

---

**THE MASSACHUSETTS  
TOXICS USE REDUCTION INSTITUTE**

---

**LEACH & GARNER COMPANY  
NORTH ATTLEBORO, MASSACHUSETTS**

**AMMONIA REDUCTION FOR HEAT TREAT  
FURNACE ATMOSPHERES**

**TOXICS USE REDUCTION INSTITUTE  
CLEANER TECHNOLOGY  
DEMONSTRATION SITES & MATCHING GRANTS  
PROGRAM**

**Technical Report No. 43**

**1997**

University of Massachusetts Lowell

**Leach & Garner Company, General Findings Division  
North Attleboro, Massachusetts**

**Ammonia Reduction for Heat Treat Furnace Atmospheres**

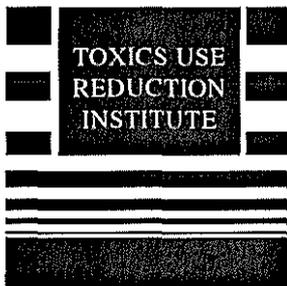
Susan A Mayo, Environmental/Safety Manager  
Leach & Garner Company, General Findings Division

Jodie Siegel, Project Manager  
Toxics Use Reduction Institute

**The Toxics Use Reduction Institute  
Cleaner Technology Demonstration Sites  
and Matching Grants Program**

**The Toxics Use Reduction Institute  
University of Massachusetts Lowell**

**1997**



All rights to this report belong to the Toxics Use Reduction Institute. The material may be duplicated with permission by contacting the Institute.

The Toxics Use Reduction Institute is a multi-disciplinary research, education, and policy center established by the Massachusetts Toxics Use Reduction Act of 1989. The Institute sponsors and conducts research, organizes education and training programs, and provides technical support to promote the reduction in the use of toxic chemicals or the generation of toxic chemical byproducts in industry and commerce. Further information can be obtained by writing the Toxics Use Reduction Institute, University of Massachusetts Lowell, One University Avenue, Lowell, Massachusetts 01854.

©Toxics Use Reduction Institute, University of Massachusetts Lowell

## Preface

In its 1997 fiscal year, the Massachusetts Toxics Use Reduction Institute combined the Cleaner Technology Demonstration Sites and Industry Matching Grants programs. The goal of the combined program is to provide companies with the opportunity to test and demonstrate new cleaner technologies as well as to promote the adoption of cleaner technologies by Massachusetts industry. Five companies were selected as demonstration sites to showcase the implementation of technologies that embrace the concepts and principles of toxics use reduction. The program, which included a series of visits to the facilities and related presentations and publications, allowed individuals and firms to observe and assess their value first-hand. Site visits were open to industry, environmental groups, community groups, the media and others.

Associate sponsors of the program included the Massachusetts Office of Technical Assistance for Toxics Use Reduction, the Executive Office of Environmental Affairs, the Department of Environmental Protection, the Environmental Protection Agency of New England, and the Associated Industries of Massachusetts.

This program will continue to provide grants to recognize the many companies across the Commonwealth that have used toxics use reduction and cleaner technologies while enhancing their firm's competitiveness.

The following report is an in-depth analysis of the cleaner technology demonstrated at Leach & Garner Company General Findings Division, North Attleboro, Massachusetts.

## Notice

This report has been reviewed by the Institute and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Toxics Use Reduction Institute, nor does the mention of trade names or commercial products constitute endorsement or recommendation for use.

## TABLE OF CONTENTS

1. INTRODUCTION .....	1
1.1 Project Intent.....	1
2. DESCRIPTION OF TECHNOLOGY.....	1
3. APPLICATION & TRANSFERABILITY .....	4
3.1 Application.....	4
3.2 Transferability .....	5
4. ENVIRONMENTAL & OCCUPATIONAL HEALTH ASSESSMENT .....	6
4.1 Chemical Hazards.....	6
4.2 Air Emissions.....	6
4.3 Toxic Chemical Use per Unit of Product .....	6
5. ECONOMIC IMPLICATIONS.....	6
5.1 Capital Expenditures .....	6
5.2 Regulatory Costs for Toxics Use.....	7
6. INSTALLATION AND VALIDATION .....	7
7. BARRIERS AND RESOURCES ENCOUNTERED.....	8

