Well Engineering & Technology Sdn. Bhd.
6.07, Level 6, Menara Hap Seng,
Jalan P. Ramlee,
50250 Kuala Lumpur, Malaysia.
Tel / Fax: (603) 2022 0803
Email : welltech@welltechengineering.com
Website: www.welltechengineering.com

HDC Mk II™ Testing

Procedures for Screening & Testing Product Performance

HDC Mk II™	**-**	**Barite and CaCO3 filtercake dissolver** **Calcium, Barium & Strontium scale dissolvers**
Pyrosol	**-**	**Calcium, Iron, Sulphide dissolver systems**
PentaFlow	**-**	**OBM/WBM Cake breaker, CaCO3 dissolver, de-emulsifier, clay inhibitor, water wetting agent**

Contents

Introduction

The development of **HDC Mk II™** 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 **Pyrosol** dissolvers. That stated, the basic screening tests are standard oil field procedure.

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.

Several conditions are required to be noted for the lab tests as below to avoid error and obtain optimal results:

Risk Highlights in Lab Testing

1. The solids remaining should *always* be washed liberally with water. This is done during the filtration process. Failure to liberally wash with water will not yield a true result as the **HDC Mk II™** reacts when 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 as it cools and can contaminate the results as it is retained by the filter paper and results in an erroneous result when weighing. This is solubilised in the wash. In the field this does not occur down hole since it does not cool to ambient subsurface.

2. **HDC Mk II™** needs free water in order for the chemical reaction to complete to saturation. In a lab situation particularly with **scale** samples, sometimes there is not enough free water to achieve maximum results. We do not normally experience this with OBM/SBM filter cakes but it should be discussed beforehand. Therefore, it is recommended that 25% water is added to the **HDC Mk II™** before **scale** lab testing. In the field this is not necessary as reservoirs are typically water wet and there is ample water to maximize performance.

3. Static testing in flasks or steel bombs are not an accurate or acceptable method of determining the effectiveness of these products in an open hole. Therefore, while in the container, stir the sample at the lowest setting, typically 10- 20 rpm's. 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. See Appendix 2.

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.**

For OBM / SBM Filtercake or Paste Dissolve Testing

Two types of tests can be run to simulate the field efficiency of the **HDC Mk II™**.

TEST 1 consists of building the SBM/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 2 consists of adding a pre-determined weight of barite to the emulsified oil and making a thick paste. The paste is subsequently added to a beaker, and procedurally washed and soaked as per the field programme.

TEST 1

Test conditions and procedures are as follows:

1. Weigh out 10.0 – 15.0 grams of OBM/SBM filter cake and put in an aging cell.
2. Add 300 mls of **PentaFlow** or **PowerPickle** (discuss prior to testing**)** to the aging cell. Heat to BHT, roll at minimum rpm (alternatively rotate 10 mins every hour) for 4 hours to simulate flow. Remove the cell from the oven and allow settling until all solids are on the bottom, then decant. Do not decant any solids.
3. Add 300 mls of **HDC Mk II™** to the same aging cell.
4. Seal well and put the cell into the HR oven for 20 hours at the well BHT of the target zone.
5. Rotate the cells at minimum rpm for 10 minutes every hour. This will replicate the pump and soak technique used in the field.
6. Remove from the oven, allow to cool. With the solids settled pour out most of the liquid **HDC Mk II™**, and then refill with water, then stir or shake for a few minutes. and filter, filter contents
7. Then decant to a filter paper and liberally wash again with water, dry, and weigh.

The soak times above generally reflect the recommended soak times in the field.

Test Type	Volume HDC MK II™	Weight Sample 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)	.05 g	99.5 %	> 40 g/l
WFI – OBM Paste (Barite)	100 mls	16.45 g	.90 g	94.5%	44.4 g/l
WFI Screening (Barite)	80 mls	8.2 g	1.5 g	83.0%	67 g/l

Note: In the tests above no PentaFlow or PowerPickle were first used and in some tests, smaller volumes of HDC Mk II were used.

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).

