

# PREVENTION, DETECTION AND MITIGATION OF RUNAWAY REACTIONS

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## ABSTRACT

The prediction, early detection and relief of uncontrolled exothermic chemical reactions which can lead to thermal runaways in reactors and vessels are of paramount importance to the chemical process industry. Apart from the release of chemical inventories into the environment, runaway reactions may lead to damage to equipment and plant and in extreme cases to catastrophic explosions and loss of life.

The recently completed European project on Advanced Warning And Runaway Disposal (AWARD) had as its aims the prevention of runaway reactions and substantial improvement in the design and reliability of pressure relief systems. An early warning detection system (EWDS) was developed which will reliably detect runaway while drastically reducing the number of false alarms and reactor shutdowns. The EWDS uses a novel detection algorithm, was proven in an industrial environment and has been developed into a commercially available system.

Work on pressure relief systems focussed on gaining a better understanding of the extent to which a two-phase vapour/liquid mixture is vented. This affects the sizing of the pressure relief system itself and any downstream disposal system to protect the environment. Improved sizing methods were developed and validated through a series of unique large-scale experiments.

## 1.0 NOMENCLATURE

$div$  divergence

$V_{PS}$  volume of phase space

$\dot{V}_{PS}$  rate of change of phase space volume

$t$  time (s)

$T$  temperature (K)

$\alpha$  void fraction

$\delta$  standard deviation

## 2.0 INTRODUCTION

The maintenance of safe operating conditions for chemical reactors is of paramount importance to avoid accidents and damage to personnel and installations and environmental pollution. The design of relief disposal systems for exothermic reactions is an important problem faced by the chemical industry across the European Union. The Advanced Warning And Runaway Disposal (AWARD) European project addressed the needs to detect runaway initiation in advance to allow countermeasures to be taken, and to design emergency relief systems for chemical reactors.

The overall strategic objective of the research was twofold:

1. to develop, validate and test in an industrial environment, a device for the early detection of the initiation of runaway events. Such a device should be able to cope with industrial conditions and in particular, noise signals, rough handling and frequent changes in process.
2. to produce a methodology for the design of disposal systems to protect the workers and the environment from the effects of pressure relief of runaway chemical reactions. The methodology needed to be capable of producing a disposal system, which is adequate but not significantly oversized.

The AWARD project was funded under the Competitive and Sustainable Growth Programme of the Fifth Framework. At the request of the European Commission, AWARD resulted from the merging of two successful complementary proposals: “Advanced Warning Against Runaway Events” (AWARE), which addressed the first objective above, and “Inherent Safe Process Design and Improved Vent Disposal System Design for Exothermic Reactors”(DISPOSE), which addressed the second. Merging these proposals into a single project brought to the fore a number of synergies between the proposals, including allowing the results of large-scale experiments within the DISPOSE proposal to also be used for the validation of the early warning device.

The project, which had a budget of € 3.7 million, was coordinated by the University of Manchester and had two technical coordinators: the Joint Research Centre (EU) for objective 1; and the Health and Safety Laboratory (UK) for objective 2. There were 15 partners from 8 European countries plus two Institutes of the Joint Research Centre (see Appendix). 13 were original partners and 2 joined in the course of the project. The project ran from 2001 to 2005.

### **3.0 DEVELOPMENT OF THE EARLY WARNING DETECTION DEVICE (EWDS)**

#### **Basis of the EWDS**

The AWARD Early Warning Detection System (EWDS) was intended to improve the quality, safety and reliability of production operations and reduce accidents in chemical plants by developing and testing a new device for early warning detection of runaway events based on the application of chaos theory techniques. The concept had already been patented [1] but required further development in order to be turned into a commercial device and to demonstrate both that it was able to distinguish between runaway and non-runaway situations and that it did not produce false alarms during controlled heating/cooling or in the case of quasi-instantaneous semibatch operation.

