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STRENGTHENING OF ESSENTIAL OILS

DP/DRK/88/001

DEMOCRATIC PEOPLE'S REPUBLIC OF KOREA

Technical report: First mission of the analytical chemist*

Prepared for the Government of Democratic People's Republic of Korea by the United Nations Industrial Development Organization, acting as executing agency for the United Nations Development Programme

Based on the work of R. Thomann, Analytical Chemist

Backstopping Officer: T. De Silva, Chemical Industries Branch

United Nations Industrial Development Organization Vienna

^{*} This document has not been edited.

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ABSTRACT

Title of the project:

Strengthening the Essential Oil

Industry in Korea

Number of the project:

DP/DRK/83/001/A/01/37

Number of the mission:

DP/DRK/88/001/11-52

Description of job mission: Analytical chemist

the UNIDO consultant fulfilled his task at the Pyongyang Essential Oil Research Centre (PEORC), during his mission, in June 1991.

The special tasks were,

- to install the laboratory equipment, deliverd from Germany, in accordance with the contract of acd-agro consult, Dresden;
- to put the laboratory equipment into operation and to train the PEORC laboratory staff in operating it;
- to discuss the organization of the laboratory work and the analytical methods (ordering solvents, glass ware, gases, and services);
- to discuss the research program for essential oils obtained by n-hexane extraction.

I. INTRODUCTION

Inis report is given by Dr. Thomann, head of the food technology department at the Institute for Cereal Processing, in 1505 Bergholz-Rehbrücke, Germany. Special objects of his department are essential oils, their production, quality control, and marketing.

The report gives a survey on the activities which were done at r.t.O.R.C.

- to install the laboratory equipment,
- to train the laboratory staff,
- to prepare the field purchase order (solvents, glass ware),
- to inspect the extraction unit in the fields,
- to organize some further activities for marketing.

the mission in Pyongyang started on June 8, 1991, and was finished on July 2, 1991, followed by debricfing stop in Vienna.

all the objectives planned in the analytical field were attained and connected with technological commercial, and scientific questions which have to be solved by the P.E.O.R.C. staff, in future.

the mission was combined with the mission of Mr. Languer, CTA, for the installation of bench scale equipment, at P.E.O.R.C.

II. FECHNICAL REPORT

A. Laboratory situation

As described in the report of Mr. Hylands (1987), the level of laboratory equipment does not meet with the requirements, at present. The few existing glass ware is available only in single items. The use for different samples is connected with long waiting periods due to cleaning. Instruments and devices for quantitative and qualitative analyses did not exist, with the exception of an antiquated gas chromatograph (CSFR - packed columns).

The available solvents and chemicals do not suffice to clean the ylass ware and instruments used or to produce the required solutions and standard mixtures for the adjustment of the instruments and to evaluate the essential oils produced.

The supply of media (cooling water, electricity, highly cleaned gases) is irregular and difficult.

The installation of media (especially of electricity) is hazardous to the health of the operators and the function of the equipment.

The laboratory is staffed with five members. The conversation in English was possible with three of them. They have good knowledge in chemical and analytical problems and are very interested in extending their knowledge. (Participation in a TLC training course, in China, in 1989.)

All the instruments have been adjusted or tested. Operating instructions (check-lists) for all new instruments have been established together with the laboratory staff.

Due to the missing appropriate standard substances/mixtures and glass ware for their preparation, simplified tests had to be performed.

After the realization of the field purchase orders for solvents (Annex IV), glass ware/standard substances, and miscellaneous, the laboratory staff immediately will be able to practice all these methods.

ror TLC, the drying oven and desiccators have not been available in the required size $(20 \times 20 \text{ cm plates})$ so that it was necessary to improvise by a hand-made drying oven and development chambers filled with drying agents. The gas chromatograph was operated at 110 V/60 Hz. The standard electricity voltage in the laboratory is 200 to 230 V. A transformer with limited performance had to be used (warning!).

The gases to be used (nitrogen, hydrogen) should be pure (99.995%) but a quality certificate was not available. The capacity of VARIAN-GC installed gas filters is limited, a more frequent replacement has to be taken into account.

