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HDC MK II Series Testing

Procedures for Screening & Testing Product Performance

HDC MK II	**-**	**Calcium, Barium & Strontium scale dissolvers**
HDC MK II	**-**	**Mud Grade Barite, Calcium Carbonate mud Cake dissolver**
Pyrosol (Series)	**-**	**Calcium, Iron, Sulphide dissolver systems**
PentaFlow	**-**	**OBM/WBM Cake breaker, metal sequestrant And anti-emulsifier preflush**
K+HDC MK II 17	**-**	**High weight HDC MK II based cake penetrator for pipe release**

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Introduction

The development of the HDC Mk II product line has led to a number of laboratories developing differing test methods to determine the product's effectiveness. In line with this ongoing evolutionary work, is the defining of the most efficient methods to use, as well as clearly marking the limitations of lab testing.

The following document highlights the most efficient methods to test HDC Mk II or the Pyrosol Series dissolvers. That stated, the basic screening tests are standard oil field procedure.

> There are times we add 25% water in the lab tests to the HDC Mk II, if the scale is particularly pure, will react to saturation and this is highly dependent upon temperature. When the sample cools, e.g., for handling and during filtration – potassium sulphate can precipitate and plug the filtration equipment and give an erroneous solids recovery. The addition of some water prevents this from happening. In the field this is not an issue because the product remains at temperature and is squeezed away or returns hot but with additional water from the formation which also prevents sulphate salt precipitation. And as above in all cases LIBERALLY wash the filter paper. This is specific to lab tests only, not the field.

A secondary objective is to demonstrate the selected product's versatility in dissolving the gross solids components of the oil based muds normally used by the client system, including carbonate material as well as barite.

More refined testing consists of cake deposition or gross core invasion by drilling fluids and subsequent clean out trials using the selected **fluid in a flow loop on actual or synthetic cores.**

Note* Static testing in bottles or steel bombs are not an accurate or acceptable method of determining the effectiveness of these products in open hole. Field data from wells using HDC MK II have validated this methodology and imply that these lab tests give a nominal result – usually 70% to 80% of a field result in similar conditions.

Ref: Case history in Appendix 2

Scale Testing

For scale testing, the test is exactly the same as for barium sulphate except that we would:

a) Run both the neat product and the dilution used in the BaSO4 test, as real scale is rarely pure and in those cases neat product can be a lot better
b) Run at downhole temperature (in hot roll cells if necessary)

c) Run the test with scale lumps (because this avoids grinding and realising radioactive dust but also because the scale sometimes just disintegrates without fully dissolving as cementaceous materials dissolve)

2.0 Summary Laboratory Results – Screening

2.1 Screening of HDC Mk II for use – quick bench testing

The decision on which HDC Mk II variant to be used in the field is made by screening the products against the solids to be dissolved. These solids are usually mud barite and or calcium carbonate weighting materials. These solids can of course be scale material, such as barium scale, sulphide scale, carbonate scale or other.

Although the method below is used for screening, it accurately determines the "Dissolving Capacity" of the HDC Mk II for the target solid. The volumes used in the bottles is standardised at 80 mls. The conversion from the end results (solids dissolved) is by simply multiplying the dissolved solids by 12.5 to obtain grams per liter (1000 mls/80mls = 12.5).

2.2 Test Method 1 – Duran Bottle Dry Weighting Material Dissolving

2.1.1 Equipment Required:

Hot Roll Oven
Duran Bottles
Weight scale
Millipore Filtration unit
1 micron or 0.5 micron Millipore filtration paper

A) Screening

Testing of cake dissolvers is basically a screening process. Often, the barite or carbonate to be removed will not be analysed or identified as to purity. The identification of the exact mineralogy of the barite is not critical unless the barite is difficult to dissolve with one of the standardised HDC MK II variants.

Initial screening is essential to identify the correct product to use for the target cake matrix solids (or scale).

