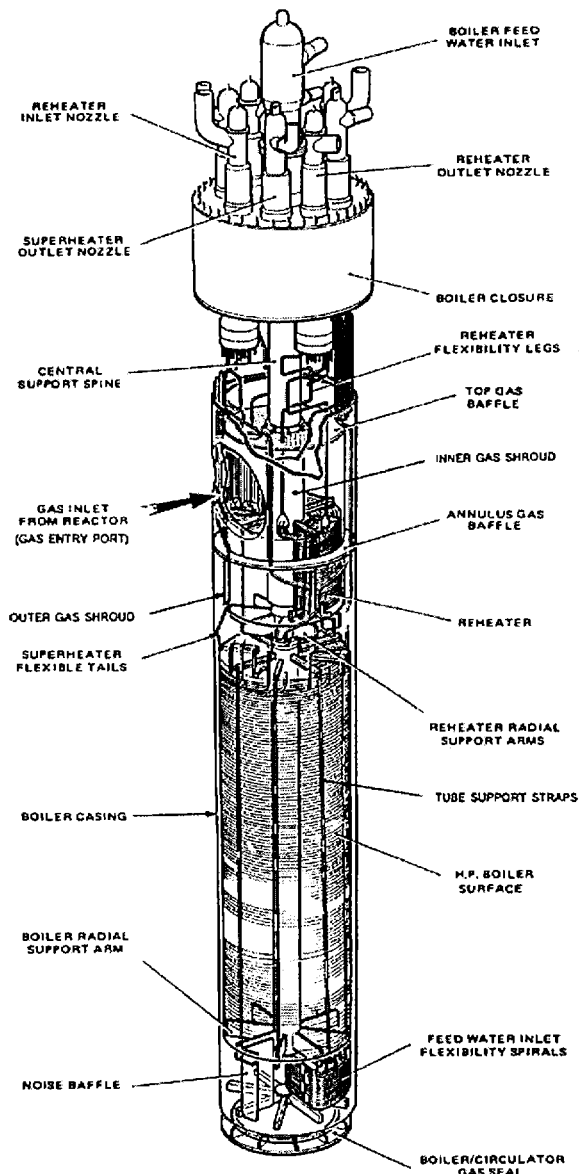


Chemical Cleaning of UK AGR Boilers

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Introduction

For a number of years, the waterside pressure drops across the advanced gas-cooled reactor (AGR) pod boilers have been increasing. The pressure drop increases have accelerated with time, which is the converse behaviour to that expected for rippled magnetite formation (rapid initial increase slowing down with time). Nonetheless, magnetite deposition remains the most likely cause for the increasing boiler resistances.



The AGR pod boilers have now operated for ~100,000 hours. They are of a once-through helical design, as shown in Figure 1. A reactor contains four boiler quadrants, each of two pods, and these are permanently encased within the reactor concrete pressure vessel. Each pod contains 285 individual boiler tubes, comprising carbon steel, 9Cr1Mo steel and type 316H stainless steel sections welded at lower and upper transition joints, respectively. The pods also house austenitic reheat tube banks, which are physically located above the boilers. Each pod boiler is designed to produce 543°C steam at 170 bar and 216 tonnes/hr. The feedwater chemistry is all-volatile with ~25 µg/kg oxygen dosed to the feedwater to inhibit flow-assisted corrosion in carbon steel sections. An overdose of hydrazine ensures that oxygen does not reach evaporator and superheater sections, thus avoiding the risk of oxygen induced localised corrosion. Feedwater quality is maintained to a very high standard, with chloride and sulphate levels <0.2 µg/kg. Finally, the feedwater iron levels are < 5 µg/kg.

A number of potential countermeasures have been considered in response to the boiler pressure drop increases. The use of alternative amines was investigated, with the aim of increasing the high temperature pH in the evaporator and thereby reducing evaporator deposition. However, this was discounted following the observation of excessive amine degradation in rig testwork. The implementation of a fully oxygenated chemistry to inhibit evaporator deposition was also investigated and this was discounted because of the risk to the evaporators and to austenitic superheaters that could be operating wet. A plant trial of elevated oxygen and reduced hydrazine has also been carried out to minimise internal boiler iron transport, particularly from upper carbon steel sections. However, there was no detectable reduction in the rate of pressure drop increase. Chemical cleaning was therefore considered and a project to substantiate and then implement chemical cleaning was initiated.

Figure 1 - Schematic of the AGR Pod Boiler

Whilst chemical cleaning is carried out routinely on conventional plant boilers, the AGR pod boilers pose a number of unique challenges for chemical cleaning, and these explain the cautious approach that has been adopted. Among the key issues are:

- The boiler tube walls are only ~2 mm, yet they need to withstand an operational differential pressure of 130-170 bar and need to last the lifetime of the station because the boilers cannot be replaced.
- Due to the once-through design, all boiler sections need to be chemically cleaned. Unlike conventional once-through boilers, there is no opportunity to clean only selected boiler sections. It is very rare to carry out operational cleans of austenitic superheaters, and sections of those in the AGR pod boilers are in a highly sensitised condition.
- The pod boilers are the sole source of decay heat removal. Therefore, a very low risk of boiler tube failure has to be demonstrated in order to ensure that a means always exists to remove decay heat. Hence, the impact of chemical cleaning on boiler tube integrity needs to be very low and clearly substantiated.
- It is conventional in chemical cleaning to establish first the nature of the deposition by taking a tube specimen. This cannot be done with the AGR pod boilers, and nor was it practicable to visually inspect the relevant waterside surfaces.
- The 7 AGR power stations had operated ~200 reactor years without boiler chemical cleaning. A pod boiler clean would therefore be a 'first of a kind'.

