FINAL REPORT

Permit Number: 80/11/05/019/52

SAHRIS Case ID: 9/2/092/0004

Author of report: Name: Prof. S.E. Lauritzen
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Date of report: 12 June 2017

SAHRA Permit Officers on Permit: 1. Mrs Colette Scheermeyer
2. Mrs Nonofho Ndobochani
SAHRA: Manager of the Archaeology, Palaeontology and Meteorites Unit

Date of permit issue: 30 June 2011

Report due date:

Permit Holder: Name: Prof. C.S. Henshilwood
Qualification: PhD
Affiliation: University of Bergen, Norway & University of the Witwatersrand, Johannesburg

Permit To:

Name of locality/site(s): De Hoop Nature Reserve, Western Cape Province.

Object ID's (or batch ID) reflected on permit:

Refer Case ID: 9/2/092/0004

EXECUTIVE SUMMARY (NO MORE THAN 1000 WORDS)

Report: Geological samples, speleothems from caves. Prof. Stein-Erik Lauritzen

A limited number of relict speleothems (stalagmites) were collected from four caves in the de Hoop area to serve as materials for a MSC- PhD project. The purpose is to map climate change during a time when critical evolution of *Homo sapiens* occurred in the area, that is the last 100,000 years. The advantage of using speleothems (i.e. stalagmites) over other climate archives (like marine sediment cores) is that the recorders were situated literally “next door” of the humans who inhabited caves nearby. Speleothems can be dated very precisely by U-series methods (measuring small, natural levels of Uranium in the samples). In addition, the contemporary climate, as recorded by the dripwater that enters the cave and form the stalagmites, can be measured in several ways. Firstly, the layering within the specimens reveal growth rate; dirt bands and discordances reveal floods and draughts, respectively. Secondly, the content of trace elements and stable isotopes (of O and C) can be translated into rainfall and temperature. Together, these records can in turn be used to trace eventual environmental changes that would have affected human occupation of the area. Speleothems are thus key materials for mapping the living conditions for early humans. In additions, new methods are currently being developed for more precise interpretation of data from speleothems.

Four caves were evaluated and sampled: Bloukrantz Cave (BL), Klipdrift Sea Cave (KDS) , Klipdrift Shelter (KDS) and West Cave (WC). The samples were all selected from already broken or damaged specimens and judged for quality on site, ie. Compact and crystalline samples were sought for; porous fabrics were avoided. The samples were cast in plaster blocks for proper orientation and cut in longitudinal slices (“wafers”) for further analyses. Each wafer was then used for further analyses as described in the MSc and PhD theses of Jane S Adigun (2016). The number of samples and each individual is described in great detail in the thesis.

One of several scientific papers are in preparation, based on the PhD Thesis, where Jane Adigun will be the first author.

The samples are currently curated at the Department of Earth Science at Bergen University, and will be returned to SA when all analyses are complete.

We gratefully thank for the permission to collect samples and will communicate further information to you as new analyses are at hand.

SAHRIS OBJECT OR SITE LINKS

METHODS AND MATERIALS

Refer thesis by Dr Jane Adigun:

PAGE 49: 2.4.2 Site selection and speleothem sampling

In April 2008, a preliminary study was undertaken to try to identify caves within the reserve that contained suitable speleothem deposits for uranium-series dating and environmental analysis. This formed the basis for the author's master's dissertation (Noah 2011). The current study builds on this initial work and includes more intensive sampling of Bloukrantz Cave and West Cave which form part of the Stilgat sea caves complex. Additional samples are taken from the Klipdrift Sea Cave and Klipdrift Shelter that collectively form part of the Klipdrift Complex.

Chapter 4 :

Chapter 4 focuses on the stalagmite samples that form the core of this thesis. These samples come from the De Hoop Nature Reserve on South Africa's southern Cape coast and were taken from Bloukrantz Cave, Klipdrift Sea Cave and Klipdrift Shelter located in the eastern section of the Reserve.

Six stalagmites were identified for analysis (BL1, BL3, BL4, KDS & KDC5) and are described individually.

Sample macromorphology

PAGE 104: **BL1** is one of four samples collected inside Bloukrantz Cave which is located on the eastern edge of the De Hoop Nature Reserve.

PAGE 110: **BL2** is the second stalagmite collected inside Bloukrantz Cave.

