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ASSISTANCE TO THE ESSENTIAL OIL INDUSTRY, ZANZIBAR

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UNITED REPUBLIC OF TANZANIA

<u>Technical report: Maximizing the capacity of the clove</u> <u>distillery at Chake Chake, Pemba</u>*

Prepared for the Government of the United Republic of Tanzania by the United Nations Industrial Development Organization, acting as executing agency for the United Nations Development Programme

> <u>Based on the work of S. Jain,</u> <u>consultant in perfumery</u>

Backstopping Officer: T. De Silva Chemical Industries Branch

United Nations Industrial Development Organization Vienna

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* This document has not been edited.

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Introduction

Cloves (Eugenia Caryophyllata) are the major crop in the Zanzibar Islands, which include Unguja and Pemba and are traditionally called the Clove Islands.

There are two major facilities for the distillation of Clove Buds and Clove Stems under the Control of the Zanzibar State Trading Company (ZSTC). One is an almost obsolete plant, nevertheless with a considerable production capacity situated at Malindi in Unguja Island. The other is modern plant at Chake Chake in Pemba Island.

In Pemba, there is an established capacity but ensurement of maintainence is important.

Attuned to the distillation capacity of the plant, the ensurement of timely collection and organised drying of raw materials is crucial.

The Government expects UNIDO to provide support to enhance the economic, commerical and technological structure to modernise the industry in Zanzibar.

POST TITLE : CONSULTANT IN PERUFUMERY

- DUTIES : The expert was expected to carry out the following in collaboration with the CTA and the Counter part staff:-
- i) Evaluate the potential of the essential oils presently being produced for the preparation of aroma chemicals and distillates.

- ii¹ Advise on methods to upgrade the quality of the Clove Oil for use in perfumery.
- iii) Train counterpart staff in methods of sensory evaluation, quality assessment and blending.
- iv) Recommend further requirements for the improvement of essential oils for use in perfumery.
- v) Submit a fully prepared report to UNIDO, at the end of the mission, embodying his findings and recommendations.

Summary

The mission took place between 3rd Feb'93 and 17th Feb'93, during which time the expert was attached to the Clove Steam Distillery of the Zanzibar State Trading Corporation, at Chake Chake, Pemba, Zanzibar.

As per the terms of reference of the assignment, the expert functioned under the supervision of the CTA, Dr. B.C. Ghulati and the NPD Mr. Nasib Omar.

At the beginning of the mission, the expert was duly briefed by the CTA, Dr. B.C. Ghulati.

The expert worked with Project Team of the Clove Stem Distillery (CSD) and exhaustively covered all aspects of his mission as set out in the job description.

The expert carried out the following functions:-

- i) Evaluated the Olfactory Quality of the essential oils being currently produced by CSD i.e. Clove Stem Oil and Clove Bud oil.
- ii) Advised on methods to upgrade the quality of the Clove Bud oil for use in Perfumery.
- iii) Evaluated the potential of usage and odour quality of the other essential oils that can be distilled from the raw materials available in Pemba.
- iv) Evaluated the potential of the essential oils being presently produced for the preparation of aroma chemicals and distillates.

- v) Trained Counterpart staff in methods of sensory evaluation,
 quality assessment and blending.
- vi) Rocommended further requirements for the improvement of essential oils for use in perfumery.

Findings, Observations and Work Performed.

Evaluation of Olfactory Quality of Essential Oils: Clove Bud
 Oil and Clove Stem Oil.

A large number of samples of essential oils such as Clove Stem oil and Clove Bud oil which are currently being produced by CSD were Olfactorily evaluated.

For details see annexure 6

It was found that in the distillation of Clove Bud oil and Clove Stem Oil, a substantial part of the oil was being lost (i.e. was not being collected) through the so-called settling tanks where the water of distillation mixed with oil was taken for settling. The percentage of oil so lost was 10 - 15% for Clove Bud oil and 15 - 20% for Clove Stem oil, as per the figures given by the production foreman.

Further after the distillation was over, the distilled oil which has moisture in it, was taken for purification under vacuum and the complete moisture plus about 0.1% of the essential oil was distilled and collected.

It was found that this small portion of the oil had the typical odour of Clove Buds and the purified oil as was being currently produced was deficient in this vital odour, the same being relatively flat and characterless. The oils obtained from the settling tank as mentioned above and from the purification were found to be black in colour and were examined Olfactorily and their GC analyses were studied.

It was found that the Black oil obtained from settling tanks was rich is Eugenol and Eugenol Acetate and also had the typical fruity odour associated with Clove Buds.

Further it was observed that the Black oil obtained from the Purification process had even a more intense typically sweet spicy Clove character and was rich in the low boilers which are reponsible for the top note of the Clove Bud oil.

It was found that by combining the 0.1% low boiling fraction obtained during purification with the purified oil after separation of mositure gave an olfactorily acceptable and complete oil representative of Clove Buds, which was in terms of odour better than the regular purified oil.

Further the results of an experiment conducted earlier in the CSD laboratory were studied in detail.

Clove Buds were distilled in the laboratory and instead of all the oil being collected as a single fraction, it was collected in four fractions successively obtained. It was found that the last fraction which constituted 1% of the total oil content was rich in primarily caryophyllene. As such if this fraction was separated and the first three fractions were taken as Clove Bud Oil, then the olfactory quality of the oil became quite comparable to the Madagascar quality which is the desired and result. Also in terms of chemical analysis, the quality got

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upgraded to the standard accepted international quality. This was confirmed by the results of GLC analysis conducted with such a sample.

A sample of Clove Bud Oil of a production lot from the factory was examined for olfactory quality and the same was found to be inferior in its typical clove character to the oil sample made by mixing the top fraction obtained in purification with the normal factory oil.

A sample of the Black oil obtained from the settling tanks was checked for odour and chemical nature. The odour was found to be similar to the typical clove odour and in analysis the Eugenol content was high and the caryophyllene was low. It was found that by adding this fraction back to the main oil, the Eugenol content could be raised and the Caryophyllene content lowered, so as to obtain a Clove Oil of a better specification.

2. Upgradation of Quality of Clove Bud Oil for use in Perfumery

It was found that Clove Bud oil being distilled in CSD was lacking in the typical top note characteristic of Clove Buds. It was found that two top fractions were being separated during distillation. These were first the top fraction rich in Eugenol being obtained from the settling tanks and second the small top fraction being obtained during the moisture removal stage.

Experiments were conducted and it was found that by mixing these fractions back, the quality could be upgraded.

These fractions have the typical sweet spicy and fruity character of Clove Buds and hence the result.

It was found again by virtue of experiments conducted that the last fraction obtained during the distillation of Clove Bud oil, which was 1% of the total oil content was poor in Eugenol content and high in Caryophyllene content. Since Caryophyllene an odour which is some what woody and greenish, it is has contrary to the odour of Clove Buds and excessive Caryophyllene content results is a Clove Bud oil of poor olfactory quality. It found that by separating this fraction and by not mixing it was with the main oil both the olfactory quality as well as the Eugenol and Acetyl Eugenol contents improved and such an oil was well within the international specifications used in the industry.

It was found that this last fraction could be fractionated further to obtain Acetyl Eugenol and Caryophyllene. Caryophyllene can be used to prepare derivatives like the acetate and the alcohol which are used in perfumery. The Eugencl/Acetyl Eugenol fraction can be used to upgrade and adjust qualities during the course of normal production.

In order to investigate further methods for upgrading the Clove Bud Oil quality, four separate distillations were conducted in the laboratory. The Clove Buds used in these distillations were of Grade I, Grade II, Grade III and Grade IV (normal distillation grade). Grades I, II and III are gradations done as per ZSTC standards and were uptill now not offered for distillation to CSD, being exclusively reserved for exports. It was found that with the fall in exports such grades could also be made available for distillation. Accordingly 4 oils were obtained from the 4 grades of cloves and were olfactorily examined. It was found that Grade I and Grade II were olfactorily much better than

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the normal oil and as such formed two superior grades. Grade III and IV were simlar to each other and like the normal oil distilled.

As a result of the analysis of the essential oils distilled from the four grades of cloves mentioned above it was seen that grades I and II which represented the two superior grades of cloves had typically a very high percentage of Acetyl eugenol and low percentage of Caryophyllene. Similarly grades III and IV which represented the lower grades of clove had low percentages of Acetyl eugenol.

Since the typical odour of clove bud oil as represented by the Madagascar quality, is due to high Acetyl eugenol content, the quality of clove bud oil was manipulated by making a predetermined mix of grades I, II, III and IV such that the desired amount of Acetyl eugenol was obtained in the distilled oil.

