Department of Energy
Office of Scientific and Technical Information
Post Office Box 62
Oak Ridge, Tennessee 37831

August 10, 2016

Re: OSTI-2016-01064-F

Dear Mr. Ravnitzky:
This is in final response to the request for information you sent to the Department of Energy (DOE), Office of Scientific and Technical Information (OSTI) under the Freedom of Information Act (FOIA), 5 U.S.C. 552 on June 22, 2016.

You requested a "copy of records, electronic, or otherwise, of each letter TO and FROM universities, companies, and organizations, from the OSTI 'cold fusion' documents collection." On July 11, 2016, you were emailed an interim response letter informing you of the need for OSTI to obtain release authorization from the Department of Energy. OSTI received notification to release the letters to you in their entirety on August 8, 2016. As a result, OSTI is releasing 72 cold fusion letters in this mailing on a CD-ROM because of the volume and file size of the PDFs.

In addition, there are approximately 13 letters that are currently being reviewed by the DOE's General Counsel Office (GC) for release or redaction. Upon receipt of guidance from GC, OSTI will release in whole or in part.

This decision, as well as the adequacy of the search, may be appealed within **90** calendar days from your receipt of this letter pursuant to 10 C.F.R. § 1004.8. Appeals should be addressed to Director, Office of Hearings and Appeals, HG-1, L'Enfant Plaza, U.S. Department of Energy, 1000 Independence Avenue, S.W., Washington, D.C. 20585-1615. The written appeal, including the envelope, must clearly indicate that a FOIA appeal is being made. You may also submit your appeal to OHA.filings@hq.doe.gov, including the phrase "Freedom of Information Appeal" in the subject line. The appeal must contain all of the elements required by 10 C.F.R. § 1004.8, including a copy of the determination letter. Thereafter, judicial review will be available to you in the Federal District Court either: 1) in the district where you reside; 2) where you have your principal place of business; 3) where DOE's records are situated; or 4) in the District of Columbia.

You may contact OSTI's FOIA Public Liaison, Charlene Luther, Office of Preservation and Technology at 865.576.1138 or by mail at the Department of Energy, Office of Scientific and Technical Information, 1 Science.gov Way, Oak Ridge, TN 37830 for any further assistance and to discuss any aspect of your request. Additionally, you may contact the Office of Government Information Services (OGIS) at the National Archives and Records Administration to inquire about the FOIA mediation services they offer.

The contact information for OGIS is as follows: Office of Government Information Services, National Archives and Records Administration, 8601 Adelphi Road-OGIS, College Park, Maryland 20740-6001, e-mail at ogis@nara.gov; telephone at 202-741-5770; toll free at 1-877-684-6448; or facsimile at 202-741-5769.

If you have any questions about the processing of the request or about this letter, please contact Madelyn M. Wilson at

Sincerely,

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ORNL/TM-11322

OAK RIDGE NATIONAL LABORATORY

MARTIN MARIETTA

A Preliminary Investigation of Cold Fusion by Electrolysis of Heavy Water

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M. Petek

OPERATED BY
MARTIN MARIETTA ENERGY SYSTEMS, INC.
FOR THE UNITED STATES
DEPARTMENT OF ENERGY

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Chemical Technology Division

A PRELIMINARY INVESTIGATION OF COLD FUSION BY ELECTROLYSIS OF HEAVY WATER

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CONTENTS

A PRELIMINARY INVESTIGATION OF COLD FUSION BY ELECTROLYSIS OF HEAVY WATER

ABSTRACT

Several tests have been made with electrolytic cells utilizing 0.1 to 0.2 N LiOD in D_2O as the electrolyte and a palladium cathode surrounded by a wire-wound platinum anode operating at cathode current densities of 100 to 600 mA/cm^2. The cathodes were swaged to diameters of 2.8 or 5.5 mm with 8.0 to 8.5 cm of active length and then annealed in some tests. The electrolyte temperature was controlled and heat was removed by flowing water in a cooling jacket, and the cell was insulated. Cooling water and electrolyte temperatures were determined by thermocouples; neutron and gamma-ray spectra were measured; and the electrolyte was periodically analyzed for tritium. In one test, an internal wire coil of platinum coated with palladium black was used in a closed system to recombine the electrolytically generated D_2 and O_2 without release of any off-gas.

The electrolyte was periodically sampled and electrolyte of the nominal concentration was added to replace the volume withdrawn; makeup D_2O was also added, when required, in those experiments which did not include a recombiner. Neutron and gamma-ray spectra were recorded on magnetic media; temperatures, coolant flow rate, and voltages were recorded and, in the last two experiments, acquired by a computer data acquisition system.

Tests up to 1000 h in duration were made, and in some experiments excess power was detected for periods of many hours, usually in the range of 5 to 15%. However, during one 12-h period, excess power of up to 50% was observed. On three separate occasions, the neutron count rate exceeded the background by three standard deviations; in addition, an apparent transient increase of tritium in the electrolyte by at least a factor of 25 occurred during one test.

1. INTRODUCTION

Recently, several research groups have reported that excess energy results during the electrolysis of heavy water with a LiOD electrolyte solution and palladium cathodes.[1-4] Neutron count rate anomalies have been observed in some cases, and an increase in tritium content of the electrolyte solution has also been measured.[4-7]

A group of Oak Ridge National Laboratory staff members, including chemists, physicists, and chemical engineers, have initiated an investigation with the goal of studying possible thermal or nuclear reactions that occur during the electrolysis of D_2O in a LiOD electrolyte with palladium cathodes. The experimental system utilizes positive heat removal by circulating cooling water, a design concept that is amenable to system scale-up and continuous operation. The goal of the research is to carry out the experiments with a very complete energy balance while simultaneously measuring the results of possible nuclear interactions.

2. MATERIALS AND METHODS

Two different primary electrolysis cell designs were used. The first was an open system in which the electrolytically produced gases, D_2 and O_2, were allowed to continuously exit the cell. A second design concept utilized a recombiner that catalyzed the recombination of D_2 and O_2 to form heavy water. The latter was a closed system that did not require replenishment of either D_2O or the electrolyte except when samples were withdrawn.

2.1 OPEN ELECTROLYSIS CELL

The open electrolysis cell was fabricated from Pyrex with a nominal ID of 4 cm and an active internal height of 12.5 cm (Fig. 1). It had an internal headspace of approximately 4 cm into which a N_2 purge gas could be introduced and removed. A Teflon cap was used to partially seal the system and allow the entrance of a glass tube for measurement of electrolyte temperatures, a glass-encased resister for internal calibration, and a polyethylene tube for the introduction of makeup D_2O and removal of electrolyte samples. The cell was surrounded by a cooling jacket with forced water flow and thermocouples inserted in the inlet and outlet streams. Two-inch-thick fiberglass insulation covered the entire exterior of the cell except for a 1- by 3-cm opening used to observe the electrolyte level.

2.2 CLOSED ELECTROLYSIS CELL

A second design concept was similar to that described above, except that the cell body was fabricated from 1.5-in. Pyrex pipe and the top was sealed with a conventional glass pipe flange (Fig. 2). Total recombination of the evolved D_2 and O_2 was carried out within an expanded gas space of the enclosed electrolysis cell. The recombiner was fabricated from 375 cm of 32-ga* platinum wire in a special coil configuration in the gas space (see Fig. 3) that had been electrochemically coated with 10 wt% palladium black. The wire was wrapped around six Teflon-sheathed screws that were attached to the top flange and extended down into the gas space. Teflon tubing, connected through the flange to the gas space, exited through a heavy-water bubbler so that off-gas could be detected. The gas-tight top flange made from Teflon provided, through conventional tubing compression fittings, electrode connections and entrance of an electrolyte thermocouple sheathed in polyethylene.

*Here, ga = gauge.

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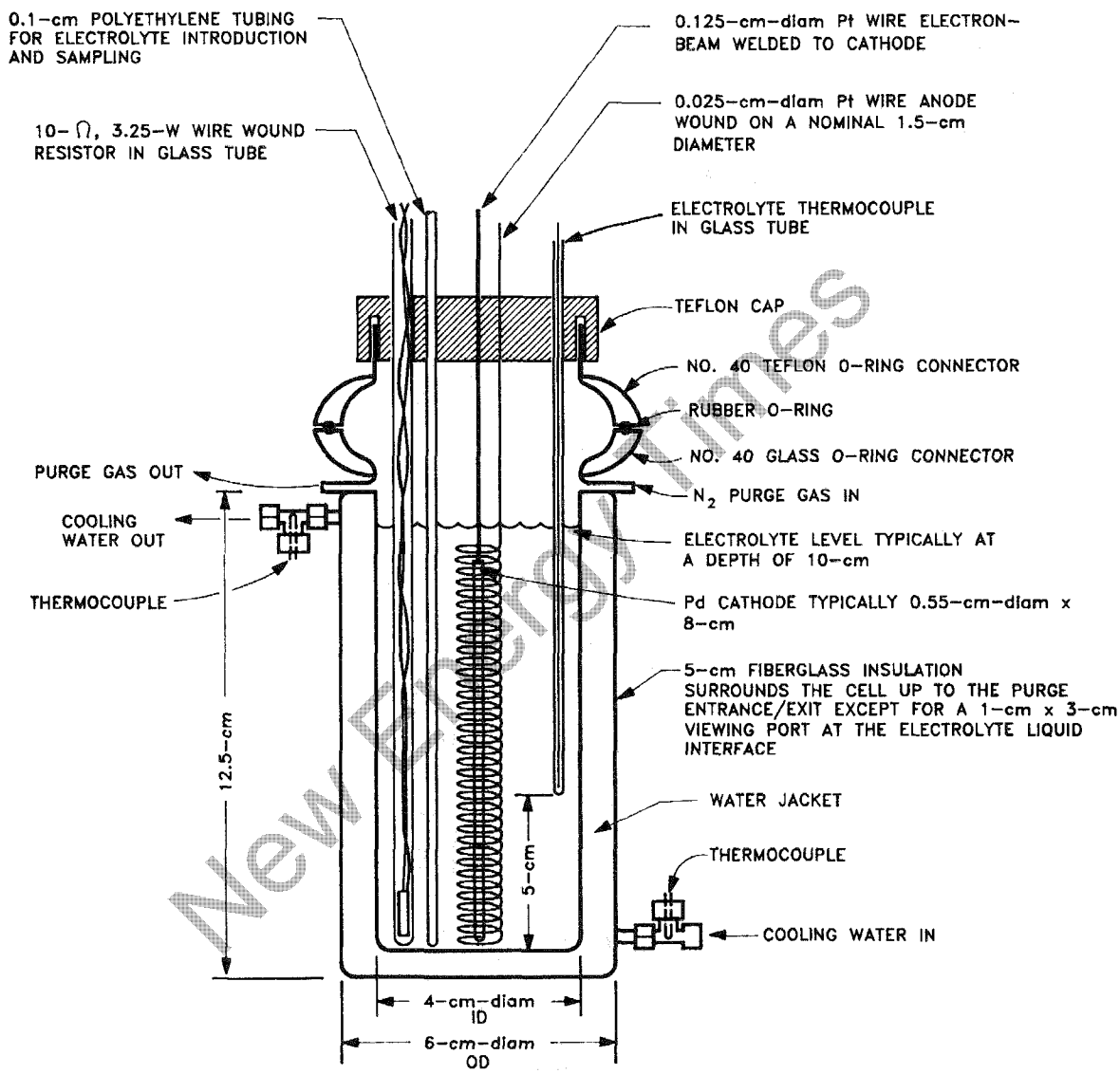

