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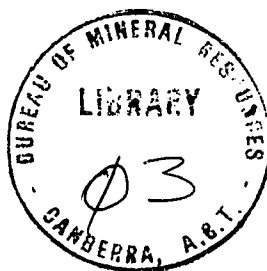
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DEPARTMENT OF NATIONAL DEVELOPMENT

BUREAU OF MINERAL RESOURCES, GEOLOGY AND GEOPHYSICS

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NOTES ON SOME PHOSPHATE TEST PROCEDURES

by
J. Barrie

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INTRODUCTION

A common reagent used to test for the presence of phosphate in rock or soil material is acid-ammonium molybdate solution. When applied to the test material the acid may dissolve any phosphate present and the ammonium molybdate reacts with the ensuing solute to form a yellow precipitate of phospho-ammonium molybdate.

This record sets forth some notes on the use of this reagent, suggested procedures, and significance of the results.

The reagent is sensitive to phosphate contents ranging from about 1000 parts per million (0.1 percent P_2O_5) upwards and its behaviour may be quite erratic. Thus it cannot be considered an ideal reagent and essentially it gives only a "yes-no" answer for the presence of phosphate. However, its use reduces by approximately half the material requiring further, more quantitative, testing. The "Phosphate Log" of a continuous series of samples has promise of being a useful parameter in general stratigraphic and paleogeographic studies.

THE REAGENT

Formula:

Dissolve 50 g of ammonium molybdate in a mixture of 70 ml of concentrated ammonia (S.G.O. 88) and 140 ml of water.

When all the ammonium molybdate is dissolved add this solution slowly with constant stirring, to a mixture of 250 ml of concentrated nitric acid and 500 ml of water. Stand for 24 hours before using; and decant from any precipitate if necessary.

The made up reagent is usually good for two weeks, but may last only a week (hot climates) or in cool dark conditions it may last up to one month. A positive sign of deterioration is the development of a yellow precipitate in the storage bottle.

If the reagent is required for a prolonged time without access to facilities for making up fresh batches then the solution may be kept in two parts - acid, and ammonium molybdate. For use portions of the two solutions may be measured volumetrically from stock. It is recommended that new mixtures be made up each week.

D. A. Haldane (Senior Chemist, B.M.R.) has advised that:

1. The reagent must be formulated in order to obtain reliable results
2. The acid concentration must be correct else if too acid - molybdic acid will precipitate; if too weak - no reaction.
3. An excess of ammonium ion is required to hasten the precipitation of phospho-ammonium molybdate. The excess of ammonium ion must be at least 5 times that of the ammonium ion in the ammonium molybdate. To obtain this, liquid ammonia is incorporated in the reagent recipe.

Also precipitation depends on only vitric acid being present (the precipitate is more soluble in hydrochloric or sulphuric acids) and the amount of ammonium vitrate must be between 5 and 15 percent of the made solution (> 15% increases the solubility of the precipitate).

MISCELLANEOUS SAMPLES

For testing miscellaneous material such as rock in hand specimen, crushed or unconsolidated material, and soil, a fragment or portion of the material should be placed on a watch glass or palette and immersed in reagent.

The observer should wait at least a minute before noting the result. In the field it is commonly more convenient to place the test material on a piece of plastic such as a sample bag and discard after use. In the absence of such inert containers leaves of trees or shrubs may be used provided that the species has first been checked to give a negative result with reagent.

The reagent may be applied directly to outcrop but the reagent must lie on the rock and not soak into the rock. The observer is especially cautioned against the possibility of a surface phosphatic film such as phosphatic lime, manganiferous or ferruginous stains or skins, as well as phosphatic dust and ash from burnt vegetation. Preferably the rock should be broken and a fresh face tested.

Due to the irregular behaviour of the reagent it is not valid to compare results of one specimen material to another.

SOME PHOSPHATE TEST PROCEDURES

CUTTINGS

Procedure

Take approximately one level teaspoon full of cuttings from each collected interval (usually 10 foot, sometimes 5 foot) and place on a watch glass. For convenience it is recommended that enough glasses be laid out to test a 200 foot interval of the well at a time. Furthermore if a glass is laid out for each collected interval and is left blank where no sample is available it ensures that the operator will not get confused with respective intervals.

Flood each glass with reagent, wait at least one minute and then read and record the results according to nil, trace, low, medium, and high. Nil = no colour; trace = yellow liquid; low = yellow liquid plus yellow precipitate; medium and high are increasing intensities of yellow precipitate. It does not matter that two operators may differ in their scaling of the results provided that the rule "one operator, one batch of field reagent, for each series of tests" is adhered to.

