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PRODUCT DIVERSIFICATION AND IMPROVEMENT OF THE QUALITY OF ESSENTIAL OILS

SI/ZIM/94/801/11-52/0730DO

ZIMBABWE

Technical report: Mission of the chemical technologist*

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

Based on the work of M. J. Milchard,
chemical technologist

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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ABSTRACT

This report arises from a 1.5 m/m mission by a Chemical Technologist (SI/ZIM/94/801/11-52/0730D0) to Marondera, Zimbabwe from 13 September to 20 October 1995. The mission's purpose was to provide high level advice for product diversification and the improvement of the quality of essential oils produced at Essen Oils Co./Redevelopments (Pvt.) Ltd.

The main conclusion from the mission is that, following the successful installation of the fractional distillation unit, Essen Oils/Redevelopments Ltd. now have the capability to improve the quality of their products by small scale processing, which should make them more acceptable to a wider market. They also will be able to evaluate the technique thereby providing information on which to base decisions on the viability of upgrading the plant by increasing its throughput capacity.

There is a future for the production of selected essential oils in Zimbabwe. There are a number of active producers and other interested parties who have formed an Essential Oils Producers Association (EOPA) to further their common interests. It is hoped that this will lead to the exchange of information and collaboration on both technical and marketing aspects of the industry.

Areas where improvements can be made were identified and recommendation are made for: further trials with the fractionation facility and improvements to the equipment; improvements to the steam distillation process for essential oils; increased efforts to maintain market awareness; quality control; and diversification.

I. INTRODUCTION

This report describes the work of the Chemical Technologist, M.J.Milchard, undertaken during a mission to Zimbabwe. It summarises the activities and is intended to provide the information on which decisions can be made affecting the future development of essential oil production in Zimbabwe.

The purpose of the mission was to provide advice for product diversification and the improvement of the quality of essential oils produced at Essen Oils Co./Redevelopments (Pvt.) Ltd. Central to the improvement of the quality of the essential oils was the provision of a fractional distillation plant. A full job description is given in Annex 1.

The mission began on arrival in the country on 13 September 1995 and finished on departure on 20 October 1995. The main activity during the mission was the assembly and commissioning of a 20 litre fractional distillation unit. A review of existing production methods and equipment for the production of essential oils and laboratory analytical and quality control facilities was undertaken and prospects for diversification were considered.

This mission resulted from an earlier visit by the backstopping officer in February 1995. During that visit specific areas requiring attention and the need for inputs by a Quality Control Chemist and a Chemical Technologist were identified. This mission took place during part of that by the Quality Control Chemist which provided the opportunity for useful collaboration and discussion of ideas.

The main objective of the mission was attained in that the fractional distillation unit was successfully assembled and commissioned. The technology of fractionation was transferred to the counterparts by demonstration of the technique during the initial trials. The results obtained (Annex 5) show that good separation of components is possible with this equipment. Further trials are necessary to determine the conditions for optimum separation, leading to quality improvements, for individual oils.

Other objectives of the mission were addressed and these are discussed in the main body of the report. Conclusions and recommendations for future action are given.

II. ACTIVITIES

On arrival in Harare a meeting was held with Mr.R.Deschamp, the UNIDO programme officer, during which my job description and proposed work programme were discussed. The following day the project and current situation concerning the collaboration between Essen Oils and Redevelopments Ltd. was discussed with one of the counterparts, Mr Craig Naude, before proceeding to the farm near Marondera where Essen Oils is situated.

A. Fractional Distillation Unit

The fractional distillation unit was packed in six wooden crates ar ' had been delivered to the farm prior to my arrival. Mr C Tippett, the counterpart from Ess. .i Oils, had already allocated and cleared an area adjacent to the essential oils production facility where the unit was to be installed. The choice of site was a good one in that the unit would be completely enclosed and therefore well protected from accidental damage. Access to the area would only be necessary when using the unit. It has a high roof and so is well ventilated. The required electricity and water services were adjacent.

Soon after the start of the construction of the galvanised metal framework it became apparent to me that there were discrepancies between the drawing of the metal framework and that of the glass unit and lengths of pipework supplied. Construction was halted and I contacted the manufacturer in the UK. I was informed that I had been given an old drawing and parts list. The design had since been changed and updated. The manufacturer agreed to send the latest drawings and parts list. This caused an unavoidable delay in the assembly of the unit. On receipt of the latest drawings the support framework was rebuilt and the assembly of the glassware proceeded with. As the fractional distillation unit is all glass the assembly had to be undertaken very precisely and very carefully to avoid damaging any of the ground glass surfaces which form the vacuum seal. This was especially true as there were no assembly instructions. Every nut and bolt joining the glass flanges had to be tightened to a precise torque which required the purchase of a special torque wrench capable of low settings. When the assembly was almost completed it was discovered that there were four flange inserts missing. This could not have been determined earlier as they were not on the parts list and through an oversight had been omitted by the manufacturer. Also the thermometer for the still pot contained internal fractures in the glass where it had been joined on two bends which were not immediately apparent. These fractures are only visible on close inspection from a certain angle and are a manufacturing fault which could cause the thermometer to shatter on continued heating and cooling. The manufacturer was informed of these points with a request for replacements which were subsequently received.

The connections to the vacuum pump and associated gauge and valves require special flanges. After very extensive enquiries it was discovered that these were not available in Zimbabwe. Advice was sought from the manufacturer in the UK and their agent in South Africa. Eventually the required parts were custom made by a local specialist experienced in precision metal turning.

On completion of these connections and the installation of the vacuum line, condenser water and electricity supplies for the heating mantle, vacuum pump and water pump the

overall assembly was complete (Annexes 7 & 8). When the unit was operated empty for the first time a very good vacuum was obtained and no leaks in the system were detected. Trials were then undertaken with eucalyptus and tea tree oils. The results of these trials are given in Annex 5. As the fractions were collected their compositions were monitored by gas chromatography. The chromatograms obtained are given in Annex 6. These trials were used to demonstrate the technique of fractional distillation to the counterpart staff and the project local consultant. The effects of changes in vacuum and temperature were clearly demonstrated together with procedures for the safe operation of the unit. It can be seen from the results that these initial trials demonstrated that good separations of components in the oils can be obtained. Improvements in the cineole content of eucalyptus oil from 52% to over 80% and the terpinen-4-ol content of tea tree oil from 6% to 25% were obtained. As each processing unit requires its own set of parameters, improvements in the separations will be achievable following the optimisation of parameters from further trials. Detailed operating instructions are given in Annex 4.

Although the unit worked well there are improvements which can be made to facilitate its operation. The configuration of the glassware is such that there is a holdup of product in certain areas. I eliminated the major one by a slight design modification and others can be improved by adjusting the fall angles of the glassware. The vacuum pump is so efficient connected to this unit that no vacuum control is afforded by the bleed valve supplied. The vacuum had to be controlled by an air leak into the unit itself. The bleed valve should be replaced with one allowing a larger volume of air to enter the system giving finer control. The vacuum gauge (0-1040 millibars) has too wide a range for this application and is only graduated in 20 millibar units. The operating vacuums for this unit will normally be in the range 0-150 millibars and so the gauge should be replaced with one covering this range with finer divisions. The electric motor operating the vacuum pump was found to be drawing at least 30% more current than its design specification. This was a cause for concern and the manufacturers advice was sought. No satisfactory explanation could be offered for this situation but an assurance was given that it would be safe to use the motor, with a higher fuse rating, as it has an internal cut out device which will protect it from damage. Apart from the motor becoming hot no problems were encountered during the trials when the current drawn remained high. The situation must be resolved as it could affect the life of the motor.

B. Essential Oil Processing

The essential oil processing capability at Essen Oils consists of three 12,000 litre still vessels constructed of mild steel with space provision for a fourth still. The internal surfaces of the stills are coated with a special epoxy resin which prevents corrosion of the surface and any reaction between the essential oil and the metal. The flat still lids have a water seal arrangement to prevent the loss of oil and water vapour. The short side takeoff from the still leads to a stainless steel vertical multiplate condenser. One still has a dedicated condenser, the other two share a common condenser with an internal mechanism for isolating the still not in use. The outer surface of each still is lagged with insulating material. Steam is introduced into the base of the stills through a sparge arrangement from a large steam boiler with a capacity of about 500 kg/hour steam at 40 psi. The condensed oil and water are separated in a large stainless steel Florentine flask.

This visit was at a time when no raw material was available so that it was not possible to observe any essential oil distillations. However the operational practices were discussed in detail with Mr Tippett. From observation and discussion it was concluded that there are areas where improvements can be made in the current operation.

The grid on which the distillation material is supported and the lifting chains for removing the spent material are made of mild steel. These should be replaced with stainless steel to prevent corrosion and possible reaction with essential oils. The plastic funnel between the condenser and the collector, observed during the visit of the backstopping officer, has been replaced by one made of stainless steel. The steam requirement for each still is such that the capacity of the boiler is only sufficient to be able to operate one still at a time. A new boiler with nearly twice the steam capacity is currentiy being installed. This means that two stills will be able to be operated simultaneously, three if the existing boiler is retained in working order. The steam pipes from the boiler to the stills should be lagged to prevent heat loss. Steam pressure gauges should be installed on the boiler side of the control valves to the stills so that inlet pressure can be related to flow rate as an aid to process optimisation. The practice of collecting the oil from the separator in plastic buckets should be abandoned in favour of using stainless steel buckets.

During the mission the feasibility of using a straw cutter on the spent material from previous eucalyptus oil distillations was investigated. Initial trials showed that this reduced the waste to a form suitable for composting. These trials should continue.

C. Quality Control

The quality control facilities at Essen Oils consist of a small laboratory with a gas chromatograph and various items of glassware and chemicals. Mr Tippett obtained quotations for the supply of additional analytical equipment for the project including a polarimeter, refractometer and analytical balance. Subsequent to providing these quotations the company ceased trading and so it was not possible to obtain these items. Although they would be a useful addition to the facilities of the laboratory the capital expenditure on these items would probably be better spent on upgrading gas chromatography facilities.

There have not been any requests to date for Essen Oils to provide any physical constants such as density, optical rotation or refractive index on any of the oils sold whereas there have been requests for gas chromatograms. The existing machine, Pye 104, is in working order and the data output is processed by a computer programme (DAPA) which was specially written for chromatography. This allows the raw data to be stored, integrated and printed in different formats. The operating parameters for the chromatograph have been refined following the mission by the Quality Control Chemist. However the main difficulty remains the reproducibility of the chromatograms between individual runs. There is considerable variation in the retention times of components from run to run making comparisons between oils and individual isolates difficult and unreliable. The machine is operating with a packed column at present as there are no fittings to instal the supplied capillary column. The use of the capillary column will certainly improve the resolution but not the reproducibility problem. This is due to the design and material of the column oven which does not respond rapidly to changes in temperature and so does not equilibrate quickly between temperature programmed runs which are necessary for the analysis of essential oils.

As gas chromatography is such an important and useful analytical technique for the analysis of essential oils consideration should be given to the purchase of a modern machine which will have a thermally stable oven and built in facilities for capillary column operation.

Clevenger type volatile oil and Dean and Stark moisture determination apparatus is available in the laboratory. These will be useful in the screening of possible new essential oils materials and monitoring trial plots or variety comparisons.

D. Diversification

As there is considerable competition for its two main products, eucalyptus and tea tree oils, from other world producers, Essen Oils is currently investigating diversification into other products. These are likely to be essential oils but other materials will be considered on their merits. One example is the production of indigo dye. Requirements for any new product would be that it is relatively drought tolerant, as there have been severe shortages of water in recent years, and that it has a high value. Export by air is the only viable option as shipment by sea has to be done through either South Africa or Mozambique with considerable delays and difficulties encountered.

Two oils which are currently showing promise are those from *Tagetes minuta* and *Lippia javanica*. The 'Tagette' oil is obtained from the collection of non cultivated material growing on land outside the control of Essen Oils. This means that the yield and quality of the oil can vary from area to area and year to year. To overcome this problem trial plots are to be established this year on Essen Oils land where growth characteristics and costs of cultivation can be monitored. Plots of *Lippia javanica* have been established and it appears that the plant is fairly drought tolerant. Sales of the oils have been made to Europe indicating that it is worthwhile pursuing their development. Other oils under investigation are those from the 'Lavender tree' and Helichrysum. Another essential oil plant which would be worthwhile investigating is *Salvia stenophylla*. This grows in Southern Africa and contains Bisabolol for which there is a demand.

Although laboratory facilities are available for the small scale distillation of these plant materials (Clevenger), in order to get more meaningful results a larger facility is required. The existing stills are far too large for trial quantities and so Mr Tippett has drawn up plans for a small field still. These were discussed and advice given on various aspects of the design. The intention is to construct the still in stainless steel so that any material can be distilled in it and to locate it adjacent to the main distillery for ready access to steam and cooling water. Mr Tippett will use it for his own trials and make it available for use by others on a confidential basis.

E. Farm Day

A farm day was organised at Essen Oils where the opportunity was provided for the other essential oil producers (Annex 2) to learn about fractional distillation and the application of gas chromatography to the analysis of essential oils. Producers were invited personally and the event was publicised in the Essential Oil Producers Association (EOPA) newsletter.

Seven people attended on the day and I explained the principles of fractional distillation and its application to essential oils. Those present were shown the unit in the course of assembly. The Quality Control Chemist demonstrated the gas chromatograph, explaining the principles behind the technique. Chromatograms of the oils of interest in Zimbabwe were available for inspection.

The opportunity was taken for discussion with those present on the current situation concerning the essential oil industry in Zimbabwe. No-one raised any specific technical problems as the main area of concern seemed to be that of marketing. I emphasised that I would be available to provide assistance to members of EOPA during my mission in Zimbabwe if required. No requests were received up to the time of my departure.

The people attending the farm day appreciated the efforts involved in organising the day and said that they found it very useful and informative.

F. Good Manufacturing Practice

The principles of Good Manufacturing Practice (GMP) were discussed with Mr Tippett relating to the production of essential oils. The importance of general cleanliness in and around the processing area, good storage conditions and readily retrievable records of individual batches processed were emphasised. Mr Tippett is well aware of the importance of safety and safe working practices both in the processing area and in the laboratory.

Further guidance on GMP is given in Annex 3.

III. CONCLUSIONS

Following the installation of the fractional distillation unit and the transfer of technology for its operation, Essen Oils/Redevelopments Ltd now have the capability to fractionate a range of essential oils. Further trials are necessary in order to determine the optimum processing conditions for individual oils. The capacity of the unit is not sufficient for the economic commercial processing of lower value oils but will provide the information on which to base decisions on upgrading to a commercial capacity. However the unit should be suitable for the isolation of high value individual components in small volumes.

Observation and discussion of the equipment and procedures for the primary distillation of essential oils at Essen Oils indicate that there are no major shortcomings in the processing of oils. Areas where improvements can be made were identified and recommendations given. There are plans to establish a small pilot still for experimental purposes which will be necessary for the screening of material for diversification.

The current quality assessment of essential oils required by buyers seems to be analysis by gas chromatography. The laboratory at Essen Oils has a chromatograph which, although an old model, is capable of producing acceptable results following the mission of the Quality Control Chemist. In the longer term this will have to be replaced by a more modern instrument and so it is not worthwhile at this stage trying to upgrade it with

accessories. Other analytical instruments such as a polarimeter, refractometer etc. would be of limited value at this stage in the development of the company.

Marketing of its products remains a major concern of Essen Oils. Severe difficulties are encountered with communication with buyers overseas because of the inadequacy of the telephone system. Without reliable and rapid international communications it is difficult to respond to buyers enquiries and to obtain up to date information on market trends and prices. International surface shipment of oils is difficult as it has to go via South Africa or Mozambique with the associated delays and difficulties. The alternative use of airfreight is only viable for high value oils due to the higher costs. Future marketing strategy should therefore be aimed at supplying high unit value products internationally by air and lower value oils within Zimbabwe for import substitution and regionally in Southern Africa. With the fractionation facility it should be possible to meet local buyers specifications. Diversification should be aimed at high unit value or value added products unless there is a clearly identified sustainable local demand for a product.

Essen Oils is a well managed commercial operation in which any developments must be shown to have an economic justification. The association with Redevelopments Ltd should improve considerably the marketing of its products. The company has benefited from the assistance provided by this mission as it would from any future help.

IV. RECOMMENDATIONS

Fractional Distillation

1. In order to prevent heat loss from the column, it is recommended that it is lagged with the insulation material obtained during the mission.