## LIST OF FIGURES

FIGURE 1: AMMONIA DISSOCIATOR .....	2
FIGURE 2: BOC MODEL II FURNACE CONTROL PANEL.....	3
FIGURE 3: TYPICAL ANNEALING/SOLDERING FURNACE.....	5

## **1. INTRODUCTION**

Leach & Garner established its manufacturing operations in the United States in 1899 and serves major jewelry markets throughout the world. The General Findings Division operates under SIC codes 3911 and 3915, and reports on several chemicals under TURA. Prior to TURA, General Findings had successfully reduced the use of sulfuric acid below reporting thresholds, eliminated freon degreasing and replaced cadmium bearing solders.

Members of the TUR Team include: Joe Sisto, Vice President/General Manager; DP Agarwal, Vice President Technology; Bill Bliss, Production Manager; Grig Raykhtsaum, Metallurgist; Marinko Markic, Director of New Technology; Jim Bolanis, Materials Engineer; Armand Richard, Project Engineer; Rick Shepard, Chief Wastewater Treatment Operator; and Sue Mayo, Environmental/Safety Manager.

### **1.1 Project Intent**

Heat treating is essential to various metal forming processes, keeping the metal soft enough to be twisted, stamped, bent, rolled, etc. The furnaces at General Findings are used for soldering in addition to heat treatment. Jewelry components are assembled in graphite trays (known as "coals"), and as they pass through the solder furnace components are bonded together (as opposed to each piece being hand-soldered). At elevated temperatures in these furnaces, metals react with air to form oxides. A protective atmosphere must be maintained to achieve the desired bright and clean finish.

This atmosphere is achieved by dissociating (splitting) anhydrous ammonia into its basic components: 75% hydrogen, and 25% nitrogen by volume. Successful tests with lower hydrogen levels were performed in-house by diluting the dissociated ammonia ("DA") with nitrogen. (See Table 1). Although this project will not decrease anhydrous ammonia consumption below reporting thresholds, the significant reduction in ammonia usage reduces the potential for worker exposure and environmental impact. This project also allows General Findings to plan for an increase in production activity without additional regulatory requirements.

## **2. DESCRIPTION OF TECHNOLOGY**

It has been a practice for many years in the jewelry industry to use hydrogen-containing atmospheres in the process of annealing and soldering precious metal alloys. High hydrogen content gas mixtures provide a strong deoxidizing potential, which allows for both the removal of any surface oxides present and prevents oxide formation during heat treatment.

In the past General Findings has used a combination of process gases, predominantly nitrogen and hydrogen. This is accomplished by dissociating commercial grade anhydrous ammonia (NH<sub>3</sub>). Ammonia vapor is processed through a retort<sup>1</sup> at elevated controlled temperatures (1650°F - 1800°F). The ammonia molecule is then “cracked” or split into its major constituents of nitrogen and hydrogen, as shown below:



This reaction yields a gas mixture fixed at 25% nitrogen and 75% hydrogen. The intention of this project is to further dilute the available hydrogen gas from this reaction by adding nitrogen.