This mix of grades can be standarised by first distilling a sample lot in laboratory and then taking the production lot in the same proporation.

By this method variations in quality due to variations in the raw material can be controlled and a product of standard specification can be produced on a repetitive basis.

The fundamental principal of quality upgradation and standardisation is that either raw material is to be manipulated or the distillation process is to be suitably controlled so as to give a product of the desired quality.

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It was found that the main buyer of the CSD was a UK dealer called John Kellys (London) Ltd, Prescot House, Prescot Street, London. This buyer was not entirely happy with the quality being supplied and as such had submitted a typical type sample of Madagascar Clove Bud Oil for matching.

This sample was examined olfactorily and it was found that the odour of this sample was similar to the sample prepared after mixing the top fraction obtained in purification back into the main oil. However the colour was slightly lighter and the Caryophyllene note was subdued. After examining this sample, the results of all the experiments were checked and it was found that John Kelly's sample could be matched by following any one of the under mentioned alternatives:-

i) a) During the distillation of normal distillation grade Cloves, the last fraction which is Caryophyllene rich should be isolated. The rest of the fractions obtained should be bulked together as Clove Bud oil.

b) The Clove Bud oil obtained above should be purified in the normal manner and moisture removed. The top fraction of oil which is removed alongwith the water should be isolated and collected. This should be subjected to flash distillation and the colourless or pale yellow product thus obtained should be added back into the purified Clove Bud Oil.

This procedure will yield an oil which will have high Eugenol, high acetyl eugenol and low caryophyllene contents. Olfactorily this oil will be as good as the best Madagascar grades.

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ii) The second alternative is to take the best quality cloves ie the Grade I Cloves which have the best flavour and fragrance and distill them. This will yield an oil which will be rich in Acetyl Eugenol and Eugenol and low in Caryophyllene.

However the efficacy of this method will depend upon the efficiency of distillation and at the moment the same suffers from a major draw back. It was found that nearly 12 - 15% of the oil which was Eugenol rich with low Caryophyllene was being lost alongwith the water of distillation as the separators and the settling tanks being used were simply inadequate to cope with the volume of the distillate. Further it was found that there is no provision for distillation using the Cohobation technique which is absolutely essential especially with oils that have both a lighter than and heavier than water fraction. It was found that the loss of the eugenol rich - Caryophyllene poor fraction was enough to make the percentage of Caryophyllene in the final oil higher then the desired amount and hence the oil poorer in odour.

3. Evaluation of the Odour Quality and Potential of Usage of Other Essential Oils whose Raw Material is available in Pemba.

It was found that Pemba was rich in diverse flora and there were many essential oil bearing plants growing wild in Pemba. The most important of these were examined and small quantities of essential oils distilled in the laboratory.

The results of the Olfactory evaluation are as under:-

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a). Bitter Orange Oil: The Bitter Orange tree grows wild in Pemba and is widely found in the forest areas. It was found that controlled harvesting could yield sufficent material for distillation to produce the oil in commercial quantities. The oil from the leaves and twigs of the tree which in commercial parlance is known as Petitgrain Oil was distilled in the laboratory.

It was found the odour was typically fresh green citrus and powerful and compared very favourably with the commercially available Petitgrain oil of Paraguay which is the only source for this oil at present.

b. Cardamom Oil : The cardamom plants grows very well on the island of Pemba in the shade of the Clove trees.

One sample of the oil distilled from the local material was available and the same was examined. The Cardamom oil distilled in CSD was found to have a typical fresh green some what minty Cineole like odour. The odour although not comparable to the best Indian Oil which is the standard used world wide, was yet, within acceptable limits. It was found that Cardamom was not yet being cultivated extensively and only trial plots had been planted. The small quantity produced was consumed locally as a spice.

c. Ylang Oil : A number of samples distilled in the laboratory were examined for Olfactory quality.

It was found that the Ylang tree is widely distributed over the island but as on date no effort had been made to exploit the oil commercially.

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It was found that although the oil was genuine and pure, in olfactory terms its odour profile was incomplete indicating either incomplete distillation or poor quality of the material distilled. Upon enquiry it was found that flowers which had naturally fallen to the ground were being distilled whereas actually fresh flowers should plucked early in the morning and distilled immediately so as to prevent fermantation and yield the best quality oil. Further as indicated by the odour analysis, it was found that complete distillation had not been carried out and as such a relatively poor quality oil had resulted. It was found however that the type and variety of Ylang available is good and acceptable and with suitable controls, a world class quality which was olfactorily acceptable could be produced.

d. Ocimum sauve : It was found that this plant grows wild all over the island and large quantities of material could become available upon harvesting the same.

Samples were distilled in the laboratory and upon olfactory evaluation it was found that the oil of Ocimum sauve had a very clean spicy clove like odour with a sweet floral character. Upon experimentation, it was found that this oil could be used very well in the modern spicy - oriental fragrances especially those designed for men.

e. Oleoresin Clove Bud : It was found that distillation grade cloves had been used to prepare the oleoresin Clove by solvent extraction.

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Upon organoleptic evaluation of these products it was found that the product was not truly representative of cloves because of the following reasons:-

- i) There were traces of solvent odour in the samples.
- ii) The samples lacked the typical odour and flavour of cloves although the taste was present.

Upon enquiry, it was determined that the usual and standard method of preparing oleoresins had not been followed in which the exhausted cloves i.e. after the distillation of essential oil are solvent extracted to extract the taste bearing resinous material and to this a predetermined quantity of the distilled essential oil is added back to obtain a standardised oleoresin which can be defined in terms of the essential oil content.

It was found that by following this method, a good quality product which was acceptable on organaleptic evaluation could be produced.

f. Lemongrass oil West Indian : A sample of the oil from the bulk lot was examined for olfactory quality. It was found that the quality of the oil was very good being typically fresh citrus lemon like, free from the grassy overtones of the East Indian quality due the presence of Methyl Heptinone in the East Indian material.

It was found that this plant grew very well in Pemba and it was possible to extend the cultivation without any major problem.

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G. Cinnamon Leaf Oil : It was found that 2-3 acres of Cinnamon had been planted on the ZSTC/CSD farm about 2 years ago. The plant growth was a little below normal because of the dry climate in that particular part of the island and also the plants had become diseased. Further it was found that coppicing was not being carried out and as such the condition of the plants was less then ideal.

A part of the leaves collected from the farm had been distilled in the lab and the same was evaluated for olfactory quality.

The Cinnamom Leaf Oil was found to be of a good quality comparable to the Sri Lankan Oil and olfactorily quite acceptable.

h. Vetiver Oil : It was found that Vetiver grew very well in the climatic conditions of Pemba especially in the areas with sandy soil.

A sample of the oil distilled from the local vertiver roots was examined for its olfactory characteristics.

It was found that the oil that had been obtained from the local vetiver roots was typically fresh, woody, earthy and sweet quite reminiscent of the North Indian type of vetivert which is considered the best quality of vetivert in the world but is not available in commercial quantities and exists only in the wild. It was estimated that this oil of considerably superior odour profile would be able to fetch a premium in the world market currently supplied be Indonesia and Haiti.

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It was found that a Pilot-scale Distillation still had already been ordered for the further distillation of Vetiver oil.

i. Artemisia camphorata : It was found that this species of Artemisia is indigenous to Pemba and has traditionally been used as an Aromatic herb. The essential cil distilled in the laboratory from this plant was examined and it was found that it has a very sweet-fruity-strong camphoraceous odour which is quite unique and suitable for use in fragrances provided the material is made available in commerical quantities.

Apart from the essential oil, the concrete and absolute of Artemesia Camphorata had been prepared in the laboratory from the fresh leaves of the plant. Upon clfactory examination it was found that the solvent extracted product was quite different in its odour characteristics from the volatile oil. The concrete and the absolute were spicy and camphoraceous but lacked the sweet fruity fragrant aspect of the distilled oil. It was found that a more useful product could be prepared by solvent extracting the exhausted leaves (i.e. after the distillation of essential oil) give a relatively neutral resinous material which can be used to such or some volafile oil can be added back to give an **8** S aromatic absolute/concrete, such that it has the desirable odour of the essential oil and the fixative power of an absolute/concrete/resinoid.

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4. Evaluation of the Potential of the Essential Oils being presently produced for the preparation of Aroma Chemicals and Distillates

It was found that at present, the following essentials having a potential for being processed for the production of Aroma Chemicals and Di tillates are being produced at CSD:-

- 1. Clove Bud Dil.
- 2. Clove Stem Dil.
- 3. Lemongrass Oil.

