Brief critique of the two latest CSIRO reports – John Black 24 November 2017

Report: Burrup Peninsula Aboriginal petroglyphs: colour and spectral mineralogy 2004-2017
by Noel Duffy, Erick Ramanaidou, David Alexander and Deborah Lau. EP161761 June 2017

The most important outcome from the latest report on changes in the colour Murujuga rock surfaces over time is that, contrary to previous reports, the authors now conclude there have been significant colour changes between 2004 and 2016. The authors now acknowledge that the main conclusion in the previous 2015 report: "*The comparison of the colour and spectral data collected and processed for both the Northern (control sites) and Southern sites has shown no consistent trend in an increasing or decreasing direction*" is not correct.

The authors also make an important statement: *"..... that the results of this report supersede all results previously published*".

This statement is an important admission to substantial errors in analysis and interpretation of all previous reports that have been used by the Western Australian and Federal governments and industry to place the ammonium nitrate production facility in the midst of the rock art and to justify its high levels of emissions.

The results provided in the current report show an average colour change of 11.94 ΔE units at the southern sites and 13.80 ΔE units at the northern sites. The international colour change measure (ΔE) has a scale of 0 to 100, which means in 13 years since measurement commenced there was a reported change in colour of approximately 13%. Such a major change over a short period of time should be of concern for preservation of the rock art.

The authors, without providing a logical reason, attempt to diminish the value of the significant of the findings relating to colour changes and to changes in lightness of the rocks.

All data collected during the first five years of measurement were discarded with results from 2009 to 2016 only being included in the final statistical analysis. Nevertheless, the statistical analyses presented in the report do show significant changes in lightness of the rocks over time of measurement. These significant changes in colour were found with both the KM and ASD instruments, despite the high variance in measurements from year to year. The authors recognise the high variance in their ASD instrument measurements (partly because there was no removal and replacement of the instrument for repeat measurements until year 2016). An improved design of the experimental procedures would have substantially reduced this between year variation.

The report also states there are no significant differences in colour change between northern and southern sites. No statistically significant differences would be expected between the two regions as shown by the independent reviewer of the previous reports appointed by the WA government. The inclusion of only two northern sites was shown to be vastly insufficient, if the aim of the research was to prove differences between locations in colour change of rocks and petroglyphs. Selection of the northern sites as 'control' sites has also been criticised and recognised by the Burrup Rock Art Technical Working Group as being inappropriate because of the large number of annual ship movements (19,000) in close proximity to the northern sites.

The independent reviewer engaged by the Western Australian Government remains highly critical of the experimental and statistical methods used, which has resulted in wide variation of measurements and difficulties with interpretations. Many of the concerns about methodology have been included in a paper "Inadequacies of research used to monitor change to rock art and regulate industry on Murujuga ('Burrup Peninsula'), Australia", published in Rock Art Research[1].

Report: Extreme weathering experiments on the Burrup Peninsula / Murujuga weathered gabbros and granophyres By Erick Ramanaidou, Gay Walton and Derek Winchester: Report EP172193 August 2017

This report is of little value for assessing the effect of nitric acid, sulphuric acid, ammonia or ammonium nitrate load on dissolution of rock surfaces or petroglyphs on Murujuga. CSIRO accept this interpretation of the research in their revised report by now stating it should be viewed as a preliminary study and should not be used as an indication for permissible emissions levels.

The first report on the extreme weathering research dated May 2017 was placed on the WA Government website around 22 June 2017. The report was reviewed by Dr Ian MacLeod (former Director of the WA Maritime Museum), Dr John Black, Dr Simon Diffey (statistician) and Dr Stephane Hoerle (French material scientist and specialist in rock art degradation). An eight-page critique of the report was provided to CSIRO and the WA Government. The initial report was removed from the website.

Issues identified with the report

False claims: Claims in the report that Gabbro rock surfaces would withstand a pH of 3.0 and granophyre rock surfaces a pH of 3.2 before dissolution of manganese and iron compounds were not supported by the results provided. For example, one third of the results for dissolution of manganese from granophyre rock, which showed significant concentrations in solution after 3-day of emersion, had pH values above 3.2. Similarly, for gabbro rocks, the results showed significant mineral dissolution at pH values above 3.0.

