REPORT ON KAVA ANALYSIS 2012-2015 (JULY)

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Introduction

Kava, Piper methysticum, is a traditional drink of Vanuatu and a number of Pacific countries such as Fiji, Tonga, Samoa and Palau. The active components in kava are kavalactones. The six major kavalactones are: [1] methysticin, [2] dehydromethysticin, [3] kavain, [4] yangonin, [5] desmethoxy yangonin,[6] dihydrokavain. Vanuatu has 12 varieties of noble kava, 79 varieties of medicinal kava and 44 varieties of two-day kava. From these three groups of kavas, the noble and medicinal can be differientiated from the two day kavas by the concentration of dihydrokavain, kavain, yangonin, methysticin and desmethoxyyangonin, in this order, or with Kavain being in highest concentration followed by dihydrokavain and then the rest of the other kavalactones.

During the mid-1990's to the year 2000, Vanuatu experienced an export boom which saw large amounts of kava being exported into Europe to Nutriceutical companies looking for new herbal supplements to combat stress, depression and other health issues. Farmers and Exporters spurred by the kava boom did not place quality as a priority. This oversight in quality control saw the export of peelings along with roots and chips. A number of deaths from liver poisoning due to the consumption of pills and other kava based products resulted in the ban of kava to Europe. This ban lasted from 2001 through to the early part of 2015. This saw a loss of VT5 billion annually for Pacific Island Countries (PINA, 2012).

In 2002, the Kava Act was drafted so as to create quality standards for the export of Kava. The Act basically outlines the standards for the farming of kava and for the exportation of Kava. (Kava Act, 2002) While the standards outlined in the Act are mandatory, the lack of a Regulation to implement this has made it difficult to regulate the Act.

In the 11th session of the Coordinating Committee for North America and South West Pacific (CCNSWP) held in Tonga in 2010, Tonga presented the discussion paper of a proposal for new work on the development of an international standard for dry kava products. In the 12th session of its meeting in Madang, Papua new Guinea in 2012, it was agreed that Vanuatu would take the lead on collecting more evidence on the safety of kava. More work is still required to be carried out on this standard.

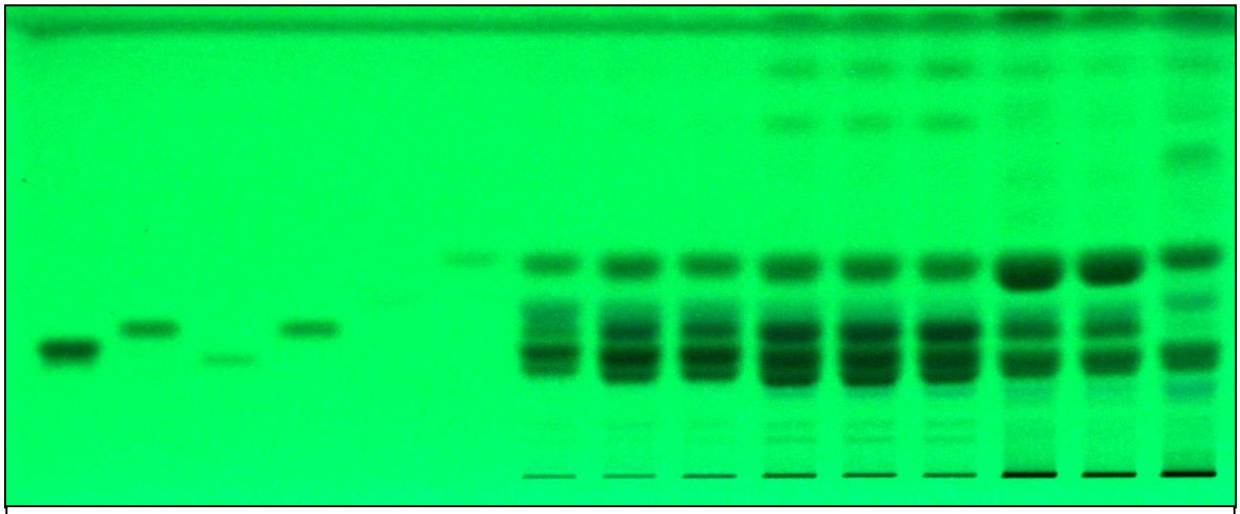
The High Level Kava Conference held in Vanuatu in early 2012 highlighted the importance of quality control of kava and the economic effects that the EU ban had on the annual revenue of all Pacific island countries who are major exporters. Renewed efforts were made then to continue the fight towards lifting the EU ban and to collect more scientific evidence for the development of an international standard for Kava. To date, BfARM the German Pharmaceutical company which caused the EU ban, has lost its court case and the EU has lifted the ban on kava exports. The lesson to be learnt from this experience is to ensure the quality of our kava exports is maintained through regular testing and traceability.

