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EXAMPLES OF USE AND INTERPRETATION OF FIELD DATA USING A PORTABLE HYDROGEN PERMEATION MONITOR

Andrea M. Etheridge
Shell Global Solutions (UK)
Cheshire Innovation Park
P.O. Box 1
Chester CH1 3SH
United Kingdom

Emma B. McDonald
Shell Global Solutions International B.V.
Badhuisweg 3
1031 CM Amsterdam
The Netherlands

Duane Go Serate
Philipinas Shell Petroleum Corporation
Tabangao Refinery
Batangas City
Philippines

Frank Dean
Ion Science Ltd
The Way
Fowlmere
Cambridge
SG8 7UJ
United Kingdom

ABSTRACT

Three refinery locations were identified for the evaluation of a hydrogen flux measurement tool. At the first, spot flux measurements were carried out at identical sites in a pair of very similar crude distillation units. Certain locations were proximal to an online wall thickness monitor. Although hydrogen activities derived from the measured flux were very low, there was correspondence in flux activities obtained from identical measurement sites. The wall thickness measurements and proximal flux measurements were co-incidentally very low.

At the second location, higher hydrogen activities were identified at a number of locations subject naphthenic acid / sulfidic corrosion. There was very significant co-trending of activities between sites at each location, but activities correlated more closely with 'Potential H2S' obtained from crude oil data than from the TAN of crude fractions tested, perhaps because the latter was moderately low. At a third location, flux monitoring of the bakeouts of three vessels yielded flux data corresponding closely with expected hydrogen content in the vessels due to prior service.

INTRODUCTION

Recently, a handheld hydrogen flux monitoring tool[1-8] was made commercially available(1). This tool measures hydrogen flux by sweeping hydrogen from the steel surface in a sample gas stream of ambient air, and measuring the increase in hydrogen concentration in this sample. The resulting hydrogen concentration is displayed in units of nl/cm^2/s.

The instrument provides measurement of hydrogen flux from steel, over a wide range of steel temperature (to 500 ^{o}C) and curvatures (>5 cm diameter). Flux is detected over 5 orders of magnitude, assisted by the ability to vary the area of hydrogen collection from a target steel surface. Measurements take 60 seconds and frequently no surface preparation is required. The instrument is intrinsically safe and it engages no consumables. The principle of measurement is discussed in more detail elsewhere[2]. The probes used are designed for either low or high temperature use. The LT (low temperature) probe is of 6 in diameter, offering high sensitivity, and usually used at temperatures of below 130 deg C. The HT (high temperature) probe is 2.25 in diameter, offering a higher dynamic range, occasionally required during its use on surfaces of up to 500 deg C.

The tool's application in the refinery is recent, having shown promising success when used to monitor hydrogen flux emanating from steel surfaces due to corrosion in the laboratory. Hydrogen is generated in the refinery as a result of corrosion under two general circumstances: due to aqueous ('*low* temperature', *i.e.* <120 ^{o}C) corrosion of steel in wet sour or acidic services, and due to sulfidic/naphthenic acid ('*high* temperature', *i.e.* > 240 ^{o}C) corrosion. Additionally, at high temperatures and pressures, substantial molecular hydrogen is solubilised and permeates steel. Moreover, hydrogen emanates from steel during bakeouts. Illustrations of the tool in use are shown in **Figure 1**.

Monitoring hydrogen flux at low temperatures:

At low temperatures, hydrogen flux can be measured as a result of corrosion of carbon steel or low alloy steel exposed to H_2S or HF. Here atomic hydrogen enters the steel and recombines at grain boundaries to produce characteristic hydrogen blistering and/or hydrogen induced cracking (HIC). However, at ambient temperatures, the breakthrough time of the hydrogen through the steel is of the order of hours or days (depending on exact temperature and thickness); there is a significant delay between the onset of corrosion at the interior steel face, and the establishment of a stable flux at the exterior face. Consequently the immediacy of flux measurement does not reflect immediate

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(1) Hydrosteel 6000 (TM), Ion Science Ltd, UK

corrosive conditions, and it is often better suited to constant monitoring. This is particularly the case for many sour applications where corrosion is episodic. Also flux is mediated by the degree of occupancy of traps within the steel.

Monitoring hydrogen flux at high temperatures:

Hydrogen flux can also be measured as a result of corrosion by naphthenic acids and hydrogen sulphide[3], usually at 280-400 ^oC. The hydrogen is evolved via the simplified reaction below:

$$Fe + 2R\text{-}COOH \rightarrow Fe(R\text{-}COO)_2 + H_2$$

At such temperatures, stable flux corresponds to sulfidic and naphthenic acid corrosion occurring tens of minutes previously. Also the permeation of hydrogen through steel at high temperature is usually independent of steel quality as traps for hydrogen play a minor role. Such active corrosion is not readily obtained by any other instrumentation, and compliments the weekly corrosion trends measurable by an online wall thickness monitor. The lack of other corroborative active corrosion data is perhaps the flux technology's most negative feature at present: it is difficult to correlate, particularly in view of high temperature corrosion being not fully understood. This paper studies the use of the flux tool in three separate trials, addressing different applications for the equipment.

Data treatment

Flux data were converted to hydrogen activities $\boldsymbol{a}$, [(bar molecular hydrogen)$^{1/2}$] on the basis of equation (i) after Grabke and Riecke[9]:

$$\boldsymbol{a} = 3.74 \times 10^{-6}.\, J_{ss}.\mathbf{w} \exp(-3586/T) \qquad \text{(i)}$$

for representative carbon steel, where J_{ss} is the steady state flux (pl.cm^{-2}.s^{-1}), $\mathbf{w}$ the steel thickness (cm) and T the temperature (Kelvin). This treatment was particularly important in the first two trials as equipment of highly variant thickness, service and temperature was evaluated. Note the square of activity provides an equivalent pressure of molecular hydrogen required at a steel face to induce the registered flux to permeate under steady state. At low temperatures, activities equivalent to several hundred thousand bar equivalent can be generated by aggressive hydrogen promoters such as sour gas. By contrast, naphthenic acid corrosion delivers robust flux (often in excess of 1000 pl.cm^{-2}.s^{-1}), which corresponds to a hydrogen activity of only a few bar. This reflects the dramatically increased permeability of steel at increasing temperature (by a factor of 70,000 between 30 and 300 ^oC), reflecting both increased solubility and diffusivity.

REFINERY FIELD EVALUATIONS

Location 1: Use of hydrogen flux monitoring in conjunction with an online wall thickness field monitor

Introduction

The flux tool was incorporated into a monitoring programme for two crude distillation units (CDU) in a refinery. This refinery processes a variety of crudes, including a number of opportunity crudes which contain high Total Acid Number (TAN) or high sulfur. Thus, the threat of naphthenic acid corrosion (NAC) is relatively high.

