# ALKOXYLATION RUNAWAY REACTION INCIDENT AT BAKER PETROLITE, HARTLEPOOL

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On 12th October 1999, a runaway reaction occurred in a 22 m<sup>3</sup> reactor during an alkoxylation reaction using Propylene Oxide. The subsequent pressure increase caused the bursting disc to rupture and a few tonnes of Propylene Oxide was released to atmosphere. The release occurred despite the presence of a knockout pot designed to capture released material. Although the incident caused no injury and only minimal plant damage, it did result in substantial costs both in terms of investment in corrective measures, and business loss. The incident and its causes are described along with improvement measures implemented to prevent recurrence and provide better protection against overpressure and environmental release. Improvement measures included improvements to process control, a re-design of the pressure relief system of all reactors in the plant, and the installation of a quench tank. The methodologies used to design the improvement measures are described. The paper also describes a later incident which provided a full scale test of the new quench tank.

KEYWORDS: Propylene Oxide, Ethylene Oxide, Runaway Reaction, Quench tank, Pressure Relief, Discharge and Disposal

#### **INTRODUCTION**

Baker Petrolite (part of the Baker Hughes group) operates an upper tier COMAH site near Hartlepool, United Kingdom. They employ approximately 85 people on a site covering approximately 11 acres. On the site, they manufacture a range of specialty chemical products. Many of these products are synthesised using semi-batch alkoxylation reactions i.e. involving Ethylene Oxide (EO) and/or Propylene Oxide (PO).

On 12th October 1999, a runaway reaction occurred. Whilst nobody was injured, the incident resulted in a significant release of Propylene Oxide to atmosphere despite the presence of a knockout pot. The incident highlighted a number of deficiencies both in the design and operation of the reactor and the pressure relief system. This paper describes the incident and its causes, the improvements made to prevent further recurrence, and the re-design of the relief and discharge and disposal system.

## **DESCRIPTION OF THE REACTOR PLANT**

Prior to the incident, Baker Petrolite had six stainless steel reactors of varying capacity in their reactor plant building. Most of the reactions carried out in these reactors involved Alkoxylation reactions, although other products were also made. Four reactors have top entry agitators and two reactors have external re-circulation loops. Each reactor was protected from overpressure by a relief system comprising of a graphite bursting disc and a vent line. Each vent line was routed independently to a common knockout pot (V7). A Photograph of V7 is given in figure 1.

V7 was a vertical vessel of approximately  $27 \text{ m}^3$  in volume and atmospheric pressure rated. It was located inside the reactor building and contained approximately 2000 kg of water (intended to provided some degree of quench and dissolution of EO or PO) maintained at a constant level by a ball cock level control device. Each of the six reactor vent lines entered the knockout pot separately. The outlet from the knockout pot was 0.6 m diameter and routed vertically to atmosphere via the roof of the building in which the reactors were housed. The discharge height was approximately 10 m above ground.

Reactors were mounted on load cells and as such the relief vents were linked to the reactor via a sliding joint arrangement originally designed by engineering consultants. This allowed sufficient relative movement to ensure effective operation of the load cells. The incident occurred in reactor V6 which is one of the reactors with an external re-circulation loop. V6 has a volume of  $22 \text{ m}^3$  and a design pressure rating of 8.6 barg. The graphite bursting disc had a burst pressure of 7.7 barg at 20°C, and a diameter of 0.3 m. The vent line from V6 to V7 had a diameter of 0.4 m.

# **DESCRIPTION OF THE INCIDENT**

The incident occurred at approximately 16:50 hours on 12th October 1999. The product being made was a polypropylene glycol in reactor V6. This involved the reaction between a low molecular weight Polyol and Propylene Oxide. The process steps involved up to and including Propylene Oxide addition are summarised below

- 1. Add starting Polyol
- 2. Add 45% Potassium Hydroxide solution (catalyst)
- 3. Perform pressure test to approximately 60 psi
- 4. Heat to approximately 110°C to dehydrate
- 5. Sample to check moisture
- 6. Apply nitrogen padding to reactor
- 7. Add Propylene Oxide at controlled rate

The Propylene Oxide was pumped to the reactor from a storage tank and the addition quantity was controlled to maintain the temperature between 110–115°C. If necessary, cooling was applied by use of water at ambient temperature: normally done automatically, it was under manual control due to external circumstances. On the day in question, the steps as far as the pressure test had been completed as normal and PO addition started about 0830 hours. The temperature of the reaction mix just prior to PO addition was between 80 and 90°C. Normally, the PO addition continues uninterrupted until all PO has been added. However, on this day, a number of interruptions to PO addition occurred one due to filling of the PO storage tanks and another due to the installation of new gas detectors in the reactor building.