This paper describes the project that has now resulted in the first full-scale AGR boiler clean. Because of the issues identified above, chemical cleaning was implemented in a staged manner, ensuring that lessons learned were incorporated into following stages of the project. The principal stages of the project are as follows:

- i) Preliminary review of existing substantiation data, chemical cleaning experience and practices, identifying candidate reagents for further investigation
- ii) Materials testwork to select a chemical cleaning reagent/procedure
- iii) Further materials testwork to substantiate the selected reagent/procedure for plant cleaning
- iv) A trial clean of a small number of boiler tubes on the plant
- v) A clean of the Wythenshawe Boiler Rig, an experimental test loop that includes a single full-scale full length boiler tube, which can be exposed to plant representative thermohydraulic conditions
- vi) The full-scale clean of a single reactor quadrant followed by a period of operation
- vii) The full-scale clean of the remaining three reactor quadrants
- viii) The full-scale cleaning of other affected reactors

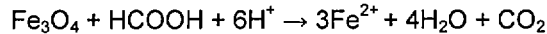
A range of oxide layers are expected to be present on the boiler tubes. Oxides formed by oxidation of the substrate are expected to be duplex in nature, with deposited oxides forming an additional outer layer predominantly on the evaporator surfaces. Oxides on carbon steel sections will be predominantly haematite, whilst the ferritic or austenitic sections will comprise an inner layer of chromium or chromium/nickel rich spinel oxide, respectively, plus an outer layer of magnetite. Prior to any chemical cleaning, it was estimated that the oxide thickness in the evaporator regions was of the order of 100-200 μm , predominantly deposited magnetite.

Reagent Qualification

Although no AGR chemical cleans had taken place prior to those described in this report, the AGR boilers were designed to be chemically cleaned. At the time of reactor commissioning, it was the policy in the UK to chemically clean power plant boilers every four years, rather than the more common approach since the 1990s of only chemically cleaning when there is an operational requirement to do so. As a result, Moore and co-workers developed a chemical cleaning procedure for the AGR boilers based on ammoniated citric acid plus formic acid, which is commonly referred to as GOM 106. Much work was done to substantiate this procedure for AGR materials in the 1980s, and experience also built up in its use in conventional plants in the UK. A similar reagent is also used in conventional plants in the US, based on ammoniated hydroxy-acetic acid plus formic acid (ref. 1). However, some concerns were raised about the ability of GOM 106 to remove relatively thick evaporator oxide deposits, as might be expected in an AGR boiler after 100,000 hours of operations.

Since the early work on GOM 106, a number of important developments have taken place in the field of power plant chemical cleaning, particularly the use of hydrofluoric acid (ref. 2) and EDTA (ref. 1) as cleaning agents. Hydrofluoric acid was a particularly promising candidate for AGR cleaning due to its rapid and efficient removal of oxides, the low volume and easy treatment of waste, and the extensive experience of use in conventional once-through boilers. GOM 106 and hydrofluoric acid were therefore selected for further investigation, whilst EDTA was also retained as a fall-back option in the event that neither of the primary candidates proved suitable.

The GOM 106 reagent cleans in part by dissolving iron oxides via complexation with citrate, and in part by undercutting. As a result, a significant amount of particulate is generated and the procedure includes intermittent surge periods at high flow velocity to flush the particulate from the boiler. The addition of formic acid improves cleaning efficiency via reductive dissolution, whereby ferric iron within the oxide is reduced to the more soluble ferrous form:



A further benefit of this process is that the ferric iron concentration is reduced, and so the risk of substrate attack is also reduced. The GOM 106 reagent is 3% citric acid, 0.5% formic acid, ammoniated to pH 3.5 and 0.05% of the film-forming corrosion inhibitor Stannine LTP. Tests were carried out at $90 \pm 5^\circ\text{C}$.

Hydrofluoric acid is much more efficient at dissolving iron by complexation than citric acid, and even in a once-through boiler clean will saturate with iron and produce no particulate. Therefore, only the required amount of hydrofluoric acid for iron removal is used. Furthermore, the process waste is treated with lime to precipitate all fluoride and metals, and so the final disposal of the waste is made much more practicable. The reagents investigated in this work were 1% hydrofluoric acid with either 0.1% Dodigen 95 or 0.2% Lithsolvent CL4 corrosion inhibitor. Tests were carried out at $60 \pm 5^\circ\text{C}$.

The candidate reagents were tested for oxide removal efficiency and corrosivity in a recirculatory test loop that incorporated plant representative tube specimens, Figure 2. The test loop was designed to replicate the flow velocities, temperatures and reagent chemistries in a plant clean, although it should be noted that a plant clean would be once-through rather than recirculatory to protect carbon steel sections from attack by ferric iron. Oxygen levels were controlled to $<30 \mu\text{g}/\text{kg}$ and the influence of dissolved iron, in particular ferric iron, was simulated by the addition of magnetite powder to the reagent prior to testing. Tube specimens were pre-oxidised, to produce layers as representative of plant water-grown and steam-grown oxides as practicable, and could be stressed using an external jig as shown in Figure 2. Heat treatments applied to austenitic specimens were also designed to produce sensitisation. Finally, sections of tube from a boiler test rig that had operated under full-power conditions for 10,000 hours were also included in the test matrix. In order to assess the cleaning performance, post-test metallographic examination was carried out, supported by measurements during cleaning for dissolved metals, ferric iron and redox potential (platinum electrode). Metallographic examination focussed on localised metal losses, for example due to flow accelerated corrosion, pitting, galvanic attack or IGA/SCC, because ultimately boiler tube failures are likely to occur at sites of deeper localised attack rather than shallower general metal loss.