Sample macromorphology

PAGE 115: **BL3** is the third sample collected inside Bloukrantz Cave and this stalagmite fragment has a height of 425mm from the top to the base and a diameter of 35 mm at the top (Fig. 4.3).

Sample macromorphology

PAGE 118: **BL4** is the fourth sample from Bloukrantz Cave and is a columnar shaped stalagmite fragment that is broken at both ends.

Sample macromorphology

PAGE 121: The **KDS sample** is a small, stalagmite that was found in an archaeological context during the 2012 excavation season at Klipdrift Shelter. The sample comes from quadrate R29c in the PBA/PBB stratigraphic unit

Sample macromorphology

PAGE 124-125: The **KDC 5 sample** has a typical stalagmite shape with a rounded top, wide 125 diameter and stacked growth layers which are clearly defined.

LOCATION DETAILS

These samples come from the De Hoop Nature Reserve on South Africa's southern Cape coast and were taken from Bloukrantz Cave, Klipdrift Sea Cave and Klipdrift Shelter located in the eastern section of the Reserve.

Location name(s):	De Hoop Nature Reserve
GPS Co-ordinates:	34°27'15.08" S | 20°23'58.63" E
Nearest town:	Bredasdorp
Local District:	Overberg
Magisterial District:	Bredasdorp
Province:	Western Cape

Approximate age of materials:
Refer thesis by Dr Jane Adigun:

Age models

PAGE 138
The StalAge algorithm discussed in the previous chapter (Chapter 3 section 3.3.7) was used to model the growth of the BL1 stalagmite. The data used to produce this age model is highlighted in Table 5.1 (**PAGE 141)** and the final age model is presented in Fig. 5.4 (**PAGE 138**)**.**

PAGE 138: Fig. 5.4 The age model obtained for the **BL1** sample from Bloukrantz Cave showing the final age model (dashed black line). Corresponding 95% confidence limits for the StalAge model are denoted by age upper limit (solid red line) and age lower limit (solid green line). The U-series ages measured by ICPMS are also shown (black dots) along with the corresponding 2σ errors.

PAGE 141: Table 5.1 Results from ICPMS U-Th analyses of the **BL1 stalagmite** from Bloukrantz

PAGE 142: *Uranium concentration and activity ratios*
Thirteen U-Th ages were obtained for the **BL3 stalagmite** and the data are summarised in Table 4.2.

Refer also **PAGE 147: Fig. 5.7** Stratigraphic position of each of the uranium series age determinations obtained for the **BL3 sample** from Bloukrantz Cave.

Age models
Using the same procedure for the BL1 stalagmite an age model was produced for the BL3 sample and is shown in Fig. 5.8. refer **PAGE 149: Fig. 5.8** The age model obtained

for the **BL3 sample** from Bloukrantz Cave showing the final age model (dashed black line).

PAGE 150: Table 5.2 Results from ICPMS U-Th analyses of the **BL3 stalagmite** from Bloukrantz Cave Cave

PAGE 151: *Uranium concentration and activity ratios*
Twelve U-Th ages were obtained for the **BL4 stalagmite.**

PAGE 155: Fig. 5.11 Stratigraphic position of each of the uranium series age determinations obtained for the BL4 sample from Bloukrantz Cave.

Age models

PAGE 156: Using the same procedure described for the previous samples (*i.e.*, BL1 & BL3) an age model was produced for the **BL4 stalagmite** and is shown in Fig. 5.12 (**PAGE 157-158)**….The data used to produce the age model for this sample is highlighted in Table 5.3 (**PAGE 159).**

PAGE 157-158: Fig. 5.12a and 5.12b. The age model obtained for the BL4 sample from Bloukrantz Cave showing the final age model (dashed black line).

PAGE 159: Table 5.3 Results from ICPMS U-Th analyses of the **BL4 stalagmite** from Bloukrantz Cave

PAGE 160: *Uranium concentration and activity ratios*
Twenty U-Th ages were obtained for the **KDS stalagmite** although one subsample (J. No. 1038) was omitted due to an analytical problem. The data for the remaining nineteen U-Th ages is summarised in Table 5.4.

PAGE 164:
Using the same procedure used for the Bloukrantz Cave samples (described in Chapter 3 section 3.3.7) an age model was produced for the **KDS stalagmite** and is shown in Fig. 5.15.

PAGE 165: Fig. 5.15 The age model obtained for the **KDS sample** from Klipdrift Shelter showing the final age model (dashed black line).

