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EXPLORATION & PRODUCTION DIVISION

PEB/ 38/81

NOVEMBER 1981

ULA WELL 7/12-6

SAMPLING AND GAS ANALYSIS

by

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1. INTRODUCTION

As well 7/12-6 was to be the final appraisal well for the Ula field, the drill stem tests would present the last opportunity of obtaining samples for any studies and analyses required during the design work for the development phase of the field. An extensive sampling programme was therefore planned for DST 2. At the same time the acid gas concentration would be checked on site using detector tubes together with observation of the procedures used by the service company for taking and handling the pressure samples. This report formally presents the results of the on site measurements and observations together with the component analysis of the separator gas samples.

SUMMARY

This report records the results of analyses of separator gas sampled from ULA Well 7/12-6 during DST 2, together with observation on the pressure sampling procedures used by service company personnel during the test.

CONTENTS

	<u>Page No.</u>
1. INTRODUCTION	1
2. ON SITE GAS ANALYSES	2
3. PRESSURE SAMPLING	3
4. SATURATION PRESSURE MEASUREMENT	4
5. ANALYSES OF SEPARATOR GAS SAMPLES	5
6. CONCLUSIONS	6

2. ON SITE GAS ANALYSES

Analyses of the separator gas stream were made on site using Siebetec detector tubes. These results have previously been reported and the values given by the tubes are given in Table 1. A box from each batch of tubes was selected and calibrated using gas mixtures of known concentrations. This resulted in calculation factors for the H₂S and methyl mercaptan tubes of 1.2 and 0.83 respectively and these factors should be applied to the values in Table 1. A summary of the calibrations is shown in Table 2 for reference.

In general, the changes in measured concentrations vary as might be expected, the H₂S starting low and finally levelling off to a corrected figure of 3.6 ppm, with the CO₂ tending to decrease at the higher separator pressures and finally settling down to just over 3% by volume at the stable separator pressure of 625 psig. At no time during the test was methyl mercaptan detected, the lower limit of detection being of the order of 0.2 ppm.

3. PRESSURE SAMPLING

In general, the procedures used by the service company personnel for taking samples were good and the only minor criticism being the length of some of the sample lines. Sufficient pressure vessels of the appropriate types were on site well before the test even though a more than expected number were used for samples from DST I a few days earlier. The four 'conditioned' 20 litre Gerzat vessels, required for analysis and total sulphur determination of the separator gas, arrived on site containing a small proportion of H_2S in nitrogen. However, the service personnel had only a vague idea of how to proceed and the reason for such conditioning. On instruction these vessels were emptied and air blown through them to remove all traces of H_2S . This was checked using gas detector tubes and when no H_2S could be detected the vessels were evacuated prior to sampling.

The pressure liquid samples taken at the well-head for reservoir fluid, or from the separator, were sampled by downward displacement of either mercury or water, the latter being used only for two 20l samples of separator oil. At all times the sample lines were bled sufficiently to ensure a representative liquid entered the sample vessel. The volume of the displaced liquid was measured and sufficient ullage for safe shipment was obtained by drawing a further quantity of mercury or water from each sample vessel. The rate of displacement was such that the liquid in the sample vessel should have remained in the single phase region.

Gas was sampled directly into evacuated vessels and again bleeding the line and sampling time was considered sufficient to obtain a good sample. However, the sample line was quite long and it would have been preferable to have a shorter line with a narrower bore. This would have increased the probability of condensed liquid being swept into the sample cylinder with its associated gas resulting in a more representative sample.

4. SATURATION PRESSURE DETERMINATION

Field saturation pressure determinations were made on the well head samples by withdrawing discreet quantities of mercury and noting the change in pressure of the sample. The sample was not shaken or stirred to obtain equilibrium and this was assumed to have been achieved when no further change in pressure occurred. The gauge used for measuring the pressure (a 10" Heise) although a quality product was in poor condition, there being no back cover. There was some tarnishing of the movement which undoubtedly contributed to the gauge appearing to be 'sticky'. We have found in the laboratory that reservoir oil and mercury can often form a stable emulsion which, if present in narrow bore tubing similar to that used to connect the gauge and sample vessel to the mercury pump, can cause partial blocking resulting in an apparent 'sticky' gauge. Either or both these factors contributed to the difficulty in obtaining and the reliability of the results obtained.