Results should be recorded on a format such as that in Fig. 7 to which may be added a summary of a lithologic well log and any subsequent assay data.

The response of the reagent to phosphatic material varies considerably. Some of the principal factors governing the reaction are temperature, age of the reagent, and the solubility of the phosphatic material. Some high grade material may be slow to react while calcareous material with less than 1 percent P_2O_5 may react immediately. The result for a 1 percent sample can be identical to that of a 40 percent sample.

However, it is contended that in a continuous series of samples such as cuttings a trace is lower than an adjacent low whereas the same trace may be either higher or lower than a trace a thousand feet deeper in the hole or a trace in any other hole. Therefore the fluctuations that develop in the systematic testing of a continuous series of material are significant.

Thus a log showing the relative distribution of phosphate through a stratigraphic succession is obtained. Quantitative data can be obtained only by assay. The qualitative log enables the selection of an optimum number of samples for quantitative assay (see "sample interval" fig. 2.)

Comments

It should be remembered that the cuttings available in a store such as the B.M.R. Core and Cuttings Laboratory Fyshwick, A.C.T., are a very

small part of the cuttings collected at the drill site, and that the teaspoon of cuttings tested with reagent may not be representative of the interval or even of the bag. Consequently it must be expected that results will not duplicate exactly. However, a reasonable degree of duplication is essential for the results to have any significance.

A series of tests have been carried out to determine the degree of duplication and are recorded in fig. 1. Results using a reagent without an excess of ammonium ion were unsatisfactory.

CORE

Procedure

Establish a sample interval. Most cores are numbered 1, 2, 3 ... and are in 5 or 10 foot intervals depending on the length of the core barrel used by the driller. Other cores, especially those of "slim-line" diamond drilling are laid out, in continuity, in core boxes with footages marked at the end of each row, as well as containing the drillers mark for the end of each lift.

For testing intervals by core numbers:

- (1) Ensure that the surface of the core is clean.
- (2) Remove the core, a piece at a time and in continuity, and scrape down the side of each piece collecting the powdered scrapings on a clean sheet of paper.
- (3) When the interval is complete empty the scrapings onto a watch glass.
- (4) When 10 or 20 or more samples are ready, test with reagent and repeat the procedure as for cuttings.

At the end of the hole all cores that gave a positive result should then be re-tested in detail according to the lithologic changes, in order to trace where the phosphate came from. Spot tests may then be taken for assay.

Spot testing A most satisfactory way of obtaining material for spot testing is to use a small hand drill with a masonry bit. The powder obtained by drilling a hole in the core is collected and need only be pulverised in a mortar and pestle before being assayed. Such a technique is expedient and avoids mutilating the core and the crushing stage of sample preparation. In the event of an intersection of ore grade material the core must be channel sampled by slitting.

EXAMPLES

Fig. 2 :- The phosphate log correlates with the stratigraphy and shows that the Upper Cambrian limestone-dolomitic sediments were barren of phosphate whereas similar sediments of the underlying Middle Cambrian contain phosphate. Such a contrast would certainly be useful for correlating drill holes in the area and should assist in the interpretation of the palaeo-environment of the Cambrian seas.

Figs. 3, 4, 5 :- The phosphate log correlates well with the Arumbera Sandstone - Pertatataka Formation - Areyonga Formation contacts. Farther down the hole (not illustrated) the phosphate log also picks out a 'spillitic zone' in the otherwise barren (of phosphate) Bitter Springs Formation.

It should be noted (Fig. 4) that the upper part of the Pertatataka Formation is essentially barren of phosphate compared with the lower part of the Formation even though the bulk of the lithology is shale.

Comparing this with the gamma ray log it is seen that the relatively consistent signature of the log changes slightly at about the same point as phosphate appears in the Formation. Either log, on its own, could not be regarded as significant; together they reflect some significant change in the palaeo - environment.

In addition to this the medium and high results obtained over the interval 3730' to 3770' are consistent with general considerations on the 'time' for phosphate deposition viz. towards, or at the end of a period of chemical deposition.

Fig. 6. :- The interval was tested specifically to examine the 'spotted shale' marker bed, hence as yet only three assays have been carried out. However, the log agrees reasonably well with the contact between Hutton Sandstone and Evergreen Shale and also correlates to a rise in the gamma ray activity.

