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Urea Reactor Liner Leakage
(A Case Study)

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***Abstract* - This paper covers background information on the 30 years old revamped plant; problems experienced at the plant after many years of continuous operation, e.g., leaks in the SS 316 L (mod) lining due to the combined action of erosion and corrosion; basis of the revamp work on the design information provided by M/S. Saipem a leader in urea production technology. The urea reactor of Urea line 1 of 11 unit recently suffered liner leakage from weep hole Number 34 on dated 28/11/217.This was the second chance of liner leakage 1st leakage was occurred on dated 28th Jan 2006.The line-1 Plant was commissioned in 1988.A total number of 95 weep hole provided in 11/21 Reactors. In any segments four number weep hole are interconnected. This paper intended how to detect leakage and repairing of the weld joint and further precaution to prevent the corrosion/erosion.**

Key Words - Urea Reactor, Weep hole, liner, Detection, Helium.

INTRODUCTION

National Fertilizers Limited, a Govt. of India Undertaking, was incorporated on 23rd August 1974. It is the second largest producer of nitrogenous fertilizer in the country and has four operating fertilizer units located at Nangal, Bhatinda, Panipat and Vijaipur with a total installed capacity of 32.083 lakh tones Urea.NFL, a Schedule 'A' & a Mini Ratna (Category-I) CompanyThe Vijaipur unit, which is an ISO 9001:2000 & 14001 certified, comprises of two streams.The Vijaipur have two ammonia plant M/S. Haldor Topsoe Technology, Denmark capacity 1750 & 1864 TPD for Line-I & line-II respectively and four urea plant of M/S. Saipem ammonia stripping process, Italy. The Line-I plant installed in 1988 and that of line –II in 1997.The capacity of Urea-I urea –II is 3030 & 3231 TPD respectively. The raw material used includes natural gas, water and power. Three Numbers Captive power plant of capacity 17 X 3 MW are used in this complex. Both the plants have consistently achieved high levels of capacity utilization.

REACTORS DETAIL

The all four reactors(R-1) of Urea line 1 &2 plants are 40 meters Height (tan to tan) and ID of the reactor is 2.04 meter. The liner of all reactors has SS 316 L (mod) while shell of line 1 & line 2 are differing. The line -1 both reactors are coil layered vessel while 31 unit has multi layered and that of 41 is mono block reactor. Detail as following tableNo-1.The passivation of reactor liner is done by air which is introduced in CO_2 compressor in 1st stage. The all four Urea reactor have 15 number of high efficiency sieve trays. These have been provided to prevent the escape of gaseous CO_2, which must react with NH_3 in the lower portion of the reactor. These trays also help in preventing the internal recycling of the reaction products with higher specific gravity products formed in the upper part of the reactor. Reactor shell is made of CS with a 7mm liner of SS316L (mod) on the inner surface. Liner of the reactor is cladded to the CS shell and longitudinal seams and circumferential seams are differing in line-1and line-2 urea reactors. For detecting any leakage in the liner a weep hole monitoring system has been provided. The line-1 urea reactors have 95 numbers of weep holes. And Urea line-2 of 31-Urea reactors has 77 numbers weep holes and 41 reactorhas 98 numbers weep holes. In the weep hole monitoring system there are two loops of weep holes covering the entire reactor. Data are following in Table No.-1, 2 & 3.

Table No.-1

Reactors Specifications			
Sr. No.	Parameters	units	Value
1	Working Pressure	Kg/cm^2	159
2	Design Pressure	Kg/cm^2	169 at top
		Kg/cm^2	169 +full of liquid at Bottom
3	Hydro Test Pressure	Kg/cm^2	219.7 (Vertical)
		Kg/cm^2	224 (Horizontal)
4	Working Temperature	^0C	188
5	Design Temperature	^0C	200
6	Liner Material		SS 316 L (mod)
7	Shell Material		CS
8	Internals & Trays		2 RE 9(25/22/2)

Table No. 2

LINE 1 & 2 REACTOR DETAIL				
Sr. No.	Specifications	11/21 -Reactor (R-1)	31-Reactor(R-1)	41 Reactor(R-1)
1	Type of vessel	Coil Layered Vessel	Multi Layered vessel	Mono wall Vessel
2	Manufactures	M.H.I Japan	BHPV(Vizag)	L & T
3	Liner, MOC	SS 316 L (mod)	SS 316 L (mod)	SS 316 L (mod)
4	Liner Thickness	7 mm	7 mm	7 mm
5	Shell MOC	SPV 46 Q	A 516 GR 70	A 516 GR 70
		MY56	A 724 GR A	
6	Shell Thickness, mm	SPV 46 Q=16 Inner	A 516 GR 70=10+3	A 516 GR 70=67
		MY56=4.5 X 8 LYR	A 724 GR A=6X12	
		SPV 46 Q=8 outer		
7	Total THK, mm	67	92	74
8	No. of weep Hole	95	77	98
9	Weight, Ton	190	225	195