A full screening will involve: HDC Mk II

a) Weigh out between 5 and 10 grams of sample
b) Record weights
c) Label bottles (Duran Bottles) - record what liquid is in each and weight of sample
d) Pour 80 mls of each test HDC Mk II fluid into bottles
e) Place lid on bottles – tighten before rolling
f) Place samples in rolling oven @ 90 deg. C or estimated BHT and age/roll for 18 to 24 hours. ***Note** Static aging does not give an accurate result and should not be done!***

* If there is a specific target well with known BHT, use the BHT of that well as the oven temperature – time can be flexible during refined testing. In general, the higher the temperature, the higher the dissolving rate and less time it takes.

g) Remove bottles from oven and allow to cool

h) Shake bottles, and pour into Millipore filtration unit; the Millipore unit should use 1 micron or 0.5 micron filter papers
i) Use plain water to wash out bottles and filter this material as well
j) Filter until liquid is gone

Note: The solids remaining should *always* be washed with several volumes of water. This can be done during the filtration process to speed up the filtration rate. Failure to wash with water will not yield a true result.* As HDC Mk II reacts in an environment where more barite is available than the product's capacity to dissolve it, potassium sulphate which is fully soluble in water remains as an intermediate by-product. This is solubilised in the wash – and in the field has never been seen in returns which implies that this condition either does not occur down hole, or the potassium sulphate is solubilised (removed) in the back flow of the KCL brine pre-flush or brine material nominally bull-headed ahead of the treatment.

j) Place filter paper with solids on glass dish and place in oven to dry for 3 hours or more (over night is OK)
h) Weigh dried material from oven
l) Subtract remaining weight (take into account weight of filter paper) from starting weight. The difference gives you the amount dissolved. This needs to be reported in % of sample dissolved and as grams per liter of product dissolved. This is calculated as below:

Example:

1-Dry Weight of Barite = 7 grams
2-Add to 80 mls HDC Mk II in Duran Bottle
3-Hot Roll 24 hours
4-Filter on 0.5 micron paper
5-Wash filter with 200 mls water
6-Remove filter paper with remaining solids – place in oven and dry thoroughly
7-Weight dried filter paper

A) Weight After drying – Weight of Filter Paper = weight remaining barite (2 grams)
B) Weight of Original Sample (7 grams) – Weight remaining (2 grams) = Dissolved (5 grams)
C) Dissolved (5 grams) X 12.5 = 62.5 grams per liter or "Dissolving Capacity of HDC MK II"
D) % dissolved = 5/7 or 71%

2.3 Test Method 2 – Duran Bottle – Gross Cake "Paste" Dissolving

Tests Method Developed With Baroid

Testing for dissolving barite or carbonate in the field condition can be done in two ways. One way is to take the dry barite or carbonate powder and make a paste with the base oil or base fluid if its water based. The second is to build filter cakes using HPHT presses. The paste method is most accurate as the weight of all components is known. It is however necessary to keep in mind that in the field, 92 to 95% of all filter cake material is composed of barite or calcium carbonate material. The weight of the paste should reflect this.

1) Procedure for making and testing barite/carbonate paste

a) Have sample of unweighted OBM or unweighted water based fluid
b) Have sample of barite or carbonate to be tested – weigh out ± 10 to 12 grams barite or 12 - 15 grams calcium carbonate (if the cake has both – mix in proportions)

c) Into dry sample, add base fluid (OBM for example) to barite in small volumes until you have a thick paste. This is normally about 4 to 7 mls of base fluid
d) The weight of the paste when complete, should be approximately ± 14 to 16 grams for the barite sample, and about 20 grams for the carbonate sample. Mixed barite carbonate samples should be proportional.

2) The procedures followed from here on in, ***should duplicate*** the field operation

For OBM

A pre flush of solvent is usually run prior to spotting an HDC Mk II fluid in OBM (Power Pickle or PentaFlow). Note: PowerPickle can be replaced by Xylene or Operator preference solvent that is compatible with PentaFlow and/or HDC Mk II.

a) add paste to Duran bottle
b) add 80 mls PentaFlow/solvent mix. Heat to BHT (if practical), roll or agitate for 15 minutes to simulate flow. Allow settling until all solids are on the bottom, then decant. Do not decant any solids.
c) add 80 mls HDC Mk II to bottle, seal well and "Hot Roll" for 18 to 24 hours @ BHT (Bottom Hole Temperature) of well – say 110 deg.C
d) remove from oven, allow to cool; filter liquid
e) to bottle with solids in it, add water, shake and filter in same filter paper as in step d; add another 80 mls water to filter vessel and continue filtering until dry
f) place filter paper and remaining solids on glass in oven and dry for 3 hours or more (overnight is best)
g) weigh dry solids

The efficiency of the operation is judged by the % barite and total cake material dissolved and g/l as in the previous testing.