The fact that a 2.8 percent P_2O_5 assay for a 'medium' is higher than an 0.9 percent P_2O_5 for a 'high' does not refute the basic principal stated on page 2 when considered in the light of reproducibility results in Fig. 1.

Fig. 7 :- Encouraged by the consistency of agreement between the Phosphate Log and stratigraphy the format for the Phosphate Log has been changed to include additional information which may also be useful. As yet not enough tests have been carried out to demonstrate this.

The left half of the page and the gamma ray log are restricted to information obtained from the well site geologist while the right half concerns the testing of the cuttings and the phosphate log.

It is considered desirable for the operator to note the lithological type and colour of the cuttings he is testing principally to highlight any marked difference between the cuttings over an interval and the reported lithology.

Material containing $CaCO_3$ typically allows any phosphate present to go into solution very rapidly and very low concentrations of phosphate may give strong reactions. To record the carbonate content in terms of + or ++ could be useful in qualifying the interpretation placed in the phosphate log. Likewise the colour of the reaction, yellow, green, blue, white, has significance in terms of the calcium and magnesium carbonate contents, and the presence of organic matter. Organic matter tends to reduce any yellow phospho-ammonium molybdate precipitate to a blue precipitate.

'Even', 'spotty', 'obs.' (=obscured) refers to the distribution of a yellow precipitate in the test container. In general an 'even' reaction may be interpreted as relevant to the interval from which the cuttings were taken, 'spotty' to one or more bands within the interval, while 'obs.' is a typical response to calcareous material and finely powdered material.

For example: in Fig. 6 the 2.8 percent assay, in the absence of additional data, must be considered as representative of the 10 foot interval. However, supposing the reaction was 'spotty' and that the spots represented 10 percent of the test material: then assuming the remaining 90 percent of the material averages 1 percent P_2O_5 the interval may contain an aggregate of 1 foot averaging 19 percent P_2O_5 .

It is emphasised that due to the uncertainty of the degree of representivity of the tested material such an interpretation must be regarded only as a 'speculation'. Should this be of particular interest then it would be worth pursuing a more quantitative approach to the study of the interval.

FIG 1

FIG 1

FORMULATED REAGENT (with excess ammonium ion)

CRUDE REAGENT (without excess ammonium ion)

WHITE PLASTIC
PALLETTE

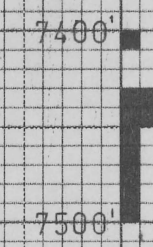
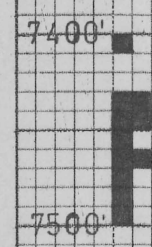
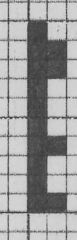
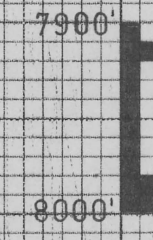
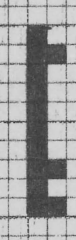
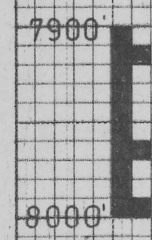
WATCHGLASS ON
STAINLESS STEEL

WATCHGLASS ON
WHITE PAPER

WHITE PLASTIC
PALLETTE

WATCHGLASS ON
STAINLESS STEEL

WATCHGLASS ON
WHITE PAPER



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PHOSPHATE LOG																							
WELL NAME (COMPANY) No. OORAMINNA (EXOIL) No1										WELL INDEX No. (C & C LAB.) 30													
LOCALITY	BASIN AMADEUS					LAT. 24 / 0 / 6 LONG. 134 / 9 / 50 E					ELEV. KB 1624'												
	STATE N.T.					1:250,000 SHEET (NAME & NUMBER) ALICE SPRINGS					T.D. 6097'												
REFERENCES																							
AGE	ROCK UNIT	DEPTH	LOG	CORE	LITHOLOGY (Remarks)	LITHO. TYPE	COLOUR	ML	TRACE	LOW	MEDIUM	HIGH	CARB	EVEN	BOOTH	REACTION	COLOUR	SAMPLE	SQ	Q	ASSAY % P ₂ O ₄	GAMMA LOG	
																						10	20
1000	CAMBRIAN				SANDSTONE																0.18		
2	ARUMBERA FORMATION																						
3	UPPER PROTEROZOIC				LIMESTONE																		
4	PERTATATAKA FORMATION				LIMESTONE, DOLO-MITE, SANDSTONE AND SHALE																		
5					SANDSTONE & SHALE																		
6					SHALE																		
7																							
8																							
9																							
2000	Molybdate Test F.W.					Analysis SQ					Analysis Q F.W.					M(Pf)/18							
Date 1964-5					Date					Date 1964-5													

