An Electrochemical Method to Predict Corrosion Rates in Soils

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Linear polarization resistance (LPR) testing of soils has been used extensively by a number of water utilities across Australia for many years now to determine the condition of buried ferrous water mains. The LPR test itself is a relatively simple, inexpensive test that serves as a substitute for actual exhumation and physical inspection of buried water mains to determine corrosion losses. LPR testing results (and the corresponding pit depth estimates) in combination with proprietary pipe failure algorithms can provide useful predictive tools in determining the current and future conditions of an asset. A number of LPR tests have been developed on soil by various researchers over the years, but few have gained widespread commercial use, partly due to the difficulty in replicating the results.

This author developed an electrochemical cell that was suitable for LPR soil testing and utilized this cell to test a series of soil samples obtained through an extensive program of field exhumations. The objective of this testing was to examine the relationship between short-term electrochemical testing and long-term in-situ corrosion of buried water mains, utilizing an LPR test that could be robustly replicated. Forty-one soil samples and related corrosion data were obtained from ad hoc condition assessments of buried water mains located throughout the Hunter region of New South Wales, Australia. Each sample was subjected to the electrochemical test developed by the author, and the resulting polarization data were compared with long-term pitting data obtained from each water main. The results of this testing program enabled the author to undertake a comprehensive review of the LPR technique as it is applied to soils and to examine whether correlations can be made between LPR testing results and long-term field corrosion.

Keywords: electrochemistry, corrosion, LPR, long-term prediction, cast iron

1. Introduction

Previous work by the author has focused on some of the physical and practical issues of short-term electrochemical testing using Linear Polarisation Resistance (LPR). The technique can estimate short-term corrosion rates and gives an insight to the different corrosion mechanisms present in a soil environment. What has not been well demonstrated, to date, is how short-term electrochemical testing relates to long-term field corrosion. A successful correlation between short-term corrosion testing and long-term field corrosion would give the asset owner access to a valuable predictive tool. The purpose of this paper is to present some preliminary results of a soil sampling and testing program utilising LPR and, in light of these results, examine the applicability of the LPR technique to the measurement of long-term corrosion rates.

The author has developed a unique electrochemical cell, which has been shown through an extensive testing program to be robust and reliable for LPR testing of soils. Previous work by the author using this electrochemical cell has focused more on the effects of both the cell components and the soil being tested, rather than the possible use of the technique as a predictive tool. What has not been demonstrated to date is a correlation between this electrochemical test and long-term field pitting. This research was conducted in conjunction with a major pipeline condition assessment program undertaken for a leading Australian water utility; this program has been undertaken over many years and has covered hundreds of kilometres of pipeline to date. A number of samples and corresponding pitting data were collected during this program, in order to compare the outcomes of testing using the electrochemical cell with field data. The history of cast iron pipe manufacture and installation has been reviewed elsewhere and will not be discussed here; however it

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should be noted that during this research program, samples have been collected on pipelines installed from around 1885 through to 1981. As such, a broad range of cast irons, corrosion rates and bedding conditions can be expected from within the analysed samples; this variation should be regarded as necessary in order to obtain a broad scope of pitting ranges required to test an overall calibration. To date, 41 samples have been collected as part of this research, while some other soils have been used for additional testing.

The overall objective of this research program is to develop a reliable, reproducible and simple electrochemical test that is able to accurately determine the corrosivity of a soil, thereby providing a predictive tool for determining the likely condition of an asset. This paper will describe the results achieved to date and will present some early conclusions about the veracity of the LPR technique, as currently applied in the water industry. Based on previous experience by a number of authors (for example⁹), it is acknowledged that there are many factors involved in both producing and interpreting electrochemical results. Furthermore, there are clearly more factors to soil corrosion than intrinsic soil attributes, such as the local environment. These are not fully understood at the present time. However the data collected as part of this research will be invaluable, should a correlation between field pitting and short-term electrochemical testing be established.