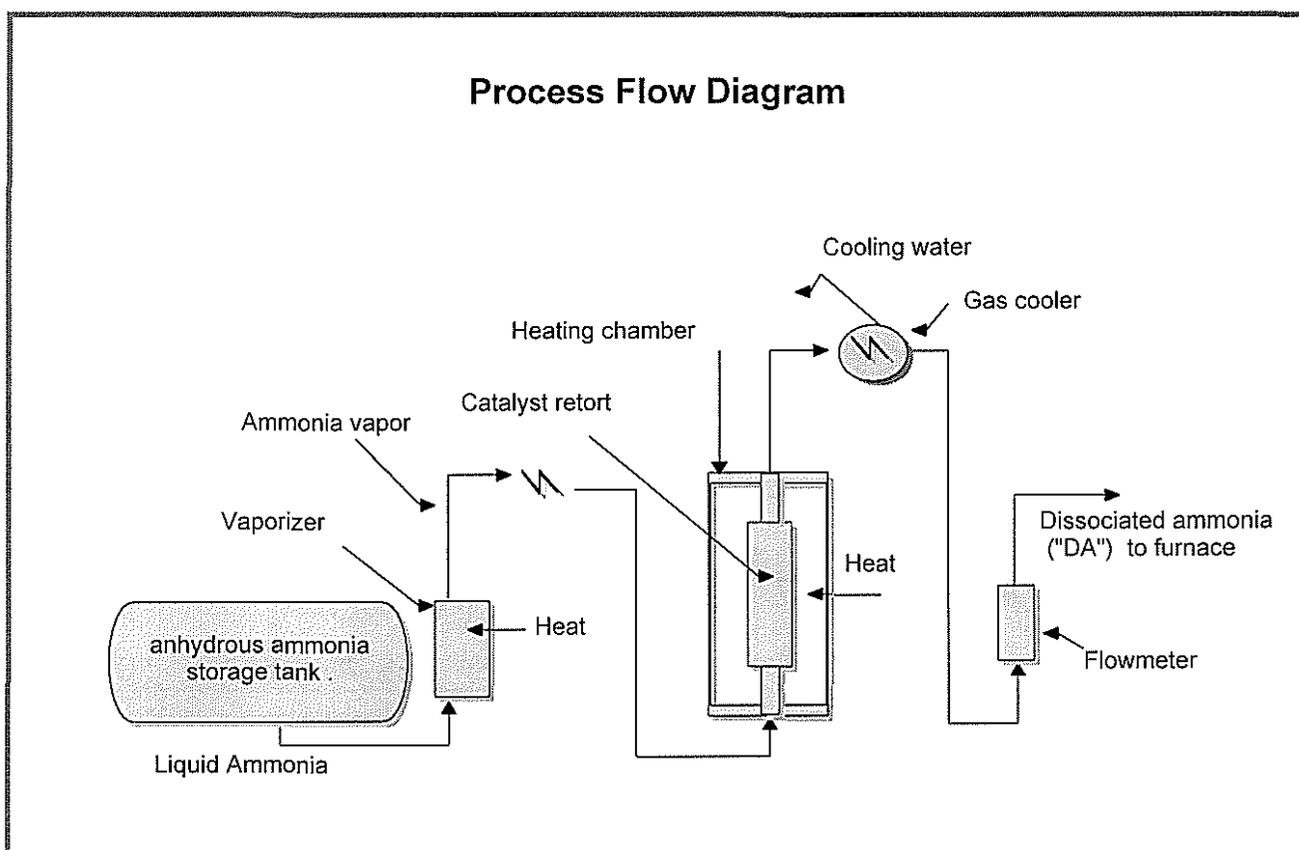
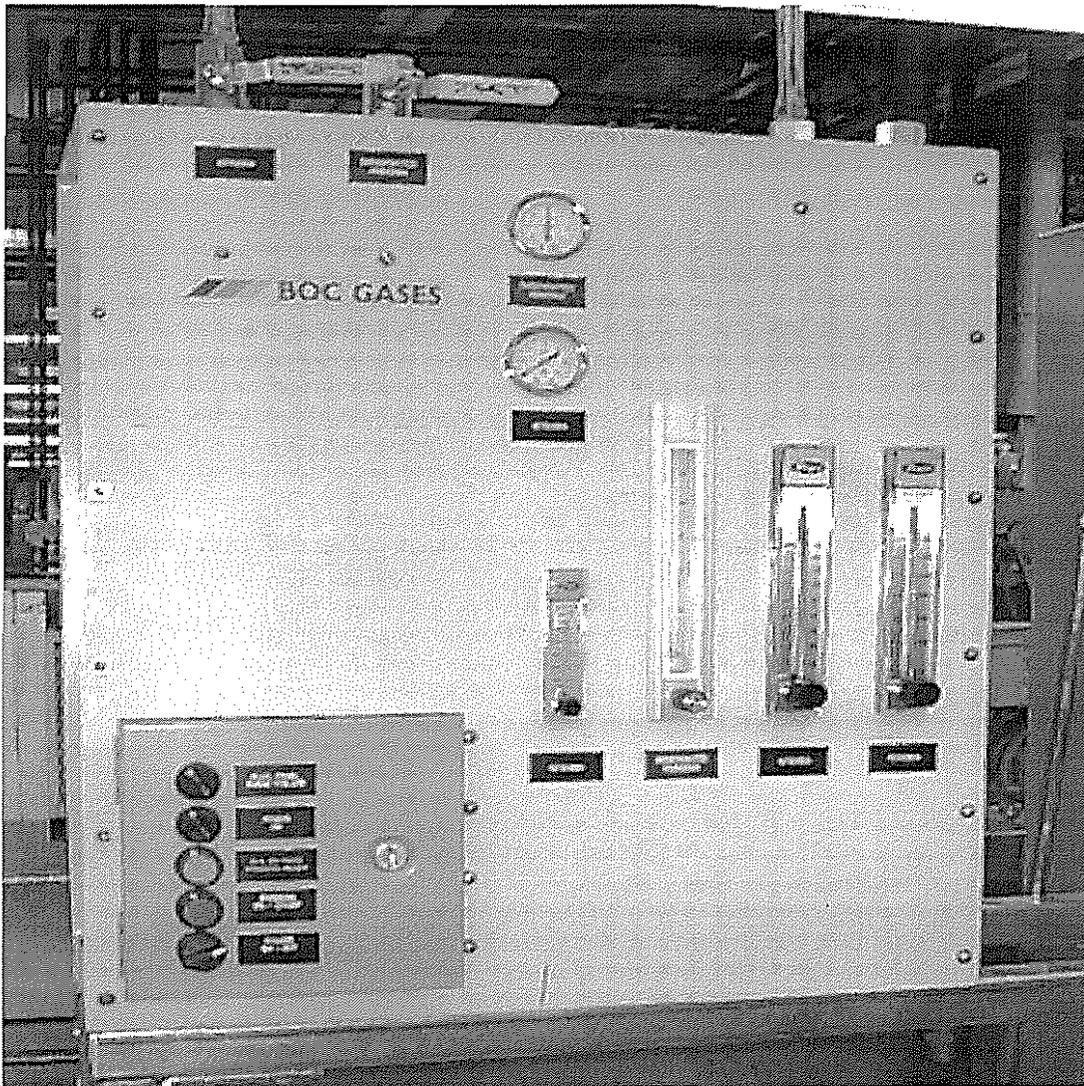


