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OAK RIDGE
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FOR THE DEPARTMENT OF ENERGY

# HASTELLOY N THERMAL CONVECTION LOOP: STATUS REPORT

**May 2002**

**D. F. Wilson, L. M. Toth, J. F. King, E. K. Ohriner, J. R. DiStefano, E. T. Manneschmidt**

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ORNL/TM-2002-112

Metals and Ceramics Division

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## EXECUTIVE SUMMARY

A Hastelloy N thermal convection loop was designed and constructed. The rationale for the use of Hastelloy N is outlined and reduced scale drawings of the design are presented. The loop, in its frame, stands 1.91 m tall; has ports for electrochemistry, vacuum, and insert gas; has hot and cold legs that contain 32 Hastelloy N specimens each; and has a surge tank and freeze valves. The volume of the loop, freeze valves, and surge tank, as constructed, is approximately 4.2 liters A description of the assembled specimens and the specimen weights are presented. The various steps in the drawing of the Hastelloy N tubing, and production and purification of the fluoride salt are described. This Hastelloy N, thermal convection loop will allow for the determination of corrosion effects such as mass transfer due to thermal gradient in a flowing molten fluoride salt at 1073 K, a temperature above the experience base.

# 1. INTRODUCTION

This report summarizes the various steps in the construction of a Hastelloy N thermal convection loop. Operations performed on the Hastelloy N material included reduction of piping, rolling of plate, swaging of tubing, welding, and quality assurance. As shown in Fig. 1, the loop in its frame stands 1.91 m (75 in.) tall; has ports for electrochemistry, vacuum, and inert gas; has hot and cold legs that contain thirty-two Hastelloy N specimens each; and has a surge tank and freeze valves. All surfaces that will be wetted by the molten fluoride salt during testing are Hastelloy N. The volume of as-constructed loop, freeze valves, and surge tank is approximately 4.2 liters.

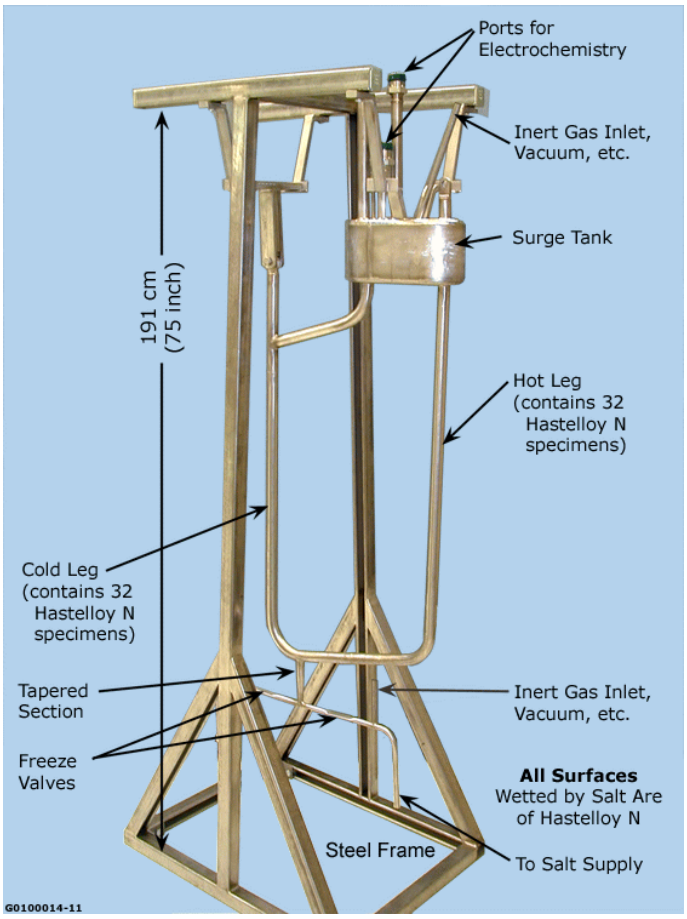


**Figure 2. Hastelloy N loop in steel frame.** The hot and cold legs of the loop contain 32 Hastelloy N specimens each. All surfaces wetted by the molten salt are Hastelloy N.

## 2. LOOP DESIGN

Details of the design of the Hastelloy N loop are contained in “Thermal Convection Loop-HTFL” drawings N3E020632A001, N3E020632A002, N3E020632A004, N3E020632A005, and N3E020632A006 which are presented in Appendix A. Of special significance are the “freeze” valves that are detailed in drawing N3E020632A005 and shown in Fig. 2. The loop is designed to maintain the molten salt within the loop via a solidified plug of salt, which, during loop operation, is obtained by cooling the flattened region. Interruption of the cooling and application of a minor amount of heat will allow for rapid emptying of the salt from the loop. The loop is designed to be loaded with salt by pressurizing heated tanks (see Fig. 3) attached to the freeze valves.

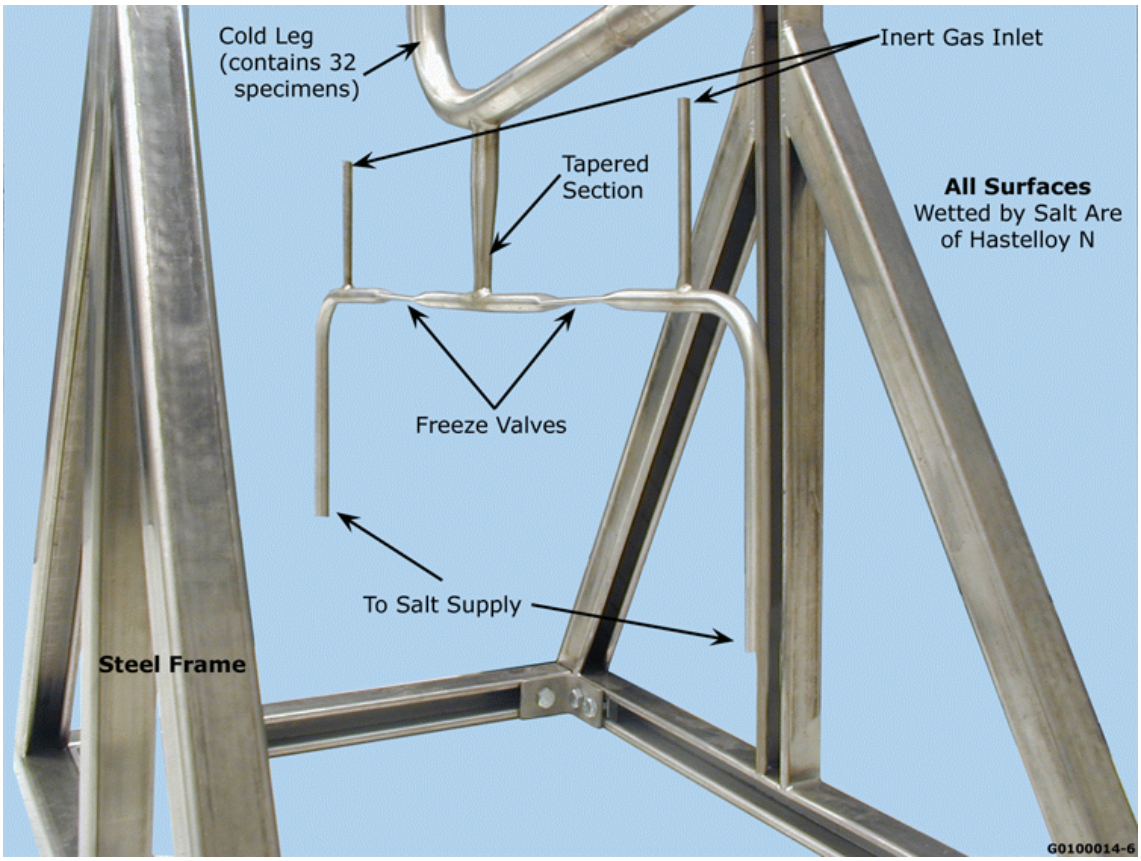


**Figure 3. View of the Hastelloy N freeze valves.** The flattened region allows for rapid cooling of the salt that creates a salt plug.