2. Corrosion Data Collection and Soil Sample Collection

The collection of soil samples and accompanying field data was an ad-hoc process, being largely determined by the requirements of the broader condition assessment program. As part of this process, selected pipes are exhumed and grit blasted to reveal the extent of corrosion; the physical exhumation served as an independent arbitrator of the condition assessment process. In general, these exhumation sites were chosen near previous failures, however this was not always possible due to the requirements of the condition assessment program and occupational health and safety issues. It was sometimes necessary to exhume pipes of a certain age, not only to confirm bedding conditions, but to confirm pipe material. Fig. 1 shows a typical cast iron pipe after exhumation and abrasive blasting, while Fig. 2 shows the typical location for soil sample collection at an exhumation site.

As each pipeline was subject to abrasive blasting to expose corrosion, the grit/metal from the sandblasting procedure was cleared prior to sampling, to ensure that only natural soil was collected. Each soil sample was bagged and labelled according to the sample location. Basic field recording was also undertaken during the collection of each sample. Descriptions of the bedding conditions and the presence of any imported backfill were recorded, and other key physical characteristics were noted for each sample, including the presence of ground water, sand backfill, physical measurements of the pipe and observations regarding the possible presence of iron and sulphate reducing bacteria. The details of this work, and the specifics of the sampling methodology, have been discussed further elsewhere⁹.

Table 1 presents the key attributes and corrosion related information for each sample. For reference, the manufacturing types shown in Table 1 are as represented as follows:

- Statically cast pipe (referred to as SC) – pipes nominally manufactured prior to 1929 (noting that both vertical and horizontal casting took place);
- Australian Iron and Steel (AIS) pipe (referred to as DEL) – pipes manufactured between 1929 and 1942 using the ‘Delevaud’ process;
Table 1. Cast Iron pipe samples and corrosion details Soil Preparation and Electrochemical Method

| Sample No. | Pipe Details | Manufacturing type | Age at inspection (yrs.) | Maximum Pit Depth (mm) | Pitting Rate (mm/yr.) |
|------------|--------------|--------------------|--------------------------|------------------------|----------------------|
| 1          | DN 375 CICL 1961 | SDELII            | 46                       | 1                      | 0.022                |
| 2          | DN 375 CICL 1955 | SDELI             | 52                       | 2.1                    | 0.040                |
| 3          | DN 375 CICL 1942 | DEL               | 65                       | 8.2                    | 0.126                |
| 4          | DN 600 CICL 1928 | SC                | 82                       | 8.2                    | 0.100                |
| 5          | DN 375 CICL 1978 | SDELII            | 29                       | 1.5                    | 0.052                |
| 6          | DN 500 CICL 1955 | SDELI             | 54                       | 8.4                    | 0.156                |
| 7          | DN 375 CICL 1969 | SDELII            | 40                       | 1.2                    | 0.030                |
| 8          | DN 300 CICL 1968 | SDELII            | 41                       | 3.1                    | 0.076                |
| 9          | DN 375 CICL 1959 | SDELI             | 51                       | 13                     | 0.255                |
| 10         | DN 200 CI 1916  | SC                | 94                       | 7.8                    | 0.083                |
| 11         | DN 250 CICL 1968 | SDELII            | 42                       | 5.2                    | 0.124                |
| 12         | DN 200 CICL 1941 | SDELI             | 70                       | 5                      | 0.071                |
| 13         | DN 200 CICL 1947 | SDELI             | 64                       | 7.2                    | 0.113                |
| 14         | DN 300 CICL 1976 | SDELII            | 35                       | 12.7                   | 0.363                |
| 15         | DN 500 CICL 1942 | SDELI             | 69                       | 6.4                    | 0.093                |
| 16         | DN 200 CICL 1970 | SDELII            | 43                       | 1.4                    | 0.033                |
| 17         | DN 200 CICL 1942 | SDELI             | 69                       | 12.9                   | 0.187                |
| 18         | DN 150 CICL 1966 | SDELII            | 46                       | 8                      | 0.174                |
| 19         | DN 200 CI 1941  | SDELI             | 71                       | 6.5                    | 0.092                |
| 20         | DN 250 CI 1936  | DEL               | 76                       | 2.5                    | 0.033                |
| 21         | DN 250 CICL 1935 | DEL               | 77                       | 7.3                    | 0.095                |
| 22         | DN 500 CICL 1964 | SDELII            | 48                       | 3.5                    | 0.073                |
| 23         | DN 500 CICL 1959 | SDELI             | 53                       | 4                      | 0.075                |
| 24         | DN 200 CI 1957  | SDELI             | 55                       | 7.6                    | 0.138                |
| 25         | DN 375 CICL 1969 | SDELII            | 43                       | 3.8                    | 0.088                |
| 26         | DN 500 CICL 1976 | SDELII            | 37                       | 1.8                    | 0.049                |
| 27         | DN 500 CICL 1976 | SDELII            | 37                       | 3.7                    | 0.100                |
| 28         | DN 200 CICL 1955 | SDELI             | 58                       | 7.6                    | 0.131                |
| 29         | DN 200 CICL 1979 | SDELI             | 33                       | 7.2                    | 0.218                |
| 30         | DN 300 CICL 1962 | SDELI             | 50                       | 7.7                    | 0.154                |
| 31         | DN 200 CICL 1962 | SDELI             | 50                       | 10                     | 0.200                |
| 32         | DN 450 CI 1923  | SC                | 89                       | 8.3                    | 0.093                |
| 33         | DN 500 CICL 1959 | SDELI             | 53                       | 7.5                    | 0.142                |
| 34         | DN 300 CICL 1967 | SDELII            | 45                       | 6.5                    | 0.144                |
| 35         | DN 500 CICL 1939 | DEL               | 73                       | 6.5                    | 0.089                |
| 36         | DN 375 CI 1884  | SC                | 129                      | 6.5                    | 0.050                |
| 37         | DN 375 CI 1884  | SC                | 129                      | 12.5                   | 0.097                |
| 38         | DN 375 CI 1884  | SC                | 129                      | 6.5                    | 0.050                |
| 39         | DN 600 CICL 1930 | SC                | 82                       | 9.2                    | 0.112                |
| 40         | DN 375 CI 1884  | SC                | 129                      | 10.5                   | 0.081                |
| 41         | DN 375 CI 1884  | SC                | 129                      | 9.5                    | 0.074                |

