INVESTIGATION REPORT

CHEMICAL MANUFACTURING INCIDENT
(9 INJURED)

KEY ISSUES:

- INTERNAL HAZARD COMMUNICATION AND PROCESS SAFETY INFORMATION
- REACTIVE HAZARD MANAGEMENT
- PROCESS SAFETY MANAGEMENT
ABSTRACT

This report explains the explosion and fire that occurred on April 8, 1998, at the Morton International, Inc. (now Rohm & Haas) plant in Paterson, New Jersey. The explosion and fire were the consequence of a runaway reaction, which over-pressured a 2000-gallon capacity chemical reactor vessel and released flammable material that ignited. Nine employees were injured, including two seriously, and potentially hazardous materials were released into the community. The key safety issue covered in this report was reactive chemical process safety management. Recommendations concerning this issue were made to Morton International, Inc. (a wholly owned subsidiary of Rohm & Haas Company); Morton International, Inc.’s Paterson, New Jersey Plant; the Occupational Safety and Health Administration (OSHA); the Environmental Protection Agency (EPA); the American Chemical Council; the Center for Chemical Process Safety (CCPS); the Paper, Allied-Industrial, Chemical & Energy Workers International Union (PACE); and the Synthetic Organic Chemical Manufacturer’s Association (SOCMA).

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*Salus Populi Est Lex Suprema*
People’s Safety Is the Highest Law
# TABLE OF CONTENTS

## EXECUTIVE SUMMARY

- ES.1 INTRODUCTION .............................................................................................................. 1
- ES.2 INCIDENT .................................................................................................................. .................................. 1
- ES.3 KEY FINDINGS .............................................................................................................. ............................ 2
- ES.4 RECOMMENDATIONS ........................................................................................................... ................... 8

## 1.0 INTRODUCTION ............................................................................................................... ............... 11

- 1.1 BACKGROUND ................................................................................................................. ........................... 11
- 1.2 INVESTIGATION PROCESS ...................................................................................................... ................ 12
- 1.3 PLANT FACILITIES ........................................................................................................... ......................... 13
- 1.4 LAYOUT AND COMPONENTS ...................................................................................................... ............ 14
  - 1.4.1 Kettle Description ....................................................................................................... ........................... 14
  - 1.4.2 Kettle System ............................................................................................................ ...................... 15

## 2.0 DESCRIPTION OF THE INCIDENT ................................................................................................ ..18

- 2.1 INCIDENT EVENTS ............................................................................................................ ........................18
- 2.2 EMERGENCY RESPONSE ......................................................................................................... ...............26
- 2.3 OFF-SITE IMPACTS ........................................................................................................... .........................27
- 2.4 PROPERTY DAMAGE ............................................................................................................. ....................28

## 3.0 ANALYSIS OF PROCESS CHEMISTRY ........................................................................................29

- 3.1 REACTIVE CHEMICAL ANALYSIS ................................................................................................. .........29
- 3.2 TESTS OF RAW MATERIALS ..................................................................................................... ...............33

## 4.0 ANALYSIS OF PROCESS DEVELOPMENT PROGRAM ................................................................34

## 5.0 ANALYSIS OF PROCESS SAFETY MANAGEMENT PROGRAM ................................................38

- 5.1 PROCESS SAFETY INFORMATION ................................................................................................. 38
- 5.2 PROCESS HAZARD ANALYSIS ..................................................................................................... 40
- 5.3 INVESTIGATION OF PROBLEMS WITH PREVIOUS BATCHES ..........................................................43
- 5.4 MANAGEMENT OF CHANGE .................................................................................................... 45
- 5.5 OPERATING PROCEDURES AND TRAINING .................................................................................. 46

## 6.0 REGULATORY OVERSIGHT ANALYSIS ........................................................................................49

- 6.1 REGULATORY COVERAGE .......................................................................................................... 49
- 6.2 MORTON PSM PROGRAM .......................................................................................................... 51
- 6.3 GOOD PRACTICE GUIDELINES ................................................................................................. 52
- 6.4 RECENT CATASTROPHIC REACTIVE CHEMICAL INCIDENTS ..........................................................53

## 7.0 ROOT AND CONTRIBUTING CAUSES ........................................................................................56

- 7.1 ROOT CAUSES ................................................................................................................ ............................ 56
- 7.2 CONTRIBUTING CAUSES ........................................................................................................ 57

## 8.0 RECOMMENDATIONS ............................................................................................................ ........59
9.0 REFERENCES .................................................................................................................. 62

APPENDIX A: INCIDENT TIMELINE ..................................................................................... 63

APPENDIX B: DEVELOPMENT TIMELINE .............................................................................. 69

APPENDIX C: ROOT CAUSE ANALYSIS DIAGRAM ............................................................. 70

APPENDIX D: MATERIAL SAFETY DATA SHEETS ................................................................. 71

# TABLE OF FIGURES

<table>
<thead>
<tr>
<th>FIGURE</th>
<th>DESCRIPTION</th>
<th>PAGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>AERIAL VIEW OF THE PATERSON PLANT</td>
<td>11</td>
</tr>
<tr>
<td>2</td>
<td>PLOT PLAN</td>
<td>13</td>
</tr>
<tr>
<td>3</td>
<td>ANNOTATED RENDERING OF A CHEMICAL REACTOR HEAD SIMILAR TO KETTLE SEVEN</td>
<td>15</td>
</tr>
<tr>
<td>4</td>
<td>SIMPLIFIED PROCESS FLOW DIAGRAM</td>
<td>16</td>
</tr>
<tr>
<td>5</td>
<td>TEMPERATURE CHART AND DIGITAL DISPLAY</td>
<td>17</td>
</tr>
<tr>
<td>6</td>
<td>MANWAY HATCH IN PLACE</td>
<td>19</td>
</tr>
<tr>
<td>7</td>
<td>CONDENSER AND VENT PIPING</td>
<td>20</td>
</tr>
<tr>
<td>8</td>
<td>GLASS SECTION OF 8-INCH LINE TO CONDENSER</td>
<td>22</td>
</tr>
<tr>
<td>9</td>
<td>RUPTURE DISK ASSEMBLY RECOVERED FROM K-7 AFTER THE INCIDENT</td>
<td>23</td>
</tr>
<tr>
<td>10</td>
<td>EXTRUDED HEAD GASKET</td>
<td>24</td>
</tr>
<tr>
<td>11</td>
<td>RECOVERED INSPECTION MANWAY COVER</td>
<td>24</td>
</tr>
<tr>
<td>12</td>
<td>POST-ACCIDENT PHOTO OF THE KETTLE</td>
<td>25</td>
</tr>
<tr>
<td>13</td>
<td>BUILDING 11’S BLOWOUT PANELS AND WINDOW FRAMES</td>
<td>28</td>
</tr>
<tr>
<td>14</td>
<td>HEAT GENERATION VERSUS HEAT REMOVAL RATE</td>
<td>31</td>
</tr>
</tbody>
</table>
EXECUTIVE SUMMARY

ES.1 INTRODUCTION

On Wednesday, April 8, 1998, at 8:18 pm, an explosion and fire occurred during the production of Automate Yellow 96 Dye at the Morton International, Inc. (now Rohm & Haas\(^1\)) Plant (the plant) in Paterson, New Jersey. The explosion and fire were the consequence of a runaway reaction, which over-pressured a 2000-gallon capacity chemical reactor vessel (or kettle) and released flammable material that ignited. Because of the serious nature of the incident, including injuries to nine employees, the release of potentially hazardous materials into the community, and extensive damage to the plant, the U.S. Chemical Safety and Hazard Investigation Board (CSB) initiated an incident investigation. The purpose of the investigation was to identify the root causes of the incident and make recommendations to prevent similar incidents.

ES.2 INCIDENT

The Paterson plant manufactures a series of dye products. Yellow 96 Dye was produced by the mixing and reaction of two chemicals, ortho-nitrochlorobenzene (\(o\)-NCB) and 2-ethylhexylamine (2-EHA). The dye was used to tint petroleum fuel products.

The investigation team determined that the reaction accelerated beyond the heat-removal capability of the kettle. The resulting high temperature led to a secondary runaway

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\(^1\) In February 1999, Rohm and Haas Company, of Philadelphia, PA, purchased Morton International. Morton is currently a wholly owned subsidiary of Rohm and Haas.
decomposition reaction causing an explosion, which blew the hatch off the kettle and allowed the release of the kettle contents. The initial runaway reaction was most likely caused by a combination of the following factors: (1) the reaction was started at a temperature higher than normal, (2) the steam used to initiate the reaction was left on for too long, and (3) the use of cooling water to control the reaction rate was not initiated soon enough.

The explosion ejected flammable vapors from the kettle into the second floor of the production building. The explosion and flash fires inside the building injured nine workers. The flashing eruption of chemicals broke through the building roof, ignited and formed a large fireball above the building, and spattered the adjacent neighborhood with a yellow-brown mixture of compounds that included Yellow 96 Dye and o-NCB.

Workers’ injuries included burns, contusions, and twisted joints. Two employees were badly burned and required extended hospitalization. All of the employees were able, some with assistance, to escape the site of the explosion.

**ES.3 KEY FINDINGS**

1. Morton’s initial research and development for the Yellow 96 process identified the existence and described the two exothermic chemical reactions\(^2\) that can occur when the chemicals used to produce Yellow 96 are mixed and heated:

   - The desired exothermic reaction, to form Yellow 96, which is initiated at an onset temperature\(^3\) of 38°C (100°F) and begins to proceed rapidly at a temperature of approximately 75°C (167°F), and

\(^2\) An exothermic reaction, or “exotherm,” generates heat as a by-product of a chemical reaction. Since reactions usually proceed faster at higher temperatures, the heat generated is typically removed from the reacting mixture by a medium such as cooling water, to allow the reaction to proceed at the desired constant temperature.

\(^3\) The onset temperature of a reaction is the lowest temperature at which exothermic activity is observed.
• the undesired, exothermic reaction that results from the thermal decomposition\(^4\) of the Yellow 96 product, which is initiated at an onset temperature 195°C (383°F).

2. The Paterson facility was not aware of the decomposition reaction. The Process Safety Information (PSI) package, which was used at the Paterson plant to design the Yellow 96 production process in 1990 and which served as the basis for a Process Hazard Analysis (PHA) conducted in 1995, noted the desired exothermic reaction, but did not include information on the decomposition reaction.

3. Neither \(o\)-NCB nor 2-EHA, individually, demonstrates exothermic activity at the normal Yellow 96 process temperatures.

4. Two discrepancies in Morton’s Material Safety Data Sheet (MSDS) for the Yellow 96 product were identified:

   • The National Fire Protection Association (NFPA) Reactivity Rating was listed as 0 (on a 0 to 4 scale, with 4 being the most reactive). The CSB determined that the proper reactivity rating for Yellow 96 is 1.

   • The Yellow 96 boiling point was listed as 100°C. The CSB determined that the boiling point was approximately 330°C, or greater than the decomposition temperature.

5. Morton did not follow up on two recommendations made by its researchers in 1989: (1) to conduct additional reactive chemical laboratory tests to determine “the rate of reaction under the worst reaction conditions ... the rate of decomposition of the finished product ... [and] pressure rise data which could be used to size emergency venting equipment ...” and, (2) to install specific control and safety devices on the Yellow 96 production vessels such as an emergency shutdown system.

\(^4\) A thermal decomposition reaction occurs when a chemical breaks up into smaller molecules during exposure to an elevated temperature for a sufficient period of time. The liberation of large amounts of heat and generation of high pressure may accompany decomposition reactions.
6. In scaling up the process from the laboratory phase to production-scale, Morton revised the process from a semi-batch process, in which the \( o \)-NCB was added in incremental stages to the full amount of 2-EHA already in the reactor, to a batch process, in which the entire quantities of the reactants were added to the reactor at the outset, and staged heating was then applied. Morton did not take into account that this change resulted in a potentially more thermally hazardous operation.\(^5\) The heat output from a semi-batch process is easier to control than is that from a batch process. In the batch process, heating the entire mass of reactants at the start of the reaction increases the likelihood of exceeding the heat removal capacity of a kettle.

7. The kettle was not equipped with safety equipment, such as a quench or reactor dump system, to shut down the process in case of a runaway reaction emergency. Also, pressure relief devices (rupture disks) were too small to safely vent the kettle in case of a runaway reaction.

8. The operators controlled process temperature manually by opening and closing steam and cooling water valves. Operating procedures, however, required that the process run at temperatures (150 to 153°C) that were near the temperature at which the reaction could become uncontrollable (195°C). Thus, normal variations in the operators’ responses to the batch temperature or delays in adjusting valves could result in overheating or undercooling, with the result that the heat generated by the reaction would exceed the cooling capacity of the kettle. In several previous instances, the operators documented on the process batch sheets that the process temperature rose at a faster-than-expected rate and exceeded the upper limit specified in the operating procedures in spite of the operators’ efforts. The temperature of these batches eventually returned to the operating limits. Management did not investigate the causes of these events.

9. In 1996, Morton transferred the Yellow 96 process from 1000-gallon to 2000-gallon kettles and increased the amount of raw materials used per batch by approximately 9 percent.

\(^5\) While this change minimized the potential for toxic effects due to employee exposure to \( o \)-NCB, it also resulted in a more thermally hazardous operation.
without using their existing Management of Change procedures. Morton did not assess the effects of these changes. While 20 percent of the 25 batches of Yellow 96 made before these changes exhibited temperature excursions, half of the 6 batches made after the changes had temperature excursions.

10. The Occupational Safety and Health Administration (OSHA) Process Safety Management (PSM) standard did not cover the Yellow 96 process. Morton covered the process under its internal PSM program, which was applied to OSHA-regulated processes and certain other processes. The CSB did not find that lack of OSHA PSM coverage directly contributed to causing the incident. The Yellow 96 process would not have been covered by the Environmental Protection Agency’s (EPA) Risk Management Program rule, which went into effect in June 1999.

11. The OSHA PSM standard covers only a limited number of reactive chemicals. EPA’s Risk Management Program rule does not cover reactive chemicals as a class. These standards provide only minimum requirements for process safety management and provide little guidance on reactive chemical process safety management issues, such as the use of chemical screening techniques, and the proper design of pressure relief, emergency cooling, and safety interlock systems.

12. Morton’s PSM program did not require adherence to a number of industry good practice guidelines for the safe management of reactive chemical processes such as those published by the Center for Chemical Process Safety or Lees’s Loss Prevention in the Process Industries.

13. Morton also did not effectively implement the requirements of its internal PSM program. The PHA conducted for the process and the operating procedures (batch sheets) did not address the consequences of potential deviations such as excessive heating, a runaway reaction, or the inability to provide enough cooling to maintain temperatures in a safe operating range. Batch sheets did not list the actions operators should take to correct or avoid deviations. Operators’ reports of significant deviations in controlling batch temperature were not acted on by management.
**Root Causes**

1. Neither the preliminary hazard assessment conducted by Morton in Paterson during the design phase in 1990 nor the process hazard analysis conducted in 1995 addressed the reactive hazards of the Yellow 96 process.

   Not addressing these hazards resulted in the following design, operational, and training deficiencies:

   - The kettle cooling system could not safely control the exothermic Yellow 96 synthesis reaction.
   - The kettle was not equipped with safety equipment, such as a quench system or a reactor dump system, to stop the process to avoid a runaway reaction.
   - Rupture disks were too small to safely vent high pressure in the kettle in the event of either of the two foreseeable runaway reactions.
   - Morton converted its Yellow 96 production from a staged, incremental addition (semi-batch) process to a staged heating (batch) process without assessing the possible hazards of this change, e.g., the increased difficulty of controlling heat output.
   - Operating procedures did not cover the safety consequences of deviations from normal operating limits, such as runaway reactions, or specify steps to be taken to avoid or recover from such deviations.
   - Training did not address the possibility of a runaway reaction and how operators should respond to avoid injury if a runaway reaction could not be controlled.

2. Process safety information provided to plant operations personnel and the process hazard analysis team did not warn them of the potential for a dangerous runaway chemical reaction.
Morton internal memoranda had documented that the desired reaction to form Yellow 96 from o-NCB and 2-EHA was exothermic and that Yellow 96 would begin to decompose rapidly (runaway) at temperatures close to the upper operating temperature. Morton researchers also had identified several situations, such as loss of cooling, which might give rise to temperatures capable of causing violent decompositions. Although information on the reactive hazards was contained in plant files, operators and supervisors were unaware that a dangerous decomposition reaction was possible.

**Contributing Causes**

1. **The hazards of previous operational deviations were not evaluated.**

   Management did not investigate evidence in numerous completed batch sheets and temperature charts of high temperature excursions beyond the normal operating range. Investigation of these incidents likely would have provided an opportunity to uncover the process’s reactive hazards and correct design and other problems.

2. **Morton did not follow their Management of Change procedures to review changes made in reaction kettle and batch size.**

   Morton changed the Yellow 96 processing equipment from 1000-gallon to 2000-gallon kettles and also increased the batch size by 9 percent in 1996. Morton did not follow its Management of Change procedures and did not review the changes for possible safety consequences. A Management of Change review likely would have provided another opportunity for Morton to uncover the process’s reactive hazards and correct design problems. The review likely would have also revealed that the changes made resulted in a decrease of 10 percent in the heat transfer area per gallon of reactants. Half of the batches made after this change exhibited temperature excursions versus 20 percent in the batches made before the change.
ES.4 RECOMMENDATIONS

Morton International, Inc. (a wholly owned subsidiary of Rohm and Haas Company)

Establish a program that ensures reactive chemical process safety information and operating experience are collected and shared with all relevant units of the company.