Methods of Analysis

The common method of analysis is high pressure liquid chromatography (HPLC). This method was employed in the late 1990's by one of the export companies, however, it is very expensive to maintain as it requires special analytical columns and chemicals specifically for HPLC.

Near Infrared Spectrophotometer (NIRS)

This was the initial method of analysis used at this laboratory. While it was fast and did not require the use of expensive chemicals it required annual calibration of the machine which would have cost VT1,000,000 which would be more than the revenue generated from the analysis. Due to this high cost,



PICTURE 1: HPTLC PLATE SHOWING THE INDIVIDUAL KAVALACTONES AND FLAVOKAVAINS

it was necessary to find another method of analysis.

High Performance Thin Layer Chromatography (HPTLC)

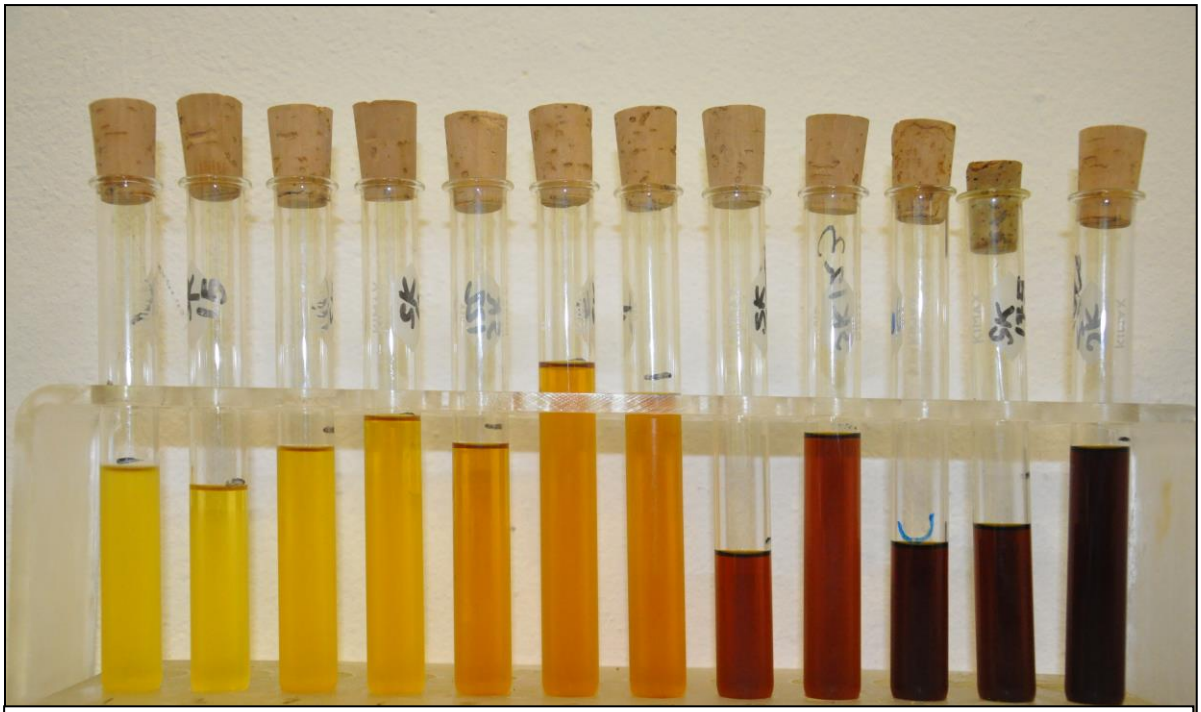
This method of analysis was investigated and found to be a lot more accurate than NIRS in that it was able to detect small differences between the noble types and non-noble types of kava. (Lebot V., Do, TK and Legendre, L. 2014). This method can only take a minimum of 18-20 samples at a time on a 10 x 20 cm HPTLC plate [Picture 1]. One analysis session takes 2 hours and in one day 144 to 160 samples could be analysed. In terms of chemicals, it uses 35mLs of a mixture of dioxane, hexane and ethyl acetate per analysis. Due to the low throughput of samples at the current time, it would not be possible to use this method of analysis.