- AIS Super ‘DeLevaud’ (referred to as SDELI) – pipes manufactured between 1942 and 1962 using the Super ‘DeLevaud’ process; and
- Pipes manufactured at Yenarra (referred to as SDELI) – that is pipes manufactured by Tubemakers between 1962 and 1978 (note that these were also manufactured using the Super ‘DeLevaud’ process).

Following on from SDELI case iron pipe, ductile iron pipes were introduced in Australia in 1978. No data was obtained for material after this date.

All collected soil samples were subject to the same soil preparation techniques. After sample collection, the soil was dried in an oven (nominally the oven was set at 110 °C); after a period of between 24 and 48 hours the sample...
was dry. The samples were mechanically broken into small pieces that were then processed through a Retch mortar and pestle grinder. This reduced the sample to a dry aggregate of the original soil. The sample was then sieved to remove particles larger than 2 mm, which are defined as gravel according to ASTM methods. Samples were then stored until use in sealed plastic buckets.

To determine the wilt point of each soil prior to any electrochemical testing, a WP4C dew point water potential meter was used as per the method set out by Campbell. Soil preparation for electrochemical testing was undertaken by manually mixing an arbitrary amount of soil on a marble bench with distilled water until the desired moisture content was achieved. Wetted soil was allowed to stabilize for at least one day prior to testing, to ensure that the moisture content was homogenous.

The electrochemical cell used for the experiment was designed by the author after careful consideration of past electrochemical cells. These factors include, but were not limited to:

- reproducibility;
- operator independence;
- sound electrochemical design;
- interchangeable components; and
- the ability to vary the compaction of the soil

With that in mind, Fig. 3 shows a schematic of the electrochemical cell in its present configuration, whilst Fig. 4 shows three electrochemical cells during an electrochemical experiment. Note that the clamps visible in Fig. 4 help to ensure that contact is maintained between the electrodes and compacted soil. Early prototypes of the cell (Dafter 2009) were unsuccessful in removing errors caused by soil/electrode interface separation.

