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ABSTRACT

Because water-cooled molten 83 a/o Pb-17 a/o Li (99.3 w/o Pb - 0.7 w/o Li) eutectic alloy is being considered as a blanket for nuclear fusion applications, it is important to understand the interactions that might occur if this alloy were inadvertently to contact liquid water. We have, therefore, investigated the behavior of 120 g of the molten alloy at 600°C when impacted with a vertical 2.4 m-tall column of water at 25°C or 60°C. The behavior was compared with similar impacts of 25°C or 60°C water on 140 g of molten lead at 600°C. (Temperatures and weights above are nominal.) Also, two preliminary experiments were performed with empty crucibles.

Multiple pressure spikes were produced with the Pb, while essentially only one initial pressurization followed by a few strongly damped minor peaks were observed with the alloy. H_2 and aqueous solutions of LiOH were generated with the alloy, but not with the lead. Also, an aerosol with an acrid odor was detected in the gases vented about six minutes after the alloy-water interactions. It was assumed to be Li-bearing, but was not studied further. The debris recovered after the water impacts on both Pb and the alloy was highly porous.

The amount of H_2 estimated by quadrupole mass spectrometry is about 20% less than that estimated on the basis of titration of the OH^- ion. This observation may be due to sampling of the gases within minutes of the interaction, before the melt had cooled completely, while the solutions for titration were collected many hours after the system had equilibrated both thermally and chemically.

Our mass spectrometric measurements of the H_2 generated per gram of alloy are compared with values determined gaseometrically both by Biney and by Herzog in earlier experiments in which the molten alloy was flooded with water. Our values agree well with those of Biney but are about twice as large as Herzog's.

The phenomena observed can be reasonably interpreted on the basis of strong hydrodynamic fragmentation of the melt during the melt-water contact, followed by chemical reactions of the melt with steam and liquid water.

ACKNOWLEDGMENTS

We are pleased to acknowledge the financial support of the U.S. Department of Energy's Office of Fusion Energy and the EG&G Fusion Safety Program provided via Dr. Kathy A. McCarthy of the National Engineering Laboratory, Idaho Falls, Idaho.

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INTRODUCTION

The lead-lithium alloy system is unusual because it is formed from lithium, a very low density, low atomic weight, highly electropositive element, and lead, a very dense metal of low reactivity and high atomic weight. This provides materials with applications as diverse as high energy batteries (Yokoyama et al., 1993) and nuclear fusion.

In nuclear fusion applications, the lead-rich eutectic alloy, 83 a/o Pb-17 a/o Li (99.3 w/o Pb - 0.7 w/o Li), offers a unique combination of breeding and neutron multiplying capabilities with a relatively low liquidus temperature of 235°C (Hansen and Anderko, 1958; but see also Hubberstey et al., 1992). For over a decade this alloy has been proposed for use as a liquid breeder/blanket material in various nuclear fusion applications (Sze et al., 1980); more recently, this material has been chosen as a candidate liquid metal blanket material in the European Union's development program for a DEMO reactor (Malang et al., 1994). In one concept being studied in this program, the molten alloy would be cooled by pressurized water.

Because water is being considered as a coolant, it is important to understand the interactions that might occur if the molten alloy inadvertently contacts water in an accident situation (Corradini and Jeppson, 1991; Kottowski et al., 1991; Piet et al., 1987). A worst-case scenario would be the release of toxic and radioactive species from containment (Jeppson and Serrini, 1989).

Although the behavior of this alloy is fairly predictable when it contacts gaseous oxidizers such as air or steam (Jeppson and Muhlestein, 1985; Coen, 1985; Hubberstey and Sample, 1993), its behavior on contact with liquid water is less well understood. There are three major areas of uncertainty associated with the interactions with liquid water: (a) possible vigorous hydrodynamic behavior (e.g., steam explosions); (b) the generation of hydrogen by the chemical reaction between water and the lithium component of the alloy; and (c) the concurrent formation of significant quantities of lithium hydroxide solutions.

Concern (a) probably would involve mostly thermal interactions that might mechanically threaten structures and containments. Little has been reported about interactions of this sort, although both Finn et al., (1980) and Kuhlbörsch and Reiter (1984) have released a few tens of grams of the molten alloy into water, noting only mild interactions. On the other hand, similar amounts of molten unalloyed lead, when similarly released into water, explodes spontaneously (Flory et al., 1969). It would seem important, then, to learn more about the hydrodynamic molten alloy-water interactions.

Concern (b), the generation of hydrogen, is cause for concern because of the possibilities for overpressurization of containments and, if somehow it were to mix with air, for damaging explosions. The generation of hydrogen by the interaction of the lithium component of the molten alloy with water has been studied in a gentle, flooding mode by Herzog (1987), Herzog and Corradini (1989) and Biney (1995). When 60°C water interacted with 600°C melt, there was considerable hydrogen generation, corresponding to the reaction of about 40% (but in several experiments as much as 70%) of the lithium present in the alloy. Other experiments were performed by Kranert and Kottowski (1991) on a small scale in which pressurized water subcooled by 43°C was released onto the eutectic alloy at 500°C in a vertical impact system. Although hydrogen production was not measured, its presence in significant quantities was apparent by the strong damping of the multiple pressure spikes normally observed when molten lead was impacted under identical conditions. (Analogous water column impact studies with molten lead have been reported by Vukovic (1994), Hillary et al., (1973) and Darby et al., (1973).) Large scale water injection ("BLAST") experiments were also performed with the alloy at roughly similar temperatures and pressures (Savatteri and Gemelli, 1991); hydrogen was also generated in these experiments and appeared to blanket the melt in such a manner that steam explosions were suppressed.

Concern (c), the possible formation of large amounts of concentrated caustic lithium hydroxide solutions from the lithium-water reaction, is primarily an environmental and personnel safety matter that, to our knowledge, has not been addressed in the readily available literature associated with this alloy. However, the ultimate compositions of these solutions ought to be directly related to both the amount of hydrogen generated and the amount of lithium removed from the alloy during the interactions with water. Therefore, study of the aqueous chemistry should provide important ancillary information about the nature of these interactions. (Related to the aqueous chemistry of these interactions are the corrosive effects of the alloy-water interaction on stainless steel reported by Agostini and Benamati (1991)).

The objective of our work has been to explore further both the hydrodynamic and the chemical aspects of well characterized and reproducible forced contact of about 0.1 kg of both molten Pb and 83 a/o Pb-17a/o Li alloy at 600°C with liquid water at either 25°C or 60°C. The water impact was generated with a vertical shock tube apparatus. Dynamic and static pressure and temperature data were recorded during the interactions. Gaseous products were collected several minutes after the interactions and measured with a quadrupole mass spectrometer-based partial pressure analyzer. The aqueous solutions produced during the interactions were separated from the debris after the system cooled and were analyzed by titration acid-base. The debris was retained for further study.

The phenomena observed can be reasonably interpreted on the basis of strong hydrodynamic fragmentation of the melt during the melt-water contact, followed by chemical reactions of the melt and subsequent solids with steam and liquid water.

EXPERIMENTAL

Water Column Apparatus

Our water contact apparatus is derived from the original concepts of Wright and coworkers (1965, 1966) as extended by others (Hillary et al., 1973, Darby et al., 1973, Patel and Theofanous, 1981; Yuen et al., 1994). In this concept, a column of water is driven forcefully downward when the diaphragm on which it rests is ruptured, due either to overpressurization or to the activation of a mechanical cutter. (In closely related experiments, a high speed valve was substituted for the diaphragm (Kranert and Kottowski, 1991).) After a short travel downward, the water column impacts a hot molten material, either held in a crucible, or levitated as an electromagnetically heated drop. The impact of the water on the hot molten material, then, may initiate a physical melt-water interaction that sometimes has an additional chemical component. Various diagnostics have been used to determine the nature of the interactions including high speed photography, flash X-ray imaging, dynamic pressure measurements and gas analyses. Examination of the debris recovered afterward also provides important information about the nature of the interactions.

In this work, we used both dynamic pressure recording and gas analyses; we also included some rapid temperature measurements. Moreover, because the Li-containing alloy produces LiOH in amounts proportional to the extent of the melt-water chemical interaction, we were able to add acid-base titration of OH^- to our diagnostic precedures. Debris was also examined to a limited extent.

The original mechanical design of the UW apparatus, documented by Raz (1991), is shown in Figure 1. An improved design due to Farahani (1995) was used in this work; it is shown schematically in Figure 2. Photographs of the apparatus as used here are shown in Figures 3a through g.

Initiation of the Water Impact

Before an experiment, the water is contained in a 2.54 cm I.D. stainless steel tube about 2.4 m-tall; the upper end of the tube is connected to a 7.6 cm I.D. x 50 cm-tall expansion chamber closed at the top with a flanged plate. There is about 1 L of air at atmospheric pressure in the head space above the water level in the upper chamber before an experiment is performed (see Figures 3a and 3b).

The column of water initially rests on a Kapton polymer foil diaphragm that closes the bottom of the 2.54 cm-ID stainless steel tube. The foil has a thickness of 0.5 mm, chosen to burst at a nominal overpressure of 1.0 MPa. This foil is held in place in the union shown in Figures 3a and 3c (assembled) and 3f and 3g (dissembled). Below the diaphragm there is a 2.54 cm-diameter 45 cm-tall volume filled with a low pressure of argon; at the lower end of this volume the 2.0 cm-diameter crucible in which the melt is held is attached via a two-piece threaded crucible holder. A schematic diagram of the crucible is shown in Figure 4.

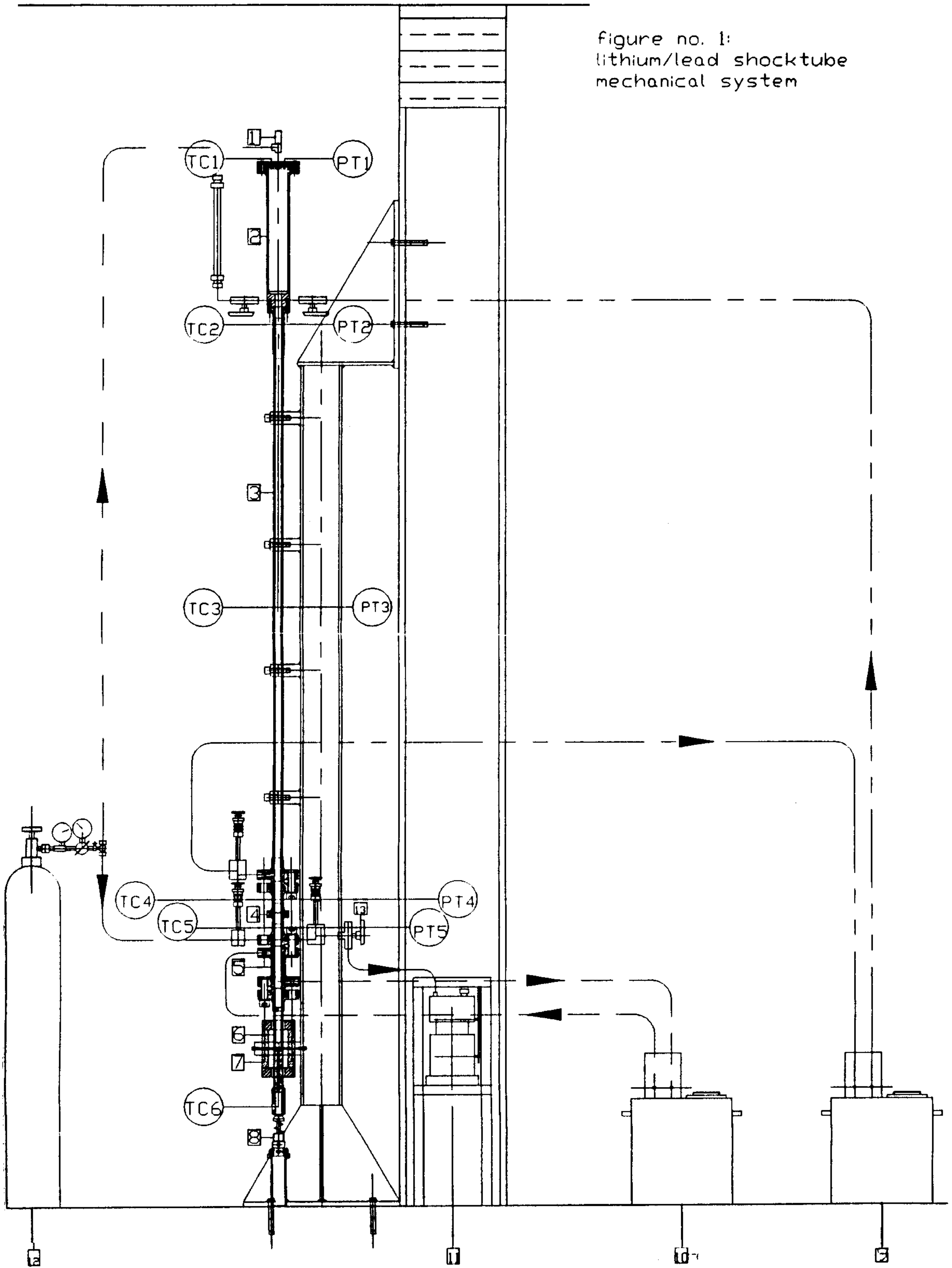

figure no. 1:
lithium/lead shocktube
mechanical system

design: mike raz
date: 11/15/90

Figure 1. Machine drawing of the vertical water column apparatus as originally constructed, reproduced from Raz (1991). Note that numbering of pressure transducers and thermocouples differs from that used in later work (Farahani, 1995; Vukovic, 1995) and in this work.

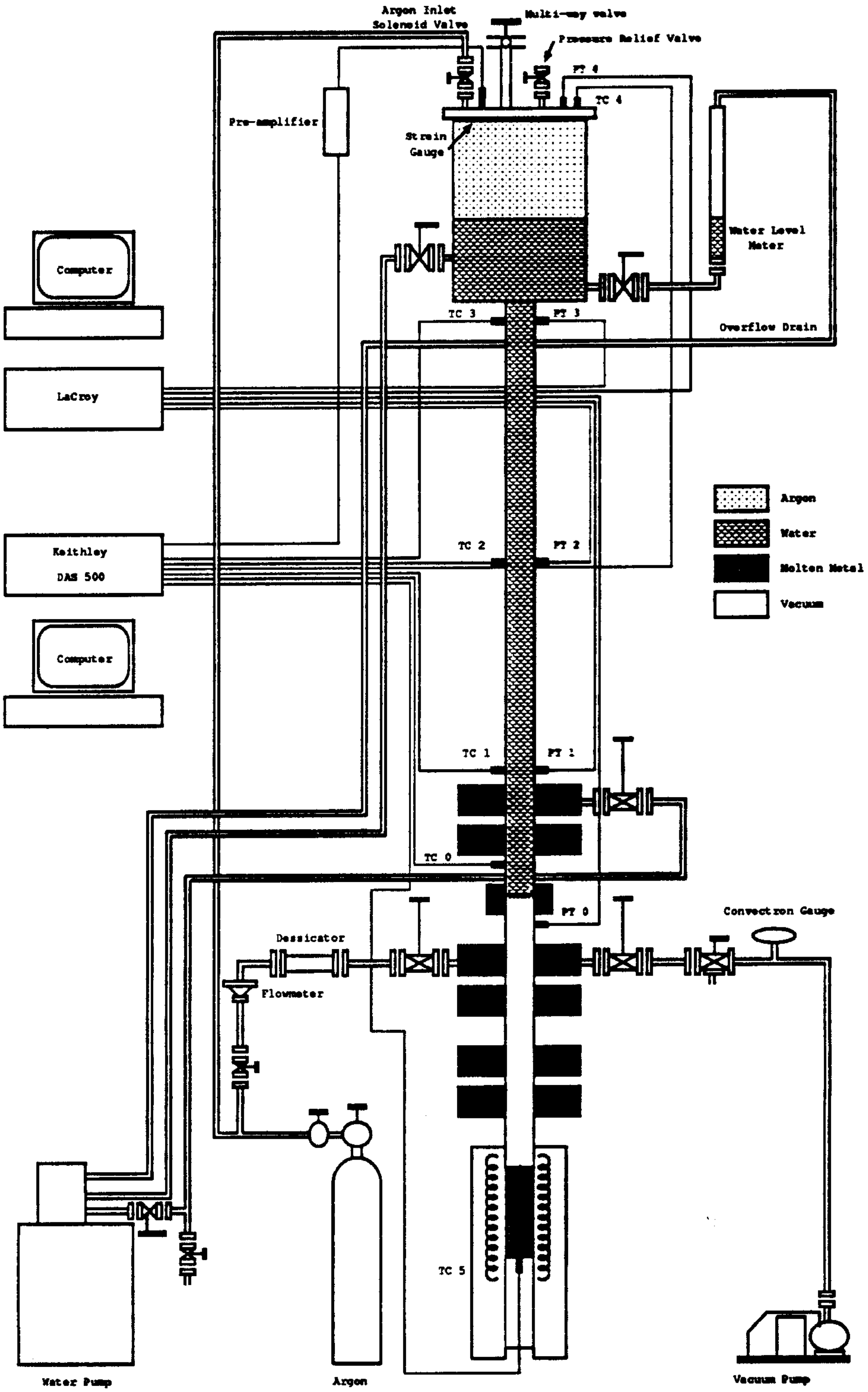

Figure 2. Schematic drawing of the vertical water column apparatus in its current format, reproduced from Farahani (1995). A major improvement over the original apparatus shown in Figure 1 is the provision for gas sampling at the multi-position valve at the top of the apparatus.

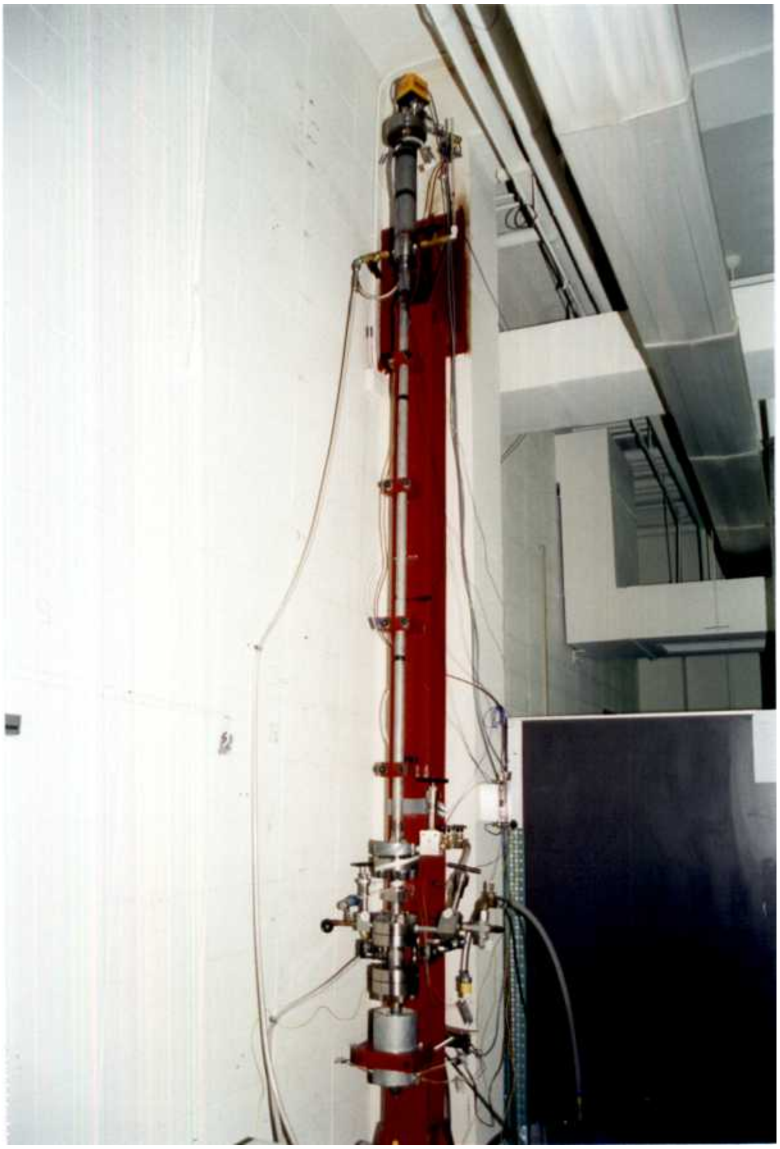

Figure 3. Photographs of the vertical water column apparatus.
Figure 3a. Overall view of the apparatus. (B-59-3)

Figure 3b. View of the upper portion of the apparatus. At the top is the multi-position electrically controlled valve. Just below it are the two gas sampling bottles. The expansion chamber and the upper end of the column are at the center and the water level sight glass is at the right. (B-56-8)

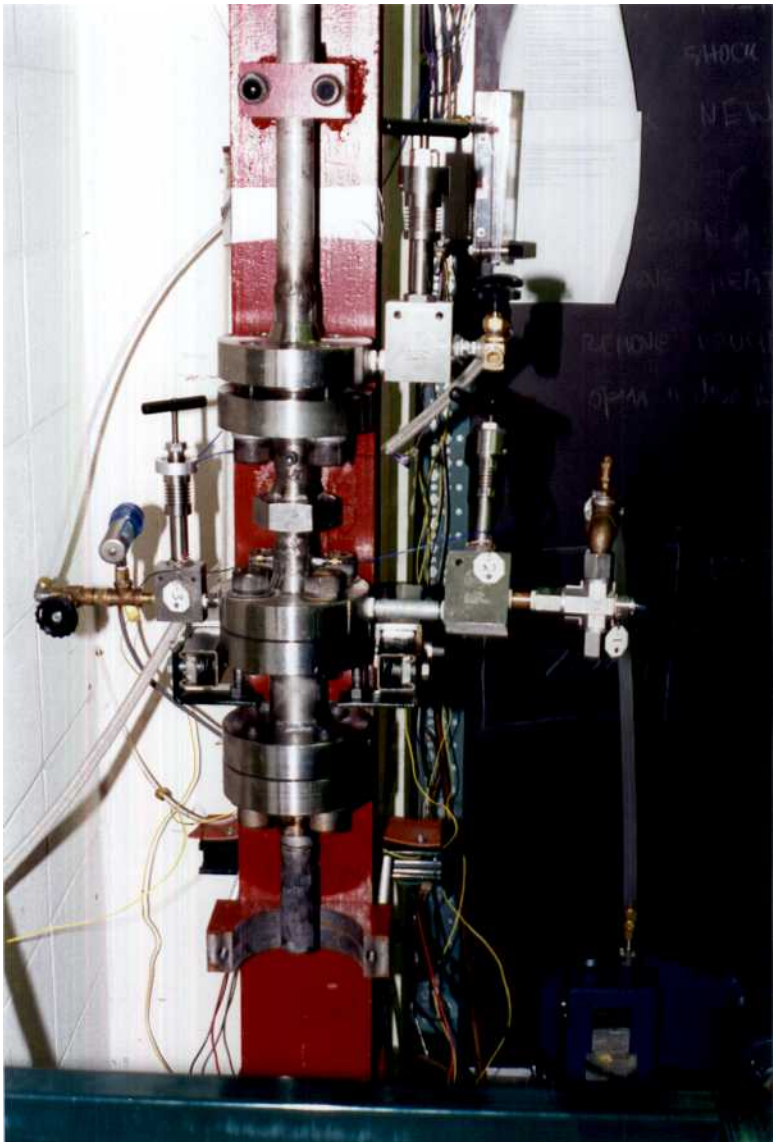

Figure 3c. View of the lower portion of the apparatus The union that holds the diaphragm is at the center of the photograph. Just below it, the pressure transducer, PT0, and thermocouple, TC0, can be seen. Above the union is the coolant entry valve. Below it are two valves that control vacuum (right) and gas inlet (left). A Pirani-type pressure gauge is visible at the far left. The upper half of the crucible holder is shown at the bottom of the apparatus. (B-56-1)

Figure 3d. (left) Crucible holder at the bottom of the apparatus. Crucible is held in the upper half of the holder (cf. Figure 3c). A Type K thermocouple, TC5, is held against the bottom of the crucible by the lower half of the holder. (B-56-2)

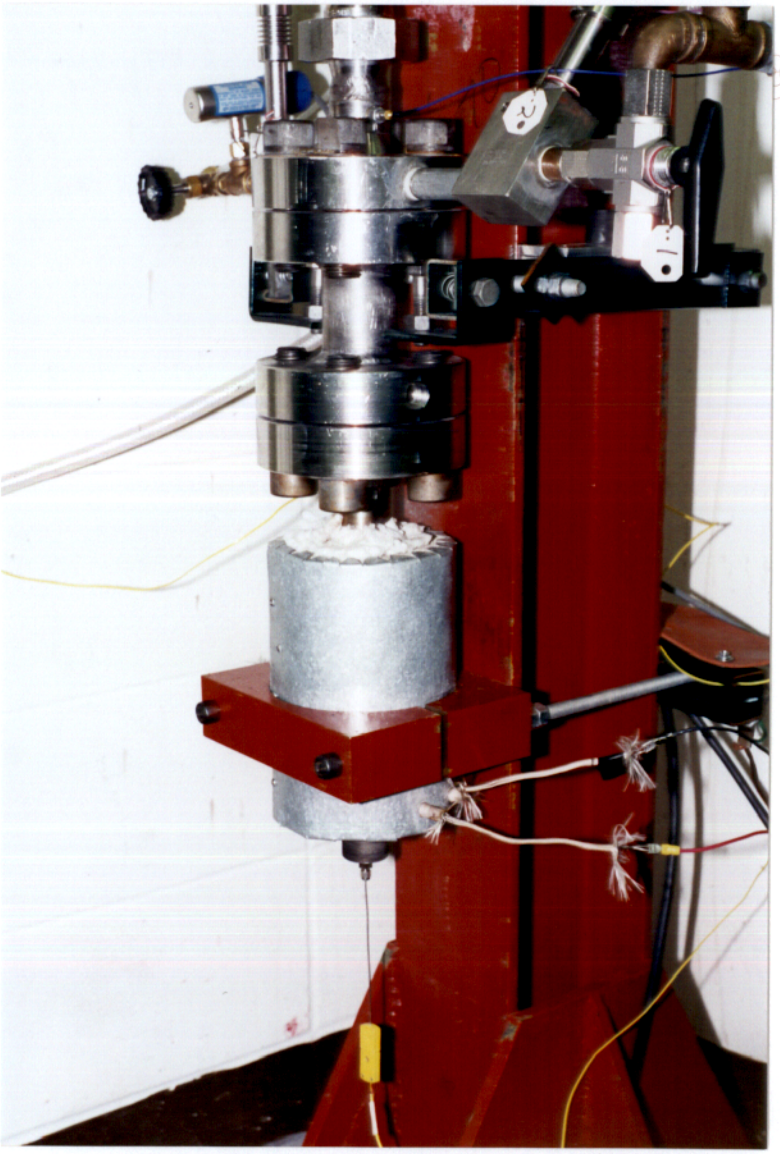

Figure 3e. (right) Crucible holder at the bottom of the apparatus shown in Figure 3d with the two half-cylindrical heaters in place. The lower end of TC5 can be seen beneath the crucible holder. (B-56-9)

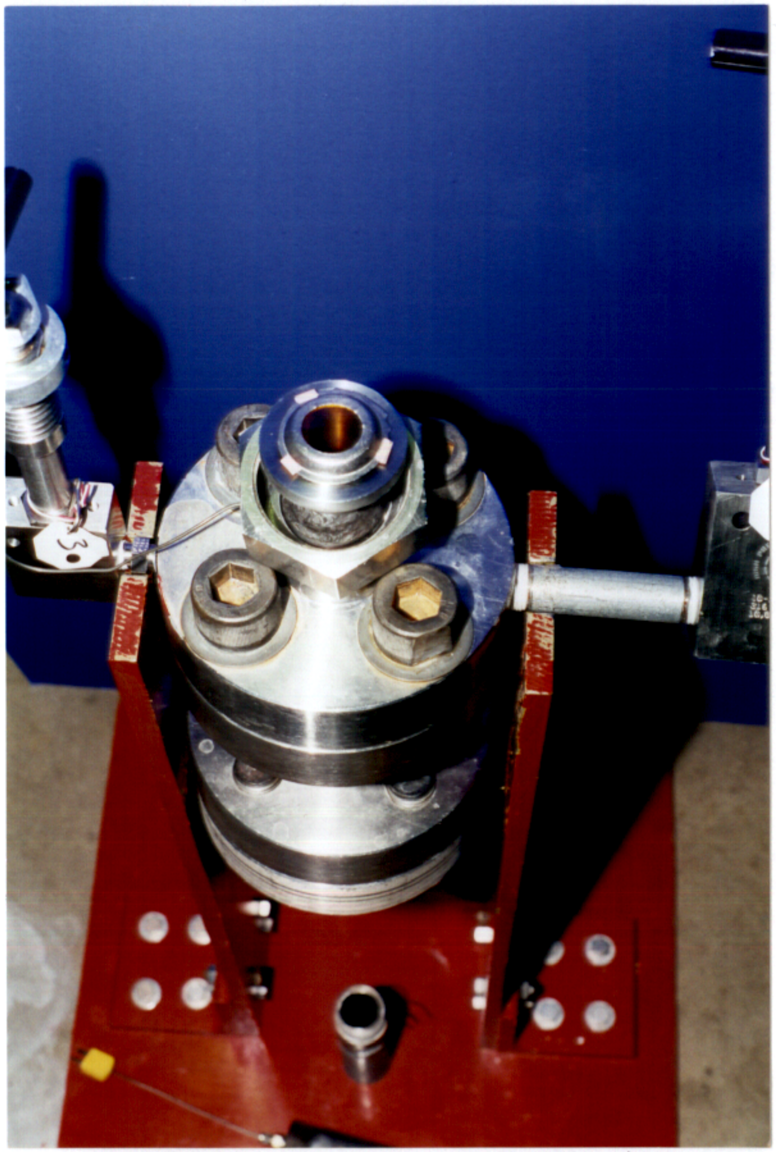

Figure 3f. (left) Dissembled union viewed from above. A new diaphragm is visible, prior to assembly. The vacuum and gas inlet valves can be seen at left and right of the photo. (B-56-12)

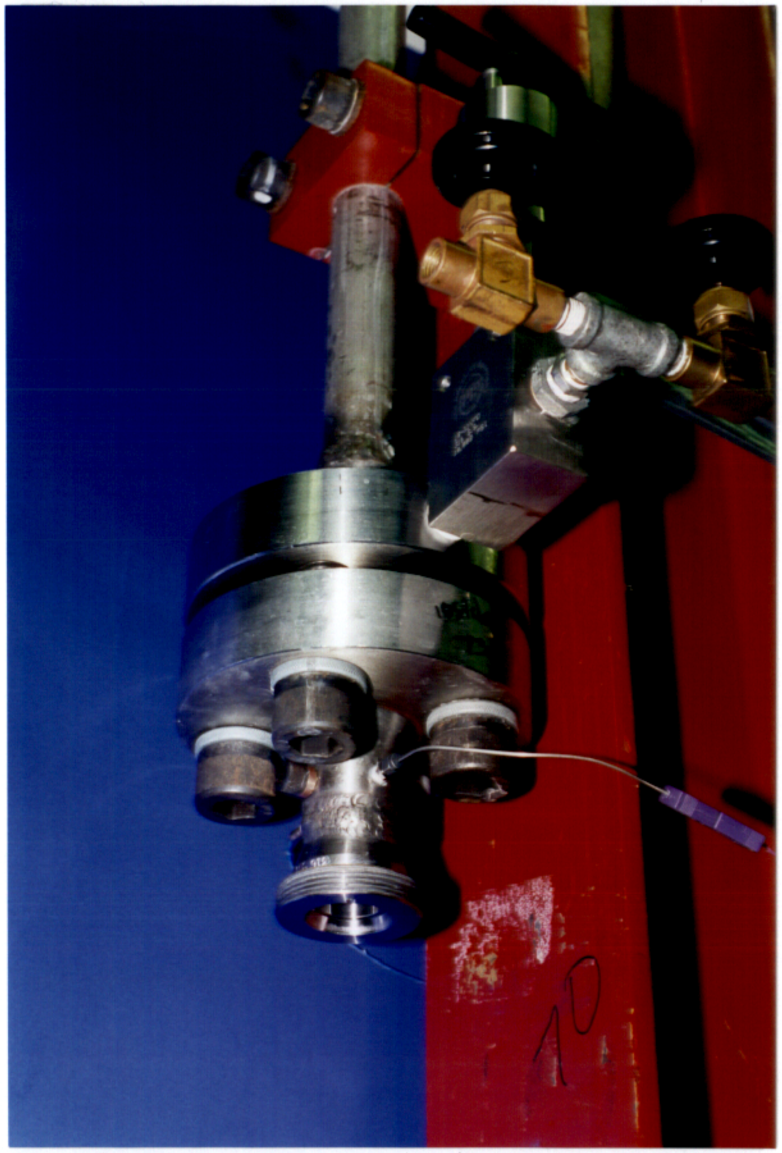

Figure 3g. (right) Dissembled union viewed from below. The thermocouple, TC0 is visible just above the threads of the union. The lower coolant valve can be seen extending from the upper flange. (B-56-14)

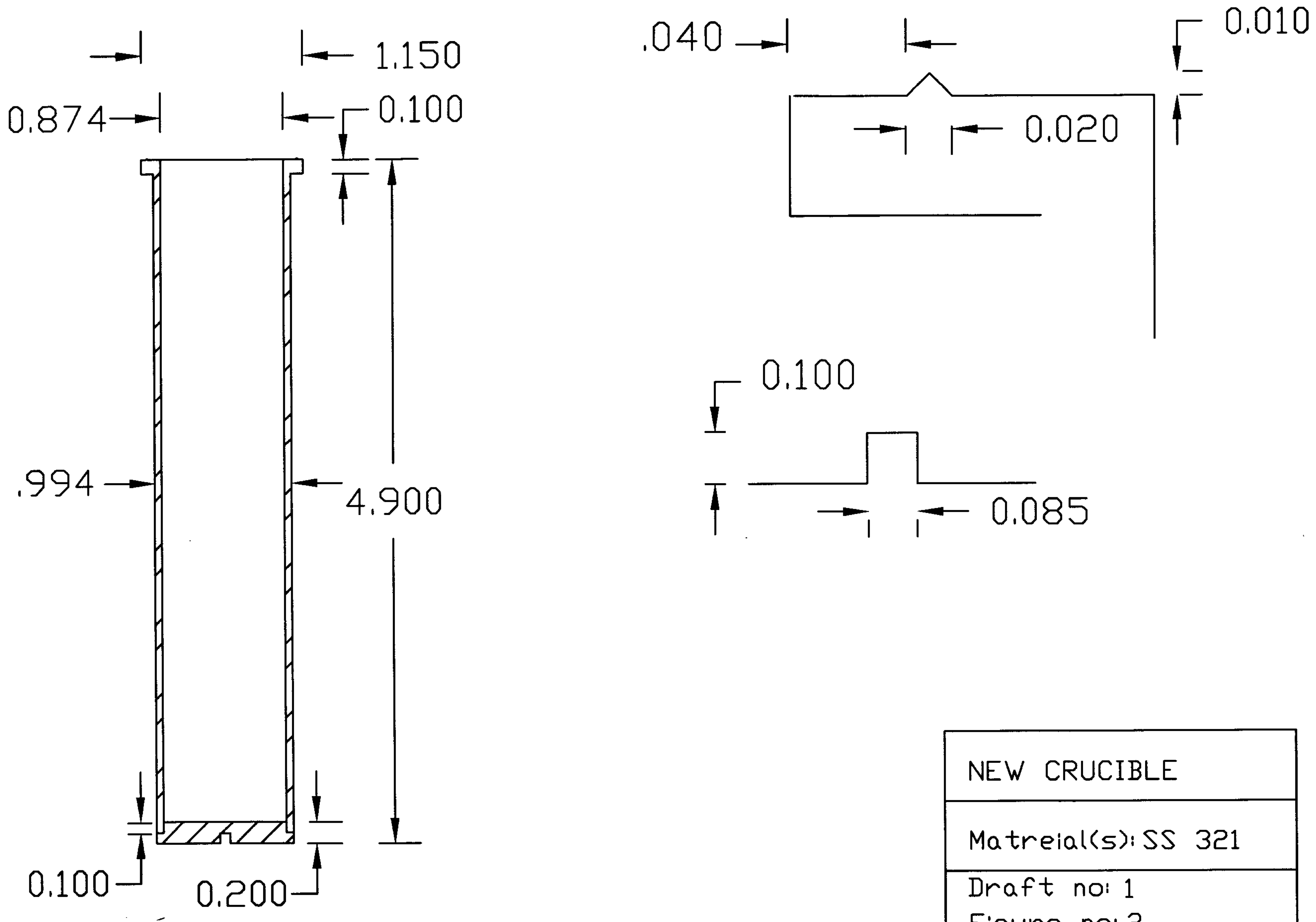

Figure 4. Schematic diagram of the crucible. At the right are details of (above) a raised edge on the lip of the crucible used for sealing against a metal gasket, and (below) the depression in the bottom of the crucible that accepts the Type K thermocouple TC5.

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Design: ALI FARAHANI

When both melt and water have been brought to the desired temperatures, the head space in the expansion chamber is pressurized with argon until the diaphragm bursts, causing the water to be driven downward into contact with the melt.

Transducers

Our water column apparatus has five high speed (2 μs) quartz transducers positioned as indicated in Figures 1 through 3. These are designated: PT0, located just below the burst disc; PT1, located just above the diaphragm; PT2 and PT3, located nominally 1 and 2 meters, respectively, above the diaphragm; and PT4, located in the gas-filled head space above the water in the upper chamber. Each transducer is connected via coaxial cable to one of the five charge amplifiers, each of which, in turn, is connected to one channel of a dual channel high speed digital data acquisition system (the "LeCroy system"). The relationships between column, transducers and the data acquisition system are shown schematically in Figure 2. Detailed descriptions of the apparatus and its electrical and an electronic components are given by Vukovic (1994) and also in Appendix A.

A sixth transducer, a slower response-time strain gauge, designated PT5, is also located in the expansion chamber. It measures the gas pressure as a function of time as the upper chamber is pressurized prior to and after the bursting of the diaphragm. The output of the strain gauge is recorded via a preamplifier by a second slower data acquisition and experiment control system (the "Keithley system"), as indicated in Figure 2. Details are to be found in Appendix A and Vukovic (1994).

Thermocouples

Five thermocouples are positioned as shown in Figures 1 and 2. They are designated TC0, TC1, TC2, TC3, and TC4, and are located at the same levels as the corresponding transducers PT0, PT1, PT2, PT3, and PT4. These thermocouples are Type E, and their outputs are recorded with the data acquisition and experiment control unit (the "Keithley system").

A sixth thermocouple, designated TC5, is Type K, and is inserted vertically upward through the lower crucible holder until it rests in a small depression in the bottom of the crucible (see Figure 4); this thermocouple monitors the temperature of the melt in the crucible. Its output is also recorded with the Keithley data acquisition and experiment control unit, as indicated in Figure 2. Further details about the thermocouples and their recording are presented in Appendix A and by Vukovic (1994).

Initiation of Data Acquisition

The output generated by transducer PT0 provides a trigger signal for both the Keithley data acquisition and experiment control unit and the LeCroy high speed data acquisition system as indicated in Figure 2, Appendix A and in Vukovic (1994). Note that a separate channel in the LeCroy high speed data acquisition system is devoted to processing and recording triggering information.

Control of the Experiment

The control of the apparatus is indicated schematically in Figure 2 and in Appendix A and Vukovic (1994). The functions controlled include:

- The two crucible heaters, each of which is in half-cylinder form to fit around the crucible holder, each powered with its own variable transformer.
- The solenoid valve that admits the pressurizing argon to the expansion chamber when the temperatures of both melt and water are deemed appropriate to initiate the interaction as determined by the thermocouple readings displayed by the Keithley experiment control unit.
- A multiple position valve that directs gas flows in the expansion chamber. This valve is electrically operated with a manual switch. The valve is normally closed when the argon is admitted via another valve into the expansion chamber to pressurize the column. The multiple position valve is next moved to a second and then a third position after the interaction to admit gas samples into the previously evacuated sample bottles A and B for gas analyses. The valve is finally moved to a fourth position to exhaust the pressurized gases outside the apparatus.

The overall sequencing of the experiments is provided by two personal computers as described by Farahani (1995), Vukovic (1994) and in Appendix A.

Water Column Height and Temperatures

Both the height and the temperature of the column of water are controlled by a temperature regulated circulating bath (called the "Haake" system). This bath contains distilled water that is pumped upward through the vertical column and exits to return to the bath at the lower end of the expansion chamber. (Further details about this circulation system are given in Appendix A and Vukovic (1994).) A sight glass is also attached to the lower end of the expansion chamber adjacent to the circulating water outlet, as indicated in Figure 2; the sight glass is visible in Figures 3a and 3b. The temperature of the water bath is adjusted to provide circulating water slightly hotter than that desired for the water column; by proper valving, just prior to an experiment, the water level is adjusted to the final height desired, as marked on the sight glass.

In our experiments with heated water, an auxiliary 300 W electrical heating tape controlled by a variable transformer was wrapped around the flange at the lower end of the water column to achieve a more uniform distribution of water temperatures throughout the height of the system. Water temperatures at various column heights were monitored with thermocouples TC0, TC1, TC2, TC3 and TC4 via the Keithley system.

Atmosphere Control During Melting

The atmosphere within the crucible and lower portion of the column (the impact volume) is controlled by a combination of evacuation and a slow purge of argon via a flow meter by careful throttling of the valves shown in Figures 2 and 3. A Pirani-type gauge monitors the argon pressure and is also used to determine the leak tightness of the crucible holder assembly.

Gas Analyses

Gas analyses were performed on the samples collected in two bottles designated A and B using a quadrupole mass spectrometer-based partial pressure analyzer. Figure 5 schematically shows the components of the partial pressure analyzer. This device is designed to measure the

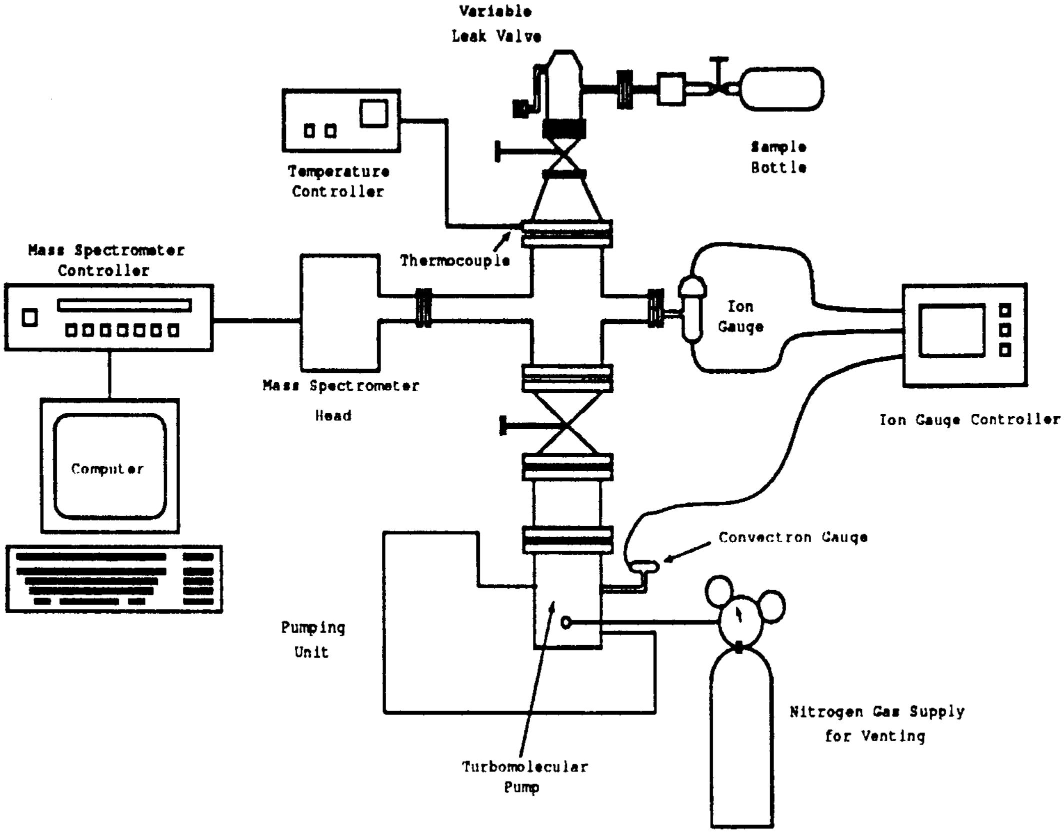

Figure 5. Schematic diagram of the quadrupole mass spectrometer-based gas partial pressure analyzer.

hydrogen concentration in the gas volume above the coolant column in the shock tube. A high vacuum is achieved using a turbo-molecular pump while the partial pressures are measured by a mass spectrometer. This measurement apparatus is comprised of four parts:

- Mass Spectrometer
- Ion Gauge
- Vacuum Pumps
- Valve Assemblies.

Mass Spectrometer

Our partial pressure gauge, supplied by Balzers (Hudson, NH), is a quadrupole mass spectrometer with the mass range from 0 to 64 amu. The sensor, the main part of the device, consists of three assemblies:

- an axially symmetric ion source, where the gas molecules are ionized and directed toward the analyzer;
- quadrupole mass analyzer, where the ions are separated by mass before they enter the detector; and
- a Faraday cup detector, where the ions are detected so the relative mass abundances can be measured.

The lowest detectable partial and total pressures as claimed by the manufacturer are the same at 1×10^{-10} torr (760 torr = 0.1 MPa). However, meaningful pressure readings were not possible at pressures less than 2×10^{-9} torr due to noise interferences.

Ion Gauge

A Granville-Phillips (Boulder, CO) Series 274 ion gauge is installed in the vacuum chamber to measure the total pressure for calibration purposes only. Its operational range is between 1×10^{-9} to 1×10^{-3} torr. It is controlled by a Series 307 vacuum gauge controller from Granville-Phillips that measures pressures from 1×10^{-9} to 1×10^{-3} torr, depending on the gauge(s) being used.

Vacuum Pumps

A Pfeifer - Turbo pumping unit Series 060, which includes the turbo pump, the backing pump and the required connecting and control elements, is used to pump down the vacuum chamber. The turbo pump has a volumetric flow rate of 56 L/s N_2 at a speed of 1500 Hz. The ultimate pressure of the turbo-molecular pump in our laboratory set up with 24 hours of bake-out at 150°C is 8×10^{-9} torr. However, pressures lower than 4×10^{-9} Torr have been reached days after each startup.

Valve Assemblies

Two VAT gate valves isolate the vacuum chamber from the pumping system and the outside air for leak testing. These two valves also act as barriers to any rush of high pressure gas into the vacuum chamber in case of pump or third valve failure. A variable leak valve is used to introduce the fresh gas into the vacuum chamber at the desired rate. This ultrahigh vacuum dosing valve has an adjustable gas flow rate in the range of 10^{-10} to ~600 mbar L/s (1 mbar = 10^{-4} MPa).

Photographs of the mass spectrometer system are shown in Figures 6a through 6c.

Materials

The lead was obtained as a 51 mm-diameter round bar from Taracorp, Inc., of Granite City, IL. This is the same material that was used as one starting material for preparation of the PbLi eutectic alloy as described by Nelson et al., (1995). It was cut from the bar and then more accurately to exact size, weighed, and otherwise handled in the laboratory in air.

The 83 a/o Pb-17 a/o Li alloy was taken from batch JK-121994-01 prepared as described in Appendix C of Nelson et al., (1995). It was cast to form six ingots in the argon-purged glove box shown in Figure 7. A photograph of the ingots is shown in Figure 8. The dimensions of the stainless steel mold were chosen to provide individual slugs of nominal weight of 120 g with diameters of 19.3 mm (to fit the 20 mm inside diameter of the crucible) and heights of 40.4 mm. This sample height was chosen to duplicate that used in the molten lead-water experiments

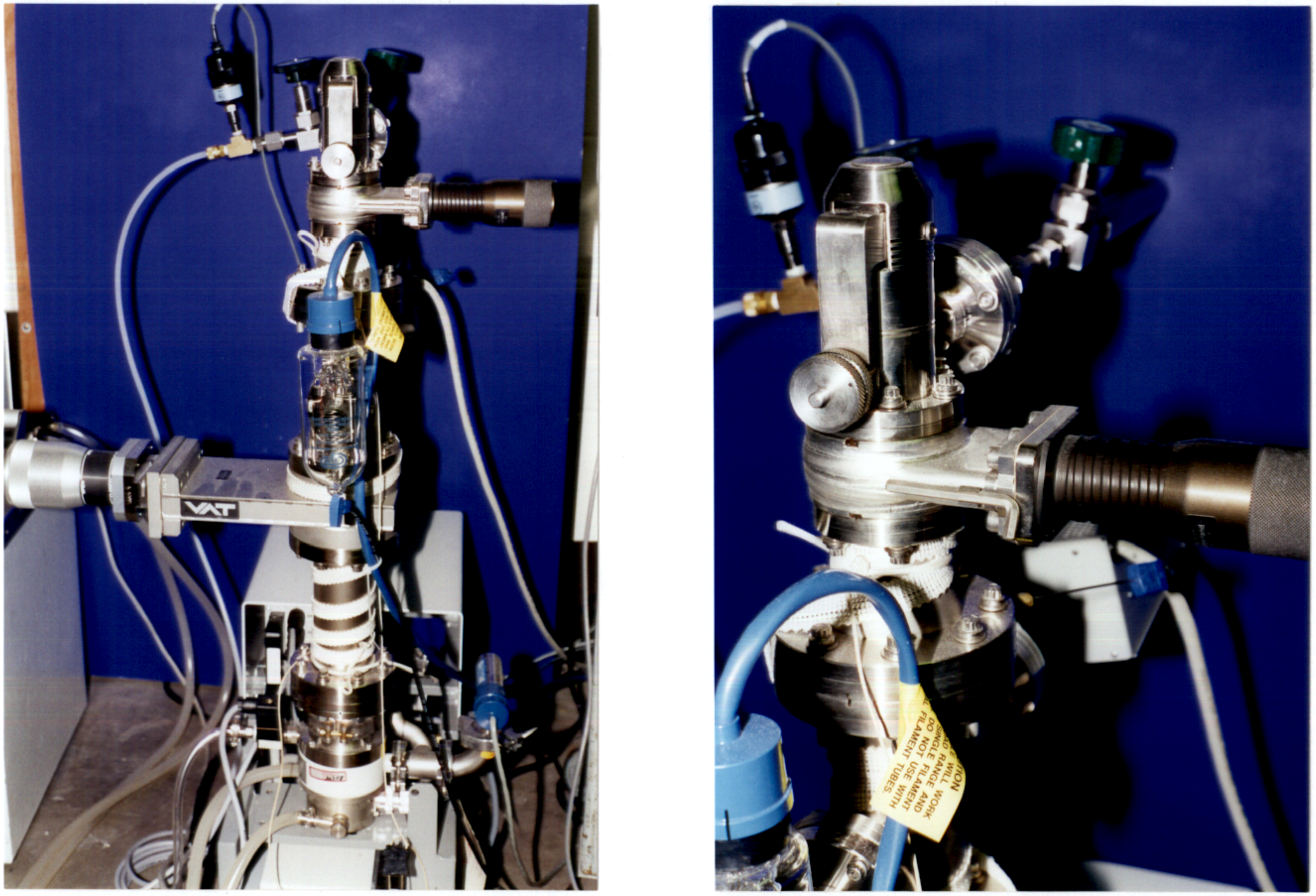

Figure 6. Photographs of the quadrupole mass spectrometer-based gas partial pressure analyzer.

Figure 6a. Overall view of the apparatus. (B-56-15)

Figure 6b. Closer view of the apparatus. (B-56-16)

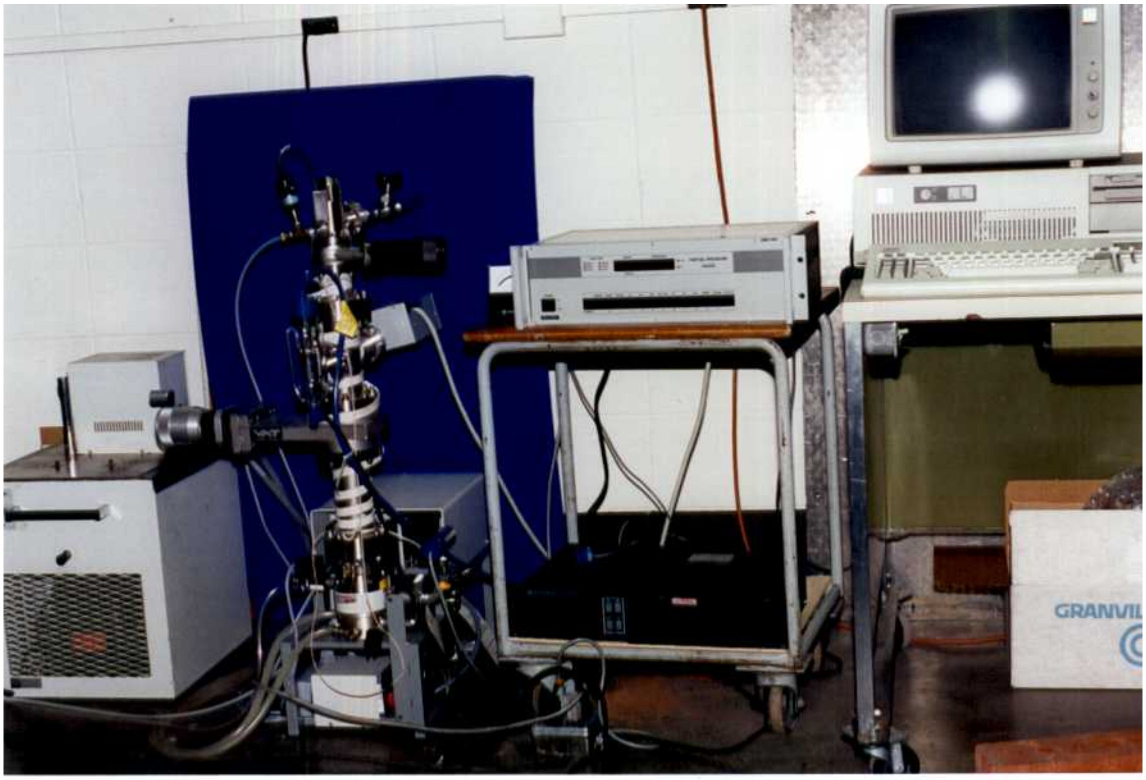

Figure 6c. Broader view of the system. From left: Constant temperature water circulator; gas analysis apparatus; control units; computer. (B-56-17)

Figure 7. Glove box in which 83a/oPb-17a/o Li alloy was cast, weighed and loaded into the crucibles. (B-56-18)

Figure 8. View of six samples of the 17 a/o Li-83a/o Pb alloy shortly after casting and removal from the molds. (B-37-1-10).

performed by Vukovic (1994). After freezing, due to favorable differences in thermal expansions, the slugs of alloy easily fell from their respective molds.

In order to load an alloy sample into the apparatus, one of the slugs was first weighed and placed in a preweighed crucible in the argon-purged glovebox. The crucible was then covered with a paraffin foil as shown in Figure 9. This gas-tight seal over the mouth of the crucible kept the slug of alloy in an argon atmosphere as the crucible was passed from the glove box and moved to the water column apparatus. The paraffin foil was carefully removed from the crucible as it was being installed in the crucible holder, as shown in Figure 10. Argon was being flushed downward from the lower part of the apparatus over the mouth of the crucible as it was being installed.

The argon used to purge the glove box and to flush the crucible during heating of the melts was commercial cylinder gas used without further purification. Its nominal composition was discussed by Nelson et al., (1995). The glove box atmosphere achieved by purging with this gas typically had a dewpoint of -60°C, with 10 ppm of impurities.

The water used in these experiments was distilled water obtained from local commercial sources.

Aqueous Solutions

After each experiment with the alloy, the aqueous solutions that remained in the column were strongly alkaline, about pH = 12, as indicated by wide range pH test paper (Hydrion Brand obtained from the Fisher Scientific Company). Each solution was collected quantitatively with appropriate rinsing with distilled water. The aqueous solutions from each experiment were collected as three segments: (i) that above the water inlet valve (just below transducer PT1, see Figure 2); (ii) that between the water inlet valve and the evacuation/gas inlet valve including the diaphragm holder; and (iii) the liquid that remained in the lowest part of the column including the crucible after the diaphragm holder was dissembled. These three segments were combined with appropriate rinsing with distilled water. Also, the spongy debris recovered from each experiment and its crucible was rinsed respectively with small amounts of distilled water until

Figure 9. Crucible containing sample of the alloy and covered with paraffin foil after removal from the glove box. (B-39-1-1)

Figure 10. Top of crucible being stripped of paraffin foil just before the crucible is installed in the apparatus. Argon is flushing downward over the mouth of the crucible from the lower opening of the apparatus during the installation. (B-39-1-2)

the rinsings were neutral as indicated by the test paper (usually about a dozen rinsings were needed). The individual rinsings were separated from the debris by decanting and filtration with more rinsing and added to the solution collected from the column. Then each overall aqueous solution (that consisted of the column solution combined with the rinsings from the debris and its crucible) was sent to the University of Wisconsin Soil and Plant Analysis Laboratory for analysis of hydroxyl ion (OH^-) concentration by acid-base titration.

To make sure no alkali remained in the column, the lower end of the upper part of the dissembled disc holder (see Figure 3g) was closed with a rubber stopper and the column and upper part of the apparatus were refilled to its usual level with fresh distilled water and drained a second time. The column rinse solution so produced was also sent for titration to determine OH^- concentration, even though it always appeared neutral toward the test paper.

The combined column solution and rinsings of the debris and crucible from one experiment with molten Pb, although not detectably alkaline toward the test paper, was also sent for OH^- analysis as a control.

Debris

The debris that remained in the lower end of the column (above and outside the crucible) after both the lead-water and alloy-water interactions was always easily removed. Also the material from the lead-water interaction was readily separated from both the column and the stainless steel crucible. However, only a portion of the debris could be removed easily from the crucible after the alloy-water interactions. (Similar behavior during Pb-Li eutectic alloy-water interactions was reported by Kranert and Kottowski (1991).) Some debris always adhered inside the lower portion of the crucible in spite of moderate mechanical probing with a bent stainless steel rod. The amounts of debris retained were determined by weighing. The crucibles from the alloy-water experiments were sectioned longitudinally with a bandsaw to permit observation of the adhered debris.

Experimental Procedures

A step-by step description of the procedures used to perform these experiments is presented in Appendix A.

RESULTS

General Observations

The results obtained from our water impact experiments consist of dynamic pressures recorded during the first seconds after each interaction, near the melt and at several heights in the water column above, and the static analyses of the gases, aqueous solutions and debris that were generated by the interactions. Also, ancillary data were collected during an interval that extends from shortly before to 5 seconds after the interaction to define temperatures, gas pressures and triggering information pertinent to each experiment. The experiments were carried out in random order to insure that the results were not affected significantly by systematic changes in the characteristics of the experimental apparatus or procedures.

We performed a total of eight experiments (temperatures and weights are nominal):

- Two dry runs without melt, one with the crucible at 25°C (B-25-1) and the other at 600 °C (B-31-1); water was at 25°C in both experiments.
- Two experiments with 140 g of molten lead at 600°C, one with water at 25°C (B-35-1) and the other with water at 60°C (B-43-1).
- Four experiments with 120 g of molten 83 a/o Pb-17 a/o Li alloy at 600°C, one pair with water at 25°C (B-39-1 and B-50-1) and a second pair with water at 60°C (B-45-1 and B-52-1).

The dynamic pressure-time traces all showed a sharp initial pressurization that accompanied the first impact of the water column, followed by:

- A sequence of regularly spaced, decreasing spikes in the dry runs when no melt was present in the crucible;
- Irregularly spaced spikes, with the second and third higher than the initial pressurization when molten lead was in the crucible; and
- Small, strongly damped and widened pressurizations when the molten eutectic alloy was in the crucible.

Our analyses indicated that 30-40 mmoles of gaseous hydrogen and 75 to 90 mmoles of lithium hydroxide were produced from each molten alloy-water interaction; neither could be detected in the dry runs or lead-water experiments. The measured amounts of hydrogen correspond to the removal of about half the lithium originally present in the melt, while the amounts of lithium hydroxide indicate the removal of nearly three fourths of the lithium.

The temperature records indicate that a significant exothermic chemical reaction between the molten Pb-Li alloy and the water occurs during at least the first 5 seconds after the impact; no exothermicity could be detected during the runs with the empty crucibles or with the molten lead.

The results of our experiments are summarized in Table 1. The pressure profiles along the shock tube and the crucible temperatures for all the experiments listed in Table 1 presented in Figures 11 through 18 and in Appendix B, as described below.

Pressure Profiles

Dynamic pressure measurements were made at five different locations along the shock tube (see Figures 1, 2 and 3 for the locations of the pressure transducers). Due to occasional difficulties with the measurement and data acquisition systems, not all of the traces from the five pressure transducers are available for all the experiments. However, we will only present and discuss the pressure traces as measured successfully by PT0 and PT4. (The transducer PT0 measures the pressure in the vacuum chamber below the rupture disk; the gas pressure in the expansion chamber at the top of the column is measured by PT4.) Although in all experiments we have used the pressure traces recorded successfully from PT1, PT2 and PT3 to understand the reflection and transmission of the shock waves, these traces have not been used in the body of this report in any of the calculations except when noted explicitly. Figures 11 through 18, a through c, show the pressure profiles recorded during experiments with different crucible loadings. (In each figure, a and b show the traces recorded with transducer PT0 dIuring 1000 ms and 30 ms time intervals centered approximately on the initial pressurization; c shows the record from transducer PT4 also over a 1000 ms time interval. A more complete set of pressure transducer records is presented in Appendix B.)

Table 1. Molten Pb and Pb-Li Eutectic Alloy-Water Interactions Initiated by the Impact of a Column of Water in a Shock Tube Geometry

								Mass Spectrometer			Titration of LiOH		
Expt. No.	Melt	Melt Weight (g)	Li Weight (g)	Li (mmole)	T_{melt} (°C)	T_{H_2O} (°C)	Injection Pressure (MPa)	H_2 Generated (mmole)	H_2 Generated (mmole/g)[b]	Lithium Reacted (%)	H_2 Generated (mmole)	H_2 Generated (mmole/g)[b]	Lithium Reacted (%)
B-25-1	None	—	—	—	14.6[a]	27.3	0.998	NA	NA	NA	NA	NA	NA
B-31-1	None	—	—	—	576.7[a]	30.7	0.951	0	0	NA	NA	NA	NA
B-35-1	Pb	140.18	—	—	590.2	29.1	1.02	0	0	NA	NA	NA	NA
B-39-1	Pb-Li	119.57	0.837	120.6	592.4	27.9	1.08	33.7*	0.282*	55.9*	45.3*	0.379*	75.0*
B-50-1	Pb-Li	116.59	0.816	117.6	598.5	27.4	1.05	42.0+	0.360+	71.5+	42.9*	0.368*	73.0*
B-43-1	Pb	146.08	—	—	587.2	61.1	0.917	0	0	NA	0	0	NA
B-45-1	Pb-Li	115.91	0.811	116.8	600.9	61.0	0.579	29.3*	0.253*	50.1*	37.6+	0.324+	64.4+
B-52-1	Pb-Li	122.42	0.857	123.5	594.1	59.6	0.912	34.4*	0.281*	55.8*	45.5*	0.372*	73.6*
							Avg of *'s	32.47 ±2.76	0.272 ±0.016	53.9 ±3.32	44.6 ±1.45	0.373 0.006	73.9 ±1.03
							Avg. of all (Includes "maverick")	34.85 ±5.27	0.294 ±0.046	58.33 ±9.19	42.83 ±3.68	0.361 ±0.025	71.5 ±4.85

[a] Temperature of empty crucible. [b] Based on melt weight. +"Maverick" value

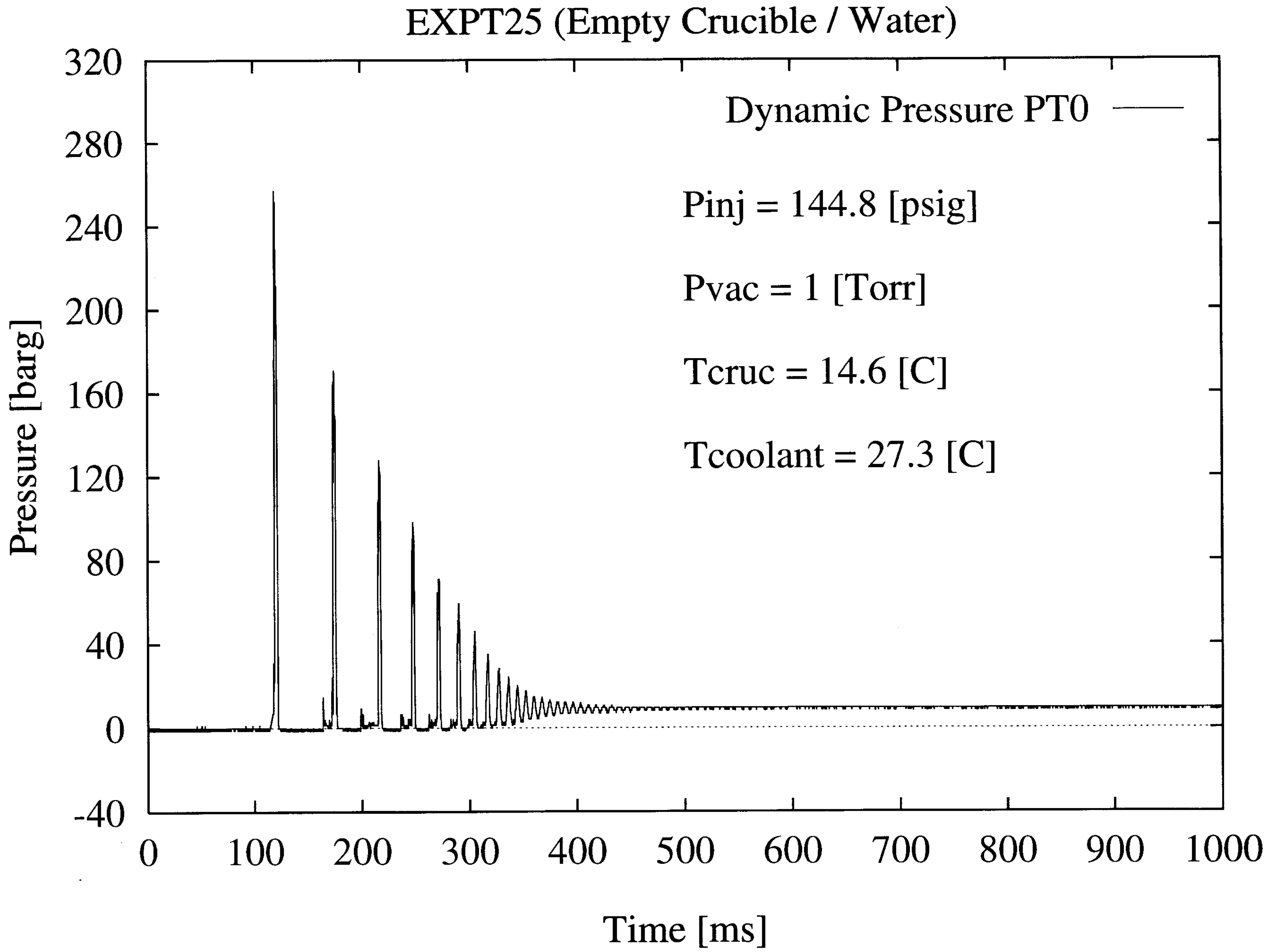

Figure 11a. Instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C. Transducer PT0, 1000 ms trace.