It was determined after observation of the existing operations that during the distillation of Clove Bud oil and Clove Stem Oil, a by-product of similar nature was being obtained from both the distillations. This was called the Black Oil and infact represented the oil lost as an emulsion with water during the course of the distillation. Since the Clove oils contain fractions which are heavier as well as lighter than water and also because the specific gravity of Eugenol which is the main constituent of Clove Oils is very close to that of water, a substantial part of the oil dissolves in the water of distillation, turns black during storage and is subsequently recovered by settling the oil/water emulsion in settling tanks.

Utilisation of Black Oil : It was found that the Black oil obtained during the distillation of Clove Bud oil and Clove Stem Oil was very rich in Eugenol containing nearly 90% of the same. Since this oil is dark in colcur, it has to be redistilled or rectified before it can be added to the main bulk of the oil. The other alternative is to utilise the oil to produce Isolates and Derivatives and in the process engage in diversification and production of value added products.

The main Aroma Chemicals which can be produced from this Black oil are as under:-

- 1. Eugenol
- 2. Isoeugenol
- 3. Acetyl Eugenol
- 4. Acetyl Isoeugenol
- 5. Methyl Eugenol
- 6. Methyl Isoeugenol
- 7. Benzyl Isoeugenol

It was explained to the project authorities that normally Clove Leaf Oil which is the cheapest Clove Oil is used for the production of these derivatives and as such by normal commerical considerations, it would not be economical or feasible to process the Clove Bud and Clove Stem Oils to produce isolates and derivatives. In the case of the Black oil which is a by product, it would be more disirable to rectify/redistill the same and utilse it to up grade the quality of Clove Bud oil and Clove Stems Oil.

However, purely from the olfactory point of view, eugenol produced from the Black Oil obtained from Clove Bud Oil and Clove Stem Oil would be superior is to the normal eugenol prepared from Clove Leaf Oil. Upon olfactory examination it was found that indeed the Eugenol obtained from Clove Stem oil and Clove Bud Oil was superior in odour being more sweet, fruity and typically clove bud like. The other derivatives such Isoeugenol and Acetyl Eugenol were also examined but from the olfactory and therefore the perfumer's point of view, no special characteristics in the odour could be discerned and as such it was found that if isolates and derivatives were to be prepared then the most suitable item would be Eugenol which could then be prepared in two grades as follows:-

- i) Eugenol ex Clove Stem Oil
- ii) Eugenol ex Clove Bud Oil

and sold as an olfactorily superior product as compared to Eugenol ex Clove Leaf Oil which is the normal commerical product.

Utilisation of Lemongrass Oil :- It was found that the West Indian type of Lemongrass oil was being cultivated in Pemba and that it had high Citral content (75%). It was also seen that the total quantity being produced, around 600 - 1000 kilos per annumn was not such that it could be sold as such since lemongrass oil is sold in large bulk with customers buying 5 M/T or 10 M/T at a time at least. Since this kind of quantity was not foreseen to be produced the only viable alternative to utlise the lemongrass oil was to make value added products i.e. Isolates and Derivatives mainly the following:-

1. Ionone Fure

- 2. Ionone Alpha
- 3. Methyl Ionone

These materials could then be sold in the international market, alongwith the Eugenol ex Clove Siem oil and Clove Bud Oil. 5. Training of Counterpart Staff in Methods of Sensory Evaluation, Quality Assessment and Blending :

It was found that members of the project team had no training or exposure to the fragrance and flavour industries. Accordingly, the principles and practices involved in the manufacture of fragrances and flavours, sensory evaluation and quality assessment were explained and demonstrated to the counterpart staff using the samples carried by the Expert especially for the purpose from India at his own cost.

It was found that proper equipment for setting up a Quality Assessment and Sensory Evaluation Laboratory was not available and as such a comprehensive list of the equipment required for this purpose was prepared and handed over to project authorities to enable them to procure the same.

Conduct of Traning Course:

It was found that the project team had no training in the techniques used for Sensory Evaluation, Quality Assessment and Blending.

As such to rectify this defficiency, a training course was conducted for the project team which covered the following topics:-

- a) Defination of Fragrances and Flavours
- b) Uses of Fragrances and Flavours
- c) Classification of Fragrances and Flavours
- d) Raw Materials used in the Fragrance and Flavour Industries both natural and synthetic.

e) Proper Methods of Odour Evaluation.

- f) Terminology used in the fragrance and flavour industries.
- combinations and Accords used in the fragrance and flavour industries.
- h) Production of fragrances and flavours
- i) Use of odour evaluation ability to do compounding and blending as well as quality control

Establishment of Laboratory for Olfactory Assessment and Sensory Evaluation.

It was found that the basic pre-requisite for doing sensory evaluation work in the field of essential oils, aroma chemicals, fragrances and flavours by way of a proper laboratory did not exist in the project. The importance of having such a facility was explained in great detail as also the method of creating the same alongwith full details of the procedures to be followed and equipment to be followed.

Glossary of Terms used in the Industry

It was found that the project team had little experience in dealing with the fragrance and flavour industry on an international basis and consequently they were generally unaware of the language of perfumery or the technical and/or commerical terms used within the industry.

It was found that because of this reason, the project team was not in a position to fully comprehend the technical literature relating to the industry and to communicate with trade meaningfully. To reactify this a full glocsery of terms used in the fragrance and flavour industry was prepared and provided to the project team.

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

1. It was concluded that for production of olfactorily acceptable qualities of the essential oils of Clove Bud and Clove Stem, it was necessary that the oil being lost with the water of distillation should be collected either by settling or by the introduction of cohobation techniques.

2. It was concluded that the oil obtained as above by settling was critical for the olfactory quality of essential oils of clove bud and clove stem because it was rich in eugenol content and poor in caryophyllene and its subtraction from the main oil resulted in the relatively harsh odour of the Zanzibar oil.

3. It was concluded that the fraction of essential oil obtained during the moisture removal stage was also very critical for the odour quality of the clove bud and clove stem oils as this fraction contained the low boilers which are responsible for the typical clove odour.

4. It was concluded that to match the Madagascar quality of clove bud oil from the olfactory as well as the chemical analysis point of view, it would be necessary to combine with the main oil the oil recovered from the settling tanks as well as the oil fraction obtained during purification.

5. It was concluded that since both the fractions of the oil described above i.e. the oil obtained from settling tanks as well as the oil recovered during the purification process, were dark in colour, it would be necessary to flash distill them so as to remove the colour before adding them to the main oil.

6. It was concluded that the vital difference between the Madagascar type clove bud oil and the Zanzibar type clove bud oil in terms of their olfactory quality was that the Madagascar oil was more sweet spicy fruity floral typically like natural clove bude whereas the Zanzibar quality was somewhat harsh in odour, lacking the sweet fruity character typical of natural clove buds.

7. It was concluded that the olfactory quality of clove bud oil as also dependent on the nature of the fractions obtained during the distillation process. Upon examination of the various fractions obtained, it was further concluded that the end fractions obtained during the distillation were rich in caryophyllene and therefore harsh in odour and that such fractions made a negative contribution to the olfactory quality of clove bud oil.

8. It was concluded that clove bud oil of a satisfactory olfactory quality could be obtained by the separation and removal of the end fractions obtained during the distillation.

9. It was concluded that the quality of clove bud oil as being currently distilled by CSD was in fact inferior to the standard Madagascar quality and as such was required to be upgraded so as to obtain a quality which would be acceptable in the international market and which would also fetch a better price.

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10. It was concluded that for the upgradation of the clove bud oil the following steps were required to be taken:-

i) black oil from settling tank should be flash distilled and added back to the main oil.

ii) dark coloured fraction obtained during purification should be flash distilled and added back to the main oil.

iii) the end fractions obtained during the distillation should be separated and removed.

iv) for overall improvement in the quality of the clove bud oil distilled, grade I and grade II cloves should be used as compared to the grade IV which is normally used. Ideally a mix of grades I, II, III and IV should be made and test distilled in the laboratory to confirm the specification of the resultant oil. Upon the Acetyl eugenol, Eugenol and Caryophyllene contents being found to be as per desired specification, the bulk lot for distillation can be mixed in the same proporation for the production batch.

11. It was concluded that from the operational point of view the capacity of the settling tanks available in conjunction with the distillation units was too small and as such a large part of the water of distillation alongwith the dissolved/emulsified oil simply overflowed in to the drain and as such the oil was lost.

It was therefore concluded that there should the one settling tank for each distillation unit so that the water of distillation from each unit can be collected separately and the oil collected therefrom after settling.

