PRESSURE RELIEF OF LIQUIDS CONTAINING SUSPENDED SOLIDS

Derick McIntosh, Simon Waldram (Hazard Evaluation Laboratory Ltd, 50 Moxon Street, Barnet, Herts. EN5 5TS) and Janet Etchells (Health and Safety Executive, Magdalen House, Stanley Precinct, Bootle, Merseyside L20 3QZ)

Abstract
The multi-phase venting of vapour, liquid and solids has been studied experimentally on the 1 and 10 litre scales. In non-reacting systems, the depressurisation profiles of superheated water or water-glycerol mixtures were studied on their own and with added glass particles. The particles were both solid and hollow, with specific gravities both greater and less than 1. Similar depressurisation experiments were made during the runaway reaction of acetic anhydride and water, both with and without solids. Relief set pressures were between 3 and 5 bara. Solids concentrations were up to 16% v/v. Nozzle to median solid diameter ratios were between 6 and 500. Experimental design techniques were used to study the effects of many factors efficiently. Depressurisation profiles with and without solids present were compared: in general the solids had little statistically significant effect. There was limited evidence that the less dense solids could increase depressurisation rates slightly. Tests with runaway reactions have highlighted some difficulties in comparing systems with and without solids present. In all cases, the vented fluids were less concentrated in solids than those in the reactor. This may have important implications for design and sizing of pressure relief vents.

INTRODUCTION

The field of two-phase liquid/vapour venting has been the subject of considerable research in recent years[1]. The situation will often arise, however, when there are solids present in the discharge stream. The solid phase might be a heterogeneous catalyst, (e.g. a platinum group metal on a porous carbon support particle, for a hydrogenation reaction,) a partially dissolved reactant or a solid product that is crystallizing as the reaction proceeds. There is little guidance about how to allow for the presence of such solids when sizing a pressure relief system, although some initial work has been carried out by Beyer and Steinbach[2]. Some preliminary information, based on recommendations of The Design Institute for Emergency Relief Systems (DIERS), is given in reference 3. However this has not been validated experimentally and the authors point out that these methods may not apply if the solids are not carried over at the same velocity as the liquid. There may also be problems if the solids affect the flow from the reactor or cause fouling in the relief system. HSE, in collaboration with a consortium of companies has, therefore, sponsored a project to investigate the problems further on the laboratory scale, and to identify the main issues involved. This paper describes the main findings, which will be published in an HSE Research Report[4].

EXPERIMENTAL PROGRAM
An experimental programme was devised to examine the effects of the addition of suspended solids on a two-phase vented system. To examine the effects of the solids, the venting profiles from tests with the addition of solids were compared to those without. Initially non-reacting systems were examined, using superheated water or water/glycerol liquid mixtures, and then reacting systems were examined, using acetic anhydride and water. Experimentation was carried out on both the 1 litre and 10 litre scales.

There are a vast number of potential variables that could be studied in this project, e.g. solid concentration, solid diameter, solid density, nozzle diameter, fill level, stir rate, relief pressure etc. In these circumstances it was important to plan experiments, execute the experimental work and process and analyse the results in a structured and efficient manner. To achieve this, factorial experimental design techniques have been used: see reference 5 for an introduction to this topic. When the effects of many factors are to be examined then very large experimental programmes are required. In these cases fractional factorial designs (e.g. half or quarter) can be used to reduce the required number of experiments (e.g. by factors of 2 or 4). However, a compromise must be made: such designs lose the ability to discriminate clearly between the effects on the response of combinations of factors.

As an example, a full level factorial design involving 6 factors would require 2^6, or 64, experiments. Statistical analysis of the results enables the effect on the response variable to be estimated not only for every factor, but also for all possible combinations of factors. A half factorial design would only involve 32 experiments, and hence half of the experimental effort, without significant loss of response data. Additionally, replicate centrepoint experiments are commonly included to give an indication of the experimental error, and this is used in the statistical analysis.

Planning such experiments, and interpreting the results from them, can be tedious and complex but the use of standard software packages avoids many of these difficulties. In this project the "Design Expert" software was used, see http://www.statease.com.

APPARATUS
A schematic diagram of the apparatus is shown in figure 1. The test rig consisted of a reactor vessel, a vent line and a catch tank. The 10 litre reactor was a purpose built, baffled, stainless steel vessel, rated to 20 bar. Thermocouples, a pressure transducer, a bleed valve, a magnetic drive coupling, an overpressure relief valve and the vent line were incorporated into the reactor top plate. Four electric rod heaters were used and entered through the bottom plate. Two Rushton type impellers and a baffle system were used for the agitation of the mixtures.

The reactor was connected to the catch tank via a 12.7 mm (½") vent line that incorporated a pneumatically operated ball valve. When open this gave full bore unobstructed flow. The vent line was connected to the reactor via a fitting, designed to hold a variety of different sized nozzles. The automatic ball valve was sited just downstream of the nozzle. The catch tank was placed on a balance to allow continuous measurement of the vented mass, as a function of time.