The EWDS requires only temperature measurements, or a representative variable of the state of the reactor [2], and is based on state space reconstruction techniques developed in non-linear dynamical systems theory and on the on-line calculation of the divergence of the system [3]. In order to calculate the divergence without needing to know the differential equations of the system we have used the theory of embedding. The extension of this theory to the case of discontinuous chemical reactors is complicated by the nonstationarity of the system that implies that the embedding parameters (time delay and the embedding dimension) are changing during the process. The divergence gives the rate of expansion or contraction of the phase space volume,  $V_{PS}$ , of a dynamical system and, and it may be calculated as:

$$div = \frac{\dot{V}_{PS}(t)}{V_{PS}(t)} \quad (1)$$

The divergence reconstruction algorithm was tested with ramped heating/cooling, batch and semibatch runaway experiments and quasi-instantaneous reaction experiments in a 2 L bench scale reaction

calorimeter (RC1 from Mettler-Toledo). It was found that compared with a conventional limit check, for example the first derivative of the reactor temperature, runaway was detected around 60 seconds in advance. In these calorimetric experiments, the selected value of  $dT/dt$  for comparison was  $0.01^{\circ}\text{C/s}$ , to avoid false alarms due to heating/cooling.

## Industrial experiments

The EWDS was intensively tested in two industrial plants at Rohm and Haas (R&H) in Italy (Figure 1) and Esteve Quimica (EQ) in Spain during normal operations. These industrial experiments allowed for testing of the robustness of the early warning detection system (EWDS) against high levels of noise and frequent manipulations by plant operators. Around 150 batches were recorded at R&H and 16 at EQ. Furthermore, offline analysis was also performed using industrial data from Repsol S.A concerning a polymerization process. It was demonstrated that the EWDS can be successfully used within an industrial environment without giving rise to false alarms.

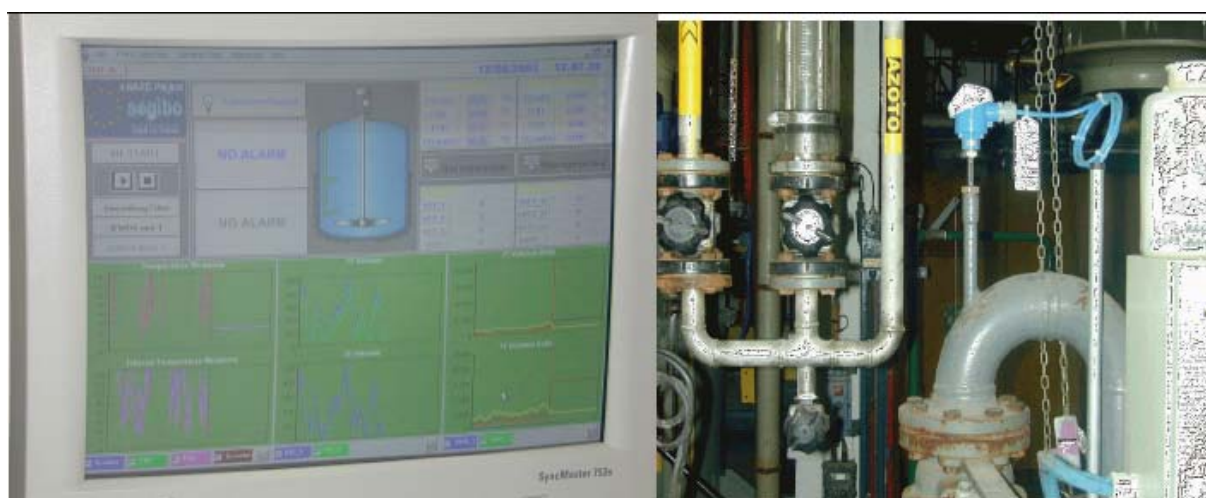


Figure 1 Prototype EWDS device installed on a reactor at R&H industrial site at Bergamo, Italy

As an example, Figures 2 and 3 show an industrial reactor for the same reaction but with a normal behaviour and with a sudden auto-acceleration. As can be seen, during the first experiment there are no alarms produced, whereas in the second experiment an alarm is triggered off 25 minutes before the end of the time sequence.

## Runaway experiments

A series of runaway experiments was carried out in the pilot plant at the Health and Safety Laboratory (Buxton, UK) using the esterification reaction between isopropanol and propionic anhydride. The model developed for this process allows the comparison between simulated and experimentally reconstructed divergences. The algorithm was able to distinguish between two clear runaway cases and a slow reaction case, close to runaway conditions [4, 5].

It was decided to use the DISPOSE large-scale experiments (see below) and analyse the results with the EWDS. This analysis demonstrated that the device was able to detect the runaway in good time despite the use of conservative parameters and the signal without filtering. Furthermore, no false alarms were triggered during the heating, addition nor cooling stages. For the final experiment, it was possible to run the EWDS algorithm in real time and to indicate the point at which the EWDS triggered on the video of the experiment.