Fig. 1. Open-system electrolysis cell used in cold fusion tests.

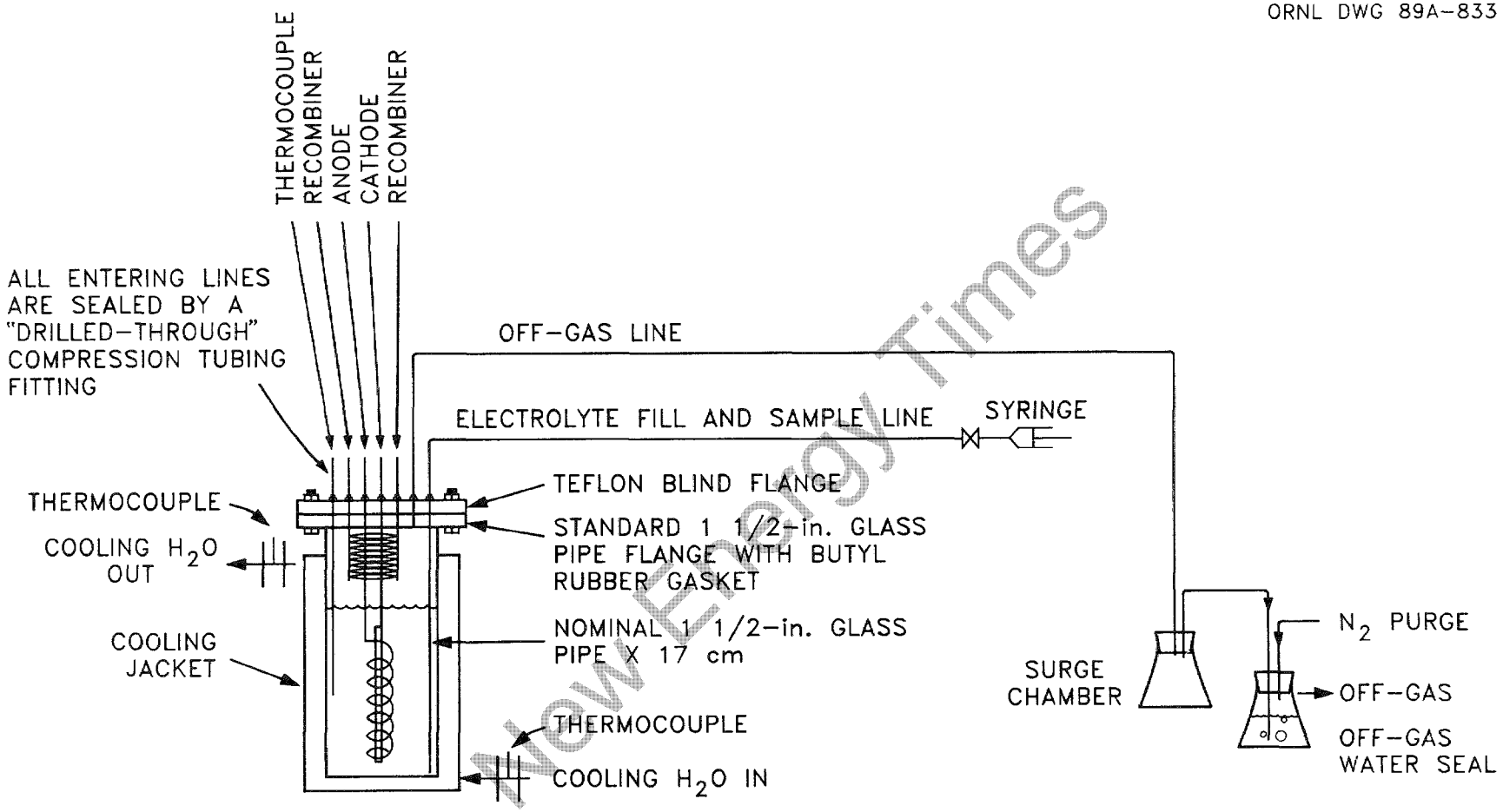

Fig. 2. Closed-system electrolysis cell used in cold fusion tests.

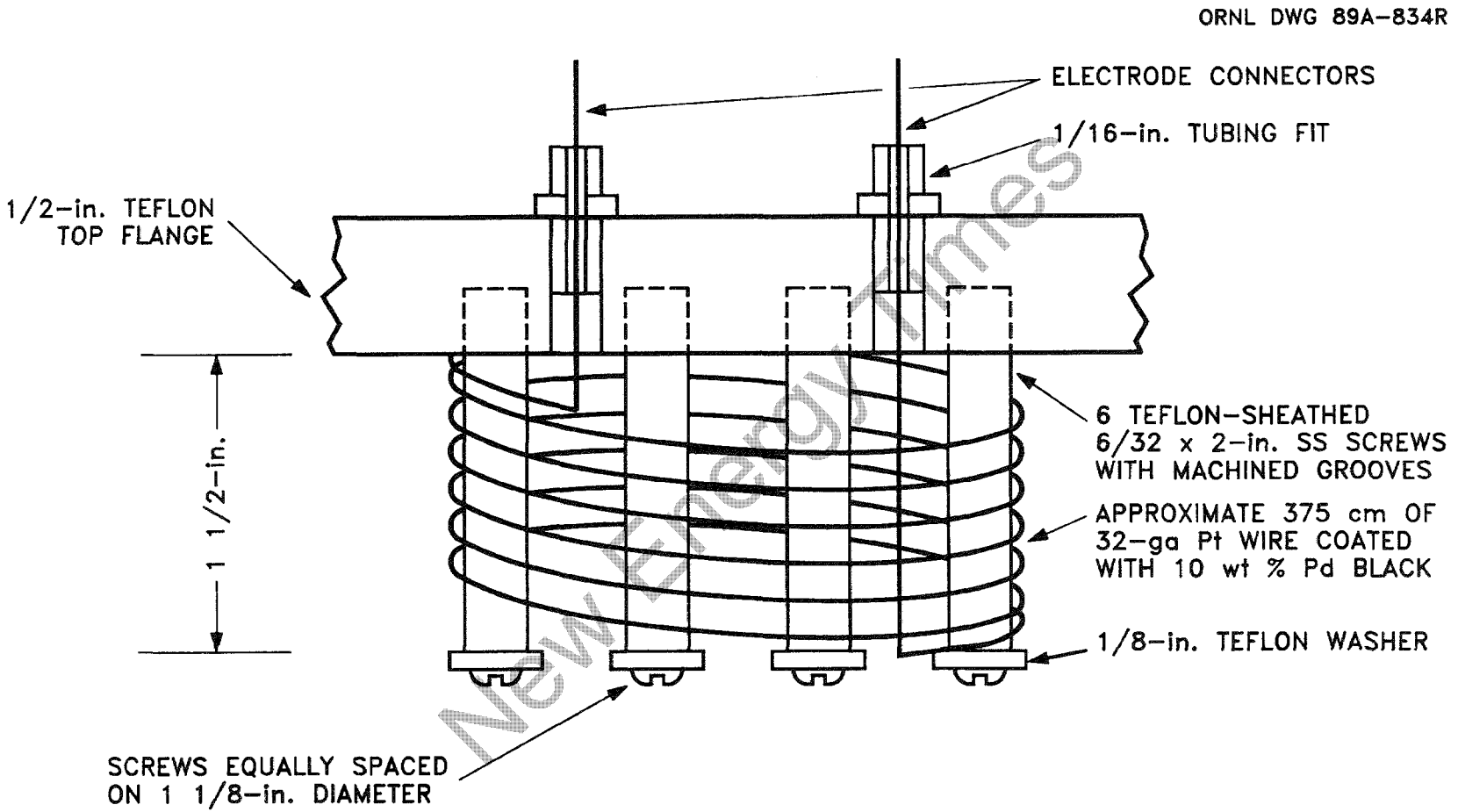

Fig. 3. Configuration of the recombiner used in the closed system.