For the tests with reacting systems, it was important to minimise discharge of vapours. The vented catch tank was replaced with a sealed vessel and downstream sparged quench

tank. To aid condensation of the vapours, approximately 8 kg of water were placed in the catch tank prior to the test. This worked well and there was very little mass loss from the system overall. A solenoid bleed valve, fitted between the catch tank and the quench tank, was opened after venting to prevent the cooling vapours in the catch tank creating a vacuum and sucking back liquid from the quench tank.

Control and data logging were achieved using HEL software. This allows data acquisition at a maximum logging rate of 10 points per second. Other than during the venting stage of the experiment, the data was only logged every 20 seconds.
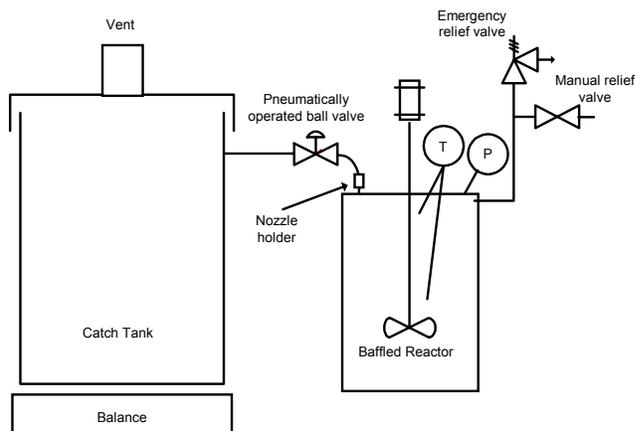
Figure 1.   Schematic diagram of the 1 and 10 litre test apparatus

EXPERIMENTAL PROCEDURES
The same general procedure was used for all the tests using non-reacting fluids:

- Charge the materials and seal the reactor.
- Pull a vacuum in the reactor vessel[1]
- Allow the reactor contents to reach the relief pressure/temperature.
- Turn off the heating and wait 6 seconds.
- Open the relief valve and vent the reactor until the temperature dropped to 101°C or the pressure reached 1.05 bara.
- Close the relief valve and allow the reactor to cool.

The 6 second delay was incorporated to allow the data logging rate to be changed to the maximum rate without any delays caused by the software controls. Once initiated, the experimental procedure was fully automated and computer controlled to allow reproducible experimental sequences.

---

[1]Note that in the case of the 1 litre tests, a vacuum was not drawn, rather the vessel was heated with the vent valve open until the liquid reached its boiling point. In this manner the water was degassed and the reactor was purged of air.

Non-reacting test series were carried out by depressurising water and water/glycerol mixtures, both with and without the addition of solid particles. Glycerol was added to vary the liquid density (liquid to solid density ratio) and the liquid viscosity. The solids used were spherical glass particles in size ranges from 4–45 μm to 250–425 μm. Additionally, low density hollow glass particles were used. These have a density of 0.6 kg m^{-3} and are in the size range 0–65 μm. This range is not too dissimilar to the 4–45 μm solid particles, and comparisons have been made between the results from tests using these two sets of particles.

In the case of reacting systems, using acetic anhydride and water, the acetic anhydride was added to the reactor and heated to 50 °C under sealed conditions. The water was then added, the reactor was re-sealed, the mixture heated to 80°C and the heaters were turned off. The exothermic reaction was then allowed to proceed naturally until the venting condition of 3 bara was reached. At this point the reactor vent valve was opened and the reactor vented to the catch tank. The reactor was re-sealed when the pressure dropped to 1.05 bara.

TESTING ON 1 LITRE SCALE

Several series of tests were carried out on the 1 litre scale. Water and water/glass mixtures were charged to the reactor and heated to a temperature of 152°C (5.06 bara) before venting. The data obtained showed that there was very little difference in the depressurisation profiles between 700 ml of water and 700 ml of water plus up to 16% by volume of solids. Indeed, the profiles from tests with the same volumetric fill levels were very similar. There was limited evidence that the presence of the hollow particles resulted in a very slightly quicker depressurisation.

Two full factorial test series, with 4 factors, using a 2 mm and a 5 mm nozzle, were performed. The data analysis on the times taken for the pressure to drop in small increments throughout the depressurisation showed that there was no statistically significant effect due to the addition of solids.

A potential problem that was identified on the 1 litre scale was that the repeatability of the test was poor, particularly when comparing many identical tests carried out over an extended time period. Tests on the 10 litre scale were more reproducible. For this reason, experimental work concentrated on the 10 litre scale.

NON-REACTING EXPERIMENTS ON THE 10 LITRE SCALE

A series of tests were carried out by depressurising water and water/glycerol mixtures, both with and without the addition of solid particles. A 6 factor, half factorial experimental design was used. The following factors were examined: solid concentration, solid diameter, nozzle diameter, fill level, stirring speed and glycerol concentration. The relief pressure was fixed at 5 bara.

In order to examine the effects of the particle density, a further series of tests were planned to complement those already performed. The low density glass has a similar diameter range to the 4–45 μm solid glass. Many of the tests already run in the previous series could be incorporated into a new experimental design, so that only 4 new tests had to be performed, all of these were with the low density glass particles. A 5 factor, half factorial

design was analysed, with the factors being: solid concentration, solid density, fill level, stir speed, and nozzle diameter. The effect of glycerol was not studied in this series.