A detailed analysis of all the experiments showed that it was possible to achieve an advance of around 10 minutes when compared with a traditional limit check as the first reactor temperature derivate (assuming the noise is eliminated and the signal may be smoothed enough to avoid continuous false alarms).

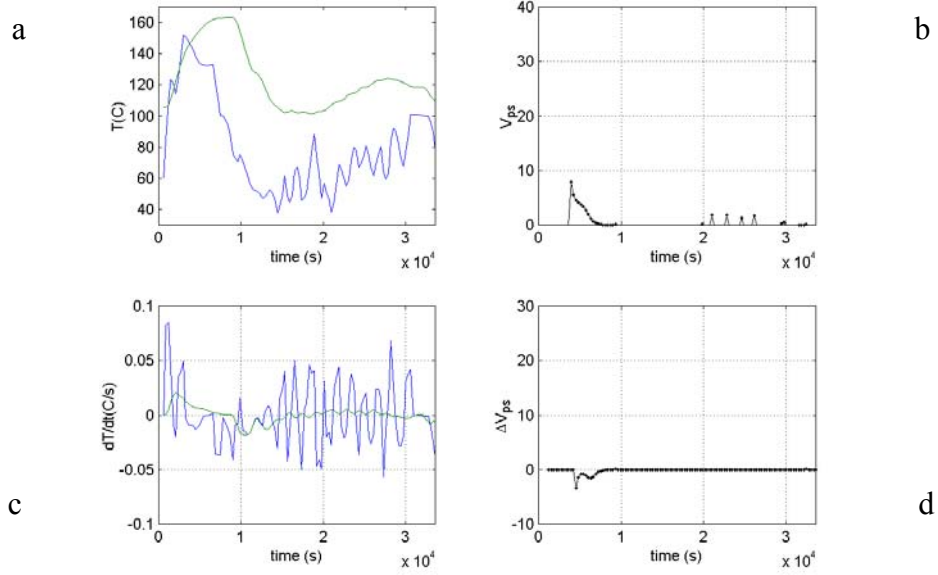


Figure 2. a/Industrial Reactor and jacket temperatures; b/  $V_{PS}$  values; c/Reactor and jacket derivatives; d/  $\Delta V_{PS}$  values. Parameters  $\Delta t=1$ ;  $\Delta V_{lim}=0.55$  (sampling is every 300 s).

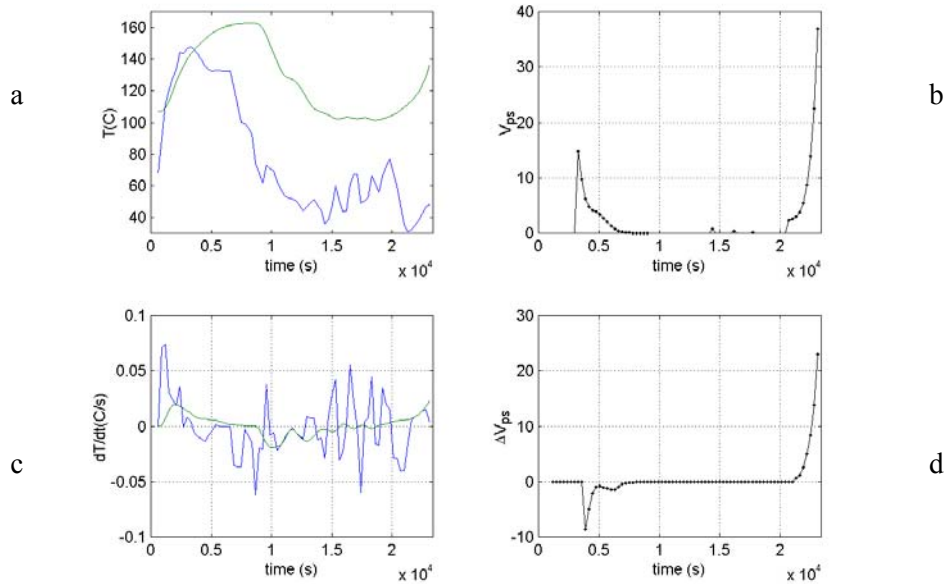


Figure 3. a/Industrial Reactor and jacket temperatures; b/  $V_{PS}$  values; c/Reactor and jacket derivatives; d/  $\Delta V_{PS}$  values. Runaway alarm starts 25 minutes before the end of the registration. Parameters  $\Delta t = 1$ ;  $\Delta V_{lim} = 0.55$ . (sampling is every 300 s).