2. As only one carbon burst disk was supplied with the unit it is recommended that spare disks are obtained as a priority. The unit cannot be operated without a burst disk.

3. It was found that very volatile components of the oils which were not condensed in the unit were condensing in the outlet pipe from the vacuum pump. It is recommended that the vacuum line is fitted with a cold trap between the unit and the vacuum pump to eliminate the possibility of damage to the pump and contamination of the oil and that a suitable trap is fitted to the exit port of the pump in order to prevent chemicals entering the atmosphere.

4. As the range of vacuum at which this unit is normally operated occupies only a small area of the scale on the gauge provided, accurate measurement of vacuum is difficult. It is recommended that the gauge is exchanged for one with a more appropriate scale such as 0-150 millibars.

5. The air leak valve does not permit sufficient air to enter the system to provide any control of vacuum. It is recommended that the valve is exchanged for one with a greater inlet volume.

6. To improve the boiling characteristics of the material in the evaporator it is recommended that suitable sized inert boiling balls are obtained which will not interfere with the operation of the drain valve.

7. In order to determine the optimum fractionation conditions for the major oils of interest an extended series of trials is necessary which were not possible during this mission. As there are considerable demands on the time of Mr Tippett on the farm for matters unrelated to essential oils it may not be possible for him to devote the continuous period of time which this would require. If this is the case it is recommended that consideration be given to a further mission by a Chemical Technologist to undertake these trials.

Essential Oil Distillation

8. The steam pipes from the boiler to the stills are not insulated. The resultant heat loss can lead to waste of boiler fuel and water condensation in the pipes. It is therefore recommended that these pipes are lagged with suitable material.

9. Apart from measuring the distillate flow rate there is no measure of steam usage during a distillation. It is recommended that steam pressure gauges are fitted adjacent to the steam inlet valves on the boiler side. This will permit measurement of inlet pressure required for a given flowrate and facilitate reproducing flowrates by setting inlet pressure for particular distillations.

10. Although the inner surfaces of the still vessels are coated with an epoxy resin the support grids and lifting chains are made of mild steel. As these are liable to corrosion and possible reaction with certain components of oils it is recommended that these are replaced with similar items made of stainless steel.

11. As certain oils can react with plastic, it is recommended that the buckets used for collecting the oils from the Florentine separator are replaced with ones made of stainless steel.

Marketing

12. It is recommended that continued efforts are made to determine the exact requirements and specifications of local users of essential oils.

13. In order to maintain awareness of prices and marketing trends it is recommended that requests are made to selected international essential oil dealers to be put on their mailing lists to receive their market newsletters. The library of Zimtrade in Harare may also be a useful source of information.

Quality Control

14. The gas chromatograph is the most useful analytical instrument at Essen Oils at present. Improvements to this technique lie in the analysis of oils on capillary columns. It is not considered worthwhile attempting to upgrade this instrument because of deficiencies in the stability of the oven temperature. It is therefore recommended that if a decision to move to capillary columns is made then the chromatograph is replaced with a modern

instrument with built in features of injection splitter, make up gas lines etc. to easily accommodate these columns.

15. As there have been no requests to date from buyers for physico-chemical data on oils it is recommended that at the present time instruments for these determinations. such as a polarimeter and refractometer, are not purchased. If such a request was received the determinations could be carried out by a local laboratory.

Diversification

16. It is recommended that any materials considered for diversification should be drought resistant and have an intrinsic high value or added value on processing.

17. As initial sales of oils from *Tagetes minuta* and *Lippia javanica* have been encouraging it is recommended that development of these two oils continues with further cultivation trials.

18. It is recommended that screening of indigenous plants such as the Lavender tree, Helichrysum and possibly *Salvia stenophylla* continues. Further development should follow if initial trials show promise.

19. As diversification into non essential oil materials is also being considered it is recommended that further information is obtained on Indigo and *Aloe vera*. Trials should then be undertaken if considered feasible.

20. As a small scale steam distillation facility will be required for trials with new materials it is recommended that the construction of the pilot still is given high priority.

ANNEX 1

JOB DESCRIPTION
SI/ZIM/94/801/11-52/0730D0

Post Title: Chemical Technologist

Duration: 1.5 m/m

Date Required: ASAP

Duty Station: Marondera, Zimbabwe

Purpose of Project: High level advice for product diversification and the improvement of the quality of essential oils produced at Essen Oils Co./Redevelopments (Pvt.) Ltd.

Duties: The expert will be required to work in collaboration with the other international experts and counterpart staff at Essen Oils Co./Redevelopments (Pvt.) Ltd. In carrying out the following duties:

1. Study the current production methods, analytical support services and quality assurance system and associated problems of the company.

2. Determine the requirements for improvement of production technologies, secondary processing and process control and full capacity utilization.

3. Demonstrate processing and fractionation methods for product improvement, (eucalyptus, tea tree, tagetes and lippia oils in particular).

4. Train counterpart staff in improved methods of distillation/fractionation and raw material and process control.

5. Introduce CGMP and safety measures.

6. Recommend a suitable quality assurance system.

7. Assess the potential of indigenous raw materials or essential oils to be used in product diversification.

8. Recommend a diversification programme together with details of processing, product development and quality control specifications.

9. Recommend equipment needed for the diversification programme.

10. Recommend further improvements to increase production and quality of essential oils.

11. Advise other companies (Four Seasons Foods Put. Ltd., Essential Extracts Company, Nature Products Company and Plant Oil Producers Association) on quality control and processing methods.

Finally the expert will furnish a report embodying his findings and progress and outlining his recommendations on product diversification, improved technologies and processing and quality improvement to both UNIDO and Essen Oils Co./Redevelopments (Pvt.) Ltd.

Qualifications: Chemist/Pharmacist/Technologist or chemical engineer with over 10 years of experience in the production and quality control of essential oils and plant based products.

Language: English

ANNEX 2

PEOPLE MET DURING THE MISSION

Counterparts

Mr C J Tippett	Managing Director, Essen Oils Co.
Mr C Naude	Redevelopments Ltd.
Mr A K Irvine	Technical Consultant

Farm Day Attendees

Mr M Jack	Four Seasons Foods
Mr I Sinclair	Director Marketing, Essential Plants Extract Company
Mr C D B Payne	Linden Grange Farm
Dr L S Chagonda	Department of Pharmacy, University of Zimbabwe
Three Staff Members	Department of Pharmacy, University of Zimbabwe

UNIDO

Mr R Deschamps	Programme Officer

Others

Dr I Southwell	Quality Control Chemist
Mr R Holland	Precision Metal Fabrication
Mr T Thompson	Scientific Equipment Supplier

ANNEX 3

GOOD MANUFACTURING PRACTICE

Good manufacturing practice is most often applied to the food industry. However due consideration should be given to the following points applicable to essential oil processing.

Plant and surroundings

1. Equipment storage
2. Raw material receipt
3. Waste disposal
4. Plant construction and design:
   a. Product flow
   b. Timely processing
   c. Construction to ensure prevention of product contamination and easy cleaning.
   d. Adequate space for safe and efficient operation.
5. Product segregation:
   a. Raw material
   b. Intermediate and finished products

Operational cleanliness

1. Maintenance of plant and equipment in clean condition
2. Cleaning materials
3. Storage of raw materials to prevent contamination
4. Vermin control
5. Control of personnel in production area

Personnel

1. Cleanliness - proper hygienic practices:
   a. Personal cleanliness
   b. Protective clothing as appropriate
   c. Protection of product and product contact surfaces
2. Education and training of employees:
   a. Proper processing techniques
   b. Appreciation of safe working practices
3. Supervision:
   Competent personnel with the responsibility for assessing compliance by all employees to good hygienic and safe working practices.

Equipment

1. Proper construction and material:
   a. Corrosion resistant material
   b. Non reactive with product
   c. Easy to clean

Production and process controls

1. Receiving, transporting, preparing, manufacturing, packaging and storage all under clean and safe conditions and practices.
2. Recording and secure archiving of processing conditions and results of every batch processed.
3. Laboratory controls:
   a. Representative sampling procedures
   b. Appropriate quality control analyses
   c. Maintenance of records of results
   d. Laboratory safety practices
4. Supervision
5. Raw materials - quality

ANNEX 4

OPERATING INSTRUCTIONS FOR THE FRACTIONAL DISTILLATION UNIT

he following step by step guide is intended for the efficient and safe operation of the 20 litre fractional distillation unit installed at Essen Oils Co. These instructions refer to the equipment as installed at the end of this mission. Procedures may have to be modified following subsequent modifications to the design or individual components.

Start Up

Ensure that the apparatus is clean. Close all of the valves in the glass unit except the valve to the vacuum line at the top of the column. The sample may be introduced at this stage or once the vacuum is established. For the former option open the quick release cover on the 20 litre flask and carefully pour the measured volume of oil into the flask. The maximum working volume is 15 litres but it was found that less bumping and frothing occurred when 12 litres was used. This volume is level with the rim of the electric heating mantle when cold.

Close and tighten the quick release cover with the wing nuts. Close the unit isolation valve next to the pump and open fully the air bleed valve. Turn on the cooling water. Switch on the vacuum pump. As the air bleed valve currently fitted does not give sufficient vacuum control it is controlled by adjusting the bleed valve on top of either of the collection flasks.

Slowly open the isolation valve until fully open. Open the vacuum valves on each of the collection vessels and adjust the system vacuum to the required level with one of the collection vessel bleed valves. A typical initial vacuum is 60 millibars (the gauge has 20 millibar graduations). If the vacuum was established with the apparatus empty the oil may now be introduced via the valve on the top of the 20 litre flask.

Operation

Observe the oil in the evaporator. If the mixture boils at ambient temperature it is unlikely that the vapours (very volatile components or water) will condense and will therefore pass through the vacuum pump as there is not a cold trap in the system. To avoid this, adjust the vacuum to 100 millibars or until the boiling stops. Then or if the oil did not boil initially, turn on the heating mantle to setting 3.

Continue heating until boiling occurs and vapour travels up the column and is condensed in the condenser. Note the temperatures in the evaporator and in the reflux divider. At this stage the reflux divider valve remains closed allowing total reflux

Close the product inlet valve on the collection flask with air inlet valve controlling the system vacuum. Open the product inlet valve on the other collection flask. When the temperatures are stable (they will not always be the same, the evaporator tending to be slightly higher) open the reflux divider valve and set the reflux ratio to about 10:1. That is

10 volumes return to the column and 1 goes to the collector. When the temperature in the reflux divider starts to rise or if several fractions are to be collected at the same temperature close the product inlet valve on the collector vessel and open the valve on the other vessel. To remove the fraction from the system close the vacuum valve on the collector flask and slowly open the air inlet valve on the same flask until the pressure in the flask is at atmospheric. The fraction may now be removed through the drain valve. This procedure is repeated for subsequent fractions. By isolating the collector vessel the equilibrium of the system is maintained. When changing between collector vessels it may be necessary to use the other air bleed valve to control the system vacuum.

The number of fractions collected and the temperatures, vacuum setting and reflux ratios will vary with different oils and the degree of separation required. Optimum conditions for individual oils will be determined by a series of trials using different combinations of these settings. During these trials a careful note should be kept of all the parameters and the composition of all the fractions should be monitored by gas chromatography.

Shut down

When the final fraction has been collected the reflux divider valve is closed and the heater switched off. The system is allowed to cool and any material remaining in the column allowed to drain into the evaporator. Close the isolation valve. Ensure that the air bleed valve on the vacuum pump is open and the gas ballast valve is closed and then turn off the vacuum pump. Slowly open one of the air bleed valves on the glass unit until the system is at atmospheric pressure. The final fraction can now be removed and the residue in the evaporator removed while it is still warm through the bottom drain valves.

Cleaning

If the plant is going to be used for the same oil soon after the finish of the current operation then complete cleaning will not be necessary, allowing the system to drain should suffice. If a different oil is to be processed of if the plant is to be left unused for some time it should be cleaned. The design of this plant is such that there is hold up of material in parts of the system. The only way to clean it thoroughly therefore would be to dismantle it, clean the parts and reassemble it. This is clearly not practical and the following procedure is recommended. Put about three litres of acetone in the 20 litre flask. Close the isolation valve and do not switch on the vacuum pump. Close the reflux divider valve. Open the air bleed valves on the collector vessels so that there is not a build up of pressure in the system which might rupture the burst valve. Gently heat the acetone until it boils and refluxes in the column. Allow boiling under total reflux for about 30 minutes. Open the reflux divider valve and collect about 500 ml. of solvent in each collector. Close the divider valve and switch off the heating. Wait until boiling has stopped and all liquid has returned to the evaporator from the column. Remove the solvent from the evaporator and the receivers. Add a small amount of fresh solvent to the evaporator, allow to stand for a short while and remove. Leave the unit with all the valves open, including the quick-release cover and all drain valves, for the last traces of solvent to evaporate. When next using the plant do not introduce any sample until all traces of solvent are removed. When using acetone for cleaning, extreme care must be taken because of its flammable nature. It may be possible to recover the used acetone for subsequent cleaning operations by laboratory distillation.

ANNEX 5

RESULTS OF THE FRACTIONAL DISTILLATION TRIALS

EUCALYPTUS OIL

Sample	Processing conditions		Volume (ltr)	Cineole content %
	Head temp (C)	Vacuum (millib)		
Whole oil			14	52
Fraction 1	73	60	1.7	15.5
Fraction 2	76	60	2.8	31.4
Fraction 3	84	60	2.2	54.6
Fraction 4	85	60	2.95	84.9
Fraction 5	58	10	1.91	82.4
Residue			1.6	9.3

Major component composition - area % by gas chromatography

Sample	Isovaleraldehyde	Alpha pinene	Cineole
Whole oil	1.4	34.3	52
Fraction 1	0.8	81.8	15.5
Fraction 2		65.7	31.4
Fraction 3		40.8	54.6
Fraction 4		1	84.9
Fraction 5		0.3	82.4
Residue			9.3

TEA TREE OIL

Sample	Processing conditions		Volume (ltr)	Terpinen-4-ol content %
	Head temp (C)	Vacuum (millib)		
Whole oil			15	6.5
Fraction 1	77	30	1.275	0.26
Fraction 2	68	20	3.45	0.8
Fraction 3	68	20	4.38	1.3
Fraction 4	68	20	2.74	8.7
Residue			2.92	25.3

Major component composition - area % by gas chromatography

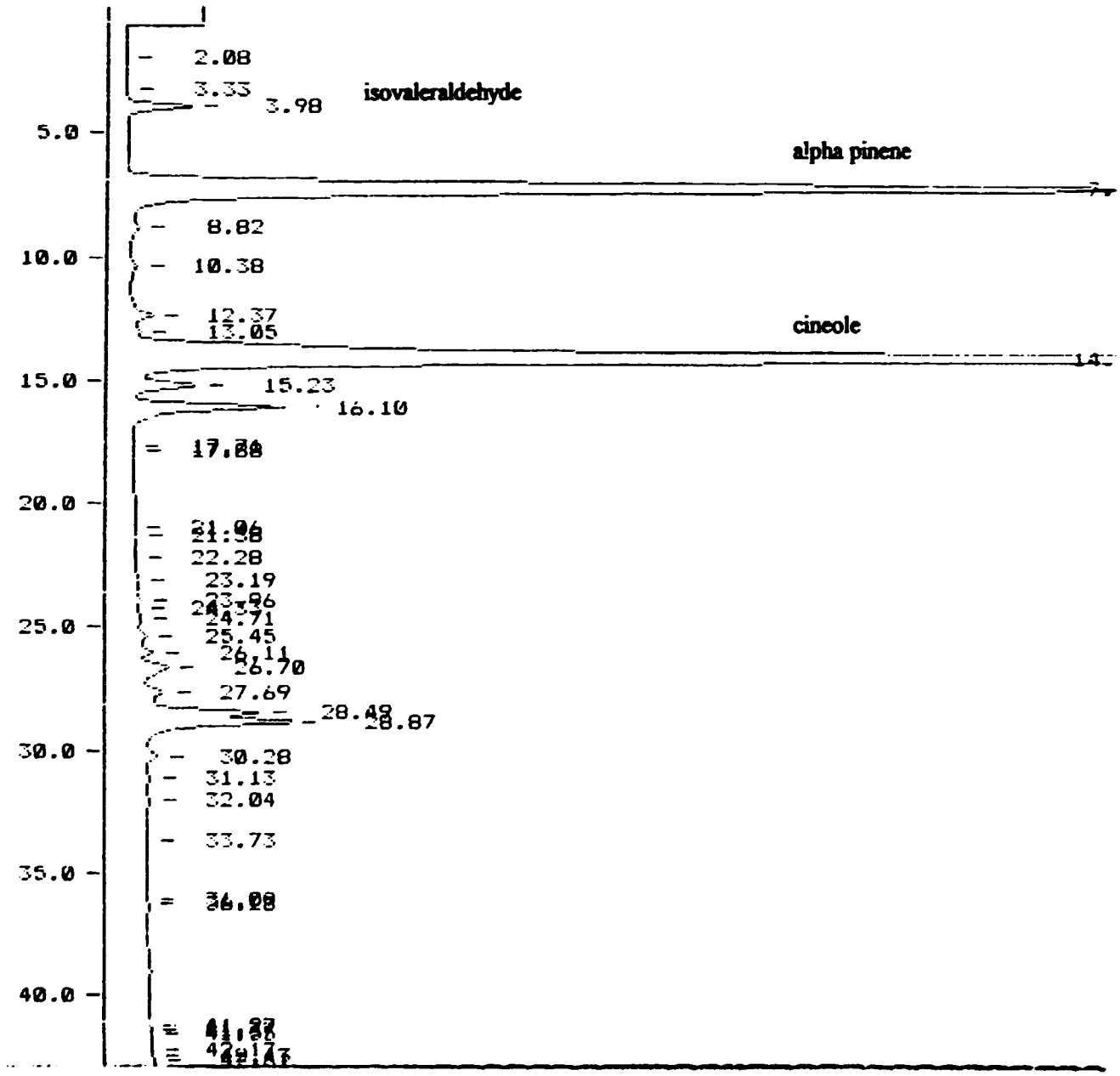
Sample	Alpha pinene	Cineole	Terpinolene	Terpinen-4-ol
Whole oil	2.3	30.9	42	6.5
Fraction 1	17	53.8	14	0.26
Fraction 2	2.7	49.4	34	0.8
Fraction 3	0.4	36.7	52.4	1.3
Fraction 4		15.7	65	8.7
Residue			1.7	25.3