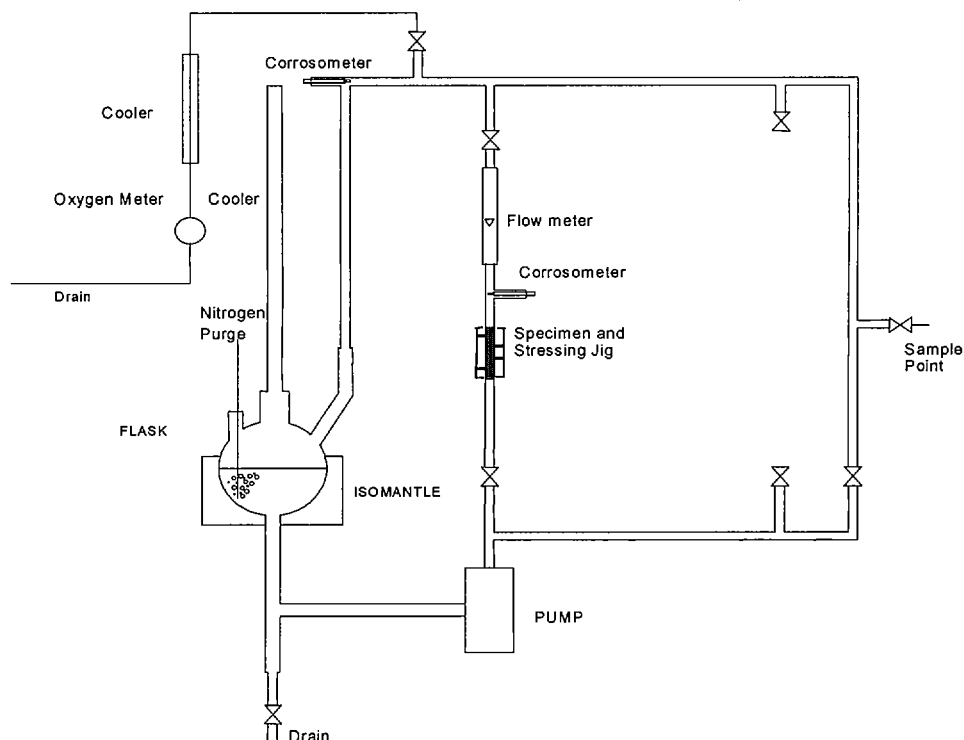


Figure 2 - Schematic of the Reagent Testing Loop

The tests considered most critical were those carried out on thick oxides on 9Cr1Mo tube and on the upper transition joint between the 9Cr1Mo and 316H sections. Both reagents performed well at removing oxides in the testing, although GOM 106 did take a relatively long time to clean the inner spinel oxide from steam oxidised 9Cr1Mo specimen. Neither reagent removed the high temperature formed inner spinel oxide from the Type 316H material. As expected, hydrofluoric acid cleaned much quicker than GOM 106. Corrosion damages were relatively low, and mostly dependent on the time spent cleaning after oxides had been removed. However, the 9Cr1Mo side of the upper transition joint appeared to be significantly more susceptible to galvanic attack in the hydrofluoric acid reagent than in GOM 106. Figure 3 shows micrographs from UTJ specimens tested in hydrofluoric acid and GOM for 3 and 14 hours, respectively. The metal loss in the hydrofluoric acid test was much more significant than any other seen in the testwork, and so GOM 106 was selected as the reagent for boiler cleaning.

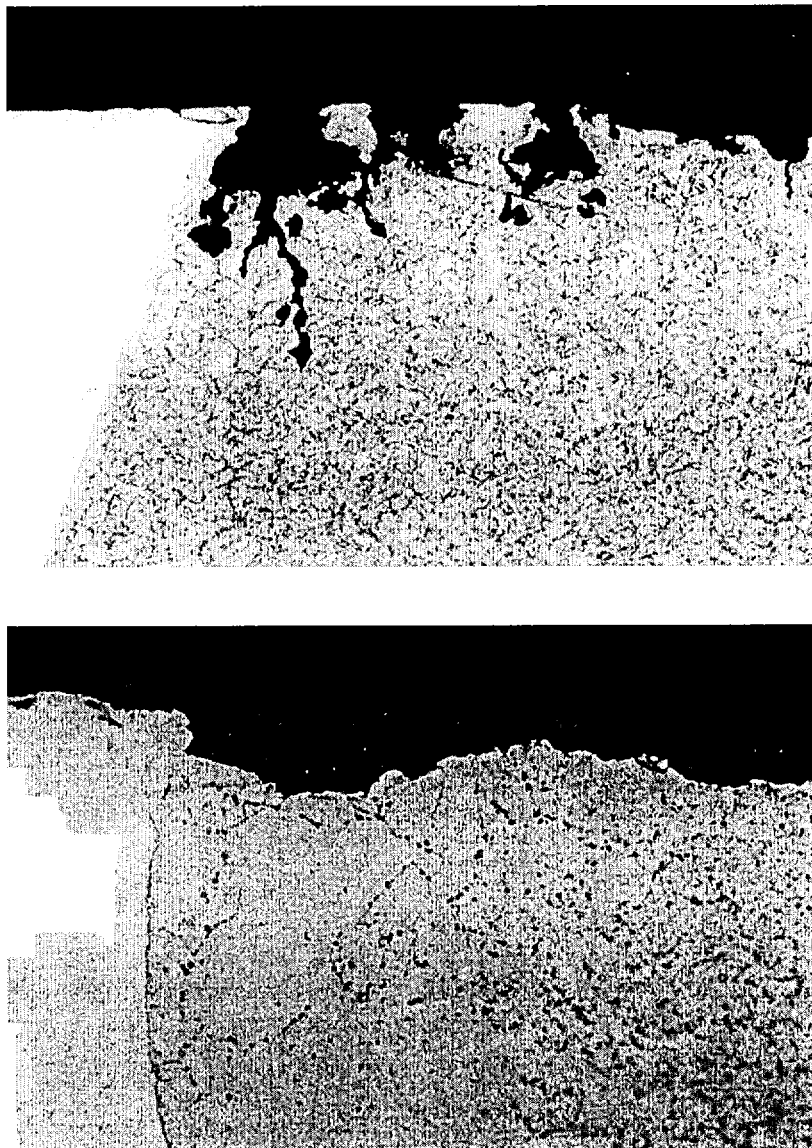


Figure 3 - Comparison of Upper Transition Joints after Cleaning with Hydrofluoric Acid for 3 hours (top - magnification 200x) and GOM 106 for 14 hours (bottom - magnification 500x).