Figure 1: Ammonia Dissociator<sup>2</sup>

The diagram in Figure 1 shows the process flow for the dissociation of the anhydrous ammonia. Furnace control panels, such as the one shown in Figure 2, were purchased from BOC Gases to mix various combinations of “cracked” ammonia and metallurgical grade nitrogen.

<sup>1</sup> A “retort” is a containment vessel with a catalyst.

<sup>2</sup> *Metals Handbook, Ninth Edition, Volume 4, Heat Treating, 1981, page 410.*



**Figure 2: BOC Model II Furnace Control Panel**

Total hydrogen content will range between 20% & 37.5%, instead of the 75% found in "DA". This will reduce the usage of "cracked" ammonia consumption by 50% to 73% for a given volume. Table 1 shows a comparison of flow rates of "cracked" ammonia and nitrogen, at a total of 150 cubic feet per hour. These hydrogen levels are still considered in the explosive range for combustible gases and should be handled accordingly.

**Table 1- Flow Rates**

<b>Hydrogen Content</b>	<b>Cracked Ammonia (cfh)</b> N <sub>2</sub> + 3H <sub>2</sub>	<b>Nitrogen (cfh)</b> N <sub>2</sub>	<b>Total Volume (cfh)</b>
20%	40	110	150
25%	50	100	150
30%	60	90	150
35%	70	80	150
36%	72	78	150
37%	74	76	150
37.5%	75	75	150

### 3. APPLICATION & TRANSFERABILITY

Leach & Garner had previously explored replacing “DA” with separate nitrogen and hydrogen systems, with the gases mixed prior to entering the furnace. With the large volume of atmospheric gas needed, a liquid hydrogen tank would have been required. Due to safety concerns and space requirements this option was rejected.