Detailed operating instructions for the cell have not been presented in this paper; they will be discussed in future publications. For now, it is sufficient to say that electro-
chemical exposures for this present work were limited to short-term exposures (typically less than 30 minutes) and involved a single EIS measurement followed by an LPR scan following potential stabilisation.

4. Results

A plot of the short-term polarisation resistance results against the pitting rate (simplified to a linear pit rate) has been presented Fig. 5 for all samples subject to this series of tests. Fig. 5 demonstrates that there is no general corre-
lation between short-term measured polarisation resistance and pitting rates. Given the breadth and diversity of the soil samples tested, respective pipe ages and variations in pitting depths, this lack of correlation is not entirely unexpected. Furthermore, there does not appear to be a general correlation between pit depths and low polarisation resistance; a number of samples reported a high polarization resistance and high pitting rates (Samples 1 and 2, for example).

Given the poor general correlation observed between the 41 samples examined for this testing, an attempt was made to distil the results into more logical subsets of data (based on key physical variables, which are known to affect the observed corrosion), and examine these subsets for a positive correlation. As such, the samples were separated out based on physical characteristics at each site (for example, the presence of a sand surround) and were examined for a relationship between $R_p$ and pitting rates. A linear fit of the data is presented for each mode of analysis.

Attempts were made to examine the data based on the following four modes of analysis, which are discussed further below:

1. This initial analysis considered all samples, with the exception of sandy soils and samples with a sand surround/bedding (Fig. 6).
2. This analysis was the same as the first, but excluded all samples where the water table had been present within the trench (Fig. 7).
3. This analysis was the same as the second, but excluded three samples where the measured moisture content at collection was not roughly halfway between the wilt point and saturation (i.e. samples are relative to the wilt point) (Fig. 8).
4. The final analysis was the same as the third, but included the three samples that had been previously excluded (Fig. 9). Note that this is therefore the same data presented in Fig. 8, but with the green data points included in the relationship presented. This figure has been included as it represents the relationship between all native soils (for the purposes of this paper this is soils in the absence of select bedding and/or under no influence from the local water table) and short-term measurement polarisation resistances.

Based on the results presented in Fig. 6 to Fig. 9, no general correlation between pitting rate and polarisation resistance was identified. Therefore, the data from the final mode of analysis (as outlined in point 4 and Fig. 9) was subsequently divided into individual pipe manufacturing types (refer to Table 1 for a full description of each manufacturing type), in order to further investigate any possible correlations between pipes of a similar age/type. Unfortunately, because the samples that involved a water table, sand bedding or a sand surround had been excluded from the entire data set, only one sample remained from the pipes that were manufactured using the SDELII process, and only two samples remained from pipes manufactured by the DEL process. For this reason, there is little value in presenting the results for these two manufacturing types (this data is, however, included for reference in the summary chart provided as Fig. 12). Therefore, only the data for the SDEL and SC samples is presented in detail.
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The SDELI samples (sample numbers 7, 10, 14, 26, 27, 31 and 41) showed a strong correlation, as shown in Fig. 10. However it should be noted that sample 31 was known to undergo periods of full submersion during high rainfall events, and removal of this sample improves the correlation from $R^2 = 0.76$ to $R^2 = 0.92$. A correlation was also identified between the SC samples (sample numbers 5, 42, 50, 51 and 53). The correlation is based on clay samples only, as the loam samples all appeared to sit in a separate datum (this includes sample numbers 11, 47 and 50, represented by green data points).

Both the SDELI and SC pipe samples demonstrate a strong correlation between polarisation resistance and corrosion rates (as shown in Fig. 10 and Fig. 11). It is noteworthy that all loam samples had a lower corrosion rate than those in clays, which is to be expected. A summary chart of the data as analysed for each pipe class is provided in Fig. 12 (note that the loam samples, indicated by green in Fig. 11, are not included in this figure).