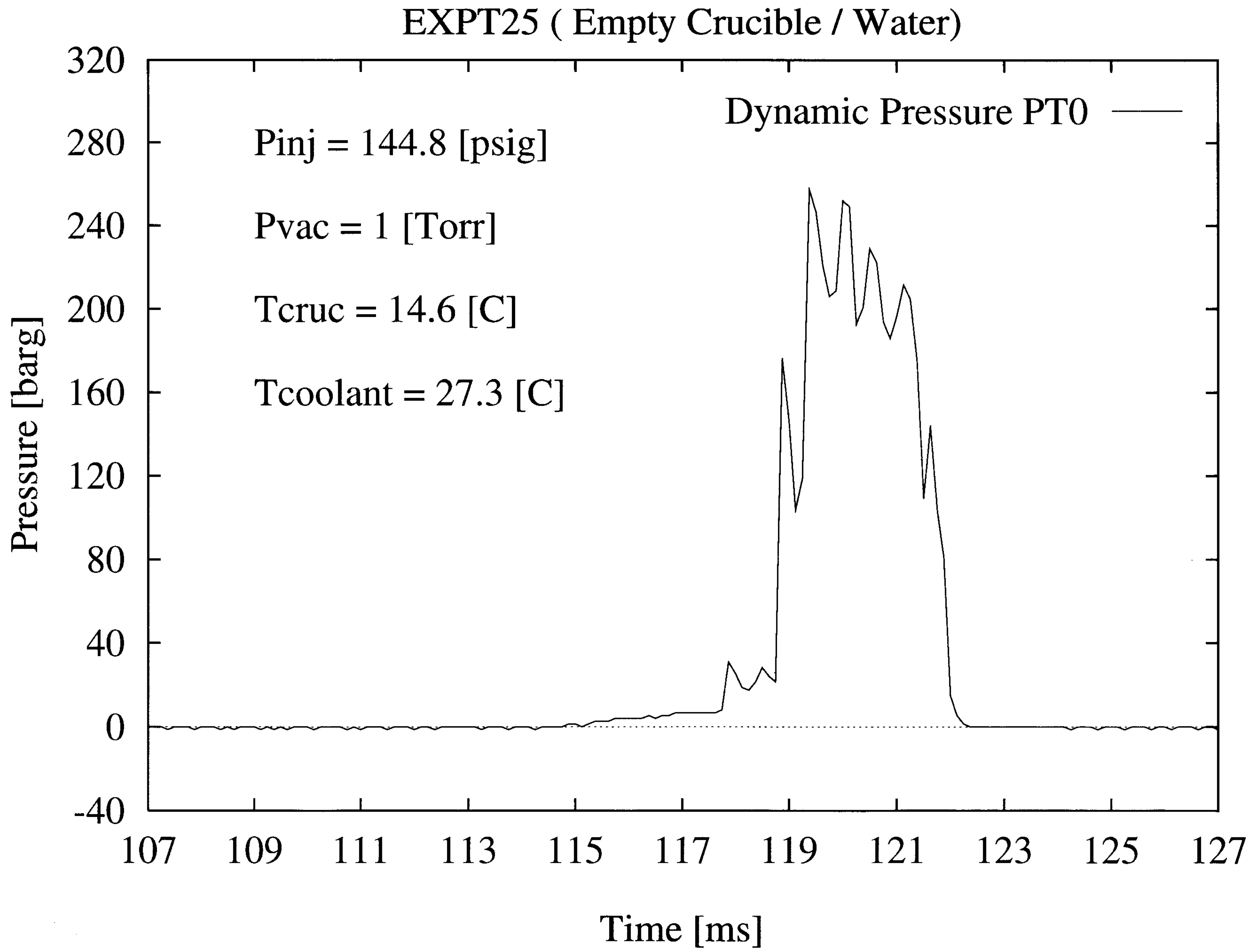

Figure 11b. Instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C. Transducer PT0, expanded time scale.

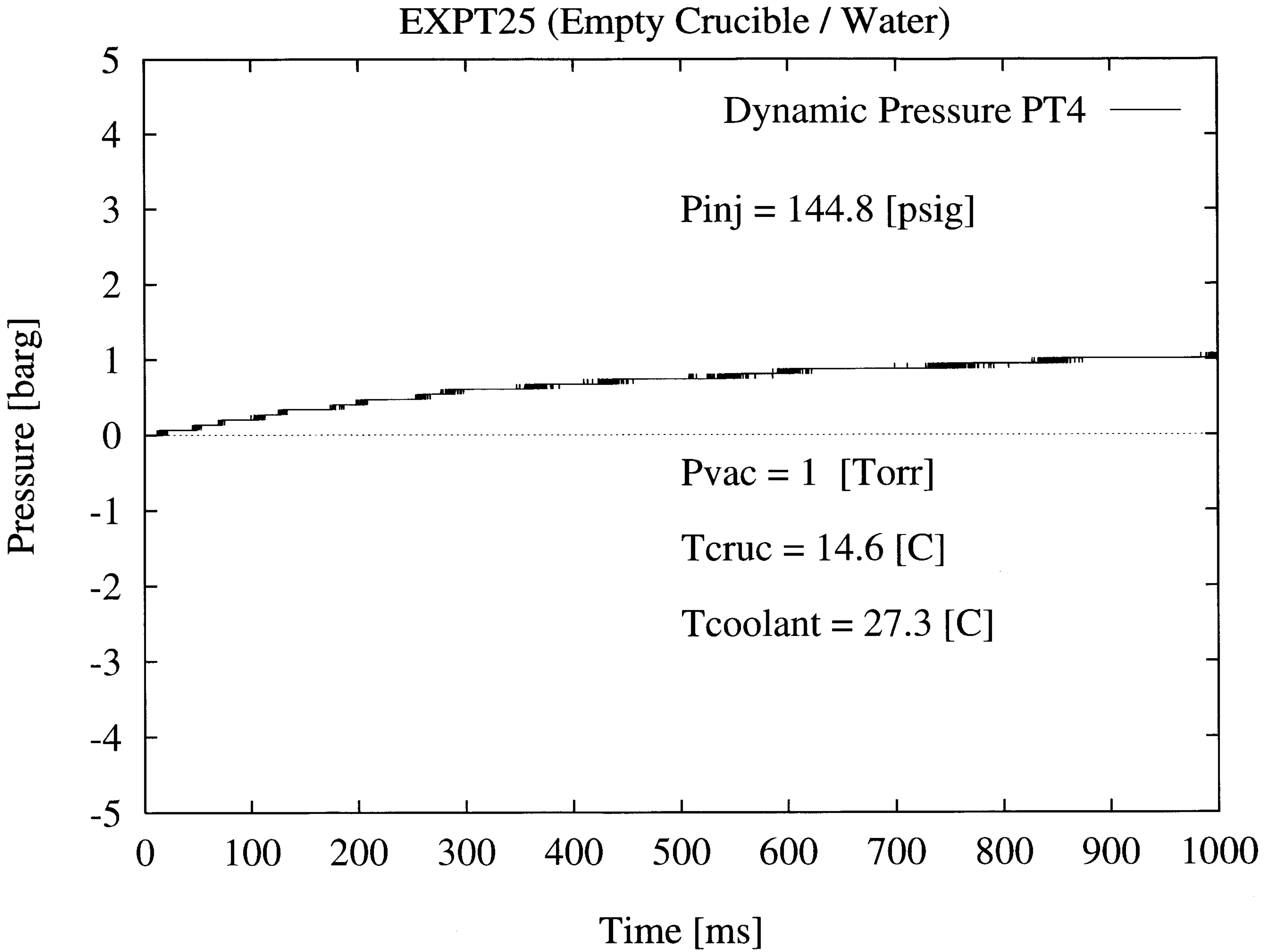

Figure 11c. Instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C. Transducer PT4, 1000 ms trace.

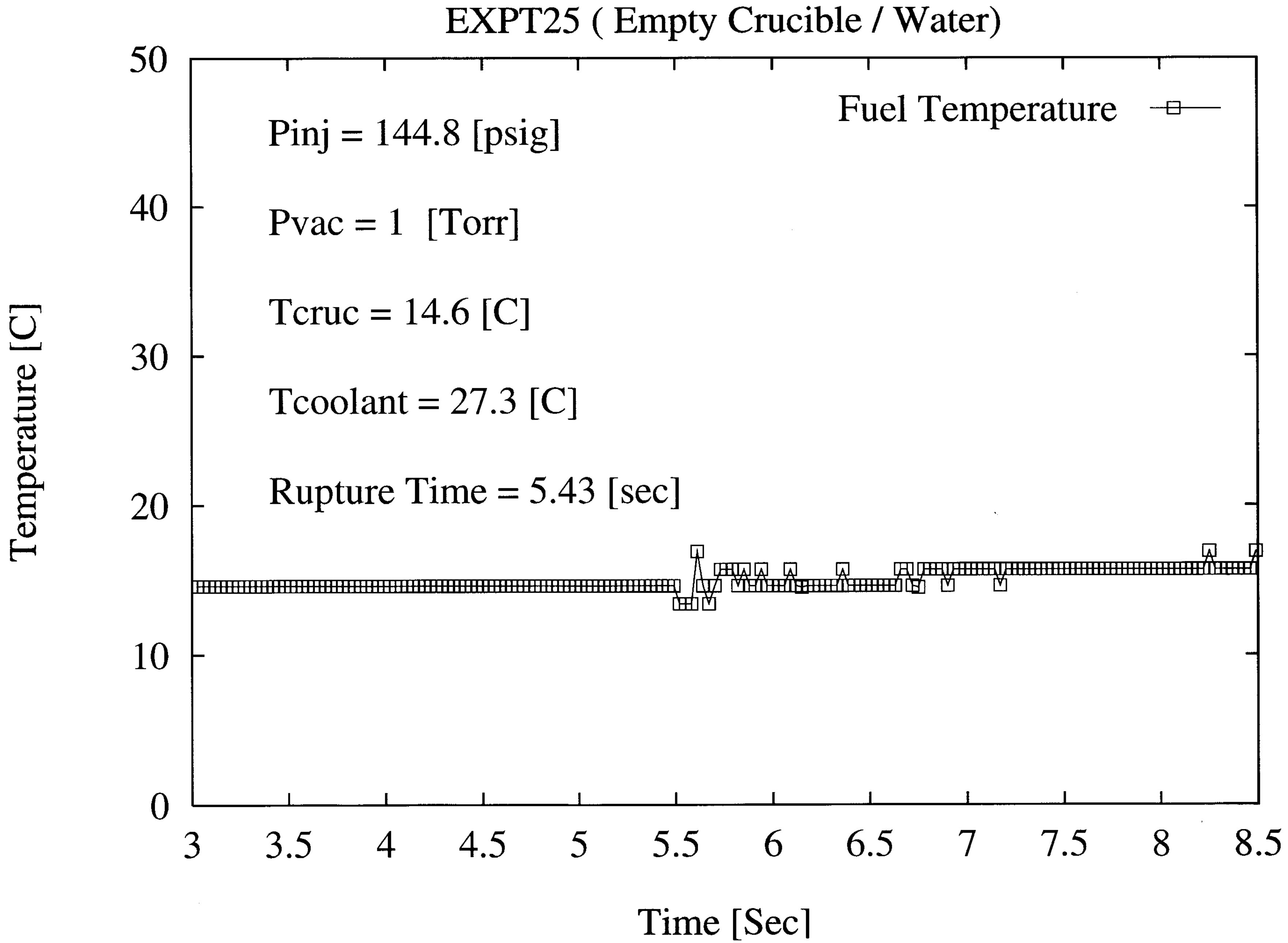

Figure 11d. Instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C. Thermocouple TC5, 5 s trace.

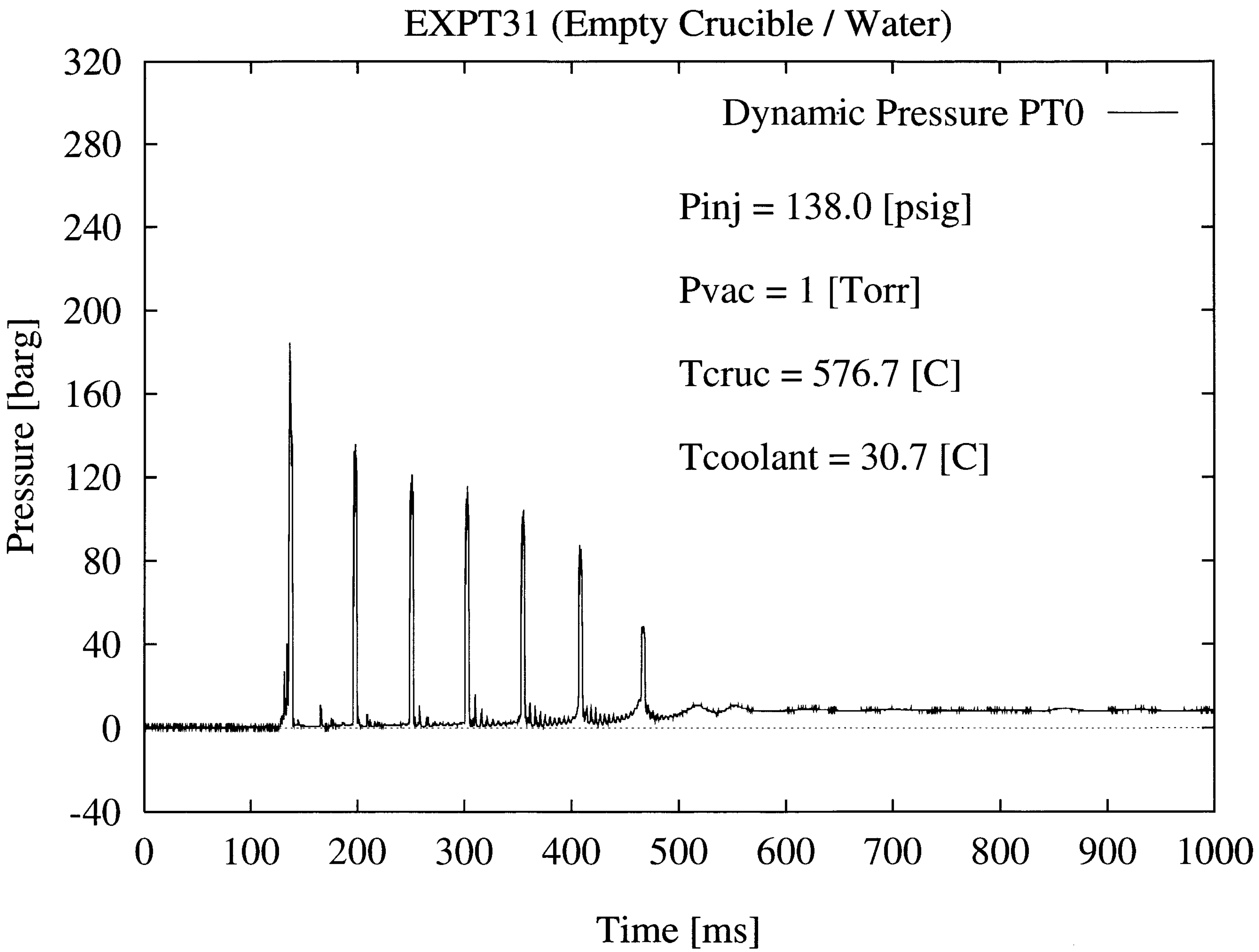

Figure 12a. Instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C. Transducer PT0, 1000 ms trace.

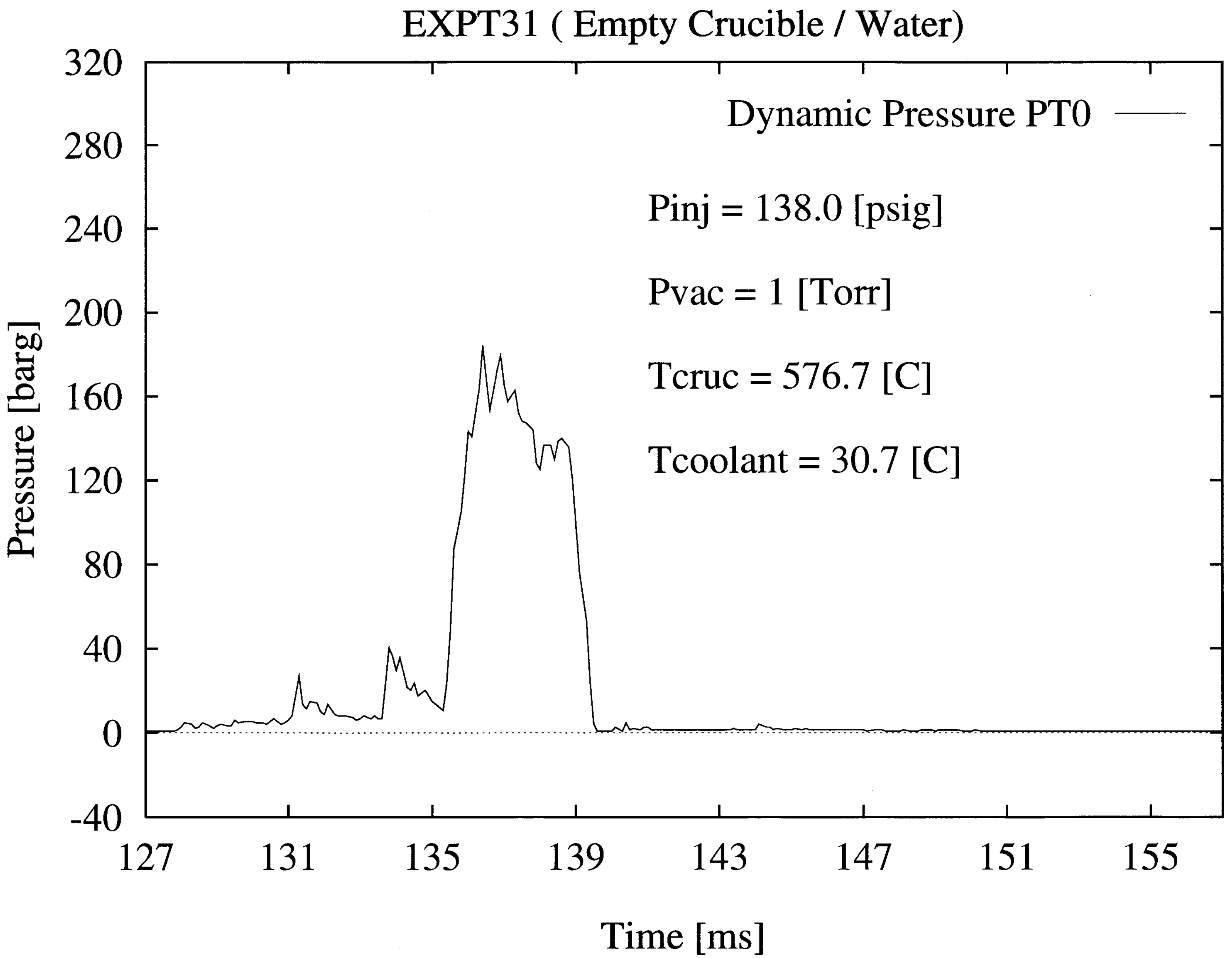

Figure 12b. Instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C. Transducer PT0, expanded time scale.

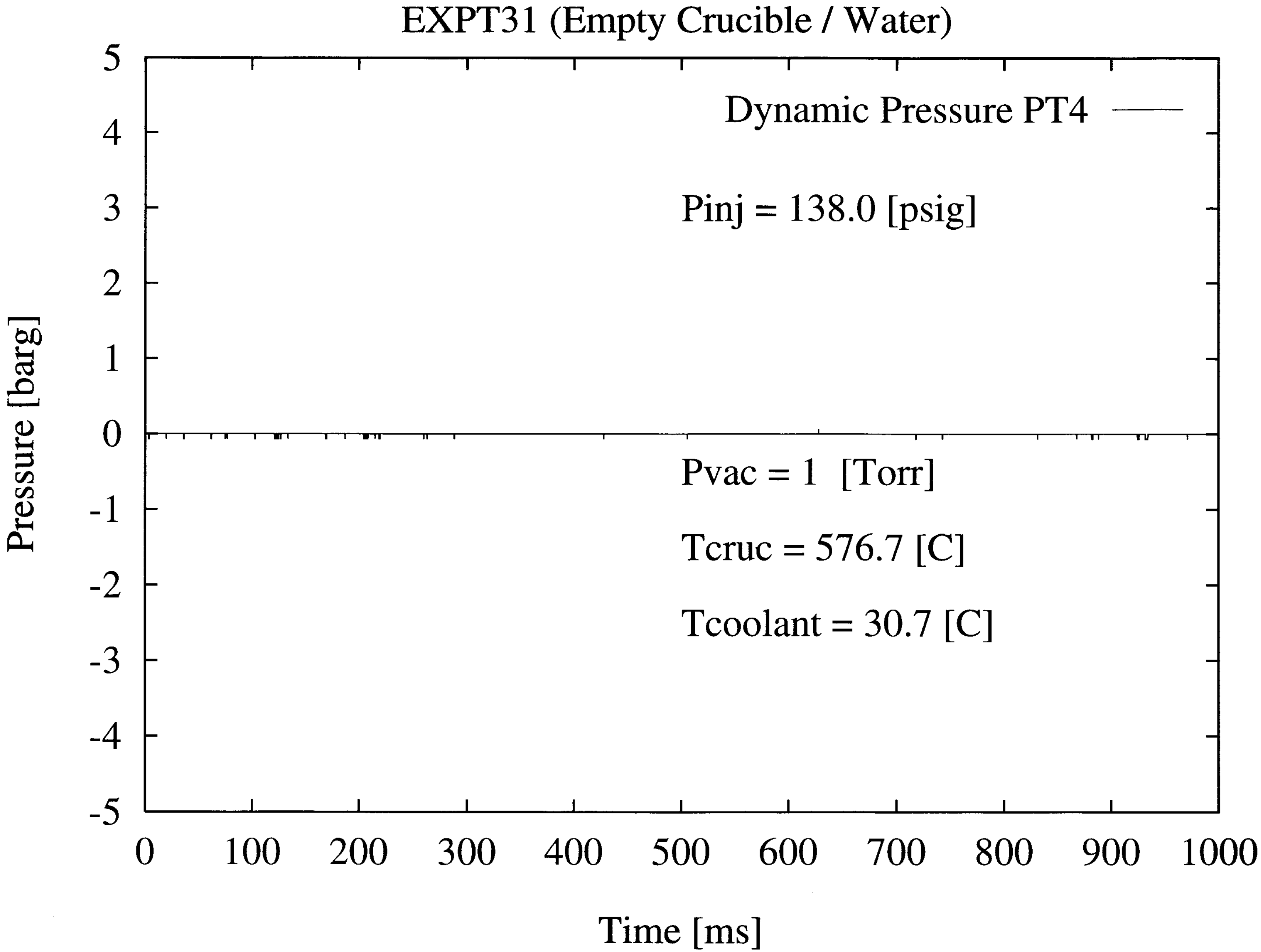

Figure 12c. Instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C. Transducer PT4, 1000 ms trace.

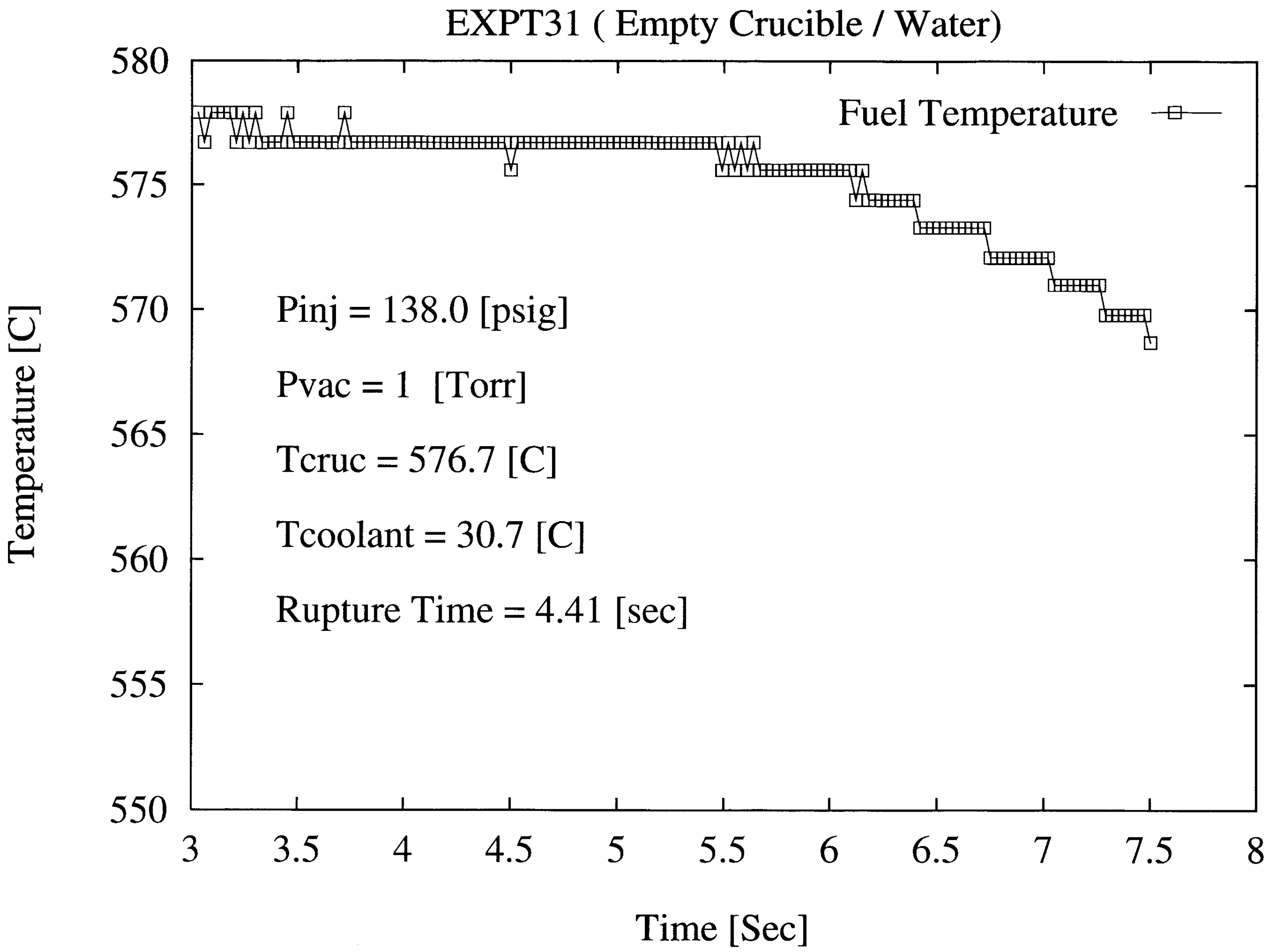

Figure 12d. Instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C. Thermocouple TC5, 5 s trace.

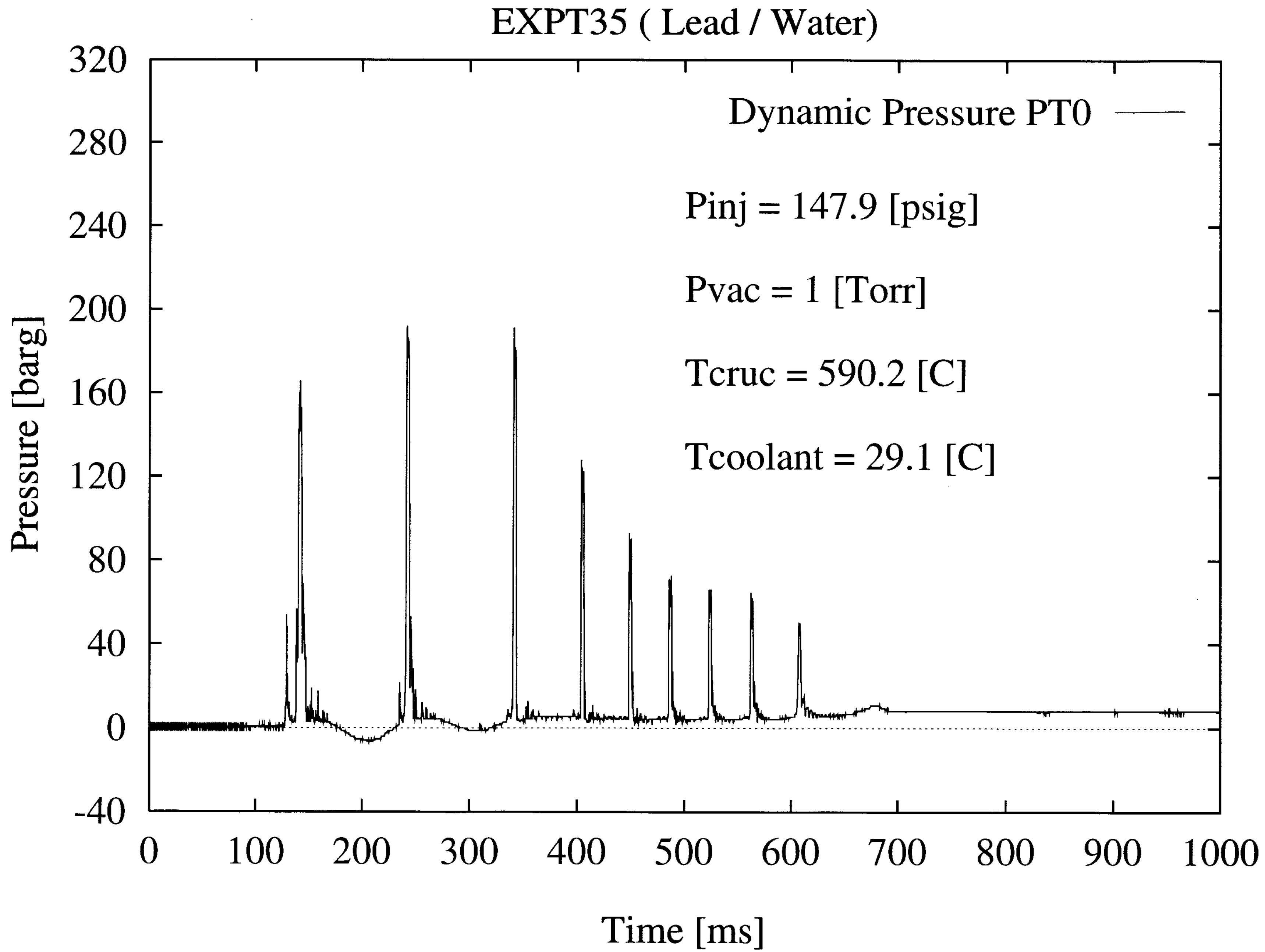

Figure 13a. Instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C. Transducer PT0, 1000 ms trace.

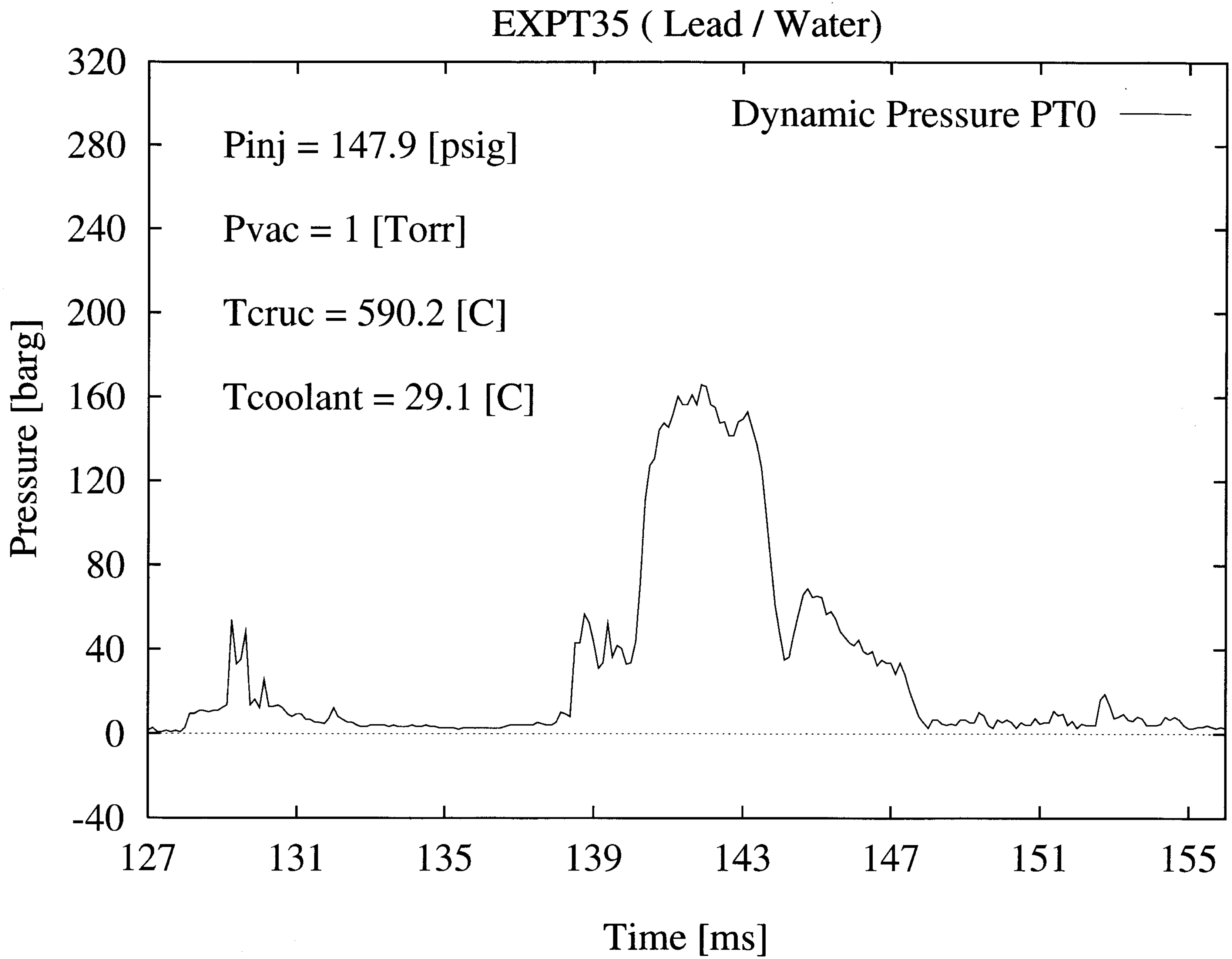

Figure 13b. Instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C. Transducer PT0, expanded time scale.

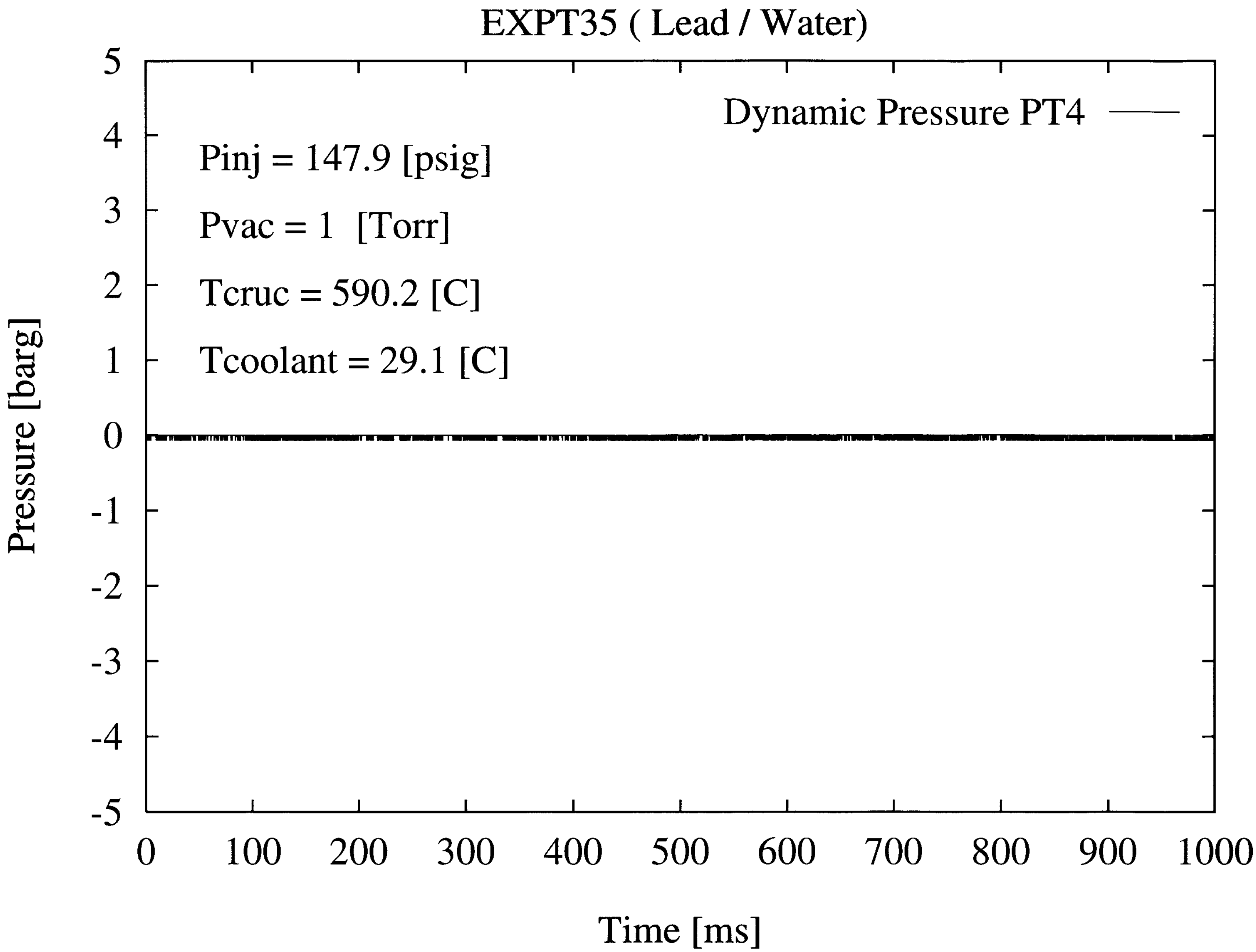

Figure 13c. Instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C. Transducer PT4, 1000 ms trace.

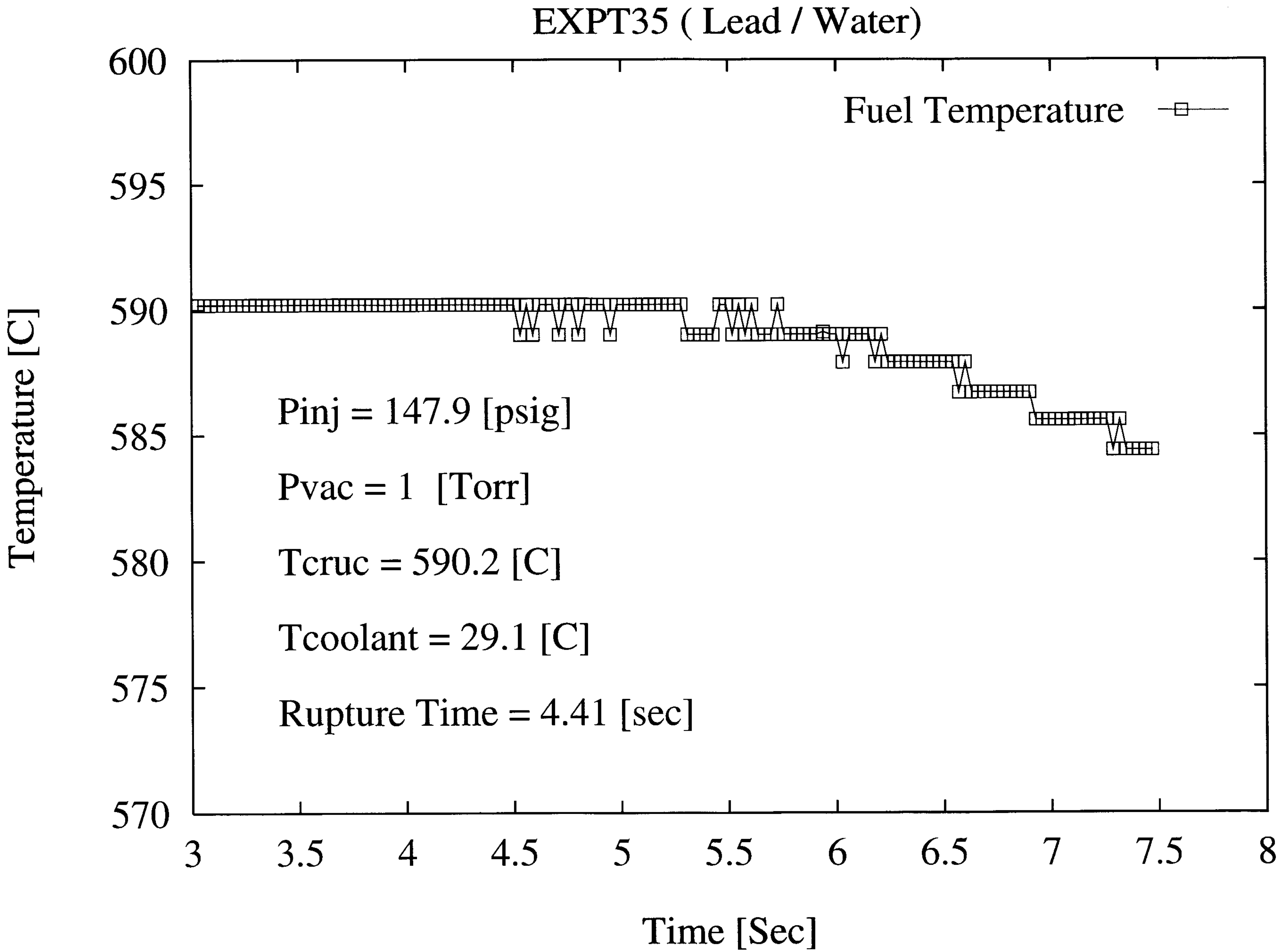

Figure 13d. Instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C. Thermocouple TC5, 5 s trace.

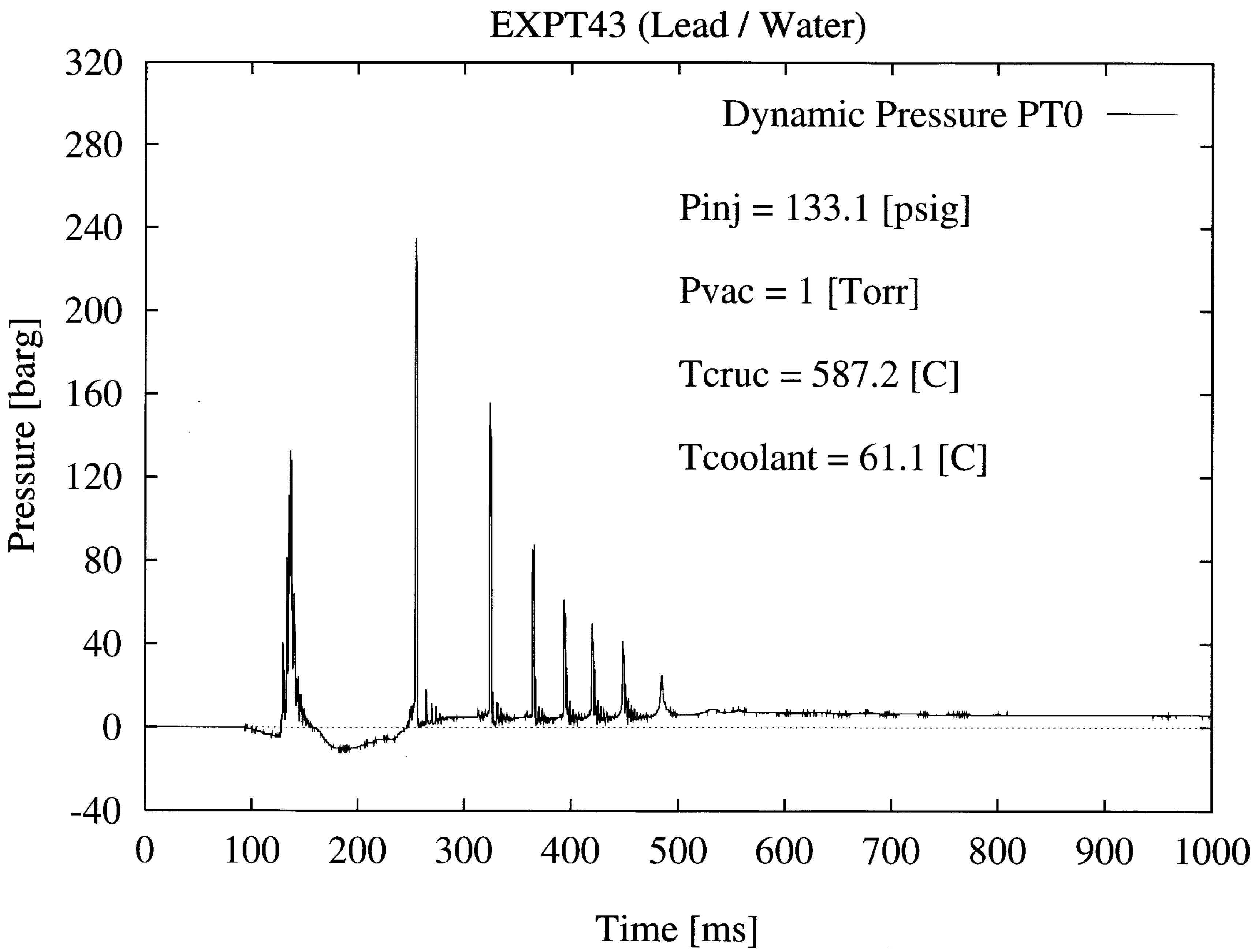

Figure 14a. Instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C. Transducer PT0, 1000 ms trace.

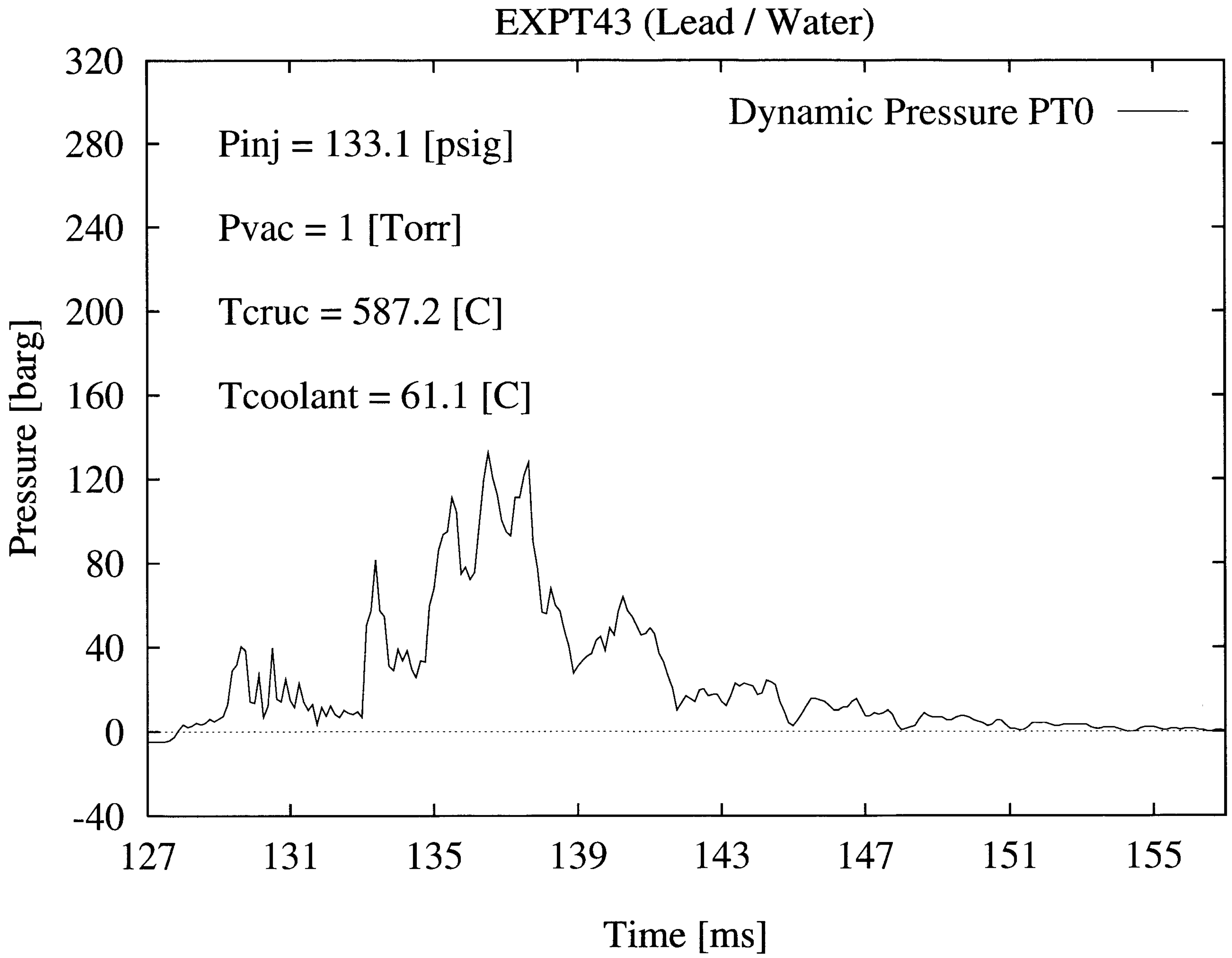

Figure 14b. Instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C. Transducer PT0, expanded time scale.

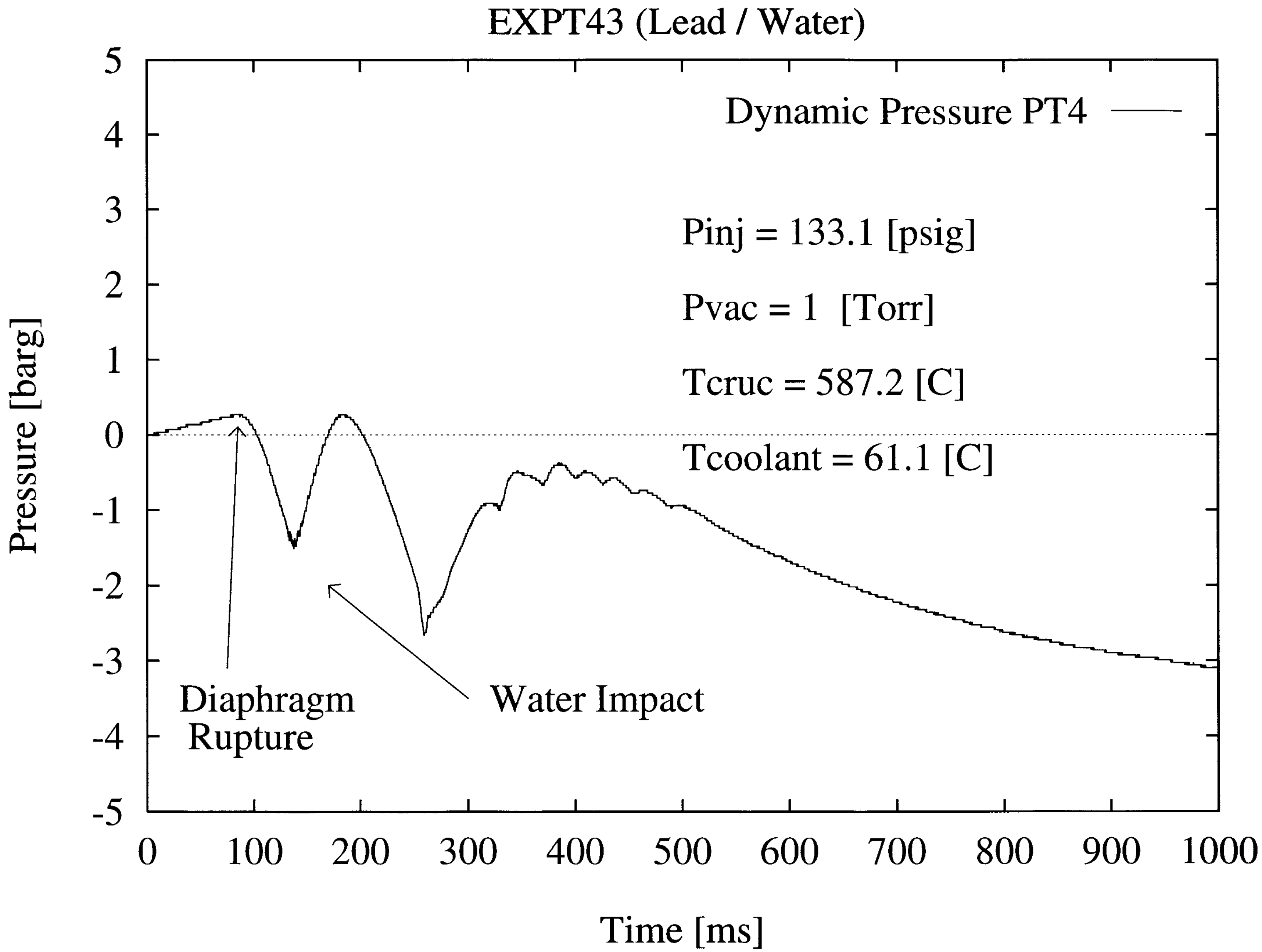

Figure 14c. Instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C. Transducer PT4, 1000 ms trace.

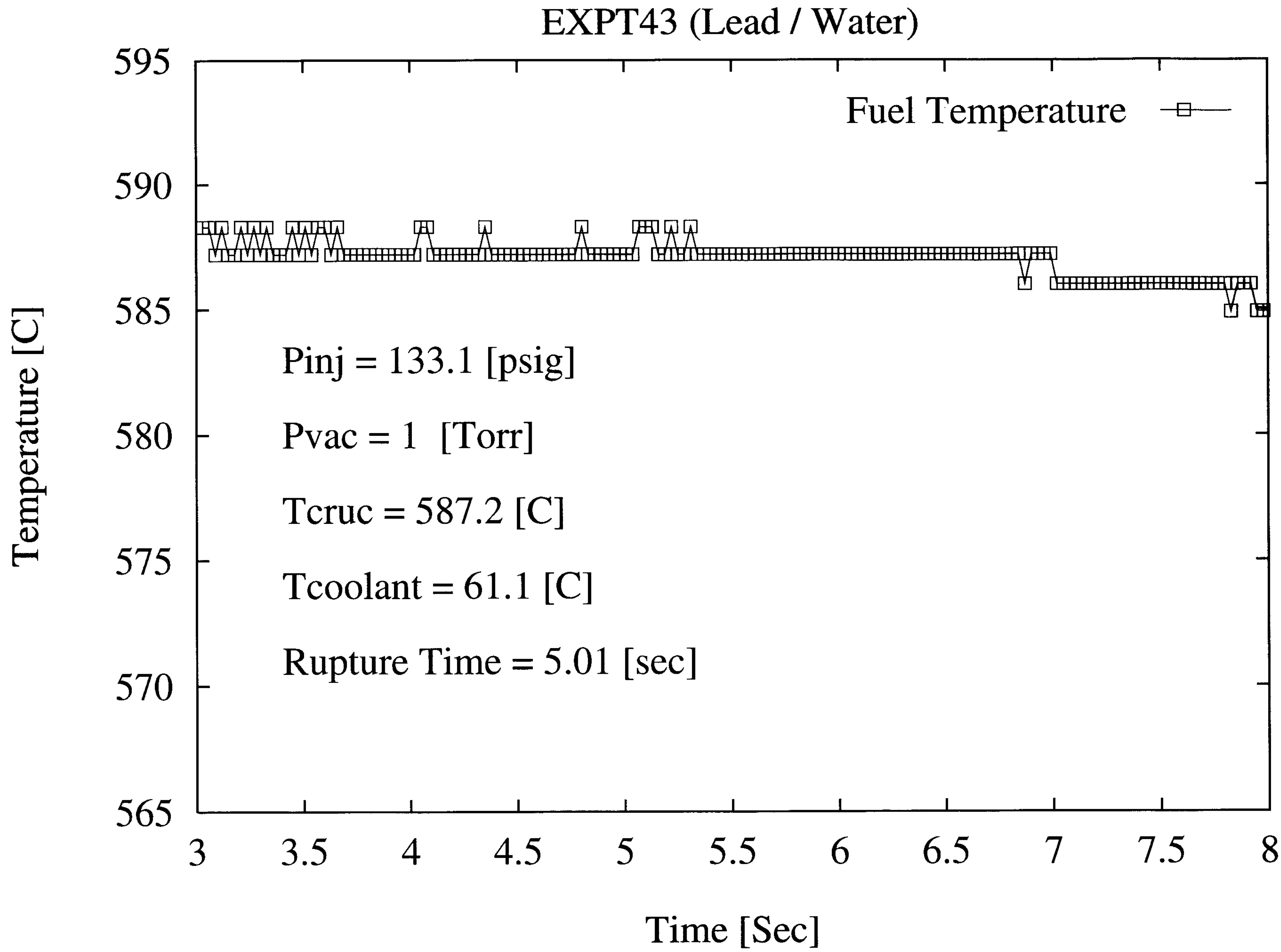

Figure 14d. Instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C. Thermocouple TC5, 5 s trace.

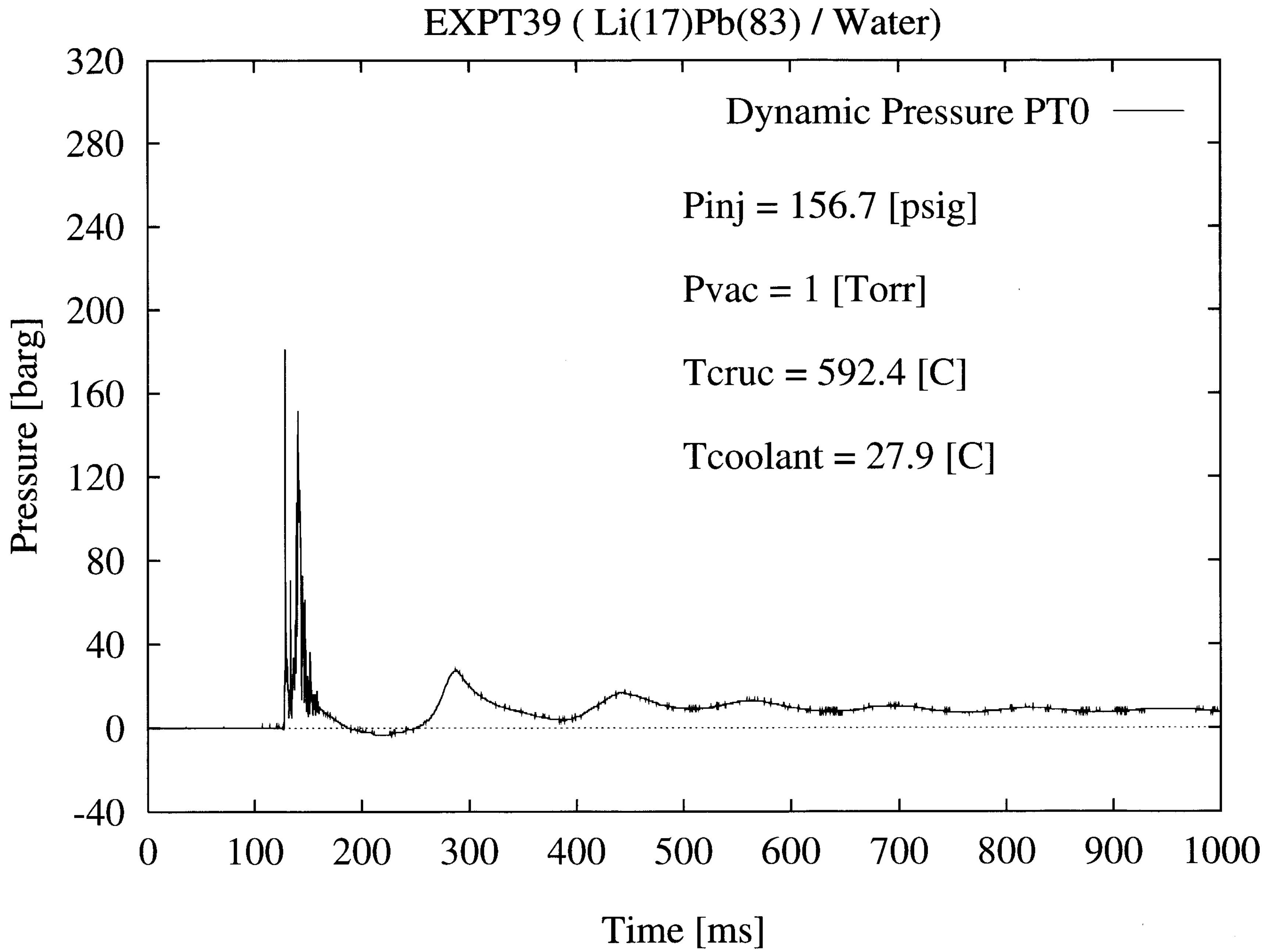

Figure 15a. Instrumental records from experiment B-39-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 592.4°C; water was at 27.9°C. Transducer PT0, 1000 ms trace.

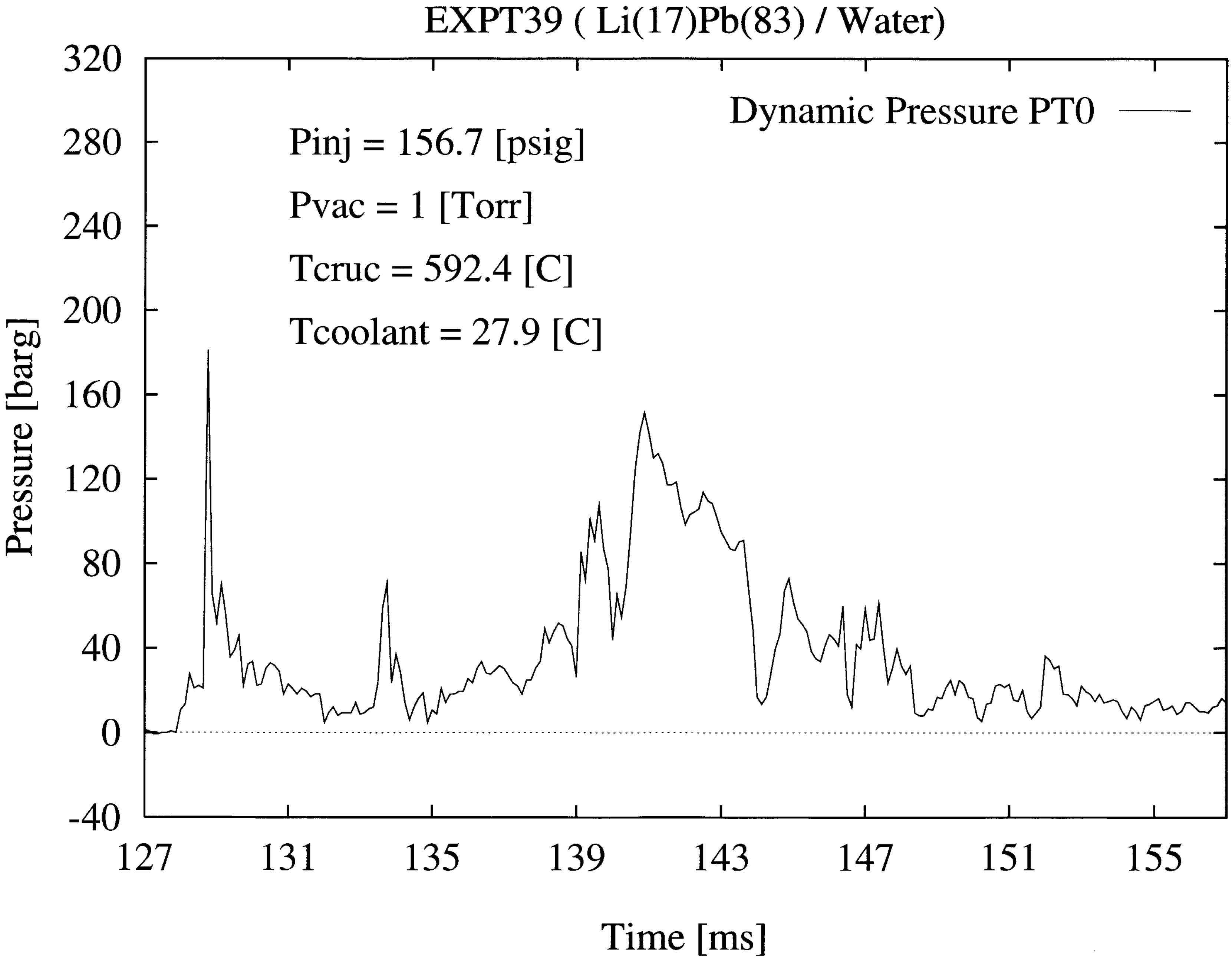

Figure 15b. Instrumental records from experiment B-39-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 592.4°C; water was at 27.9°C. Transducer PT0, expanded time scale.

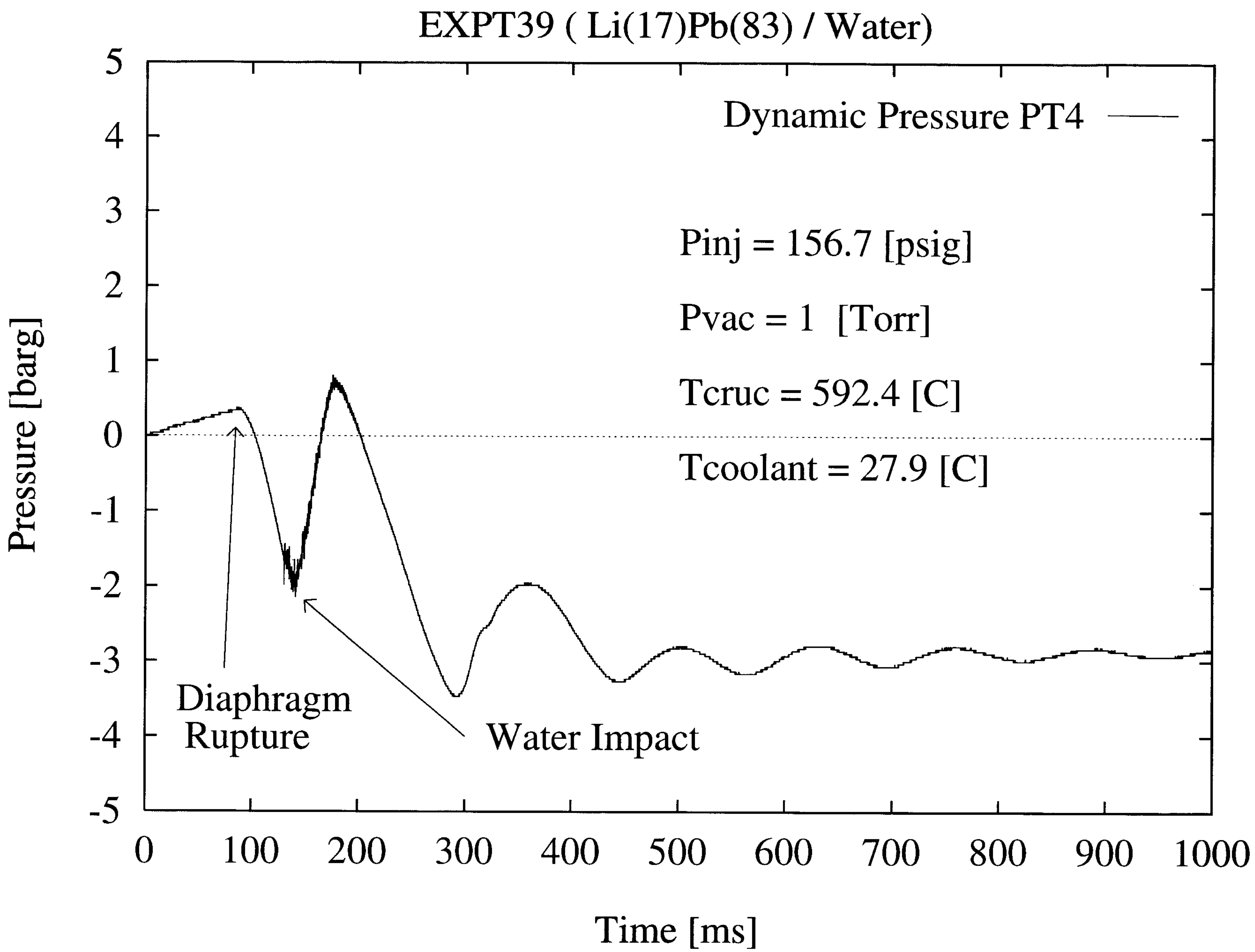

Figure 15c. Instrumental records from experiment B-39-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 592.4°C; water was at 27.9°C. Transducer PT4, 1000 ms trace.