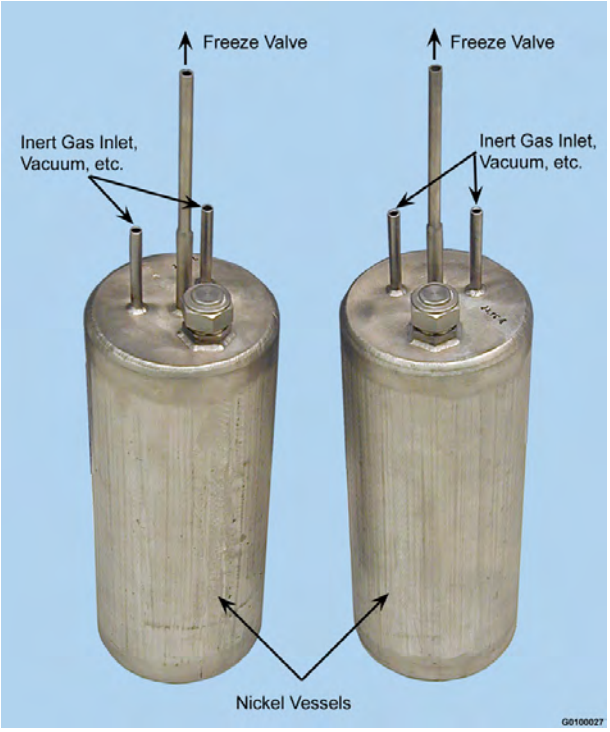


**Figure 4. Nickel vessels for salt supply.**

The weight of the loop and its salt content is supported from the top of the loop via attachments to a steel frame. The steel frame is designed to allow for movement of the loop due to expansion on being heated to, and contraction on being cooled from, the operating temperature. As shown in Fig. 4, loop supports from the top of the cold leg of the loop and from the top of the surge tank are free to slide within guides attached to the steel frame. Stops on the end of the guides prevent excess movement during normal handling of the loop. In addition, as shown in Fig. 5, the hinged attachment at the top of the cold leg allows for minor rotation in the vertical plane, which may be necessary due to the various thermal gradients across the loop and surge tank. Rotation is accommodated on the cold leg side of the loop because it is less massive than the hot leg and attached surge tank.

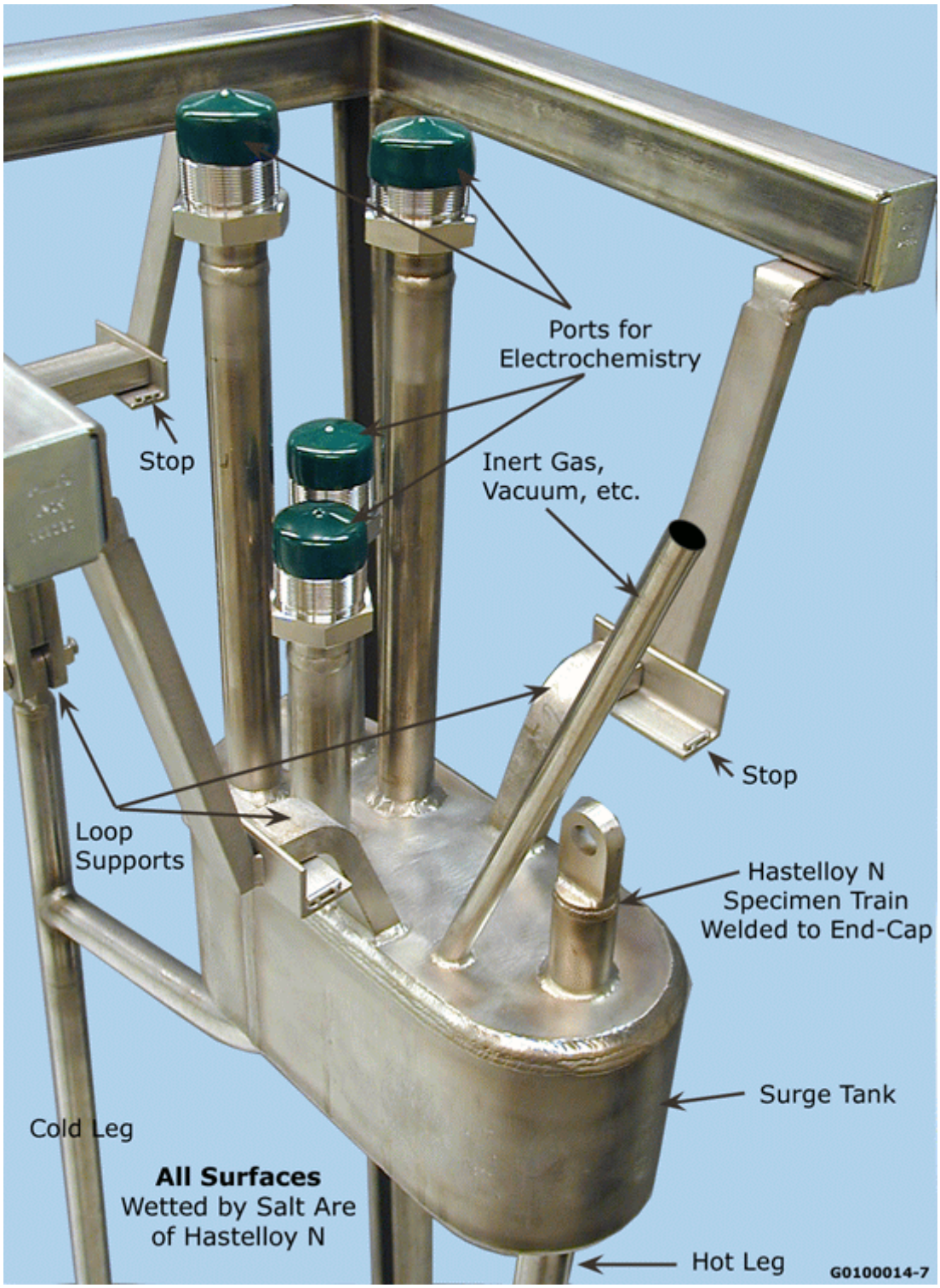


**Figure 5. Details of the loop supports.** Thermal expansion is accommodated via sliding (hot and cold legs) and rotation (hinge at cold leg) of the loop.

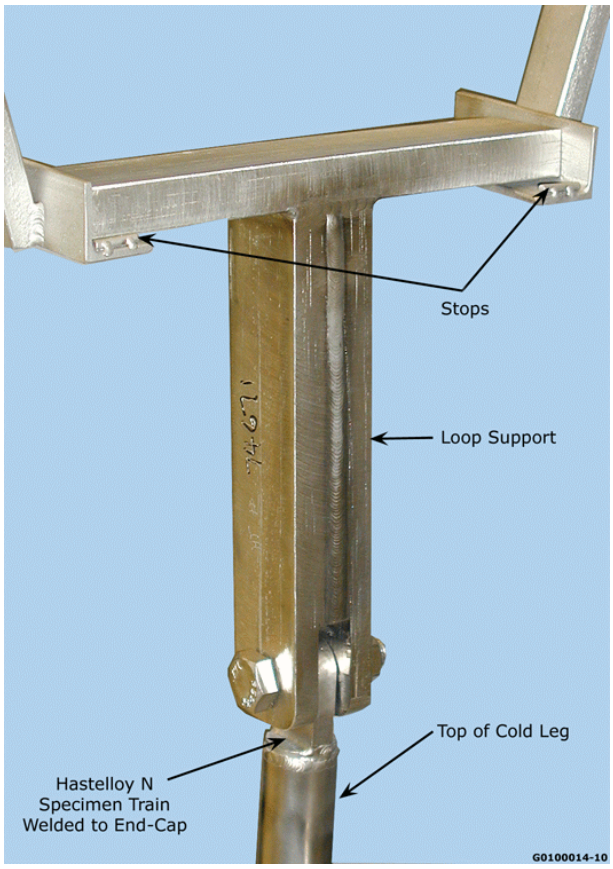


**Figure 6. Support at the top of the cold leg showing hinged connection.**

As constructed, each leg of the loop holds thirty-two Hastelloy N specimens while allowing for minimal disturbance of the flowing molten salt. Specimens are designed with notches at two ends of the specimen to allow for interlocking of them, thus providing some rigidity to the arrangement. As shown in Fig. 6, the interlocked specimens are wired with Hastelloy N wire to a Hastelloy N weld rod, which was subsequently welded to the end cap of each leg.