After all interruptions were over, PO addition was resumed although at this point the temperature of the reaction mixture had dropped to 60°C. Over the next 2 hours, the temperature of the reaction mix increased from 60°C to 100°C and the pressure from 2.8 to 4.1 barg at which time the operator stopped PO addition and applied cooling. The pressure started to fall so the operator isolated the cooling and left the control room to carry out other

duties. A few minutes later, a loud bang was heard. Immediately following the bang, a spray of liquid was seen leaving V7 vent on the reactor plant roof directing towards 400 and 700 tank farms. Eyewitness reports indicated release duration of approximately 60 seconds. Subsequent inspection showed that reactor V6 bursting disc had ruptured.

## **CONSEQUENCES AND INVESTIGATION OF THE INCIDENT**

Fortunately, nobody either on or off-site was injured. However some storage tanks were splashed by released material. In addition, some liquid reached a branch railway line serving industrial users and which runs close to the Eastern edge of the site boundary.

A mass balance indicated that approximately 4775 kg of liquid and vapour was released to atmosphere. This included approximately 1300 kg of water from the knockout pot. This indicated that about 3475 kg was lost from reactor V6. The water in V7 was removed probably by a combination of vaporisation and displacement. The amount of pure PO released to atmosphere was difficult to determine. Subsequent calorimetry on a similar process indicated that in order to reach the disc burst pressure, approximately 30–40% of the PO charged would need to accumulate and much of this would have relieved to V7 due to its volatility. This would have amounted to a few tonnes. It was difficult to predict how much pure PO was released to atmosphere as some may have reacted with water in V7 to form Propylene Glycol. However, due to the short contact time and design of V7 (dip tubes into water only) only a fraction is likely to have reacted so much of the PO remaining in the reactor was assumed to have been released to atmosphere.

The amount of material released from V6 was about 30% by weight of the total. This indicated that homogeneous two-phase flow almost certainly didn't occur although some liquid was entrained in the venting stream. There was no visible damage to V6, V7 or the vent line from V6 to V7. In addition, V6 passed a subsequent pressure test indicating no distortion of flanges etc. Although this didn't necessarily indicate that the relief system was adequately sized i.e. design pressure not exceeded, it did demonstrate that the relief event didn't subject V6 to a pressure beyond its yield. Lack of any visible damage to V7 was perhaps more surprising due its atmospheric design pressure rating. This was probably attributable to the fact that the discharge line from V7 was large in diameter i.e. 0.6 m, straight and short in length, thus giving minimal back pressure. Figure 2 shows the internals of V7 after the incident. The original water level can be clearly seen from the watermarks on the dip pipes.

## CAUSE OF THE INCIDENT

The cause of the incident was almost certainly due to accumulation of PO at the end of the addition period prior to the incident. The reaction rate is known to be slow at 60°C resulting in accumulation of PO. Nevertheless some reaction occurs at 60°C and this slowly heated the reaction mix to about 100°C over a 2 hour period accelerating all the time but without consuming much PO. In addition, because little reaction was evident, PO continued to be added, further increasing accumulation. At 100°C, the reaction rate is much faster and a large amount of the PO would have suddenly reacted as if the process was batch. Clearly,

isolation of the cooling just prior to the incident was also a significant contributory factor although by this time the amount of accumulation would likely have made the event unrecoverable by normal cooling alone.

Although not the direct cause interruptions to PO addition meant that the reactor was allowed to cool to 60°C prior to resuming PO addition. There was a low temperature trip (softwired) set at 60°C preventing addition of PO below this temperature.

