FIELD APPLICATION OF A NEW, PORTABLE, NON-INTRUSIVE HYDROGEN MONITOR FOR SOUR SERVICE

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ABSTRACT

In oil and gas production, corrosion caused by hydrogen sulfide can result in hydrogen induced cracking in susceptible steels. Hydrogen induced cracking is dependent upon the concentration of hydrogen in the steel matrix, and, therefore, the hydrogen flux through the steel. A novel, non-intrusive, rapidly portable, detector has been designed to measure hydrogen flux. The equipment has been applied to demonstrate the success of a corrosion inhibitor program in a wet sour gas gathering system. Hydrogen emanating from the external pipe surface was continuously collected in a stream of air and conveyed to a remote hydrogen analyzer. The method enabled reliable measurement of hydrogen flux within 1 minute to a resolution of ± 2 picoliters /cm²/s.

INTRODUCTION

A new "enhanced" corrosion inhibition program was initiated to prevent hydrogen induced cracking and general corrosion in an extensive wet sour gas transportation system in Saudi Arabia. Since hydrogen damage was the critical aspect to be controlled, the development of an accurate method to determine hydrogen flux through the pipe wall was essential.

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The Saudi Aramco master gas system¹ was developed in the mid 1970's to transport gas from 32 gas/oil/water separation plants (GOSPs) to 3 gas plants where sweetening, fractionation, and liquefaction occur, prior to shipping to industrial customers. Prior to sweetening, the system carries wet sour gas in 49 pipelines with a total length of several hundred kilometers. The size range is from 24- to 48-inches in diameter. There are also numerous lateral tie-ins.

The system was constructed using spiral and longitudinally welded API-5L X52 pipe. Although the system was built from pipe specified to be resistant to hydrogen damage, experience proved that the chemistry limitations imposed on the pipe at that time were not sufficient to prevent hydrogen damage.² As a result, company specifications were further developed, limiting steel chemistry and manufacturing processes for pipe being purchased after the early 1980's. Inhibition programs were used to reduce corrosion in the system.

Due to the lack of in-service corrosion failures, it was generally considered that the inhibition program was successful. However, between 1988 and 1993, instrumented scraper runs were performed on twenty wet sour gas pipelines. Varying degrees of internal corrosion damage including hydrogen induced cracking (HIC) were found on 30 percent of the lines. Damage indications from instrumented scraper runs were confirmed by bell-holing and external ultrasonic thickness measurements at selected test sites. Figure 1 presents an example of data provided by the instrument scraper runs for Pipeline A in 1989 and 1992. Table 1 summarizes the level of corrosion damage found in these surveys.

Pipeline A is a 36-inch pipeline built in 1978. It is about 16.3 km long with a wall thickness of 0.267-inches from kilometre 0 to 13.6, and a wall thickness of 0.371-inches from kilometre 13.6 to the gas plant. The line ends at a scraper trap, nominally 42-inches in diameter with a wall thickness of 0.438 inches.

The pipe was made from a low sulfur steel with copper additions. However, inclusion shape control with either calcium or rare earth additions was not attempted. The result was a structure with elongated inclusions.

In order to understand the inadequacies of the existing corrosion inhibitor system, a detailed analysis of the phase behavior of the system was performed. A review of system operating parameters and phase simulations using HYSIS found that the gas phase in the pipelines is under-saturated with respect to liquid hydrocarbon, and that water condensation occurred in the lines before hydrocarbon condensation.

Sour gas entering the pipeline system is processed as shown in the simplified Figure 2. Gas from the high pressure production separator and low pressure production separator is fed to compression, and from there to a heat exchanger and an air-cooler to condense liquid hydrocarbons, before being reheated to approximately 70°C for transport in the pipeline.

After reheating, the gas is a single phase fluid, under-saturated with respect to liquid hydrocarbon and water. However, as the gas moves through the pipeline, cooling occurs and the water dewpoint can be reached resulting in corrosion.

Figure 3 presents the temperature-pressure hydrocarbon phase envelope for the gas fed to Pipeline A. The actual operating conditions of Pipeline A are indicated by the open triangles, and these open triangles indicate that liquid hydrocarbon will not condense.