The main conclusions from the report were a misrepresentation of the statistically significant results presented.

Well accepted statistical methods for predicting 'threshold' or 'breakpoint' for mineral dissolution in relation to pH were not used. These statistical methods provide the likely threshold pH and the statistical confidence for the predicted number – showing the range in pH which could be the threshold value. These analyses were not included in the revised report despite CSIRO being given details of the statistical procedures.

There were several other major criticisms of the research.

Too much solution: The volume of liquids containing the test used to dissolve the rock surface layer was so large that a very high proportion of the minerals would need to be dissolved before they could be detected by the measuring instruments. The amount of mineral in solution would vary for the rock samples because of differences in thickness of the patina layer. For this reason, only a small number of solutions showed mineral dissolution. The reviewers suggested that change in pH over time for each sample would be another method for assessing dissolution because hydrogen ions are removed during mineral dissolution and pH would rise.

Ignore much data: Many other measurements were made including optical microscopy for measuring morphological and colour changes; spectrophotometry for measuring changes in colour space variables L*, a* and b*; and reflectance spectroscopy for measuring mineralogy and colour space variables. Results from these measurements were not analysed but could have been used to corroborate the mineral dissolution results. An extraordinary claim was made in the report "the KM spectrophotometer cannot provide discrimination between sample before and after exposure.", despite 70 of the 78 comparisons of L*, a* and b* showing significant ($P<0.05$) differences from before and after treatment.

Revised report

The revised August 2017 report does not address the major criticisms of the May 2017 report, but develops dubious reasons why those significant mineral dissolution results with pH above either 3.0 for gabbro or 3.2 for granophyre can be regarded as outliers. However, the revised report does cast great doubt on the value of the research for assessing the effects of pollutants on Murujuga rocks.

Reference

[1] Black JL, Box I, Diffey S (2017). Inadequacies of research used to monitor change to rock art and regulate industry on Murujuga ('Burrup Peninsula'), Australia. *Rock Art Research* **34**, 130-148.

Extreme weathering experiments on Burrup Peninsula / Murujuga weathered gabbros and granophyres

By Erick Ramanaidou, Gay Walton and Derek Winchester

Report EP172193   May 2017

Comments by John L Black, Ian D MacLeod and Simon Diffey

10 July 2017

General comments:

Understanding the impact of pollutants emitted by industry on the surface characteristics of Murujuga rocks and the implications for petroglyphs is extremely important for long-term preservation of the rock art. However, the conclusions from this report that dissolution of Murujuga rock surfaces commences at pH of 3.0 for gabbro and pH 3.2 for granophyre, with implications that rock art will not be damaged while rock surface pH is above these values, are not justified or supported by the data presented in the report.

We have serious concerns about the experiment design, methods, statistical analysis of the data and the selectivity of the measurements used to draw the report conclusions. A large amount of information was collected during the experiment including: ICP measured mineral dissolution; scanning electron microscopy for measuring changes in mineral composition; optical microscopy for measuring morphological and colour changes; spectrophotometry for measuring changes in colour space variables L*, a* and b*; and reflectance spectroscopy for measuring mineralogy and colour space variables. Values for each measurement type were obtained before and after immersion in the test liquids. However, only the ICP mineral dissolution measurements were analysed in relation to pH and used to draw conclusions. The collected data relating to colour and mineral changes has not been used to assess the impact of the pollutants or used to corroborate the results from the mineral dissolution measurements.

A great deal of the data collected has not been included in the report. A comprehensive statistical analysis of all data collected must be undertaken to determine the extent of change for each characteristic of the rocks measured and the pH at which these changes occur. Suitable statistical models have previously been agreed upon by Simon Diffey and David Alexander from CSIRO. Identifying pollutant concentrations when changes in colour occur is particularly important for preservation of the Murujuga rock art.

The veracity of the dissolution data presented is highly suspect. Iron dissolution was recorded for only 21, and manganese dissolution for only 29, of the 110 samples examined. These low recorded rates of dissolution for iron and manganese from desert rock surfaces with increasing acidity are at odds with previous published information. We suspect the discrepancy is due to the large amount of test solution used relative to the quantity of minerals within the leaching zone of the rock surface, associated with the high detection limits of the instrument used to make measurements. A calculation, using the information presented in the report, indicates that 76% of the manganese and 16% of the iron would need to be dissolved before detection. There is also sound evidence suggesting the use of silicon on the rock surface during sample preparation would reduce solubility of minerals.