Further materials tests were carried out to fully substantiate the GOM 106 reagent for plant cleaning. A difficult area to replicate was the degree of sensitisation of the type 316 austenitic stainless steel. It is known that short-term high temperature heat treatments can not achieve the same degree of sensitisation as longer term lower temperature treatments, i.e. those experienced in the plant. Therefore, tests were carried out on coupons of steam exposed material taken from superheater headers on the plant, to ensure that sensitisation levels representative of the plant were tested. The test programme also addressed other stages of the cleaning procedure, for example the interim passivation, and the consequences of parameters being out of specification. More detail on the chemical clean materials testwork programme can be found elsewhere (refs. 3,4).

The testwork enabled a fully substantiated cleaning procedure to be produced, which could then be applied in plant cleans. The procedure comprised six principal stages, as follows:

- Stage 1: Pre-clean flush with boiler feedwater, to remove any debris or particulate in the boiler prior to cleaning.
- Stage 2: Acid clean with 3% citric acid, 0.5% formic acid, 0.05% Stannine LTP inhibitor, ammoniated to pH 3.5. Surge flow periods take place for a couple of minutes every half-hour, in order to flush particulate from the boiler. Target temperature 90°C.
- Stage 3: Acid rinse with 0.2% citric acid ammoniated to pH 3.5, to remove inhibitor and residual iron salts from the boiler.
- Stage 4: High velocity flush with boiler feedwater, to remove partially detached oxide scales from the boiler.
- Stage 5: Interim passivation with 1% citric acid, 0.5% sodium bromate, ammoniated to pH 10.0. Target temperature 55°C. This reagent was used for full-scale cleaning with slightly different versions used in the demonstration cleans. This reagent is equally suited for copper removal, and so this was also used to demonstrate that negligible copper was present in the boiler deposits (normally determined from plant specimens).
- Stage 6: Final rinse with ammoniated water, to displace the passivation solution and to leave the boiler in an adequate storage solution.

To support this procedure, a number of key monitoring parameters were specified to ensure that a safe and effective clean would be carried out, including alarm and limit values. These included principal process parameters, such as flow, temperature and pH, plus other supporting parameters, such as oxygen, chloride and carbon steel corrosion rate (by LPR). In order to minimise the risk of corrosion, the acid clean reagent was required to be deaerated (target 30 µg/kg oxygen) and chloride levels of <2 mg/kg were also specified. The LPR corrosion rate meter was demonstrated to give a very clear response to loss of inhibitor.

Demonstration Cleans

Two demonstration cleans were carried out prior to full-scale cleaning. The first was the trial clean of six boiler tubes on the plant, which represented one of the most significant milestones in the project. It was the first occasion that plant tubes had been cleaned in any AGR, and was the first opportunity to get plant data on the nature of the boiler deposits. It would also confirm the pressure drop benefit from chemical cleaning, and indicate the time required to clean. The second demonstration clean was of the Wythenshawe Boiler Rig, a full scale single boiler tube with about 25,000 hours of operation under plant thermohydraulic conditions. The Boiler Rig permitted a clean that closely simulated a plant clean, but, unlike a plant clean, allowed sections to be removed for inspection. Therefore, the Boiler Rig clean provided final confirmation of the corrosion losses during a genuine once-through clean.

The plant trial clean took place in May 2000, taking advantage of the planned removal of feed and superheater headers during an outage. The three tube pairs were selected to represent inner, outer and middle rows of the pod boiler, noting that tube pairs had to be cleaned because tubes are bifurcated at the end of the superheater sections. The tube pairs were also selected to optimise the amount of post-clean data that could be obtained, as some tubes have instrumented inlet orifices to measure tube flow and some have outlet thermocouples to measure tube pair outlet temperature.

In the plant trial clean, heated deaerated demineralised water was supplied to a nitrogen sparged chemical cleaning mixing tank, into which concentrated chemicals were pumped in the appropriate proportions. The resulting reagent was then pumped through the tubes selected for cleaning. The majority of boiler outlet liquid was directed to a waste tank, although during the passivation stage, the solution was returned to the mixing tank to minimise chemicals usage. Tube inlet and tube pair outlet sample lines were connected to allow various continuous and grab sample analyses. The reactor gas-side was set-up at the process temperature in order to maintain the required temperature throughout the tubes. A gas-side overpressure was also maintained during cleaning, to prevent ingress of chemicals into the reactor in the event of a leak, and this principle has been adopted in all plant cleaning.

The results of the trial clean were very promising. The acid clean stage was allowed to proceed for about 13 hours, although the iron measurements, Figure 4, suggested that cleaning was complete much earlier (after about 8 hours). During conventional plant cleaning, the appearance of a plateau indicates that all relevant oxides have been removed, with the residual iron due to corrosion of the substrate metal. However, in this case, the plateau indicated the complete removal of haematite and magnetite deposits, with the residual iron due at least in part to the slow removal of the inner spinel oxide on the 9Cr surfaces. This observation was confirmed

by the chromium measurements, Figure 4, and by the continued removal of chromium rich particulate during surge flows in the plateau region. Nonetheless, it is still concluded that the optimum time to end cleaning is once the iron plateau has established. This is an important and somewhat unexpected conclusion, in light of earlier concerns about the ability of GOM 106 to clean in a reasonable timescale. Nickel measurements, Figure 4, showed that nickel was removed from the outer evaporator deposits but that very little was removed otherwise, confirming negligible attack of the inner oxide on the austenitic surfaces.