## EWDS development from prototype to commercial system

One of the AWARD partners was Segibo, an industrial company operating in the sector of plant engineering and control systems manufacturing, particularly for fine chemical and pharmaceutical production. Within the project Segibo was tasked with developing the EWDS into a commercial system and for its subsequent exploitation.

Based on the preliminary experiments and calorimetric laboratory tests, a prototype version of the EWDS was developed and the runaway detection algorithm implemented. Two prototype units were produced for testing. After the industrial site demonstration and the runaway experiments at HSL were analysed, a modified version of the algorithm was developed to:

- improve the signal to noise ratio;
- take out the jacket temperature from the state space reconstruction algorithm and use it only as an indicator (it was observed that the jacket temperature was not always correlated with the dynamics inside the reactor, e.g. the same coolant was used for other purposes).

The improved algorithm was tested by analysis of the existing experiments [4, 5]. The results showed that from the point of view of the time offered for implementing countermeasures to be taken and of the robustness to operator intervention, this new device outperforms existing commercially available early warning detection systems.

The EWDS has now been implemented in several configurations by Segibo for commercialization as shown in Figure 4. There is one version as an industrial PC, another version that may be incorporated in a rack, a PLC version and finally, dedicated stand alone software that may be introduced into the data acquisition and control system of the client.

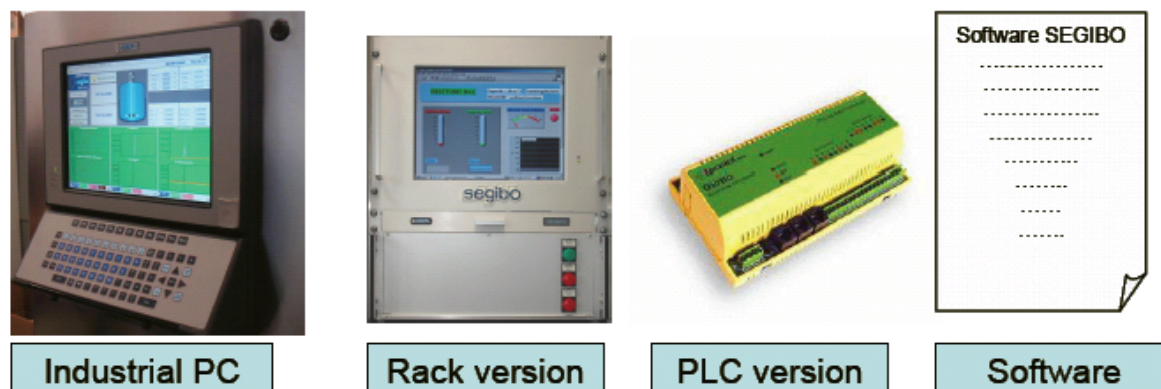


Figure 4. Configurations of the EWDS

## 4.0 DEVELOPMENT OF A METHODOLOGY FOR DISPOSAL SYSTEM SIZING

### Objectives

This part of the project was aimed at producing a methodology for the design of disposal systems to protect the environment from pressure relief of a runaway chemical reaction. This included simplified guidelines for use by small and medium enterprises (SMEs) and software for more detailed analysis. SMEs were a specific target for the results of the work because of their important wealth generating role within Europe and the fact that a substantial proportion of businesses in the European Union which are classified as SMEs carry out industrial processes which involve exothermic reactions or

chemical operations which require a good understanding of reactive hazards and of the design and operation of safety venting devices. It was recognised that the different requirements of health and safety regulation and environmental regulation may require resources which are beyond the reach of most SMEs. One of the AWARD partners was Arran Chemical Company, a chemical industry SME in Ireland, which took the role of helping to target the output for SMEs.

In order to develop this methodology, the following aspects had to be considered:

- Development of reliable methods to predict the level swell in a venting chemical reactor during exothermic runaway and hence the flow rate and quantity of liquid which needs to be retained by the disposal system;
- Testing both the level swell methods and the design/sizing methods against large-scale experiments.