It would be useful to connect the gas chromatograph with one of the personal computers at P.E.O.R.C. The details should be discussed with the regional service office of VARIAN, in Hong Kong.

b. New laboratory equipment

According to the contract with agro-consult, Dresden, the laboratory equipment was installed, at the P.E.O.R.C. laboratory. Any transport damage or defect of single items was recorded in a protocol. The supplier was informed correspondingly. He will send substitutes, as soon as possible. Thanks to the relatively unimportant runction of the damaged parts, the laboratory activities/performance as a whole was not affected severely.

the new installation consists of,

- top load balance (Annex VI, fig. 1)
- precision balance (Annex VI, fig. 1)
- thin layer chromatography equipment (Annex VI, fig. 2)
 - . automatic and manual coater
 - . development chambers (different sizes)
 - . glass plates
 - . UV chamber
 - . different tools and reagent feeder
- automatic polarimeter with micro tubes (Annex VI, fig. 3)
- Abbe refractometer (Annex VI, fig. 4)
- rotational vacuum evaporator (Annex VI, fig. 5)
- gas chromatograph (VARIAN 3400)
 - . with split/splitless injector
 - . different packed and capillary columns
 - . FID and TCD as detectors

C. Essential oils and their processing

The management of P.E.O.R.C. organized a trip to the extraction unit for rosa rugosa, at Chong Pyong. Wild roga rugosa plants and flowers could be inspected. The area near Chong Pyong which is designated for harvest has an acreage of about 100 ha.

A further area of about 2000 ha is said to exist near the Chinese/korean border. In the area near Chong Pyong, 400 workers can be employed for harvesting. Every one of them will collect daily 5 to 10 kgs of flowers. 1500 tons of rosa rugosa flowers will be collected and extracted annually.

The extraction with n-hexane was performed in one of the five extractors (240 1, each)(Annex VII, fig. 3, 4, 5, and 6). The flower/solvent relation is 1:3. The extraction time for the first step is 1 hour. Most of the essential oil (especially Eugenol) is extracted. In a second extraction step, the flowers are "washed with fresh n-hexane.

The nexane has not the required quality (perfume grade). In an analysis of two samples, performed in Germany in July 1991, contents of n-hexane of 25 % and 40 %, respectively, were detected in the redistilled sample (Annex V). 2000 kg flowers (70 % moisture) are yielding 1 kg of "concrete oil".

the five extraction vessels may be used daily up to three times, thus facilitating the processing of 3 tons of rosa flowers.

the extraction residue is steam treated to obtain the remaining n-hexane. The extract is concentrated on a normal-pressure fall-film evaporator which is situated outside the extraction building. Water steam is used as heating medium. The residence time of the extract in the fall-pipe is 7 sec. at a capacity of about 10 kg/h (Annex VII, fig. 7, 8, 9, and 10).

Sometimes, a steam-heated distillation unit is used for solvent refractionation (Annex VII, fig. 11).

After experimental testing the bench scale fall-fill vacuum evaporator, in Pyong Yang (time of delivery November, 1991), the equipment in Chong Pyong will be reconstructed according to the possibilities of Korean engineering and the demands of their commercial partners.

the enriched extracts were subjected to vacuum rectification in a bench vacuum evaporator or a laboratory scale evaporator (Annex VII, fig. 12), in Pyongyang.

The situation of extract processing will be changed drastically with the new equipment which will be deliverd, in autumn 1991.

A verbal report on the water steam distillation was given by Mr. Ur. Choi. The technical requirements to this technology are realizable and can be met without any problems. It is estimated that the size of the equipment, which exists in the D.P.R.K., will cover the demands of the industrial production of different Korean plants.

the following plants for water steam distillation are planned,

- Acorus calamus L. var. anyastatus Berrer
- Angelica qigas Nakai*
- Acorus gramineus solunder
- Agastache rugosa (Fischer et Meyer)
- Thymus vulgaris L.
- Angara schnifolia Eugler
- Thuja orientalis L.**
- Ledum palustre L.
- Convallaria Keiskei Mig.
- Syringia dilatata Nakai
 - * 1000 kg essential oil of the 1990 harvest available
 - ** 5000 kg essential oil of the 1990 harvest available

If the planned objectives, such as

- bench scale equipment installation,
- glass ware,
- solvents for laboratory and 1000 l for extraction tests,
- miscellaneous,
- VARIAN service,

are realized by the management of P.E.O.R.C. and the started activities for commercialization of essential oils (1st step - water steam distillates, 2nd step - hexane extracts) are continued, there will be good chances for fulfilling the planned outputs.