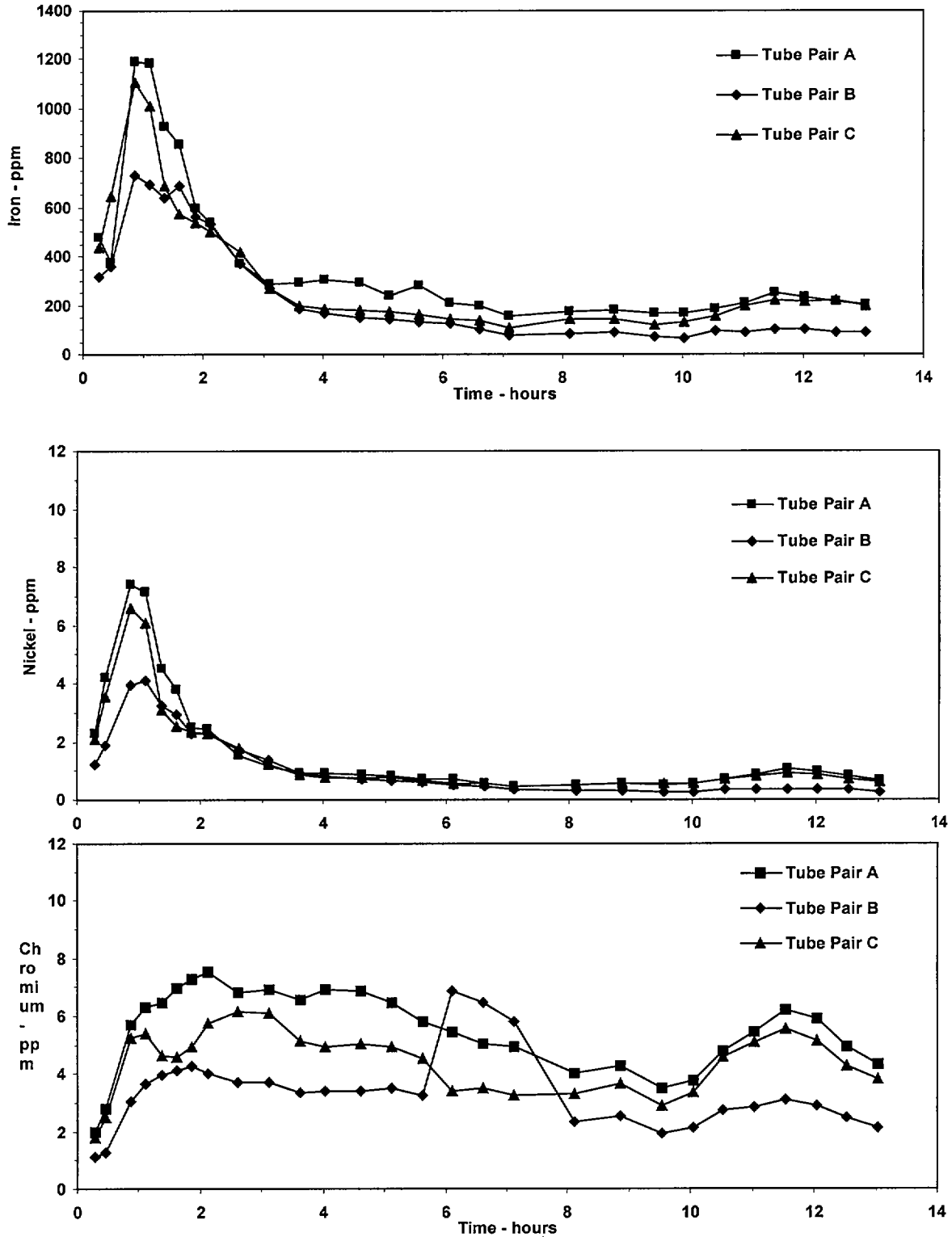


Figure 4 - Dissolved Iron, Chromium and Nickel Concentrations during the Plant Trial Clean (by ICP-OES).

It was estimated that about 4 kg of particulate and soluble iron was removed from the six tubes. The surge flow periods were very effective at removing iron from the boiler, and it is estimated that particulate iron accounted for about 45% of the total iron burden. The surge flow period samples were heavily discoloured with particulate, and it was possible to differentiate visually between material removed from upper boiler sections (magnetite) and from lower boiler sections (haematite). The total amount of iron removed suggested that the evaporator deposits were of the order of 100 μm . There are clear differences in the metal concentrations between the three tube pairs in Figure 4. These are believed to be due to flow differences between the tube pairs, indicating that the tubes clean at a similar rate even if the flow resistances are different.

A significant single phase pressure drop recovery of about 30% was observed between the pre-clean flush and the post clean flush. The resulting on-load improvements were observed as flow increases in the cleaned tubes rather than pressure drop recoveries, because of the small number involved. Nonetheless, these flow increases indicated a similar level of improvement when the reactor returned to power, thus confirming that a significant operational benefit would be realised by chemical cleaning.