## **IMPROVEMENTS**

The incident was reportable under RIDDOR and as a result The Health and Safety Executive were notified. An improvement notice was issued which required consideration of

- 1. Prevention of such an incident happening again
- 2. Review of adequacy of pressure relief system
- 3. Review of discharge and disposal arrangements

This incident clearly showed that the knockout pot (V7) was ineffective and steps were taken to commission the design of a replacement.

#### PREVENTION

With semi-batch alkoxylation reactions such as these, safety is often based upon prevention of accumulation of unreacted EO or  $PO^1$ . Accumulation can occur in a number of ways

- 1. EO or PO added at too low a temperature
- 2. EO or PO added too fast
- 3. Agitation failure
- 4. Catalyst not added
- 5. Combinations of the above

Some controls were already in place at the time of the incident to avoid these scenarios. These included

- Low Temperature trip to prevent addition of EO or PO below 60°C
- High pressure trip to indicate accumulation of EO or PO although this was shown to be ineffective for PO due to the lower vapour pressure of PO
- Agitator sensing interlocked to EO/PO feed systems
- Exotherm detection on some reactors

Adiabatic dewar calorimetry was carried out on a similar PO process to determine the effect of accumulation of PO on peak reaction pressure and rate of temperature and pressure rise. The results are presented in figure 3. The minimum reactor design pressure is 8.6 barg and the disc burst pressure is 7.7 barg on all reactors. This indicates that provided the accumulation is not allowed to increase beyond about 30%, the disc burst pressure shouldn't be exceeded. Subsequent isothermal calorimetry indicated that addition of PO at 60°C and at the then maximum allowable feedrate of 3500 kg/hr, resulted in accumulation levels at least as high as 40%. Using DIERS methodology<sup>2,3</sup>, the required vent diameter at 40%

accumulation was calculated to be 0.7 m. The vent diameter on V6 and other similar sized reactors was 0.3 m and it was not practical to increase to 0.7 m due to space restriction on the reactor top dish.

A detailed risk assessment was then carried out for the process involved in the incident. One of the outcomes of this assessment was to review the low temperature trip settings and maximum allowable feedrates of EO and PO. Further calorimetry work was undertaken. At 95°C, isothermal calorimetry showed that addition of PO or EO at a maximum rate of 2000 kg/hr would result in a maximum accumulation level of about 15% i.e. 15% of the reaction heat was released at the end of the addition of PO/EO. From figure 3, this indicated a peak pressure of 2.6 barg for the PO product tested, which is below all reactor design pressures. The equivalent maximum pressure in the case of EO (based upon a typical process) was 7.8 barg, again below the design pressure of all the reactors, although not below the disc burst pressures. The adiabatic results for EO at 15% accumulation are shown graphically in figure 4.

As a result, the low temperature trip was increased to 95°C. In addition, restrictor orifices (ROPs) were fitted to the EO and PO feed systems to restrict feedrates to 2000 kg/hr. Whilst it was shown that 2000 kg/hr was acceptable as a maximum feedrate, 1500 kg/hr has been adopted as a maximum operating rate to provide an additional margin of safety particularly for the EO processes and to keep peak pressure below disc burst pressure. A margin of safety was also required to account for the fact that the PO and EO processes tested were being assumed to be representative of a range of PO and EO reactions carried out. However, the two selected were considered to be worst case representations of the PO and EO processes respectively. The ROPs, low temperature trips and agitator sensors and interlocks are clearly safety critical in terms of preventing accumulation to levels whereby a relief system upgrade was not required.

#### PRESSURE RELIEF SYSTEM

Based upon the improvements made to the process control to prevent accumulation, it was decided that the relief system would not need to be designed to deal with runaway reaction scenarios. Work is still ongoing to study the required safety integrity levels of all safety critical systems according to the requirements of IEC61508. Most of the other non-alkoxylation reactions carried out on the plant were shown not to present a high potential for runaway. This was because many of the reactions were operated semi-batch and it was shown that even at room temperature, accumulation was insignificant. Again, process control is used as the primary basis of safety.