Table No. 3

Urea Reactors Trays Detail			
Trays No.	Material	No. of Holes	Pitch
1 to 5 (top)	2 RE 69	641	Triangular
6 to 10 (middle)	2 RE 69	1282	Triangular
11 to 15 (Bottom)	2 RE 69	1923	Triangular

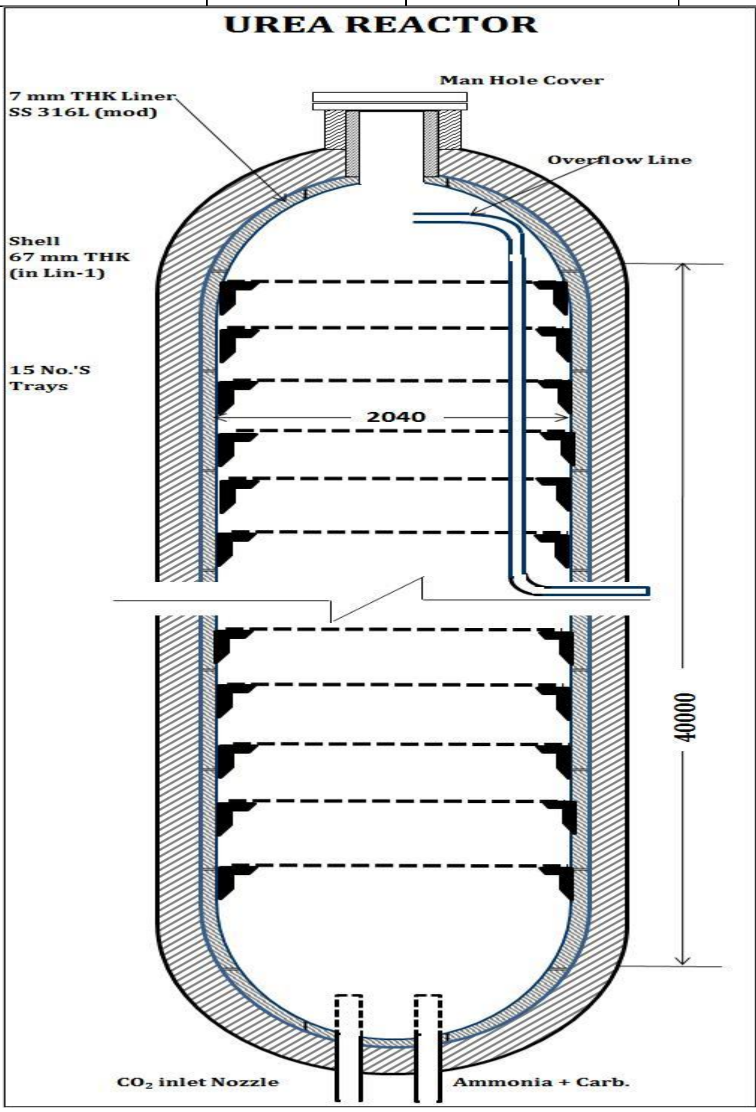

Fig. No. 1

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REACTORS LEAKAGE DETECTION AND REPAIRING

Reactor Liner Leakage on 28/01/2006.

In Urea Line-I and 2 plant all the weep holes are connected to conductivity meter based monitoring system. on dated 28/01/2006 the leakage alarm appeared in weep hole number 23 of 11 stream.The weep hole was checked physically and vapour analysed in laboratory found ammonia vapour and CO_2.Immedially shut down was taken and leakage detected with instrument air and soap solution the leakage easily detected. For find out the leakage the liner plate behind the weep hole was pressurized with instrument air at the pressure of 0.5 kg/cm^2 and other three interconnected weep holes were plugged. Soap solution applied on opposite side of liner. The pin hole leakage easily detected. The pin hole repaired by welding. The repaired portion was passivate with 10 % HNO_3 solution and rinse with Demineralized water. The repaired portion was further checked with instrument air and soap solution no leakage was found. The shutdown duration was only 69hrs. Feed cut to feed in.

Reactor Liner Leakage on 28/11/2017.