### Level swell methodologies

During venting of a runaway chemical reactor, the volume of the reactor contents rises or swells with the volume of gas and/or vapour bubbles formed within the liquid. The ability to model level swell is essential in order to determine how much liquid the disposal system must be designed to handle. Most existing methodologies are based on a ‘drift flux’ approach in which the level swell is correlated with the rise velocity of a typical bubble, or the fall velocity of a single liquid droplet.

A dynamic reactor relief software code (RELIEF), which had been developed by the Joint Research Centre (JRC), was used as a means of evaluating different level swell models for validation against experimental data. JRC modified the RELIEF code to allow new models for level swell, kinetics etc to be introduced. The bubble slip models developed by Kataoka & Ishii [6] and by Boesmans [7], on the basis of bubble column experiments, linked with the droplet slip model by Wallis [8], were chosen for implementation following a critical review of available models, carried out by Inburex of Germany [9].

The review of level swell models identified that the slip models were difficult to use in a numerical analysis because of a discontinuity after transition to the droplet flow regime. RELIEF overcomes this discontinuity but in a way that does not give a good fit for the droplet regime. Inburex developed an improved level swell model in which the slip undergoes a smooth transition from whichever model is used in the liquid continuous regime, to Wallis’s droplet slip model. This new model [9], which is illustrated in Figure 5, describes bubble flow independent of droplet flow and is capable of describing a breakup zone where liquid starts to break up and droplets are formed.

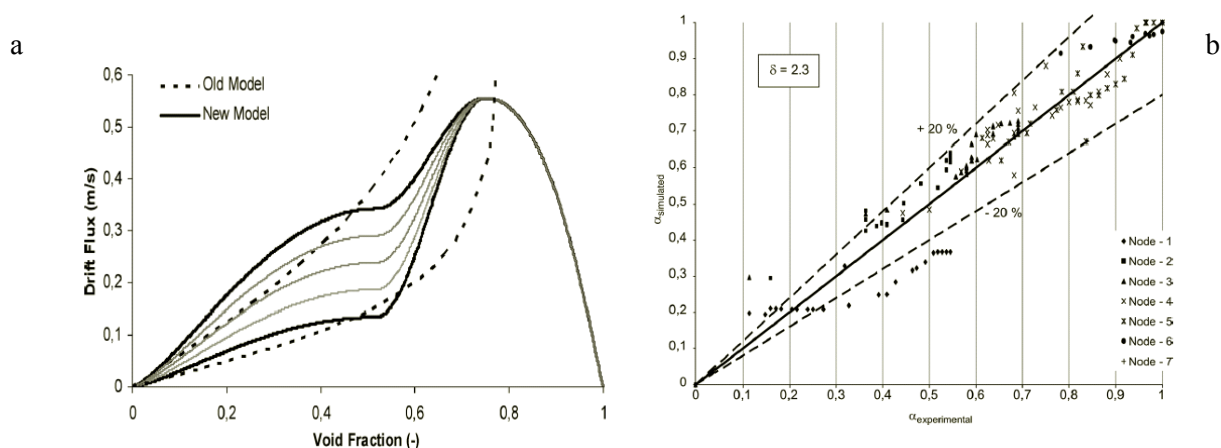


Figure 5. a/ Illustration of behaviour of new and old models; b/ Comparison of proposed model with the data of Sozzi [10]



Potential versions of the new model were compared with water blowdown data produced in experiments by Sozzi [10] (see Figure 5), by DIERS, and by the AWARD large-scale experiments (see below). The results show that the inclusion of a separate droplet flow model for high void fractions is important. The best correlation of both the level swell in the vessel and the vented mass was obtained with the Kataoka & Ishii bubble slip model, which was further modified to allow for the continuous variation of the gas flux. This was linked with the Wallis droplet model at void fractions above 0.75. The new level swell model shows a big improvement in the emergency relief computer simulation.

The University of Manchester had the task within the AWARD project to consider physical phenomena that are not taken into account within drift flux models. This particularly included foaminess and non-equilibrium effects. A detailed dynamic model was produced together with a simpler formulation for foaminess in terms of the drift flux methodology [11].

### Large-scale experiments

There was a lack of suitable data for the validation of level swell models, particularly under conditions of full scale runaway chemical reaction. As part of the AWARD project, a unique large-scale facility [8] was constructed at the Health and Safety Laboratory using a reactor comparable in size to industrial vessels. The facility, shown in Figure 6, comprises a 2,500 litre reactor connected via a 200 mm diameter vent line to a 13,000 litre catch tank. A large number of temperature and pressure transducers were installed at various points in the reactor, vent line and catch tank.