ANNEX 6

G C TRACES FROM THE FRACTIONAL DISTILLATION TRIALS

EUCALYPTUS OIL

Whole oil



Whole oil

INTEGRATION REPORT FOR FILE EUC-IND2

SAMPLE ID = EUC-IND2 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 100 3 .1 0

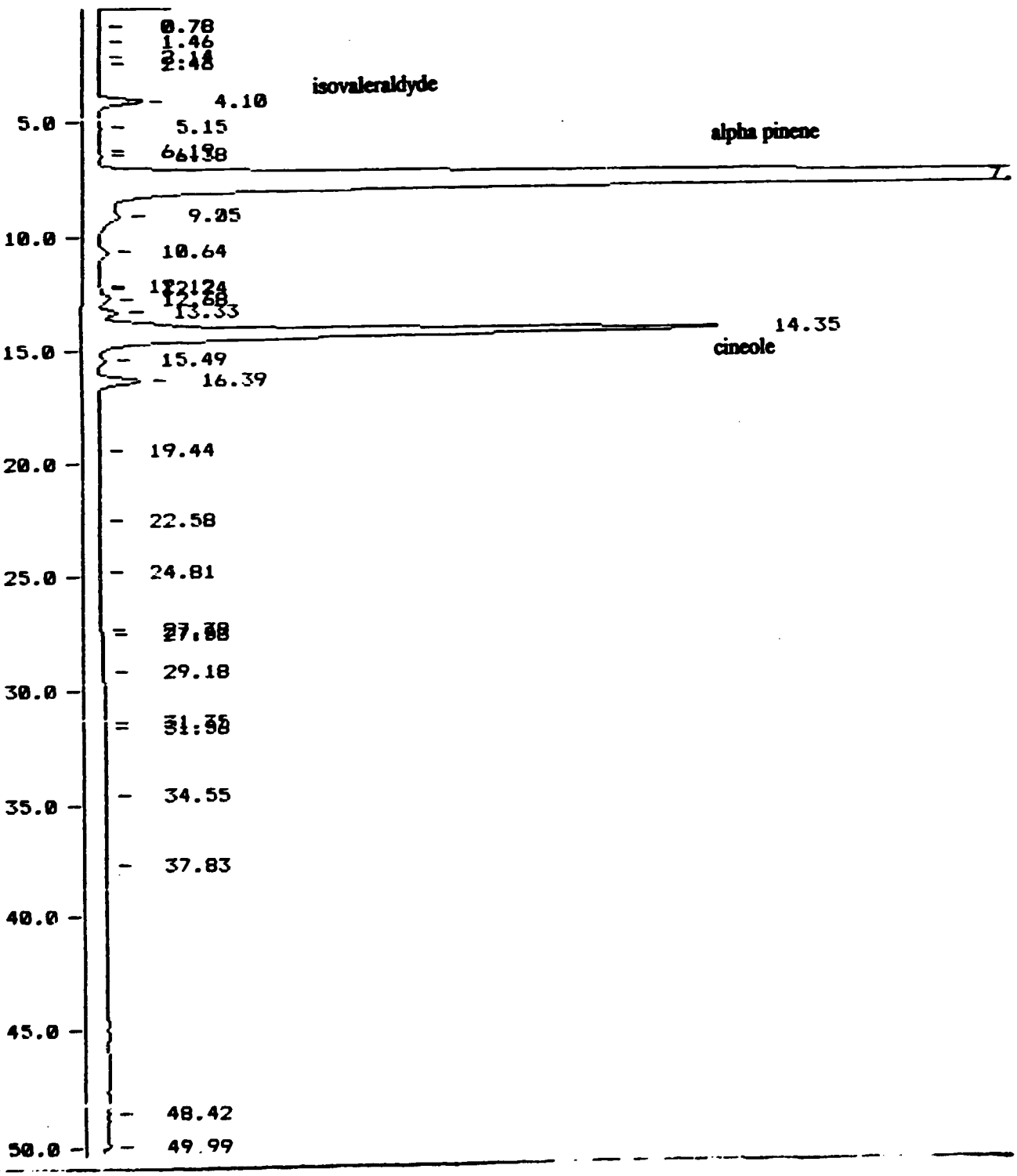
DATE 10-19-1995 TIME 06:46:00

TEXT:EUC-INDUSTRIAL GRADE BATCH A9

PLOTTING IS FROM 0.00 to 55.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	2.08	28	2	BV	1	13	0.0067
2	3.33	19	1	BV	4	11	0.0045
3	3.98	5841	165	BB	6	55	1.3898
4	7.24	143999	2675	BB	12	107	34.2649
5	8.82	539	13	BB	29	44	0.1283
6	10.38	562	13	BB	21	44	0.1338
7	12.37	3091	57	BV	18	65	0.7355
8	13.05	223	8	VB	30	26	0.0531
9	14.13	218569	4186	BV	40	100	52.0089
10	15.23	5560	144	VV	55	48	1.3231
11	16.10	15711	347	VB	32	76	3.7384
12	17.71	16	1	BV	25	7	0.0038
13	17.88	16	1	VV	27	10	0.0039
14	21.06	18	1	BV	32	10	0.0043
15	21.38	50	3	BV	33	10	0.0119
16	22.28	15	1	BV	34	8	0.0036
17	23.19	154	6	BB	36	26	0.0367
18	23.96	369	12	BB	37	37	0.0877
19	24.33	17	2	BV	38	8	0.0040
20	24.71	77	3	BB	44	20	0.0182
21	25.45	873	22	BV	42	36	0.2077
22	26.11	718	25	BV	57	30	0.1709
23	26.70	3220	64	VB	53	53	0.7661
24	27.69	1758	45	BV	64	33	0.4183
25	28.49	9374	263	BV	83	32	2.2305
26	28.87	7467	219	VB	271	56	1.7767
27	30.28	1197	28	BV	66	48	0.2847
28	31.13	167	6	BB	64	27	0.0397
29	32.04	74	3	BB	65	21	0.0175
30	33.73	17	1	BV	64	8	0.0040
31	36.09	37	2	BV	64	15	0.0088
32	36.28	21	2	VV	65	10	0.0051
33	41.27	42	2	BV	71	15	0.0099
34	41.37	16	1	VV	72	7	0.0039
35	41.55	16	1	VB	74	10	0.0038
36	42.18	29	1	BV	73	10	0.0069
37	42.43	82	4	VV	74	15	0.0195
38	42.61	31	2	VV	79	11	0.0075
39	45.15	207	4	BB	72	49	0.0491
40	48.11	17	1	BV	69	10	0.0039
41	54.53	18	2	BB	73	11	0.0042

Fraction 1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12.24
12.68
13.33
14.35
cineole
15.0
15.49
16.39
20.0
19.44
22.58
25.0
24.81
27.38
27.88
29.18
30.0
31.35
31.58
35.0
34.55
37.83
40.0
45.0
48.42
50.0
49.99

Fraction 1

INTEGRATION REPORT FOR FILE EUC-IND4

SAMPLE ID = EUC-IND4 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 100 3 .1 0

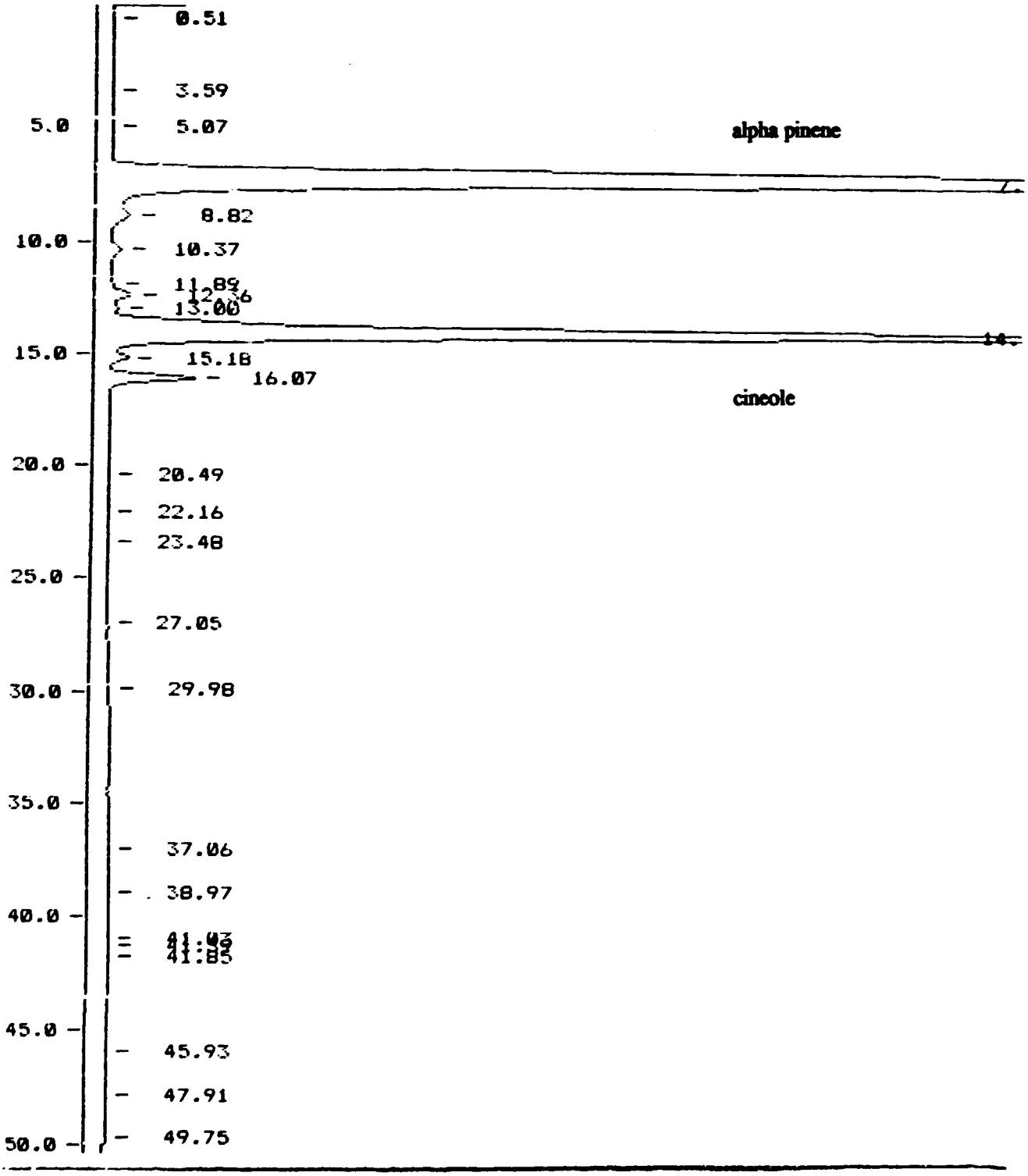
DATE 10-19-1995 TIME 13:04:36

TEXT:EUC-IND A-PINENE?

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.04	272	17	BV	-17	8	0.0485
2	0.78	19	3	BV	0	7	0.0034
3	1.46	37	2	BV	-1	14	0.0066
4	2.14	49	2	BV	-0	17	0.0087
5	2.46	48	2	VB	1	18	0.0085
6	4.10	4485	121	BB	3	47	0.7984
7	5.15	31	2	BB	4	16	0.0056
8	6.19	18	1	BV	2	6	0.0032
9	6.38	57	2	VV	4	15	0.0102
10	7.62	459412	8162	BV	2	116	81.7831
11	9.05	1349	27	BB	45	59	0.2401
12	10.64	153	5	BB	2	29	0.0272
13	12.12	77	5	BV	1	13	0.0137
14	12.24	18	2	VV	6	8	0.0033
15	12.68	1044	26	VV	8	41	0.1858
16	13.33	2001	51	VV	10	35	0.3562
17	14.35	86844	1670	VB	28	98	15.4597
18	15.49	591	16	BV	11	34	0.1052
19	16.39	4924	118	BB	3	58	0.8766
20	19.44	50	2	BB	1	19	0.0089
21	22.58	31	2	BV	-1	12	0.0055
22	24.81	35	1	BV	2	16	0.0062
23	27.39	28	1	BV	6	11	0.0050
24	27.58	19	2	VB	7	11	0.0034
25	29.18	19	2	BV	8	9	0.0033
26	31.35	24	1	BV	13	13	0.0042
27	31.56	16	2	VV	13	9	0.0028
28	34.55	18	2	BV	14	10	0.0033
29	37.83	15	1	BV	15	10	0.0027
30	48.43	16	1	BB	17	11	0.0028
31	49.99	44	8	BB	18	4	0.0078

Fraction 2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15.18
16.07
cineole
20.0
20.49
22.16
23.48
25.0
27.05
30.0
29.98
35.0
37.06
38.97
40.0
41.03
41.85
45.0
45.93
47.91
50.0
49.75

Fraction 2

INTEGRATION REPORT FOR FILE EUC-IND5

SAMPLE ID = EUC-IND5 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 100 3 .1 0

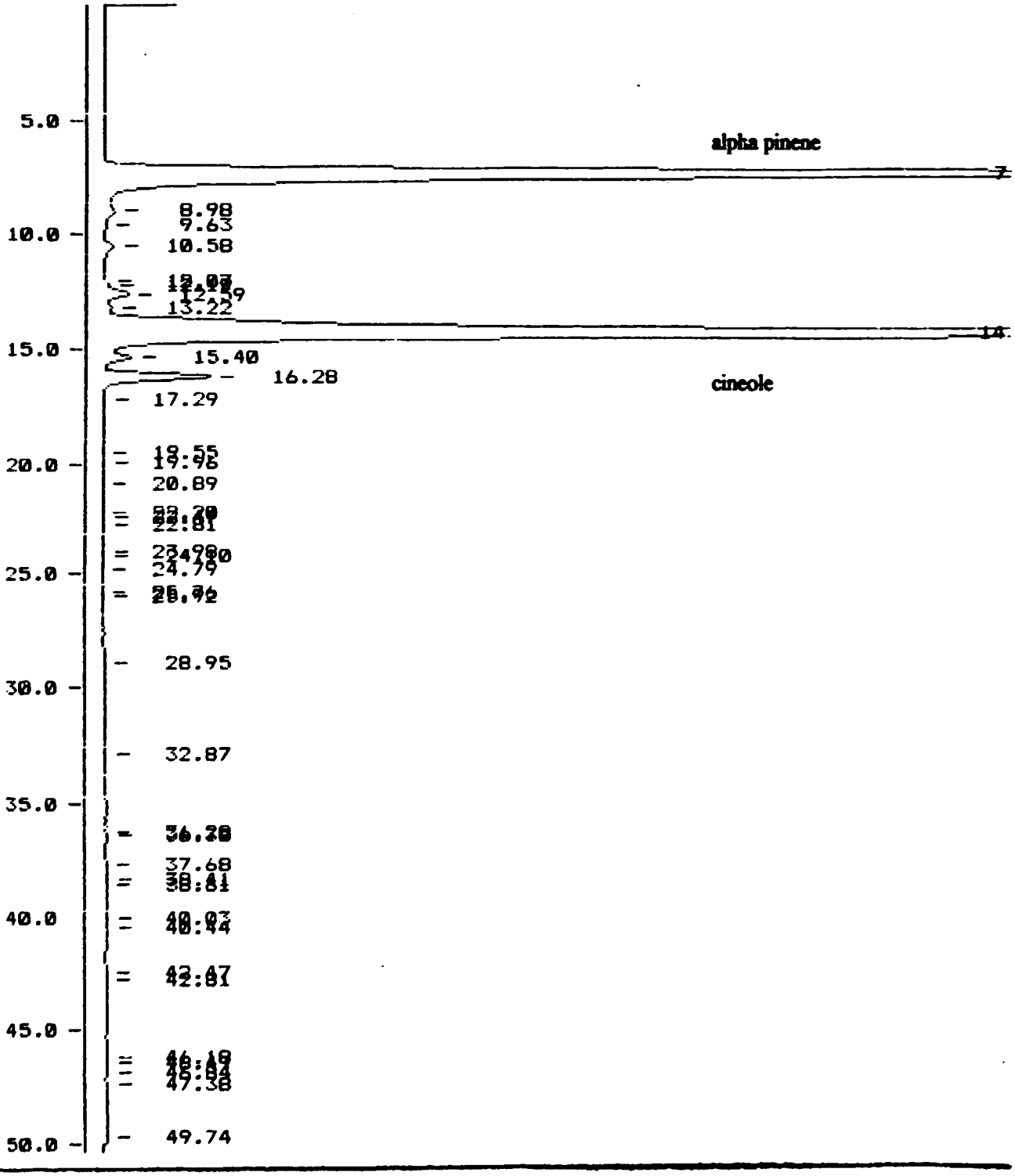
DATE 10-19-1995 TIME 14:49:38

TEXT:EUC-IND CINEOLE?