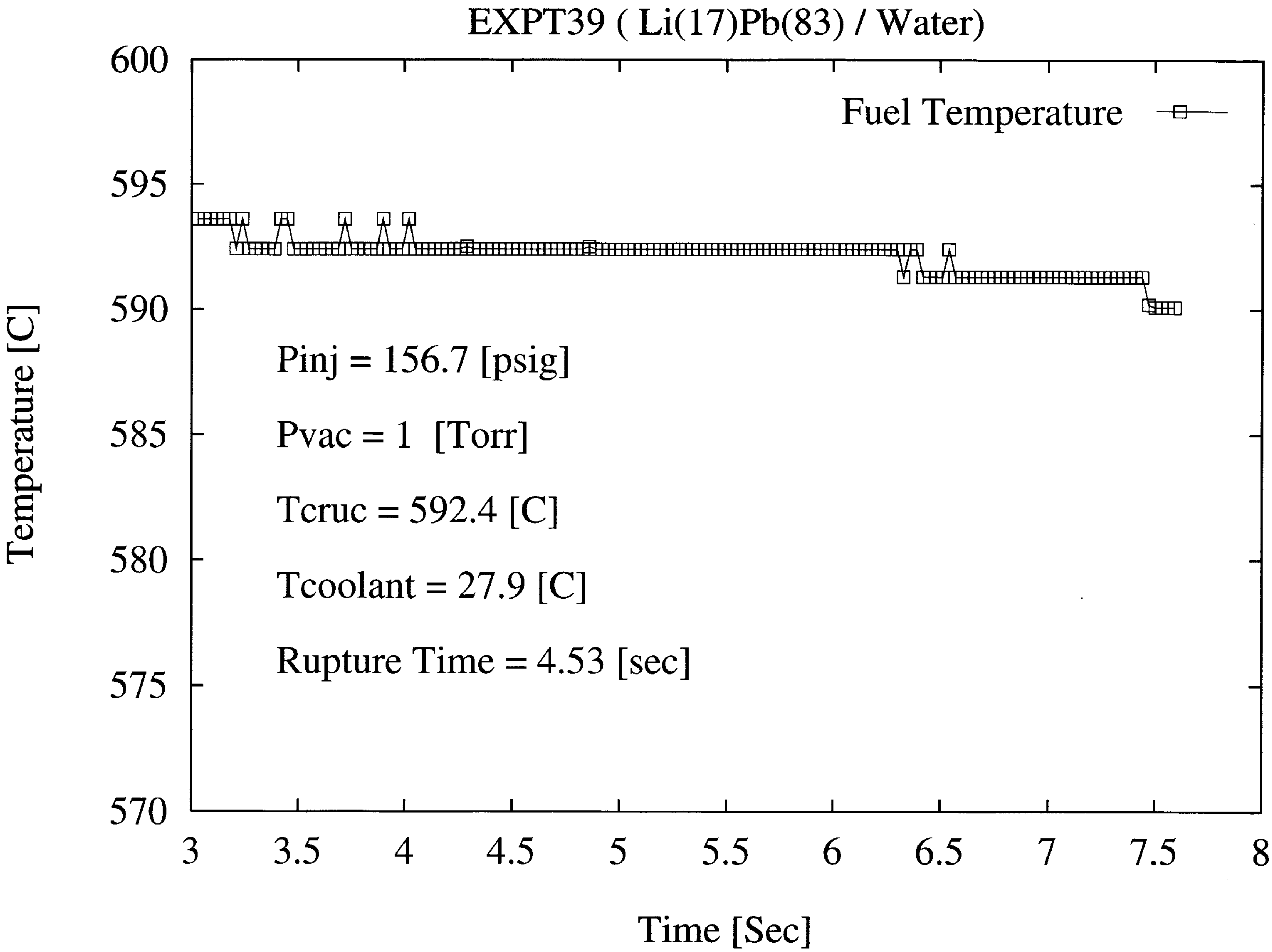

Figure 15d. Instrumental records from experiment B-39-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 592.4°C; water was at 27.9°C. Thermocouple TC5, 5 s trace.

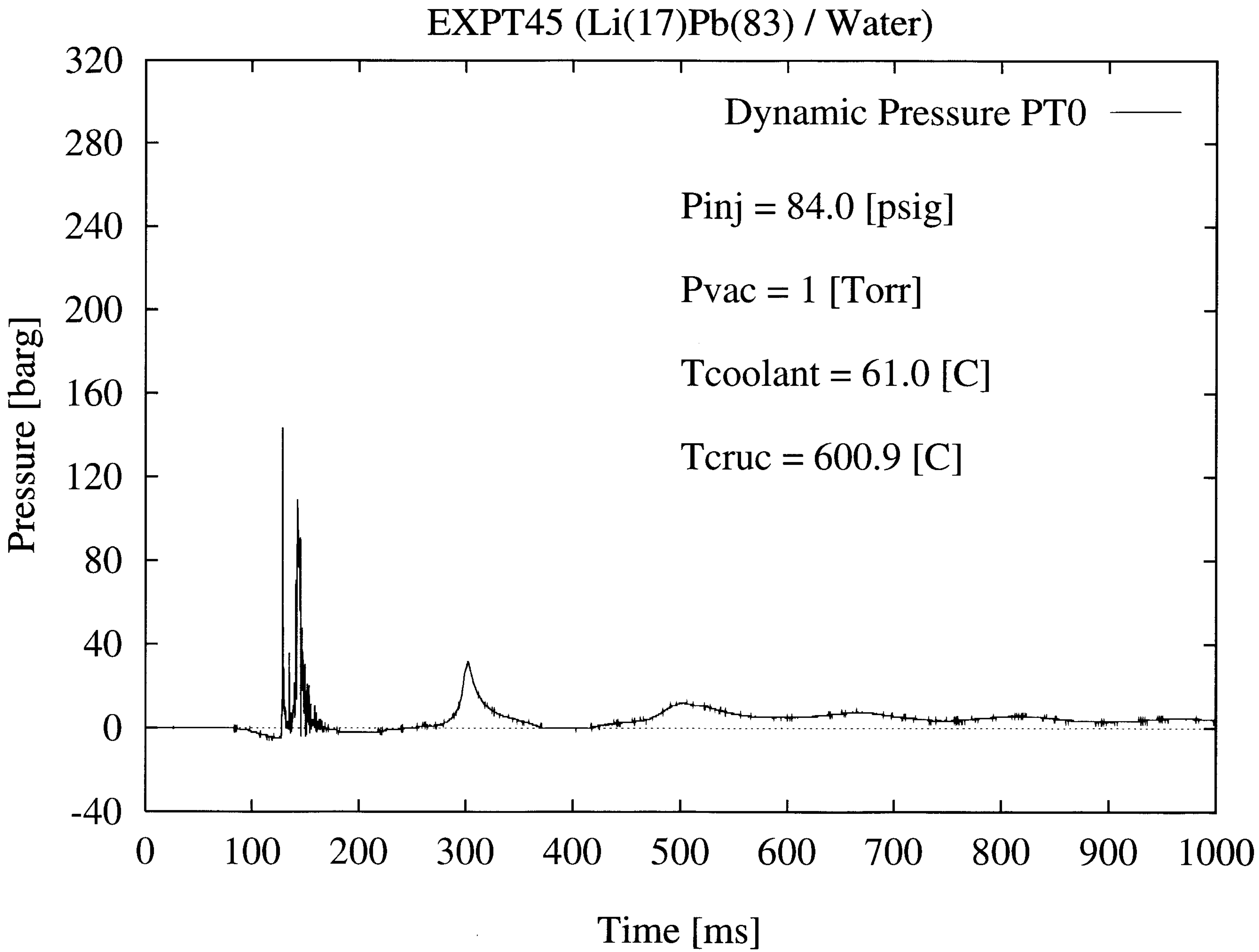

Figure 16a. Instrumental records from experiment B-45-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 600.9°C; water was at 61.0°C. Transducer PT0, 1000 ms trace.

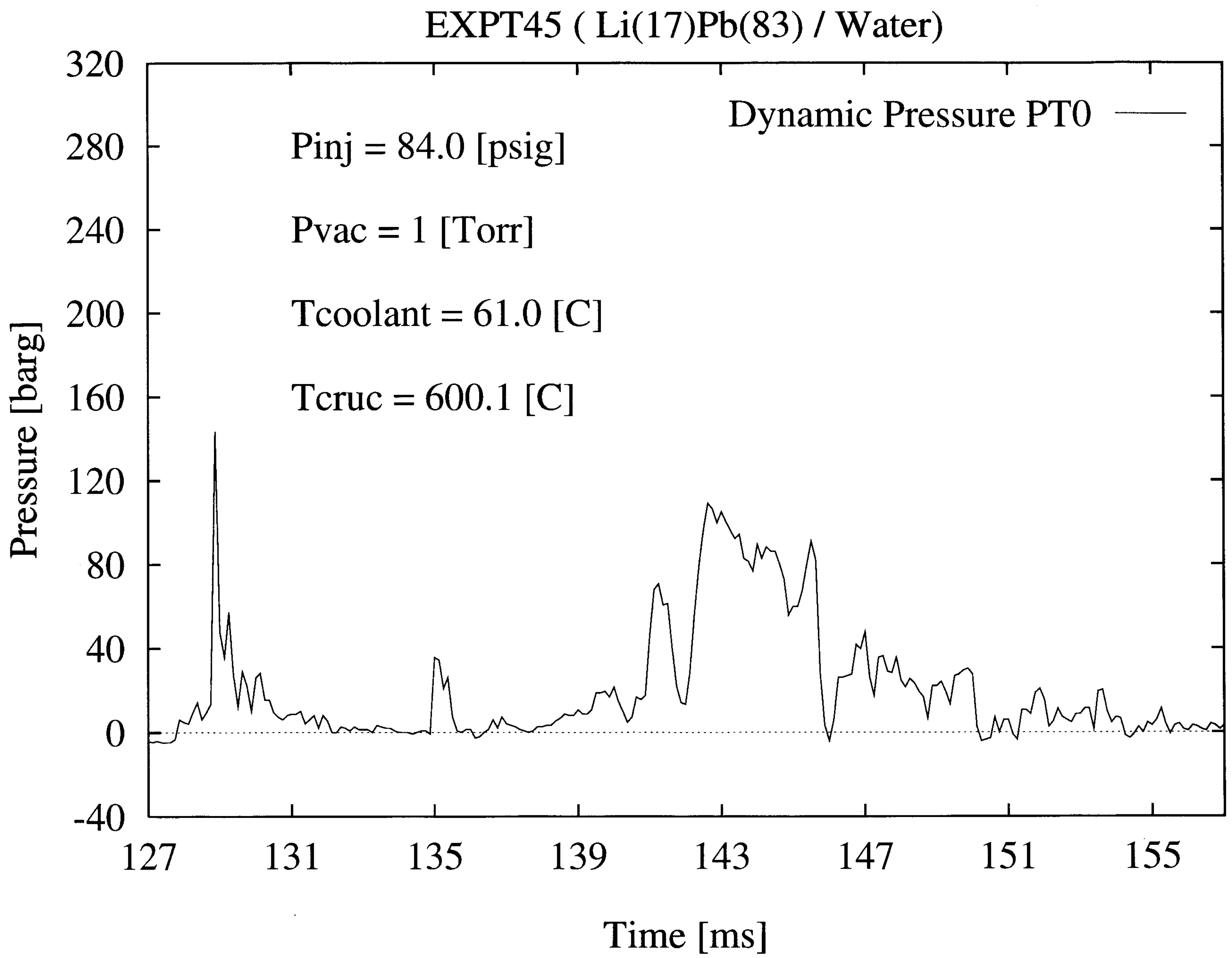

Figure 16b. Instrumental records from experiment B-45-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 600.9°C; water was at 61.0°C. Transducer PT0, expanded time scale.

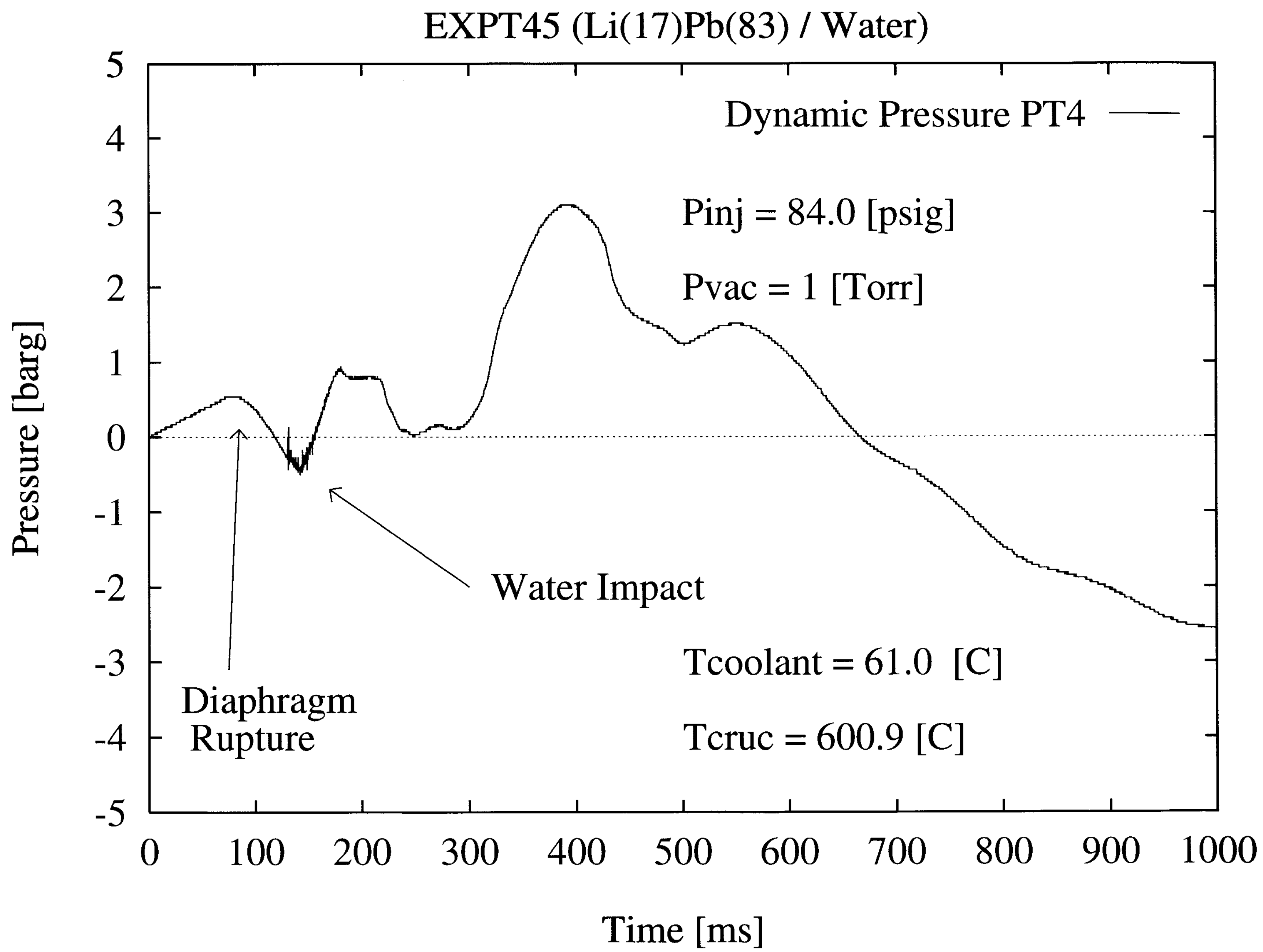

Figure 16c. Instrumental records from experiment B-45-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 600.9°C; water was at 61.0°C. Transducer PT4, 1000 ms trace.

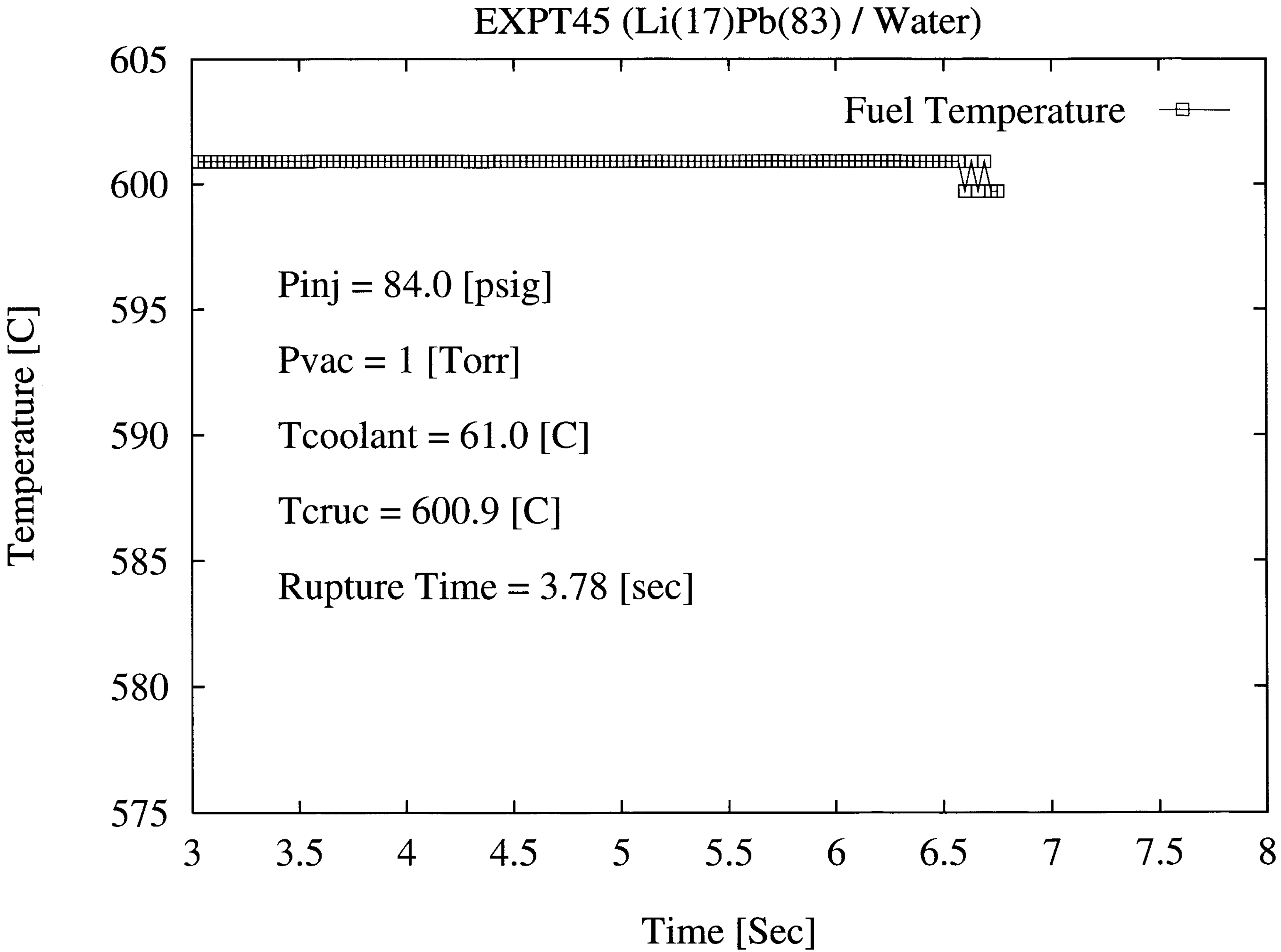

Figure 16d. Instrumental records from experiment B-45-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 600.9°C; water was at 61.0°C. Thermocouple TC5, 5 s trace.

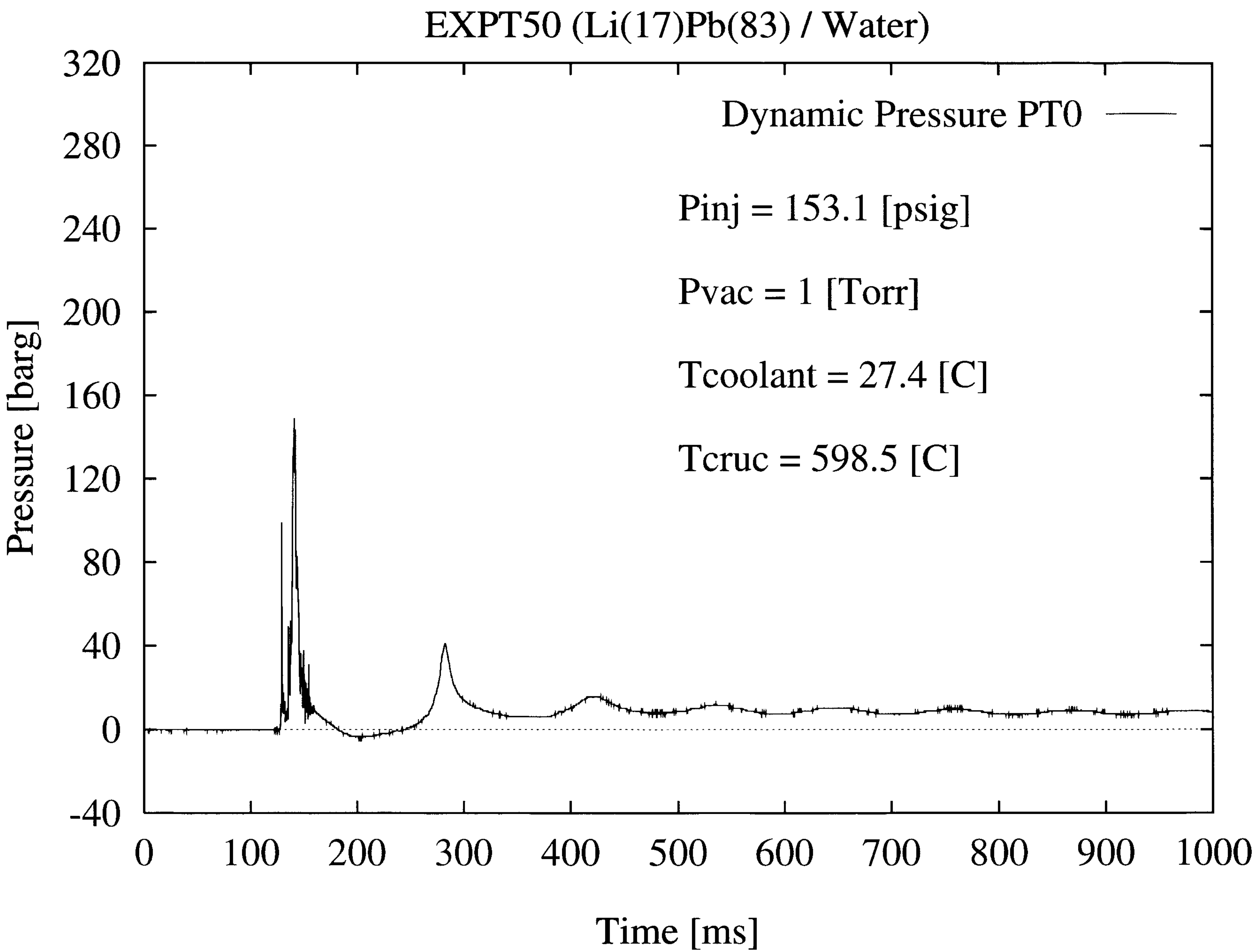

Figure 17a. Instrumental records from experiment B-50-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 598.5°C; water was at 27.4°C. Transducer PT0, 1000 ms trace.

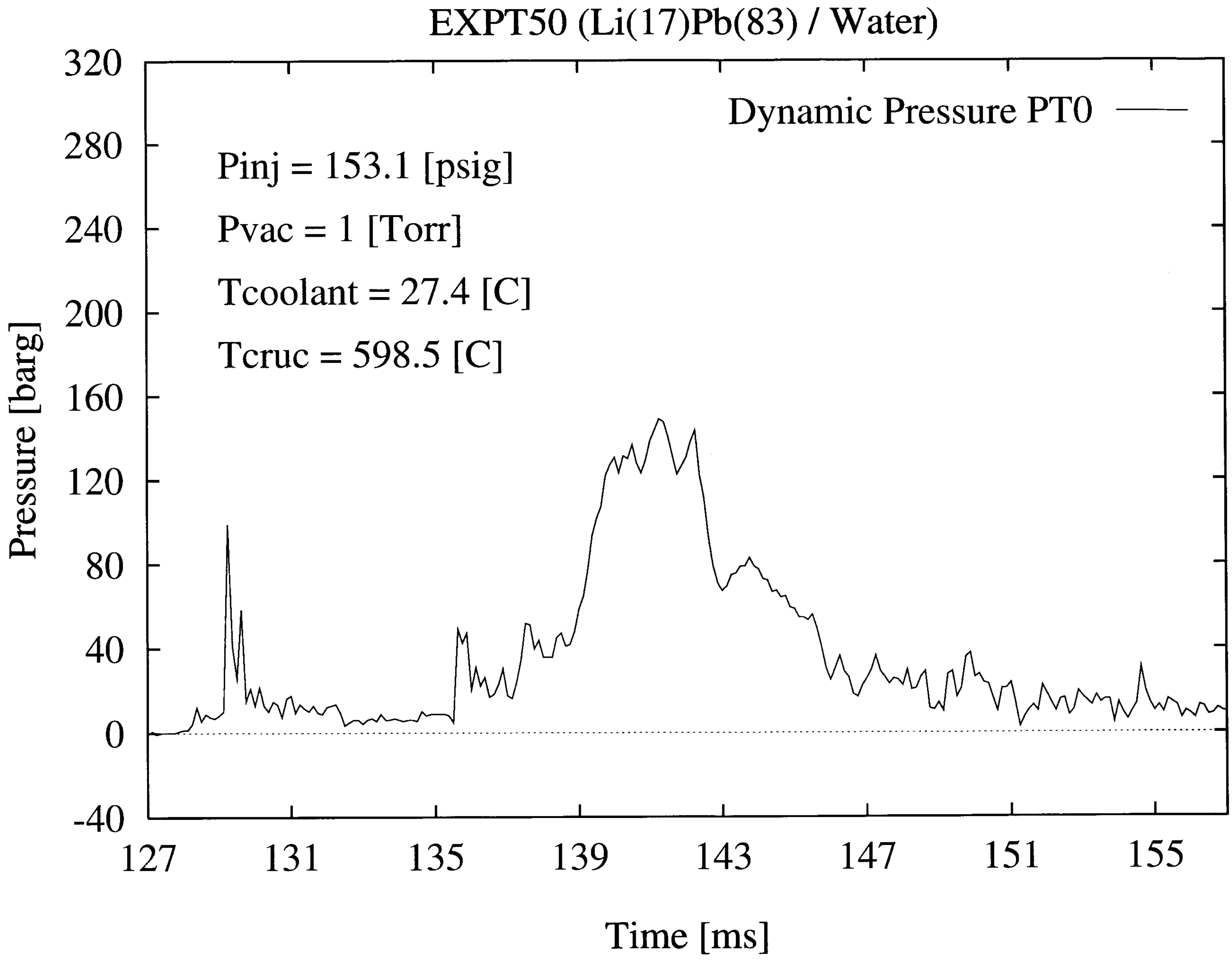

Figure 17b. Instrumental records from experiment B-50-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 598.5°C; water was at 27.4°C. Transducer PT0, expanded time scale.

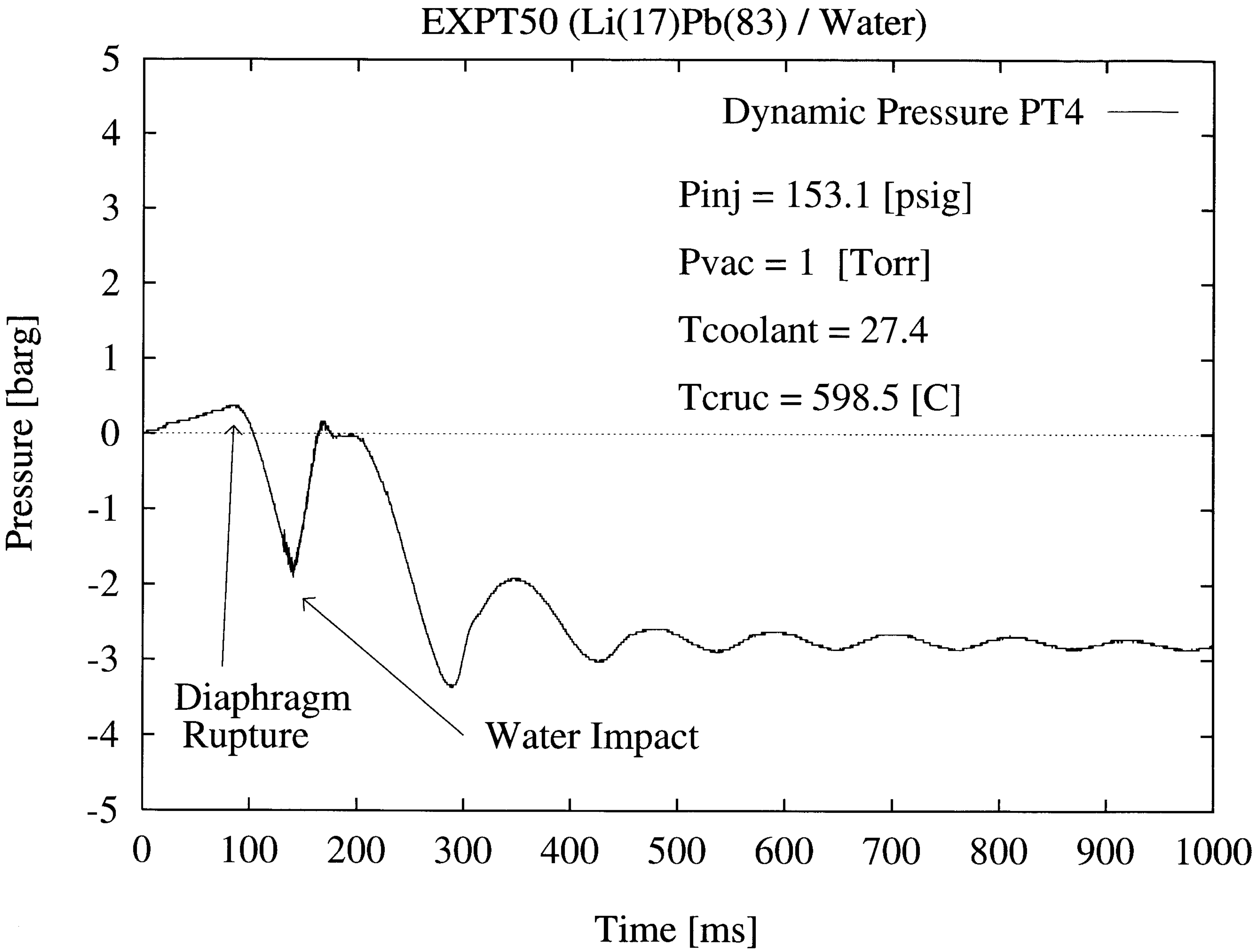

Figure 17c. Instrumental records from experiment B-50-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 598.5°C; water was at 27.4°C. Transducer PT4, 1000 ms trace.

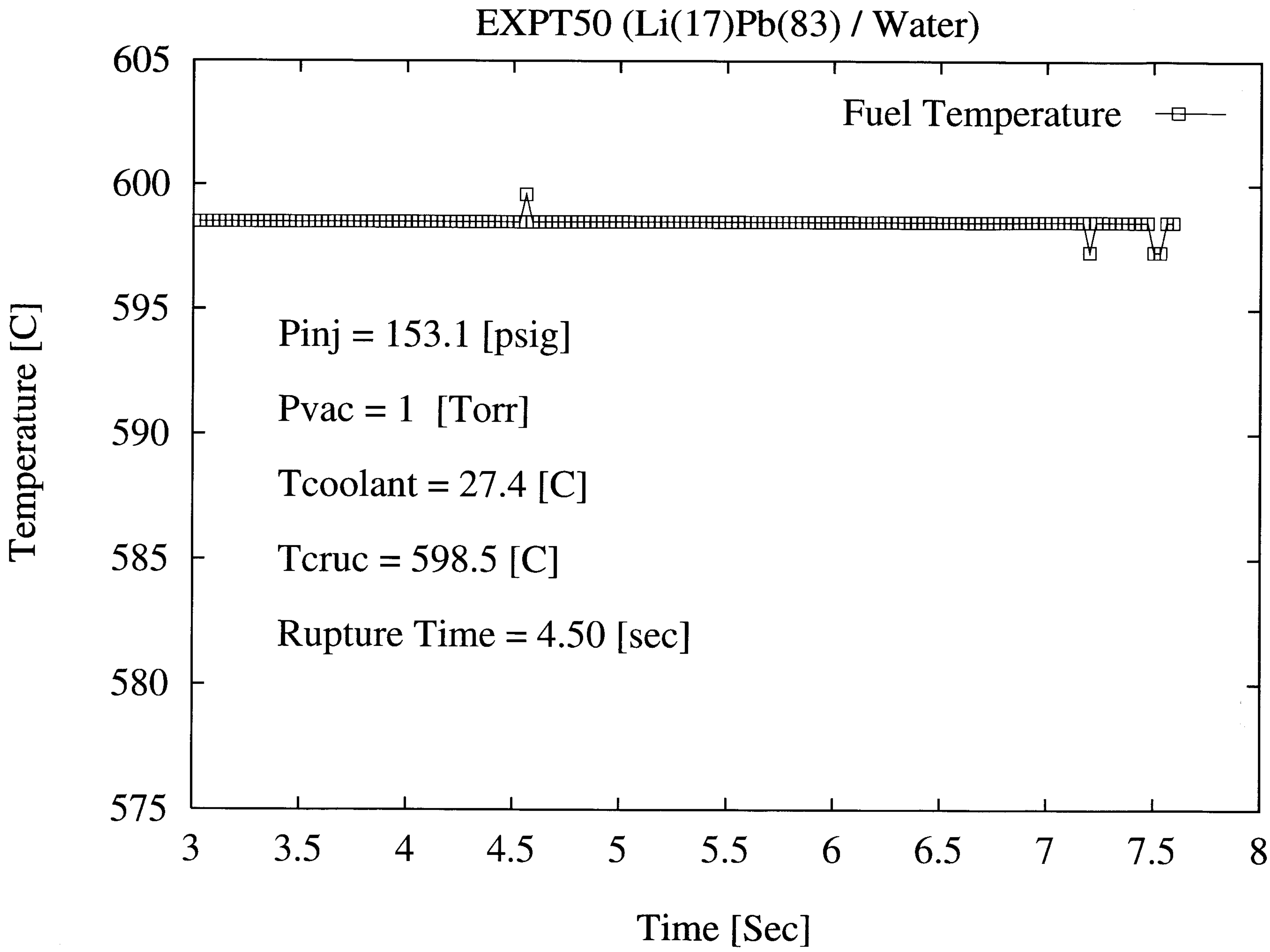

Figure 17d. Instrumental records from experiment B-50-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 598.5°C; water was at 27.4°C. Thermocouple TC5, 5 s trace.

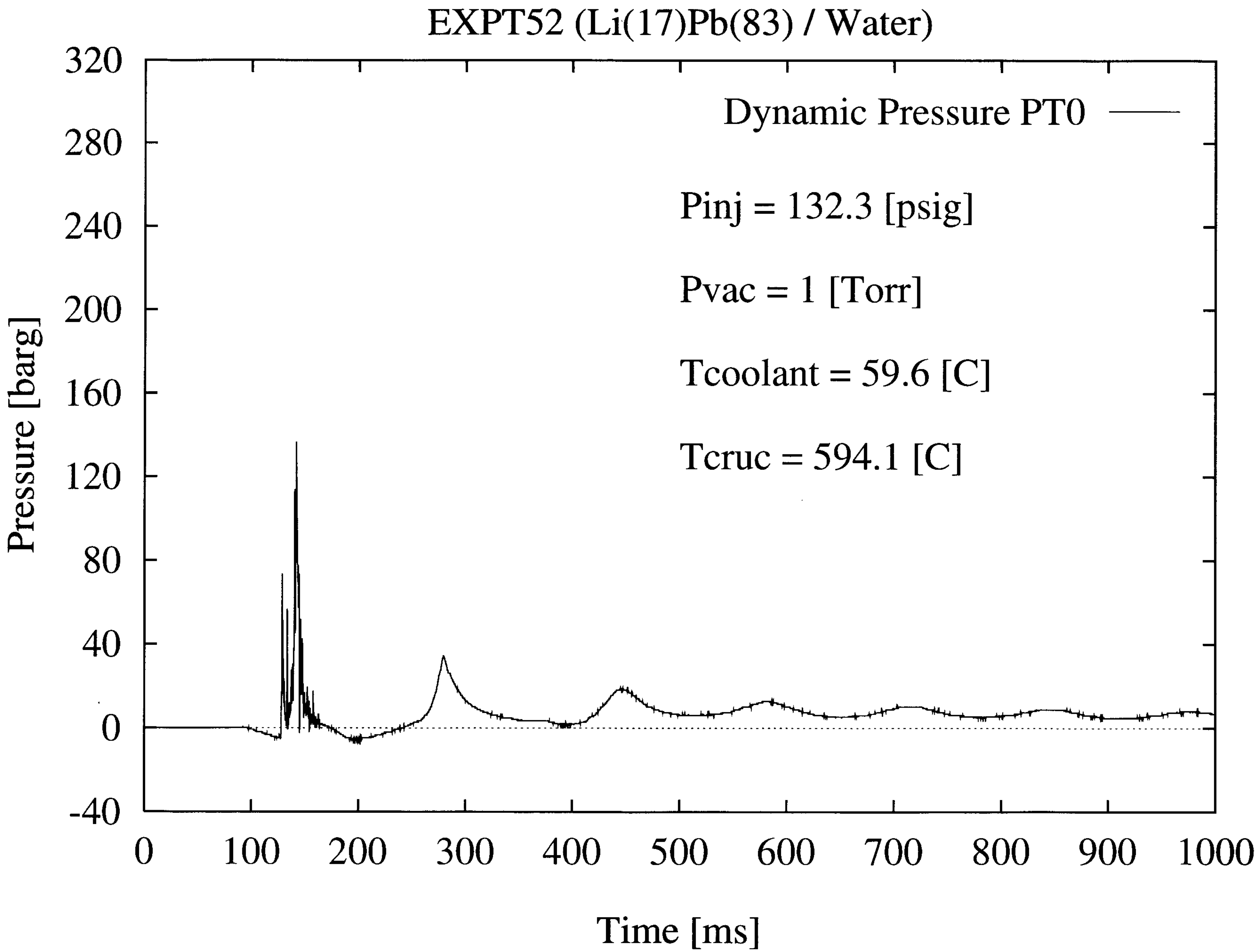

Figure 18a. Instrumental records from experiment B-52-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 594.1°C; water was at 59.6°C. Transducer PT0, 1000 ms trace.

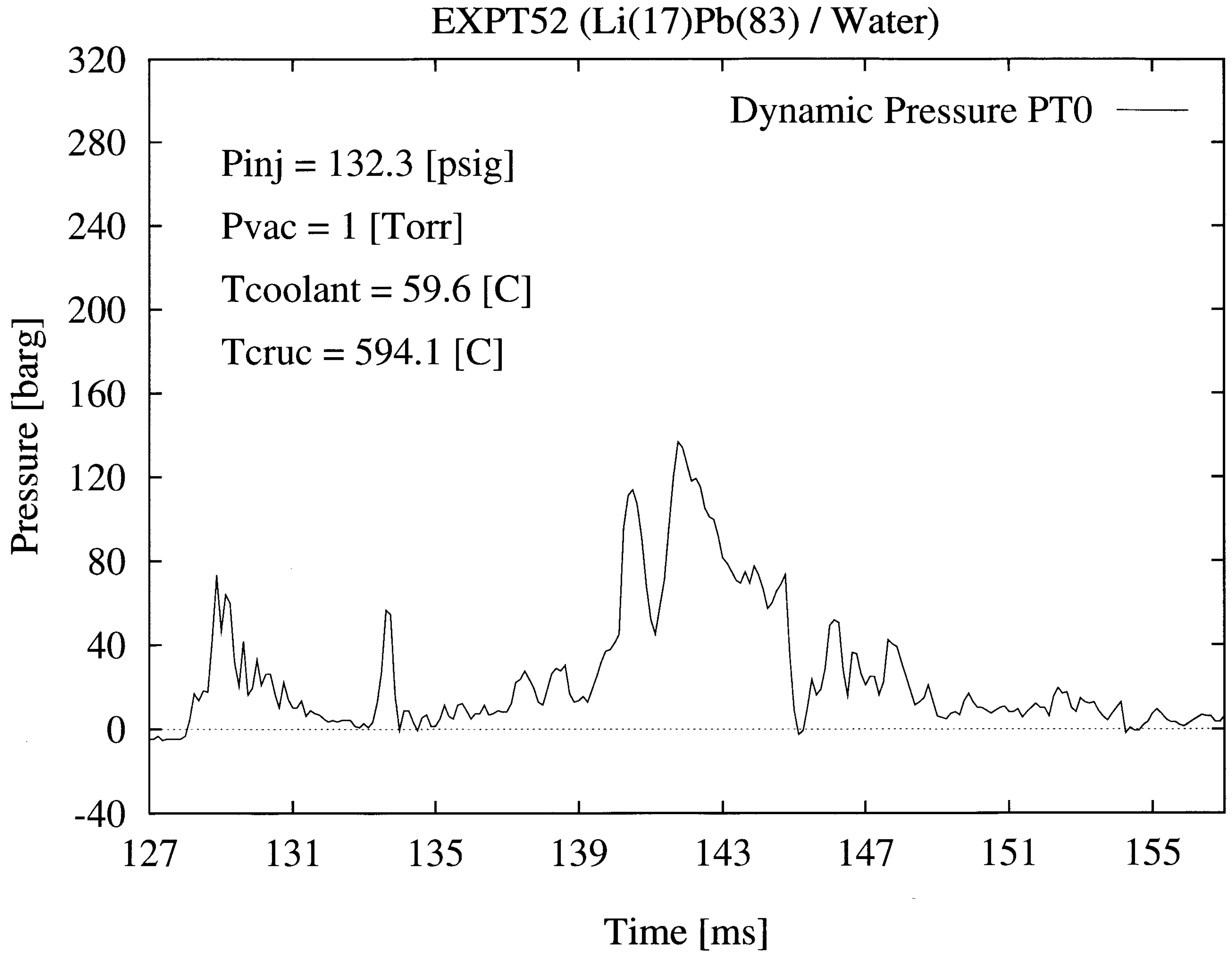

Figure 18b. Instrumental records from experiment B-52-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 594.1°C; water was at 59.6°C. Transducer PT0, expanded time scale.

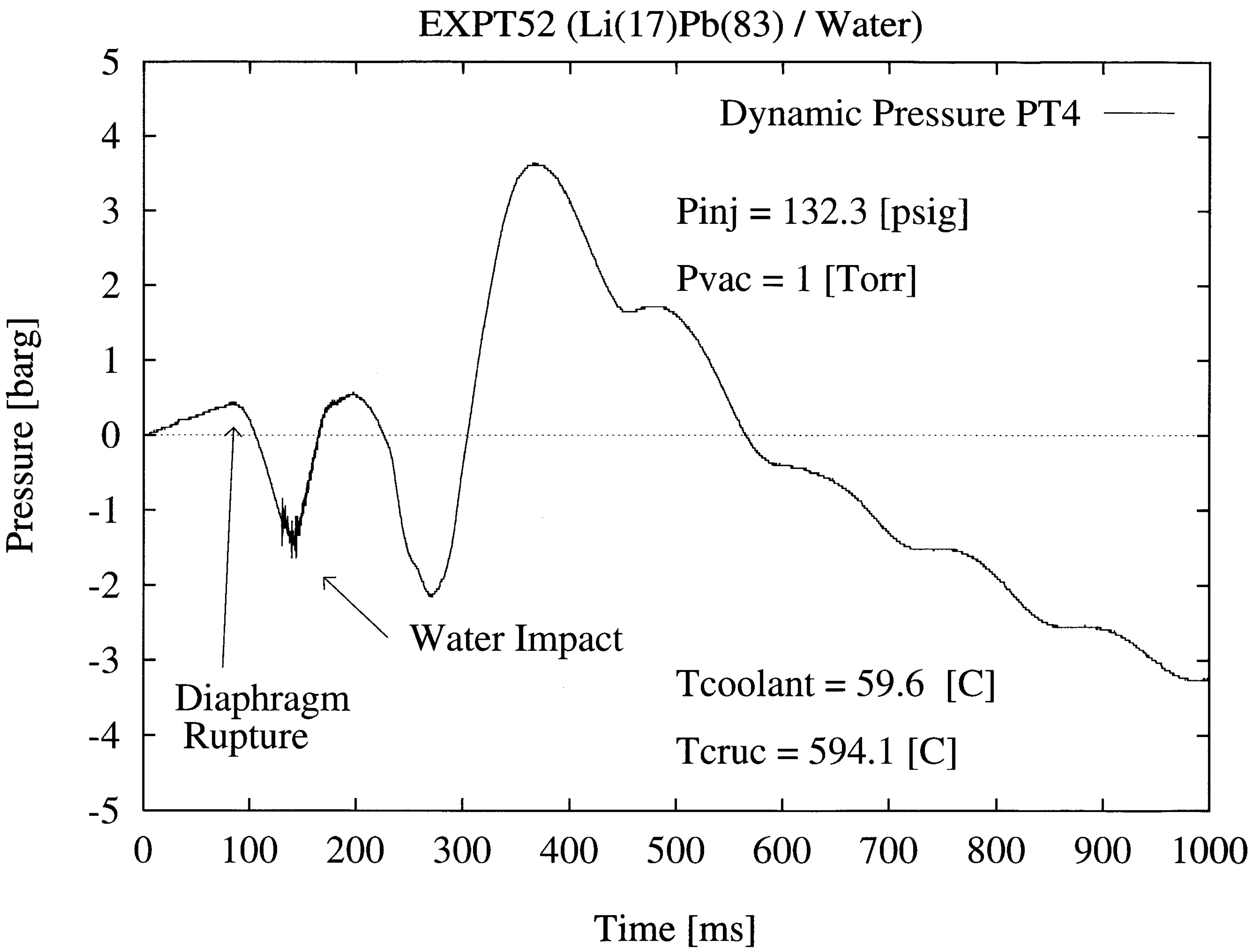

Figure 18c. Instrumental records from experiment B-52-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 594.1°C; water was at 59.6°C. Transducer PT4, 1000 ms trace.

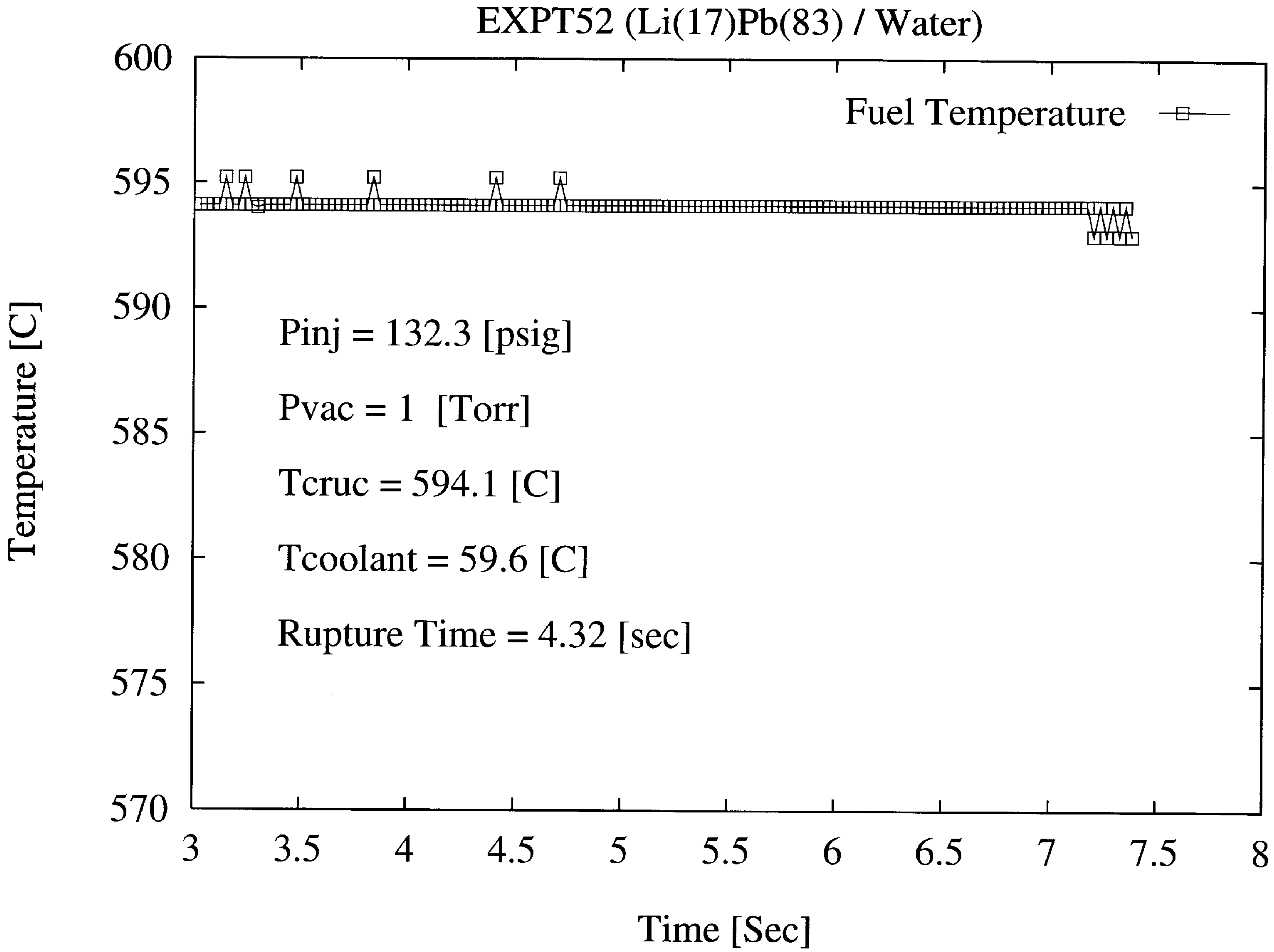

Figure 18d. Instrumental records from experiment B-52-1. Crucible contained molten 83a/o Pb-17a/oLi alloy at 594.1°C; water was at 59.6°C. Thermocouple TC5, 5 s trace.

Typical pressure profiles recorded during the impact of a pressurized water column upon the surface of an empty crucible, at room temperature, are shown in Figures 11a, b and c. The value of the pressure at the peak of the first rise in the PT4 in trace Figure 11c corresponds to the injection pressure as measured by the strain gauge transducer in the upper chamber. Therefore, the PT4 trace indicates only the changes in the gas pressure during the experiment and not the absolute values of the pressure. The absolute value of the gas pressure at any time during the experiment can be obtained by simple calculations using the strain gauge and the PT4 records. However, matching the pressure data from PT5, the strain gauge, with those of PT4 will underestimate the injection pressures (this is due to the different data acquisition frequencies for the two transducers) and hence, the gas pressure at all other times. Using the slope of the pressure as measured by the strain gauge transducer, we found that the maximum error in the value of the injection pressure cannot be greater than 20 kPa. The traces in Figures 11 through 18, a and c, are shown between 0 and 1000 ms. Time zero does not correspond to the actual beginning of an experiment; it is recalibrated here in order to eliminate the unnecessary data from the opening of the solenoid valve until some time before the diaphragm ruptures.

After the three-second countdown, the solenoid valve will be opened and the argon gas will rush into the expansion vessel. The rush of the argon gas will pressurize the water column and will force the diaphragm to rupture. During the pressurization the transducer PT4 records the pressure as shown in Figures 11c through 18c. When the diaphragm ruptures, the coolant column starts to move downward; hence, the pressure will decrease in the vicinity of the transducer PT1 (see Appendix B). An expansion wave will carry this information upward to the expansion vessel. As the water moves down toward the bottom of the crucible, the water vapor that was produced because of the vacuum below the rupture disk will be pressurized. The vapor pressurization can be seen better if we replot PT0.

Figures 11b through 18b show traces recorded on expanded time scales from transducer PT0 at the time of the first impact of the water with the empty crucible or the melt. The vapor, which was formed at the leading surface of the water column in the vacuum, will eventually condense in whole or in part. The condensation is accompanied by a decrease in the pressure as measured by PT0 and is shown in all of the traces in Figures 11b through 18b. However, we speculate that high pressure vapor may be produced should there be any source of heat that is hot enough to force vapor generation. Therefore, in cold empty crucible experiments such as experiment B-25-1, one does not expect to see any second pressure peak prior to the impact of the water mass upon the surface of the crucible. We believe that the existence of two pressure peaks prior to the main impact of the water upon the fuel or the crucible surface indicates the presence of high pressure water vapor. The collapse of the second vapor film might trigger thermal- or ignition-type vapor explosions.

The shape of the pressure trace from transducer PT0 for the main impact is affected by the crucible loading. The pressure trace has a plateau at its maximum in an empty crucible, commonly called solid impact experiments at room temperature (see experiment B-25-1 in Figure 11b). The time interval of the pressure plateau is the same as the actual acoustic relief time, which is the time for a pressure signal to travel from one end of the tube to the other end and return, for that specific experiment. The actual pressure relief time is affected by void fraction and its distribution, presence of contact surfaces, and boundary layer; hence, it is impossible to calculate the actual pressure relief time if the flow patterns within the tube are not completely understood. Theoretical pressure relief time may be calculated when some assumptions regarding the flow patterns are made in order to determine the sound speed, C, in the flow. The theoretical acoustic relief time, t^{th}, can be determined by equation (1) when C is known:

$$t^{th} = \frac{2h_c}{C} \tag{1}$$

where h_c is the length of the coolant column. The speed of the sound was determined to be ~1400 m/s in the volume occupied between transducers PT1 and PT3 (see Appendix B). The maximum hydrodynamic pressure generated due to the impact of the water column upon the surface of an empty crucible at vacuum, Δp^{th} (the formation of any water vapor because of the vacuum will reduce the impact pressure), can be estimated

$$\Delta p^{th} = p_c C_c v_c \qquad (2)$$

$$v_c = v' t_{im} \qquad (3)$$

$$v' = g + \frac{P_{burst}}{p_c h_c} \qquad (4)$$

$$t_{im} = \sqrt{\frac{2h_f}{n'}} \qquad (5)$$

where p_c, C_c, v_c, h_f, v', and P_{burst} are coolant density, sound speed in the coolant, velocity at impact, height of the coolant fall, coolant acceleration, and pressure difference across the coolant column, respectively. Due to additional losses because of the coolant phase change, the pressure plateau has a small negative slope with time in solid impact-like experiments at higher temperatures. The change in the pressure plateau in a hot solid impact (i.e., hot empty crucible) experiment can be seen in experiment B-31-1 (see Figure 12). The impact of water upon liquid surfaces does not have the pressure plateau anymore, but a series of pressure oscillations suggestive of coarse melt and coolant mixing. Figure 15 shows the trace for transducer PT0 for experiment B-39-1, which illustrates the pressure transient of the coolant impact upon the surface of the molten lithium-lead alloy in the crucible.

The generation of water vapor and hydrogen gas can contribute significantly to the pressurization of the gas in the expansion chamber during the experiment. The increase of the pressure inside the upper chamber beyond the initial driving pressure is part of the work done in the melt-coolant interaction.

Temperature Profiles

Figures 11d through 18d show the temperature profiles for the eight experiments conducted. The temperature traces are plotted for approximate time intervals of 4 or 5 seconds. The initial time in the plots, 3 s, corresponds to the end of the three second-long countdown and the opening of the solenoid valve. The Keithley data acquisition system continues to record data for a period of another three seconds after a triggering signal from transducer PT0. The signal from PT0 corresponds approximately to the time of the rupture disk failure. The rupture disk failure time is noted on all of the temperature plots. The crucible temperature, indicated here by TC5, is measured by a thermocouple inserted in a 2.5 mm depression in the bottom of the crucible (see Figure 4). Because of the positioning of the thermocouple, the temperatures measured are the crucible temperatures which are taken to be essentially the same as the melt temperatures. The thermocouples reach 63.2% of a step change in temperature in about 275 ms; thus, not only the temperature traces as measured by these sensors are slightly lower than the actual melt temperature, but also, rapid changes in the system temperature cannot be captured at all. Thermocouple TC5 indicates a fall in the crucible temperature in experiment B-31-2, where coolant at room temperature impacted a hot crucible (Figure 12d) and in experiments B-35-1 and B-43-1 where room temperature and hot coolant, respectively, impacted a hot molten lead surface (with no lithium present) (Figures 13d and 14d). However, when lithium was present, thermocouple TC5 indicated essentially no change in the crucible temperature when hot or cold coolant impacted the molten Pb-Li in experiments B-39-1, B-45-1, B-50-1 and B-52-1 (Figures 15d, 16d, 17d and 18d).

We attribute the failure of the crucible to cool quickly, when it contains the molten eutectic Pb-Li alloy, to the evolution of heat during the interaction with water; its rapid cool-off when the crucible is empty or when it contains molten Pb is attributed to the evolution of little or no heat during the interaction.

Hydrogen

Hydrogen analyses were performed on the gases that were sampled from the expansion vessel at the top of the column one (bottle A) and five (bottle B) minutes after the interactions for all experiments except preliminary experiment B-25-1; these sampling times were chosen for uniformity with the analyses of hydrogen produced with aluminum-containing melts in the same apparatus by Farahani (1995). The results of these analyses are presented in Table 1 as the total number of millmoles of hydrogen generated, as the number of millimoles generated per gram of metal initially placed in the crucible, and as the percentage of lithium originally present in the metal sample that would have been removed to produce the number of millimoles of hydrogen generated according to equation (7) below.

Typical quadrupole mass spectrometer plots are shown in Figure 19 for an experiment with molten lead (B-43-1) and in Figure 20 for an experiment with the molten eutectic alloy (B-45-1); both plots were produced with sample bottles B. Notice the strong hydrogen peak at mass 2 in Figure 20 that does not appear in Figure 19. The procedures used to quantitatively convert peak heights such as these into hydrogen concentrations are presented in Appendix C.

In preparing Table 1, we have estimated the percentage of lithium originally present in the eutectic alloy that would have been removed to produce the measured amounts of hydrogen according to the equations

$$2\,Li + H_2O \rightarrow Li_2O + H_2 \tag{6}$$

and

$$2\,Li + 2H_2O \rightarrow 2\,LiOH + H_2 \tag{7a}$$

or

$$2\,Li + 2H_2O \rightarrow 2\,Li^+ + 2OH^- + H_2\,. \tag{7b}$$

Notice that although the amount of water that reacts with each lithium atom in equation (7), the water-rich situation, is twice that in equation (6), the lithium-rich situation, each lithium atom still generates the same amount of hydrogen (one H atom per Li atom or 1/2 H_2 molecule per Li atom), regardless of whether the reaction follows path (6) or (7), or some fractional combination of the two reactions. (See Hubberstey and Sample (1993) for a careful thermodynamic study of these reactions.)

Aqueous Solutions

The aqueous solutions that remain after the alloy-water interactions should contain a number of moles of LiOH (i.e., number moles of both Li^+ and OH^- ions) equivalent to exactly twice the number of moles of hydrogen gas (H_2) generated, provided the water-rich equation (7)

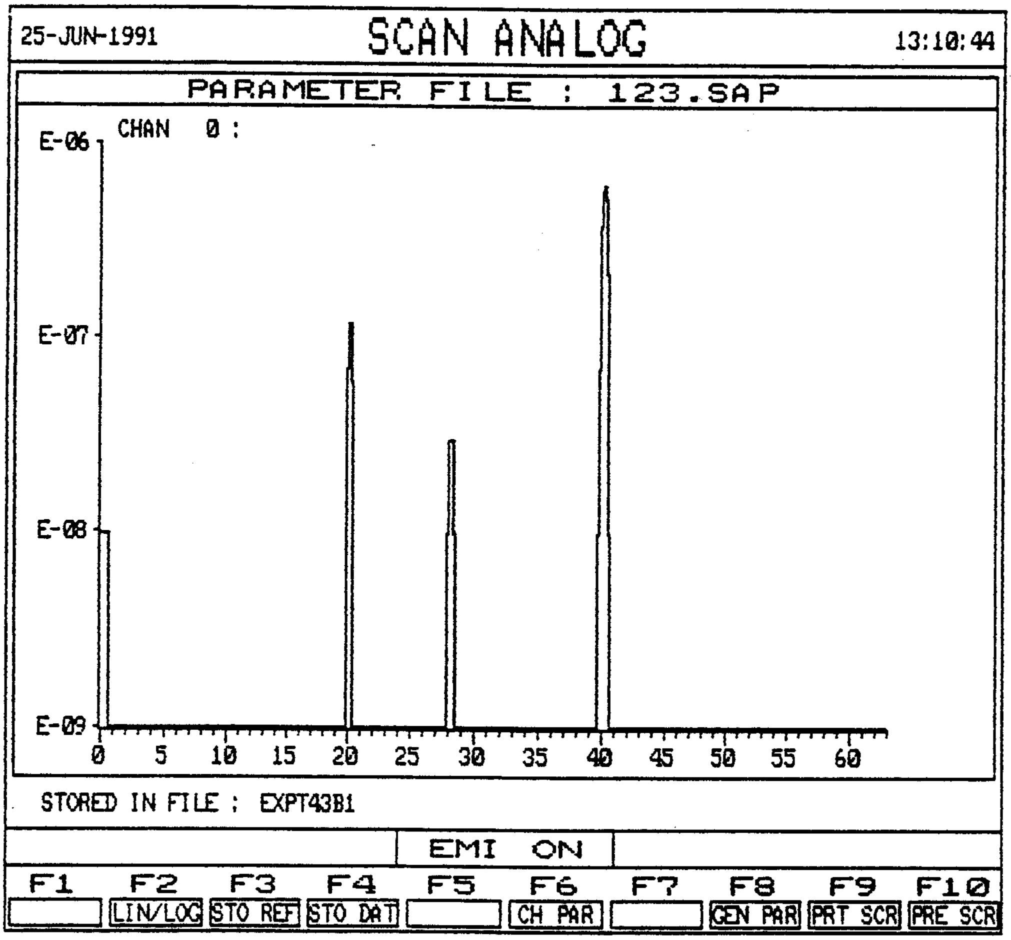

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Figure 19. Typical mass spectrum for gases in Bottle B sampled 5 minutes after impact in experiment B-43-1; molten Pb was at 587.2°C, water was at 61.1°C. Note absence of peak at mass 2.

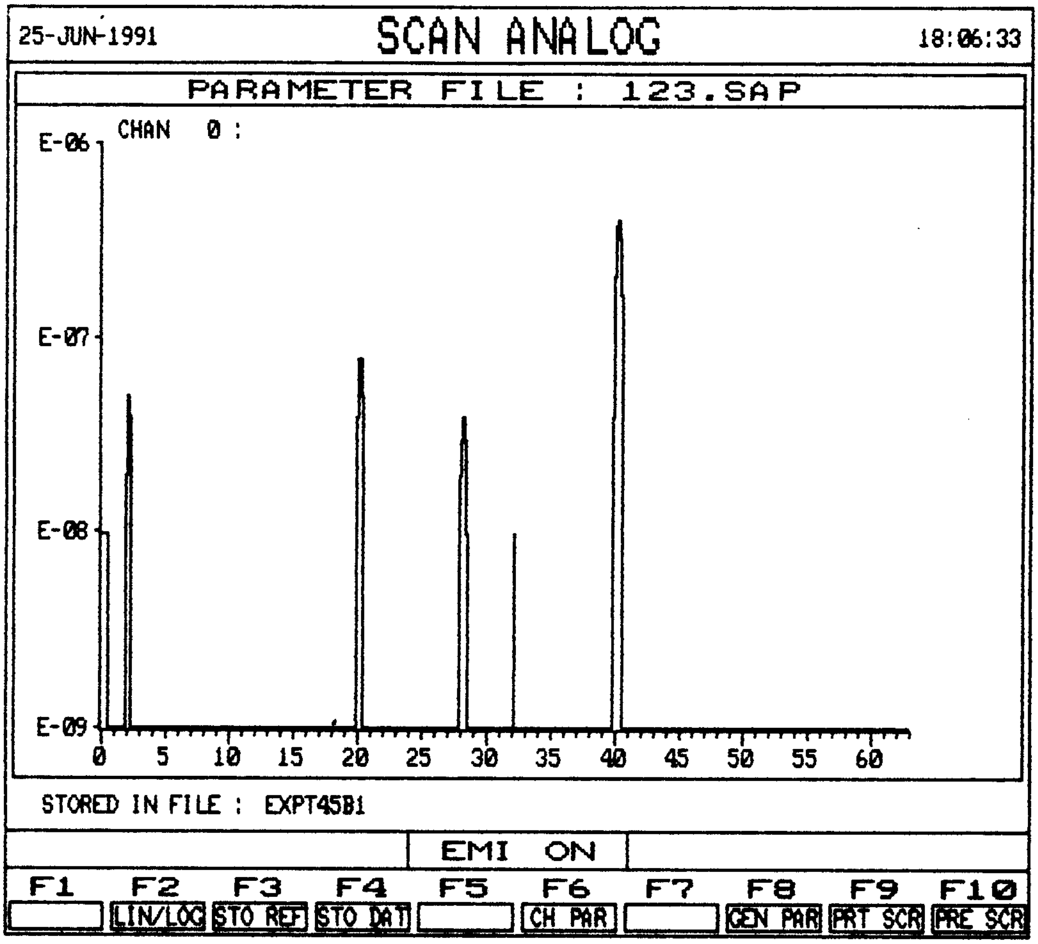

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Figure 20. Typical mass spectrum for gases in Bottle B sampled 5 minutes after impact in experiment B-45-1; molten eutectic Pb-Li alloy was at 600.9°C, water was at 61.0°C. Note strong peak at mass 2.

is the governing reaction (which should be the case in our experiments). Because it is important to know the amount of lithium removed from the alloy during a melt-water interaction in an accident situation, we have investigated the amount of hydroxyl ion (OH^-) produced because (a) it should provide a reliable indicator of both the amount of hydrogen generated and the amount of lithium removed from the alloy; and (b) the analysis is very straightforward, accurate, fast and inexpensive.

We therefore quantitatively collected the alkaline aqueous solutions (pH $\cong$ 12 as indicated by the wide range pH test paper) that remained after each of the four alloy-water interactions and after one of the lead-water interactions (B-43-1) (neutral to the test paper) as a control. Each solution was both titrated with standard acid to determine OH^- concentration (moles/kg), and weighed to determine total amount of solution; the product of these quantities yields the total number of moles of OH^- generated. (See Appendix D for the original analytical reports.)

The results of these titrations are included in Table 1, both as millimoles of H_2 equivalent to the total number of millimoles of OH^- collected (i.e., 1 mole of OH^- = 1/2 mole H_2) and as millimoles of H_2 per gram of alloy used; we have also converted these values to the percentage of lithium removed from the alloy to produce that amount of H_2. It should be noted in Table 1 that the amounts of lithium removed as determined by titration appear to be about 20% greater than the analogous amounts determined by the gaseometric analyses of hydrogen.

Aerosols

An important qualitative observation is the acrid odor that was noticed roughly six minutes after each alloy-water interaction when the gases in the expansion vessel at the upper end of the column were vented to the room after the last gas sample was taken. (The odor was not noticed during venting after either the dry runs or the Pb-water experiments.) The acrid odor was similar to the "lithium odor" noticeable when small bits of metallic lithium react with liquid water in an unvented area. Although we did not study this odor production further, we believe it to have resulted from lithium-bearing (and possibly lead-bearing) aerosols being carried into the upper chamber by the gases generated in the alloy-water interaction as they bubble upward through the column of water. (Aerosols that contained both lithium and lead were collected above the site of water injections into molten eutectic alloy by Jeppson and Serinni (1989).)

There is a second observation consistent with the evolution of H_2 and its bubbling up through the column minutes after the water impacts the molten alloy: that the gas pressures in the expansion chamber above the column fluctuated considerably during the 5 minute-long active data acquisition period. (These pressure fluctuations were not observed during the same intervals after interactions of the water with the molten lead or the empty crucibles.) These fluctuations (or their absence) could be seen clearly on the numerical displays for the strain gauge presented by the Keithley digital acquisition system (see Appendix A). The strain gauge is located in the expansion chamber at the top of the chamber (see Figure 2). Although the Keithley system recycled through and displayed both strain gauge and thermocouple inputs once every 30 s, none of its outputs was saved beyond 3 s (cf. Figures 11d through 18d).

Debris

Overview

The debris recovered from both the lead-water and the eutectic alloy-water interactions was highly porous and spongy and in irregular chunks several centimeters across; moreover, this material crumbled easily, preventing any meaningful measurements of particle sizes. There were some debris fragments found in the vicinity of the burst disc and below when the apparatus was dissembled at the union that holds the diaphragm (see Figures 1, 2 and 3c, f and g). Finding debris fragments this far above the crucible (about 45 cm) indicates that a moderately vigorous melt-water explosive interaction had occurred during the contact. (In this same appartus, Farahani (1995) found fragmented debris well above the burst disc in several of his very energetic interactions between molten aluminum and water.)