	Sample Weight (grams)	**Fluid Tested**
Start	16.5	HDC Mk II
End	0.95	Volume = 80 mls
Grams Dissolved	15.55	Grams/Liter = 30
% Dissolved	94.24%	

Calculated By: Start weight – End Weight = Grams Dissolved
Grams Dissolved / Start Weight = % Dissolved

Grams per liter is calculated by: Grams Dissolved X 12.5

2) procedure for testing HPHT cakes

This test was developed with SOWSCO & Shell Nigeria. Note: based on the results of the tests run below, ADDAX Nigeria ran the system to clean out an OBM and acid impacted drilled liner with zero production rates and returned the well to 4600 BFPD after a 48 hour operation. Case History available on request.

Testing filter cakes are the same as testing the paste. The cakes are built in the HPHT press. The cakes are carefully removed and weighed. The cakes are treated in the same manner as the paste before hot rolling, filtering and drying. This is very painstaking and

probably, not as accurate as material can be lost during transfers. It is however, as requested by Shell – a test that can be run quickly on the table top without having to use expensive high tech equipment.

Oil Based Mud and Oil Environment Dissolving Testing

The oil based mud as used by Baroid in Port Harcourt (XP07) was delivered to the lab. The mud was field grade, in average condition and contained a substantial volume of unidentified drill solids and carbonate material. The emulsion was tight as there was little fluid loss. It was very difficult to build cakes. Eventually, 500 psi differential, 250°F, and five hours were required to achieve a 2/32nds 10 to 12 gram quantity of cake.

Two types of test were run to simulate the field efficiency of the HDC Mk II.

Test 1 consisted of adding a pre determined weight of barite to the emulsified oil and making a thick paste. The paste was subsequently added to a beaker, and procedurally washed and soaked as per the field programme. *Aberdeen

Test 2 consisted of building the OBM cake from the field mud as received, weighing out a fixed amount into a beaker, and procedurally washing and soaking as per the field programme.

Test conditions and procedures were as follows:

1- Weigh out barite sample – 15.0 grams for screening – 10.28 grams for the OBM Cake
2- OBM cake only - Wash/soak dynamically in PowerPickle/PentaFlow 150 to 250 mls depending on flask size, at 90°C for 15 minutes then decant
3- Add HDC Mk II to Erlenmeyer flask – 300 mls in this test
4- Add OBM cake – as lifted from paper to flask of HDC Mk II
5- Note* weight of any loss material from cake on filter paper was noted and found to be less than 0.05 grams or negligible
6- Turn on magnetic stirrer and heating element until flask temperature is 90°C
7- Allow stirring to continue and flask heating for a minimum of 5 hours on this type of demonstration
8- Decant liquid, add 50% solution of HDC Mk II + water – re-heat @ 90° C and stir for additional 1 hour
9- Cool, filter contents, dry and weigh

Test Type	Volume HDC Mk II Used	Weight Sample Solids Before	Weight Sample After	% Dissolved	Grams/Liter Dissolving Capacity
SOWSCO – Barite Screening	300 mls	15 g	2.1 g	90.0%	46 g/l
SOWSCO – Field OBM Cake (Test 1)	300 mls	10.28 g (barite + oil + drill solids)	0.05 g	99.5 %	> 40 g/l
*** OBM Paste (Barite)**	100 mls	16.45 g	0.90 g	94.5%	44.4 g/l
*** Screening (Barite)**	80 mls	8.2 g	1.5 g	83.0%	67 g/l

Comments:

- Indicates paste test run in Aberdeen on samples run prior to testing at SOWSCO premises using hot roll equipment for screening & OBM testing.

The results of the laboratory test are very consistent. Re-runs of the field grade mud supplied to build the filter cakes for the tests did not take into account the composition of the drill solids (the drill solids were not analysed).