On dated 28/11/2017, a weep hole leakage alarm from Weep Hole No. 34 was appeared on lectrotek system. Immediately checked the weep hole No.-34 and vapour analysed in laboratory found ammonia & CO_2 vapour and leakage was confirmed. Immediately shut down was taken at 11.15 hrs on dated 28/11/2017.draining of the H.P loop started at 11.15 hrs. For air purging 11 PT-05 A/B cover open for air purging. Man entry permitted at 18.15 hrs on dated 29/11/2017 after confirming ammonia contents less than 25 ppm and Oxygen contents more than 20 %. Four Numbers man ways were opened for detecting leakage the leakage above 5th tray. So the 4 man ways opened. first of all, leakage detection tried with instrument air & soap solution. For detecting the leakage, the instrument air introduced at the pressure of 0.5 kg/cm^{2}and other three interconnected weep hole plugged so that the pressure retains inside the liner and shell. Soap solution applied on liner for detecting the leakage. Four hour tried to leakage but success did not get. After that our further step to detect the leakage with ammonia and phenolphthalein indicator. But leakage could not have detected. Timing tabulated in Table No-4.

As the weep hole leakage was not clear, the exact location the leak could not be found out with Soap solution and ammonia test.Finally, the DP check and number of the spot, pin point found. All the circumferential Seam and longitudinal seam opposite of weep hole No. 34 between 4th& 5th trays welding done. After that ammonia is introduced at the pressure of 0.4 kg/cm^{2}in weep hole No. 34 and hold up for 10 hrs. and liner and all welding seam checked by phenolphthalein indicator. No leakage was found and man hole cover boxed up. Start-up activities started and feed in done on dated 01/12/2017 at 11.15 hrs. Total time taken for this shut down was 72 hrs (3 days) feed cut to feed in. Typical lifetime of the 316L Urea Grade protective layer of a urea reactor is 20-30 years depending upon passivation and plant load number of the tripping of the plant process variations etc. Typical lifetime of a urea plant is 40-50 years. Every urea reactor will finally operate close to the end of lifetime conditions of the protective layer meaning that at a certain moment a leak in the protective layer is nearly unavoidable.

WEEP HOLE MONITORING

Urea line-1 weep hole monitoring with lectrotek system (Pune). In Urea line-2 Masibus weep hole monitoring system is incorporated. The Micro genie scan is a microprocessor based scanner which has been specially designed for resistance input to monitor reactor leakage easy and very simple in installation low pricing equipment’s and easy in maintenance.

Table No. 4

DETAIL OF SHUT DOWN TIMING AND ACTIVITIES					
Sr. No.	Activities	Date	Duration	Time	Parallel Jobs
1	11 Reactor Feed Cut	28-11-2017	11.15	0	
2	H.P. Loop draining started		11.15 hrs. to 17.15 hrs	6	Weep hole cleaning with steam
3	L.S. Purging		17.15 hrs to 21.15 hrs	4	
4	H.W filling		21.15 hrs to 00.15 hrs	3	Instrument air connection provided with pressure regulator with P.G
5	H.W overflowing at the pressure 30 kg/cm^2	29-11-2017	00.15 hrs to 04.15 hrs	4	
6	H.W Draining		04.15 hrs to 12.15 hrs	8	Reactor man hole cover opening
7	Air cooling		12.15 hr to 18.15 hrs	6	Air sampling

8	Man entry permitted for Reactor trays man ways opening up to 34 Number weep hole.		18.15 hrs. to 21.15 hrs	3	PT-5 A/B opening for air purging
9	Leakage checking with Inst air and soap solution	30-11-2017	21.15 hrs to 01.15 hrs	4	No Leakage found
10	Leakage checking with Ammonia & phenolphthalein indicator solution		01.15 hrs to 08.15 hrs	7	No Leakage found
11	Finally welding by hit and trial on opposite of W.H. No34, based on DP pin points		08.15 hrs to 13.15 hrs	5	
12	Further checking with ammonia & phenolphthalein indicator solution		13.15 hrs to 23.15 hrs	10	No Leakage found
13	Man hole cover boxed up	01-12-2017	23.15 hrs to 03.15 hrs	4	Bottom PT-5 A/B boxed up
14	L.S Heating for H.P. loop		03.15 to 06.15 hrs	3	
15	K.W.Heating, up to 140-150^{0}C		06.15 hrs to 08.15 hrs	2	
16	Ammoniation		08.15 hrs to 11.15 hrs	3	
17	Reactor Feed In		11.15 Hrs.		
			Total Time	72	

