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Yours faithfully For SGS India Pvt Ltd.

S. Suresh Kumar Technical Manager - National Environment Services

SGS India Private Limited, Chennai and Central Pollution Control Board, Zonal Office (W), Vadodara

PROJECT TEAM

Principal Project Co-ordinator	-	Shri J.S. Kamyotra, Member Secretary, CPCB, Delhi
Project Co-ordinators	-	Shri B. R. Naidu Zonal Officer, CPCB, Vadodara Shri Prasoon Gargava, Scientist "C", CPCB, Vadodara
Monitoring & Support Team	-	CPCB, West Zone Office, Vadodara Shri. Prasoon Gargava, Scientist "C" Shri S. Pradeep Raj, Scientist "B" Shri. Nishchal. C, Scientist "B" Shri H. Roat, Jr Scientific Assistant
		SGS, Chennai Shri S. Suresh Kumar, Manager - Env Shri Pughazhandi, Incharge - FO Shri Anand, Chemist Shri Haribabu, Chemist Shri Rajan, Field Assistant
Report Preparation	-	Shri S. Suresh Kumar, Manager - Env.
Report Editing & Finalization	-	Shri B. R. Naidu, Zonal Officer Shri Prasoon Gargava, Scientist "C"

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1.0 INTRODUCTION:

Volatile organic compounds (VOCs) are emitted from various sources, both anthropogenic and biogenic. They are important precursors in photochemical reactions, and the formation of secondary aerosols. Therefore in controlling ozone concentration & secondary aerosols, sources of precursors need to be defined. Volatile Organic Compounds (VOCs) mostly industrial solvents which are used in Chemical Industries (Pharmaceuticals, Pesticide, Dye & Dye Intermediates & other Chemicals) are emitted in to the environment in most of the industrial estates. Some of them being known carcinogens can be identified as Hazardous Air Pollutants (HAPs) and needs special attention. Uncertainties in emission inventory are greater for VOCs than the criteria pollutants such as TSPM, SO₂ and NOx. The diversity of VOCs sources includes vehicles, industry, solvent-containing products and biological processes. An estimation of volatilization is very difficult due to uncertainties in estimating solvent use, temperature and manner of applications. In urban areas, VOCs are mainly from anthropogenic sources such as vehicle tailpipes emitting gases from both the complete and incomplete combustion of gasoline, evaporation of gasoline from engines & tanks, solvents used in industry as well as in household products.

Ambient air levels of VOC is required to be monitored primarily because of their role in adverse impacts on human health, as well as on ecology, and also on the adverse impact on atmospheric factors relating to other environmental changes (Ozone Layer Depletion etc) i.e. increases in levels of troposphere (ground-level) ozone and decreases in levels of stratospheric ozone. Most of the chlorinated VOCs may contribute for 35 - 55% of outdoor air borne cancer risk.

The development of effective strategies for controlling VOCs refers on quantifying the ambient concentrations and the identification of emission sources of these pollutants. However, information on ambient concentrations and the composition of VOCs in India still limited.

In India, due to the absence of standards for VOCs (except Benzene in ambient air) and recommended monitoring procedure, it is difficult to initiate appropriate action by the regulatory and enforcing authorities. Many countries have standards

for many chemicals of concern, however in the absence of standards in India, it is utmost important that precautionary measures are taken promptly. Presently, criteria pollutants viz. SPM, PM 10, PM 2.5, SO₂, NOx and other pollutants viz. NH₃, Cl₂ & Acid Mist etc. are monitored regularly to know the concentrations and impact on the ambient air quality. The aim of this study was to investigate composition of VOCs and their concentrations in ambient air, fugitive emissions and industrial effluents in Lote industrial area. The effort has been made in this study to generate baseline data for future policy decisions. Central Pollution Control Board, Western Zonal Office along with M/s SGS India Pvt. Ltd, has carried out the indepth baseline study to monitor the VOCs concentration in ambient air and fugitive emissions & effluent discharge from the industries. CPCB engaged M/s SGS India Pvt Ltd., Chennai for carrying out sampling and analysis.

For the purpose of monitoring the VOCs in the ambient air as well as from fugitive sources in the industries, the Lote Parshuram industrial area has been selected due to its complex nature of industries consisting of Pharmaceuticals, Dyes & Dyes Intermediates, Pesticides and Paint units etc. Many of these industrial sectors are known sources of Volatile Organic Compounds (VOCs).

This study is carried in order to prioritize some potential HAPs for standard development, subsequently enforcing measures for them and also to address complaints from Public/NGOs in future.

2.0 Volatile Organic Compounds (VOCs) :

Volatile Organic Compounds (VOCs) are organic chemical compounds that have high enough vapor pressures under normal conditions to significantly vaporize and enter the atmosphere. A wide range of carbon-based molecules, such as aldehydes, ketones, and other light hydrocarbons are VOCs.

2.1 Some Definitions:

There is no clear and widely supported definition of a VOC. VOC is a term used more in relation to air quality and environmental studies. From a chemistry viewpoint "Volatile Organic Compound" can mean any organic compound (all chemical compounds containing carbon with exceptions) that is volatile (evaporating or vaporizing readily under normal conditions). This is a very broad set

of chemicals. Definitions vary depending on the particular context and few are quoted below:

• Canada

Health Canada classes VOCs as organic compounds that have boiling points roughly in the range of 50 to 250 $^{\circ}$ C (120 to 480 $^{\circ}$ F). The emphasis is placed on commonly encountered VOCs which would have an effect on air quality.

• European Union

A VOC is any organic compound having an initial boiling point less than or equal to 250 °C measured at a standard atmospheric pressure of 101.3 kPa.

• United States

A VOC has high vapor pressure and low water solubility. The US EPA's Terms of Environment defines a VOC as any organic compound that participates in atmospheric photochemical reactions except those designated by the EPA as having negligible photochemical reactivity. Under the Code of Federal Regulations it is similarly defined as any compound of carbon, excluding carbon monoxide, carbon dioxide, carbonic acid, metallic carbides or carbonates, and ammonium carbonate, which participates in atmospheric photochemical reactions.

Worldwide, legal definitions of the term "VOC" are in many respects, more a matter of policy than a matter of science.

2.2 Classification of the VOCs: Some of the classifications are given as:

2.2.1 Based on Boiling Point:

•	VVOC : Very Volatile Organic Compounds	:	< 0 up to 50 - 100°C
•	VOC : Volatile Organic Compounds	:	50-100 up to 250 - 260°C
•	SVOC : Semi Volatile Organic Compounds	:	250 - 260 up to 380 - 500°C

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• POM : Polycyclic Organic Materials :> 380°C

2.2.2 Based on Toxicity to Human Health:

• Highly harmful:

Highly harmful VOCs are the substances such as Benzene, Vinyl Chloride and 1,2 dichloromethane pose serious health risks to humans.

• Class A VOCs:

Class A VOCs are the VOCs which may contribute substantially to the creation of photochemical ozone, depletion of stratospheric ozone or global warming. These are considered as having a medium degree of harmfulness. Examples include carbon tetrachloride, 1,1,1-trichloroethane, trichloroethylene and trichlorotoluene.

Class B VOCs:

Class B VOCs are the remaining majority of VOCs are considered as having a lower degree of harmfulness than class A VOCs. Examples include butane and ethyl acetate.

2.3 Effects of VOCs:

The following are the effects of VOCs:

- Human health impacts; some VOCs are known or suspected carcinogens;
- Nuisance effects such as odours;
- Contribute to global warming; and
- Some VOCs also react with nitrogen oxides in the air in the presence of sunlight to form ozone.

The list of 60 VOCs which are identified by USEPA and mostly impart toxic effects to human health considered for the sampling and analysis is enclosed as **Annexure**-I and the important physical properties of all 60 VOCs is enclosed as **Annexure** - II.

3.0 OBJECTIVE:

The Objectives of the study are as below:

The aim of the study was to identify typical levels of VOCs in this industrial cluster where monitoring of this unregulated but important group of pollutants has rarely been conducted. The objectives of the study can be listed as below:

- To identify and quantify the VOCs presence in the ambient air and wastewater
- To prioritize the most toxic and most predominant VOCs in the Ambient air
- To generate the database for future monitoring and subsequent standard development for enforcement
- To develop a protocol for sampling and analysis method for VOCs in Indian context
- To compare concentration with International AAQ standards/guidelines

Each of these objectives requires a proper selection of the various elements constituting the sampling strategy such as type and number of locations to be included in the study, the sampling method, frequency and duration.

4.0 METHODOLOGY:

The definition of VOC is based on ranges of boiling points. According to this definition, VOCs are volatile organic compounds which boil between 50° C and 260° C. However, no exact limit exists between the said four categories. The reason is that in practice, the categories are determined by the methods used to sample organic pollutants from air. Most of the methods rely on pre concentration of organic compounds on adsorbents and therefore the type & amount of adsorbent & the sample volume will to a certain extent influence the boiling point range of the trapped compounds. Using combinations of appropriate adsorbents very volatile (VVOCs) and volatile organic compounds (VOCs) can be sampled simultaneously, and, hence the distinction between them may even become redundant.

The volatile organic compounds (VOCs) constitute a wide range of carbon molecules i.e., aliphatic & aromatic hydrocarbons, ketones, aldehydes, chlorinated hydrocarbons. The industries like refineries, petrochemicals, pharmaceuticals,

pesticides, dye and paints & varnish are the major sources, which emit VOCs in the atmosphere. As the industries are using different solvents in their processes they emit huge amount of VOCs to the environment. The aliphatic, aromatic and chlorinated hydrocarbons were the target volatile organic compounds. The sampling for volatile organic compounds was done for ambient air, fugitive emissions and effluent. For ambient air and fugitive emissions, the sampling was conducted using calibrated SKC low flow sampler at low flow rates ranging between 200 ml/min and 500ml/min using Tenax and Carboxen multi tube. The VOC adsorbed samples in the multibed adsorbent tubes were transferred to lab desorbed thermally by using Active Thermal Desorption System & then analyzed using capillary GC-MS to find out the VOC content in the sample. The steps involved in the study are:

- Selection of Industrial Area of LOTE Parashuram for the VOC monitoring;
- Collection of Information and dry data from Industries as well as from Maharashtra State Pollution Control Board & Maharashtra Industrial Development Corporation;
- Literature review;
- Selection of ambient air quality monitoring locations primarily based on wind direction, proximity to residential/commercial area, indicative concentration shown by Photo ionic detector and safety, availability & accessibility of locations;
- Selection of wastewater sampling locations based on wastewater sources, pollution potential etc. in industrial area, Inlet of CETP; and
- The monitoring of VOCs covered following:
 - I. VOC Monitoring: Air
 - Ambient air quality monitoring (two rounds for 24 hrs each) was conducted across the industrial estate at five locations which covers upwind, downwind, center and crosswind directions of the industrial estate.
 - Fugitive emission monitoring was carried out at selected industries and CETP.

Ambient air samples were withdrawn by a pump and adsorbed on sorbent tubes, ¹/₄ inch O.D., 3.5 inch long, packed with 100 mg of carboxen and 50mg Tenax (Supelco). Air collection flow rate was designed at 500 ml/ min. The actual flow

rate was 0 - 1000 ml/min. \pm 2 ml/ min. Sampling time was eight hours. When the sampling was finished, the sorbent tube was capped with brass Swagelok, wrapped with aluminum foil and put in a plastic bag, which was kept in an ice box and transported to the laboratory, and the samples were stored in a solvent-free refrigerator. The analysis work was carried out within one week after the sampling. Sorbent tubes for air sampling were cleaned each time before usage at 240 °C for 180 minutes.

Air samples were analyzed by the thermal desorption-GC/MS at the laboratory. The Thermal Desorber Unit (TDU) was from Unity, Markes International Limited. The TDU temperature program used was as follows: Purge for 5 minutes, and desorb at 190 °C for 20 minutes to cold trap -10 °C for 15 minutes and immediately heat to 310 °C to GC column. The GC/MS-QP5000 Column: was a Capillary column SPB TM 624, 60 m. long, 0.32 mm. i.d., thickness 1.8 μ m. from Agilent. Mass spectrometry analysis used an Electron Impactor, Quadrupole mass spectrometer.

II. VOC Monitoring: Wastewater

• Wastewater monitoring was carried out at CETP Inlet and also at different segment industries across industrial estate.

The analysis results are compiled, collated and interpreted. Based on the interpretation, observations and recommendations are prepared and presented in the report.

5.0 ABOUT THE AREA:

5.1 ABOUT LOTE PARSHURAM INDUSTRIAL ESTATE

Lote Parshuram industrial area is developed by Maharashtra Industrial Development Corporation (MIDC) in Ratnagiri district of Maharashtra state. This area spreads on the land of about 574 ha and accommodates 347 plots. This area is situated on the hilly area and in the catchment areas of Jagbudi River, which meets Vashishti River and forms Karambavane creek, and ultimately meets Arabian Sea as a Dabhol creek. Lote-Parshuram industrial area is established on both sides of Mumbai-Goa National Highway-17 [NH-17]. This area is 12 km from Chiplun taluka (towards Mumbai) and 17 km from Khed taluka (towards Goa) and 115 km from Ratnagiri. It is about 250 km from Mumbai and 350 km from Goa. It may be seen in the following schematic map of the industrial estate that it has typical linear strip type development along the national highway.