The clean of the Wythenshawe Boiler Rig followed soon after the plant trial clean, with the acid clean stage taking about 6 hours. Short sections of boiler tube were removed from the Rig both before and after cleaning to establish the pre- and post-clean tube condition. Metallographic examination revealed that the majority of oxide had been removed effectively and only minor corrosion had taken place. Negligible single phase pressure drop recovery was obtained, although rippled magnetite was present in the evaporator section. More interestingly, a clear two-phase pressure drop recovery was observed in the lower superheater section. This is believed to be the result of magnetite deposition in the superheater during earlier low temperature Rig tests, where the lower superheater was operated as part of the evaporator. This enhanced effect of roughness in steam, due to the much higher fluid flow velocities, may be a contributing factor to the pressure drop increases observed on the plant. More information on the clean of the Wythenshawe Boiler Rig can be found in ref. 3.

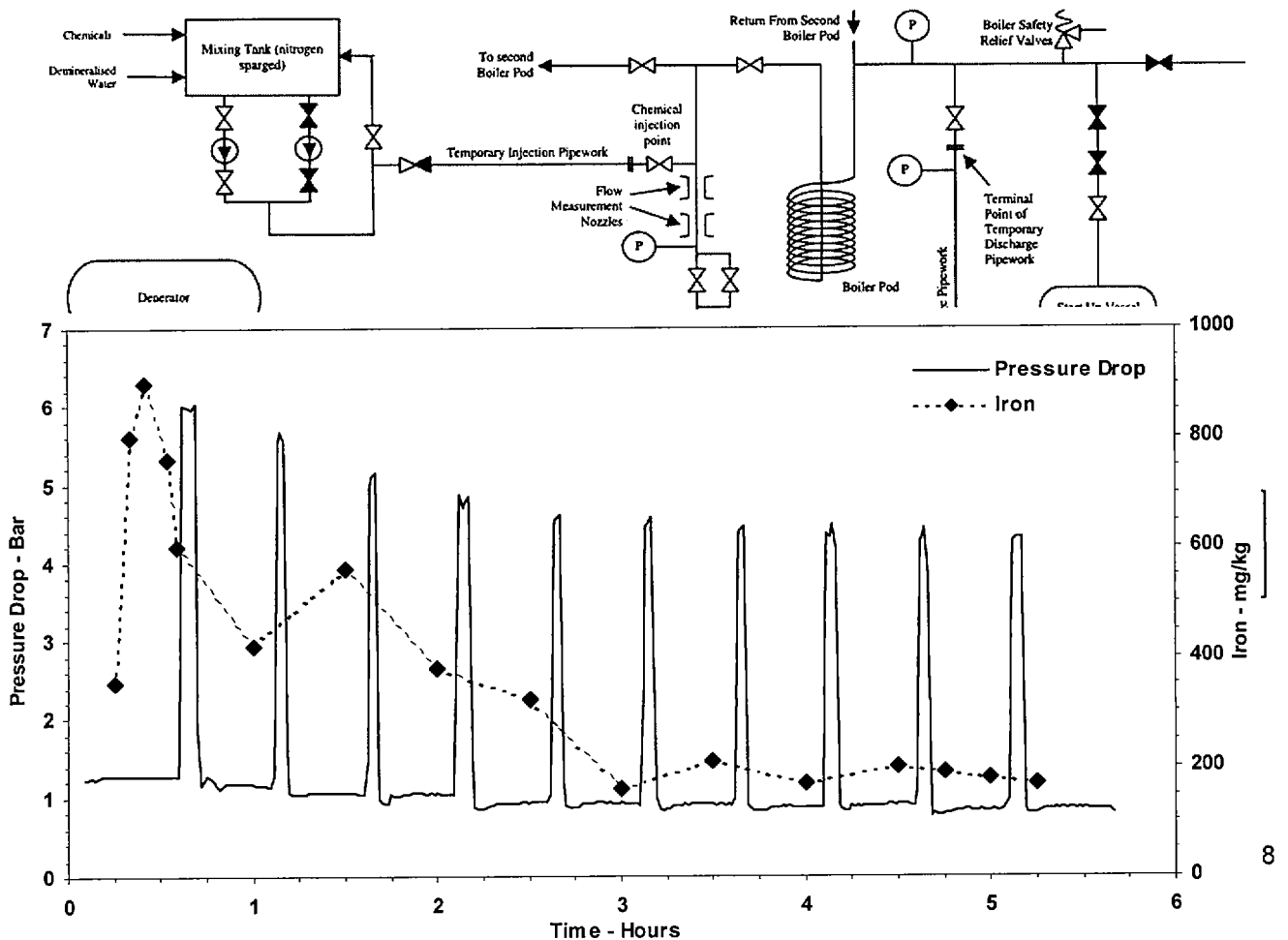
Full-Scale Cleaning

In line with the staged approach described earlier, the first reactor full-scale clean took place in two stages, with the first quadrant cleaned in July 2001 and the remaining three quadrants in December 2001. A schematic of the system design for full-scale cleaning is shown in Figure 5. The principle is similar to the trial clean except that a concentrated solution is prepared in the chemical mixing tank, which is injected into the boiler feed pipework where it is diluted to the correct strength with boiler feedwater. All temporary pipework and the inlet injection points were fabricated specifically for the chemical clean project. An existing steam pipework connection was made use of for connecting the outlet temporary pipework, which directed the cleaning waste to a large waste storage tank. The concentrated chemicals are not heated, other than by the heat of dissolution, and so the required reagent temperature was achieved by pre-heating the diluting water using the station start-up feedheater. It was not practical for either the start-up feedheater or the chemical injection skid to respond directly to each short surge flow period, and so there were temperature and reagent concentration transients during and immediately after each surge period. However, this did not appear to significantly impact the cleaning effectiveness. In order to meet oxygen level targets, additional deoxygenation of plant make-up water was carried out by a mobile trailer. The acid clean reagent oxygen levels were typically 10-30 $\mu\text{g}/\text{kg}$. Routine control of feedwater impurities plus care in the standard of bulk chemicals also ensured that chloride levels were <2 mg/kg in the cleaning reagent.