However, the relief systems would need to be able to deal with the most demanding of other non-reactive relief cases. It was shown that the most demanding of the non-reactive relief cases was external fire (as is often the case). Relief calculations showed that the existing vents had sufficient capacity to cope with all vapour flow due to fire boil up. The vents also had sufficient capacity for homogeneous two-phase flow in the event of external fire. However, it was considered that for alkoxylation reactions at least, homogeneous two-phase flow was unlikely as demonstrated by the incident itself. Two-phase flow calculations were performed using DIERS methodology.<sup>2,3</sup>

## DISCHARGE AND DISPOSAL

One of the most important improvement measures was how to prevent further releases of PO or EO to the atmosphere. Clearly V7 was ineffective as a knockout pot as it contained very little of the relieving fluid during the incident. Because of the high volatility of EO and PO, it was decided that in the event of pressure relief, EO or PO would flash to vapour and thus a knockout pot or catch tank would be ineffective in preventing atmospheric release. However, EO and PO are highly soluble in water, so it was decided to design and install a quench tank. A quench tank has a number of advantages over a simple knockout pot in that it would

- 1. Achieve partial if not complete absorption of EO or PO preventing atmospheric release
- 2. Cool the mixture thereby reducing the likelihood of any on-going reaction

The ideal solution would have been to install a dedicated quench tank for each reactor. However, this would not only have incurred very high cost and leadtime, but more importantly, insufficient space was available for more than one tank. However, use of a single tank meant that the relief systems would need to be combined prior to entering the tank. This presented a few issues which needed consideration and which are discussed later in the paper.

Detailed bench scale quench tests were not performed. Whilst such tests are normally required to prove that a particular reaction will quench<sup>4</sup> (and recommended by the authors), their omission in this case was justified for the following reasons

- 1. Following involvement of the HSE, time was very tight to complete the design. Although not a key factor where safety is concerned, the company was keen to ensure continuity of supply to key customers.
- 2. The primary purpose of the quench tank was to absorb EO or PO rather than to achieve full reaction quenching. The ability of a particular design to do this would be less easy to demonstrate by small-scale tests.
- 3. Due to the process control improvements made, it was unlikely that runaway reactions on PO processes would need quenching as they would be contained.
- 4. The worst credible EO runaway following improvements also wouldn't exceed the reactor design pressures, although did approach the disc burst pressure. The need to quench EO reactions was therefore considered a possibility. However, because the remaining EO quantity available to react would be small, it was considered that only a small proportion of the reaction mix would need quenching.
- 5. In the case of external fire, it was also estimated that much of the EO or PO would have reacted prior to the disc bursting, again reducing the emphasis on the need to quench.
- 6. Many alkoxylation reaction products are highly soluble in water. Thus it was likely that water would be an effective cooling medium.

In addition, to compensate for absence of quench test data and to satisfy the HSE, some pessimistic assumptions were made in the design of the quench tank

1. Heat balance assumed that all the contents of the largest reactor (18000 kg) could be released to the quench tank at maximum EO reaction temperature of 200°C

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- 2. Heat balance took into account heat of solution of EO in water
- 3. Heat balance assumed that unreacted EO would continue to react in the quench tank

The heat balance assumed a final temperature below 80°C where it was known that the normal reaction at least, would slow down appreciably. In addition, the 80°C was below the atmospheric boiling point of the most concentrated EO solution likely to form in the quench tank. The heat balance showed that 35000 kg of quench fluid was required. The quench fluid was a 25% solution of Glycol in water to prevent freezing. This is a very important consideration with quench tanks and provision needs to be made to sample the quench fluid to check Glycol levels. Another important factor was how to obtain a suitable vessel for the quench tank within a very short timeframe. A second hand pressure vessel had become available ex Chlorine duty. This was a horizontal vessel of approximately 74 m<sup>3</sup> in volume, with a design pressure of 13 barg. This was sufficiently large to contain all the quench fluid and provide adequate freeboard height to allow for liquid swell and to prevent droplet entrainment.