Lote Parshuram, MIDC, was established in the year 1982 for chemical industries (Pesticides, Dyes & Dyes Intermediates, Paint, Pharmaceuticals, etc.) along with few engineering units. Approximately 248 industries are located in this area. The category-wise break-up of operational units are as given below:



Source: MPCB



VIEW OF LOTE INDUSTRIAL AREA FROM ACROSS THE HIGHWAY

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This area is declared as a Chemical Zone after TTC Industrial area at Navi Mumbai, Ambernath, Dombivali, Kalyan and Tarapur in Maharashtra state. The water supply to the MIDC, Lote is from Vashishti River. The capacity of the water supply system provided by the MIDC is 24 MLD (maximum), whereas the present water supply is only to the tune of 7 - 7.5 MLD for industrial as well as domestic purposes. The total wastewater generation is about 5 - 5.5 MLD.



VIEW OF RIVER VASHISHTHI - A MAJOR SOURCE OF WATER FOR MIDC - LOTE

Increased pollution problems from SSI and MSI red category industries made MPCB to promote a CETP in the area. The Common Effluent Treatment Plant (CETP) is located at P-30, Lote Parshuram MIDC, Taluka Khed, District Ratnagiri, established in the year 2003 by Lote Parshuram Environment Protection Co-operative Society Ltd., a subsidiary organization of Lote Parshuram Industries Association. Around 141 numbers of industries have become member to the CETP. The designed capacity of CETP is 6 MLD, consisting of primary and secondary treatment units. The actual effluent load coming is reportedly around 4.6 MLD.



VIEW OF CETP OF LOTE INDUSTRIAL ESTATE

The treatment units provided by the CETP are collection cum equalization tank, flash mixer, primary clarifier, bio-reactors I & II (diffused aeration), secondary clarifier, oxidation tank (diffused aeration), treated effluent collection tank, decanter and sludge drying beds. The effluent from the CETP is disposed to the Karambavane creek through the 12 km long closed conduit, where the Jagbudi River (a small seasonal river) joins a perennial river Vashishti before meeting the Arabian Sea. The disposal location, which was reportedly finalised in consultation with NIO, is submerged in water at a depth of around 1.5 m and it is equipped with a diffuser system to evenly distribute the effluent at the confluence location.

6.0 METHODS FOR SAMPLING:

6.1 Air sampling:

It is decided to follow EPA TO-17 for sampling of Ambient Air Quality and Fugitive emissions using pre and post calibrated personal samplers.

The criteria of choosing the Method TO-17 for sampling are:

• Many compounds which boils at above 100°C also efficiently collected by these methods

- These method have a flexibility of sampling the gas stream at a high flow rate of 1 lpm and at low flow rate of 0.1 to 0.5 lpm
- These methods have an option of sampling smaller volumes at lower flow rates and should be used when the boiling points of the VOCs of interest are below 35°C.
- The target detection limit of these methods is $0.1\mu g/m^3$.

Measurements of VOCs are usually subdivided into sampling and analytical steps, of which the sampling is carried out in the ambient environment. Adsorption/thermal desorption with multisorbent air-sampling cartridges was developed for the determination of 60 analytes including halogenated alkanes, halogenated alkenes and aromatics. The volatilities of the compounds ranged from that of dichlorofluoromethane to that of 1,2,3-trichlorobenzene. The 60 volatile compounds were determined using a 240-L air sample and a sample cartridge containing 100 mg of Tenax and 125 mg of Carboxen. Analysis and detection were done by gas chromatography/mass spectrometry. The minimum detectable level (MDL) concentration values ranged from 0.1 parts per billion by volume (ppbv) for all volatiles. No breakthrough was detected with the prescribed sample volumes with the spiked VOCs observations and the analyte stability on the cartridges was very good. Excellent recoveries were obtained with independent check standards. Travel spike recoveries ranged from 90 to 110% for 57 of the 60 compounds. The recoveries were less than 70% for bromomethane and chloroethene and for a few compounds such as vinyl chloride, Dichlorofluormethan that are subject to losses by high volatility, the lowest travel spike recovery was obtained for bromomethane (62%). Blank values for all compounds were either below detection or very low. Analytical precision was measured using duplicate sampling. There are no significant variations observed.

The choice of the sampling method depends on the objective of an investigation and the VOCs of interest.

6.2 About Sorbent Tubes:

Multi sorbent tubes packed in order of increasing sorbent strength are used to facilitate quantitative retention and desorption of VOCs over a wide volatility range. The higher molecular weight compounds are retained on the front, least

retentive sorbent, the more volatile compounds are retained farther into the packing on a stronger adsorbent. The higher molecular weight compounds never encounter the stronger adsorbents, thereby improving the efficiency of the thermal desorption process.

6.3 Selection of sorbent tube for air sampling:

The selection of tube and sorbent material for sampling based on safe sampling volumes and breakthrough characteristics of sorbents. The selection of the adsorbent is an essential part of the measurement and sampling strategy. Therefore adsorbents have to be selected very carefully.

Tubes which contain two or more adsorbents to retain a broad range of VOCs are commercially available. Despite some advantages of such mixed sorbent tubes, several disadvantages are known. The sorbents in the same tube differ in their optimal desorption temperature and high humidity may also cause problems, especially when an adsorbent for polar compounds is included.

The newly packed sorbent tubes is conditioned at least 120 minutes at 350° C by passing 50ml/min. of pure helium carrier gas through them. After conditioning the tubes are sealed with brass 1/4 inch Swagelok fittings and PTFE ferrules then wrapped by uncoated aluminium foil. The prepared sorbent tubes are to be kept in a clean, airtight, opaque container and stored in a Refrigerator (organic free) or cooler box to maintain the temperature of 10° C.

6.4 Sample size

The sample size is an important determinant of the precision of a study. For instance, the precision of the mean value of the VOC level estimated from a sample of Ambient by the standard deviation which usually decreases with the number of samples taken. Thus, when the sample size increases the precision of the estimated sample mean value increases. A reliable estimate of this part of the distribution might require a large sample size.

When the distribution of the relevant determinants is known and the sample is carefully stratified for these determinants, the complete distribution can be obtained.

Two situations can be distinguished for deciding the sample size. In one situation, there is little information available on which agent(s) might cause the complaint. VOC measurements will then be a kind of 'fishing expedition' where one collects samples and waits to see which VOC turns up at what concentration. In a given situation a considerable sample size is necessary to obtain a reasonable statistical power for testing whether or not the observed. In second situation, VOC concentrations are extreme compared to a common distribution of ambient VOCs.

The selection of the method of sampling and analysis mainly depend on three important common factors.

- **Representation:** the extracted gases must be representative of the gas stream within the ambient air.
- Integrity: the extractive system and any sampling media used for grab sampling must be managed and operated in a manner which maintains integrity of the sample. If any of the VOCs are lost or changed during delay between sampling and analysis, then it must be known and understood both the extent and nature of these threats to sample integrity.
- Validity: The methods used for sampling and analysis must be valid, unbiased, accurate and precise within the ranges defined by the criteria of acceptance.

6.5 Wastewater Sampling:

Wastewater samples were collected from CETP and on segment wise at different identified industries.

The water samples collected in purge & trap amber vials directly with neck full of samples which can avoid any head space formation. After collection the samples were preserved in cooler with proper identification mark and the same is sealed.

7.0 SAMPLING PROCEDURE FOR AMBIENT AIR QUALITY AND FUGITIVE EMISSION MONITORING

7.1 About Adsorbent Tubes:

Based on the merits and demerits, it was decided to adopt EPA TO-17 method (as detailed below) for the sampling of VOCs at ambient air as well as fugitive emissions.

7.1.1 Active Thermal Desorption (ATD) combination tube: -

Compendium Method TO-17 using Tenax and Carbopack ATD Method. Thermal desorption (TD) is a complementary gas extraction technique whereby sorbent tubes (depicted in Figure below) are heated in a flow of carrier gas. Trapped vapours desorb from the sample tubes into the gas stream and are transferred, via a refocusing device, into the GC/MS analyzer. Conversely, thermal desorption allows complete transfer of all target analytes to the analytical system, with no dilution or solvent interference. Detection limits offered by thermal desorption methods are typically at least 1000 times higher than equivalent solvent extraction methods, facilitating ambient monitoring at ppt/ppb levels as well as higher ppm (and %-level) concentrations.

The advantage of Method TO-17:

- No contamination occur since the sample is directly injected
- Wide spectrum of high molecular weight chlorinated and aromatic VOCs are captured
- Easy to capture



ATD Tubes



Pictorial presentation of inside of ATD Tube:-

Thermal desorption efficiency is readily validated and is always above 95%, independent of ambient conditions and the nature of the target analytes (polar/nonpolar, volatile/semi-volatile, etc.).

7.2 Sampling Preparation:

The steps followed are as given below:

- Determined the extent of the sampling effort, the sampling methods to be employed the type and amounts of equipment and allied supplies needed;
- Organized the necessary sampling and monitoring equipment;
- Sampling pumps are pre calibrated before sampling with soap bubble techniques;
- Prepared a schedule in consultation with CPCB and SPCB officials; and
- Performed a general site survey prior to the study, in accordance with the site specific health and safety plan for sampling locations.

7.3 Calibration Procedure:

To save time in the field, sampling pumps are pre-calibrated in the laboratory prior to arrival on-site. The calibration is checked in the field prior to and upon completion of sampling.

The steps followed are as given below

- Assembled the calibration train using a calibrated (external) rotameter, sampling pump, a tube holder system and an adsorbent tube. The adsorbent tube is a representative tube from the same lot of tubes that is used for sampling.
- Turned on the pump and adjust the flow using the flow adjust mechanism on the tube holder until the float ball on the rotameter is aligned with the rotameter's pre-calibrated flow rate value.
- Marked to the manifold and pump indicating the pre-calibrated flow rate and sampling media.
- Calibrated sampling system, flow regulator and tubing kept ready before sampling. Broke both ends of the adsorbent tube before sampling and ensured that each opening is at least one half the inside diameter of the tube.
- Kept one field blank at each sampling station and three at laboratory blank with opened both ends of the ATD tube. This blank will give if any contamination in field during sampling as well as during the analysis in the laboratory.
- Fixed the ATD tube into sampling line and placed back-up section nearest to the pump. Position the tube in a vertical position to avoid channeling of air through adsorbent section.

7.4 Field Operation:

- Mobilized the pre-calibrated sampling equipment to save the time at the sampling site and fine tuning of the flow should be required.
- Placed the sampling tubes in the breathing zone. The pump and adsorbent tubes are placed on any solid stationary surface.
- Removed the caps of ATD tubes and Cracked the adsorbent tube ends using a glass tube cracker.

• To set up the sampling train with ATD tube, Tenax end of the adsorbent tube is open to atmosphere and Carbopack back up portion is towards the manifold of the sampler. The manifold is attached to the inlet plug on the pump. The photograph of sampling arrangement is shown below:



- Adjusted time on the pump to required sample time i.e. 8 hrs for each cycle. The sampling flow rate of 0.5 LPM is constantly maintained at all locations.
- Two rounds (one round- 24 hrs consists of three cycles of eight hrs each) of monitoring i.e. first round on 28-29.12.2010 and second round on 29-30.12.2010.
- Verified regularly the sampling flow using the calibrated rotameter. Recorded the final flow rate on the air sampling data sheet.

7.5 Post Operating Procedure:

- Recorded the sampling time on the air sampling data sheet
- Removed the adsorbent tube from the sampling pump
- Covered the adsorbent tubes with teflon tape followed by Caped adsorbent tubes with plastic caps immediately after sampling. Never use rubber caps.
- Placed the sample in a whirl bag labeled with sample ID Number
- Recorded all applicable information on the air sample data sheet (sample volume, ID number, location of the sampling, date and weather parameters)

• Sampled adsorbent tubes are stored in a cooler box (less than 10°C) and the maximum storage time is one week from the date of sampling.

7.6 Sample Storage and Preservation:

- 1. After sampling the identified samples are capped securely.
- 2. Samples collected in adsorbent tubes should not be kept in warm places or exposed to direct sunlight. After sealed the samples are kept in a cold storage box.
- 3. Samples are transported immediately after the sampling is over and stored under refrigeration until they are analyzed. Samples were analyzed within two to four days.

7.7 Quality Assurance / Quality Control:

Based on protocol for a measurement will give information on the quality control performed. At least 10 % of samples taken should be duplicated. Blanks and field blanks have to be analyzed in conjunction with the samples. The use of standards (internal and external), avoidance of contaminants and losses during sampling & storage also must be documented.

Quality Assurance and Quality Control (QA/QC) constitute an integral and important part of the entire sampling strategy and sampling management. The extent of QA/QC work will heavily depend on the sampling objective and other related factors.