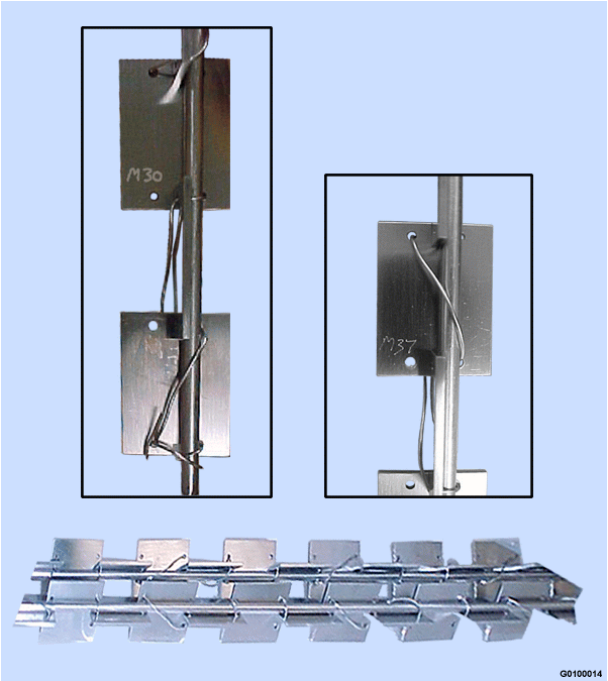


**Figure 7. Interlocked Hastelloy N specimens (1.91 x 2.25 x 0.13 cm) wired to a Hastelloy N weld wire (0.32 cm diam).**

## 3. MATERIAL SELECTION

Hastelloy N (INOR-8),* which has chemical composition shown in Table 1, was chosen as the materials of construction for the loop for the following reasons:[1]

- designed for compatibility with molten fluoride salts,
- minimum Cr but sufficient to provide good oxidation resistance[2] (Fig. 7),
- solid solution strengthened with molybdenum to provide adequate high temperature yield[3] and tensile strengths[4] at 1073 K (Fig. 8),
- alloy does not embrittle upon aging,
- limited aluminum, titanium, and carbon contents minimize fabrication and corrosion problems,
- limited boron content prevents weld cracking, and
- carbides are stable to 1255 K.

*INOR 8 is the designation of the Hastelloy N while it was under development at ORNL.

**Table 1. Chemical composition (wt%) for Hastelloy N (INOR-8) material**

| Element | Requirement* | Tubing as analyzed† | Specimen as analyzed† |
|---|---|---|---|
| Nickel | Remainder | 71.18 | 72.17 |
| Molybdenum | 15.00–18.00 | 16.18 | 16.11 |
| Chromium | 6.00–8.00 | 7.26 | 6.31 |
| Iron | 5.00 | 3.71 | 4.03 |
| Carbon | 0.04–0.08 | | 0.03 |
| Manganese | 1.00 | 0.53 | 0.53 |
| Silicon | 1.00 | 0.58 | 0.38 |
| Tungsten | 0.50 | 0.06 | 0.06 |
| Aluminum + titanium | 0.50 | 0.02 (Al) | <0.01 (Al) |
| Copper | 0.35 | 0.01 | 0.01 |
| Cobalt | 0.20 | 0.12 | 0.15 |
| Phosphorous | 0.015 | 0.01 | 0.006 |
| Sulfur | 0.020 | | 0.003 |
| Boron | 0.010 | | |
| Others, total | 0.50 | 0.02 (Mg), 0.27 (V) | 0.01 (Mg), 0.20 (V), <0.01 (Ca), <0.01 (Zr) |

*Source: R. B. Briggs, Program Director, *Molten-Salt Reactor Program: Semiannual Progress Report for Period Ending July 31, 1964*, ORNL-3708, p. 332, Oak Ridge National Laboratory, November 1964. Single values are maximum percentages.
† Quantitative analysis using Inductively Coupled Plasma (ICP), Dirats Laboratories, Westfield, MA.

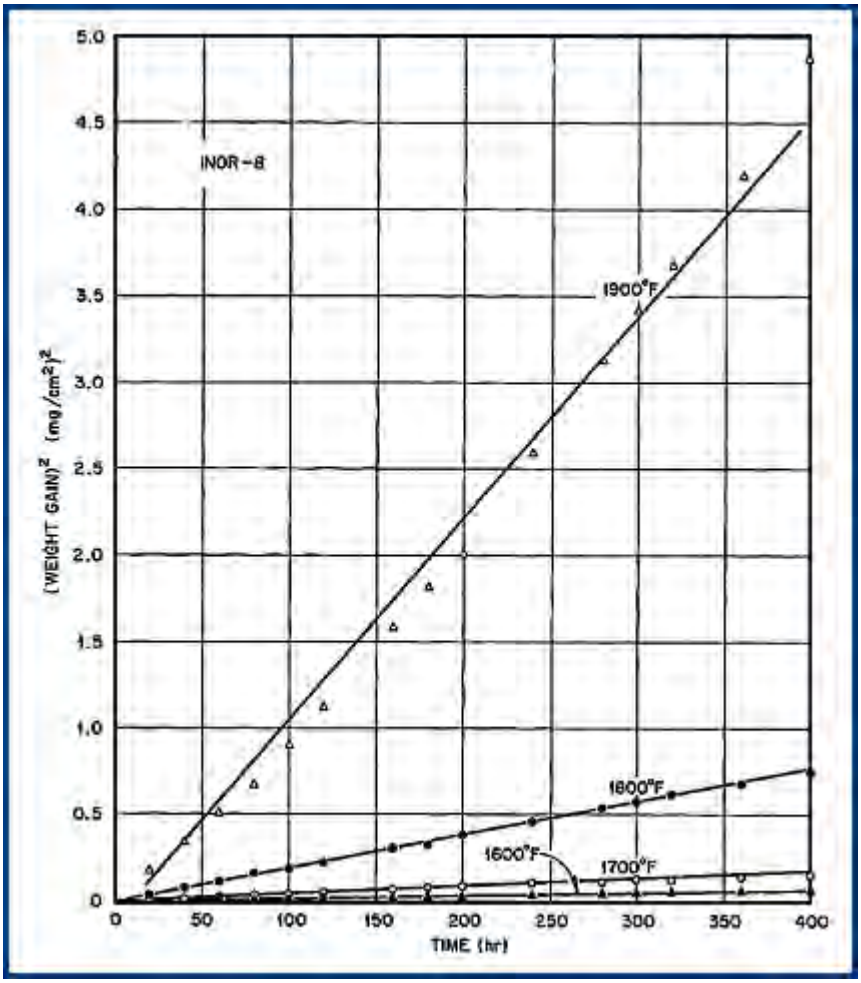


**Figure 8. Oxidation resistance of Hastelloy N is adequate for this application.** (Reference 2.)

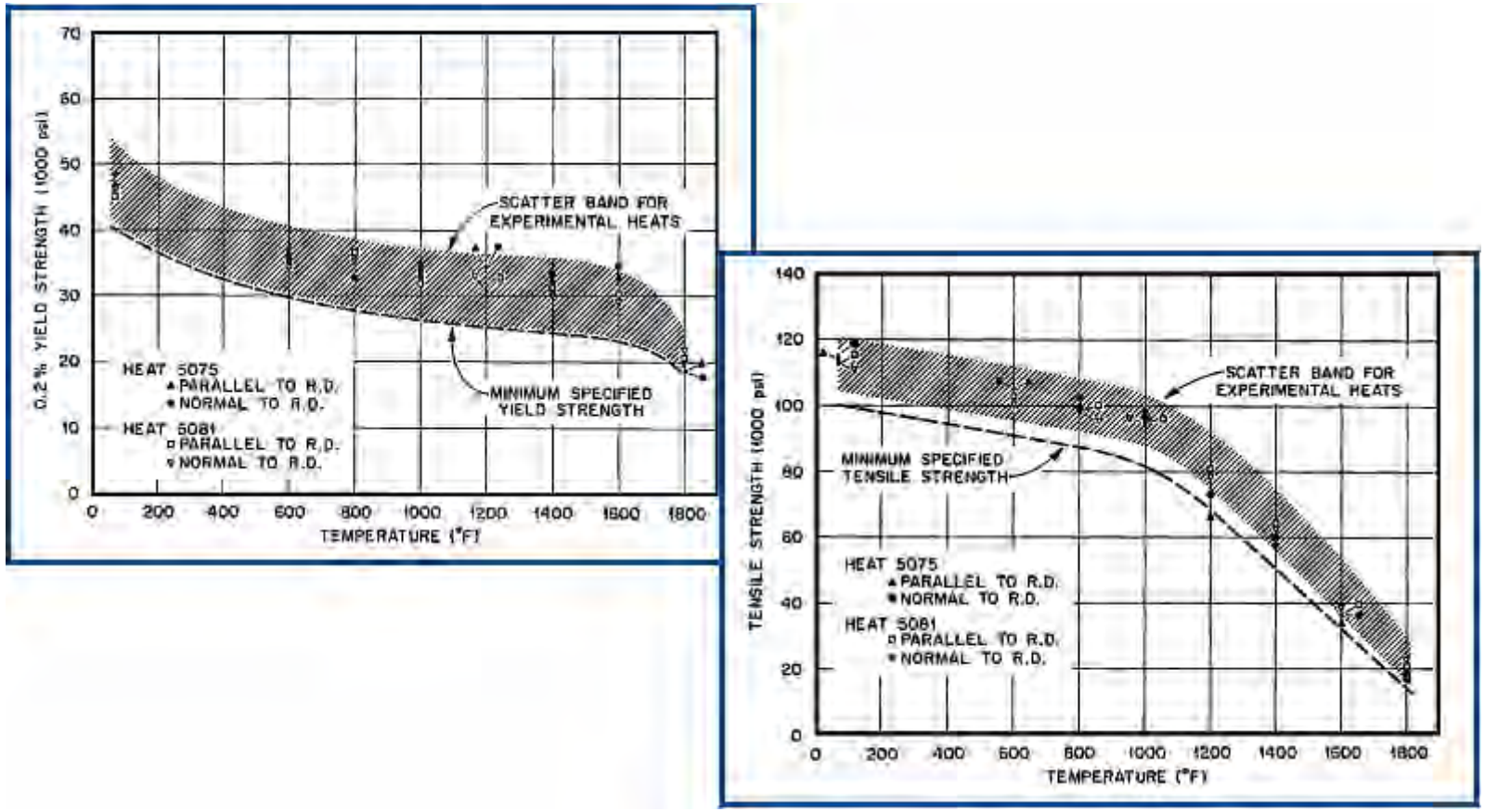


**Figure 8. Yield and tensile strengths of Hastelloy N permit operations at 1073 K.** (References 3 and 4, respectively).