BUREAU OF MINERAL RESOURCES, GEOLOGY AND GEOPHYSICS												Sheet									
PHOSPHATE LOG												of									
WELL NAME (COMPANY) No. OORAMINNA (EXOIL) No 1								WELL INDEX No. (C & C LAB.) 30													
LOCALITY		BASIN AMADEUS				LAT. 24/0/6 S LONG. 134/9/50 E				ELEV. KB. 1624'											
		STATE N.T.				1:250,000 SHEET (NAME & NUMBER) ALICE SPRINGS				T.D. 6097'											
REFERENCES						CUTTINGS		MOLYBDATE TEST		ASSAY % P ₂ O ₅											
AGE	ROCK UNIT	DEPTH	LOG	CORE	LITHOLOGY (Remarks)	LITHO. Type	COLOUR	NIL	TRACE	LOW	MEDIUM	HIGH	CARB.	EXTR.	BOTT.	REACT.	COLOUR	SQ	Q	GAMMA Log	
UPPER PROTEROZOIC	PERTATATAKA FORMATION	2000																			
		1					SHALE														
		2																			
		3																			
		4					SHALE													0.10	
		5																		0.15	
		6																			
		7					SHALE													0.12	
		8																		0.09	
		9																			
3000																					

NOTE:

Correlation of phosphate log with gamma ray log

DIAGRAMMATIC →

Molybdate Test	F. W.	Analysis SQ	Analysis Q	F. W.	M(Pf)/18
Date	1964 - 5	Date	1964 - 5	Date	

BUREAU OF MINERAL RESOURCES, GEOLOGY AND GEOPHYSICS

PHOSPHATE LOG

Sheet
ofWELL NAME
(COMPANY) No.

OORAMINNA (EXOIL) No 1

WELL INDEX No.
(C & C LAB.)

30

LOCALITY

BASIN

AMADEUS

LAT. 24/0/6 S LONG. 134/9/50 E

ELEV. KB.

1624'

STATE

N.T.

1:250,000 SHEET
(NAME & NUMBER)

ALICE SPRINGS

T.D.

6097'

REFERENCES

CUTTINGS

MOLYBDATE TEST

ASSAY
% P₂O₄

AGE	ROCK UNIT	DEPTH	LOG	CORE	LITHOLOGY (Remarks)	LITHO. TYPE	COLOUR	W/T	LOW	MEDIUM	HIGH	CARB.	EVEN	SPOTTY	OTHER	COLOUR	SAMPLE	SQ	Q	10	20	GAMMA Log
3000																						
1					SHALE																	
2																						
3	UPPER PROTEROZOIC				SHALE																	
4	PERTATATAKA FORMATION																					
5																						
6					SHALE																	
7																						
?	?	3725			LIME STONE														0.34			
																			0.25			
																			0.32			
8					LIMESTONE & SHALE																	
9	AREYONGA FORMATION																		0.26			
					SHALE																	
4000																			0.28			
Molybdate Test F.W.					Analysis SQ					Analysis Q F.W.					M(Pf)/18							
Date 1964-5					Date					Date 1964-5												