Thin Layer Chromatography (TLC)

This method is similar to that of HPTLC but is simpler and does not require high set up costs. It is however, a qualitative method. This means that it can determine the difference between noble and non-noble kava but it is not able to quantify the individual kavalactones.

Colorimetry

This method uses the different colours of the acetone extract to identify the kava variety. The kava powder when mixed with acetone gives different coloured solutions. The colour of the solution determines the kava variety with noble varieties giving a yellow solution, two day varieties giving an orange to dark orange solution and wild varieties giving brown to dark brown solution [Picture 2].



PICTURE 2: EXTRACTS OF NOBLE AND NON-NOBLE KAVA. FROM THE FAR LEFT THE FIRST FOUR TEST TUBES SHOW THE YELLOE COLOUR OF THE NOBLE VARIETIES, THE NEXT FIVE TEST TUBES SHOW THE ORANGE COLOUR OF THE TWO DAY VARIETIES, THE LAST THREE TEST TUBES ON THE RIGHT SHOW THE COLOUR OF THE WILD KAVA VARIETIES.

This is the simplest and easiest method to use to determine the differences between noble and non-noble kava through the colour of the extract. In terms of set up costs, it requires the use of a UV-Vis spectrometer and can be used in conjunction with the TLC method. In terms of costs to the Exporter it would not be expensive to run and in the long run may be developed as a test kit for exporters.

Standard Operating Procedures

Over the past three years a process has been set up for analysis. This is illustrated in the flow diagram illustrated in Figure 1.

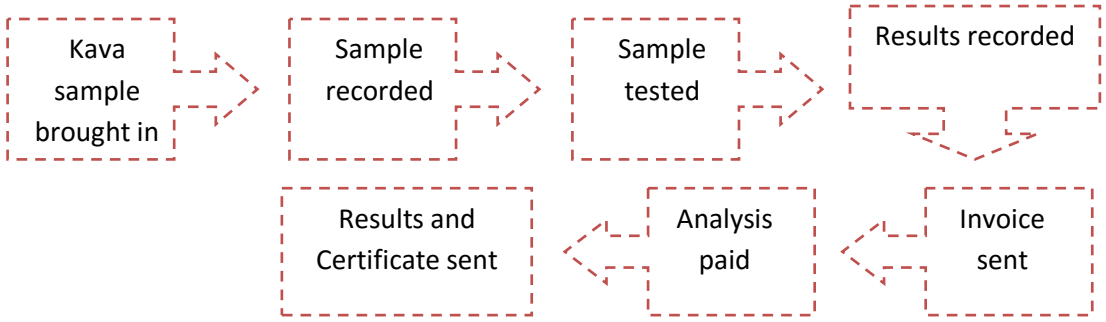


FIGURE 1: FLOW CHART OF ANALYTICAL PROCEDURE

Details of this process can be viewed in the Policy and Procedures Manual for the laboratory [Annex 1].