Morton International, Inc.’s Paterson, New Jersey Plant

1. Revalidate PHAs for all reactive chemical processes in light of the findings of the CSB report and upgrade, as needed, equipment, operating procedures, and training.

2. Evaluate pressure relief requirements for all reaction vessels using appropriate technology, such as the Design Institute for Emergency Relief Systems (DIERS)\(^6\) method and test apparatus and upgrade equipment as needed.

3. Evaluate the need for and install, as necessary, devices, such as alarms, added safety instrumentation, and quench or reactor dump systems to safely manage reactive chemical process hazards.

4. Revise operating procedures and training for reactive chemical processes as needed, to include descriptions of the possible consequences of deviations from normal operational limits and steps that should be taken to correct these deviations, including emergency response actions.

5. Implement a program to ensure that deviations from normal operational limits for reactive chemical processes that could have resulted in significant incidents are documented and investigated and necessary safety improvements are implemented.

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\(^6\) DIERS is a consortium of companies, formed in 1976 by the American Institute of Chemical Engineers, to develop methods for the design of emergency relief systems to handle runaway reactions. The DIERS group has developed computer programs and a bench-scale test apparatus for assessing the venting requirements of runaway reactions and two-phase flow.
6. Revise the Yellow 96 Material Safety Data Sheet (MSDS) to show the proper boiling point and NFPA reactivity rating. Communicate the MSDS changes to current and past customers (who may retain inventories of these products).

**Occupational Safety and Health Administration (OSHA) and Environmental Protection Agency (EPA)**

1. Issue joint guidelines on good practices for handling reactive chemical process hazards. Ensure that these guidelines, at a minimum, address the following issues:

   - The evaluation of reactive hazards and the consequences of reasonably foreseeable and worst-case deviations from normal operations.
   - The importance of reporting and investigating deviations from normal operations.
   - The determination of proper design for pressure relief capability, emergency cooling, process controls, alarms, and safety interlocks, as well as other good-practice design features for handling reactive substances.
   - The appropriate use of chemical screening techniques such as differential scanning calorimetry.

2. Participate in a hazard investigation of reactive chemical process safety conducted by the CSB. The objectives of the hazard investigation will include:

   - Determine the frequency and severity of reactive chemical incidents.
   - Examine how industry, OSHA, and EPA are currently addressing reactive chemical hazards. Determine the differences, if any, between large/medium/small companies with regard to reactive chemical policies, practices, in-house reactivity research, testing, and process engineering.
   - Analyze the effectiveness of industry and OSHA use of the National Fire Protection Association Reactivity Rating system for process safety management purposes.
• Develop recommendations for reducing the number and severity of reactive chemical incidents.

American Chemistry Council; Center for Chemical Process Safety (CCPS); Paper, Allied-Industrial, Chemical & Energy Workers International Union (PACE); Synthetic Organic Chemical Manufacturer’s Association (SOCMA)

Communicate the findings of this report to your membership.
1.0 INTRODUCTION

1.1 BACKGROUND

On Wednesday, April 8, 1998, at 8:18 pm, nine workers were injured in an explosion and fire at the Morton International, Inc. (now Rohm and Haas Company) plant in Paterson, New Jersey.7 This incident was the result of a runaway chemical reaction during the production of Automate Yellow 96 Dye. Because of the serious nature of the incident, which caused the hospitalization of two employees, the release of chemicals into the community, and damage to the plant, the U.S. Chemical Safety and Hazard Investigation Board (CSB) sent a team to investigate the incident.

Morton International, Inc. is a major salt producer and maker of specialty chemicals for a variety of applications. Morton developed the Automate Yellow 96 Dye product in the 1980s.

7 Morton International, Inc. was purchased in the spring of 1999 as a wholly owned subsidiary of Rohm and Haas Company, which is a publicly held Fortune 500 company.
Combined with other dyes, Automate Yellow 96 produced bright green shades of tinting for fuels. Initial laboratory testing and pilot plant scale-up were conducted at Morton facilities in Hounslow, England in 1986–1989. During this development phase, Morton researchers observed exothermic activity and determined that batch temperature control was key to preventing the reaction from reaching runaway conditions. In 1988 and 1989, the Morton research groups in Paterson, New Jersey, and Ringwood, Illinois, developed a staged heating process for Yellow 96 and reported no temperature control problems during laboratory and pilot-scale work. The first production-scale batch of Yellow 96 was manufactured at Paterson on September 6, 1990.

1.2 INVESTIGATION PROCESS

The CSB received and shared information with the Occupational Safety and Health Administration (OSHA), the Environmental Protection Agency (EPA), and local emergency response organizations. The CSB examined physical evidence from the incident, conducted interviews with Morton personnel, and reviewed relevant documents obtained from Morton.

The CSB was assisted in its initial field investigation work by contractors from the Department of Energy’s Oak Ridge National Laboratory and the National Aeronautics and Space Administration’s (NASA) White Sands Test Facility. Calorimetric testing of the Yellow 96 process and process materials was conducted for the CSB by both the White Sands Test Facility and Chilworth Technology, Inc.

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8 The field phase of the investigation was delayed by approximately 6 weeks due to problems between the CSB and Morton in obtaining proper credentials for the contract investigators. This delay did not affect the CSB’s key findings or conclusions regarding the causes of the Morton incident.
1.3 **PLANT FACILITIES**

The Paterson facility is located in Passaic County, on a seven-acre site surrounded by other industrial establishments and residential homes. On-site support facilities include offices, laboratories, shops, lunchrooms, and locker rooms. The plant is bordered on the west by New Jersey Route 20 (McLean Blvd.) and on the east by the Passaic River (Figure 1).

The incident occurred in Building 11 (B-11), a three-floor building on the east end of the complex. A facility plot plan is provided in Figure 2. The plot plan identifies the location of each of the process buildings, the K-7 chemical reactor vessel used on April 8 to produce Yellow 96, and the hot box, which was used to preheat the ortho-nitrochlorobenzene (o-NCB) for use in the Yellow 96 process.

![Figure 2: Plot Plan](image-url)
Industrial dye products were manufactured at the plant via batch processing. Various raw materials were mixed in reaction vessels (kettles) and heat was applied to drive the reaction process. The resulting dyes were further processed after the reaction steps to remove residual chemicals and waste products. The final product was put into drums or transferred to product storage tanks to be shipped off-site or blended with other products to produce different colors as final products.

1.4 LAYOUT AND COMPONENTS

1.4.1 Kettle Description

From 1990 to 1996, Morton produced Yellow 96 in 1000-gallon kettles. In September 1996, Morton switched production to 2000-gallon kettles to minimize color contamination between different dyes produced in the same kettles. Kettle number seven (K-7), which was involved in the incident, was one of the 2000-gallon kettles.

Kettle K-7 was designed and manufactured in 1962 (Figure 3 is a drawing of a kettle similar to K-7). The interior of the kettle was glass lined to prevent corrosion of the carbon steel shell and heads. The inside diameter of the kettle was 78 inches and the height was about 107 inches. The kettle heads were dished, with nominal thicknesses of 7/8 inch and 15/16 inch for the top and bottom heads, respectively. The nominal thickness of the shell was 15/16 inch. A heating and cooling jacket, or outer case, extended from the top head-to-shell weld downward to the middle of the bottom head and surrounded the outside of the kettle. The kettle had a Maximum Allowable Working Pressure9 (MAWP) of 100 psig. The MAWP for the jacket was 90 psig. Kettle rupture disks were set at 10 psig.10

9 The Maximum Allowable Working Pressure (MAWP) is the maximum pressure at which a vessel is designed to operate safely. Typically, vessels are operated at pressures significantly below the MAWP to provide a margin of safety.
10 Rupture disks are emergency safety devices designed to open to relieve high pressure in vessels and protect equipment and personnel from catastrophic failure of the vessel.
Nozzles were located in the jacket and on the top head of the kettle to provide piping connections. The nozzle at the bottom of the vessel served as a drain. The most pronounced feature of the top head was a 30-inch-diameter bolted closure. A glass-coated, single-speed agitator was attached to the top surface of the bolted closure. The bolted closure contained a 14- by 18-inch elliptical manway secured by four C-type clamps.

The CSB determined that the kettle and jacket design, fabrication, inspection, hydro testing, and registration stamping met the requirements of the American Society of Mechanical Engineers, Section VIII, Boiler and Pressure Vessel Code.

1.4.2 Kettle System

The entire K-7 process system, with the exception of the thermal oxidizer and pressure-relief catch tank, was located inside Building 11. The kettle extended vertically from the ground level to the second floor. The operators had access to the kettle’s top head and associated nozzles on the second floor where the raw materials were added. Valve handles and temperature readout and recording instruments were also located on the second floor.

Figure 3: Annotated Rendering of a Chemical Reactor Head Similar to Kettle Seven

Besides the agitator bolted closure, the chemical reactor’s head had the following:

(A) a 14- x 18-inch elliptical inspection manway,
(B) a penetration for the thermocouple,
(C) an 8-inch nozzle to the rupture disks and the 10-inch pressure manifold leading to the catch-all tank,
(D) a connection for the liquid condensate return, from the liquid separator,
(E) an acid feed line into the kettle,
(F) a direct kettle vent line to the B-11 manifold that collected organic vapors and delivered them to the plant’s thermal oxidizer,
(G) an 8-inch line to the condenser on the third floor of B-11. A raw material feed nozzle is located behind the 15-hp. electric agitator motor.

(http://www.pfaudler.com/gle/vessels.html)
Each valve manually from the second floor. The third floor housed the condenser and exhaust piping leading outside to the thermal oxidizer and pressure-relief catch tank.

Temperature in the vessel was controlled by adjusting the flow of cooling water and steam piped to the kettle’s jacket with manually operated valves. The operators determined the degree to which these valves were opened based on their experience in running the process. The timing of switching from heating to cooling or vice versa was also based on the operators’ experience. The cooling-water inlet was located at the bottom and the steam inlet was located at the top of the jacket. The water outlet was shared with the steam outlet at the top of the jacket. Steam that condensed during heating operations was drained from the bottom of the jacket and directed to the boiler condensate return. Figure 4 is a simplified flow diagram of the K-7 reactor system.

Figure 4: Simplified Process Flow Diagram
Reactor instrumentation provided measurements of reactor temperature, reactor pressure, and cooling water pressure. Reactor temperature was measured by one thermocouple\textsuperscript{11} connected to two temperature readouts: a digital display and a circular recording chart. The recording chart could not record temperatures greater than 150°C, although the digital readout continued to display temperatures above 150°C (see Figure 5). The kettle was not equipped with temperature or pressure alarms and there were no automatic shutdown devices.

\textsuperscript{11} A thermocouple measures temperature. A thermocouple is composed of two dissimilar metals fused together; the junction between the two metals generates a voltage that is a function of temperature.
2.0 DESCRIPTION OF THE INCIDENT

2.1 INCIDENT EVENTS\textsuperscript{12}

On April 8, 1998, the second work shift began at 4:00 pm. During this shift, Yellow 96 was scheduled to be prepared in K-7, a 2000-gallon jacketed kettle located at the east end of Building 11 (B-11). Plant batches of Yellow 96 had been numbered sequentially from the beginning of production in 1990 and this was to be batch #32. The supervisor assigned two chemical operators to the production and provided them with the batch sheets for Yellow 96.

Operators used batch sheets for step-by-step direction in performing the process. The batch sheets were written and approved by plant management, and supervision and operations personnel. The Yellow 96 batch sheet was nine pages long and had abbreviated safety data sheets (listing only the key health hazards and protective equipment) for the raw materials and products as well as a list of raw materials.\textsuperscript{13} The batch sheets outlined the procedures for each step in the process. Operators were required to record the time when each step began and ended, the temperature of the kettle contents, and their initials. Each batch was assigned a sequential lot number. The lead operator performing the process controlled the batch sheets throughout the production of Yellow 96. Upon completion of the batch, the operator returned the sheets to the supervisor along with the temperature recording charts generated during the batch.

Both operators involved in the incident had made Yellow 96 before. The lead operator had 31 years of plant experience and the assistant operator had more than 3 years of experience.

Before beginning batch 32, the operators inspected the kettle through the manway to ensure that it was clean. The lead operator recalled that the temperature of the kettle was approximately 20 to 30°C (68 to 86°F) during and after the inspection. Once the inspection was completed, the

\textsuperscript{12} This narrative can be followed on the Incident Timeline in Appendix A. It describes the actions taken by the operators working on the second floor operating deck of B-11 and the subsequent emergency response activities.

\textsuperscript{13} Complete Material Safety Data Sheets for Yellow 96, o-NCB, and 2-EHA may be found in Appendix C.
manway cover was closed and clamped onto the kettle using four C-type clamps (see Figure 6).

The first processing step was the addition of ortho-nitrochlorobenzene (o-NCB) to the kettle. Because o-NCB has a melting point of 32°C (89°F), the drums required for a batch were placed in a hot box for several days to be melted before use. The hot box was an enclosed room, with a steam heater, large enough to contain the drums (the hot box is shown in Figure 2, to the west of Building 11).

The operators prepared to transfer the o-NCB from the drums by ensuring that all kettle valves were closed, except for the vacuum valve, and the agitator was started. A vacuum was used to draw the o-NCB from the supply drum to the kettle through a combination of piping and flexible hose. This operation was repeated until all drums were empty. The transfer of the o-NCB was normal, taking approximately 30 minutes (until 5:15 pm) indicating that the vacuum was adequate and the o-NCB was well liquefied. Once the transfer of the o-NCB was completed, the temperature of the o-NCB inside the kettle was reported by the lead operator to be “60-something degrees, almost 70°C” (158°F), while the second operator recalled that it was “about 44°C.” The U.S. Chemical Safety and Hazard Investigation Board (CSB) determined through interviews that the operators did not adjust the initial mix temperature to between 40 to 42°C (104 to 108°F) as required by the batch sheet. This temperature-reduction step would have reduced the initial rate of the reaction to produce Yellow 96.

The next processing step was the addition of the 2-ethylhexylamine (2-EHA). The operators readied the kettle by ensuring the vacuum valves and drain isolation valves to the kettle were closed. They opened the kettle vent to ensure there was no buildup of pressure when the 2-EHA

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**Figure 6: Manway Hatch in Place**

As seen in this photograph of a similar kettle, four clamps hold the inspection manway hatch in place.
was added. The operators left this vent valve open for the duration of the operation, allowing the process to be performed at atmospheric pressure (see Figure 7).

Next, the operators added 2-EHA to the kettle. The 2-EHA was pumped into the kettle from an underground storage tank through a meter that stopped the flow when the preset amount of 2-EHA had been pumped. The kettle agitator continued to operate during the 2-EHA addition. The transfer took approximately 25 minutes. After adding the 2-EHA, the operators left the building for dinner, returning at approximately 7:35 pm.

After the addition of the 2-EHA, which was at ambient temperature, the operators stated that the temperature of the mixture was approximately 44 to 48°C (111 to 118°F). Since the onset temperature for the reaction was 38°C, it is likely that the reaction to produce Yellow 96 was already occurring at a very low rate at this time.

2-EHA is a combustible material, with a flash point\(^{14}\) of 52°C (126°F). \(o\)-NCB has a flash point of 127°C (261°F). Thus, both substances were above their flash points during a portion of the normal process and during the runaway incident.

The staged heating procedure used by Morton to produce Yellow 96 started with an initial heatup of the reaction mixture to 90°C, with gradual increases thereafter to 150 to 153°C. With

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\(^{14}\) Flash point is the minimum temperature at which a liquid gives off sufficient vapor to form an ignitable mixture with air near the surface of the liquid.
both the \(o\)-NCB and the 2-EHA now in the kettle, the operators slowly raised the temperature to initiate the reaction by opening and closing valves to allow steam to enter the kettle jacket in short bursts, typically lasting several minutes. This processing step was designed to raise the temperature slowly. The expected reaction time for a batch was 6 to 8 hours.

At approximately 7:40 pm, the lead operator began to raise the temperature of the mixture by introducing steam to the kettle’s jacket. The operating procedures stated that the heating process was necessary to start the exothermic reaction used to produce the dye. To introduce steam to the kettle’s jacket, the operator opened the kettle jacket’s steam-outlet and steam-inlet valves and watched for steam to appear at the steam blow-off line. The steam and cooling-water valves were grouped on opposite sides of the kettle and were operated by hand-wheels on extension rods to the valve bodies below the operating deck. Once the jacket was filled with steam, the steam-outlet valve was closed. The lead operator applied steam to the kettle for about 10 minutes, stopping when the jacket’s pressure gauge read 5 to 10 psig. He repeated this step two more times, watching the digital temperature readout. As the temperature rose, he noticed that the rate of the kettle’s temperature rise was unusually fast. He recalled that the temperature of the batch rose quickly from 70 to 80°C (158 to 176°F) and that the tenths decimal reading on the digital temperature indicator was “moving very fast.” This rapid temperature rise began within the first 15 minutes after the start of heating. The typical heat-up rate during this phase of the process was 1–2 degrees per minute.