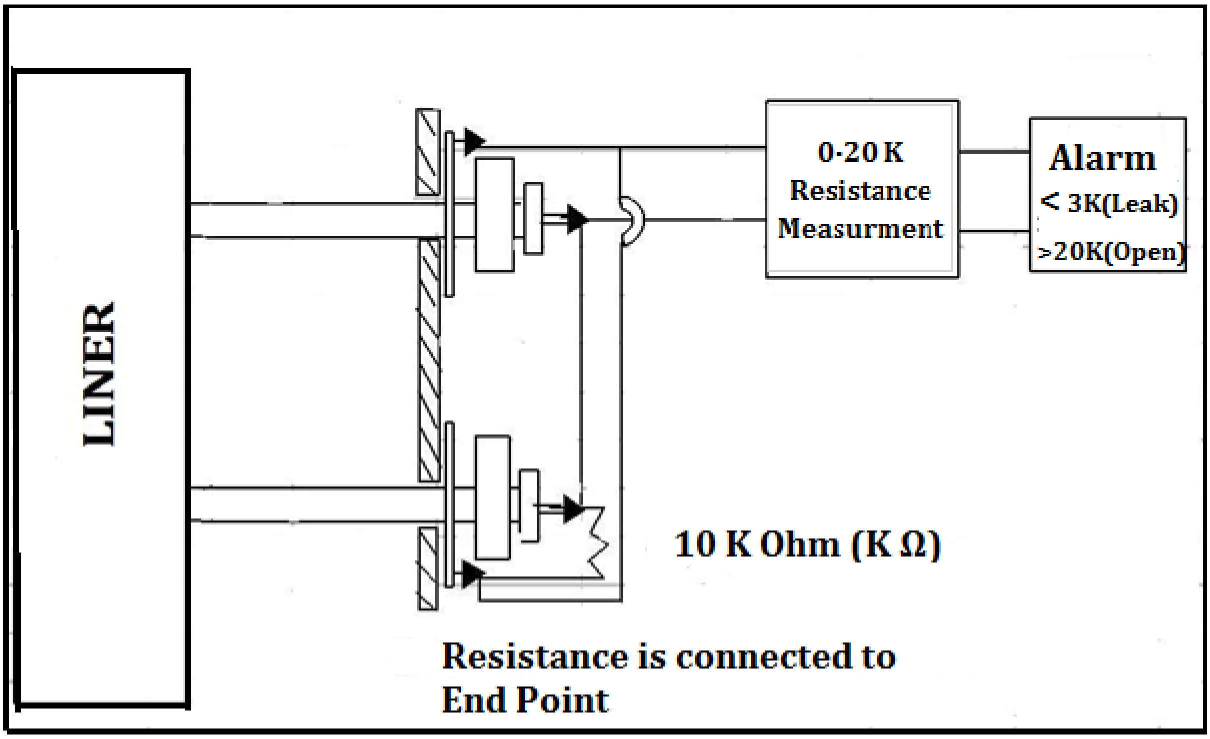

Fig. No. 2

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This programme containing non-volatile memory for setup data, 4 duals and 4 digit display and LED bar for information RS -232 –C serial port

The instrument is housed in a ½ DIN extruded Aluminum rack suitable for control room mounting. A small but highly functional Keyboard consisting of 4 membrane keys is provided as the as the instrument operator interface electrical and I/O temperature are instruments rear Via Screw type terminals. The electronics is located on modularly designed board for easy field trouble shooting and maintenance. All cards are plug in type and segregated by their function Viz –including power supply CPU, A/D display.

All the weep holes have to be checked by passing instrument air every week/fortnight for any leakage and blockage & the pressure of the testing medium should not be more than 0.5 kg/cm^2

SAFETY RISKS OF UREA HIGH PRESSURE EQUIPMENT

1. High pressures
2. High Temperatures
3. Various kinds of corrosion phenomena (inside and outside)
4. Crystallization risks.
5. Large volumes
6. Release of toxic ammonia in case of a leak

Integrity of carbon steel pressure bearing wall can be threatened by:

1. Carbamate corrosion due to damage of protective layer with (corrosion rate 1000 mm/year):
2. An early and reliable detection is a must.
3. Stress corrosion cracking behind loose liner when water and contaminants are present.
4. Stress corrosion cracking from outside when water and contaminants are present