Figure 6. Large-scale facility showing reactor (within building), vent line and external catch tank

Level swell and the density distribution in the reactor are determined using differential pressure and a specially designed scanning gamma-ray system (see Figure 7). Collimated gamma ray beams emerge from source holders on one side of the reactor and rod-shaped detectors on the other side are used to monitor changes in attenuation caused by variations in the two-phase density in the vessel during pressure relief. The sources are rotated in order to record the variation in attenuation as the angle of the beam changes. The response from the detectors can be related to the axial variation of void fraction in the vessel. One of the differential pressure transducers records the pressure difference between diaphragms at the top and bottom of the reactor. The response is directly proportional to the mass of

the contents and is not affected by the density distribution or absolute pressure in the reactor. Two other transducers cover the middle and top sections of the reactor.

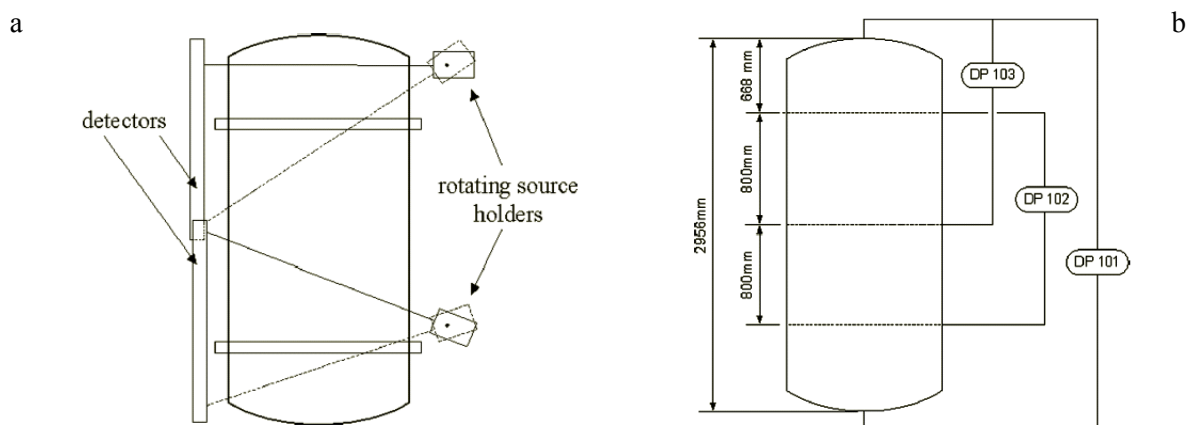


Figure 7. Instrumentation for measuring level swell: a/ Scanning gamma ray densitometer system; b/ configuration of differential pressure measurement

Typical results for two vented runaway reaction experiments, conducted with and without surfactant to modify the foaminess and level swell behaviour, are shown in Figure 8. The gamma ray attenuation shows both the effects of two-phase flow and an underlying effect from a flange in the upper part of the reactor. A procedure was developed for analyzing this data to obtain void fractions at different levels in the reactor.