At approximately 8:05 pm, with the temperature at 100°C (212°F) and rising rapidly, the operators switched from heating the reactor to cooling by closing the jacket steam valves and opening the cooling water valves.

Three other experienced operators became aware of the problem and came to assist. They inquired if the cooling water was on, and at least one of them confirmed the valve positions. The lead operator re-verified the positioning of the valves to ensure that the cooling-water inlet and outlet valves were full open and that the steam-inlet and steam-condensate valves were closed. The operators stated in interviews that they had heard the sound of the cooling water flowing through the piping, another indicator they used to ensure cooling water flow. The CSB
confirmed that the valves had been working properly.

Approximately 2 minutes passed. The operators called the supervisor to assist and he also verified that all valves were configured properly. The temperature chart recorder, which had a maximum reading of 150°C, was now off scale and of no use to the operators, but the digital gauge showed that the temperature continued to rise. The kettle began to vibrate and very turbulent vapor appeared in the glass section of pipe above the kettle leading to the condenser (Figure 8). This vapor was composed of 2-EHA and gases generated by the decomposition reaction.

The kettle’s emergency relief system, consisting of two 6-inch rupture disks set to open at 10 psig, activated. The operators estimated that the temperature at the time the rupture disks vented was about 180 to 190°C (356 to 374°F). Figure 9 shows the recovered dual rupture disk assembly. The rupture disks were installed on the top of the kettle in a 6-inch diameter line that vented into a 10-inch diameter manifold. This manifold dumped the vapor and liquid aerosol flow into a catch tank outside Building 11. A total of 100 gallons of various chemicals were recovered from this tank after the incident. However, without information on
the contents of the tank before the incident, the CSB could not determine which materials were vented during the event.

The batch temperature continued to increase rapidly, passing the Yellow 96 decomposition onset temperature of 195°C (383°F). The kettle began to rumble and shake more violently. One operator noticed that material was being blown up from the kettle and into the condensate return separator, indicating that the boiling of 2-EHA and the generation of gases from the reactions had exceeded the capacity of the condenser. The condenser’s tubes were flooded with liquid, and the system was trying to relieve its pressure by backward flow up the condensate return line and through the condenser vent.

At the point of the disk failure, an operator who was standing near the kettle reported that he saw the digital temperature reading increase from 190 to 260°C in less than 30 seconds. He then shouted to several other operators and started to run towards an exit. As he reached the top of the stairs located in the middle of the second floor, he heard additional sounds, including a gush of air, which he associated with the failure of the glass condensate return separator assembly. At ground level, he shouted a warning to three more workers who were unaware of the danger. They all ran toward an exit.
At almost the same time, the operator and supervisor still at the kettle reported in interviews that they observed the temperature on the digital temperature readout at about 200°C (392°F). They remained unaware that the batch temperature was above the decomposition temperature of the Yellow 96 product. At this point, the operator and supervisor ran toward the second-floor northeast exit. The reaction in the kettle was vibrating the second-floor steel decking and there was a very loud rumbling, which other workers on the site thought was the sound of a train passing.

At 8:18 pm, the pressure in the kettle blew the inspection manway off the vessel. The manway was found 15 feet from the kettle, and the heat and pressure extruded the head gasket of the vessel through the bolted connection of the 30-inch bolted closure with the top head of the kettle (Figures 10 and 11).

From the open vessel manway, a jet of hot reactants erupted, emptying the vessel. The ejected material penetrated the third floor and the roof. An aerosol mixture of gas and liquid shot above the roof and spattered the adjacent neighborhood with a yellow-brown mixture of compounds including Yellow 96 and

![Figure 10: Extruded Head Gasket](image1.jpg)

This was a result of the heat and pressure that developed during the explosions.

![Figure 11: Recovered Inspection Manway Cover](image2.jpg)

This photo shows the scars where the clamp tore away.
\( o\)-NCB. This aerosol plume ignited forming a large fireball above the roof. The recoil from the expanding gases of the explosion twisted the kettle off its mounts. The kettle dropped 4 feet onto the ground level below and was found leaning against a cinder block wall. Figure 12 shows the final resting position of the vessel after the blasts and fire.

As the operator and supervisor reached the second-floor exit landing, the explosion blew them to the mid-level landing and flash fires spread throughout the building. The building’s wall panels and windows were blown out by the blast, absorbing much of the explosion’s energy and preventing greater damage. The lead operator and the supervisor were blown off the mid-level stair landing onto to the ground. They suffered second- and third-degree burns, requiring their hospitalization in intensive care for 5 days. Injuries to the other workers included first-, second-, and third-degree burns, contusions, abrasions, lacerations, and muscle strains.
2.2 EMERGENCY RESPONSE

The Paterson Fire Department (PFD) arrived at 8:24 pm. The Paterson fire chief assumed command of the fire-fighting effort and called for the Hazardous Materials (HazMat) team. Fire was visible burning through the roof of the building. Before the PFD began their attack, plant personnel assembled the necessary information on the location and materials involved in the fire and the Paterson fire chief determined whether the presence of water-reactive chemicals precluded the use of water to fight the fire.

Once determined safe, water was applied to the fire. The flames were suppressed in about 1 hour, but the PFD continued the water deluge to facilitate the HazMat Team’s entry and initial survey at 9:44 pm. The HazMat Team conducted a primary and secondary search throughout the building, which indicated that all workers had escaped. The PFD team then entered the east end of the building on the second floor with a portable dry chemical fire-extinguishing unit to extinguish the remaining fires. The firewater was stopped as soon as possible to prevent washing contaminants into the Passaic River, which borders the plant’s eastern side. The fire was reported under control at 11:27 pm. The K-7 fire-suppression system, located over the top of the kettle, and the B-11 fixed fire-water deluge system were damaged by the explosion and did not aid fire suppression.

Morton collected on-site approximately 310,000 gallons of contaminated water from fire-fighting operations and next-day rain. Less than 10,000 gallons of contaminated firewater/stormwater reached the Passaic River, according to an estimate by Morton.15

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2.3 Off-Site Impacts

During the fire, the ambient temperature was about 40°F. It was a clear night, with light winds mostly from the northeast, switching to the southeast. These winds blew the plumes of reactants, products, and smoke off the plant site. The fallout was mainly to the west of the plant. Spots were reported on cars in the neighborhood, at an adjacent candy factory, and at a car dealership one-half mile away.

Nearby residents were ordered to shelter in-place during and immediately after the fire-fighting attacks. The shelter-in-place was conducted by the Paterson Police Department and encompassed a 10-block by 10-block area around the plant. The shelter-in-place lasted for approximately 2 to 3 hours.

During and following the fire, air monitoring was performed by various organizations, including the Passaic County Department of Health, Paterson Office of Emergency Management, the Environmental Protection Agency, and Morton. Tests conducted during the fire by the Passaic County Department of Health were negative for benzene, halogenated hydrocarbons, and nitrous compounds.

Workers at two neighboring businesses and fire fighters reported throat irritation and a slight burning in their eyes and on their skin. Several odor complaints were also received from community members.

Approximately 20 hours after the incident, a health warning statement, prepared by the Passaic County Department of Health and issued jointly with the Paterson Mayor’s office, was distributed to the local community. It advised residents to avoid contact with the deposited material, listed steps to be taken in case of health effects, and instructed residents on how to handle contaminated items.

Wipe samples were taken of visible material deposited on automobiles and buildings near the Morton facility. These samples contained measurable quantities of Yellow 96 Dye and o-NCB.
2.4 PROPERTY DAMAGE

The open flooring in B-11, in conjunction with the blowout walls and windows, minimized the fire and blast damage to the load-bearing structure of the building.

The blast blew out the windows, doors, and blowout walls of B-11 (Figure 13). While there was some blast damage in the immediate vicinity of this reactor vessel, most of the damage was caused by the ensuing fire. The second blast was outside, above the roof.

Structural consultants subsequently determined that the load-bearing columns were not severely damaged. Overall, the building was still structurally sound and some of the equipment was salvageable.
3.0 ANALYSIS OF PROCESS CHEMISTRY

3.1 REACTIVE CHEMICAL ANALYSIS

The runaway chemical reaction incident in the Yellow 96 process involved two reactions: (1) the desired synthesis reaction to form the product, and (2) the undesired decomposition reaction of the product.

The heat produced by the exothermic reaction that formed the product exceeded the heat-removal capacity of the kettle and rapidly raised the temperature of the batch. That heat generation caused the vapor pressure of the 2-ethylhexylamine (2-EHA) to rise until it boiled and the reaction released additional gases, pressurizing the vessel. The heat released also raised the temperature of the batch above the decomposition temperature of Yellow 96, at which point the gaseous products of the decomposition reaction resulted in even greater pressure, culminating in the explosion and the final release of material from the kettle.

Chilworth Technology, Inc., of Monmouth Junction, New Jersey, was hired by the U.S. Chemical Safety and Hazard Investigation Board (CSB) to conduct a battery of calorimetric tests analyzing the Yellow 96 synthesis and decomposition reactions. Two exothermic reactions were observed: Reaction 1, the desired reaction of 2-EHA with ortho-nitrochlorobenzene (o-NCB) to form Yellow 96 had an onset temperature ($T_o$) of 38°C. Reaction 2, the decomposition reaction of the Yellow 96 had an onset temperature of 195°C. (The onset temperature is the lowest temperature at which exothermic activity is observed.) Chilworth also determined that the onset temperatures for the generation of permanent gases were 139°C for the desired synthesis reaction and 201°C for the decomposition reaction.16

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The heat released during the synthesis reaction raised the temperature of the reaction mixture. The rate of the temperature rise depended on the balance between the reaction rate, the heat capacity of the chemicals, and the rate of heat removal by the reactor’s cooling water jacket. To hold a constant temperature in the kettle after the synthesis reaction had begun, the operators manually adjusted the flow of cooling water through the water jacket, trying to balance the heat removal rate with the heat generation rate. Without any indicators for water-flow rate or for cooling-water inlet and outlet temperatures, the operators controlled the kettle’s temperature based on in-house training and experience. The operators also had to take into consideration a time lag in the temperature response of the reaction mixture to the manual adjustments in the cooling-water flow.

The released heat not only raised the batch temperature but also accelerated the reaction rate, making the temperature rise more difficult to control. The accelerating reaction rate made it more likely that the heat generated by the reaction would exceed the rate of heat removal for which the kettle was designed, resulting in a runaway, uncontrollable condition. The runaway condition occurs because the rate at which heat is removed through the jacket increases linearly with increasing temperature, while the rate at which heat is generated by the reaction increases exponentially (see Figure 14).

The reaction rate is determined both by the volumetric concentrations of the two reactants and the temperature. The rate of a reaction increases exponentially with temperature. At the beginning of the reaction, the reactants are at their maximum concentrations. As the reaction proceeds to completion, the starting materials are depleted and the reaction slows down. Therefore, at the start of the batch, with a high inventory of reactants, the influence of temperature on the heat generation rate is greatest and there is the most danger of overrunning the heat removal capacity of the kettle. As will be discussed in Section 4.0, Analysis of Process Development Program, use of a semi-batch process instead of a batch process, could have provided the operators with more control over the reaction.

The operating procedures for Yellow 96 stated that the operators should heat the reactant mixture to 90°C to initiate the reaction; however, in the operators’ experience, following the
operating procedures resulted in a temperature increase that was difficult to control. The operators believed that the reaction between 2-EHA and o-NCB started as early as 75°C (167°F) and so most operators turned the steam heating off when the reaction temperature reached approximately 70 to 80°C. The CSB confirmed the operators’ observations: calorimetric testing determined that the onset temperature for the Yellow 96 synthesis reaction was 38°C. Therefore, the reaction would proceed slowly even at temperatures lower than was thought by the operators.

The operating procedure required that the operators should begin the heating process from a temperature of 40 to 42°C (104 to 108°F), but the operators usually started heating the initial mixture at whatever the batch temperature was after the addition of the 2-EHA to the o-NCB. The review of completed batch sheets as well as interviews showed that the starting temperature was greater than 40 to 42°C (104 to 108°F) in a number of cases, including batch 32 (as determined in interviews). Since the reaction began at 38°C, starting from a lower temperature and a correspondingly lower initial reaction rate would have given the operators more time to react and possibly control the rising temperature before the heat generation rate overran K-7’s

![Figure 14: Heat Generation versus Heat Removal Rate](image)

If the reaction temperature exceeds point A, the rate of heat generation exceeds the rate of heat removal and the reaction will run away.

(HSE, Designing and Operating Safe Chemical Reaction Processes, page 10)
cooling capacities. The operators had been able to do just that during previous batches that exhibited temperature excursions (see Section 5.3).

If the cooling water is incapable of removing all of the heat generated by the reaction, the temperature rises and accelerates the reaction rate. In batch 32, the accelerating synthesis reaction continued to generate heat, raising the temperature. As a result of this heating, the residual 2-EHA in the kettle began to vaporize rapidly as was observed in the 8-inch glass line between the kettle and the condenser by the operators.

In some batch operations evaporation and condensation (refluxing) can be an effective heat-removal technique. Heat generated by the reaction is used to boil a solvent or other materials in the reactor. This vaporized material is then cooled and converted back to liquid in a condenser. The heat is thus transferred to the water used to cool the condenser and removed from the process. The Yellow 96 researchers in the United Kingdom considered using evaporation and condensation as a heat-removal mechanism but it was not considered in the Paterson plant design. Vaporization and condensation, however, did occur in batch 32, but the vaporization proceeded at too fast a rate for all the materials to condense, while additional noncondensable gases were also being generated. Operators had seen vigorous refluxing during the reaction in only one previous batch (batch 31), a high-temperature excursion beyond the normal operating limit.

As the reaction temperature approached 195°C (383°F), the undesired exothermic decomposition reaction of Yellow 96 began producing more heat and noncondensable gases, which further pressurized the vessel. The pressure in the vessel rose higher than the rupture disk pressure setting (10 psig). At about 180 to 190°C (356 to 374°F), the rupture disks vented, allowing material to escape to the catch tank and to the atmosphere. However, the venting area available through the rupture disks was insufficient to relieve the pressure being generated by the reaction, so the pressure in the kettle continued to rise. The 8-inch glass line to the condenser (see Figure 9) and the glass 2.5-inch condensate return line shattered, also venting reactants.
The pressure resulting from the Yellow 96 decomposition continued to increase rapidly and exceeded the kettle’s ability to relieve through the several now-opened lines. This pressurization eventually caused the manway inspection hatch to be blown off. The expanding vapors and the decomposition gases propelled the reaction mixture through the third floor and roof as a two-phase flow of vapors and liquid.\textsuperscript{17}

\section*{3.2 Tests of Raw Materials}

NASA’s White Sands Test Facility\textsuperscript{18} and Chilworth\textsuperscript{19} conducted differential thermal analysis\textsuperscript{20} tests for the CSB on the two raw materials. No exothermic reactions or pressure spikes were detected for the individual reactants until temperatures were above the maximum Yellow 96 operating temperature (160°C). After the incident, the Occupational Safety and Health Administration (OSHA), the Environmental Protection Agency (EPA), and Morton analyzed the unused raw materials and determined that the reactants used were within the expected quality range. These tests confirmed that the runaway reaction was not the result of contamination from the raw materials.

\textsuperscript{17} An analogy for the eruption of two-phase flow from the kettle is the release of gas, liquid, and foam observed when a container of carbonated beverage is shaken and then opened.


\textsuperscript{20} Differential Thermal Analysis (DTA) is a preliminary screening test, similar to the Differential Scanning Calorimetry (DSC) performed by Morton in 1987. DTA is used to determine whether an exotherm occurs, the exothermic onset temperature, and the amount of heat generated. The DTA test used small samples, e.g., several milligrams. The sample is heated at a controlled rate. The onset temperature, \( T_o \), is the temperature at which the sample first shows an observable thermal response due to reaction or decomposition.
4.0 ANALYSIS OF PROCESS DEVELOPMENT PROGRAM

The Yellow 96 process went through several research phases at different branches of Morton. (The Timeline in Appendix B summarizes important dates in the development and production of Yellow 96.) The company began its research with small-scale reactions in 1986 at their Hounslow, England, facility. Larger experimental batches of Yellow 96 were produced in 1987 and 1988. Morton researchers observed exothermic activity in these tests. Morton contracted Brunel University in the United Kingdom to perform differential scanning calorimetry (a typical screening test for exothermic activity) on the reaction in 1987. In these tests, “the material was found to decompose with considerable generation of heat at above 220°C.” Morton developed a semi-batch, staged addition process to control the exothermic reaction rate in these early trials. The researchers added the ortho-nitrochlorobenzene (o-NCB) to the 2-ethylhexylamine (2-EHA) in four equal portions and allowed the reaction to run its course and subside between o-NCB additions.

In 1989, the researchers at Hounslow wrote a review of the process in which they made a number of recommendations regarding the reaction control and safety. This review was faxed to the Paterson plant on April 28, 1989. Among other things, the researchers recommended controlled cooling-water addition directly into the reactor as an emergency method to stop a runaway reaction.

This process review memo also included the recommendation that accelerating rate calorimetry testing be performed to determine “the rate of reaction under the worst reaction conditions ... the rate of decomposition of the finished product ... [and] pressure rise data which could be

22 During much of the development work, Morton referred to the Yellow 96 material as “Liquid Yellow 152” or “Sudan Yellow 152.”
used to size emergency venting equipment ....”