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.06	180	18	BV	-19	5	0.0313
2	0.51	23	1	BV	-2	9	0.0041
3	3.59	20	2	BV	-0	8	0.0035
4	5.07	22	2	BV	-1	14	0.0038
5	7.29	376469	6361	BV	-0	118	65.7300
6	8.83	1151	25	BB	37	50	0.2009
7	10.37	1243	25	BB	2	50	0.2171
8	11.89	229	9	BV	2	19	0.0400
9	12.36	2083	48	VV	12	48	0.3636
10	13.00	84	4	VB	13	19	0.0147
11	14.08	179919	3446	BV	18	103	31.4131
12	15.18	1498	40	VV	23	43	0.2615
13	16.07	9607	231	BB	8	56	1.6773
14	20.49	18	2	BV	-1	8	0.0031
15	22.16	17	2	BV	0	8	0.0029
16	23.48	19	1	BV	0	12	0.0033
17	27.05	16	1	BV	5	7	0.0028
18	29.98	15	2	BV	10	9	0.0026
19	37.06	16	1	BV	13	10	0.0027
20	38.97	15	1	BB	14	11	0.0026
21	41.03	19	1	BV	13	12	0.0033
22	41.39	26	1	BV	13	12	0.0045
23	41.85	19	2	BV	14	10	0.0034
24	45.93	17	1	BV	13	11	0.0029
25	47.91	27	2	BV	13	13	0.0048

Fraction 3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45.0
50.0
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9.63
10.58
13.22
15.40
16.28
17.29
20.89
24.79
28.95
32.87
37.68
49.74

Fraction 3

INTEGRATION REPORT FOR FILE EUC-IND6

SAMPLE ID = EUC-IND6 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 100 3 .1 0

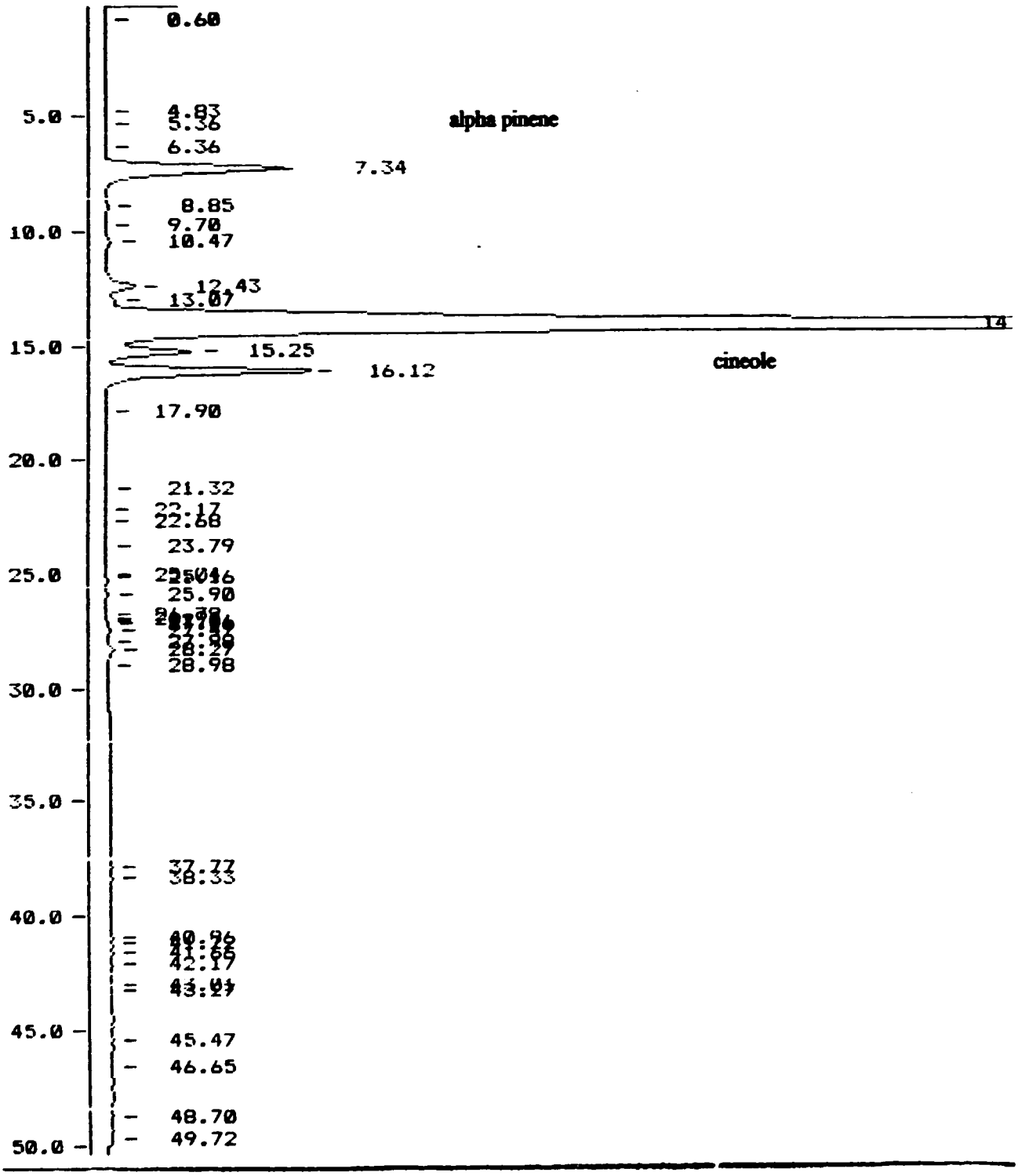
DATE 10-19-1995 TIME 16:22:46

TEXT:EUC-IND FRACTION 3

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.06	124	9	BV	-7	7	0.0291
2	7.47	173879	3243	BB	3	111	40.7647
3	8.98	45	3	BB	21	21	0.0105
4	9.63	16	1	BB	8	11	0.0037
5	10.58	1297	26	BB	4	53	0.3040
6	12.08	103	7	BV	6	11	0.0240
7	12.19	31	2	VV	15	8	0.0072
8	12.59	2396	61	VB	18	41	0.5618
9	13.22	122	5	BB	15	20	0.0286
10	14.33	232943	4493	BV	21	104	54.6118
11	15.40	2110	57	VV	31	45	0.4947
12	16.28	12694	300	VV	7	73	2.9761
13	17.29	20	1	BV	2	8	0.0048
14	19.55	17	1	BV	2	12	0.0041
15	19.96	21	2	BV	2	12	0.0048
16	20.89	18	1	BV	1	10	0.0041
17	22.20	52	3	BV	1	17	0.0122
18	22.47	77	3	VV	1	17	0.0181
19	22.81	30	2	BV	2	9	0.0071
20	23.98	16	1	BV	3	6	0.0037
21	24.10	18	1	VB	5	11	0.0042
22	24.79	22	3	BV	0	8	0.0051
23	25.76	31	1	BV	2	9	0.0072
24	25.93	61	5	VV	-1	7	0.0142
25	28.95	18	1	BV	7	9	0.0043
26	32.87	15	2	BV	10	8	0.0036
27	36.28	23	2	BV	13	9	0.0054
28	36.38	15	1	VV	14	9	0.0036
29	37.68	25	3	BV	14	8	0.0060
30	38.41	19	2	BV	14	11	0.0045
31	38.61	15	1	VV	14	10	0.0035
32	40.03	58	4	BV	14	13	0.0136
33	40.44	16	1	BB	15	10	0.0038
34	42.47	16	2	BV	12	7	0.0036
35	42.81	32	3	BB	13	14	0.0075
36	46.19	19	2	BV	13	8	0.0045
37	46.49	49	3	BV	16	19	0.0115
38	46.84	31	2	VB	12	14	0.0073
39	47.38	49	3	BV	14	12	0.0115

Fraction 4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12.43
13.07
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15.25
cineole
16.12
17.90
21.32
22.17
22.68
23.79
25.90
28.98
37.77
38.33
40.96
42.17
45.47
46.65
48.70
49.72
5.0
10.0
15.0
20.0
25.0
30.0
35.0
40.0
45.0
50.0

Fraction 4

INTEGRATION REPORT FOR FILE EUC-IND7

SAMPLE ID = EUC-IND7 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 100 3 .1 0

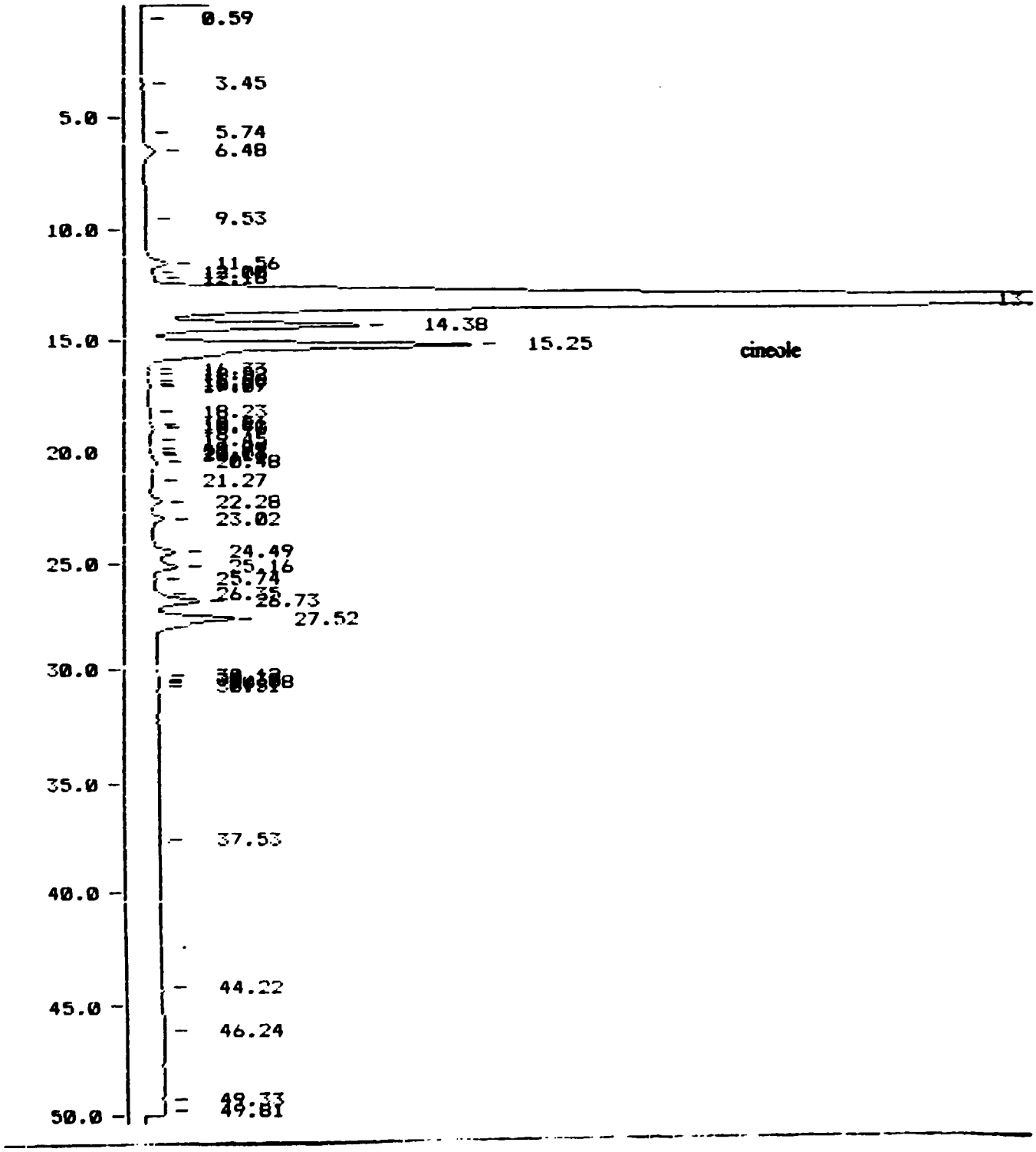
DATE 10-19-1995 TIME 18:21:53

TEXT:EUC-IND FRACTION4

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.06	156	13	BV	-10	6	0.0366
2	0.60	38	2	BV	1	12	0.0089
3	4.83	29	2	BV	0	10	0.0068
4	5.36	30	2	BV	-0	13	0.0070
5	6.36	17	2	BB	-1	13	0.0039
6	7.34	26305	483	BV	6	85	6.1569
7	8.85	31	2	BB	5	19	0.0073
8	9.70	24	2	BV	1	13	0.0057
9	10.47	139	6	BB	2	31	0.0326
10	12.43	4387	84	BV	2	67	1.0269
11	13.08	110	5	VB	16	22	0.0259
12	14.24	362679	6863	BV	26	102	84.8884
13	15.25	7450	199	VV	57	46	1.7437
14	16.12	24537	571	VB	14	76	5.7430
15	17.90	15	1	BV	1	8	0.0036
16	21.32	86	3	BB	2	23	0.0201
17	22.18	20	1	BV	0	10	0.0046
18	22.68	20	2	BV	1	12	0.0046
19	23.79	85	5	BV	2	13	0.0198
20	25.04	24	2	BV	2	8	0.0057
21	25.16	27	2	VV	5	7	0.0063
22	25.90	18	1	BB	9	10	0.0042
23	26.79	21	2	BV	3	10	0.0049
24	26.94	20	2	VV	3	7	0.0046
25	27.06	19	3	VV	6	7	0.0045
26	27.16	21	2	VV	8	8	0.0050
27	27.47	117	5	VB	9	22	0.0273
28	27.98	25	2	BV	7	8	0.0058
29	28.28	471	15	VB	10	31	0.1103
30	28.98	24	1	BV	7	9	0.0056
31	37.77	18	1	BB	19	9	0.0042
32	38.33	39	3	BB	17	17	0.0090
33	40.96	19	2	BB	18	11	0.0045
34	41.23	19	1	BV	18	9	0.0045
35	41.66	17	1	BB	17	14	0.0040
36	42.17	31	2	BV	18	12	0.0072
37	43.01	50	3	BV	17	16	0.0116
38	43.27	23	2	VV	17	10	0.0053
39	45.47	42	3	BV	18	14	0.0098
40	46.65	15	1	BB	17	11	0.0035
41	48.70	25	2	BB	18	16	0.0058