Appendix 1 - Barite Dissolving Photos

1.0 Screening

Before Hot Rolling

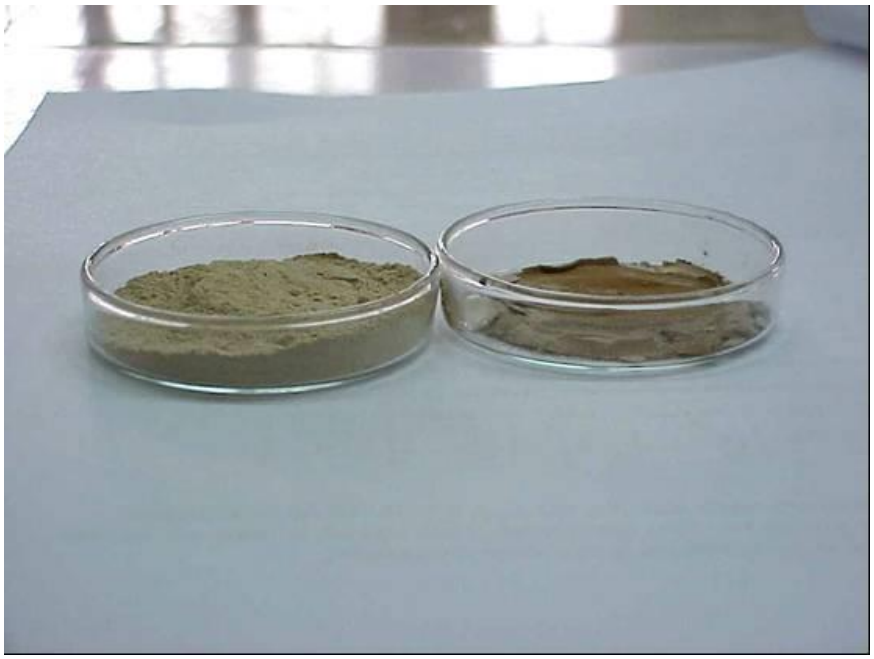
Bottle Testing HDC MK II Variants with standard weight so test barite 5 to 10 grams/80mls. Samples are hot rolled at BHT (90°C) for 18 hours

After Hot Rolling

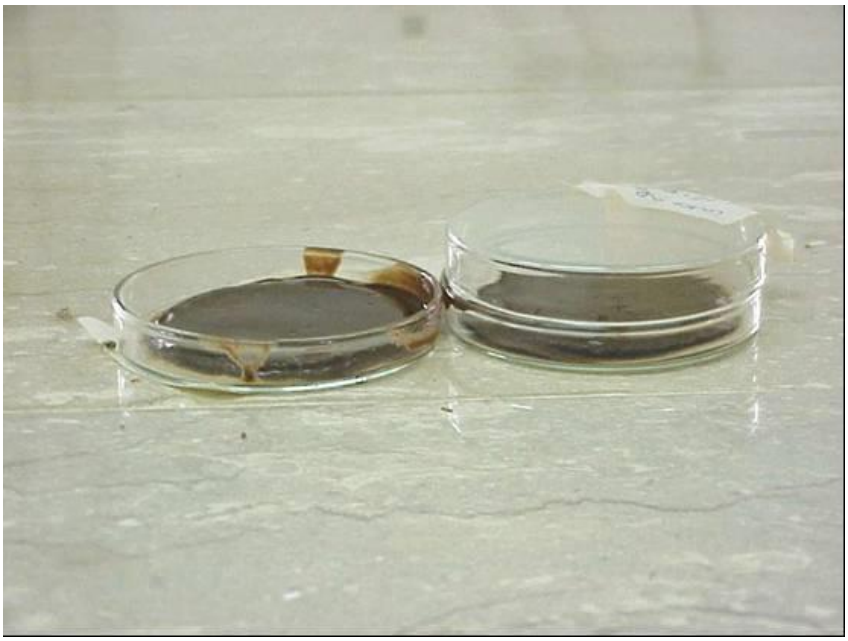
2.0 The reactants are cooled, filtered in 0.5 to 1 micron Millipore filters and washed with water 2 to 3 volumes

The residues are oven dried and the remains calculated

The photo below is of 15 grams barite (left) before testing. The dish on the right contains the residues after testing – 2.1 grams remaining in a barite of 95% purity.