Some factors, e.g. fill level and nozzle diameter, are already known to affect the depressurisation. As this project is aimed at investigating the effects of solids, the prime interest was to identify those experiments in which solid diameter, or solid concentration, combined with the other factors affected any aspect of the depressurisation profile.

The response variables chosen for the analysis of this data are the times taken for incremental depressurisation, in steps of 0.1 bar down to a pressure of 4 bara, then in 0.2 bar steps to 3 bar, and thereafter in 0.5 bar steps. The results showed that the nozzle diameter has an effect on the times throughout the depressurisation, and the fill level has an effect down to 2.5 bara. This would be expected. Additionally, almost throughout there was the effect of an interaction between the fill level and the nozzle diameter. The stirrer speed was identified as having an effect in the early stages, down to 4.7 bara.

The first design showed that the solid concentration had an effect on the initial depressurisation, with the incremental depressurisation times between 4.5 bara and 3.0 bara reducing with increasing solids concentration (ie increasing the solids, reduced the time for depressurisation). This may be simply because at a fixed reactor fill level the liquid contents reduce at increased solid concentration, and the amount of vapour produced as the reactor contents cool to atmospheric conditions will therefore become smaller at high solids concentration. This may affect the transition point between single and multi-phase flow. Otherwise the presence of the solids had no statistically significant effect on the depressurisation.

It is interesting to note that there was no effect on any of the response variables due to the amount of glycerol added. This suggests that the viscosity and density difference between the water and water/glycerol mixtures had no effect on depressurisation for the range of variables studied.

The analysis of the second experimental design showed similar findings to the previous design in that neither the solid concentration nor the solid density had a statistically significant effect on the response variables. Fill level and nozzle diameter had the main effect on the response factors studied.

REACTING SYSTEM

Several properties were desirable when choosing the reacting system. The reaction needed to be proceeding at a relatively high rate during venting so as to promote multiphase flow and to test venting under demanding conditions. Additionally, the 10 litre vessel has a high thermal inertia (ϕ factor) and therefore a high fraction of the heat of reaction would be absorbed by the vessel itself during slow periods of an exotherm.

The reaction of water and acetic anhydride to produce acetic acid was eventually chosen for several reasons – in particular, the reaction mechanism is relatively simple, and the flammability hazards are minimised by using an aqueous reaction. The stoichiometry of this reaction is 1:1, but examination of vapour pressure data indicated that a slight excess of water would generate a higher pressure, and hence the reaction was run with a 3:2 water:acetic anhydride mole ratio. Perhaps more importantly, this vigorous exothermic reaction has been involved in a several publicly reported incidents[6,7,8]. Leigh and

Krzeminski[8] quote an incident where water had entered a storage tank containing acetic anhydride and 15% acetic acid at ambient temperature. The resultant overpressure and vessel rupture killed one person and injured 20 more.

This series of tests was initially planned as a factorial design, with factors of fill level, glass diameter and glass concentration. The high, low and centrepoint values used in the factorial design are given in table 1. Note that reactant volume and glass concentration are independent factors. Combinations of these at high, centrepoint and low values will determine the actual reactor fill level.

A 10.5 mm diameter constriction was installed on the reactor. This was chosen following small scale PHI-TEC tests, and was predicted to give significant overpressure during the venting under the conditions of the reaction. A relief set pressure of 3 bara was used.

In order to give the same thermal inertia, as tests run with glass particles, the tests run at 0% suspended solids concentration were, in fact, run with 15% v/v of 3 mm diameter glass beads present. The diameter of these beads was so large as to ensure that they always settled out in the bottom of the reactor and were not entrained in the vented flow. This was thought to be a good method of keeping the heat capacity of the system constant and at the same time the glass ballast would not affect the two-phase venting rate from the reactor.

Additionally, two tests (one at each fill level) were run using the hollow glass beads (density 0.6 kg m^{-3}) for comparison with the tests with the 4–45 μm solid particles (density 2.5 kg m^{-3}).

RESULTS

Table 2 gives the conditions for each test. The test numbers in table 2 have been sorted in terms of reactant volume and have been given a letter designator for clarity.

The main conditions of the tests are given in Table 2. Figures 2 and 3 give the respective pressure and temperature profiles during venting for the tests using 5000 ml of reactants +15% v/v solids. It can be seen that the tests A and B (both with 3mm glass beads), which had two-phase flow gave very much higher overpressures than the tests with three-phase flow: tests C and D resulted in virtually no overpressure. This was not expected, as in the non-reacting systems, the presence of solids had very little effect on blowdown profiles.

Figure 4 shows the self-heat rate profiles for the tests starting from the temperature at which the electrical heating was turned off (80°C). It is clear that tests A and B (with 3 mm glass beads as thermal ballast) reached much higher heat-rates before venting commenced at 3 bara. As the test recipes are exactly the same, there must be an effect due to the diameter of the solids. Transient heat transfer calculations confirm that the large glass beads absorb less of the heat from the reaction, due to their smaller external surface area and low thermal diffusivity[9]. This will become increasingly apparent at high runaway reaction rates. The fact that glass is a relatively poor conductor of heat will also have an effect, with smaller glass particles heating up more quickly and more uniformly than larger particles.