Kava Analysis

Exporter	2012	2013	2014	2015 (July)
South Seas Commodities	111	10	37	5
Vanuatu Kava Store	1	6	-	5
The Kava House	12	-	2	5
Kava Emporium	2	7	1	6
VRRP	11	24	66	8
Valele Trust	1	-	-	-
De Quiros Exporters	-	1	-	1
American Kava Association (AKA)	-	-	26	-
VCMB			9	1
Kava House	-	-	10	6
Nakamal trading				1
Hugaituva Kava Store				1
Private Individuals	17	-	9	1
TOTAL	**155**	**96**	**160**	**40**

TABLE 1: EXPORTERS VERSUS NUMBER OF SAMPLES ANALYSED

In 2012, analysis of kava commenced with the first 14 samples coming from South Seas commodities, a kava exporting company which exports primarily to New Caledonia. Kava samples were analyzed using the Near Infrared Spectrophotometer (NIRS) initially as a pilot study. It was slow to take off [Table 1] as Exporters were not aware of the service or were unsure about the accreditation status of the laboratory. In 2013, a problem with the accuracy of the NIRS caused analysis to be stopped in 2013 hence the low number of analysis.

Figure 2 gives a picture of the Percentage of Kava samples analysed per Exporter by Kava type (noble or two day) who have utilized the testing services of the Food Technology Development Centre and Analytical Unit to verify the quality of their samples. Certain Exporters have increased the number of samples they have brought in for analysis while others have reduced their samples. This fluctuation in analyses is directly related to the lack of implementation of Kava Standards given that it is a mandatory standard.