PAGE 166: Table 5.4 Results from ICPMS U-Th analyses of the **KDS stalagmite** from Klipdrift Shelter with problematic ages shaded.

PAGE 167: *Uranium concentration and activity ratios*
Twelve U-Th ages were obtained for the **KDC 5 stalagmite** and the data are summarised in Table 5.5.
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PAGE 170: Fig. 5.18 Stratigraphic position of each of the uranium series age determinations obtained for the **KDC 5 sample** from Klipdrift Cave.

PAGE 173: Fig. 5.19 The age model obtained for the **KDC 5 sample** from Klipdrift Sea Cave showing the final age model (dashed black line).

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LIST OF ALL PARTICIPATING RESEARCHERS

Name: Prof. C.S. Henshilwood
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CURATION OF MATERIALS

Name of Institution:
University of Bergen

Name of Curator:
Prof. Stein-Erik Lauritzen

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Institutional Address:
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How is the material being curated?
The material is stored in the U- series laboratory and in the GEO compact storage area at UiB.

The samples are currently curated at the Department of Earth Science at Bergen University, and will be returned to SA when all analyses are complete.

GEOLOGICAL COLLECTIONS AND EXCAVATIONS

Responsible person 1:	Prof. C.S. Henshilwood, University of Bergen, Norway & University of the Witwatersrand, Johannesburg
Responsible person 2:	Prof. S.E. Lauritzen, University of Bergen, Norway
Responsible person 3:	Dr Jane Adigun, University of the Witwatersrand, Johannesburg
Number of participants:	8
Duration of fieldwork	12th March – 2nd April 2008 and 28th February – 4th March 2011
Excavation equipment used	Hammer and chisel, if not loose pieces
Description of work/methodology	Refer thesis by Dr Jane Adigun
Site management	Cape Nature
Description of work/methodology; *Refer thesis by Dr Jane Adigun:*	

PAGE 71: 3.2.3 Method for preparing speleothem samples
PAGE 72: 3.2.4 Measuring U-series isotope ratios using the Element 2

CHAPTER 3 provides an overview of the theoretical basis for the method used in this study. The specific procedure used for selecting sub-samples for dating and stable isotope analyses are discussed. Here the discussion focuses on the principles, applications, and constraints of the 230Th/234U dating methods applied to each of the samples. In the final section of the chapter, the discussion focuses on the sampling approach used for extracting stable isotopes of carbon and oxygen and the age 60 models used to constrain the isotopic data. An overview of the dripwater and temperature reconstruction analyses are also presented.

CHAPTER 4 In addition, to describing the physical appearance and other morphological attributes of the samples, the methods used to prepare each of the samples for uranium-series dating are also discussed. In addition, the procedure used to prepare the samples for isotopic analyses are also presented here.

BL1 sample
PAGE 108: Sample preparation for U-series dating by 234U/230Th
PAGE 110:Sample preparation for stable C and O isotope analysis
BL2 sample
PAGE 114: Sample preparation for U-series dating by 234U/230Th
PAGE 114: Sample preparation for stable C and O isotope analysis
BL3 sample
PAGE 117: Sample preparation for U-series dating by 234U/230Th
PAGE 118: Sample preparation for stable C and O isotope analysis
BL4 sample
PAGE 120: Sample preparation for U-series dating by 234U/230Th
PAGE 120: Sample preparation for stable C and O isotope analysis

KDS sample
PAGE 124: Sample preparation for U-series dating by 234U/230Th
PAGE 124: Sample preparation for stable C and O isotope analysis

KDC5 sample
PAGE 126: Sample preparation for U-series dating by 234U/230Th
PAGE 127: Sample preparation for stable C and O isotope analysis

CONCLUSIONS REACHED

Refer reports by Prof. Prof. S.E. Lauritzen, University of Bergen, Norway
and thesis by Dr Jane Adigun, University of the Witwatersrand, Johannesburg

Refer thesis by Dr Jane Adigun: PAGE 289:

General conclusion

In this thesis speleothems collected in the De Hoop Nature Reserve, southern Cape that contained palaeoclimate records from MIS 5a to MIS 3 were analysed. The data I present indicates shifting palaeoenvironmental conditions across this time period with warmer relatively stable conditions in early MIS 5a. In late MIS 5a and early MIS 4 there is greater variability in comparison with early MIS 5a. However, MIS 3 is the most variable period recorded here. In this study I have emphasised that much greater control is needed on in-cave variables, for example air temperature and atmospheric CO2. This is particularly relevant to the interpretation of the 18O signal in terms of temperature and rainfall. I also indicate that the ages for some of the known archaeological data for this region, especially from the older excavations, is not sufficiently accurate to allow for direct comparison with the speleothem signals. This integration will become more productive as a fine-grained understanding of the ages of the archaeological deposits becomes available.