Operation of the recombiner was initiated by imposing an electrical current of 0.7 A (2.4 V) over a period of 4 to 6 h, after which no detectable off-gas from the system was observed. After initiation, the recombiner electrical current was required only if the cell current has been stopped for an extended period and then restarted.

The cooling jacket did not extend over the entire outside surface of the cell because space was needed for the glass pipe flanges. Thus, the recombiner section, probably operating at a relatively high temperature in comparison to the electrolyte, did not have access to primary heat removal by the cooling water. This necessitated adding four more inches of fiberglass in order to reduce the heat loss at the top of the cell.

2.3 SYSTEM DESIGN

Heat removal from the electrolysis cell was determined by the temperature increase of the forced flow of distilled water. A circulating coolant bath with the temperature controlled to within 0.1°C was used, along with an external positive-displacement pump that controlled the coolant flow rate with an accuracy of 0.5% (Fig. 4). The N_2 purge gas was monitored by a rotameter, while D_2O was added and electrolyte was sampled by means of a syringe connected to a Teflon tube that entered through the flange and extended to the bottom of the cell.

The electrical power supply operated at a constant current with an accuracy of ±1 mA. Electrolyte and coolant water inlet and outlet temperatures were measured by calibrated thermocouples, and the overall electrode voltage was determined to within 0.01 V. These system parameters were recorded on a strip-chart recorder every 6 s. With some tests, an on-line data acquisition system was also used to acquire all temperature and voltage measurements. Calibration of the thermocouples, including curve fitting by fourth-order

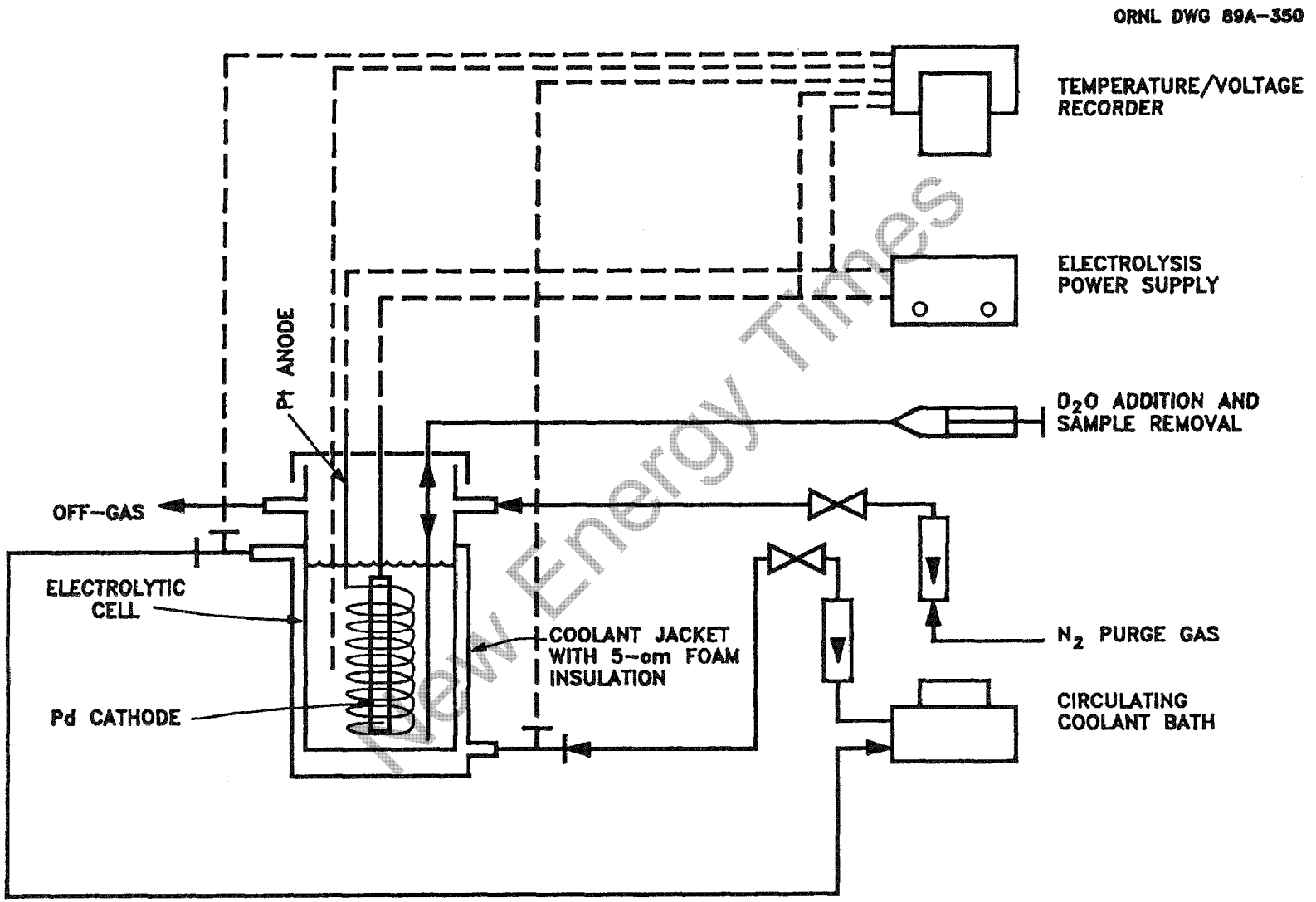

Fig. 4. Schematic of cold fusion equipment.

polynomials, allowed temperature measurements to within 0.05°C. This PC-supported system also calculated a heat balance at 1-min intervals. The calculated energy measurement error for this experiment was approximately 0.2 W.

2.4 RADIATION DETECTION

The experimental system was contained within a 2-ft-thick concrete enclosure in order to reduce the neutron background. Neutron count rates were measured by an NE-213 scintillator that was placed immediately adjacent to the insulated cell (Fig. 5) and employed pulse-shape discrimination having a threshold of 1.2 MeV. The overall detection efficiency was 1.46×10^{-3} as determined by a ^{252}Cf source. The gamma-ray contribution to the neutron peak was 4.1% of the gamma peak, or approximately 20% of the neutron peak. The reported neutron count rates were corrected for this contribution. The neutron detector had a typical sensitivity at three standard deviations of 3×10^{-24} fusion per deuterium pair per second. The same scintillation system allowed the detection of gamma rays with energies above a threshold of about 3 MeV.

A separate gamma-ray spectrometer (NaI detector) was also used for the detection of neutrons via a polyethylene converter. This system was lead shielded and contained in an airtight enclosure to reduce the background gamma-ray spectrum. A multichannel analyzer with the window set for the expected capture gamma-ray region was used, and the system had an overall detection efficiency of 5.75×10^{-5} as determined by a ^{252}Cf source; however, it had somewhat less sensitivity due to the higher gamma-ray background. Both the neutron and the gamma-ray spectra were periodically recorded on magnetic media by a small computer system. The tritium concentration in the electrolyte was measured periodically by removing small volumes of the electrolyte and counting with a liquid scintillation system.

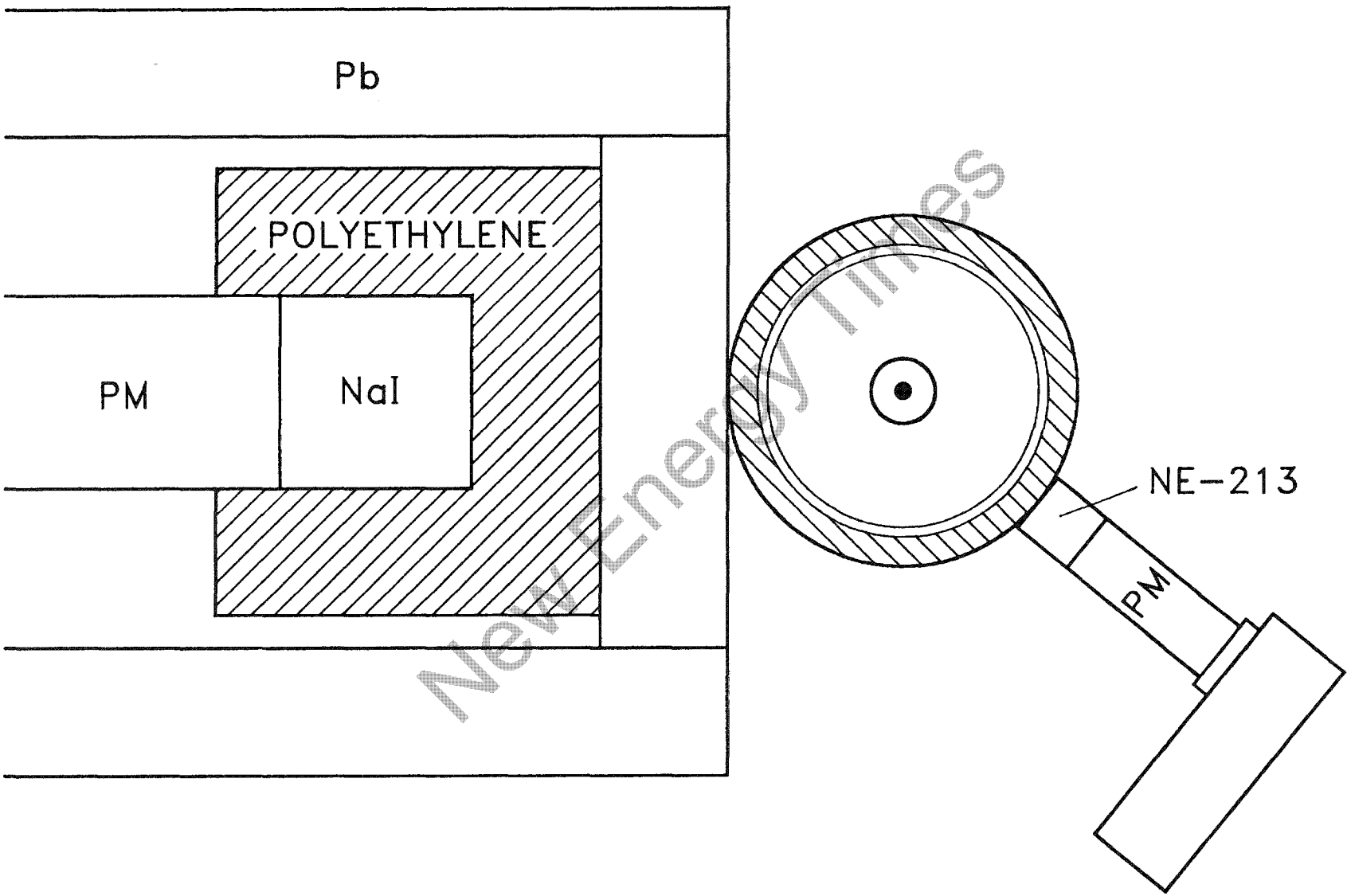

Fig. 5. Detection of neutrons and gamma rays.