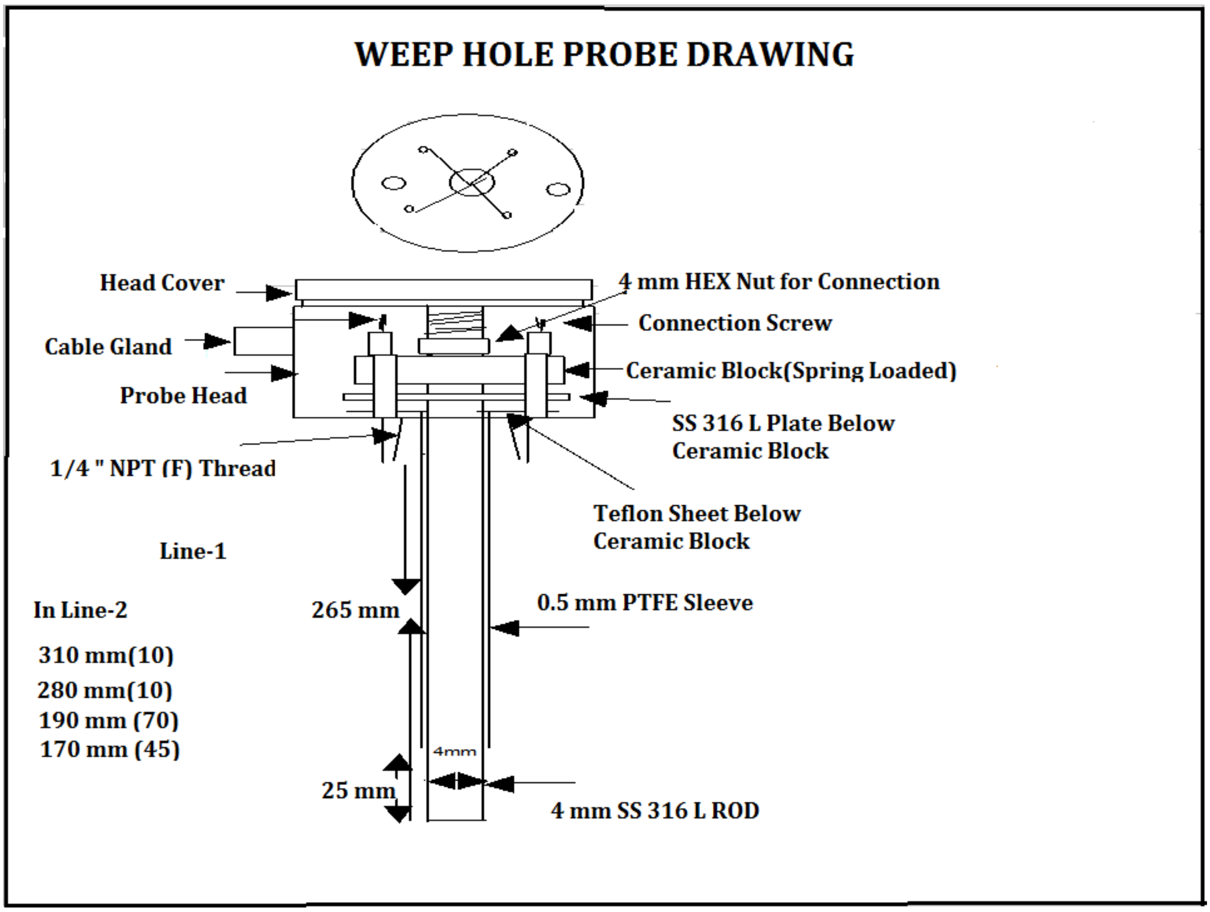

Fig No.-3

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Other methods of Leak detection

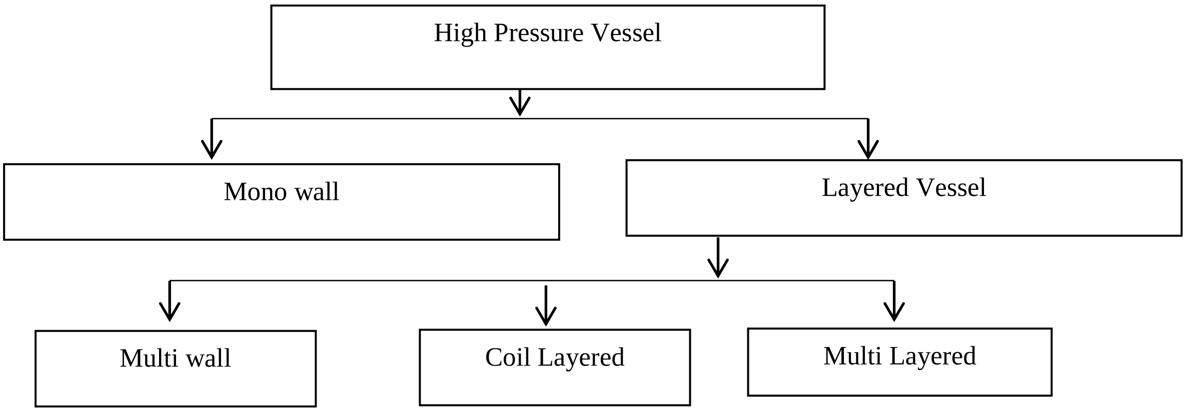
Number of methods are available in market as following.

1. Instrument air & soap solution
2. Ammonia with phenolphthalein indicator.
3. Helium leak detection.
4. Vacuum testing method.
5. Ultrasonic flaw detection
6. DP Test method.

PRESSURE VESSEL

Pressure vessels are leak proof containers which contain media under pressure and temperature May be of any shape milk bottles, shaving cream, tires, tanks, reactors etc. The term pressure vessel referred to those reservoirs or containers, which are subjected to internal or external pressures.For higher operating pressures and higher temperature, new technologies have been developed to handle the present day specialized requirements. Multilayer Pressure Vessels have extended the art of pressure vessel construction and presented the process designer with a reliable piece of equipment useful in a wide range of operating conditions for the problems generated by the urea processes.