III. RECOMMENDAT ONS

A. Technical section

- 1. For the continuous work of the gas chromatograph the acquisition of four new gas steel-flasks (2 nitrogen, 2 hydrogen) with precision manometers is recommended to the institute.
- 2. Sufficient electrical connections should be available to the laboratory equipment in order to meet with the safety requirements for the operators and instruments (sockets with earthing).
- 3. Water supply should be installed in separate water traps with threads and belonging connections to rubber/plastic tubes.
- 4. For strengthening the international connections, computer software programs should be used which will increase the efficiency of writing letters, offers, requests.
- s. It is recommended to raise the budget 1991 by the following positions,

- 1000 l hexane (perfune grade)	1000 US-\$
- stock of solvents (Annex IV)	1000 US-\$
- VARIAN service consultation	
in November 1991	1000 US-\$

o. Staff section

Any participation in the international communication is advisable. inis could be done by

- continuation of English language training to the laboratory staff,
- subscription to scientific publications (journals, reprints, or copies),
- participation in international workshops,
- organizing national workshops with universities, pharma-enterprises at P.E.O.R.C.,
- participation in the 1st World-Congress on Medicinal and Aromatic Plants for Human Welfare (The Netherlands, 1992) by presenting a poster on P.E.O.R.C. activities (Annex VIII),

- intensification of the correspondence with essential oil processing enterprises, universities, and research centres (contact letters have been drafted),
- issuing publications on results of their own as "short informations" to international journals and publication bulletins,
- working out a P.E.O.R.C. offer.

C. Commercial section

- Promising prospects on the market are expected, first of all, for the essential oils from

Angelica gigas and Thuja orientalis.

Contacts to different trading organizations should be advisable for price and quality comparison purposes.

- Before offering the Rosa rugula extracts, investigations on hexane extracts (perfume grade) for quality determining criteria should be performed (import of 1000 l pure n-hexane at a price of 1000.- US-\$).
- Ucpending on the demands on the international market, further essential oils should be offered, if the plants are available, in U.P.R.K.
- High prices should be argued as follows, natural, wild growing plants are used; no use of plant protection substances (analytical data); utilization of pure solvents (?!) or water steam; qualified quality control (when established).



UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

JOB DESCRIPTION

DP/DRK/88/001/11-52

Post title

Analytical Chemist

Duration

1 month

Date required

Hay or June 1991

Duty station

Pyongyang

Purpose of project Utilization of indigenous essential oils to develop suitable fragrance materials for local industry and export.

Duties

The expert will work under the supervision of the National Project Coordinator and will have particular responsibility for assisting in the design and fabrication of improved distillation and extraction . equipment for local construction and supervise their initial fabrica- . tion.

The expert will participate at the preparatory stages for evaluation of the project and assist in formulating proposals for a time-framed workplan of future activities for the project.

The expert will prepare a full report on his findings and recommendations

Qualifications

A chemical design engineer with a thorough grasp of the up-to-date technology of distillation and extraction of essential oils who will assist local personnel in the design and fabrication of improved stills for local construction. The expert should have recent experience of field distillation, preferably in a developing country.

Language English

Background information As part of a new initiative, the Government of DPR Korea has recently established the Pyongyang Essential Oil Research Centre (P.E.O.R.C) to act as a central body to supervise the production of essential oils and related products, principally from rose, lilac and lily of the valley, to serve a growing domestic market for scented toiletries as well as export. The Government is receiving assistance from UNDP through a UNIDO project designed to facilitate the development of P.E.O.R.C. The project will enhance rural development and provide raw materials for local industry (including that with high added value such as comestics) in a hitherto somewhat neglected sector of the economy.

Ultimately, by producing material for export, will contribute to foreign exchange

Annex II: List of partners

Mr. Dr. Choi Dung Guang
Mr. Dr. Li Myong Ho
Miss Hong Dong Suk
Miss Kang Gum Suk
Mr. Dr. Kim Dyong Chol
Mr. Dr. Karcher
Mrs. Williams
Mr. Li
Miss Rieger

Addresses:

director of P.E.O.R.C.
assistant director
laboratory staff
laboratory staff
technologist
interpreter
U.N.D.P. representative
U.N.D.P. assistant representative
U.N.D.P. senior officer
U.N.D.P. junior officer

Pyongyang Essential Oils kesearch Centre,
Bucksu, Daedonggang,
Pyongyang
D.P.R. of Korea

P.O. Box 27

Pyongyang

D.P.R. of Korea

Annex III: List of recommended equipment for 1992 ff.