The full-scale cleans were implemented successfully, and the general trends were as for the earlier plant trial clean. The overall temperatures were slightly higher than during the plant trial clean, and this appeared to improve the cleaning efficiency, with the acid clean stages all taking about 6 hours including a period after the iron plateau to confirm that the end point had been reached. It is estimated that about 400 kg of iron was removed from each boiler quadrant, in good agreement with the value derived from the plant trial clean. Valuable additional information was also obtained by monitoring the boiler pressure drops during cleaning. Figure 6 shows the typical behaviour and comparison with the iron trends. The sharp pressure drop peaks indicate the surge flows, and the reduction in surge flow pressure drop with time is a direct measure of the benefit of cleaning. These data support the conclusion that the end point for the clean is reached once the iron plateau has established.

One of the significant issues for full-scale cleaning using the GOM 106 reagent is the volume of waste created. Cleaning the full reactor generated about 5,000 m^3 of liquid waste, typically pH ~4 and about 1.5% in citric acid. The waste is disposed of over several months via slow discharge into the station cooling water (sea-water), in accordance with an agreed discharge consent.

Additional monitoring and control was carried out during the post clean storage and then return-to-power of the cleaned boilers. Adequately protecting the cleaned surfaces from enhanced corrosion before operational oxide layers have been allowed to form is an important feature of any chemical clean. Here, a high ammonia regime (~5 mg/kg) was adopted during storage, seeking to keep oxygen levels as low as practicable. A flow was maintained wherever possible to avoid stagnant conditions, and boiler outlet monitoring confirmed that iron was not being released from the freshly cleaned carbon steel surfaces. Once the boilers started warming during reactor start-up, oxygen dosing was commenced, whilst maintaining the required overdose of hydrazine, to ensure that a protective haematite layer formed on the carbon steel surfaces. The ammonia concentration was allowed to reduce back to normal operating levels as the feed flow increased. Some residual cleaning reagent was retained in the remaining oxides on the boiler surfaces, shown by the appearance of a broad boiler outlet after-cation conductivity transient as boiler temperatures increased.



Following the single quadrant clean, feed pipework valving arrangements were modified on the cleaned boiler to simulate the resistance which had been removed by cleaning. This was done as a conservative measure to maintain a symmetric pattern of heat exchange whilst there were both cleaned and uncleaned boilers in service. In this way, no operational benefit was taken from the first quadrant clean. Conversely, the full benefit of cleaning was realised following the subsequent three quadrant clean and boiler feed pressures reduced to levels commensurate with early station life. The improvements, shown in terms of normalised pressure drop in Figure 7, represent a significant operational benefit for the station.

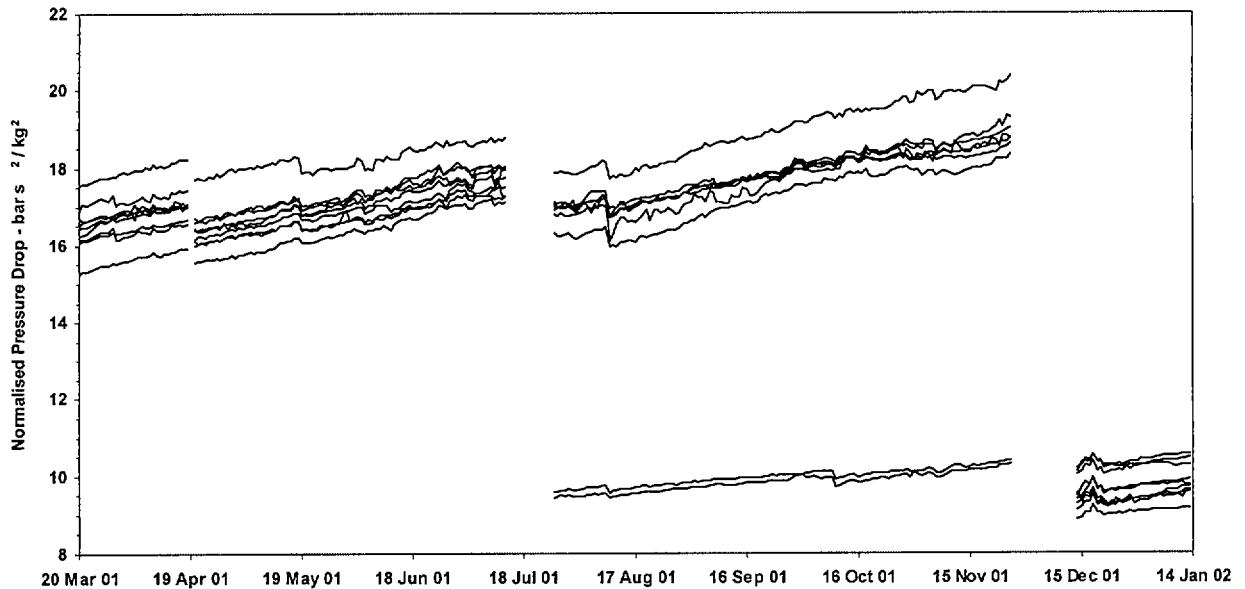


Figure 7 - The Effect of Full Scale Cleaning on Operational Boiler Pod Pressure Drops

Discussion

The adoption of a cautious and staged approach to the implementation of chemical cleaning has been important in this project, because of the significant potential risk posed by the lack of access to the AGR boilers and their sensitivity to corrosion losses. It has taken the project about 3 years from inception to the first completed reactor clean, and this does not include the significant work carried out in the 1980's by the C.E.G.B. on the development of the GOM 106 process.