## 4. SPECIMENS

The Hastelloy N specimens were manufactured by Metal Samples, Incorporated,* from materials supplied by ORNL (chemical composition shown in Table 1), with a 32 RMS surface finish, per specifications. The specimens were "spot checked" dimensionally to check for variations and verify consistency. The dimensional ranges were as follows:

Width - 1.9037 to 1.9062 cm (0.7495 to 0.7505 in.),

Length - 0.2527 to 0.2540 cm (0.0995 to 0.1000 in.), and

Thickness - 0.1295 to 0.1321 cm (0.0510 to 0.0520 in.).

After cleaning in acetone, Hastelloy N specimens were assembled into two chains, each approximately 68.58 cm (27 in.) long, for the hot and cold legs. The specimens are connected together using Hastelloy N weld wire that had been drawn down to 0.061 cm (0.024 in.) diameter. The wire is threaded through the holes in each end of each specimen and around a 0.318 cm (0.125 in.) diam weld rod, whose purpose is to hold the specimens straight and reasonably rigid during the test. Figure 6 shows the interlocked specimens wired with Hastelloy N wire to a Hastelloy N weld rod. The weld rods, which extended above the top of the specimen chains, were welded to the end caps of the loop. Specimens, scribed in the lower left hand corner of each, were labeled M1 through M32 for the hot leg and M33 through M64 for the cold leg. The pre-test weights of these specimens are presented in Table 2.

---

*Metal Samples Company Incorporated, Munford, AL 36368.

**Table 2. Weights of Hastelloy N specimens within legs of Hastelloy N loop**

| Hot leg | | | | Cold leg | | | |
|---|---|---|---|---|---|---|---|
| **Name** | **Weight (g)** | **Name** | **Weight (g)** | **Name** | **Weight (g)** | **Name** | **Weight (g)** |
| M1 | 5.45654 | M17 | 5.54883 | M33 | 5.51528 | M49 | 5.55842 |
| M2 | 5.50922 | M18 | 5.55475 | M34 | 5.50708 | M50 | 5.52191 |
| M3 | 5.38392 | M19 | 5.54458 | M35 | 5.33355 | M51 | 5.43062 |
| M4 | 5.56149 | M20 | 5.54798 | M36 | 5.48549 | M52 | 5.53954 |
| M5 | 5.52542 | M21 | 5.45402 | M37 | 5.56768 | M53 | 5.52538 |
| M6 | 5.49638 | M22 | 5.52714 | M38 | 5.54722 | M54 | 5.53096 |
| M7 | 5.56318 | M23 | 5.53318 | M39 | 5.55606 | M55 | 5.50745 |
| M8 | 5.44616 | M24 | 5.55787 | M40 | 5.56418 | M56 | 5.55193 |
| M9 | 5.53828 | M25 | 5.51313 | M41 | 5.40985 | M57 | 5.31465 |
| M10 | 5.49267 | M26 | 5.55940 | M42 | 5.54172 | M58 | 5.51047 |
| M11 | 5.44970 | M27 | 5.44984 | M43 | 5.38037 | M59 | 5.55317 |
| M12 | 5.36093 | M28 | 5.43638 | M44 | 5.54054 | M60 | 5.56752 |
| M13 | 5.52311 | M29 | 5.53089 | M45 | 5.55960 | M61 | 5.53845 |
| M14 | 5.46306 | M30 | 5.54406 | M46 | 5.51715 | M62 | 5.56342 |
| M15 | 5.55594 | M31 | 5.54307 | M47 | 5.43368 | M63 | 5.47146 |
| M16 | 5.53030 | M32 | 5.47470 | M48 | 5.54220 | M64 | 5.40773 |

## 5. MATERIALS PROCESSING

Tubing, which has chemical composition presented in Table 1, was produced at the Oak Ridge National Laboratory from Hastelloy N schedule 40 [4.82 cm (1.9 in.) outer diameter (O.D.), 4.06 cm (1.6 in.) inner diameter (I.D.)] pipe from in-house stock. The measurements made on the finished tubing are presented in Table 3. Due to the high strain hardening rate of this material,[5] as shown in Fig. 9, nine anneals were performed during the reduction to tubing with an average O.D. 0.4155 cm (1.0554 in.) and average I.D. 0.3612 cm (0.9174 in.). The average wall thickness obtained was 0.0265 cm (0.0673 in.) with percentage variation of 4.1%. The reductions in diameters and associated anneals are presented in Table 4. Because the inside of the tubing has drawing marks, evaluation of materials performance should be concentrated on the specimens rather than on the tubing.

**Table 3. Dimensions of finished Hastelloy N tubing used in construction of loop[a]**

| Tubing # | Length (in.) | Outer diameter (in.) | Inner diameter (in.) | Minimum wall thickness (in.) | Maximum wall thickness (in.) | Variation in wall thickness (%) |
|---|---|---|---|---|---|---|
| 1A | 54.75 | 1.0570 | 0.9195 | 0.0665 | 0.0680 | 2.2 |
| 1A | | 1.0515 | 0.9175 | 0.0657 | 0.0687 | 4.4 |
| 1B | 48.25 | 1.0565 | 0.9200 | 0.0657 | 0.0690 | 4.8 |
| 1B | | 1.0515 | 0.9180 | 0.0650 | 0.0697 | 6.7 |
| 2A | 64.88 | 1.0575 | 0.9180 | 0.0650 | 0.0680 | 4.4 |
| 2A | | 1.0585 | 0.9190 | 0.0653 | 0.0693 | 5.8 |
| 2B | 62.25 | 1.0530 | 0.9165 | 0.0647 | 0.0690 | 6.2 |
| 2B | | 1.0550 | 0.9180 | 0.0640 | 0.0703 | 9.0 |
| 2C | 69.00 | 1.0620 | 0.9180 | 0.0663 | 0.0675 | 1.8 |
| 2C | | 1.0590 | 0.9160 | 0.0655 | 0.0707 | 7.4 |
| 2D | 60.38 | 1.0515 | 0.9190 | 0.0647 | 0.0690 | 6.2 |
| 2D | | 1.0600 | 0.9195 | 0.0643 | 0.0707 | 9.1 |
| 2E | 65.50 | 1.0525 | 0.9170 | 0.0655 | 0.0683 | 4.1 |
| 2E | | 1.0520 | 0.9195 | 0.0653 | 0.0677 | 3.5 |
| 3A | 62.5 | 1.054 | 0.912 | 0.067 | 0.0685 | 2.2 |
| 3A | | | | 0.067 | 0.0685 | 2.2 |
| 3B | 63 | 1.055 | 0.915 | 0.0662 | 0.0678 | 2.4 |
| 3B | | | | 0.067 | 0.0685 | 2.2 |
| 4A | 59.4 | 1.055 | 0.915 | 0.068 | 0.069 | 1.4 |
| 4A | | | | 0.067 | 0.0679 | 1.3 |
| 4B | 38.4 | 1.055 | 0.915 | 0.0675 | 0.069 | 2.2 |
| 4B | | | | 0.067 | 0.0675 | 0.7 |
| Average | | 1.0554 | 0.9174 | 0.0673 | | 4.1 |

[a] Measurements were made in inches.