Perhaps the key message I would like to emphasise in this thesis is that *Homo sapiens* in this region faced highly variable environmental and climatic conditions during MIS 5a to MIS 3. Yet, despite this variability it seems that these early humans were highly adaptable, both in terms of changing technologies but also in their demographic and subsistence patterns. This might be one of the reasons that when *Homo sapiens* left Africa at *c.* 60 ka they so easily adapted to the very variable conditions found in other continents.

ADDITIONAL DOCUMENTS

Refer thesis by Dr Jane Adigun:

List of speleothems recovered (see also page 3 in this report)

Sample macromorphology
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Sample macromorphology
PAGE 124-125: The **KDC 5 sample** has a typical stalagmite shape with a rounded top, wide 125 diameter and stacked growth layers which are clearly defined.

Map of collection points including GPS coordinates

Location name(s): De Hoop Nature Reserve
GPS Co-ordinates: 34°27'15.08" S | 20°23'58.63" E

Location and environmental setting of the De Hoop Caves and Klipdrift Sea Cave
Location
The De Hoop Nature reserve is *c.* 260 km east from Cape Town and is located along the southern cape coast of South Africa (Fig. 2.10). The terrestrial section of the reserve covers an area of *c.* 340 km2 and extends for approximately 5 km towards the sea.

Refer thesis by Dr Jane Adigun:

PAGE 47

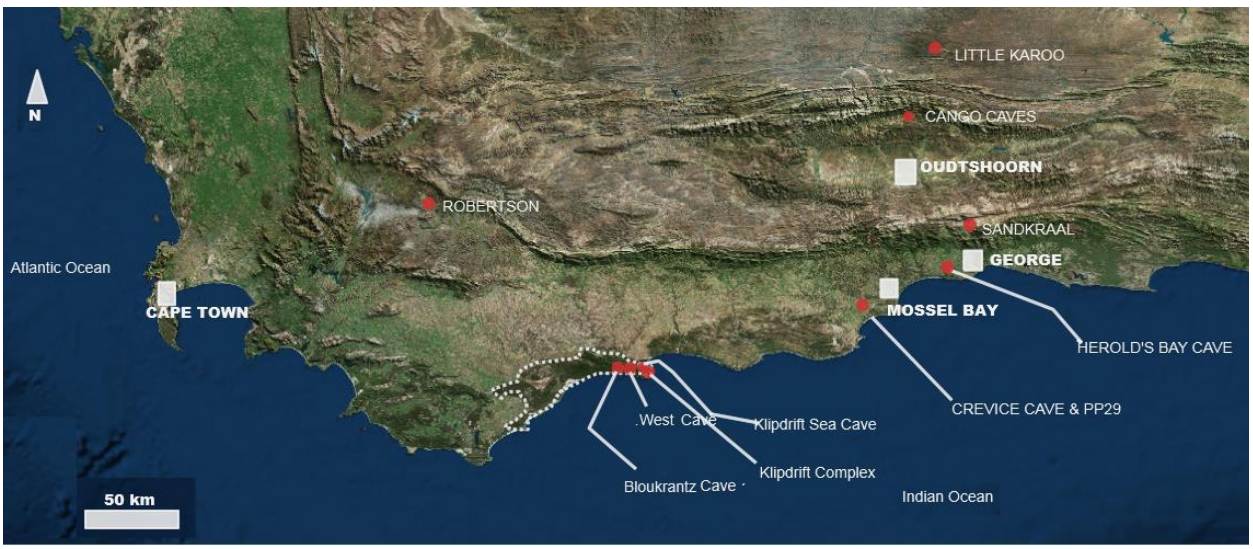

Fig 2.10a Location map of the De Hoop Nature Reserve (study area) containing Bloukrantz Cave (BLOU), West Cave (WC) and Klipdrift Sea Cave (KDC). The extent of the reserve is shown (- - - -) and the location of the other speleothem bearing caves in the southern Cape. These sites are the Cango Cave, Pinnacle Point (containing PP29 & Crevice Cave), the Little Karoo, Sandkraal Cave and Herold's Bay Cave