The results of this analysis indicate that there is a good correlation between short-term electrochemical testing and...
long-term pitting observed within pipes of a similar age. However it should be stressed that only clay samples were used for the analysis presented in Fig. 12. This data also indicates that the corrosion rates observed for SDELI pipes are significantly higher than the rates identified for SC pipes. This suggests that SDELI pipes are more susceptible to corrosion than SC iron pipes (it is acknowledged that there was insufficient data in relation to the DEL and SDELI pipes for these particular manufacturing types to be accurately critiqued in the same manner). Given the positive correlation for SDELI and SC pipes, it is postulated that a similar relationship could also be identified for DEL pipes given sufficient samples. The author is of the opinion that such a relationship should also occur on SDELI pipes. However, further testing would be required to demonstrate such relationships. Obtaining sufficient samples that fit the criteria of having no imported sand bedding or select backfill would be the principal difficulty with undertaking this additional testing. Of the 13 samples collected for this research on SDELI pipes, only two were not impacted by bedding or sandy soils of some kind.

5. Discussion

The correlations derived from the analysis of specific pipe types (as summarised in Fig. 12) are considerably stronger that the relationships derived from the general modes of analysis undertaken on all samples collectively. This outcome indicates that the LPR technique can be used successfully in specific applications to estimate the long-term corrosion rates of ferrous pipes in soils.

The results presented in this research offer some significant advantages to those achieved by Ferguson \(^{13}\) and Hay \(^{16}\), in that for each separate pipe class (or age) there is a separate calibration. This is important, as there is clear evidence that pit rates diminish with time. The use of a single calibration is of little use for older cast irons and this second calibration is essential in order to not over-estimate corrosion losses.

It should be noted that cast iron pipes might have had an initial protective coating of bitumen. This is anecdotal however, and some pipes may have had no coating at all \(^{15}\). A bitumen coating, depending on the thickness, may have an effect on the corrosion rate of a soil towards ferrous materials. This effect may be greater for older cast irons that were dipped in hot bitumen, rather than having a brush applied coating \(^{15}\). It is possible that the significant difference in the calibrations obtained for vertically cast iron pipes and Super DeLevaud pipes may be due to this variable. However no information is available for each of the pipe samples obtained for this research, so it cannot be considered as a separate variable and has thus been ignored.

As briefly outline in Section 4, the maximum pit depth measured on cast iron pipes was found to have no correlation with a short-term electrochemical test in certain scenarios. These scenarios can be summarised as follows:

- Sand backfill/sandy soils – There were five pipes that were buried in native sand soils. For the corresponding soil samples (#1, 2, 23, 29, and 30), the measured polarisation resistance was higher than 10000 ohms-cm\(^2\). It is worth noting that only a single sample (#30) from within this class of samples recorded a pitting rate higher than 0.05 mm/yr.

- Imported bedding – The pipes installed with imported bedding can be divided into two categories: (i) those where the bedding had been installed correctly (100 mm full sand surround), or (ii) those where bedding was poorly installed and the bedding and native soil were spatially separate and both were in contact with the pipe. It should be noted that for all these pipes, native soil was selected to perform the calibration against pitting; there was no correlation between LPR results and corrosion of pipes with a sand surround. It should also be noted that for soils where the bedding was correctly installed and intact (samples 6, 8, 9, 17, 25, 26 and 28), the measured corrosion rates were less than 0.07 mm/yr in all but one case (sample 25). For the three samples where the bedding was poor or only partially surrounding the pipe (samples 15, 38 and 39), the corrosion rate was considerably higher than LPR would predict. For one pipe sample, a corrosion rate of 0.34 mm/yr. was recorded.

- Water table – There were nine pipe samples that were, to varying degrees, partially submerged under the water table (samples 4, 13, 16, 20, 21, 44, 45, 46 and 48). The corrosivity of these samples varied, with measured pitting rates between 0.07 and 0.18 mm/yr. Note that if a single sample is removed from this data set, an inverse relationship is observed to those previously presented (lower R\(_p\) results in a lower corrosion rate).

It was observed during the field data collection for this work that the corrosion observed on bedded and sand surround pipes was often the result of native soil infiltrating the imported bedding. Due to this, pitting rates can actually be quite high (in the order of 0.1 mm/yr.), however the amount of metal lost is, in comparison to a wholly native soil, several orders of magnitude less, principally due to increased drainage.