For more reliable saturation pressure measurements to be made in the field it is suggested the following procedure be used.

The mercury pump and all lines to the sample vessel and gauge should be filled with clean mercury, the gauge movement proved to be free over its range and the gauge calibrated at the well site using a dead weight tester. This calibration should preferably be witnessed and included in the sampling report. After a sample vessel has been filled, a further quantity of mercury should be run out say 15-25 ml allowing the sample to become 2 phase. The sample vessel should then be connected to the mercury pump and the line bled in the usual way. Discreet volumes of mercury should then be added to the vessel using the mercury pump and after each addition the vessel should be stirred by inverting it several times. Equilibrium will have been achieved when further stirring produces no change in pressure. Pressure against mercury injected may then be plotted to give the saturation pressure. The temperature of the sample at the time of measurement is obtained by strapping a thermometer to the sample vessel during the saturation pressure determination in the usual way. The vessel is then disconnected and mercury run out to provide ullage for shipment.

The above method ensures the flow of mercury is always into the sample vessel thus preventing any oil/mercury emulsion from entering the lines to cause partial blocking which would result in an apparent 'sticky' gauge.

5. ANALYSES OF SEPARATOR GAS SAMPLES

The bottles containing the samples of separator gas were heated for 24 hours before samples were taken for component analysis by chromatography. Results of these analyses are given in tables 3 and 4 and are presented on an 'air free' basis, i.e. any oxygen found to be present is assumed to have come from the sub-atmospheric sample taken from the pressure sample and transferred to the chromatograph, such oxygen together with the appropriate quantity of nitrogen is then subtracted from the analysis. The density of each sample at 760mm Hg and 60°F is also given and was calculated from the analysis. Sulphur compound determinations were made on the gas from bottle no. A4508 resulting in no measurable quantities being detected. The vessel was not heated before gas was sampled for these determinations. A different approach was therefore required for the other samples. For safety considerations, we were unable to connect the 20 litre Gerzat sample bottles to the total sulphur apparatus and there was no direct means of determining the weight of gas used. It was decided to adopt the following procedure.

The 20 litre sample bottles were heated for at least 24 hours to re-evaporate any condensed liquid and gas was taken sampled into 550 ml aluminium bottles. These were then weighed and heated by immersion in hot water before the entire contents were used for a total sulphur determination. The empty bottle was then reweighed and the weight of gas determined by difference.

This procedure was used to give the results, shown in Tables 3 and 4 for the remaining samples and show values of between 1.9 and less than 1 ppm weight for the total sulphur content. At these levels it would be almost impossible to determine H_2S or mercaptan concentrations and so these are not quoted. The revised procedure was also used on the original sample, contained in bottle A4508, giving a figure of 5 ppm weight. The earlier sulphur compound analysis carried out on this sample and reduced the pressure when heated to less than half, about 280 psig, of the original pressure. This much higher figure indicates most of the sulphur compounds are associated with the condensed liquids and were concentrated by the earlier sulphur compound determinations using gas from a cold sample bottle.

6. CONCLUSIONS

The field measured value for CO₂ of about 3.1% by volume is in reasonable agreement with the laboratory figure of about 2.7% mol. However, the figure for total sulphur obtained in the laboratory when expressed as H₂S is somewhat lower than the correct field measured values and points to some reaction with the vessel walls, even though it was conditioned prior to sampling. As might be expected from the H₂S values, mercaptans were not detected at any time.

