Improving the Stability of Standard Platinum Resistance Thermometers

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Abstract

In order to improve the reliability and performance of standard platinum resistance thermometers (SPRTs), much research and investigation has been done at Fluke Calibration. A new short capsule SPRT was designed, gas mixtures for filling long-stem SPRTs were investigated, and the sensor support used in high-temperature SPRTs was redesigned. Through these efforts, the performance of SPRTs have improved. However, there are some difficult challenges to further improve the performance of SPRTs. In the paper, recent work to make further improvements is summarized.

1. Introduction

Standard platinum resistance thermometers (SPRTs) are used to interpolate temperature in the range from 13 K to 961.78 °C on the International Temperature Scale of 1990 (ITS-90). In the past decades, the reliability and performance of SPRTs has been troubled by instability. The uncertainty caused by SPRT instability is becoming a more significant proportion of total uncertainty in fixed point calibrations, since other uncertainty components have been reduced with technology improvements. Metal purity, for example, has increased, and resistance bridge accuracy has also improved. During recent CCT and regional key comparisons, it was found that the selected SPRTs were less stable than desired. Similar problems were noted by other national metrology institutes (NMIs). Performance, particularly the stability, of SPRTs—which include low-temperature capsule SPRTs, mid-temperature long-stem SPRTs, and high-temperature SPRTs—has seen little improvement over the years. In response to requests from NMIs, Fluke Calibration has been working to improve the performance of SPRTs, but there are some difficult challenges.

As one result of the investigation, it was found that it is extremely important that SPRTs be handled and operated correctly. SPRTs are quite fragile and are often inadvertently damaged by severe conditions or even routine use. A user must be able to recognize when an SPRT has been impaired, whether it can be restored, and what action is appropriate to take. In the paper, the recent improvements will be summarized and methods for evaluating the condition of SPRTs are presented and discussed.

2. Improving Standard Platinum Resistance Thermometers

2.1. Short capsule SPRT

As a result of needs of the Kelvin-Boltzmann projects and other research such as noise thermometry, a new 25-ohm glass capsule standard platinum resistance thermometer (SPRT) with a 20-mm long sensor and a 35-mm long capsule was developed. A glass capsule with the same thermal expansion coefficient as that of platinum is used. A new quartz cross support is specially designed. The main challenges are to shorten the length of capsule SPRTs from 65 mm to 35 mm, improve the stability further, and even expand the temperature range.

The temperature range of the capsule SPRT is from 13 K to 232 °C. Its rate of resistance drift at the triple point of water (Rtp) is better than the equivalent of 0.5 mK after the SPRT is exposed to liquid nitrogen (LN2) for 100 hours. The repeatability of the thermometer is better than 0.5 mK. As an example, the test results of three thermometers are shown in Figure 1, Figure 2, and Figure 3. The National Institute of Metrology of China (NIM) reported that the stability of this type of SPRT reached 0.03 mK when used at room temperature during a one year period.

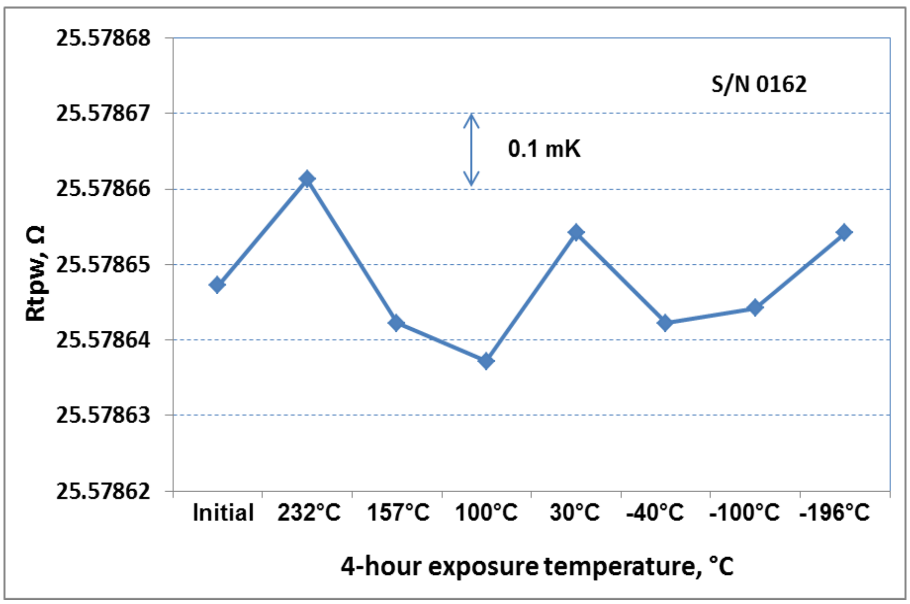

Fig1. RTPW drift was evaluated after each of the eight SPRTs was held in LN_2 (-196 °C) for 100 hours