#### 3.1 Application

This project will accomplish essentially the same thing by diluting the “DA” with nitrogen. The nitrogen and dissociated ammonia flow to a specially designed mixing control panel (see Figure 2), where the gases are mixed to the desired percentages. The initial control panels have a range of 40-400 cfh nitrogen, 10-100 cfh nitrogen (exit end of furnace) and 10-250 cfh dissociated ammonia, and meet NFPA 86C (National Fire Protection Association) requirements. The 40-400 cfh nitrogen is used for purging and to mix with the “DA” for an atmospheric gas, the 10-100 cfh nitrogen at the exit end of the furnace acts as a curtain to keep the protective atmosphere in and air drafts out of the furnace. See Figure 3 for an example of a typical belt furnace.

Due to the large volume of nitrogen used to dilute the “DA,” the 1,500 gallon nitrogen tank was replaced with a 6,000 gallon tank. This also required a new, larger cement pad. A telemetry unit allows the scheduling center to “call-up” the tank to check volume levels and a low level alarm automatically sends a signal to the scheduling center.

Initially the “DA” will be diluted 50% with nitrogen to provide a 37.5% hydrogen atmosphere. As testing proceeds, General Findings will further reduce the hydrogen to between 20% and 37.5% by volume. A hydrogen mix of 5%-10% could be utilized, but a 20%-37.5% mix will be used to maintain the burnoff flame on the entrance end of the furnace as an operator safety precaution. At least 20% hydrogen is required to maintain a visible flame, which indicates hydrogen flow through the furnace and is a constant visual feedback that the operators are used to.

### 3.2 Transferability

This application could easily be used at another facility that uses dissociated ammonia for a protective furnace gas atmosphere where the mixing of nitrogen and "DA" may be done with BOC or similar panels. Although each furnace and application is unique, BOC Gases has provided vital technical support and experienced troubleshooting skills to enable a smooth start-up. Typical applications including precious metal annealing, joining and hardening are:<sup>3</sup>

#### Annealing

- Ferrous and Non-Ferrous
- Stainless Steel
- Spheroidizing
- Lamination Decarburization

#### Joining

- Carbon Steel Brazing
- Stainless Steel Brazing
- Glass to Metal Sealing
- Ceramic Metallizing

#### Powder Metallurgy

- Ferrous and Non-Ferrous Sintering
- Carbide Sintering
- Powder Reduction and Annealing