- water thermostales
- precision manometers
- drying ovens (for TLC)
- semiautomated burettes
- apparatus for essential oil determination (Clevenger)
- store flasks (glass and aluminium)
- collectors for used solvents, scaled for solvent recycling and water protection
- rubber and plastic tubes
- apparatus for distilled or deionized water
- electrical extension flex
- watertraps with thread
- desk exhausters (toxic gases)
- eye glasses (window glass)
- refrigerator for samples and standards
- fastening for steel flasks
- transformer 110 V
- compressor for low oil air (gas chromatography)
- gas filters/absorbers (gas chromatography)
- VARIAN service (check/consultation/PC adaption)

Annex IV: List of solvents

Variant I 1 or kg	Variant II 1 or kg
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Annex V: Analytical data of solvent used (n-hexane)
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                                              #2.3-DH-H
26.51
                          3241 BC
          26.51#
                                       .037
                                              #2-11 110
 26.76
          26.76#
                          1335 BB
                                       .036
                                              #4 11-110
 27.03
          27.03#
                          365 BD
                                       .010
                                             #3.4 DH-H
27.36
                         3579 BC
597 BB
          27.36#
                                       .000
                                              #3-M INFIGETSTHOP
27,32
          27.02#
                                       .016
                                              #10204-TMCD (2.2.5TM)
20.51
20,23
29,22
          20.51#
                           210 BD
                                       .021
                                             #1T40MCH11C3DMCH
          20.93#
                          212 80
                                       .006
                                             #1-H C-3-ECP
          20.22#
                          141 BV
                                       .004
                                             #1-M-T-3-ECP
29:131
          29.31#
                          337 VB
                                       .000
                                             #1-M-T-2-ECP
20.50
          30.50#
                          150 BC
                                       . 004
                                             #1-H-1-ECP
20.60
          30.ce#
                         2170 BC
                                       .059
                                             #OCTANI1C2C3THCP
30,29
33,07
          39.29#
                          123 80
                                       .003
                                             #I -PP- CP
          33.07#
                          131 CC
                                       .003
55 31
34 56
45 65
46 55
76 55
                                              #N-LU-CL
          33.01#
                          206 BB
                                       .000
                                             #102-5MCH
          34.50#
                          711 BC
                                       .019
                                              #ECH+3.5-04-HP
         35.25#
                          192 00
                                       .005