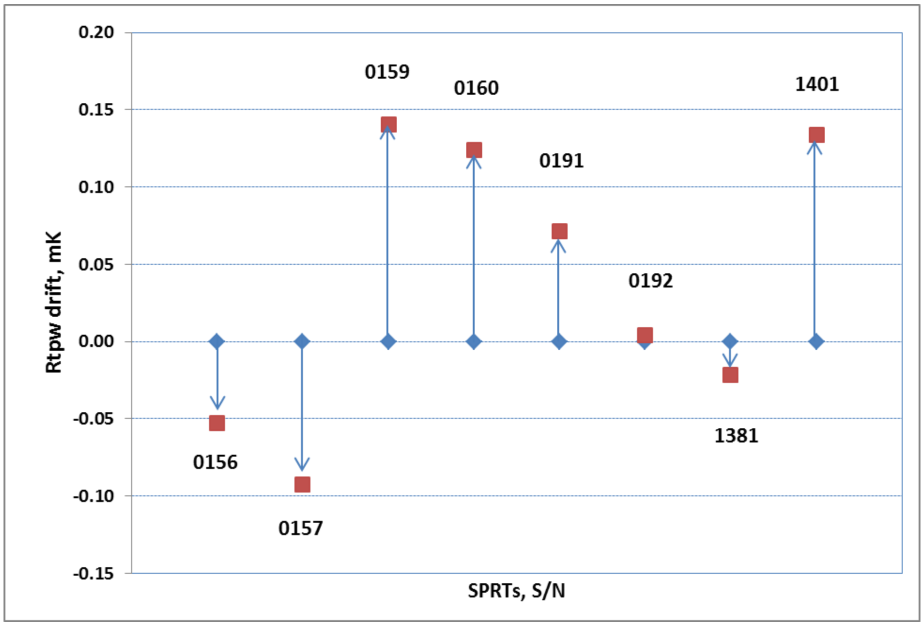

Fig. 2 Rtpw drift was evaluated after each of the eight SPRTs was held in LN_2 (-196 °C) for 100 hours

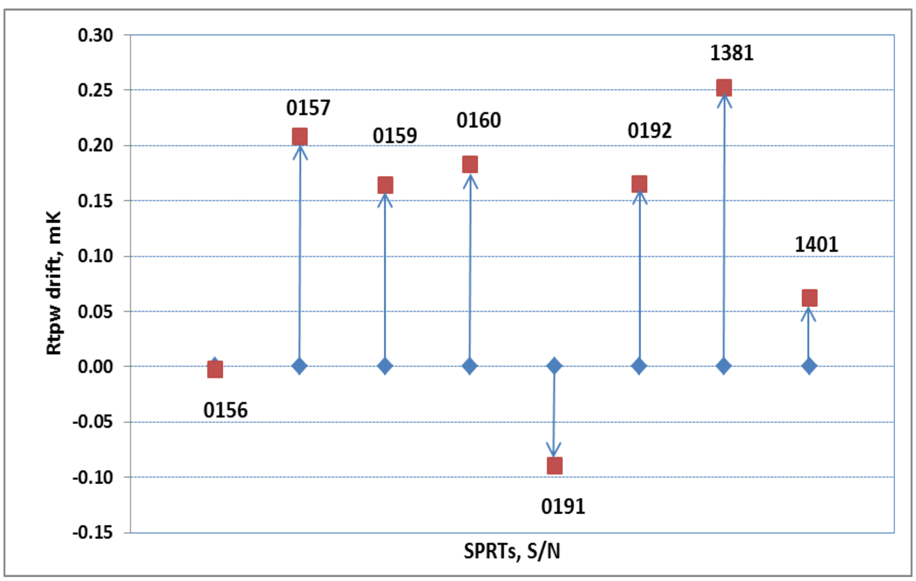

Fig. 3 Each of eight SPRTs was thermal cycled 10 times from LN_2 (held for 20 min) to 232 °C (stop at room temperature for 10 min)