Figure 4 shows similar data for the water component of the stream. Water condensation is expected to occur in Pipeline A during the summer months, resulting in corrosion. Figure 4 demonstrates two cases, summer and winter. The gas will carry much more water in the summer than in the winter because of the effect of the air-coolers shown in Figure 2. In the summer, the air-coolers will cool less efficiently and a greater amount of water will be retained in the gas stream. The condensing water dissolves the corrosive constituents of the gas phase, resulting in pH values calculated to be in the region of 3.6.

DESIGN OF THE INHIBITION SYSTEM

The corrosion inhibitor package is composed of two components, the active inhibitor chemistry and a carrier solvent to dissolve the inhibitor molecules.

In the original design of the inhibition system, insufficient corrosion inhibitor and carrier solvent were injected into the pipelines. Accordingly, the phase behavior analysis suggests that the inhibitor carrier solvent would flash off soon after injection and the inhibitor active component would be extremely poorly distributed in the pipeline. The expected result would be that the inhibitor would be ineffective, which is confirmed by the field observations.

The new or "enhanced" corrosion inhibitor package was started in 1994 and takes into account the hydrocarbon dew-point behavior. The inhibitor package was reformulated with a greater dilution of carrier solvent and the injection rate increased. The program is designed to ensure adequate delivery of corrosion inhibitor throughout the length of each wet sour gas pipeline in order to minimize the risk of further internal corrosion damage and minimize the risk of failure due to HIC. This objective is achieved by injecting inhibitor plus sufficient diesel carrier solvent to ensure the presence of a liquid hydrocarbon film throughout the length of the lines.

Figure 5 shows the calculated hydrocarbon phase behavior for the enhanced package for Pipeline A. The pipeline now falls within the liquid hydrocarbon envelope indicating that hydrocarbon will be present to carry the inhibitor along the line. Also, hydrocarbon condensation occurs at a higher temperature than the water condensation, and so much, if not all, of the pipeline surface will be expected to be hydrocarbon-wet.

MONITORING METHODS

To verify the performance of the enhanced inhibitor system, three approaches were used. (a) Perform fluid sampling at the gas plant to assure that the inhibitor chemistry was present. (b) Monitor the further development of HIC damage at selected bell-holes. (c) Develop a reliable monitoring system that could demonstrate that the hydrogen flux had been reduced below the level required to cause HIC along the length of the pipeline.

Method (a) proved to be problematic. While it proved possible to easily detect at the gas plants the presence of the diesel solvent from the inhibitor formulation, it proved much more difficult to verify by analytical methods whether or not the inhibitor itself was present. Further it was impossible to determine analytically if the concentration of inhibitor reaching the gas plants was sufficient to prevent HIC. The quantity of diesel reaching the gas plants was less than that predicted by the theoretical modeling.

Method (b) indicated that any crack growth was below the limits measurable by conventional ultrasonic inspection over the test period.

Method (c) required the development of a reliable, rapid-response hydrogen permeation monitoring system that was rugged enough to withstand the Arabian environment, readily field portable, easy to install, non-intrusive, and not subject to large variations in performance with temperature.

DESIGN OF THE NEW HYDROGEN MONITOR

The new hydrogen monitor³ is known as HydrosteelTM. The monitor consists of a collector, that continuously captures hydrogen emanating from the steel pipework, and a hand-held detector module, that continuously draws the captured hydrogen into the detector and records data. The unit is shown in Figure 6.

Collection system

Hydrogen emanates from the outer pipe surface, as a consequence of migration of atomic hydrogen from the pipe interior walls, through the steel under diffusion control, association at the pipe's exterior surface, and subsequent molecular hydrogen desorption. By attaching a plate or hydrogen 'collector' of well defined geometry to the pipe's exterior surface, ambient air may be drawn between the steel surface and collector plate so as to effectively capture desorbing hydrogen. The air stream is then conveyed through the collector handle into a capillary, leading to the hydrogen analyzer.

The collector plate is flexible and is attached to the pipe surface magnetically. The plate and the magnets are supported by a bracket by which the collector can be manually maneuvered, providing intimate contact of the collector with the steel pipe and providing for simple attachment and detachment. With present designs, pipes or vessels with diameters of 16 inches or greater can be accommodated.