The possibility of simultaneous relief (i.e. relief from more than one reactor at the same time) was considered. Simultaneous relief would have required a quenchtank of at least twice the volume i.e. at least  $150 \text{ m}^3$ . This would have required construction of a new tank at increased cost and leadtime. Whilst simultaneous relief could not be totally discounted, it was considered that the frequency was sufficiently low such that installation of a larger quenchtank was not justified on reasonably practicable grounds. The next challenge was to design the tank internals i.e. the internal sparger. Factors to consider here were:

- Selection of appropriate hole size e.g. to minimise blockage and water hammer risk
- Determination of number of holes
- How to ensure even distribution of relieving liquid and vapour amongst the holes
- How to maximise contact time and area with the quench fluid

To design the sparger for the tank, guidance given in the CCPS guide on Pressure Relief and Effluent handling<sup>5</sup> was used. This was found to be a very useful and practical source of information. The final design consists of a central manifold made of 0.76 m diameter pipe, with twelve 0.15 m diameter pipe branches. The holes were drilled into the branches uniformly around the circumference to even out reaction forces as much as possible. In total, 1540 holes of 10 mm diameter were used. The final sparger arrangement inside the quench tank is shown diagrammatically in figure 5.

A sparger with side arms was used to maximise contact area with the quench fluid. A single central pipe would probably have been adequate but this wouldn't have as good use of all available water volume and would thus be less efficient. The sparger was positioned as low as possible within the tank to maximise depth of submergence and thus maximise the likely contact time for heat and mass transfer. However, the sparger had to be positioned sufficiently far above the base to permit the side branch length to be long enough to fit on the required number of holes whilst achieving minimum hole spacing in line with recommended guidance. All in all, the design process was iterative.

The relief line from the reactors entered axially primarily to facilitate installation of the 0.76 m central manifold into the tank. The side branches were fitted onto the central

manifold within the tank. This required construction of a new manway on one dished end of the vessel. The design pressure rating of the tank needed to be maintained. Special supports were designed to secure the sparger arrangement within the tank to withstand likely reaction forces. The tank itself rested on saddles and it was shown that the friction generated by the weight of the tank would be sufficient to withstand axial reaction forces as the vented liquid first came into contact with the quench fluid. However, additional restraints were fitted to provide greater security. The final installed quench tank is shown in figure 6.

Another factor to consider was the effect of the nitrogen padding in the reactor. The nitrogen wouldn't be dissolved within the quench fluid and would need to disengage from the liquid. Because the reaction products were effectively surfactants, it was considered likely that some foaming would occur during sparging. It was considered possible that the foam would prevent disengagement of the nitrogen gas without carry over of foam out of the new tank. Whilst release of foam to atmosphere did not carry a high risk, it was considered unacceptable to risk any release to atmosphere. For this reason, a second tank was installed to catch any foam or liquid which might get entrained by the nitrogen. Installation of this tank was an accepted option for the Health and Safety Executive. A photo of the second containment tank is given in figure 7.

The next step was to design the final atmospheric stack from the second containment vessel. It was decided to adopt a pessimistic approach and design the stack on the basis that no EO would be absorbed by the quench fluid. Dispersion modelling was performed using PHAST version  $6^6$ . The modelling considered the stack height and diameter required to avoid a potentially flammable cloud impinging upon a nearby elevated light. It was also necessary to avoid ground level concentrations both on and offsite which could cause acute harm to people. The concentrations of interest were the Lower Flammable Limit (LFL) for flammable effects and the Immediately Dangerous to Life and Health (IDLH) concentration for toxic effects. For EO, these concentrations were 30000 parts per million (ppm) and 800 ppm respectively. To provide an added margin of safety, the flammable cloud at LFL/2 was modelled. Weather conditions 1.5F and 5D were used which are present 70–80% of the time in the area.

After consideration of wake effects of nearby tank farms, it was decided to install a stack of 0.6 m diameter, 15 metres high with a 0.3 m diameter tip to increase vertical velocity and aid dispersion. This gave a comfortable clearance of the LFL/2 plume envelope from the light and other obvious ignition sources. Also, at no point would the ground level concentration have exceeded the IDLH concentration. The final stack design is also shown in figure 7 in relation to the elevated light.