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12. It was concluded that it was essential to introduce cohobation techniques in the distillation of clove bud oil and clove stem oil so as to prevent the substantial loss of oil currently taking place through the settling tanks.

13. On the strength of evaluation of odour quality and potential of usage it was concluded that the following essential oils should be taken up for regular commericial production:-

i) Bitter Orange Oil

ii) Ylang Oil

iii) Ocimum sauve oil

iv) Lemongrass [i]

v) Cinnamon Leaf Oil

vi) Vetiver Oil

vii) Artemisia camphorata oil

14. On the st-ength of the evaluation of the potentia⁻, it was concluded that the following essential oils should be further exploited for the production of aroma chemicals and distillates:-

i) Clove Bud Oil Black Oil from settling tank

ii) Clove Ster Oil Black uil from settling tank

iii) Lemongrass Oil

to produce the following aroma chemicals:-

i) Eugenol

ii) Iso Eugerol

iii) Acetyl Eugenol

iv) Acetyl Isseugenol

v) Methyl Eugenol

vi) Methyl Isteugenol

- vii) Benzyl Isoeugenol
- viii) Ionone Pure
- ix) Ionone Alpha

x) Nethyl Ionone

15. Upon interaction with the counterpart staff, it was concluded that extensive training in and exposure to the fragrance and flavour industries was required to be given to them so as to enable them to understand the principles and practices involved in the manufacture of fragrance and flavours, sensory evaluation and quality assessment.

16. It was concluded that a proper quality assessment and sensory evaluation laboratory was required to be set up to enable the project staff to properly evaluate the essential oils and aroma chemicals produced by CSD.

RECOMMENDATIONS

1. It is recommended that for the production of olfactorily acceptable qualities of the essential oils of clove bud and clove stem, the oil being lost with the water of distillation should be collected either by settling or by the introduction cohobation techniques.

2. It is recommended that in order to match the Madagascar quality of Clove bud oil from the olfactory as well as the chemical analysis point of view the oil recovered from settling tanks as well as the fraction obtained during purification shc_ld be combined with the main oil.

3. It is recommended that both the oil obtained from setting tanks as well as the cil recovered during the purification process should be flash distilled so as to remove the colour.

4. It is recommended that the end fractions obtained during the distillation of clove bud oil should be separated and removed so as to upgrade the olfactory quality of the balance oil.

5. It is recommended that steps should be taken to suitably modify the existing production procedures in line with the findings of this mission so as to upgrade the Zanzibar quality to make it equivalent to the Madagascar quality and therefore more acceptable in the international market.

6. It is recommended that the following steps be taken to upgrade the quality of clove bud oil being distilled by CSD:-

i) black oil from settling tank should be flash distilled and added back to the main oil.

ii) dark coloured fraction obtained during purification shouldbe flash distilled and added back to the main oil.

iii) the end fractions obtained during the distillation should be separated and removed.

iv) for overall improvement in the quality of the clove bud oil distilled, grade I and grade II cloves should be used as compared to the grade IV which is normally used.

7. It is recommended that the number of settling tanks available for separation of dissolved/emulsified oil in the water of distillation should be increased such that one tank should become available for each distillation unit.

8. It is recommended that cohobation be introduced in the distillation of clove bud oil and clove stem oil so as to maximise the yield of essential oil and prevent loss of oil through dissolution/emulsion in the waters of distillation.

9. It is recommended on the basis of odour evaluation and potential of usage that the following essential oils be taken up for production for sale in the international markets:-

- i) Bitter orange oil
- ii) Ylang oil
- iii) Ocimum sauve oil
- iv) Lemongrass oil
- v) Cinnamon leaf oil

vi) Vetiver oil

vii) Artemisia camphorata oil

10. It is recommended that the following essential oils should be further exploited for the production of aroma chemicals and distillates:-

i) Clove Bud Oil Black oil from settling tank
ii) Clove Stem Oil.... Black oil from settling tank
iii) Lemongrass oil

to produce the following aroma chemicals:-

- i) Eugenol
- ii) Iso Eugenol
- iii) Acetyl Eugenol
- iv) Acetyl Isoeugenol
- v) Methyl Eugenol
- vi) Methyl Isoeugenol
- vii) Benzyl Isoeugenol
- viii) Ionone pure
- ix) Ionone Alpha
- x) Methyl Ionone

11. It is recommended that extensive training in and exposure to the fragrance and flavour industries should be given to the counterpart staff to enable then to understand the principals and practices involved in the manufacture of fragrance and flavours, sensory evaluation and quality assessment. 12. It is recommended that a proper quality assessment and sensory evaluation laboratory be set up to enable the project staff to properly evaluate the essential oils and aroma chemicals produced by CSD.

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UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

13 September 1991

JOB DESCRIPTION DP/URT/86/026/11-55

Post Title: Consultant in Perfumery

Duration: 0.5 v/m

Date Lequired: November 1991

Duty Station: Chake, Chake, Perba

Purpose of Project: Maximising the capacity of the Clove Distillery in Chake, Chake

Duties: The expert is expected to carry out the following in collaboration with the CTA and the counterpart staff.

i) Evaluate the potential of the essential oils presently being produced for the preparation of aroma chemicals and distillates.

ii) Advise on methods to upgrade the quality of the clove oil for use in perfumery.

111) Train counterpart staff on methods of sensory evaluation, quality assessment and blending.

iv) Recommend further requirements for the improvement of essential oils for use in perfumery.

After the end of the mission the expert will be required to present to UNIDO a fully prepared report embodying his findings and recommendations.

Qualifications: A graduate in Commistry with considerable experience in Perfumery, Fragrance formulation and sensory methods of analysis.

Language: English

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Background Information: Cloves (<u>Eugenia caryophyllata</u>) are the major crop in the Zanzibar Islands, which include Unguja and Pemba and are traditionally called the Clove Islands.

There are two major facilities for the distillation of clove buds and clove stem under the control of the Zanzibar State Trading Company (ZSTC). One is an almost obsolete plant, nevertheless with a considerable production capacity situated at Malindi in Unguja Island. The other is a modern plant at Chake Chake in Pemba Island.

In Pemba here is an established capacity but ensurement of maintenance is important.

Attuned to the distillation capacity of the plant the ensurement of timely collection and organized drying of raw materials is crucial.

The Government expects UNIDO to provide support to enhance the economic, commercial and technological structure to modernise the industry in Zanzibar.

ANNEXURE 2

CONDUCT OF TRAINING COURSE FOR TECHNICIANS OF CSD

The course was attended by the following personnel of CSD:-

- 1. Slim Rashid Juma
- 2. Khamis Mwinyi Khamis
- 3. Abdalla Suléiman Haji
- 4. Issa Hassan Khatib
- 5. Mussa Hamad Bakar

The details of the topics covered were as follows:-

- 1. Fragrance Industrial
 - (a) <u>Definition</u>: Any mixture of two or more odouriferous substances, of a type used in industry.
 - (i) Of a type used in food FLAVOUR (ii) All others FRAGRANCES

Thus it was explained that for the purposes of the programme which was mainly concerned with the industrial use of fragrances, it was necessary to abide by the aforementioned definition which is now used internationally as per the Brussels Trade Nomenclature.

(b) <u>Uses</u>: Since the programme was concerned with the use of industrial fragrances, the possible use of such fragrances were explained and are listed below:-

(i) Household Products

Soaps and Detergents Cleaners Disinfectants Polishes Paints Adhesives Air Fresheners

(ii) <u>Personal products</u>

Cosmetics : Make up products Toilet and Beauty preparations Perfumes and Toilet Waters

(iii)<u>Industrial Products</u>

Dry cleaning Leather & Rubber Articles Artificial Leather Linoleum Plastics Printing inks, Perfumed Board & Paper Textiles

(iv) Agricultural Products

Insecticides Insect and Animal Repellents Animal Baits & Attractants Veterinary Products Cattle Feeds

(c) <u>Classification</u>

After detailing the uses of various fragrances, the major classes/categories into which fragrances can be divided were described and the same are detailed below:-

> Green Fresh 1. Green Balsamic 2. Fruity Fresh 3. Floral Fresh 4. 5. Floral Rose 6. Jasmine 7. Floral - sweet (Lilac) Aldehydic - Floral 8. Aldehydic - Floral-woody-powdery 9. 10. Fresh - Mossy - Aldehydic (CHYPRE) Floral - Mossy - Animalic (CHYPRE) 11. Mcssy - Fruity (CHYPRE) 12. Oriental 13. Leather 14. 15. Fougere 16. Citrus - classic 17. Citrus - Fixed 18. Cool - Floral 19. Lavender Notes Spicy Notes 20. 21. Woody Musk Notes 22.