The design of the collector is critical to the success of the monitoring system. The magnetic attachment and lack of any liquids in the collector head enables the collector to be attached in the 12, 3, 6, or 9 o'clock position on the pipe circumference without any restrictions, unlike some of the wet cells used by earlier workers.⁴ The position of the collector is critical in lines with stratified flow because water puddling may be very localized. The system can be easily used on a vertical pressure vessel wall.

The collector plate was sized based on the detection limits of the detector unit and the minimum permeation rate that needed to be detected. Literature reports various hydrogen fluxes that result in sufficient hydrogen concentration within the steel matrix to cause HIC. Taira et al.⁵ report hydrogen fluxes of 1,400 pl.cm⁻².sec⁻¹. Cayard et al.⁶ report hydrogen fluxes of the order of 650 pl.cm⁻².sec⁻¹ on a 12.6 mm thick HIC resistant plate. Hay and Rider⁴ report fluxes of the order of 30 pl.cm⁻².sec⁻¹ from a 9.27 mm thick, HIC susceptible API-5L Grade B pipe.

The highest diffusible concentration of hydrogen in the steel will be at the hydrogen entry surface. However, it must also be appreciated that the steel matrix itself is not uniform, and that a lower concentration of hydrogen may be necessary to cause HIC at a mid-wall defect where segregation has occurred in the metallurgical structure.⁷

HydrosteelTM: Ion Science Limited, The Way, Fowlmere, Cambridgeshire, SG1 7QP, England

Under Fick's first law, under steady state conditions, permeation flux is defined by the relationship:

$$J_{\infty} = FDc_0/L \qquad (i)$$

where, J_{∞} is the steady state flux, F is the Faraday, D is the hydrogen diffusion coefficient, c_0 is the hydrogen concentration at the inner pipe surface, and L is the membrane (or pipe wall) thickness. It is assumed that the exit sub-surface concentration is zero.⁸

From equation (i), it is apparent that the permeation flux is inversely proportional to membrane thickness. Various workers also report that for different steels the hydrogen diffusion coefficient, D, can vary.^{4,6} Therefore, hydrogen flux, per se, is not an absolute measure but is dependent on factors such as pipe wall thickness, steel composition, and even measurement technique. Measurements made with an intrusive probe based on a thin steel membrane would be expected to be different to measurements made through a pipe wall in the field by an external detection device.

To provide an assurance that a "safe" hydrogen flux can be accurately determined, the new hydrogen monitor must therefore be able to detect hydrogen fluxes at least an order of magnitude smaller than the smallest critical flux reported for the typical pipeline wall thickness of interest. The required minimum measurement is therefore of the order of 2 pl.cm⁻².sec⁻¹.

Ideally, tests on pipe samples removed from the field would be used to develop data on the critical concentration required to cause HIC and therefore the critical permeation flux that will result in that concentration. However, this was not possible during the initial phases of the present study.

Detector

A low voltage, pneumatic pump, draws the air stream from the collector plate across the sensing face of a detector. The detector's high sensitivity and reproducibility, described below, is achieved on account of very well defined diffusion boundary conditions prevailing between the sensing face and the anode of the amperometric cell, at which hydrogen is efficiently oxidized, and where, therefore, the concentration of hydrogen is practically zero. The detector current is amplified and output, either as an analogue or digital signal.

Power is supplied to the detection system from four 1.5V alkaline batteries, to enable roaming application of the detection system. It includes a digital display and data storage facility, and is designed to meet current BASEEFA (BS EN 50020:1995) intrinsic safety standards (EEx ia IIC T4), enabling its deployment in all hazardous areas.

Calculation of hydrogen concentration

The flow of the air, F, across the steel pipe surface is carefully regulated by means of restrictions and a flow bypass between the pump and detector to ensure it is smooth and remains constant. This is particularly important as the flux of hydrogen emanating from the steel J is given by:

$$J = c \ge F/A$$
(ii)

where A is the effective area over which hydrogen is captured, and c the enhanced hydrogen concentration resulting from hydrogen entrainment.

System calibration

The zero hydrogen datum point for the measurement is the background hydrogen concentration in air at sea level, namely 0.5 ppm.⁹ Rapid diffusion and hence rapid dispersion of hydrogen in air results in a very low and hence stable background concentration prevailing in the ambient air of most environments, enabling very slight enrichment of the hydrogen content of air collected from a steel surface to be monitored.