Morton did not perform these additional tests or install the recommended safety equipment.

In late 1989, Morton transferred its research effort on Yellow 96 to the United States. Initial research was conducted at the Paterson plant. Paterson revised the process from semi-batch to a full-batch process in order to minimize employee exposure to o-NCB. In the batch process, the full amounts of o-NCB and 2-EHA were added to the reactor first, and heat was then applied to initiate the reaction and drive it to completion. This procedure, with one total addition of o-NCB versus the four partial additions in the semi-batch process, was used throughout Yellow 96 production at Paterson. Morton did not take into account that switching from their initial, semi-batch process to the full-batch process resulted in a more hazardous condition.

In *Inherently Safer Chemical Processes*, the Center for Chemical Process Safety notes: “Semi-batch or gradual addition batch processes [Morton’s initial process design] limit the supply of one or more reactants, and increase safety when compared to batch processes in which all reactants are included in the initial batch charges [Morton’s production process]. For an exothermic reaction, the total energy of reaction available in the reactor at any time is minimized.”

The U.K. Health and Safety Executive (HSE) writes in a recent publication that semi-batch processes “reduce the quantity of reactant present and controlling the addition rate may stop the reaction in the event of a hazard arising.” At the start of the batch process, when the reactant concentrations were at their maximum levels, the influence of temperature on the rate of heat generation was greatest, and there was the most danger of exceeding the heat removal capacity of the kettles.

Paterson’s research did not specifically test for the exothermic reactions and their lab-scale work did not reveal the runaway potential of these reactions. After receiving additional

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documentation from Hounslow alerting them to the possibility of a runaway exothermic reaction, including the process review memo noted above, Paterson researchers performed further experiments and determined that they could adjust the temperatures of the reaction to “reduce the potential for overheating the mixture.”

Morton also conducted research at its Ringwood, Illinois, semiworks facility, producing six trial batches of material, in sizes of 80 to 425 gallons. In these pilot-scale batches, Morton was able to control the exothermic reaction within operating limits.

Paterson staff used a “Premanufacturing Process Review Checklist” as a guide for bringing Yellow 96 to full-scale production in 1990. The Yellow 96 checklist contained an information package (process transmittal) received from the Ringwood semiworks facility. The information included batch sheets from the Ringwood batches, material safety data sheets (MSDSs) for the materials used in the process, and several memos and notes relating to the process. The memos noted the presence of an exothermic reaction but stated that the process could be controlled by correctly following the operating steps.

Morton did not conduct a preliminary hazard assessment when they brought Yellow 96 to production in 1990. Preliminary hazard assessment and Process Hazard Analysis techniques were in use throughout the chemical process industries at this time and had been gathered and published by the Center for Chemical Process Safety in 1989, 3 years before OSHA’s promulgation of the Process Safety Management rule.27,28

The observations by the Morton researchers of the several laboratory and pilot-scale batches performed in the United States were the determining factor in Paterson’s analysis of the safety of the process. To design a safe reaction process, certain basic information is required. The

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empirical ability to control the process in laboratory and pilot plant should have been augmented by additional key engineering work, such as:

- Conducting additional calorimetric testing when the process was changed from staged addition of o-NCB to staged heating of the entire reaction mass.

- Calculating a heat and mass balance to determine if there was sufficient heat removal capability in the reactors to handle foreseeable events and to determine the influence of reactor vessel size on this function.

- Determining the worst case venting scenario and sizing the reactor’s safety relief devices accordingly. The pressure relief devices on the Paterson reactors had been sized for the scenario of an external fire that would boil xylene in the kettle. Morton did not run calculations to determine if the relief devices could safely vent the pressure generated by a runaway exothermic reaction or if the reaction vessels were strong enough to safely contain the maximum expected pressure.
5.0 ANALYSIS OF PROCESS SAFETY MANAGEMENT PROGRAM

Morton’s Process Safety Management (PSM) program did not require adherence to a number of industry good practice guidelines for the safe management of reactive chemical processes. Morton also did not effectively implement important requirements of their internal PSM program. Deviations in the following areas were important factors leading to the accident:

- Process Safety Information
- Process Hazard Analysis
- Investigation of Problems with Previous Batches
- Management of Change
- Operating Procedures and Training

5.1 PROCESS SAFETY INFORMATION

Paterson facility personnel were not aware of the existence of the decomposition reaction. The Process Safety Information (PSI) package, which was used by the Paterson personnel to design the Yellow 96 production process in 1990 and which served as the basis for a Process Hazard Analysis (PHA) conducted in 1995, noted the desired exothermic synthesis reaction, but did not include information on the undesired exothermic decomposition reaction.

The Yellow 96 process safety information package did not contain details of the research on the exothermic reactions performed at Hounslow nor did it contain the recommendations made by Hounslow for process safety, control, and additional testing in the “LY 152 Process Review” memo, which had been faxed to Paterson on April 28, 1989. Inclusion of this information in the process safety information package was a requirement of Morton’s PSM program.
Morton’s Material Safety Data Sheet (MSDS) for Yellow 96 Dye stated that the National Fire Protection Association (NFPA) Reactivity Hazard Rating\textsuperscript{29} for this material was a 0 (on a 0 to 4 scale, with 4 being the most reactive). The U.S. Chemical Safety and Hazard Investigation Board (CSB) has determined that the proper reactivity rating for Yellow 96 Dye is 1.\textsuperscript{30,31} The reactivity rating is a ranking of the “degree of susceptibility of materials to energy release. Some materials are capable of rapid release of energy by themselves, through self-reaction or polymerization, or can undergo violent explosive reaction through contact with water or other extinguishing agents or with certain other materials.”\textsuperscript{32} The NFPA defines 0 materials as “materials that in themselves are normally stable, even under fire conditions,” while 1 materials are “normally stable, but ... can become unstable at elevated temperatures and pressures.”\textsuperscript{33} The NFPA ratings are used by emergency responders, employees, and customers, as an indicator for the degree of hazard associated with a chemical. Erroneous information regarding reactive hazards can result in errors in handling or responding to emergencies. In this incident, the difference did not affect the emergency response activities.

The Yellow 96 Dye MSDS also stated that the compound’s boiling point was 100°C. The CSB determined that the boiling point is actually much higher, approximately, 330°C, which is greater than the decomposition onset temperature of 195°C. While not contributing to causing the incident, the foregoing MSDS discrepancies are examples of shortcomings in Morton’s Process Safety Information package.

\textsuperscript{29} NFPA704, “Standard System for the Identification of the Hazards of Materials for Emergency Response,” 1996 edition, pages 704–707. The NFPA hazard rating system is intended to provide basic information for fire fighters and emergency responders. In addition to reactivity, the NFPA system rates fire and health hazards on the same 0 to 4 scale.
\textsuperscript{31} Based on determination of the Instantaneous Power Density (IPD) of Yellow 96 per the method of NFPA 704, 1996 edition.
\textsuperscript{33} Ibid., pages 704–708.
5.2 PROCESS HAZARD ANALYSIS

Process hazard analysis (PHA) is a structured, in-depth examination of a chemical plant process. The objectives of a PHA are to:

- identify the factors in the process that have the potential to cause undesired events (process hazards);
- uncover the foreseeable sequences of events (scenarios) that could lead to the realization of these hazards;
- evaluate the consequences of these scenarios should they occur;
- propose improvements, as warranted, to equipment and procedures that either reduce or eliminate the hazards, prevent the scenarios from occurring or mitigate their consequences.

The PHA conducted for the Yellow 96 process did not address the consequences of potential deviations such as excessive heating, a runaway reaction, or the inability to provide enough cooling to maintain temperatures in a safe operating range.

The formal PHA for Yellow 96 was conducted in January 1995, approximately 4 years after the first batch was produced at Paterson. The PHA was performed using the “What If?” method of analysis. The PHA team analyzed the raw material storage tanks; the weigh tanks; the kettle vessel/agitation/instrumentation; the kettle condensers, receivers, piping, valves, and pumps; the elevator and pallet jack; the water scrubber; the dye coupling tanks; and the various site support systems. The PHA did not specifically address the processing of Yellow 96 in K-7 but generally covered all of the reactor kettles, both 1000- and 2000-gallon capacity.

As noted earlier, the Paterson Plant received a number of memos from the Ringwood facility that indicated the potential for a thermal runaway in the Yellow 96 process. Ringwood stated, “If the batch [ortho-nitrochlorobenzene (o-NCB) charge to the kettle] is left to warm, then it

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will exotherm strongly upon the addition of the 2-ethylhexylamine.”

In another memo, referring to the first 100-gallon, pilot-scale batch, Ringwood noted, “The concern about exothermic conditions as expressed by... [the Morton Hounslow facility] had been seen previously during the initial R&D [research and development] work. This can easily be handled by starting the reaction at ambient temperature and proceed with staged heat-up with holds at predetermined temperature ranges.”

When the question, “What if runaway reaction occurs?” was asked, the PHA team recorded the hazard and consequences as “Not Applicable.” The PHA team relied on the information from the Ringwood pilot plant and the success of the pilot batches and the apparent success of the Paterson batches to reach this conclusion. As noted earlier, PHA team members stated that they were not aware of the work done in Hounslow to characterize the exothermic reactions although this work had been sent to Paterson in 1989. The PHA team did not take into consideration the potential for a runaway reaction, although the potential was evident in the product development information.

An effective PHA program requires examination of deviations from normal operations that could turn an exothermic reaction into a runaway situation. The PHA team should have considered such deviations as: “What if the o-NCB is warmer than specified prior to 2-EHA addition?” and “What if the predetermined temperature ranges of the staged heat-up process cannot be met due to equipment or instrumentation malfunction?” The PHA addressed the question of the failure of the heating system, but it did not ask the question, “What if there is inadequate cooling?”

The PHA team did not consider the potential ramifications of the high temperature excursions in batches 3, 5, 14, and 15 (see Section 5.3 – Investigation of Problems with Previous Batches). Investigation of these occurrences would have provided an opportunity to correct design problems, which likely would have prevented the incident.

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As a result of not recognizing the potential for a runaway reaction, the PHA team did not consider the need for additional safeguards, such as the ones Hounslow recommended in the 1989 process review memo. These recommendations included: temperature alarm, agitator failure alarm, level alarm, pressure alarm, and emergency cooling water addition directly into the reactor.\(^\text{36}\)

The late Frank P. Lees, an internationally recognized process safety expert, stated, with regard to emergency safety measures:

> There are a number of emergency measures that can be taken if a process deviation occurs which threatens to lead to a runaway reaction. The prime measures are:
> 
> (1) inhibition of reaction;
> (2) quenching of reaction;
> (3) dumping.

The reaction may be stopped by the addition of an inhibitor, or short stop. This involves the use of an inhibitor specific to the reaction in question....

Quenching involves adding a quenching agent, usually water under gravity, to the reaction mass to cool and dilute it....

The third method is dumping, which involves dropping the reactor charge under gravity into a quench vessel beneath which contains a quench liquid.

Other methods, which can be used to slow the reaction but are not generally ranked as prime emergency safety measures, include:

(1) shut-off of feed;
(2) direct removal of heat;
(3) indirect removal of heat;
   (a) full normal cooling;
   (b) emergency cooling.\(^\text{37}\)

The Hounslow recommendations of 1989 did suggest controlled quenching of the reaction with water as an emergency safety measure. The Lees publication does not list the measures actually used at Paterson (direct removal of heat and full normal cooling) as prime emergency safety

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measures. Quenching with water may not have been appropriate given the fact that the process was being run above the boiling point of water (condenser and relief vents would have had to be sized to safely handle the water vapor generated as the heat of reaction was removed from the kettle); however, this is another example of a possible safety improvement that should have been considered during the process design effort.

The PHA team also did not question whether the relief device sizing was adequate. Effective PSM programs require that PHA teams consider scenarios that might require pressure relief, such as runaway reactions, loss of cooling, loss of agitation, or errors in the addition of reactants. The necessary venting area is then determined through laboratory testing and calculations. Venting scenarios such as these were not discussed during the Yellow 96 PHA. Instead, the PHA participants relied on the information they had received from the Ringwood pilot plant that a runaway situation was not expected. As a result, the kettle’s venting systems were not designed to handle the pressure generation rate of the runaway reaction but had instead been sized for a scenario of an external fire that would boil xylene in the kettle.

After the incident, Morton calculated that a vent area of 116 square inches would have been required to properly vent the two-phase flow mixture that resulted from the decomposition reaction. 38 On K-7, the 6-inch rupture disk had a venting area of only 28.3 square inches.

5.3 INVESTIGATION OF PROBLEMS WITH PREVIOUS BATCHES

A number of previous batches of Yellow 96 had exhibited unexpected exothermic activity as shown by high temperature excursions beyond the normal operating range. These batches exhibited unusual temperature profiles or exceeded the maximum operating temperatures. In

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38 Morton calculated the required vent area using the DIERS methodology. DIERS (Design Institute of Emergency Relief Systems) is a consortium of companies, formed in 1976 by the American Institute of Chemical Engineers, to develop methods for the design of emergency relief systems to handle runaway reactions. The DIERS group has developed computer programs and a bench-scale test apparatus for assessing the venting requirements of runaway reactions and two-phase flow.
each of these incidents, the operators were able to regain control of the batch temperature and complete the batch. Management did not investigate these warning events in the processing history of Yellow 96 and did not consider these previous incidents during the PHA.

Significant prior incidents involving 1000-gallon reactor vessels included the following:

- **Batch 3 (1991):** The operators noted on the batch sheet “cooling not controlling temp.” The operators recorded a temperature of 134°C during the step for heating the batch from 90 to 110°C.

- **Batch 5 (November 1991):** The operators noted “Temp override to 120 ... cooling inadequate to control temp” during a specified temperature hold at 108 to 110°C.

- **Batches 14, 15, and 18 (July and September 1994, and September 1995):** The temperature recording charts for these batches indicate that the temperature rose quickly and remained off-scale (greater than 150°C) for approximately 30 minutes for batches 14 and 15 and 75 minutes for batch 18.

Starting with batch 25 in 1996, Morton began producing Yellow 96 in 2000-gallon reactor vessels. Significant occurrences following this change included:

- **Batch 28 (April 1997):** The operators noted on the batch sheet “cooling water of no use,” when trying to maintain the batch temperature at 110°C. The batch sheets indicate that batch temperature rose much more quickly than expected.

- **Batch 30 (October 1997):** The temperature of this batch rose quickly and remained off the temperature recording chart (maximum temperature of 150°C) for approximately 30 minutes.

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39 Two Morton batch sheets were labeled as batch 3. The batch in question was labeled CSBINSP003200-3213 in the data received from Morton.
Batch 31 (March 1998): As in batch 30, the reactor temperature rose quickly and remained off the chart for approximately 45 minutes. Based on operators’ comments during interviews, the temperature of this batch reached 180–200°C.

The operators’ written notes in the batch sheets, detailed above, should have served as notification to management of the temperature control problems and prompted the supervisors to investigate these problems. However, the operators did not always record high temperatures on the batch sheets and they did not verbally inform supervisors of the temperature excursions when they were able to regain control of a batch. One operator reported in an interview that he had brought these issues to the attention of his supervisor, but no supervisors recalled this contact. Several supervisors were aware that high temperatures had occurred in the past. However, supervisors stated in interviews that high temperatures were considered a quality concern, not a safety issue. The batch sheets for Yellow 96 mirrored this quality concern, stating, “DO NOT heat batch above 160°C or yield and quality will be lower.”

5.4 MANAGEMENT OF CHANGE

Beginning with batch 25, in 1996, Paterson began producing Yellow 96 in 2000-gallon kettles versus the 1000-gallon kettles that had been used for the first 24 batches. At that time, Paterson also scaled-up the Yellow 96 batch size by approximately 9 percent. The CSB determined that the combination of these two changes resulted in a reduction of approximately 10 percent in available heat transfer area per gallon of material. Therefore, the batches produced in the 2000-gallon kettles had less cooling per gallon of reacting material than batches made in the 1000-gallon kettles. Given the fact that the reaction was being run at temperatures very close to the runaway conditions, it was less likely that the operators would be able to control the process in the larger kettles.

Paterson did not use its management of change procedures to review the safety of these changes even though they met the definition of a change in Paterson’s PSM program. The batch sheets
were never modified to reflect the use of 2000-gallon kettles. The batch sheet in use at the time of the April 1998, incident was last revised in August 1996. It specified the use of a 1000-gallon kettle. The scaled up volumes of \textit{o}-NCB and 2-EHA were indicated on the batch sheets by crossing-out the old volumes and writing in the increased amounts.

5.5 **Operating Procedures and Training**

The Yellow 96 batch sheet contained little direction for the operators on how to manage the exothermic reaction between \textit{o}-NCB and 2-EHA. As a result, each operator ran the Yellow 96 process slightly differently. This lack of direction had safety consequences, as discussed in the examples below.

- The batch sheet stated, after adding \textit{o}-NCB to the kettle, to “Adjust temperature of kettle to 40–42°C with mixing.” With an onset temperature of 38°C, higher temperatures could lead to early initiation of the reaction and make it less likely that the operators could control the reaction.