The debris from the lead-water interactions did not adhere to the stainless steel crucibles. Thus it could be recovered quantitatively from both the crucible and from the column above. (For example, the crucible recovered from experiment B-43-1 was clean enough to permit its reuse in a later experiment.) A photograph of the debris recovered from the lead-water experiment B-35-1 is shown in Figure 21. Also included in this photograph are the ruptured diaphragm and a metal gasket recovered along with the debris.

The debris from the alloy-water experiments, although easily removed from the column, could not quantitatively be removed from the crucibles by simple probing with a bent stainless steel rod as had the lead-water debris. This was true whether the debris in the crucible was moist or had been dried on a hot plate at low temperature. We determined the weights of material that had adhered to the crucibles as shown in Table 2. (It should be noted that debris also adhered to the stainless steel crucibles in the eutectic Pb-Li alloy-water impact interactions reported by Kranert and Kottowski (1991).)

Table 2. Amounts of Debris that Adhered to the Stainless Steel Crucibles During Molten Eutectic Pb-Li Alloy-Water Interactions

Expt. No	Weight of Adhered Debris (g)	Water Temperature (°C)
B-39-1	20.12	27.4
B-45-1	44.90	61.0
B-50-1	49.11	27.4
B-52-1	24.52	59.6

The moist debris recovered after the alloy-water interactions was strongly alkaline, as indicated by the pH test paper. The alkali was easily removed from the spongy material with about a dozen repetitive rinsings with small amounts of distilled water. Most of the rinse water could be removed by decanting, but the last step in the rinsing procedure was to collect the debris in a cone of filter paper in a funnel, and rinse until the rinsings draining from the funnel were neutral, as indicated by the test paper. After the last rinsing had drained away and had been combined with the parent solution, the debris was allowed to dry overnight in air in the filter paper (the funnel and cone of filter paper were covered lightly with a paper towel).

Sometimes after about 15 hours of air drying, the filter paper cone was still moist. When a piece of test paper was pressed against the moist filter paper, it showed an alkaline response

Figure 21. Photograph of the debris recovered from the lead-water interaction experimenmt B-35-1. Also included are the ruptured diaphragm and a metal gasket recovered along with the debris. The diameter of the white circle is 24 cm. (B-35-1-1)

that had been absent during the filtration the day before. This suggests that there might have been a slow reaction of the porous debris with liquid water and/or air to produce still more lithium hydroxide after the amount initially produced in the melt-water interaction had been removed. This secondary production of alkali was investigated a bit further by placing a chunk of the debris from experiment B-39-1 that had been carefully rinsed and air-dried into distilled water. The next morning, however, no alkalinity was detected in the water by the test paper.

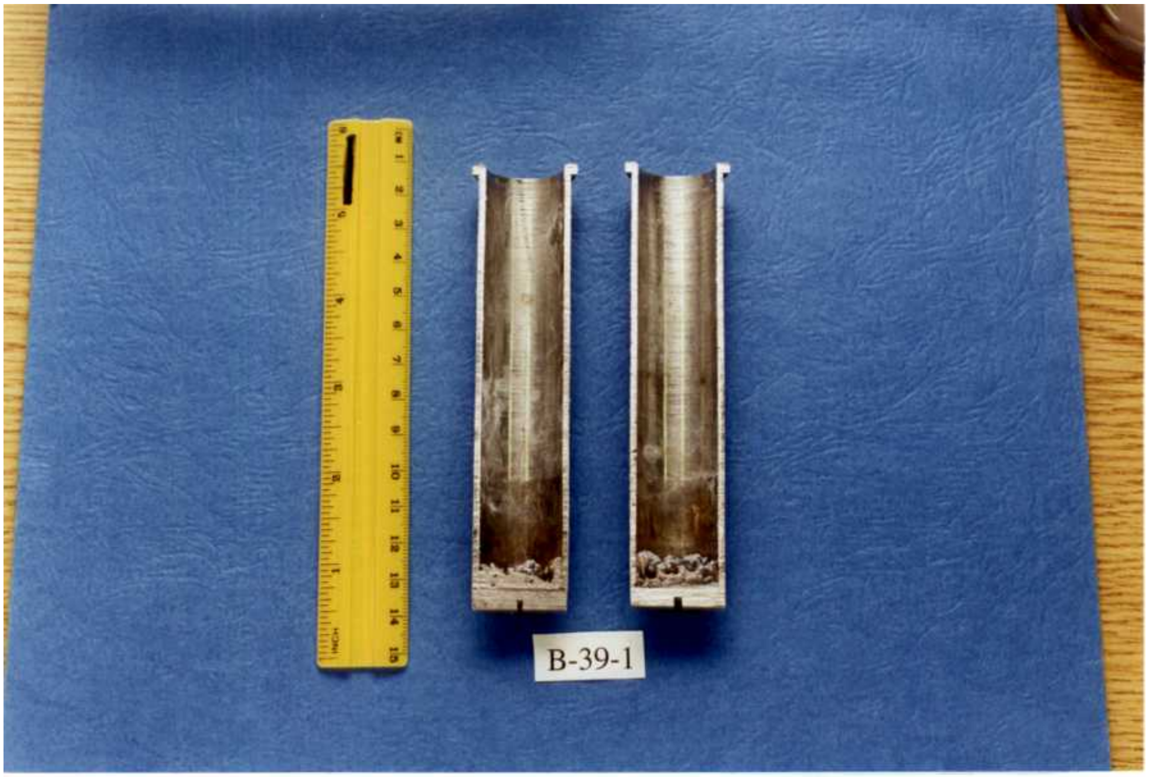
Another significant qualitative observation is the formation of brown stains on the filter papers that had been in contact with the moist alloy (as in the filter drying situation described in the previous paragraph). These were not major stains, but were nevertheless very noticeable in each of the alloy-water interactions. The presence of iron-bearing impurities in the solutions was considered as a simple explanation, but was rejected because only high purity metals were used to prepare the alloy (Nelson et al. 1995); moreover no signs of corrosion of iron-related materials such as stainless steel were noticed either during preparation or casting of the alloy or in the water column impact experiments described here.

Also, after the debris samples from the alloy-water experiments had been stored in contact with room air for several weeks, they developed a brown external coloration, almost a rust-like appearance. In comparison, the debris samples from the lead-water experiments, when stored similarly in air, developed a grey-white coloration.

We did not investigate the formation of either the brown stains or the brown or white colorations on the debris further.

Sectioning of the Crucibles

After several weeks of storage in air, the four crucibles from the molten alloy-water interactions were cut in half lengthwise to examine the debris that had adhered (see Table 2). The sectioning was done similarly to that reported by Kranert and Kottowski (1991). The cutting was done with a bandsaw without water or other coolant or lubricant. Photographs of both sections from each experiment are shown in Figures 22 through 25; the (a) photographs were taken essentially perpendicular to the axes of the crucibles while the (b) photographs were taken



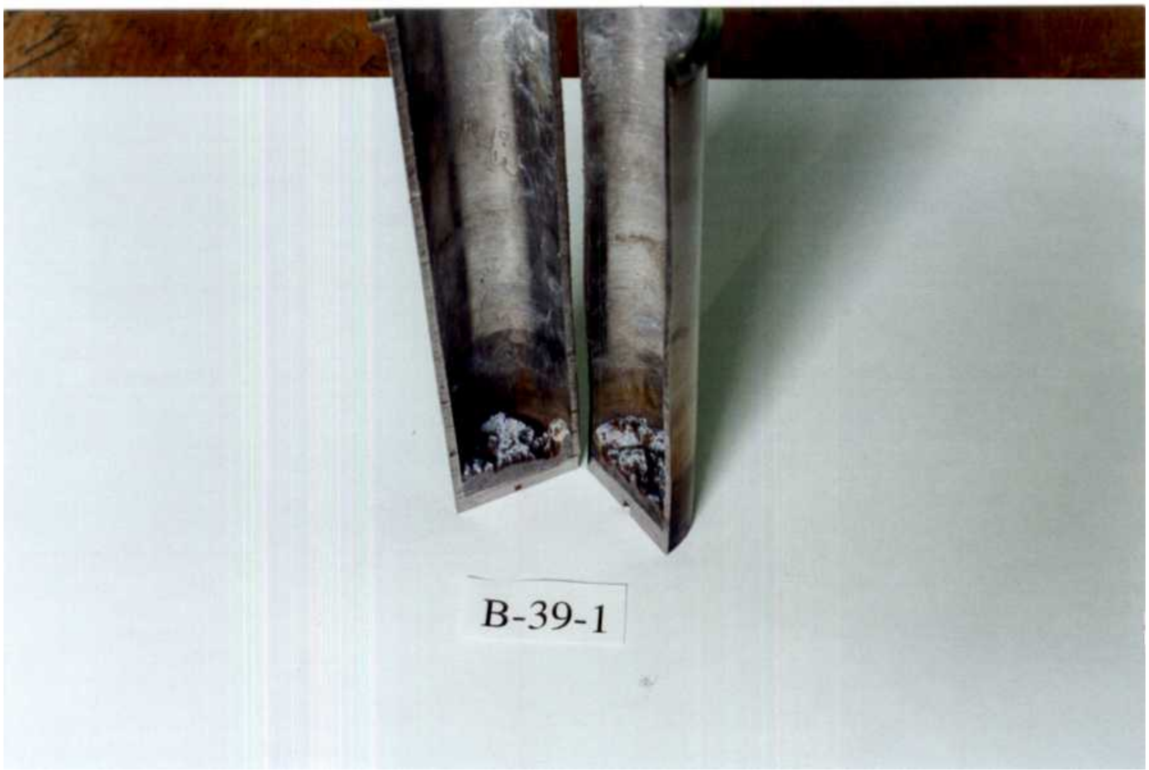


Figure 22. Photographs of the crucible with adhered Pb-Li debris from experiment B-39-1 after longitudinal sectioning: (a) (above) essentially perpendicular view (B-39-1); (b) (below) view from about 45˚ to show the upper surface of the adhered debris. (B-45-1-4). Notice in (b) that essentially no debris was freed by the sectioning.

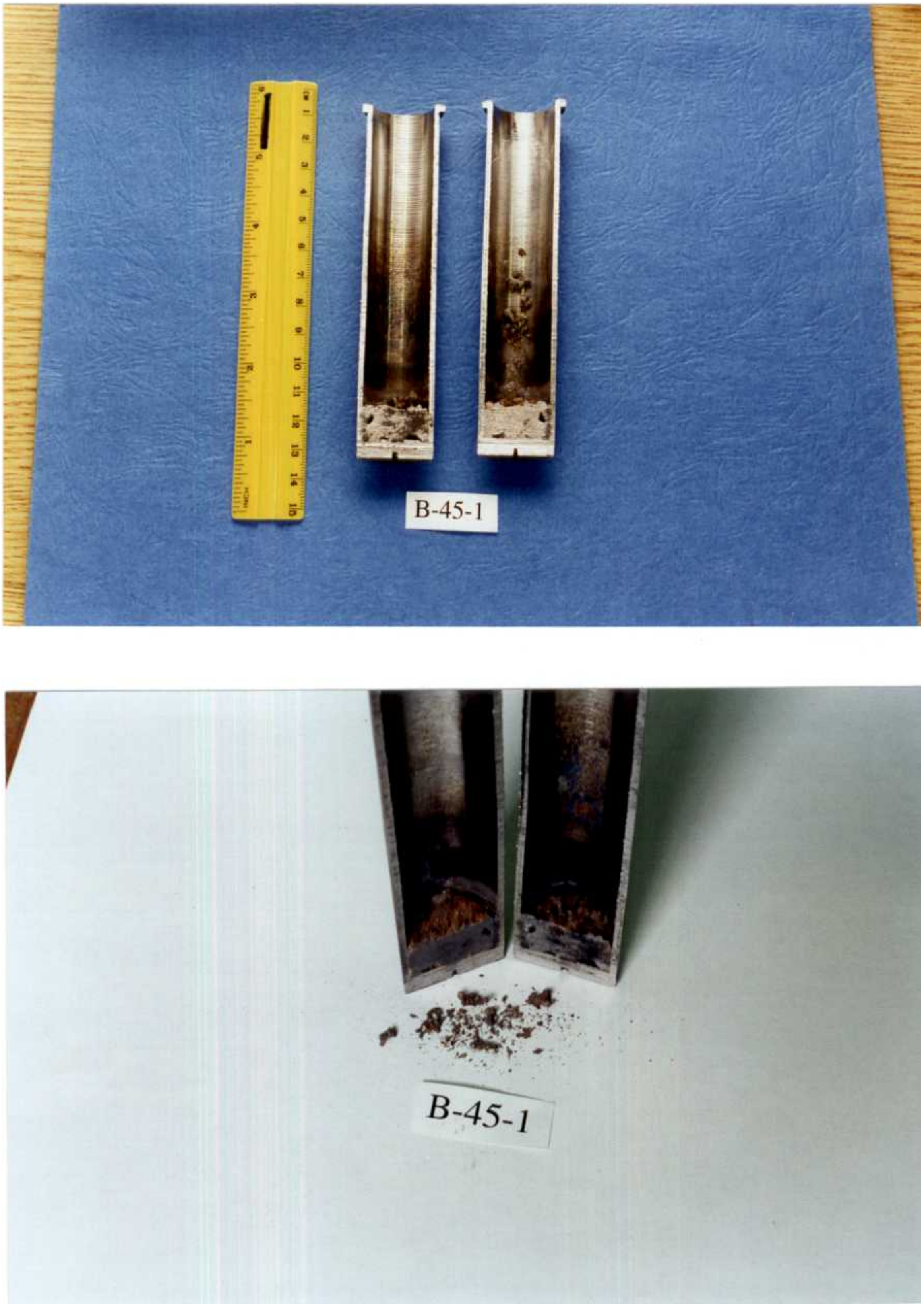


Figure 23. Photographs of the crucible with adhered Pb-Li debris from experiment B-45-1 after longitudinal sectioning: (a) (above) essentially perpendicular view (B-45-1-1);(below) view from about 45° to show the upper surface of the adhered debris (B-45-1-4). Notice in (b) the small amount of red-brown debris freed by the sectioning.

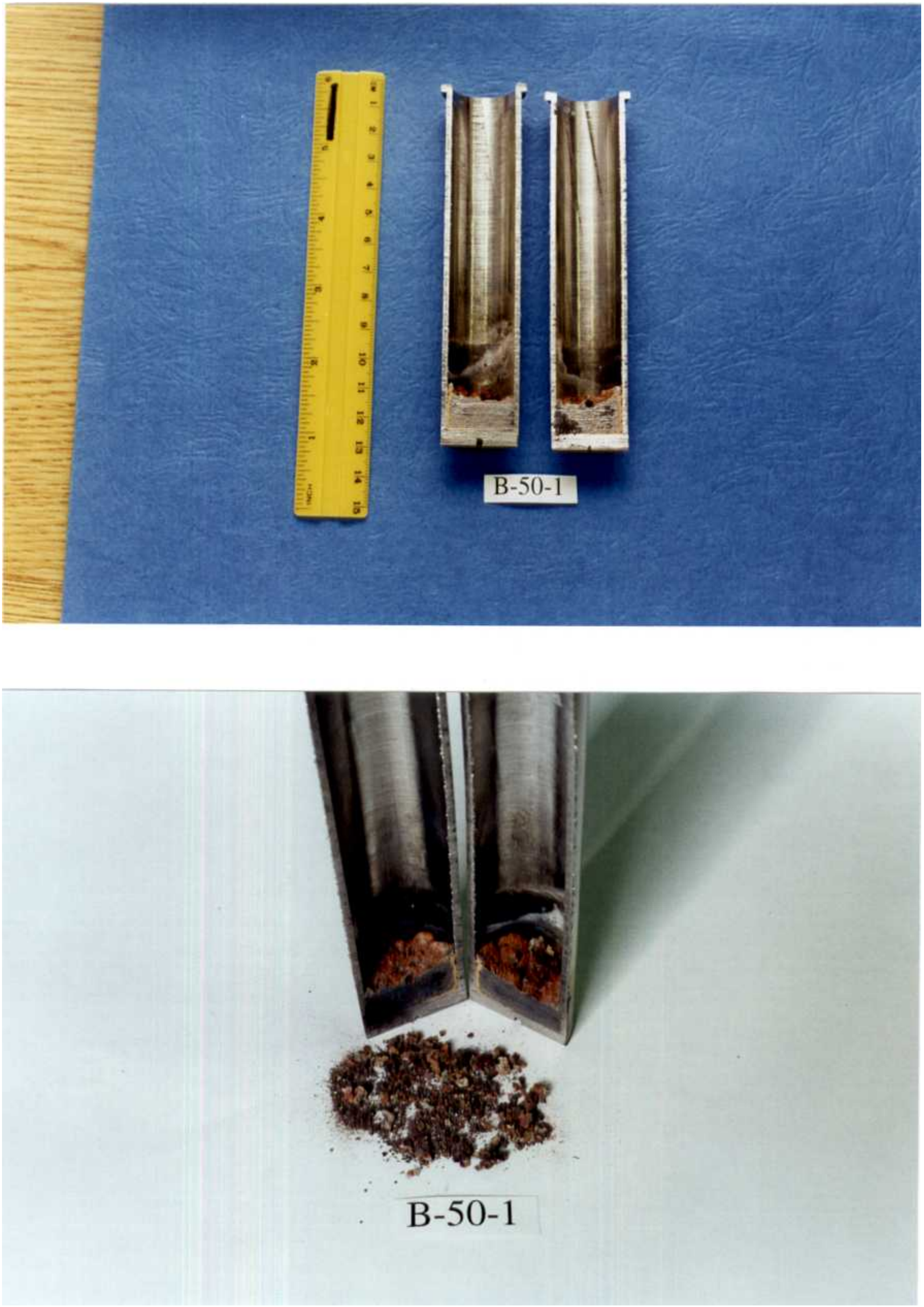


Figure 24. Photographs of the crucible with adhered Pb-Li debris from experiment B-50-1 after longitudinal sectioning: (a) (above) essentially perpendicular view (B-50-1-1); (b) (below) view from about 45˚ to show the upper surface of the adhered debris (B-50-1-5). Notice in (b) the large amount of red-brown debris freed by the sectioning.

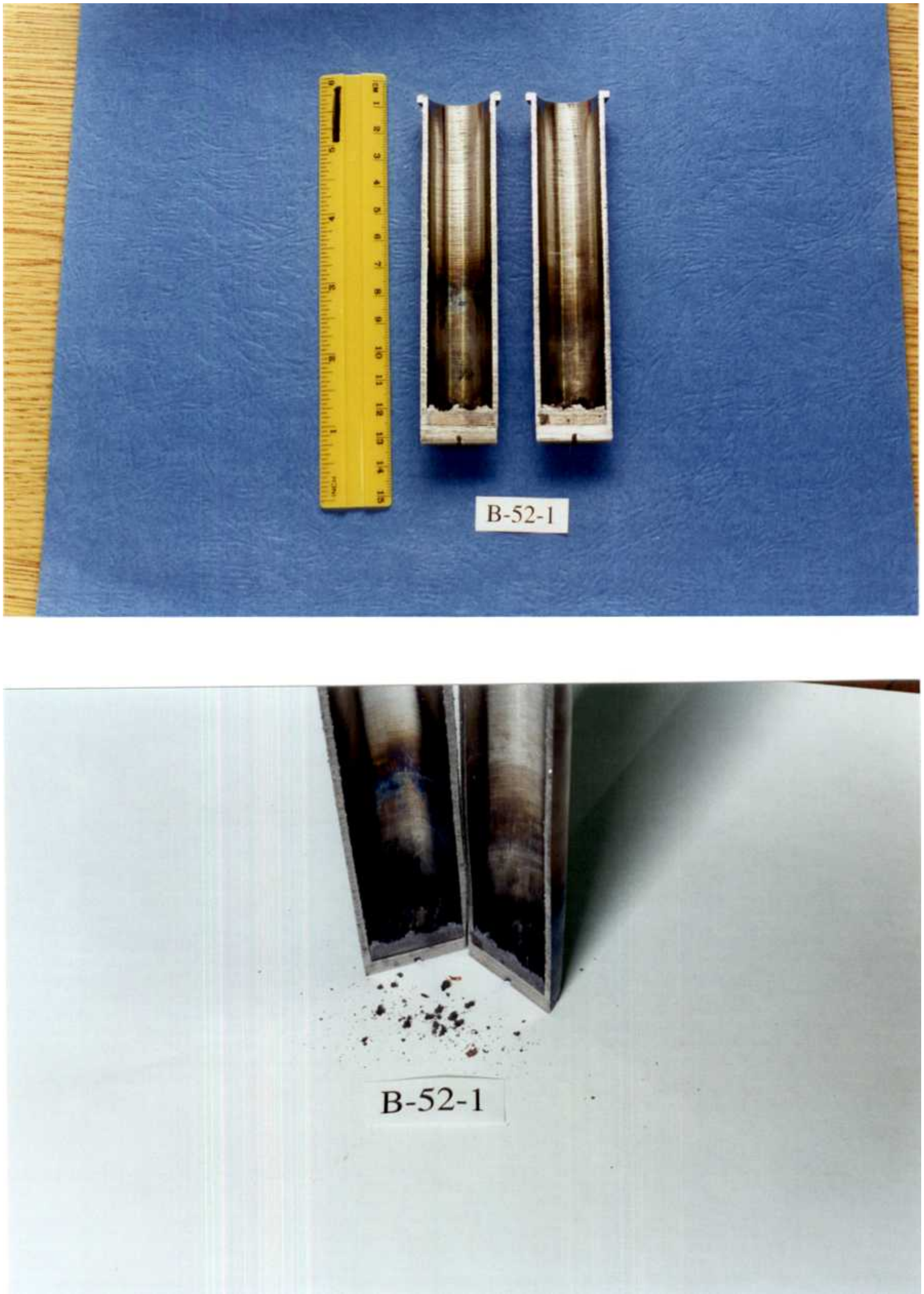


Figure 25. Photographs of the crucible with adhered Pb-Li debris from experiment B-52-1 after longitudinal sectioning: (a) (above) essentially perpendicular view (B-52-1-1); (b) (below) view from about 45° to show the upper surface of the adhered debris (B-52-1-4). Notice in (b) the small amount of debris freed by the sectioning.

at an angle of about 45° in order to view the upper surfaces of the adhered debris. In the experiments where a significant amount of granular debris was released by the sawing, it was collected and placed in a small pile at the base of the standing crucible halves. Particularly noteworthy is the unusually large amount of red-brown, almost crystalline debris freed during the sawing of the crucible from experiment B-50-1 (see Figure 24b); only minor amounts were freed from the other three crucibles.

DISCUSSION

Overview

The primary objective of this work has been to study the contact with water of nominally 120 g of the eutectic Pb-Li alloy when molten and at 600°C. In so doing, we have conducted four successful experiments where forceful contact between the molten alloy and the coolant was achieved by pressurizing the water column inside a 2.54 cm ID shocktube and impacting it upon the surface of the melt. Two experiments with empty crucibles (only air and argon at a low total pressure as specified for each experiment) and two experiments with molten lead (nominally 140 g at 600°C) have been performed to provide baseline information; these baseline experiments also can be compared with earlier analogous experiments of Vukovic (1994) and Farahani (1995), who have used the same apparatus as the one used for these eight experiments. A comparison of our experimental results for the Pb-Li alloy with those of Herzog (1987) and Biney (1995) also will be presented along with the chemical analyses later in this chapter.

Experiments with Empty Crucibles

Two baseline experiments were performed here, one with the crucible at room temperature (B-25-1) and the other with the crucible heated to 577°C (B-31-1). The crucibles were left empty in these experiments; that is, crucible volumes were occupied by a mixture of residual air and argon at low pressure prior to the coolant impact. The gas volume below the rupture disk is comprised of mostly argon. As explained earlier in the experimental section, the volume below the rupture disk is purged continuously with argon and evacuated at the same time. The pressure inside this volume is measured and recorded immediately before each experiment, after all valve closures prior to the initiation of disc rupture. This low pressure in the crucible is called the vacuum pressure (usually about 100 Pa) and is noted as P_{vac} on all traces (see Figures 11 through 18 and Appendix B).

Pressure Profiles; Empty Crucible Experiments

In Figures 11 and 12 and Appendix B we show the pressure traces for experiments B-25-1 and B-31-1. The shapes of the pressure traces are consistent with the work of Vukovic (1994) and Farahani (1995). The PT0 traces in Figures 11a and 12a show the multiple pressure spikes of decreasing magnitude in time, with shorter time intervals between consecutive spikes, which are the characteristics of the impact of the water upon the surface of an empty crucible. When the diaphragm ruptures, there will be some vaporization from the coolant front. The generated vapor will occupy the volume between the location of the rupture disk and the bottom of the crucible. The vapor volume will be pressurized as the coolant column moves downward.

For the crucible at room temperature this increase in the pressure is recorded by PT0 and can be seen in the expanded PT0 trace in Figure 11b for experiment B-25-1. As shown in this figure, the increase in the pressure upon impact does not occur as a completely sharp step because the coolant front does not have a flat or uniform shape due to the way the diaphragm ruptures and the presence of the water vapor immediately following the diaphragm rupture. Vukovic (1994) showed this trend when the impact was filmed at high speed using a transparent crucible. When the crucible is held at room temperature, this vapor may condense in whole or partially under the coolant column pressure. The effects of vapor condensation and the non-uniform water front cause pressure oscillations prior to the first main impact shown in Figure 11b.

When the crucible is hot, as in experiment B-31-1, the heat transfer from the crucible to the vapor volume will increase the vapor pressure and temperature of the water. Thus the vapor volume will condense at higher pressures because of this heat transfer. Not all of the vapor will condense because of the physical boundary which is present in hot crucible experiments. The pressure trace as measured by PT0 will have more and larger oscillations in hot crucible experiments (see the expanded PT0 trace for experiment B-31-1 in Figure 12b) than those recorded in experiments with the empty crucibles at room temperature (Figure 11b). These physical events may be the processes by which melt and coolant mixing could occur prior to the main coolant impact.

When the bulk of the water column impacts the surface of the crucible, the pressure increases due to the water hammer effect. After the first impact, the water column rebounds and moves upward toward its original location. But because of losses associated with the impact, namely friction and phase change, it does not completely reach its initial position. Hence the first pressure peak has to be the largest one in the absence of any appreciable energy source in the experiments with the empty crucibles at room temperature. Each pressure spike in the PT0 pressure trace shown in Figure 11a is indicative of a water impact. The time that it takes for the water column to move up and down once decreases as the distance that it travels up and down shortens. If the crucible is heated, however, this time period will not decrease uniformly for consecutive impacts because the presence of considerably more vapor will damp the water movement. As a result, in the hot crucible experiments the water does not impact the crucible as many times as it does in the experiments with the empty crucible at room temperature (compare Figures 11a and 12a for experiments B-25-1 and B-31-1).

The gas pressure inside the expansion vessel above the water column increases when the solenoid valve opens and the argon gas rushes in. The increase in the pressure inside the expansion vessel is recorded by transducer PT4 located in the top flange of the shock tube. As explained in the previous section, however, not all the piezoelectric transducer traces were recorded for all the experiments. In experiments where PT4 traces are missing, we can use the strain gauge transducer traces for the analyses. The strain gauge traces for the pressurized gas for experiments B-25-1 and B-31-1 are shown in Figures B-1g and B-2g in Appendix B. The data acquisition period for the strain gauge transducer was 30 seconds and continued until three seconds after the rupture disk failure for all experiments. The pressure value at the first maximum, as recorded by the strain gauge transducer, is the injection pressure and is noted as P_{inj} on all traces in Figures 11 through 18 and in Appendix B.

The pressure decreases shown by the strain gauge after the first maxima indicate that the diaphragm has ruptured and that the coolant is moving downward. The expansion of the pressurized gas continues until the water column is stopped by the impact upon the bottom of the crucible and starts moving up, toward the expansion vessel. The compression of the gas inside the expansion vessel upon the upward motion of the coolant column is a measure of the combined effects of the water hammer pressure and the energy transfer due to the melt-water interaction.

A comparison of the strain gauge traces for experiments B-25-1 and B-31-1 in Figures B-1g and B-2g in Appendix B shows the effects of heating the empty crucible on the work output. The heat transferred from the hot crucible to the coolant contributes to compressing the expansion gas. If an explosive melt-water interaction occurs, one could easily observe maximum pressures in second or later peaks higher than in the first peak (for example, see the pressure traces recorded when the crucible contains the molten eutectic Pb-Li alloy in experiment B-39-1 discussed later in this report and shown in Figures 15 through 18 and in Appendix B; also see Farahani (1995)). Note also that the pressure peaks measured at the top of the column by transducer PT4 or the strain gauge are not as sharp and large in magnitude as those measured by transducer PT0 because of the differences in the speed of the sound in gas and liquid phases. The existence of a number of surfaces, e.g., liquid-gas surface between the coolant and the expansion gas, contributes significantly to the reflection and transmission of the pressure disturbances. The pressure disturbances lose their magnitude and sharpness as they move from the liquid phase into the gas phase.

Crucible Temperatures; Empty Crucible Experiments

Although seven thermocouples were used in each experiment to measure the ambient, crucible, gas, and coolant temperature (at four different heights along the shock tube) we will only show and discuss the records of TC5, the crucible temperature traces. The readings of the other thermocouples were used mainly for controlling the experiments during heating and at other times when became necessary to check for leaks.

In the two baseline experiments performed with water impacts into empty crucibles, one at room temperature (B-25-1) and the other heated to 577°C (B-31-1), the records for the crucible thermocouple showed no change, and a rapid cooling by about 9°C, respectively. The traces for these experiments are shown in Figures 11d and 12d and again in Appendix B. These temperature traces were recorded and plotted starting after the pressurization sequencing began and ending three seconds after the diaphragm rupture. (Note that the total temperature recording period can be somewhat different for each of the experiments. The diaphragm rupture time is noted on the temperature plots for all the experiments, however.)

Hydrogen Measurement; Empty Crucible Experiments

No hydrogen was found in either of the two sample bottles A and B that were filled with the expansion gas one and five minutes after the coolant impacted the surface of either empty crucibles in experiments B-25-1 or B-31-1.

Experiments with Molten Lead

In experiments B-35-1 and B-43-1, the water column impacted 140.18 g and 146.08 g of molten lead at 590.2°C and 587.2°C, respectively. The water was at 29.1°C in the first experiment and at 61.1°C in the second.

Pressure Profiles; Molten Lead Experiments

Figures 13a and 14a show the pressure transducer PT0 traces for experiments B-35-1 and B-43-1. These pressure traces differ from those recorded for the empty crucible experiments. The first pressure peaks in both PT0 traces are no longer the highest peaks; now the maximum pressures occur on the second water impact in both figures. Also, the time interval between the consecutive pressure spikes does not decrease as it did in the experiment with the empty crucible at room temperature. We attribute these stronger second peaks to the heat transfer from the molten lead that generates water vapor at high pressures, which in turn helps to move the water column further upward upon its expansion. On its downward return path toward the crucible, the water column possesses more energy and hence, will impact the crucible with a higher velocity resulting in a larger water hammer pressure. Also, the molten lead may fragment further, beyond that from the first melt-coolant impact because of the tremendous pressure pulse generated in the second impact. The additional heat transferred on the second impact probably explains the larger third peak observed in experiment B-43-1 and shown in Figure 14a.

The expanded PT0 traces for experiments B-35-1 and B-43-1 are shown in Figures 13b and 14b. Although the traces in these figures look similar, they have some distinguishable differences; the traces before the main impacts and the shape of the pressure traces for the main impacts need to be explained. The expanded PT0 pressure trace for experiment B-35-1 in Figure 13b shows a short interval of pressure oscillations at 128 to 130 ms prior to the first main impact at about 139 ms. The expanded pressure trace for experiment B-43-1 in Figure 14b shows a more complex set of pressure oscillations that starts at about 129 ms and does not end until the first main impact at about 133 ms.

We speculate that the absence of any significant disturbance of the molten lead by small water drops and water vapor prior to the main impact resulted in the relatively simple pressure trace shown in Figure 13b. In line with the above explanation, we assume that in experiment B-43-1, the water column front was not as flat as it was in experiment B-35-1. Hence, the shape of the coolant front could explain the differences between the pressure oscillations before the main impacts in these experiments. Actually, the expanded pressure trace of the first main water impact in experiment B-35-1 shown in Figure 13b is very similar to the trace of the first impact in experiment B-31-1 shown in Figure 12b. But in experiment B-31-1, the water impacted the surface of an empty crucible at 576.7°C, whereasin experiment B-35-1, the water impacted molten lead at 590.2°C. The similarity between the expanded pressure traces of the main water impact in experiments B-31-1 and B-35-1 seems to suggest very limited fragmentation of the lead during the first water impact in experiment B-35-1 and subsequently less energy transfer between the molten lead and water.

Crucible Temperatures; Molten Lead Experiments

The temperature of the molten lead decreased when impacted by the coolant in both experiments B-35-1 and B-43-1 as shown in the traces for the crucible thermocouple, TC5, in Figures 13d and 14d, respectively. The temperature of the crucibles that contained molten lead dropped about 6°C in experiment B-35-1 and 3°C in experiment B-43-1 within the 3 second recording time after the initial water impact. When room temperature water impacted a heated empty crucible, the crucible temperature decreased about 9°C in experiment B-31-1 within the same 3 second recording interval (see Figure 12d). We attribute this to the fact that the heat content of the empty crucible in experiment B-31-1 is lower than the combined heat content of the lead and the crucibles in experiments B-35-1 and B-43-1, causing a larger terperature drop in experiment B-31-1 following the coolant impact. The difference between the crucible temperature drops in experiments B-35-1 and B-43-1 may be caused by the higher coolant temperature in experiment B-43-1 (61°C) than in experiment B-35-1 (28°C). This will result in a smaller crucible temperature drop because of the lower heat transfer rate due to smaller temperature gradients between the hot melt and the coolant.

Hydrogen Measurement; Molten Lead Experiments

As in the empty crucible experiments, no hydrogen was detected in the bottles that were filled at either 1 or 5 minutes with the expansion gas from the experiments B-35-1 and B-43-1.

Experiments with the Molten Pb-Li Eutectic Alloy

Four experiments were conducted where 120 g of the Pb-Li eutectic alloy at 600°C interacted with water at 25 or 60°C (weights and temperatures are nominal). The interactions between the alloy and water were characteristically different than those involving empty crucibles or molten lead. The presence of extensive melt-water chemical reaction significantly increased the available heat transferred to the coolant. Moreover, the resulting hydrogen gas from the chemical reaction between the alloy and water provided the reaction vessel with a cushion that damped the impact pressure produced by the returning water column.

Pressure Profiles: Molten Pb-Li Alloy Experiments

Figures 15a through 18a show the PT0 pressure profiles for experiments B-39-1, B-45-1, B-50-1, and B-52-1, respectively. The PT0 pressure profiles for each experiment with the alloy show only one major pressurization followed by two or three low, broad pressurizations, compared to the multiple sharp pressure spikes produced in the molten lead and empty crucible experiments (see Figure 11a through 14a). Also, the expanded PT0 pressure profiles for the initial impact, shown in Figures 15b through 18b indicate that the events leading up to the initial water impact with the molten alloy differ from those in the molten lead experiments. In the experiments with the alloy, the pressure oscillations prior to the main impact are of the same magnitude as the maximum pressure recorded during the first impact, while the magnitude of the peaks of the pressure oscillations as recorded by PT0 prior to the main impact for both the molten lead and the empty crucible experiments were noticeably smaller than the pressures recorded during the main impact.

The presence of chemical reaction between the molten alloy and water seems to explain the differences between the pressure profiles recorded during the alloy experiments and the lead experiments. As explained earlier, upon the diaphragm rupture, the volume below the disk quickly will be occupied by water vapor which will be forced into intimate contact with the surface of the molten alloy beneath the moving water column. The exothermic chemical reaction between the alloy and the water vapor will generate both heat and hydrogen gas. The addition of hydrogen gas and more heat to the already pressurized water vapor will cause a rapid increase in the pressure within the reaction volume which will be measured best by the closest pressure transducer, PT0. The water column will be slowed momentarily by the expansion of the water vapor and the generated hydrogen gas. Eventually the bulk of the coolant will impact the molten alloy. After the impact, the water column moves upward under the force of the water hammer pressure and the expanding hydrogen gas. Upon the return of the water column to the crucible, the generated hydrogen gas provides a cushion and slows the coolant column; hence, the second impact generates a very much smaller and broader water hammer pressure.

The pressure profiles measured in the expansion vessel by PT4 for the molten alloy-water experiments B-39-1, B-45-1, B-50-1, and B-52-1 are shown in Figures 15c through 18c, respectively. These traces show a characteristic W-form that consists of three major peaks corresponding to the pressure at the time the diaphragm ruptures, the maximum upward motion of the water column and a broad late pressurization; these pressurizations are separated by minima that correspond to the first and second impacts of the water column on the crucible and its contents. The "W's" are "tilted" differently depending on the height of the third peak, which varies depending on the nature of the interaction and the generation of gaseous products. The temperature of the water in the column at the time of interaction also seems very important.

As shown in Figure 14c for experiment B-43-1, a lead-hot water interaction, the "W" is tilted only slightly toward the right; that is, the three maxima are not too different. But in Figures 15c and 17c, for alloy-cold water experiments B-39-1 and B-50-1, the "W's" are tilted strongly to the right; that is, the third maxima are very small. And in Figures 16c and 18c for alloy-hot water experiments B-45-1 and B-52-1, the tilt is strongly to the left, due to the large third maxima in both experiments.

The "W's" tilt because travelling pressure disturbances are affected significantly by the contact surfaces and the media through which they pass. Travelling from a liquid medium, e.g., room temperature water, to a gaseous medium, e.g., argon, a pressure disturbance will lose its sharpness and magnitude because of the lower density of the gas and the fact that sound travels slower in gases than in liquids. Thus the expansion gas in experiments B-45-1 and B-52-1, because of the higher initial coolant temperature, has more water vapor than the expansion gas in experiments B-39-1, and B-50-1. The presence of a higher pressure of water vapor causes the unusually high third peaks in the PT4 pressure profiles for experiments B-45-1 and B-52-1. Also, the expansion gas will have an increasing capacity for holding water vapor, proportional to the temperature of the expansion gas. This is reflected in the equilibrium expansion gas temperatures of 35.8°C and 33.0°C for the two molten alloy-cold water experiments B-39-1 and B-50-1, and 53.4°C and 54.2°C for the two molten alloy-hot water experiments B-45-1 and B-

52-1, respectively. These temperatures were determined from the outputs of thermocouple TC4 in the expansion vessel (see Figure 2) displayed by the Keithley data acquisition system.

Crucible Temperatures; Molten Pb-Li Alloy Experiments

The crucible temperature profiles recorded with thermocouple TC5 for experiments B-39-1, B-45-1, B-50-1, and B-52-1 are shown in Figures 15d through 18d, respectively. Except for experiment B-39-1 where the crucible temperature decreased only 2.4°C within three seconds after the rupture disk failure, the crucible temperature remained constant for this same time interval for the other three alloy experiments. This essentially negligible cooling should be compared to the temperature drops of 9°C for the heated empty crucible experiment (B-31-1) and the 6°C and 3°C drops for the molten lead-water experiments (B-35-1 and B-43-1) all measured over the identical 3 second interval after disc rupture. The fact that the alloy reacted chemically with water according to Equations (6) and (7) explains the crucible temperature profiles for alloy experiments: the additional heat from the exothermic chemical reaction between the alloy and water compensated for the cooling effects of the water column.

Hydrogen Measurements; Molten Pb-Li Alloy Experiments

The results of the mass spectrometer analyses of hydrogen in the four molten alloy-water impact experiments are shown in Tables 1 and 3. The procedures for analyzing the peak heights of the mass spectra of the sort shown in Figures 19 and 20 are summarized in Appendix C. Discussion of these analyses will be presented in the next sections.

Comparisons Between Our Gaseometric and Titrimetric Analyses

Tables 1 and 3 indicate the significant differences between the extent of molten eutectic Pb-Li alloy-water chemical reaction determined by our two analytical techniques—quadrupole mass spectrometry and acid-base titration. These measurements are expressed in the tables as both the number of millimoles of gaseous hydrogen generated and the percentages of lithium originally present in the alloy that were removed during the interactions; these quantities have been interconverted using equations (6) and (7). Three of the four titration results indicate 25% to 30% greater reaction than the gas analyses, while the fourth, epxeriment B-50-1, shows only

Table 3. Comparisons of Hydrogen Generated and Lithium Removed During Three Different Sets of Experiments in Which Molten 83a/o Pb-17a/o Li at 600°C Interacted with Liquid Water

Experiment No.		T_{H_2O} (°C)	Wt. of Alloy (g)	No. mmoles of Li	H_2 Generated (mmole)			H_2 Generated/Wt. Alloy (mmole/g)			Lithium Removed (%)		
					Δ p	ms	OH^-	Δ p	ms	OH^-	Δ p	ms	OH^-
Biney	L11	60	44.0	44.37	8.05	----	----	0.183	----	----	36.3	----	----
(1995)	L12	60	38.0	38.32	10.7	----	----	0.282	----	----	55.8	----	----
	L14	60	49.0	49.42	12.6	----	----	0.257	----	----	51.0	----	----
	L21	60	41.0	41.35	8.76	----	----	0.214	----	----	42.4	----	----
	L22	60	35.0	35.30	11.1	----	----	0.317	----	----	62.9	----	----
					10.24			0.251			49.7		
					±1.837			±±0.053			±10.6		
Herzog	16	60	47.0	47.40	7.01	----	----	0.149	----	----	29.58	----	----
(1987)	17	60	56.3	56.78	6.48	----	----	0.115	----	----	22.82	----	----
	20	90	39.8	40.14	7.47	----	----	0.188	----	----	37.22	----	----
	21	70	67.9	68.48	6.97	----	----	0.103	----	----	20.36	----	----
	22	80	51.3	51.74	4.84	----	----	0.094	----	----	18.71	----	----
	29	90	25.9	26.12	6.94	----	----	0.268	----	----	53.14	----	----
	30	70	19.0	19.16	6.12	----	----	0.322	----	----	63.88	----	----
	42	60	35.5	35.80	8.68	----	----	0.245	----	----	48.49	----	----
	43	60	32.8	33.08	6.40	----	----	0.195	----	----	38.69	----	----
					6.768			0.187			36.988		
					±1.039			±0.079			±15.754		
This Work	B-39-1	27.9	119.57	120.6	----	33.7	45.3	----	0.282	0.379	----	55.9	75.0
	B-50-1	27.4	116.59	117.6	----	42.0	42.9	----	0.360	0.368	----	71.5	73.0
	B-45-1	61.0	115.91	116.9	----	29.3	37.6	----	0.253	0.324	----	50.1	64.4
	B-52-1	59.6	122.42	123.5	----	34.4	45.5	----	0.281	0.372	----	55.8	73.7
						34.85	42.83		0.294	0.361		58.33	71.45
						±5.27	±3.68		±0.046	±0.025		±9.19	±4.85

Experimental Techniques: Δ p = pressure measurements; ms = quadrupole mass spectrometry; and OH^- = acid-base titration of hydroxyl ion.

about 2% greater reaction. Because both methods of analysis are believed to be reasonably reliable, it is necessary to consider possible sources of the apparent discrepancies.

It should be noted that the two analytical procedures were performed on samples taken at very different times after the interactions, as indicated in Table 4: the gaseous samples were taken within a few minutes of the interaction while the aqueous solutions were in contact with the alloy for many hours during which the melt had solidified andcooled to room temperature and equilibrated with the water both chemically and thermally.

Table 4. Time Intervals Between the Molten Pb/Li-Water Interactions and the Analytical Sampling

Expt. No.	H_2 Sampling(min)	Removal of the Solutions from the Column (hr)	Rinsing of the Debris (days)*
B-39-1	1, 5	21	10
B-45-1	1,5	~20	18
B-50-1	1,5	~20	15
B-52-1	1,5	20	0.8

* The dried deis was exposed to the ambient room atmosphere during this period.

A likely explanation for these discrepancies is that the alloy-water chemical reaction continued well after the gases were sampled at one and five minutes. Several lines of reasoning seem to support this idea:

- First, the interaction between the molten eutectic alloy and water is known to be exothermic, as observed by Jeppson and Muhlestein (1985) and by Crocker as cited by Coen (1985). In their experiments, steam at 335°C was sparged into 200 kg of the melt at 500°C under adiabatic conditions. Within 4.5 minutes, the melt temperature had risen 370°C and the interaction caused vigorous shaking of the apparatus. Although we used much less melt, our apparatus is considerably smaller and probably not as well insulated and our water was liquid, thus it is possible that the temperature in our crucible and the lower end of the interaction chamber remained quite high (perhaps even increased), over time intervals at least as long as the 4.5 minutes reported in the references just cited.

- The exothermicity of the Pb-Li melt-water reaction is consistent with the essentially constant temperatures of the crucibles that contained the alloy after the interactions compared to the distinct coolings observed with the empty crucible and in the Pb-water interactions shown in the outputs of thermocouple TC5 (see Figures 12d through 18d). Because we stopped recording both thermocouple and pressure transducer data 3 s after the interactions, however, we have no useful information about the temperatures of the melt 1 and 5 minutes later when the gas samples were taken. Another observation that supports the continued reaction between melt and water is that the analyses of each bottle B, the second sample of gas taken 5 minutes after the interaction, always showed 10 to 15% more hydrogen than the corresponding bottle A sampled 1 minute after the interaction.
- Still another observation that might be related to the possible continuation of the chemical reaction and the retention of high alloy temperatures for times of five minutes or more is the detection of an acrid "lithium odor" when the upper chamber of the apparatus was vented to the atmosphere of the laboratory about 6 minutes after the interaction. This odor is thought to be the result of fine mists being formed as the hydrogen gas bubbled upward through the alkaline solution-filled column after the pressure of the driving gas was released from the expansion chamber. The vigorous nature of the bubbling and the stirring it must produce can be visualized by comparing the volumes of hydrogen that were released at the bottom of the column with the total amount of liquid water in the column. As shown in Table 1, 30-40 mmoles of hydrogen were released, according to the mass spectrometer analyses. This corresponds to 0.7 to 0.9 L of gas at room temperature and atmospheric pressure or perhaps 2 or 3 L at atmospheric pressure and 600°C. The volume of water in the column is about 10 L, estimated for a 2 m-tall column

with a 2.54 cm inner diameter, and not including the volume of the upper chamber. The active ebullition in the column and the upper chamber is not difficult to visualize, particularly if the alloy is still hot at the bottom of the column.

Also consistent with the vigorous bubbling up of H_2 through the column minutes after the water impacted the molten alloy are the fluctuating gas pressures observed with the strain gauge in the expansion chamber above the column (see Results section). These pressure fluctuations were not observed during the same intervals after the water had impacted the molten lead or the empty crucibles.

Another process that may add significant amounts of OH^- to the solutions is the possible continuation of the alloy-water reaction, probably at a much slower rate, after the melt had solidified as fragmented, porous debris and after the system had cooled and essentially equilibrated. Although the temperatures were low, the contact times were long — about 20 hours as indicated in Table 4. Thus, if there is a slow reaction between the alloy and water at room temperature, according to equations (6) and (7), lithium might be continuously extracted from the solid debris with the gradual evolution of hydrogen that would diffuse away unnoticed. Because of the excess of water in the column and in the rinsing operations any Li_2O produced would be converted to LiOH.

There is also the possibility that some OH^- could have been added to the solutions via rinsing of the solidified debris after it had been exposed to ambient air for even longer contact times up to 18 days as indicated in Table 4. The observations that (a) moist filter paper in contact with moist debris changes from neutral to alkaline overnight, and (b) that "dry" debris somehow produces a reddish coating after several weeks of exposure to air, suggest that some slow reactions may occur between the porous solid alloy and the air (probably only with oxygen and water vapor, not with the nitrogen; see Hubberstey and Sample (1993)).

Our four experiments with the alloy do not shed much light on how the long term reactions of the porous alloy with liquid water proceed because the contact times before the aqueous solutions were drained from the column and the debris were about the same and never less than 20 hours for each experiment (see Table 4). We did have some very different times for exposure of the debris to air before rinsing out the LiOH in preparation for titration, namely, experiment B-45-1 which was allowed to dry and was exposed to air for 18 days prior to rinsing and experiment B-52-1, which was rinsed immediately after it was removed from the column. There does not seem to be a major difference shown in Table 1 between the short and the long exposures of the debris to air shown in Table 4, however.

In the preceding paragraphs, explanations have been given for why the amounts of OH^- have increased above those amounts of hydrogen produced in the first minutes after the interactions, thus producing the discrepancies noted in Table 1. However, we cannot rule out another possibility, namely, that some hydrogen could have been removed before the analyses in these first minutes and thus also contributes to the discrepanices. It is possible that, at least early in the interaction, the hydrogen may have been hot, perhaps at temperatures that approached or even exceeded the 600°C melt temperature, as it bubbled through the water surface in the expansion chamber. Because there is some residual air gas mixture (i.e., Ar+air) in the upper chamber, some of the hot hydrogen might have ignited spontaneously and burned. (The spontaneous ignition temperature of hydrogen in air at atmospheric pressure is about 500°C (Conti and Hertzberg, 1988); it should be even lower at higher pressures. The spontaneous ignition and combustion of bubbles of hot hydrogen gas as they emerge from water into an air atmosphere have been reported by Nelson et al., (1992).) The combustion of a fraction of the hydrogen would thus tend to reduce to an amount of hydrogen estimated by mass spectrometry.

Initially, there was about 1.1 liters of air at local atmospheric pressure and room temperature in the expansion chamber prior to the pressurization with argon that ruptures the diaphragm. If the combustion had gone to completion, the amount of hydrogen removed would correspond to twice the amount of oxygen contained in 1.1 liters of air, which is 0.22 liters, or 10 millimoles. Thus as much as 20 millimoles of hydrogen could have been removed by combustion. It may be only a coincidence in Table 1 that the amounts of hydrogen generated as estimated by gas analyses are 10 to 15 millimoles lower than those estimated by titration. (The removal of gaseous hydrogen by combustion, of course, would not be expected to affect the titration results.)

Comparisons with Earlier Research

Our work should be compared with two sets of closely related molten eutectic Pb-Li alloy-water interactions reported earlier; those of Herzog (1987) (see also Herzog and Corradini (1989)) and those of Biney (1995). In both sets of experiments, the molten alloy was flooded relatively gently with water and the gaseous hydrogen was measured by changes in the system pressure (Δp). In Table 3, we have collected the results obtained with the alloy at 600°C by these authors; our results with 600°C melt obtained by mass spectrometry (ms) and titration (OH^-) are also included in the table for comparison. (The symbols in parentheses above in this paragraph are used in Table 3 to indicate the analytical techniques used to estimate the extent of melt-water reaction.)

In the work reported by Herzog, masses of the eutectic alloy between 20 g and 65 g were melted in a flowing argon atmosphere in a cylindrical cavity 2.54 cm in diameter and 3.18 cm deep. The melt was then flooded with about 1 L of water. Melt temperatures were between 350°C and 600°C, while water temperatures ranged from 60°C to 90°C. The initial pressure of argon in the chamber was nominally atmospheric. Hydrogen generation was measured by changes of pressure in the chamber, which was made gas-tight by appropriate valve closures just before the interaction. The measurements of pressure were recorded at 200 s (3.3 min), after the melt and water were considered to have equilibrated chemically and thermally.

Biney placed 37 g to 56 g samples of the eutectic alloy at the bottom of a cylindrical cavity 2.54 cm in diameter and 17.78 cm deep in a cylindrical stainless steel block. Melt temperatures were in the range 350 to 650°C, very similar to Herzog's. Hydrogen was measured by two procedures: pressure changes and thermodynamic calculations based on thermocouple responses. The main difference between Herzog's and Biney's apparatus seems to be that Biney placed his melt at the bottom of a cavity much deeper (17.78 cm-deep), than Herzog's, who used a cavity that is only 3.18 cm deep; the diameters of both cavities are identical, 2.54 cm. (Our cavity (the crucible) is 2.0 cm in diameter and 11.94 cm deep; see Figure 4.)

Extent of the Chemical Reaction

Using Table 3, it is possible to compare the extent of the molten eutectic alloy-water chemical reaction that occurred in the three sets of experiments as estimated by the three different analytical procedures — pressure changes (Δp), quadrupole mass spectrometry (ms) and titration (OH^-). The measurements by the three techniques are assumed to be completely interconnectable by means of equations (6) and (7).

(Note: In the preparation of Table 3, we have included data only for the alloy temperature common to all three sets of experiments, namely 600°C. Moreover, we have not tried to include the effects of water temperature because Herzog showed that, for molten alloy at 600°C, changing the temperature of the water over the range 60°C to 90°C did not seem to affect the amount of hydrogen generated. Moreover, we have assumed that this insensitivity applies to the entire range of water temperatures studied, nominally 20°C to 95°C.)

In Table 3 the total amounts of reaction (i.e., H_2 generated) are lowest on the average for Herzog's experiments, about twice as large for Biney's work, and about five (ms) and six (OH^-) times larger for our work. Herzog's and Biney's work should be directly comparable because of the similarity of the weights of molten alloy studied, the apparatus used, and the relatively gentle modes of flooding. Our work might be expected to differ because we used two to four times more melt and a significantly more vigorous mode of melt-water contact.

If we look at the extent of the chemical reaction per unit weight of alloy (H_2 Generated/Weight of Alloy), however, the extents of reaction are closer to each other. Again, on the average, Herzog's values are lowest, while both Biney's and ours (ms) are about 60% greater. (Our titration-based values (OH^-) are about 90% greater, however.) Note the relatively good agreement between both sets of recent gaseometric values: our mass spectrometer analyses and Biney's measurements.

Because they are calculated from essentially the same data, the percentages of lithium removed during the molten alloy-water interactions shown in Table 3 have the same relationships as the amounts of hydrogen generated per weight of alloy: Herzog's are lowest and the two recent gaseometric values — Biney's and our mass spectrometer data — are about 60% greater and show good agreement, while our titration data are about 90% greater. (Note: The recent gaseometric values of about 0.3 mmoles of H_2 per gram of alloy also show good agreement with the value of 0.25 mmole/g determined in a larger scale experiment by Jeppson et al., (1983).)

The amounts of lithium removed are large: on the average 37% in Herzog's work, 60% in the recent gaseometric measurements (Biney's and ours) and about 72% in our titrations. (Herzog, however, did achieve 64% removal of lithium in one experiment, no. 30, of the same order of magnitude as the average of the recent gaseometric values; see Table 3.)

It should be pointed out that as lithium is removed from the alloy by the metal-water reactions the liquidus temperature of the alloy will increase (Hansen and Anderko, 1958). Ultimately, if all the lithium is removed, the melting temperature of pure lead, 327°C, will be reached, as was achieved in prolonged sparging of the molten alloy with steam by Jeppson and Muhlestein (1985). Increases of as much as 100°C in the liquidus temperature of the alloy by removal of lithium might be a significant factor in postaccident cleanup in large fusion installations.

Herzog introduced the concept of calculating the amount of hydrogen generated per unit area of melt exposed to the water during the interaction. He used the area of the opening into his crucible, a 2.54 cm hole, for these calculations. For example, when this melt was at 600°C, the average amount of hydrogen generated per unit area of melt in 200 s was 13.4± 0.61 moles/m^2. The corresponding average value for Biney's interactions with 600°C alloy is 24.2 moles/m^2, using the same orifice area as Herzog's. Using the 2 cm inside diameter of our crucible (see Figure 4), the mass spectrometer data given in Tables 1 and 3 indicate that our averaged hydrogen generations over 300 s were about 112 moles/m^2 (assumes 25°C and 60°C water temperatures are equivalent). If we use the amounts of hydrogen in Tables 1 and 3 that correspond to the amounts of OH^- generated, this quantity increases to about 136 moles/m^2. It should be recognized, however, that the areas used here are those of the orifices of the three reaction chambers. The significance of this parameter becomes uncertain, however, when extensive fragmentation and porosity is induced in the alloy by the interaction. It may apply best to Herzog's situation where flooding was intentionally gentle to minimize melt stirring and breakup. In our water impact experiments it is probably of limited value because we intentionally caused the melt to break up during the interaction. Biney's situation is probably intermediate because his flooding was somewhat more disruptive of the melt than Herzog's but much less than ours.

Our "Maverick" Experiments

In Tables 1 and 3, it can be seen that, in both sets of our four analyses of extent of chemical reaction (i.e., hydrogen generation) determined by mass spectrometry or by titration, there are three analytical results close together and a fourth some distance away. Unfortunately, the value that is "off" is not for the same experiment in the two cases. Thus in Table 1 for experiments B-39-1, B-45-1 and B-52-1, the mass spectrometric estimates of the amounts of hydrogen generated per weight of alloy are 0.282, 0.253 and 0.281 mmole/g (with asterisks) while the "maverick" value is 0.360 mmole/g for experiment B-50-1. Similarly, the titration analyses indicate 0.379, 0.368 and 0.372 mmole/g for experiments B-39-1, B-50-1 and B-52-1 (with asterisks), while the "maverick" value is 0.324 mmole/g for experiment B-45-1.

While two sets of four experiments each cannot offer a statistically ideal situation, we can at least mention several observations on the two "maverick" experiments that might give some insight about why they differ from their "non-maverick" cohort experiments.

Experiment B-45-1

This was the first experiment with the molten eutectic alloy in which the water temperature was "hot" (61°C). Perhaps as a result of this, the diaphragm ruptured early to give an injection pressure of only 0.579 MPa; the usual rupture of a Kapton disc of this thickness (0.5 mm) produces an injection pressure of nominally 1.0 MPa, as indicated for the other seven experiments listed in Table 1. It is possible, then, that the lower injection pressure in experiment B-45-1 somehow contributed to the generation of a lesser amount of LiOH (OH^-) than its three cohorts, B-39-1, B-50-1 and B-52-1 over the 20 or 21 hours of contact between alloy and the column solution, yet did not affect the short-term analyses of hydrogen by mass spectrometry.

One possible explanation might be that, in experiment B-45-1, the lower injection pressure produced less porous, less fragmented debris that reacted about the same as the other samples on the short-term to produce hydrogen gas for the mass spectrometer but less completely over the long-term to produce the LiOH (or (OH^-) for the titration. We have no evidence to support this explanation other than a qualitative observation that the debris recovered from experiment B-45-1 seemed less bulky than that retrieved from the other three experiments with the eutectic alloy.

<u>Experiment B-50-1</u>

This experiment is the "odd" one from standpoint of the mass spectrometer analyses, showing a "high" value of 0.360 mmoles of H_2/g of alloy compared to its cohorts' values of 0.282, 0.253 and 0.281 mmoles of H_2/g for experiments B-39-1, B-45-1 and B-52-1, respectively. Moreover, its mass spectrometer value is almost identical to its titration-determined value of 0.368 mmole/g and also to the values of 0.379 and 0.372 mmoles/g for experiments B-39-1 and B-52-1, respectively.

We can speculate that the red-brown granular debris freed from the crucible from experiment B-50-1 as it was sawed, shown in Figure 24, may have quickly presented a very bubbly open structure to the water as it reacted, yielding essentially all of the hydrogen for mass spectrometry in the first few minutes after the interaction rather than more slowly for its three cohorts. This bubbly structure may have essentially frozen in place in the crucible, giving the friable material that was freed during the sawing operation.

<u>The Red-Brown Debris</u>

It has been noted that one common characteristic for the two "maverick" experiments with the alloy is the presence of red-brown debris on the surfaces of the two samples of debris from experiments B-45-1 and B-50-1 that were exposed when the crucibles were sectioned (see Figures 23b and 24b). There was <u>no</u> red-brown debris present in the two "non-maverick" experiments, B-29-1 and B-52-1 (see Figures 22b and 25b).

In order to learn more about the red-brown granular debris freed by sawing the crucible from experiment B-50-1 (see Figure 24b), we had some of this material analyzed by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) at the University of Wisconson Soil and Plant Analysis Laboratory. (We used this procedure previously to analyze samples of the alloy; see Nelson (1995).) The analysis indicated 0.25 w/o Li and 97.41 w/o Pb (see analytical report reproduced in Appendix D), or 9.0 a/o Li and 90.9 a/o Pb. Thus the alloy sample that began the experiment with 17 a/o Li now yields debris with only 9.0 a/o Li, suggesting that 47.1 a/o of its Li had been removed. This value is significantly less than the 71.5 a/o (ms) and 73.0 a/o (OH^-) removal indicated in Tables 1 and 3.

At present, we cannot offer an explanation for the differences between the "maverick" and "non-maverick" experiments and their relationships to the red-brown debris, if any.

Suggestions for Future Studies

In retrospect, we make several suggestions for improving the information that may be obtained from molten lithium-lead eutectic alloy interactions in a water impact apparatus, or in other similar experiments:

- Extend the measurements of temperature of the melt and the surrounding solution (i.e., TC5, TC0 and TC1 in our apparatus) to times after impact commensurate with the length of appreciable chemical reaction. This could be tens of minutes or even hours in our apparatus.
- Collect gas samples over longer time intervals. One or more samples should be taken after the apparatus and melt have cooled to room temperature and the melt has equilibrated with the aqueous solution that surrounds it.
- Prevent the gases generated in the experiment from contacting air in the expansion vessel before samples can be taken. In our apparatus, this would involve simply purging the

upper chamber with argon or other inert gas before starting the pressurization to initiate bursting of the diaphragm. Although we cannot be sure that combustion of hydrogen is occurring in the residual air present in this upper chamber, the argon purge would be a simple precaution that would require no change in the apparatus and only a minor change in procedure. Significant changes in the amounts of hydrogen measured with and without purging might indicate that combustion can affect the analyses.

- Record the equilibrium expansion gas temperatures with thermocouple TC4 after the impact. At present, this temperature is copied from the data displayed visually on the Keithley monitor.

In addition to the largely procedural changes suggested above, we also urge several fundamental changes in the design of the compact apparatus:

- Improve the design of the rupture disc holder to permit easier and more reproducible disc loading and rupture.
- Improve the melt heating arrangement to provide better uniformity in melt and furnace temperatures and easier positioning of the furnace around the crucible.
- Improve the design of the crucible and its holder to make it simpler and less costly to fabricate and to prevent unwanted chemical reaction and alloying, especially in theevent of crucible failure.

CONCLUSIONS

In this work, we have studied the impact of a water column driven downward by a pressurized gas onto molten eutectic Pb-Li alloy. The experiments were performed to investigate phenomena that might occur during accidental contact of this molten material with liquid water during a hypothetical fusion reactor malfunction. We observed significant physical and chemical interactions during the experiments, primarily fragmentation of the melt and its reaction with water to generate hydrogen and lithium hydroxide solution. Only moderate pressure transients were produced during the interactions and there was no sign of runaway combustion or ignition.

Overpressurization of containments might be a concern, however, because more than half the lithium present in the melt reacted with the water to produce hydrogen. Also, significant gaseous explosion hazard might be introduced if somehow the hydrogen were to mix with air. Moreover, the caustic lithium hydroxide solutions produced during the interactions could cause concern from personnel safety, corrosion and environmental standpoints.

In our work, we used two techniques for estimating the amounts of hydrogen generated during the interactions — quadrupole mass spectrometry and acid-base titration of the lithuim hydroxide generated. The titrations indicated significantly greater amounts of hydrogen than the mass spectral analyses. Our current interpretation of the differences between the mass spectrometer and titration analyses of the eutectic alloy-water interaction is that both procedures yield essentially correct values for the extent of the chemical reactions, but are applied over vastly different time periods — up to 5 minutes for the massspectrometry and tens of hours for the titration.

We have also compared our experimental results with nonreactive control situations in our own apparatus (impact volume filled with molten Pb or left empty), as well as studies by other workers with both reactive and nonreactive melt-water interactions.

As a result of the previous and present studies, we believe that a reasonable understanding of the behavior that might be expected during an accidental contact ofthe molten eutectic Pb-Li with liquid water is beginning to emerge.

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Appendix A

Information Pertinent to Performing the Experiments

The vertical water impact apparatus (also referred to as the "shock tube") involves a complex assemblage of experimental components and procedures. In this appendix, we describe the data acquisition and experiment control systems, summarize typical procedures and present a prototype check list. We hope to provide a background for future experimentation with this apparatus and to assist in the evaluation of data obtained with it.

Data Acquisition and Experiment Control

The data acquisition and experiment control systems are shown schematically in Figures A-1 and A-2, respectively.

The experimental sequence consists of two basic operation phases: the first phase is a heat-up phase where the melt below and the coolant above are brought to the desired initial conditions. Heat-up can extend to 30 min or longer depending on the temperatures required. The second phase is the reaction phase and begins when the rupture disk breaks and the coolant comes in contact with the melt.

The heat-up phase begins by turning the heaters on with dual manual switches. AC power is delivered through the switches to a set of Variacs and on to the heaters. The Variacs adjust the voltage and determine the rate of heating. At this time the Keithley 500 Data Acquisition and Control system (the "Keithley") is used to monitor the thermocouples (TC0-TC5) at the melt and along the tube. A static transducer labeled PT5 is mounted in the upper driver section; PT5 is a strain gauge type transducer. The amplifier box connected to the transducer provides the excitation voltage for the transducer and output to the Keithley. The PC-AT attached to the Keithley operates and records information from the Keithley. During this phase the temperatures (TC0-TC5) and pressure (PT5) are displayed on the PC screen. The PC uses the Keithley operating software called KDAC500. KDAC500 uses a basic program written for this experiment called SS-V.ASC. KDAC500 requests a keystroke when heat-up is complete. When the keystroke is applied the PC will begin a 3 second countdown and store 3 seconds of data before rupture.

The Keithley initiates the reaction phase by sending a TTL level signal to a solid state relay located on the relay board. AC power is delivered via the relay to the argon solenoid valve. The solenoid valve opens and argon pressurizes the upper tube section (the expansion vessel) which causes failure of the rupture disc. Failure of the disc allows coolant contact with the melt causing mixing and a shock pressure pulse. The PC stores an additional 8 seconds of temperature and pressure data received from the Keithley after a 3 second countdown.

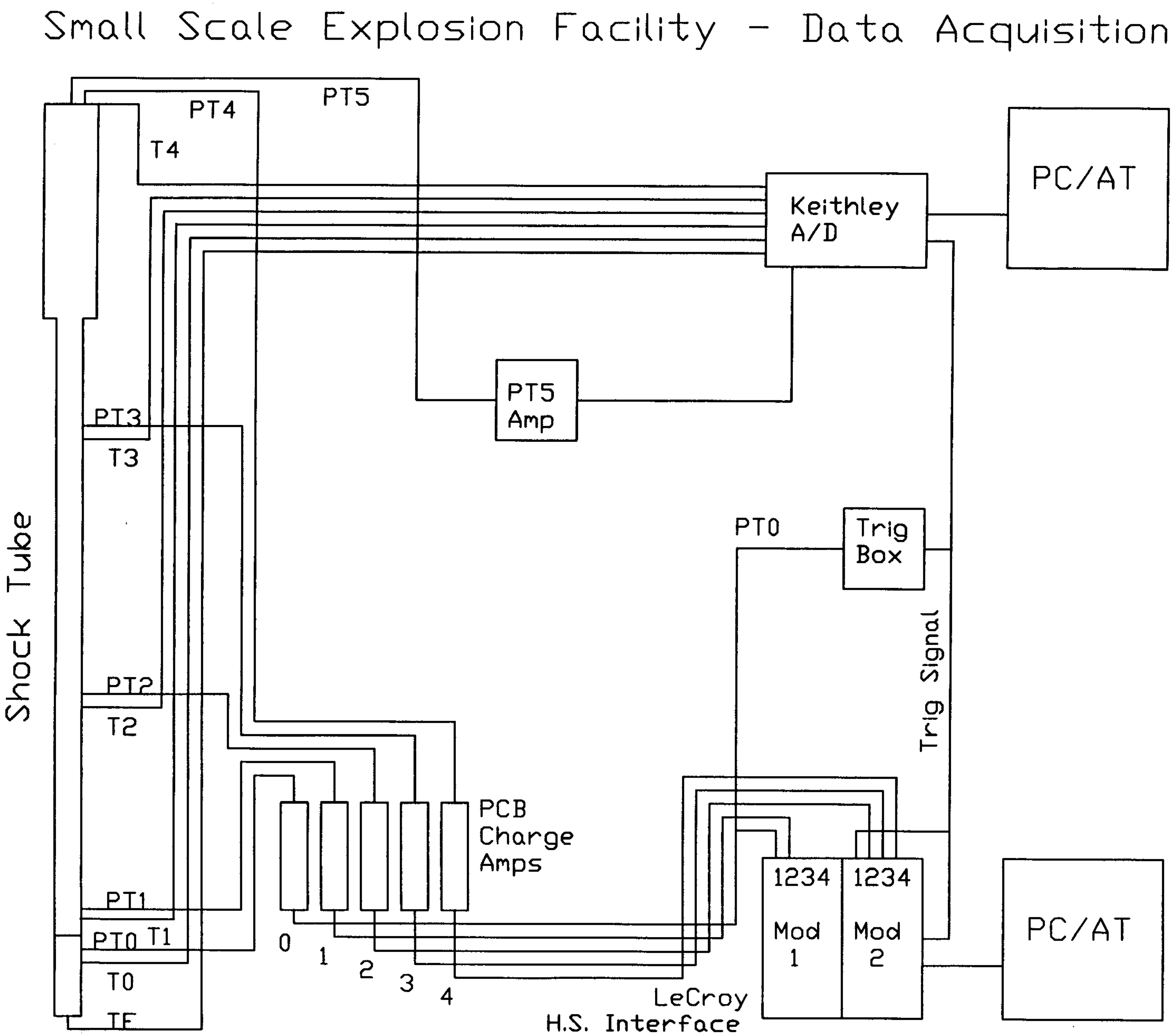


Figure A-1. Schematic diagram of the data generation and acquisition system used with the vertical water impact (shock tube) apparatus.