Procedure

Potential monitoring locations were identified on two very similar crude distillation units 'CDU-1' and 'CDU-2' where corrosion resulting in efflux of hydrogen was likely. Although both units are almost identical, the feed diets vary slightly. However, it is expected that identical locations would exhibit similar hydrogen activities. These included a selection of both low temperature sites, where sour water corrosion was possible, and high temperature sites, where naphthenic acid attack and sulfur corrosion could occur. In parallel, an online wall thickness monitor was used to measure wall losses in both units[10]. Some of the sites identified for the flux measurement were directly comparable between the two units. Others were proximal to an online wall thickness monitor.

After initial measurements, it was clear that some sites were subject to very low hydrogen activity, so it was decided to reduce the number of sites to those which showed the highest hydrogen activity, which in number would also be more manageable for the operators (who carried out the measurements during their daily rounds).

Results and Discussion

Hydrogen flux was converted into hydrogen activities according to equation (i) above, and results are presented on a log scale in **Figure 2**. The activity from all points was relatively low as compared to laboratory studies of flux, and the limited published data of field trials using the tool. For example, Grabke and Reicke[9] quote activities $\boldsymbol{a}$ of 1500 bar$^{1/2}$, equivalent to over 2 million bar, cathodically induced with concentrated sulfuric acid, and Dean[8] reported a monitored flux incident due to sour corrosion of amine service steel of 600 bar$^{1/2}$. There are a few significant peaks from test sites on either crude distillation units, and variation in activities less than 0.0001 bar$^{1/2}$ are completely attributable to noise.

However, **Figure 2** does show some clear trending. Firstly, the activity from any particular site tended to remain within a band of activity spanning one or two decades. Furthermore, identical site pairs on the two distillation units provided indicated similar activities. This is particularly underscored by the time averaged activity for each site, for which there is close correspondence in values obtained from identical sites.

Over the trial period, wall thickness measurements were also being made on the CDUs, at some points which corresponded to the Hydrogen monitor steel locations. These readings were also consistently low, corroborating with the indication of low corrosion activity provided by the flux tool. While some high TAN crudes were processed, these were blended in such a way that the overall TAN level was not in excess of approximately 0.7, again, consistent with low hydrogen activities measured at the high temperature measurement sites, one of which is shown in **Figure 2**.

During the trial period at the refining sites, operators of the equipment made the following additional observations concerning the flux tool:

- Quick and easy, can fit into normal operator rounds
- Magnets lost power on HT probe
- Difficult to fit on non-standard geometries – LT only bends in one plane.
- Difference between after 1 min and maximum saturation

Measurements at high temperatures were found to be affected by cooling of the surface by the high temperature roaming probe used (HT-R). It is understood that the manufacturers of the technology have since ensured that insulation is used to minimise cooling of this probe. A fixed probe would also avoid this, but is more appropriate for long term logging and inspection at high criticality sites. The effect of temperature in measurements at Location 2 was improved by taking measurements after five minutes, rather than 60 s.

Conclusions

While no direct correlations between hydrogen flux and wall thickness measurements could be made at Location 1, consistently low flux readings and wall thickness readings were considered to serve as a zero datum from which to compare future readings should more opportunity crudes be utilised. The tool is thus considered favourably as part of a bigger monitoring package in conjunction with crude oil risk assessment programs.

At Location 1 is considered that the roaming flux tool is beneficial in providing operators with notification of a potential change in corrosion rate, which is possible within a much shorter time-frame than with an online wall thickness monitor or other monitoring tools.

Location 2: Correlation of hydrogen flux measurements due to napthenic acid / sulfidic corrosion with the corrosivity of crudes.

Introduction

The study was aimed at correlation of high temperature corrosivity of crude with hydrogen flux measurements in different refinery field service environments.

Procedure

An identical probe to that used at Location 1 was employed to investigate NAC / sulfidic corrosion at temperatures of >200°C. Spot measurements were taken at various locations in the refinery.

Steel surface temperatures were also measured using a portable pyrometer gun(1); bulk temperature of the process streams was also measured using on-line temperature indicators. Both of these measurements were directly input into the Location 2 data control system (DCS). Thickness measurements were obtained by an ultrasonic test method(2), incorporating a probe enabling measurements on steel of up to 500°C. Although these instruments give a tolerance of error of +/- 0.1mm, as they are inherently difficult to use at high temperatures a tolerance of +/- 0.5mm has been used for high temperature corrosion rate prediction. All of the instruments used during the field measurements were used in accordance of the manufacturers procedures.

Following an initial review of corrosive areas within the refinery, five locations were identified as being prone to NAC, based on several years of inspection data. They were located in the refinery's atmospheric crude distillation unit (CDU):

HGO Outlet Headers (vulnerable to sulfidic corrosion and NAC)
Lower Circulating Reflux – Heavy Gas Oil stream (vulnerable to NAC)
Furnace Outlet (vulnerable to sulfidic corrosion and NAC)
Crude Transfer Line (vulnerable to sulfidic corrosion and NAC)

Initially, between 20-30 measurements were made at each of the locations. After three inspections some 20 sites exhibiting the highest flux were identified for continued inspection over the remainder of the eight month trial. Some representative sites are shown in **Figure 3a-d**.

The field results for the hydrogen flux, thickness measurements and temperatures were then correlated with the process stream variables such as Crude TAN (bulk TAN and crude fraction LTAN), crude sulfur levels , H_2S and potential H_2S levels were all taken from the crude oil assay sheets. The crude blend entering the CDU was usually blended from four out of approximately eight crudes. Stream velocity could not be measured directly, however it could be correlated qualitatively using the throughput of the distiller during the time of hydrogen flux monitoring.

Results and Discussion

The field measurements are shown in **Figures 4-8**, together with relevant operational parameters. In view of the correlation between equivalent sites in Location 1, **Figure 2**, it can be immediately appreciated that certain sites show characteristically more hydrogen activity than others. For example, sites 2,5,7 and 9, **Figures 4-5**.

In contrast to Location 1, this data shows strong trending between sites over time, not least because flux, and hence hydrogen activities, were generally higher from Location 2 high temperature sites. Particularly noteworthy was the data collected from the CDU furnace outlets, **Figure 6**, congruent with their very similar functionality and the equivalence of these sites as portrayed in **Figure 3c**. Likewise, the monitoring of three circumferential sites from the crude transfer line, **Figure 3d**, yielded closely trending

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(1) Parmer (IR)
(2) Panametrics 26DL, with high temperature dual probe D790

activities, **Figure 7**. Indeed, an important result from this work was in showing strong co-trending of flux activity at diverse test sites over many months of operation.

Regarding the metallurgical aspects of the areas under study, the lower circulating reflux steel sites (**Figures 3a** and **3b**) are carbon steel, whereas the furnace outlet (**Figure 3c)** and transfer line sites (**Figure 3d)** are 5Cr-0.5Mo steel. Although average flux values shown in **Figure 8** do not indicate a significant reduction in average hydrogen activity from the latter two locations, the peak activities obtained from the lower circulating reflux sites, **Figure 4**, were significantly higher than observed elsewhere.