The full-scale cleans required the coordinated activities of personnel at the cleaning skid, at the station central control room, at a remote monitoring station and at other locations around the station. In order to demonstrate that this process could be carried out correctly, a series of detailed procedures were formulated, all of which were called upon by an overall quality plan and managed by a site team leader for the project. This approach proved a very effective means of ensuring that a relatively complex and potentially hazardous process was implemented safely and efficiently.

Whilst the chemical cleaning process employed here is not novel, the sensitivities of the pod boilers required significant additional investigations that would not normally be considered for conventional plant cleans. The detailed materials testwork highlighted the importance of understanding and then assessing the specific plant condition at all locations that will be exposed to the cleaning reagent. An AGR is one of the few cases where an operational clean of superheater boiler sections has to be carried out, and the materials testwork confirmed that these were the most sensitive to corrosion, particularly as they are exposed to the highest ferric iron levels. In conventional cleaning, corrosion is assessed retrospectively as general metal losses, typically using corrosion coupon weight loss measurements. However, the greater risk to boiler tube integrity is often more likely to come from localised corrosion, for example IGA/SCC, galvanic notching and pitting attack.

Further valuable information has been obtained in this project from the extensive monitoring and analysis of plant cleans, again much of which would not normally be considered for conventional plant cleans. Monitoring of boiler pressure drops allowed the benefit of cleaning to be followed in real-time, improving the confidence in the end point determination. ICP-OES and ion chromatography were valuable tools for post clean analysis of

solutions. A good example of the information obtainable by ICP-OES is shown in Figure 4. However, a wide range of other elements can also be determined accurately by this method, and these provided valuable information on the nature of the boiler deposits. This technique also provided an accurate means of measuring the inhibitor concentration, which had proved very difficult by other methods. This worked by detecting the sulphur in the inhibitor active component. Ion chromatography was found to be the only successful method for determining the chloride concentration in the acid cleaning reagent, and could measure the much higher levels of the reagent components at the same time. Figure 8 shows an example anion chromatogram from an acid cleaning solution, taken at the boiler outlet during a plant clean. Finally, the in-situ LPR corrosion rate measurements provided a valuable in-situ means of demonstrating that the inhibitor was present and inhibiting effectively.

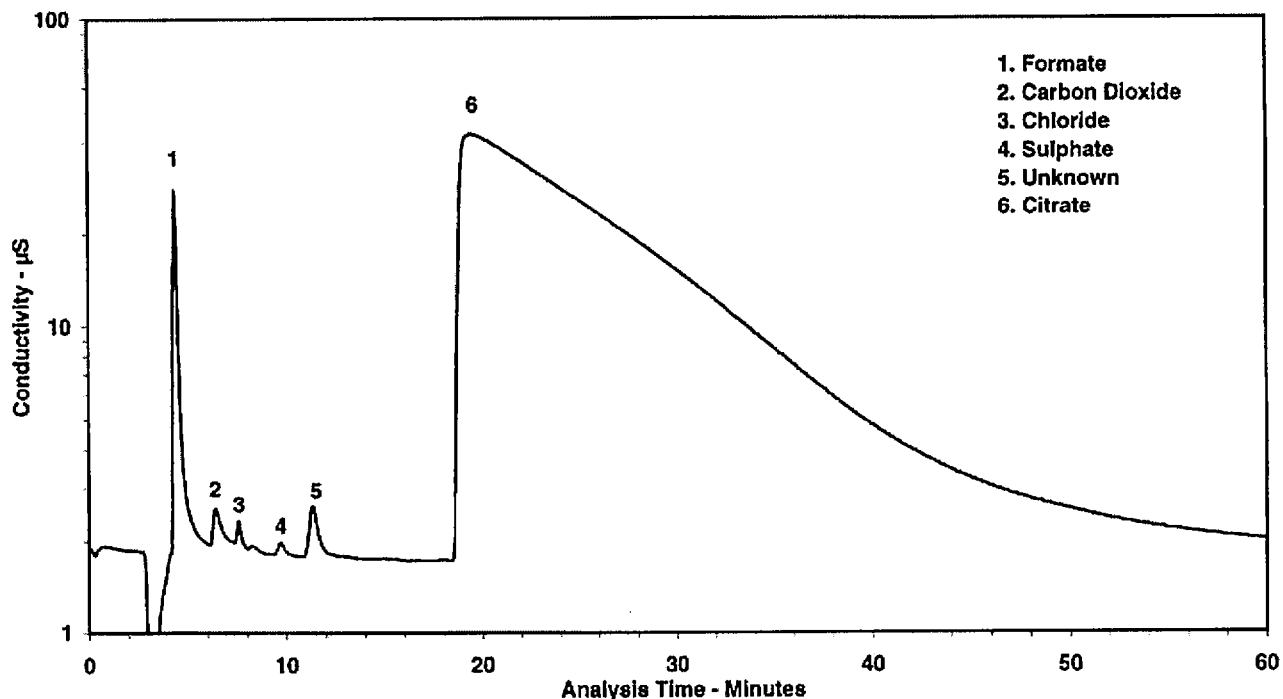


Figure 8 - Example Anion Chromatogram of a GOM 106 Solution

An important benefit of the staged approach has been to allow lessons learned to be fed into subsequent cleaning activities. Equally, as confidence in the process grows, the need for some of the more conservative controls can be reviewed. Among the issues under consideration for future cleans are whether the interim passivation is required, and whether there are any further steps to reduce the volume of waste generated. As for the chemically cleaned reactor, the future development of the boiler pressure drops will be closely monitored to establish the likelihood of further chemical cleaning during the remaining operating life.

Acknowledgements

A large number of people from several organisations have participated in this chemical cleaning project. Key to this success has been the adoption of a single team approach, and all involved are commended for their efforts. The Chemical Processes Technical Group of the UK nuclear industry's Industrial Management Committee are also thanked for supporting the presentation of this paper.

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