The minimum detectable flux of hydrogen emanating from a surface, $J_{\text{sen,min}}$ is given by

$$J_{\text{sen,min}} = (F_{\text{sam}} \ge c_{\text{min}}) / A_{\text{eff}}$$
(iii)

Where F_{sam} is the sample flow rate, c_{min} is the minimum detectable concentration of hydrogen and A_{eff} the effective area over which hydrogen is collected.

To determine these parameters, experiments were carried out in clean laboratory air. The sample flow rate, F_{sam} , was determined volumetrically at 0.52 ± 0.005 ml.s⁻¹ through the cell, irrespective of whether or not the collector was attached to steel. The response of the sensor to hydrogen was evaluated by stepwise addition of hydrogen to a large enclosed volume of agitated air. A linear sensor response was obtained in the range 0 to 200-ppm v/v hydrogen in air. c_{min} was established at 0.28-ppm hydrogen in air equivalent, based on the standard deviation of 5-s time averaged data. A_{eff} was determined by admitting from a pressurized source a known concentration and flow of hydrogen in air through a pinhole leak emanating from the center of a gradated square steel plate. This enabled the efficiency of hydrogen capture from the pinhole leak emanating from any location beneath the collector to be evaluated. By summing the detector response to the pinhole leak captured from all representative plate zones, A_{eff} was calculated at 102 cm². From (iii) we arrive at a value for

$$J_{\rm sen,min} = 1.43 \ \rm pl.s^{-1}.cm^{-2}$$
 (iv)

All measurements in the field on the calibrated system appeared to bear out this sensitivity limit. Further details of how these parameters were evaluated are given elsewhere.⁸

Probe response

Figure 7 illustrates the speed of the probe response. The 6-inch diameter collector was attached to a 2-mm thick mild steel plate that was electrochemically charged with hydrogen at a constant rate. After approximately 750 seconds, the collector was detached from the surface. The probe response is approximately 30 seconds to clear from the reading to zero. This process is reversed at time = 1350 s. The probe response is approximately 30 seconds to rebuild from zero to 90% of the stable reading on reattachment.

Temperature sensitivity

Collector components in the present development are designed to tolerate steel surface temperatures of up to approximately 70°C. Collectors with much higher temperature ratings have been developed for specific applications in the steel industry. The tool reported in this paper was designed to meet the intent of BASEEFA T4 intrinsic safety rating, which, by definition, covers the temperature range -20 to $+40^{\circ}$ C. The ambient air/hydrogen mixture being drawn across the sensor must be limited to the maximum working temperature of the sensor, which is 50°C.

The detector sensitivity and mass flow through the analyzer pump both increased by about 10% on increasing the temperature between 20 and 50°C. Thus the measured flux, (from equation (ii)) was not significantly affected by temperature shifts in this range. For the greatest precision, it is beneficial to calibrate the instrument for the ambient temperature range in which the readings will be taken, and verify the flow rate past the detector cell. In the fieldwork where ambient conditions were about 50°C, it was found to be beneficial to allow the detector to stabilize for about ten minutes after transporting in an air-conditioned car at about 20°C.

FIELD MEASUREMENTS ON INHIBITED PIPELINE

The new hydrogen monitor was used to determine hydrogen flux rates along the length of Pipeline A. Tests were performed at three bell-holes and in the destination gas plant. Data in the gas plant were compared with a proprietary intrusive electrochemical cell. Readings were taken by magnetically attaching the probe to the coated pipe surface.

Along the length of the pipeline, the hydrogen permeation rate was found to be less than 2 pl.cm⁻².sec⁻¹. Readings were taken at 4, 8, and 12 kilometers along the line in early and mid summer, the time expected to be most corrosive due to the less efficient operation of the air coolers in the GOSPs. The 12-kilometer part of the line was of particular interest because hydrogen damage in this area had necessitated replacement of this part of the line.

The line terminated at the gas plant in a scraper trap that was provided to accommodate recovery of cleaning scrapers and intelligent scrapers used for pipeline inspection. In the gas plant, readings were taken as shown in Figure 8. Upstream of the scraper trap, readings were also found to be less than 2 pl.cm⁻².sec⁻¹. Between the mainline take-off and kicker line take-off, a hydrogen flux of 5 pl.cm⁻².sec⁻¹ was recorded. Between the kicker line and the end of the scraper trap, a hydrogen flux of 8 pl.cm⁻².sec⁻¹ was measured. All readings were detectable only at the 6 o'clock location on the piping system.