Figure 5 shows the initial month's data for the Lower Circulating Reflux (HGO stream). All parameters monitored remain fairly invariant between 14 and 22 Oct 2002. However on the 24 Oct 2002 the flux data for most tested sites change abruptly. However, when the TAN number of the crude then rises significantly on 13 Nov. 2002, the hydrogen at most sites decreased and became less variant over time and test location. The 'turbulence' in hydrogen activity during this period coincides with a step change in TAN, and was not wholly unexpected. As reported by Kane and Cayard[11], the progress of NAC is determined not only by naphthenic acids, but by the dissolution of iron sulfide films to form iron naphthenate.

Indeed, 'potential H_2S' provided the strongest hydrogen activity trend indicator out of all the parameters evaluated. Crude Fraction TAN was not nearly so influential, which is perhaps not surprising give the low and fairly constant TAN values. This is particularly apparent in **Figures 6** and **7**. By contrast, in **Figure 4** it is apparent that position #2 activities changed at complete variance with the others. Position #2 peak activities preceded those at most other sites then subsided sharply, consistent with a high rate of corrosion leading to more rapid passivation.

Conclusions

'Spot' correlations of the hydrogen flux measurements could not be made against the process parameters as it was acknowledged that the corrosive mechanisms prevailing during NAC corrosion are not instantaneous, but subject to a complex sequence of conditions, most significantly with respect to the formation of passivating sulfide scale. What has been confirmed is an internal consistency in the flux measurements obtained and the complexity of naphthenic acid corrosion in refinery operations. The consistent trending between all of the points measured indicates good sensitivity of the hydrogen flux tool.

Although crude and fraction TAN values were measured, it was not seen to be as influential in this case as had been previously thought. This is probably due to the comparatively low TAN values that were measured. The results as a whole indicate that in this study the potential H_2S measurements gave the strongest correlation with measured flux.

The lower peak hydrogen activities in the 5Cr-0.5Mo areas also provided some confirmation that Cr in steel provides protective film against sulfidic corrosion and this can slow the onset of NAC.

Location 3: Bakeout application

Introduction

A trial was undertaken to show whether bakeout of vessels was required to achieve low risk of weld cold cracking during nozzle modifications, as a result of dissolved hydrogen in the base metal from hydrogen charging which may have occurred in service.

Bakeouts are normally performed to reduce the concentration of dissolved hydrogen in the vessel walls so that weld modifications can take place with a low risk of cracking caused by the presence of dissolved hydrogen. The dissolved hydrogen usually originates from internal corrosion by sour water or sour amine during normal operations.

Hydrogen fluxes from the walls of three vessels in the Delayed Coker Unit of a refinery were measured during hydrogen bakeout. Bakeout treatments had previously been recommended in preparation for weld modifications during the shutdown. The vessels studied were:

- Unsaturated Fuel Gas Amine Knock Out Drum (V1)
- Unsaturated Fuel Gas Amine Absorber (C1)
- Sponge Absorber (C0)

The vessels were tested for hydrogen charging during the bakeout treatments to show the levels of dissolved hydrogen and to provide guidance for future bakeout treatments. This information could be used in future to shorten the bakeout cycles or to gain confidence needed to forego bakeout treatments when repairs/modifications are required.

Procedure

The bakeout was conducted by placing electric resistance heating pads, covered by insulating blankets, on the vessel shells around the nozzles. The heating sequence consisted of ramping the temperature at 100 ^{o}C per hour up to 350 ^{o}C, which was maintained for 4 hours, followed by still air cooling. Vessel wall temperatures were recorded throughout the bakeout cycle.

A series of hydrogen flux measurements were taken periodically using the flux monitoring tool employed elsewhere in this work, incorporating the high temperature roaming probe, during hydrogen out-gassing from three vessels during bakeout heat treatment prior to welding. Measurements were made on the top nozzle of the unsaturated fuel gas amine knockout drum, the vapour inlet nozzle of the unsaturated fuel gas amine absorber, and the inlet nozzle of the sponge absorber. This was performed by placing the probe in contact with the vessel wall adjacent to the nozzle after peeling back the insulation blanket. Stable measurements were obtained after one minute of contact.

Measurements were conducted during the ramping up phase and into the hold time of each bakeout.

Paint was removed from the measurement area (as indicated in the results tables) by scraping and abrading with emery paper after heating had begun.

Results and Discussion

It was estimated that a peak outgassing rate of 4 nl/cm^2/s is indicative of dissolved hydrogen concentrations of 1-2 ppm in the steel thickness range being measured. This is the critical level below which weld cold cracking is unlikely to occur. Results are shown in **Tables 1-3** and **Figure 9**.

Prior to testing the bakeout of the unsaturated fuel gas amine knock out drum (V1) was thought to be unnecessary since past experience has showed that corrosion and accompanying hydrogen charging is usually not severe enough to cause welding problems. This was confirmed by the measurements made, as no flux measurements >1.5 nl/cm2/s were measured. A small rise in hydrogen flux was seen when the paint was removed, but this immediately returned to low values, indicating hydrogen degassing was not occurring.

The sponge absorber (C0) was in sweet gas service, and again the bakeout was deemed unnecessary prior to testing. The flux measurements were initially small, however one hour and 10 minutes into the test oil contamination was found on the surface. This was confirmed by the measurements made. The presence of paint or oil or any other source of hydrocarbons such as binder in heat insulation on the surface tend to generate hydrogen as they inefficiently combust above 200 ^{o}C, leading to false flux indications. The presence of oil on the insulation is believed to have also given a very high reading obtained during the treatment of the sponge absorber (C0). Peak hydrogen flux occurred between 300-350^{o}C. Hydrogen flux fell rapidly after reaching the peak flux.

Bakeout was considered necessary for unsaturated fuel gas amine absorber (C1), which was in sour amine service. This was confirmed as the hydrogen flux measurements increased with time to a maximum of 4.1 nl/cm2/s, and then decreased to 0.7 nl/cm2/s.

Conclusions

The generally accepted threshold for hydrogen charging is that more than 1-2 ppm dissolved hydrogen in the steel can lead to weld cracking. Flux greater than 3-4 nl/cm^2/s were seen on the fuel gas amine absorber (C1), confirming the need for degassing as recommended. High values were also seen on sponge absorber (C0) but these were attributed to oil contamination rather than hydrogen degassing. Low values were seen for unsaturated fuel gas amine knock out drum (V1) confirming that there was a low risk of weld cracking and hence degassing was not necessary. The following points have therefore been concluded:

1. The hydrogen flux measurement results were consistent with predictions of likely hydrogen charging of the vessels, which were based on Shell and industry experience.
2. Peak hydrogen fluxes were greater than an estimated threshold value of 3-4 nl/cm2/s during bakeout of the amine absorber (C1), but less than this threshold for the other two other vessels tested.
3. Bakeout was justified for the amine absorber (C1).
4. Future hydrogen bakeouts of the amine absorber (C1) may be of shorter duration than the treatment done this turnaround and this should be monitored by hydrogen flux monitoring during the bakeout to optimise the bakeout duration.
5. Future bakeout treatments on other vessels can be monitored with the hydrogen flux monitor used in this work.