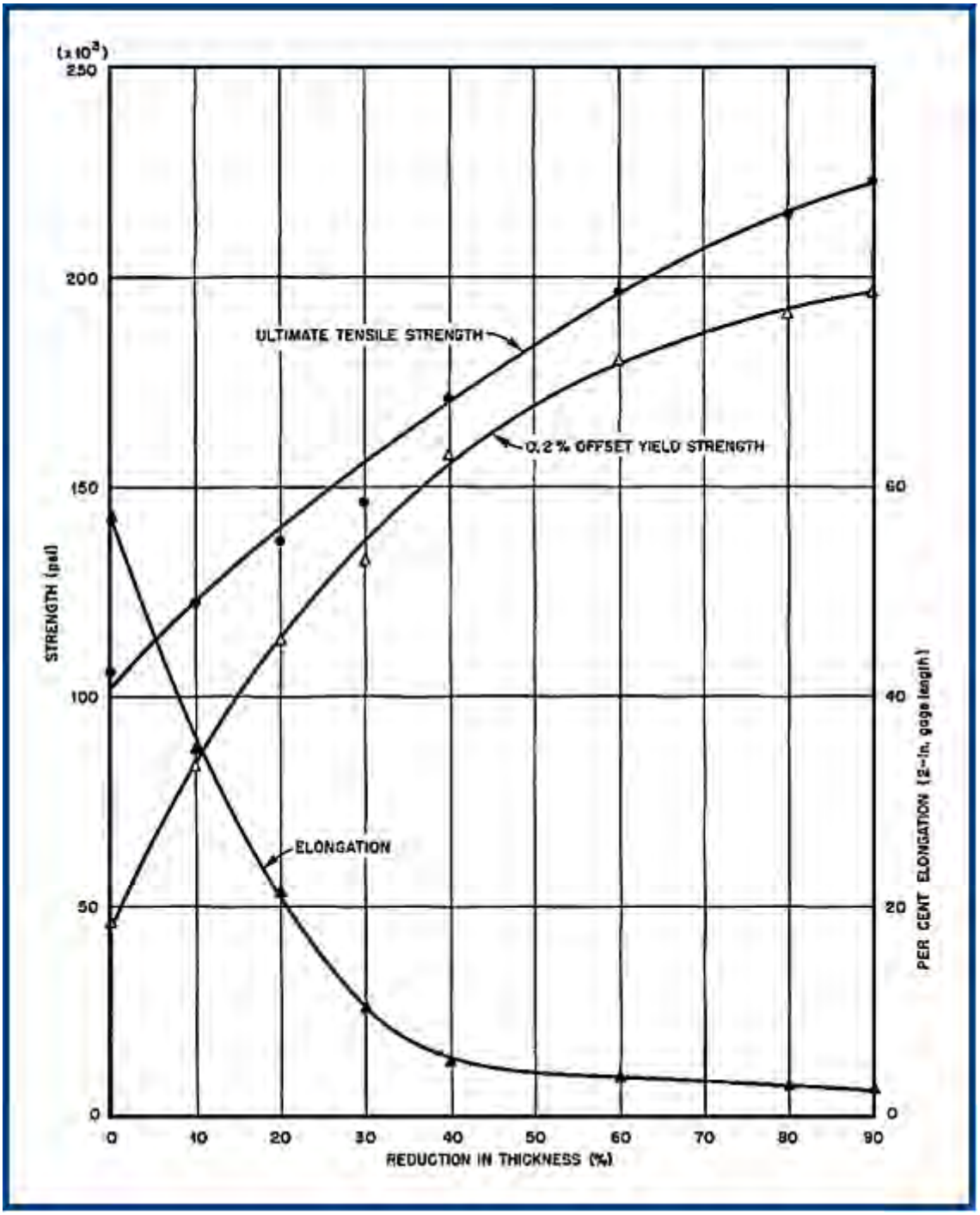


**Figure 9. Due to rapid strain hardening, cold forming requires frequent anneals.** (Reference 5.)

**Table 4. Drawing and annealing schedule for production of Hastelloy N tubing[a]**

| Draw | I.D. (in.) | O.D. (in.) | Wall thickness (in.) | Cross sectional area (in.$^2$) | % reduction | % wall reduction |
|---|---|---|---|---|---|---|
| | | | Drawing over Plug Mandrels | | | |
| 0 | 1.900 | 1.600 | 0.150 | 0.825 | | |
| 1 | 1.850 | 1.560 | 0.145 | 0.777 | 5.8 | 3.3 |
| 2 | 1.775 | 1.490 | 0.143 | 0.731 | 5.9 | 1.7 |
| 3 | 1.725 | 1.450 | 0.138 | 0.686 | 6.2 | 3.5 |
| | | | Anneal | | | |
| 4 | 1.675 | 1.410 | 0.133 | 0.642 | 6.4 | 3.6 |
| 5 | 1.625 | 1.370 | 0.128 | 0.600 | 6.6 | 3.8 |
| 6 | 1.575 | 1.330 | 0.123 | 0.559 | 6.8 | 3.9 |
| 7 | 1.550 | 1.290 | 0.130 | 0.580 | -3.7 | -6.1 |
| 8 | 1.525 | 1.270 | 0.128 | 0.560 | 3.5 | 1.9 |
| | | | Anneal | | | |
| 9 | 1.500 | 1.235 | 0.133 | 0.569 | -1.7 | -3.9 |
| 10 | 1.460 | 1.200 | 0.130 | 0.543 | 4.6 | 1.9 |
| 11 | 1.440 | 1.165 | 0.138 | 0.563 | -3.6 | -5.8 |
| | | | Anneal | | | |
| 12 | 1.420 | 1.155 | 0.133 | 0.536 | 4.7 | 3.6 |
| 13 | 1.400 | 1.140 | 0.130 | 0.519 | 3.2 | 1.9 |
| | | | Anneal | | | |
| | | | Drawing over Bar Mandrels | | | |
| 14 | 1.375 | 1.125 | 0.125 | 0.491 | 5.4 | 3.8 |
| 15 | 1.355 | 1.125 | 0.115 | 0.448 | 8.7 | 8.0 |
| | | | Anneal | | | |
| 16 | 1.312 | 1.080 | 0.116 | 0.436 | 2.7 | -0.9 |
| 17 | 1.290 | 1.080 | 0.105 | 0.391 | 10.3 | 9.5 |
| | | | Anneal | | | |
| 18 | 1.250 | 1.040 | 0.105 | 0.378 | 3.4 | 0.0 |

**Table 4. Drawing and annealing schedule for production of Hastelloy N tubing[a]**

| Draw | I.D. (in.) | O.D. (in.) | Wall thickness (in.) | Cross sectional area (in.$^2$) | % reduction | % wall reduction |
|---|---|---|---|---|---|---|
| 19 | 1.22 | 1.040 | 0.090 | 0.319 | 15.4 | 14.3 |
| 20 | 1.205 | 1.040 | 0.083 | 0.291 | 8.9 | 8.3 |
| | | | Anneal | | | |
| 21 | 1.160 | 1.000 | 0.080 | 0.271 | 6.7 | 3.0 |
| 22 | 1.145 | 1.000 | 0.073 | 0.244 | 10.0 | 9.4 |
| | | | Anneal | | | |
| 23 | 1.085 | 0.950 | 0.068 | 0.216 | 11.7 | 6.9 |
| 24 | 1.055 | 0.920 | 0.067 | 0.209 | 2.9 | 0.0 |
| | | | Anneal | | | |

[a] Measurements were made in inches.

Annealing was performed in a retort, which was purged with argon-4% hydrogen gas for at least one half hour at a sufficient flow rate to obtain a volume of 10 cubic feet. To avoid gas entrapment, the tubes, which are closed at one end, were purged separately prior to loading into the retort. Each tube was purged for approximately 5 minutes with the same gas using a long fill tube, which reaches near to the bottom of the tube as it is held with closed side down. The tubes were heated for 1 hour [starting when the furnace recovered to a temperature of 1121°C (2050°F)] at 1149°C (2100°F).

After the one hour anneal, the retort was removed from the furnace and forced-air cooled using fans. Argon-4% hydrogen gas flow was maintained until the retort cooled to about 427°C (800°F). The retort was not opened until it cooled to below 94°C (200°F). Thermocouple measurements of both internal and external retort temperature were recorded periodically during the heat treatment.

Sections of the Hastelloy N tubing, that were bent, were filled with Cerrabend filler prior to bending. The residual Cerrabend filler that was not removed by melting was removed by a NaOH soak, which was followed by a dilute $HNO_3$ wash prior to welding.

## 6. SALT PRODUCTION AND PURIFICATION

Two 4-kg batches of fluoride salt (LiF-NaF-KF; 46.5-11.5-42.0 mol %) were prepared using a hydrofluorination technique. This approach allows for the removal of impurities, which significantly impacts corrosion, by treatment with hydrogen fluoride/hydrogen mixtures and hydrogen. The impurity removal reactions include:

- oxide removal
  - $O^{2-} + 2HF \quad 2F^- + H_2O$
  - $OH^- + HF \quad F^- + H_2O$
- sulfur removal
  - $SO_4^{2-} + H_2 \quad S^{2-} + H_2O$
  - $S^{2-} + 2HF \quad 2F^- + H_2S$

Residual hydrogen fluoride and some impurity metal fluorides can be removed by sparging with hydrogen, helium, or hydrogen/argon mixtures.