- The batch sheet stated, after adding the 2-EHA, to “Make sure cooling water is on the condenser then carefully heat batch to 90°C. Note: DO NOT overheat or batch will start to exotherm.” The next step was to “[c]arefully give batch small shots of steam to raise the temperature 2–3°C if necessary. Watch batch temperature carefully as batch will exotherm from 90°C–110°C over 10 minutes.” There were no instructions on how much steam to use or how long to apply steam. Consequently, each operator had their own technique and looked for different temperature milestones to determine when to switch from steam to cooling water. The operators who ran batch 32 stated that they let steam enter the vessel jacket to heat up the batch for 5 to 10 minutes with 5 to 10 pounds of steam pressure on the jacket. They did not apply cooling water until the batch reached a temperature of 100 to 110°C. This was contrary to the experience of other operators and supervisors who stated
that the amount of steam admitted into the jacket should be minimal, lasting 1 to 5 minutes, and cooling water should be applied when the batch temperature reached 70 to 80°C.

- A note following the final reaction step stated, “DO NOT heat batch above 160°C or yield and quality will be lower.” Operators and supervisors stated during interviews that they had not been trained regarding the risk of a runaway exothermic reaction in the Yellow 96 process. They believed that temperatures higher than the maximum temperature of 160°C would result only in quality problems with the finished product. In addition, the temperature chart could not record temperatures above 150°C, limiting Morton’s ability to document the temperature profile of a batch and identify abnormal temperature deviations.

The additional steam used in batch 32 raised the reaction rate of the Yellow 96 synthesis reaction to a point at which it was generating heat faster than it could be removed by cooling water. The immediate cause of the incident highlights the dangerous circumstances under which the operators were instructed to run the Yellow 96 process:

- The batch sheet provided incomplete information on how much heating the batch required. New operators were trained on-the-job by working with an experienced operator, but batches of Yellow 96 were made infrequently, and so on-the-job training was infrequent. Each operator used their own experience as their guide.

- There was no instrumentation that operators could use to measure the amount of steam being used or cooling water temperatures to ensure consistency of batch temperatures from batch to batch.

- While the operators and supervisors knew the batch was sensitive to heat, they were unaware of the possible outcomes of a runaway reaction.

Plant operating procedures (batch sheets) did not address the handling of emergency situations in the kettles. Operators were not sufficiently trained to understand fully, or to react appropriately, to the runaway reaction. Other than the instruction to ensure that the cooling water was on, the operators were given no other direction for emergency situations. They were
not trained to recognize the hazards associated with the Yellow 96 process and consequently did not recognize when they should have evacuated the building. Operators were instructed to obtain help from supervision when unusual events occurred.

On the evening of the incident, the supervisor, the two principle operators, and two other chemical operators stayed by the runaway kettle while: (1) the rapidly rising batch temperature exceeded the operating procedure limit of 160°C, (2) the residual 2-EHA started boiling and the reaction generated additional gases, (3) the violently boiling mixture flooded the condenser, (4) the kettle rupture disks actuated because of the vessel’s pressurization, (5) the kettle began rumbling and shaking, and (6) the kettle temperature exceeded the decomposition onset temperature range for Yellow 96 of 195°C. After the operators established that heating was off, full cooling was on and the temperature of the batch continued to rise, their presence could not contribute anything to preventing an incident such as the one that occurred.
6.0 REGULATORY OVERSIGHT ANALYSIS

The Occupational Safety and Health Administration’s (OSHA) Process Safety Management (PSM) standard (29 CFR 1910.119) provides only minimum requirements for process safety management and provides little guidance with respect to reactive chemical process safety management. The lack of guidance did not contribute directly to causing the incident. However, additional guidelines in such areas as reporting and investigating deviations from normal operations, the use of chemical screening techniques, and proper design for pressure relief, emergency cooling, and safety interlock systems would likely have helped the Morton Paterson staff to recognize the hazards of the Yellow 96 process and take steps to avoid the incident.

6.1 REGULATORY COVERAGE

The OSHA PSM standard did not cover the Yellow 96 process. However, Morton included the process under its internal PSM program, which was applied to OSHA-regulated processes and certain other processes. In most respects, Morton patterned its program after the OSHA standard; however, there were significant omissions and differences. Also, the Morton program did not require adherence to a number of industry good practices for the safe management of reactive chemical processes.

OSHA only refers to reactivity hazards as two of the information requirements in the PSM standard’s Process Safety Information element subheading, “Information pertaining to the hazards of the highly hazardous chemicals in the process,” 1910.119(d)(1): “(iv) Reactivity data [and] (vi) Thermal and chemical stability data.” In addition, the Non-Mandatory Appendix to the standard states,

“The information to be compiled about the chemicals, including process intermediates, needs to be comprehensive enough for an accurate assessment of the fire and explosion characteristics, reactivity hazards, the safety and health hazards to workers, and the corrosion and erosion effects on the process equipment and monitoring tools. Current material safety data sheet (MSDS)
information can be used to help meet this requirement which must be supplemented with process chemistry information including runaway reaction and over pressure hazards if applicable.”

The OSHA PSM standard covers certain reactive chemicals ranked as 3 and 4 under the National Fire Protection Association’s (NFPA) 0 to 4 reactivity ranking system. Reactive chemicals are not covered under the Environmental Protection Agency’s (EPA) Risk Management Plan rule. Some reactive chemicals are listed by the EPA because they qualify for inclusion due to their toxic or flammable properties. The raw materials and the Yellow 96 Dye were all assigned a 0 reactivity rating on their respective material safety data sheets. However, as was the case with the Yellow 96 process, many chemicals with low reactivity rankings can be very reactive in combination with other chemicals or at temperatures that are reached only as a result of deviations from normal operating conditions.

In 1995, OSHA received a petition to promulgate an Emergency Temporary Standard (ETS) as the result of an incident at Napp Technologies, Inc., in Lodi, New Jersey, on April 21, 1995, that killed five people. The petition stated that the PSM standard “fails to comprehensively cover reactive chemicals” and requested that OSHA reopen the standard. As of August 2000, OSHA has not acted on this petition.

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40 In NFPA 704, the NFPA defines “3s” as “materials that in themselves are capable of detonation or explosive reaction, but that require a strong initiating source or that must be heated under confinement before initiation.” “4s” are defined as “materials that in themselves are capable of detonation or explosive reaction at normal temperatures and pressures.”

41 Eric Frumin, et al., to Mr. Joe Dear, Asst. Secretary for Occupational Safety and Health, “Petition for Emergency Revision of Standards on Process Safety Management and Emergency Response,” October 23, 1995. The petitioners were Union of Needletrades, Industrial and Textile Employees; United Steelworkers of America; Oil, Chemical and Atomic Workers; AFL-CIO; International Association of Fire Fighters and International Chemical Workers Union.
Morton’s written Process Safety Management program did not include a number of OSHA PSM requirements. The U.S. Chemical Safety and Hazard Investigation Board (CSB) also identified areas in which Morton did not implement their PSM program.

Examples of inadequacies in Paterson’s PSM program included the following:

- Under the requirements for what the Process Hazard Analysis (PHA) shall address, the Morton program did not include the OSHA requirement for “The identification of any previous incident which had a likely potential for catastrophic consequences in the workplace.” As stated earlier, investigation of these occurrences would have provided an opportunity to correct design problems, which likely would have prevented the incident.

- Under OSHA PSM requirements, an operating procedure is required for Emergency Shutdown. Morton’s program simply lists “Emergency Shutdown” in a list of required procedures and omitted OSHA’s requirements that the procedures should state the conditions under which shutdown is required and the operator’s responsibilities. Inclusion of this information in operating procedures and training likely would have led the operators to evacuate sooner, thus preventing their injuries.

Examples of inadequacies in Paterson’s implementation of its PSM program included the following:

- Morton’s program required that the process safety information package should contain “Copies of any laboratory, pilot plant, or other testing work (including that performed outside the company) which pertains to the hazards posed by the chemicals used in the process.” There were a number of memos and notes that touched on the exothermic nature of the Yellow 96 process, but the testing results and memos that explicitly discussed the runaway potential were not provided to the PHA team or used to inform operations personnel. Using this information in process reviews and in training would have made personnel more aware of the hazards of the process.
• Morton’s program required information on chemical reactions and equations for primary and important secondary, or side reactions addressing all chemicals associated with the process, including discharge and by-products. Also required, if available, were kinetics data for important process reactions, including order, rate constants vs. time, approach to equilibrium, and kinetics data for undesirable reactions, such as decompositions and autopolymerizations. This information was not compiled for the Yellow 96 process although the information had been developed and was available.

• The change from the 1000-gallon to the 2000-gallon kettle was not approved through a Management of Change process, although the change met Morton’s definition of a change. A change of this magnitude should also have been reviewed in a PHA per Morton’s procedure. Again, Morton missed an opportunity to assess the hazards of the process and take steps to avoid an incident.

6.3 GOOD PRACTICE GUIDELINES

Good practice guidelines, such as the Center for Chemical Process Safety publications,42,43 were available prior to the startup of Yellow 96 production in 1990. Other guidelines (see References) became available prior to the Yellow 96 Process Hazard Review in 1995. Use of these guidelines would have provided the Morton Paterson staff with information on the means to identify the potential reactive hazards and would likely have led them to take steps to prevent the incident.

Additional guidelines are now available from the Center for Chemical Process Safety’s books on reactive chemicals (see References), Lees’s Loss Prevention in the Process Industries44 and

the U.K. Health and Safety Executive’s booklet, “Designing and Operating Safe Chemical Reaction Processes.”⁴⁵ The U.K. guidelines, published in May 2000, are a consensus product of government, industry, and labor. Use of these types of guidance documents by Morton staff could have provided them with sufficient information and awareness of the reactive hazards that likely would have helped them to take actions to prevent the incident.

Government-issued safety guidelines on other topics, such as those issued by OSHA and EPA, have been used extensively by industry. For example, the EPA’s Off-Site Consequence Analysis Guidance is used by industry in complying with the EPA’s Risk Management Plan requirements.

### 6.4 Recent Catastrophic Reactive Chemical Incidents

The Chemical Safety Board, and safety professionals in general, currently face limitations in their ability to obtain information on reactive chemical incidents. Detailed analysis of root causes is only available for several major incidents in recent years. Expanded information on the root causes of incidents would provide data on trends in reactive chemical process safety management and on the effects of federal and state regulations and consensus standards on the frequency and severity of reactive chemical incidents.

**Napp Technologies Incident⁴⁶**

The 1995 Napp explosion and fire in Lodi, New Jersey resulted in five deaths as well as injuries, public evacuations, and serious damage both on and off the site. A joint EPA/OSHA team investigated this event. At the time of the incident, Napp was conducting a mixing operation involving water-reactive chemicals. The operation, which should have been completed in less

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than 1 hour, continued for nearly 24 hours. Operators noticed an unexpected reaction taking place in the blender, producing heat and the release of a foul-smelling gas. During an emergency operation to remove the contents of the blender, the material ignited and a deflagration occurred that resulted in the deaths of the employees and the destruction of the facility.

According to the EPA/OSHA report, the most likely cause of the incident was the inadvertent introduction of water into water-reactive materials (aluminum powder and sodium hydrosulfite) during the mixing operation. The water caused the sodium hydrosulfite in the blender to decompose, generating heat, sulfur dioxide, and additional water. The decomposition process, once started, was self-sustaining. The reaction generated sufficient heat to cause the aluminum powder to react rapidly with the other ingredients and generate more heat, until the point of deflagration was reached. As in the Morton case, the chemicals and chemical reactions involved in the Napp incident were not covered under the OSHA PSM standard.

Georgia-Pacific Resins Incident

In 1997, an explosion at a resins production unit at Georgia-Pacific Resins, Inc. in Columbus, Ohio, killed one worker and injured four others. This explosion was caused by a runaway reaction. As detailed in an EPA Chemical Safety Case Study on this event, “the runaway was triggered when, contrary to standard operating procedures, all the raw materials and catalyst were charged to the reactor at once followed by the addition of heat. Under the runaway conditions, the heat generated exceeded the cooling capacity of the system and the pressure generated could not be vented through the emergency relief system causing the reactor to explode.”

The PHA conducted for the Georgia-Pacific process did not consider the failure to control the rate of chemical addition to the process. In addition, the pressure relief system was not sized to handle the pressure rise from such an event.

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Napp, Morton, and Georgia-Pacific were three of the most significant and highly studied reactive chemical incidents to occur in the United States in recent years but they were not the only incidents:

- In its Safety Alert on the Georgia-Pacific incident, EPA noted six other phenol-formaldehyde reaction incidents that had occurred since 1989.\(^{49}\)

- Databases such as the EPA’s Emergency Response Notification System, OSHA’s incident statistics, and the U.K. Institution of Chemical Engineers’ incident database contain over 30 events in the last decade that were characterized by keywords such as “exothermic reaction” or “runaway reaction.”

7.0 ROOT AND CONTRIBUTING CAUSES

7.1 ROOT CAUSES

1. Neither the preliminary hazard assessment conducted by Morton in Paterson during the design phase in 1990 nor the process hazard analysis conducted in 1995 addressed the reactive hazards of the Yellow 96 process.

Not addressing these hazards resulted in the following design, operational, and training deficiencies:

- The kettle cooling system could not safely control the exothermic Yellow 96 synthesis reaction.

- The kettle was not equipped with safety equipment, such as a quench system or a reactor dump system, to stop the process to avoid a runaway reaction.

- Rupture disks were too small to safely vent high pressure in the kettle in the event of either of the two foreseeable runaway reactions.

- Morton converted its Yellow 96 production from a staged, incremental addition (semi-batch) process to a staged heating (batch) process without assessing the possible hazards of this change, e.g., the increased difficulty of controlling heat output.

- Operating procedures did not cover the safety consequences of deviations from normal operating limits, such as runaway reactions, or specify steps to be taken to avoid or recover from such deviations.

- Training did not address the possibility of a runaway reaction and how operators should respond to avoid injury if a runaway reaction could not be controlled.
2. Process safety information provided to plant operations personnel and the process hazard analysis team did not warn them of the potential for a dangerous runaway chemical reaction.

Morton internal memoranda had documented that the desired reaction to form Yellow 96 from ortho-nitrochlorobenzene (o-NCB) and 2-ethylhexylamine (2-EHA) was exothermic and that Yellow 96 would begin to decompose rapidly (runaway) at temperatures close to the upper operating temperature. Morton researchers also had identified several situations, such as loss of cooling, that might give rise to temperatures capable of causing violent decompositions. Although information on the reactive hazards was contained in plant files, operators and supervisors were unaware that a dangerous decomposition reaction was possible.

7.2 Contributing Causes

1. The hazards of previous operational deviations were not evaluated.

   Management did not investigate evidence in numerous completed batch sheets and temperature charts of high temperature excursions beyond the normal operating range. Investigation of these incidents likely would have provided an opportunity to uncover the process’s reactive hazards and correct design and other problems.

2. Morton did not follow their Management of Change procedures to review changes made in reaction kettle and batch size.

   Morton changed the Yellow 96 processing equipment from 1000-gallon to 2000-gallon kettles and also increased the batch size by 9 percent in 1996. Morton did not follow its Management of Change procedures and did not review the changes for possible safety consequences. A Management of Change review likely would have provided another
opportunity for Morton to uncover the process’s reactive hazards and correct design problems. The review likely would have also revealed that the changes made resulted in a decrease of 10 percent in the heat transfer area per gallon of reactants. Half of the batches made after this change exhibited temperature excursions versus 20 percent in the batches made before the change.
8.0 RECOMMENDATIONS

Morton International, Inc. (a wholly owned subsidiary of Rohm and Haas Company)

Establish a program that ensures that reactive chemical process safety information and operating experience are collected and shared with all relevant units of the company (98-006-I-NJ-R01).

Morton International, Inc.’s Paterson, New Jersey Plant

1. Revalidate Process Hazard Analyses for all reactive chemical processes in light of the findings of the U.S. Chemical Safety and Hazard Investigation Board (CSB) report and upgrade, as needed, equipment, operating procedures, and training (98-006-I-NJ-R02).

2. Evaluate pressure relief requirements for all reaction vessels using appropriate technology, such as the Design Institute for Emergency Relief Systems (DIERS)\(^{50}\) method and test apparatus and upgrade equipment as needed (98-006-I-NJ-R03).

3. Evaluate the need for and install, as necessary, devices, such as alarms, added safety instrumentation, and quench or reactor dump systems to safely manage reactive chemical process hazards (98-006-I-NJ-R04).

4. Revise operating procedures and training for reactive chemical processes as needed, to include descriptions of the possible consequences of deviations from normal operational limits and steps that should be taken to correct these deviations, including emergency response actions (98-006-I-NJ-R05).

\(^{50}\) DIERS is a consortium of companies, formed in 1976 by the American Institute of Chemical Engineers to develop methods for the design of emergency relief systems to handle runaway reactions. The DIERS group has developed computer programs and a bench-scale test apparatus for assessing the venting requirements of runaway reactions and two-phase flow.
5. Implement a program to ensure that deviations from normal operational limits for reactive chemical processes that could have resulted in significant incidents are documented and investigated and necessary safety improvements are implemented (98-006-I-NJ-R06).