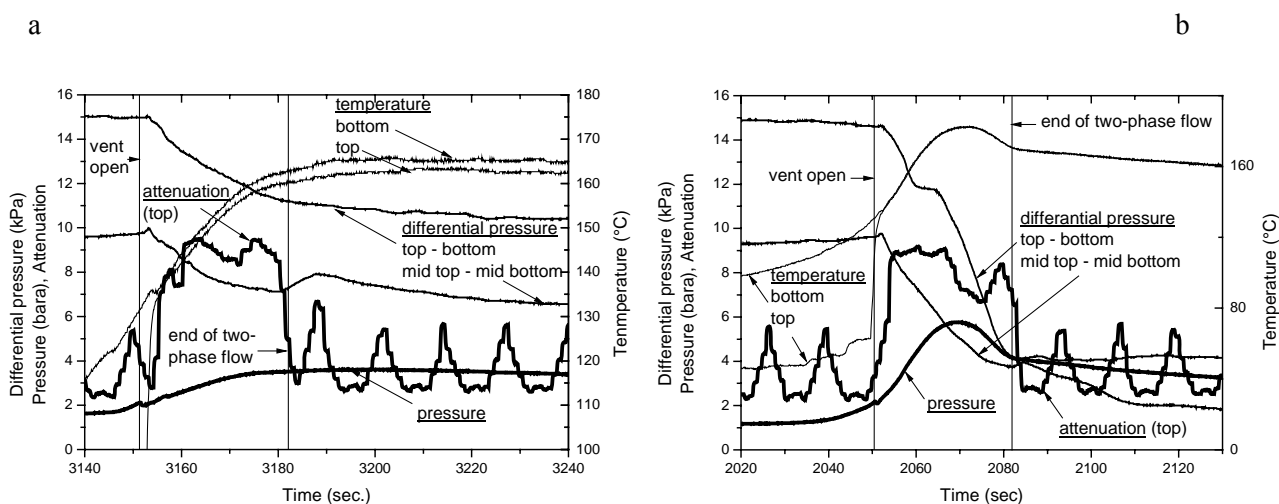


Figure 8. Variations in temperature, pressure, differential pressure and gamma-ray attenuation during large-scale venting of hydrolysis of acetic anhydride with 50% fill. a/ without surfactant b/ with the addition of surfactant

Blowdown experiments using this facility were used to validate improved new level swell models (see above). Runaway reaction experiments were used to validate simplified methodologies for both level swell and relief system sizing [12, 13]. These methodologies (see below) were confirmed as being conservative for the large-scale experiments which were carried out during the AWARD project. However, the high quality of instrumentation allowed, for the first time, a scientific validation of the empirical assumptions made within simplified vent sizing equations [12]. This showed that, although



the vent sizing calculations had been conservative, the underlying assumptions were not a true reflection of the physical processes which occurred during venting. The maximum pressure was shown to have been limited by reactant consumption rather than by tempering of the reaction by removal of latent heat by venting, as is assumed in the simplified methodologies.

### **Simplified guidance**

It was recognized that, if SMEs carry out vent and disposal system sizing themselves, they would do so using simple calculation procedures rather than sophisticated and expensive software. The Centre for Chemical Process Safety (CCPS) “Guidelines for Pressure Relief and Effluent Handling Systems” [14] gives suitable methods for the sizing of the disposal system, provided that input data about the quantity of liquid vented and the flow rate of the two-phase flow is known. The guidance produced by the AWARD project was therefore focused on these aspects.

Guidance [15] has been made available on the AWARD project web site which compares simplified sizing methods with large-scale experimental results from the AWARD project. Based on this limited comparison, recommendations are given for the estimation of:

- The quantity of material vented to the disposal system.
- The flow rate to the disposal system.
- The pressure in the reactor (which is an input to calculating the flow rate).

A brief overview is also given of the AWARD project as a whole and it is made clear that the dynamic modelling approaches (see above) have the potential for more accurate sizing of disposal systems.

## **5.0 BEYOND THE AWARD PROJECT**

### **Dissemination**

The dissemination of the findings is obviously an important part of any research project. The AWARD project did this in a number of ways, including links with relevant industry groups and networks including the European and USA DIERS User Groups (concerned with the design of pressure relief systems) and the HARSNET and S2S European networks. An AWARD web-site was set up early in the project and used for communication between the partners as well as between the partners and the public. Well over 100 papers, articles and technical reports have been produced by the project.

The AWARD European Technical Workshop on Advanced Warning and Runaway Disposal which took place in Lisbon, Portugal on 21 and 22 April 2005 was aimed at those working in the chemical process industry and, in particular, in SMEs. The meeting attracted over 50 participants including many representatives from organisations and companies outside the AWARD Partnership. Twenty four presentations were made at the Workshop. The workshop proceedings have been incorporated in a CD which is available to the public.

### **EWDS**

One of the objectives of the project was the development of the EWDS into a commercial system. This has been achieved and Segibo had the responsibility for its exploitation. Segibo have prepared a business plan for the commercial exploitation of the EWDS and have signed a license agreement with the European Commission for the use of the JRC patent [1], which constitutes the technological backbone of the EWDS system. Segibo has presented the EWDS at a number of international trade fairs including Achema at Frankfurt am Main; the International ESMG Symposium on Process Safety and Industrial Explosion Protection in Nürenberg; and several conferences and fairs in Italy.

Interest in the EWDS from industry has been high due to its proven advantages over existing systems and the inclusion of successful industrial trials during the AWARD project. Segibo has installed several equipments for trial at different companies. However, despite this, commercial exploitation has progressed slowly. A specialist supplier/ manufacturer of safety systems has shown its interest and an agreement to include the EWDS in their sales catalogue has been concluded. This may prove to be the best way to achieve take-up by industry.