The 4-kg batches were prepared in a graphite-lined vessel using several steps:

- dry the individual components (99+% purity, Reagent Grade) in a vacuum oven for at least 24 hours at 423 K (150 °C),
- weigh and mix raw materials,
- load materials into graphite-lined vessel,
- heat vessel to 973K (700°C) under helium flow (~100 ml/min),

  (temperature of at least 200°C above melting point),
- insert graphite dip leg(s) and decrease temperature to 823 K (550°C),

  (temperature 50-100°C above melting point),
  - Switch to hydrogen flow through dip leg(s) (~100 ml/min)
- add hydrogen fluoride (20 ml/min) to hydrogen flow,
- continue $H_2$/HF flow to achieve HF exposure of 10 wt %,

- 10 wt % = 10 g HF per 100 g salt, and
- at 25 ml/min --- (~8 days for a 4 Kg batch of salt).

After required 10 wt% treatment, remove hydrogen fluoride gas:

- switch off hydrogen fluoride,
- continue hydrogen flow until,

  HF in exit gas titrates to less than 0.1 % of original, and
- replace hydrogen with helium.

Continue with digestion and cool down:

- raise dip leg(s) above melt,
- raise temperature to 973K (700°C) (1 h),
- decrease temperature to 733K (460°C) (1 h),
- cool salt slowly (10K/h) through melting point 723K (450°C), and
- turn off power and allow salt to furnace cool
  - maintain helium flow.

## 7. ACKNOWLEDGMENTS

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## 7. REFERENCES


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3. R. B. Briggs, Program Director, *Molten-Salt Reactor Program: Semiannual Progress Report for Period Ending July 31, 1964*, ORNL-3708, p. 350, Oak Ridge National Laboratory, November 1964.

4. R. B. Briggs, Program Director, *Molten-Salt Reactor Program: Semiannual Progress Report for Period Ending July 31, 1964*, ORNL-3708, p. 349, Oak Ridge National Laboratory, November 1964.

5. R. B. Briggs, Program Director, *Molten-Salt Reactor Program: Semiannual Progress Report for Period Ending July 31, 1964*, ORNL-3708, p. 361, Oak Ridge National Laboratory, November 1964.

## APPENDIX A

Details of the design of the Hastelloy N loop are contained in “Thermal Convection Loop-HTFL” drawings N3E020632A001, N3E020632A002, N3E020632A004, N3E020632A005, and N3E020632A006. Greatly reduced images of these drawings follow.