2.5 MATERIALS

Some of the earlier tests were made with heavy water* that was 99.5% D_2O with an initial tritium content of 1 x 10^6 Bq/L. The most definitive tests were made with a very pure deuterium oxide obtained from Aldrich Chemical Company, Inc., and designated as 99.9 atom % D with a tritium content of 2000 Bq/L. The electrolyte was prepared by dissolving reagent-grade, natural lithium in the D_2O at a nominal concentration of 0.1 to 0.2 N.

The cathode material was 99.9% palladium (Materials Research Corporation) that was cast in argon and then swaged to the desired diameter. This cylindrical material was then cut to the desired length; a 0.13-cm platinum connecting wire was electron-beam-welded to the top; and, in some tests, the cathode was annealed at 900°C for 2 h in vacuum (Table 1).

All of the anode material and the recombiner were fabricated from 99.9% platinum wire in the size range of 24 to 32 ga (Englehard Corporation). The anodes consisted of wire coils made by wrapping the wire around the exterior of a skeletal glass mandrel (four 0.15-cm-diam glass rods with cross bracing) that surrounded the cathode and provided an electrode spacing of 0.3 to 0.5 cm.

*Available from previous work.

Table 1. Operating conditions for the cold fusion tests

Test	Cathode	LiOD concentration (N)	Cathode current density (A/cm^2)
CF-1	0.28-cm-diam x 8.5 cm; cast and swaged but not annealed	0.2 for entire test	100 for entire test
CF-2	0.56-cm-diam x 8.5 cm; cast and swaged but not annealed	0.2 for entire test	100 for 0 to 169 h 200 for 169 to 172 h 400 for 172 to 220 h 200 for 220 to 241 h 400 for 241 to 363 h
CF-3	0.28-cm-diam x 8.5 cm; swaged and annealed at 900°C for 2 h in vacuum	0.2 for 0 to 621 h 0.3 for 621 to 660 h 0.5 for 660 to 886 h 1.0 for 886 to 1050 h	200 for 0 to 504 h 400 for 337 to 501 h 600 for 50l to 1050 h
CF-4	0.28-cm-diam x 8.0 cm; swaged and annealed at 900°C for 2 h in vacuum	0.1 for entire test	100 for 0 to 426 h 200 for 426 to 667 h 300 for 667 to 953 h 400 for 953 to 1000 h

2.6 START-UP PROCEDURES

The tests were initiated by loading the prepared electrolytic cell with approximately 125 mL of the electrolyte, starting the purge gas at approximately 1 mL/s, if required, and turning on the electrical current. The electrolyte temperature was controlled by the cooling water flow rate and was usually maintained in the range of 28 to 32°C, but there were some controlled excursions up to 70°C and down to 10°C. More D_2O was added to the electrolyte every 8 h in order to maintain a constant electrolyte inventory for the open-system tests, and electrolyte samples were taken periodically (every 2 to 3 d). The gamma-ray and neutron spectra were measured continuously and recorded every 4 h.

2.7 ENERGY BALANCE

An energy balance was determined for each test based on the following assumptions: (1) the electric current is 100% efficient for the electrolysis of D_2O; (2) the system operates at quasi-steady state with a constant inventory of deuterium in the cathode; (3) except where internal recombination was used, all of the D_2 and O_2 exit the electrolysis cell without recombination; (4) the exiting gases, including a purge gas, were saturated with D_2O that was at equilibrium with heavy water at the temperature of the electrolyte; and (5) there was no heat loss to the ambient environment.

The second assumption will obviously not be correct when a major portion of the formed D_2 is being adsorbed by the cathode, but this will be true for only a very short period in the early phases of the test. Since the volume of makeup heavy water required to be added to maintain a constant inventory in the electrolysis cell was approximately equal to the volume electrolyzed and evaporated for the open systems, it was assumed that very little recombination of the electrolysis gases occurred. Conversely, no addition of D_2O or electrolyte was required for the closed systems with recombination except to replace samples that were withdrawn. There was obviously some heat loss to the ambient, although the system was well insulated, but this would result in a conservative estimate of the recovered heat. Necessary chemical and physical properties for D_2O were obtained from reference handbooks.[8]

2.7.1 Open System

The resulting energy balance for the open systems can be represented by:

ENERGY IN:

(Volts) x (Amps);

ENERGY OUT:

ELECTROLYSIS (Typically >30%)

D_2O --> D_2 + 1/2 O_2 (-Heat of Formation);

FORCED COOLING (Typically >60%)

(Cooling Water Temperature Increase) x (Flow Rate)

LATENT HEAT (Typically <2%)

(Heat of D_2O Vaporization).

2.7.2 Closed System

The closed system with recombination allows a much simpler energy balance with fewer assumptions:

ENERGY IN:

(Volts x Amps);

ENERGY OUT:

(Cooling Water Temperature Increase) x (Flow Rate).

3. RESULTS AND DISCUSSION

Four long-term (hundreds of hours) electrolysis tests have been made in which a palladium cathode and a platinum anode were used in the presence of 0.1 to 1 N LiOD in D_2O (Table 1). Although each electrolysis cell and supporting equipment are similar, there has been an evolution of design, culminating with a totally closed system in which internal D_2-O_2 recombination was achieved. In each test, the total heat removed was determined by the increase in temperature of the circulating cooling water in a water jacket

around the electrolysis cell, and neutron and gamma-ray measurements were made throughout the test period. Tritium measurements were made on electrolyte samples that were periodically removed from the cell.

The results of these tests are very preliminary and certainly do not confirm "cold fusion"; however, there have been (1) periods of several hours when apparently a significant amount of excess heat was produced, and (2) periods when the neutron counts were greater than three standard deviations above the previously measured background. Tritium increase was indicated on one occasion.

3.1 OPERATION OF THE INTERNAL RECOMBINER

It is well known that platinum and/or palladium can be effectively used as a catalyst for the recombination of H_2/D_2 and O_2. Since we were interested in a well-controlled catalytic system that could be effectively used in the gas space of an electrolytic cell, several tests were made on the use of platinum wire in a coil configuration (Fig. 6). The first tests were made with a coil of 24-ga platinum wire in the gas space of an enclosed electrolytic cell utilizing platinum electrodes in an electrolyte containing 0.2 N LiOH in H_2O. Electrical current could also be imposed on the recombiner wire for heating purposes.

It was found that with the electrolysis cell operating at 100 mA and ambient temperature and pressure, it was necessary to introduce over 2 W of energy into the platinum recombiner coil in order to induce a measurable decrease in the off-gas (Table 2). However, when 3.3 W of electrical energy was imposed on the coil, all of the off-gas stopped, indicating complete recombination; in fact, an appreciable vacuum could be maintained on the system. It was necessary to continue to add heat to the coil in order to ensure recombination. When the cell current was increased to 150 mA, it was impossible to completely stop the off-gas — a situation that indicates incomplete recombination.