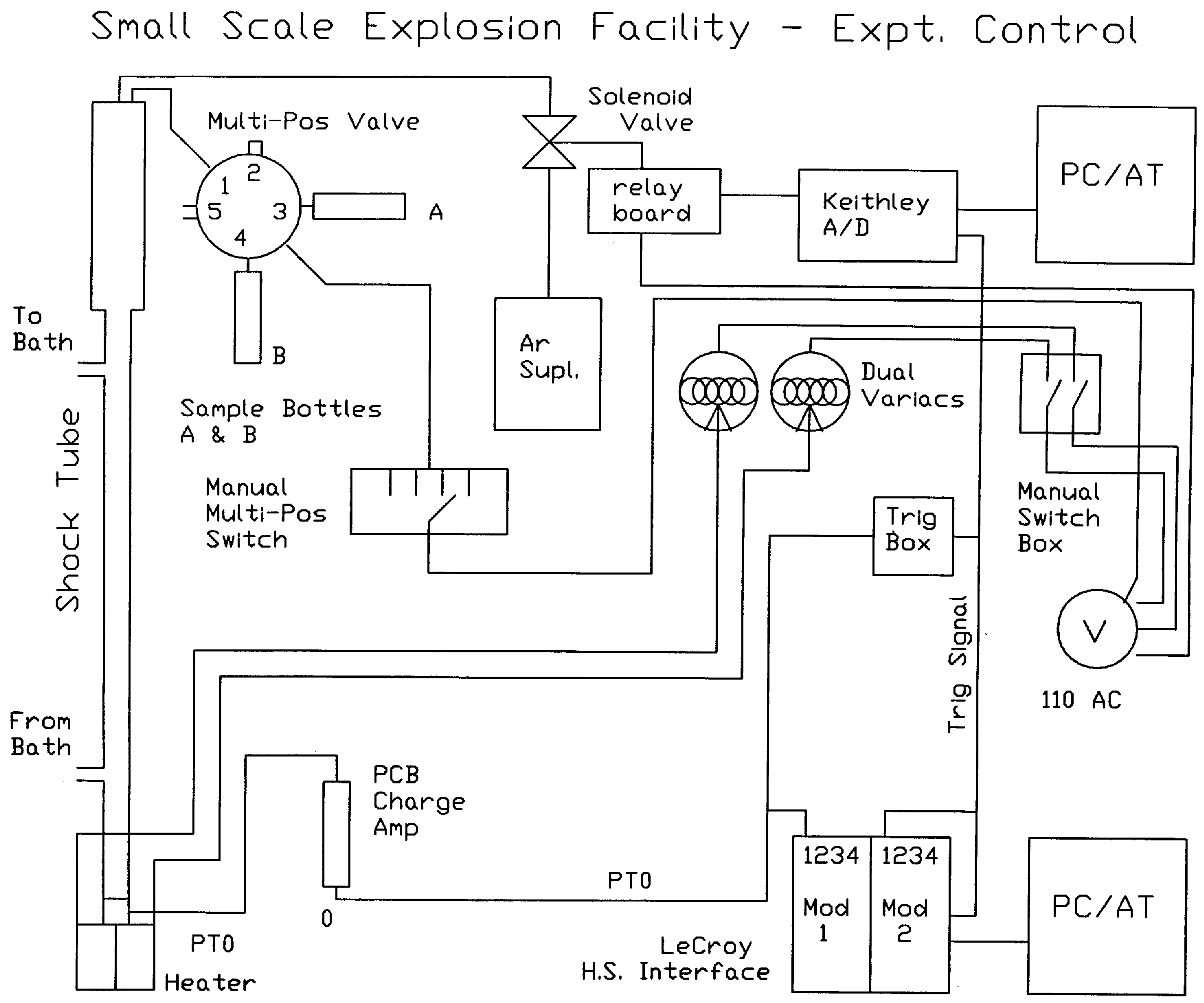


Figure A-2. Schematic diagram of the system used for control and recording of temperatures, pressures, gas flows and sequencing pertinent to the vertical water impact (shock tube) apparatus.

PT0 through PT4 are piezo type transducers located along the tube to measure the dynamic pressure changes associated with the shock. The piezo type transducers are paired with charge amplifiers which convert the transducer capacitance change to a voltage signal. A high speed data acquisition system is required to record and store the signals produced by the charge amplifiers. This experiment uses a LeCroy High Speed Interface (the "LeCroy")to record and store the signals from PT0 through PT4.

The LeCroy is controlled by a PC AT using Catalyst software. The program carlst.exe is used to prepare the LeCroy for operation. The single trigger option is used which requires a trigger signal of TTL positive to initiate data acquisition by the LeCroy.

PT0 is the first transducer to sense a pressure pulse. A trigger box containing a compactor and amplifier is connected in parallel with the LeCroy to the output of the PT0 charge amplifier. A rise of voltage in the PT0 charge amplifier causes the trigger box to send a TTL positive trigger to the LeCroy and to the Keithley. The Keithley simply records the trigger signal to be used as a reference.

The multi-position valve at the tube upper section (expansion vessel) is kept in the closed position (2) during pressurization and rupture. After one minute the manual multi-position switch is rotated to move the valve into the bottle A position (3) to obtain a gas sample. Five minutes after rupture the switch is rotated to move the valve into the bottle B position (4) to obtain another gas sample. After closing their valves, the sample bottles are removed and the multi-position valve is moved to the exhaust position (5) to relieve pressure built-up in the column and expansion vessel.

Experimental Operations

In this section, we present a chronological listing of the steps taken in a normal water column impact on a heated melt. This listing could be used as a checkoff list for the performance of an experiment if desired.

- Weigh crucible with two washers with and without material to be melted. This is done in an argon-flushed glove box if the material is air-sensitive, e.g., eutectic Pb/Li alloy.
- Install diaphragm and put lower part of the apparatus in place.
- If the crucible loading operation begins in the glove box, cover the top of the crucible with paraffin foil to minimize contact with air during the transfer.
- Transfer the crucible to the impact apparatus.

- Flush the lower portion of the impact apparatus with argon, which will exit downward from the crucible mounting.
- Install the crucible with washers in its sample mounting, removing the paraffin foil if used. Secure crucible with lower mounting. Install thermocouple TC5 through the lower mounting, inserting it in the indentation in the bottom of the crucible.
- Check mechanical vacuum pump oil; change if necessary.
- Test lower portion of the apparatus for vacuum-tightness with convectron gauge.
- Test all transducers for baseline drift, and overall operability.
- Check voltages of 9 V trigger and 12 V strain gauge batteries.
- Assemble two heater halves around crucible holder; attach electrical leads from Variacs.
- If water at an elevated temperature is used in the experiment, set and operate circulating bath heater to provide water a few degrees above that desired for the experiment. Install and operate auxiliary heating tape just above the flange above the diaphragm. Adjust bath and tape temperatures to provide desired water temperature that is uniform along the height of the column using thermocouples TC0 through TC4..
- Evacuate mass spectrometer sample bottles to 300-400 mbar (1 mbar=10^2 Pa). Install both bottles on multi-position valve at top of the column.
- Turn on both crucible heaters. Adjust current manually on two Variacs to achieve desired melt temperature as read on TC5.
- Provide inert melting atmosphere in the crucible and lower portion of the apparatus by control of evacuation, leak tightness and argon flow.
- Prepare to run by closing valves to lower chamber and redirecting argon flow at increased pressure to upper chamber.
- Arm triggering unit.
- Start control of experiment by Keithley unit. Turn off crucible heaters.
- Overpressurization of upper chamber will cause disc to burst and drive water column downward.

- Impact will trigger data acquisition systems via signal from transducer PT0. Data will be collected by the two personal computers.
- Immediately after the experiment, safeguard data on computers to prevent inadvertent loss.
- Open multi-position valve to sample bottle A during first minute after diaphragm burst.
- Take sample at 1 minute by opening valve on the bottle A. Listen for gas flow into bottle A.
- During next four minutes, rotate multi-position valve to sample bottle B.
- Take second gas sample by opening valve on bottle B at 5 minutes. Listen for gas flow into bottle B.
- Release gas pressure in upper chamber by rotating multi-position valve. Note unusual odors, etc., as gas is exhausted into the room atmosphere.
- Remove sample bottles A and B from the multi-position valve.
- Analyze gases in the two sample bottles A and B with mass spectrometer.
- Collect, convert and transfer stored transducer and thermocouple data to floppy discs.
- After apparatus cools, remove and collect aqueous solution from the column with rinsing if analyses are desired. Use distilled water and continue rinsing until solution draining from the apparatus appears neutral toward wide range pH test paper.
- Remove debris for analyses later. Rinse with distilled water, if desired; collect and combine rinsings until neutral toward test paper for titration later.
- If debris adheres to the crucible attempt to remove it by gentle probing. If this is unsuccessful, weigh the crucible after it is dry. Section crucible if desirable.
- Print out transducer and thermocouple traces as desired.
- Convert mass spectrometer data to partial pressures.

Experimental Outline

A prototype experimental outline that may be used as a check list during and after the vertical water column impact experiments is reproduced in Figure A-3; it was prepared by A. Farahani.

Experiment Number:

Date and Time of Experiment	
Components: Fuel / Coolant	
Expected Set-Up:	Tf = C Pdr = bar Pline = psig Pvac = torr
People Present	
Ambient Temperature and Pressure	T = C Pa = " Hg
Height of Water Column / pH Factor of Coolant	260 cm /
Mass and Density of Fuel	Mf = g
Height of Fuel in Crucible	
Crucible Number and Weight	# Wc = g
Rupture Disk Thickness	
Preamplifier Supply / Trigger Box Voltages	
Charge Amplifier Descriptions pt0 Range = psig/Volt pt1 Range = psig/Volt pt2 Range = psig/Volt pt3 Range = psig/Volt pt4 Range = psig/Volt	 Time Constant = Sensitivity = Time Constant = Sensitivity = Time Constant = Sensitivity = Time Constant = Sensitivity = Time Constant = Sensitivity =

Time at the Start of the Heating Process	
Time at the End of the Heating Process	
Time at the Opening of the Solenoid Valve Vacuum Pressure	 torr
Time at the Opening of Bottle #1 /pressure/temp.	
Time at the Opening of Bottle #2 /pressure/temp.	
Data Files Keithley LeCroy	
Actual Set-Up	Tf = C Pdr = psig
Weight of Retrieved Fuel	
Weight of Crucible plus Remainder of Fuel	
Fuel Loss %	

Comments:

Figure A-3. Experimental guide and checklist.

Appendix B

Instrumental Records

A complete set of the traces recorded from transducers PT0, PT1, PT2, PT3, PT4, the strain gauge and the thermocouple TC5 for each of the eight experiments is presented in Figures B-1 through B-8. A few unsuccessful records are not available and are omitted.

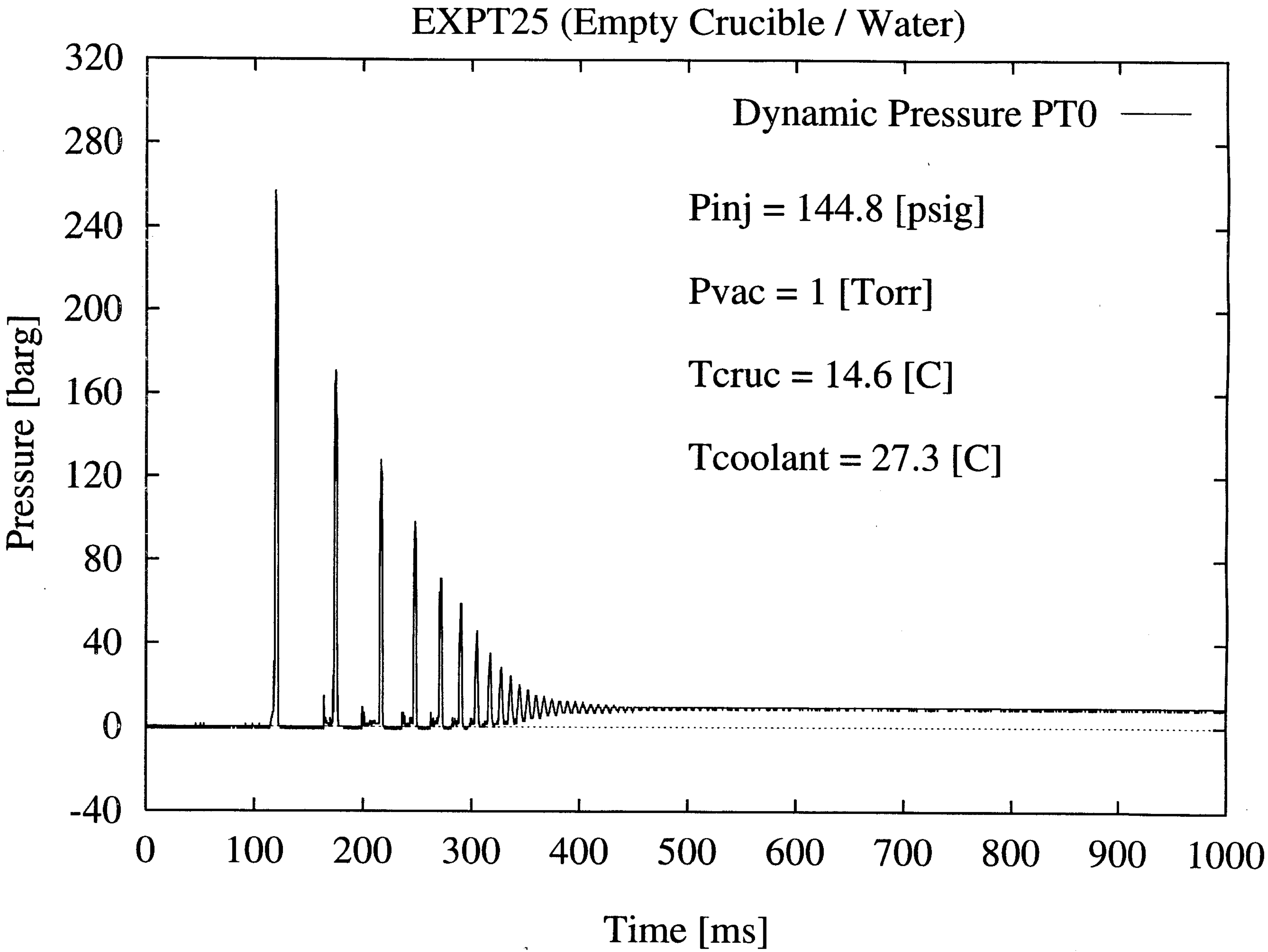


Figure B-1. Complete set of instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C.
a. Transducer PT0,1000 ms trace.

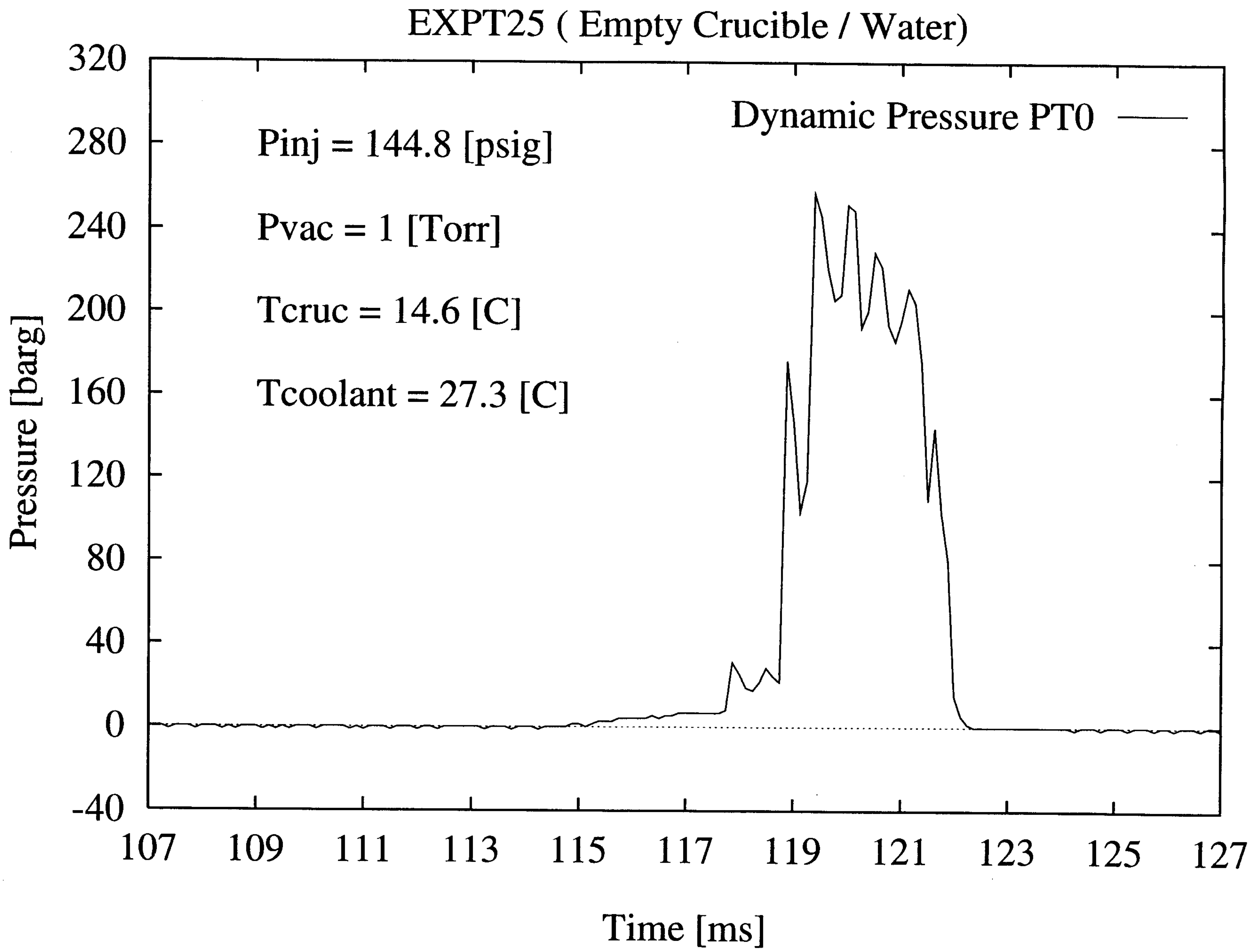


Figure B-1. Complete set of instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C.
b. Transducer PT0, expanded time scale.

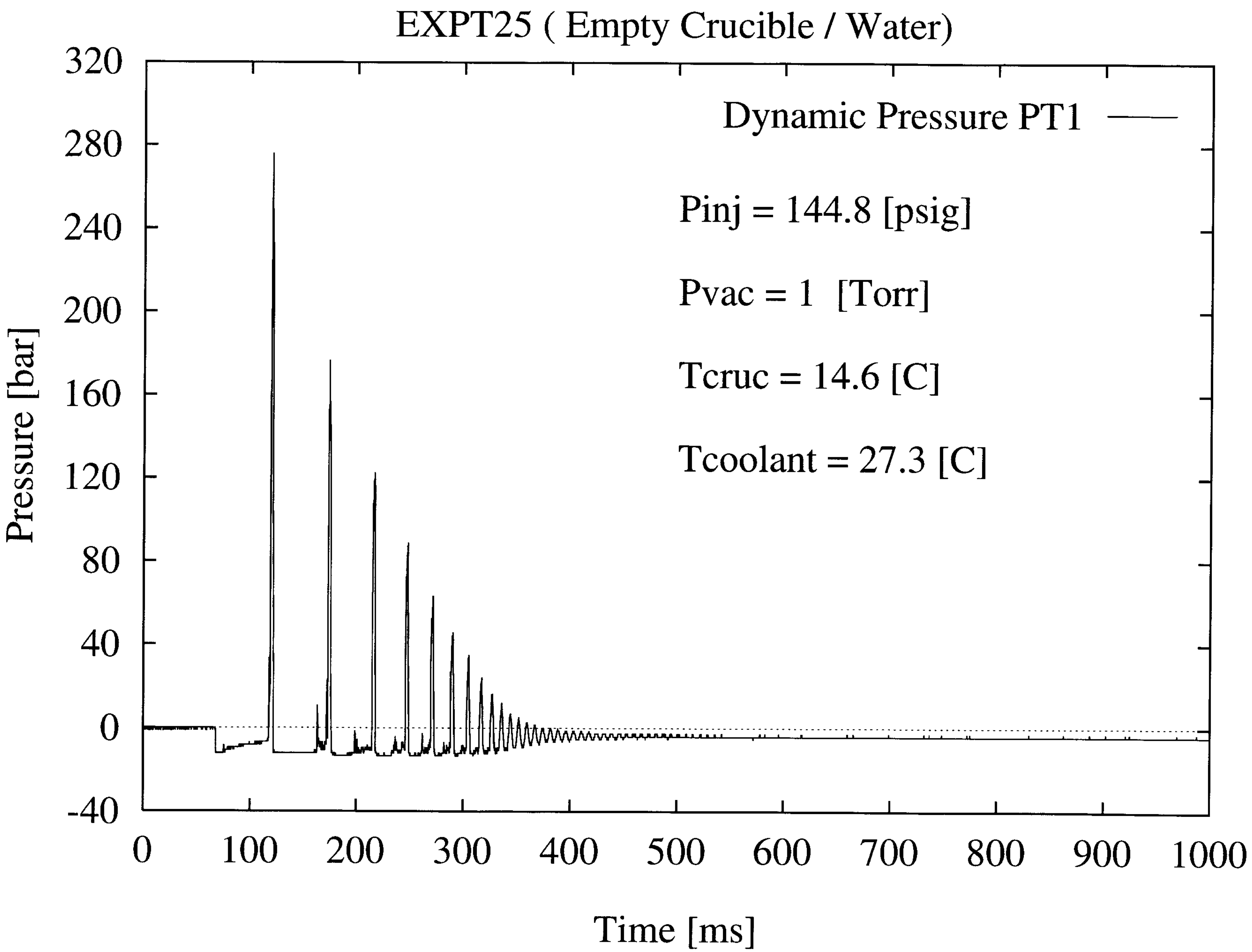


Figure B-1. Complete set of instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C.
c. Transducer PT1, 1000 ms trace.

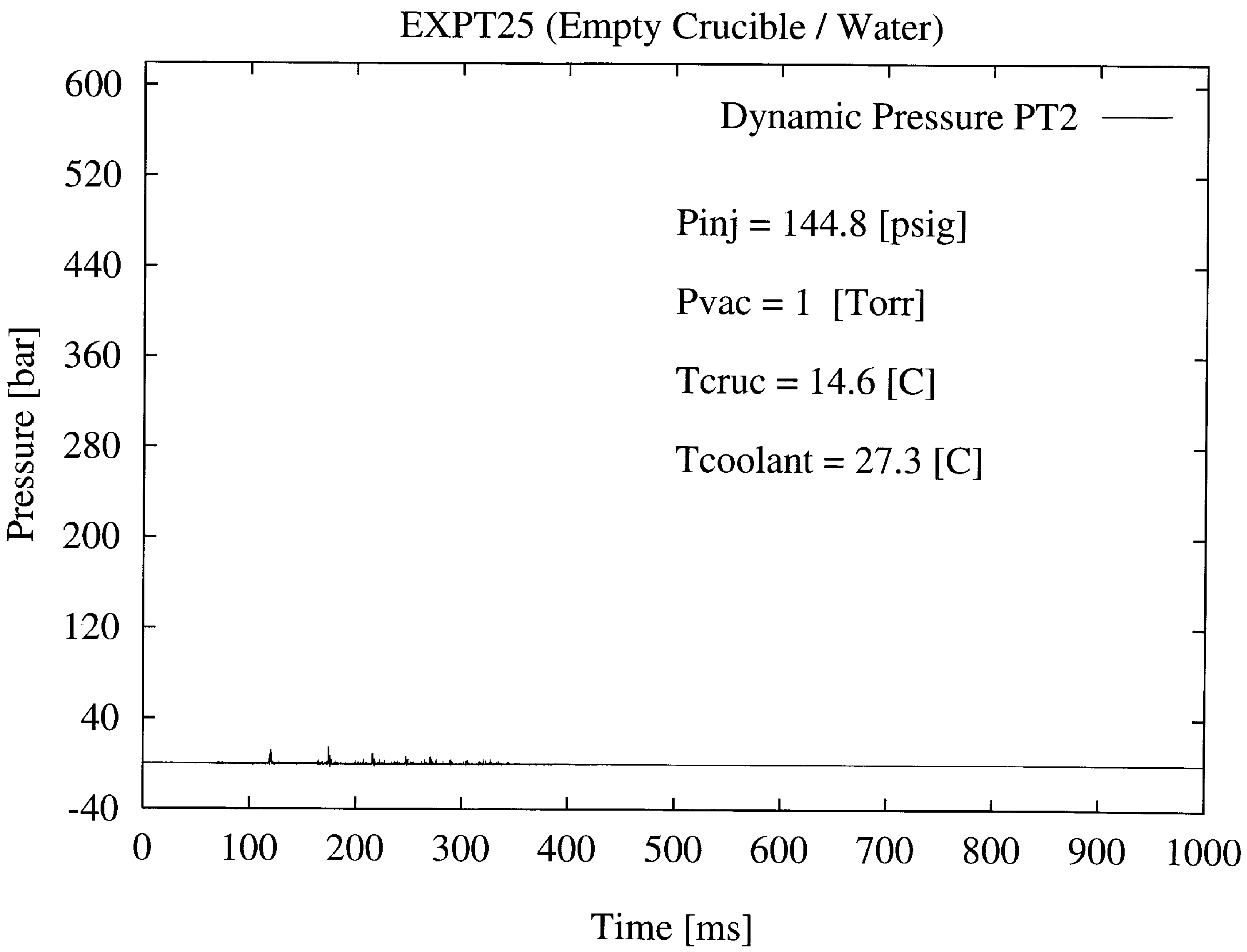


Figure B-1. Complete set of instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C.
d. Transducer PT2, 1000 ms trace.

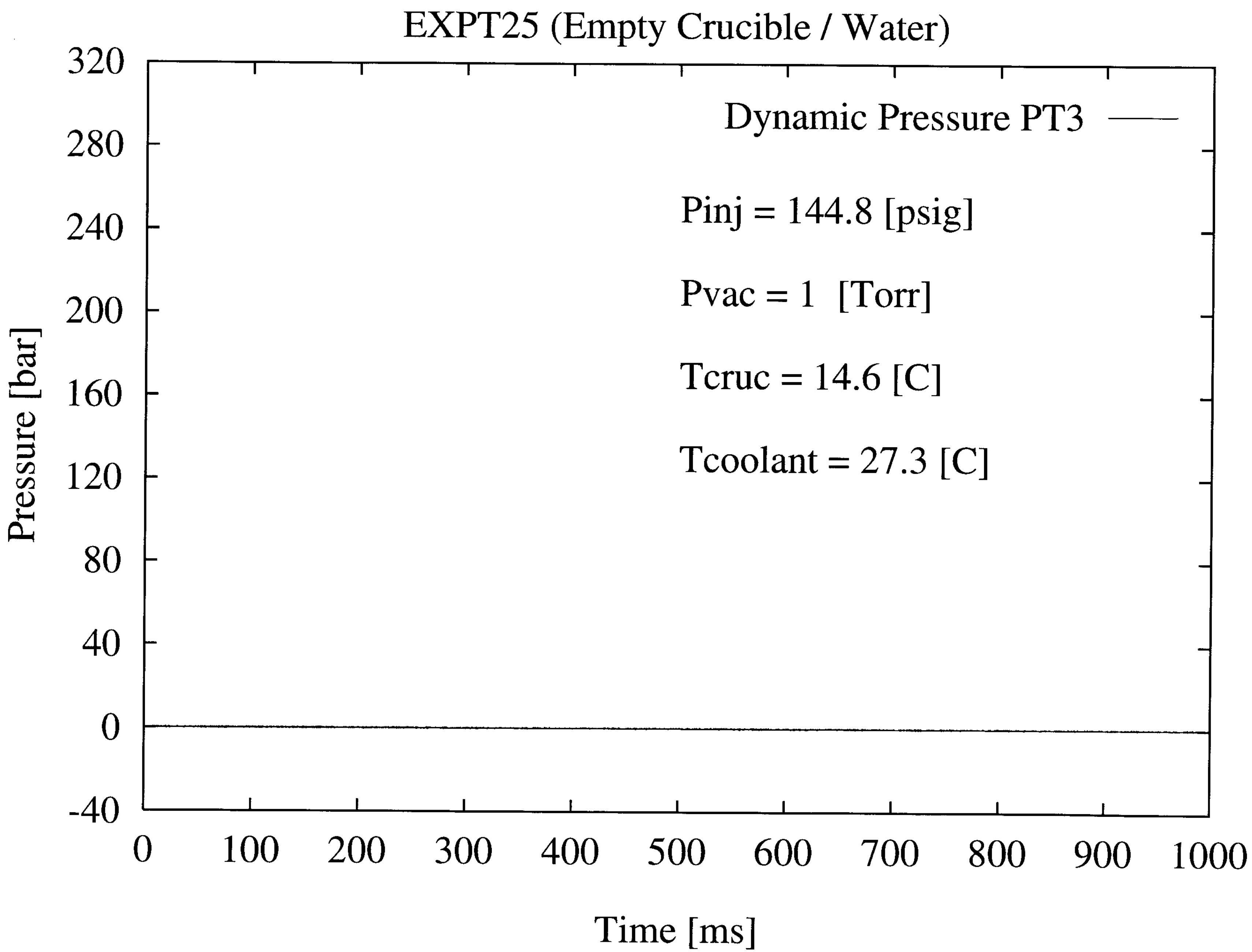


Figure B-1. Complete set of instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C.
e. Transducer PT3, 1000 ms trace.

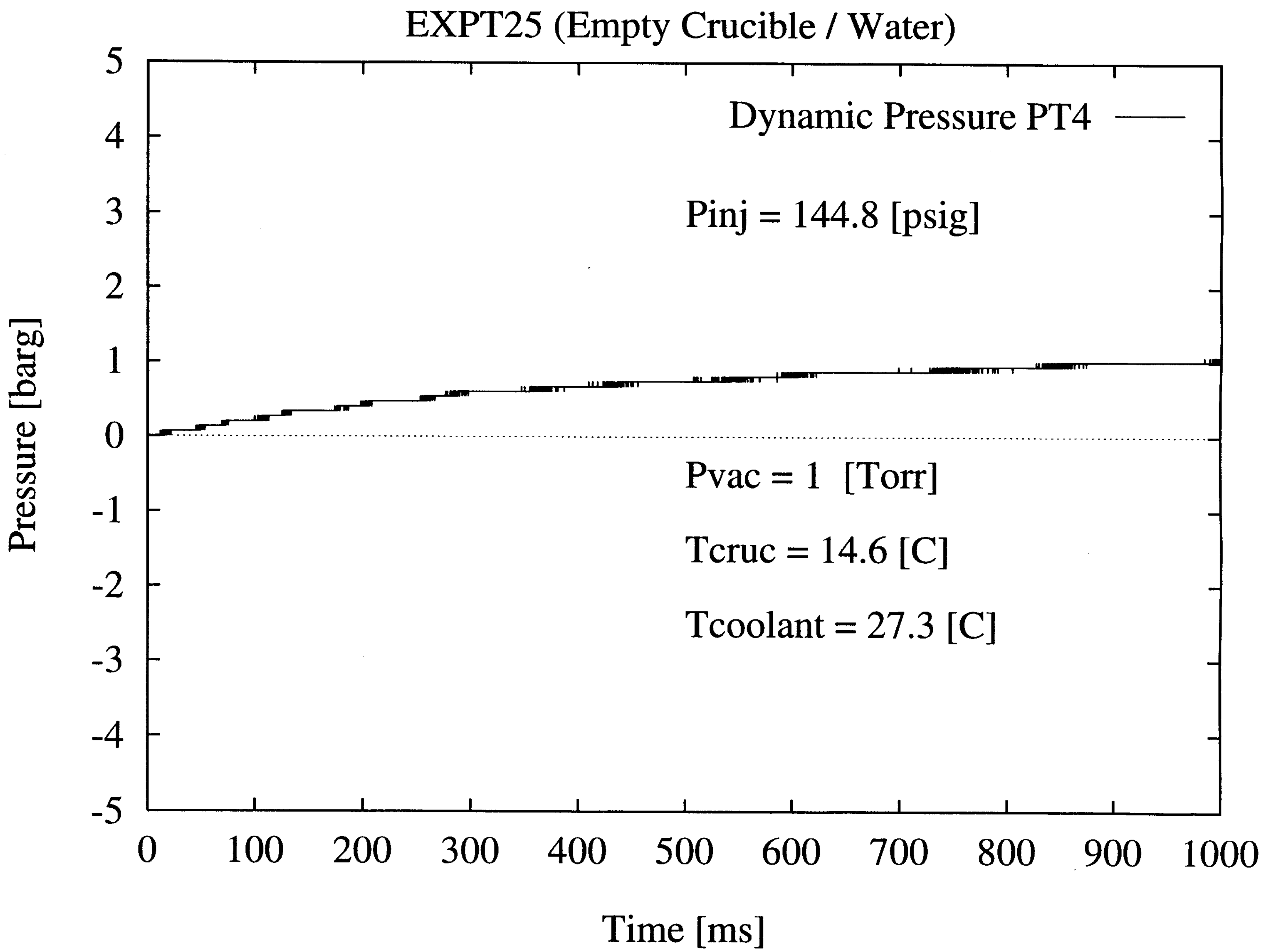


Figure B-1. Complete set of instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C.
f. Transducer PT4, 1000 ms trace.

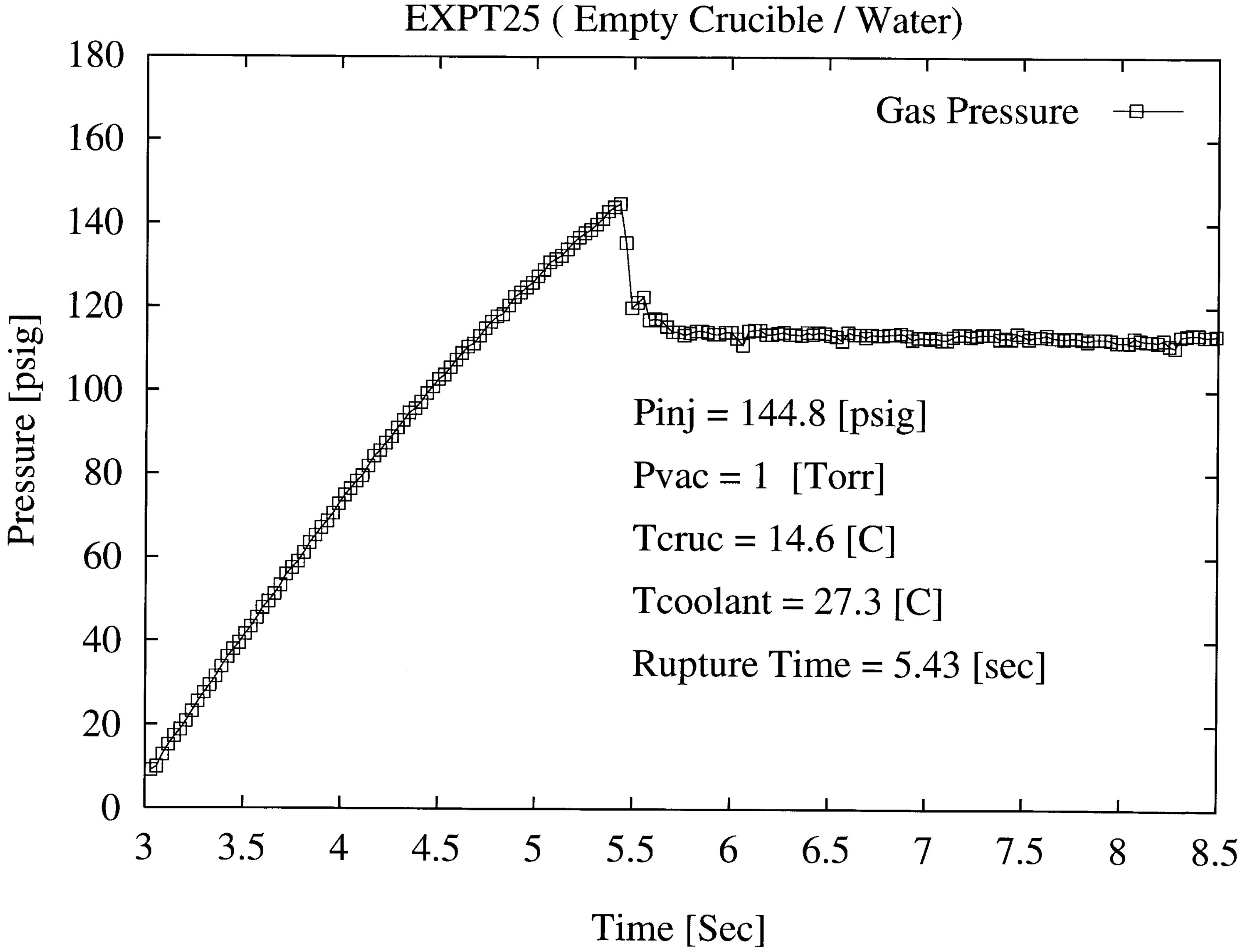


Figure B-1. Complete set of instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C.
g. Strain gauge PT5, 5 s trace.

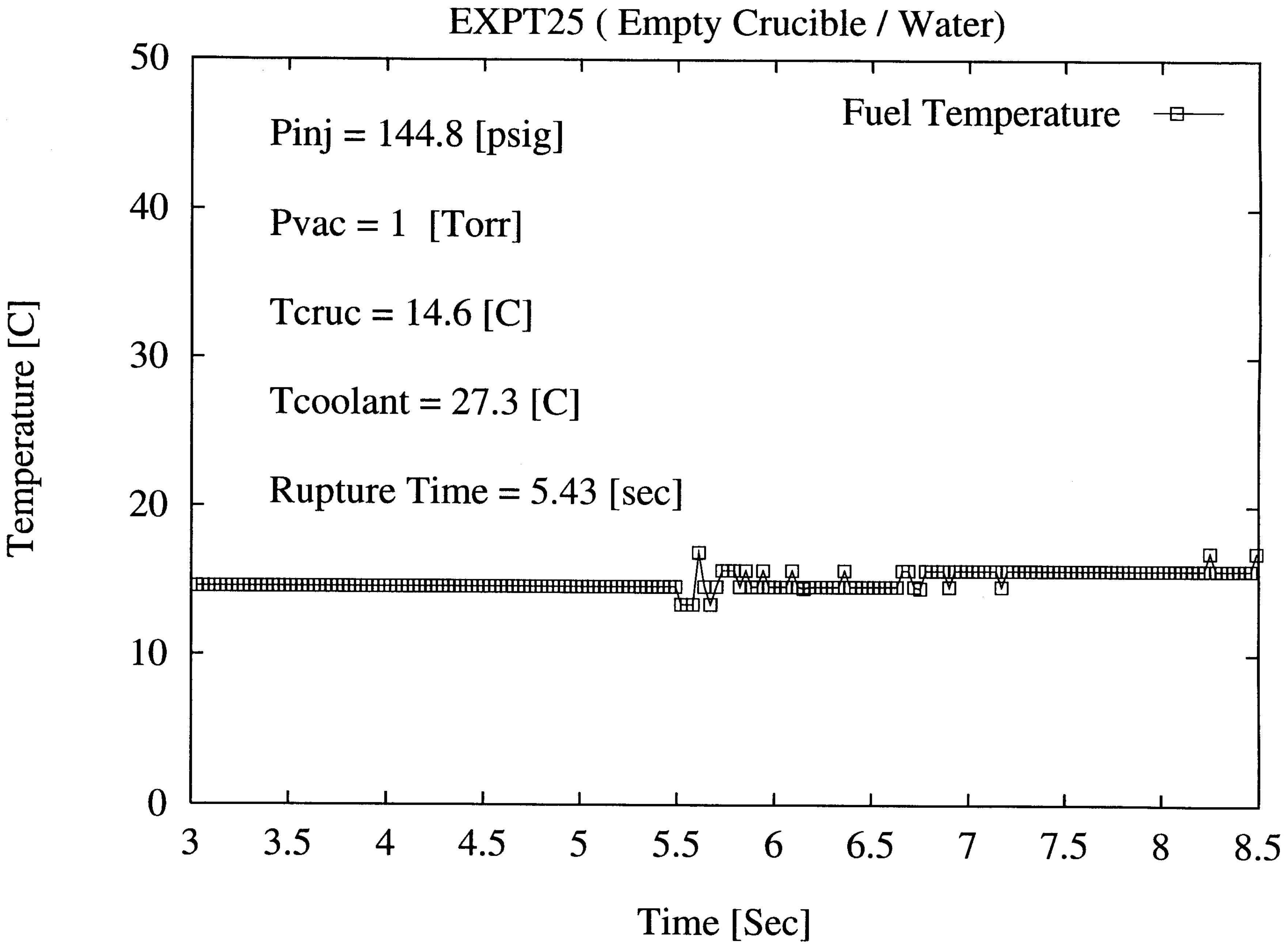


Figure B-1. Complete set of instrumental records from experiment B-25-1. Crucible was empty and at room temperature; water was at 27.3°C.
h. Thermocouple TC5, 5 s trace.

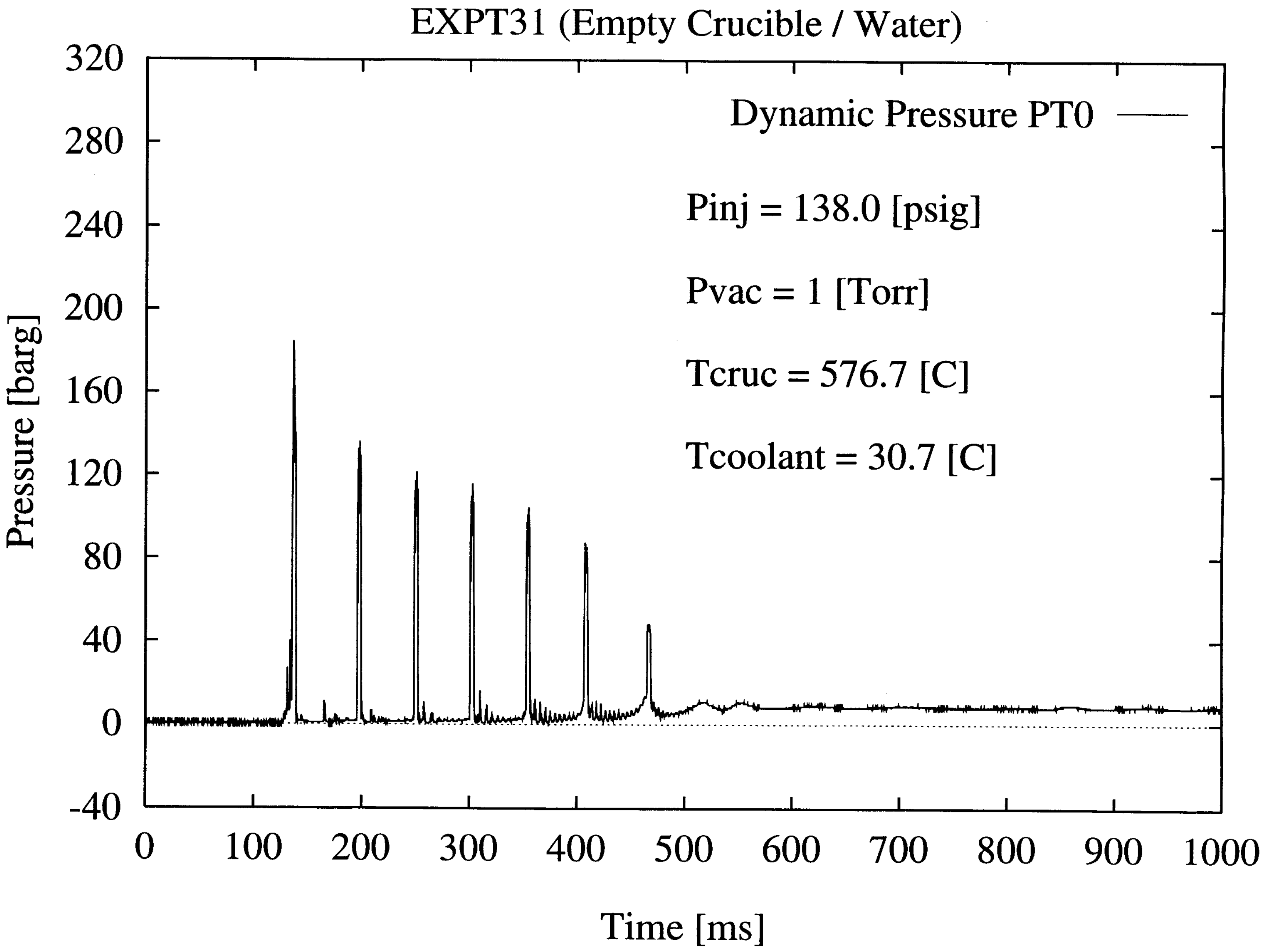


Figure B-2. Complete set of instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C.
a. Transducer PT0,1000 ms trace.

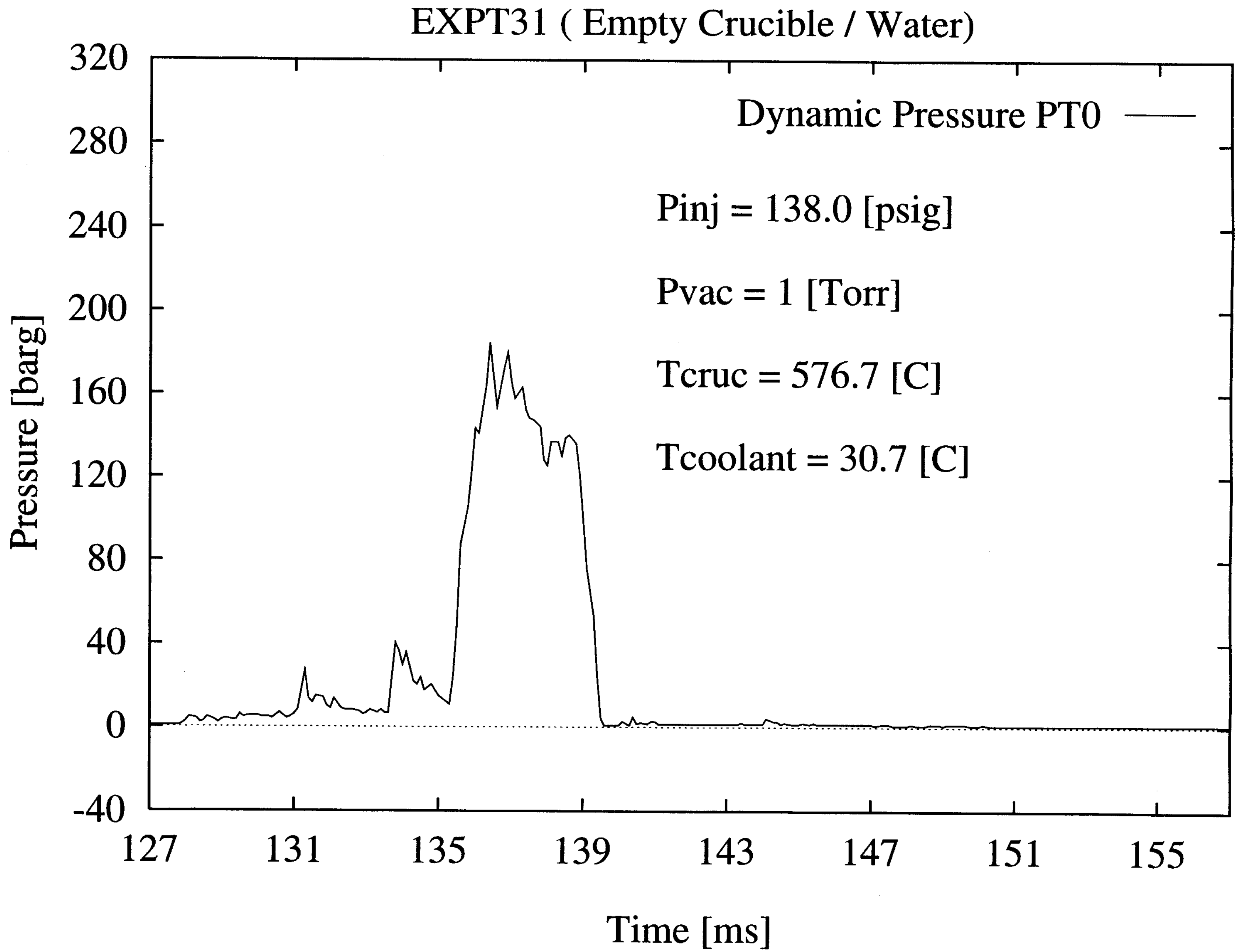


Figure B-2. Complete set of instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C.
b. Transducer PT0, expanded time scale.

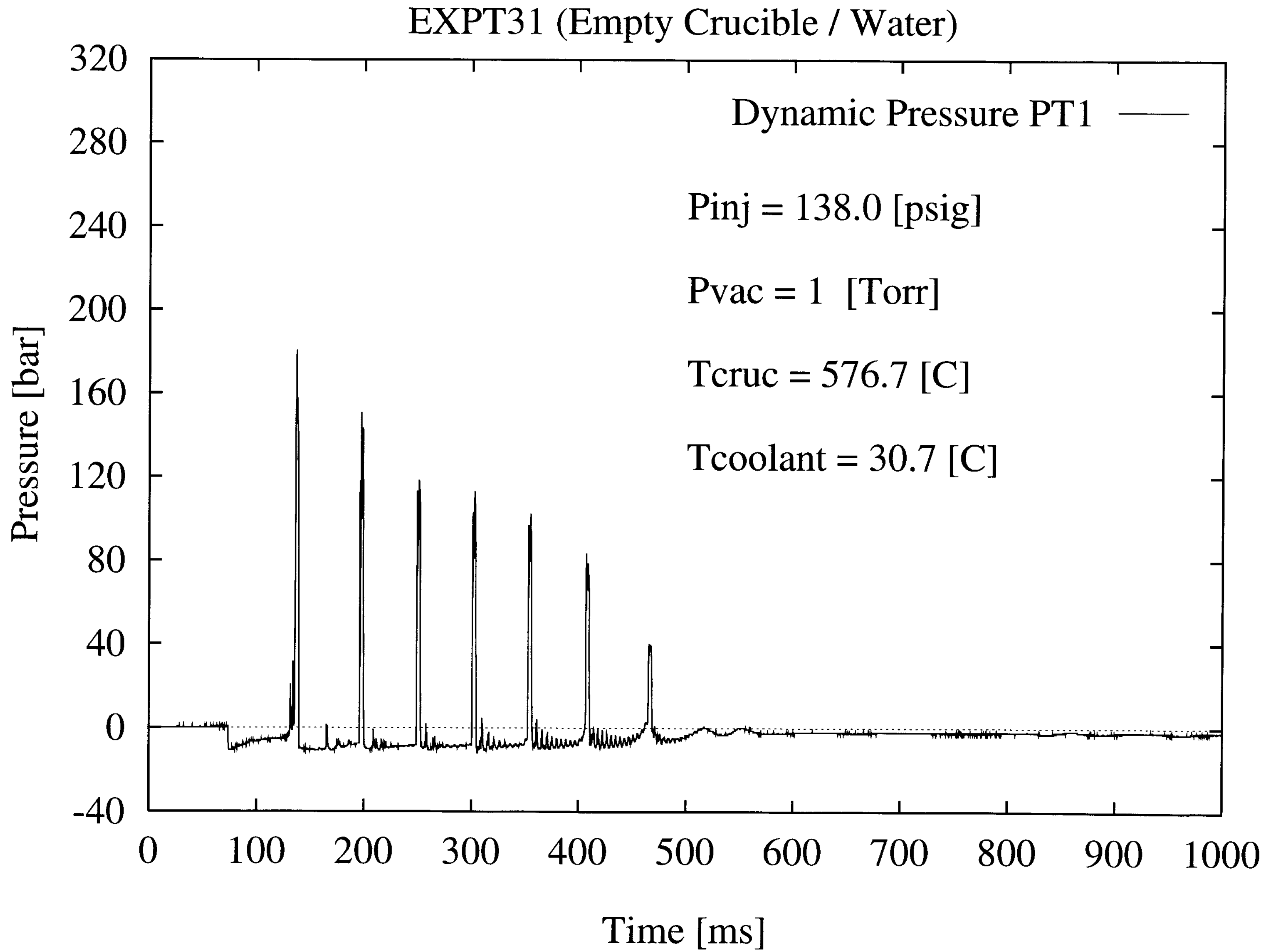


Figure B-2. Complete set of instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C.
c. Transducer PT1, 1000 ms trace.

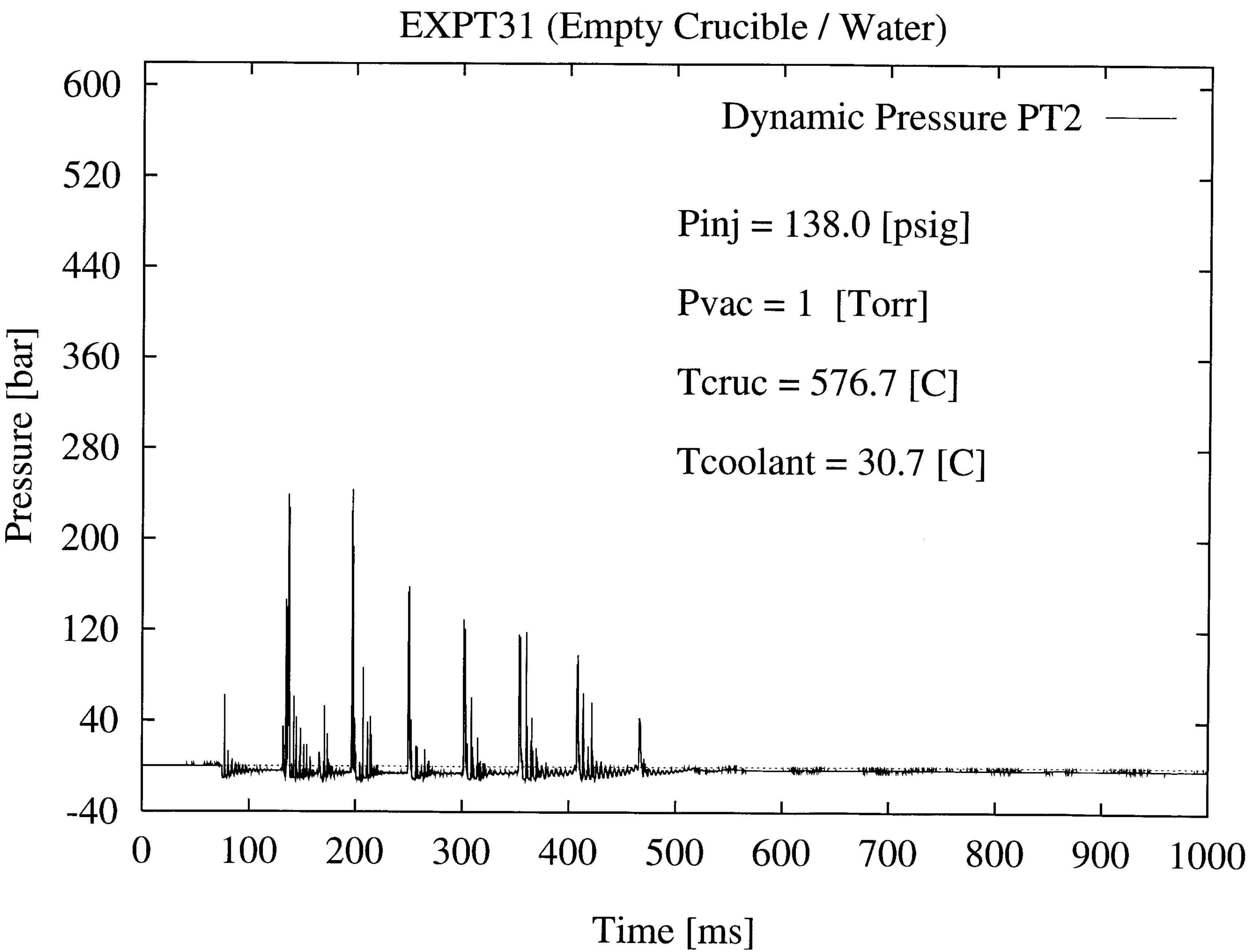


Figure B-2. Complete set of instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C.
d. Transducer PT2, 1000 ms trace.

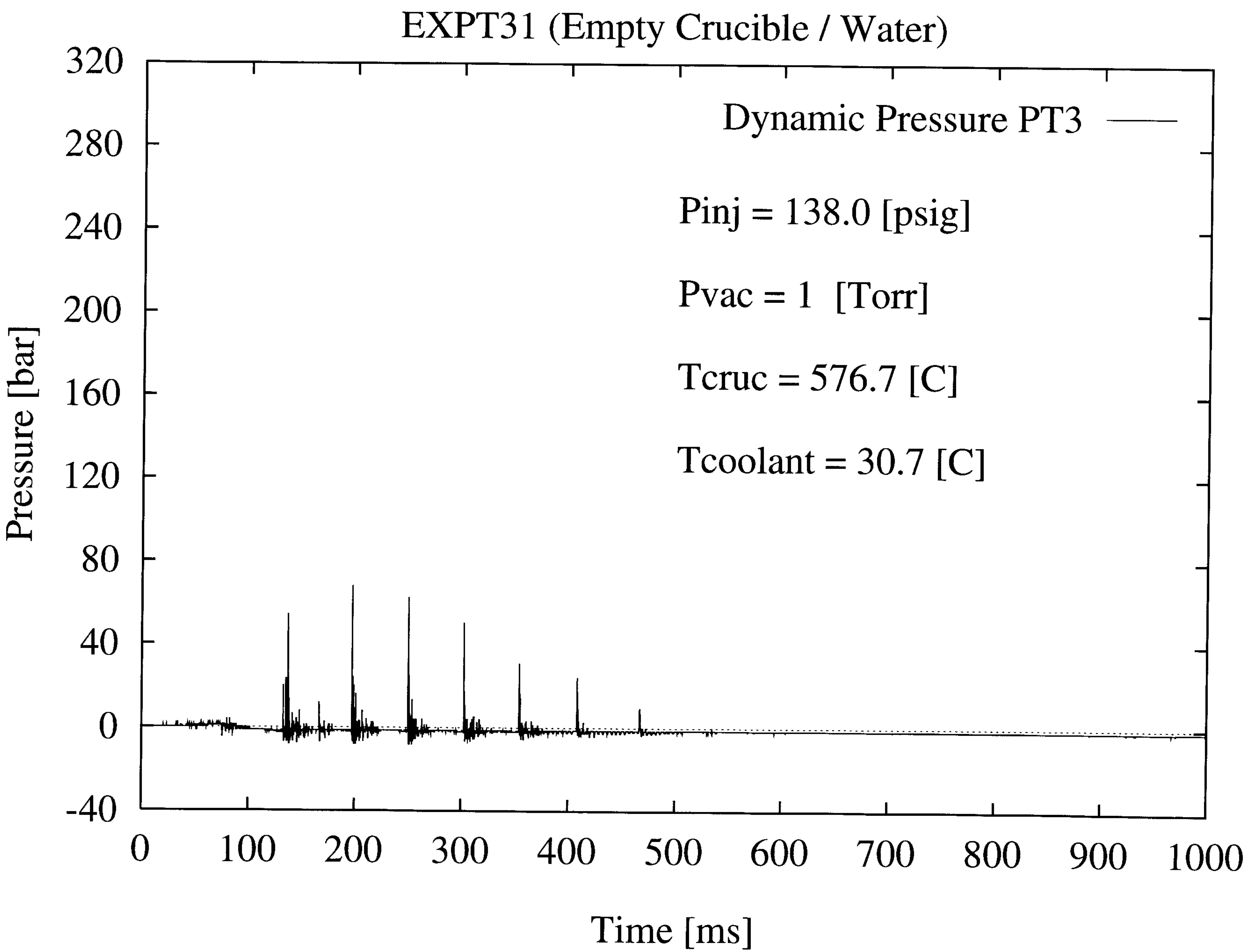


Figure B-2. Complete set of instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C.
e. Transducer PT3, 1000 ms trace.

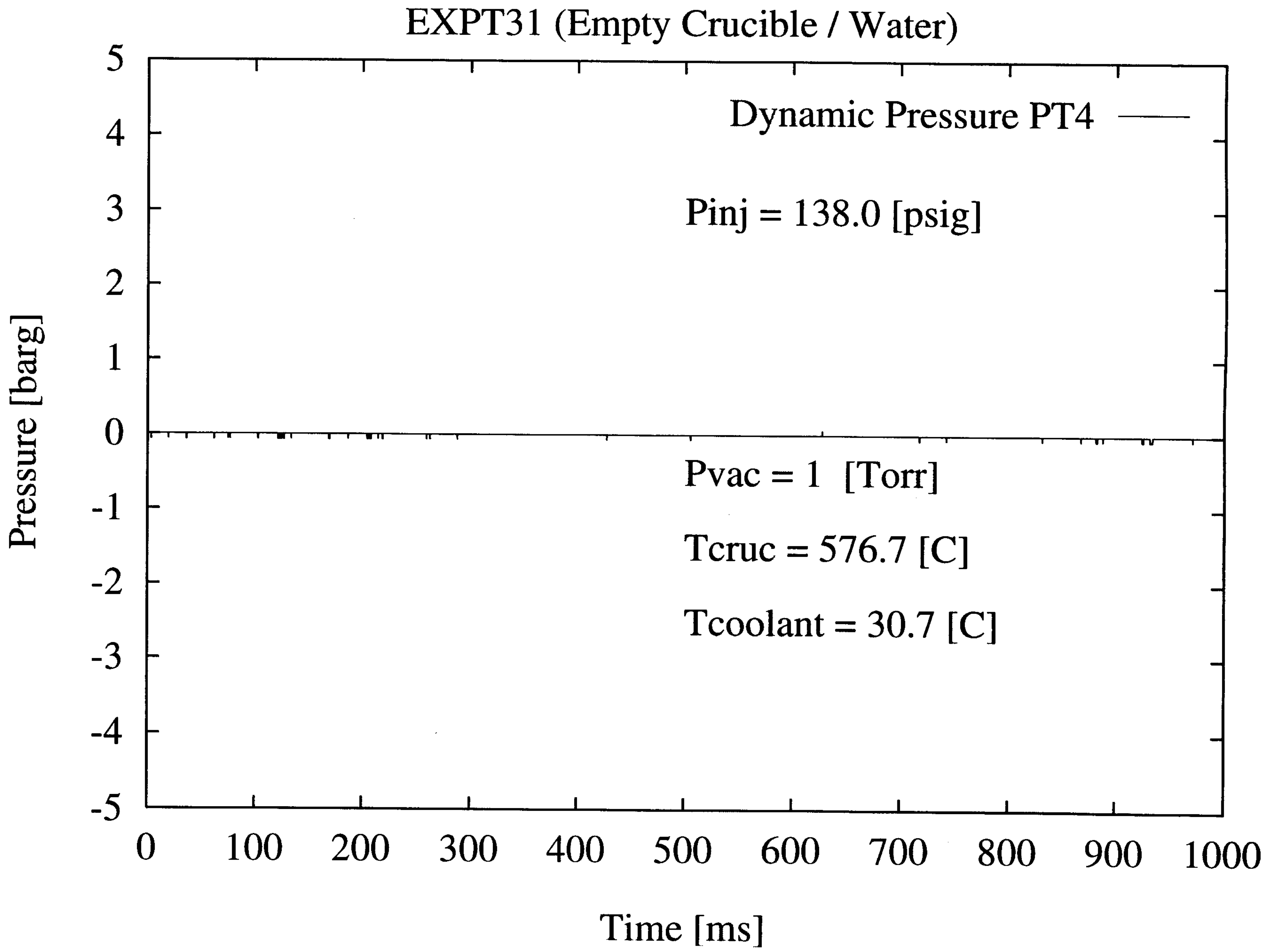


Figure B-2. Complete set of instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C.
f. Transducer PT4, 1000 ms trace.

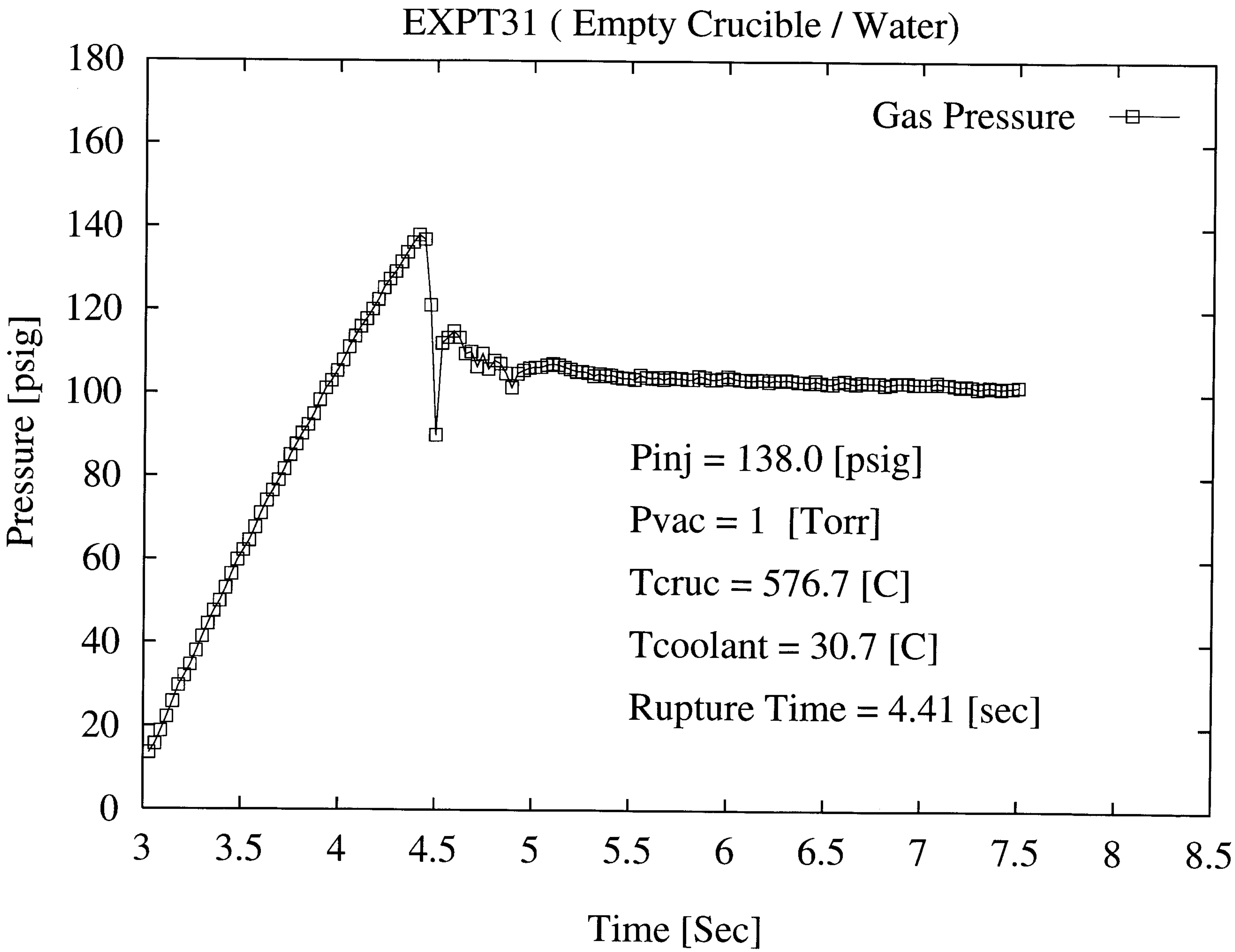


Figure B-2. Complete set of instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C.
g. Strain gauge PT5, 5 s trace.