Some of the salient features also been considered with regard to QA/QC of VOC measurements are: definition of the sampling & data quality objectives, design criteria for sampling, sampling media, sampling location and chemical analysis. In addition, the data quality objective is decided depending on the sampling objective. The choice of sampling parameters such as time, duration and location of sampling, are all closely related to the choice of the sorbent and the sampling technique. Many pitfalls are to be associated with sampling. The quality of the analytical results depends mainly on internal laboratory control, but it is equally important to know potential artifact formation in the sampling or in the analytical

system. In addition, active participation in international inter-comparisons, round robin tests and certified reference materials should be used for validation & quality control. Compliance of following is necessary to ensure QA/QC:

- Data pertaining to the VOCs study are documented on a data sheet.
- All instruments are operated in accordance with operating instructions as supplied by the manufacturer.
- Equipment check and calibration activities were carried out before and after sampling.
- For every 20 samples one field blank and lab blank were set aside. These field blanks will be handled in the same manner as the sampling tube (break, seal and transport) except that no air is drawn through the tube.
- At all sampling stations, the samples were collected in ATD adsorbent tube.

7.8 **Data Validation:**

Results of the quality control samples are evaluated for contamination. This information is utilized for quality assessment of the environmental sample.

The process flow chart for VOC sampling in ambient air is given at Annexure - IIIA, IIIB & IIIC

8.0 **MONITORING:**

8.1 Ambient Air Quality Monitoring:

8.1.1 Selection of Ambient Air Quality Monitoring Locations:

Within one industrial cluster, different types of industries generally have different VOC emissions and moreover sources are not necessarily evenly distributed all over the industrial areas. Transfer of pollutants from one location to the other, depends on the air flow pattern in the industrial area. As the number of locations that can be taken generally is limited, the appropriate selection of locations for sampling becomes necessary. The selection of locations are the obvious for some study objectives e.g., maximum solvent used in industries located, type of industries located, wind direction, geographical spread of the industrial area etc.. When

there is no specific guidance given for location of the sampling, the up and down wind should normally be recommended for the sampling of VOCs in a industrial cluster and rest will be cross wind stations that too based on the segments of industries spread.

The level and homogeneity of the VOC concentration in a industrial area depends on a number of meteorological conditions like wind speed, wind direction, temperature, relative humidity etc.. Some of these conditions have a direct or indirect influence on source emissions; others influence the dilution and mixing of VOCs in a sampling location and thus have an effect on the results. Source emissions may depend on temperature and surface air velocity.

The sampling location is governed by:

- Objectives of study;
- Instrumentation and method involved availability;
- Accessibility of the sampling site;
- Obstruction (tall trees, wall etc.,) free environment. Trees, walls etc. can obstruct the free air flow and can reduce pollutant concentrations in ambient air;
- At a height of minimum 5M from ground level so that the interferences by locally generated pollutants are minimized;
- Consideration of up wind down wind method; and
- Security against loss and tampering of instrument.

The wind direction was South East to North West and some time North East to South West. The ambient temperature was in the range between $20 - 32^{\circ}C$ and the Relative humidity in the range of 45 - 75%.

8.1.2. Sampling Duration, Time and Frequency:

The sampling duration depends clearly on the objectives, but it is also tightly linked to the sensitivity of the analytical method, the stability of the samples and the breakthrough volumes of the compound(s) of interest on the particular sorbent. These factors have to some extent to govern the decisions on sampling parameters.

It has to be taken into account that short-term sampling may lead to a loss of sensitivity whereas during long-term sampling some pollutants of interest may not be quantitatively collected.

Sampling duration is a very important factor to be considered if health effects are suspected to be related to indoor air quality / Ambient Air Quality. It has to be taken into account that short term sampling easily leads to a misrepresentation of a "true" average value due to the selected time for sampling.

Long-term sampling (from several hours to a few weeks) is suitable to assess average air pollutant concentrations. It is especially appropriate for ambient air quality investigations. When the objective is the evaluation of quantifying the volatiles presence in the atmosphere, the sampling duration must be in accordance with that applied in defining the safe sampling volume.

Concerning the sampling frequency, a single sample is rarely sufficient to assess ambient air quality. Minimum four to five locations across the industrial area based on the wind pattern to be required for getting representative data.

8.1.3 Ambient Air Quality Monitoring (AAQM) Locations at Lote:

The background information and dry data about industrial estate were collected from MPCB and MIDC. The ambient monitoring locations were worked out on the basis of dry data collected and preliminary survey of the industrial estate.

The ambient and fugitive emissions are monitored in the industrial estate to know the concentrations of each individual VOC and to understand its potential impact on the human health and the environment. The main parameters which affect the distribution & concentration of VOCs in the ambient air are its distribution into the atmosphere by predominant wind speed & its direction, distribution of industries across the industrial estate. The monitoring stations for both ambient and fugitive emissions were selected on the basis of total VOC concentration persists in the ambient air as well as in the industry. The team carried out preliminary survey of entire industrial estate with Photo Ionization Detector (PID), which is capable of detecting the total VOC concentration in terms of PPM and also in terms of PPB. The instrument is normally used to determine the VOC leak detections in the work

environment / plant / process areas. The ambient air quality monitoring locations were established on the basis of total VOCs concentration showed by PID, covering different directions of industrial estate and of course considering the safety of the instrument at monitoring place. The following table depicts the location, duration and salient features of the ambient air monitoring locations in Lote for VOCs.

	Sampling		Date		
Location	location	Duration	Round - I	Round - II	Salient features
M/s. Ratnagiri Telecom Exchange, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri.	Roof top of admin block, SE of MIDC	24 hr	28.12.2010 to 29.12.2010	29.12.2010 to 30.12.2010	Approximate height from ground level: 20 feet. South side of sampling point - M/s. Rallis India Ltd, north side is covered with little vegetation, hillocks and NH - 7 passes through that area.
M/s. Ganesh Ice Factory, B -69/2, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri.	Roof top of ice factory Center of MIDC	24 hr	28.12.2010 to 29.12.2010	29.12.2010 to 30.12.2010	Approximate height from ground level: 15 feet. Sampling location is surrounded by other Ice factory and little vegetation.
M/s. Amico Pesticides Ltd, B - 1/1, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri.	Roof top of admin block W of MIDC	24 hr	28.12.2010 to 29.12.2010	29.12.2010 to 30.12.2010	Approximate height from ground level: 20 feet. Back side of sampling location- M/s. Gharda chemicals & USV limited, front side- MIDC road, M/s. Hindustan Unilever Ltd & Lote CETP.
M/s. Kansai Nerolac Paints Ltd, F - 1/2, 1/3, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri.	Roof top of HR block NW of MIDC	24 hr	28.12.2010 to 29.12.2010	29.12.2010 to 30.12.2010	Approximate height from ground level: 25 feet. Front side of sampling location- thick vegetation, M/s. Pentoky chemicals, m/s. Gharda chemicals back side- M/s. Kansai Nerolac's ETP.
CETP - Lote, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri.	Near to admin block - open space, SW of MIDC	24 hr	28.12.2010 to 29.12.2010	29.12.2010 to 30.12.2010	Approximate height from ground level: 03 feet. The CETP plant boundary is covered with green belt, besides M/s. Hindustan Unilever Ltd.

No. of ambient air (VOC) samples: 30



Environmental Sampling and Analysis of Volatile Organic Compounds, Lote Industrial Estate, Maharashtra

MAP SHOWING THE AMBIENT AIR QUALITY MONITORING STATIONS ACROSS LOTE MIDC

Based on the objective of the study and location selection criteria, five locations, as detailed below, were selected for Ambient Air Quality Monitoring:

Up wind	: (1)	Premises of Telephone Exchange South East direction of MIDC
		Industrial area
Center	: (2)	Premises of M/s Ganesh Ice Factory, Center of MIDC Industrial
		area.
Cross Wind	: (3)	Premises of M/s AIMCO Pesticides, West direction of the MIDC
		industrial area.
Down Wind	: (4)	Premises of M/s Nerolac Paints Ltd., North West direction of
		MIDC Industrial area.
Cross Wind	: (5)	Premises of M/s Common Effluent Treatment Plant (CETP South
		West direction of MIDC Industrial Area

Two rounds of monitoring were carried out for all five locations. Each round of 24 hours duration has three cycles of eight hours each.

The Ambient Air Quality Monitoring locations at LOTE MIDC are depicted at **Annexure-IV.** VOC sampling details viz. Date of sampling, time of sampling, sampling locations, flow rate, sampling duration and volume of air sampled for LOTE MIDC is given in **Annexure-VA & VB** respectively.

8.2 Fugitive Emission Monitoring:

Industries were selected from the inventory available with MPCB. Collection of relevant information from industries and detailed information of plant premises was carried out to finalize the important locations for monitoring from fugitive emission point of view. Locations of fugitive monitoring in industries were selected on the basis of several factors viz. solvent handling and its storage practices, leakages in the flanges, reactor vessels, high fugitive emission potential activities like filtration, centrifugation etc.. Because of its versatility in nature the maximum total VOC concentration will be in the work place, where the workers are more prone to the occupational health hazards. The PID was used in finalizing the locations for fugitive emission monitoring in industries. Based on the above observations and by keeping in the view point of occupational health related hazards the following locations were selected. The following table depicts the location, duration and salient features of the sampling locations:

Location	Sampling location	Duration	Date	
M/s. Rallis India Ltd, D - 26, MIDC, Lote Parshuram Industrial	Between Centrifuge No.7 & FBD No.2 (inside the production plant- second stage of production plant)	4 hr	29.12.2010	
Alea, la - Kileu, Dist - Katilagili.	Near material equipment lifting bay	4 hr	29.12.2010	
M/s. Kansai Nerolac Paints Ltd, F	Grinding area of plant/thinning	4 hr	30.12.2010	
- 1/2, 1/3, MIDC, Lote	(second stage of process)			
Parshuram Industrial Area, Ta -	Down wind/back side of the unit. b/n	4 hr	30.12.2010	
Khed, Dist - Ratnagiri	resin block and main plant			
	Plant No. 2 first floor, near filter No.	4 hr	30.12.2010	
M/s. Charda Chomicals I td	F - 2503/ centrifuge area			
M/S, Gharda Chemicals Etd	Down wind of 1,2,3 production block	4 hr	30.12.2010	
	Down wind of 4,5,6 production block	4 hr	30.12.2010	
No. of fugitive emission (VOC) samples: 07				

No. of fugitive emission (VOC) samples: 07

8.3 Wastewater Monitoring:

Wastewater samples were also collected from few industries covering different industrial sectors like Pharmaceutical, Pesticides, and Paints etc. and also from CETP to know the characteristics of the effluent in terms of VOCs and also to know the indicative losses of VOCs in environment through effluent. The details of samples collected are given below:

Location	Sampling location	Type of sampling	Date
M/s. Rallis India Ltd, D - 26, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri	Raw effluent collection sump	Grab	29.12.2010
M/s. USV Limited, B - 1/8, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri	Raw effluent collection sump	Grab	29.12.2010
M/s. Kansai Nerolac Paints Ltd, F - 1/2, 1/3, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri	Raw effluent collection sump	Grab	30.12.2010
M/s. Gharda Chemicals Ltd.	Raw effluent collection sump	Grab	30.12.2010
CETP - Lote, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri	Raw effluent collection sump cum aeration tank	Grab	30.12.2010

No. of wastewater samples (VOC): 05

9.0 METHODS FOR ANALYSIS:

9.1 Analysis for Ambient Air Quality & Fugitive Emission Samples:

Method EPA TO-17 (Active Thermal Desorption) method of analysis is used for Ambient Air Quality and Fugitive emission samples. EPA TO-17 Method is described at **Annexure- VI** and this annexure includes method validation also.

9.2 Analysis for Wastewater Samples:

EPA Method 5035A/8260B is adopted for the analysis of VOCs in wastewater samples which is described in **Annexure - VII**.

One Set of Chromatogram is given at Annexure-VIII

- 10.0 RESULTS & DISCUSSIONS:
- 10.1 Results & Discussion for Ambient Air Quality Monitoring:-
- Station- 1: The location:1 at M/s. Ratnagiri Telecom Exchange, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri is in south East direction of MIDC Industrial area.

The AAQM Station-1 was identified as up-wind station as per the prevailing wind direction at the time of the study. The wind direction was South-East to North-West and some time North-East to South-West. The ambient temperature was in the range between 20 - 32° C and the Relative humidity in the range of 45 - 75%. Approximate height from ground level is 20 feet. South side of sampling point - M/s. Rallis India Ltd, north side is covered with little vegetation, hillocks and NH - 7 passes through that area.