Inadequacy of the method for measuring mineral dissolution is corroborated by the observed substantial increases in solution pH when the rocks were immersed over time in weak nitric and sulphuric acids. Dissolution of rock surface minerals results in the consumption of acid (H^+) and an increase in pH as was observed in samples immersed in weak acids where dissolution of the minerals was not detected.

The authors of the report used pH of solutions after 3 days immersion as the critical pH from which dissolution occurs without providing a justification. Mineral dissolution was claimed to commence at pH 3.0 for gabbro and 3.2 for granophyre rocks. This claim is not supported by the results presented using their criterion for commencement of dissolution. Six of the 18 results presented in Table 10 where dissolution of manganese was significant for granophyre rock had pH values after 3 days immersion above 3.2, with one as high as 7.1.

Statistical models exist for predicting the 'threshold' or 'break point' for when the relationship between two variables (for example manganese dissolution and pH) occurs and the accuracy of that prediction. We have used one such model for the 1 month manganese dissolution results presented in Table 10. Predicted manganese dissolution commenced at pH 3.91, with 95% confidence limits ranging from pH 2.34 to pH 5.45. However, because of the low number of samples showing manganese dissolution this predicted pH for manganese dissolution must be treated with extreme caution. Similar statistical analyses should be conducted for all the types of measurements made during the experiment, including those showing a significant increase in pH over time.

A great deal more work is required to present and analyse the results from this experiment before any reliable conclusion can be made about the impact of pollutants on the Murujuga rock surfaces. Until this work is conducted, it is unclear whether reliable information about the effects of industrial pollutants on Murujuga rock surfaces can be obtained. The design of the experiment, apparent lack of measurement replication and methods used may severely restrict the usefulness of the experiment.

We believe that the report currently on the DER website should be removed until this full analysis is completed because it is providing false conclusions not supported by the data presented in the report.

Purpose of experiment and experimental design

The purpose of the study was outlined in the 'Introduction' to the report as follows:
> "This study is aimed at simulating the effect of air pollution from the industrial complexes including iron ore port, liquefied natural gas, and salt production and, ammonia and ammonium nitrate plants. The study consists in (1) collecting samples of weathered gabbros and granophyres sampled at each of the original 7 sites in the Burrup Peninsula/Murujuga; (2) characterising the weathered surfaces of the samples; (3) immersing the samples in reaction vessels at 50°C with 5 solutions including distilled water (control), nitric acid, sulphuric acid, ammonium nitrate and ammonia at a range of concentrations and (4) characterising the weathered surfaces of the samples after exposure."

The statement indicates the importance of the experiment for: i) identifying the effects of the seven sites, with these covering the existing range of gabbro and granophyre rock types; ii) identifying the independent effects of each of five different solution types when provided at different concentrations.
If these were the objectives of the experiment, the following samples would be required:
- Several rock samples from each site (minimum of 3) to provide replication to identify site effects
- At least 13 cylinders from each rock collected because there are 13 treatments. However, some of these cylinders would need to be replicated up to 3 times to identify the variance in treatment for the same rock

A balanced design to meet the stated objectives would be 7 rocks x 3, 13 cylinders x 3 = 819 solutions. Some treatments could be partially replicated in a balanced method to reduce this total number if required.

The most important point is that statistical analyses to identify the independent and interacting effects of site, rock type and pollutant can be readily evaluated when a considered experimental design is applied.

If sites were considered not to be important, but rock type was, rocks could be collected from two sites only. Such a design would reduce the number of samples to 234 (2*3*13*3).

The actual allocation of rock samples to treatments is not described in the report and appears to have no design. An inspection of Table 9 shows there are only 10 samples from Site 7, which is not sufficient to cover all treatments. The samples from Site 7 are concentrated in the NH_3 test solutions. Similarly for Table 10, many of the sites are not represented across all treatments. Because of the lack of a consistent design, it is impossible to determine the impact of site (although site undoubtedly will influence the results). However, if the measurement accuracy is sufficient, the results could be analysed to identify the effects of rock type, pollutant and concentration using standard statistical models generally agreed by Simon Diffey and David Alexander from CSIRO. This was not done in the report, but would add greatly to the understanding if applied to all the different measurements made on the samples.