Classification of Pressure Vessel



Multi wall Vessel	Coil Layer vessel	Multi-layer vessel
They are the proprietary construction of STRUTHERS WELLS CORPORATION U.S.A	Required thickness is calculated as per solid wall construction formulas subsequently no. of layers and their thickness is determined	Required thickness is calculated as per solid wall construction formulas subsequently no. of layers and their thickness is determined
Designed to various international codes	Unit cylinders consist of an Inner shells, steel hoop (3 – 6 mm thk) coiled around the inner shell in volute and split outer shell of 6-to 12 mm thickness	Inner shells of suitable material are used as per process requirement
Required thickness is calculated as per solid wall construction formulas subsequently no. of layers and their thickness is determined	Uninformative priestess can be obtained by coiling of hoop around inner shell	Then thin plates of high T.S. are wrapped on outside of inner shell to obtain required thk.
Cylindrical shells are rolled to desired Dia and welded (plates THK in the range of 1" to 21/2")	Vent holes are provided in all layered sections except for inner shell lining	Vent holes are provided in all layered sections except for inner shell lining.

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Cylindrical shells are consecutively shrink fitted over each other to obtain total wall thickness		Higher the operational pressure or pressure vessel diameter or length the more favourable the weight ratio between multilayered and monowall design.
Heating rate for shrink fitting is as per ASME SEC. VIII Cooling after shrink fitting is done in still air		

ADVANTAGES		
Multi wall Vessel	Coil Layer vessel	Multi-layer vessel
Layers of different material can be used as per process requirement.	Does not suffer from lack of uniformity of material.	Does not suffer from lack of uniformity of material.
Unlimited size of the vessel	Can be provided with innermost layer in cladding orcorrosion resistant material as per requirement.	Can be provided with innermost layer in cladding or corrosion resistant material as per requirement
Does not involve any full depth heavy longitudinal weld	Lighter in weight due to use of high T.S. steel plates. Vent holes are provided for leak detection in welds.	Lighter in weight due to use of high tensile strength steel plates.
Vent holes are provided for leak detection in welds	These are also profitable in the point of anti-hydrogen attack .Unlimited size of the vessel	No PWHT is required except in special cases.Vent holes are provided for leak detection in welds.

Normally uneven corrosion in reactor occurs due to poor feed rate and quality, N/C ratio; high inert; chloride and sulphides and also iron precipitated water. Lack of passivation. 316 L Urea Grade needs a continuous oxygen supply to assure a passive layer builds up. Sulphides cause stress cracking in stainless steel. Chlorides are somewhat corrosive to stainless steel. The combination is not good. It sounds like you might be getting a lot of impurities in there from somewhere. Very small quantities of these things (other than halides) are not likely to be very much of a problem, but you need to have an analysis made of the sludge you are getting to determine what all you have in there. Then it will be easier to judge how bad the situation is. With this kind of mix (if you are getting all of those things) you can get a lot of side reactions. If you have that many anions, you will need to get cautions from something. It is possible for it to find them in the stainless steel. Passivation air must not be less than 0.3% in compressor house in third stage. Following points to avoid corrosion/erosion.

1. Chloride in passivation air-seldom present in atmosphere.
2. N/C ratio should be proper.(In M/S. Saipem C/N ratio is 3.5 and in M/S. Stami is 2.8
3. Prevent iron precipitated water.
4. Check CO_2 purity - 99.3%, Hydrogen and Iron present in carbon dioxide.
5. Oil content in liquid ammonia feed, as few grades of oil contains sulphur.
6. Holding of solution in shut down must be less than 48 hrs. However M/S. Saipem recommended 72 hrs. Initially, but after some cases of corrosion/erosion was observed further recommended for 48 hrs.
7. Ammonia liquid feed in reactor must be more than 132.6 ^{0}C(critical temp. of ammonia)
8. During holding of solution liquid ammonia to be given to reactor for 10 minutes and during start up ammonia feed 10 minutes prior to carbon dioxide feed.
9. Pay attention to depletion of oxygen due to dead spots.