A similar effect is likely when φ factors are considered: at very high self-heat rates the observed φ factor may be much closer to unity than expected from simple calculation,

simply due to the fact that the heat transfer to the reactor vessel cannot occur at a sufficiently high rate for the reactants and vessel to be at an essentially identical temperature at any time. This means that, during fast thermal runaways, the phi factor may approach unity irrespective of the thermal mass of solids that may be present or of the reactor vessel itself. As a consequence, large diameter inert solids result in a much more vigorous reaction compared with the same mass of smaller glass particles.

Table 3 gives the mass balance information and maximum temperature and pressure data for the tests with 5000 ml of liquid. Again the effects of the glass diameter can be seen. The finest particles, which also result in the lowest heat rate due to reaction, result in a greater mass retained in the reactor. The lower heat rate and hence lower rate of vapour generation should give a lower superficial velocity and would be expected to result in less carryover.

Similar observations can be seen in figures 5 to 7 for the tests with the higher fill level. The data is also summarised in table 4. It is interesting to note from figures 3 and 6 that there is slight variation between the temperatures at the start of venting even though the pressure was identical each time. This could be attributable to experimental errors. However, the lower fill level does appear to result in slightly higher relief temperatures. One possible explanation is that the density of the acetic anhydride and acetic acid mixture falls with increasing temperature and the compression of the head space gas may be a significant factor. At higher fill levels the compression effect would be greater and venting would occur slightly earlier in the runaway. The data from a test with 6500 ml of reactants but without glass solids is also shown, and the significant effect of adding glass can be seen on the reaction rate.

The data for the repeat centrepoint tests are shown in figures 8 to 10 and in table 5. These tests show excellent repeatability in terms of blowdown profiles and self heat rates: the lines on the graphs virtually overlap each other.

NOTE ON MIXING
Table 6 shows the mass balance data for the glass particles in each test. In the case of an ideally mixed system, with no slip between the solid and liquid phases, it could be expected that the glass concentration in the reactor and the vented fluid would be the same. Table 6 gives the fraction of the initial liquid and glass masses that were drained from the reactor. In every case, there is an increased concentration of solids in the reactor following venting. It is likely that there was some accumulation of solids towards the bottom of the reactor with the solid glass, particularly with the larger solids. The nature of the reactor vessel makes it impossible to make a visual inspection and qualitative judgement of the stirring efficiency. However, based on visual observation on the 1 litre scale, at similar tip speeds, it was believed to be good at the centrepoint and high stirring rates.

It is very interesting to note the data from the tests using the hollow solids. From previous observations on the one litre scale, these particles are very easily mixed with the liquid, and float when not agitated. Therefore, with poor mixing, any increase in solid concentration would be towards the top of the reactor. It would be expected that as this will vent first, and that a greater fraction of the glass would be vented than that in the reactor as a whole. If the mixing

was always perfect, then an equal fraction of solid and liquid would remain in the reactor and the catch tank. The data in table 6 shows that this is not the case, and a greater fraction of the liquid was vented. It is clear therefore that the liquid is vented preferentially even though the solids are lighter than the liquid. If the solids are taking part in the reaction, then this will need to be considered in the design of the relief system, particularly if it means that the reactants could be concentrated, either in the reactor, or in the downstream containment or disposal system.

CONCLUSIONS

From the testing and analysis described above the following can be concluded.

A. Non-reacting tests
  a. Pressure and temperature profiles versus time during venting were in general not influenced to a statistically significant extent by the presence of solids.
  b. There is limited evidence that, under some circumstances, the presence of solids can increase depressurisation rates, particularly at intermediate times. It is likely that the presence of solids may promote even more vapour bubble nucleation and promote bubbly or homogeneous rather than churn turbulent flow.
  c. During venting, liquid is discharged preferentially to the solids. This was observed for both naturally floating and sedimenting particles, i.e. both less and more dense than the fluid in which they are suspended.

B. Reacting systems
  a. Adding inert particles to a reacting system increases the phi factor and hence reduces the reaction runaway rates. This effect is highly non-linear with particle mass.
  b. Depressurisation of reacting systems containing inert solids was highly reproducible on the 10 litre scale.
  c. The temperature of inert particles suspended in a liquid whose temperature is changing rapidly may lag behind the fluid temperature. This means that the effective phi factor can change during the course of a fast runaway. For this reason, large inert particles appeared to accelerate the runaway reaction relative to the same mass of smaller particles and this can lead to larger overpressures during venting.
  d. Heat transfer limitations to the body of a large or massive reactor vessel during a fast runaway may mean that the average temperature in the reactor body is much lower than in the reacting fluid. In an analogous way to item c this can lead to a shifting value of the phi factor as a reaction proceeds. The reaction may become much faster than that anticipated from small scale studies at an analogous phi factor.
  e. Direct comparison of the results from the reacting system tests with, and without, solids is very difficult because of the change in the phi factor (and hence reaction rate) and the ability of inert solids to accelerate a runaway reaction, see items a, and c.
  f. Production of vapour and preferential flow of the liquid (relative to the solid) in the vent discharge will enhance the solid concentration in the reactor. This will then alter the phi factor and hence the runaway rate. If the solids are participating in the reaction this may also affect the reaction rate per unit volume.