2.2. Long-stem SPRT

The most important improvement of long-stem SPRTs is with the gas mixtures used to fill the sheath. The mixture is adjusted accordingly to the operating temperature range. Oxidation of the platinum wire is the main cause of resistance instability in the range from about 250 °C to

450 °C, aside from mechanical shock during handling. The drift rate caused by platinum oxidation strongly depends on the partial oxygen pressure. Higher partial oxygen pressure causes instability of the SPRT in the range from about 250 °C to 450 °C. An SPRT with partial oxygen pressure of 0.4 kPa showed no platinum oxidation at all [1]. As an example, the test results of an SPRT with partial oxygen pressure of 0.4 kPa is shown in Fig. 4.

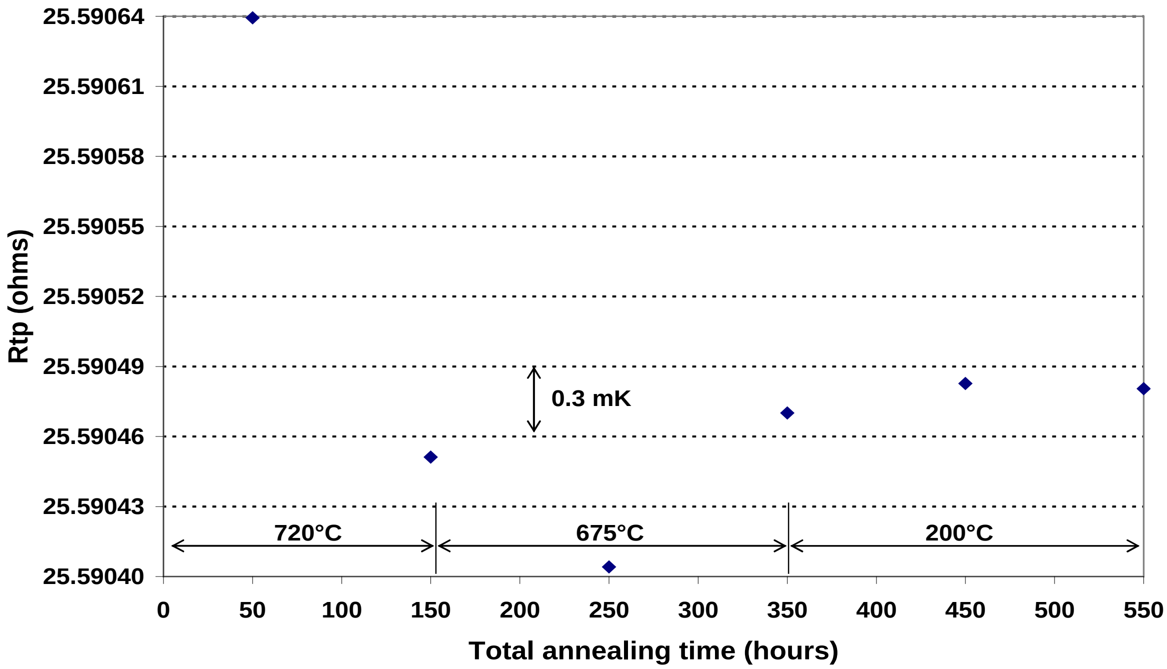

Fig. 4 Stability of an SPRT with partial oxygen pressure of 0.4 kPa

There is a little difference in behavior from oxidation effects between quartz sheathed SPRTs and Metal sheathed SPRTs [2]. Because of slow oxidation of the metal sheath and the consequent loss of oxygen in a metal sheathed PRT, the oxygen content of a metal sheathed SPRT element can significantly affect the performance if its element is not sealed separately from its metal sheath. It is very difficult to maintain a balance between the oxidation effect and element contamination in a metal sheathed SPRT, since the oxygen content in the thermometer can vary after a period of operation. A thermometer would show an oxidation effect after it was made, but no contamination problem. However, when no oxidation effect was detected, the thermometer was likely to become contaminated if it continued to be exposed to high temperature.