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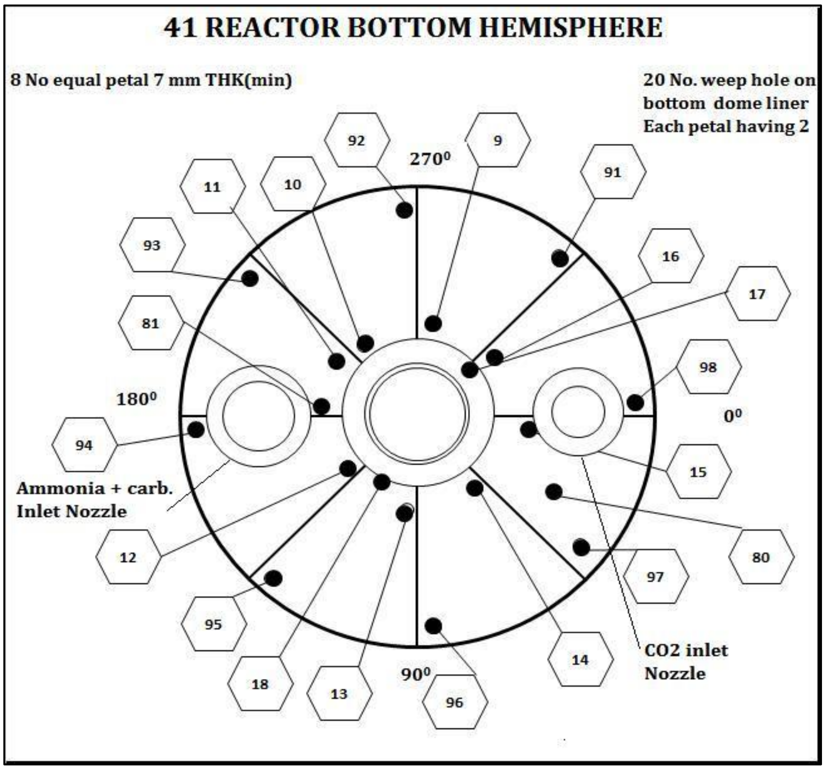