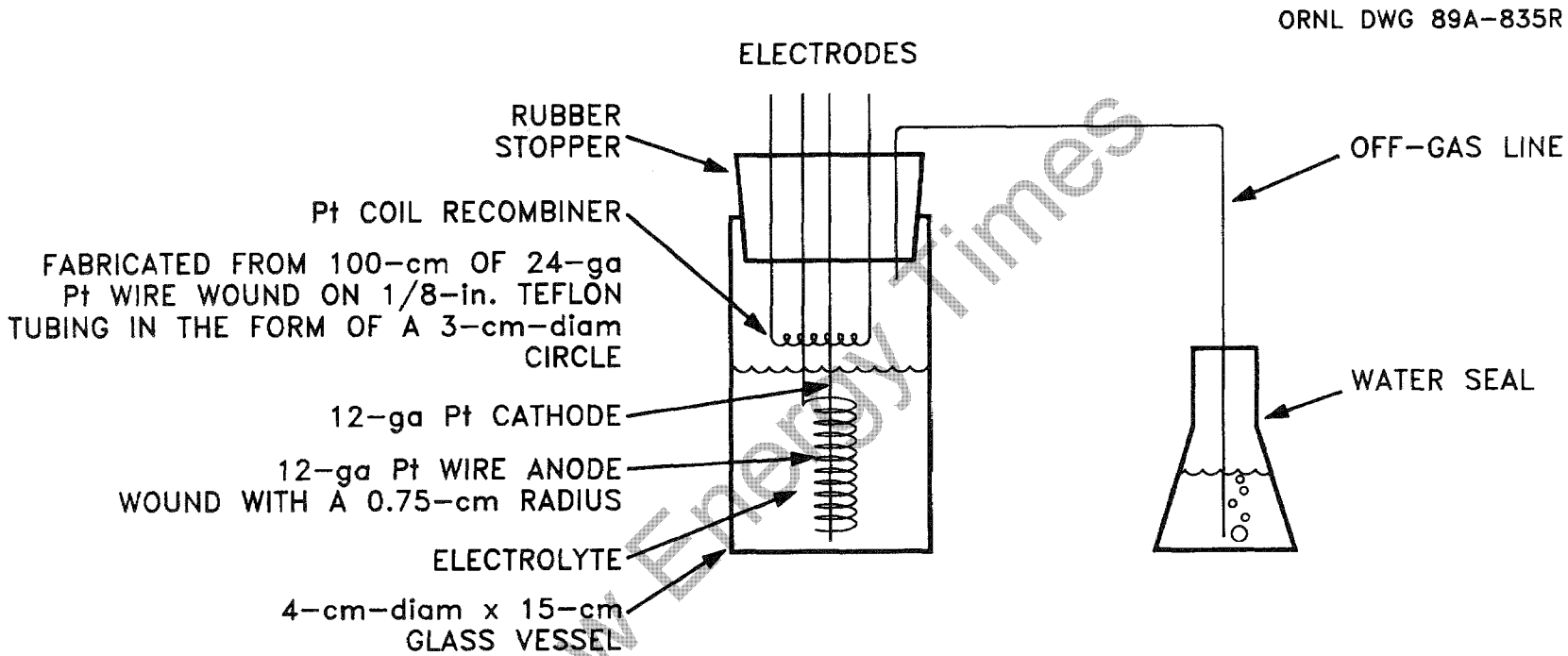

Fig. 6. Experimental apparatus for the testing of a platinum-wire recombiner in a closed electrolysis cell.

Table 2. Test of a platinum wire coil as a H_2-O_2 recombiner in a closed electrolysis cell[a]

Recombiner power (W)	Relative off-gas rate (% of initial)	Comments
0.0	100	
0.22	100	
0.92	100	
2.10	80	
3.28	0	5-cm H_2O vacuum
2.66	60	
2.16	90	
3.20	0	4-cm H_2O vacuum
3.20[b]	80	

[a]An enclosed electrolysis cell with an off-gas line exiting to a water trap was used at ambient conditions. The electrodes were 12-ga platinum wire suspended in 0.2 N NaOH, and the recombiner coil was 1 m of 24-ga platinum wire wound on 1/8-in.-diam Teflon tubing in a circular configuration. The electrolysis current was 100 mA unless otherwise specified.

[b]The electrolysis current was 150 mA.

It was expected that palladium with a large amount of surface area would be more effective as a recombination catalyst. Thus, a second catalytic system was fabricated from platinum wire that had been previously coated with palladium black (Fig. 2). It was found that the palladium-coated wire was extremely efficient as a recombiner with electrolysis currents in the range of 0.71 to 3.5 A (100 to 500 mA/cm^2) as long as the interaction was first initiated by electrically heating the wire coil with approximately 1.6 W (Table 3). After initiation, the heat of recombination was sufficient to maintain complete recombination, and the system was operated under a slight vacuum.

Table 3. Test of a palladium-coated platinum wire as a H_2-O_2 recombiner in a closed electrolysis cell[a]

Electrolysis electrical current (A)	Recombiner power (W)	Relative off-gas rate[b] (% of initial)
0.71	0	100
0.71	0.12	100
0.71	0.48	100
0.71	1.14	91
0.71	1.60[c]	0

[a]An enclosed electrolysis cell with an off-gas line exiting through a water seal was used at a temperature of 25 to 30°C. The configuration and operating conditions are described in Fig. 2 and Table 1.

[b]Values after operation at 5 to 15 min at these conditions.

[c]After initiation, the recombiner power was reduced to 0. The electrolysis current was later increased to 3.5 A without any indication of an off-gas.

3.2 EXCESS HEAT

Several periods of apparent excess energy have been observed in both the open and the closed systems; some of these lasted for many hours.

3.2.1 Open System

With the various assumptions described above, including no recombination of D_2 and O_2 within the cell, heat in excess of that provided by joule heating was detected for periods of several hours in two different open-system experiments. This excess heat was usually in the range of 5 to 15%; however, during one 12-h period of Run CF-2, up to 50% was seen (Fig. 7). The experimental uncertainty during this time was calculated to be 3 to 5%.

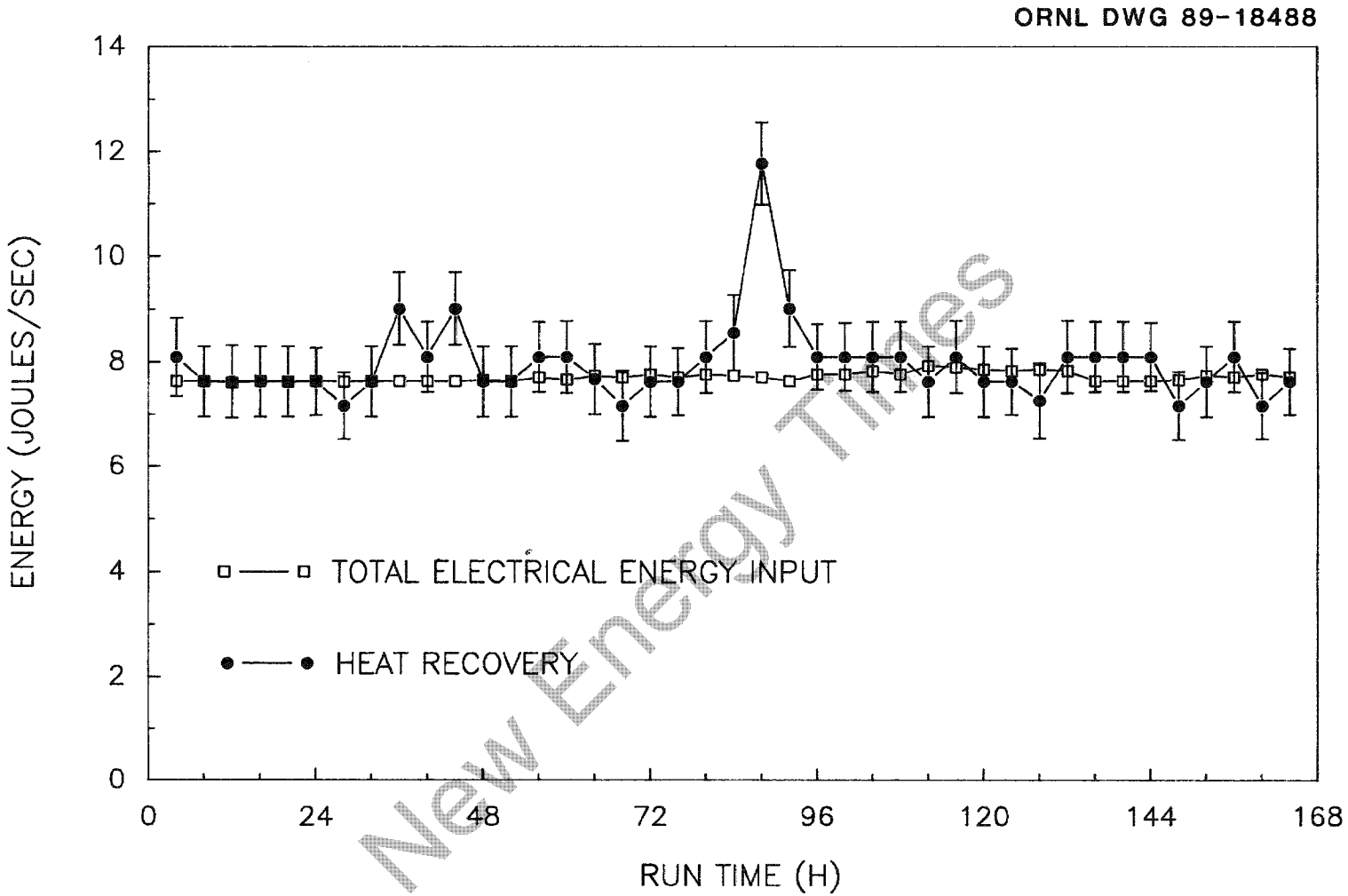

Fig. 7. Energy balance for Test CF-2.

There was no indication of extended excess power generated during Run CF-1. One extended period of excess power in Run CF-3 coincided with an increase in the cathode current density and was extended by perturbing the system — for example, by changing the electrolyte temperature or increasing the electrolyte concentration (Fig. 8).

3.2.2 Closed System

During the first 740 h of operation with the closed system, the power balance was slightly negative (typically 1 to 2% after additional insulation was included), indicating a slight heat loss through the cell insulation. Starting at about 740 h and continuing for an additional period of more than 200 h, an increasing positive power balance in excess of 5% was observed at some points (Fig. 9). The estimated error of measurement for the test is approximately ±2%; however, this discrepancy may be partially offset by the apparent 1 to 2% heat loss to the environment.