#### Hardening

- Low and High Carbon Steels
- High Alloy and Tool Steels
- Carbon Restoration

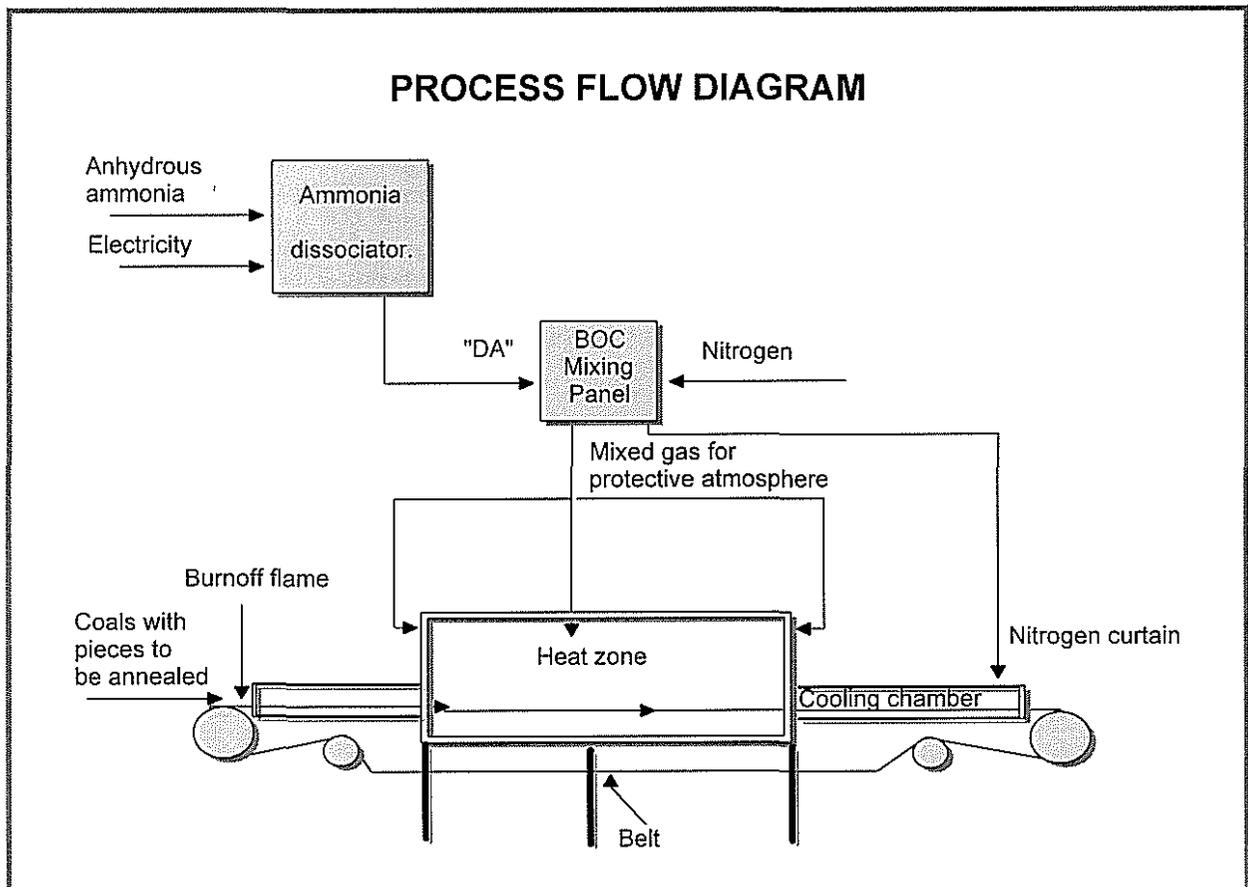


Figure 3: Typical Annealing/Soldering Furnace

<sup>3</sup> Air Products, Nitrogen-based Atmosphere System Flow Control Panels, Models 3000H/3000M, 1984.

## **4. ENVIRONMENTAL & OCCUPATIONAL HEALTH ASSESSMENT**

### **4.1 Chemical Hazards**

Ammonia, CAS # 7664-41-7, is listed as an extremely hazardous substance in Appendix A to Part 355 of 40 CFR, and is thus reportable under EPCRA Section 313 and TURA. It has the potential to cause severe eye and respiratory irritation. Skin burns and blisters occur in an atmosphere of 1.5-2.0% ammonia.<sup>4</sup>

The ammonia is directly piped to the dissociators from an exterior storage tank in the lower receiving dock area. Worker exposure could result from a spill during an ammonia delivery, pipe leak or dissociator malfunction. This new system will require fewer ammonia deliveries, thus reducing the chances for a spill, and will allow the shutdown of one dissociator unit.

### **4.2 Air Emissions**

It is expected that reduced ammonia usage will result in decreased residual ammonia generated by the dissociators. Also, the decreased chances of a spill reduce the chances of spilled ammonia vaporizing and entering the air handling system.

### **4.3 Toxic Chemical Use per Unit of Product**

In 1996 General Findings used 104,318 pounds of anhydrous ammonia to process 202,271,413 units through the furnaces, resulting in 1,938 units/pound of ammonia. At 37.5% hydrogen (50% reduction) General Findings will process 3,877 units/pound of ammonia and at 20% hydrogen, 7,262 units/pound of ammonia. This reflects reductions in ammonia use of 50% and 73% respectively.

## **5. ECONOMIC IMPLICATIONS**

Chemical cost savings of \$11,461/year are anticipated at a 20% hydrogen mix. The cost of nitrogen was reduced due to increased usage, rental costs increased for the larger nitrogen tank, and ammonia prices have remained the same although reduced usage may lead to price increases. Additional savings will be realized from reduced electrical usage (shut-down of one ammonia dissociator) and reduced heat load on the air conditioning system (from the shut-down dissociator unit) to total \$13,226/year. Payback with the matching grant is 4½ years. Additional savings will be realized when the new Raw Material area goes into production.