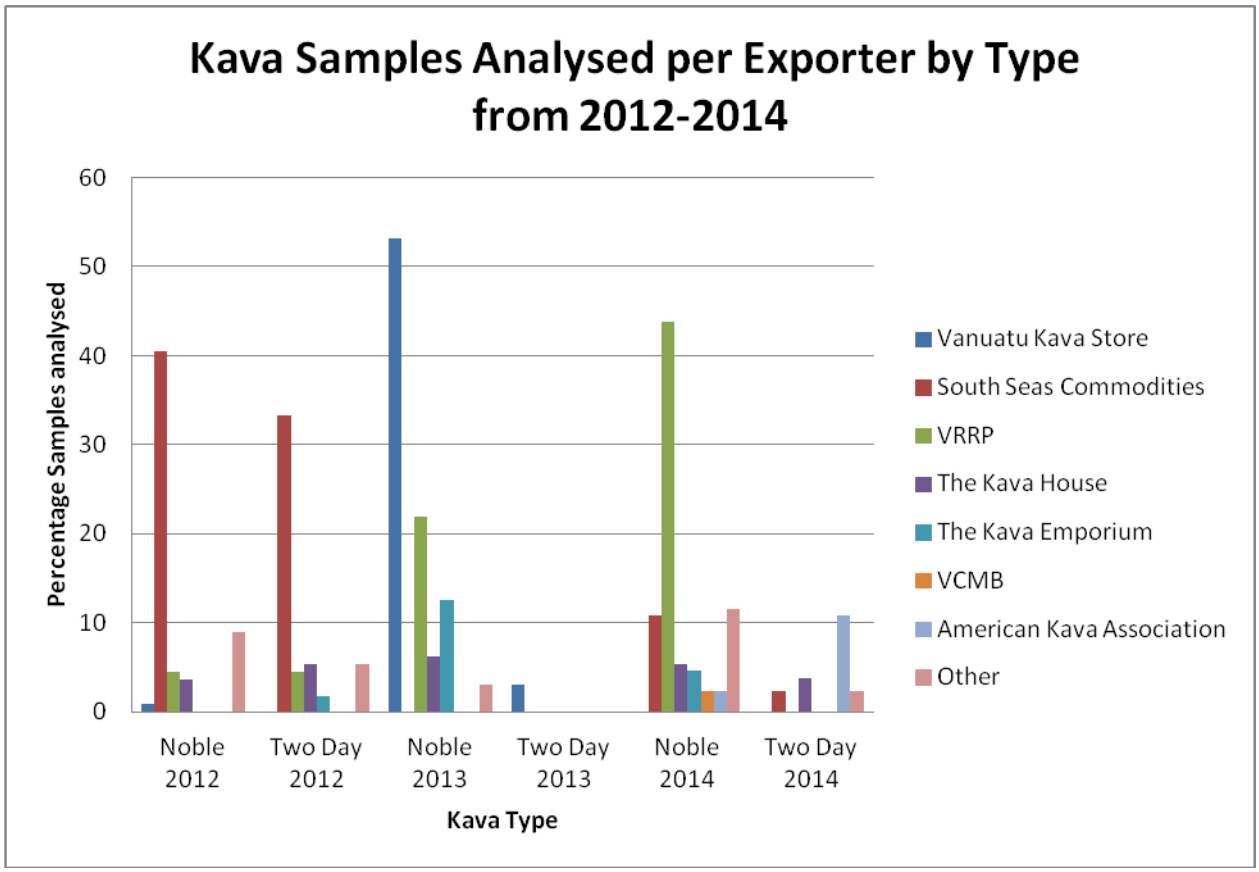


FIGURE 2: COMPARISON OF NOBLE AND TWO DAY KAVA BROUGHT IN BY EXPORTERS FOR ANALYSIS

Trends in kava quality

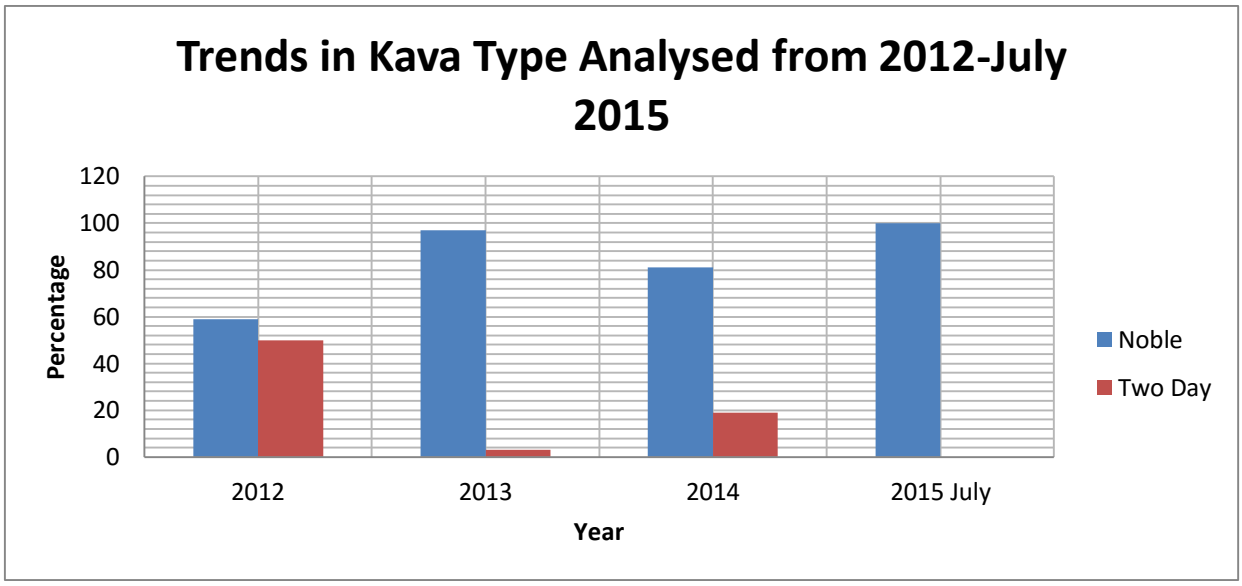


FIGURE 3: COMPARISON OF KAVA TYPES ANALYSED SINCE 2012 TO MID-JULY

Since kava analysis commenced there has been a marked increase in the noble varieties analyzed. It must be noted here that this data reflects samples that have been brought in for analysis only. Figure 3 shows the trend in the quality of kava types brought in for analysis for the past 3 years.

While some Exporters are ensuring that all kava exported are analyzed, there are still a certain number who do not check the quality of the kava exported. This is evident in Kava Exporters residing in Santo. Current arrangements with the Vanuatu Commodities Marketing Board (VCMB) have seen attempts by the Board to collect samples for analysis with the Exporter paying for these tests.

Experience has seen that Kava consignments that have been exported into the United States, through the American Kava Association [Figure 2], have had samples sent back to Vanuatu for testing as per the Food Drug Administration (FDA) rules.