AMBIENT AIR QUALITY - VOC (ROUND - 1)

SAMPLING AREA: MIDC LOTE

SAMPLING SITE: TELECOME EXCHANGE - 28.12.10 TO 29.12.10

PARAMETERS	TIME:13.00-21.00	TIME:21.10-05.10	TIME:05.20-13.20	AVERAGE (µg/m3)	
	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)		
Benzene	9.7	BDL	BDL	3.2	
Ethylbenzene	31.7	13.3	BDL	15.0	
Isopropylbenzene	3.9	BDL	BDL	1.3	
Napthalene	BDL	21.8	104.5	42.1	
o-Xylene	35.8	56.5	9.6	34.0	
TOTAL VOC =	81.1	91.6	114.1	95.6	

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AMBIENT AIR QUALITY - VOC (ROUND - 2)								
SAMPLING AREA: MIDC LOTE								
SAMPLI	NG SITE:TELECO	ME EXCHANGE -	29.12.10 TO 30.	12.10				
	TIME:16.45-0.45 TIME:0.50-08.50 TIME:08.55-16.55							
PARAMETERS	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)	(µg/m3)				
Benzene	BDL	48.4	11.5	20.0				
Carbon tetrachloride	BDL	3.5	BDL	1.2				
Chlorobenzene	BDL	113.6	BDL	37.9				
Napthalene	3.5	BDL	BDL	1.2				
Tetrachloroethane	12.4	78.4	BDL	30.3				
Toluene	96.5	205.8	BDL	100.8				
o-Xylene	29.7	93.8	21.5	48.3				
TOTAL VOC =	142.1	543.5	33.0	239.7				

Total average concentration of VOCs in first round of monitoring was found to be less than the results of second round of monitoring. Three VOCs were found common in both the rounds of monitoring out of total detected 09 VOCs. Concentration of Naphthalene was found to be highest in first round of monitoring, however concentration of Toluene found to be highest in second round of monitoring at station-1.



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Station - 2: M/s. Ganesh Ice Factory, B -69/2, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri., Center of MIDC Industrial area.

The AAQ station-2 was identified as Center station of the MIDC LOTE industrial area as per the prevailing wind direction at the time of the study. Approximate height from ground level is 15 feet. Sampling location is surrounded by other Ice factory and little vegetation. The ambient temperature was in the range between 20 - 32°C and the Relative humidity in the range of 50 - 75%.

AMBIENT AIR QUALITY - VOC (ROUND - 1)						
SAMPLING AREA: MIDC LOTE						
SAMPLING SI	TE:GANESH ICE	FACTORY - 28.1	2.10 TO 29.12.1	0		
	TIME:13.50-21.50	TIME:22.00-06.00	TIME:06.10-14.10	AVERAGE		
FARAMETERS	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)	(µg/m3)		
Benzene	26	BDL	BDL	8.7		
Sec-Butylbenzene	BDL	15.5	BDL	5.2		
Chloroform	BDL	5.9	BDL	2.0		
1,2- Dichlorobenzene	BDL	168.8	79.1	82.6		
Ethylbenzene	BDL	51.9	7.2	19.7		
Isopropylbenzene	BDL	6.1	BDL	2.0		
Napthalene	BDL	372.6	34.9	135.8		
Tetrachloroethene	4.2	BDL	BDL	1.4		
Toluene	BDL	247.6	BDL	82.5		
Styrene	10.3	192.8	210.2	137.8		
1,2,4-Trichlorobenzene	BDL	6.8	BDL	2.3		
1,3,5-Trimethylbenzene	BDL	67.9	7.7	25.2		
o-Xylene	59.6	158.5	112.6	110.2		
TOTAL VOC =	100.1	1294.4	451.7	615.4		

AMBIENT AIR QUALITY - VOC (ROUND - 2)				
SAMPLING AREA: MIDC LOTE				
SAMPLING SITE: GANESH ICE FACTORY - 29.12.10 TO 30.12.10				
PARAMETERS	TIME:16.55-0.55	TIME:01.00-09.00	TIME:09.05-17.05	AVERAGE (µg/m3)
	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)	
Bromobenzene	BDL	94.3	BDL	31.4
1,2- Dichlorobenzene	BDL	70.7	BDL	23.6
Ethylbenzene	55.9	58.1	13.3	42.4
Napthalene	BDL	51.2	BDL	17.1
Tetrachloroethane	6	1.3	3.2	3.5
Toluene	BDL	201.5	122.3	107.9
1,2,4-Trimethylbenzene	BDL	69.2	BDL	23.1
Styrene	161.5	418.1	BDL	193.2
1,3,5-Trimethylbenzene	BDL	19.7	BDL	6.6
o-Xylene	133.5	169.7	72	125.1
TOTAL VOC =	356.9	1153.8	210.8	573.8

Total average concentration of VOCs in first round of monitoring was found to be more than the results of second round of monitoring. Eight VOCs were found common in both the rounds of monitoring out of total detected 15 VOCs. Average concentration of Styrene was found to be highest in both the rounds of monitoring at station - 2.


Station-3: Premises of M/s. Aimco Pesticides Ltd, B - 1/1, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri, West direction of the MIDC industrial area.

The AAQM Station-3 was identified as cross-wind station as per the prevailing wind direction at the time of the study. The wind direction was South East to North West and North East to South West (some time). The ambient temperature was in the range between $20 - 32^{\circ}$ C and the Relative humidity in the range of 50 - 70%. Approximate height from ground level: 20 feet. Back side of sampling location- M/s. Gharda chemicals & USV limited, front side- MIDC road, M/s. Hindustan Unilever Ltd & Lote CETP.

AMBIENT AIR QUALITY - VOC (ROUND - 1)								
SAMPLING AREA: MIDC LOTE								
SAMPLING	SITE: AIMCO PES	STICIDES - 28.12	.10 TO 29.12.10)				
	TIME:15.30-23.30	TIME:23.40-07.40	TIME:07.50-15.50	AVERAGE				
FARAMETERS	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)	(µg/m3)				
Benzene	BDL	8.4	16.3	8.2				
Bromobenzene	BDL	BDL	77.9	26.0				
Sec-Butylbenzene	1.8	3.5	35.3	13.5				
Chloroform	BDL	11.2	5.9	5.7				
1,2- Dichlorobenzene	101.4	122.6	161.8	128.6				
Ethylbenzene	122.1	102.4	84	102.8				
Isopropylbenzene	6.3	BDL	6.4	4.2				
Napthalene	105.7	144.2	198.7	149.5				
Propylbenzene	19.8	20.6	102.6	47.7				
Toluene	222.4	256	107	195.1				
Styrene	12.1	15.7	18.1	15.3				
1,3,5-Trimethylbenzene	89.3	56.8	99.8	82.0				
o-Xylene	259.6	226.8	218.8	235.1				
TOTAL VOC =	940.5	968.2	1132.6	1013.8				

AMBIENT AIR QUALITY - VOC	(ROUND - 2)
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SAMPLING AREA: MIDC LOTE

SAMPLING SITE: AIMCO PESTICIDES - 29.12.10 TO 30.12.10

	TIME:17.10-1.00	TIME:01.05-09.05	TIME:09.10-17.10	AVERAGE (μg/m3)	
FARAMETERS	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)		
Benzene	10.2 BDL		BDL	3.4	
Ethylbenzene	BDL	BDL	122.6	40.9	
Napthalene	124.6	22.9	BDL	49.2	
Tetrachloroethene	1.4	BDL	BDL	0.5	
1,2,4-Trimethylbenzene	BDL	3.2	BDL	1.1	
1,3,5-Trimethylbenzene	BDL	2	BDL	0.7	
o-Xylene	18.9	14.4	56.8	30.0	
TOTAL VOC =	155.1	42.5	179.4	125.7	

Total average concentration of VOCs in first round of monitoring was found to be much higher than the results of second round of monitoring. Five VOCs were found common in both the rounds of monitoring out of total detected 15 VOCs. Average concentration of o-Xylene was found to be highest in first round of monitoring and concentration of naphthalene was found to be highest in second round of monitoring at station - 3.



Station-4: Premises of M/s. Kansai Nerolac Paints Ltd, F - 1/2, 1/3, MIDC, Lote Parshuram Industrial Area, Ta - Khed, Dist - Ratnagiri., Roof top of HR block NW of MIDC, West direction of Industrial estate.

The AAQM Station-4 was identified as cross-wind station as per the prevailing wind direction and some time which will be down wind station based on wind direction at North East to South West at the time of the study. The wind direction was South East to North West and North East to South West. The ambient temperature was in the range between 20 - 32°C and the Relative humidity in the range of 50 - 70%. Approximate height from ground level: 25 feet. Front side of sampling location-thick vegetation, M/s. Pentoky chemicals, m/s. Gharda chemicals back side- M/s. Kansai Nerolac's ETP.

AMBIENT AIR QUALITY - VOC (ROUND - 1)									
	SAMPLING AREA: MIDC LOTE								
SAMPLING SI	TE:NEROLAC PA	INTS - 28.12.10	TO 29.12.10						
	TIME:16.3030	TIME:0.40-08.40	TIME:8.50-16.50	AVERAGE					
FARAMETERS	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)	(µg/m3)					
Benzene	50.2	1.9	BDL	17.4					
Bromobenzene	7.2	14.2	BDL	7.1					
Chlorobenzene	151.8	BDL	BDL	50.6					
1,2- Dichlorobenzene	27.7	49.9	BDL	25.9					
Ethylbenzene	287	157.4	400.3	281.6					
Isopropylbenzene	18.5	BDL	25.3	14.6					
Napthalene	178.8	25.3	69.3	91.1					
Propylbenzene	106.7	30	65.9	67.5					
Tetrachloroethene	78.5	BDL	BDL	26.2					
Toluene	182.1	BDL	107.1	96.4					
1,2,4-Trimethylbenzene	228.9	155.4	BDL	128.1					
1,3,5-Trimethylbenzene	130.9	110.3	70.3	103.8					
o-Xylene	299.1	197.8	331.3	276.1					
m&p –Xylene	65	52.5	99.1	72.2					
TOTAL VOC =	1812.4	794.7	1168.6	1258.6					

AMBIENT AIR QUALITY - VOC (ROUND -2)								
SAMPLING AREA: MIDC LOTE								
SAMPLING	SITE:NEROLAC P	AINTS - 29.12.1	0 TO 30.12.10					
	TIME:17.20-1.20	TIME:1.25-09.25	TIME:9.30-17.30	AVERAGE				
FANAMETERS	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)	(µg/m3)				
Benzene	3.8	5.3	BDL	3.0				
Ethylbenzene	136.4	56.6	31.1	74.7				
Isopropylbenzene	2.1	BDL	BDL	0.7				
Napthalene	56.1	22.2	BDL	26.1				
Propylbenzene	57	BDL	BDL	19.0				
Tetrachloroethene	BDL	BDL	8.5	2.8				
Toluene	BDL	76.7	189.6	88.8				
1,2,4-Trimethylbenzene	144.3	BDL	BDL	48.1				
1,3,5-Trimethylbenzene	106.5	BDL	BDL	35.5				
o-Xylene	194.2	186	33.1	137.8				
m&p -Xylene	2.9	BDL	BDL	1.0				
TOTAL VOC =	703.3	346.8	262.3	437.5				

Similar to the trend of station -3, total average concentration of VOCs in first round of monitoring was found to be much higher than the results of second round of monitoring. Out of the total 14 detected VOCs, 11 VOCs were found common in both the rounds of monitoring. Average concentration of Ethyl benzene was found to be highest in first round of monitoring and concentration of o-Xylene was found to be highest in second round of monitoring at station - 4.



Station-5: Premises of CETP - Lote, MIDC, Lote Parshuram Industrial Area, Ta -Khed, Dist - Ratnagiri. South West direction of MIDC Industrial Area. Near to admin block - open space, SW of MIDC

The AAQM Station-5 was identified as cross-wind station as per the prevailing wind direction at the time of the study. The wind direction was South East to North West and North East to South West. The ambient temperature was in the range between 20 - 32° C and the Relative humidity in the range of 50 - 75%. Approximate height from ground level: 03 feet. The CETP plant boundary is covered with green belt, besides M/s. Hindustan Unilever Ltd.