2. Raw Materials used in Fragrance Industry

The variety of raw materials used in the fragrance industry was discussed and the diversity of sources from which these raw materials are derived was discussed and explained. The technical classification of raw materials was also discussed and explained.

3. Proper Methods of Odour Evaluation

The importance of precise and correct odour and Sensory evaluation in the industry was discussed in detail. The importance thereof in industry from the technical as well as the commercial points of view was explained.

4. <u>Terminology used in Fragrance/Flavour Industries</u>

The technical and commercial terms used in the international perfumery industry were explained and a full glossary of the terms used was prepared and explained.

5. <u>Combinations and Accords used in Fragrance &</u> <u>Flavour Industries</u>

The formulations used to produce fragrances as per the classification mentioned above were discussed and typical formulas for each category were provided.

5. Production of Fragrances and Flavours

The practical procedures used in production, the correct withodology for the same, the equipment required and the precautions to be taken were explained and discussed in detail.

7. Use of Odour Evaluation Ability to do Compounding and Blending as well as Quality Control

Olfactory assessment and instrumental quality control methods and their technical and commercial importance was explained. The methodology of selection of a fragrance for any particular applications and the technical and commercial considerations involved were explained.

Concepts of standardization as well as custom production were explained.

METHOD FOR CREATION OF AN OLFACTORY ASSESSMENT AND SENSORY EVALUATION LABORATOY FOR NATURAL SYNTHETIC PERFUMERY MATERIALS.

Background:

1. Natural and synthetic perfumery materials such as essential oils, aromatic chemicals, etc, are used primarly for their odour appeal. Although the analytical characteristics which are commonly determined may provide some assurance regarding the chemical purity of an odoriferous substance, they do not necessarily indicate the "purity" of odour. Hence, olfactory evaluation has been practised for centuries and, in the perfumery trade, it has formed the basis of acceptance or rejection of odoriferous materials.

This methodology has been formulated with a view to introduce standard methods of testing for olfactory assessment of natural and synthetic perfumery materials.

2. Olfactory assessment has been the target of some criticism as it is a subjective test. Numerous attempts on basic odour research and, more particularly, on objective measurement techniques have been made from time to time but none of these has so far wide acceptence. Whereas objective methods are the goal of all odour research, there is, at present, no technique which may replace sensory detection and evaluation of odours.

Terminology

1. Top note: The initial and primary odour effect perceived by the olfactory nerves on smelling a strip freshly impregnated with the material being tested. The top note(s) is (are) usually of a short duriation and may or may not be coperceived along with the middle note.

2. Middle note: The secondary overall odour effect experienced by the olfactory nerves on smelling a strip impregnated with the material after the initial top note has evaporated. It lasts for a longer time on the strip than the top note.

3. Residual note (Dry-out Note): The tertiary odour effect experienced by olfactory nerves on smelling a strip impregnated with a material after the top and the middle notes have disappeared. Besides indicating the lasting character and strength of the material, it may also reveal the nature of the lesser volatile materials.

4. By note: An odour effect, additional to the normal pattern of odours associated with the material, experienced by olfactory nerves on smelling an impregnated strip during any stage of evaporation. It is generally regarded as an index of foreign odour and/or undesirable adulterant and alien.

5. Odour Description: Due to the absence of precise terms, descriptive words which are subjective in nature are commonly used to express the odour sensations perceived in the top, middle, residual and by-notes. Some of these terms are given below but the list is not intended to be exhaustive:

acid acrid aldehydic amber animal balsamic bitter burnt camphoraceous choking citrus cloying cool dry dull earthy exalting faccal fatty fishy floral fungal fresh fruity goaty grassy green heavy herbal honey intense leafy leathery minty mossy mushroomy musky musty nauscating nutty oriental peppery persistent

phenolic piney powdery pungent refreshing sappy sharp sickly smokey SOUT spicy stemlike still odour sulphuraceous sultry sweet tarry tart woody

Requirements

General Requirements: The following general precautions are required to be noted.

Selection and Training:Better results are obtained if individuals with a keen sense of ll and ability to distinguish between different odours are selected for training in ctory assessment.

gue: Continuous smelling causes olfactory fatigue and decreases critical odour eption. To avoid this, the number of samples assessed during a session should be ted as far as is practical. Further, during smelling, the body should be relaxed. Resting in interval between smelling different samples is also advantageous. If the number of ples to be tested is fairly large, it is advisable to examine last those materials which are wn to be pungent or strong in odour.

t should be borne in mind that inability to correctly identify certain odours may arise 1 natural deficiencies such as specific anosmia. For instance, some people are unable to eive musky odour.

lias: The necessity of minimizing all differences between samples other than that of ir in order to prevent the prejudicing of results is stressed. 'Blind' tests should be ucted by ensuring that the markings on the smelling strip do not disclose the origin of amples.

"ime of Olfactory Assessment: The evidence relating to the most favourable time for ucting olfactory assessment is somewhat conflicting. However, the morning appears e generally favoured. In general, olfactory assessment should be done after a mable interval of time has elapsed after a meal or a beverage has been taken.

reedom from Contaminating Odours: It is necessary to ensure that the hands, nose smelling strips arc free from contaminating odours as these are likely to vitiate the ts. It is recommended that the individual responsible for assessing odour should wash er hands several times during a smelling session as well as clear his /her nose.

faterial Requirements: The following materials, apparatus and environmental itions are required.

ibrary of Standard Samples: For each essential oil, aromatic chemical or other imery material, there shall be a standard sample of approved odour value.

he standard samples shall be kept in well-stoppered, air-tight, neutral amber-coloured bottles and when not in use, they shall be stored in a refrigerator at about 5°C.

he odour characteristics of standard samples are likely to change over a period of time ver well they may be stored. Some materials improve in odour as a result of maturing others deteriorate because of minute oxidative changes. An alteration in the odour acteristics of standard samples is not desirable and, in such cases, fresh standards ld be adapted. Generally, all perfumery materials recommended shelf life and the le should be changed thereafter.

thyl alcohol: Perfumery grade.

viethyl Phthalate: Perfumery grade.

melling strips: These shall preferably be 1 cm wide and 15 cm long. They shall be : from odourless, thin, absorbent paper and shall be sufficiently stiff so that the strips >t bend under their own weight when held in a horizontal position.

bsorbent paper of substance ranging from 100 to 280 g/m² is commonly used. Paper de entirely from the best cotton material, and is usually in the form of cotton or 'innen or a mixture of both. It should be free from any trace of chemicals. Also the water in making such paper should be pure and completely free from odours, chemicals or The paper should be neutral and should have been kept away from odorous materials :nvironment all the time. These considerations should be useful in evaluating the ty of the paper used for preparing smelling strips.

melling strips shall be packed in air-tight, odour-free containers and stored in a clean r-free room. Those intended for daily use shall preferably be kept in a wide-mouthed bottle covered by a beaker.

rips Stand: A cruciform patterned 3-clip stand, approximately 21 cm high, or any suitable device, to hold impregnated smelling strips.

Environment: A well-ventilated room, as free as possible from all outside disturbances. Ideally, the temperature and humidity suited are about 20°C and 80 percent RH (Relative Humidity), respectively. The colouring of the room shall be sober and the furnishing restricted. The general environment shall have a restful rather than a distracting effect.

Procedure

One end of each smelling strip shall be clearly marked before use. Dip the unmarked end of one strip (about 0.5 to 1.0 cm) in the material under examination and of another strip to the same depth in the standard sample after it has attained room temperature. For certain perfumery materials, such as fatty aldehydes, absolutes and solids, use 1 to 10 percent solutions in ethyl alcohol or diethyl phthalate for olfactory assessment.

For semi-solids, solids and strong-smelling substances, use the procedure as given below.

For semi-solid materials: The odour of semi-solid materials such as guaiacwood oil, oakmoss resinoid and absolute, labdanum resinoid and absolute, etc, should be taken on smelling strips but only after melting the contents completely under controlled temperature below 100°C preferably on water-bath.

For strong smelling materials: In order to have a better perception, strong smelling substances irrespective of their physical appearance may also be smelt after dilution to about 1 to 10 percent such as indole, fatty aldehydes, etc, using ethanol or diethylphthalate as a diluent.