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TABLE 1

ANALYSES OF GASES USING DETECTOR TUBES

DATE	TIME	SAMPLE POINT	SEPARATOR PRESSURE (GAS LINE)	H ₂ S ppm VOL	CO ₂ %VOL	MERCAPTON ppm VOL
15/7	2120	RIG FLOOR ATMOS.	-	N.D.		
	2130	RIG FLOOR ATMOS.	-	N.D.		
	2240	SEPARATOR GAS	~400		3.1	
	2245	"				N.D.
	2250	"		1.0		
	2350	"		0.8		
16/7	0001					N.D.
	0010		700		3.0	
	0015		700			N.D.
	0040		975		2.7	
	0050		975	0.6		
	0325		625	0.5		
	0330				3.2	
	0340					N.D.
	0730		625		3.2	
	0750					N.D.
	0800		625	3.0		
	0830				2.6	
	0840					N.D.
	1005		625		3.1	
	1015					N.D.
1020				3.0		
1030				3.0		

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WELL: 7/12-6

TABLE 2

CALIBRATION OF SIEBETEC DETECTOR TUBES

SIEBETEC DETECTOR TUBES - CARBON DIOXIDE 2L BATCH NO. QC90947

TUBE READING % VOL	CONCENTRATION CO ₂ BY GAS CHROMATOGRAPHY % VOL
0.7	0.7
0.9	0.9
1.25	1.24
2.70	2.60

GREATEST ERROR +3.8% OF AMOUNT AT 2.7% VOL LEVEL
THIS IS NOT SIGNIFICANT - NO CORRECTION TO BE APPLIED

SIEBETEC DETECTOR TUBES - HYDROGEN SULPHIDE 4LL BATCH NO. QC01116

THE TUBES WERE CALIBRATED AGAINST A STANDARD ATMOSPHERE OF H₂
OVER A RANGE 4-60 PPM. THEIR RESPONSE WAS LOW (~20%) BUT
WITHIN SPECIFICATION (±25%).

TO OBTAIN TRUE CONCENTRATION MULTIPLY TUBE READING BY 1.20

SIEBETEC DETECTOR TUBES - METHYL MERCAPTAN 71 BATCH NO. QC00722

THE TUBES RESPONSE WERE ~20% HIGH WHEN CALIBRATED AGAINST
KNOWN ATMOSPHERES OF MERCAPTAN IN THE 0.5 - 5 PPM RANGE.

TO OBTAIN TRUE CONCENTRATION MULTIPLY TUBE READING BY 0.83

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FIELD: ULA
WELL: 7/12-6 DST 2

TABLE 3

ANALYSIS OF SEPARATOR GAS

BOTTLE NO.	A4508	A7343
COMPONENT	%MOL	%MOL
CO ₂	2.70	2.67
N ₂	4.56	5.13
C ₁	66.63	67.05
C ₂	13.21	13.25
C ₃	7.25	7.36
1C ₄	.97	.91
nC ₄	2.38	2.16
1C ₅	.52	.43
nC ₅	.55	.46
1C ₆	.31	.19
nC ₆	.14	.10
C ₇	.25	.13
C ₈	.11	.06
C ₉	.06	.04
C ₁₀	.03	.02
BENZINE	.03	.02
TOLUENE	.03	.02
	<u>100.00</u>	<u>100.00</u>

DENSITY (CALCULATED)	1.020 Kg/m ³	.994 Kg/m ³
TOTAL SULPHUR ppm/w	5.0*	1.9 Repeat 1.2 Determinations

* THIS FIGURE WAS OBTAINED FROM A LOW PRESSURE SAMPLE SEE TEXT.

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FIELD: ULA
WELL: 7/12-6 DST 2

TABLE 4

ANALYSIS OF SEPARATOR GAS

BOTTLE NO.	A4867	A11275
COMPONENT	%MOL	%MOL
CO ₂	2.69	2.75
N ₂	6.83	4.61
C ₁	65.36	67.20
C ₂	13.04	13.32
C ₃	7.32	7.50
1C ₄	.91	.92
nC ₄	2.17	2.17
1C ₅	.43	.42
nC ₅	.45	.43
1C ₆	.24	.22
nC ₆	.13	.11
C ₇	.19	.14
C ₈	.09	.07
C ₉	.06	.04
C ₁₀	.03	.02
BENZINE	.04	.06
TOLUENE	.02	.02
	<u>100.00</u>	<u>100.00</u>
DENSITY (CALCULATED)	1.010 Kg/m ³	.998 Kg/m ³
TOTAL SULPHUR ppm/W	1.2	>1.0
		>1.0