6. Revise the Yellow 96 Material Safety Data Sheet (MSDS) to show the proper boiling point and National Fire Protection Association reactivity rating. Evaluate the need for and change, as necessary, the MSDSs for other Morton dyes. Communicate the MSDS changes to current and past customers (who may retain inventories of these products) (98-006-I-NJ-R07).

Occupational Safety and Health Administration (OSHA) and Environmental Protection Agency (EPA)

1. Issue joint guidelines on good practices for handling reactive chemical process hazards (98-006-I-NJ-R08).

Ensure that these guidelines, at a minimum, address the following issues:

- The evaluation of reactive hazards and the consequences of reasonably foreseeable and worst-case deviations from normal operations.

- The importance of reporting and investigating deviations from normal operations.

- The determination of proper design for pressure relief capability, emergency cooling, process controls, alarms, and safety interlocks, as well as other good-practice design features for handling reactive substances.

- The appropriate use of chemical screening techniques such as differential scanning calorimetry.

2. Participate in a hazard investigation of reactive chemical process safety conducted by the CSB (98-006-I-NJ-R09).
The objectives of the special investigation will include:

- Determine the frequency and severity of reactive chemical incidents.
- Examine how industry, OSHA, and EPA are currently addressing reactive chemical hazards. Determine the differences, if any, between large/medium/small companies with regard to reactive chemical policies, practices, in-house reactivity research, testing, and process engineering.
- Analyze the effectiveness of industry and OSHA use of the National Fire Protection Association Reactivity Rating system for process safety management purposes.
- Develop recommendations for reducing the number and severity of reactive chemical incidents.

American Chemistry Council; Center for Chemical Process Safety (CCPS); Paper, Allied-Industrial, Chemical & Energy Workers International Union (PACE); Synthetic Organic Chemical Manufacturer’s Association (SOCMA)

Communicate the findings of this report to your membership (98-006-I-NJ-R10).
9.0 REFERENCES


APPENDIX A: INCIDENT TIMELINE

Shift starts at 1600, April 8, 1998

Operator 1 held over from last shift.

Operator 1 & 2 suit up in protective clothing

Operators 1 & 2 inspect K-7

Inspection manway hatch is secured with C clamps.

K-7 empty and clean.

Temperature of empty K-7 is 20 to 30 o C (ambient).

Start

Temperature of a 2,000-gallon chemical reactor.

This is the fourth batch of Automate Yellow 96 Dye to be prepared in K-7.

This is to be the 32nd plant batch of Automate Yellow 96 Dye.

Kettle 7 (K-7) is a 2,000-gallon chemical reactor.

Operators 1 & 2 secure inspection manway hatch on the head of K-7.

Operators 1 & 2 inspect K-7 and prepare to start batch 32 of Yellow 96.

A three-man team makes final preparation to load ortho-nitrochlorobenzene (o-NCB).

Laborers suit up in protective clothing with supplied air.

Operators 1 & 2 secure inspection manway hatch on the head of K-7.

Operators 1 & 2 inspect K-7 and prepare to start batch 32 of Yellow 96.

The B-11 evacuation is scheduled.

Second-shift foreman assigns Operators 1 & 2 to K-7 in Building 11 (B-11) and gives them Automate Yellow 96 Dye batch sheets.

Operators 1 & 2 inspect K-7 and prepare to start batch 32 of Yellow 96.

The B-11 supervisor and 1st-shift foreman schedules production and work assignments.

Speaker and 1st-shift foreman schedules production and work assignments.

Note: All times are expressed as military time.

APPENDIX A: INCIDENT TIMELINE

REVISED – 7/5/2000
On second floor, laborer brings drums of o-NCB from elevator to K-7. (1645)

A hand-pulled forktruck is used to bring the pallet of drums from elevator to kettle on second floor of B-11.

The pallet of drums is brought to the second floor of B-11 by an elevator at the west end.

A laborer has prepared a solution of water and detergent in 5-gallon pail.

A laborer has prepared a solution of water and detergent in 5-gallon pail.

K-7 agitator is turned on.

K-7 feed manifold and dip tube flushed with 5 gallons of detergent solution. (1720)

Operators 1 & 2 leave B-11 for airing out period. (1730)

Team unsuits.

K-7 feed manifold and dip tube flushed with 5 gallons of detergent solution. (1720)

Temperature of drums is too hot to handle with gloved hand.

Temperature after loading o-NCB was reported by operators as from "about 44" to "60-something almost 70.

K-7 vacuum pulls o-NCB through dip tube and feed manifold. (1715)

Transfer of o-NCB takes about 25 minutes and is uneventful.

Vacuum system is working. Vacuum gauge on head of K-7. No unusual occurrences noted.

Pallet of empty drums is returned to B-11 elevator.

B-11 unoccupied for mandatory 30 minutes while "airing out."

Temperature of drums is too hot to handle with gloved hand.

K-7 agitator is turned on.

On second floor, laborer brings drums of o-NCB from elevator to K-7. (1645)
Shots of steam are added by opening the steam-in valve for a few minutes.

Operators 1 & 2 return to charge 2-ethylhexylamine (2-EHA). (1800)

2-EHA flows into K-7 through feed manifold from underground storage tanks. (1830)

Operator 1 adds first steam to K-7 jacket. (1940)

Operator 1 fills K-7 jacket with steam.

Operators estimate that loading 2-EHA takes 25 minutes.

Based on experience, Operator 1 aligns valves and sets 2-EHA feed meter.

Operators know that Yellow 96 has a very difficult exotherm to control.

Operators and supervisors know that exotherm of Yellow 96 starts before 90 °C.

Individual operators work from experience and develop their own batch heating techniques.

Vent to condenser above K-7 is open.

Digital meter is set for 2-EHA charge and controls remote pump at storage tank.

Jacket steam pressure is 5 to 10 psi on dial gauge.

Lag time between temperature rise in kettle and steam addition is a few minutes.

There are no temperature or flow indicators on steam supply.

Operators' dinner break. (1900–1935)

Operator 1 adds one or two more shots of steam.

At the beginning of heating, the temperature of the 2-EHA and o-NCB mixture is at 44 °C.

Jacket steam pressure is 5 to 10 psi on dial gauge.

One line in operating instructions in batch sheet says only, “heat carefully to 90°C.”

Operators and supervisors know that exotherm of Yellow 96 starts before 90 °C.

Individual operators work from experience and develop their own batch heating techniques.

Operators estimate that loading 2-EHA takes 25 minutes.

Based on experience, Operator 1 aligns valves and sets 2-EHA feed meter.

Operators know that Yellow 96 has a very difficult exotherm to control.

Operators and supervisors know that exotherm of Yellow 96 starts before 90 °C.

Individual operators work from experience and develop their own batch heating techniques.

Vent to condenser above K-7 is open.

Digital meter is set for 2-EHA charge and controls remote pump at storage tank.

At the beginning of heating, the temperature of the 2-EHA and o-NCB mixture is at 44 °C.

Jacket steam pressure is 5 to 10 psi on dial gauge.

Lag time between temperature rise in kettle and steam addition is a few minutes.

There are no temperature or flow indicators on steam supply.

Operators estimate that loading 2-EHA takes 25 minutes.

Based on experience, Operator 1 aligns valves and sets 2-EHA feed meter.

Operators know that Yellow 96 has a very difficult exotherm to control.

Operators and supervisors know that exotherm of Yellow 96 starts before 90 °C.

Individual operators work from experience and develop their own batch heating techniques.

Vent to condenser above K-7 is open.

Digital meter is set for 2-EHA charge and controls remote pump at storage tank.

At the beginning of heating, the temperature of the 2-EHA and o-NCB mixture is at 44 °C.

Jacket steam pressure is 5 to 10 psi on dial gauge.

Lag time between temperature rise in kettle and steam addition is a few minutes.

There are no temperature or flow indicators on steam supply.
The K-7 temperature rises rapidly to 100° to 110°C. (2000)

Operators overhear water flow and steam bumping in K-7 jacket, verifying water is on.

Operators estimate that it takes 2 minutes to switch to cooling water.

Valves are grouped on opposite sides of K-7.

Operators' past experience indicates that the exotherm of Automate Yellow 96 may begin around 75°C.

Operators 1 & 2 estimate about 15 minutes have passed since steam addition began.

K-7 temperature rises rapidly to 150°C and keeps rising rapidly.

K-7 temperature rises to over 175°C. (2015)

K-7 starts to vibrate the second-floor decking.

Three other operators on the second floor of B-11 are now aware that K-7 has a problem and start over to see.

Other second-floor operators have gathered around in the aisle next to K-7.

The shift supervisor arrives and rechecks valves.

The temperature in K-7 has exceeded the boiling point of residual 2-EHA.

Permanent gases are being generated by the reaction.

Cooling water to K-7 confirmed to be full-on. Cooling water pressure is normal.

The shift supervisor arranges to come over.

Three other operators on the second floor of B-11 are now aware that K-7 has a problem and start over to see.

The experienced operators have never before observed a significant condensate return in a Yellow 96 batch.

The experienced operators have never before observed a significant condensate return in a Yellow 96 batch.

Operators 1 & 2 see shift supervisor on his rounds and call him to come over.

Operators 1 & 2 estimate about 15 minutes have passed since steam addition began.

Operators 1 & 2 hear water flow and steam bumping in K-7 jacket, verifying water is on.

Operators 1 & 2 estimate that it takes 2 minutes to switch to cooling water.

Operators 1 & 2 recheck cooling water valve positions and find them correct.

The cooling water line-in or line-out have no temperature or flow indicators.
Two 6-inch rupture disks in series pop in 6-inch line from K-7 reactor head to B-11 catch tank. (2015)

- Rupture disks are set for 10 psi.
- The temperature in K-7 is between 180°C and 190°C.
- Mixture in K-7 is above the Yellow Dye decomposition exotherm onset temperature of 175°C-195°C.
- Heat removal by condensing vapors in the condenser and returning reflux to the kettle cannot keep up with reaction heat.
- K-7 rumbling violently as mix's boiling rate rapidly increases.
- K-7 temperature climbs to over 200°C. (2017)
- Last K-7 temperatures observed were between 200°C and 290°C.
- An experienced observing operator reads digital temperature gauge at 290°C.
- There is a loud roar; other plant workers think that a train is passing the plant.

Remaining group of operators and shift foreman run toward B-11 exits by different routes.

K-7 contents erupt from manway, starting flash fires that catch workers trying to escape second floor, B-11. (2018)

Aerosol plume of K-7 reactants and products blows through the third floor and roof of B-11 and ignites.

Operator 1 and shift foreman at second floor east end door are blown down steps to landing.

Secondary ignition of aerosol release blows them off landing onto ground.

They suffer severe third-degree burns and require intensive care.

Witness observes large fire ball above roof.

K-3 operator shouts “get out” and runs to stairs toward the west end of B11.

To this point, the only pressure relief for K-7 was the 2.5-inch condenser vent line through ball valve to receiver vented to catch tank.

On all other occasions, the Yellow 96 batches had recovered from these temperature excursions.
Call to Paterson Fire Department (PFD) at 2020.

- Multiple 911 calls.
- Structure fire on the roof at the east end of B-11.
- Sounds second alarm for more equipment, (2044)

Battalion 1 arrives on scene at 2024.

- All plant personnel accounted for.
- Victims are sent by ambulance to three area hospitals, two most serious to St. Joseph’s.
- Nine injured workers assemble at the McLean Blvd. plant entrance.

PFD positions ladder pipe truck and begins placing water on west end of B-11 roof.

- Uncertain as to whether chemicals involved are water reactive.
- Full protective gear used.

Fire under control at 2327.

PFD repositions ladder pipe truck, increases pressure, and douses east end of B11 roof.

- Fire knocked down at 2144, but water flow maintained to cover entry teams.
- Light vapor fires put out with wheeled dry chemical unit.
- Fire units remain in case of hot spots.
- Site secured as a crime scene by Paterson police.

End

- Investigative teams begin.
- One fireman punctures boot and is exposed to site chemicals.
- Primary and secondary building searches are completed.
- All firemen decontaminated before returning. No ill effects.
- One fireman decontaminated before returning. No ill effects.
- HAZMAT and fire teams suit up for entry.
- Site secured as a crime scene by Paterson police.
- Water flow from ladder pipe is stopped to prevent washing contaminants into Passaic River.

5
APPENDIX B: DEVELOPMENT TIMELINE

June/July 1986
Initial Lab Experiments at Hounslow, UK

10/2/87
Differential Scanning Calorimetry testing in UK identified decomposition exotherm

4/8/88
Memo, "Thermal Stability of Liquid Yellow 152," Discussion of thermal stability work at Brunel University

4/8/89
Handwritten Memo—"LY 152 Process Review" recommending additional testing and safety instrumentation

4/28/89
"LY 152 Process Review" transmitted to Paterson/Ringwood

5/1/89
Development of stepwise heating process to minimize exposure to ortho-nitrochlorobenzene

5/12/89
First Ringwood Batch —100 gallons

6/25/90
Ringwood Batch 90-9495
Operator's Note: "This batch wants to run, 112-133°C"

8/22/90
Ringwood Semiworks Process Transmittal sent to Paterson

9/6/90
First Paterson batch

1991
Batch 4—High Temp occurrence —1000-gal kettle "Cooling not controlling temp"

11/14/91
Batch 5—High Temp occurrence —1000-gal kettle "Cooling inadequate to control temp"

7/27/94
Batch 14—High Temp occurrence 1000-gal kettle

9/13/94
Batch 15—High Temp occurrence 1000-gal kettle

January 1995
PROCESS HAZARD ANALYSIS

9/14/95
Batch 18—High Temp occurrence 1000-gal kettle

1996
Process Change to 2000-gal kettles and increased batch size

4/2/97
Batch 28—High Temp occurrence 2000-gal kettle

3/31/98
Batch 31—High Temp occurrence 2000-gal kettle

4/8/98
Batch 32—Explosion and Fire K-7—2000-gal kettle

Revised 7/5/2000
APPENDIX C: ROOT CAUSE ANALYSIS DIAGRAM

Injuries and Physical Damage

Gas pressure exceeds strength of manway clamps

Employees present at kettle side

Employees attempt to salvage product

Employees unaware of hazards

Inadequate hazard assessments

Inadequate mgmt. of change

Employees had saved previous batches

Majority of batches had been smaller (1000 vs 2000 gal)

Timing of steam/cooling water had been favorable

Venting capacity insufficient to relieve pressure

Runaway reaction generates permanent gases and boiling of 2-EHA

Cooling water system unable to control heat of reaction

Operating procedures/training inadequate

Steam added at too high a rate

Switching to cooling water too late

Inadequate transfer of research information

Operating procedures/training inadequate

Inadequate transfer of research information

Inadequate implementation of plant programs

Inadequate design — no heat/mat'l balance

Not required by Morton PSM program

Inadequate guidance on hazard assessments

Inadequate guidance on hazard assessments

Inadequate transfer of research information
APPENDIX D: MATERIAL SAFETY DATA SHEETS

MATERIAL SAFETY DATA SHEET

1. CHEMICAL PRODUCT AND COMPANY INFORMATION

Product Name: Acumatic Yellow 96
Trade Name: Acumatic dye
Product Form: Solid

For customer service/technical information, contact Morton Adhesives & Chemical Specialties
100 N. Riverside Plaza
Chicago, IL 60606
1-800-279-1777

MSDS version by:
Technology and Regulated Substance Compilations
David Misakian, P. E.
100 N. Riverside Plaza
Chicago, IL 60606
1-800-279-1777

3. CHEMICAL PRODUCT AND COMPANY INFORMATION

2. COMPOSITION/INFORMATION ON INGREDIENTS

COMMON NAME
C.I. Solvent Yellow
Non-hazardous and other ingredients
Below reportable levels

Hazardous Level
Benzanilide, N[(2-ethylhexyl)2-nitro (CAS No. 85128-20-2)

4. HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW: May cause digestive tract irritation. May cause eye irritation. Prolonged or repeated contact may cause skin irritation. Prolonged exposure may cause eye, nose, and respiratory tract irritation. See sections 2, 6, and 7 for additional information.

PRIMARY MODES OF EXPOSURE: Eye, Skin

EYE CONTACT: May cause slight to mild irritation.

SKIN CONTACT: Prolonged or repeated contact may cause irritation.

INHALATION: Irritation to the nose, mouth, throat, and stomach.

INGESTION: Irritation to the mouth, throat, and stomach.

TARGET ORGANS/CHRONIC EFFECTS: Eye

CONDITIONS AGGRAVATED BY EXPOSURE: Exposure to this product is not expected to contribute, worsen or aggravate any pre-existing medical conditions.
4. FIRST AID MEASURES

EYE CONTACT: Immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention if irritation persists.

SKIN CONTACT: Immediately flush with water. Remove contaminated clothing and shoes. Get medical attention if irritation persists. Professionally wash clothing and shoes before reuse.

INHALATION (Breathing): Provide fresh air. If symptoms develop, seek immediate medical attention. If not breathing, give artificial respiration.

INGESTION (Swallowing): Get medical attention. Immediately induce vomiting as directed by medical personnel. Never give anything by mouth to an unconscious person.

NOTES TO PHYSICIANS: Treatment should be directed at preventing absorption, administering no symptoms till they occur, and providing supportive therapy.