OVERALL CONCLUSIONS

At each of the locations studied, the conclusions for that site have been presented.

Overall, the Hydrogen monitor has been used successfully to measure hydrogen activities at high temperatures, with a fast response time. This information, together with operational conditions, is able to provide valuable insight into varying high temperature corrosion rates within the refinery.

The tool has also been used effectively to monitor hydrogen bakeout of vessels during a refinery shut down and could be used in the future to shorten this heat treatment duration.

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TABLES

Table 1. Hydrogen Flux Measurements for Unsaturated Fuel Gas Amine Knock out Drum (V1)

Time	Temperature [C]	Hydrogen Flux [nl/cm2/s]	Remarks
0815	200	0.07	Paint intact
0850	245	0.82	
0856	245	0.35	Paint removed
1000	350	1.50	
1100	350	0.56	
1130	350	0.47	
1145	350	0.00	

Table 2. Hydrogen Flux Measurements for Unsaturated Fuel Gas Amine Absorber (C1)

Time	Temperature [C]	Hydrogen Flux [nl/cm2/s]	Remarks
0820	160	0.26	Paint intact
0830	190	1.10	
0840	190	2.17	Paint removed
1005	290	4.1	
1105	350	1.7	
1145	350	1.17	
1450	350	0.71	

Table 3. Hydrogen Flux Measurements for Sponge Absorber (C0)

Time	Temperature [C]	Hydrogen Flux [nl/cm2/s]	Remarks
0030	220	.48	Paint removed
0038	240	.45	
0051	262	.42	
0100	270	.76	Additional paint removal
0110	280	.75	
0120	300	.67	
0130	320	1.37	
0140	340	4.4	Oil contamination
0145	340	1.16	
0200	350	1.07	
0230	350	0.44	

FIGURES

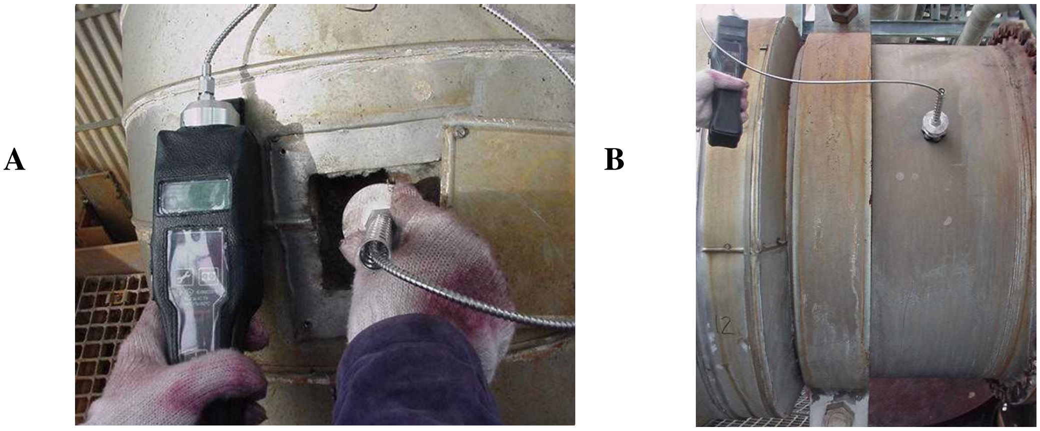

Figure 1. Two examples of flux probe deployment (from location 2). The high temperature roaming (HT-R) probe is shown. Note in the case of **A**, the probe is adpressed by the operator over the measurement period to ensure good contact between the probe's flexible underside and steel surface on a pipe bend.

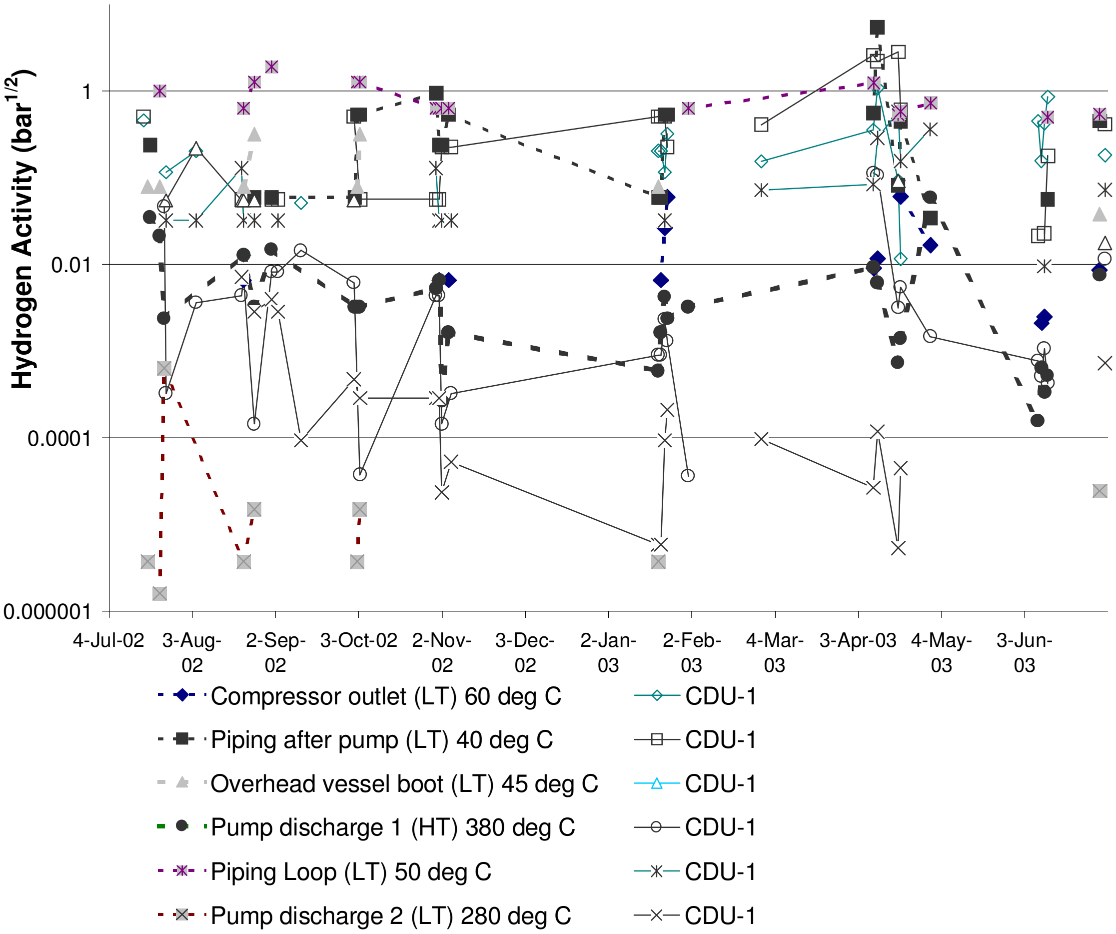

Figure 2. Location 1 activities for the six pairs of identical sites in two crude distillation units could be tested. CDU-1 data is shown by open symbols and solid lines, CDU-2 with matching but closed symbols and broken lines.