There are many unexplained aspects of the experiment that may have a major impact on the results.
- The largest number of samples (110) was exposed to solutions for only 3 days. Was there a reason to expect major changes in mineral dissolution in 3 days, rather than conducting a time series of immersions? Were any preliminary experiments conducted to identify the time course of dissolution of minerals?
- How were the samples and solutions treated prior to sampling for analysis and for pH measurement to ensure thorough mixing of the solutions?
- How many replicate samples were taken from each solution for measurement of mineral dissolution and pH at each sampling time? That is, what was the repeatability of single measurements to provide standard errors of the estimates?
- Twenty six samples from the 110 were used for more extensive measurements. Presumably these were selected to be one for each treatment for gabbro and granophyre. Why were only 26 selected and what was the basis for selecting these particular samples?
- Fifty eight of the 110 samples remained in solution for 1 month. Why 58, when only 26 were removed for more detailed analyses?

There was mention of identifying the effect of pH in the statement of objectives. There is a strong interaction between pH and EH as indicated in Pourbaix diagrams that effects solubilisation of compounds. Measurements of EH would have substantially increased understanding of the reactions that occur with each of the pollutants tested and be another assessment of mineral dissolution.

ICP-AES mineral dissolution

Conclusions about the effects of pollutant compounds on rock surfaces were based entirely on the mineral dissolution studies. Results presented in Tables 9 and 10 show dissolution of minerals on the rock surface occurred in only a very small number of the acid treatments. This result differs from published information on the effects of acid on rock patina (Bednarik 1979, 2002, 2009; Gordon and Dorn 2005; Black et al 2017b). A likely explanation for the inability to detect mineral dissolution is the experimental method combined with the detection limits of the ICP-AES instrument used. There are two concerns about the experimental method: i) the volume of liquid for immersion and ii) the use of silicon to protect the surface layer while the resin was applied (steps 4 & 5 of the method).

Volume of liquid: The volume of liquid in which the samples were immersed is not provided in the report. However, an estimate of approximately 200 ml can be made from Figure 6, which shows an immersed rock cylinder, using the 10 mm diameter of the cylinder as a scale. The weight of the rock surface leaching zone is given on page 20 as 1.123 g. The chemical composition of the rock surfaces is given in Table 3 as approximately 0.7% for MnO and 11% for Fe_2O_3. Thus, there is 7.86 mg MnO and 123 mg Fe_2O_3 in the leaching zone. If all the MnO and Fe_2O_3 were dissolved the concentrations in solution would be 0.0393 mg/l and 0.618 mg/l, respectively for MnO and Fe_2O_3. The detection limits of the instrument for MnO and Fe_2O_3 were 0.03 mg/l and 0.1 mg/l, respectively.

These calculations mean that 76% of the MnO in the leaching zone would need to be dissolved before it was detected and 16% of the iron. If the assumption that the amount of liquid added was 200 ml is correct, a substantial proportion of metals would need to be dissolved before detection, particularly for Mn. The calculation also assumes that the leaching zone for all rocks sampled was 5.5 mm. The leaching zone will not be the same for every sample and if less than 5.5 mm, 100% of dissolved metals may not be detected by the methods used.

Use of a silicon coating to protect the rock surface: Neutral silicon that was applied to rough rock surfaces would be slow drying and penetrate deep into holes and crevices on the rock surfaces making it extremely difficult to remove completely by peeling away (Stambolov 1971; Ward 1991). Silicon is hydrophobic and any silicon remaining would prevent the immersion liquids reaching the full rock surface. Residual silicon in combination with the high dilution factor could be an explanation for low detection of iron solubilisation and the low detection rate of all mineral dissolution.

Analysis of dissolution results

No statistical analyses of the results were undertaken, except for a t-tests for differences between before and after immersion. Detailed statistical analyses of the data could be undertaken to identify effects of fixed and random variables. This should be undertaken to determine whether there are significant effects of rock type, solution used, concentration of solution, time of immersion etc. However, the large number of zero results may influence the value of such an analysis.