Hold the strip impregnated with the standard sample at such a distance from the nose that there is incipent yet distinct perception of odour. While smelling, concentrate wholly on the sensations received and make mental observations. Repeat the procedure with the strip impregnated with the test sample. After about a minute's rest, repeat the comparison reversing the order of smelling the two strips. Finally, compare the two strips for their odour in a "blind" test. If a difference in odour is observed, repeat the "blind" test on the two strips five times. Record the observations of each "blind" test.

It is important to note that although the room shall be well-ventilated, the strips kept under examination should not be exposed to a direct draught.

After this initial assessment for top notes, fix the two strips on a stand keeping them sufficiently apart to avoid inter-contamination. Examine the strips periodically by the "blind" test and note the changes in quality and intensity of odour. Continue in this manner as long as the odour on each strip remains perceptible.

Report

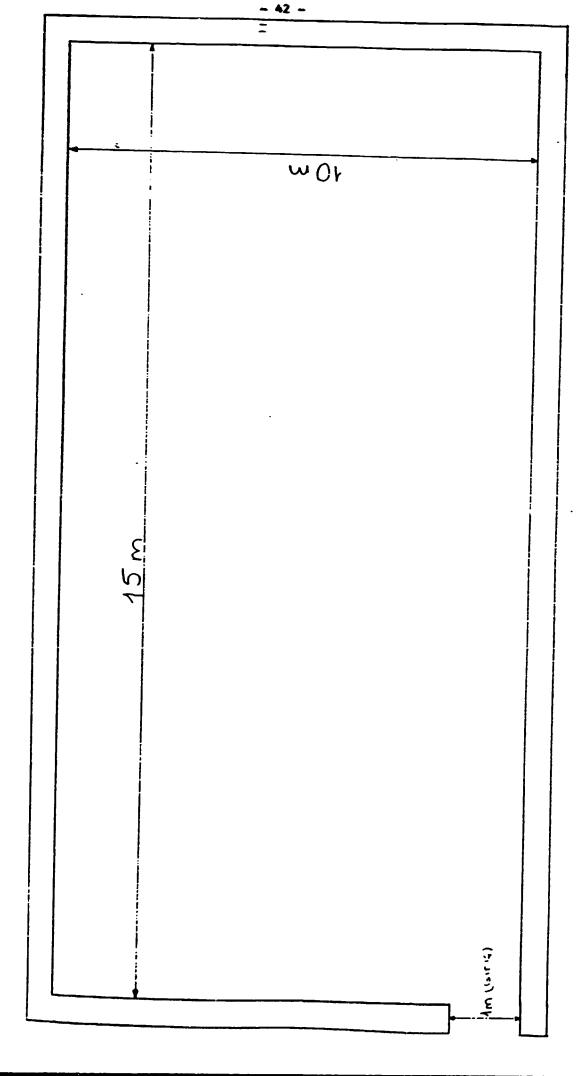
Report the top, middle and residual odour assessment of the test sample as compared the odour of the standard sample at corresponding stages of assessment.

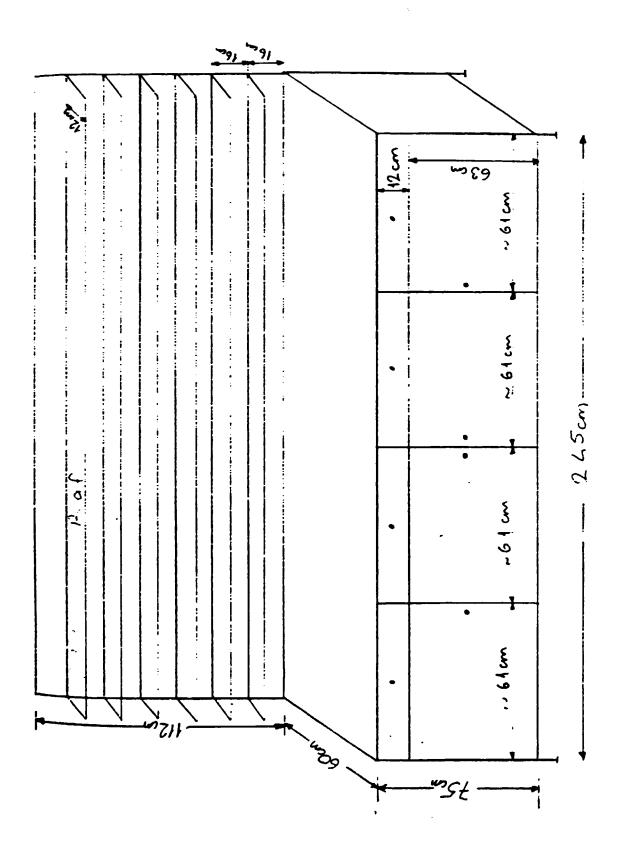
<u>Criterion for Judgement of Ouality:</u> The odour of the material under examination shall espend to that of the standard at all stages of assessment. If it does not and the pattern dour is considered to be inferior to that of the standard, the quality of the material shall egarded as not satisfactory.

<u>Referee Test</u>: In case of dispute, present the individual assessing odour with three able coded smelling strips, two of which have been dipped in the material under mination and the remaining one in the standard sample (or *vice-versa*). If the 'odd' ple is consistently picked five times in a 'blind' test, the material shall be deemed to a pattern of odour different from that of the standard sample.

TAILS OF FACILITIES REQUIRED IN THE SENSORY EVALUATION AND GRANCE CREATION LABORATORY

- 1. Refrigerated storage of standard samples of raw materials and finished products.
- 2. Samples of raw materials in bottles of proper design for daily working.
- 3. Weighing balances of accuracy to third decimal place.
- 4. Magnetic stirrer and heater.
- 5. Water bath
- 5. Working tables with shelves up the eye level.
- 7. Efficient exhaust and ventilation system
- 3. Wash basin
- 9. Conical flasks, beakers, pipettes, droppers, funnels and aluminium foil.
- 0. Detached smelling room free from all odours for odour evaluation fitted with an efficient exhaust and filtered air inlet system.







GLOSSARY OF TERMS RELATING TO NATURAL AND SYNTHETIC PERFUMERY MATERIALS

in the preparation of this glossary, most of the terms currently in use in natural and synthetic perfumery trade and industry together with their synonyms and more common terms in vogue internally and also in other countries have been included.

Terminology

- 1. Absolutes: An ethanolic extract of a concrete or a resinoid which contains the maximum concentration of odoriferous components and is free from natural waxes and/or any solvent used in the processing.
- 2. Acid Value: It is numeric value equivalent to the number of milligrams of potassium hydroxide required to neutralize the free acids present in 1 g of the material.
- 3. Alcohol Perfumery Grade, Denaturated: Rectified ethyl alcohol, specially denatured for perfumery industry, and by the addition of denaturants it thus not at any undesirable by-odours to it.
- 4. Aldehydic Blend: See 13
- 5. Amber Note: A heavy full-bodied warm ambergriss like note.
- 6. Animal Note: Odours or notes with a sensuous character.
- 7. Aromatic Chemicals/Aroma Chemicals: Organic chemicals derived by organic synthesis or as isolate from natural essential oils possessing distinct aroma. Used as raw material for the preparation of perfumery blends or flavours.
- 8. Aromatic Plants: See 92
- 9. Aromatic Water: Aqueous odoriferous condensate of hydro-distilled and/or steam-distilled material of vegetable origin containing fully dispersed essential oil.
- 10. Attar (Indian): A perfume concentrate characteristic of single flower or a mixture of flowers and/or other materials of plant or animal origin with oil of sandalwood as the base.
- 11. Balsam: An odoriferous exudate from plants/trees which flows naturally or is artificially induced by incision.
- 12. Blend: Harmonious combination of two or more odouriferous materials.
- 13. Blend Aldehydic: Blend deriving their unique character from the predominance of aldehydic notes.
- 14. Blend, Cologne: Any harmonious combination of fragrances, the main characteristics of which are derived from citrus oils.
- 15. Blend, Oriental: A blend with heavy, full-bodied sweet balsamic and animal note.
- 16. Blend, Spicy: Any fragrance combination having spicy overtone.
- 17. Blend, Woody: Any fragrance dominated by a woody character.