An intrusive hydrogen probe^{$l\theta$} was mounted at the 4:30 location, upstream of the scraper trap, at the same position along the line as the first external measurement at 6 o'clock. With the intrusive probe, the steady-state reading was in the region of 4 pl.cm⁻².s⁻¹.

As a first order approximation, a constant hydrogen diffusion coefficient (D) can be assumed for the pipe material and the intrusive probe membrane. The membrane thickness (L) was 0.75 mm for the intrusive probe, and the pipe thickness (L) 9.42 mm. Using Equation (i), the expected reading on the external monitoring device in the same location would be of the order of 0.3 pl.cm⁻².s⁻¹, which is less than the minimum flux detectable by the probe. At the exact location, no hydrogen activity was detected by the external probe. Two metres downstream, in the scraper trap where there was less flow,

an external hydrogen flux of 5 pl.cm⁻².sec⁻¹ was recorded. The readings obtained on the two different probes are to a first approximation, comparable and of the same order.

In order to provide additional confidence in the validity of the data, it is reasonable to ask if the reading variation seen along the scraper trap is sensible. The line upstream of the scraper trap is a 36-inch diameter line. After entering the scraper trap, the main line exit is also a 36-inch line. About half way down the scraper trap is the kicker line. This line is a small diameter line. In normal operation it is kept flowing. Because of the flow difference between the 36-inch main line and the very much smaller kicker line, the flow in the scraper trap will be much slower, possibly allowing drop out of water and certainly supplying less inhibitor to that part of the system. Accordingly, a reading that shows greater activity in the scraper trap between the main line and the kicker line is quite reasonable.

This argument can be easily extended to the end of the scraper trap where there would be no flow. The even higher reading in the dead-end of the scraper trap makes intuitive sense. All readings, however, are less than the critical threshold permeation flux, and indicate that the inhibitor program is satisfactorily doing its job.

FIELD MEASUREMENTS ON OTHER SYSTEMS

Objective

While the satisfactory range of the field readings taken on the wet sour gas system was reassuring, it was decided that additional field data collection would be beneficial in order to demonstrate that high readings could be obtained on a system that is expected to be corrosive. Suggestions that the inhibitor system on the wet sour gas system be turned off for a period were not accepted. Therefore another system was selected to demonstrate that high permeation readings could indeed be detected. A nominally horizontal portion of a flare gas line in a GOSP was selected for these additional tests. This line operates between 30 and 100 psig with sour fluids. Exact conditions are unknown due to the uncertainty in which systems are being routed to the flare. The pipe was manufactured to meet API-5L Grade B.

Results

Hydrogen flux readings around the pipe. Spot measurements were taken at 12, 3, 6 and 9 o'clock around the horizontal pipe as shown in Table 2. Hydrogen activity was only detectable in the 5 to 7 o'clock locations where a repeatable measurement of 64 $pl.cm^{-2}.sec^{-1}$ was obtained. The logical conclusion is that this was the only water-wetted part of the pipe. This illustrates how critical the selection of monitoring location is when evaluating corrosion, especially by hydrogen permeation techniques.

Hydrogen flux readings along the pipe. A second series of readings was taken from sites at regular intervals of about 0.3 m (1 ft) along a 10-metre length of pipe, exclusively at the 6 o'clock position. Results are presented in Table 3 and Figure 9. The striking features of these readings were (A) the repeatability and (B) the variation between locations.

With regards to repeatability, it was possible to return to the sites of significant readings and obtain further readings with only small variation. Furthermore, sites where no flux was indicated consistently yielded zero readings upon re-testing. Table 2 also bears out the repeatability of tests at a specific 6 o'clock site of moderate hydrogen flux; there was a difference of only 5% between the highest reading and lowest reading.

The possible reasons for the range of readings along the pipe are variations in water pooling inside the pipe, variations in surface films, and possibly variations in hydrogen traps in the steel matrix itself. The important factor is that it illustrates the benefits of an easily portable, rapid-reading hydrogen flux monitor. It also helps to explain the field observation that hydrogen damage is usually quite sporadic.