No standard errors of measured values are presented. Does this mean that there were no replicate measurements of mineral dissolution or of pH at each sampling? If so, random errors due to variation in methods make it difficult to differentiate treatment effects from experimental error. Close examination of repeat pH measurements over time for individual samples, raises great concern about the accuracy of these measurements. For example, the pH values recorded for sample S1 224 were 4.9, 5.9, 7.4, 6.1, 7.0, respectively, for the initial solution and after 1, 2, 3 day and 1 month immersion in weak nitric acid. The authors must provide evidence of the repeatability of the dissolution and pH measurements undertaken.

If mineral dissolution is taking place, consumption of acid would be observable through a change in pH, particularly with weaker sulfuric and nitric acid solutions, which have little buffering capacity. Changes in pH of the ammonium nitrate solution would be less dramatic because of the NH_4^+/NH_3 buffer system of the weak acid and base system. Similarly, any acid mobilisation of aluminium will involve protonation of the aluminosilicates and again consume acid with a concomitant increase in the pH. Tables 9 and 10 of the report confirm that pH increased when samples were immersed in weak concentrations of nitric and sulphuric acids, even when no significant dissolution of minerals was detected. One example is sample S1 224, which was immersed in 0.00001 M HNO_3, showing a 100-fold increase in acidity with a change in pH from 4.9 for the initial solution to 7.0 after 1 month immersion. Similar examples are seen for samples immersed in weak sulphuric acid solutions.

Statistical procedures should be used to analyse the significance of the slopes in pH change over time of immersion as an alternate method for assessing whether dissolution of minerals had occurred.

The mobilisation of aluminium is to be expected in both acidic and alkaline solutions since the oxides and compounds of aluminium are amphoteric. The authors have failed to note that the mobilisation of aluminium from the rock crusts will fundamentally alter the pH balance and capacity of the mineral crust to have a buffer absorption capacity so that subsequent acid loadings will have a much more pronounced effect on the mobilisation of both iron and manganese.

Interpretation of results

The authors have used pH of the solution after 3 days as the critical pH from which dissolution occurs. A justification for selecting pH after 3 days immersion is needed in the report.

The authors state: "For the gabbros and after 3 days at 50°C, dissolution starts either at pH 3 or below for Al, Mn and Fe or more than pH 11 for Al. For the granophyres, dissolution starts at pH 3.2 and below for Al, Mn and Fe or more than pH 11 for Al."

This statement is not supported by the results presented in Tables 9 and 10 of the report. For example, for granophyre rock samples from the data provided in Table 10, there are significant differences from before and after treatment with 3 day pH as high as 7.1. Six of the 18 samples of granophyre that showed significant dissolution of Mn had 3 day pH values greater than 3.2. This is one third of the values (Table 1).

Table 1. Results from Table 10 of the Extreme Weathering report (Ramanaidou et al. 2017) showing 3 day immersion pH values for granophyre rock samples that had significant (P<0.05) Mn dissolution. pH values above 3.2 are highlighted.

Sample name	Immersion solution	pH after 3 days
S6 244	H_2O	6.9
S6 243	0.00001 M NHO_3	7.1
S8 260	0.0001 M NHO_3	4.6
S4 236	0.0001 M NHO_3	4.5
S4 237	0.001 M NHO_3	3.2
S8 259	0.001 M NHO_3	3.2
S4 80	0.001 M NHO_3	3.0
S4 62	0.087 M NHO_3	2.1
S8 251	0.087 M NHO_3	2.1
S4 231	0.087 M NHO_3	2.1
S5 111	0.087 M NHO_3	2.2
S4 235	0.001 M H_2SO_4	2.8
S8 256	0.001 M H_2SO_4	2.8
S8 253	0.0111 M H_2SO_4	1.9
S6 247	0.0111 M H_2SO_4	1.8
S4 73	0.0111 M H_2SO_4	1.9
S8 262	1 M NH_4NO_3	5.5
S6 241	0.001 NH_4NO_3	6.5

Why the authors of the report conclude that pH 3.2 is the value from which Mn dissolution occurs is totally mystifying. The conclusion is NOT supported by the data presented, which shows the 3 day pH at which significant Mn dissolution occurred was as high as 7.1. The graph in Figure 14 of the report shows clearly there was significant dissolution of Mn at pH values above 3.2. The figure is reproduced below.