#### **MECHANICAL DESIGN OF PRESSURE RELIEF SYSTEM**

The new position of the quench tank required a re-design of the pressure relief pipe work routings. It was also necessary to link all the relief pipework together prior to entering the quench tank. Because a choke would likely occur upon entry to the quench tank, a relief event in one reactor would create backpressure within the vent network and impose backpressure on other reactor discs. Also leakage from one disc could allow product to accumulate on the vent side of another disc. There were therefore several challenges in the re-design of the vent system pipework. Some of these included

- 1. Routing of pipework to minimise stress e.g. due to thermal expansion
- 2. Design of pipework supports for reaction forces
- 3. Design of flexible connections to allow load cells to function
- 4. Prevention of discs rupturing in reverse direction
- 5. Provision of a method to inspect discs without removing vent pipework.

The most difficult challenge was in the design of the flexible connections. Bellows joints were not capable of withstanding the reaction forces, particularly in the event of two-phase flow. The final design used a vertical sliding joint arrangement, which provided just enough movement to allow correct function of the load cells at the same time providing sufficient strength along its axis to withstand at least 20 tonnes force. An example of the final design is given in figure 8. Prevention of discs rupturing in the reverse direction required consideration of two factors

- 1. Static pressure in vent network during venting
- 2. Reflected pressure when vented fluid first contacts stationary water in spargers.

Due to the complexity of the system and the wide range of likely relief scenarios and products, it was difficult to evaluate such pressures with confidence. However, the existing graphite discs had a reverse pressure capability of only a fraction of the forward burst pressure. Therefore it was decided to replace the discs with stainless steel reverse acting discs of a design to provide a higher reverse pressure strength than forward burst strength. This was certainly adequate to handle the maximum vent static pressure which would not exceed forward burst pressure. It was estimated that the fluid static pressure when it first contacted the quench fluid would be about 4 barg. The reverse pressure capability of the new discs was in excess of 8 barg which was more than twice the static pressure at the point of quench fluid contact. This was considered adequate in the absence of a more complex analysis. In order to inspect the discs in situ, a manhole was installed as shown in figure 8.

# SECOND RUNAWAY REACTION INCIDENT

In January 2002, control was lost of another reaction on the reactor plant. This was not an alkoxylation reaction. The bursting disc on the reactor ruptured and several tonnes of hot material was released as a two-phase flow to the new quench tank. Whilst this was not a reaction for which the tank was primarily designed, it did provide a full-scale test of the new system which performed well. In particular

- No material was carried beyond the quench tank
- No other discs burst in reverse
- The flexible joints performed well
- Pipework and supports withstood reaction forces
- The sparger holes didn't block despite the very high viscosity of this particular reaction product (very much higher than a typical alkoxylated product)

The good performance of the system was not surprising bearing in mind the safety factors built into the design to account for uncertainties (uncertainties are always present with such designs). However, there is now a higher degree of confidence that the system will perform when required to do so. Also, the system design is being adopted as best practice within the company and designs are underway for a similar system at another UK site.

## LESSONS LEARNT

A number of general lessons can be learnt from this incident. Some of these are highlighted below

- 1. Incidents cost money. Due to the use of a common manifold, all reactors had to be shut down during installation resulting in lost production, in addition to the cost of the plant improvements.
- 2. Semi-batch reactions can be as inherently unsafe as pure batch reactions, unless the correct operating conditions to prevent accumulation are known. Calorimetry is required to determine such safe operating conditions. The cause of the incident was directly related to inadequate understanding of the reaction thermochemistry.
- 3. Process control can often be used effectively as a means to reduce relief demands due to runaway reaction and thus reduce required relief size. However, consideration needs to be given to the required safety integrity required.
- 4. Designing pressure relief systems is not just a case of determining a correct vent diameter. Close attention to the mechanical and discharge and disposal design is also required. This is particularly true when linking several reactors into a common header.
- 5. Discharge and disposal Systems e.g. Knockout pots, are unlikely to work effectively unless properly designed.

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Figure 1. V7 knockout pot



Figure 2. Inside of V7 after incident

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Figure 3. Peak reactor pressure as a function of propylene oxide accumulation



Figure 4. Adiabatic dewar data for a typical EO reaction at 15% accumulation

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Figure 6. New quench tank

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Figure 7. Secondary containment vessel and vapour discharge stack



Figure 8. New sliding joint arrangement on pressure relief pipework