$ m^2
$V_{portable\ tank}$	=	Volume of portable tank	=	20 m^3
$V_{test\ vessel}$	=	Volume of test vessel	=	0.01 m^3
$A_{portable\ tank}$	=	$20 * (9.5 \cdot 10^{-5} / 0.01)$	=	0.19 m^2

Utilizing the peak pressure curve for a given standard peroxide generated in a 10-liter study in Section 4, another peroxide exhibiting a lower peak pressure for the same venting ratio may use the same full scale container without further testing of the container or the peroxide in that container (full scale). Likewise, another peroxide which exhibits a higher pressure for a given venting ratio in the 10 liter 6-point study may be used in the full scale container so long as the container's venting ratio is increased so as to produce a lower peak pressure than the standard organic peroxide previously tested.

Figure 1: 10 LITER VESSELS FOR VENTING TESTS

Note: It is recognized in developing this method for vent sizing that dicumyl peroxide behaves differently than most other organic peroxides in vent testing. Therefore dicumyl peroxide can continue to be shipped in the portable tanks and IBCs currently approved by the DOT based on extensive testing and proven performance.