AMBIENT AIR QUALITY - VOC (ROUND- 1)								
SAMPLING AREA: MIDC LOTE								
SAMPLIN	IG SITE:CETP PREA	AISES - 28.12.10) TO 29.12.10					
	TIME:17.45-1.45	TIME:1.50-09.50	TIME:10.00-18.00	AVERAGE				
FARAMETERS	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)	(µg/m3)				
Benzene	5.2	BDL	BDL	1.7				
Bromobenzene	BDL	BDL	943.5	314.5				
Tert-Butylbenzene	BDL	31.6	BDL	10.5				
Sec-Butylbenzene	BDL	165.3	7.6	57.6				
1,2- Dichlorobenzene	BDL	122.2	90.9	71.0				
Ethylbenzene	BDL	80.3	10.6	30.3				
Napthalene	BDL	BDL	153.7	51.2				
Propylbenzene	BDL	BDL	18.4	6.1				
Toluene	69	BDL	54.9	41.3				
1,2,4-Trimethylbenzene	BDL	156.7	BDL	52.2				
Styrene	BDL	BDL	5.8	1.9				
1,3,5-Trimethylbenzene	BDL	89.8	35	41.6				
o-Xylene	98.4	367.4	168.7	211.5				
Bromomethane	BDL	1654.6	BDL	551.5				
TOTAL VOC =	172.6	2667.9	1489.1	1443.2				

AMBIENT AIR QUALITY - VOC (ROUND- 2)								
SAMPLING AREA: MIDC LOTE								
SAMPLIN	NG SITE:CETP PR	EMISES - 29.12.	10 TO 30.12.10					
	TIME:17.30-1.30 TIME:1.35-09.35		TIME:9.40-17.40	AVERAGE				
PARAMETERS	Cycle - I (µg/m3)	Cycle - II (µg/m3)	Cycle - III (µg/m3)	(µg/m3)				
Bromobenzene	3792.8	1697	227.8	1905.9				
Tert-Butylbenzene	20.7	56.3	BDL	25.7				
Carbontetrachloride	3.3	3.5	4.0	3.6				
Chlorobenzene	588.8	17.0	BDL	201.9				
1,2- Dichlorobenzene	129.2	9.6	109.3	82.7				
Ethylbenzene	231.2	22.7	BDL	84.6				
Napthalene	467.2	455.2	140.8	354.4				
Propylbenzene	52.0	60.3	BDL	37.4				
Tetrachloroethene	13.9	BDL	BDL	4.6				
Toluene	1128.3	114	BDL	414.1				
1,2,4-Trimethylbenzene	291.9	471.2	157	306.7				
1,2,4-Trichlorobenzene	24.8	BDL	BDL	8.3				
1,3,5-Trimethylbenzene	154.3	240.8	BDL	131.7				
o-Xylene	435.8	BDL	140.8	192.2				
m&p -Xylene	1.3	246.5	BDL	82.6				
TOTAL VOC =	7335.5	3394.1	779.7	3836.4				

Total average concentration of VOCs in first round of monitoring was found to be much lesser than the results of second round of monitoring. Average concentration of Bromomethane was found to be highest in first round of monitoring, however concentration of Bromobenzene found to be highest in second round of monitoring at station-5. The total average concentration of VOCs found during second round of monitoring at station-5 is highest among average concentrations of all stations during both the rounds of monitoring.



10.1.2 LOCATION WISE VOC CONCENTRATION IN AAQM:

Concentration of each VOC in round -1 & round 2 is summarized in Tables given below and effort has been made to depict the distribution of the individual detected VOCs in five locations. The plates given subsequent to each of the following Tables show the indicative % distribution & occurrence of specific VOC at any given location with respect to its total occurrence in all five monitored locations viz. about 8.8% of benzene occurrence was detected near Telephone Exchange, about 23.9% near Ganesh ice Factory, about 14.8% near Aimco Pesticides, about 47.8% near Nerolac Paints and about 4.7% near the CETP area during first round of monitoring.

AMBIENT AIR QUALITY - VOC PARAMETER-WISE PRESENCE OF VOC IN ALL LOCATIONS								
Parameter	Telephone Exchange	Ganesh Ice Factory	Aimco Pesticides	Nerolac Paints	СЕТР			
	Round1	Round1	Round1	Round1	Round1			
Benzene	3.2	8.7	8.2	17.4	1.7			
Ethyl Benzene	15	19.7	102.8	281.6	30.3			
Naphthalene	42.1	135.8	149.5	91.1	51.2			
o-Xylene	34	110.2	235.1	276.1	211.5			
Carbon Tetrachloride	BDL	BDL	BDL	BDL	BDL			
Chlorobenzene	BDL	BDL	BDL	50.6	BDL			
Tetrachloroethane	BDL	1.4	BDL	26.2	BDL			
Toluene	BDL	82.5	195.1	96.4	41.3			
Chloroform	BDL	2	5.7	BDL	BDL			
1,2-dichlorobenzene	BDL	82.6	128.6	25.9	71			
Styrene	BDL	137.8	15.3	BDL	1.9			
1,2,4-Trimethylbenzene	BDL	2.3	BDL	128.1	52.2			
1,3,5-Trimethyl benzene	BDL	25.2	82.0	103.8	41.6			
Bromobenzene	BDL	BDL	26	7.1	314.5			
m&p – Xylene	BDL	BDL	BDL	72.2	BDL			
Bromomethane	BDL	BDL	BDL	BDL	551.5			

ROUND 1 (28.12.10 - 29.12.10)



ROUND 2 (29.12.10 - 30.12.10)									
AMBIENT AIR QUALITY - VOC									
PAR/	PARAMETER-WISE PRESENCE OF VOC IN ALL LOCATIONS								
Parameter	Exchange	Ice Factory	Pesticides	Paints	СЕТР				
	Round2	Round2	Round2	Round2	Round2				
Benzene	20	BDL	3.4	3	BDL				
Ethyl Benzene	BDL	42.4	40.9	74.7	84.6				
Naphthalene	1.2	17.1	49.2	26.1	354.4				
o-Xylene	48.3	125.1	30	137.8	192.2				
Carbon Tetrachloride	1.2	BDL	BDL	BDL	3.6				
Chlorobenzene	37.9	BDL	BDL	BDL	201.9				
Tetrachloroethane	30.3	3.5	0.5	2.8	4.6				
Toluene	100.8	107.9	BDL	88.8	414.1				
Chloroform	BDL	BDL	BDL	BDL	BDL				
1,2-dichlorobenzene	BDL	23.6	BDL	BDL	82.7				
Styrene	BDL	193.2	BDL	BDL	BDL				
1,2,4-Trimethylbenzene	BDL	23.1	1.1	48.1	306.7				
1,3,5-Trimethyl benzene	BDL	6.6	0.7	35.5	131.7				
Bromobenzene	BDL	31.4	BDL	BDL	1905.9				
m&p – Xylene	BDL	BDL	BDL	1	82.6				



10.1.3 TOTAL VOC CONCENTRATION

Individual VOC concentrations (Average) at each location for two rounds of monitoring are summarized in the following table and round-wise variations of each VOC at all monitored locations is graphically depicted in plate given subsequent to the following Table.

PARAMETER WISE AMBIENT AIR QUALITY MONITORING RESULTS OF VOC IN LOTE MIDC INDUSTRIAL AREA										
Locations→	Telephone	e Exchange	Ganesh Io	e Factory	Aimco Pesticides		Nerola	c Paints	CETP	
Parameter	Round1	Round2	Round1	Round2	Round1	Round2	Round1	Round2	Round1	Round2
Benzene	3.2	20	8.7	BDL	8.2	3.4	17.4	3	1.7	BDL
Ethyl Benzene	15	BDL	19.7	42.4	102.8	40.9	281.6	74.7	30.3	84.6
Naphthalene	42.1	1.2	135.8	17.1	149.5	49.2	91.1	26.1	51.2	354.4
o-Xylene	34	48.3	110.2	125.1	235.1	30	276.1	137.8	211.5	192.2
Carbon Tetrachloride	BDL	1.2	BDL	BDL	BDL	BDL	BDL	BDL	BDL	3.6
Chlorobenzene	BDL	37.9	BDL	BDL	BDL	BDL	50.6	BDL	BDL	201.9
Tetrachloroethane	BDL	30.3	1.4	3.5	BDL	0.5	26.2	2.8	BDL	4.6
Toluene	BDL	100.8	82.5	107.9	195.1	BDL	96.4	88.8	41.3	414.1
Chloroform	BDL	BDL	2	BDL	5.7	BDL	BDL	BDL	BDL	BDL
1,2-dichlorobenzene	BDL	BDL	82.6	23.6	128.6	BDL	25.9	BDL	71	82.7
Styrene	BDL	BDL	137.8	193.2	15.3	BDL	BDL	BDL	1.9	BDL
1,2,4-Trimethylbenzene	BDL	BDL	2.3	23.1	BDL	1.1	128.1	48.1	52.2	306.7
1,3,5-Trimethyl benzene	BDL	BDL	25.2	6.6	82.0	0.7	103.8	35.5	41.6	131.7
Bromo Benzene	BDL	BDL	BDL	31.4	26	BDL	7.1	BDL	314.5	1905.9
m&p – Xylene	BDL	BDL	BDL	BDL	BDL	BDL	72.2	1	BDL	82.6
Bromomethane	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	551.5	BDL
Tert Butylbenzene	BDL	BDL	BDL	BDL	BDL	BDL	BDL	BDL	10.5	25.7
Propylbenzene	BDL	BDL	BDL	BDL	47.7	BDL	67.5	19	6.1	37.4
Isopropylbenzene	1.3	BDL	2	BDL	4.2	BDL	14.6	0.7	BDL	BDL
Sec Butylbenzene	BDL	BDL	5.2	BDL	13.5	BDL	BDL	BDL	57.6	BDL
1,2,4 Trichlorobenzene	BDL	BDL	2.3	BDL	BDL	BDL	BDL	BDL	BDL	8.3







10.1.4 <u>AMBIENT AIR QUALITY MONITORING: LOTE MIDC - ROUND-1</u> (28/12/10 - 29/12/10)

24 hours' average concentration of total VOCs at each location with average of each cycle in first round is depicted in following Table for the ease of comparison.

Station No	Sampling Locations		(1	TVOCs Concentrations micro gram / cubic meter	·s)		
	Premises of M/s. Ratnagiri Telecom Exchange, MIDC, Lote	(13:00	81.1) Hrs - 21:00Hrs)	91.6 (21:10 Hrs - 05:10 Hrs)	114.1 (05:20 Hrs - 13:20Hrs)		
1	Parshuram Industrial Area. South East direction of MIDC Industrial area.		Av	verage Concentration : 95	5.6		
2	Premises of M/s. Ganesh Ice Factory, B -69/2, MIDC, Lote Parshuram Industrial Area,	(13:50	100.1) Hrs - 21:50Hrs)	1294.4 (22:00 Hrs - 06:00Hrs)	451.7 (06:10 Hrs - 14:10Hrs)		
	Center of MIDC Industrial area.		Ave	erage Concentration: 61	5.4		
	Premises of M/s. Amico Pesticides Ltd, B - 1/1, MIDC,	940.5 (15:30 Hrs - 23:30Hrs)		968.2 (23:40 Hrs - 07:40Hrs)	1132.6 (07:50 Hrs - 15:50Hrs)		
3	West direction of the MIDC industrial area.		Average Concentration : 1013.8				
	Premises of M/s. Kansai Nerolac Paints Ltd, F - 1/2, 1/3, MIDC,	(16:30	1812.4) Hrs - 00:30Hrs)	794.7 (00:40 Hrs - 08:40Hrs)	1168.6 (08:50 Hrs - 16:50Hrs)		
4	Lote Parshuram Industrial Area, West direction of Industrial estate.		Average Concentration : 1258.6				
5	Premises of CETP - Lote, MIDC, Lote Parshuram Industrial Area, South West direction of MIDC	(17:4	172.6 5 Hrs - 01:45Hrs)	2667.9 (01:50 Hrs - 09:50Hrs)	1489.1 (10:00 Hrs - 18:00Hrs)		
	Industrial Area. Near to admin block - open space, SW of MIDC		Average Concentration : 1443.2				
A A Q M St A A Q M St A A Q M St	ation in up wind direction ation in down wind direction ation in Cross wind direction	: Station-1 : Station-2 & 4 : Station-3 & 5					
Land Use	Land Use Classification			Industrial Zone			
Sky Condit	Sky Conditions		Sunny Sky				
Ambient T	emperature		20°C to 32°C				
Predomina	ant Wind Directions		South East to No	orth West and North East	to South West		
Sampling and Analysis Methodology Adopted: EPA TO-17							

<u>10.1.5 AMBIENT AIR QUALITY MONITORING: LOTE MIDC;</u> <u>ROUND-2 (29/12/10 - 30/12/10)</u>

24 hours' average concentration of total VOCs at each location with average of each cycle in second round is depicted in following Table for the ease of comparison.