                                              #1.1.3THCH12.3.5THD
        736.288
                          313 BC
                                       .000
         36.55#
                          463 DB
                                       .012
                                             #MO:
30.17
                          162 88
                                       . 003
                                             #4-11-0
79 115
79 12
79 71
41 22
          38,79
39,15#
39,71#
41,22#
                            271 CB
                                        .007
                                                STYREN
                           500 CD
505 CD
                                       .007
                                               #0%+3-HO
                                       .000
                                               #3110
                            247 CC
                                       .006
                                               #CUMCH
 42,14
46,16
           42,14#
                            269 DB
                                        .007
                                               #11OIIC#1
                            20C CD
                                               #P-ET
                                        .005
 147,23
                            200 00
                                        .000
                                               AL. ET
 47,60
                            210 00
                                        .000
 47.97
           47.97#
                            220 00
                                        .000
                                               #O-CT
 49,26
           40.26
                            242 VB
                                        .005
                                               SEC.BO
           40.26#
                           130 00
                                        ,003
                                               #MCSITYLCH 4HH
           50.62#
                           131 00
                                        .003 #1.2.4-TH-D
```

Annex VI: The new equipment



Figure 1: The top load balance (left) and the precision balance (right)

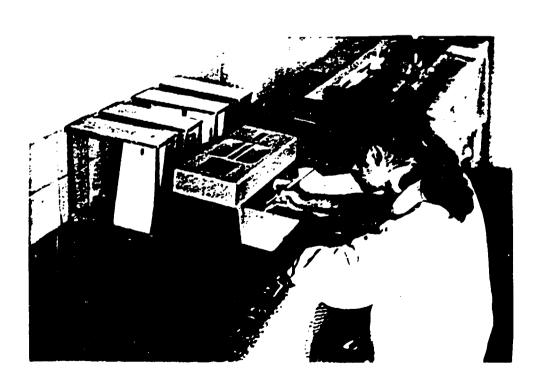


Figure 2: The TLC equipment. The UV chamber (in operation) and four developing chambers.

Annex VI



Figure 3: The automatic polarimeter "POLAMAT A"

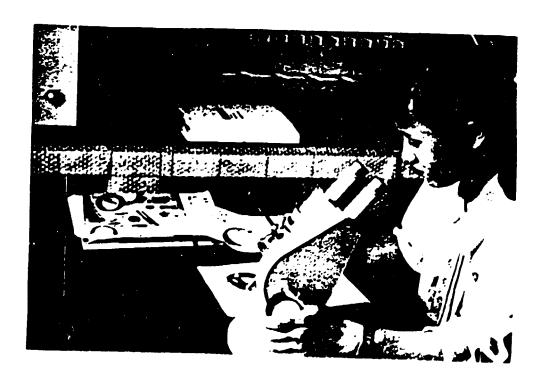


Figure 4: The Abbe-refracrometer

Annex VI

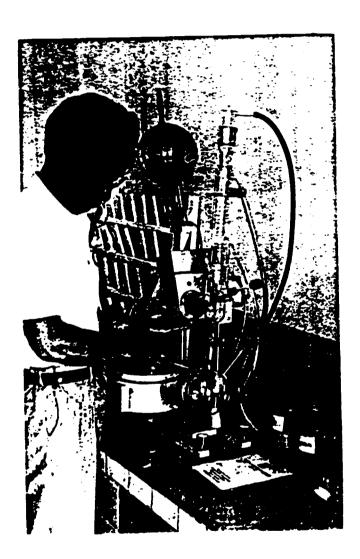


Fig. 5: The rotational vacuum evaporator.

Annex VII: The oil processing equipment





Figures 1 and 2: The rosa rugosa flowers.

Annex_VII



Figure 3: One or the extraction Vessels

Figure 4: Director Choi presents the mobility of the vesse;



Annex_VII

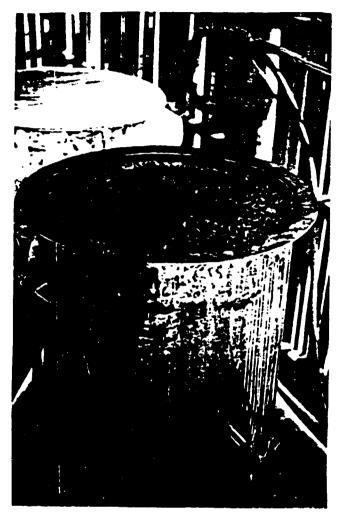


Figure 5: The extraction Vessel, filled with rose flowers and n-hexane (1:3)

Figure 6: Four of the five extraction vessels (two of them filled)



Annex VII



Figure 8: The fall-film evaporator and the condenser for the solvent (left).

Figure 7: The fall-film evaporator. The extract is pressed into the storage container (A) by pressure.



Annex VII

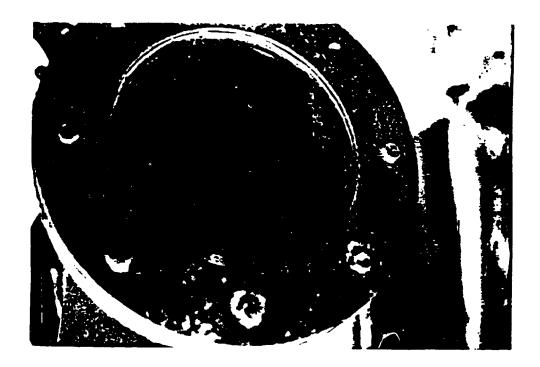


Figure 9: The evaporation speed is observed at the window between fall-pipe and the collector (down)

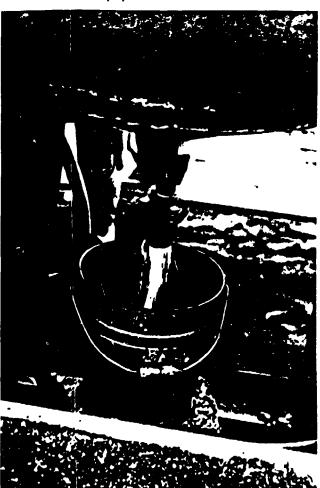