Fraction 5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24.49
25.74
28.73
27.52
37.53
44.22
46.24
48.33
49.81
5.0
10.0
15.0
20.0
25.0
30.0
35.0
40.0
45.0
50.0

Fraction 5

INTEGRATION REPORT FOR FILE EUC-IND7

SAMPLE ID = EUC-IND7 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 20 3 .1 0

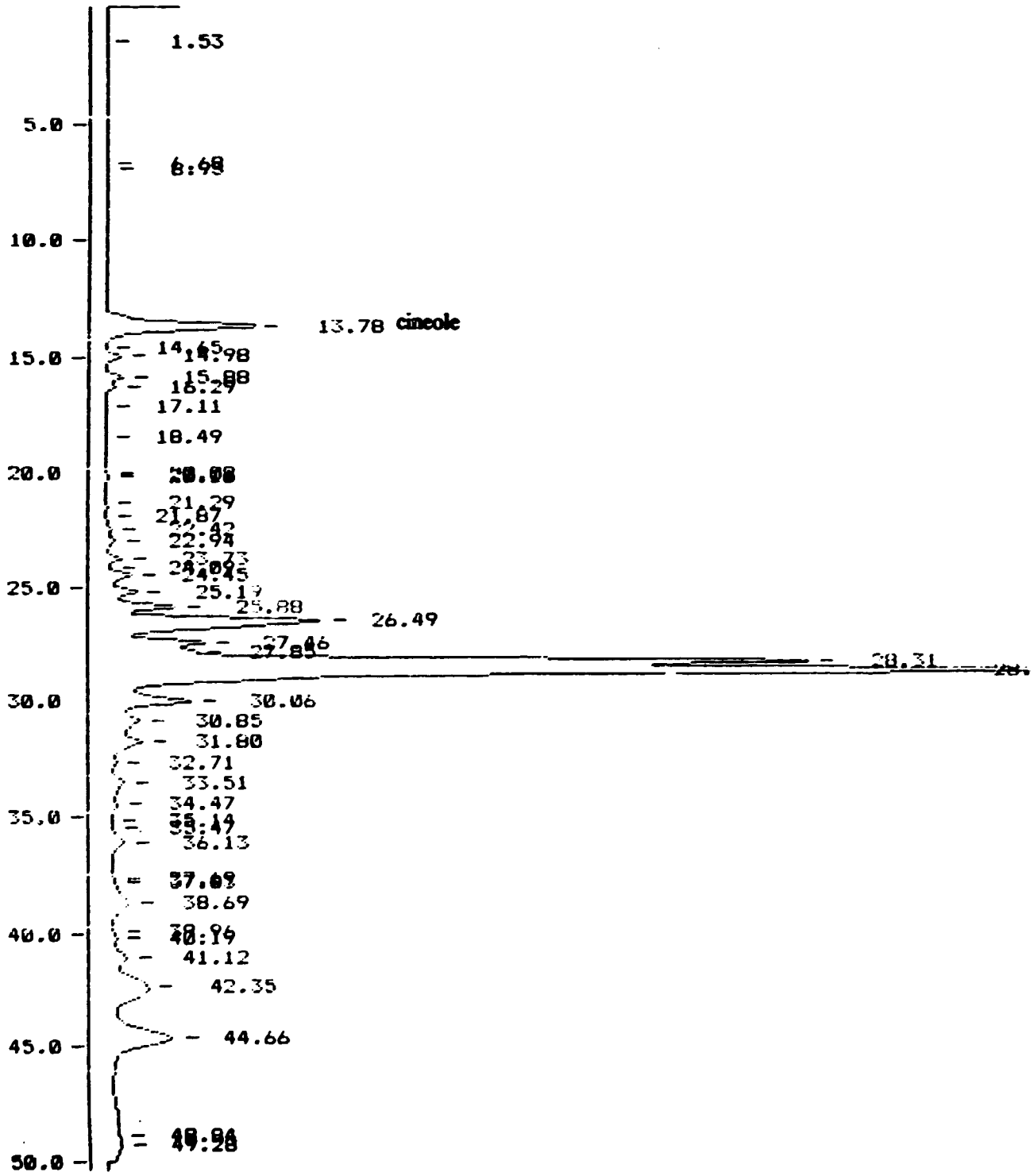
DATE 10-20-1995 TIME 05:51:09

TEXT:EUC-INDUSTRIAL BATCH A9

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.06	131	14	V	-14	5	0.0248
2	0.59	19	1	BV	-1	11	0.0036
3	3.45	21	1	BV	5	12	0.0040
4	5.74	16	1	BV	8	10	0.0030
5	6.48	1445	29	BB	10	52	0.2736
6	9.53	19	1	BV	15	10	0.0035
7	11.56	3098	58	BV	16	64	0.5864
8	12.00	28	2	VV	30	8	0.0054
9	12.18	151	7	VV	34	15	0.0286
10	13.37	435342	7958	VV	40	105	82.4163
11	14.38	21425	552	VV	96	49	4.0560
12	15.25	42227	880	VB	46	79	7.9941
13	16.33	46	3	BB	22	11	0.0087
14	16.53	21	3	BV	22	10	0.0039
15	16.88	39	3	BV	21	8	0.0073
16	16.99	27	3	VV	24	7	0.0050
17	17.07	16	2	VV	27	7	0.0030
18	18.23	25	2	BV	25	7	0.0047
19	18.81	132	8	BV	28	14	0.0251
20	18.90	99	3	VV	38	20	0.0188
21	19.45	22	2	BB	29	12	0.0041
22	19.92	22	2	BV	26	7	0.0042
23	20.03	29	3	VV	29	7	0.0055
24	20.13	16	2	VV	32	7	0.0030
25	20.48	418	12	BV	38	33	0.0791
26	21.27	62	4	BV	30	15	0.0118
27	22.28	1195	31	BB	30	41	0.2262
28	23.03	1531	40	BV	30	42	0.2899
29	24.49	2595	63	BV	36	40	0.4914
30	25.16	2019	57	VV	57	42	0.3821
31	25.74	36	2	BV	41	13	0.0067
32	26.35	297	16	BV	43	14	0.0563
33	26.73	4054	110	VB	61	43	0.7675
34	27.52	11061	216	BB	52	71	2.0940
35	30.12	23	1	BB	49	13	0.0044
36	30.30	62	4	BV	50	6	0.0117
37	30.38	318	73	VV	39	8	0.0603
38	30.51	59	15	VV	47	8	0.0112
39	37.53	20	2	BV	50	12	0.0039
40	44.22	17	2	BV	53	11	0.0031
41	46.24	22	2	BV	54	13	0.0042
42	49.33	18	1	BV	54	10	0.0035

Residue


1.53
6.68
13.78 cineole
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25.88
26.49
27.46
28.31
30.06
30.85
31.80
32.71
33.51
34.47
36.13
38.69
41.12
42.35
44.66
5.0
10.0
15.0
20.0
25.0
30.0
35.0
40.0
45.0
50.0

Residue

INTEGRATION REPORT FOR FILE EUC-IND8

SAMPLE ID = EUC-IND8 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 20 3 .1 0

DATE 10-20-1995 TIME 07:03:24

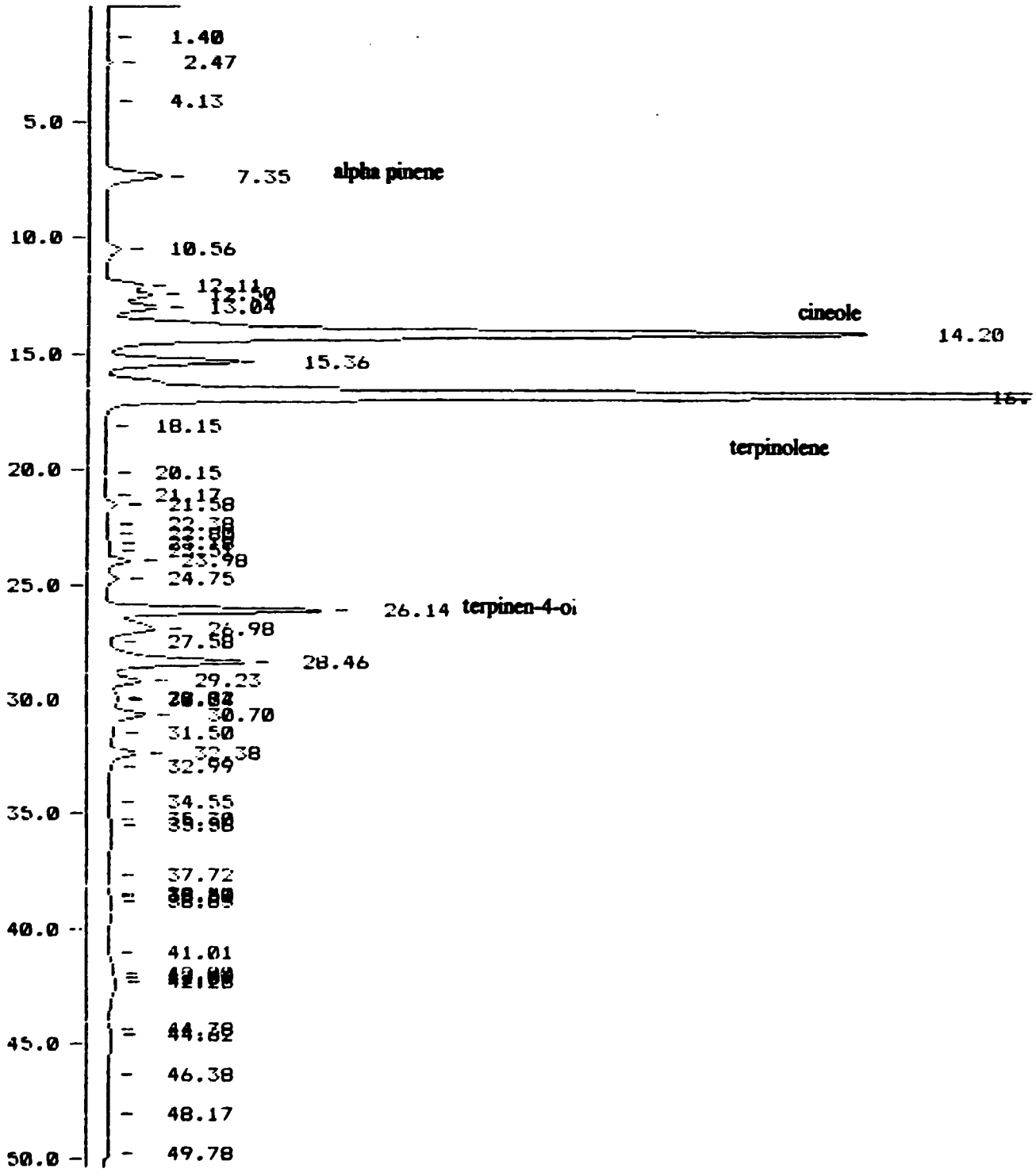
TEXT:EUC-INDUSTRIAL BATCH A9 (RESIDUAL FRACTION IN POT)

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.12	190	11	BB	-20	11	0.0770
2	1.53	26	2	BV	-4	15	0.0104
3	6.68	21	2	BV	-2	8	0.0085
4	6.93	44	2	VB	0	17	0.0178
5	13.78	22860	414	BV	0	97	9.2860
6	14.65	-9	0	TAN	2	6	-0.0036
7	14.98	1561	39	TVV	6	50	0.6341
8	15.88	1909	48	BV	3	36	0.7753
9	16.29	432	14	VB	22	34	0.1753
10	17.11	15	1	BV	-1	10	0.0061
11	18.49	31	1	BV	-1	13	0.0126
12	20.08	132	8	BV	4	14	0.0538
13	20.16	37	1	VB	13	20	0.0152
14	21.29	25	3	BV	4	8	0.0102
15	21.87	22	2	BV	3	8	0.0088
16	22.43	159	9	BV	11	13	0.0646
17	22.94	593	17	BB	15	37	0.2407
18	23.73	1245	39	BV	11	33	0.5056
19	24.09	18	1	VV	13	8	0.0073
20	24.45	2605	60	VV	15	41	1.0583
21	25.19	2530	68	VV	26	40	1.0279
22	25.88	5780	160	VV	39	38	2.3479
23	26.49	28686	514	VV	68	61	11.6526
24	27.46	7637	203	VV	76	31	3.1022
25	27.85	848	42	VV	206	16	0.3447
26	28.31	61214	1664	VV	247	34	24.8661
27	28.73	70286	2007	VV	1432	67	28.5514
28	30.06	7191	169	VV	83	55	2.9209
29	30.85	1255	35	VB	56	41	0.5099
30	31.80	2896	60	BB	53	59	1.1763
31	32.71	239	7	BB	28	31	0.0969
32	33.51	2142	32	BV	21	61	0.8699
33	34.47	31	2	BB	29	17	0.0128
34	35.14	16	1	BV	19	7	0.0064
35	35.47	2	1	TAN	21	7	0.0007
36	36.13	2276	33	TVB	23	80	0.9247
37	37.69	121	6	BV	21	20	0.0492
38	37.83	24	2	VV	28	8	0.0095
39	38.69	3678	40	BB	31	95	1.4742
40	39.96	24	2	BV	25	7	0.0098
41	40.19	82	4	VV	28	16	0.0334
42	41.12	1413	24	BB	30	58	0.5739
43	42.35	814	10	BB	37	50	0.3307
44	44.66	15021	153	BB	34	121	6.1017
45	48.84	55	3	BV	33	13	0.0224