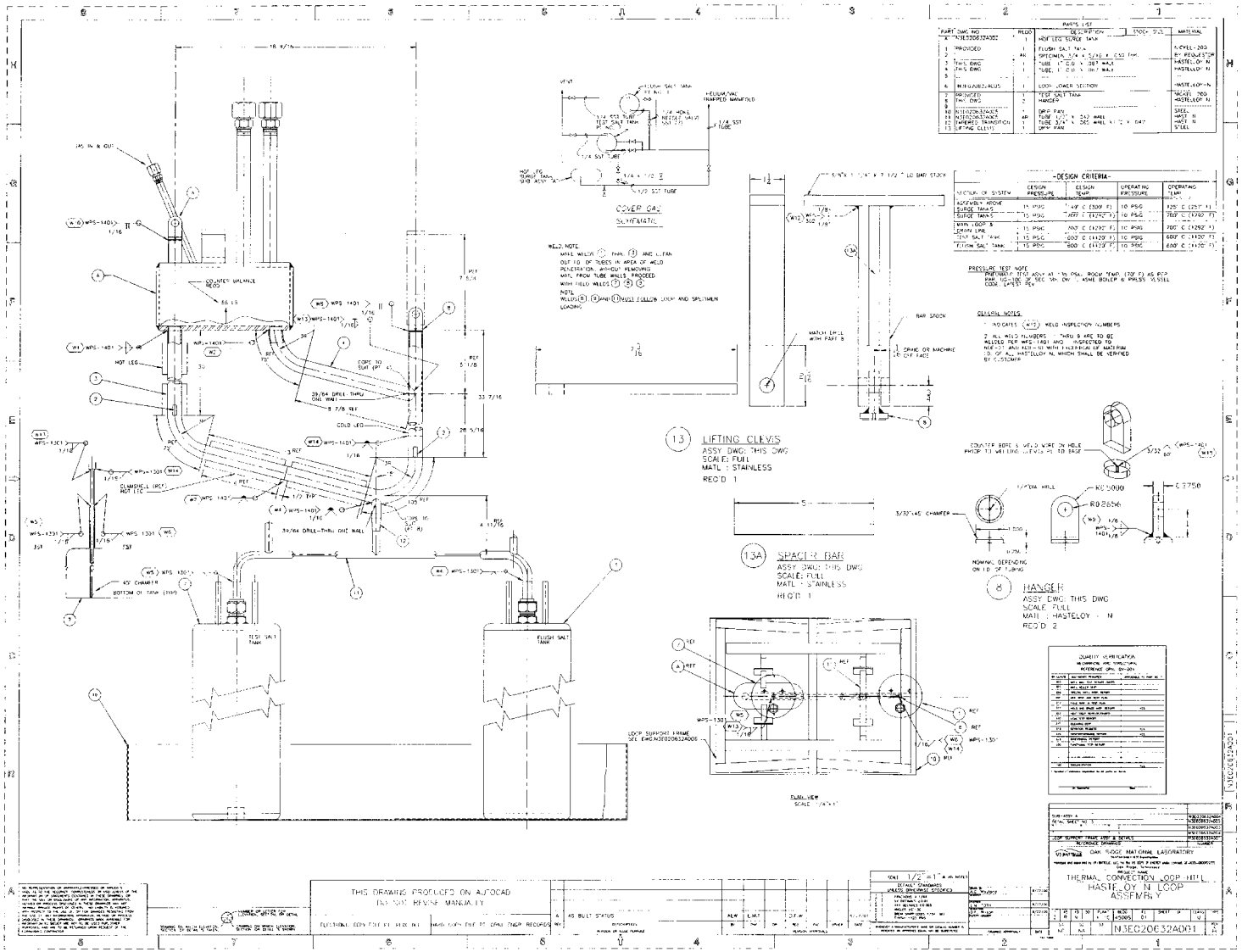


**Figure 10. Hastelloy N loop assembly drawing (N3E020632A001).**

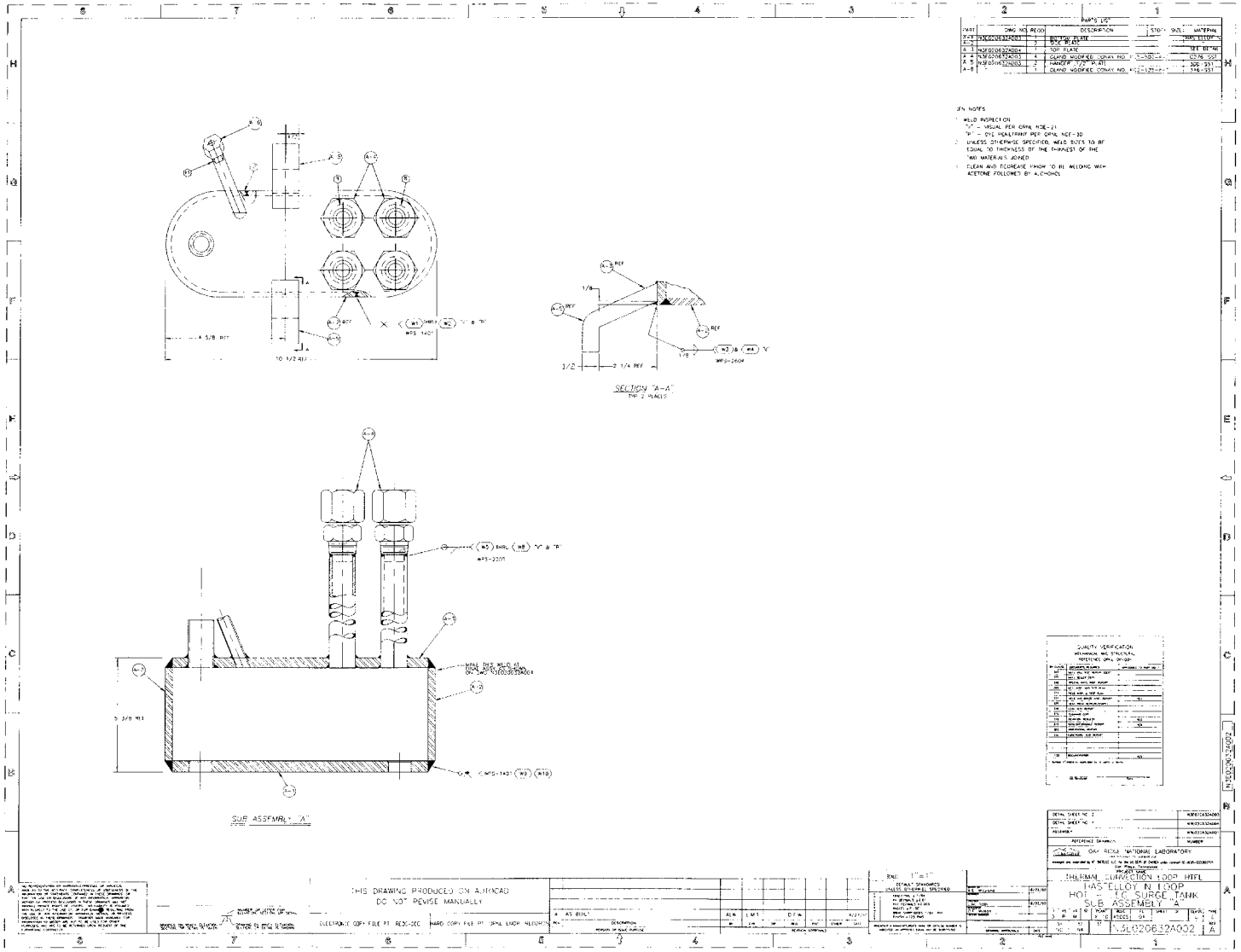


**Figure 11. Hastelloy N surge tank sub-assembly (N3E020632A002).**

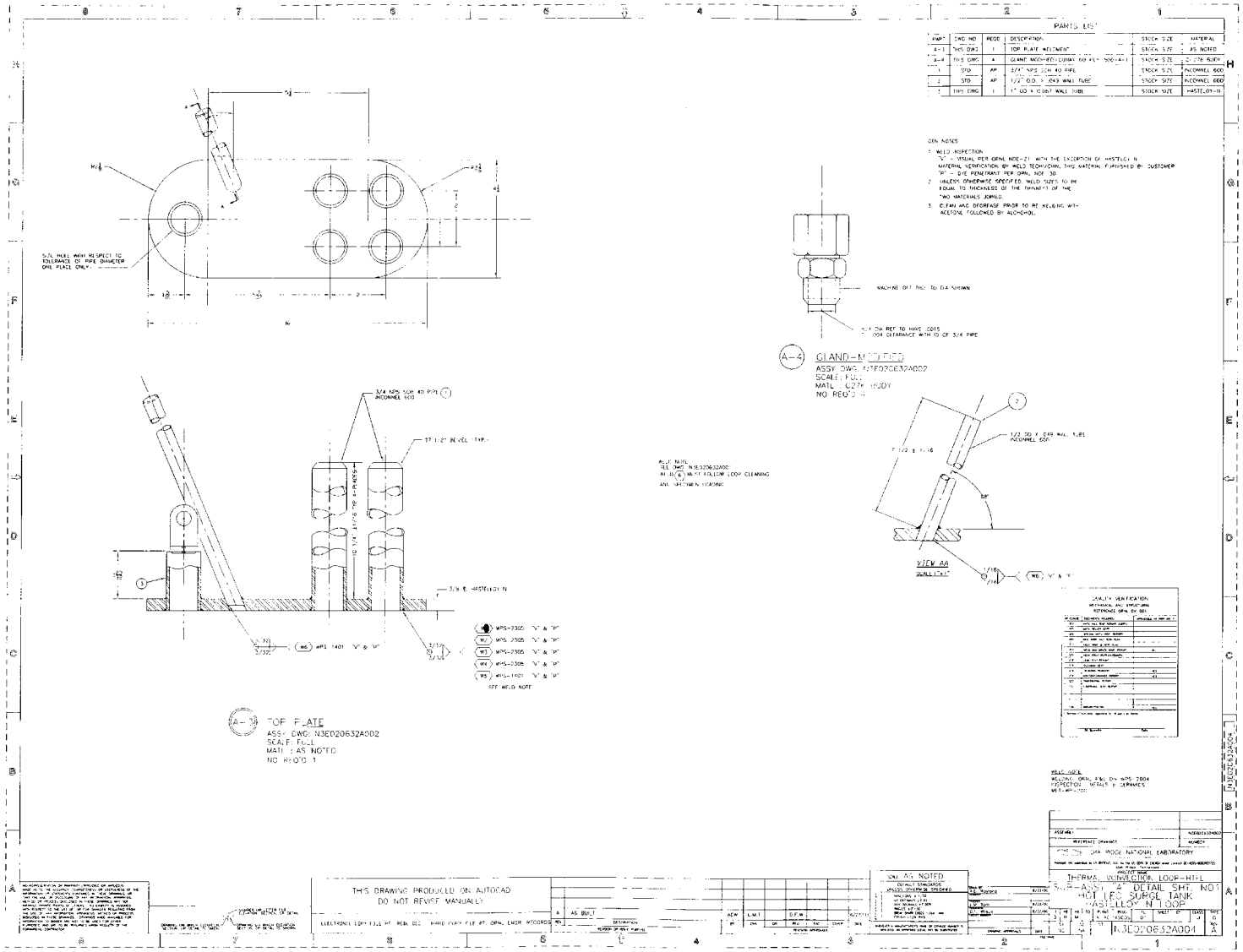


**Figure 12. Hastelloy N loop sub-assembly detail sheet one (N3E020632A004).**

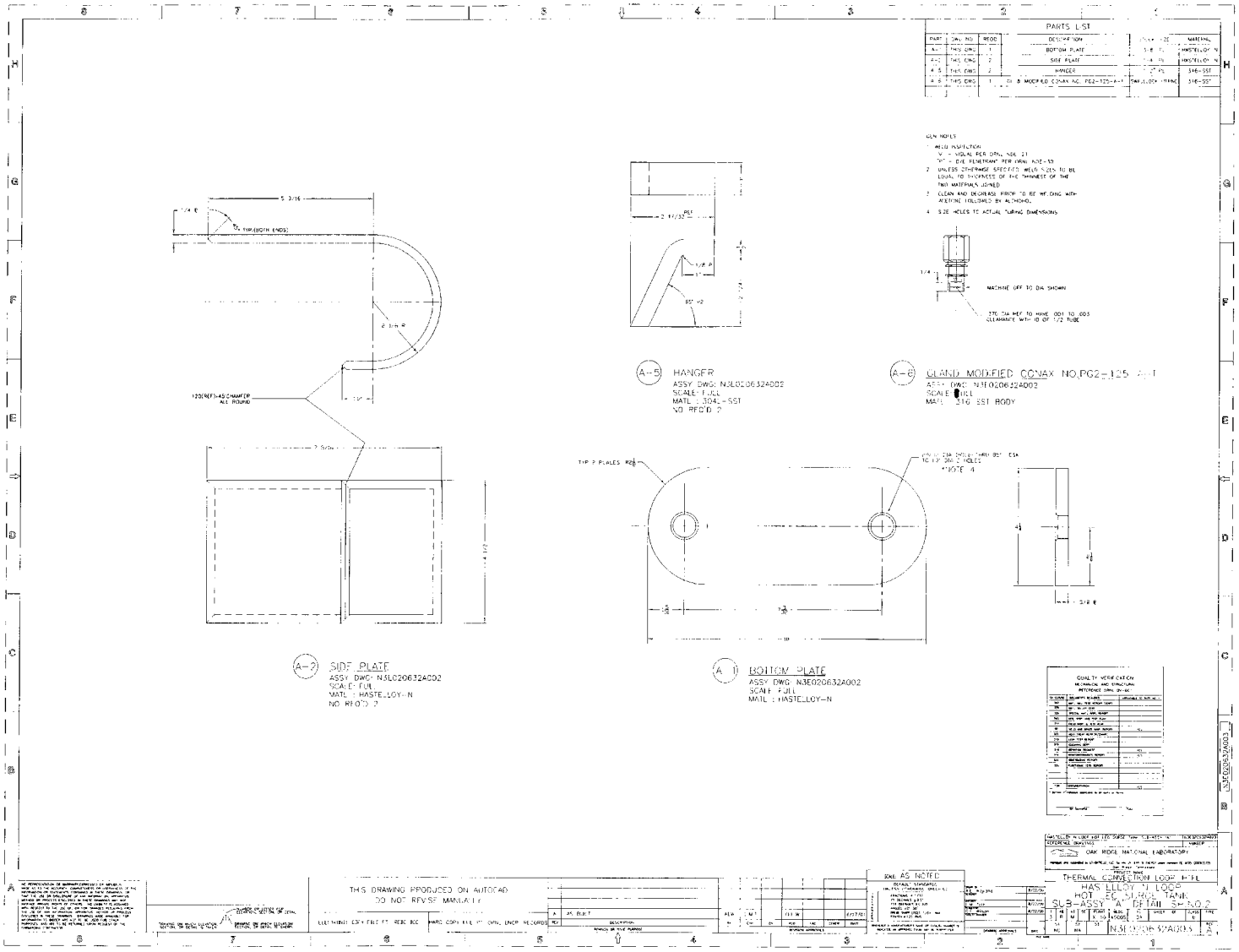