As a general conclusion these preliminary studies show that, for the ranges of variables studied, small diameter inert solids have little influence on the rates of depressurisation achieved. Although it was outside the scope of this project, there are several issues that would need to be carefully considered before calculating the required vent areas in the case of three-phase flow. Any effects of concentration of the solids during venting must be considered in selection of the calorimetric test methods used for relief line sizing. Solid deposition and downstream fouling may also be issues.

REFERENCES

1. Fisher, H.G., Forrest, H.S., Grossel, S.S., Huff, J.E., Muller, A.R., Noronha, J.A., Shaw, D.A. and Tilley, B.J: "Emergency relief system design using DIERS technology, The Design Institute for Emergency Relief Systems (DIERS) project manual," 1992, AIChE, NY. ISBN 0-8169-05668-1.
2. Beyer, R. and Steinbach, J: "Source term characterization for three-phase venting scenarios." Paper presented at the 10th International Symposium on Loss Prevention and Safety Promotion in The Process Industries, Stockholm, June 19th –21st, 2000.
3. Etchells, J. and Wilday, J: "Workbook for chemical reactor relief system sizing," 1998, HSE Books. ISBN 0-7176-1389-5
4. McIntosh, R.D. and Waldram, S.P.: "Reactor pressure relief of fluids containing suspended solids" HSE Contract Research Report, project reference 4187/R05.101.
5. Davies, L., Efficiency in research, development and production: the statistical design and analysis of chemical experiments, 1993, Royal Society of Chemistry, ISBN 0 85186 137 7
6. Barton, J. and Rogers, R.L.: "Chemical reaction hazards," second edition, 1997, I.Chem.E. ISBN 0-85295-341-0
7. I.Chem.E, Industrial accidents database, 2002, available from http//www.icheme.org.uk.
8. Leigh, W.R.D., and Krzeminski, Z.S.: "The uncatalysed reaction of acetic anhydride with water," Chemistry and Industry, pp778-779, April 28, 1962
9. Welty, J.R., Wicks, C.E. and Wilson, R.E.: "Fundamentals of momentum, heat, and mass transfer." John Wiley and Sons, New York 1976. ISBN 0-471-93354-6

Table 1. Variables and values used in factorial design for the reacting system

Variable	High	Centrepoint	Low
Reactant volume (ml)	6500	5750	5000
Solid concentration (% v/v)	15	7.5	0*
Solid diameter (μm)	150–250[2]	70–110	4–45

*15% of 3 mm glass ballast was added in these cases

Table 2. Data from 10 litre blowdown tests with the reacting system

Test Number	Reactant volume (ml)	Glass diameter (μm)
A	5000	3000
B	5000	3000
C	5000	4–45
D	5000	150–250
E	5750	70–110
F	5750	70–110
G	6500	3000
H	6500	4–45
I	6500	150–250
J	6500	250–425[3]
K	6500	0–65 (hollow glass)
L	5000	0–65 (hollow glass)

Note: the tests with 3000 μm glass should be treated as 0 % solids (i.e. 2-phase venting with no solid carryover)

---

[2]This value was chosen as the slightly larger particles (250–425 μm diameter) resulted in no solid carry over.

[3] This test gave no solid carryover and is included for comparison only

Table 3. Data obtained from acetic anhydride/water tests with 5000 ml of reactants

Test	Glass diameter (µm)	Glass carryover to catch tank (g)	Glass carryover to catch tank (ml)	Liquid retained in catch tank (g)	Liquid retained in reactor (g)	Maximum pressure (bara)	Maximum temperature (°C)
A	3000	0	0	2425	2880	5.24	169
B	3000	0	0	2523	2045	5.07	168
C	4–45	44.69	17.9	1048	4050	3.05	145
D	150–250	20.36	8.1	2449	2736	3.08	145
L	0–65 (hollow glass)	69.78	116.3	1707	3508	3.09	144

Table 4. Data obtained from acetic anhydride/water tests with 6500 ml of reactants

Test	Glass diameter (µm)	Glass carryover to catch tank (g)	Glass carryover to catch tank (ml)	Liquid retained in catch tank (g)	Liquid retained in reactor (g)	Maximum pressure (bara)	Maximum temperature (°C)
G	3000	0	0	3479	2693	4.39	162
H	4–45	299.0	119.6	2989	3619	3.10	147
I	150–250	24.58	9.8	4190	2443	3.92	157
J	250–425	0	0	3471	2898	4.67	165
K	0–65 (hollow glass)	219.56	365.9	3811	3048	3.47	151
M	No glass	N/A	N/A	3350	3571	7.68	195

Table 5. Data obtained from centrepoint cases

Test	Glass diameter (μm)	Glass carryover to catch tank (g)	Glass carryover to catch tank (ml)	Liquid retained of catch tank (g)	Liquid retained of reactor (g)	Maximum pressure (bara)	Maximum temperature (°C)
E	70–110	19.92	8.0	3116	2767	4.05	159
F	70–110	19.82	7.9	3063	2939	4.05	159