While there has been an improvement in the quality of kava exported, there is room for more improvement especially in the regulating of Exports. It must be noted that the data presented come from only a small number of Exporters based in Port Vila and the occasional sample from Santo.

Revenue earned from Tests

Year	Total Samples analyzed	Total revenue earned
2012	155	No fees charged
2013	96	9,600

2014	160	160,000
2015 (July)	38	38,000
Total	**449**	**207, 600 estimates**

TABLE 2: REVENUE EARNED PER YEAR [NOTE: VALUES ARE CALCULATED FROM THE NUMBER OF SAMPLES ANALYSED IN OUR DATABASE ONLY AND IS NOT FROM THE DEPT OF FINANCE. THERE MAY BE DIFFERENCES IN VALUES]

During the first year of analysis, no fees were charged as the Analytical Unit was trialing out the tests as this was the first time that the Unit was analyzing kava. The low number of samples and revenue in 2013 was related directly to problems with the NIRS machine not giving accurate readings. Analysis increased in 2014 due to the use of the colorimetric method of analysis.

The fees that are currently charged for analysis are 1000VT for samples that have not been ground up as extra time is taken to powder the samples, while VT500 is charged for powdered samples. A fee of VT500 is charged for the Certificate of Analysis (COA).

At the current time there is no legal framework allowing for fees to be prescribed so the fees will remain at this level until such time.

Conclusion

Kava analysis will come ever more important as more and more importing countries become more knowledgeable about kava. This means that they will demand good quality kava. For this reason, it is necessary that the Kava Act is implemented to ensure that the loss of revenue experienced during the EU ban is not repeated. It also means that alternative markets need to be sought closer to home. The lesson learnt from the EU ban was that quality and proof of quality should never be overlooked.

Recommendations

The following are recommendations made to improve the current testing capacity

- **Encourage** Bio-security Vanuatu and Department of Agriculture and Rural Development (DARD) to enforce Kava Regulations to implement the Kava Act to continue to improve the quality of kava exported.
- **Increase the awareness** of farmers on the negative impacts of poor quality kava on the market.
- **Enhance** testing and calibration records and standard operating procedures
- **Equip** the analytical unit with appropriate equipment to increase its current testing capacity

- **Develop** the colorimetric method of analysis to be used in conjunction with the TLC method as a quick and less costly method for routine analysis.
- **Work** towards GLP (Good laboratory practices) Certification for Kava Analysis.

References

Pacific Kava Producers losing over VT5 Billion in EU Ban. PINA , 2012. http://www.pina.com.fj/?p=pacnews&m=read&o=131321920250b6d31161f35dab3820. Date

Kava Act, Vanuatu Government, 2002.

Food Chem. 2014 May 15;151:554-60. doi: 10.1016/j.foodchem.2013.11.120. Epub 2013 Nov 27. Date of Access 3/8/2015

ftp://ftp.fao.org/codex/meetings/CCNASWP/ccnaswp12/na12_08e.pdf. Date of Access 3/8/2015

Annex 1

Policy

and

Procedures Manual

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INTRODUCTION

The key to quick and efficient laboratory testing lies in the processes and procedures in a laboratory and how well laboratory technicians know and implement them. Knowing what to do in a laboratory will ensure that quality is maintained at all times.

This Policy and Procedures Manual serves as a guide to all current and new staff of the Food Technology Development Centre and Analytical Unit as to the policies that guide the standard operating procedures of this laboratory and the rationale behind them.

This guide is to ensure a standard procedure is in place on how to go about the day today running of the Laboratory and maintain transparency and Traceability.

As time goes by additional policies and procedures will be added to ensure that these policies are as up-to-date as possible.

QUALITY POLICY STATEMENT

To ensure accurate and timely analytical testing services and to continuously meet or exceed the stated or implied expectation our customers through day-to-day interactions.