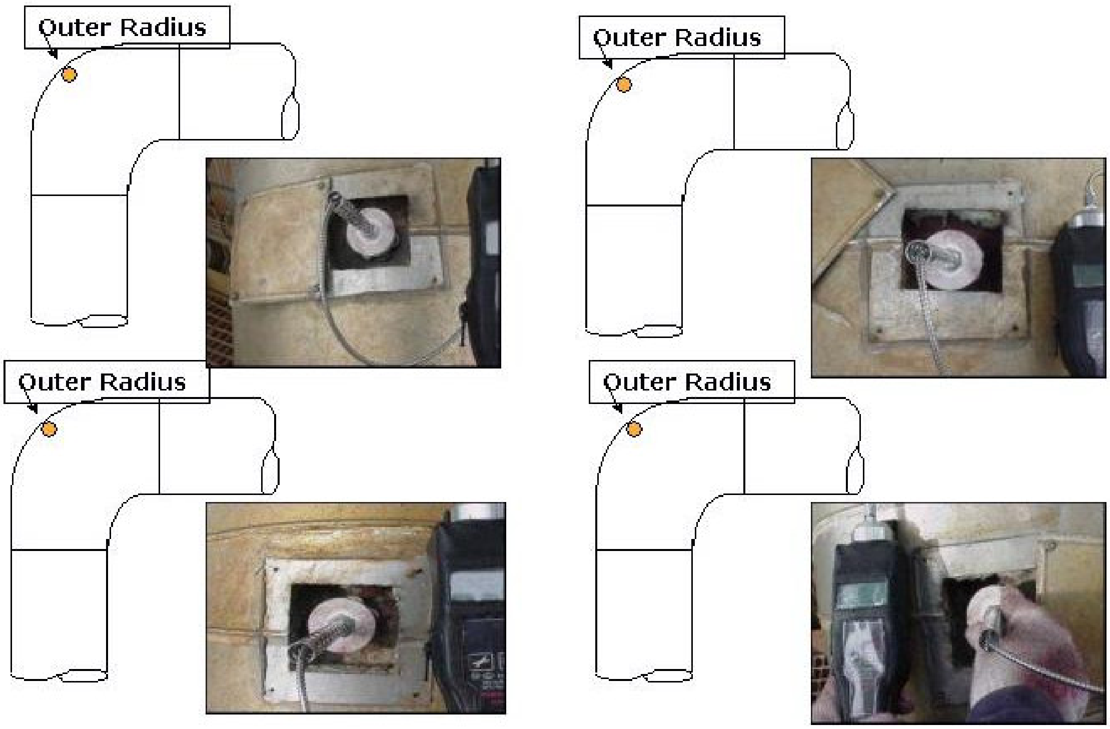

Figure 3a. Location 2, HGO outlet headers

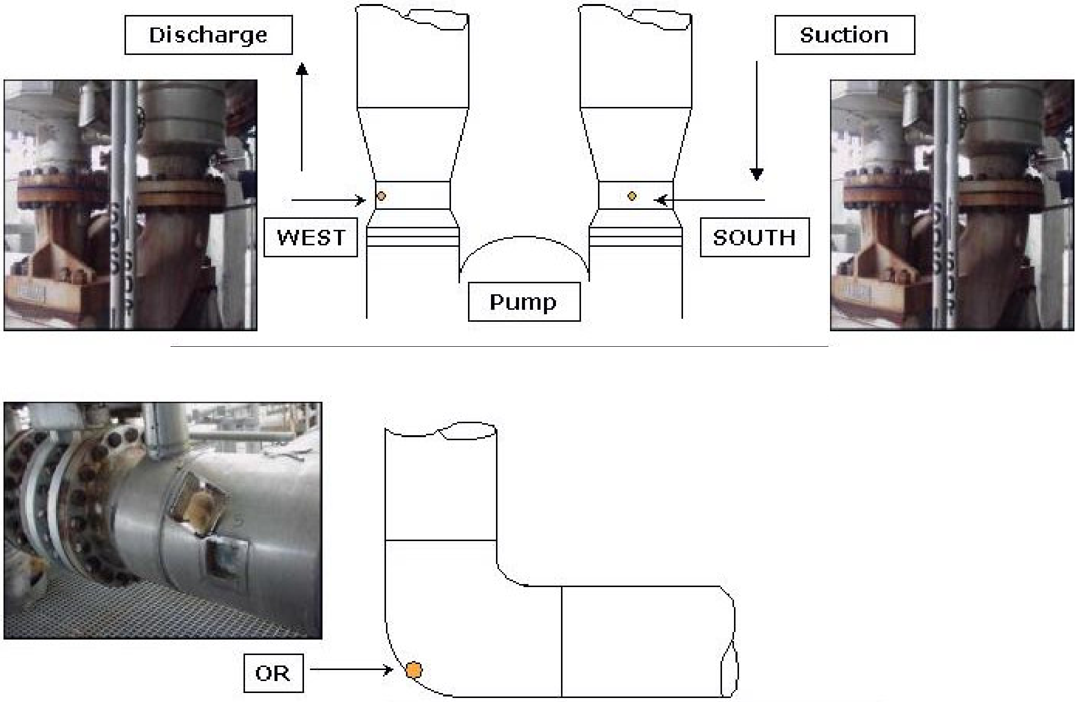

Figure 3b. Location 2, HGO Stream

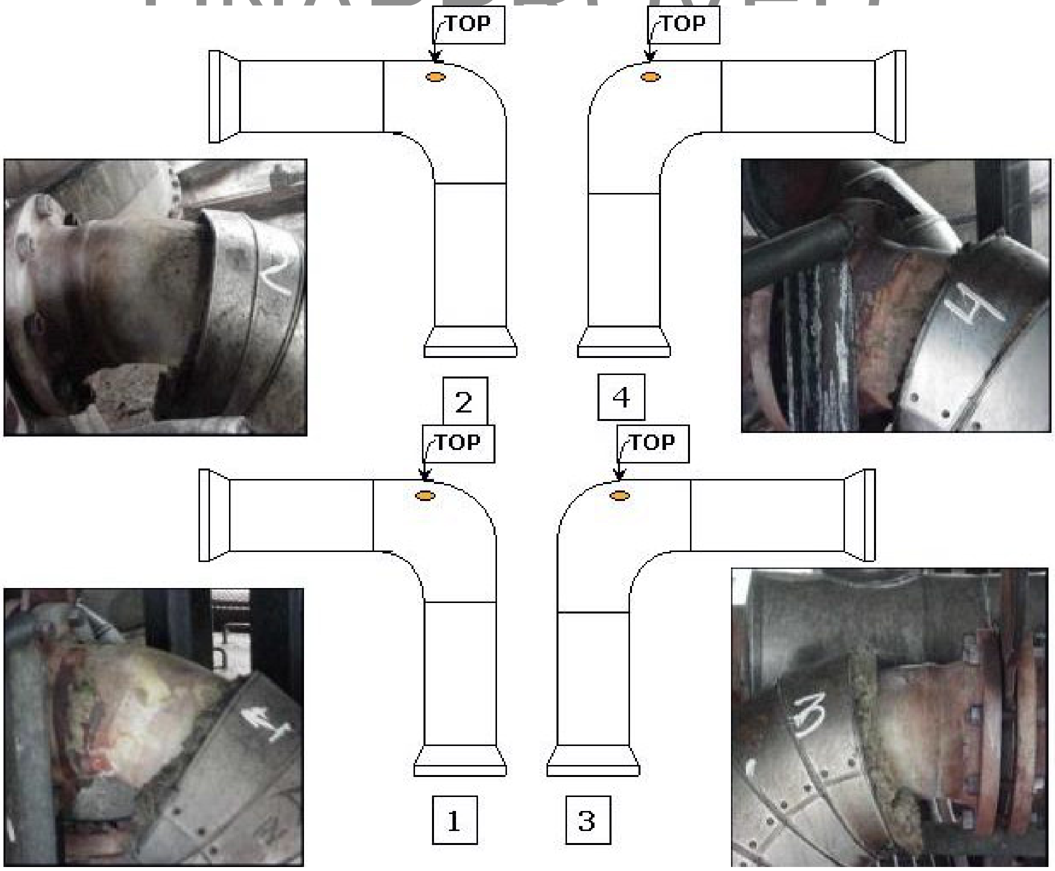

Figure 3c. Location 2, furnace outlet headers

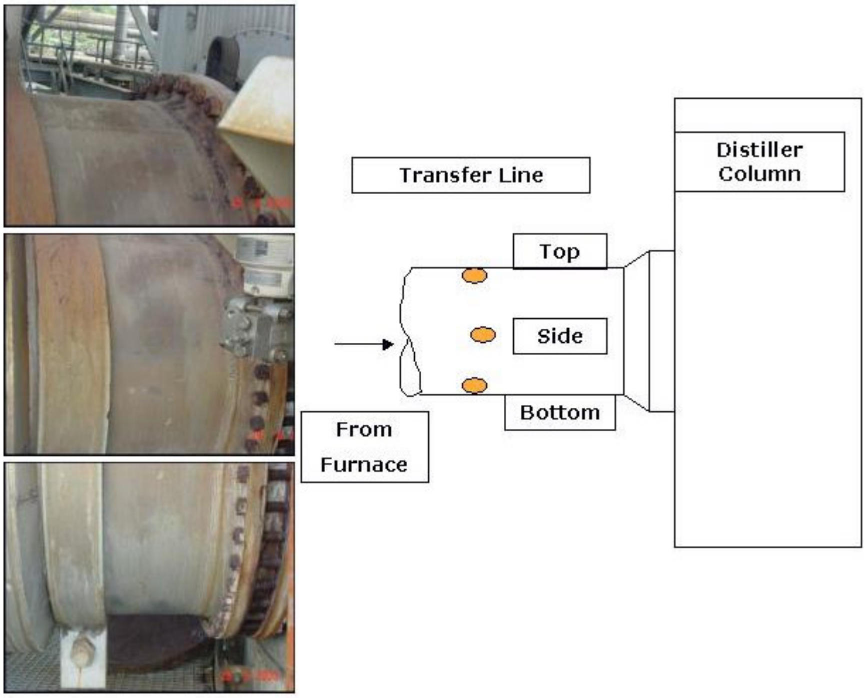