Table 6. Selected mass balance data from acetic anhydride/water tests

Test	Glass diameter (μm)	Initial glass charge (g)	Glass carryover to catch tank (g)	Glass remaining in reactor (g)	Fraction of initial glass remaining in reactor	Fraction of initial liquid charge remaining in reactor
A	3000	1875	0	1868	1.00	0.54
B	3000	1875	0	1870	1.00	0.38
C	4–45	1875	45	1782	0.95	0.76
D	150–250	1875	20	1806	0.96	0.51
E	70–110	1078.1	20	1042	0.97	0.45
F	70–110	1078.1	20	992	0.92	0.48
G	3000	2437.5	0	2421	0.99	0.39
H	4–45	2437.5	299	2078	0.85	0.52
I	150–250	2437.5	25	2354	0.97	0.35
J	250–425	2437.5	0	2362	0.97	0.43
K	0–65 (hollow glass)	585	220	346	0.59	0.44
L	0–65 (hollow glass)	450	70	383	0.85	0.66
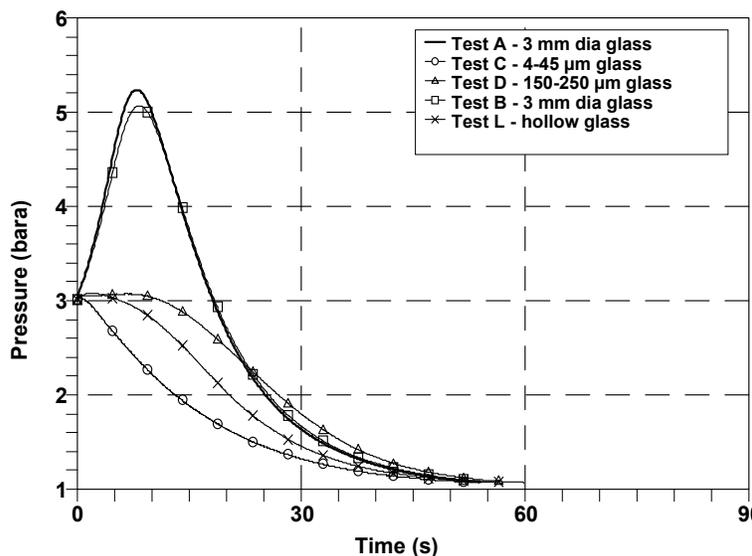
Figure 2.   Pressure profile for venting of reacting system tests at low fill level
*10 litre scale, 5750 ml charge (5000 ml reactants + 15% by volume glass), water and
acetic anhydride (mole ratio 1.5) 9 mm nozzle, 200 rpm stirring*
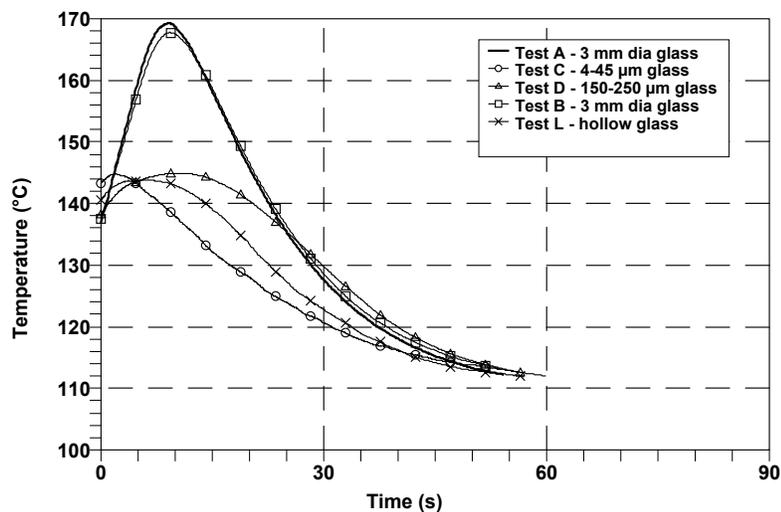
Figure 3.   Temperature profile for venting of reacting system tests at low fill level
*10 litre scale, 5750 ml charge (5000 ml reactants + 15% by volume glass), water and