Effective date: April 1st, 2012

a) Management commitment to good professional practice and quality of services provided to the customer: Tests are always carried out in accordance with stated standardized methods and customers' requirements. Requests to perform tests that may jeopardize an objective result or have a low validity are rejected.
b) Standards of service include:
    a. Customer satisfaction
    b. Accurate
    c. Timely

    Excellence in the workplace is promoted by providing all employees with the knowledge, training, and tools necessary to allow for the completion of accurate and timely work
    .

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SUMMARY OF ANALYSIS PROCESS

Process	Procedure	Form
1. Analysis Request	1.1 – 1.4	• Form AR 1.1
2. Sample Receipt	2.1 – 2.3	• Database • D.B.K. 1.1
3. Sample Analysis	Laboratory Manual	• Worksheets
4. Analysis Report	4.1 – 4.3	• AR 4.1 Certificate of Analysis and Analytical
5. Payment	5.1 – 5.3	• Invoice • Receipt

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1.0 ANALYSIS REQUEST

Policy:	All information pertaining to analysis shall be recorded and given correctly and in an efficient manner and shall be with regards to the test required for the sample in question only.
Rationale:	Customers who request analysis must always be given the information regarding analysis as correctly and efficiently as possible in order to make it easy for them to make a decision about whether to analyze or not.
Procedure:	**AR 1.1 Record information in Analysis Request form**. 1.1. Find out what kind of test the customer wants. a. If Customer tells you the type of test they want then follow procedure 1.2. b. If the Customer does not know what type of test is require for their sample follow procedure 1.3. 1.2. Give details about the test, a. Payment method b. When Customer should expect their test results c. Other information about the particular test they are requesting. 1.3. Inform the customer about the different tests and what they determine in terms of their product and similar information to procedure 1.2. 1.4. Place request form on the "IN" tray on the Laboratory Technicians desk.
Forms:	• CHF1.1 Analysis Request Form
Other Documents:	• Confidentiality Policy,

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A.R.1.1. Analysis request form

Date of Receipt: _____ Reference Number _____

Office use only

Client Information

Client Name _____

Company Name _____

Company Address _____

Telephone Number _____

Email Address _____

Sample Information

Sample Name _____ Sample Type _____

Sample Description _____

Initials of Reviewer(s):…… Revision date:…../…/…

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Testing Information

Type Of Test

- ☐ pH
- ☐ Iron test
- ☐ Iron Content
- ☐ Cut Test
- ☐ % Fat
- ☐ Kava Quality test
- ☐ Moisture
- ☐ SAP test
- ☐ HPTLC
- ☐ Esterification Value

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2.0 RECEIVING SAMPLES

Policy:	All samples received for analysis must be recorded on the Sample Receipt form, labeled and recorded in appropriate database immediately on day of receipt.
Rationale:	It is good laboratory practice to record all samples received for analysis on a database or record book. This makes it easy to collect statistics on tested samples and also for traceability purposes. All samples recorded are given a laboratory sample number which is used on all forms related to the sample.
Procedure:	2.1. Upon receiving a sample, record all information in the sample analysis request form in the Database in the Main station PC. The analysis request form will determine what type of sample is to be analyzed and what tests need to be carried out. 2.2. Find the appropriate worksheet needed for this testing method and attach it to the sample. 2.3. Place the sample along with its worksheet in the "For Analysis" tray.
Forms:	Analysis Database (main computer) • For Kava samples, Open Data base D.B.K. 1.1 • For Cocoa sample, open Database D.B.CC 1.2 • For coconut oil sample, open Data base D.B.E.C. 1.3 • For Imported foods, Open Data base D.B.I. 1.4
Other Documents:	• Worksheets [Laboratory Manual]

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D.B.C. 2.1 Commodities Sample Receipt Database

ID	Date Receipt	Client Name	Owners sample code	Lab. Sample code	Sample Description	Method of Analysis	Date of Analysis	Analysis Result	Price per analysis	Invoice number	Reference Number	VAT	Amount Owed	Receipt Number	Date of Payment
1	3/27/2012	VKS	10	10B2412	Dried kava roots & chips	NIRS	27/03/12	Noble	VT1000	01/2012	01VKE060144	1.25	1,125VT	75582	5/27/12