Fig. No. 4

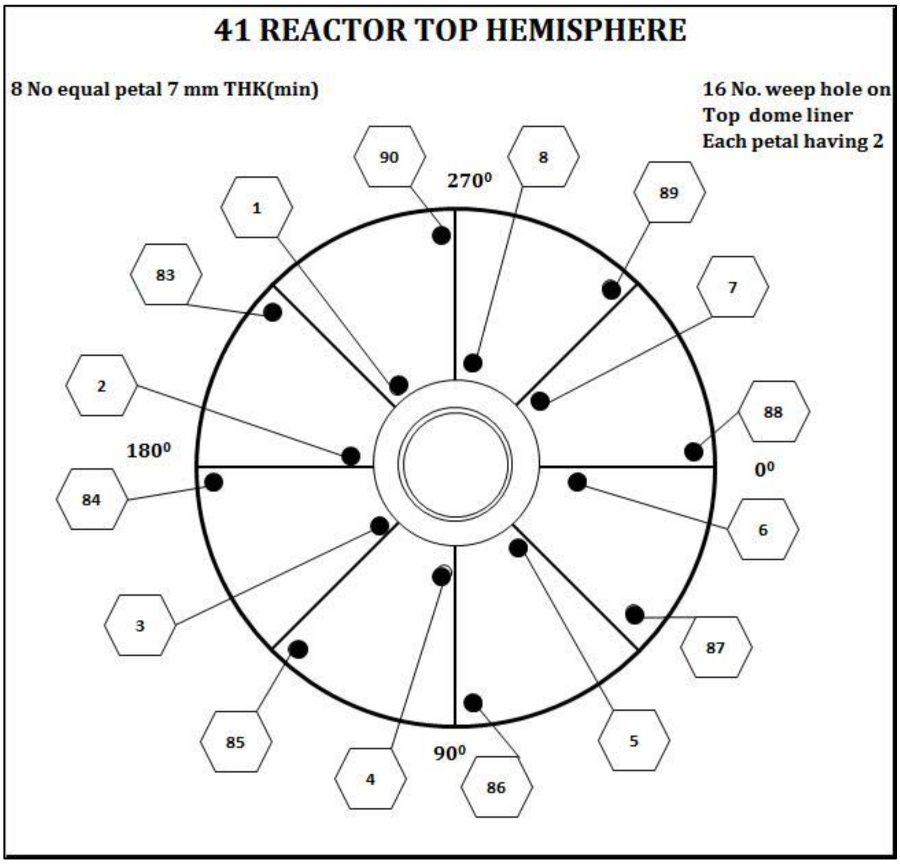

Fig. No. 5

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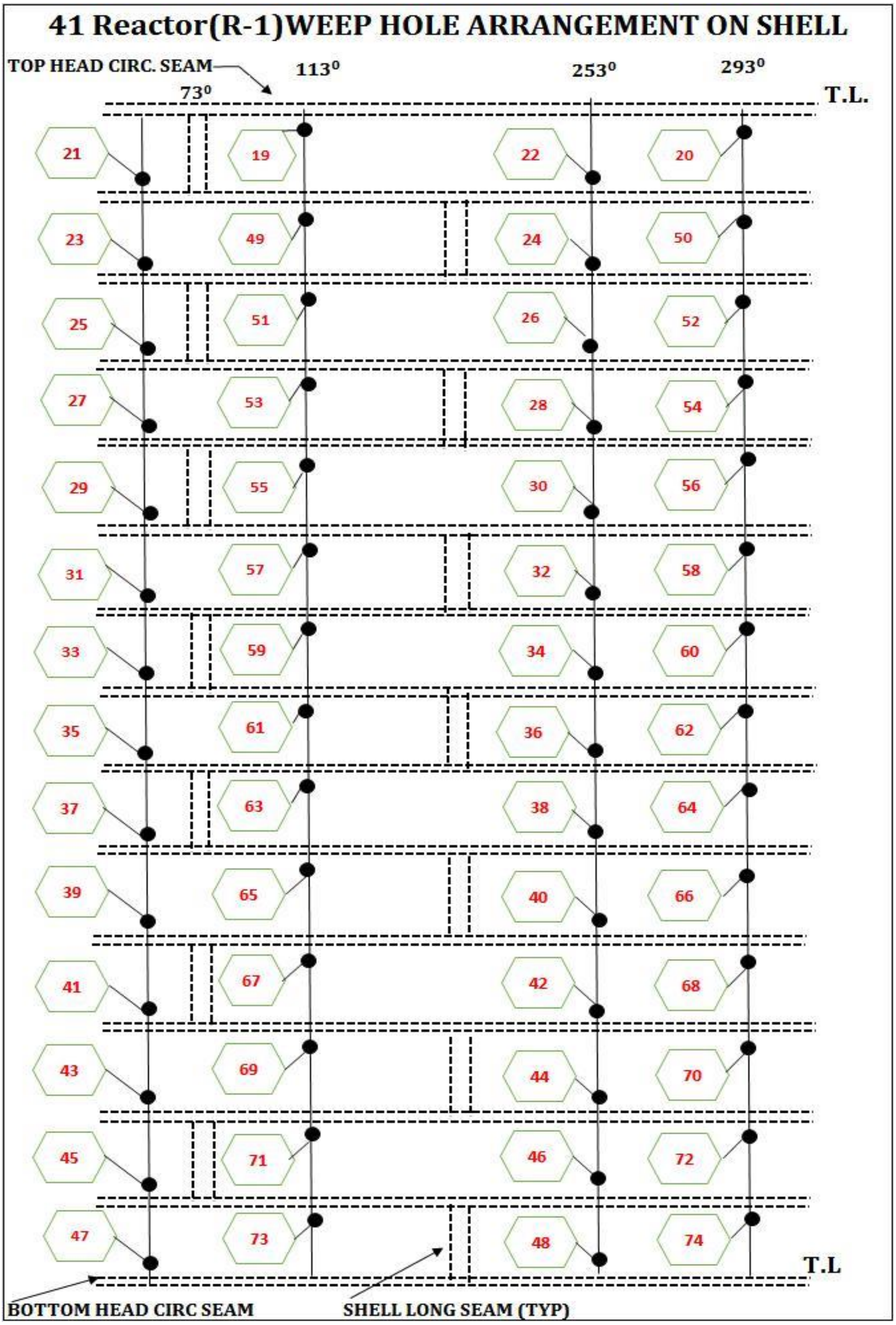

Fig. No. 6

CONCLUSION

My experience says that reactors always leak from the weld joints of liner initially. However, if the leak is not attended and carbamate comes in contact with shell. The damages are far beyond imagination. Weld joints are always more vulnerable to fail. The probability and consequence are combined to produce an estimation of risk. The results show that the lining leakage is the most severe risk and the SCC of layer shells risk is the second. Keep weep holes always open and inspect for leaks periodically. Urea reactors should be shut down immediately when a leakage is detected. Don't use vapour to detect the leakage of urea reactor vessel, in

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case it should be used, the vapour quality should be ensured. The risk of highly corrosive urea-carbamate solution leaking through these liners always exists in this equipment which might eventually damage the carbon steel walls. The places having notches like corners of support cleats or crevices in the weld joints are more prone for SCC.The reactor liner leakage is the serious phenomenon. As and when urea reactor liner leakage started shut down must be taken as soon as possible otherwise carbon steel corrosion rate is high. The subject of pressure vessel technology is central to industries as a whole. The balance of safety with a sound economic approach is a major consideration to the designer, manufacturer and operator. The legislation and standard must be such that these requirements are met.

LEGENDS

CS-carbon steel, TPD-ton per hour, MW-Megawatt. SS-stainless steel. SCC –stress corrosion cracking, DP-Dye penetrating Test. Circ.-Circumferences, Long.-longitudinal. HP-High Pressure, L.S -low pressure Steam, K.W-High Pressure flushing water. H.W-about Medium pressure flushing water.ID-inner dia. P.W.H.T.-Post weld heat treatment. T.S. -Tensile strength