acetic anhydride (mole ratio 1.5) 9 mm nozzle, 200 rpm stirring*
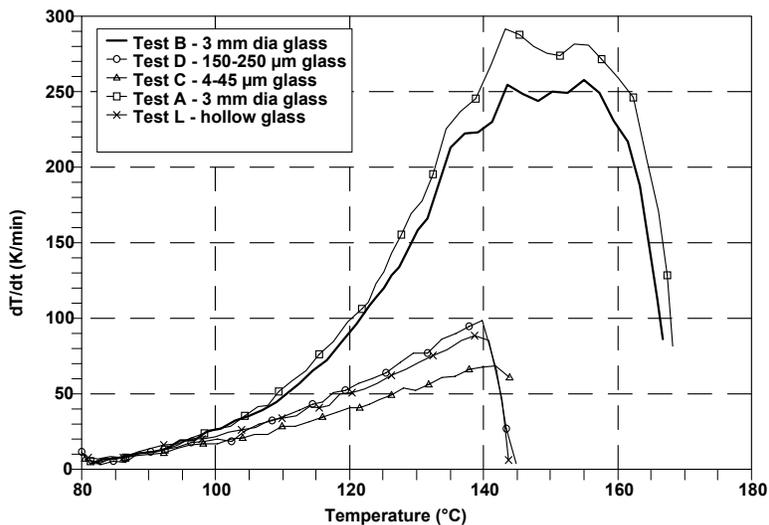
Figure 4. Heat-rate profiles of reacting system tests at low fill level
*10 litre scale, 5750 ml charge (5000 ml reactants + 15% by volume glass), water and
acetic anhydride (mole ratio 1.5) 9 mm nozzle, 200 rpm stirring*
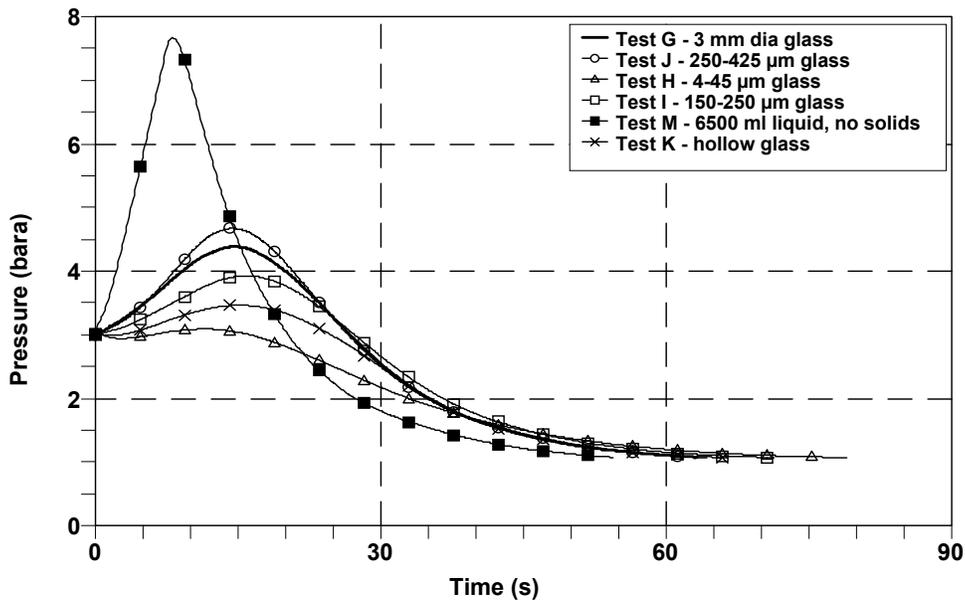
Figure 5. Pressure profile for venting of reacting system tests at high fill level
*10 litre scale, 7475 ml charge (6500 ml reactants + 15% by volume glass), water and
acetic anhydride (mole ratio 1.5) 9 mm nozzle, 200 rpm stirring*
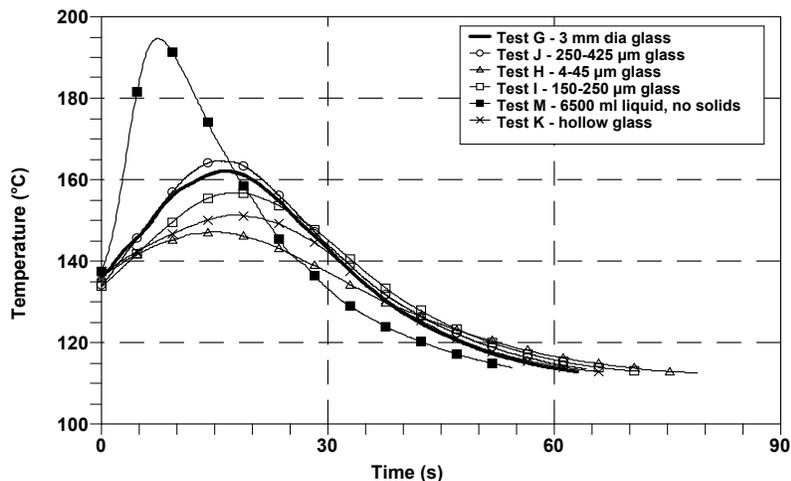
Figure 6. Temperature profile for venting of reacting system tests at high fill level
*10 litre scale, 7475 ml charge (6500 ml reactants + 15% by volume glass), water and
acetic anhydride (mole ratio 1.5) 9 mm nozzle, 200 rpm stirring*
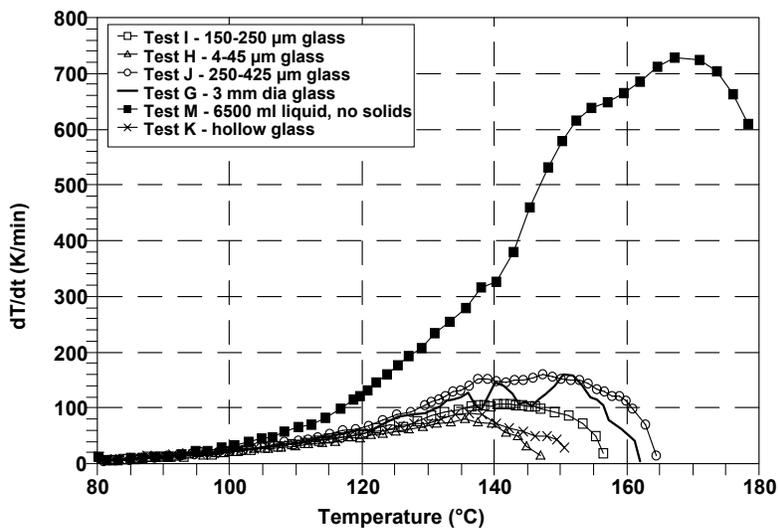
Figure 7. Heat-rate profiles of reacting system tests at high fill level
*10 litre scale, 7475 ml charge (6500 ml reactants + 15% by volume glass), water and
acetic anhydride (mole ratio 1.5) 9 mm nozzle, 200 rpm stirring*