- 18. Body: Main fragrance theme.
- 19. Boiling Range: Sec 40
- 20. Bouquet: Generally a harmonius combination of two or floral notes.
- 21. By-Note: A temporary or permanent odour effect additional to the main pattern of odour effect additional to the main pattern of odour associated with the material.
- 22. Carbonyl Value: It is numerically equivalent to the number of milligrams of potassium hydroxide, that is, equivalent to the amount of hydroxylamine required to oximate the carbonyl compounds present in 1 g of material.
- 23. Cell: A unit of the plant tissue
- 24. Cellular: Composed of cells.
- 25. Chypre: A mossy-woody fragrance, complex with a characteristic sweet citrus top note, frequently encompassing some floral tones.
- 26. Citrus: Odours reminiscent of citrus fruits, such as orange, lemon, bergamot, grapefruit, etc.
- 27. Cologne: Name used traditionally for solution of citrus perfume blends in aqueous ethanol (also see 113).
- 28. Cologne Blend: See 14
- 29. Concentration: See 94
- 30. Concentrated Perfume: Sec 86
- 31. Concrete: A material derived from a single source of vegetable or animal origin by extraction with a suitable solvent. It generally contains non-odouriferous constituents, such as waxes, coloring matter etc, in addition to odoriferous components and is free from any solvent used in the process.
- 32. Condensate: Vapours that have been condensed.
- 33. Condenser: Part of distillation apparatus where the hot vapours are cooled and condensed for recovery.
- 34. Congealing Point: It is the maximum constant temperature at which liquefied solid resolidifies.
- 35. Deterpenized Oil: Natural essential oils which are free from terpenes and/or sesquiterpenes.
- 36. Diffusion: The ability of a fragrance to radiate and permeate the environment.
- 37. Distillation: A process of evaporation and recondensation used for purifying liquids.
- 38. Distillation, Dry: Distillation of semi-solid and solid materials in the absence of steam, water, or any other solvent.
- 39. Distillation, Hydro: Distillation of a substance carried out by indirect contact with boiling water.
- 40. Distillation Range: It is the range of temperature within which a specified percentage of the material distils.
- 41. Distillation Steam: Distillation of a substance by passing steam through it.
- 42. Distillation, Vacuum: Distillation of a substance under reduced pressure.
- 43. Distillation, Water: See 39.

- 44. Dry Distillation: Sec 38.
- 45. Dry Out: Final phase of the main fragrance after the main volatile constituents have evaporated.
- 46. Enfleurage: Process of extracting fragrance of fresh flowers by intimate contact with mixture of purified fats preferably at low temperatures.
- 47. Essential Oil: It is volatile perfumery material derived from a single source of vegetable or animal origin by a process, such as hydrodistillation, steam distillation, dry distillation or expression.
- 48. Essential Oil, Synthetic: It is a composition generally consisting of natural essential oils, aromatic chemicals, resinoids, concretes, absolutes, etc, but exluding animal or vegetable non-essential oils and not having a non volatile residue in excess of 10 percent by mass. It is so composed that it bears a close resemblance primarily in odour to a naturally occurring essential oil.
- 49. Ester Value: It is numerically equivalent to the number of milligram of potassium hydroxide required to neutralize the acids liberated by the hydrolysis of the esters present in 1 g of the material. It represents the difference between the saponification value and the acid value of the material.
- 50. Ester Value After Acetylation: It is numerically equivalent to the number of milligrams of potassium hydroxide required to neutralize the the acids liberated by the hydrolysis of 1 g of acetylated material.
- 51. Evaporation Residue: Represents the percentage of perfumery material which is not volatile when heated on a steam-bath under specified conditions.
- 52. Expression: The process of extracting essential oil from the plant cells by application of mechanical pressure.
- 53. Extract: A concentrated product obtained by treating a natural perfumery material with a solvent which is subsequently evaporated.
- 54. Extraction: The process of isolating essential oil with the help of a volatile solvent.
- 55. Extrait, Alcoholic: A French word, now universally used in perfumery, meaning an alcoholic extract of odorous parts of a pomade. It is generally used to mean alcoholic solution of a perfume concentrate.
- 56. Fixative: A substance which is compatible with and provides body and substantivity and rounds off a perfume composition by regulating the rate of evaporation of its volatile constituents.
- 57. Flavour: A combined organoleptic sensation of aroma and taste in a flavouring material is also called a flavour.
- 58. Floral: The fragrance characteristic of an existing known flower type.
- 59. Fore Runnings: Initial fractions of the distillate obtained during a distillation process.
- 60. Fougere: Perfume composition having a citrus/lavender top note with sweet powder rosaceous body with mossy/woody background.

- 61. Fractionation: The process of distillation by which an essential oil is separated into various fractions.
- 62. Fruit Flavour/Essence: Suitably blended mixtures of flavouring materials, permitted chemicals and food colours, in a solvent medium of either ethanol or the permitted non-alcoholic solvents.
- 63. Fruity Note: The impression of fruit odours within the fragrance theme.
- 64. Full Bodied: A well-rounded-out fragrance that possess depth and substantivity.
- 65. Green Note: Notes that recall fresh-cut grass, leaves and stems or other parts of plants.
- 66. Gum: A natural water soluble anionic material, often of glycoside-like structure and of high molecular mass which collects in or exudes from certain plants. It forms neutral or slightly acidic solution or a sol with water and has a typical mild odour.
- 67. Gum Resin: Natural exudation from plants and trees consisting of gums and resin with very small amounts of essential oils.
- 68. Harmonius: Order, accord and symphony in a fragrance.
- 69. Heavy: Oriental balsamic as against floral/green.
- 70. Hydro Distillation: See 39
- 71. Infusion: A process of treating a substance with water or organic solvvent
- 72. Isolate: Either a single constituent or a multi-component fraction or a composited fraction, rich in dezired odoriferous components and derived from a natiral perfumery material.
- 73. Lasting Qualities: The ability of a fragrance to retain its character over a given period of time.
- 74. Leathery Note: Any fragrance conveying the dominant characteristic of tanned leather.
- 75. Melting Point: The temperature at which the material melts and becomes liquid throughout as shown by the formation of a definite meniscus.
- 76. Melting Range: The range between temperatures at which the material begins to form droplets and at which it becomes liquid throughout.
- 77. Middle Note: The main overall odour effect experienced by olfactory nerves on smelling a strip impregnated with a material and exposed to the atmosphere for some time.
- 78. Mossy Note: The notes that recall to mind moist dark forest having moss on the trees.
- 79. Natural Perfumery Materials: Perfumery materials of natural origin.
- 80. Odour: That property of a substance which stimulates and is perceived by the olfactory sense.
- 81. Oleoresin: Exudations from tree trunks or barks of trees and are characterized by the fact that these consist of entirely or mainly resin accompanied with an essential oil in varying percentages, soluble in organic solvents.
- 82. Oleoresin Gum: An exudation from plants mainly consisting of essential oil, resin and gum.

- 83. Oleoresin, Spice: Extractables of spice having resin and essential oil obtained by solvent extraction.
- 84. Oriental Blend: Sec 15.
- 85. Perfume: A solution of perfumery compound/compounds in ethanol or other suitable solvents meant for use as a personal adornment. Here ethanol or other suitable odourless solvents are used as carriers for the fragrances.
- 86. Perfume Concentrate: A non-alcoholic concentrated perfume blend.
- 87. Perfamery Compound: A concentrated base which is further diluted with or without toning and further modifications to suit various end-uses.
- 88. Perfumery Grade Alcohol: See 3
- 89. Perfumery Material: A naturally occurring substance, or a derived material, or a preparation obtained by physical and/or chemical means, which diffuses or imparts an odour or a flavour.
- 90. Perfumery Materials, Natural: Sec 79.
- 91. Perfumery Materials, Synthetic: See 107.
- 92. Plant, Aromatic: Plant bearing a characteristic aroma.
- 93. Pomade: Refined and deodorized animal fat (s) saturated with volatile oils present in and exhaled from the flowers especially the rose and the jasmine.
- 94. Rectification: Method of separation of undesirable substance to improve the quality of the materials.
- 95. Relative Density: The ratio of density of material at 27°C to that of distilled water at 27°C or 4°C when all masses are made in air is called relative density at 27°C or 4°C. Originally, it was known as specific gravity.
- 96. Residual Note (Dry Out Note): An odour effect experienced by olfactory nerves on smelling a strip impregnated with a material and exposed to the atmosphere for a period of time when the top and the middle notes have disappeared.
- 97. Resin: Solid or semi-solid translucent exudation from trees of plants. These are soluble in organic solvents.
- 98. Resinoid: A semi-fluid or a solid material obtained from a single resinous source of vegetable or animal origin by extraction with a suitable solvent and is free from solvent used in the process.
- 99. Saponification Value: It is numerically equivalent to the number of miligrams of potassium hydroxide required to neutralize the free acids liberated by hydrolysis of the esters present in 1 g of the material. It represents the sum of acid value and ester value.
- 100.Saponification Value After Acetylation: It is numerically equivalent to the number of milligrams of potassium hydroxide required to neutralize the free acid and the acids liberated by hydrolysis of the esters present in 1 g of the acetylated product.
- 101.Sesquiterpene: Term denoting a hydrocarbon composed of one-and-a-half terpene units, a single terpene unit being equal to two isoprene units.