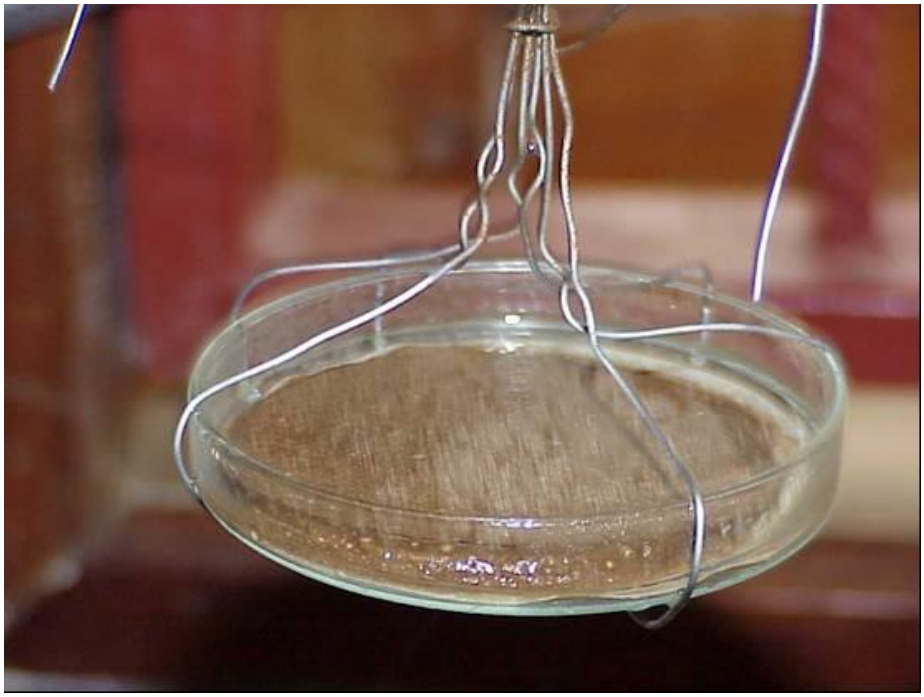
Below is an example of test run on HPHT OBM cakes. Whilst not the ideal method to determine actual performance, OBM cakes can serve as an indicator of dissolver performance.



Cake on the left is to be dissolved – the right kept for comparison.

The cake is washed in solvent solution – in this instance PowerPickle for a fixed time up to 15 minutes at 90°C

The cake is drained carefully so as not to lose solids which may have become disturbed.



The cake should be intact but the outer oil film removed.

Ideally the cake can be lifted from the filter paper prior to rolling. The cake is rolled for 18 hours @ 90°C, filtered, washed, dried and weighed.

The photo above is the remains of the cake – and filter paper. The starting weight (gross) of the cake was 11.2 grams. The final weight was 0.5 grams.

Appendix 2: Petronas Case History

Minerals Dissolved In Table II

Case History July 2002

HDC MK II - OBM Barite Dissolving
Petronas Carigali – Resak A10

Summary: Resak A10 was designed to produce 25 mmcfd, but only produced 10 mmcfd and falling. After pumping 13.0 ppg OBM to kill the well, 250 bbls of acid was pumped. Production dropped to below 1 mmcfd. Besides damage from acid, it was also believed that some of the barite from the OBM had settled and covered some of the perforations. In an attempt to recover some production, a year later almost 70 bbls of HDC MK II was bullheaded into the well over 26 hours resulting in production of 7 mmcfd.

Resak A10 is a dual string gas producer drilled by Carigali offshore Terengganu in 1999. The well was completed as a dual string completion to isolate a higher pressure reservoir at the bottom from intermediate production zones above.

Due to communication between the completions and lack of heavy brine, the well was killed and suspended in 13.0 ppg OBM. On re-entry, it was found that most of the perforations (in both zones) were partially buried under settled barite and OBM.