TEST 1 with a WBM

The procedures are almost identical but the **PentaFlow** or **PowerPickle** can be considered optional unless $CaCO3$ is used in the recipe. PentaFlow with a pH of +/- 2.0 will dissolve $CaCO3$. In the lab, they serve to dissolve the oil film around the particles. In the field, even if the well has been drilled with an WBM, either will still be used as a 1st stage simply because they provide other benefits not see in the lab such as water-wetting, dissolution of hydrocarbon residues, dissolving any emulsion blockages, dissolving other minerals in the reservoir, and de-hydration or inhibition of clays, etc.

TEST 2

A pre flush of **PowerPickle** or **PentaFlow** is usually run prior to spotting an **HDC Mk II™** fluid in OBM.

a) add paste to Duran bottle
b) add 80 ml **PentaFlow or PowerPickle**. 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 ml **HDC Mk II™** to bottle, seal well and hot roll in the oven for 18 to 24 hours at the BHT (Bottom Hole Temperature) of the well.
d) remove from the 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.

Scale Testing

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

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)

Test Method 1 – Hot Plate Magnetic Stirrer Dissolving

Equipment Required:

Oven
Thermometer
Hot Plate
Magnetic stirrer
Beaker
Weight scale
Millipore Filtration unit
1 micron or 0.5-micron Millipore filtration paper

a) Dry the filter paper at temperature of 105ºC for 1 hour.
b) Allow cool in desiccator, then weigh the filter paper, record (W2).
c) Weigh the empty 300 ml beaker glass, record (W0).
d) With the spoon; add and weight the SCALE sample of 5-10 grams sample into the beaker, and record (W1).
e) Transfer 200 ml of the dissolver into the test bottle. (For **HDC Mk II™**, use 150 ml and add 50 ml water for 25% dilution).
f) Place the thermometer in the beaker. Cover the bottle with a cap to minimize evaporation. Turn on the stirrer with a minimum rotation (typically 20 rpm).
g) Heat the mixture to the desired temperature (either target temperature if less than 100°C or if not known, 90°C) and maintain for 4 hours.
h) While sitting, stir the sample for 5 minutes at the lowest setting, typically 20 rpm’s, every hour. This to replicate the pump and soak procedure in the field.

i) After completing 4 hours, add 800 ml of distilled hot water at the temperature that the lab testing was run into the sample solution. Mix well the sample mixture.
j) Filter the residue, using a filter paper with a pore size of 2 – 3 um. Liberally wash the filtered residue with hot distilled water, again at the temperature that lab testing was run. As the sample cools in this process, sometimes precipitation can occur depending on the dissolver and sample, so in all cases LIBERALLY wash the filter paper. This is specific to lab tests only, not the field.
k) Place the filter paper with solids on a glass dish and place in an oven to dry at 105^{o}C for 24 hours. Weigh the dry residue, record (W3).
l) Calculate the loss (%).

Test Method 2 – Duran Bottle Dry Weighing Material Dissolving

The volumes used in the bottles is standardised at 80 ml. The conversion from the end results (solids dissolved) is by simply multiplying the dissolved solids by 12.5 to obtain grams per litre (1000 ml/80ml = 12.5).

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 at 90°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
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 ml **HDC MK II™** in Duran Bottle
3-Hot Roll 24 hours
4-Filter on 0.5-micron paper
5-Wash filter liberally with 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%