5. FIRE FIGHTING METHODS

Flash Point: > 200°F Method: ........ Self-Flash Closed Cup Explosive Limit: LEL/UL Not Determined UN Listed: Not Determined

HAZARDOUS COMBUSTION AND DECOMPOSITION PRODUCTS: Smoke, heat, and toxic/irritating fumes, e.g., carbon dioxide, carbon monoxide, etc. Oxides of nitrogen.

FIRE AND EXPLOSION HAZARDS: During a fire, irritating and highly toxic gases may be generated during combustion or decomposition.

EXTINGUISHING MEDIA: SMALL FIRES: Any chemical, carbon dioxide, water spray, or foam. LARGE FIRES: Water spray, ESG, or alcohol foam.

FIRE FIGHTER PRECAUTIONS/EQUIPMENT: Fire fighters and others who may be exposed to the products of combustion should be equipped with NIOSH approved positive pressure self-contained breathing apparatus (SCBA) and full protective clothing.

6. ACCIDENTAL RELEASE MEASURES

EVACUATION: Isolate hazard area. Keep unnecessary and unprotected people from entering.

CONTAINMENT: Safely stop discharge. Contain material, as necessary, with a barrier or barrier.

COLLECTION AND DISPOSAL: Stop discharge if safe to do so. Use proper protective equipment. Cover spills with absorbent clay or sand and place in closed chemical waste containers. Disposal according to applicable local, state and federal regulations.
1. HANDLING AND STORAGE

STORAGE CONDITIONS: Store in cool, dry, well-ventilated area.

TRANSFER: No special precautions are needed. Follow good warehouse and handling practices.

PERSONAL HYGIENE: Wash thoroughly after handling, especially before eating, drinking, smoking, and using restrooms. Change contaminated clothing, if need be, and wash. Routine handwashing is not usually necessary.

EMPTY CONTAINER PRECAUTIONS: Attention! This container can be hazardous when empty. Follow label warnings even after containers are emptied since empty containers may retain product residue. Do not reuse empty containers without professional cleaning for food, clothing, or products for human or animal consumption or whose skin contact can occur.

2. EXPOSURE CONTROLS/PERSONAL PROTECTION

EXPOSURE GUIDELINES:

OSHA - TLV - No regulated ingredients.

ENGINEERING CONTROLS/VENTILATION: Local exhaust ventilation is recommended when vapors, mists, or dusts can be released.

EYE PROTECTION: Wear chemical splash goggles or safety glasses with side shields.

SKIN PROTECTION: Wear protective clothing and appropriate impervious gloves. A variety of protective gloves exist. Consult glove manufacturers to determine the proper type for a specific operation.

RESPIRATORY PROTECTION: Wear NIOSH/MSHA-approved equipment. Determine the appropriate type by consulting the manufacturer. High airborne concentrations may necessitate the use of self-contained breathing apparatus (SCBA) or a supplied air respirator. Respiratory protection programs must be in compliance with 29 CFR 1910.134.

3. PHYSICAL AND CHEMICAL PROPERTIES

Appearance: Dark
Physical State: Liquid
pH: Not Applicable
VOC Material: No Determined

Specific Gravity: 1.0

NOTE: The physical data presented above are typical values and should not be considered as a specification.

10. STABILITY AND REACTIVITY
CHEMICAL STABILITY: Stable under normal conditions of use.

HAZARDOUS DECOMPOSITION ENDPRODUCTS: None known.

UNCONDITIONAL WARNING: Not applicable.

INCOMPATIBILITY WITH OTHER MATERIALS: None known.

11. TOXICITY INFORMATION

COMPONENTS:

C.I. Solvent Yellow
May cause temporary irritation to eyes and/or skin.

12. ECOLOGICAL INFORMATION

No data are available on this product.

13. DISPOSAL CONSIDERATIONS

DISPOSAL: Dispose in accordance with all local, state, and federal regulations.

GENERAL STATEMENTS: Federal regulations may apply to empty containers. State and/or local regulations may be different.

GENERAL RECOMMENDATIONS: Of the methods of disposal currently available, it is recommended that an alternative be selected according to the following order of preference, based upon environmental acceptability: (A) recycle or rework, if feasible; (B) treat waste at an authorized waste treatment facility.

SPECIAL INSTRUCTIONS: Be sure to contact the appropriate government environmental agency if further guidance is required.

14. TRANSPORT INFORMATION

Weight (lb) Shipping Name Non-regulated 49 CFR DATA B40 Y Y

DOT Label...: Not applicable
UN/NA No.: Not applicable

15. REGULATORY INFORMATION

FEDERAL:

This product is regulated hazardous under the OSHA Hazard Communication Standard 29 CFR 1910.1200.

SARA Title III Section 311: Hazard Categories:

X Ozone Depleting Chemicals - No regulated ingredients.
SARA Section 302 Extremely Hazardous Mat - No regulated ingredients.

SARA Section 401 Toxic Chemicals - No regulated ingredients.

STATE RIGHT TO KNOW.

Pennsylvania - New Jersey - NY
C.I. Solvent Yellow 5 GHS Code: H315, H319, H320
Non hazardous trade secret ingredient

California - California Proposition 65 - No regulated ingredients.

CONEG - No data available.

Canada:
This is a "controlled product" under the Canadian Workplace Hazardous Materials Information System (WHMIS).

Class 2 Division 1 Subdivision B

CBFA - NPH - No regulated ingredient.

Canadian Chemical Inventory

Domestic Substance List

Listed.

14. OTHER INFORMATION

USER RESPONSIBILITY: A bulletin such as this cannot be expected to cover all possible individual situations. As the user has the responsibility to provide a safe workplace, all aspects of an individual operation should be examined to determine if, or where, precautions - in addition to those described herein - are required. Any health hazard and safety information herein should be passed on to your customers or employees, as the case may be.

DISCLAIMER OF LIABILITY: The information contained herein is, to the best of our knowledge and belief, accurate. However, since the conditions of handling and use are beyond our control, we make no guarantee of results, and assume no liability for losses incurred by use of this material. All chemicals may present inherent health hazards and should be used with caution. Although certain hazards are described herein, we cannot guarantee that these are the only hazards which exist. Final evaluation of suitability of the chemical is the sole responsibility of the user. No representations or warranties, either expressed or implied, of merchantability, fitness for a particular purpose or any other nature are made herewith with respect to the information contained herein or the chemical to which the information relates. It is the responsibility of the user to comply with all applicable federal, state and local laws and regulations.

End of Material Safety Data Sheet
1. CHEMICAL PRODUCT AND COMPANY IDENTIFICATION

PRODUCT NAME  ORTHO-NITROCHLOROBENZENE
MSDS Number  00008733
Date  August 4, 1999
Chemical Name  Benzene, 1-chloro-2-nitro
Synonyms  OMCB, o-Nitrochlorobenzene, ortho-chloronitrobenzene, OCNB

Solutia Inc., 10500 Olive Boulevard, P.O. Box 68700, St. Louis, MO 63166-6709

FOR CHEMICAL EMERGENCY, SPILL, LEAK, FIRE, EXPOSURE, OR ACCIDENT
Call CHEMTREC - Day or Night - 1-800-424-9300, Toll free in the continental U.S., Hawaii, Puerto Rico, Canada, Alaska, or Virgin Islands. For calls originating elsewhere: 703-272-3387 (with code 601 847 8010)

For additional non-emergency information, call: 314-674-6651

2. COMPOSITION/INFORMATION ON INGREDIENTS

Component  CAS No  % by weight
o-Nitrochlorobenzene  98-78-3  99.0 - 100

3. HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW
Appearance and Odor: yellow oily solid with a sweet aromatic odor

WARNING STATEMENTS

DANGER
MAY BE FATAL IF SWALLOWED, INHALED OR ABSorbed THROUGH SKIN. CAUSES METHEMOGLOBINEMIA, DECREASING OXYGEN IN BLOOD

POSSIBLE HEALTH EFFECTS

Likely Routes of Exposure: skin contact and inhalation
This product is HAZARDOUS by all routes of exposure. Exposure to this product should be tightly controlled. This product has a high potential to cause changes in the blood cells which decrease the oxygen-carrying capacity of the blood. This condition is called methemoglobinemia. As the condition progresses, oxygen deficiency increases (cyanosis occurs), producing possible headaches, weakness, fatigue, dizziness, shortness of breath and unconsciousness. The appearance of methemoglobinemia may be delayed 8 to 12 hours after exposure.

**EYE CONTACT:** This product is no more than slightly irritating to the eye based on toxicity tests.

**SKIN CONTACT:** This product may be fatal if absorbed through the skin. This product is no more than slightly irritating to the skin based on toxicity tests.

**INHALATION:** This product may be fatal if inhaled.

**INGESTION:** This product may be fatal if swallowed.

Refer to Section 11 for toxicological information.

### 4. FIRST AID MEASURES

**POISON: GET MEDICAL ATTENTION. CONTACT A POISON CONTROL CENTER.**

Get patient to fresh air. Remove contaminated clothing but keep patient warm. Start decontamination and indicated treatment immediately. Rescuers must use care to prevent contact with the material.

**IF IN EYES OR ON SKIN:** Immediately flush with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. As soon as available, wash skin thoroughly with soap and water. Wash clothing thoroughly before reuse. Destroy contaminated shoes.

**IF INHALED:** remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen.

**IF SWALLOWED:** do NOT induce vomiting. Rinse mouth with water. Get medical attention. Contact a Poison Control Center. NEVER GIVE ANYTHING BY MOUTH TO AN UNCONSCIOUS PERSON.

NOTE TO PHYSICIANS: Call a Poison Control Center for guidance. If absorbed, this product may cause methemoglobinemia which reduces oxygen carrying capacity of blood. Methemoglobin spontaneously but slowly reverts to hemoglobin after termination of exposure. Asymptomatic patients with methemoglobin levels under 30% generally respond to bed rest, oxygen, and supportive care. Patients with symptoms or with methemoglobin greater than 30% may also respond to intravenous injection of metylene blue. See package insert for indications, dosage, and precautions. Repeat methemoglobin level one to two hours after injection to assess response. Thorough cleansing of contaminated skin, nails, and hair is essential to prevent continued absorption and methemoglobin formation.

### 5. FIRE FIGHTING MEASURES

**FLASH POINT:** 261 degrees F (127 degrees C)  
**METHOD:** Cleveland Open Cup

HAZARDOUS PRODUCTS OF COMBUSTION: Carbon monoxide (CO), carbon dioxide (CO2), hydrogen chloride (HCl), oxides of nitrogen, soot, smoke and chlorides.

**EXTINGUISHING MEDIA:** In case of fire, use water spray (foam), foam, dry chemical, or CO2.
UNUSUAL FIRE AND EXPLOSION HAZARDS: None known.

FIRE FIGHTING EQUIPMENT: Fire fighters and others expected to products of combustion should wear self-contained breathing apparatus. Equipment should be thoroughly decontaminated after use.

6. ACCIDENTAL RELEASE MEASURES

Keep people away. Keep upwind. Shut off leak if without risk. If necessary to enter spill area, wear self-contained breathing apparatus and full protective clothing including boots. Do not contain liquid until good vacuum liquid or shovel solid into dry, clean containers. Keep out of sewers, water sheds, and waterways. If not possible, notify pollution control authorities promptly.

Refer to Section 13 for disposal information and Section 15 for reasonable quantity information.

7. HANDLING AND STORAGE

Handling:
- Do not get in eyes, on skin, or on clothing.
- Do not breathe vapor.
- Do not taste or swallow.
- Keep container closed.
- Use only with adequate ventilation or wear a suitable respirator.
- Wash thoroughly after handling.

Empty container retains vapor and product residue. Observe all labeled safe-guards until container is destroyed.

Refer to Section 13 for product and container disposal.

Storage:
- Store in a cool, well ventilated place away from foodstuffs, reducing agents, acids and strong oxidizing agents.

8. EXPOSURE CONTROLS/PERSOAL PROTECTION

EYE PROTECTION: This product does not cause significant eye irritation or eye toxicity requiring special protection. Use good industrial practice to avoid eye contact.

SKIN PROTECTION: This product may be absorbed through the skin causing methemoglobinemia. Care must be taken to prevent skin contact. Wear chemical resistant gloves and boots, and protective long sleeved clothing when there is potential for skin contact. If handled as a liquid, wear a face shield and chemical resistant apron when splashing is likely. Wash contaminated skin immediately and thoroughly with soap and water. Launder contaminated clothing and clean protective equipment before re-use. Provide a safety shower at any location where skin contact can occur. Wash thoroughly after handling.

RESPIRATORY PROTECTION: Avoid breathing dust, vapor, and mist. Use NIOSH/MSHA approved respiratory protection equipment (type facepiece recommended) when airborne exposure is excessive. If used, full facepiece respirators need a face shield. Consult respirator manufacturer to determine appropriate type equipment for given application. Observe respirator use limitations specified by NIOSH/MSHA or the manufacturer. Respiratory protection programs must comply with 29 CFR 1910.134.

79
VENTILATION: Provide natural or mechanical ventilation to minimize exposure. If practical, use local mechanical exhaust ventilation at sources of air contamination such as open process equipment.

AIRBORNE EXPOSURE LIMITS:

<table>
<thead>
<tr>
<th>Product/Component</th>
<th>OSHA PEL</th>
<th>ACGIH TLV</th>
</tr>
</thead>
<tbody>
<tr>
<td>ortho nitrochlorobenzene</td>
<td>None established</td>
<td>None established</td>
</tr>
</tbody>
</table>

* Solvias has adopted an internal exposure guideline of 1 mg/m³, 8-hour time-weighted average. Skin for this product. Skin notation means that skin absorption of this material may add to the overall exposure. Avoid skin contact.

8. PHYSICAL AND CHEMICAL PROPERTIES

<table>
<thead>
<tr>
<th>Property</th>
<th>Specification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chemical Formula</td>
<td>C7H5ClNC</td>
</tr>
<tr>
<td>Appearance</td>
<td>Yellow, oily solid</td>
</tr>
<tr>
<td>Odor</td>
<td>Sweet nitro odor</td>
</tr>
<tr>
<td>Boiling Point @ 760 mm Hg</td>
<td>248 degrees C</td>
</tr>
<tr>
<td>Melting Point</td>
<td>31.8 degrees C</td>
</tr>
<tr>
<td>Vapor Pressure</td>
<td>0.4 mm Hg @ 25 degrees C</td>
</tr>
<tr>
<td>Viscosity</td>
<td>1.664 cpois @ 105 degrees C</td>
</tr>
<tr>
<td>Specific Gravity</td>
<td>1.351 @ 25/15.5 degrees C</td>
</tr>
<tr>
<td>Solubility in Water</td>
<td>0.028 % by wt @ 20 degrees C</td>
</tr>
<tr>
<td>Vapor density</td>
<td>3.44 (air = 1)</td>
</tr>
</tbody>
</table>

NOTE: These physical data are typical values based on material tested but may vary from sample to sample. Typical values should not be confused as a guaranteed analysis of any specific lot or specification for the product.

10. STABILITY AND REACTIVITY

STABILITY: Product is stable under normal conditions of storage and handling.

MATERIALS TO AVOID: Strong oxidizing agents; acids; reducing agents.

CONDITIONS TO AVOID: Long-term storage in steel @ 140 degrees C darkens this product.

HAZARDOUS DECOMPOSITION PRODUCTS: Decomposition starts at 283 degrees C.

HAZARDOUS POLYMERIZATION: Does not occur.

11. TOXICOLOGICAL INFORMATION

NOTE: Human experience has shown that man is much more sensitive to methamoglobinemia caused by these type of compounds than the rat or the rabbit.
Data from Solina single-dose (acute) animal studies with this material are given below:

<table>
<thead>
<tr>
<th>Route</th>
<th>Toxicity</th>
<th>Dose</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oral LD50</td>
<td>Slightly Toxic</td>
<td>(Rat 560 mg/kg)</td>
</tr>
<tr>
<td>Dermal LD50</td>
<td>Moderately Toxic</td>
<td>(Rabbit 400 mg/kg)</td>
</tr>
<tr>
<td>Eye irritation</td>
<td>Slightly irritating</td>
<td>(Rabbit (HIS) 4.1 on a scale of 0-8)</td>
</tr>
<tr>
<td>Skin irritation</td>
<td>Nonirritating</td>
<td>(Rabbit (HIS) 0.0 on a scale of 0-8)</td>
</tr>
</tbody>
</table>

Rats were exposed by inhalation to this product's vapor concentrations of 10, 30, or 60 mg/l for 6 hours/day, 5 days/week for four weeks. An increase in blood erythrocyte and a decrease in hemoglobin, hematocrit, and red blood cell count values were noted in the mid- and high-concentration groups. Mean spleen and liver weights were also elevated in the mid- and high-exposure groups. Microscopic changes observed in the spleen included extramedullary hematopoesis in the mid- and high-exposure groups and an increase in hemosiderosis at all exposure levels.