To accompany Record No 1965/77

FW

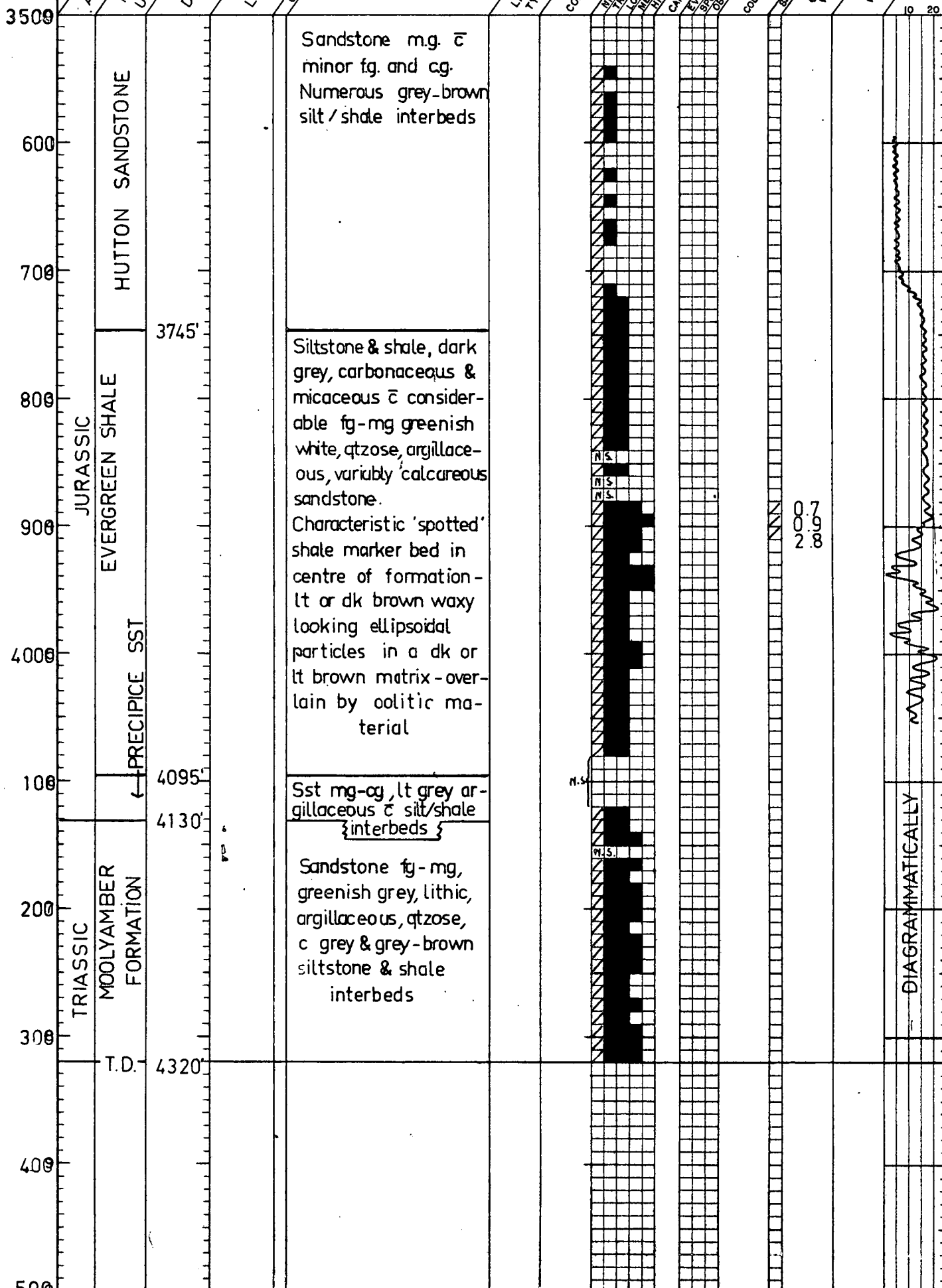
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PHOSPHATE LOG

Sheet
of

WELL NAME (COMPANY) No.			BLYTHWOOD	(AAO)	No 1	WELL INDEX No. (C & C LAB.)
LOCALITY	BASIN		LAT.		LONG.	ELEV. KB. 957'
	STATE	QUEENSLAND	1:250,000 SHEET (NAME & NUMBER)			T.D. 4320 / 4337'

REFERENCES	Q 55-56 P / 172	CUTTINGS	MOLYBDATE TEST	ASSAY
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[illegible]

Molybdate Test	J.B.	Analysis SQ	Analysis Q	S.C.G.	M(Pf)/18
Date	1964	Date	Date	1964	

BUREAU OF MINERAL RESOURCES, GEOLOGY AND GEOPHYSICS														Sheet		
PHOSPHATE LOG														of		
WELL NAME (COMPANY) No.										WELL INDEX No. (C & C LAB.)						
LOCALITY	BASIN				LAT.		LONG.		ELEV. KB.							
	STATE				P250,000 SHEET (NAME & NUMBER)				T.D.							
REFERENCES						CUTTINGS		MOLYBDATE TEST				ASSAY % P ₂ O ₅				
AGE	ROCK UNIT	DEPTH	LOG	CORE	LITHOLOGY (Remarks)	LITHO. TYPE	COLOUR	REL. PRG. LOW	MED. HIGH	CARB.	REACTN. EVEN SPOTTY OBS.	COLOUR	SAMPLE	SQ	Q	GAMMA LOG
0																
1																
2																
3																
4																
5																
6																
7																
8																
9																
0																
Molybdate Test						Analysis SQ				Analysis Q				M(Pf)/18		
Date						Date				Date						