3.3 NEUTRON COUNT RATES

In Run CF-1, in which a 0.28-cm-diam x 8.5-cm palladium cathode was used at a current density of 100 mA/cm^2, the neutron count rate displayed an average background of 13.1 neutrons/4 h with a standard deviation of 5.3 neutrons/4 h. An interesting period was observed during the first few hours of this test when the neutron count rate was 32 neutrons/4 h, which exceeded the background by about three and one-half standard deviations (Fig. 10). This result can be further put in perspective by considering a frequency histogram in which the above-mentioned neutron count rate for the 4-h period was well outside the assumed binomial distribution of the count rates; thus, it represents an anomaly of unknown genesis (Fig. 11). On two other occasions, the neutron count rate exceeded the previously measured average values by greater than three standard deviations:

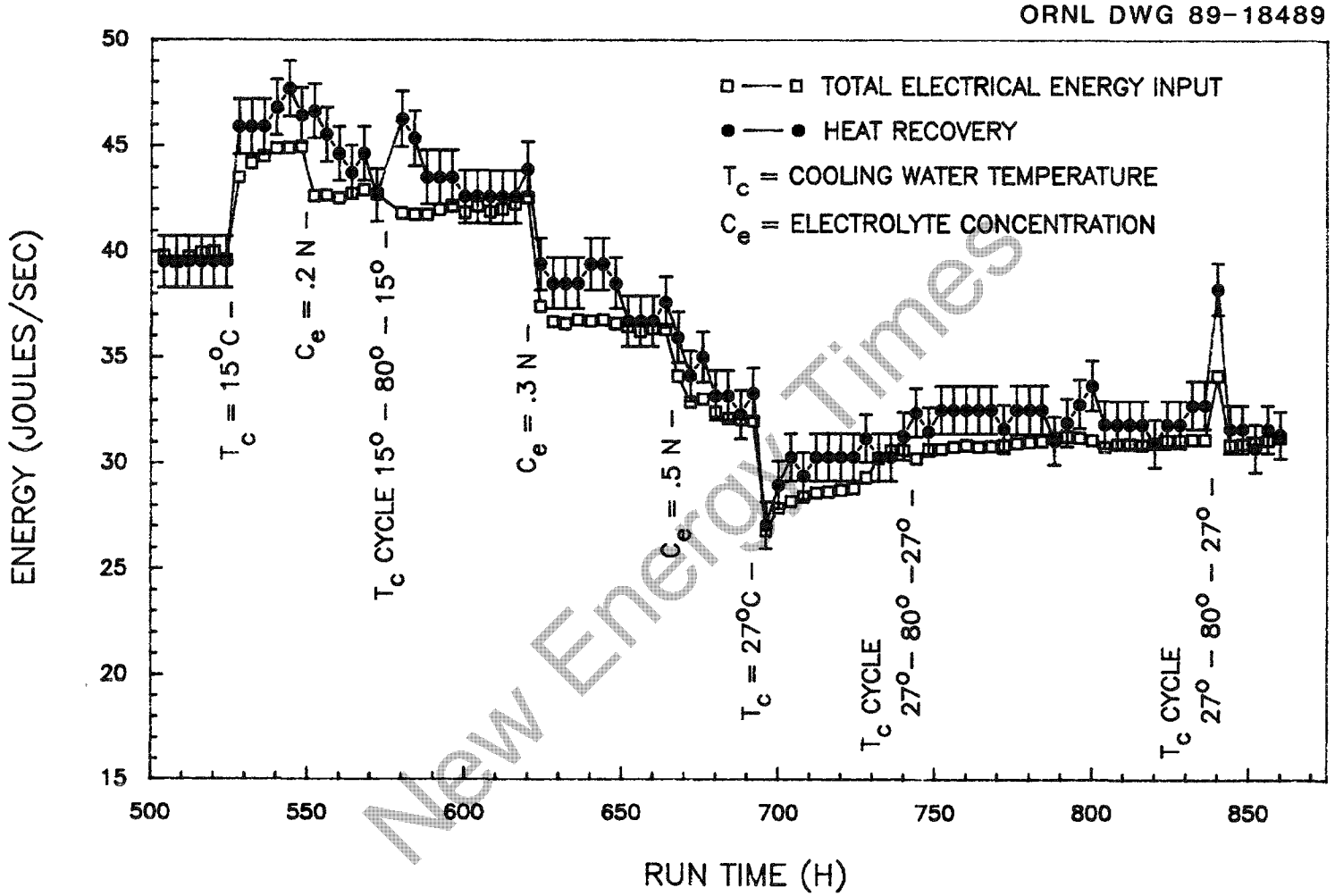

Fig. 8. Energy balance for last 500 h of Test CF-3.

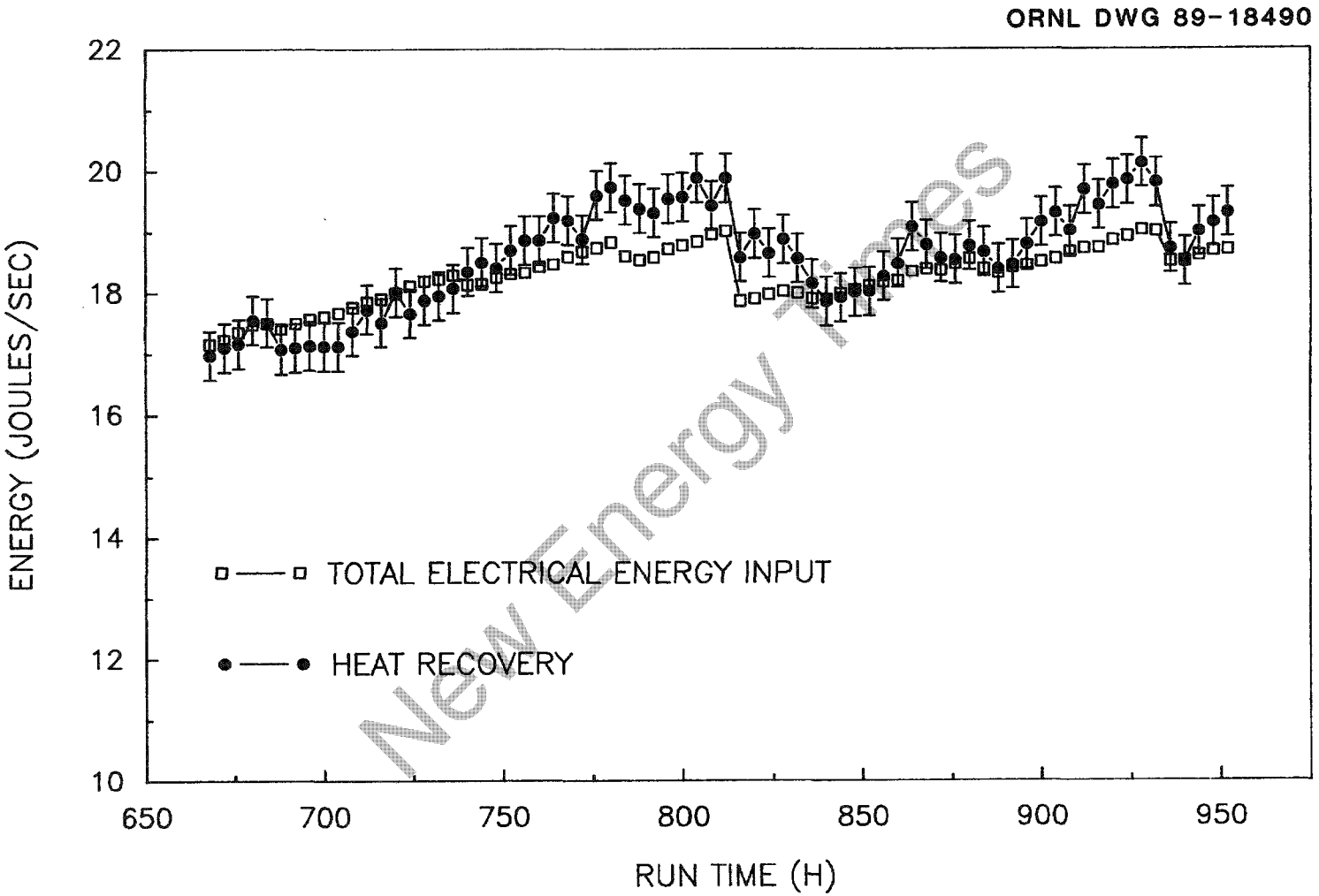

Fig. 9. Energy balance for last 300 h of Test CF-4.