D.B.I. 2.2 Import Sample Receipt Database

ID	Date Receipt	Client Name	Owners sample code	Lab. Sample code	Sample Description	Method of Analysis	Date of Analysis	Analysis Result	Price per analysis	Invoice number	Reference Number	VAT	Amount Owed	Receipt Number	Date of Payment
1												1.25	1,125VT	75582	5/27/12

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3.0 ANALYSING SAMPLES

Policy:	All samples received must be analyzed according to procedures in the official Laboratory manual.
Rationale:	All test methods used in this laboratory can be found in the Laboratory Manual. Test methods that are beyond our capabilities are not recorded.
Procedure:	**Refer to Laboratory Manual for appropriate Test method for the sample.**
Forms:	Refer to Laboratory Manual for Worksheets and Analysis Result Sheets.
Other Documents:	Laboratory Manual.-Volume 1 : CHEMISTRY

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4.0 ANALYSIS Report

Policy:	Analyses shall be reported on the official analysis results (AR) form and certificate of analysis (COA) only with endorsement by the designated head of the laboratory.
Rationale:	All results reported must be on the official laboratory letterhead only and signed off by the head of the laboratory. Results that have been reported otherwise will not be regarded as official and as having come from the laboratory.
Procedure:	4.1 Prepare a Certificate of Analysis (COA) and an Analysis Report (AR) from the data on the appropriate worksheet. 4.2 Submit the CoA and AR to the Manager for approval and signature 4.3 Place CoA and AR in the "CoA and AR" box or if payment was received for analysis on deposit of sample then a scanned copy of CoA and AR should be emailed immediately to the client.
Forms:	• Analysis Report Form • Certificate of Analysis
Other Documents:	• Worksheets

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AR 4.1 Certificate of Analysis and Analysis Report

Kava Sample Receipt and Analysis Report Form **Reference: 29VRRP300415**

Date of Receipt:	30/04/2015	**Sample Type:**	Powder
Clients Name and Address: **Telephone**: **Email address**: **Test Results**:	VRRP Limited vrrp@vanuatu.com.vu	**Method of Analysis**:	TLC and Qualitative

Sample code	% Kavalactone						TLC results	Chemotype	Qualitative Type Test
	1. DMY	2. DHK	3. Y	4. K	5. DHM	6. M			
445B300415/ TKK							Noble		yellow
456B300415/ PP							Noble		yellow

Key: Noble=yellow; Two-day= orange; Wild=brown

Both kava samples are of noble kava variety and can therefore be used for consumption or commercial purposes.

Analyst:_____Beverly Nishai____ Report Authorised by: _Ruth Amos_____ Date: 13/05/2015

Initials of Reviewer(s):…… Revision date:…./…/…

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CERTIFICATE OF ANALYSIS

Reference	29VRRP300415	
Date	13/05/2015	
Clients Name & Address	VRRP Limited Email:- vrrp@vanuatu.com.vu	
Method of Analysis	TLC and Qualitative Type Test	
Number of Samples Tested:	2	
Names of Samples tested:	**TLC Results**	**Qualitative Type Test**
455B300415	Noble	Yellow
456B300415	Noble	Yellow

	Analysts name	**Analysts signature**	**Date**
Authorised by:			
	Ruth Amos		13/05/2015
	Manager	**Signature**	**Date &Stamp**

Initials of Reviewer(s):……

Revision date:…../…/…

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5.0 PAYMENT

Policy:	Analysis results shall only be released after payments have been made.
Rationale:	All analyses must be paid before the results can be released to the Customer as there has been experience in the past of results being released with no payments ever been made even after invoices have been sent numerous times to the customer concerned.
Procedure:	5.1. Prepare an invoice for the analysis carried out. 5.2. Email invoice to client and Manager and inform client that hard copies of the CoA and AR are ready to be collected at the FTDC-AU office 5.3. Write a receipt in the Government Receipt Book and give the first copy (original) to the client with the COA and AR.
Forms:	• Invoice
Other Documents:	• Receipt book • Government receipt

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PYT 5.1 Invoice

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Sample

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PYT 5.2 Receipt

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