TEA TREE OIL

Whole oil



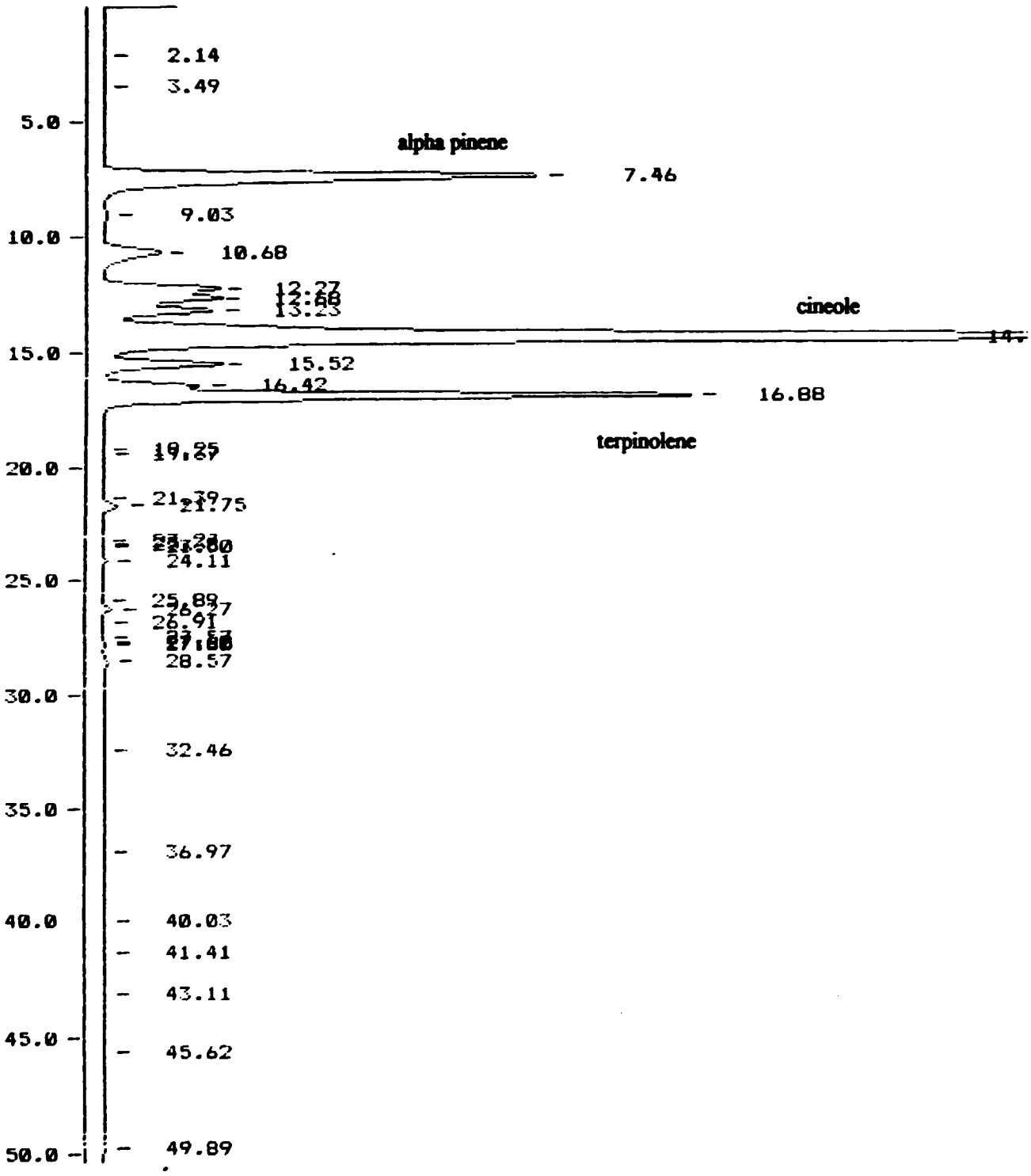
TEA TREE OIL

Whole oil

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.04	76	8	BB	-7	9	0.0217
2	1.40	21	1	BB	1	14	0.0061
3	2.47	281	14	BB	2	23	0.0803
4	4.13	24	2	BB	2	14	0.0067
5	7.35	8030	151	BV	4	72	2.2901
6	10.56	1841	34	BB	3	62	0.5249
7	12.11	4115	101	BV	3	38	1.1736
8	12.50	1735	57	VV	83	30	0.4949
9	13.04	3438	98	TAN	61	37	0.9806
10	14.20	108411	2061	TVV	31	131	30.9166
11	15.36	14453	351	VV	20	55	4.1218
12	16.78	147319	3523	VB	12	109	42.0123
13	18.15	17	1	BV	2	11	0.0048
14	20.15	18	2	BV	3	7	0.0051
15	21.17	16	2	BV	3	7	0.0046
16	21.58	1144	28	VB	5	44	0.3263
17	22.38	32	3	BV	7	9	0.0092
18	22.80	16	1	BB	11	7	0.0045
19	23.18	60	4	BV	11	13	0.0170
20	23.51	47	3	BB	16	16	0.0133
21	23.98	2169	63	BV	13	41	0.6185
22	24.75	482	18	BB	17	25	0.1375
23	26.14	22679	591	BV	9	52	6.4675
24	26.98	5625	104	VB	56	57	1.6042
25	27.58	22	2	BV	20	8	0.0063
26	28.46	17726	373	VV	22	74	5.0551
27	29.23	2757	74	VV	36	43	0.7863
28	29.93	260	10	VV	29	22	0.0741
29	30.04	22	2	VB	38	11	0.0063
30	30.70	3685	88	BB	32	55	1.0509
31	31.50	125	5	BV	27	29	0.0356
32	32.38	3497	76	VV	19	56	0.9972
33	32.99	19	1	BB	24	13	0.0055
34	34.55	16	1	BB	16	12	0.0045
35	35.30	36	2	BV	17	11	0.0102
36	35.58	102	3	VV	20	20	0.0291
37	37.72	19	1	BV	16	11	0.0054
38	38.50	17	2	BV	17	9	0.0047
39	38.58	20	1	VV	18	8	0.0056
40	38.83	33	2	VV	19	11	0.0095
41	41.01	18	2	BV	16	10	0.0051
42	42.00	36	2	BV	28	12	0.0101
43	42.09	16	2	VV	30	6	0.0046
44	42.28	62	3	VV	31	13	0.0176
45	44.38	20	1	BV	18	10	0.0057
46	44.62	61	3	VV	19	16	0.0174
47	46.38	15	1	BV	16	9	0.0043
48	48.17	23	1	BV	16	16	0.0067

Fraction 1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10.68
12.27
13.23
cineole
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15.52
16.42
16.88
terpinolene
20.0
21.75
24.11
25.0
25.89
28.57
30.0
32.46
35.0
36.97
40.0
40.03
41.41
43.11
45.0
45.62
50.0
49.89

Fraction 1

INTEGRATION REPORT FOR FILE TTLHICI1

SAMPLE ID = TTLHICI1 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 20 3 .1 0

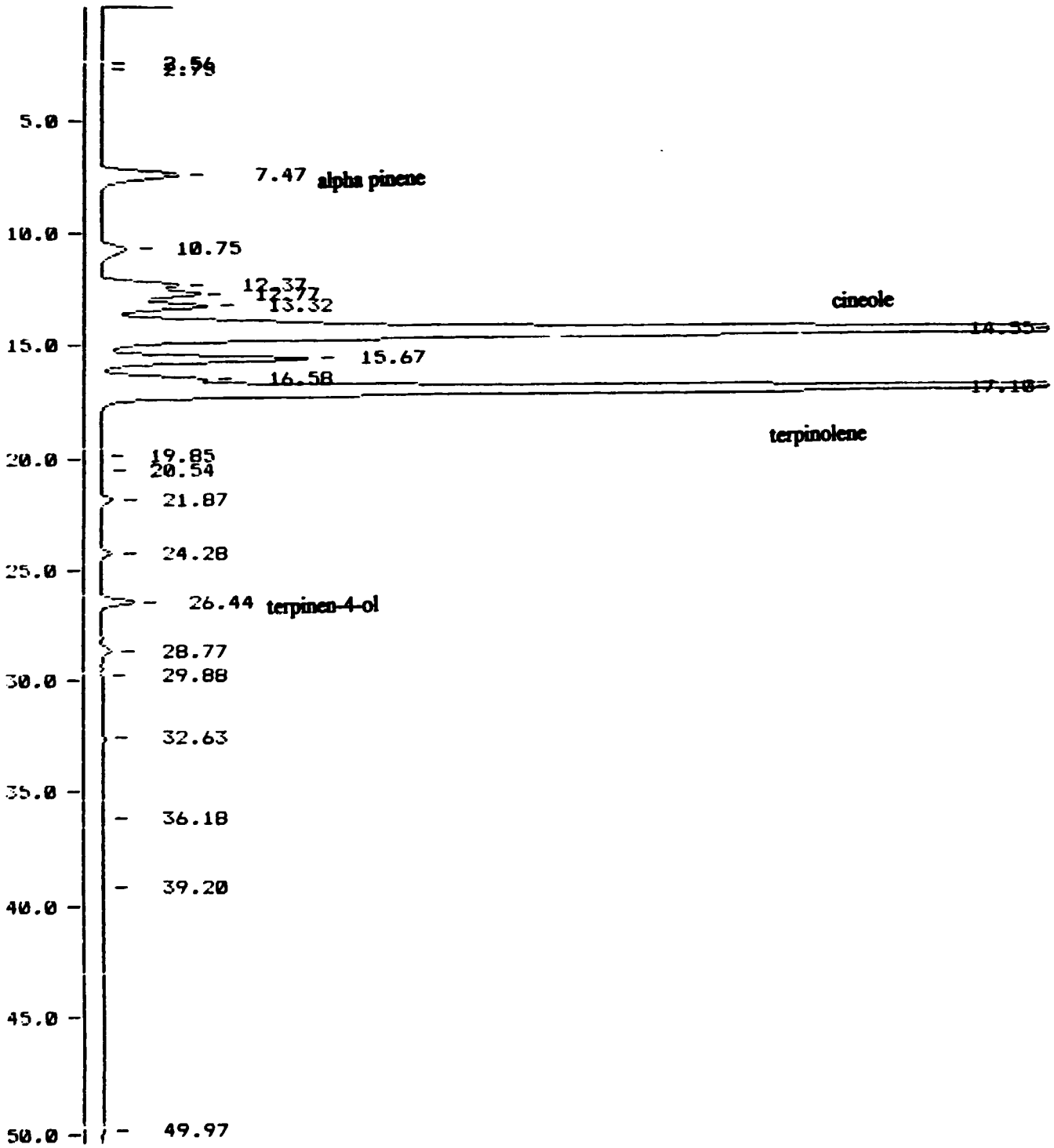
DATE 10-20-1995 TIME 10:18:38

TEXT:TEA-TREE HI CIN FIRST FRACTION

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.07	54	4	BV	-5	7	0.0139
2	2.14	28	2	BV	-1	13	0.0073
3	3.49	23	1	BV	-1	11	0.0059
4	7.46	66010	1199	BB	-2	106	16.9984
5	9.03	40	2	BB	7	17	0.0103
6	10.68	9669	158	BB	0	79	2.4899
7	12.27	14066	324	BV	2	41	3.6223
8	12.68	3778	129	VV	239	30	0.9729
9	13.23	6893	197	TAN	141	36	1.7751
10	14.43	208998	4079	TVV	57	132	53.8200
11	15.53	12274	307	VV	34	51	3.1607
12	16.42	8513	252	VV	12	32	2.1923
13	16.88	54326	1467	VB	232	62	13.9898
14	19.25	21	3	BV	0	7	0.0053
15	19.37	16	1	VB	3	11	0.0040
16	21.39	33	2	BV	-1	8	0.0085
17	21.75	1721	43	VV	4	46	0.4432
18	23.28	22	2	BV	1	8	0.0056
19	23.38	15	2	VV	3	7	0.0039
20	23.50	16	2	VV	5	8	0.0041
21	24.11	413	12	BV	2	30	0.1064
22	25.89	24	2	BV	1	8	0.0062
23	26.27	1000	27	VV	3	41	0.2576
24	26.91	18	2	BV	3	8	0.0047
25	27.57	24	2	BV	4	9	0.0063
26	27.68	17	2	VV	6	7	0.0043
27	27.80	18	2	VB	9	11	0.0048
28	28.57	161	7	BB	9	22	0.0415
29	32.46	17	2	BV	7	8	0.0043
30	36.97	16	1	BV	7	13	0.0041
31	40.03	24	1	BV	8	11	0.0062
32	41.41	18	1	BV	8	10	0.0047
33	43.11	40	2	BV	9	16	0.0104
34	45.62	20	2	BV	9	10	0.0052

Fraction 2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15.67
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19.85
20.54
20.0
21.87
24.28
25.0
26.44 terpinen-4-ol
28.77
29.88
30.0
32.63
35.0
36.18
39.20
40.0
45.0
49.97
50.0

Fraction 2

INTEGRATION REPORT FOR FILE TTLHICI2

AMPLE ID = TTLHICI2 PLOT Squeeze = 10 times ATTEN = 4

ampling Frequency [2] Hz Tg/Pk/Sp/Wth= 20 3 .1 0

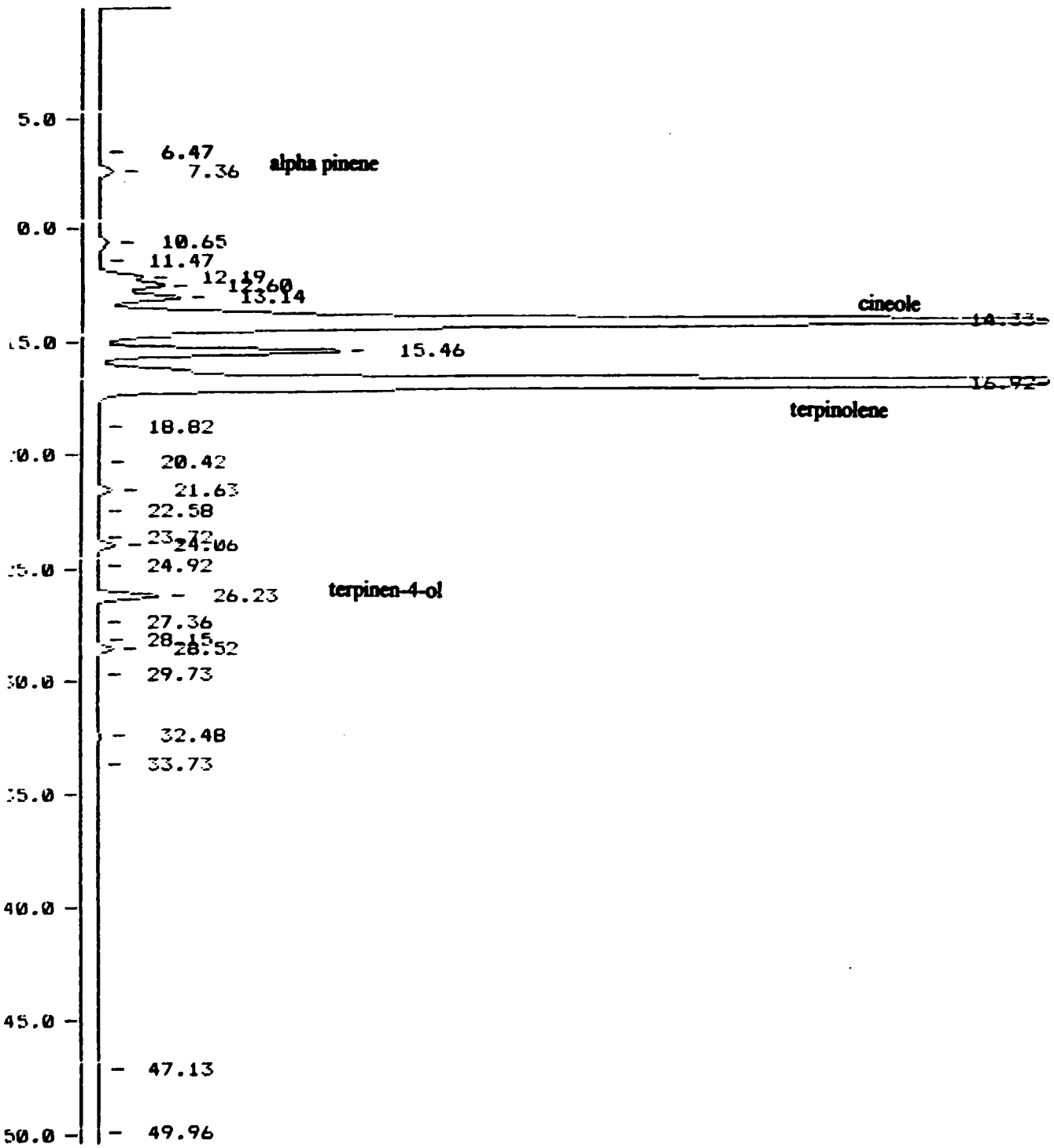
ATE 10-20-1995 TIME 11:36:05

TEXT:TEA-TREE HI CIN SECOND FRACTION

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.08	171	14	BV	-14	7	0.0378
2	2.56	19	1	BV	-2	11	0.0041
3	2.75	23	2	VV	-2	9	0.0050
4	7.47	12151	220	BB	0	74	2.6827
5	10.75	4442	74	BB	1	76	0.9807
6	12.37	9158	219	BV	3	37	2.0218
7	12.78	3747	123	VV	182	31	0.8271
8	13.33	7270	202	TAN	127	38	1.6049
9	14.55	223585	4273	TVV	60	133	49.3617
10	15.67	22213	545	VV	38	55	4.9041
11	16.58	8458	277	VV	18	28	1.8674
12	17.10	154337	4009	VV	284	82	34.0736
13	19.85	17	2	BV	-1	8	0.0037
14	20.54	28	3	BV	2	7	0.0062
15	21.87	1393	35	BB	2	44	0.3075
16	24.28	936	29	BB	8	33	0.2067
17	26.44	3598	97	BV	7	47	0.7943
18	28.78	1275	29	BV	5	47	0.2814
19	29.88	47	3	BV	5	12	0.0104
20	32.63	51	4	BV	11	13	0.0112
21	36.18	16	1	BV	10	10	0.0035
22	39.20	18	1	BV	9	10	0.0040

Fraction 3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12.19
12.60
13.14
cineole
15.46
terpinolene
18.82
20.42
21.63
22.58
23.72
24.06
24.92
26.23
terpinen-4-ol
27.36
28.15
28.52
29.73
32.48
33.73
47.13
49.96

Fraction 3

INTEGRATION REPORT FOR FILE TTLHIC3

SAMPLE ID = TTLHIC3 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 20 3 .1 0