102.Sesquiterpeneless Oil: An isolate obtained by suitably removing the sesquiterpenes (O₁₅H₂₄) from an essential oil.

103.Specific Gravity: See 95.

104.Spice Oleoresin: Sec 83.

105.Spicy Blend: See 16.

- 106.Steam Distillation: Sec 41
- 107.Synthetic Perfumery Materials: Man-made single perfumery materials, by chemical processes.
- 108. Tail Running: The last fraction of distillate obtained in a distillation process.
- 109. Terpeneless Oil: An isolate obtained by removing almost all monoterpenes (C₁₀H₁₆) from an essential oil.
- 110.Thin: The lack of body, richness and substantivity.
- 111. Tincture: A cold alcoholic extract of the soluble part of a natural fragrant material of vegetable or animal origin, the solvent being left in the extraction as a diluent.
- 112. Tissue: Plant structure composed of cells.
- 113.Toilet Water: Sec 27.
- 114.Top Note: The first odour effect experienced by olfactory nerves on smelling a strip freshly impregnated with a perfumery material.
- 115. Vacuum Distillation: See 42.
- 116. Vacuum Distillation Residue: It is the percentage of material left behind undistilled when a known quantity of the material is distilled in vacuum at specified temperature and pressure.
- 117. Volatile: A material is said to be volatile when it has the property of evaporating at room temperature when exposed to atmosphere.
- 118.Water Distillation: See 39.
- 119.Woody Blend: Sec17.
- 120.Woody Note: The impression of wood or woody odours within the fragrance theme.

LIST	OF CHEMICALS PROVIDED TO CSD FOR SETTING UP THE SENSORY
EVALU	TIGN LABORATORY.
1.	Sandalwood Oil
	Iso Butyl Quinoline
3.	Costus Oil
4.	Geranium Oil
5.	Lavender Oil
6.	Patchouli Oil
7.	Bergamot Cil
8.	Lavandin Oil
9.	Ylang Ylang III
10.	Lemon Oil Italian
11.	Vetivert Oil
12.	Petitgrain Oil
13.	Aldehyde C-12
14.	Para Tertiary Butyl Cyclo Hexyl Acetate
15.	Aldehyde C-10
16.	Aldehyde C-8
17.	Aldehyde C-11
18.	Aldehyde C-9
19.	Aldehyde C-12 MNA
20.	Methyl Heptin Carbonate
21.	Phenyl Acetaldehyde Dimethyl Acetal
22.	Anisic Aldehyde
23.	Benzyl Phenyl Acetate

- Resinoid Irris 24.
- Cinnamic Alcohol 25.

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- 26. Hydroxy Citronellal
- 27. Phenyl Ethyl Acetate
- 28. Phenyl Ethyl Formate
- 29. Phenyl Ethyl Alcohol
- 30. Styrallyl Acetate
- 31. Iso Eugenol
- 32. Linalol
- 33. Linalyl Acetate
- 34. Benzyl Acetate
- 35. Benzyl Formate
- 36. Methyl Anthranilate
- 37. Terpeneol
- 38. Phenyl Acetaldehyde 50%
- 39. Geraneol
- 40. Amyl Cinnamic Aldehyde
- 41. Citronellol
- 42. Dimethyl Octanol
- 43. Methyl Ionone
- 44. Nerol
- 45. Ionone 100%
- 46. Ionone Alpha
- 47. Guaiacwood Oil
- 48. Heliotropin
- 49. Rose Crystals
- 50. Coumarin
- 51. Fixolide
- 52. Resinoid Oakmoss
- 53. Para Cresyl Phenyl Acetate
- 54. Indole

- 55. Phenyl Acetic Acid
- 56. Castoreum
- 57. Resinoid Labcanum
- 58. Vanillir

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LIST OF SAMPLES OF CLOVE BUD OIL AND CLOVE STEM OIL TAKEN FOR ----------OLFACTORY EVALUATION ------1. Black oil from purification tank 2. Black oil from settling tank 3. Clove Bud oil -- Fraction A π π π ___ π 8 Π Π __ Π C " " -- End Fraction 11 4. Clove Bud oil Bulk Sample 5. Clove Stem oil Bulk Sample 6. Clove Bud (Green Bud) oil 7. Clove Bud (Khokar Cloves) oil 8. Clove Bud oil from Grade I (Wete & Mkoani) -"" "Grade II (") " " Grade III (**" " "** -) " " " Grade IV (" " " .) 9. Oleoresin Clove Bud

10. Clove Bud oil Madagascar

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TABLE 1 FRACTIONS OF CLOVE BUD OIL DISTILLATION IN LAB.

Clove buds used : 100 g.

Fraction	Time (Hrs.)	oil B	Low boilers	G C COMPOSI Eugenol	<u>TION</u> Beta elemene	Beta caryophy- llene	Humulene	Eugenyl acetate	High boilers	
1.	2.5	3.2	0.8884	92.4904	-	2.1392	0.2652	3.9932	0.2234	
2.	4.5	3.2	0.3381	89.8870	**	3.0586	0.3927	6.0600	0.2638	
3.	7.0	3.6	0.2304	85.5360	-	3.8477	0.4006	9.7753	0.2100	I
4.	10.5	3.2	0.2391	77.4909	-	5.0377	0.5520	16.3966	0.2838	5 4
5.	22.5	3.6	0.3699	53.5560	0.0728	15.9281	2.2409	27.4104	0.4218	·
6.	32.0	1.2	4.4273	10.9338	1.0732	63.0368	8.1197	9.9289	2.4805	

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Total oil % 18

S.No.	Sample	Oil recovered	distillation	-	Phenols	\$ (G C)				
			Hrs.	still ('C)	Total	Eugenol	Caryo- phyllen	Humulene e	Eug. acetate	
1.	CB-1A	4.42	6	110	88	79.45	10.26	1.19	7.67	
2.	CB-1B	4.42	11	100	92	84.01	7.63	0.91	6.77	
3.	CB-1C	4.30	22	100	94	77.91	5.50	0.42	15.69	
4.	CB-1-End	1.0	27	100	85	45.70	16.22	2.09	34.63	
5.	CB-1-Bulk		• =			80.07	7.00	0.57	11.77	

TABLE 2 CUTS FROM CLOVE BUD OIL DISTILLATION IN DISTILLERY

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Total oil \$ 14.14

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TABLE 3 G C COMPOSITIONS OF CLOVE BUD OIL AND BYE-PRODUCTS OF DISTILLERY

S.No.	Product	Low boilers	Eugenol	Beta elomene	Beta- caryophyllene	Humulene	Eugenly- acetate	High boilers	
	Clove bud oil normal	0.75	78.79	0.08	10.52	1.25	8.44	0.16	
	Clove bud oil (black) from purification tank	5.57	42.79	1.61	45.26	3.90	0.67	0.19	- 56 -
	Clove stem oil (black) from water storage tank	0.65	91.53	0.05	6.58	0.74	0.44	0.02	

TABLE 4 G C COMPOSITIONS OF CLOVE BUD OIL FROM DIFFERENT STAGES OF ITS GROWTH

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S.No.	Product	Low boilers	Eugenol	Beta- elemene	Beta- caryophyllene	Humulene	Eugenly- acetate	High boilers	
1.	Green clove bud oil-brown	1.12	83.25	0.06	7.78	0.95	6.76	0.08	
2.	Green clove bud oil-light color	0.79	, 72.80	Tr.	3.09	0.41	22.90	0.01	- 57 -
3.	Khokar clove bud oil	0.46	75.42	0.07	7.89	0.65	15.48	0.03	

Backstopping Officer's Technical Comments based on the work of Mr. S. Jain DP/URT/86/026/11-55

The consultant has described the valuable contributions that he has made during his two weeks assignment in Tanzania. The methods for improvement of clove oil to the expected sensory standards should be followed in order to compete in the world market. If these recommendations were received much earlier in project implementation, the problems of marketing could have been overcome. Either cohobation or use of settling tanks to recover the oil should be immediately implemented. Facilities for flash evaporation have to be obtained. Therefore, much more work is necessary to improve the quality of the major product of the distillery before additional activities are to be undertaken.

BSO fully agrees with the consultant's recommendations for improvement of quality of clove oil and expects the project authorities to take necessary action in this regard. The consultant has fulfilled his obligations successfully.