PAGE 57:

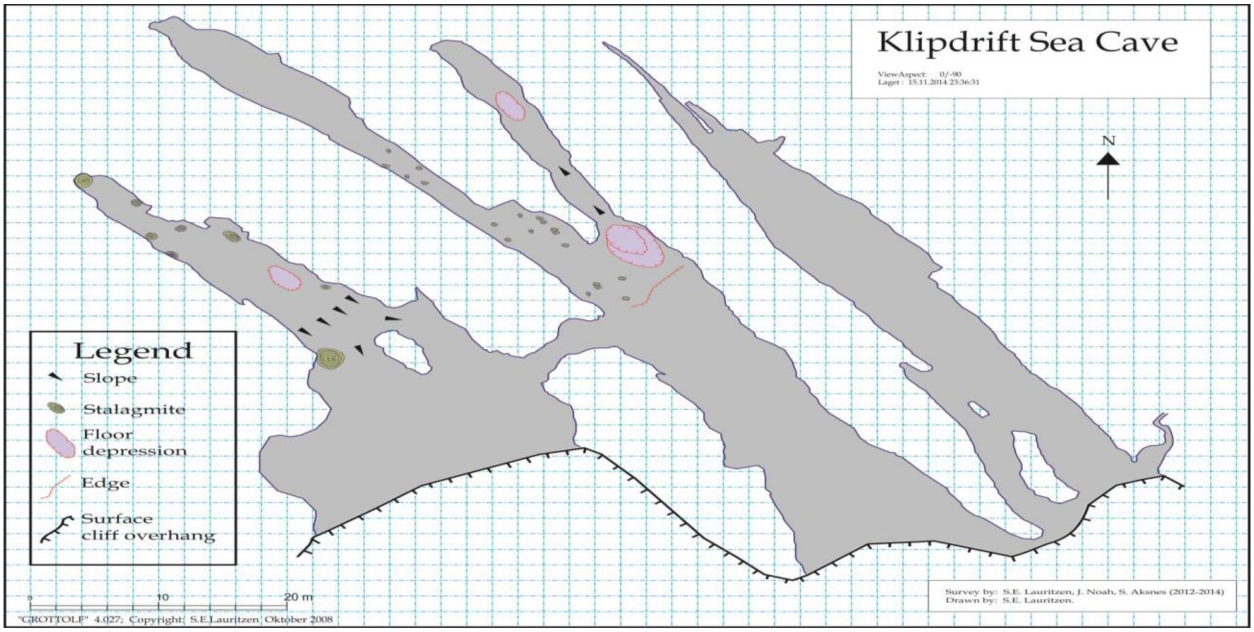

Fig. 2.15 Survey map of Klipdrift Sea Cave showing the passages and location of stalagmite deposits inside the cave and its extension of Klipdrift Sea Cave East

DESTRUCTIVE ANALYSIS

Detailed description of how the samples were taken

Description of the analytical techniques used

Refer thesis by Dr Jane Adigun:

PAGE 71

3.2.3 Method for preparing speleothem samples
Each of the samples were first cast and cut parallel to the central growth axis. The plaster-of-Paris cast was removed by soaking the sample in water and then cleaning the surface of the speleothem with 37% (technical grade) HCl. Once the samples were cleaned, subsamples of *c.* 0.5g weight and a thickness of 5-10mm each were drilled out from the individual stratigraphic layers. These samples were conditioned for heating inside a Binder drying oven and then ignited at high temperature (*c.* 600-850oC) for *c.* 4-6hrs. After ignition, the samples were dissolved in 14M HNO3, spiked with uranium and centrifuged to remove insoubles at 2,500 rpm for *c.* 5min. The samples were then evaporated to almost dryness and re-dissolved in 1M HNO3 up to a volume of 10ml. Actinides in the samples were extracted via ion exchange chromatography using Eichrom TRU resin columns (100-150µg grain size). The resin is activated in 1M HNO3

and is used to bind U and Th actinides which form complexes with the nitrates in the

solution.

Discussion on the efficacy of the technique

The methods are front- end techniques for dating and extracting climatic information.

Summary of the results

See Adigun thesis and reports above.

Accidental damage

There is no accidental damage of samples. They were all cut intentionally on a rock saw and then subsamples were drilled out for analysis.