The correlations presented in Fig. 12 also suggest that long-term electrochemical testing of soils may be unnecessary, and that meaningful results can be obtained.
with a short-term test at $E_{corr}$ stabilisation. However in order to examine this in depth, field pitting data is required to calibrate the results. This is not to suggest that longer-term electrochemical testing of corrosion rates is invalid, as many authors have achieved good correlations using electrochemical techniques\(^{(15)}\), but that caution needs to be taken in their interpretation. Further discussion on measurement of corrosion rates in soils will be presented in subsequent publications.

6. Conclusions

Based on the data presented herein, the following conclusions can be made:

- Short-term testing of forty-one soil samples using electrochemical testing showed that there is no clear general correlation between polarisation resistance and pitting rates. There was, however, a good correlation between polarisation and pitting rates for specific cohorts of cast iron pipe in native soils. Based on this result, LPR is considered to be a valid experimental method to predict pitting rates in the long-term.

- No correlation was obtained for pipe samples buried in a partial or full sand surround or for cast iron pipes in contact with the water table. For cast iron pipes buried in the water table, the inverse of the above relationship was observed (i.e., lower $R_p$ = lower pitting rate). This outcome indicates that LPR testing is not suited to the prediction of pitting rates in these specific circumstances.

- Despite the limitations of short-term testing versus long-term corrosion processes\(^{(17)}\), the electrochemical test developed as part of this research provides a simple and cost effective way to estimate the pitting rates of cast iron pipes in soils.

In addition to the results presented above, the following should also be considered:

- The packing technique used to compact soil into this electrochemical cell is not truly operator dependent\(^{(12)}\). For this reason, it is suggested that results from different laboratories looking to replicate this technique may not be comparable. Further work is required to address this variable.

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References

1. D. M. F. Nicholas, Review of the Linear Polarisation Resistance Technique (LPR) Methodology for the Condition Assessment of Buried Ferrous Pressure Pipelines, WSAA (2009).
2. M. R. Dafter, Proceedings of the ACA Conference on An Electrochemical Method to Measure Corrosion in Soils, ACA, Coffs Harbour (2009).
3. M. R. Dafter, Proceedings of the ACA Conference on Practical Considerations for Electrochemical Testing of Soils, ACA, Adelaide (2010).
4. M. R. Dafter, Proceedings of the ACA Conference on Electrochemical Impedance Spectroscopy of Soils, ACA, Perth(2011).
5. M. R. Dafter, D. M. F. Nicholas and R. E. Melchers, Proceedings of the ACA Conference on Prediction of Long-Term Corrosion in Soils using Electrochemical Tests, ACA, Melbourne (2012).
6. M. R. Dafter, Proceedings of the ACA Conference on Long-Term Corrosion of Buried Watermains Compared with Short-Term Electrochemical Testing, ACA, Brisbane (2013).
7. D. M. F Nicholas, and G. Moore, Proceedings of the ACA Corrosion Conference on Corrosion of Ferrous Watermains: Past Performance and Future Prediction: a Review, ACA, Sydney (2007).
8. I. S. Cole and D. Marmey, Corros. Sci., 56, 5(2012).
9. R. B. Petersen, M. R. Dafter and R. E. Melchers, Proceedings of the ACA Corrosion Conference on Modelling the Long-Term Corrosion of Cast Iron Pipes, ACA, Brisbane (2013).
10. ASTM D6913-04, Standard Test Methods for Particle-Size Distribution (Graduation) of Soils using Sieve Analysis (2009).
11. G. S. Campbell, Determining the -15bar (permanent with) Water Content of Soils with the WP4C, Application Note, http://www.decagon.com.
12. M. R. Dafter, Ph. D. Thesis, University of Newcastle (2014).
13. M. Heathcote and D. M. F. Nicholas, Life assessment of Large Cast Iron Watermains, UWRRA Report 146 (1998).
14. L. Hay, Ms. Thesis, The University of Sydney (1984).
15. D. M. F. Nicholas, Private correspondence (2014).
16. M. Norin and T.-G. Vinka, Mater. Corros., 54, 641 (2003).
17. R. E. Melchers, Proceedings of the ACA Conference on Long-Term Corrosion of Grey Cast Iron in Marine Environments, Melbourne (2012).