Figure 3d. Location 2, Transfer line

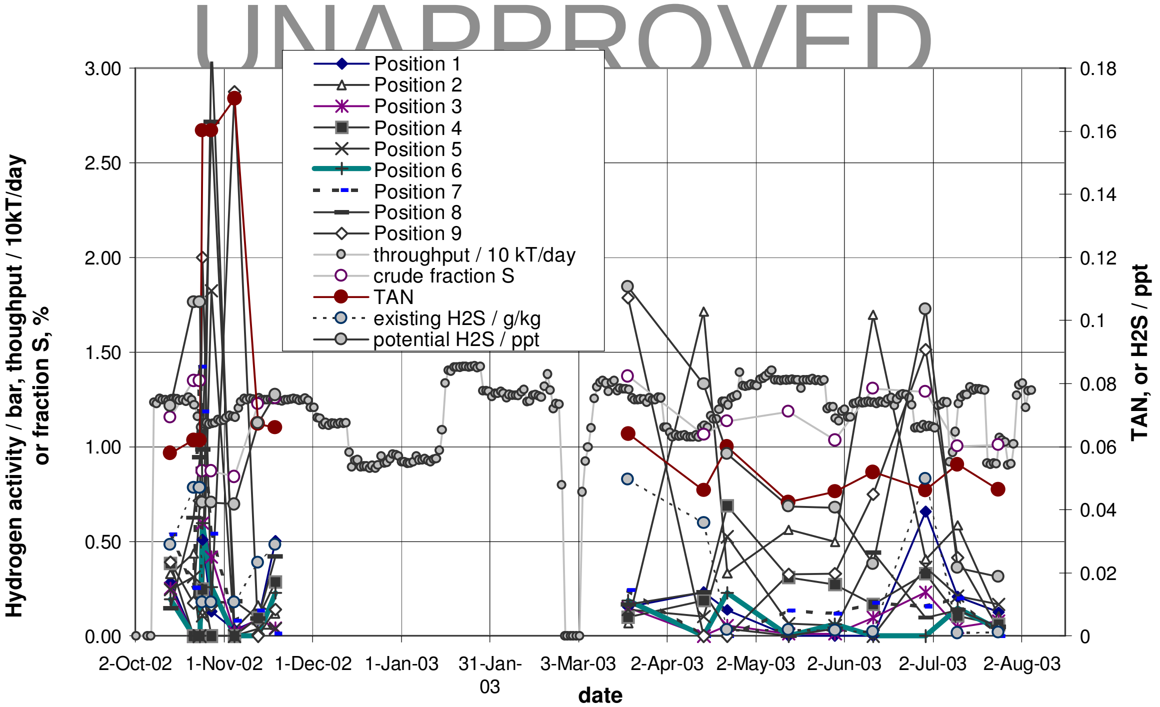

Figure 4. Location 2, hydrogen activities and operating parameter for the Lower Circulating Reflux (HGO stream). Hydrogen activities are given as equivalent molecular hydrogen pressures in bar.