After 3 days exposure for granophyres
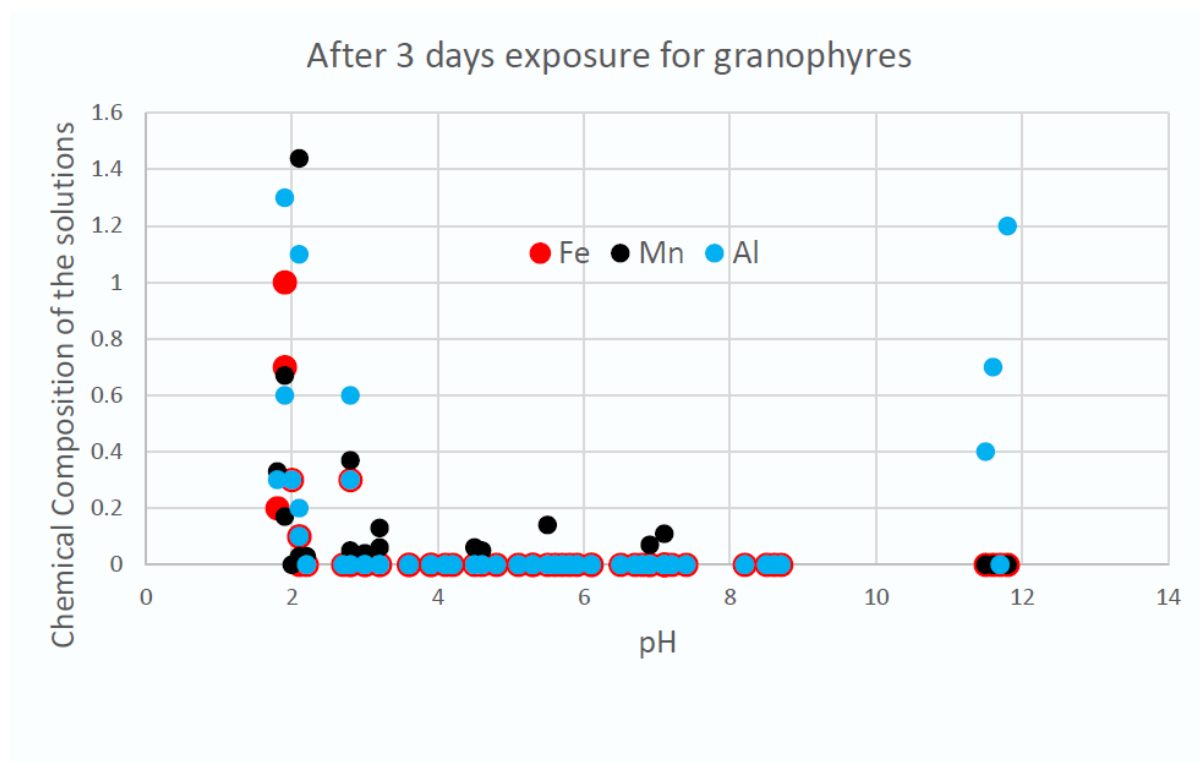
The results presented in Table 10 can be analysed using 'threshold' of 'break point' statistical models to estimate statistically the pH when Mn dissolution occurred and the standard error or confidence limits of the estimate.

We have used one statistical 'threshold' model (Muggeo 2003, 2008), to analyse the 1 month pH values given in Table 10 for manganese dissolution for granophyre rocks. The 'break point' analysis indicates that Mn dissolution commences at an estimated pH of 3.91, with confidence limits at 95% probability of pH 2.37 to pH 5.45. This result means that for the data obtained for dissolution of Mn, dissolution could start as high as pH 5.45 or as low as 2.37. However, because of the low number of samples showing dissolution due to 78% of the Mn needing to be dissolved before detection and apparent lack of replication of dissolution measurement, the values obtained should be treated with considerable caution if used as permitted rock surface acidity when petroglyphs will be eroded.