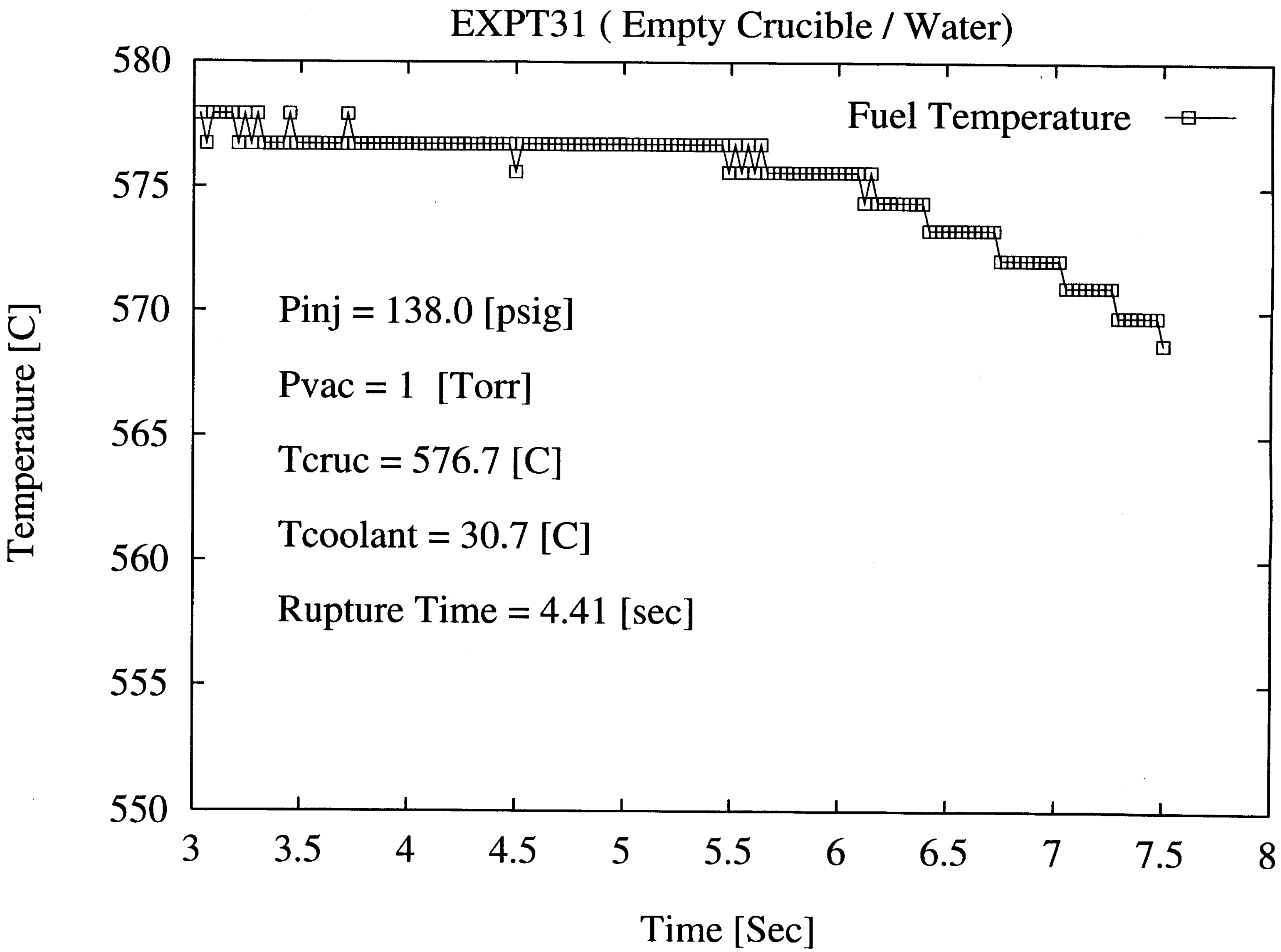


Figure B-2. Complete set of instrumental records from experiment B-31-1. Crucible was empty and at 576.7°C; water was at 30.7°C.
h. Thermocouple TC5, 5 s trace.

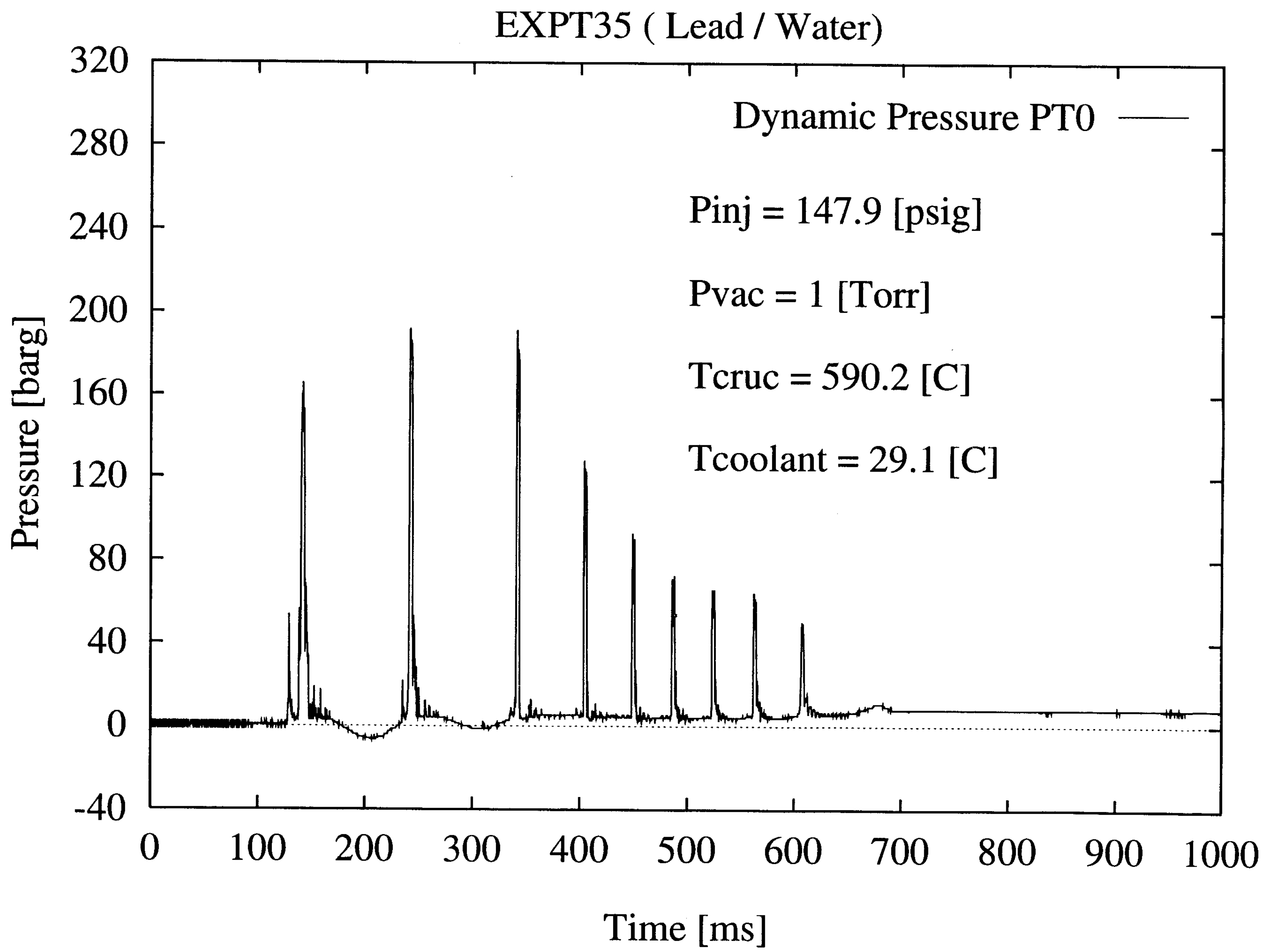


Figure B-3. Complete set of instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C.
a. Transducer PT0,1000 ms trace.

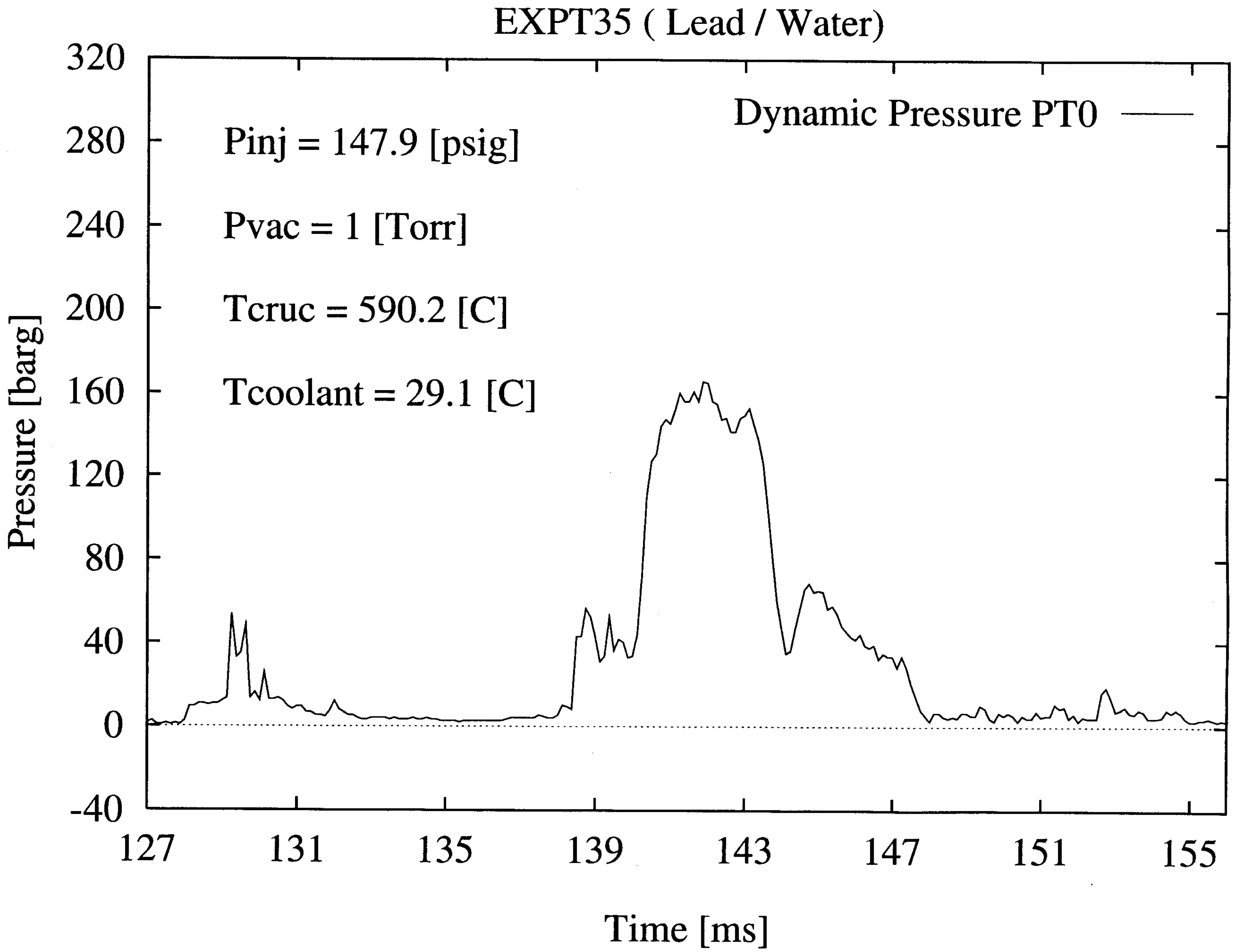


Figure B-3. Complete set of instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C.
b. Transducer PT0, expanded time scale.

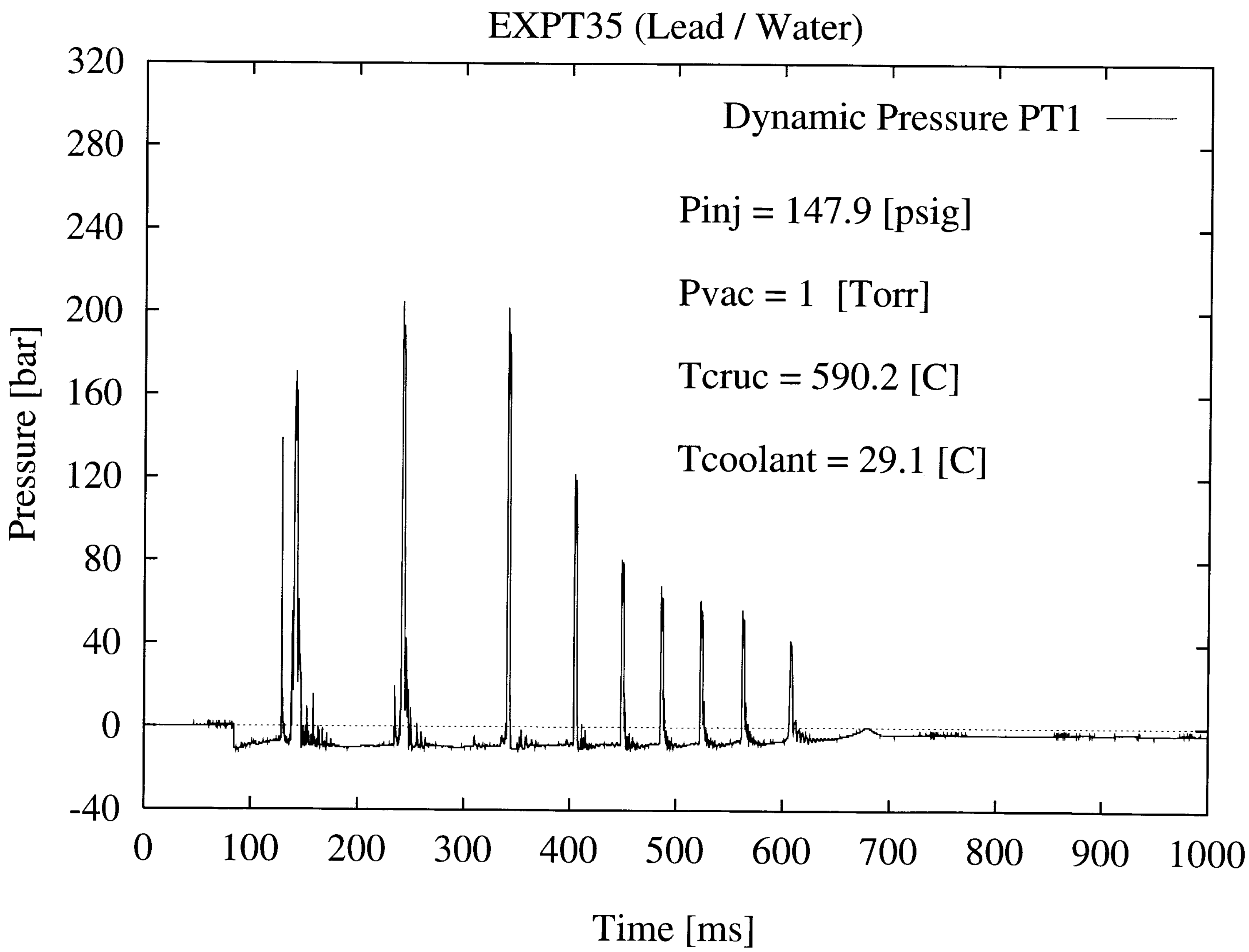


Figure B-3. Complete set of instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C.
c. Transducer PT1, 1000 ms trace.

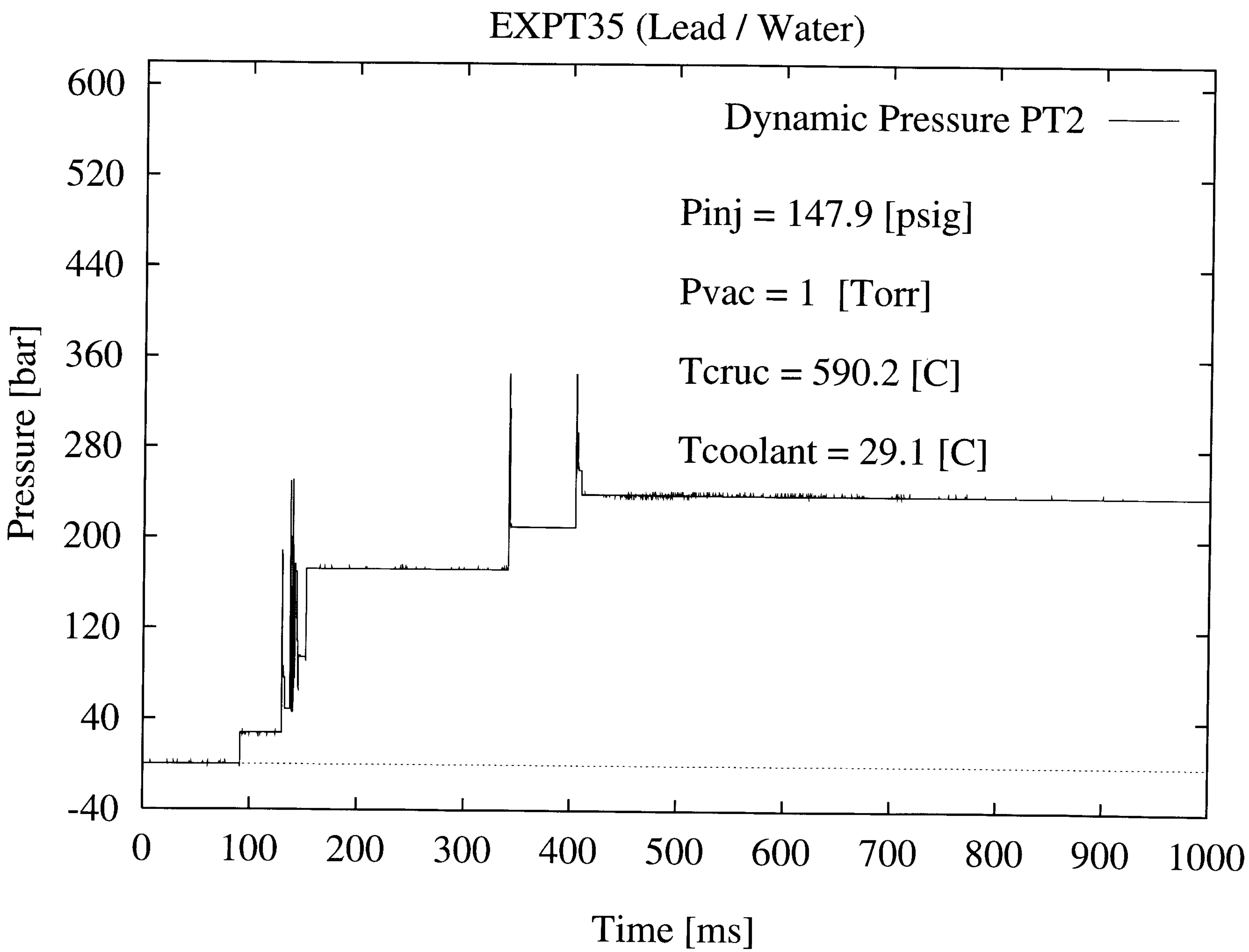


Figure B-3. Complete set of instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C.
d. Transducer PT2, 1000 ms trace.

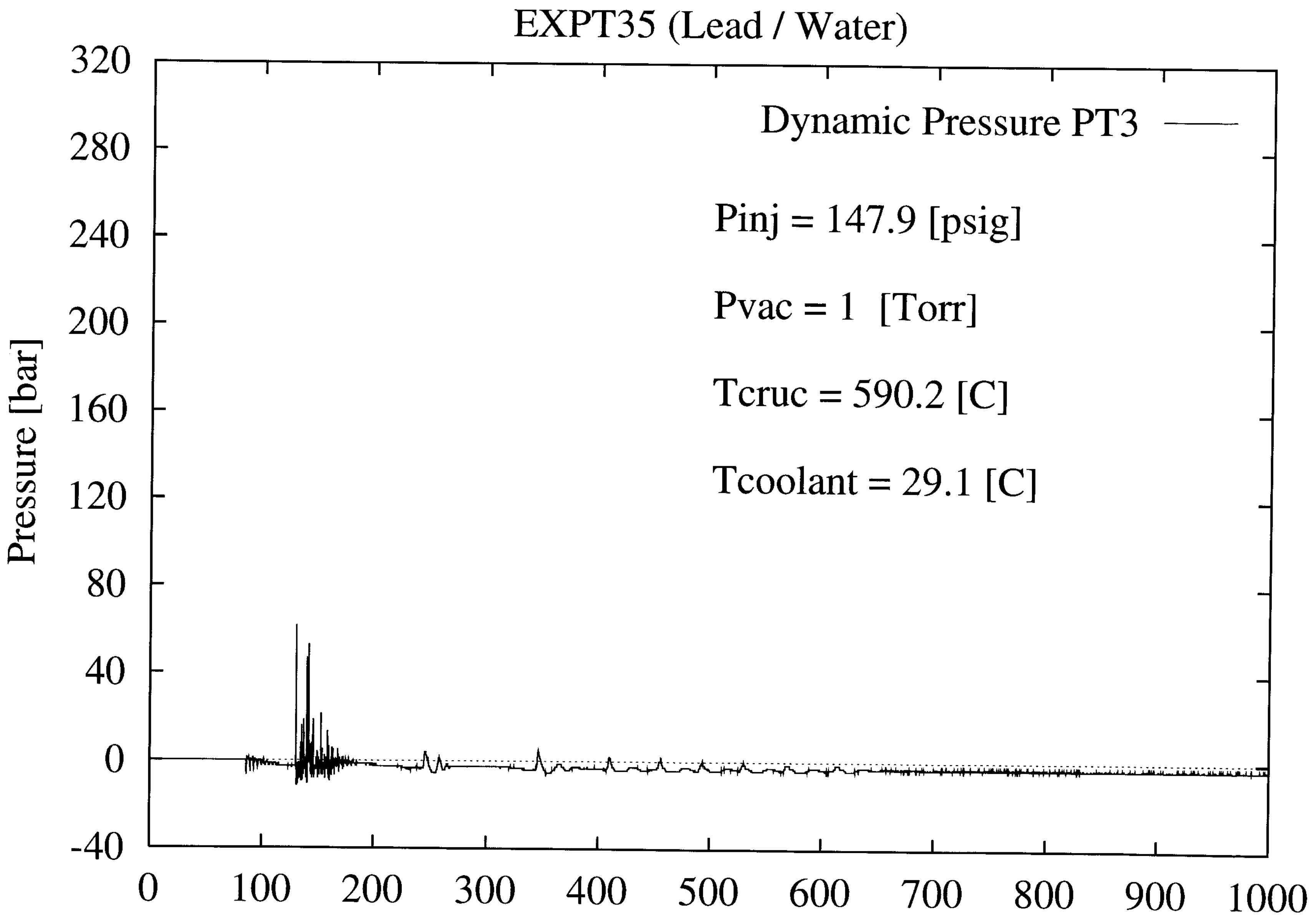


Figure B-3. Complete set of instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C.
e. Transducer PT3, 1000 ms trace.

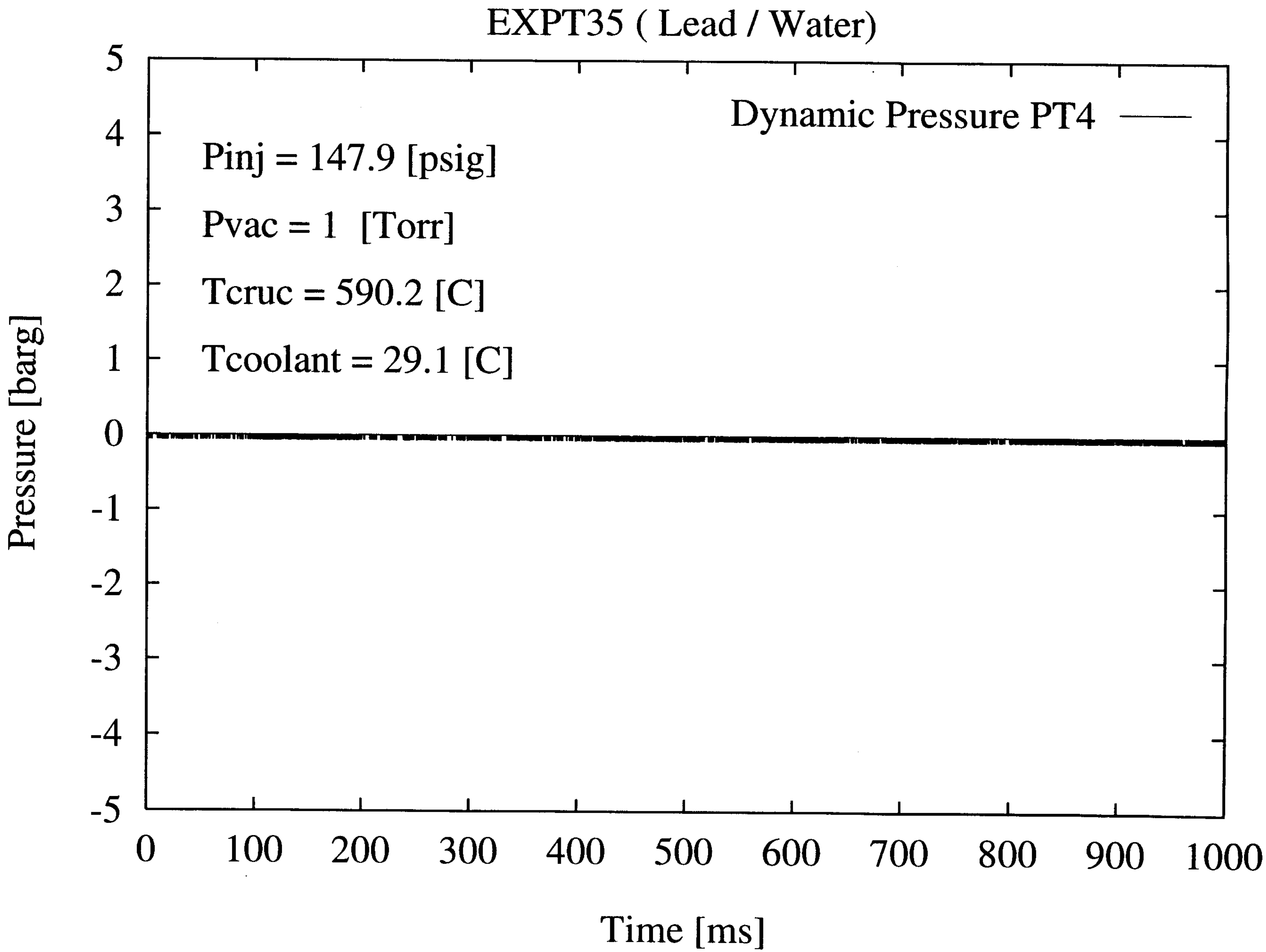


Figure B-3. Complete set of instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C
f. Transducer PT4, 1000 ms trace.

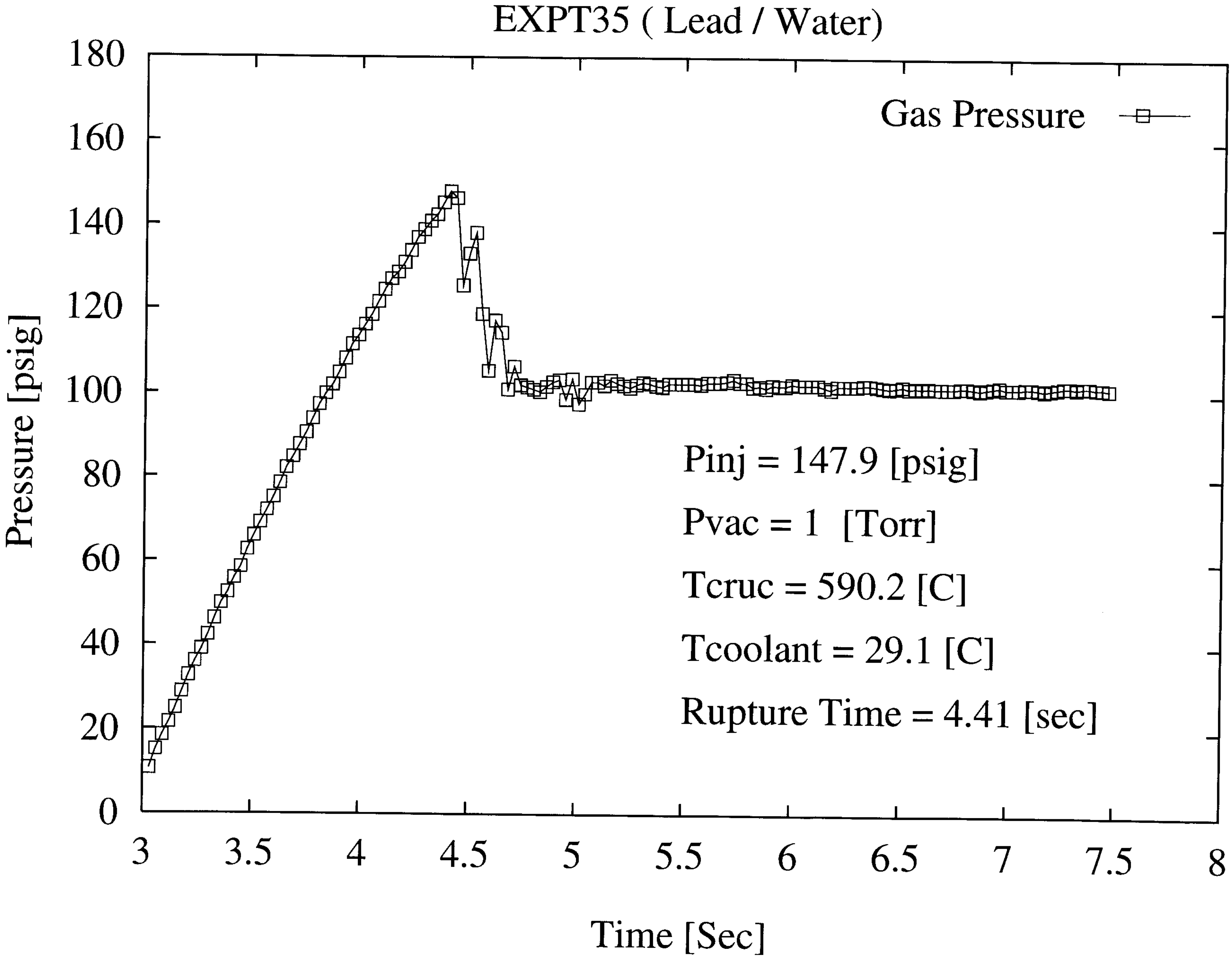


Figure B-3 Complete set of instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C g. Strain gauge PT5, 5 s trace.

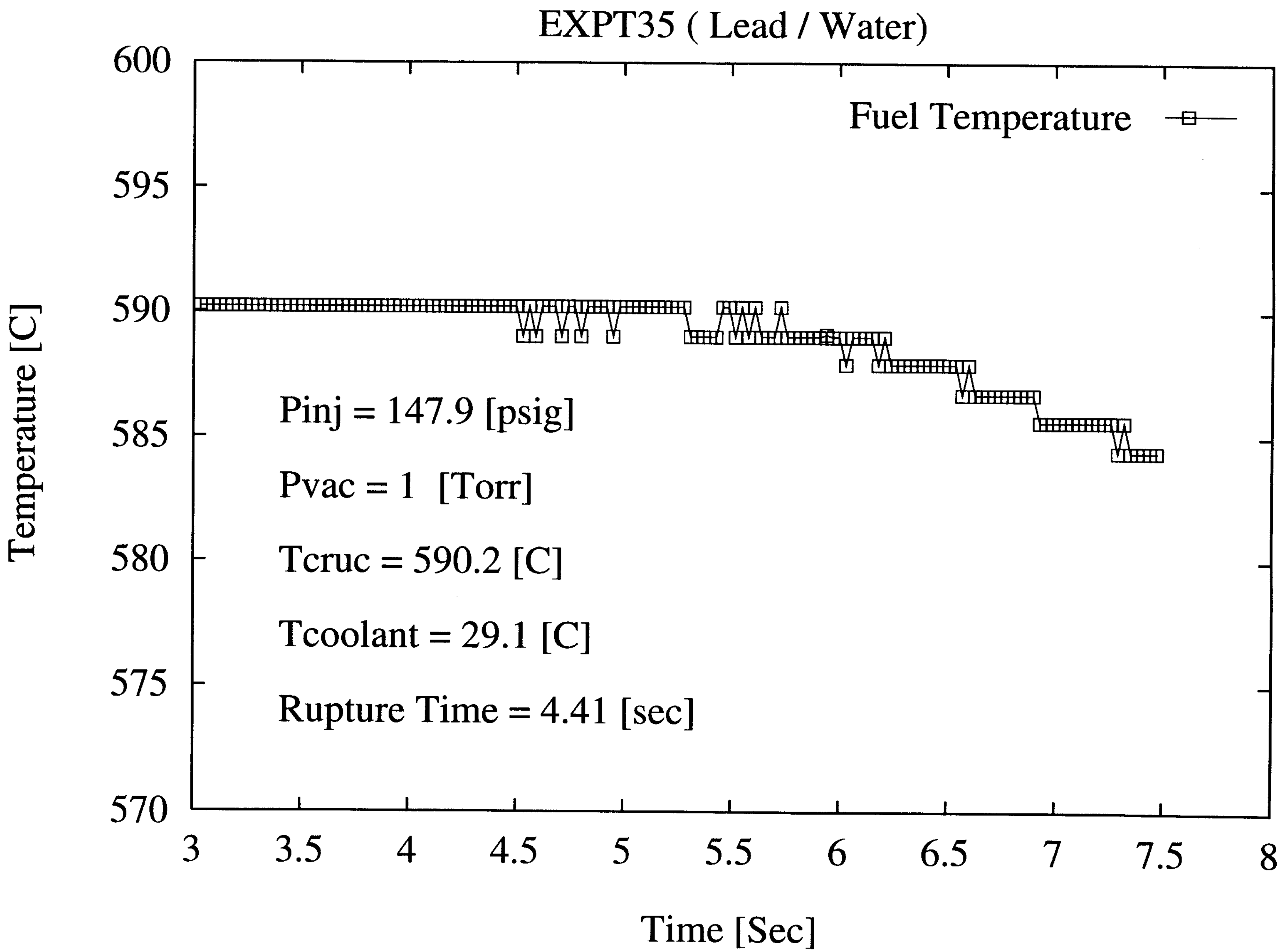


Figure B-3 Complete set of instrumental records from experiment B-35-1. Crucible contained molten Pb at 590.2°C; water was at 29.1°C
h. Thermocouple TC5, 5 s trace.

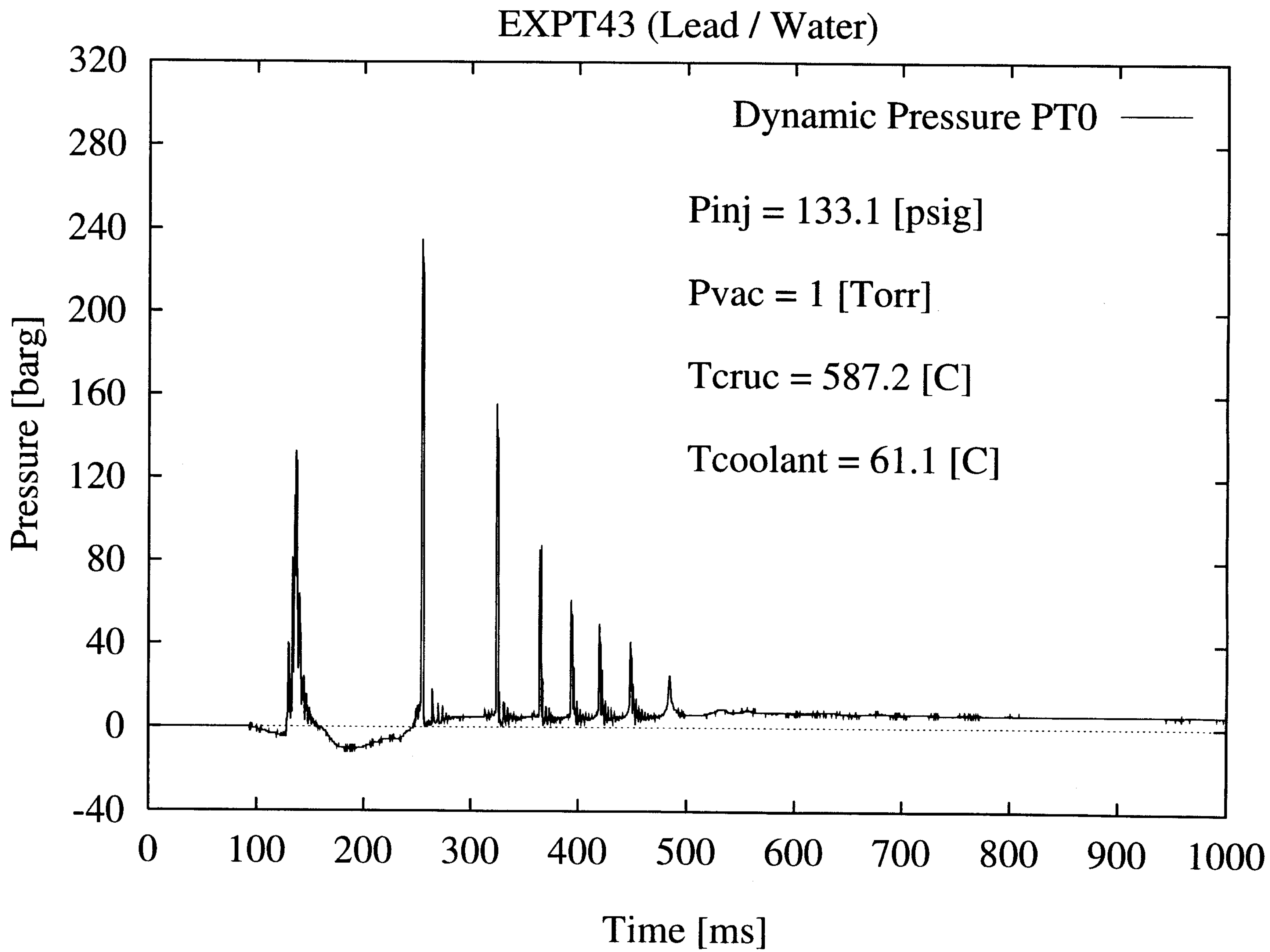


Figure B-4. Complete set of instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C.
a. Transducer PT0,1000 ms trace.

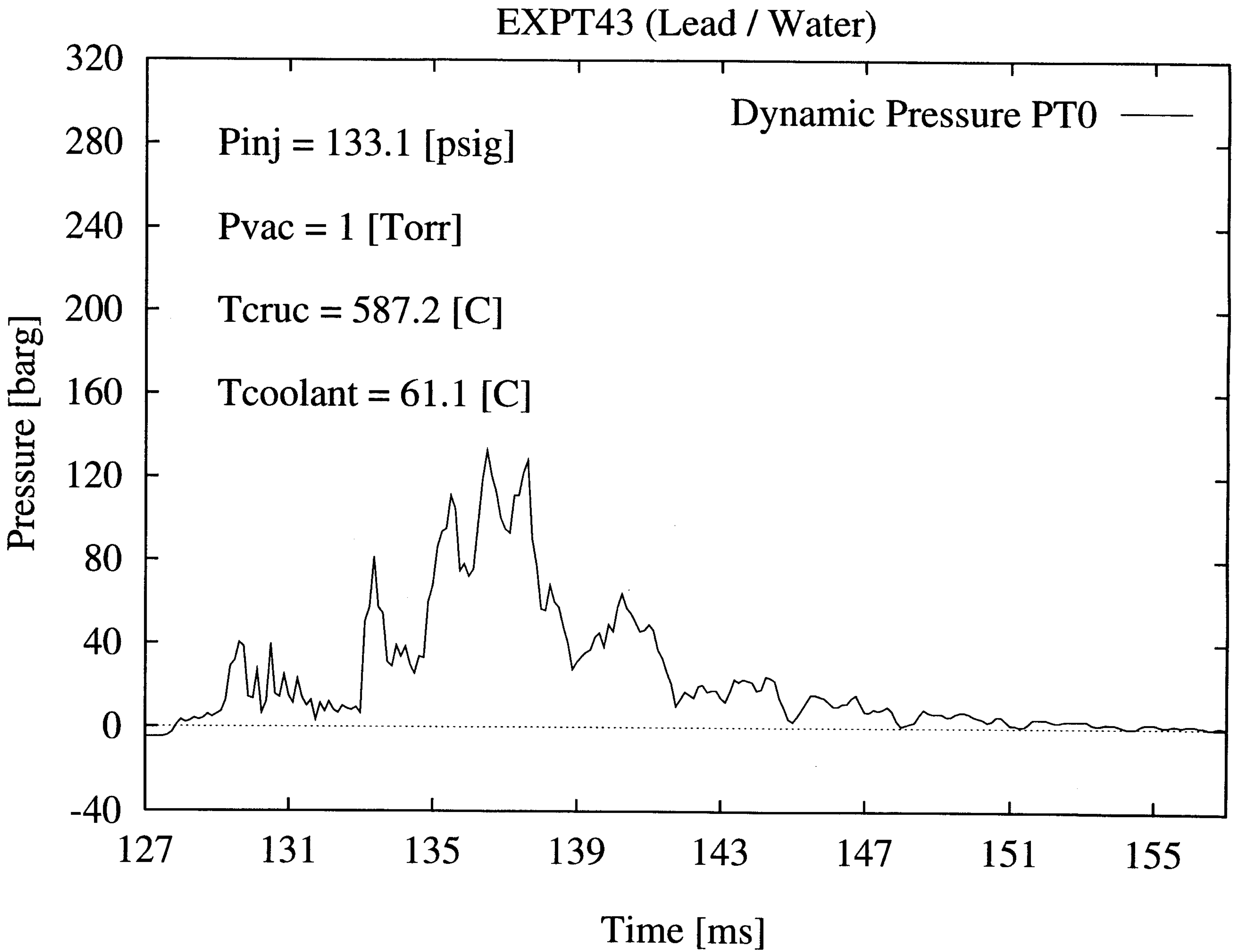


Figure B-4. Complete set of instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C.
b. Transducer PT0, expanded time scale.

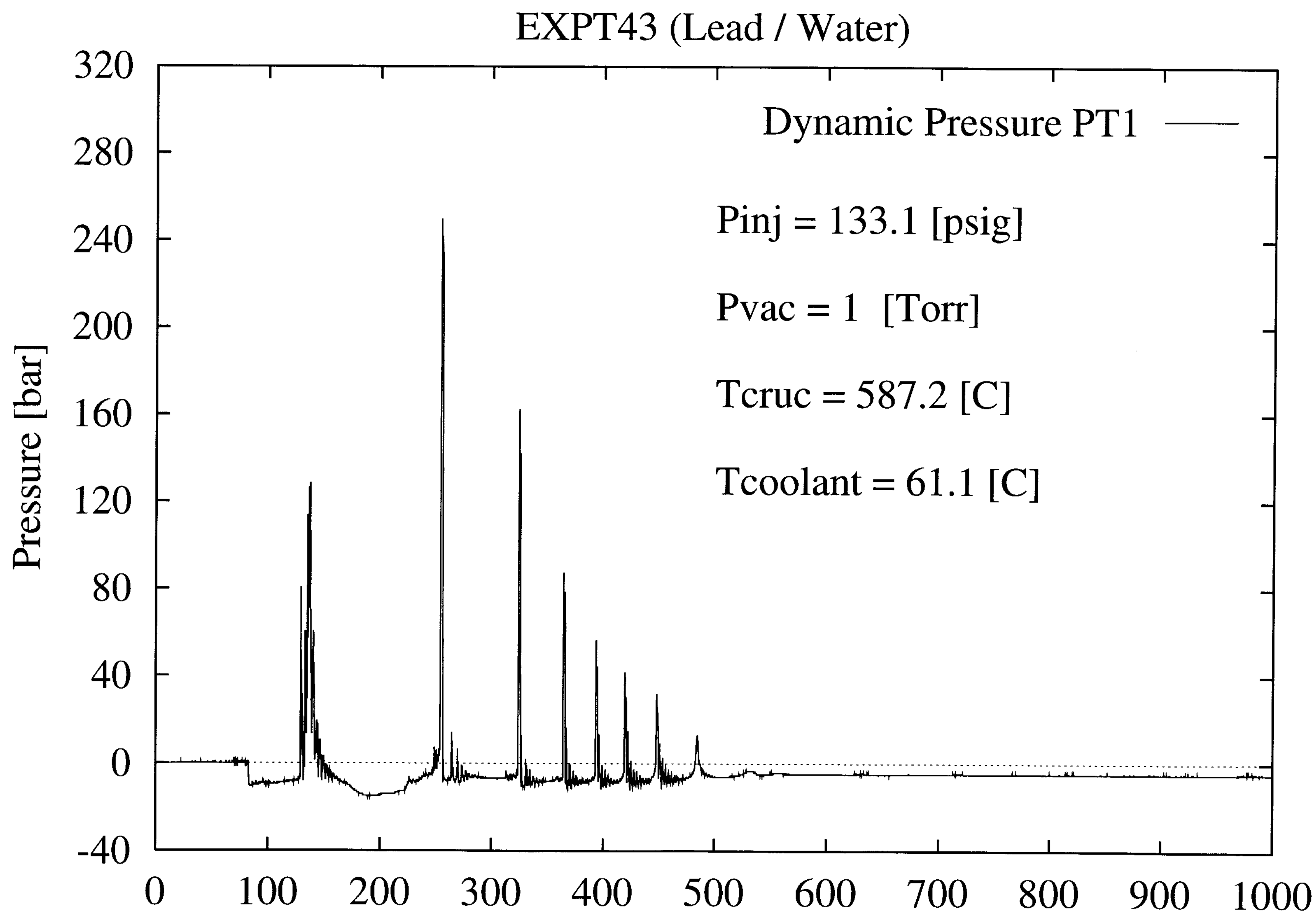


Figure B-4. Complete set of instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C.
c. Transducer PT1, 1000 ms trace.

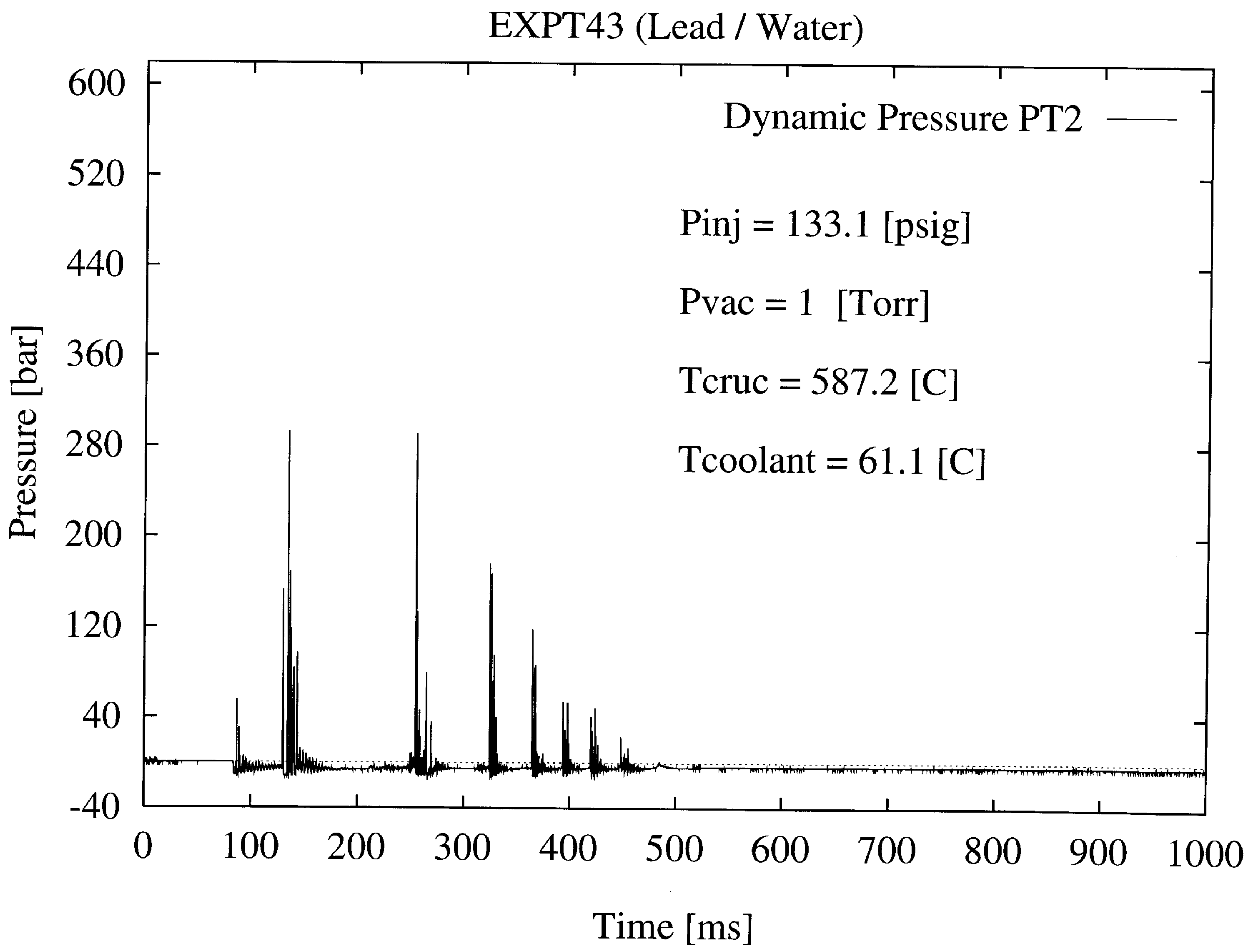


Figure B-4. Complete set of instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C.
d. Transducer PT2, 1000 ms trace.

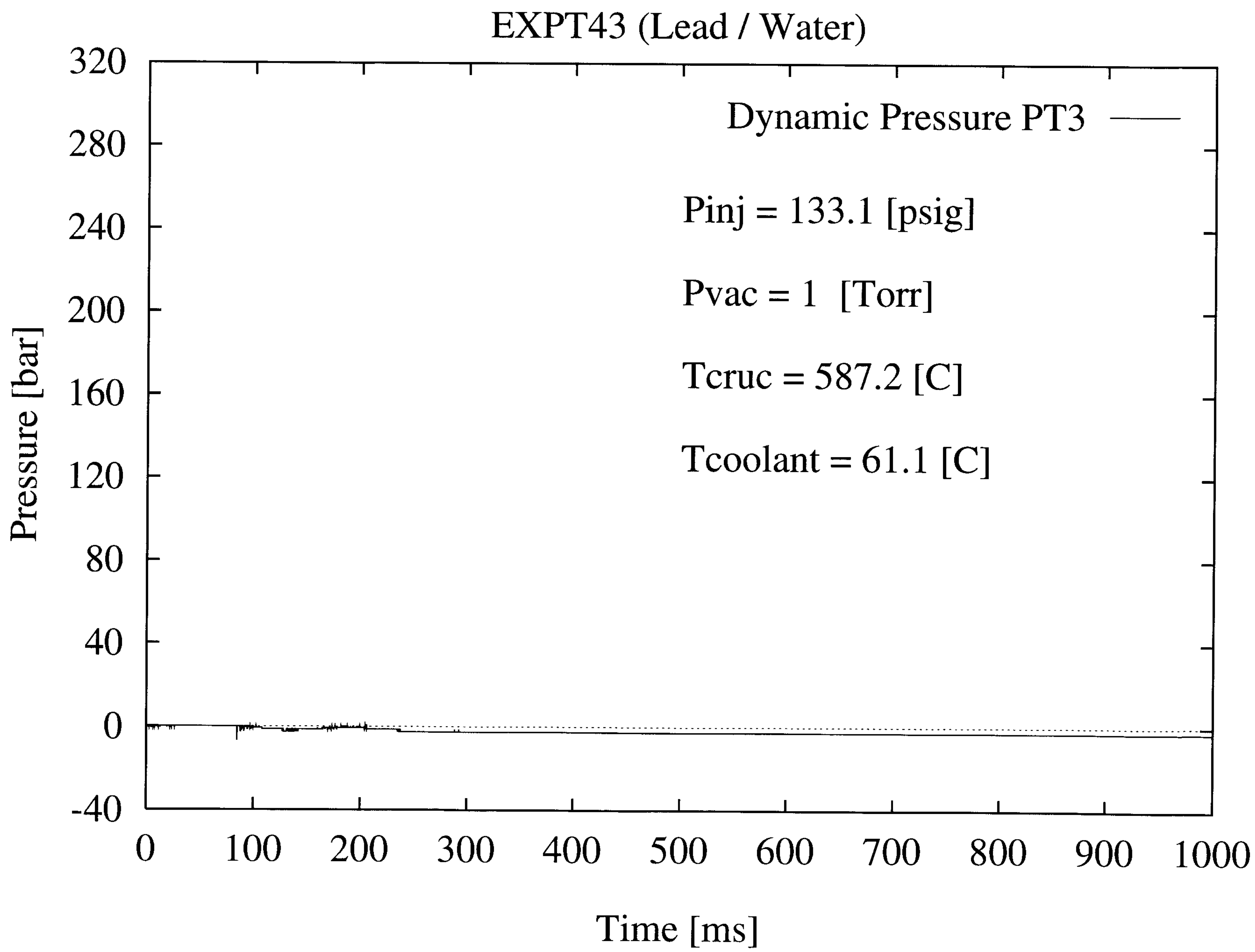


Figure B-4. Complete set of instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C.
e. Transducer PT3, 1000 ms trace.

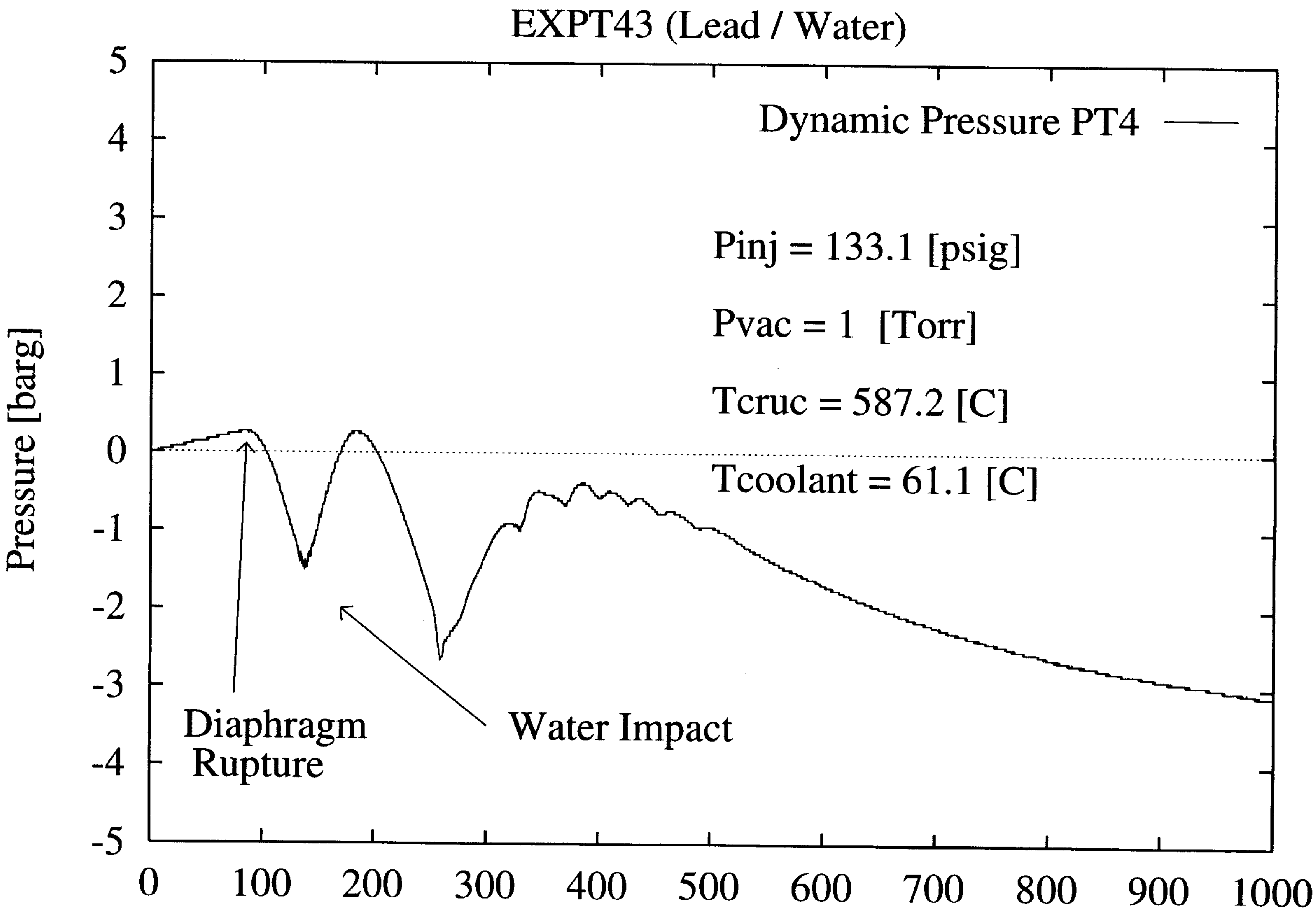


Figure B-4. Complete set of instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C.
f. Transducer PT4, 1000 ms trace.

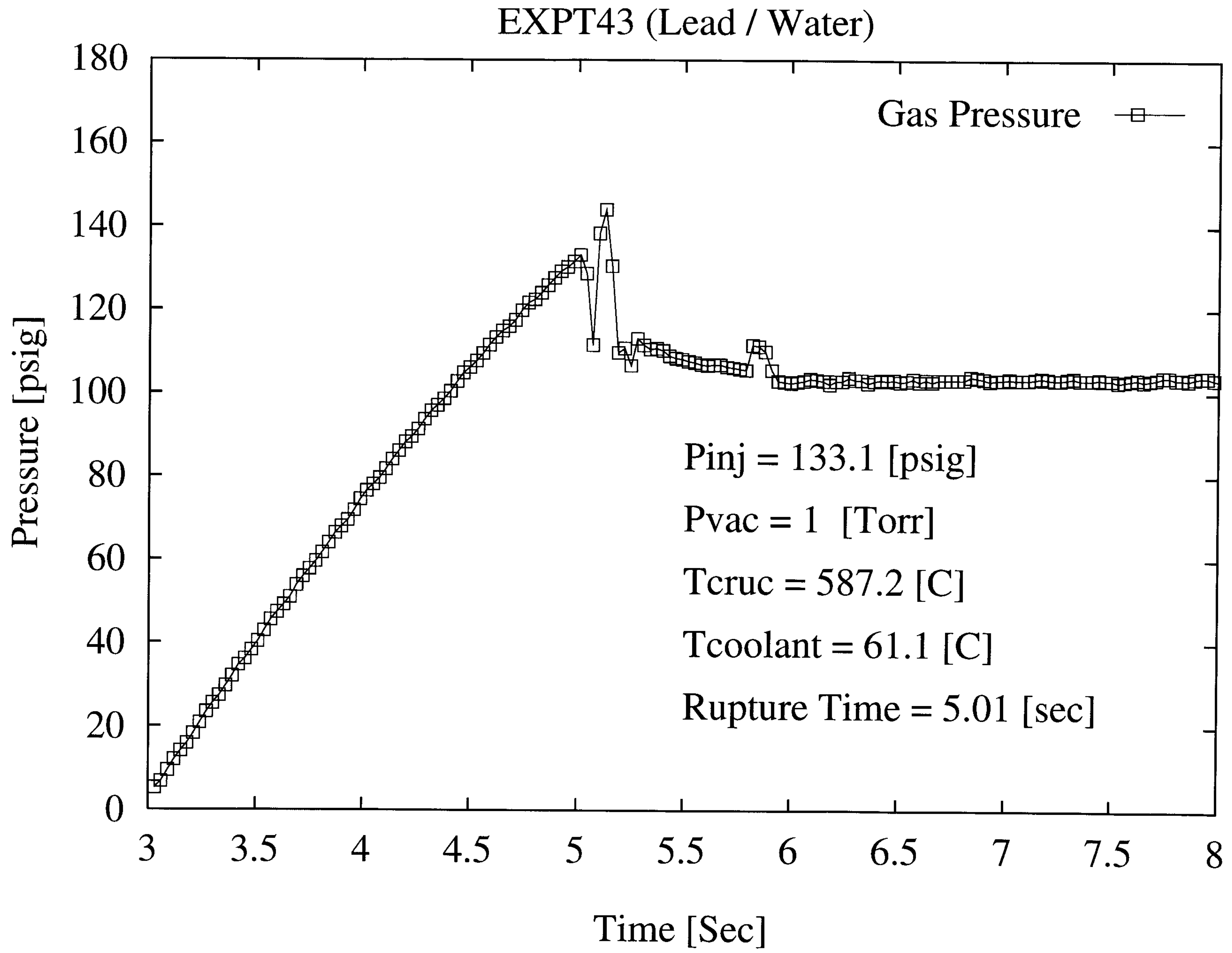


Figure B-4. Complete set of instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C.
g. Strain gauge PT5, 5 s trace.

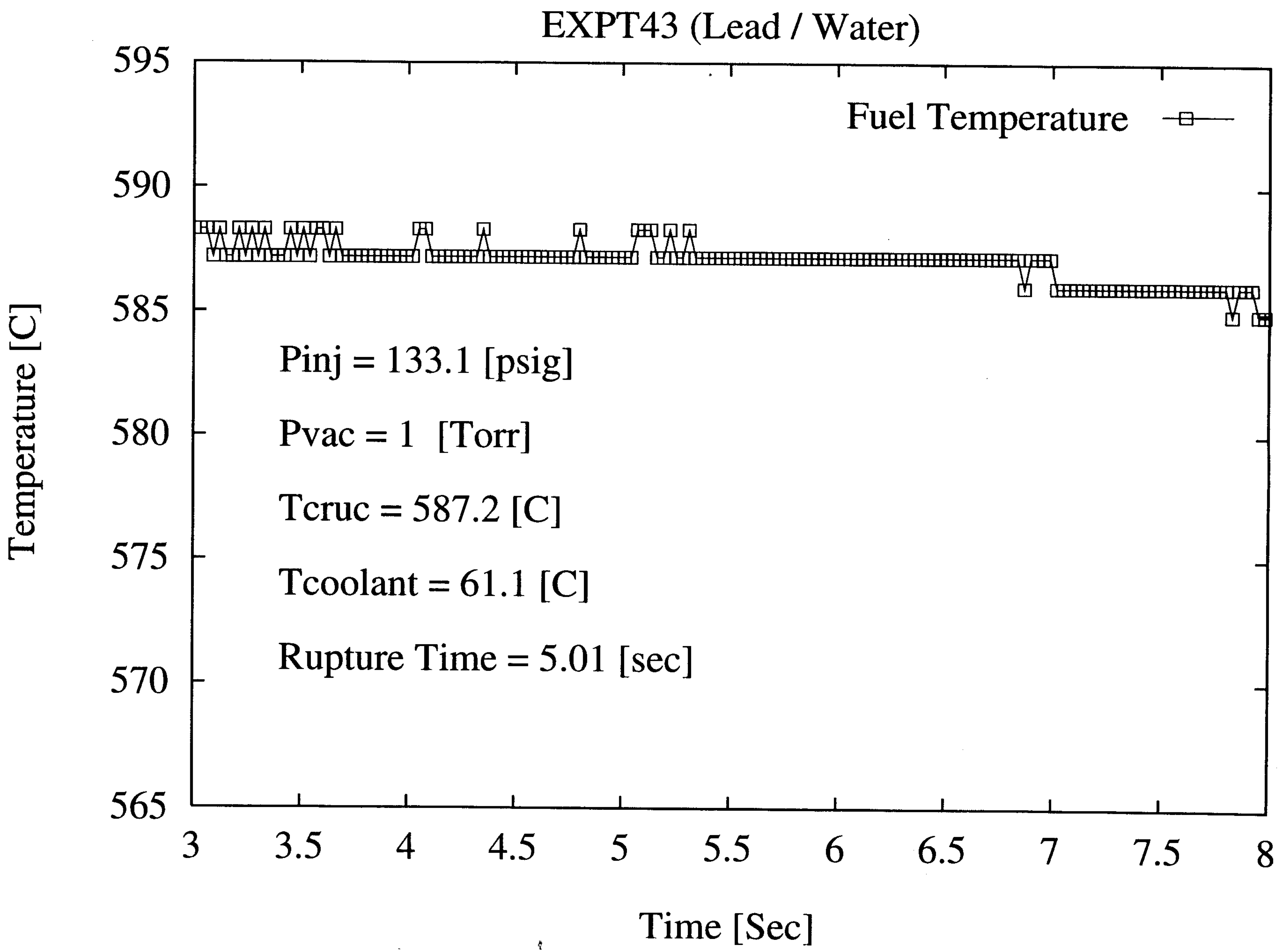


Figure B-4. Complete set of instrumental records from experiment B-43-1. Crucible contained molten Pb at 587.2°C; water was at 61.1°C.
h. Thermocouple TC5, 5 s trace.

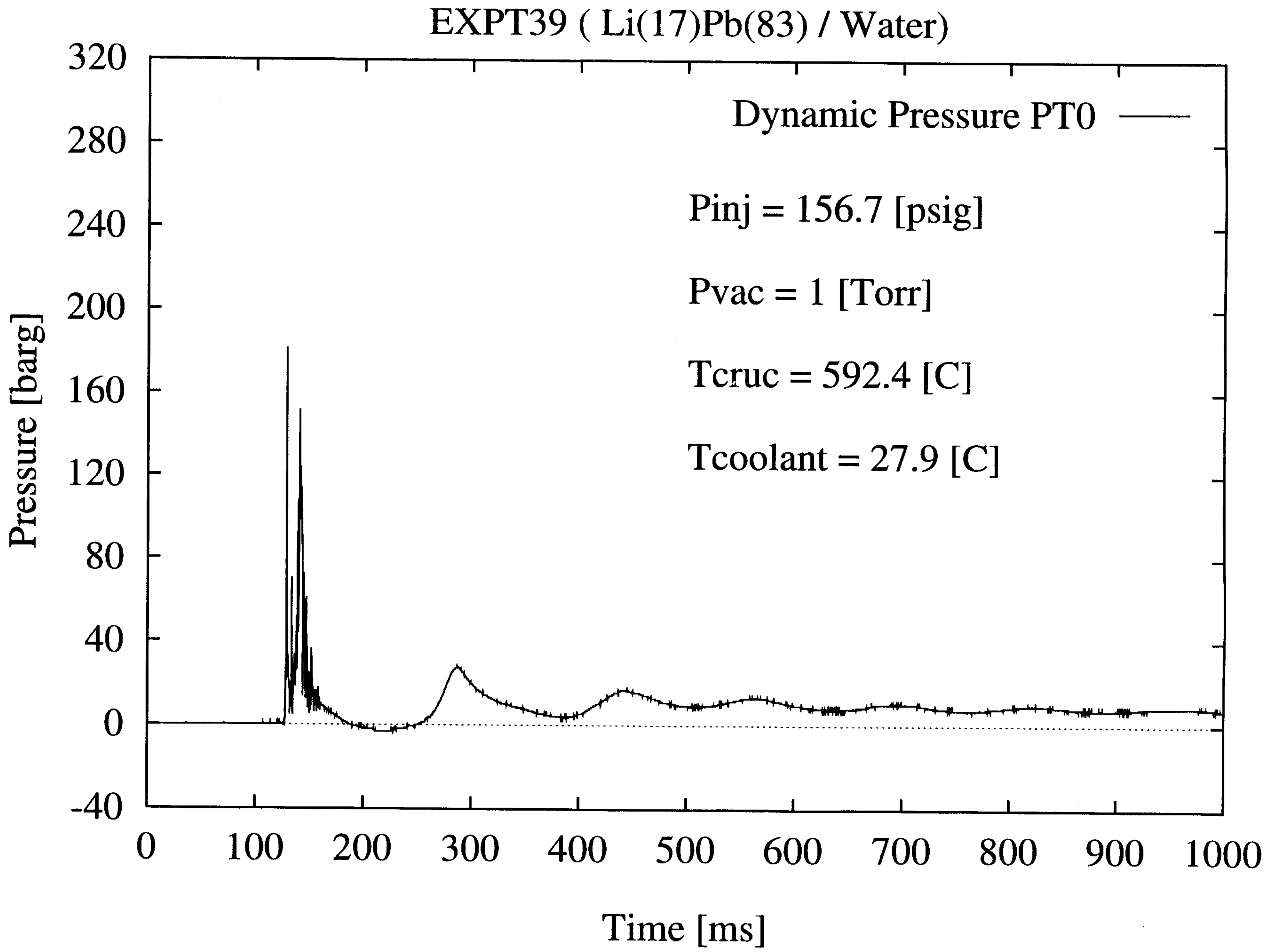


Figure B-5. Complete set of instrumental records from experiment B-39-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 592.4°C; water was at 27.9°C. a. Transducer PT0, 1000 ms trace.