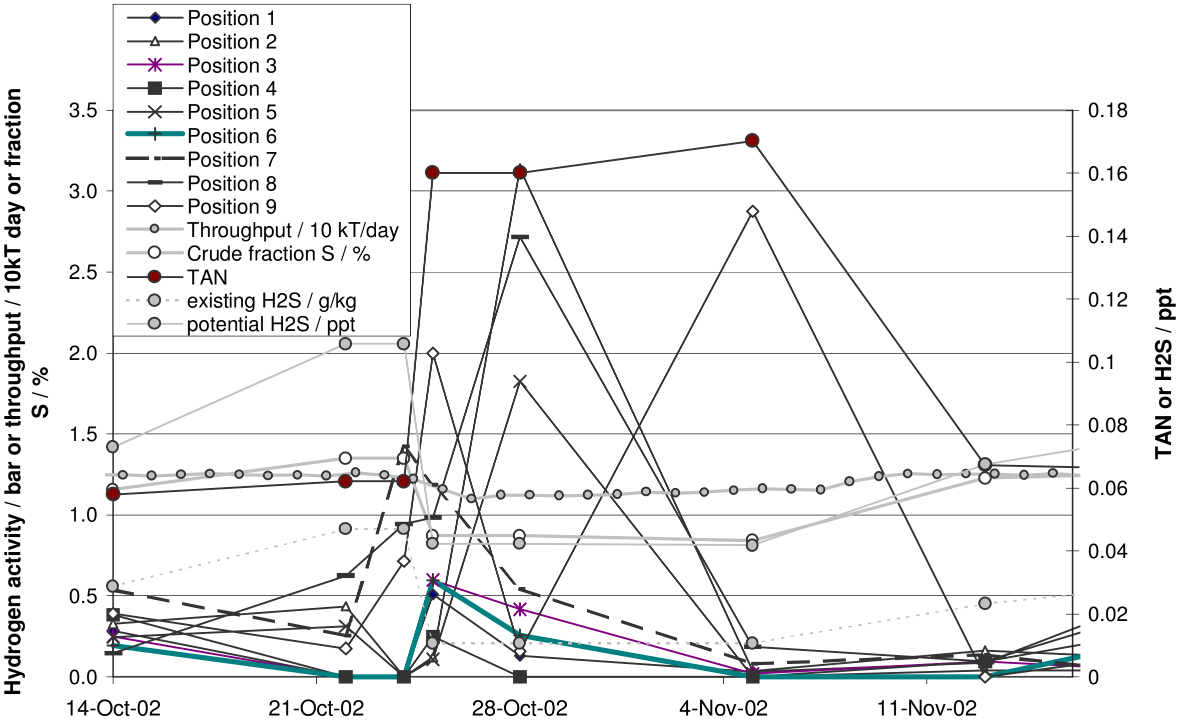

Figure 5. Location 2, detail of **Figure 4** (14 Oct. to 13 Nov. 2002).