Station No	Sampling Locations		(m	TVOCs Concentrations icro gram / cubic meter	s)		
	Premises of M/s. Ratnagiri Telecom Exchange, MIDC, Lote	(16	142.1 5:45 - 00:45Hrs)	543.5 (00:50 - 08:50 Hrs)	33.0 (08:55 - 16:55Hrs)		
1	Parshuram Industrial Area. South East direction of MIDC Industrial area.		Aver	rage Concentration : 23	9.7		
	Premises of M/s. Ganesh Ice Factory, B -69/2, MIDC, Lote	(16:5	356.9 55 Hrs - 00:55Hrs)	1153.8 (01:00 Hrs - 09:00Hrs)	210.8 (09:05 Hrs - 17:05Hrs)		
2	Parshuram Industrial Area, Center of MIDC Industrial area.		Aver	rage Concentration: 57	3.8		
	Premises of M/s. Amico Pesticides Ltd, B - 1/1, MIDC,	155.1 (17:00 Hrs - 01:00Hrs)		42.5 (01:05 Hrs - 09:05Hrs)	179.4 (09:10 Hrs - 17:10Hrs)		
3	Lote Parshuram Industrial Area, West direction of the MIDC industrial area.		Average Concentration : 125.7				
	Premises of M/s. Kansai Nerolac Paints Ltd, F - 1/2, 1/3, MIDC,	703.3 (17:20 Hrs - 01:20Hrs)		346.8 (01:25 Hrs - 09:25Hrs)	262.3 (09:30 Hrs - 17:30Hrs)		
4	Lote Parshuram Industrial Area, West direction of Industrial estate.		Average Concentration : 437.5				
	Premises of CETP - Lote, MIDC,	(17:3	7335.5 30 Hrs - 01:30Hrs)	3394.1 (01:35 Hrs - 09:35Hrs)	779.7 (09:40 Hrs - 17:40Hrs)		
5	South West direction of MIDC Industrial Area. SW of MIDC		Avera	age Concentration: 383	6.4		
A A Q M Station in up wind direction : A A Q M Station in down wind direction : A A Q M Station in Cross wind direction :			: Station-1 : Station-2 & 4 : Station-3 & 5				
Land Use Classification			Land Use Classification				
Sky Condit	tions		Sky Conditions				
Ambient T	emperature		Ambient Temperature				
Predomina	ant Wind Directions		Predominant Wind	Directions			
Sampling and Analysis Methodology Adopted: EPA TO-17							

10.1.6 AVERAGE CONCENTRATIONS OF ROUND1 & 2

24 hours' average concentrations of total VOCs at each location for both the rounds of monitoring are summarized in following Table for the ease of comparison.

Station No	Sampling Locations	Average VOCs Concentrations (micro gram / cubic meters)				
	Sampling Locations	ROUND - 1 (28.12.10 - 29.12.10)	ROUND - 2 (29.12.10 - 30.12.10)			
1	Premises of M/s. Ratnagiri Telecom Exchange, MIDC, Lote Parshuram Industrial Area. South East direction of MIDC Industrial area.	95.6	239.7			
2	Premises of M/s. Ganesh Ice Factory, B -69/2, MIDC, Lote Parshuram Industrial Area, Center of MIDC Industrial area.	615.4	573.8			
3	Premises of M/s. Amico Pesticides Ltd, B - 1/1, MIDC, Lote Parshuram Industrial Area, West direction of the MIDC industrial area.	1013.8	125.7			
4	Premises of M/s. Kansai Nerolac Paints Ltd, F - 1/2, 1/3, MIDC, Lote Parshuram Industrial Area, West direction of Industrial estate.	1258.6	437.5			
5	Premises of CETP - Lote, MIDC, Lote Parshuram Industrial Area, South West direction of MIDC Industrial Area. SW of MIDC	1443.2	3836.4			

10.1.7 COMPARISON OF MONITORING RESULTS WITH INTERNATIONAL STANDARDS/GUIDELINES

Detected VOC results of both the rounds of monitoring are compared with available international VOC standards for 24 hours average in following Tables. The referred international VOC standards/Guidelines are for Ontario (Canada), Arizona (USA), Alberta(Canada), Texas CEQ (USA) and Perth (Australia).

	Ambient Air Quality Standards (24 hours Average) µg/m3						Concentration (24 hours Average) µg/m ³			
PARAMETERS	Ontario	Arizona	Alberta	Texas CEQ ESL	Perth	Station - 1 Telephone Exchange		Station-2 Ice Factory		
						Round-1	Round-2	Round-1	Round-2	
Benzene	0.3	51	30 (1 hr)	75		3.2	20.0	8.7	BDL	
Chloroform	1	16		98	1	BDL	BDL	2.0	BDL	
Chlorobenzene	3500 (1hr)	2560		460		BDL	37.9	BDL	BDL	
1,2- Dichlorobenzene		2400				BDL	BDL	82.6	23.6	
Ethylbenzene						15.0	BDL	19.7	42.4	
Napthalene	22.5	400				42.1	1.2	135.8	17.1	
Tetrachloroethene						BDL	30.3	1.4	3.5	
Toluene	2000	3000	700	1880	4110	BDL	100.8	82.5	107.9	
1,2,4-Trimethylbenzene	12	280		1350	12	BDL	BDL	2.3	23.1	
1,3,5-Trimethylbenzene	22.5	400				BDL	BDL	25.2	6.6	
o-Xylene	730	3500		3700	1190	34.0	48.3	110.2	125.1	
m&p -Xylene	730	3500		2079	1190	BDL	BDL	BDL	BDL	
Carbon Tetrachloride	65	13		126	2.4	BDL	1.2	BDL	BDL	
Styrene	400	1700				BDL	BDL	137.8	193.2	

	Ambi	ent Air Qual	lity Standards (Concentration (24 hours Average) µg/m3					
Parameter	Ontario	Arizona	Alberta	Texas CEQ ESL	Perth	ΑΙΜϹΟ		Nerolac	
						Round-1	Round-2	Round-1	Round-2
Benzene	0.3	51	30 (1 hr)	75		8.2	3.4	17.4	3.0
Chloroform	1	16		98	1	5.7	BDL	BDL	BDL
Chlorobenzene	3500 (1hr)	2560		460		BDL	BDL	50.6	BDL
1,2- Dichlorobenzene		2400				128.6	BDL	25.9	BDL
Ethylbenzene						102.8	40.9	281.6	74.7
Napthalene	22.5	400				149.5	49.2	91.1	26.1
Tetrachloroethene						BDL	0.5	26.2	2.8
Toluene	2000	3000	700	1880	4110	195.1	BDL	96.4	88.8
1,2,4-Trimethylbenzene	12	280		1350	12	BDL	1.1	128.1	48.1
1,3,5-Trimethylbenzene	22.5	400				82	0.7	103.8	35.5
o-Xylene	730	3500		3700	1190	235.1	30.0	276.1	137.8
m&p -Xylene	730	3500		2079	1190	BDL	BDL	72.2	1
Carbon Tetrachloride	65	13		126	2.4	BDL	BDL	BDL	BDL
Styrene	400	1700				15.3	BDL	BDL	BDL

	Ambient	Air Quality	y Standards (Concentration (24 hours Average) µg/m3			
Parameter	Ontario	Arizona	Alberta	Texas CEQ ESL	Perth	CETP	
	Oncario					Round-1	Round-2
Benzene	0.3	51	30 (1 hr)	75		1.7	BDL
Chloroform	1	16		98	1	BDL	BDL
Chlorobenzene	3500 (1hr)	2560		460		BDL	201.9
1,2- Dichlorobenzene		2400				71.0	82.7
Ethylbenzene						30.3	84.6
Napthalene	22.5	400				51.2	354.4
Tetrachloroethene						BDL	4.6
Toluene	2000	3000	700	1880	4110	41.3	414.1
1,2,4-Trimethylbenzene	12	280		1350	12	52.2	306.7
1,3,5-Trimethylbenzene	22.5	400				41.6	131.7
o-Xylene	730	3500		3700	1190	211.5	192.2
m&p -Xylene	730	3500		2079	1190	BDL	82.6
Carbon Tetrachloride	65	13		126	2.4	BDL	3.6
Styrene	400	1700				1.9	BDL

10.2 Result and Discussion of Fugitive Emission Monitoring:

Unit - 1: <u>RALLIS INDIA LTD: (Pesticide Manufacturing Industry)</u>

Product	:	Acephate Technical (Pesticide)					
Qty. Production	:	4400 MT/Annum					
Raw Material Used	:	Dimethyl Phosphoro Amido Thiote (DMPAT)					
		Acetic Anhydride					
		Dimethyl Sulphate (DMSO ₄) - Catalyst					
		Sulfuric Acid - Catalyst					
		Liq. Ammonia - 22 - 24% (Neutralizing Agent)					
		Methylene Dichloride					
		Ethyl Acetate					
<u>Reaction:</u>							
DMPAT + Acetic An	hydrid	e Acetic Acid + Acephate MDC EAA Extract Purification					
Per Batch Raw Material consumption: Batch Size = 2200kgs							
DMPAT	=	2545 Kgs					
Acetic Anhydride	=	1770 Kgs					
Dimethyl Sulphate	=	299 Kgs					
Sulfuric Acid	=	18 Kgs					
Liq. Ammonia		= 1500 Kgs					
Methylene Dichloric	le	= 10,000 Lits. (BP = 39°C & Recovery = 96%)					
Ethyl Acetate		= 3000 Lits (BP = 85°C & Recovery = 94%)					
<u>Hazardous Waste G</u>	Hazardous Waste Generation during Process:						

Distillation Residue (MDC + EAA) = 2200 Lits per Month





Schematic showing fugitive emission monitoring locations

Location :1 - Production Block

Near Centrifuge (No.7) and Dryer Area (No.2)

2nd stage of Production Block (2nd floor)

Start Time: 11:35 Hrs and End Time: 15:35 Hrs

Sample ID : FUG/RALLIS/LOTE/01

Solvents are pumping in barrels.

Location :2 - Reactor Bottom Area (down wind of entire production block)

Stream stripping is one of the sources emitting maximum concentration of VOCs.

Start Time : 12:00 HrsEnd Time: 16:00 HrsFlow Rate: 300ml/min.Sample ID : FUG/RALLIS/LOTE/02

Sampling collected at two different places;

- 1. Materials Lifting Bay 2 hours
- 2. Between Production Block and Solvent Storage Area 2 Hours.

Process Flow Chart:



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Methylene Dichlo	ride :	Boiling Point is 39°C			
	:	96% Recovery			
	:	Loss is 400 lits in air, water and solid waste			
Ethyl Acetate	:	Boiling Point is 85°C			
	:	94% Recovery			
	:	Loss is 180 Lits in air, water and solid waste.			

The analysis results of fugitive emission monitoring are depicted in plate given below. The product, process reaction details and materials consumption details of the reported running product as given by the unit are furnished above. It is surprising that information given by the unit about solvent used is contrary to the actual VOCs found in the monitoring results. The results show that minimum six types of solvents were in use/stored by the unit. The concentration of Naphthalene found to be high near centrifuge area. More than 50% of Naphthalene is found in total VOCs detected at both the monitored locations. More than 25% Toluene is found in total VOCs detected between solvent storage & production block.

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Result of Fugitive Emission Monitoring in Rallies India Ltd.:



Unit - 2: USV Limited:

Segment	:	Active Pharma Ingrediants
Product	:	Nimodipine, Ropinirole HCl, Irbesartan,
		Clopidogrel Bisulfate, Glipizide and Glimepiride
Production Capacity	:	69 MT/Annum

Solvents Used:

Toluene, Ethyl Acetate, O-Xylene, Isopropyl Alcohol, Methylene Dichloride, Di Methyl Formamide, Acetone and Methanol.

Products Manufactured:

Nimodipine, Ropinirole HCl, Irbesartan, Clopidogrel Bisulfate, Glipizide and Glimepiride

Total Solvents loss/batch during manufacturing processes of above products:

Toluene + IPA + Methanol - 494 liters MDC + Acetone - 1595 liters

Due to non-operation of the plant, the fugitive emission monitoring was not carried out however, the stored effluent samples was collected from sump. The effluent analysis result is discussed separately.

Unit – 3: NEROLAC KANSAI LTD (Paint Manufacturing Industry)

Types of Products:

Water Based Paint

Industrial and Automotive Paints (solvent based)

Powder Coating Paint

Resins

Basic Raw Materials:

Resin / Medium / Binder

Pigment (Powder)

Solvent (Liquid)

Additives (Enhancement)

Processing:

Premixing (Thorough mixing)

Grinding (Ball Mill, Sand Mill, Dyna Mill & Arbitator) : Sand & Dyna mills are semi closed

Thinning – Mixer (500 lits to 10 KL)

Tinting

Filling & Packing



Location 1: Grinding Area



Max. TVOC in the Grinding and Tinning Area is 47ppm

Date : 30/12/2010 Start Time: 11:35 Hrs End Time: 15:35Hrs Flow:300ml/min.

Sample ID: FUG/NEROLAC/LOTE/03





Date : 30/12/2010Start Time: 12:00 HrsEnd Time: 16:00HrsFlow:500ml/min.

Sample ID: FUG/NEROLAC/LOTE/04

The analysis results of fugitive emission monitoring are depicted in plate given below. The product, process reaction details and materials consumption details of the reported running product as given by the unit are furnished above. The results show that minimum five types of solvents were in use/stored by the unit. The concentration of Ethyl benzene is found to be very high near grinding area. Concentration of other VOCs like Propylbenzene, Toluene, o-Xylene and m&P Xylene also found to be significantly high near grinding areas.

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Unit-4: GHARDA CHEMICALS: (Pesticide Industry)

Products Manufactured during Sampling:

Permethrin, Deltamethrin, Indoxacrab, Chloropyriphos, Trichlorpyr Easter

Solvents Used Processes:

Methylene Dichloride, Ethylene Dichloride, Toluene, O-Xylene, Chloroform



Location1: Production Block -2, Centrifuge Area - Near F- 2503

Date : 30/12/2010 Start Time: 12:00 HrsEnd Time: 16:00Hrs Flow:500ml/min.