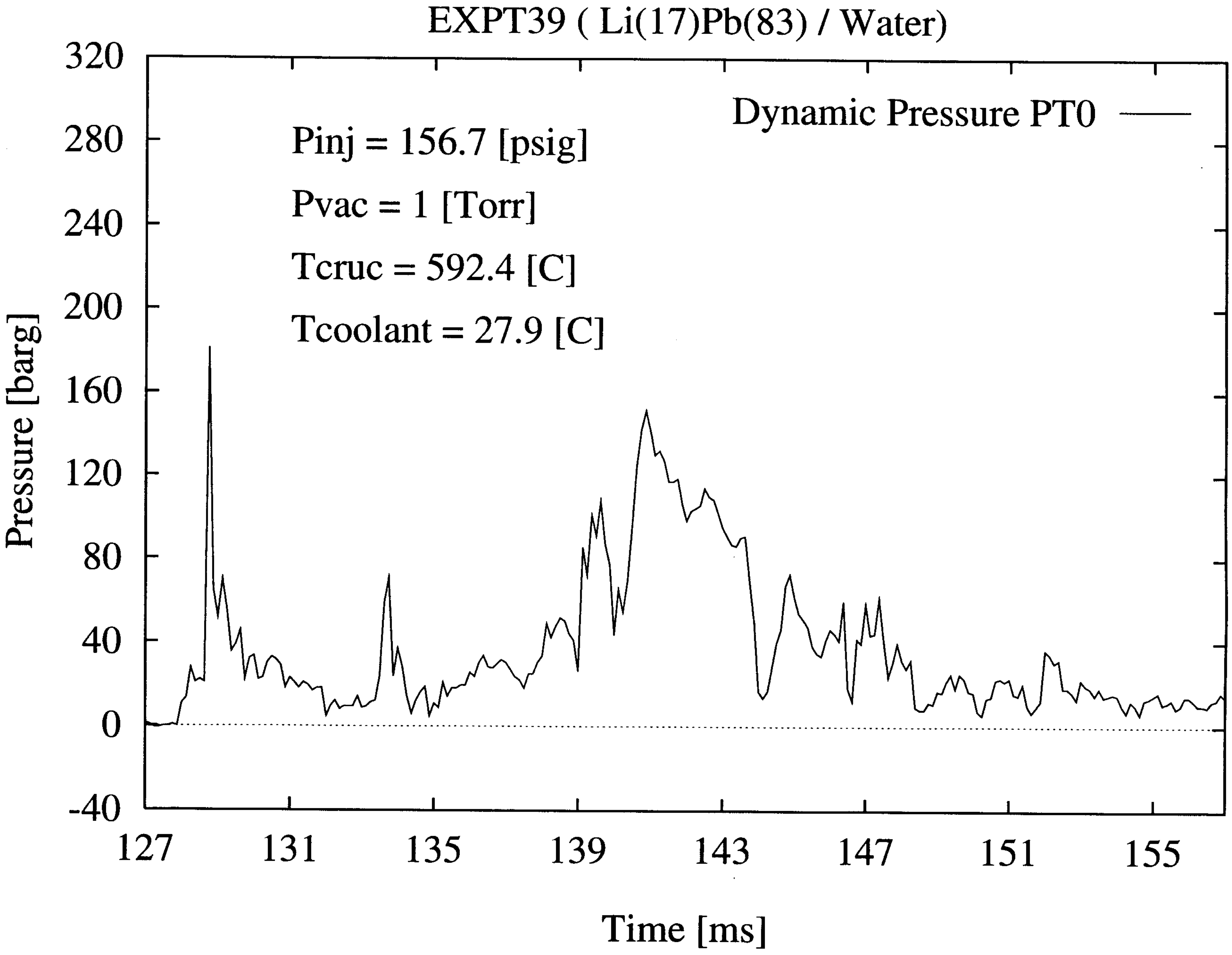


Figure B-5. Complete set of instrumental records from experiment B-39-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 592.4°C; water was at 27.9°C.
b. Transducer PT0, expanded time scale.

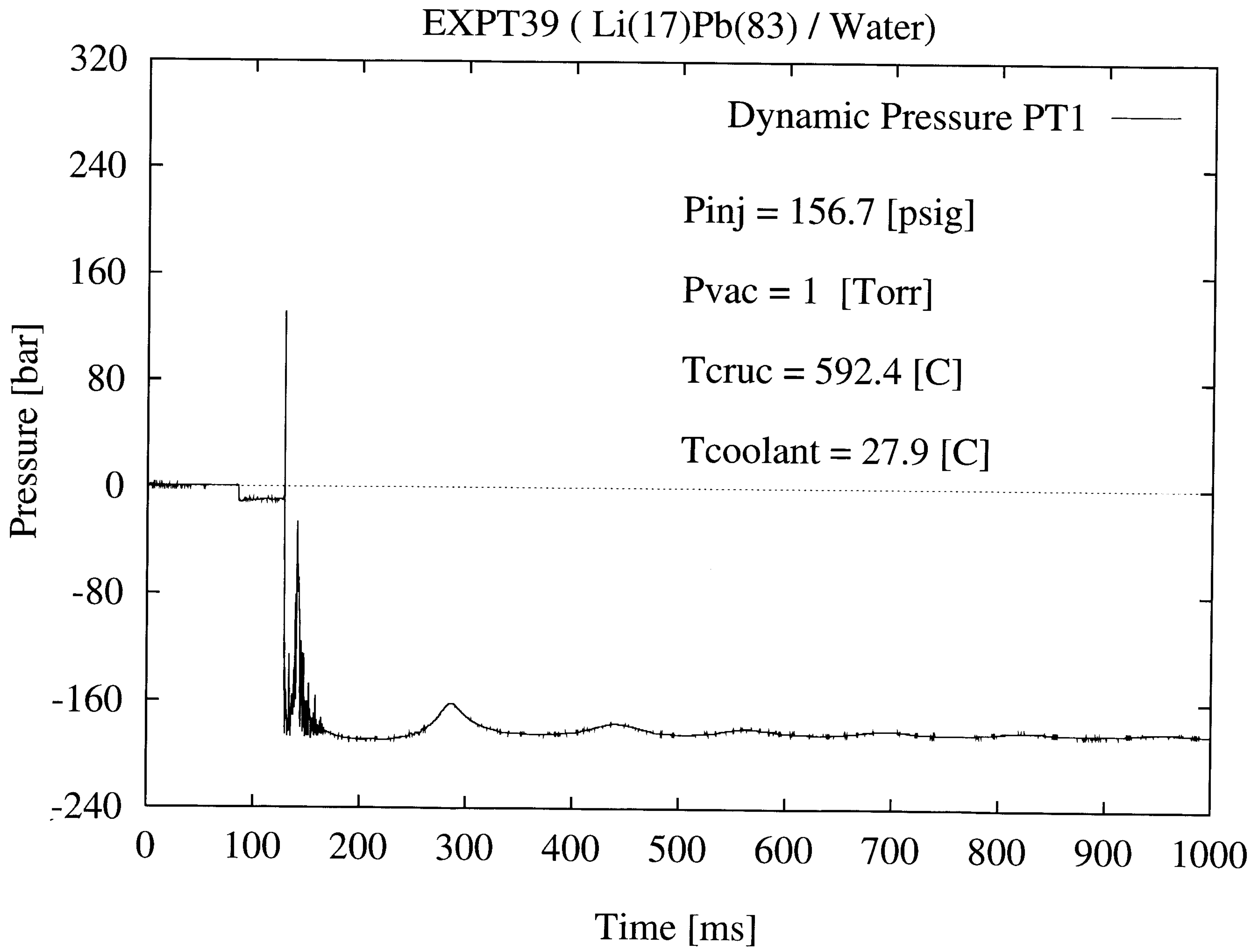


Figure B-5. Complete set of instrumental records from experiment B-39-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 592.4°C; water was at 27.9°C. c. Transducer PT1, 1000 ms trace.

NO USEFUL RECORD

Figure B-5. Complete set of instrumental records from experiment B-39-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 592.4°C; water was at 27.9°C. d. Transducer PT2, 1000 ms trace.

NO USEFUL RECORD

Figure B-5. Complete set of instrumental records from experiment B-39-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 592.4°C; water was at 27.9°C. e. Transducer PT3, 1000 ms trace.

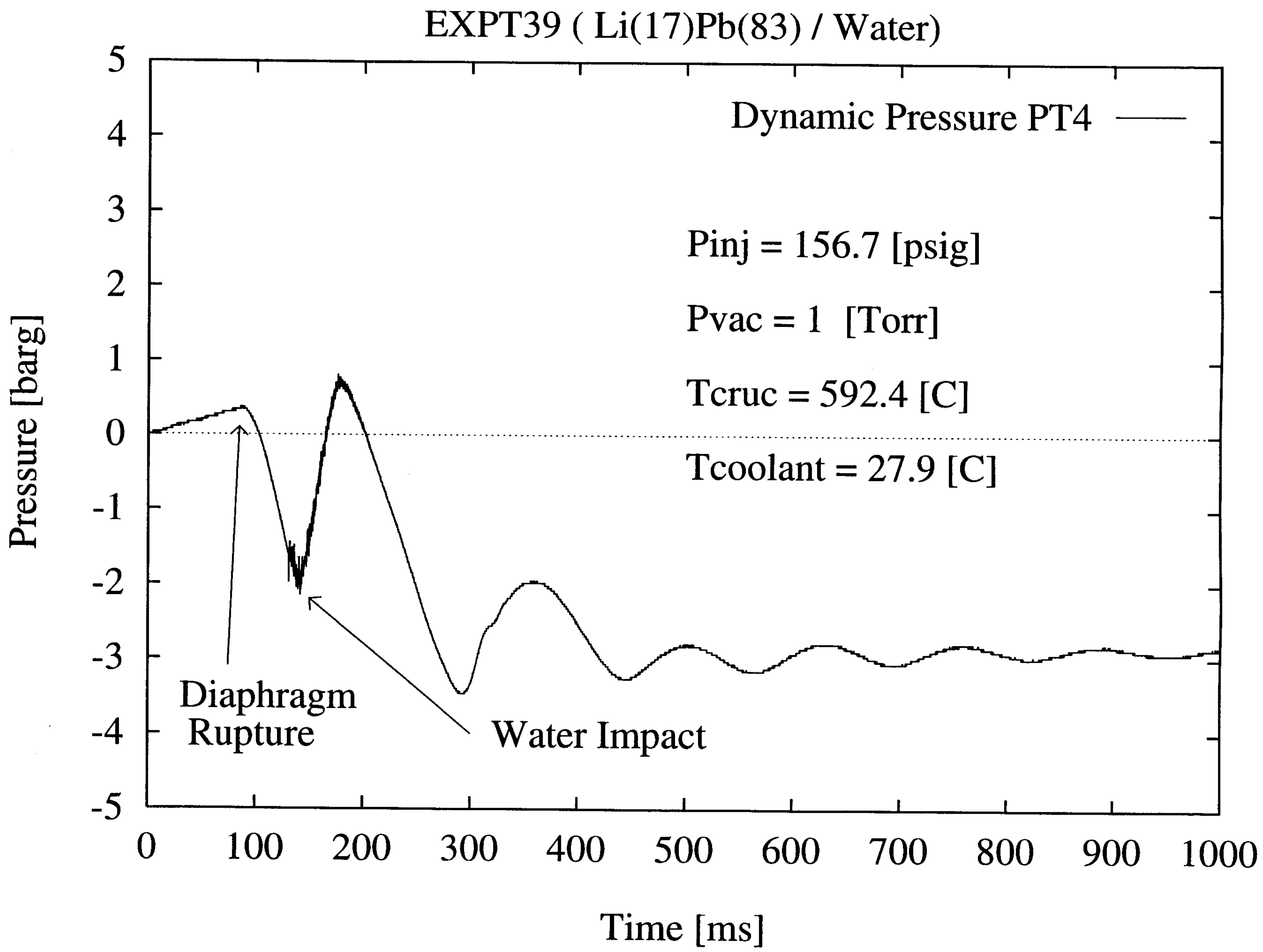


Figure B-5. Complete set of instrumental records from experiment B-39-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 592.4°C; water was at 27.9°C. f. Transducer PT4, 1000 ms trace.

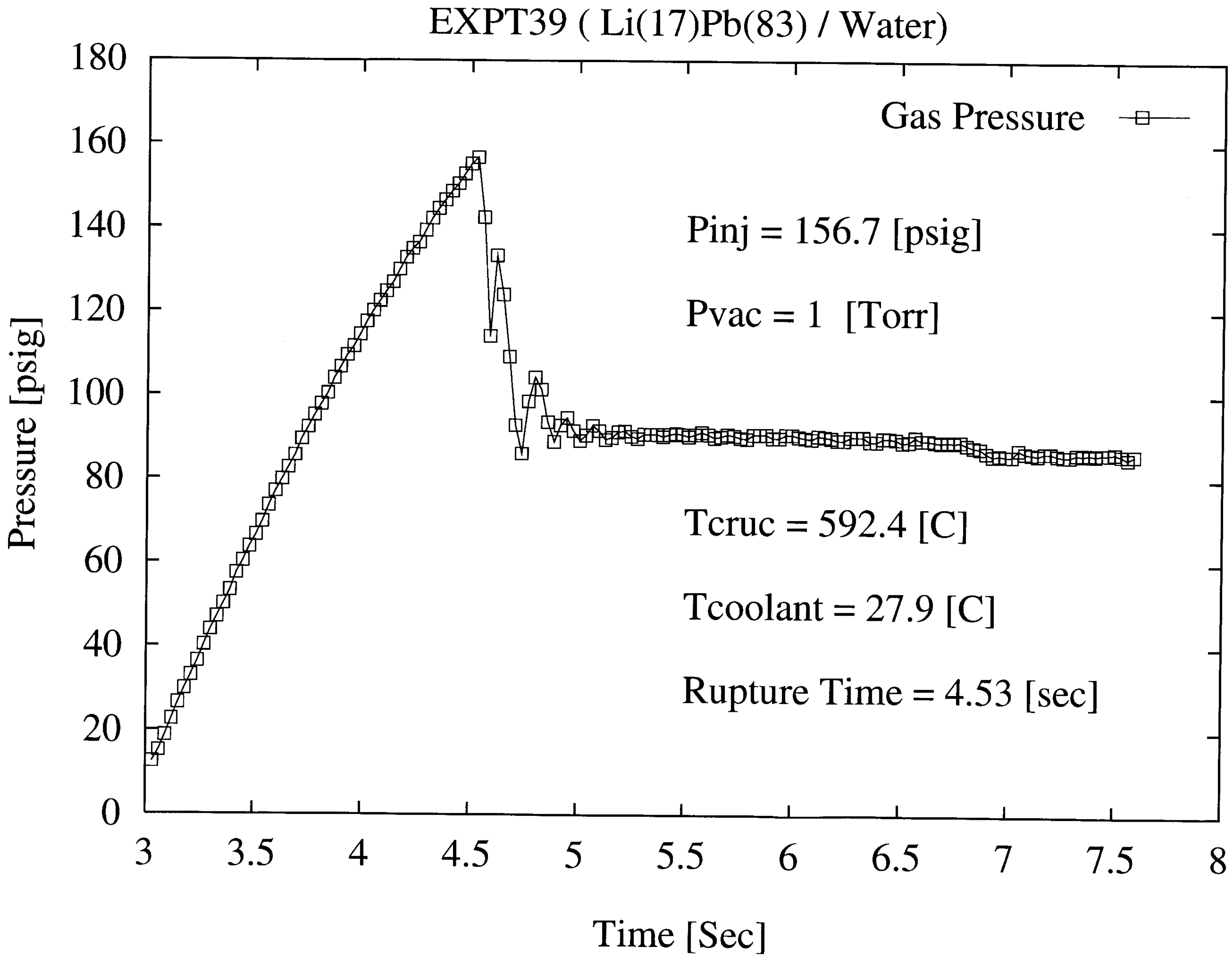


Figure B-5. Complete set of instrumental records from experiment B-39-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 592.4°C; water was at 27.9°C. g. Strain gauge PT5, 5 s trace.

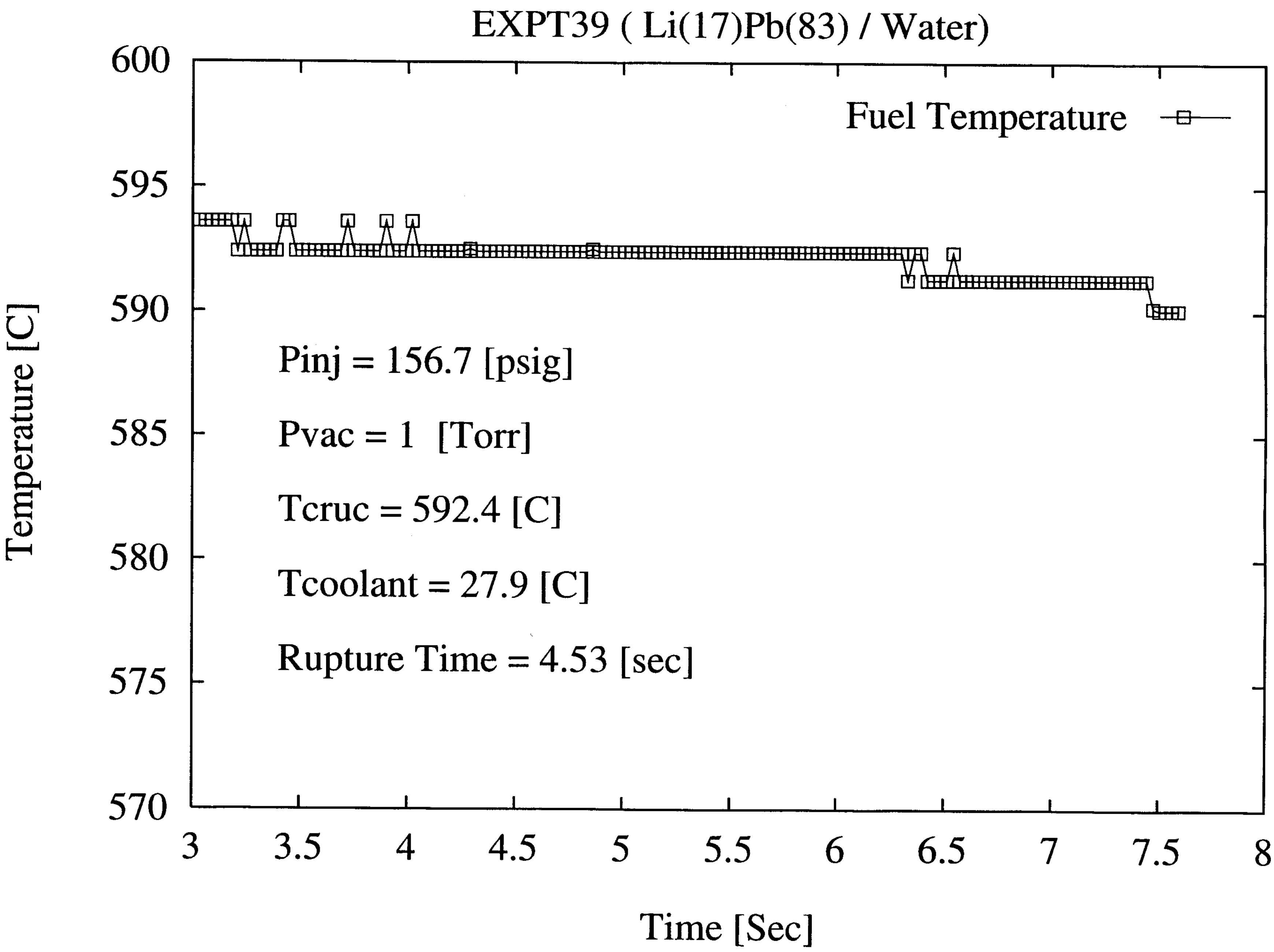


Figure B-5. Complete set of instrumental records from experiment B-39-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 592.4°C; water was at 27.9°C. h. Thermocouple TC5, 5 s trace.

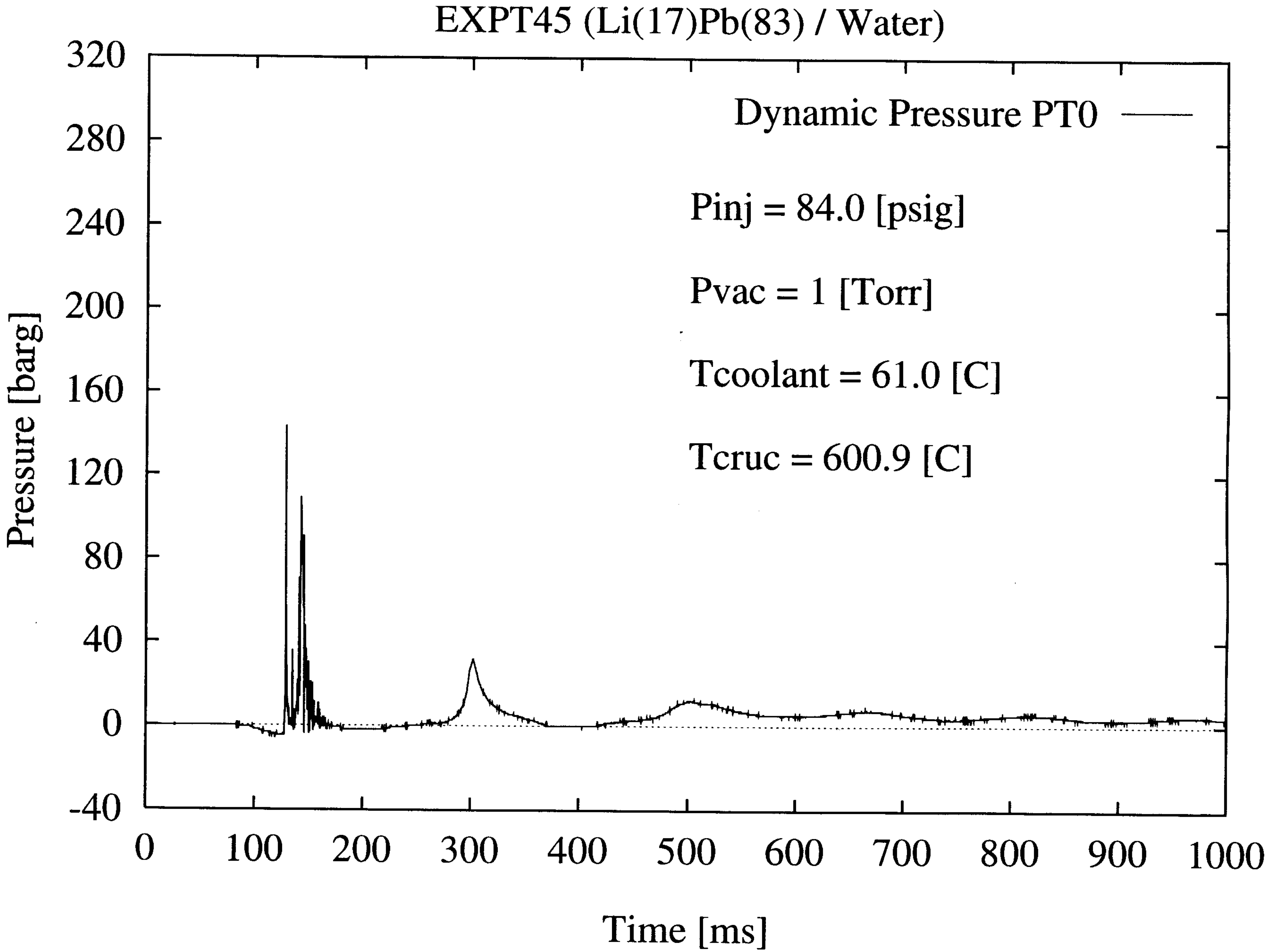


Figure B-6. Complete set of instrumental records from experiment B-45-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 600.9°C; water was at 61.0°C.
a. Transducer PT0, 1000 ms trace.

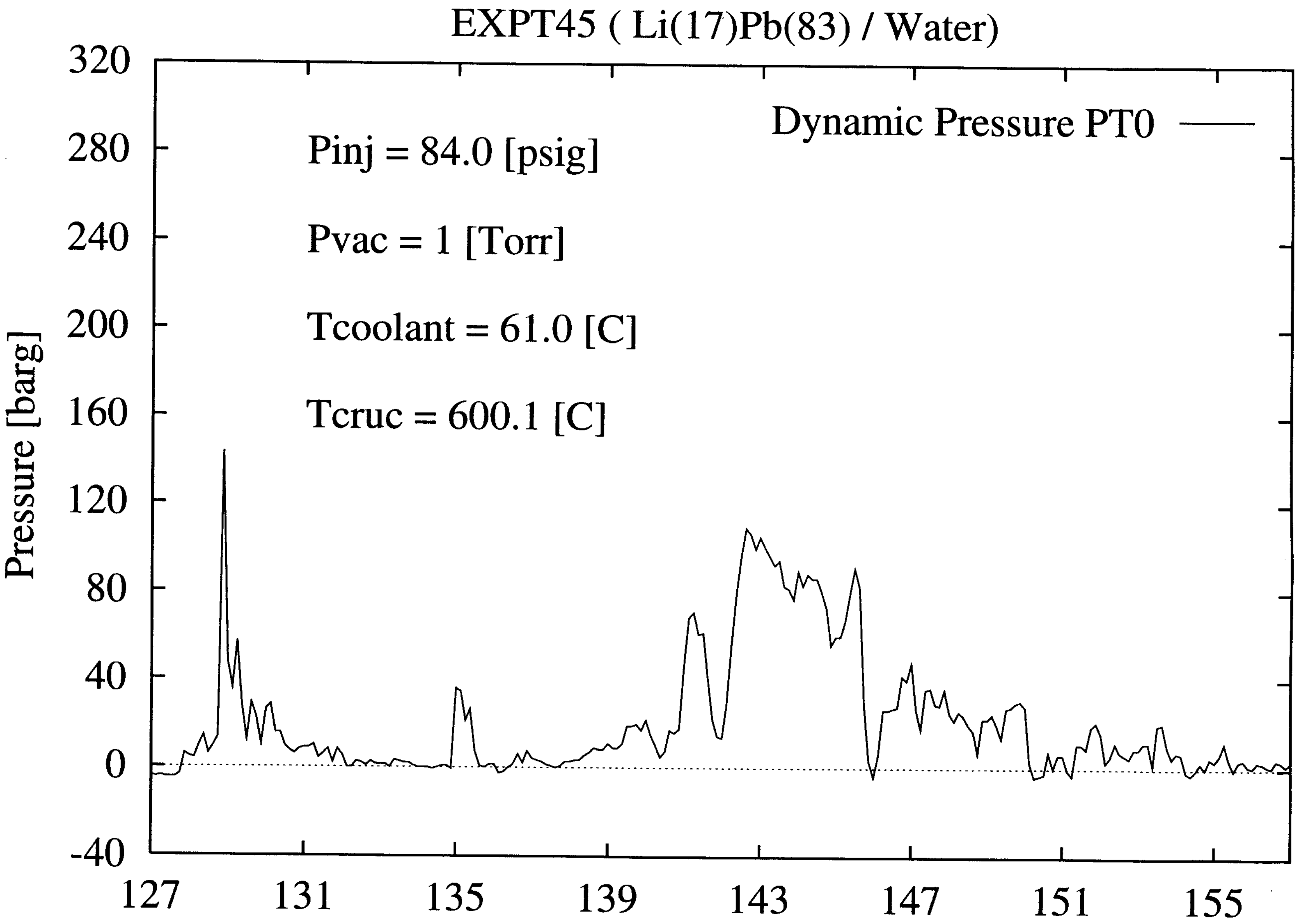


Figure B-6. Complete set of instrumental records from experiment B-45-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 600.9°C; water was at 61.0°C.
b. Transducer PT0, expanded time scale.

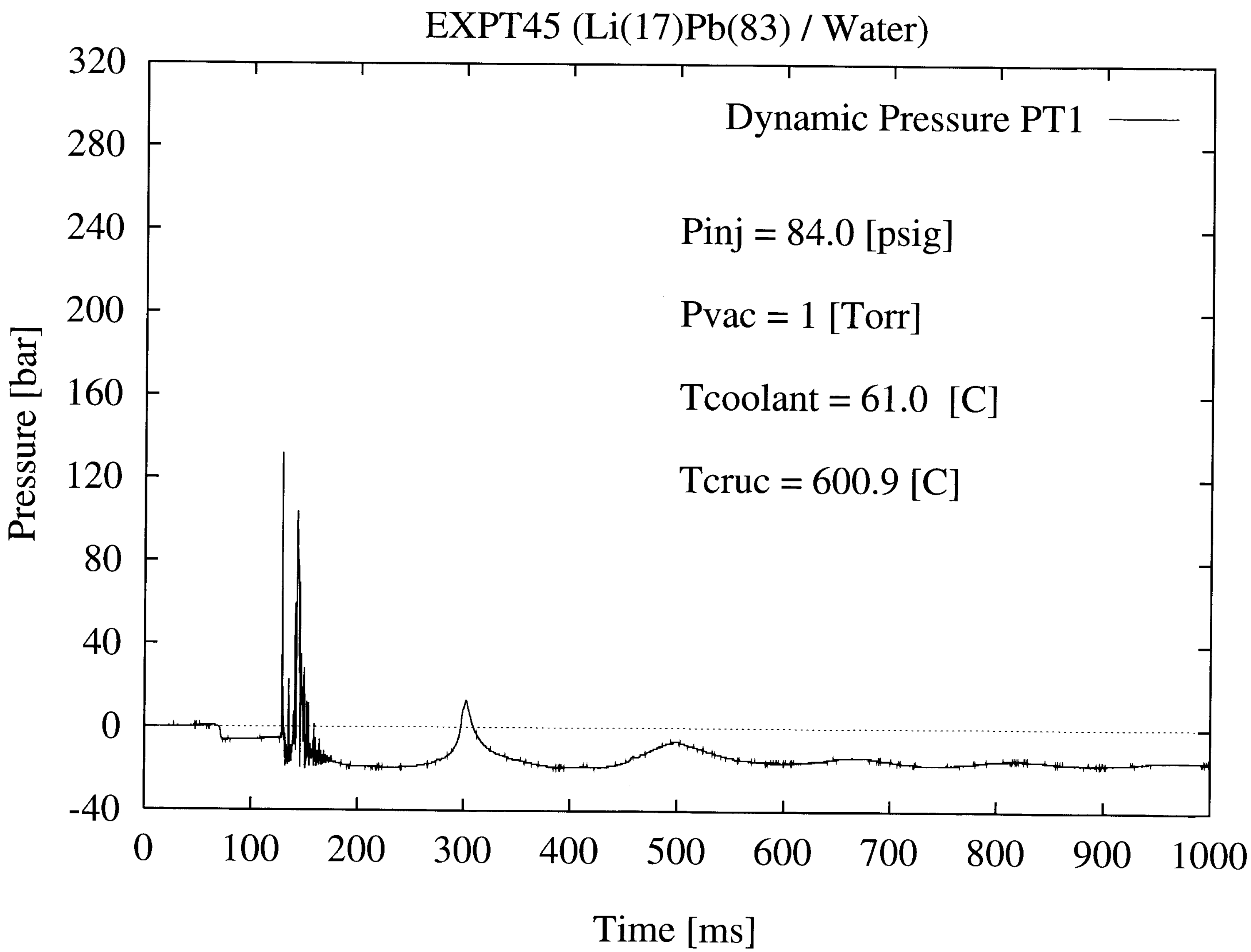


Figure B-6. Complete set of instrumental records from experiment B-45-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 600.9°C; water was at 61.0°C. c. Transducer PT1, 1000 ms trace.

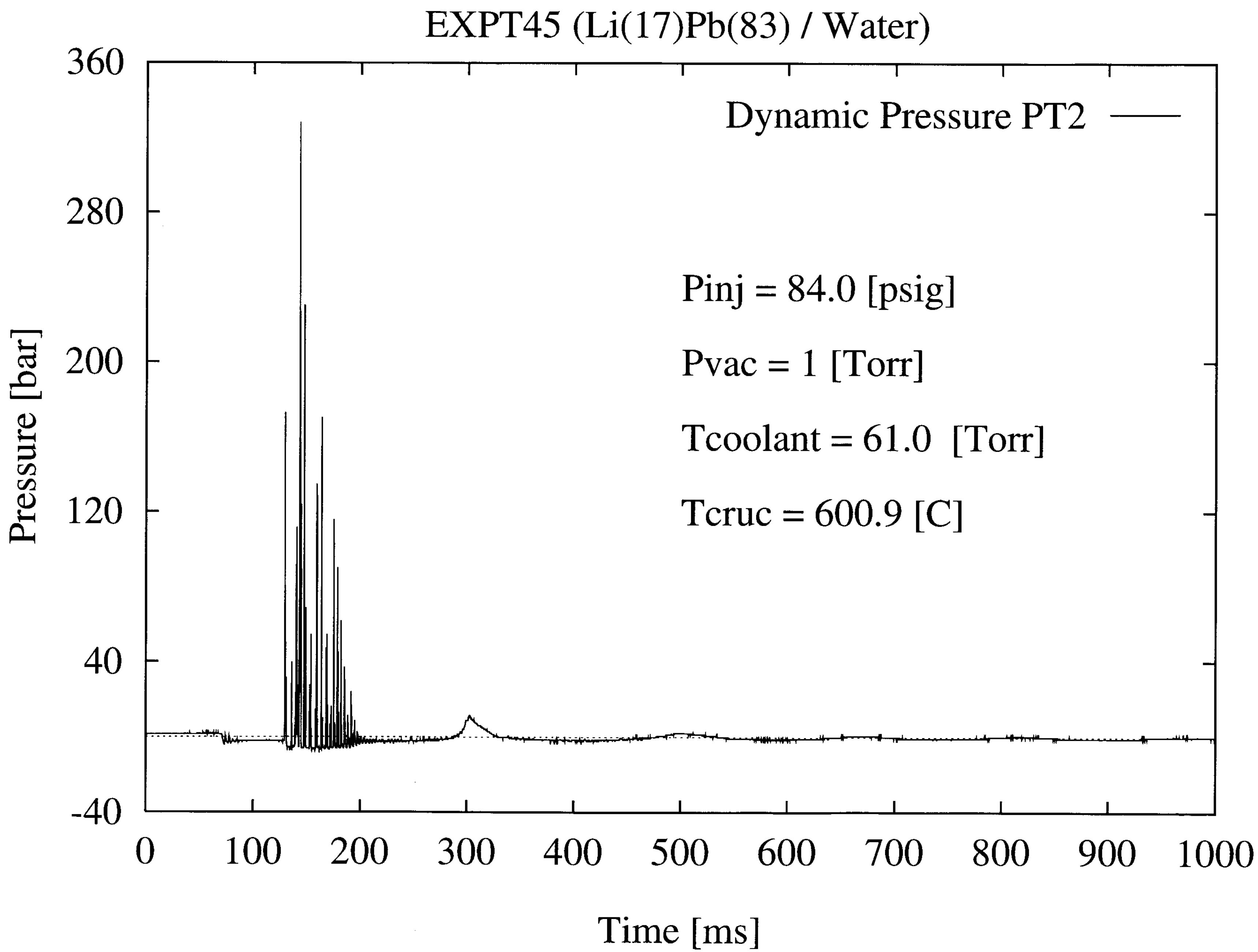


Figure B-6. Complete set of instrumental records from experiment B-45-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 600.9°C; water was at 61.0°C. d. Transducer PT2, 1000 ms trace.

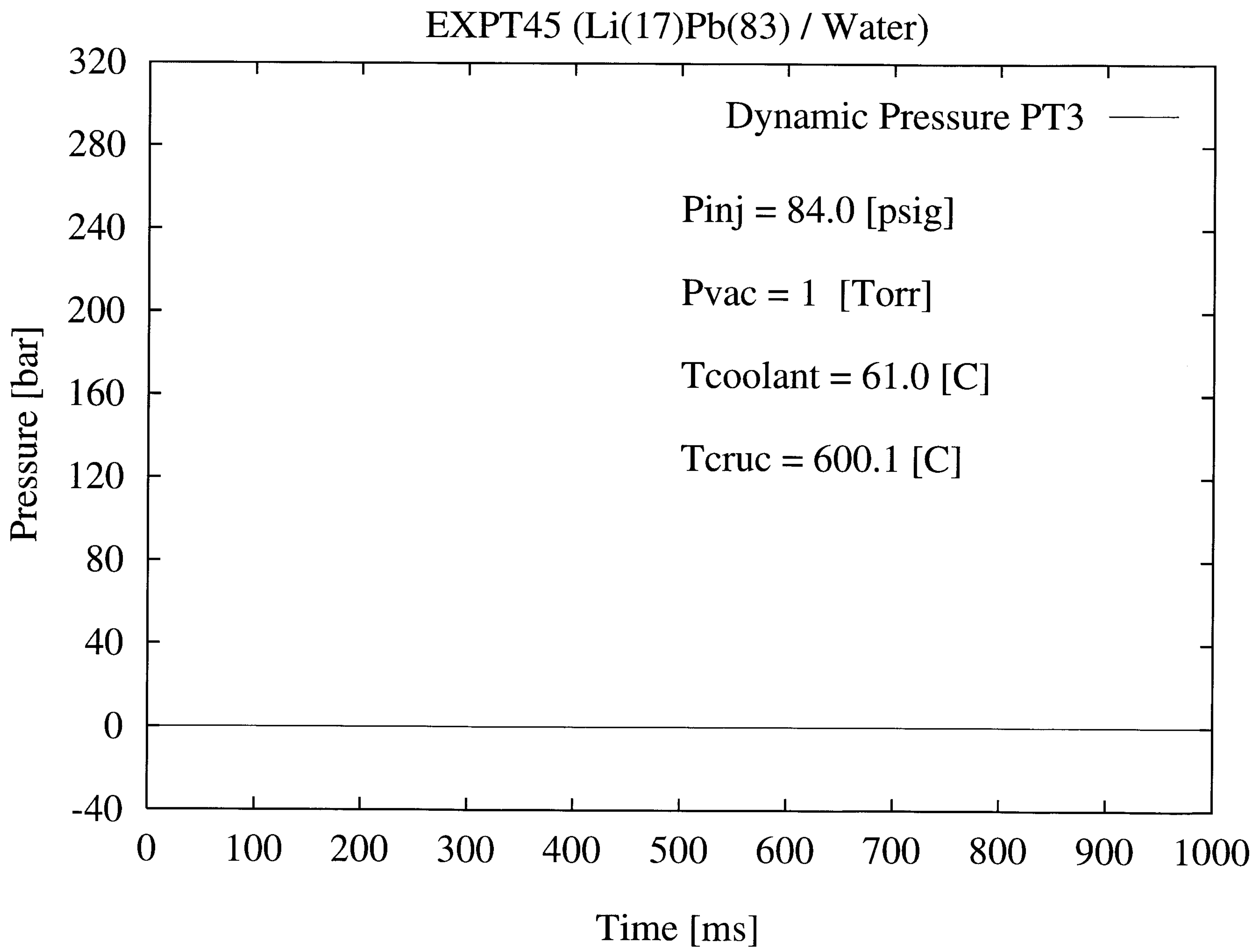


Figure B-6. Complete set of instrumental records from experiment B-45-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 600.9°C; water was at 61.0°C. e. Transducer PT3, 1000 ms trace.

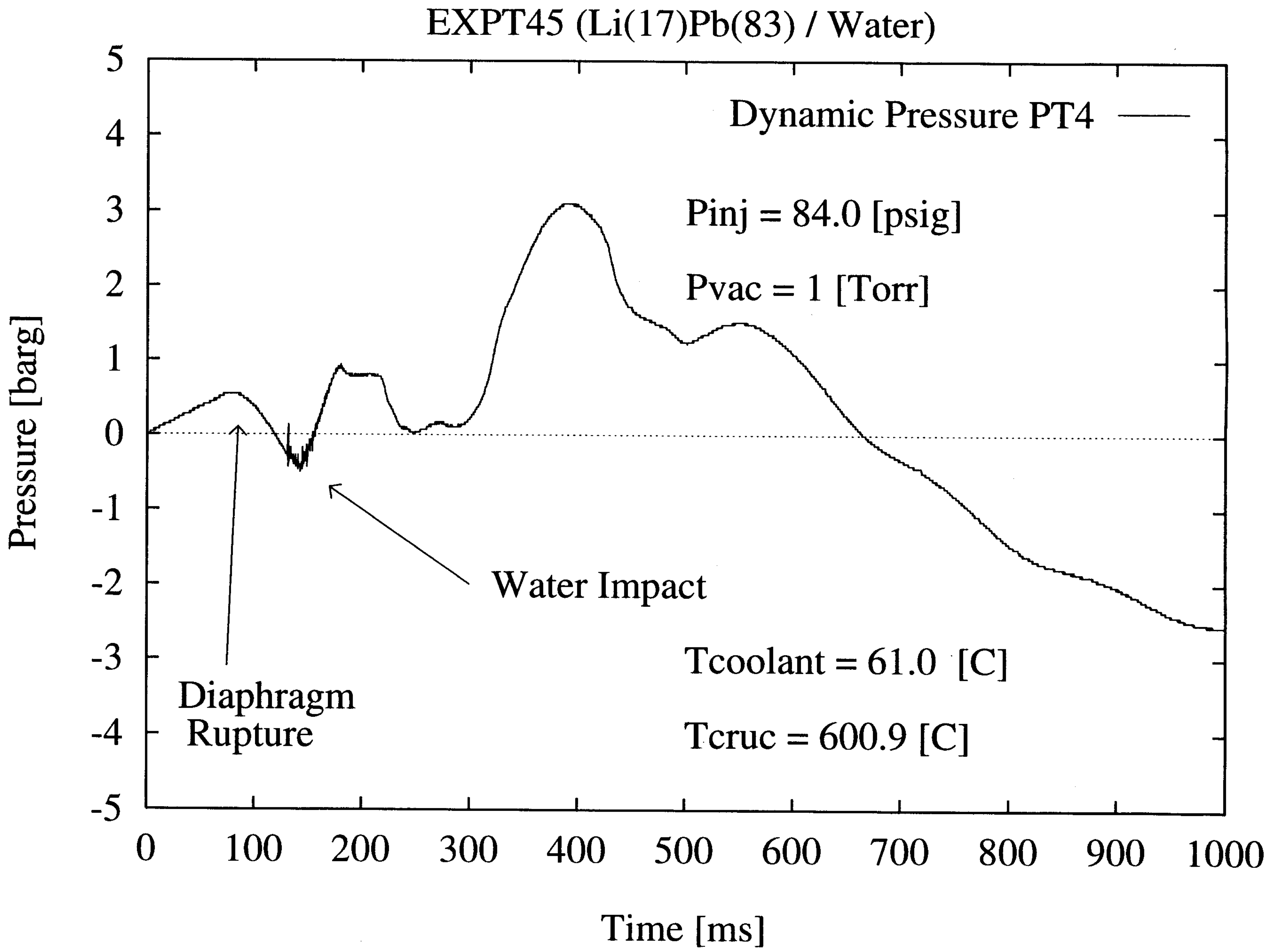


Figure B-6. Complete set of instrumental records from experiment B-45-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 600.9°C; water was at 61.0°C. f. Transducer PT4, 1000 ms trace.

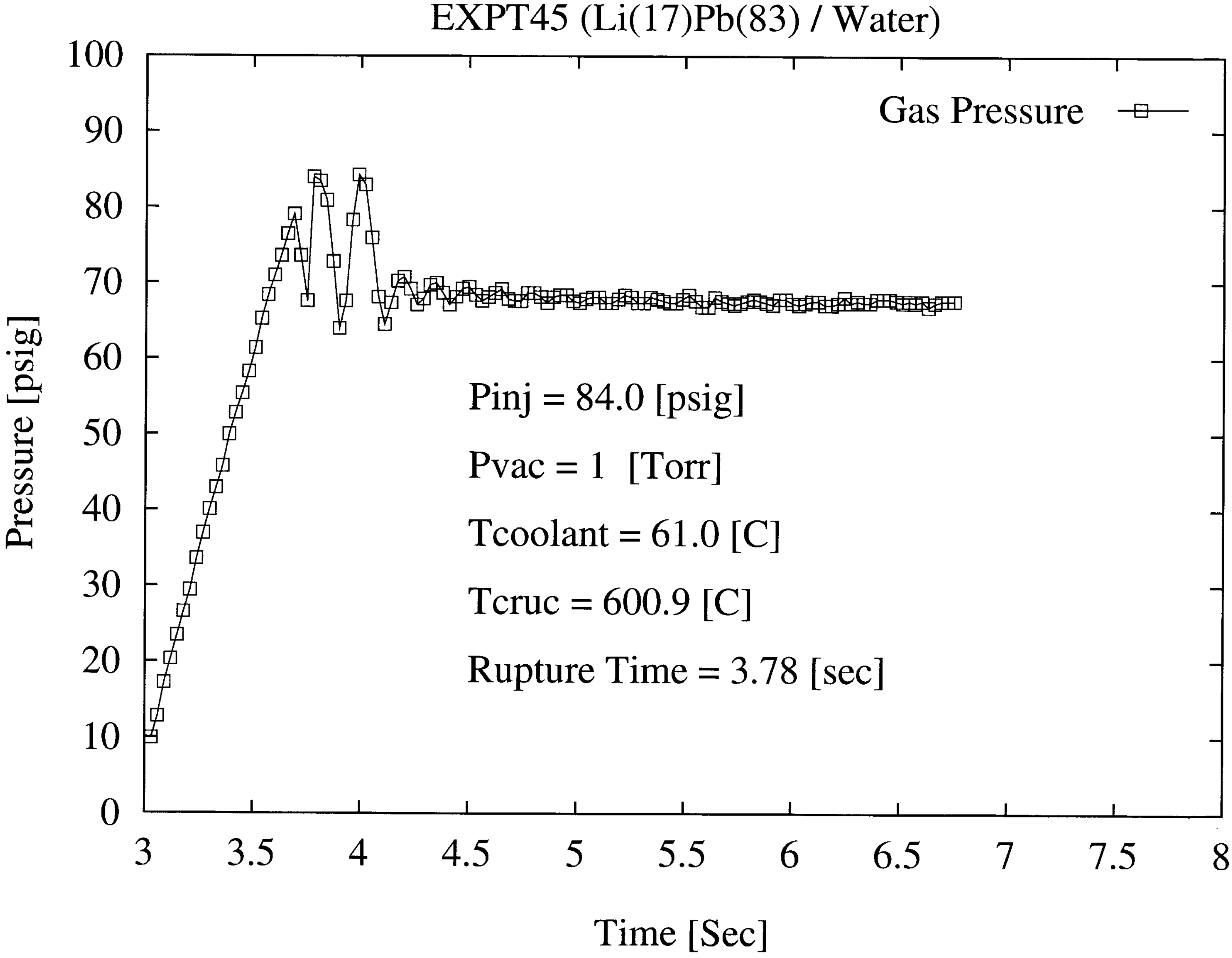


Figure B-6. Complete set of instrumental records from experiment B-45-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 600.9°C; water was at 61.0°C. g. Strain gauge PT5, 5 s trace.

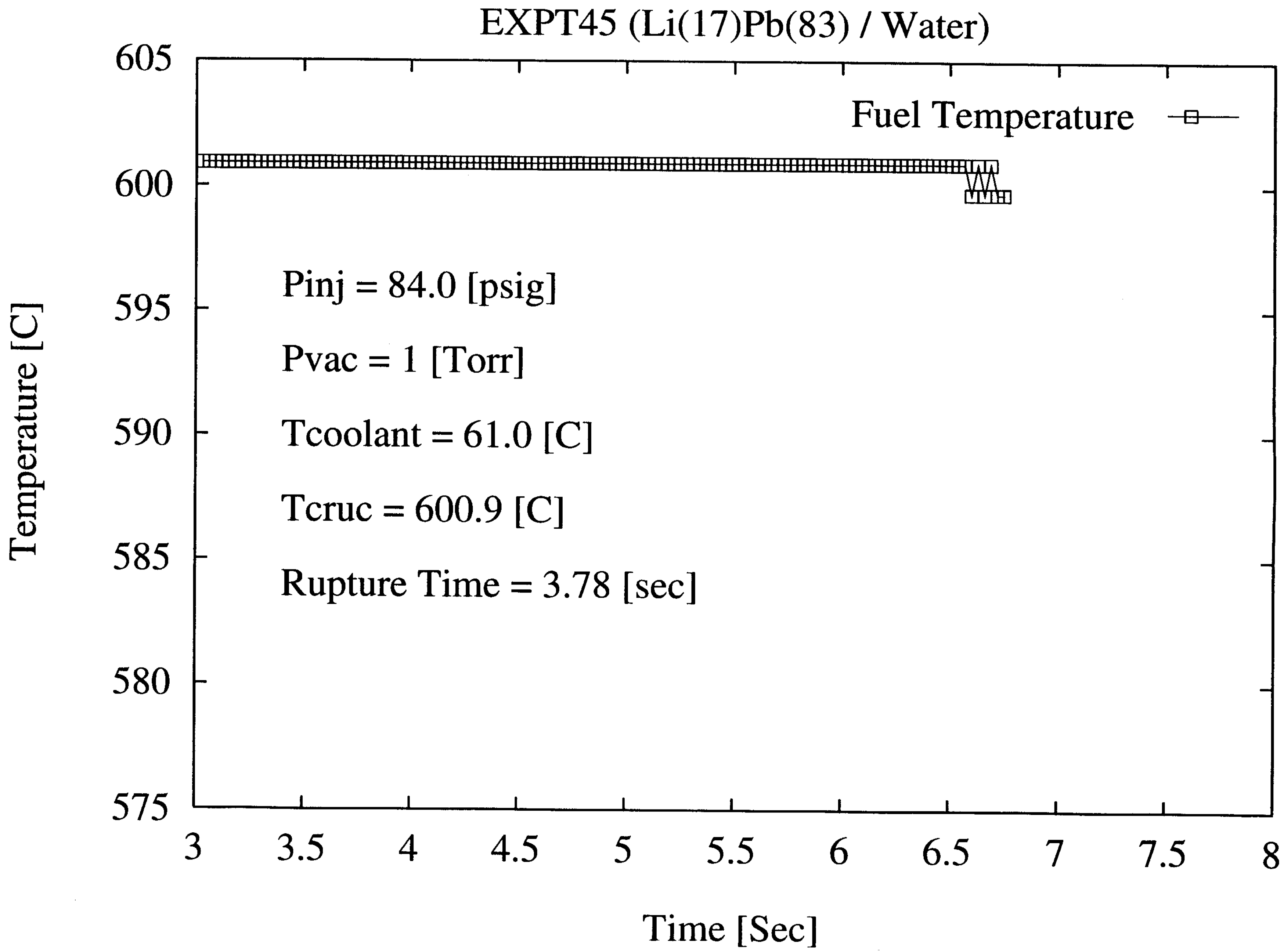


Figure B-6. Complete set of instrumental records from experiment B-45-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 600.9°C; water was at 61.0°C. h. Thermocouple TC5, 5 s trace.

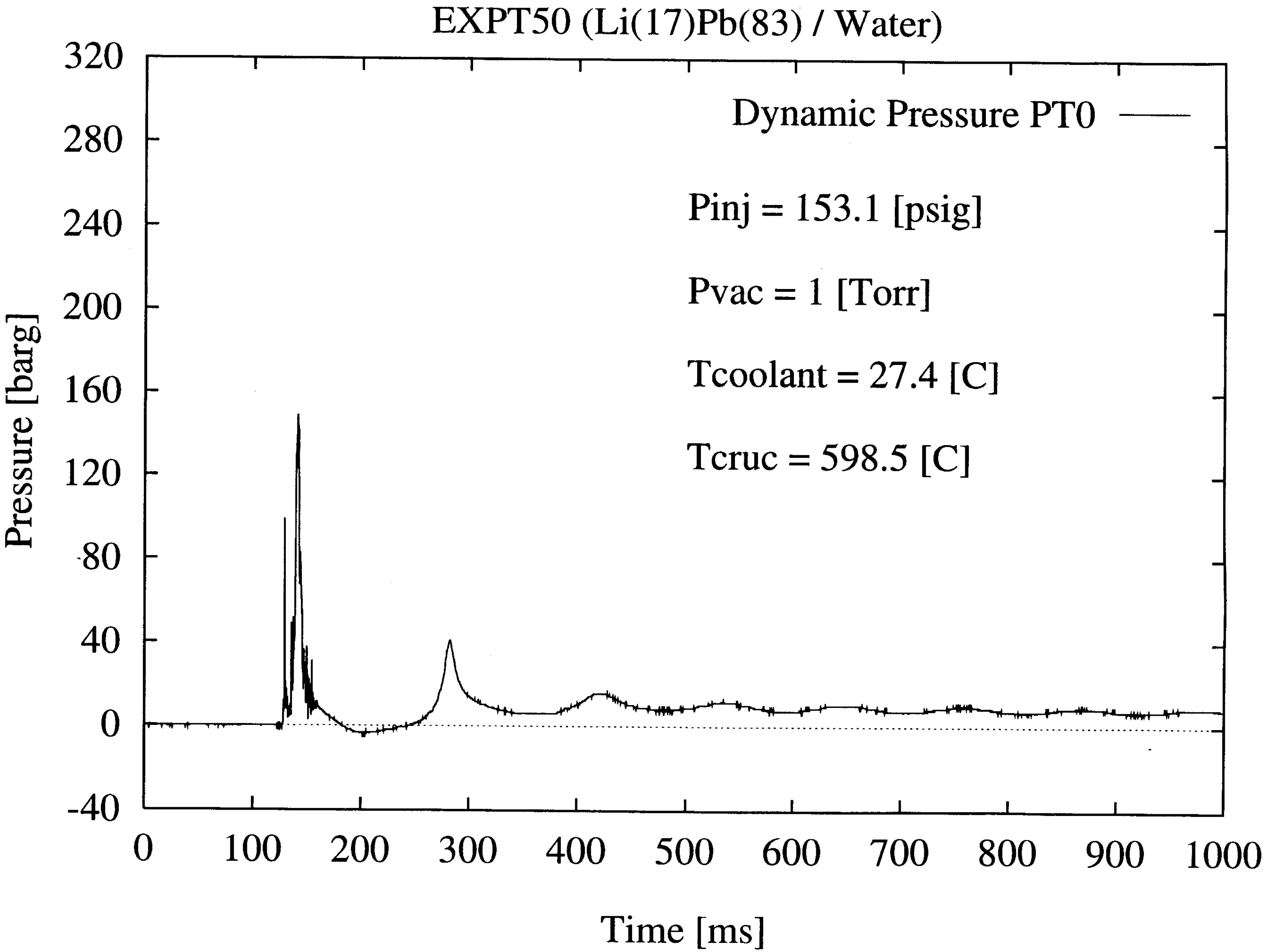


Figure B-7. Complete set of instrumental records from experiment B-50-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 598.5°C; water was at 27.4°C.
a. Transducer PT0, 1000 ms trace.

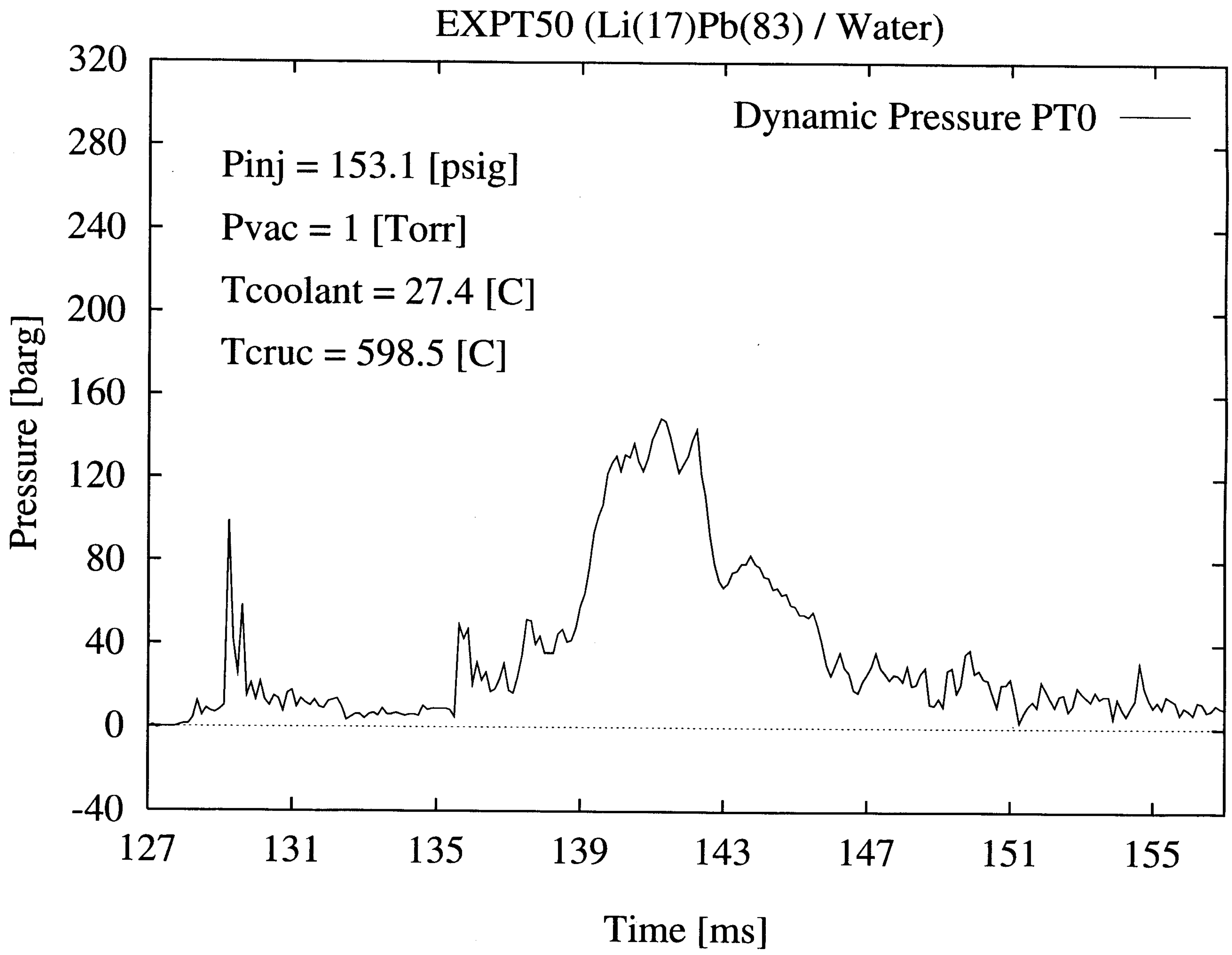


Figure B-7. Complete set of instrumental records from experiment B-50-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 598.5°C; water was at 27.4°C. b. Transducer PT0, expanded time scale.

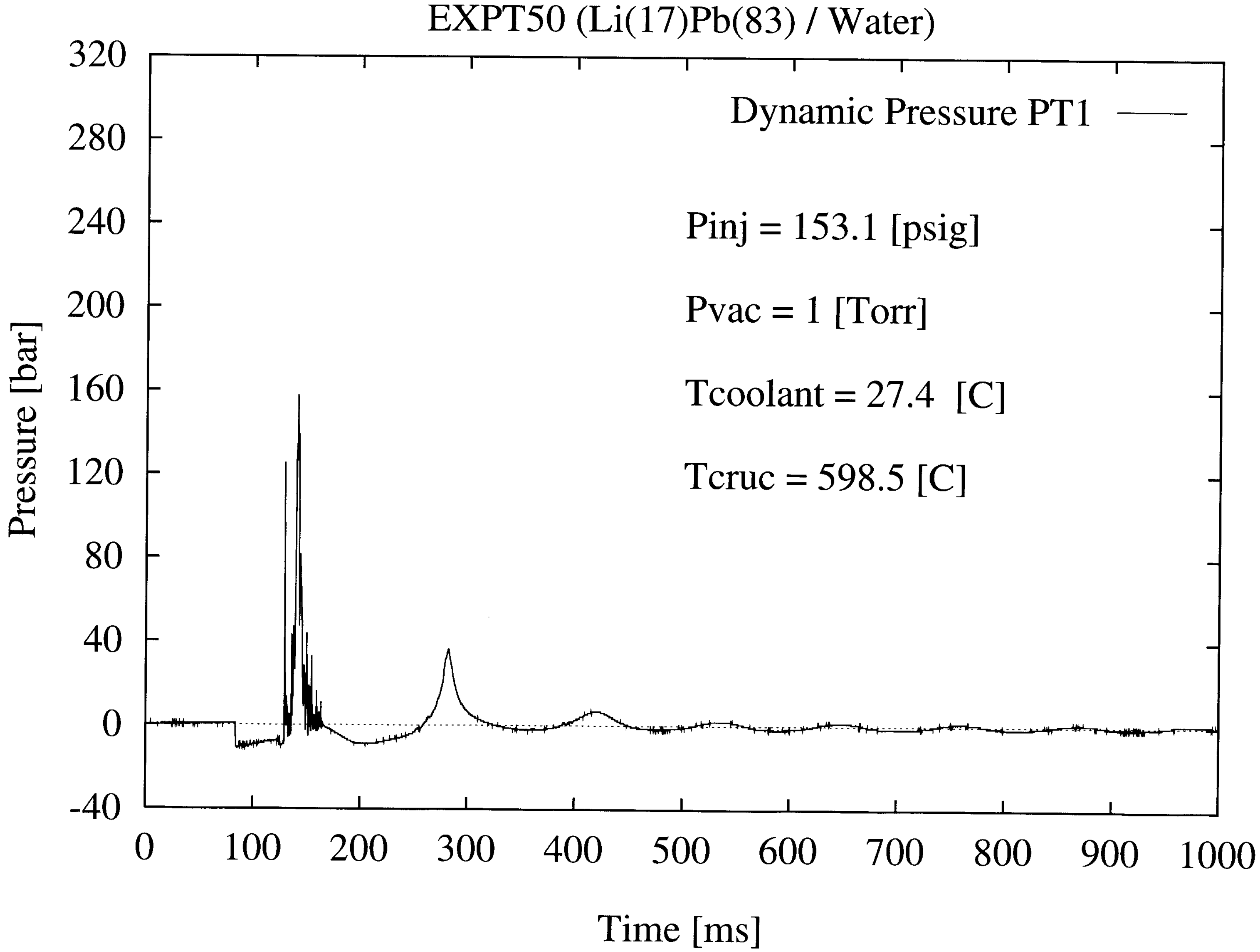


Figure B-7. Complete set of instrumental records from experiment B-50-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 598.5°C; water was at 27.4°C. c. Transducer PT1, 1000 ms trace.

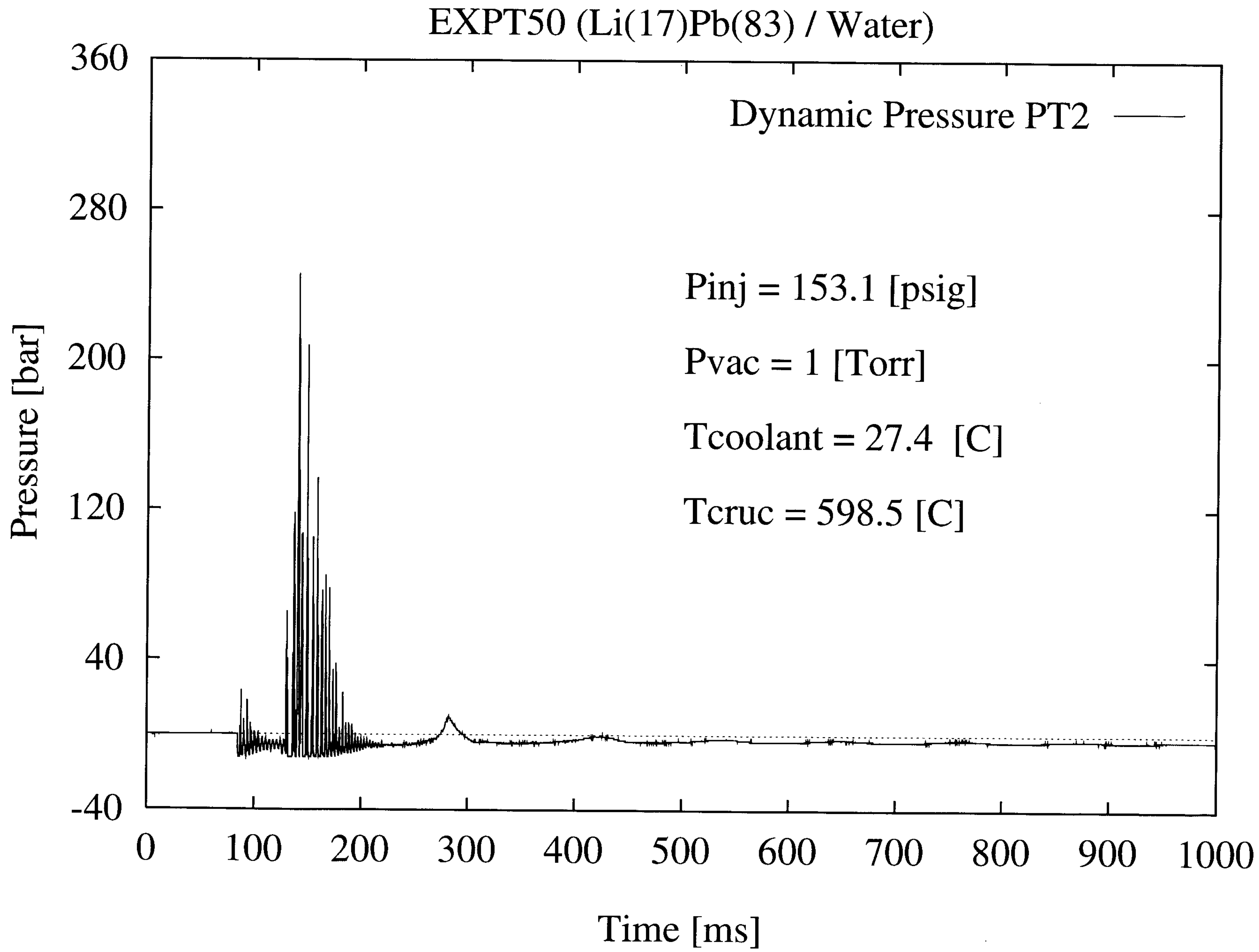


Figure B-7. Complete set of instrumental records from experiment B-50-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 598.5°C; water was at 27.4°C. d. Transducer PT2, 1000 ms trace.

NO USEFUL RECORD

Figure B-7. Complete set of instrumental records from experiment B-50-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 598.5°C; water was at 27.4°C. e. Transducer PT3, 1000 ms trace.