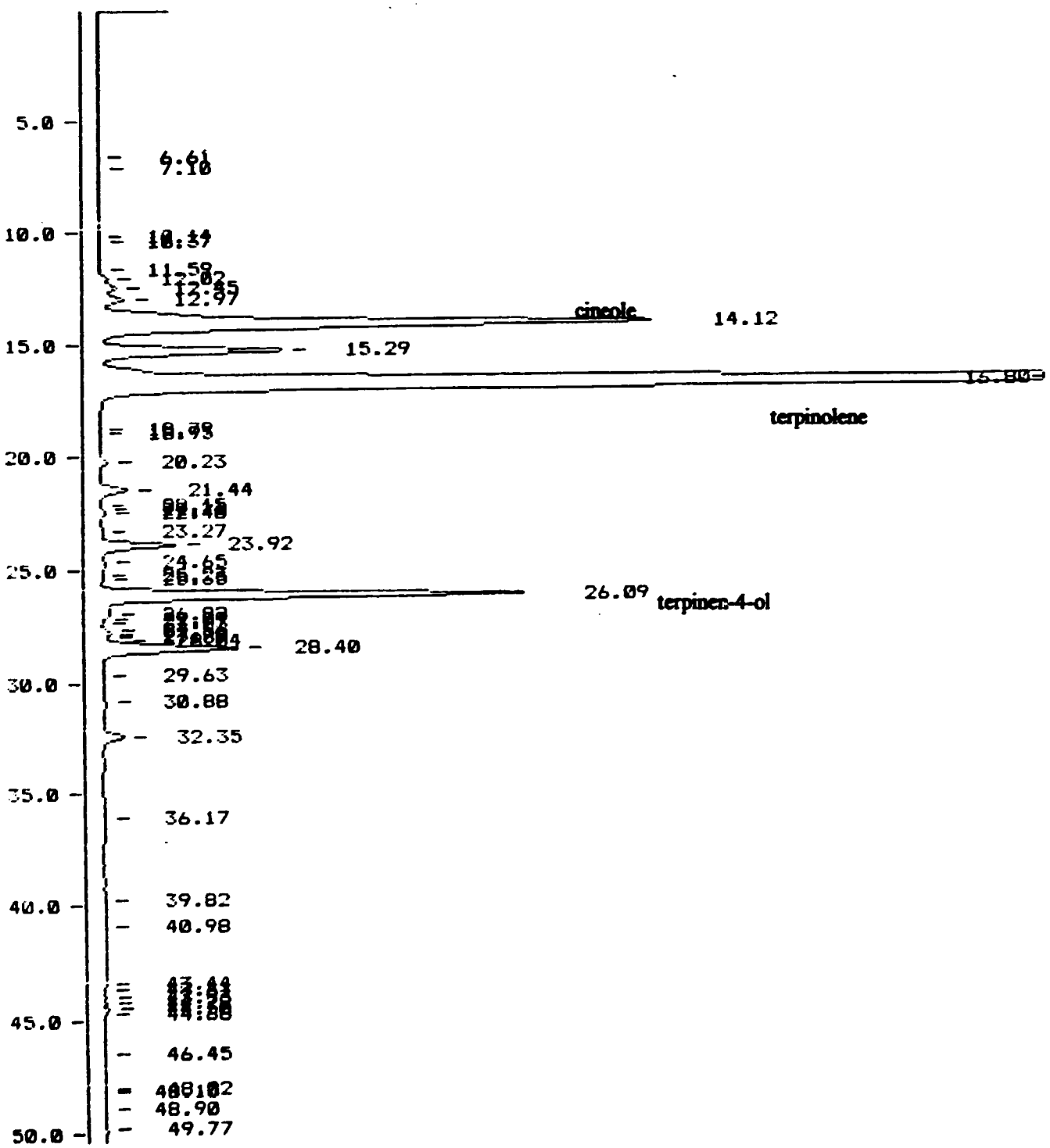
DATE 10-20-1995 TIME 15:27:23

TEXT:TEA-TREE HI CINEOLE THIRD FRACTION

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.07	115	9	BV	-9	7	0.0222
2	6.47	21	2	BV	-2	10	0.0040
3	7.36	2154	42	BB	-1	59	0.4166
4	10.65	1167	24	BB	-2	49	0.2257
5	11.47	17	1	BV	-1	13	0.0033
6	12.19	4806	122	BV	4	31	0.9294
7	12.60	2719	89	VV	106	31	0.5258
8	13.14	5553	158	TAN	89	38	1.0738
9	14.33	189848	3656	TVV	46	131	36.7125
10	15.46	27080	661	VV	32	56	5.2366
11	16.92	270922	6357	VB	17	115	52.3902
12	18.82	16	2	BV	-4	9	0.0032
13	20.42	329	10	BB	-3	32	0.0636
14	21.63	1756	43	BB	-3	46	0.3397
15	22.58	35	3	BV	-1	11	0.0068
16	23.72	31	3	BV	-3	8	0.0060
17	24.06	2020	57	VV	-0	43	0.3906
18	24.92	18	1	BV	-2	8	0.0034
19	26.23	6705	177	BV	-1	46	1.2967
20	27.36	27	3	BV	-2	8	0.0052
21	28.15	40	3	BV	2	10	0.0078
22	28.53	1586	40	VB	6	44	0.3068
23	29.73	22	2	BV	2	11	0.0042
24	32.48	97	3	BB	3	25	0.0188
25	33.73	21	2	BV	2	11	0.0041
26	47.13	15	1	BB	3	12	0.0030

Fraction 4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50.0
cineole
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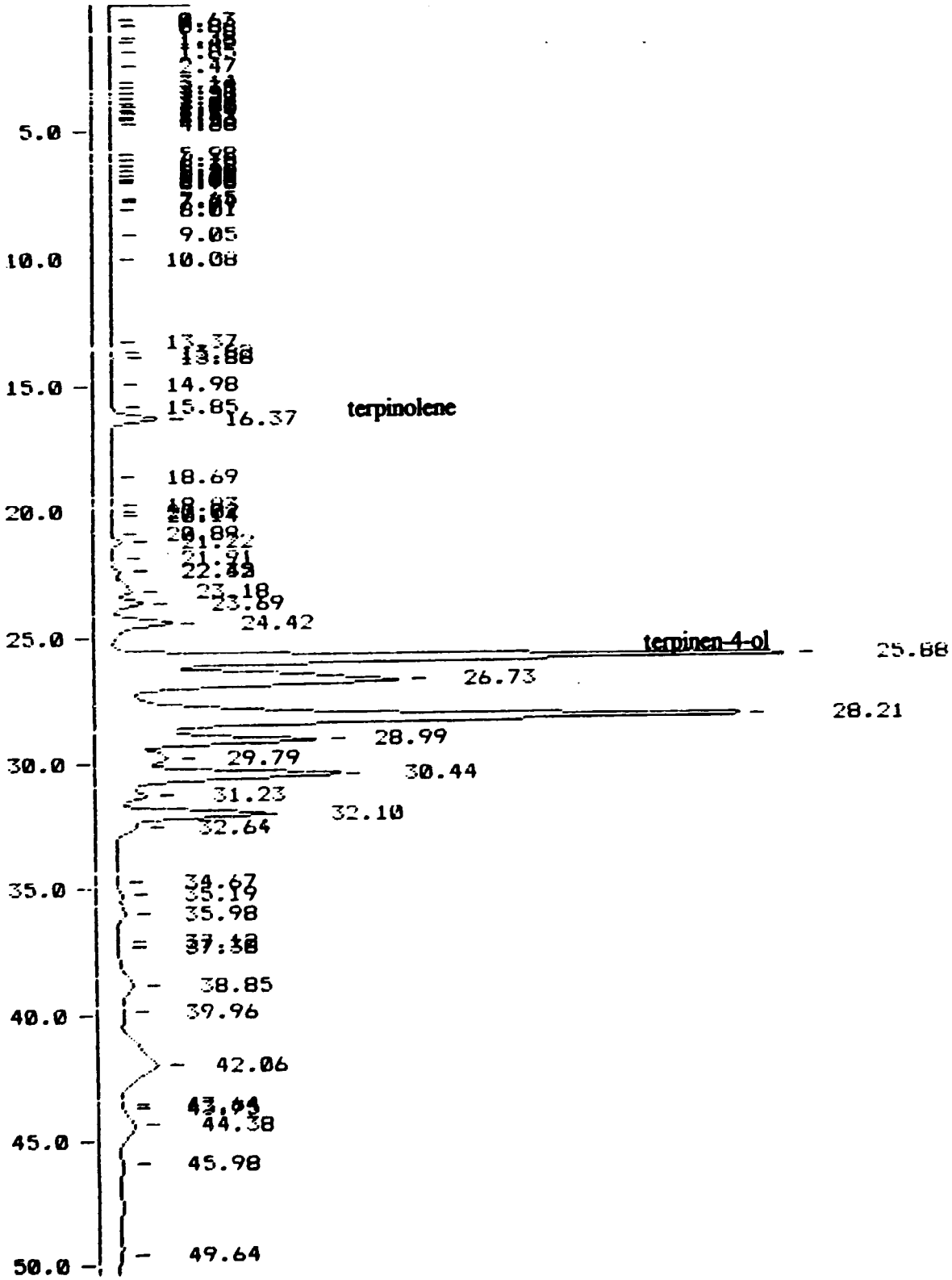
15.29
terpinolene
20.23
21.44
23.27
23.92
24.65
26.09
terpinen-4-ol
28.40
29.63
30.88
32.35
36.17
39.82
40.98
46.45
48.90
49.77

Fraction 4

PLOTTING IS FROM 0.00 to 50.00 MINUTES

PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.05	70	8	BB	-7	8	0.0136
2	6.61	15	1	BV	1	8	0.0029
3	7.10	23	2	BV	3	9	0.0046
4	10.14	25	2	BV	1	13	0.0049
5	10.37	35	2	VV	2	10	0.0067
6	11.59	15	1	BV	1	8	0.0030
7	12.03	182	7	BB	6	23	0.0354
8	12.45	903	28	BV	24	31	0.1755
9	12.97	1842	52	TAN	28	38	0.3578
10	14.1[illegible]	80858	1519	TVV	18	129	15.7050
11	15.29	21545	512	VV	17	60	4.1846
12	16.80	334802	7805	VB	16	110	65.0279
13	18.79	70	5	BV	-4	11	0.0135
14	18.95	33	3	VV	-0	11	0.0065
15	20.23	836	24	BB	4	37	0.1624
16	21.44	3269	81	BV	4	51	0.6349
17	22.15	28	3	BV	3	7	0.0054
18	22.26	32	3	VV	7	7	0.0062
19	22.48	91	5	VB	11	16	0.0178
20	23.27	19	2	BV	7	7	0.0037
21	23.92	7563	205	BB	4	52	1.4689
22	24.65	120	6	BV	10	16	0.0234
23	25.23	32	3	BV	6	8	0.0062
24	25.35	27	3	VB	9	11	0.0052
25	26.09	44544	1158	BV	5	62	8.6517
26	26.92	38	3	BB	27	8	0.0073
27	27.09	36	5	BB	16	11	0.0071
28	27.33	92	7	BV	1	9	0.0178
29	27.66	700	29	VV	4	19	0.1359
30	27.78	77	10	VV	28	7	0.0149
31	27.89	37	6	VV	20	7	0.0071
32	28.04	290	28	VV	26	8	0.0563
33	28.40	12682	337	VB	66	53	2.4632
34	29.63	21	2	BV	6	10	0.0040
35	30.88	27	2	BV	13	8	0.0053
36	32.35	2504	59	BB	10	49	0.4863
37	36.17	291	24	BV	-14	8	0.0566
38	39.82	18	1	BV	7	11	0.0036
39	40.98	16	1	BV	7	11	0.0031
40	43.44	20	3	BV	7	10	0.0040
41	43.61	21	2	VV	5	9	0.0040
42	43.93	137	6	BV	5	16	0.0266
43	44.22	203	17	VV	0	13	0.0394
44	44.50	503	18	VV	1	21	0.0978
45	44.68	17	1	VV	10	10	0.0032
46	46.45	99	4	BV	2	19	0.0192
47	48.02	17	2	BV	5	8	0.0033
48	48.13	17	1	VV	5	10	0.0032
49	48.90	19	1	BV	4	11	0.0036

Residue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15.85
16.37
terpinolene
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24.42
terpinen-4-ol
25.88
26.73
28.21
28.99
29.79
30.44
31.23
32.10
35.98
38.85
39.96
42.06
44.38
45.98
49.64

Residue

INTEGRATION REPORT FOR FILE TTLHIC5

SAMPLE ID = TTLHIC5 PLOT Squeeze = 10 times ATTEN = 4

Sampling Frequency [2] Hz Tg/Pk/Sp/Wth= 20 3 .1 0

DATE 10-20-1995 TIME 18:00:11

TEXT:TEA-TREE HI CINEOLE THE REMAINDER IN POT

PLOTTING IS FROM 0.00 to 50.00 MINUTES

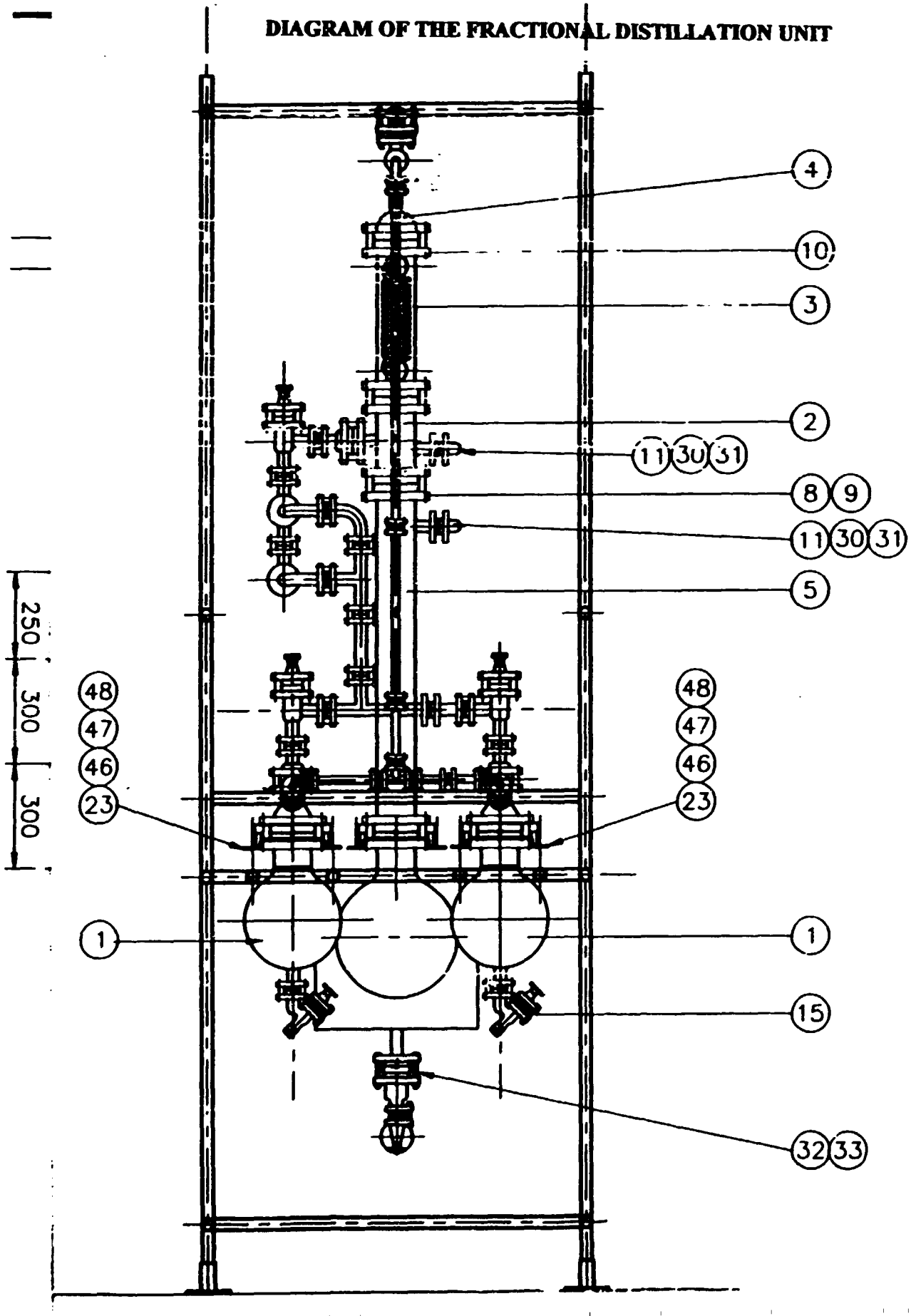
PK#	RT	AREA	HT	PK TYPE	B/Line	WTH	AREA%
1	0.18	183	7	BB	-9	18	0.0697
2	0.63	17	2	BV	-7	6	0.0065
3	0.86	48	2	VB	-6	17	0.0184
4	1.38	70	6	BV	-6	11	0.0268
5	1.45	17	2	VV	-1	8	0.0063
6	1.85	18	3	BV	-3	10	0.0069
7	2.47	15	1	BV	-2	13	0.0059
8	3.14	26	2	BB	-4	12	0.0098
9	3.39	27	2	BV	-5	26	0.0101
10	3.59	33	3	TAN	-5	14	0.0124
11	3.78	37	3	VV	-6	18	0.0141
12	3.93	23	4	TAN	-4	8	0.0088
13	4.05	22	2	VV	-5	16	0.0084
14	4.19	21	4	TAN	-5	7	0.0081
15	4.31	31	4	VV	-6	14	0.0117
16	4.43	14	4	TAN	-3	6	0.0054
17	4.53	27	1	VV	-6	9	0.0102
18	4.68	33	5	VV	-5	9	0.0126
19	5.98	17	2	BV	-4	21	0.0063
20	6.15	38	3	TAN	-6	11	0.0145
21	6.38	52	4	BV	-7	20	0.0199
22	6.56	15	3	TAN	-5	9	0.0056
23	6.72	16	2	VV	-6	8	0.0061
24	6.88	31	2	VV	-6	10	0.0118
25	6.98	35	2	VV	-6	12	0.0133
26	7.65	30	4	BV	-5	16	0.0113
27	7.74	17	3	TAN	-4	9	0.0066
28	8.01	34	3	BV	-6	11	0.0127
29	9.05	20	2	BB	-4	10	0.0074
30	10.08	18	1	BV	-5	9	0.0069