Figure 10: The enriched extract can be taken at the bottom of the collector.

Annex VII

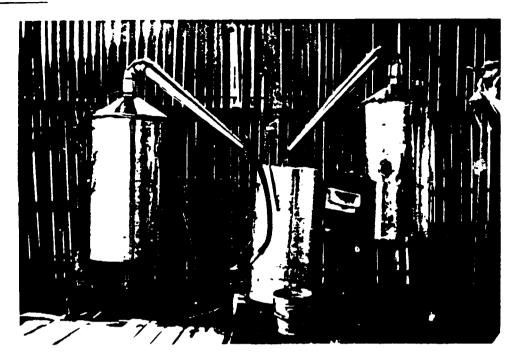


Figure 11: The fifth extractor, prepared for evaporating the residue of solvent from the extracted rosa flowers (left), the cooler (middle), and a distillation unit (right)

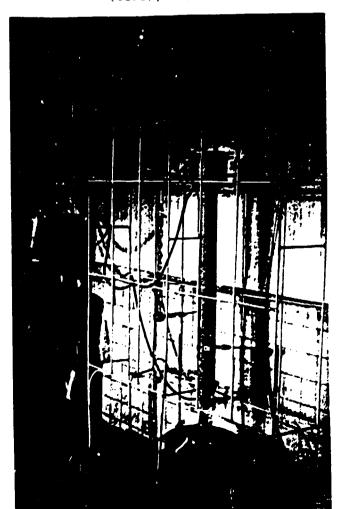


Figure 12: The laboratory scale fall-film evaporator.

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WORLD CONGRESS ON MEDICINAL AND AROMATIC PLANTS FOR HUMAN WELFARE - WOCMAP -20-24 July, 1992

Plant originated products are becoming ever more popular these days. A 'Green Wave' is driving people of many industrialized countries to 'return to nature'. In developing countries traditional medicine reflects their therapeutical qualities.

The Medicinal and Aromatic Plants Section of ISHS would like to accumulate data on the usefulness of these plants. The 500-year anniversary of Columbus' arrival in America is a timely occasion to evaluate the plant resources of the Western Hemisphere. Only 10% of all flowering species have been examined so far in this respect. Loss by extinction is threatening.

WOCMAP will outline aspects of:

- raw materials
- genetic improvement
- agro-systems
- environmental conditions
- processing
- quality control
- energy.

Other topics of interest are:

- product innovation
- pharmaceutical and pharmacological aspects of plants
- international projects and networks
- commercial aspects, marketing
- trade and product regulations
- preservation of natural resources
- quality of life, food, healthcare.

The demand for pharmaceuticals, cosmetics and flavours derived from the plant resources is estimated at 30 billion US Dollars, materials used by healers are not included.

All colleagues dealing with the cultivation, breeding, processing, marketing and social impact of these plants are invited to the WOCMAP meeting in Maastricht, Netherlands, in July 20-24 1992, where exchanges and contacts among researchers, producers, users and consumers can be made.

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The Congress Center of MECC (Maastricht Exposition and Congress Center) offers excellent possibilities for expositions next to the main conference hall and to the restaurant.

Hotel accommodation

Accommodation in different price levels is available in Maastricht, Aachen and Liège.

Correspondence should be addressed to:

IUBS - 51 Boulevard de Montmorency, 75016 Paris, France ISHS - Englaan 1, 6703 ET Wageningen, Netherlands or the Local Organizing Committee - ISHS

Englaan 1 6703 ET WAGENINGEN Netherlands. Phone: (0)8370 - 21747 Fax : (0)8370 - 21586

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28 .

Backstopping Officer's Technical Comments on Technical Report by Dr. Ralph Thomann DP/DRK/88/001/11-52

The report describes the present status of the activities at the Pyongyang Essential Oil Research Centre (PEORC) and the recommendations for development of processing and the achievement of objectives. The installation of equipment have been described together with photographs of the available equipment for processing. The training in the use of gas chromatograph for quality control and analysis has to be extended further in order to achieve the objectives as envisaged. The duties of evaluation and formulating proposals for future activities as given in the job description have been carried out.