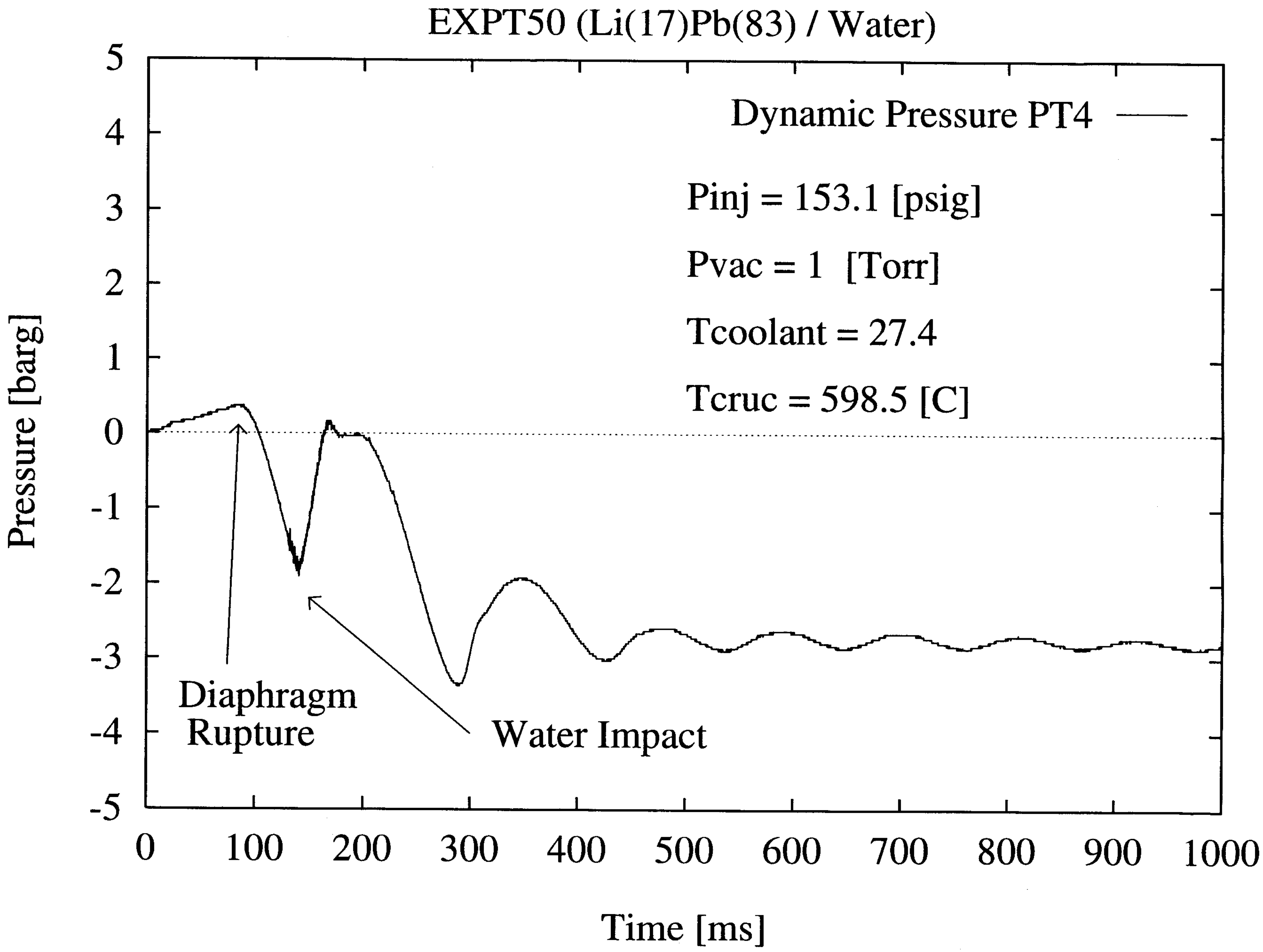


Figure B-7. Complete set of instrumental records from experiment B-50-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 598.5°C; water was at 27.4°C. f. Transducer PT4, 1000 ms trace.

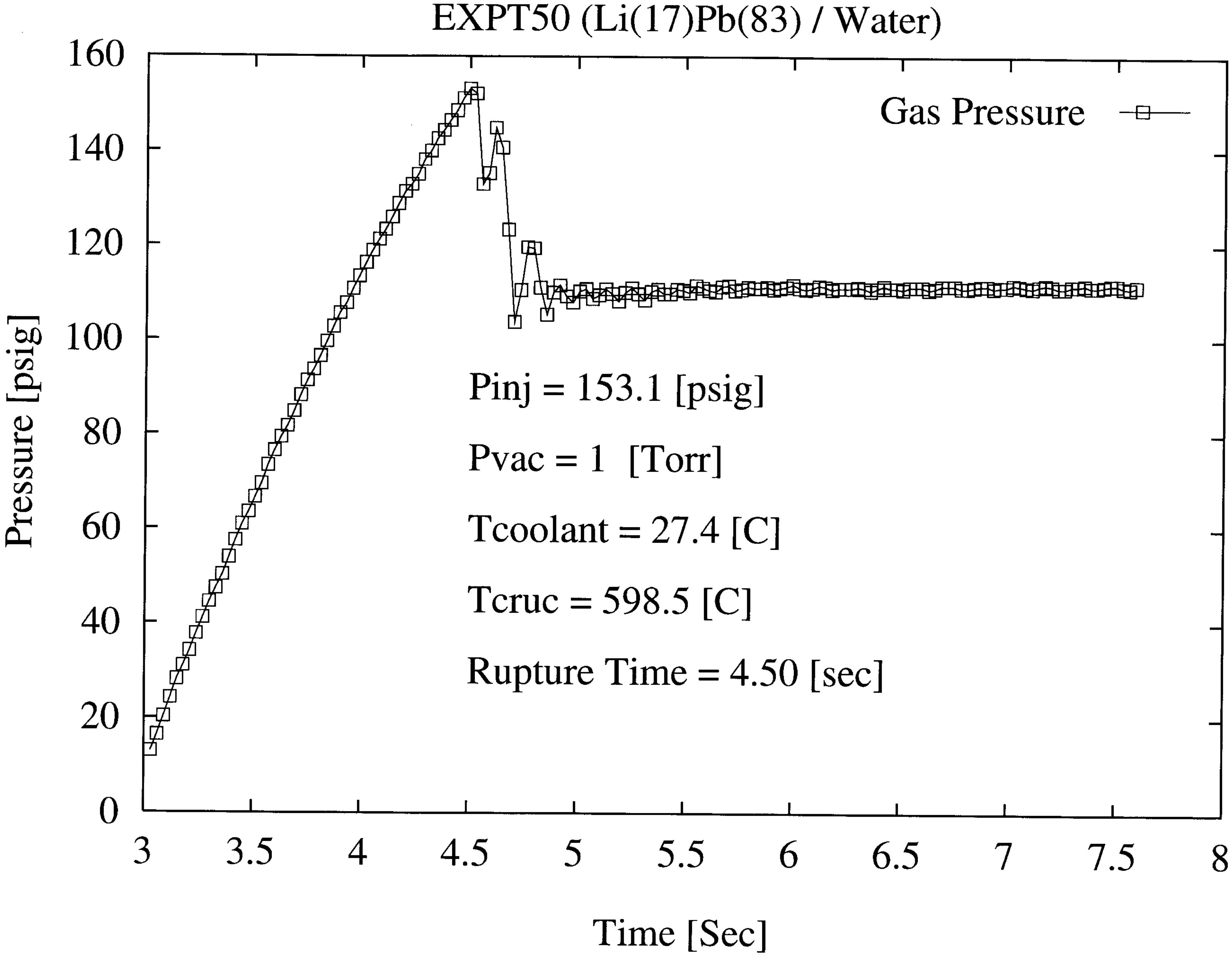


Figure B-7. Complete set of instrumental records from experiment B-50-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 598.5°C; water was at 27.4°C. g. Strain gauge PT5, 5 s trace.

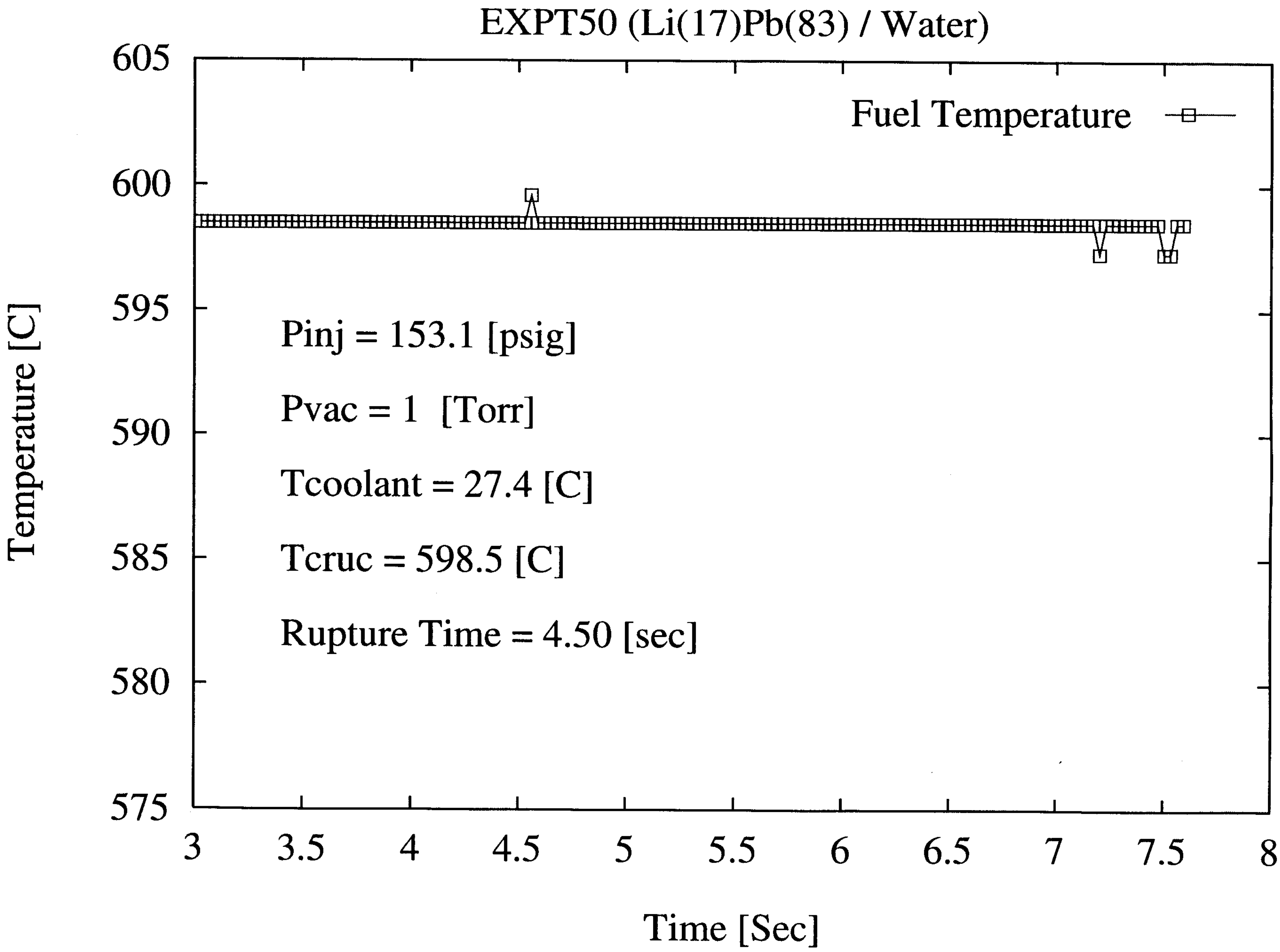


Figure B-7. Complete set of instrumental records from experiment B-50-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 598.5°C; water was at 27.4°C. h. Thermocouple TC5, 5 s trace.

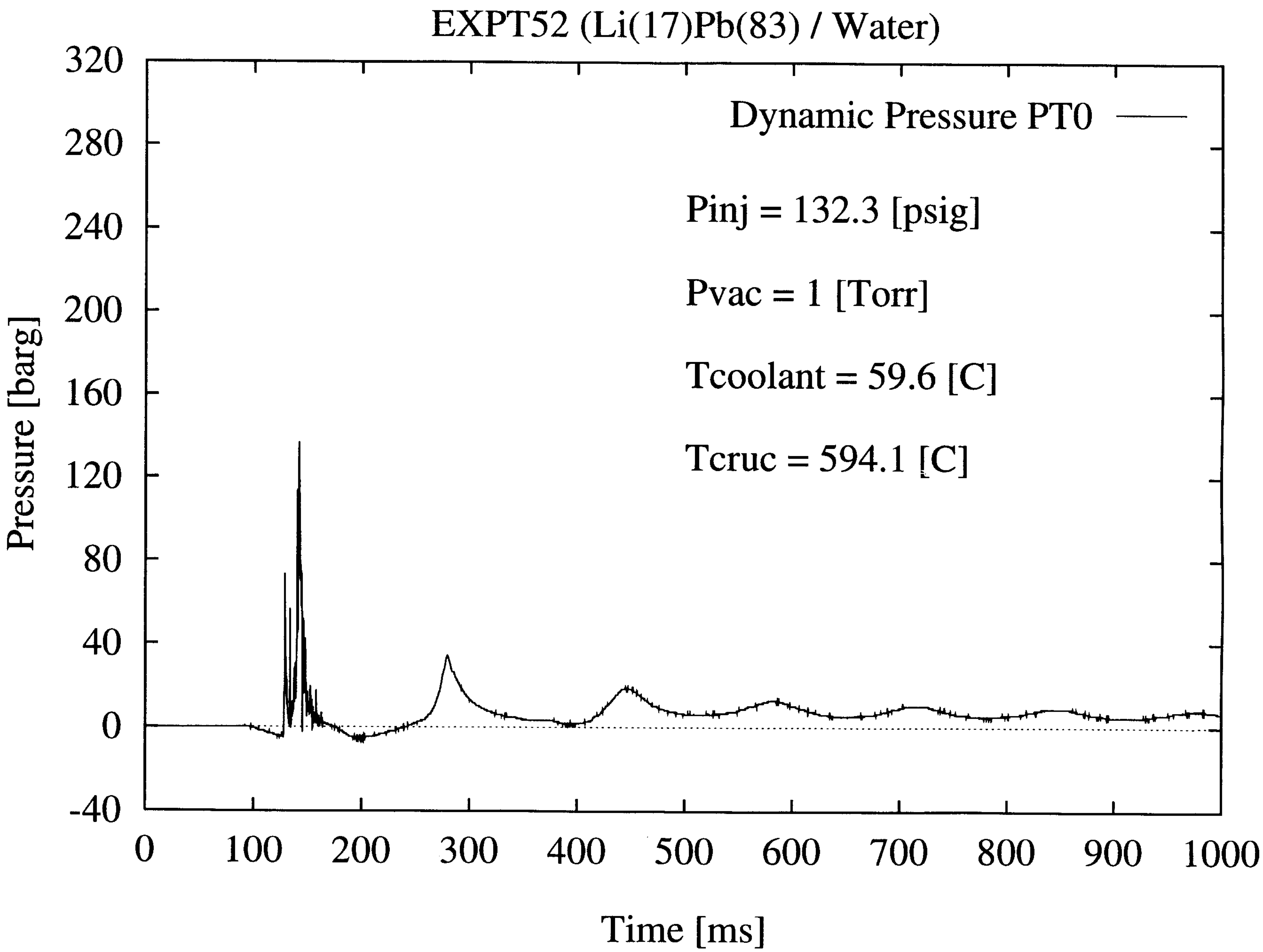


Figure B-8. Complete set of instrumental records from experiment B-52-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 594.1°C; water was at 59.6°C.
a. Transducer PT0, 1000 ms trace.

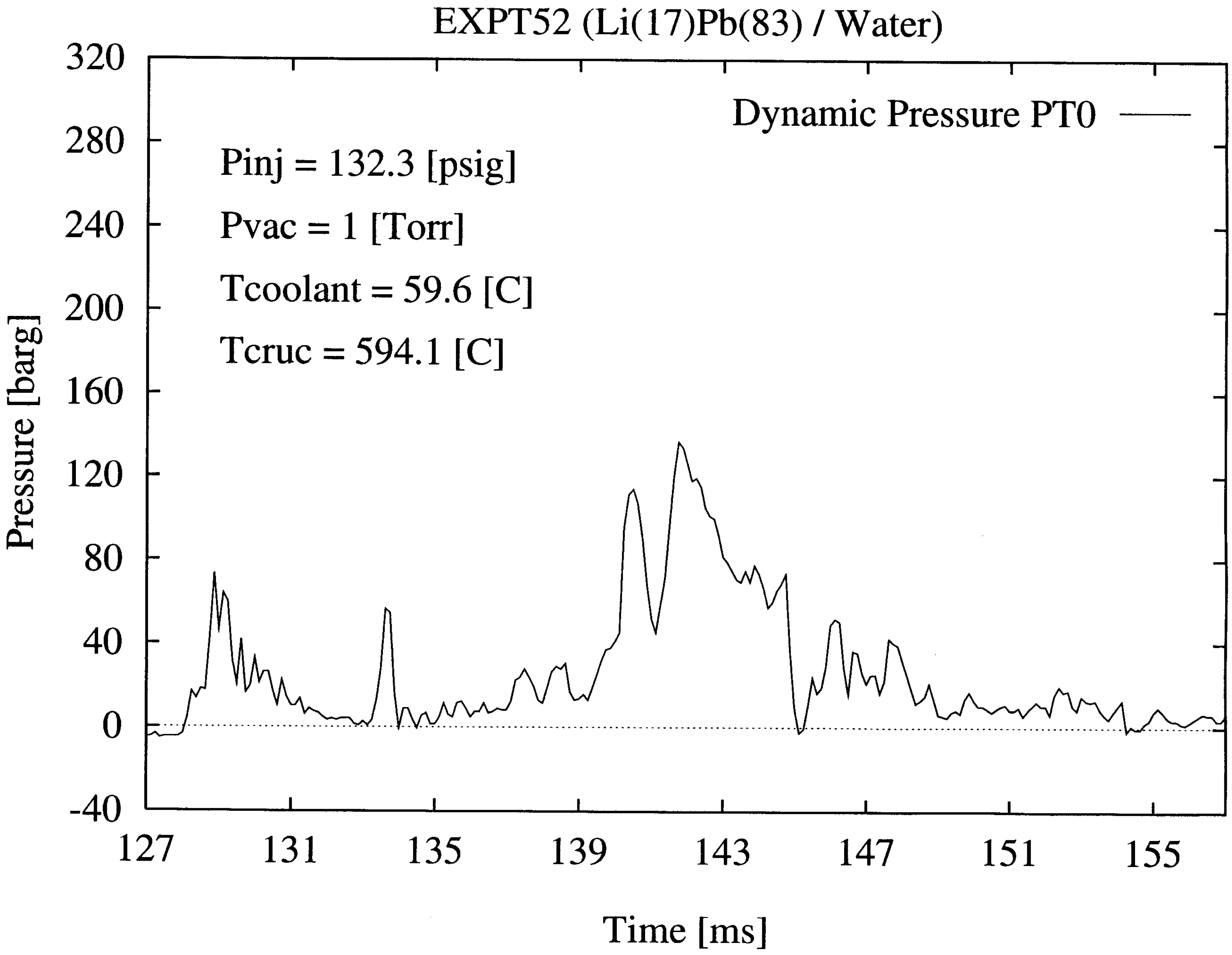


Figure B-8. Complete set of instrumental records from experiment B-52-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 594.1°C; water was at 59.6°C.
b. Transducer PT0, expanded time scale.

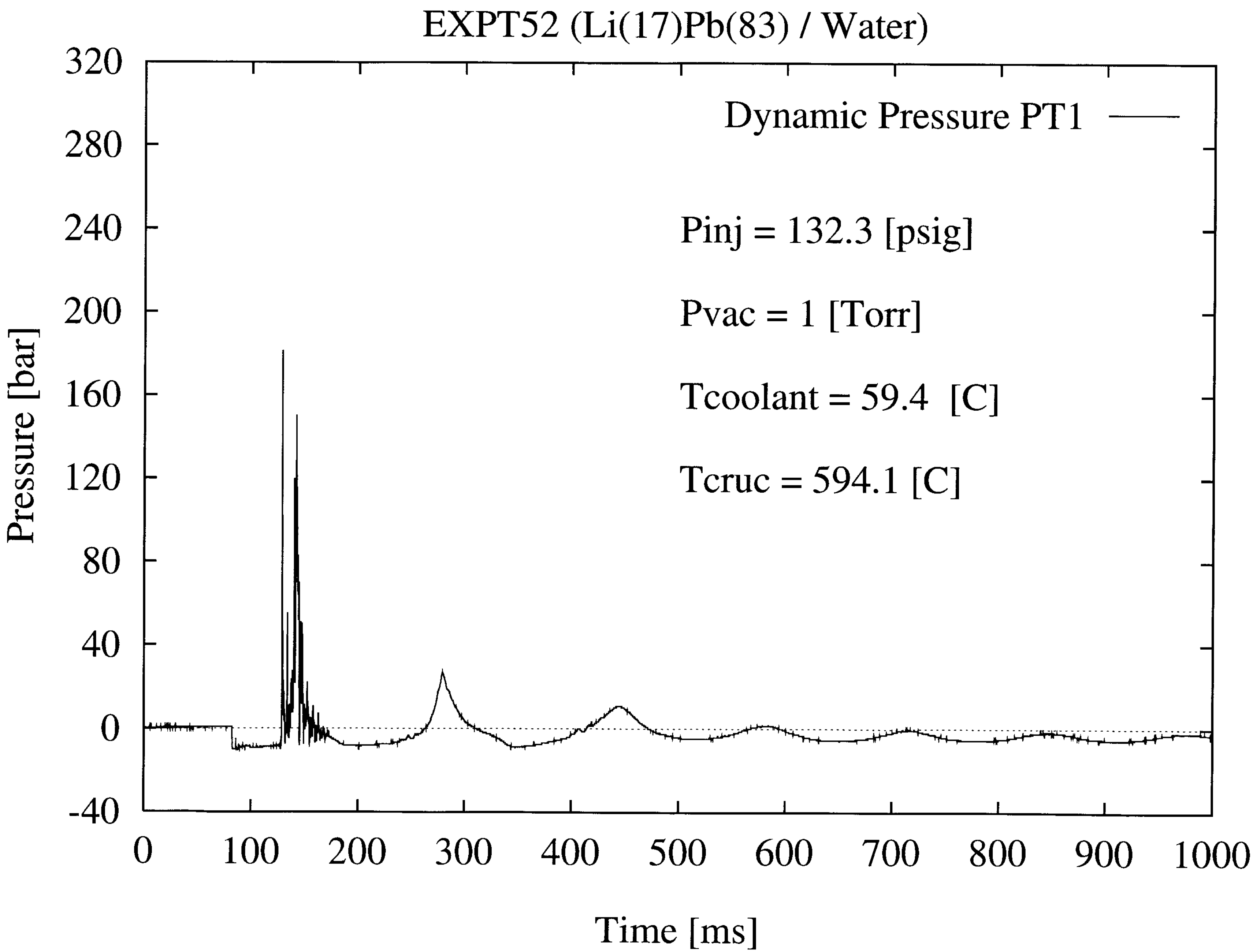


Figure B-8. Complete set of instrumental records from experiment B-52-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 594.1°C; water was at 59.6°C. c. Transducer PT1, 1000 ms trace.

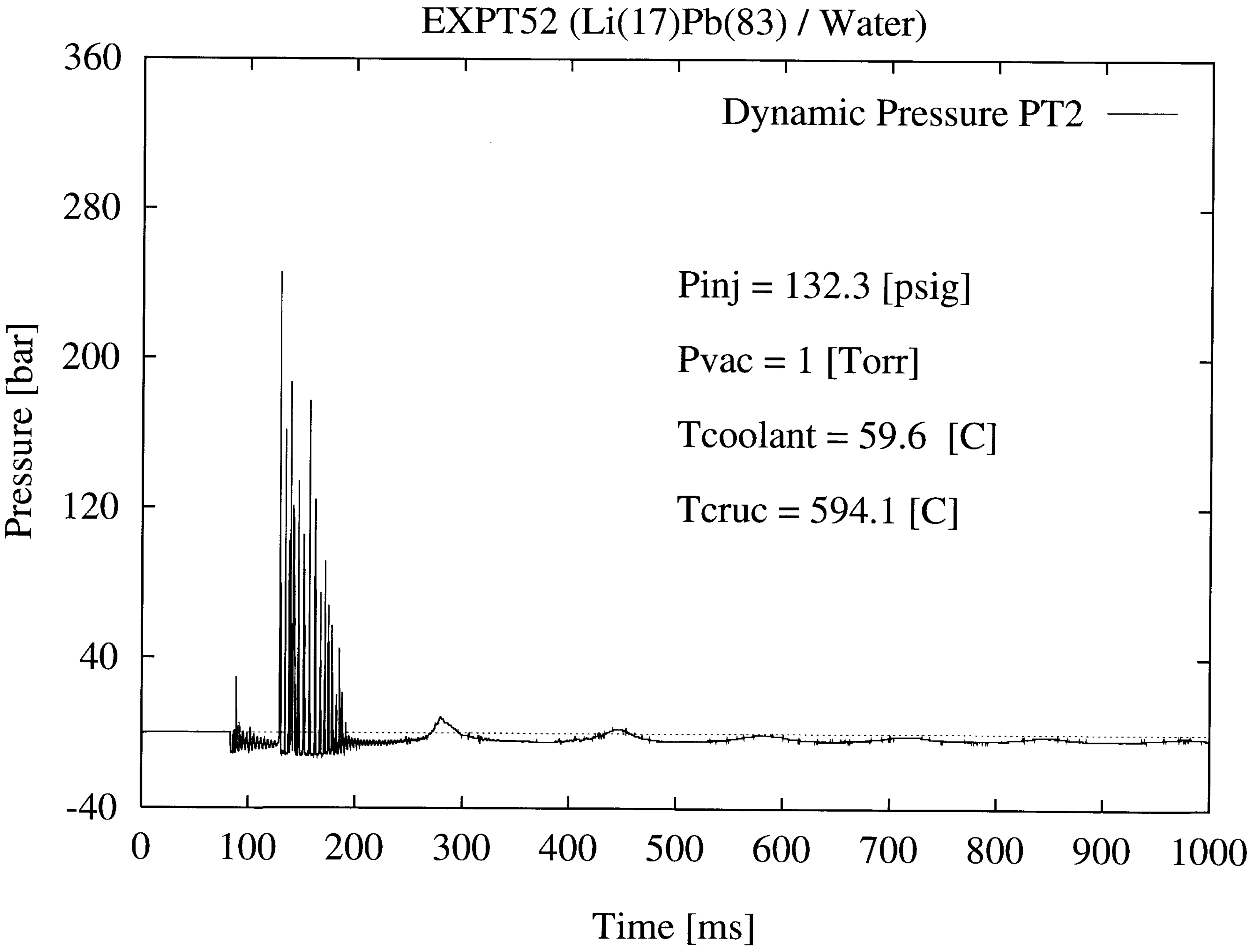


Figure B-8. Complete set of instrumental records from experiment B-52-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 594.1°C; water was at 59.6°C. d. Transducer PT2, 1000 ms trace.

NO USEFUL RECORD

Figure B-8. Complete set of instrumental records from experiment B-52-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 594.1°C; water was at 59.6°C. e. Transducer PT3, 1000 ms trace.

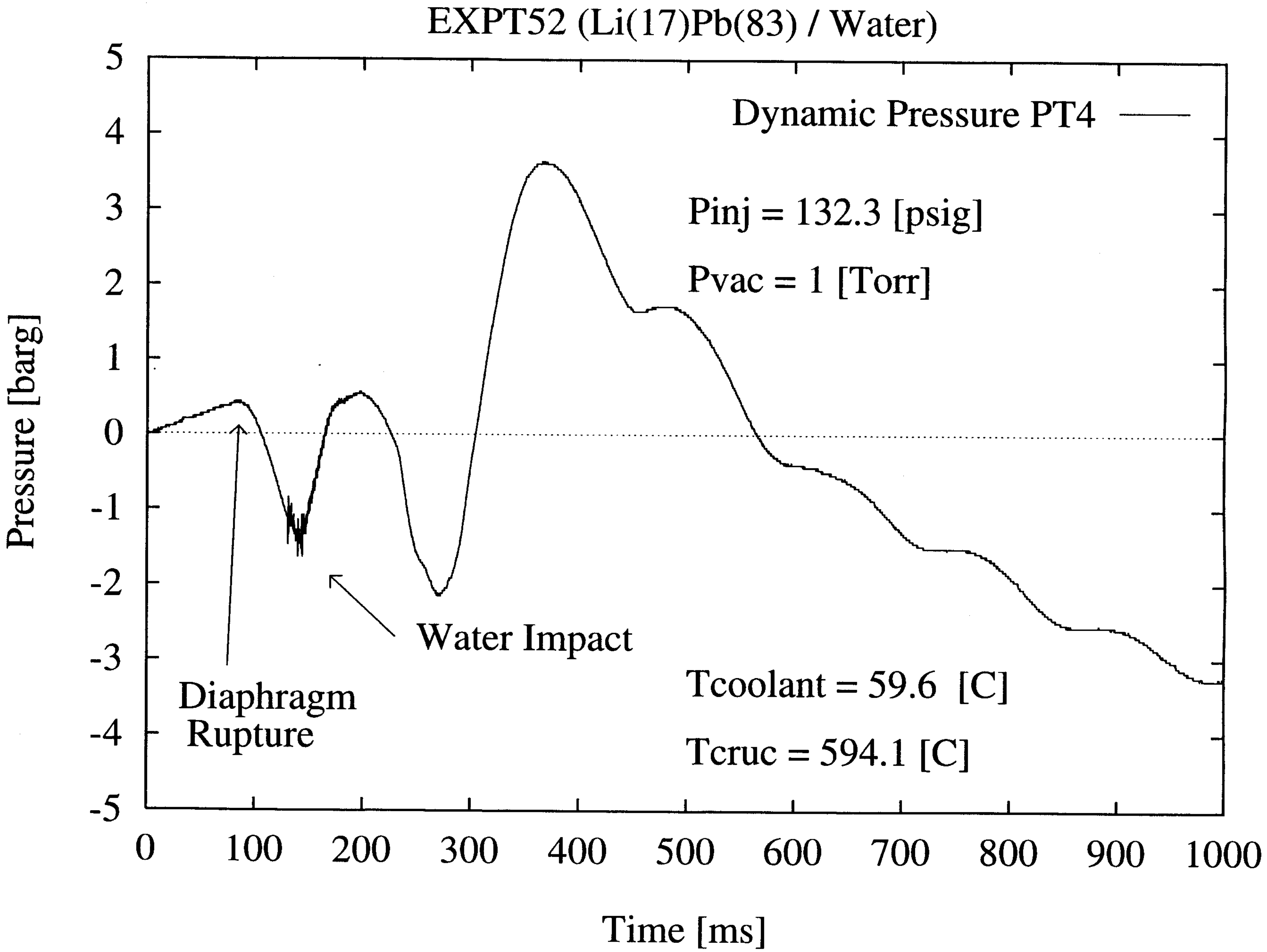


Figure B-8. Complete set of instrumental records from experiment B-52-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 594.1°C; water was at 59.6°C. f. Transducer PT4, 1000 ms trace.

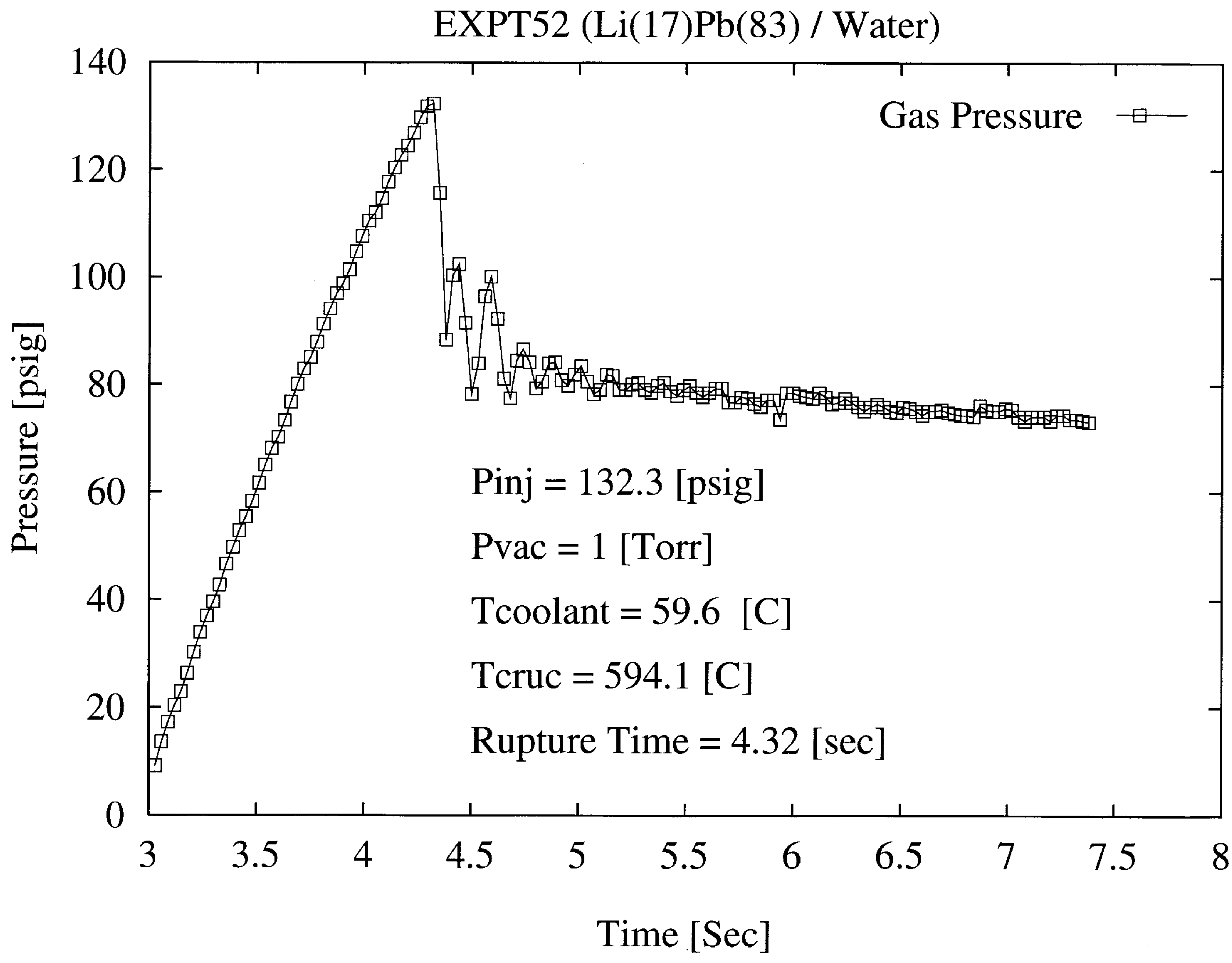


Figure B-8. Complete set of instrumental records from experiment B-52-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 594.1°C; water was at 59.6°C. g. Strain gauge PT5, 5 s trace.

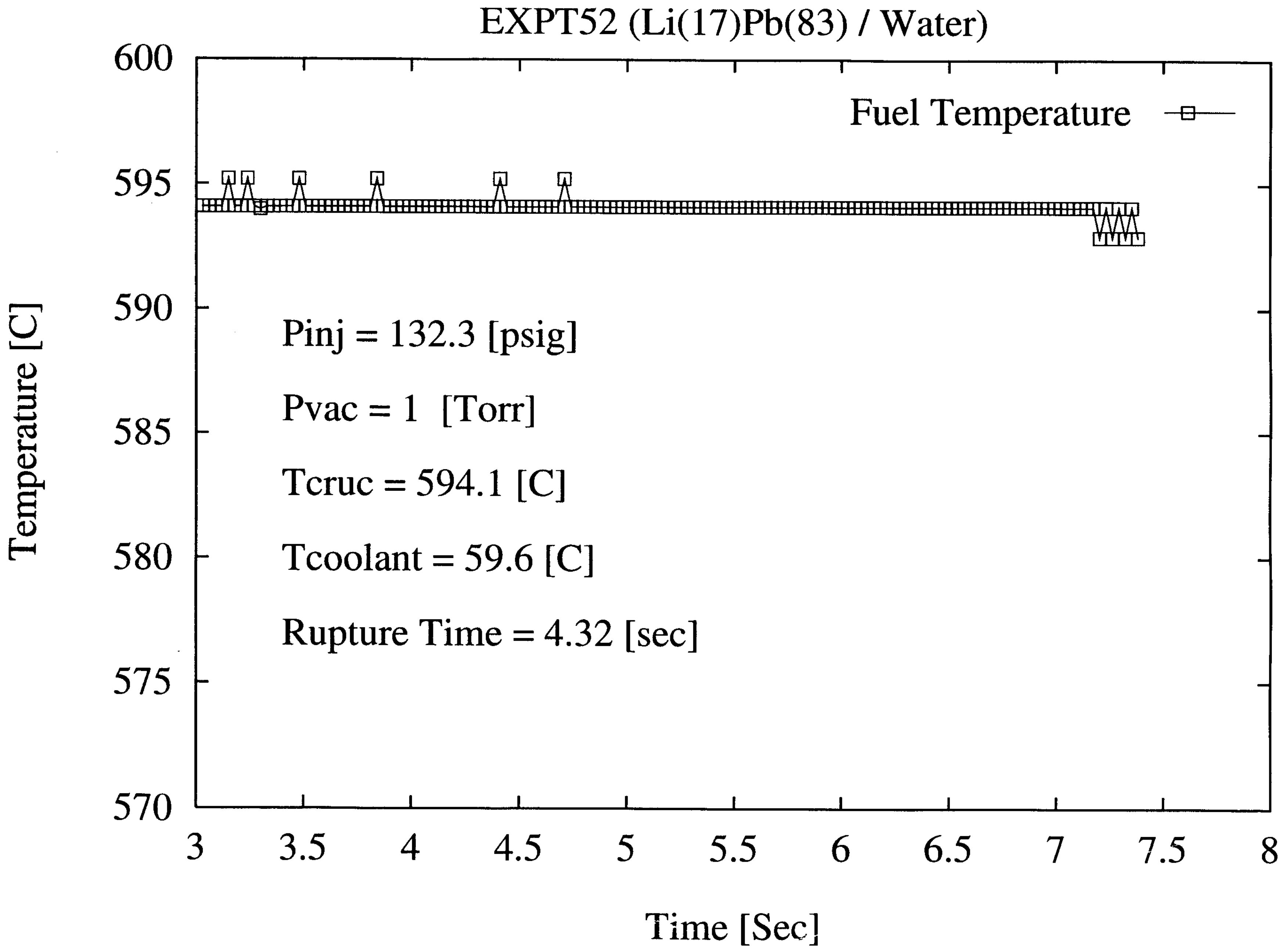


Figure B-8. Complete set of instrumental records from experiment B-52-1. Crucible contained molten 83a/oPb-17a/oLi alloy at 594.1°C; water was at 59.6°C. h. Thermocouple TC5, 5 s trace.

Appendix C

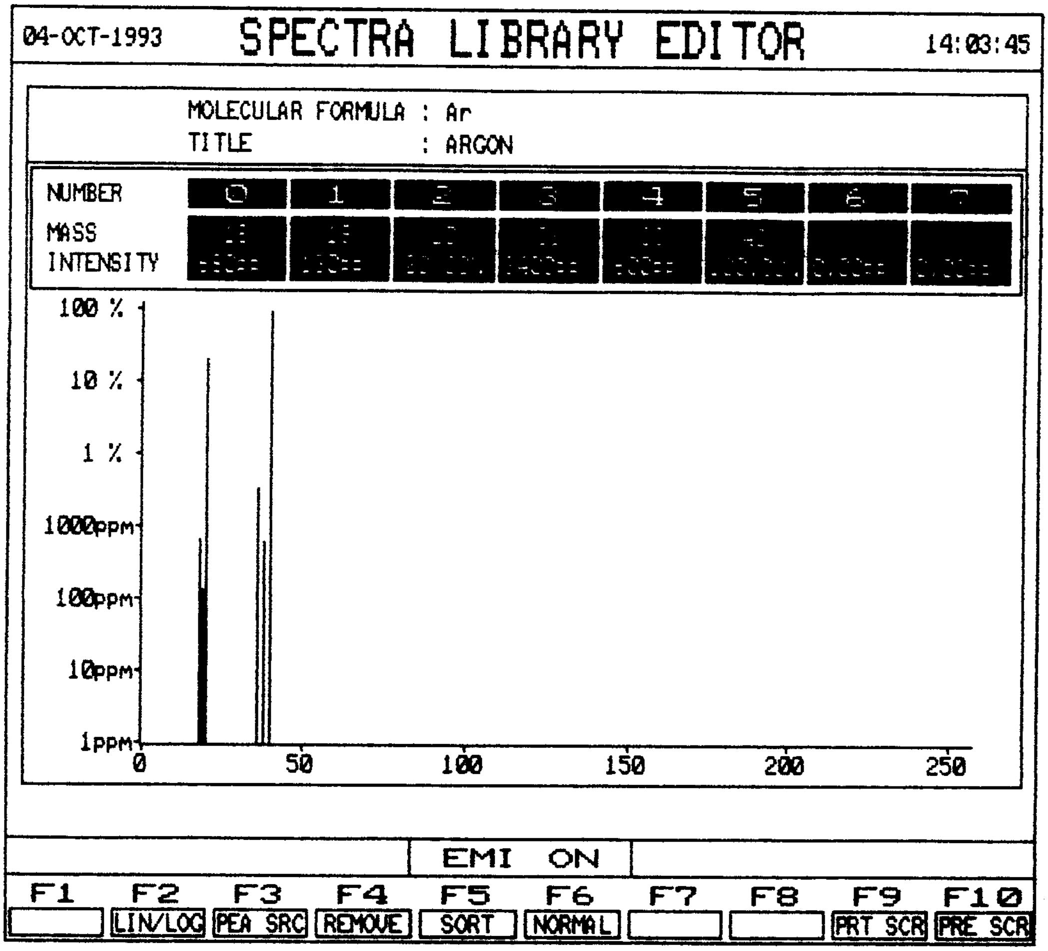
Conversion of Mass Spectrometer Peak Heights to Amounts of Hydrogen

To determine the extent of the chemical reaction between the molten metal and water, samples of gas were taken from the expansion vessel at one and five minutes after the impact experiments. The gas samples were collected in two bottles that had previously been evacuated to below 100 Pa. The total pressure of gas in the expansion vessel at the time of sampling was about 5 MPa.

Analysis of the pressure peaks for each sample (see Figures 19 and 20 in the main body of the report for typical mass spectra) were done as follows:

- The values of the pressure peak height for argon at ~ 40 m/e, oxygen at ~ 32m/e, nitrogen at ~ 28 m/e, water, if any, at ~ 18 m/e, and hydrogen at ~ 2m/e were obtained from the mass spectrometer output files.
- The cracking pattern spectra of the above elements, shown in Figures C-1 through C-4, were used to calculate the actual pressure of each element.Thus, measured pressure peaks for argon, oxygen, and nitrogen were multiplied by the factors 1.2048, 1.1190, and 1.0800, respectively, taken from the cracking patterns in these figures, to find the actual peaks. The pressure peaks for water and hydrogen were corrected to exclude the effects of the residual water andhydrogen in our partial pressure analyzer. When the pressure in the vacuumchamber was at ~ 4×10^{-9} mbar (1 mbar=10^2 Pa), the pressure peaks for water and hydrogen were reduced by 2×10^{-9} mbar and 2×10^{-9} mbar, respectively,to account for the pressure in the vacuum chamber. The pressure peak forwater was also corrected for the cracking characteristics of argon. The newpeak values for water and hydrogen were then multiplied by 1.2450 and 1.0500, respectively.
- The values of the pressure peaks for all the elements were added together toobtain the total chamber pressure.
- By dividing the actual peak pressure of each element by the total chamberpressure, the partial pressure for that element was calculated.
- The total pressure of hydrogen was calculated by multiplying the elementpartial pressure by the bottle pressure.

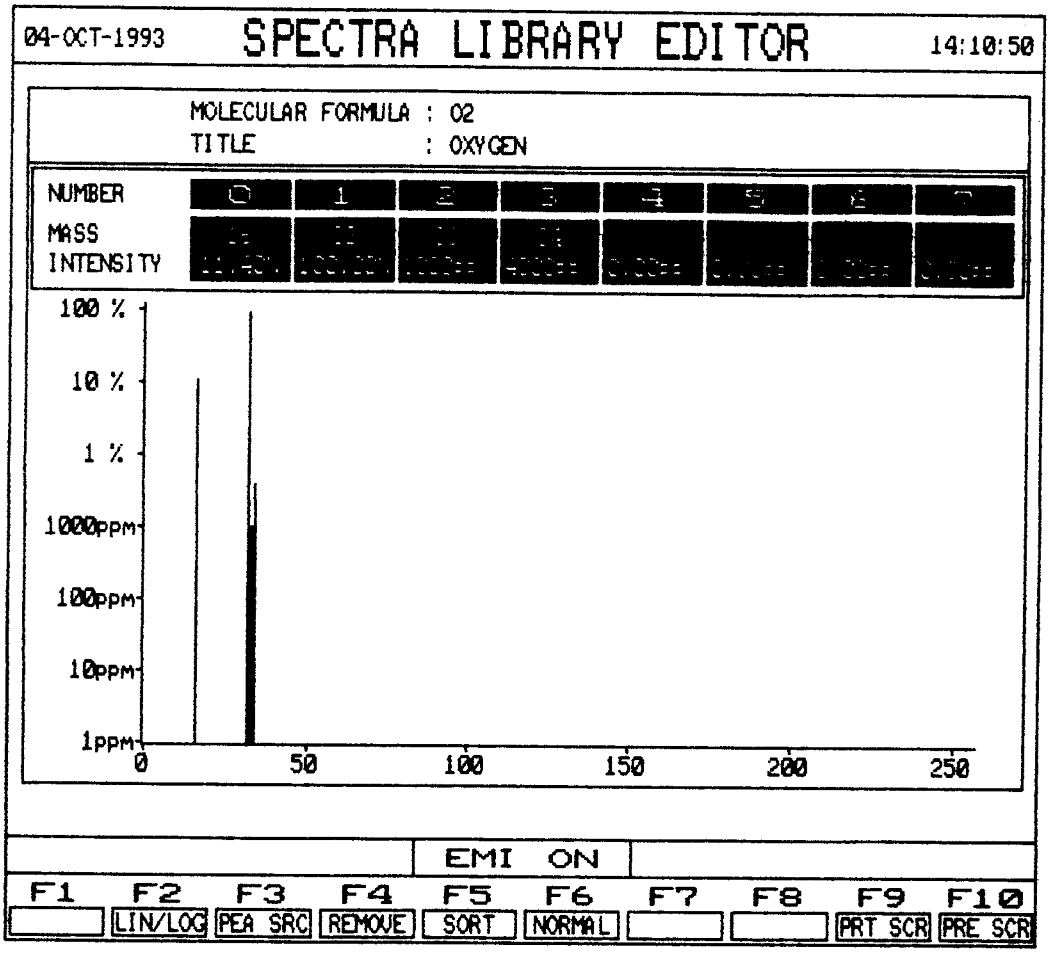
The weight percent (w/o) of the lithium contained in the melt that was oxidized (that is, removed from the melt) is based on the stoichiometric reaction between the water and the lithium given in equations (6) and (7) (see main body of the report).



04-OCT-1993 SPECTRA LIBRARY EDITOR 14:03:45

MOLECULAR FORMULA : Ar
TITLE : ARGON

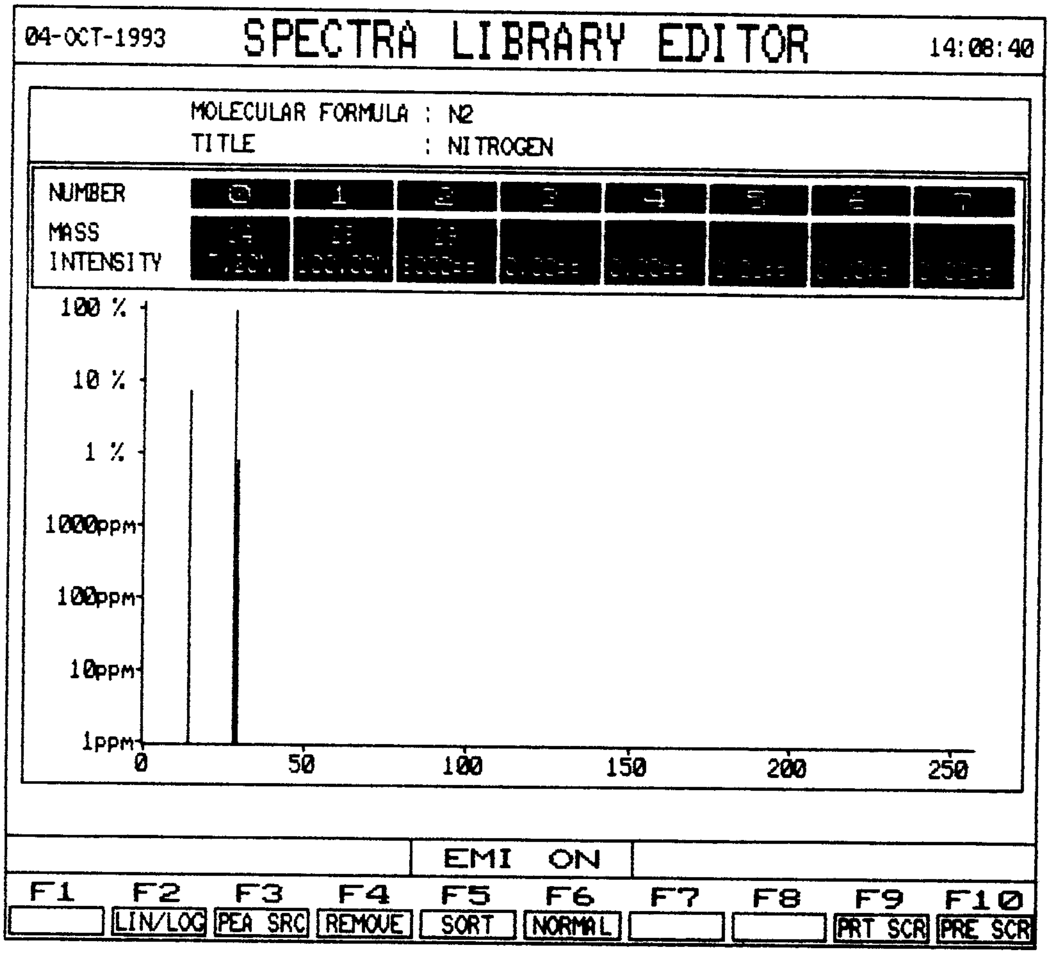
Figure C-1. Cracking pattern mass spectrum for argon. Peaks numbered 0 through 5 have these masses and intensities: 0, 18, 680 ppm; 1, 19, 130 ppm; 2, 20, 20.00%; 3, 36, 3400 ppm; 4, 38, 600 ppm; and 5, 40, 100%.



04-OCT-1993 SPECTRA LIBRARY EDITOR 14:10:50

MOLECULAR FORMULA : O2
TITLE : OXYGEN

Figure C-2. Cracking pattern mass spectrum for oxygen. Peaks numbered 0 through 3 have these masses and intensities: 0, 16, 11.40%; 1, 32, 100%; 2, 33, 1000 ppm; 3, 34, 4000 ppm.

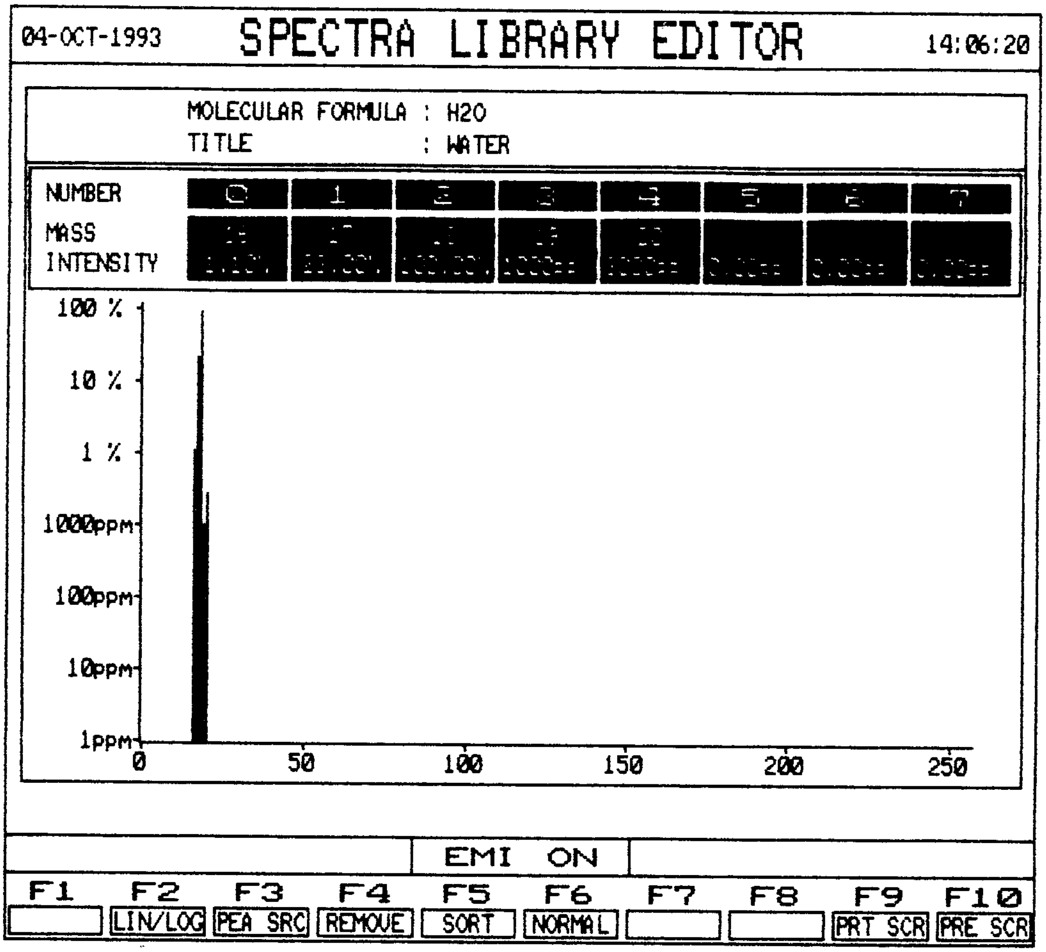


04-OCT-1993 SPECTRA LIBRARY EDITOR 14:08:40

MOLECULAR FORMULA : N2
TITLE : NITROGEN

NUMBER	0	1	2	3	4	5	6	7
MASS	14	28	29					
INTENSITY	7.20%	100.00%	3000ppm	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

Figure C-3. Cracking pattern spectrum for nitrogen. Peaks numbered 0 through 2 have these masses and intensities: 0, 14, 7.20%; 1, 28, 100.00%; 2, 29, 3000 ppm.



MOLECULAR FORMULA : H2O
TITLE : WATER

Figure C-4. Cracking pattern mass spectrum for water. Peaks numbered 0 through 4 have these masses and intensities: 0, 16, 1.10%; 1, 17, 23.00%; 2, 18, 100.00%; 3, 19, 1000 ppm; 4, 20, 3000 ppm.

As an example, the following calculations indicate how we estimated the amount ofhydrogen generated and the percentage of the lithium that took part in the melt/waterchemical reaction for experiment B-45-1. The sample analyzed in this example was the second, taken 5 minutes after the impact (bottle 2, also called bottle B). Note: This sample produced the mass spectrum shown in Figure 20 in the main body of the report.

$$P = 4.1 \times 10^{-7} \text{ [mbar]} \qquad \text{at} \qquad m/e = 40.375 \tag{C-1}$$

(The pressure values and the mass numbers (m/e) for the peaks are taken fromthe mass spectrometer output files for this experiment; these files are labeled expt45bl.asa and expt45d1.asa, which are reproduced in Figures C-5 and C-6.)

$$P_{Ar} = (1.2048) \times 4.1 \text{ x } 10^{-7} = 49.40 \times 10^{-8} \text{ [mbar]} \tag{C-2}$$

$$P = 1.0 \times 10^{-8} \text{ [mbar]} \qquad \text{at} \qquad m/e = 32.375 \tag{C-3}$$

$$P_{O_2} = (1.1190) \times 1.0 \times 10^{-8} = 1.12 \times 10^{-8} \text{ [mbar]} \tag{C-4}$$

$$P = 4.5 \times 10^{-8} \text{ [mbar]} \qquad \text{at} \qquad m/e = 28.375 \tag{C-5}$$

$$P_{N_2} = (1.0800) \times 4.5 \times 10^{-8} = 4.86 \times 10^{-8} \text{ [mbar]} \tag{C-6}$$

$$P = 5.5 \times 10^{-8} \text{ [mbar]} \qquad \text{at} \qquad m/e = 2.2500 \tag{C-7}$$

$$P_{H_2} = (1.05) \times (5.5 \times 10^{-8} - 2 \times 10^{-9}) = 5.56 \times 10^{-8} \text{ [mbar]} \tag{C-8}$$

$$P_{Chamber,Total} = P_{Ar} + P_{O_2} + P_{N_2} + P_{H_2} + \ 60.94 \times 10\text{-}8 \text{ [mbar]} \tag{C-9}$$

$$P_{H_2} = \frac{5.56}{60.94} = \underline{0.0912} \tag{C-10}$$

$$P_{bottle2} = 54 \text{ [psig]} \qquad \text{from the strain gauge} \tag*{(C-11)^a}$$

$$P_{bottle2} = 473686.5 \text{ [Pa]} \tag*{(C-12)^a}$$

$$T_{bottle2} = 40.2°\text{C} \tag*{(C-13)^a}$$

Figure C-5. Mass spectometer output file expt45b1.asa for sample bottle B taken 5 minutes after the impact in experiment B-45-1. Each entry represents 1/16th of a mass unit, e/m; two lines are required per mass unit. This file is plotted in Figure 20 in the main body of the report. The maximum partial pressure in this file is 10×10^{-7} torr.

```
ASCII-CYCLEDATAFILE EXPT45B1.ASA
DATE, 25-JUN-1991,TIME, 18: 4:00,NUMBER OF SCANS - 1
"FIRST MASS =   0.00","MASS WIDTH = 63","RANGE = E-07","VALUES = 1010","UNIT = [mbar]"
0.0000E+00,1.0000E-08,1.0000E-08,1.0000E-08,1.0000E-08,1.0000E-08,1.0000E-08,1.0000E-08
1.0000E-08,1.0000E-08,1.0000E-08,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
1.0000E-08,2.0000E-08,4.0000E-08,5.0000E-08,5.0000E-08,4.0000E-08,2.0000E-08,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
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0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
```

```
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00
```

Figure C-6. Mass spectrometer output file expt45d1.asa for sample bottle B taken 5 minutes after the impact in experiment B-45-1. Each entry represents 1/16th of a mass unit, e/m; two lines are required per mass unit. The maximum partial pressure in this file is 10 × 10^{-8} torr.

```
ASCII-CYCLEDATAFILE EXPT45D1.ASA
DATE, 25-JUN-1991,TIME, 18: 1:00,NUMBER OF SCANS - 1
"FIRST MASS =   0.00","MASS WIDTH = 63","RANGE = E-08","VALUES = 1010","UNIT = [mbar]"
0.0000E+00,1.0000E-08,1.2000E-08,1.2000E-08,1.0000E-08,1.1000E-08,1.3000E-08,1.1000E-08
1.0000E-08,1.2000E-08,1.3000E-08,8.0000E-09,6.0000E-09,6.0000E-09,3.0000E-09,0.0000E+00
1.0000E-09,2.0000E-09,1.0000E-09,0.0000E+00,2.0000E-09,1.0000E-09,0.0000E+00,0.0000E+00
1.0000E-09,1.0000E-09,0.0000E+00,1.0000E-09,2.0000E-09,1.0000E-09,0.0000E+00,5.0000E-09
1.1000E-08,2.9000E-08,4.2000E-08,5.5000E-08,5.5000E-08,4.6000E-08,3.1000E-08,1.0000E-08
1.0000E-09,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00
0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,2.0000E-09,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,1.0000E-09,0.0000E+00,0.0000E+00,1.0000E-09
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09
0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00
0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00
0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00
1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00
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0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00
0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
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0.0000E+00,0.0000E+00,1.0000E-09,2.0000E-09,1.0000E-09,1.0000E-09,2.0000E-09,1.0000E-09
0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00
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0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00
1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,1.0000E-09
0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,2.0000E-09,2.0000E-09
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00
0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,1.0000E-09,1.0000E-09,0.0000E+00
0.0000E+00,2.0000E-09,1.0000E-09,0.0000E+00,0.0000E+00,2.0000E-09,1.0000E-09,3.0000E-09
2.3000E-08,4.4000E-08,6.3000E-08,7.8000E-08,8.8000E-08,8.2000E-08,7.1000E-08,5.0000E-08
1.6000E-08,2.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09
0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00
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```

```
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0.0000E+00,0.0000E+00,1.0000E-09,1.0000E-09,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00
0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00
0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00
1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09
```

```
0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09,0.0000E+00,0.0000E+00,0.0000E+00,1.0000E-09
0.0000E+00,0.0000E+00
```

$$V_{\text{expansion vessel}} = 1766 \times 10^{-6}\ [m^3] \tag{C-14}$$

$$M_{H_2} = \frac{P_{H_2\ \text{expansion vessel}} / V_{\text{expansion vessel}}}{R_{H_2}\ T_{\text{expansion vessel}}} \tag{C-15}$$

$$M_{H_2} = \frac{(.0912 \times 473686.5)1766 \times 10^{-6}}{4124.2 \times (273.15 + 40.2)} = 59 \times 10^{-6}\ [kg] \tag{C-16}$$

$$2Li + H_2O \rightarrow Li_2O + H_2 \qquad \text{(C-17)}^{b}$$

From the stoichiometric reaction between lithium and water, for every gram of hydrogen, 6.94 grams of lithium is needed.

$$m_{Li,\text{reacted}} = 6.94 \times 59 = 409.9\ [mg] \tag{C-18}$$

$$W\%Li_{\text{reacted}} = \frac{409.9}{789.8} \times 100 = 51.9\% \tag{C-19}$$

---

[a] Bottle 2 is also called bottle B in the main body of the report

[b] See also equation (6) in the main body of the report.

Appendix D

Analytical Reports

The original analytical reports obtained from the University of Wisconsin Plant and Soil Analysis Laboratory, Madison WI, are reproduced in this appendix. They arepresented as Figures D-1, D-2 and D-3.

AUG 28 1995

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Madison, Wisconsin 53705-4453
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College of Agricultural and Life Sciences

CORRECTED REPORT

Date 8/25/95

Lab S00180

Acct 900

To Lloyd S. Nelson
Dept Nuclear Energy

From Soil & Plant Analysis Lab
Sherry Combs, Director

Subject Results of analyses on 8 solution samples

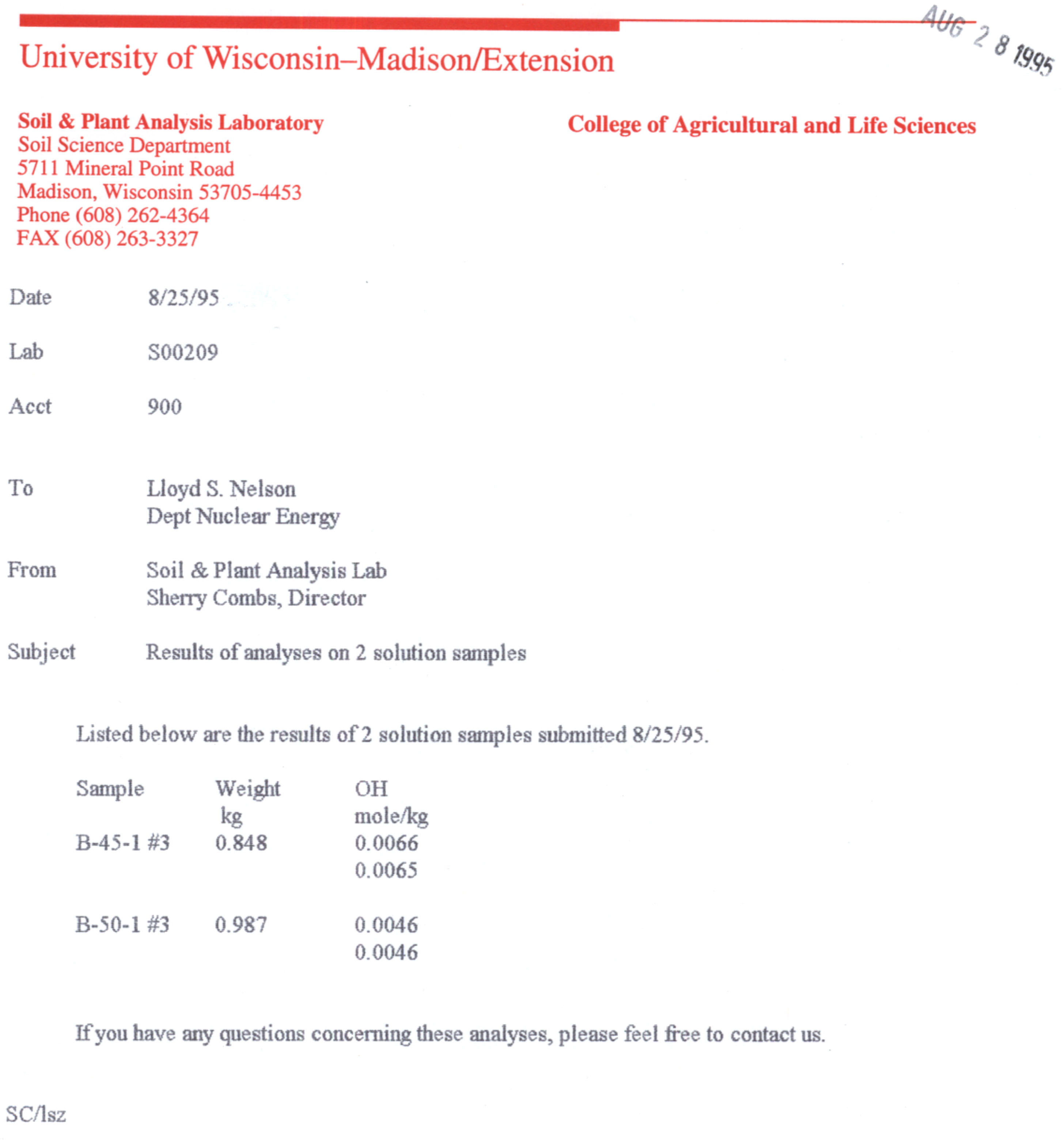
Listed below are the results of 8 solution samples submitted 8/15/95.

Sample	Weight kg	OH mole/kg
B-39-1	3.257	0.0280
		0.0276
B-43-1	1.069	0.0003
B-45-1	2.488	0.0279
		0.0280
B-50-1	2.226	0.0364
		0.0365
B-52-1	3.179	0.0288
		0.0283
B-45-1 #2	2.052	0.0003
		0.0003
B-50-1 #2	2.488	0.0003
		0.0002
B-52-1 #2	2.039	0.0002

If you have any questions concerning these analyses, please feel free to contact us.

SC/lsz

Figure D-1. Report of Titration Analyses of Eight Solutions of Hydroxyl Ion.



AUG 28 1995

University of Wisconsin–Madison/Extension

Soil & Plant Analysis Laboratory
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College of Agricultural and Life Sciences

Date 8/25/95

Lab S00209

Acct 900

To Lloyd S. Nelson
Dept Nuclear Energy

From Soil & Plant Analysis Lab
Sherry Combs, Director

Subject Results of analyses on 2 solution samples

Listed below are the results of 2 solution samples submitted 8/25/95.

Sample	Weight kg	OH mole/kg
B-45-1 #3	0.848	0.0066
		0.0065
B-50-1 #3	0.987	0.0046
		0.0046

If you have any questions concerning these analyses, please feel free to contact us.

SC/lsz

Figure D-2. Report of Titration Analyses of Two Solutions of Hydroxyl Ion.

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College of Agricultural and Life Sciences

November 2, 1995
Acct. No. **900**
Lab No. **S428**

TO: Lloyd Nelson
Nuclear Engr. Dept.
1500 Johnson Drive
U. of W., Madison
CAMPUS

FROM: Soil & Plant Analysis Lab
Sherry M. Combs, Director

SUBJECT: Results of analyses on 1 alloy sample.

Please find enclosed results of analyses on 1 alloy sample submitted October 25, 1995.

If you have any questions concerning these analyses, please feel free to contact us.

Enclosure(s)

SC/ss

Figure D-3. Report of the Inductively Coupled Plasma Mass Spectrometer Analysis of the Red-Brown Debris from Experiment B-50-1.

UW-Soil Plant Analysis Lab
5711 Mineral Point Road
Madison, WI 53705

S-428, Acct-Lloyd Nelson

Phone (608)262-4364
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11/1/95

All units = weight percent (%)
Results reported on a dry weight basis

Li	Cr	Ni	Pb	Sum
0.25	0.05	0.02	97.41	97.73

Note: the sample was dissolved with 5 ml of HNO3 at 140C for 4 hours, then the sampl was diluted to 50 ml with distilled de-ionized water.
Some residual was still left after digestion. This may have caused the sum below 100%.