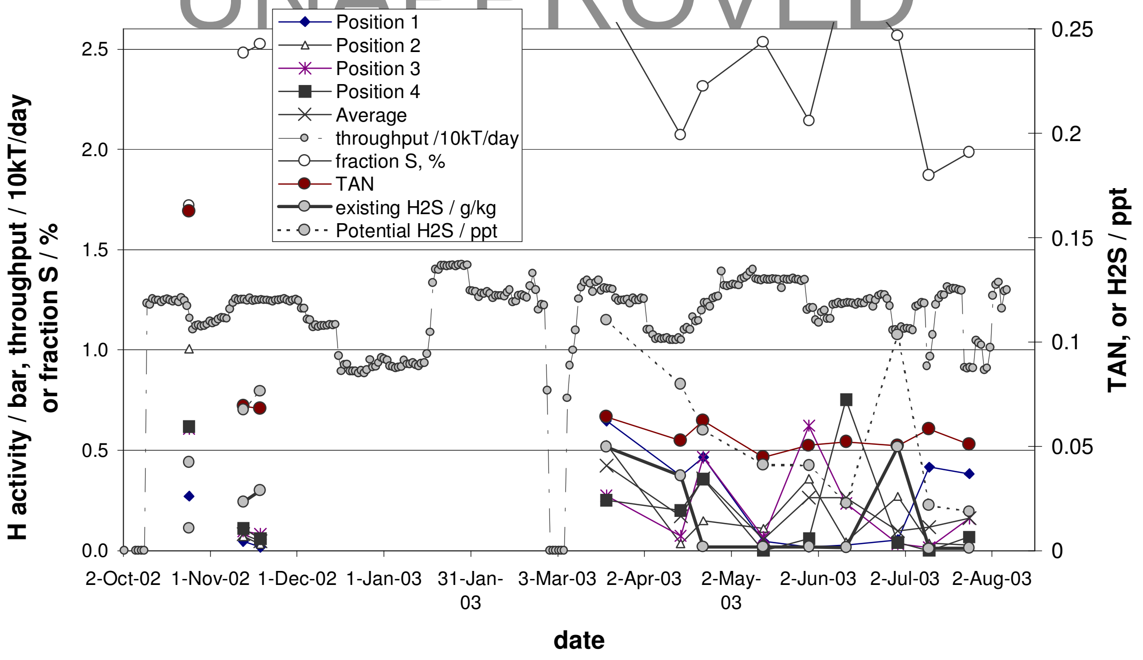

Figure 6. Location 2, data set for CDU Furnace Outlets

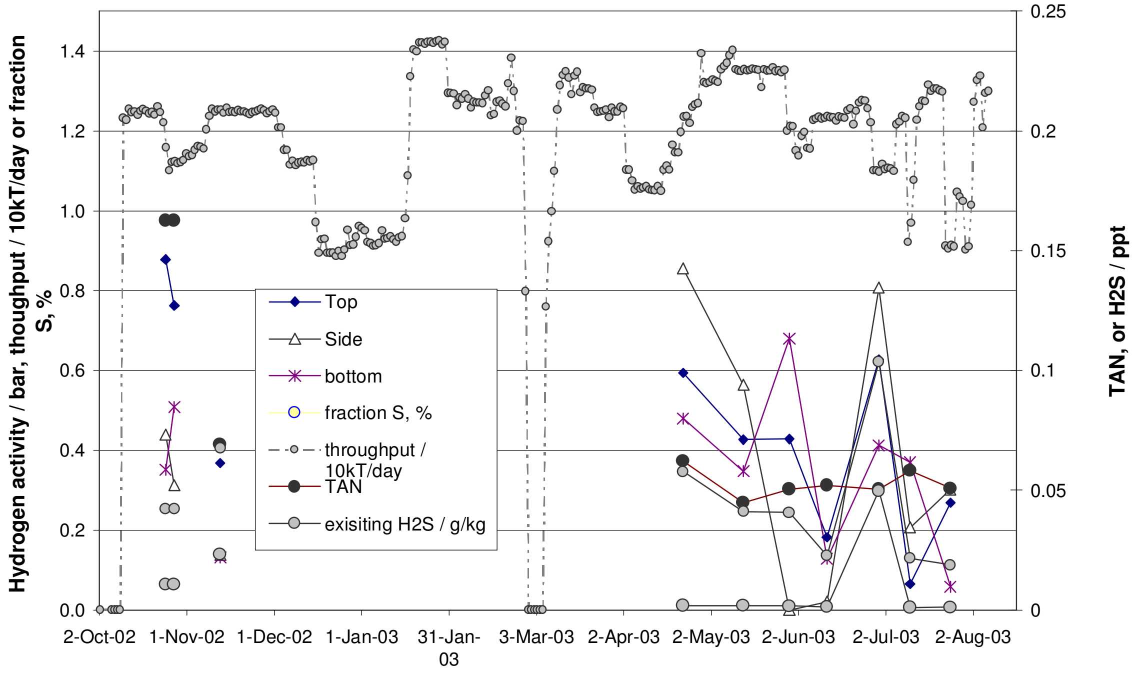

Figure 7. Location 2, complete data set for the Transfer line.

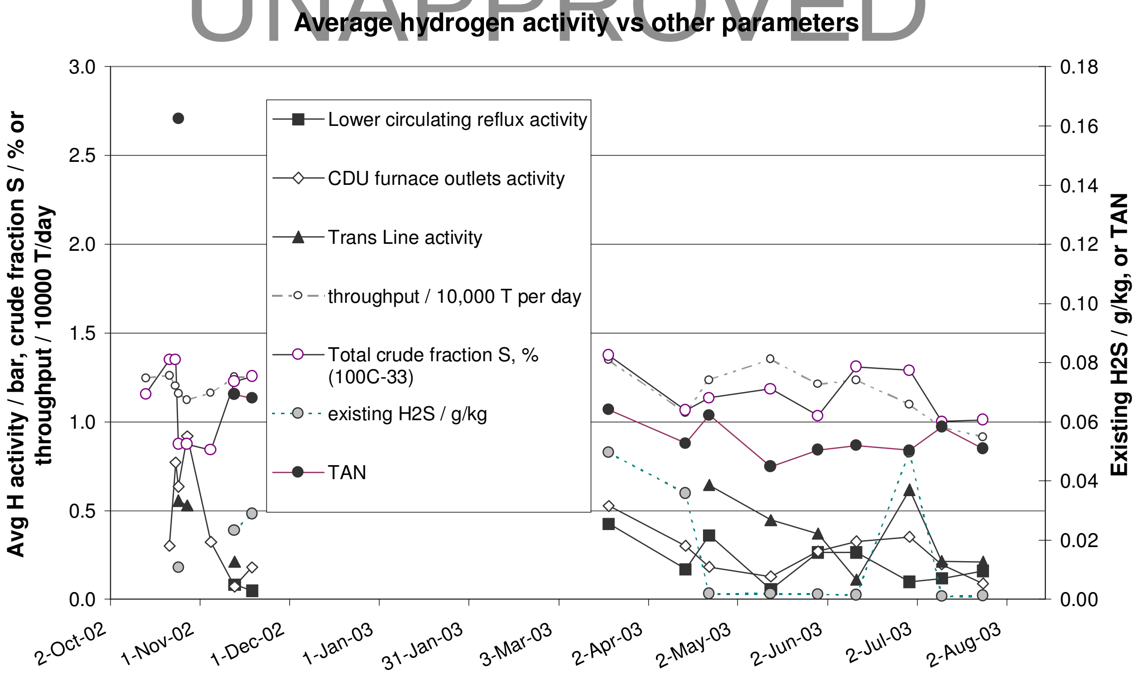

Figure 8. Location 2, Average hydrogen flux measurements, and operating parameters, for the whole trial and all locations.

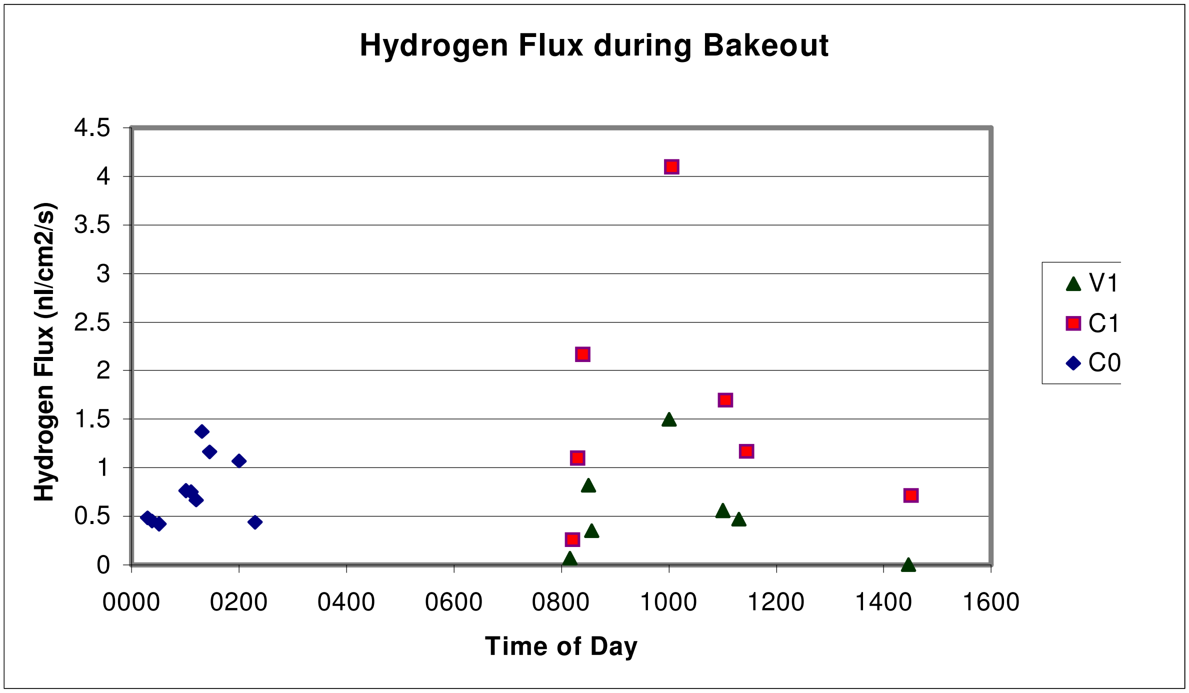

Figure 9. Location 3, Hydrogen flux readings obtained during bakeout.