In a limited oncogenicity study, this product was fed to the diet to male rats and mice of both sexes. One group of rats (low-dose) received diets containing 1,000 ppm of product for 6 months, 500 ppm for the following 12 months, and control diets for an additional 6 months. A second group of rats (high-dose) were given 2,500 ppm in the diet for 5 and 6 months, 1,000 ppm for the following 12 months, and control diets for an additional 6 months. Male and female mice were administered 3,000 or 6,000 ppm in the diet for 10 months; dietary concentrations were reduced to dosages of 1,500 or 3,000 ppm for the next 10 months. For the final 3 months, these animals received control diets. An increase in multiple tumors was reported for the low-dose rats; no increase in tumors was reported for the high-dose rats. An increase in hepatocellular carcinoma was found in female mice of both the low- and high-dose groups and in male mice only at the low-dose level. Although an increase in tumors was reported in low-dose male rats and mice, no dose-related effect was present for the males of either species.

Authors of this report considered the significance of the results of this screening study to be equivocal due to the low number of animals evaluated, the high incidence of spontaneous tumors, and the lack of a dose response in some groups.

This product is not administered by gavage to female rats at dosages of 5, 25, 75 or 150 mg/kg/day on days 6 through 16 of gestation. A second, specific study was conducted in which rats received either 100 mg/kg in corn oil or corn oil (control) on a comparable regimen. Due to significant maternal toxicity and mortality at 150 mg/kg, no fetal evaluations were performed at this dosage. No teratogenic, embryotoxic or fetotoxic effects were observed at dosages of 100 mg/kg or less. Reduced maternal body weight gain and/or food consumption were noted at 75 and 150 mg/kg/day.

No mutagenic or genotoxic potential was observed in an in vitro Chinese hamster ovary (CHO) cell line mutation assay or in a rat hepatocyte primary culture/ARA repair assay. Studies in the scientific literature report both positive and negative results in microbial mutagenesis assays with Salmonella typhimurium strains. This product has also been reported to cause DNA damage in liver and kidney cells of rats after a single intraperitoneal dosage of 50 mg/kg. Another group of investigators reported that this product did not induce sex-linked recessive lethal mutations in Drosophila. Following oral administration of this product to rabbits, the major urinary metabolites were identified as 2-chloro-3-nitrophenol and 2-chloro-3-nitrophenol analogues.

12. ECOLOGICAL INFORMATION

The following data have been classified using the criteria adopted by the European Economic Community (EEC) for aquatic organism toxicity. A legend summarizing the classification scheme appears below:

<table>
<thead>
<tr>
<th>Waterbody</th>
<th>48-hr LC50 (mg/l)</th>
<th>96-hr EC50 (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Freshwater</td>
<td>48-hr LD50 (mg/l)</td>
<td>96-hr LC50 (mg/l)</td>
</tr>
<tr>
<td>Marine</td>
<td>41 mg/l, Harmful</td>
<td>41 mg/l, Harmful</td>
</tr>
</tbody>
</table>
13. DISPOSAL CONSIDERATIONS

Product Disposal:
This material when discarded is not a hazardous waste as that term is defined by the Resource, Conservation and Recovery Act (RCRA), 40 CFR 261. Dispose of by incineration or recycle in accordance with local, state and federal regulations. Consult your attorney or appropriate regulatory officials for information on such disposal.

This product should not be dumped, spilled, chased or washed into oceans or public waterways.

Container Disposal:
Soluble recommends that empty containers be disposed of using the best available technology (incineration). Dispose of containers in accordance with local, state and federal regulations. Soluble does NOT recommend these containers be reused for any services.

14. TRANSPORT INFORMATION

The data provided in this section is for information only. Please apply the appropriate regulations to properly classify your shipment for transportation.

US DOT Classification: Chlordrin/obenzene, 6.1, UN1676, II

US DOT Label: POISON

IMDG Code: See US DOT

IATA/ICAO: See US DOT

15. REGULATORY INFORMATION

TSCA Inventory: Listed.
SAFETY DATA SHEET

 expiry date: 01/01/98

ORTHOCYCLIC HDNODIZING

MSDS No.: 00266223

SAFETY DATA SHEET

Immediate, delayed
Section 102 Extremely Hazardous Substances: Not Applicable
Section 112 Toxic Chemical(s): Not Applicable

CERCLA Reportable Quantity: Not Applicable

California Proposition 65: Not Applicable

This product is considered a hazardous chemical under OSHA.

Refer to Section 13 for RCREA classification

OTHER INFORMATION

Reason for revision: Routine review and company name change to Solviva. Supersedes MSDS dated 01/01/98

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Although the information and recommendations set forth herein (hereinafter "Information") are presented in good faith and believed to be correct as of the date hereof, Solviva Inc. makes no representations as to the completeness or accuracy thereof. Information is supplied upon the condition that the person receiving same will make their own determination as to its suitability for their purposes prior to use. In no event will Solviva Inc. be responsible for any damages of any nature whatsoever resulting from the use of or reliance upon Information. No representations or warranties, either express or implied, of merchantability, fitness for a particular purpose or of any other nature are made hereunder with respect to Information or the product to which Information refers.

Ortho-Nitrochlorobenzene.885(2)656
1. CHEMICAL PRODUCT and COMPANY IDENTIFICATION

Product Name: 2-ETHYLHEXYLAMINE
Product Code: 627911
HSDS Number: 140

Celanese Division / Celanese, Ltd.
15950 W. LBJ Freeway
PO Box 819005
Dallas, TX 75288-6005
USA

TRANSPORTATION EMERGENCY PHONE NUMBER (24 HOURS/DAY):
In USA, call: ...........800 474 9306
Outside USA, call*: ....212 643 7517,
*collect calls accepted

2. COMPOSITION / INFORMATION on INGREDIENTS

<table>
<thead>
<tr>
<th>COMPONENT</th>
<th>CAS NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-ETHYLHEXYLAMINE*</td>
<td>104-75-6</td>
</tr>
<tr>
<td>2-ETHYLCETANOL*</td>
<td>104-76-7</td>
</tr>
</tbody>
</table>

*OSHA hazardous according to 29 CFR 119, 120

3. HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW:
2-Ethylhexylamine is a clear to pale yellow liquid with an
ammonia-like odor.

CAUTION:
Combustible liquid and vapor (IP: 118 F, TOG)
Vapor is heavier than air and can travel considerable
distance to a source of ignition and flash back
In a confined space, overpressurization and container
failure could result

POTENTIAL HEALTH EFFECTS

ROUTE OF EXPOSURE:
Skin, eyes, inhalation, ingestion.

* TRANSPORTATION EXCERPT: ............(800) 434-3100
PRODUCT EXCERPT: .............(800) 885-6135
Celanese, Inc. Houston
PRODUCT INFORMATION: ..........(713) 641-4000
(713) 832-4155, Ext. 900
3. HAZARDS IDENTIFICATION (Continued)

IMMEDIATE EFFECTS

SKIN:
Liquid can cause severe skin effects, including burns and skin corrosion.

EYES:
Contact with liquid can be corrosive to the eyes, resulting in irreversible damage. Contact with vapor can cause eye irritation. Contact with excessive vapors may cause transient visual disturbances characterized by mildly blurred vision and a blue-gray disorientation of sight. This effect is commonly referred to as blue haze, or halo vision, with halo vision getting its name from the appearance of a halo when looking at light sources. These effects are due to mild central irritation and edema and normally disappear several hours after exposure ceased.

INHALATION:
Can cause irritation of the respiratory system. Exposure symptoms may include coughing, chest pain, breathing difficulty, loss of muscle coordination, headache, dizziness and nausea. Can cause pulmonary edema (accumulation of fluids in the lungs); signs and symptoms can be delayed for several hours.

INGESTION:
May cause irritation or burns of the mouth, throat, stomach and esophagus. May cause nausea, vomiting and diarrhea.

MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE:
significant exposure to this chemical may adversely affect people with chronic disease of the respiratory system and skin. May provoke asthmatic response in persons with asthma, who are particularly sensitive to respiratory irritants.

FOR FURTHER INFORMATION, SEE:
Section 4 - First Aid Measures
Section 5 - Fire Fighting Measures
Section 6 - Accidental Release Measures
Section 8 - Exposure Control/Personal Protection
Section 9 - Physical and Chemical Properties
Section 10 - Stability and Reactivity
4. FIRST AID MEASURES

SKIN:
For 15 minutes, wash with large amounts of 5% acetic acid (aqueous), alternating with soap and water. The acetic acid solution may be discontinued once the stickiness is gone. In all cases, plain water rinsing for 5 minutes is the final step. Significant signs of irritation should be seen by a physician.

EYES:
Flush eyes with water for at least 15 minutes. Contact a physician immediately.

INHALATION:
Remove patient from contaminated area. If breathing has stopped, give artificial respiration, then oxygen if needed. Contact a physician immediately.

INGESTION:
Patient should be made to drink large quantities of water. Do not induce vomiting. Contact a physician immediately.

NOTE TO PHYSICIANS:
Treat as an alkaline substance (similar to ammonia).

5. FIRE FIGHTING MEASURES

FLAMMABLE PROPERTIES
FLASHPOINT CLOSED CUP: 127 °F (53 °C)

UPPER EXPLOSIVE LIMIT: 19.8 PERCENT
In air by volume
LOWER EXPLOSIVE LIMIT: 0.67 PERCENT
In air by volume

AUTOIGNITION TEMP: 435 °F (224 °C)

HAZARDOUS PRODUCTS OF COMBUSTION:
In the presence of sufficient oxygen, combustion may produce oxides of nitrogen and carbon dioxide. Nitrogen oxide can react with water to produce nitric acid. Combustion under oxygen-starved conditions may produce numerous toxic products including carbon monoxide, cyanides and nitriles.

II TRANSPORTATION EMERGENCY: (800) 426-1308
PRODUCT INFORMATION: (912) 447-1000
86
5. FIRE FIGHTING MEASURES (Continued)

EXTINGUISHING MEDIA
Use carbon dioxide or dry chemical for small fires; adequ.: type aqueous film-forming foam or water spray for large fires. Water may be ineffective but should be used to cool fire-exposed structures and vessels.

FIRE FIGHTING INSTRUCTIONS:
If potential for exposure ... or products of combustion exists, wear complete personal protective equipment, including self-contained breathing apparatus with full facepiece operated in pressure demand or other positive pressure mode. Use water spray to cool fire-exposed structures and vessels. Material creates a special hazard because it floats on water.

6. ACCIDENTAL RELEASE MEASURES

Eliminate ignition sources. Avoid eye or skin contact; see "Section 8 - Exposure Controls/Personal Protection" for respirator information. Place leeking containers in well-ventilated area with spill containment. If fire potential exists, blanket spill with alcohol-type aqueous film-forming foam or use water spray to disperse vapors. Contain spill to facilitate clean-up. Clean-up methods may include absorbent materials, vacuum truck, etc. Avoid runoff into storm sewers and ditches which lead to natural waterways.

Call the National Response Center (800 424-8802) if the quantity (of any component) spilled is equal to or greater than the reportable quantity (RQ) under CERCLA 'Superfund': 100 kg/day.

For more information, see "Section 15 - Regulatory Information".

In U.S.: CERCLA - 24 HR/365 DAY 800 424-8802
PRODUCT INFORMATION: 813-665-6713
PRODUCT INFORMATION: 813-665-6000
7. HANDLING and STORAGE

Store in a cool, well-ventilated area. Keep away from heat, sparks and flame. Keep containers closed when not in use. Always open containers slowly to allow any excess pressure to vent. Use only DOT-approved containers. When transferring, follow proper grounding procedures. Use with adequate ventilation. Avoid breathing vapor. Avoid contact with eyes, skin and clothing. Wash thoroughly with soap and water after handling. Change soaked clothing thoroughly. Discard contaminated leather clothing. Electrical installations should be in accordance with Article 501 of the National Electrical Code for Class I locations. Exhaust systems, transfer pumps and other handling equipment must be equipped with explosion-proof motors.

8. EXPOSURE CONTROLS / PERSONAL PROTECTION

ENGINEERING CONTROLS:

General or dilution ventilation is frequently insufficient as the sole means of controlling employee exposure. Local ventilation is usually preferred.

PROTECTIVE EQUIPMENT

A safety shower and eye flush should be readily available.

SKIN:

For operations where spills or splashing can occur, use chemical protective clothing, including gloves (neoprene or rubber) and boots.

EYES:

Chemical safety goggles.

INHALATION:

Based on contamination levels and working limits of the respirator, use a respirator approved by MSHA or following are the minimum recommended equipment:

× 10 and ≤ 10 ppm - Air-purifying respirator with full facepiece and organic vapor cartridges or air-purifying full facepiece respirator with an organic vapor canister or a full facepiece powered air-purifying respirator fitted with organic vapor cartridges.

TRANSMISSION EMERGENCY: (800) 828 4155 IN U.S., 24 HOURS/7 DAYS
PRODUCT EMERGENCY: (800) 828 4155Celifene, 24 HOURS/7 DAYS
PRODUCT INFORMATION: (872) 423 4000 [24/7 AN TO 4:15 PM, CST]
INHALATION: (Continued)

> 100 ppm < 1000 ppm - Positive-pressure full facepiece
  supplied-air respirator, or continuous-flow full facepiece
  supplied-air respirator.

> 1000 ppm or unknown concentration (such as in emergency
  cases) - Positive-pressure self-contained breathing apparatus
  with full facepiece. Positive-pressure supplied-air respirator
  with full facepiece equipped with an auxiliary
  positive-pressure self-contained breathing apparatus-escape
  system.

EXPOSURE GUIDELINES:

No OSHA REL or ACGIH TLV has been established.

Immediately Dangerous to Life or Health (IDLH) level:
No value established.

5. PHYSICAL AND CHEMICAL PROPERTIES

APPEARANCE: Clear to pale yellow liquid.

ODOR: Ammonia-like odor.

PHYSICAL STATE: Liquid.

VAPOR PRESSURE: 1.2 mm Hg

VAPOR DENSITY: 4.49,

Boiling Point: Calculated.

Freezing Point: -27.9°C [-65°F]

SOLUBILITY: Nearly insoluble.

SPECIFIC GRAVITY: 0.792

% VOLATILES: No information.

% VOLATILES: 100.0% by volume.

MOLECULAR WEIGHT: 129.25
10. STABILITY and REACTIVITY

CHEMICAL STABILITY:
- Stable.

CONDITIONS TO AVOID:
- Heat, sparks, flame.

INCOMPATIBILITY:
- May react vigorously with oxidizing agents; include perchlorates, nitrates, peroxides, chlorates, nitric acid, chromic acid, halogens and hydrogen peroxide. A reaction accompanied by a large heat release occurs when mixed with acids; heat generated may cause boiling, splashing and splattering. Avoid nitrates or sources of NOx; may form nitramines which are suspect carcinogens. Corrosive to copper and copper-containing alloys.

HAZARDOUS DECOMPOSITION PRODUCTS:
- In the presence of sufficient oxygen, combustion may produce oxides of nitrogen and carbon dioxide. Nitrogen oxides can react with water to produce nitric acid. Combustion under oxygen-starved conditions may produce numerous toxic products including carbon monoxide, cyanides and nitriles.

HAZARDOUS POLUTMENTATION:
- Will not occur.

11. TOXICOLOGICAL INFORMATION

Oral LD50 : 450 mg/kg (rats); Moderately toxic to animals.

Dermal LD50 : 600 mg/kg (rabbits); Moderately toxic to animals by absorption.

Inhalation LC50 : > 125 and < 250 ppm (rats, 4 hr); Moderately toxic to animals.
12. ECOLOGICAL INFORMATION

No information

13. DISPOSAL CONSIDERATIONS

All notification, clean-up or disposal should be carried out in accordance with federal, state and local regulations. Preferred methods of waste disposal are incineration or biological treatment in federal/state approved facility.


14. TRANSPORT INFORMATION

Shipping name: 2-Ethylhexylamine
Hazard class: 3, Flammable Liquid
Subsidary hazard: 8, Corrosive Liquid
United Nations no.: UN2776
Packing group: III
North American ER Guide: 132
DOT Reportable Quantity (RD): None assigned

15. REGULATORY INFORMATION

RECIPIENT MUST COMMUNICATE ALL PERTINENT INFORMATION HEREBIN TO EMPLOYEES AND CUSTOMERS.

STATE REGULATIONS

The following chemicals associated with the product are subject to the right-to-know regulations in these states:

2-ETHYLHEXYLAMINE (104-75-6): FL, MA, NJ, PA

2-ETHYLHEXANOL (104-26-7): FL, MA, PA

U.S. FEDERAL REGULATIONS

We certify that all components are either on the TSCA inventory or qualify for an exemption.
16. OTHER INFORMATION

The following sections have been revised:

SECTION 14: TRANSPORT INFORMATION

The information contained herein is accurate to the best of our knowledge. We do not suggest or guarantee that any hazards listed herein are the only ones which exist. Celanese, Ltd. makes no warranty of any kind, express or implied, concerning the safe use of this material or in its process or in combination with other substances. Effects can be aggravated by other materials and/or this material may aggravate or add to the effects of other materials. This material may be released from gas, liquid, or solid materials made directly or indirectly from it. User has the sole responsibility to determine the suitability of the materials for any use and the manner of use contemplated. User must meet all applicable safety and health standards, our knowledge. We do not suggest or guarantee that any hazards listed herein are the only ones which exist.

TRANS. TRANSPORTATION BY RAIL: (CAUTION) 406 B3X 1600

TRANS. TRANSPORTATION BY WATER: (CAUTION) 406 B3X 1600

TRANS. TRANSPORTATION BY AIR: (CAUTION) 406 B3X 1600

92