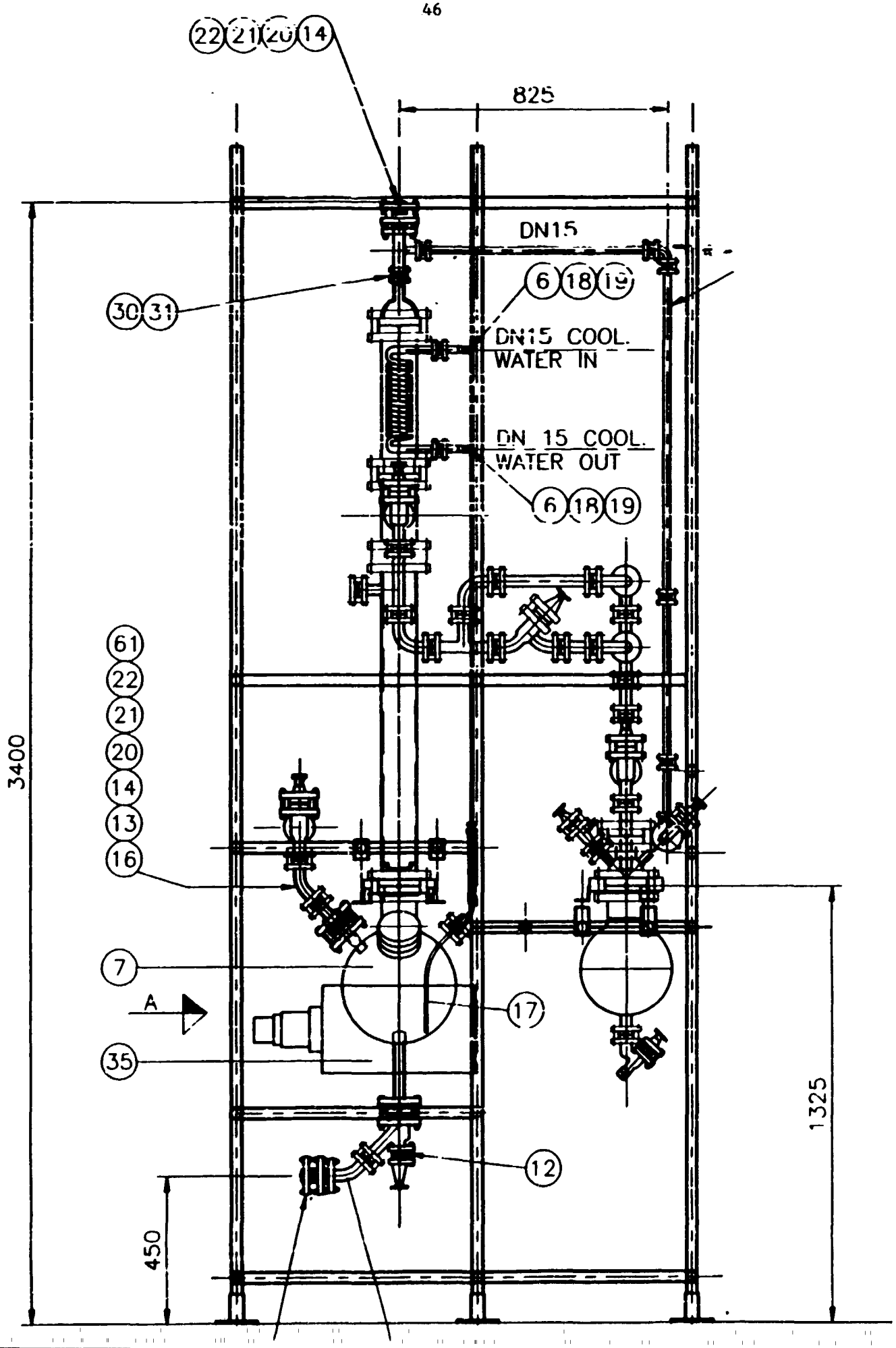
Residue continued

31	13.37	22	2	BV	-5	8	0.0085
32	13.82	219	12	BV	-2	17	0.0832
33	13.88	36	1	VV	9	20	0.0137
34	14.98	16	1	BB	-1	8	0.0061
35	15.85	139	9	BV	-3	14	0.0528
36	16.37	4536	116	BB	8	52	1.7241
37	18.69	25	2	BB	-7	14	0.0095
38	19.83	23	2	BV	-4	8	0.0089
39	20.03	43	3	VV	-1	11	0.0164
40	20.14	16	1	VB	0	13	0.0063
41	20.89	21	2	BV	-5	7	0.0080
42	21.22	1095	28	VV	-1	40	0.4161
43	21.91	162	9	BV	-3	15	0.0616
44	22.35	256	16	BV	5	14	0.0974
45	22.42	22	2	VB	23	11	0.0082
46	23.18	2169	39	BV	14	50	0.8244
47	23.69	2014	64	VV	23	35	0.7654
48	24.43	5443	136	BB	19	50	2.0686
49	25.88	66603	1723	BV	4	59	25.3134
50	26.73	33419	596	VV	160	62	12.7014
51	28.21	75234	1551	VV	58	84	28.5937
52	28.99	14015	385	VV	154	43	5.3265
53	29.79	3425	59	VV	78	43	1.3019
54	30.44	20261	491	VV	99	56	7.7004
55	31.23	1374	42	VV	58	35	0.5223
56	32.10	15714	352	VV	25	56	5.9722
57	32.64	510	14	VB	54	38	0.1938
58	34.68	22	1	BV	6	9	0.0085
59	35.19	246	10	BV	10	23	0.0934
60	35.98	132	4	BB	18	31	0.0501
61	37.12	34	3	BV	8	12	0.0128
62	37.38	47	2	VV	10	16	0.0177
63	38.85	323	6	BB	16	49	0.1226
64	39.96	15	1	BV	19	9	0.0057
65	42.06	13211	86	BB	18	160	5.0209
66	43.64	34	3	BV	14	10	0.0130
67	43.75	19	2	VV	18	7	0.0074
68	44.38	1129	22	VV	21	41	0.4290
69	45.98	104	4	BV	13	17	0.0395

ANNEX 7

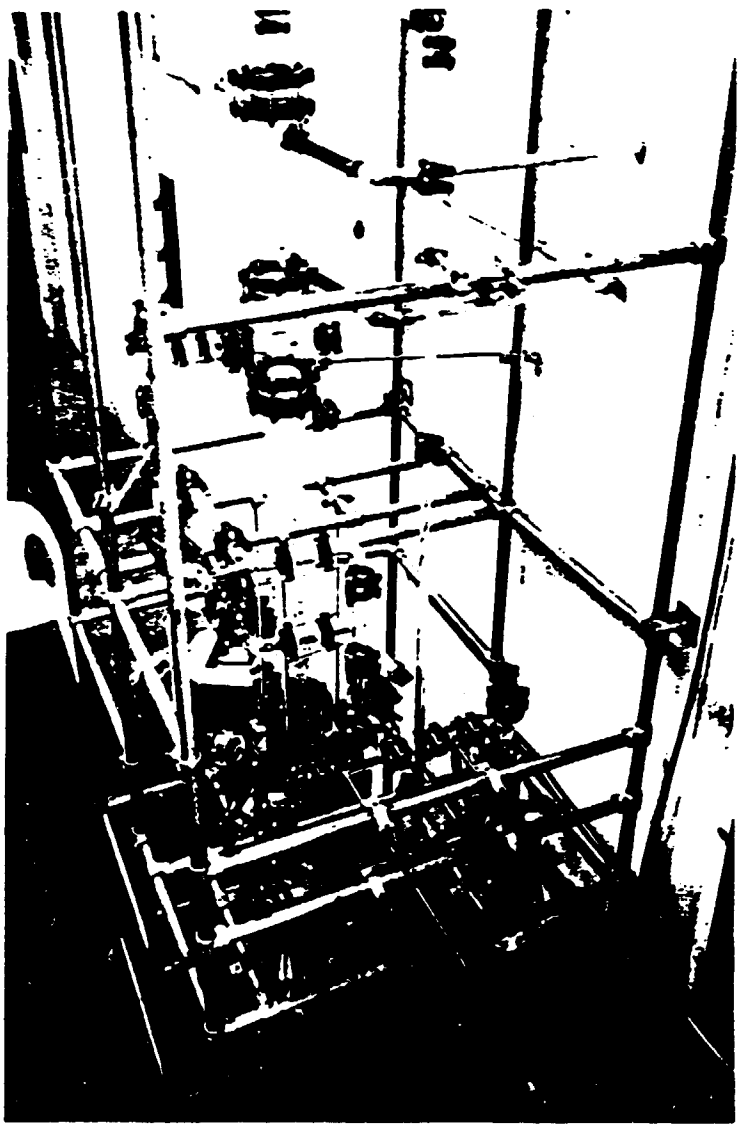
DIAGRAM OF THE FRACTIONAL DISTILLATION UNIT




22
21
20
14
825
DN15
6
18
19
30
31
DN15 COOL.
WATER IN
DN 15 COOL.
WATER OUT
6
18
19
61
22
21
20
14
13
16
3400
7
A
17
35
12
1325
450

ANNEX 8

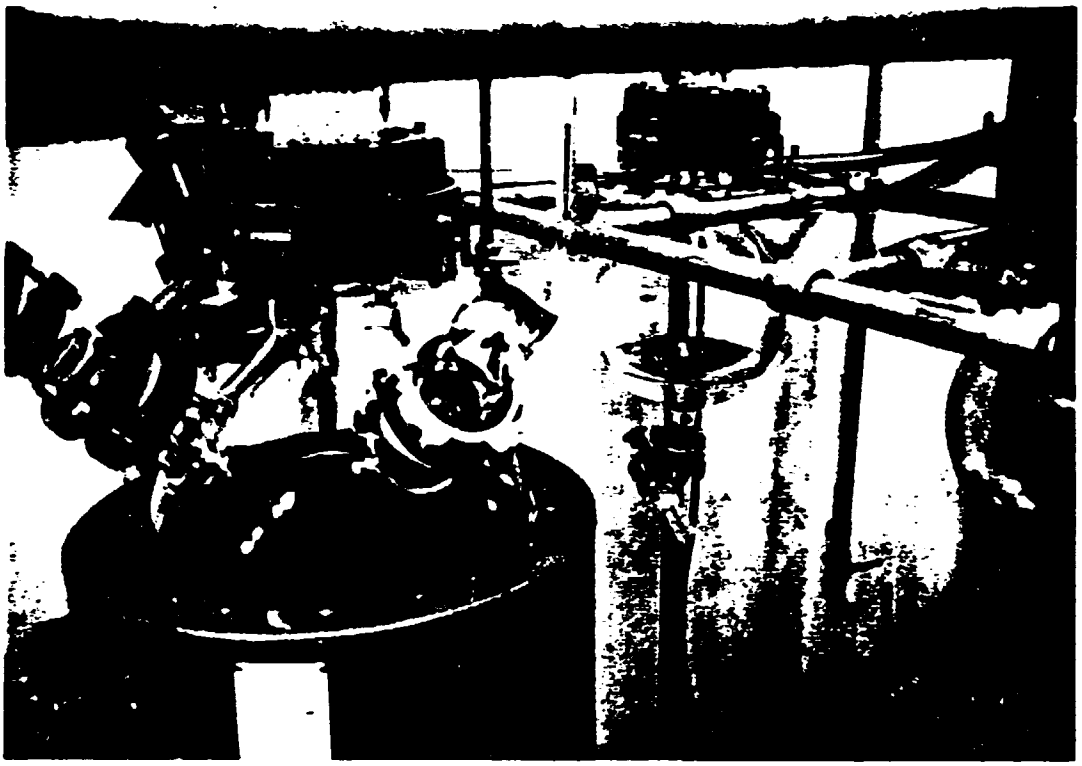
PHOTOGRAPHS OF THE FRACTIONAL DISTILLATION UNIT

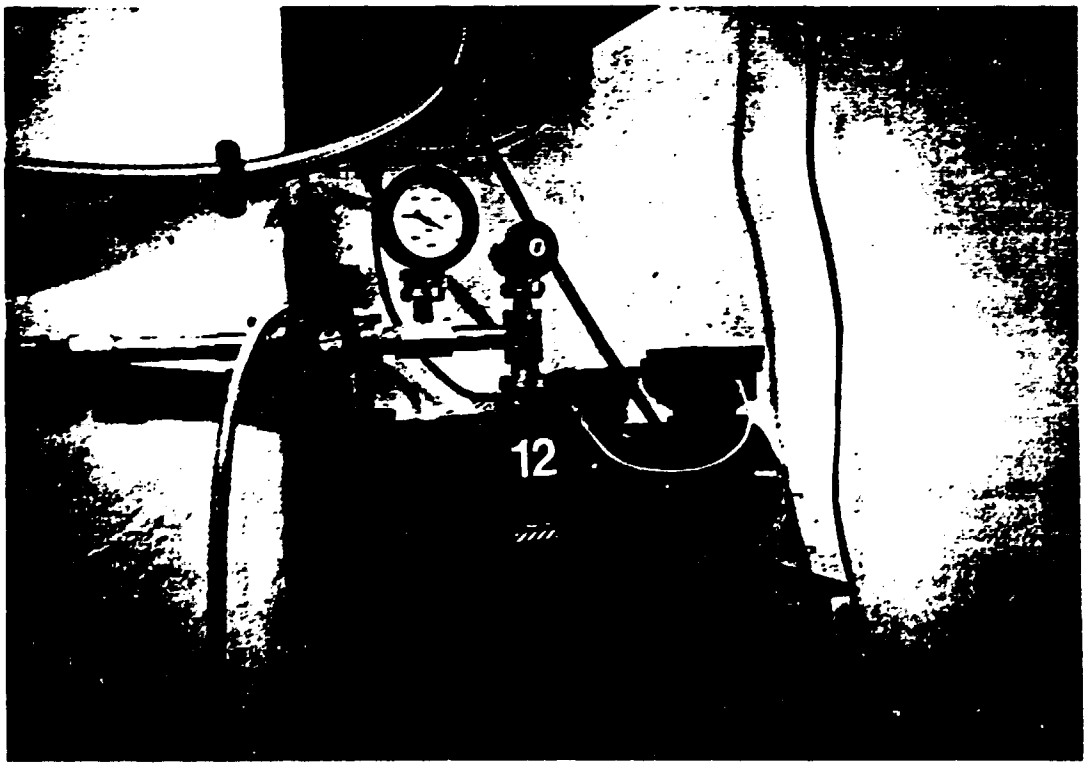


View of the unit from above

Page 50 Top - Evaporator in heating mantle with collecting vessels behind

Bottom - Vacuum pump with manifold and air bleed, gauge and isolation valve

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