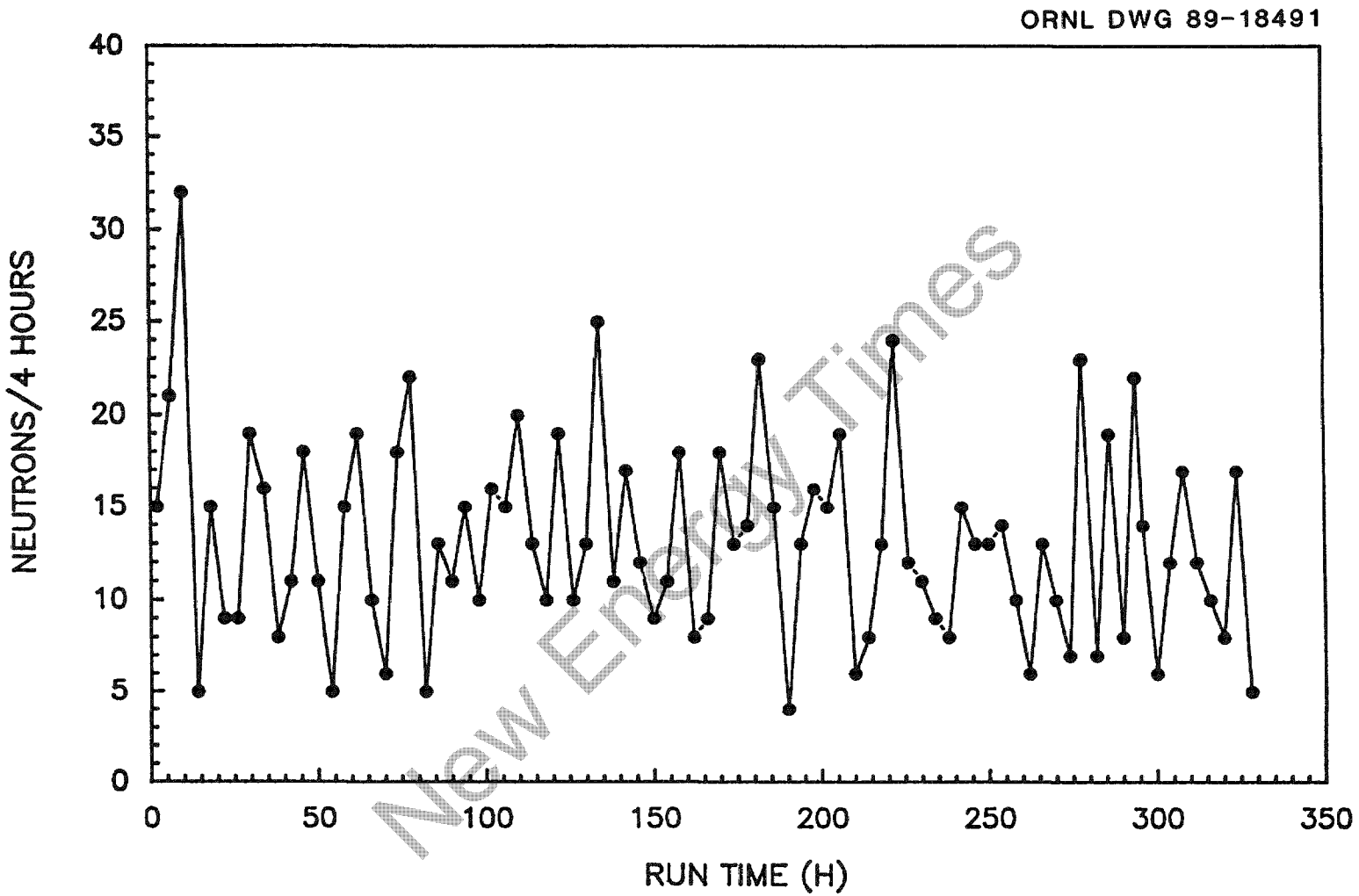

Fig. 10. Neutron count rate (corrected for overlapping gamma rays) for Test CF-1.

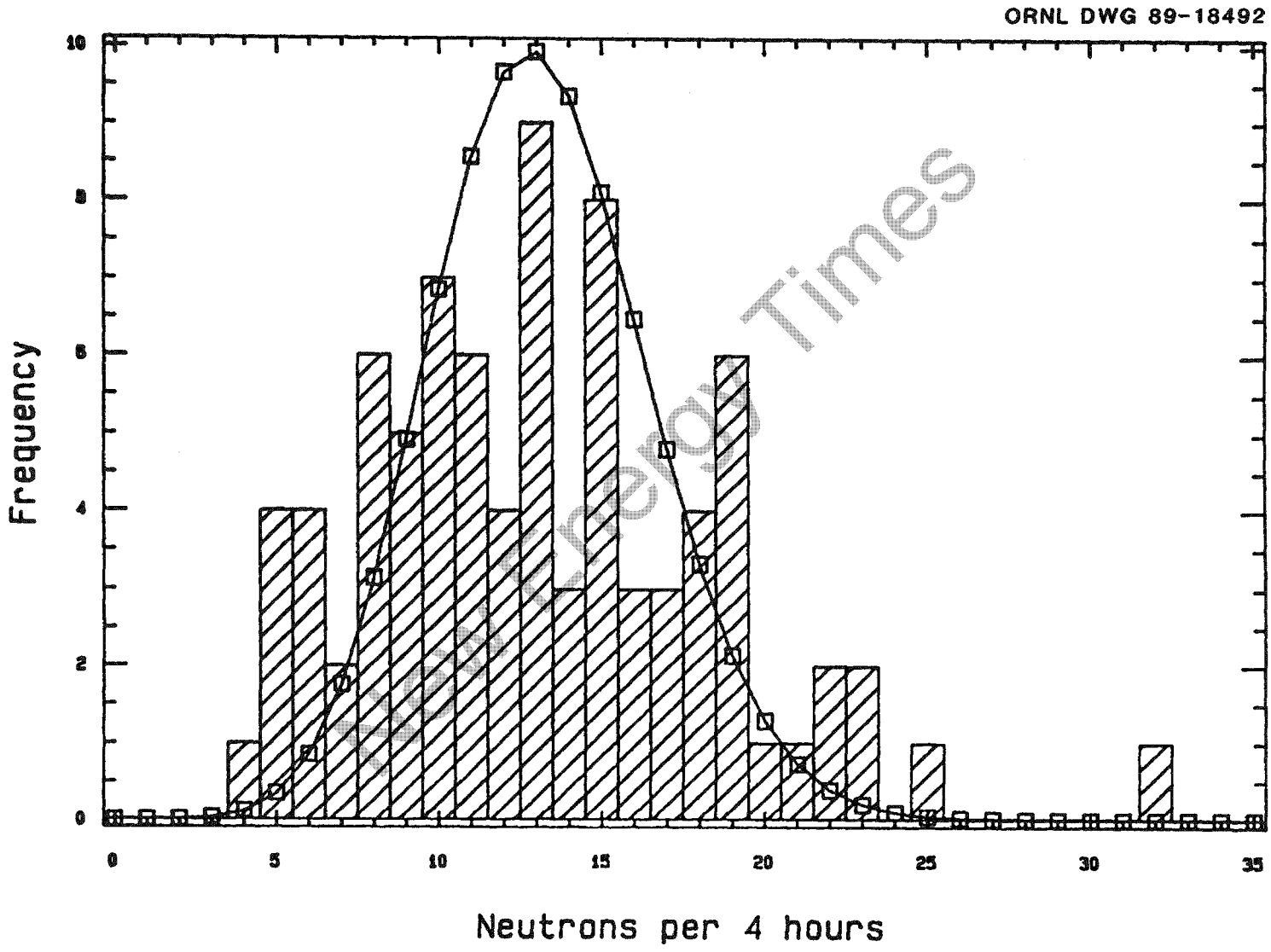

Fig. 11. Frequency histogram of the neutron count rate (corrected for overlapping gamma rays) for Test CF-1 imposed on an assumed binomial distribution.

(1) at about 90 h into Run CF-3 and (2) after more than 700 h into Run CF-4 (Figs. 12 and 13). The latter measurement occurred during the period of an increasing power excess.

3.4 TRITIUM

During Test CF-3, the tritium concentration in the electrolyte apparently increased by about a factor of 25 during the first 2 d of the experiment (Fig. 14). The tritium content then decreased to the starting concentration as fresh D_2O was added to the electrolyte solution to make up the electrolysis losses. Although contamination of the electrolyte could have produced such results, there was no indication that this happened. At other times, small tritium increases were measured in the electrolyte, but they did not exceed the possible increase due to the evaporation and electrode processes.

3.5 ADDITIONAL OBSERVATIONS

In all tests except CF-4, D_2O was periodically added to maintain the heavy-water inventory. This resulted in a changing LiOD concentration and, thus, unsteady-state operation. A series of tests was also made at different electrolyte temperatures over the range of 30 to 70°C with no apparent effect except when the cycling period was relatively short.

4. CONCLUSIONS

Preliminary tests of the electrolysis of D_2O utilizing LiOD electrolytes and palladium electrodes have not confirmed the "cold fusion" phenomena. However, there have been several apparently anomalous neutron count rates, one unexplained 25-fold increase in tritium, and periods of many hours of apparent excess energy. None of these results has been precisely reproduced, nor can they be explained by conventional nuclear or chemical theory.