573
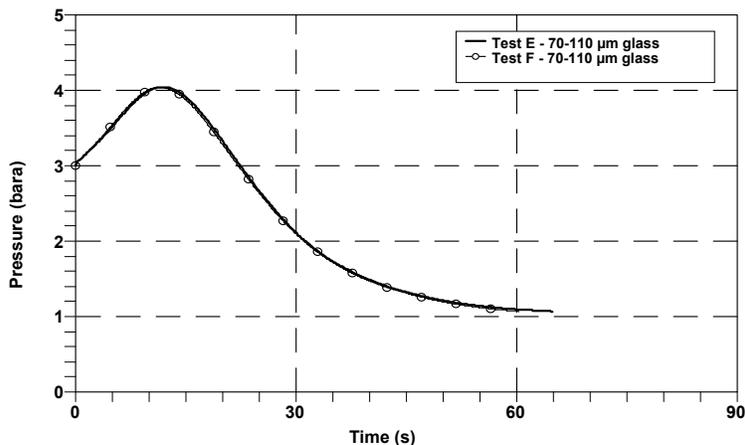
Figure 8.   Pressure profile for venting of reacting system tests at centrepoint
*10 litre scale, 6210 ml charge (5750 ml reactants + 8% by volume glass), water and
acetic anhydride (mole ratio 1.5) 9 mm nozzle, 200 rpm stirring*
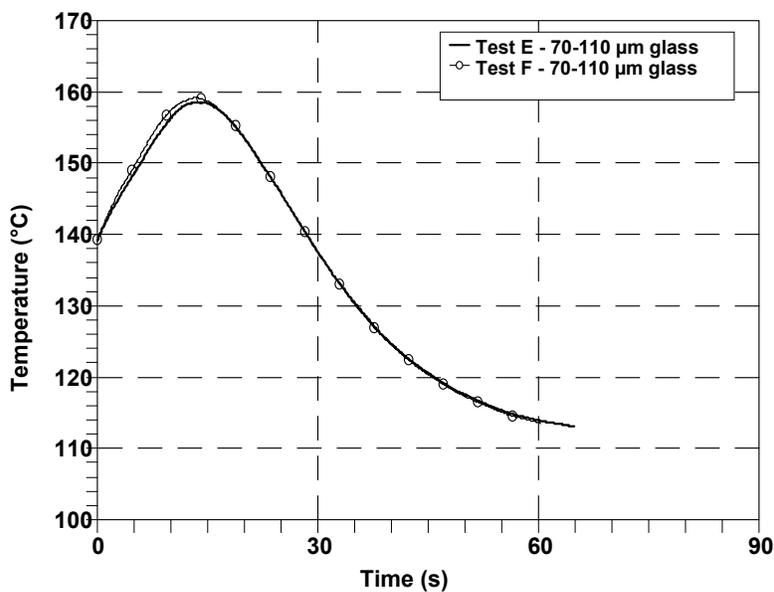
Figure 9.   Temperature profile for venting of reacting system tests at centrepoint
*10 litre scale, 6210 ml charge (5750 ml reactants + 8% by volume glass), water and
acetic anhydride (mole ratio 1.5) 9 mm nozzle, 200 rpm stirring*
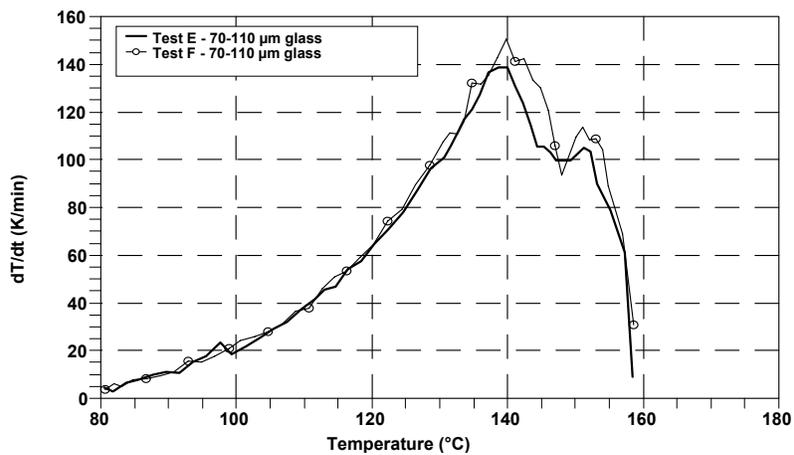
Figure 10. Heat rate profiles for reacting system tests at centrepoint
10 litre scale, 6210 ml charge (5750 ml reactants + 8% by volume glass), water and acetic anhydride (mole ratio 1.5) 9 mm nozzle, 200 rpm stirring