IMPROVEMENTS OVER OTHER HYDROGEN MONITORING SYSTEMS

Measurements were performed on Pipeline A at one location (10.5 km) in 1991 using a magnetically attached, palladium coated, sealed Devanathan cell type device. Although data were obtained, several difficulties were involved that discouraged further field use. The pipeline surface required cleaning to bare metal. The cell output was found to be strongly dependent upon small variations in the pipeline surface temperature at the temperature of interest, approximately 50°C. The cell required sealing to the pipe surface with silicone grease. Under the field conditions, blowing sand entered the silicone grease and may have impacted the data.

CONCLUSIONS

- A new, intrinsically safe, non-intrusive, rapid-response hydrogen monitor has been developed and applied to verify the effectiveness of a corrosion inhibition program designed to prevent HIC. Hydrogen flux through the steel pipeline has been reduced to insignificant levels, well below the level of hydrogen flux normally associated with HIC.
- The ease of external mounting and the ability to assimilate accurate data rapidly allow multiple readings to be taken around or along a piece of pipe or a vessel. Taking advantage of this portability, data has been generated that shows hydrogen fluxes varying over two orders of magnitude in a few metres length of pipe. Similarly, significant differences have been detected around the circumference of the pipe. The 6 o'clock location, as would be expected, proved to be the most useful.
- With the recent acquisition of pipe samples from Pipeline A, additional laboratory testing can be performed to verify the critical concentration of hydrogen necessary to cause HIC of the steel, and calculations performed to determine the actual critical permeation rates for these two pipe spools.

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ACKNOWLEDGEMENTS

- The authors wish to acknowledge the Saudi Arabian Ministry of Petroleum and Mineral Resources, the Saudi Arabian Oil Company (Saudi Aramco), and Ion Science Limited for granting permission to present and publish this paper.
- The United Kingdom Department of Trade and Industry, Welding Alloys Ltd, Fowlmere, and Saudi Aramco's Engineering Services Technology Program for support in the development of the detection system.
- J. Perez and his colleagues in Saudi Aramco's Pipelines Department for facilitating field testing.
- G. Wilken for theoretical development of the enhanced inhibition system.
- A. L. Lewis, Saudi Aramco, for permeation data developed by the intrusive electrochemical probe.
- D. M. McIntyre for useful suggestions.

NOTES ON UNITS

- Gaseous volumes are quoted at 298 K and 1013 mbar
- Gaseous concentrations in parts per million by volume (ppm)
- Bulk hydrogen in steel values in parts per million by mass (ppm)
- Hydrogen flux values in picolitres (10⁻¹² litres) gaseous hydrogen per square centimetre of steel surface per second (pl.cm⁻².s⁻¹)
- Gas flows in millilitres per second at one atmosphere (ml.s⁻¹)
- Diffusion coefficients, D, are usually quoted in units of cm².s⁻¹
- 1 ppm hydrogen in steel = $9.6 \times 10^7 \text{ pl.ml}^{-1}$. Thus, for a steel in which there is a hydrogen concentration gradient of 1 ppm.cm⁻¹, and for which D = $1.00 \times 10^{-5} \text{ cm}^2 \text{ s}^{-1}$, Fick's First Law predicts a flux of 960 pl.cm⁻².s⁻¹

 Table 1: Intelligent Scraper Data

Amount of metal loss	1989	1992
Light (0-20%)	1091	429
Medium (20-40%)	184	409
Heavy (>40%)	0	196

Table 2: Hydrogen Flux Readings Around Pipe

Position on Pipe (o'clock)	$\frac{\text{Hydrogen Flux}}{(10^{-12} \text{ litre. cm}^{-2} \text{ sec}^{-1})}$	
12	0	
3	0	
6	64	
6	67	
6	64	
9	0	

Table 3: Hydrogen Flux Readings Along the Pipe

Position Along Pipe (feet)	Hydrogen Flux (10 ⁻¹² litre.cm ⁻² .sec ⁻¹)	
1	0	
2	3	
3	7	
4	13	
5	23	
6	37	
7	114	
8	67	
9	58	
10	37	
11	99	
12	98	
13	flange	
14	215	
15	116	
16	145	
17	175	
18	96	
19	82	
20	179	
21	0	
22	80	
23	40	
24	48	
25	176	
26	80	
27	0	
28	45	
29	8	
30	20	
31	64	
32	54	
33	13	
34	3	
35	3	
36	6	
37	0	



