Sample ID: FUG/NEROLAC/LOTE/04

Location2: Near Canteen Downwind of Production Block 4,5 & 6

Date : 30/12/2010Start Time: 12:00 HrsEnd Time: 16:00Hrs Flow:500ml/min.Sample ID: FUG/NEROLAC/LOTE/05

Location3: Downwind of Production Block III

Date : 30/12/2010Start Time: 12:00 HrsEnd Time: 16:00Hrs Flow:500ml/min.Sample ID: FUG/NEROLAC/LOTE/06

The analysis results of fugitive emission monitoring are depicted in plate given below. The product, process reaction details and materials consumption details of the reported running product as given by the unit are furnished above. The results show that minimum 13 types of solvents were in use/stored by the unit. The concentration of Toluene is found to be very high near downwind of production block-III. However, concentration of 1-2 Dichloroethane found to be very high near Canteen, i.e. downwind of production block 4,5,6. VOCs found below detection limit near production block-2.


10.3 RESULTS AND DISCUSSIONS OF WASTWATER MONITORING:

10.3.1 Rallis India Ltd.,:

	RALLIS I D101-O&G TRAP (NDIA LTD COLLECTION TANK												
	SAMPLING DA	TE: 29.12.2010	Volatile Organic Compound - Wastewater - Rallis India Ltd., Lote											
	PARAMETERS	RESULT(µg/I)		D101 - O&G Collection Tank										
1	Ethylbenzene	1.5		100										
2	4-Isopropyltolune	247.3		س 75										
3	Napthalene	13.68		8n1 uc 50										
4	Styrene	11.51		tratio										
5	Toluene	11.8		25	1.5		13.68	11.51	11.8	1.93	4.4	2.38		5.6
6	1,2,4-Trimethyl benzene	1.93		0 Co		4-				1,2,4-		_		
7	Xylene	4.4			ene	z Isopropy tolune	ne Napthale	Styrene	Toluene	Trimethyl benzene	Xylene	m&p- Xylene	thane	Bromome thane
8	m&p-Xylene	2.38		Series	l 1.5	247.3	13.68	11.51	11.8	1.93	4.4	2.38	159.9	5.6
9	Chloromethane	159.9												
10	Bromomethane	5.6												
	TOTAL VOC =	460												

Contrary to the information provided by the unit 10 VOCs are found in the raw effluent sample collected with highest concentration of 4-Isoproyltoluene followed by Chloromethane.

10.3.2 : USV India Ltd:

0.110	SAMPLING SITE: USV SAMPLING LOCATION:COLLECTION SUMP											
5.NO	SAMPLING DATE:30.12.2010					Vola	atile Organic Co USV Ltd	mpound - Wa Collection Sur	stewater			
	PARAMETERS	RESULT(µg/l)	150.0									138.2
1	N-Butyl benzen	5.0	125.0			118.6	_					
2	Ethylbenzene	2.1	°_ 100.0									
3	4-Isopropyltolune	118.6	u/8ri u									
4	Dichloromethane	380636.2	0.57									
5	Napthalene	11.9	50.0 CO									
6	Styrene	2.9	25.0	5.0				11.9	2.0	2.1		
7	Xylene	3.1	0.0	N-Butyl benzen	2.1 Ethylbenzene	4-	Dichloromethane	Napthalene	2.9 Styrene	Xylene	1.7 m&p-Xylene	Chloromethane
8	m&p-Xylene	1.7	Series1	5.0	2.1	isopropyitolune	380636.2	. 11.9	2.9	3.1	1.7	138.2
9	Chloromethane	138.2										
	TOTAL VOC =	380919.7										

Contrary to the information provided by the unit 9 VOCs are found in the raw effluent sample collected with exceptionally highest concentration of dichloromethane.

10.3.3 KANSAI NEROLAC PAINTS:

I. Collection Sump:



14 VOCs are found in the raw effluent sample collected with highest concentration of dichloromethane followed by Xylene & ethylbenzene.

10.3.4 GHARDA CHEMICALS : COLLECTION SUMP 1

S No	SAMPLING DATE:30.12.2010					
3.NO	PARAMETERS	RESULT(µg/l)				
1	Benzene	498.0				
2	Bromobenzene	331.9				
3	Bromochloromethane	212.3				
4	Chloroform	40.8				
5	Bromoform	1.2				
6	N-Butyl benzen	2.6				
7	Chlorobenzene	4716.0				
8	2-Chlorotoluene	25.7				
9	1,2-Dibromomethane	1.5				
10	1,2-Dichlorobenzene	4.9				
11	1,3-Dichlorobenzene	72.5				
12	1,4-Dichlorobenzene	84.3				
13	1,1-Dichloroethane	4.6				
14	Ethylbenzene	395.1				
15	Isopropylbenzene	23.2				
16	Propylbenzene	99.0				
17	4-Isopropyltolune	21.5				
18	Dichloromethane	35480.2				
19	Napthalene	3.5				
20	1,2,4-Trimethyl benzene	658.7				
21	Xylene	741.7				
22	m&p-Xylene	326.0				
23	1,3,5-Trimethylbenzene	128.3				
24	Chloroethane	122.6				
25	Chloromethane	28.0				
26	Bromomethane	11.2				
27	Vinyl chloride	414.1				
TOTAL	_ VOC =	44449.3				



Total 27 VOCs are found in the raw effluent sample collected from sump-1 with exceptionally high concentration of dichloromethane followed by Chlrobenzene.

GHARDA CHEMICALS : COLLECTION SUMP 2

S No	SAMPLING DATE	:30.12.2010		
onto	PARAMETERS	RESULT(µg/I)		
1	Bromobenzene	504		
2	Chloroform	19.14		
3	Bromoform	38.5		
4	Dibromochloromethane	6.23	SAMPLING SITE: GHARDA CHEMICALS	
5	Chlorobenzene	761	SAMPLING LOCATION:COLLECTION SUMP SAMPLING DATE: 30.12.2010	
6	2-Chlorotoluene	6.7	2500	
7	Dibromomethane	8.4		
8	1,2-Dichlorobenzene	715.3	2000	Bromoform
9	1,3-Dichlorobenzene	6.7		Dibromochloromethane
10	1,4-Dichlorobenzene	7.5	<u>5</u> 1500	■Chlorobenzene
11	1,2-Dichloroethane	9227.7		2-Chlorotoluene
12	Ethylbenzene	81.4	2 1000	■1,2-Dichlorobenzene
13	4-Isopropyltolune	22.1		■1,3-Dichlorobenzene
14	Dichloromethane	23155	500	■1,4-Dichlorobenzene
15	Napthalene	689.7	223.c 19 1 4 8.56 23 6 7 8 4 6 7 7 5 81.422 1 1 2 84.669.7 □	 I,2-Dichloroethane Ethylbenzene
16	Tetrachloroethene	1.2		■4-Isopropyltolune
17	1,2,4-Trimethyl benzene	742.6		
18	Xylene	736.1		
19	m&p-Xylene	84.6		
20	1,3,5-Trimethylbenzene	69.7		
21	Chloromethane	223.6		
-	TOTAL VOC =	37107.17		

Total 21 VOCs are found in the raw effluent sample collected from sump-2 with exceptionally high concentration of dichloromethane followed by 1,2-Dichloroethane.

10.3.5 COMMON EFFLUENT TREATMENT PLANT : COLLECTION SUMP AFTER RECEIVER

S No.	SAMPLING DATE:30.12.2010						
3.110	PARAMETERS	RESULT(µg/I)					
1	Benzene	17.0					
2	Bromobenzene	311.4					
3	Chloroform	8.2					
4	Bromoform	24.8					
5	Dibromochloromethane	4.9					
6	1,2 dichlorobenzene	1027.3					
7	Chlorobenzene	307.9					
8	1,4-Dichlorobenzene	13.3					
9	Ethylbenzene	37.6					
10	4-Isopropyltolune	13.3					
11	Napthalene	724.6					
12	1,2,4-Trichlorbenzene	3.2					
13	Trichloroehtylene	1.3					
14	1,2,4-Trimethyl benzene	761.2					
15	1,2,3-Trimethyl benzene	3.2					
16	Xylene	826.1					
17	m&p-Xylene	48.8					
18	1,3,5-Trimethylbenzene	54.8					
19	Chloromethane	72.4					
20	Vinyl chloride	1.5					
	TOTAL VOC =	4262.8					
Tatal	20 VOCa are found	in the rout off.					



Total 20 VOCs are found in the raw effluent sample collected from CETP with highest concentration of 1,2 Dichlorobenzene followed by Xylene.

11 QUALITATIVE ANALYSIS REPORT OF SAMPLES:

As mentioned at the beginning of this report that major focus of the study is on 60 parameters but efforts is also made to list out the presence of other VOCs on the basis of qualitative analysis showing only presence or absence of other VOCs. The presence of VOCs other than major 60 parameters summarized for AAQ and effluent samples and depicted in following Tables:

VOLATILE ORGANIC COMPOUND 2110000499,501,502 AAQ							
SAMPLING AREA: MIDC LOTE							
SAMPLING SITE: TELECOME EXCHANGE							
PARAMETERS	DATE:28.12.10 TIME:13.00-21.00	DATE:28.12.10-29.12.10 TIME:21.10-05.10	DATE:29.12.10 TIME:05.20- 13.20				
	RESULT	RESULT	RESULT				
Acetone	Present	**	**				
Hexane	Present	Present	**				
Ethylacetate	Present	Present	**				

11.1 Qualitative Analysis of AAQ Samples

VOLATILE ORGANIC COMPOUND 2110000503 ,504,505 AAQ							
SAMPLING AREA: MIDC LOTE							
SAMPLING SITE: TELECOM EXCHANGE, LOTE							
PARAMETERS	DATE:29.12.10 - 30.12.10 TIME:16.45-0.45	DATE:30.12.10 TIME:0.50-08.50	DATE:30.12.10 TIME:08.55-16.55				
	RESULTS	RESULTS	RESULTS				
Acetone	**	Present	**				
Hexane	Present	Present	Present				

VOLATILE ORGANIC COMPOUND 2110000506 ,507,508 AAQ							
SAMPLING AREA: MIDC LOTE							
SAMPLING SITE: GANESH ICE FACTORY							
PARAMETERS	DATE:28.12.10 TIME:13.50-21.50	DATE:28.12.10 -29.12.2010 TIME:22.00-06.00	DATE:29.12.10 -29.12.2010 TIME:22.00-06.00				
	RESULTS	RESULTS	RESULTS				
Acetone	**	**	**				
Hexane	Present	Present	**				

VOLATILE ORGANIC COMPOUND 2110000509 ,510,511 AAQ							
SAMPLING SITE: GANESH ICE FACTORY							
PARAMETERS	DATE:29.12.10 -30.12.10 TIME:16.55-0.55	DATE:30.12.10 TIME:01.00-09.00	DATE:30.12.10 TIME:09.05-17.05				
	RESULTS	RESULTS	RESULTS				
Acetone	**	**	**				
Hexane	Present	Present	Present				
Ethylacetate	Present	**	Present				
VOLAT	VOLATILE ORGANIC COMPOUND 2110000512 ,513,514 AAQ						
	SAMPLING AF	REA: MIDC LOTE					
	SAMPLING SITE:	AIMCO PESTICIDES					
PARAMETERS	DATE:28.12.10 TIME:15.30-23.30	DATE:28.12.10 - 29.12.201 TIME:23.40-07.40	0 DATE:29.12.10 TIME:07.50-15.50				
	RESULTS	RESULTS	RESULTS				
Acetone	**		Present				
Hexane	Present		Present				
Ethylacetate	Present		Present				
Ethylaclcohol	**		Present				
Methyl isobutyl ketone	**		Present				

VOLATILE ORGANIC COMPOUND 2110000515 ,516,517 AAQ						
SAMPLING AREA: MIDC LOTE						
SAMPLING SITE: AIMCO PESTICIDES						
PARAMETERS	DATE:29.12.10 -30.12.10 TIME:17.10-1.00	DATE:30.12.10 TIME:01.05-09.05	DATE:30.12.10 TIME:09.10-17.10			
	RESULTS	RESULTS	RESULTS			
Acetone	**	**	**			
Hexane	Present	Present	**			

VOLATILE ORGANIC COMPOUND 2110000518 ,519,520 AAQ							
SAMPLING AREA: MIDC LOTE							
SAMPLING SITE:NEROLAC PAINTS							
PARAMETERS	DATE:28.12.10 TIME:16.3030	DATE:29.12.10 TIME:0.40-08.40	DATE:29.12.10 TIME:8.50-16.50				
	RESULTS	RESULTS	RESULTS				
Acetone	Present	**					
Hexane	Present	**					
Ethylacetate Present **							
Methyl Isobutyl ketone	Present	**	Present				

VOLATILE ORGANIC COMPOUND 2110000521 ,522,523 AAQ							
SAMPLING AREA: MIDC LOTE							
SAMPLING SITE:NEROLAC PAINTS							
PARAMETERS	DATE:29.12.10 -30.12.10 TIME:17.20-1.20	DATE:30.12.10 TIME:1.25-09.25	DATE:30.12.10 TIME:9.30-17.30				
	RESULTS (µg/m3)	RESULTS (µg/m3)	RESULTS (µg/m3)				
Acetone	**	**	**				
Hexane	Present	**	Present				

VOLATILE ORGANIC COMPOUND 2110000524 ,525,526 AAQ							
	SAMPLING AREA: MIDC LOTE						
SA	SAMPLING SITE: COMMON EFFLUENT TREATMENT PLANT						
PARAMETERS	DATE:28.12.10 TIME:17.45-1.45	DATE:29.12.10 TIME:1.50-09.50	DATE:29.12.10 TIME:10.00-18.00				
	RESULTS (µg/m3) RESULTS (µg/m3) RESULTS (µg/m3)						
Acetone ** ** **							
Hexane Present Present **							
Ethylacetate	**	Present	**				

VOLATILE ORGANIC COMPOUND 2110000527 ,528,529 AAQ						
	SAMPLING AREA: MIDC LOTE					
SAI	SAMPLING SITE: COMMON EFFLUENT TREATMENT PLANT					
PARAMETERS	DATE:29.12.10-30.12.2010 DATE:30.12 TIME:17.30-1.30 TIME:1.35-09		DATE:30.12.10 TIME:9.40-17.40			
	RESULTS (µg/m3)	RESULTS (µg/m3)	RESULTS (µg/m3)			
Acetone	Present					
Hexane Present		Present	Present			
Ethylacetate	Present					

It may be seen from the above results that Hexane is present at almost all locations.