Our research confirms that a proper gas mixture (with a particular oxygen content) can be found that reaches a balance between the oxidation effect and element contamination [2]. The oxygen content in the gas mixture can be high enough to protect the sensor from contamination but low enough to minimize the oxidation effect. Because the sealed element can maintain its oxygen content for a long time, it becomes possible to build a metal sheathed SPRT with excellent performance at temperatures up to 675 °C.

2.3. High-temperature SPRT

Over the past 20 years, the reliability and performance of HTSPRTs has been troubled by instability and short circuits in the sensor coils. During the European Association of National Metrology Institutes (EUROMET) regional key comparison, EUROMET.T-K4 found that the selected HTSPRTs were less stable than desired, and one of the Bureau International des Poids et Mesures (BIPM) HTSPRTs failed due to a short circuit in the sensor [3]. Similar problems were reported in other laboratories such as National Metrology Institute of Japan (NMIJ).

To prevent short circuits, improve stability, and raise the upper temperature limit to the freezing point of copper (1084.62 °C), the high-temperature standard platinum thermometer (HTSPRT) was re-designed [4]. The most important change was an improvement in the structure of the sensor support. The strip support was replaced by a new specially designed cross support. Thorough testing, including observation of the long-term stability, short-term stability, and thermal cycling response, showed that the new thermometer has excellent performance in the temperature range from the triple point of water to the freezing point of silver. The long-term R(tpw) stability was improved from the original specification of 3.0 mK per 100 hours at 1070 °C to less than 1.5 mK per 100 hours at 1085°C. The long-term stability and thermal cycle test results support an extended the temperature range up to 1085°C, near the temperature of the freezing point of copper. As an example, the long-term stability and short-term stability at the triple point of water and the freezing point of silver are shown in Fig. 5, Fig. 6, Fig. 7 and Fig. 8.