### **Vent and disposal sizing methodologies**

The short guideline for SMEs [10] has been put into the public domain through the AWARD website. HSL developed spreadsheets to implement the proposed methodology as part of the AWARD project, but they could not be made generally available because of software quality assurance issues. HSL have subsequently obtained funding from the UK Department of Trade and Industry (DTI) to assess the viability of developing these spreadsheets into a commercially available tool, which it is hoped will make the methodology even more accessible to SMEs.

There are no existing international standards for two-phase pressure relief of chemical reactors, but Working Group 1 of ISO Technical Committee (TC) 185 is developing ISO EN 4126-10, a new standard and European norm which will cover two-phase pressure relief via safety valves and includes level swell. Both HSL and Inburex are represented on this working group, ensuring that the output of the AWARD project will become incorporated into standardization.

Concerning the more detailed software produced by the project, JRC have made a decision in principle to release the RELIEF code as an open source code. Its use for modeling is also available via Inburex Consulting, the partner in the project who developed the improved level swell model. The new model has been adequately described in the open literature [6] so that it can be incorporated in other relevant commercial software for vent sizing. The University of Manchester also decided to release in freeware and open source the software packages it had developed.

### **Further research on vent sizing**

As discussed above, the use of the large-scale experimental results to validate vent sizing methodologies led to the conclusion that commonly used vent sizing equations are based on fragile assumptions which were shown not to be valid during these experiments [8]. It should be made clear that the AWARD large-scale experiments were designed primarily to measure level swell and so the conditions were relatively unusual in terms of vent sizing; also that the use of the equations led to conservative vent sizes for these experiments. Nevertheless, following discussions at the European DIERS User Group, several industrial companies were interested in further experiments to check whether the assumptions are valid under more usual design conditions.

Accordingly, a jointly funded club project, the Chemical Reaction Assessment Group (Venting) (CRAG), is in negotiation. It is proposed that HSL will carry out further experiments in its large-scale facility using larger vent sizes than were used for the AWARD experiments. It is possible that there will also be a second phase of experiments to consider gas-generating reactions, which it was not possible to include in AWARD.

## **6.0 CONCLUSIONS**

The AWARD project successfully achieved its technical objectives to produce a commercial version of an early warning detection device (EWDS) to prevent runaway and to develop a methodology for the sizing of disposal systems to protect the environment from pressure relief of a runaway reaction. The EWDS is available in four different configurations. The methodology for disposal systems concentrated on the missing link, which was adequate level swell models. Unique new data for model validation was generated in a large-scale facility and improved software-based models were developed. In addition, simplified guidance suitable for SME's has been made available on the

AWARD project web site. The outputs from the project have been widely disseminated via a large number of open publications.

Uptake of the EWDS has not progressed as planned, with industry reluctant to take advantage of this new device which provides clear benefits over existing systems. Nevertheless, this improved technology is now available.

Work is in hand to further exploit and develop the vent and disposal sizing methodology. This includes incorporation in standardization; software tools; and further research to check the validity of common vent sizing equations under usual vent design conditions.

## 7.0 ACKNOWLEDGEMENTS

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## 8.0 DISCLAIMER

The opinions expressed in this paper are those of the authors and not necessarily those of the Health and Safety Laboratory nor the Health and Safety Executive.

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## **APPENDIX: PARTNERS IN THE AWARD PROJECT**

The following were the partners in the AWARD project:

1. University of Manchester/UMIST	UK
2. Health and Safety Executive (HSL)	UK
3. European Commission, Joint Research Centre, EIS (EIS-JRC)	EU
4. European Commission, Joint Research Centre, IPSC (IPSC-JRC)	EU
5. Institut Químic de Sarrià (IQS)	E
6. Università Carlo Cattaneo (LIUC)	I
7. Università degli Studi di Messina (UM)	I
8. Sanofi Chimie (Sanofi)	F
9. Arran Chemical Company Ltd (Arran)	IRL
10. Rohm & Haas Italia (R&H)	I
11. Esteve Quimica S.A. (EQ)	E
12. Segibo Srl. (Segibo)	I
13. Investigacao e Desenvolvimento em Engenharia e Ambiente Lda (IRRADIARE)	P
14. Warsaw University of Technology (WUT)	PL
15. Inburex Consulting GmbH (Inburex)	D