In the upper zone, coiled tubing could not be used to attempt a wash out, so the zone was acidized with 350 bbls of SWIK Halliburton formulation.

The zone was originally designed to produce 25 mmscfd. At the time of acidizing, the zone was producing between 10 and 7 mmscfd and falling. After acidizing, the production dropped to less than 4 million, dwindling to less than 1.5 mmscfd by June 2002 with over 200 bpd water.

In preliminary meetings and subsequent lab confirmation by Petronas, it was agreed that a cost effective trial using HDC MK II would entail attempting to recover some of the buried perforations, and reversing if possible some of the HCL damage. Due to the high volume of acid used (over 300 barrels) it was felt that attempting to reach the complete step out radius of the acid impact on the initial treatment stage was too expensive on an experimental basis.

The HDC MK II job design was a staged bullhead operation through a cement unit. The job design was based on staged displacements of HDC MK II Mark II over three hour intervals in a “dissolve” – “wash” – “dissolve” sequence to induce removal of barite from the lower perforations and flowing through them as the chemical depleted. The final stage consisted of displacing the entire volumes into the formation and static soaking for 12 hours. The entire operation was completed in 26 hours.

At the end of 26 hours, a partial nitrogen gas lift was used although the well began cleaning up naturally. Within 24 hours of lifting, the well was producing 4.5 mmscfd, going up to 6 mmscfd within 96 hours and over 7 mmscfd after five days, with 80 bpd water, and 5 cubic meters per day of condensate. The production has continued in excess of 7 mmscfd on a 19% choke through to the last tests held 45 days after the well was stimulated.

The condensate production results indicate a clear response from the previously buried perforations although the actual gas production source remains questionable.

The actual mineral species and weights dissolved as analyzed from the returns are tabulated in Table 1. (Note: The Barite used contained high volumes of Hematite)

Barium Sulphate	55.48	Kilograms
Hematite	600.13	Kilograms
CaSO4	22.65	Kilograms
CaCO3	418.96	Kilograms
Other	22.65	Kilograms
Total	1119.86	Kilograms

Based on the average specific gravity of the dissolved compounds, the mass of solids indicate that theoretically 63' of settled barite and mud could have been removed from the well. That stated, the figure of 63' is erroneous however, as it is impossible for the HDC MK II to have uniformly contacted sufficient surface area during the stage displacements to address the bulk solids in the 7". Basing the active HDC MK II on a 25% to 50% activity within the 7", over the period of each displacement, it is estimated with a high degree of confidence that between 19' and 30' of perforations in the lower liner was re-exposed