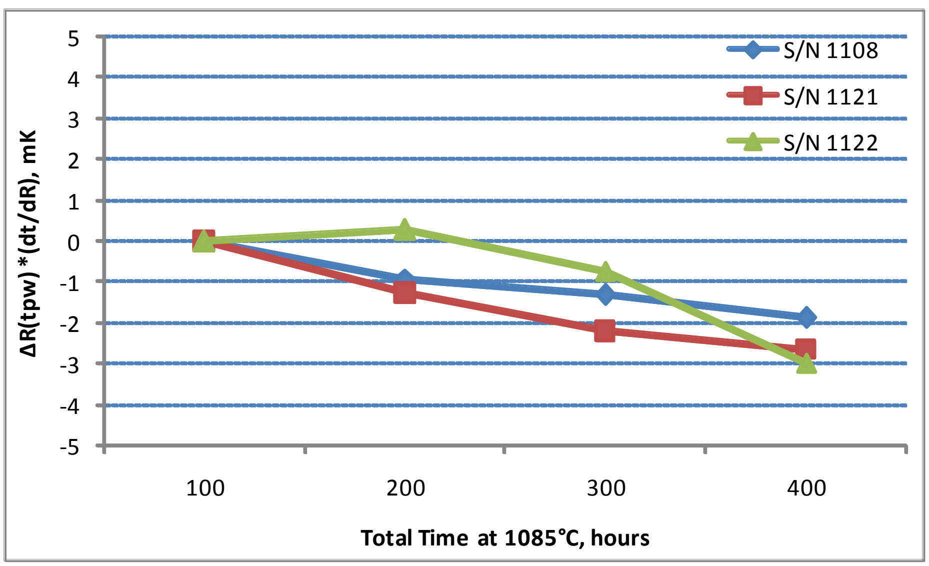

Fig. 5. Long-term stability at the triple point of water

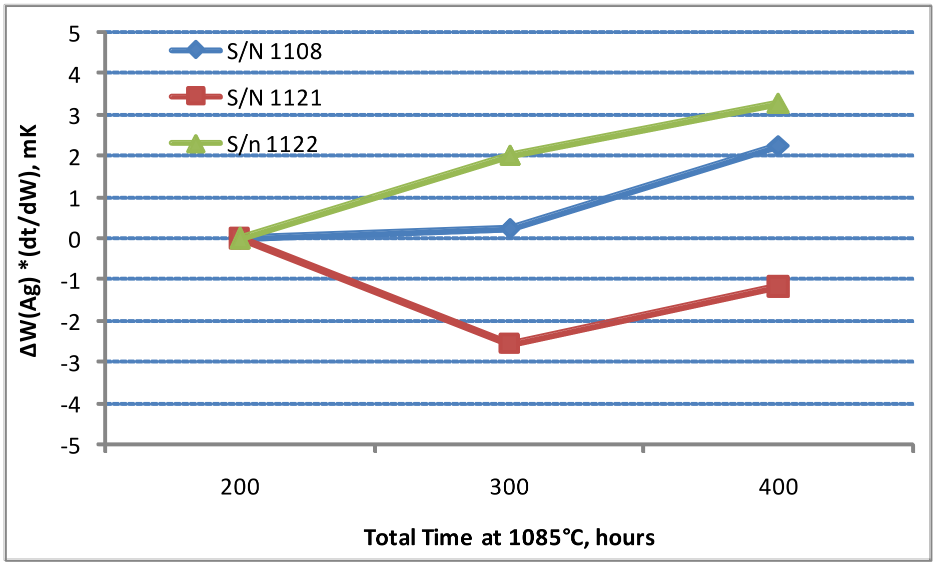

Fig. 6 Long-term stability at the freezing point of silver

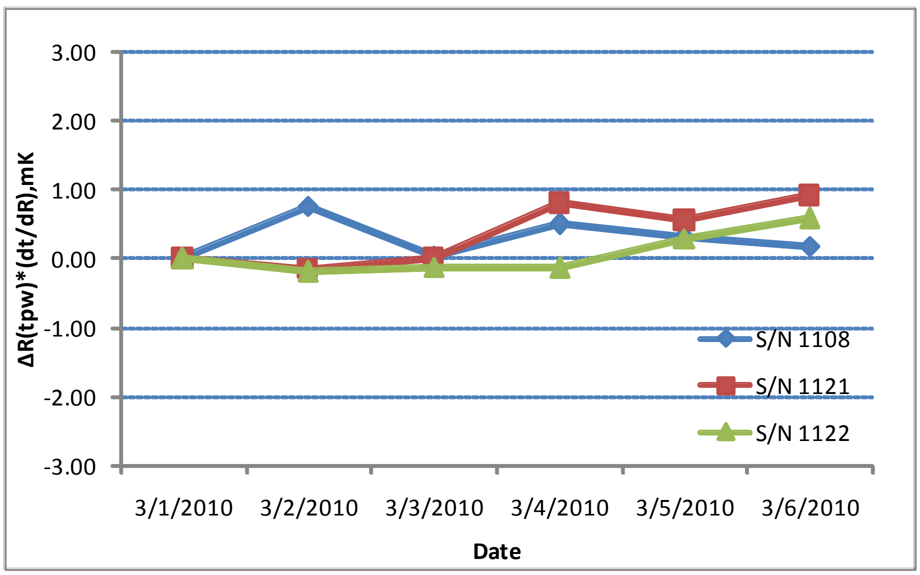

Fig. 7. Short-term stability at the triple point of water

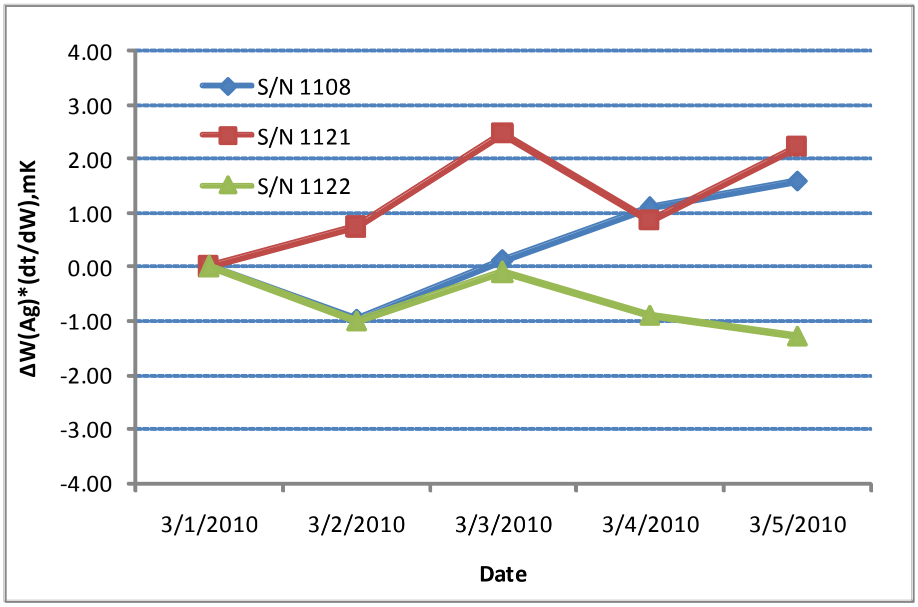

Fig. 8. Short-term stability at the freezing point of silver

3. SPRT handling and evaluation

Upon investigation of service records of SPRTs and PRTs, the primary cause of drift observed in SPRTs and PRTs was found to be improper handling. Being fully annealed during manufacturing, the platinum wire is relatively soft. Particularly with SPRTs, the sensor coils can easily move and touch adjacent coils, causing a short circuit. Vibration, shock, and temperature cycling during handling and use cause strain in an SPRT, which changes its resistance.

Annealing is a recommended procedure that usually can reverse effects of minor strain and oxidation in an SPRT or PRT and restore performance. It involves heating the thermometer to a high temperature and holding it there for a period of time. Because of susceptibility to further oxidation, performance of an SPRT or PRT may degrade if it is annealed at a temperature below about 450 °C. The annealing temperature should be at the maximum operating temperature of the thermometer, preferably between 550 °C to 660 °C if possible. It is suggested that the initial annealing period should be four hours. The R(tpw) should be measured before and after annealing. If the annealing temperature is higher than 500 °C, it is recommended to finish the process by gradually reducing the temperature to between 480 °C and 500 °C at a rate of 1.8 °C per minute, and then remove the thermometer from the annealing furnace. Proper annealing removes strain and oxidation effects and is required to maintain accuracy and stability

The block in the annealing furnace should be of a non-contaminating material such as graphite or alumina rather than a base metal. The thermometer sheath should be carefully cleaned with ethanol before it is inserted into the annealing furnace.

SPRTs and PRTs should be regularly tested to check for damage or adverse effects that degrade accuracy [5]. Evaluation can be based on comparison results from simple measurements. Required equipment includes a non-contaminating annealing furnace, a resistance measurement instrument such as a Fluke Calibration model 1594A or 1595A Super-Thermometer, a triple point of water cell and its maintenance bath, a melting point of gallium cell and its maintenance device, and/or other fixed-point cells.