### **5.1 Capital Expenditures**

Capital expenditures include the new furnace control panels and installation, new concrete pad for the larger nitrogen tank, new fencing for the nitrogen tank, portable nitrogen tank rental during switch-over, and a larger nitrogen pipeline throughout the facility. Facilities with fewer furnaces, a large enough nitrogen tank and/or cement pad if a bigger nitrogen tank is required, and ample sized piping will experience much lower capital costs. Table 2 shows the capital expenditures for this project.

---

<sup>4</sup> Carbonaire, Inc., *Material Safety Data Sheet*, revised 1/92.

**Table 2 - Capital Expenditures**

ITEM	COST
Control panels	\$52,650
Panel installation	\$4,500
Cement pad	\$7,778
Fence	\$950
Portable tank rental	\$400
Temporary liquid nitrogen cylinder	\$405
Piping	\$4,154
<b>TOTAL</b>	<b>\$70,837</b>

The control panel for a “DA” system costs a bit more than the panel for a hydrogen/nitrogen system, as the piping and flow meters must be ammonia resistant stainless steel. Copper or brass would be adversely affected by the small amount of residual ammonia in the “DA”.

**5.2 Regulatory Costs for Toxics Use**

A permit for the larger nitrogen tank was not necessary. Ammonia usage will remain above reporting thresholds so TURA reporting fees remain the same.

**6. INSTALLATION AND VALIDATION**

All new furnace control panels are built to current NFPA (National Fire Protection Association) and internal safety standards. As each panel is installed, extensive testing of atmosphere mixtures and products will be conducted as furnaces vary with design, manufacturer and age. Tables 3 & 4 show test results from two sets of internal tests.

**Table 3 - Initial Testing, February 14, 1997 - Hayes Furnace**

Material tested: 14K Leach & Garner Gold, 10K

Test Data	1 <sup>st</sup> test	2 <sup>nd</sup> test	3 <sup>rd</sup> test
Temperature, °F	1500	1300	1300
Belt Speed, in./min.	10	10	10
Nitrogen, cfh	80	80	85
Dissociated ammonia, cfh	85	85	85
Nitrogen, %	61.4	61.4	62.5
Hydrogen, %	38.6	38.6	37.5
Result : color	Good (overheated)	Good	Good
Burnout flame	Visible	Visible	Visible

**Table 4 - Additional Testing, March 20, 1997 - Hayes Furnace**

Material tested: 14K Leach & Garner gold, copper oxide

Test Data	Hoop earrings	Hoop earrings	Copper oxide
Temperature, °F	1550	1550	1550
Belt Speed, in./min.	15, heat zone 7, cool zone	11, heat zone 6, cool zone	11, heat zone 6, cool zone
Nitrogen, cfh	130	130	130
Dissociated ammonia, cfh	25	25	25
Nitrogen, %	88.0	88.0	88.0
Hydrogen, %	12.0	12.0	12.0
Result : color	Good (not soldered)	Good	Oxides removed
Burnout flame	Not visible	Not visible	Not visible

## 7. BARRIERS AND RESOURCES ENCOUNTERED

As with any new technology, there are always “bugs” or technical difficulties to work out. Flow switches in the panels were replaced with differential pressure switches, and the “DA” pressure exiting the dissociator unit was raised.

The first panel went on-line June 25, 1997. The “DA” to this furnace was immediately cut in half (50% reduction or 37.5% hydrogen) and no color problems were encountered. The second panel will go on-line during shutdown, reducing the load to one of the dissociators. Two additional panels will be tested in the new production area, while other panels have been ordered and will go on-line this fall.

With a lower hydrogen content, parts may emerge from some furnaces hotter, thus appropriate glove protection will be reviewed.

As with any process change, operator involvement, training and acceptance is an important factor. BOC Gases has been very supportive and will conduct initial operator training and provide technical support and assistance.