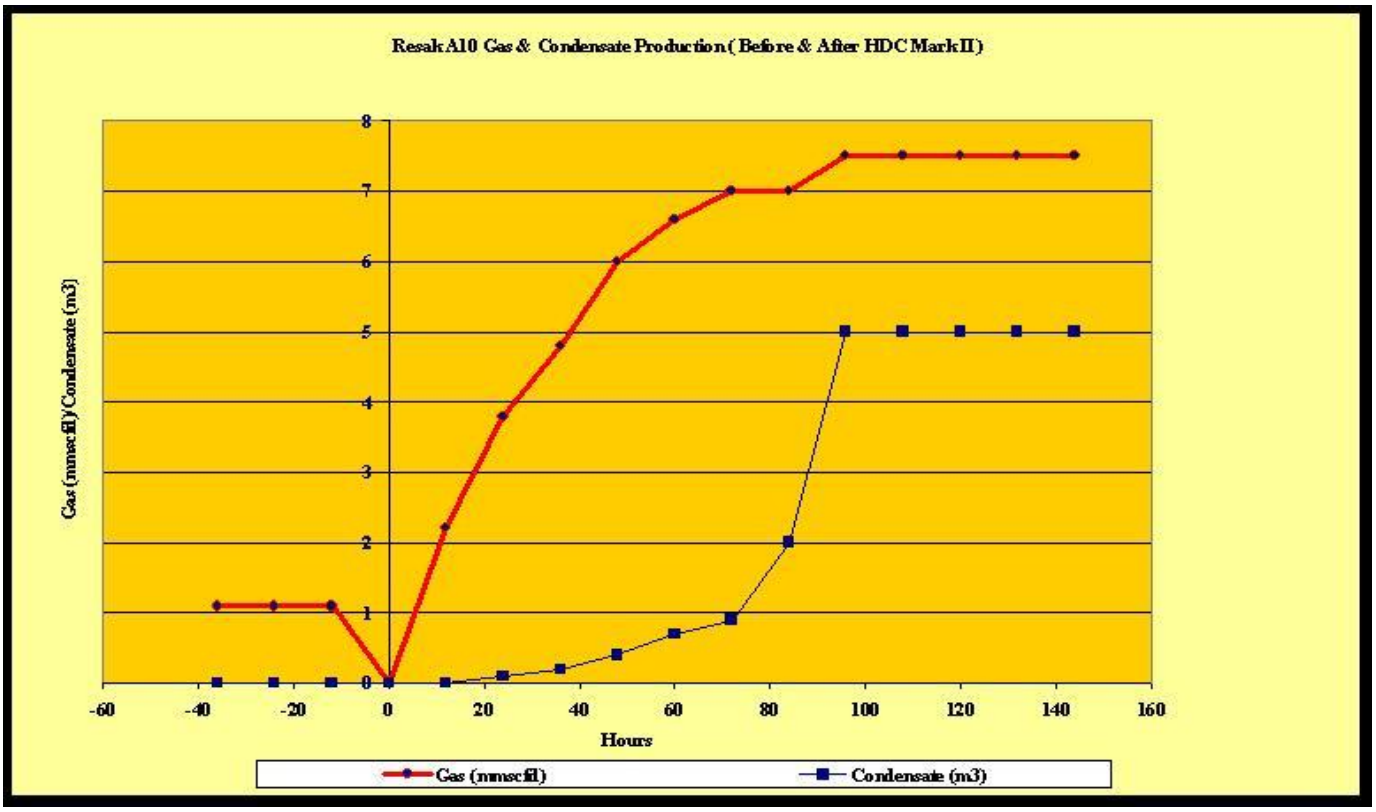
Table 2 reveals the actual analytical breakdown of the liquid volume flow back and dissolving rate of the HDC MK II in each volume.

Table 2: Separator Volumes, Weights Dissolved / HDC MK II Capacity

Sample	1	2	3	4	5
Volume bbls	40	40	160	40	40
Cum. Volume bbls	40	80	240	280	320
g/l dissolved minerals	24.24	22.34	32.50	10.83	4.82
Total Litres	6392	6392	25568	6392	6392
Total Grams	154,949.62	142,794.72	830,960.00	69,247.35	30,781.32
Total Kilograms	154.95	142.79	830.96	69.25	30.78

% SD27X In Sample	28.00%	24.65%	31.50%	10.65%	5.00%
Litres SD27X	1,789.76	1,575.63	8,053.92	680.75	319.60
Dissolved g/l	86.58	90.63	103.17	101.72	96.31

Chart 1: Production Gas Rates Before & After HDC MK II Treatment



Reference: Kasim Selamat - Senior Production Engineer kasimse@petronas.com.my

Other HDC MK II & HDC MK II Variant Reference Studies & Trials:

"HDC MK II – Enhanced Oil Recovery By Barite Removal – Ninian CCC61/39-B2"
References: Paul Beilby – CNR - Ranger Guildford, +01483-401401
David Brankling – PC david.brankling@octl.co.uk
Aberdeen, +01224-248113

"HDC MK II – Pipe Release Studies & Open Hole Obm Cake Removal Study: Bp Sunbury"
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Pete Wilson - BP Aberdeen wilsonp3@bp.com

"HDC MK II – Ferrout Barium Scale Removal & Iron Sulphide Dissolving – BHP – Liverpool Bay"
David Brankling – BHP Aberdeen david.brankling@octl.co.uk

"HDC MK II – OBM Filtercake Removal & Barite Dissolving – BHI – Aberdeen – BP Deveneck"
Bruce Ewen – BHI Aberdeen bruce.ewen@inteq.com

"HDC MK II- Barium Scale Removal – BP Aberdeen - BP Montrose A-15"
Harry Frampton – BP Sunbury framtoh@bp.com