The evaluation relies on observation of the thermometer's R(tpw). As part of the calibration quality assurance, the R(tpw) of an SPRT or a secondary PRT should be measured regularly and tracked. If it is found to have drifted, further examination is needed. Ideally, the W(Ga) should then be measured. If the melting point of gallium is not available, other fixed-point cells, such as tin or zinc, may suffice.

With metal-sheathed SPRTs and PRTs, the insulation resistance can be checked by measuring the resistance between the sheath and any of the leads with a megohmmeter after the thermometer has been exposed to 0 °C or lower for some time and then returned to room temperature. If the insulation resistance is lower than that specified by the manufacturer, the seal might be compromised and the thermometer should be repaired. With a fused-quartz-glass SPRT, insulation resistance cannot be measured directly. However, moisture can be detected by inserting the SPRT into a cold bath or container of crushed dry ice for a few hours and looking for condensation that appears inside the sheath. Methods of evaluating SPRTs were published in a previous paper [6].

4. Conclusions

Through years, the performance of all three typeof SPRTs—low-temperature capsule SPRTs, mid-temperature long-stem SPRTs, and high-temperature SPRT—have improved. However, there are still some difficult challenges to improve the stability of SPRTs further. It may be necessary to cooperate with sensor wire manufacturers to improve the reference grade platinum wire, since it was found that the quality of platinum wire varies. Improvements in the filling gas mixture may also lead to better stability by properly balancing oxidation and contamination effects. More research on oxidation in SPRT sensors is necessary.

As one result of the investigation, it was found that it is extremely important that SPRTs be handled and operated correctly. SPRTs are quite fragile and are often inadvertently damaged by severe conditions or even routine use. A user must be able to recognize when an SPRT has been impaired, whether it can be restored, and what action is appropriate to take.

Acknowledgments

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