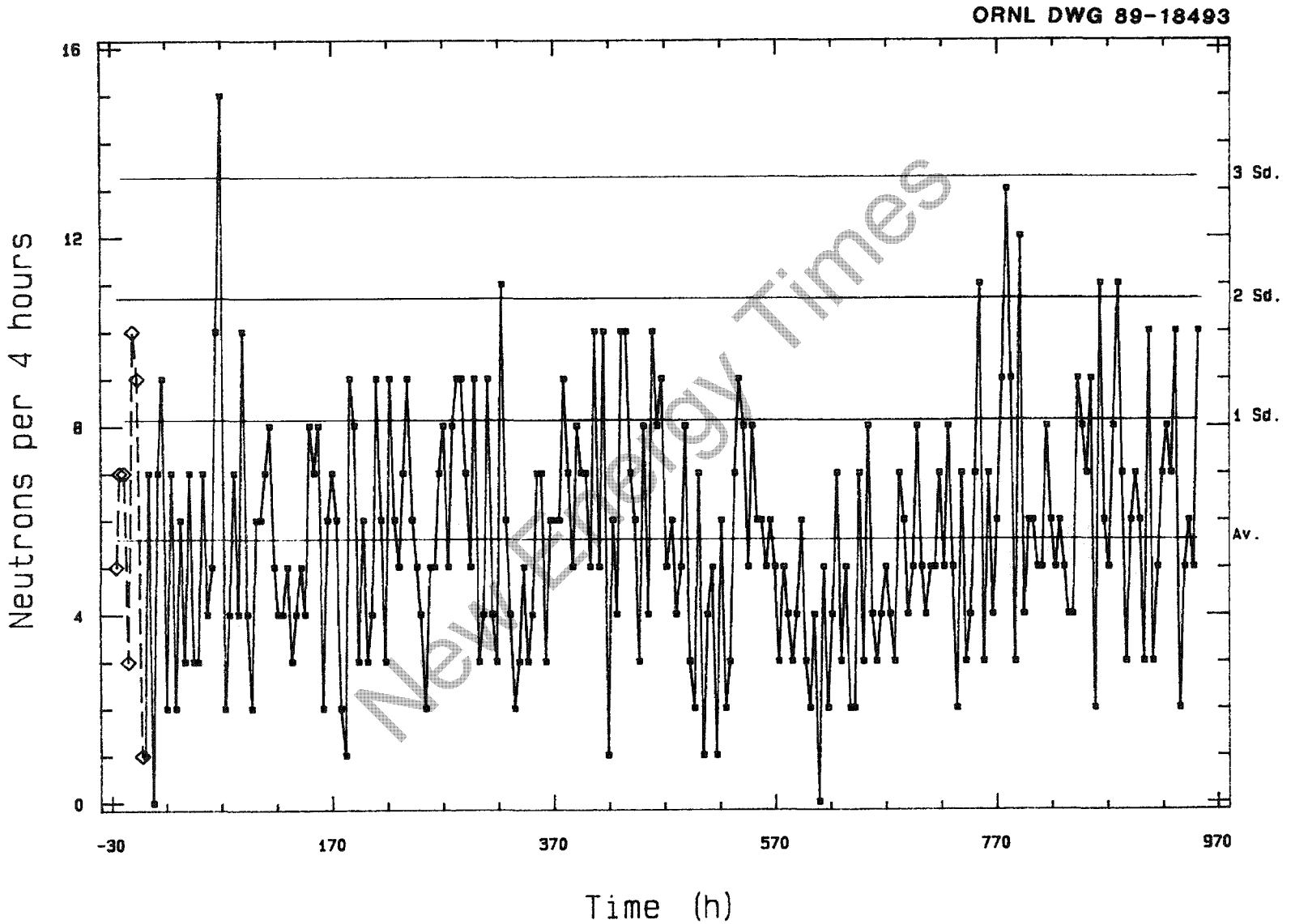

Fig. 12. Neutron count rate corrected for overlapping gamma rays for Test CF-3.

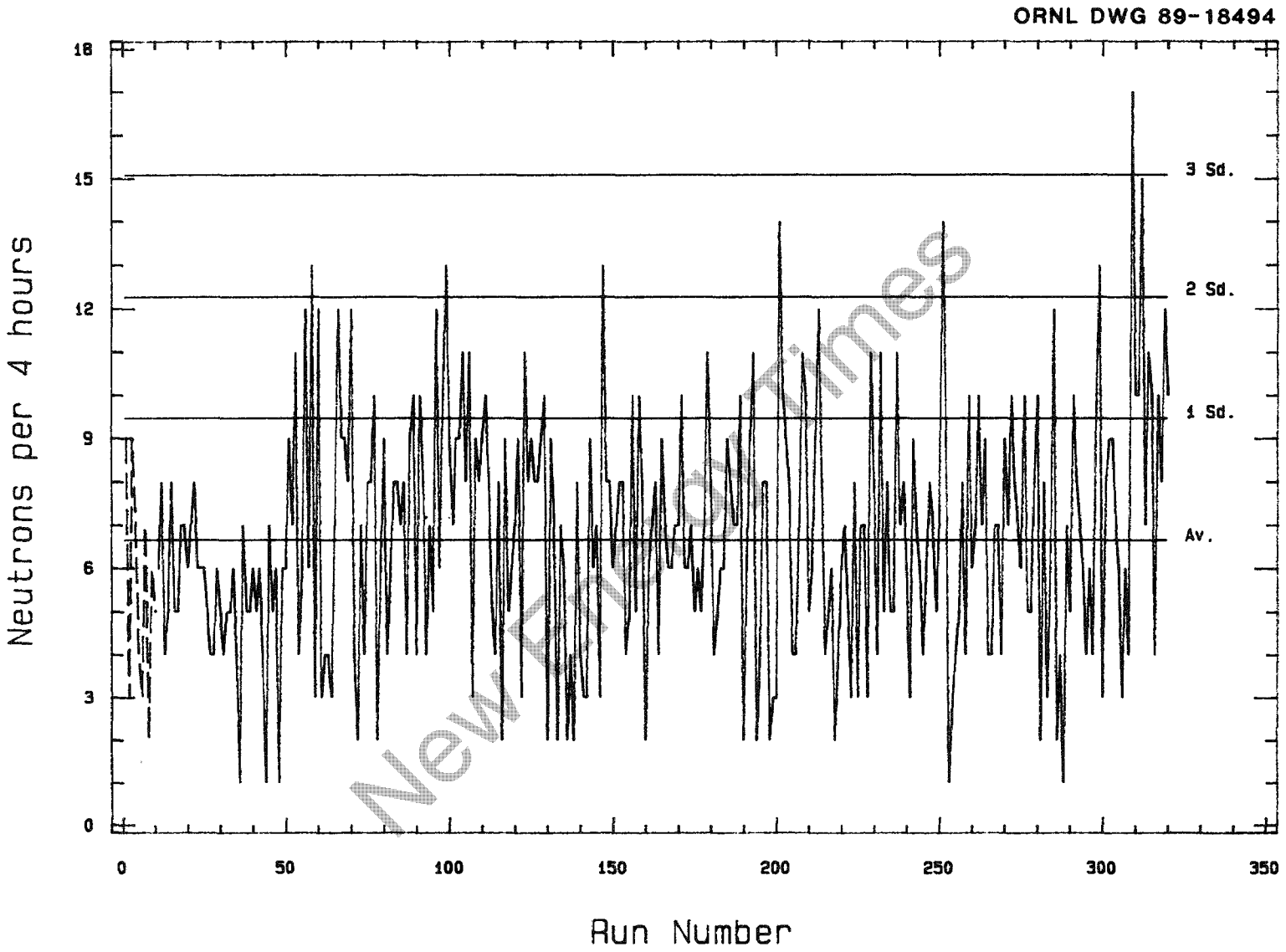

Fig. 13. Neutron count rates corrected for overlapping gamma rays for Test CF-4.

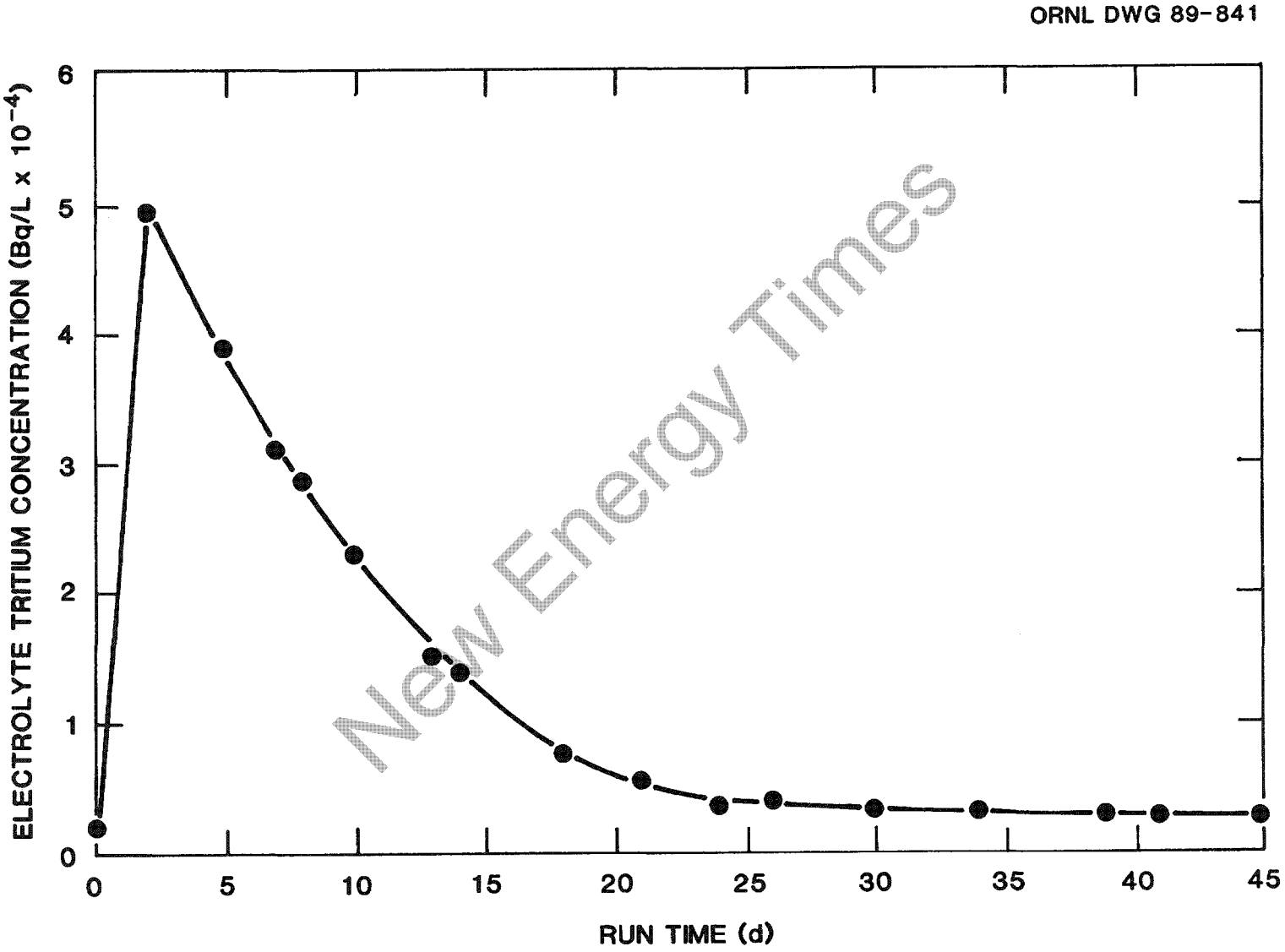

Fig. 14. Electrolyte tritium content during the first 40 h of Test CF-3.

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