Test Method 3 – Duran Bottle – Gross Cake “Paste” Dissolving

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

Appendix 1 - Barite Dissolving Photos

1.0 Screening

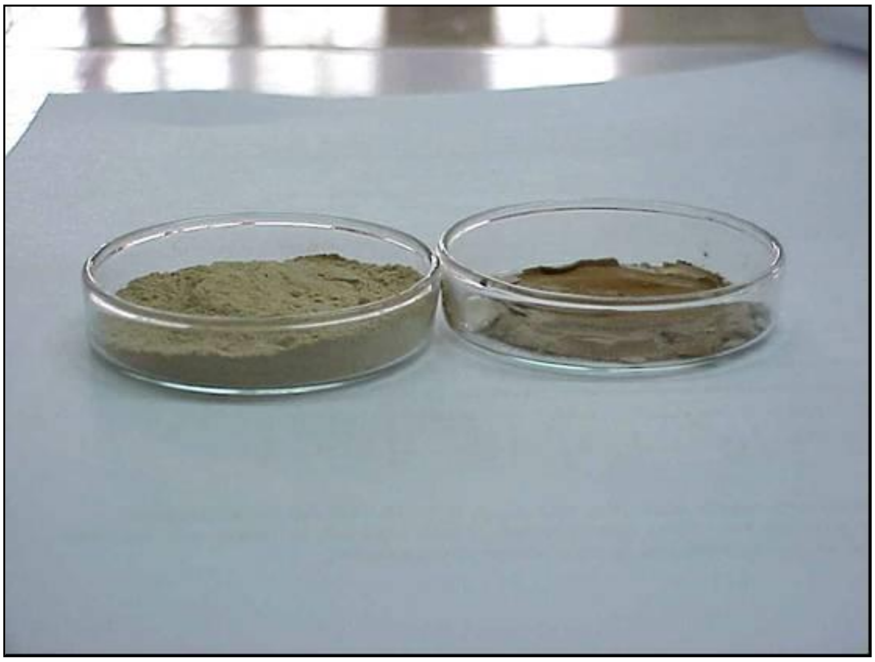
Before Hot Rolling
Bottle Testing HDC MK II™ Variants with standard weight so test barite 5 to 10 grams/80mls. Samples are hot rolled at BHT (90°C) for 18 hours

After Hot Rolling

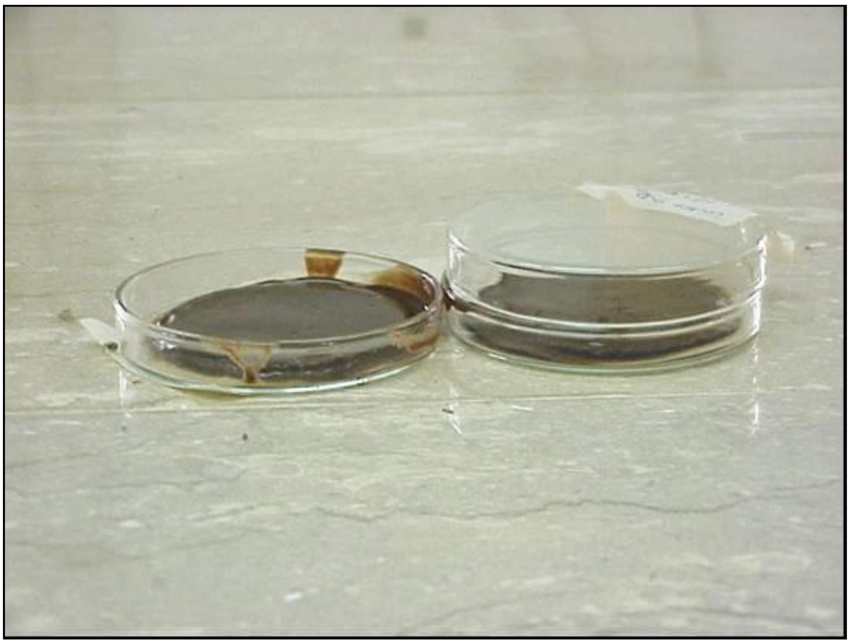
2.0 The reactants are cooled, filtered in 0.5 to 1 micron Millipore filters and washed with water 2 to 3 volumes

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 at 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

WellTech
Engineering & Technology

Well Engineering & Technology Sdn. Bhd.
6.07, Level 6, Menara Hap Seng,
Jalan P. Ramlee,
50250 Kuala Lumpur, Malaysia.
Tel / Fax: (603) 2022 0803
Email : welltech@welltechengineering.com
Website: www.welltechengineering.com

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, 300 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 19 mmcfd and consequently choked back to 7 mmcfd for production.*

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™** 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 19 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 indicates that theoretically 63' of settled barite and mud could have been removed from the well. That stated, the figure of 63' may be 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**

% HDC Mk II In Sample	**28.00%**	**24.65%**	**31.50%**	**10.65%**	**5.00%**
Litres HDC Mk II	**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**