**Figure 13. Hastelloy N loop sub-assembly detail sheet two (N3E020632A003).**

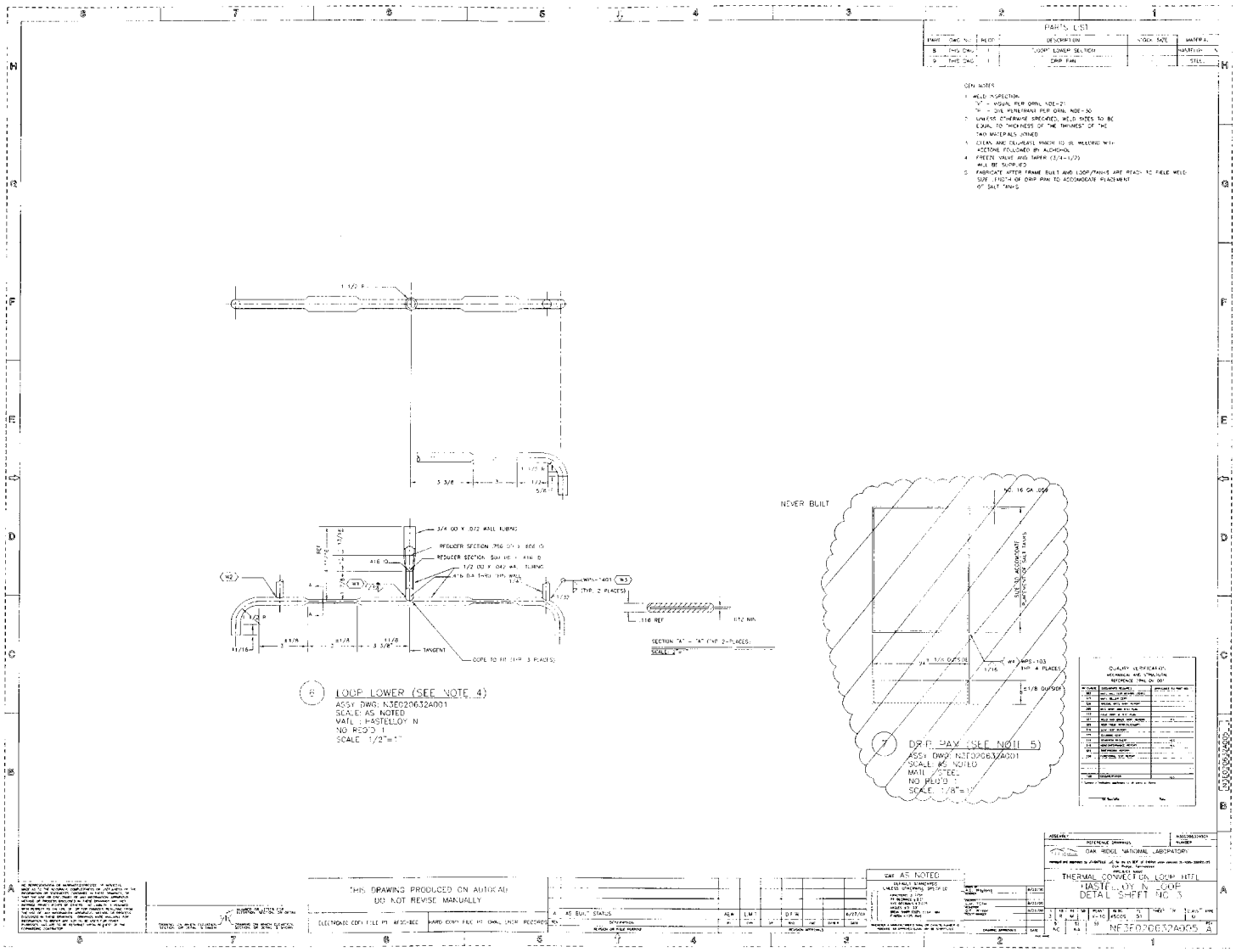


**Figure 14. Hastelloy N "freeze valve" detail sheet three (N3E020632A005).**

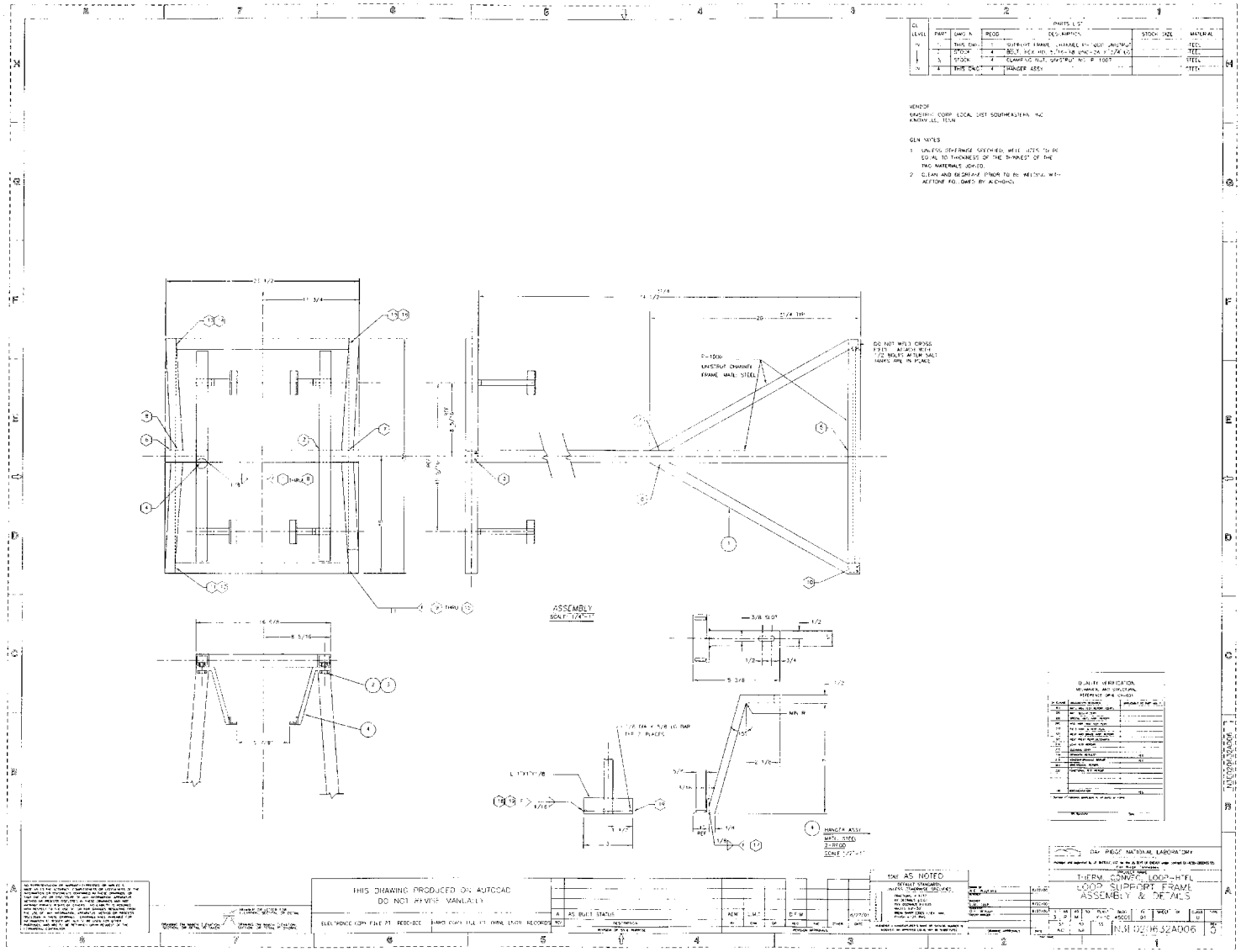


**Figure 15. Hastelloy N loop support frame assembly and details (N3E020632A006).**