Summary of assessment of mineral dissolution results

- The conclusion stated in the report that dissolution of minerals commences at pH values of 3.0 or 3.2 is a misrepresentation of the results obtained. Since this is the primary conclusion of the report's Executive Summary, it potentially has extremely serious implications for preservation of petroglyphs on Murujuga.
- The experimental methods used appear to be incapable of detecting small amounts of mineral dissolution. A calculation from the data presented for one rock surface leaching zone suggests 78% of Mn would need to be dissolved before it could be detected. There would be variation in the depth of the leaching zone in the rock samples used, so it is possible that even 100% dissolution would not be detected for rocks with smaller leaching zones. This lack of ability to measure low level dissolution of minerals, casts great doubt on the value of the experiment for drawing valid conclusions about the effects of the pollutants tested on survival of the rock art.
- Corroboration of the mineral dissolution measurements can be made by fitting statistical models to determine whether the slope of pH change in solutions over time was significantly positive. This analysis would be particularly useful for the weak nitric and sulphuric acid solutions.

- Additional useful data indicating the extent of mineral dissolution would be obtained by EH voltage measurements if samples from the experiments at various times from immersion are available.
- The lack of correspondence between the stated project aims and the design of the experiment means the effects of site cannot be determined. Commonly accepted statistical analyses should be conducted to identify the relative effects of the pollutant examined and their concentrations.

Other measurements

Several measurements in addition to mineral dissolution were made on 26 samples (one for each treatment for gabbro and granophyre rocks). These measurements included scanning electron microscopy, optical microscopy, spectrophotometry and reflectance spectroscopy. Results from these measurements would provide additional evidence for the effects of the tested pollutants on Murujuga rock surfaces and the likely impact on petroglyphs. A comparison of mineralogy changes identified by scanning electron microscopy with the mineral dissolution results would provide some validation of the dissolution method for assessing the effects of pollutants on Murujuga rock surfaces. Presentation of data from the non-dissolution measurements in the report is selective and incomplete. In fact, the potential value of these measurements has been dismissed by comments such as "the KM spectrophotometer cannot provide discrimination between sample before and after exposure." This comment was made despite 70 of the 78 comparisons of L*, a* and b* showing significant (P<0.05) differences from before and after treatment.

We recommend:
- The individual results from all measurements made on the 26 samples are included in the report. This would include individual L*, a* and b* results determined by KM spectrophotometry, ASD and optical microscopy methods. Currently, actual values are not presented and only the P statistics for before and after immersion. Actual changes in these values are important for understanding the impacts on rock art, particularly to know whether they have become lighter. CIE ΔE colour changes following immersion should also be calculated for each method.
- These data from each measuring device are analysed for trends in relation to concentrations of pollutants and pH.
- A statistical comparison is made between mineralogy changes identified from the mineral dissolution, scanning electron microscopy and ASD wavelength analysis. Such an analysis will provide some confidence in the extent of mineral changes occurring.
- The optical microscopic photographs are analysed to determine the actual before and after L*, a* and b* values for individual pixels and average colours using the method: **Prediction of L*, a* and b* values from colour images** described by Black et al (2017a). The before and after colour changes (ΔE values) can then be calculated for every test solution. A comparison of L*, a* and b* from the three methods (KM, ASD, optical microscopy) should be made to provide confidence in the measurements being made.

Presentation of the report

Understanding the experimental design and methods used is difficult because these are described at different parts of the report. For example, the Executive Summary is the only place in the report identifying that twenty six samples were used for measurements other than dissolution measurements. The Methods section is extremely cryptic and does not provide details on how sampling was conducted or the number of replicate measurements made at each sampling.

Numerous results are dismissed as 'errors'. Such practice does not follow standard scientific procedures. Standard errors for each measurement must be presented and statistical tests undertaken to show whether the mineral dissolution values that are lower after one month immersion than after 3 day immersion are statistically different. A comment and evidence are needed on the accuracy of the

various measurements. Presumably, colour standards were used to verify the L*, a* and b* values obtained from the various procedures. The comparisons between the standards and machine measurements should be included in the report.

Many statements about results are not supported by the data provided. These are noted in the Report and should be rectified.

The discussion of results is rudimentary. Several results differ from what would be expected as judged from previous published literature. Very few of the relevant publications or an explanation about why the results from the current experiment differ from those publications are presented. The Discussion should be greatly expanded to show why the results obtained are likely to be correct and different from earlier published information.

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