11.2 Qualitative Analysis of Effluent Samples

VOLATILE ORGANIC COMPOUND 2110000713 EFFLUENT WATER					
SAMPLING SITE: RALLIS INDIA LTD					
SAMPLING LOCATION:	SAMPLING LOCATION:D101-O&G TRAP COLLECTION TANK				
SAMPLING AREA: LOTE	INDUSTRIAL ESTATE , MAHARASTRA				
SAMPLING DATE:29.12.2010					
PARAMETERS RESULT					
Isopropyl alchohol	Present				
Ethylalcohol	Present				
Ethylacetate Present					
Dimethyl disulfide	Present				

VOLATILE ORGANIC COMPOUND 2110000714 EFFLUENT WATER					
SAMPLING SITE: USV Ltd					
SAMPLING LOC	CATION:COLLECTION SUMP				
SAMPLING AREA: LOTE	SAMPLING AREA: LOTE INDUSTRIAL ESTATE , MAHARASTRA				
SAMPLI	NG DATE:30.12.2010				
PARAMETERS	RESULI				
	Present				
Ethylaconol	Present				
Ethylacetate	Present				
Dimethyl disullide	Present				
Dimethyl trisulfide	Present				
Dimetry trisuinde	Present				
VOLATILE ORGANIC COM	POUND 2110000715 EFFLUENT WATER				
SAMPLING SITE	: KANSAI NEROLAC PAINTS				
SAMPLING LOC	CATION:COLLECTION SUMP				
SAMPLING AREA: LOTE	INDUSTRIAL ESTATE , MAHARASTRA				
SAMPLI	NG DATE:30.12.2010				
PARAMETERS	RESULT				
Isopropyl alchohol	Present				
Ethylalcohol	Present				
Acetone	Present				
Aceticacid	Present				
Dimethyl sulfide	Present				
Methyl isobutyl ketone	Present				
	TE: CHARDA CHEMICALS				
SAMPLING AREA: LOTE					
SAMPLI	NG DATE:30.12.2010				
PARAMETERS	RESULT				
Isopropyl alchohol	Present				
Ethylalcohol	Present				
Ethylacetate	Present				
Hexane	Present				
Dimethyl disulfide	Present				
VOLATILE ORGANIC COM	POUND 2110000717 EFFLUENT WATER				
SAMPLING SI	TE: GHARDA CHEMICALS				
SAMPLING LOCATION:COLLECTION SUMP					
SAMPLING AREA: LOTE INDUSTRIAL ESTATE , MAHARASTRA					
SAMPLI	NG DATE:30.12.2010				
PARAMETERS	RESULT				
Ethylalcohol	Present				
Aceticacid	Present				
Hexane	Present				
Dimethyl disulfide	Present				

SGS India Private Limited, Chennai and Central Pollution Control Board, Zonal Office (W), Vadodara

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VOLATILE ORGANIC COMPOUND 2110000718 EFFLUENT WATER				
SAMPLING SITE: COM	MON EFFLUENT TREATMENT PLANT			
SAMPLING LOCATION:	COLLECTION SUMP AFTER RECEIVER			
SAMPLING AREA: LOTE	INDUSTRIAL ESTATE , MAHARASTRA			
SAMPI	_ING DATE:30.12.2010			
PARAMETERS	RESULT			
Ethylalcohol	Present			
Aceticacid	Present			
Hexane	Present			
Ethylacetate	Present			
Dimethyl disulfide	Present			

12.0 OBSERVATIONS:

Findings on the basis of predominant VOCs' concentration are summarized and discussed in subsequent paragraphs.

Observation on Ambient Air Quality at LOTE Industrial estate, Maharashtra:

• The 24 hours concentration of VOCs during first round of monitoring at locations 2,3 & 4 found to be higher than second round of monitoring, however reverse scenario found at location no. 1 & 5.

Sr. No.	VOCs
1	Benzene
2	Ethyl Benzene
3	Napthalene
4	o-Xylene
5	Carbon Tetrachloride
6	Chlorobenzene
7	Tetrachloroethane
8	Toluene
9	Chloroform
10	1,2-dichlorobenzene
11	Styrene
12	1,2,4-Trimethylbenzene
13	1,3,5-Trimethylbenzene
14	Bromobenzene
15	m&p-Xylene
16	Bromomethane
17	Tert Butylbenzene
18	Propylbenzene
19	Isopropylbenzene
20	Sec Butylbenzene
21	1,2,4 Trichlorobenzene

• A total 21 VOCs are detected, as tabulated below, in the ambient air.

- Naphthalene and o-Xylene are detected at all the location during both the rounds of monitoring.
- The concentration of Bromobenzene, Bromomethane, 1,2,4-Trimethylbenzene, Toluene, Naphthalene, Styrene, Ethyl Benzene, o-Xylene,Chlorobenzene,1,2dichlorobenzene and Toluene are found to be higher at one or the other locations than other detected VOCs.
- The total 24-hours average VOCs' concentration was found to be highest during both the rounds of monitoring at location no. 5 i.e. in premises of CETP (crosswind direction of the estate; close to south-west of industrial estate). Highest 24 hours average concentration of VOCs during first round of monitoring was found to be 1443.2 microgram/cubic meters. Highest concentration during second round of monitoring was found to be 3836.4 microgram/cubic meters.
- The concentrations of Benzene in terms of μ g/m³ (24hrs Average) at sampling locations are given in the table below.

Benzene	SAMPLING LOCATIONS				
	TELEPHONE EXHANGE	ICE FACTORY	AIMCO	NEROLAC	СЕТР
Round-1	3.2	8.7	8.2	17.4	1.7
Round-2	20.0	BDL	3.4	3.0	BDL

Though, the ambient air quality standard for benzene is prescribed for annual average but an attempt is made here to compare it with concentration found in 24-hours monitoring. It is observed that the concentrations of Benzene (24 hrs average) during first round of monitoring at Nerolac Paints, Ice Factory and AIMCO are higher than the NAAQM standard i.e.5 μ g/m³(annual average) and found below the NAAQM standard at rest of the locations. However, during

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second round of monitoring it is found more only at location of Telephone exchange. Though the highest concentration of total VOCs are found at location no. 5 i.e. premises of CETP but concentration of Benzene found to be lowest at this location. When compared with international 24 hours average VOC standards/Guidelines prescribed by Ontario, most of the locations are exceeding the specified limit. EPA has classified benzene as a Group A, human carcinogen.



Health Data from Inhalation Exposure

Benzene

• The concentrations of Toluene in term of $\mu g/m^3$ (24hrs Average) at sampling locations are given in the table below.

TOLUENE	SAMPLING LOCATIONS				
	TELEPHONE EXHANGE	ICE FACTORY	AIMCO	NEROLAC	СЕТР
Round-1	BDL	82.5	195.1	96.4	41.3
Round-2	100.8	107.9	BDL	88.8	414.1

Toluene concentrations of all ambient locations are lesser when compared with international AAQM standards/guidelines. The highest concentration of 414.1 μ g/m³ is found at CETP premises during second round of monitoring.

- The concentrations of Chloroform detected in only two locations during first round of monitoring i.e., GANESH ICE FACTORY -2µg/m3 and AIMCO PESTICIDIES
 - 5.7µg/m3. The standard for Chloroform is very stringent due to high toxicity and prescribed as 1µg/m3 in the Ontario ambient air quality standard/guidelines.
- Very high concentration of Bromobenzene (Boiling Point is 156°C) is detected in CETP premises during second round of monitoring i.e. 1905.9 µg/m3. It clearly shows some industries are using or emitting large quantity of this solvent since the high concentration is found at CETP inlet effluent too. The toxicity of Bromobenzene is harmful if inhaled, swallowed or absorbed through the skin. It has been categorized as severe irritant, harmful to aquatic organisms and may cause long-term damage in the environment. However, no exposure limits are established for Bromobenzene. The acute and chronic health effects are further described below:

Potential Acute Health Effects:

Very hazardous in case of eye contact (irritant), ingestion, inhalation. Hazardous in case of skin contact (irritant, permeator). Inflammation of the eye is characterized by redness, watering, and itching.

Potential Chronic Health Effects:

Very hazardous in case of eye contact (irritant), ingestion, inhalation. Hazardous in case of skin contact (irritant, permeator). The substance is toxic to kidneys, the nervous system, and liver. Repeated or prolonged exposure to the substance can produce target organs damage.

• The concentrations of Napthalene (Boiling 239° C) in terms of μ g/m³ (24hrs Average) at sampling locations are given in the table below.

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NAPTHALENE	SAMPLING LOCATIONS				
	STATION-1 TELEPHONE EXHANGE	STATION-2 ICE FACTORY	AIMCO	NEROLAC	СЕТР
Round-1	42.1	135.8	149.5	91.1	51.2
Round-2	1.2	17.1	49.2	26.1	354.4

 Naphthalene is detected at all the locations during both rounds of monitoring with maximum concentration (354.4 µg/m3) at CETP during second round of monitoring. It carries potential health hazard to human due to high exposure, harmful if swallowed and may cause respiratory, skin or eye irritation. Threshold Limit Value is 10 ppm for 8 hours exposure. It is sensitizer and possible carcinogen. EPA has classified naphthalene as a Group C, possible human carcinogen.



Health Data from Inhalation Exposure

- The concentration of Bromomethane in terms of µg/m³ (24hrs Average) at CETP is detected 551.5 µg/m³ during first round of monitoring and 1905.9 µg/m³ during second round of monitoring. However, concentration is found to be below detection limit at all other locations. The toxicity of Bromomethane may cause damage to the following organs: kidneys, astrointestinal tract, upper respiratory tract, skin, eyes, central nervous system (CNS). Carcinogenic effects are classified & (Proven) by NIOSH. Exposure Limits of Bromomethane as given by ACGIH TLV (United States, 1/2009) if absorbed through skin is TWA: 3.9 mg/m³ 8 hour(s) & TWA: 1 ppm 8 hour(s).
- The detected concentration of Styrene in terms of µg/m³ (24hrs Average) is found maximum at Ganesh Ice Factory with value of 193.2µg/m³ during second round of monitoring. It is hazardous if emitted into the atmosphere. Acute (short-term) exposure to styrene in humans results in mucous membrane & eye irritation and gastrointestinal effects. Chronic (long-term) exposure to styrene in humans results in effects on the central nervous system (CNS), such as headache, fatigue, weakness, depression, CSN dysfunction, hearing loss, and peripheral neuropathy.



Health Data from Inhalation Exposure

The o-Xylene is detected at all the locations and the concentration ranges from 30 to 235.1µg/m³. It is hazardous if emitted into the atmosphere. Acute (short-term) inhalation exposure to mixed xylenes in humans results in irritation of the eyes, nose, throat, gastrointestinal effects, eye irritation, and neurological effects. Chronic (long-term) inhalation exposure of humans to mixed xylenes results primarily in central nervous system (CNS) effects, such as headache, dizziness, fatigue, tremors, and in coordination; respiratory, cardiovascular, and kidney effects have also been reported. EPA has classified mixed xylenes as a Group D, not classifiable as to human carcinogenicity.



- The maximum average total VOC concentrations during first round of monitoring are found at CETP premises [1443.2 µg/m³; south-west direction of the MIDC industrial area] followed by M/S Kansai Nerolac Paints Ltd., [1258.6µg/m³; west direction of the MIDC industrial area] & M/S Amico Pesticides [1013.8µg/m³; Centre of the MIDC industrial area].
- The maximum average total VOC concentrations during second round of monitoring are found at CETP premises [3836.4 µg/m³; south-west direction of the MIDC industrial area] followed by M/S Ganesh Ice Factory, [573.8 µg/m³; centre of the MIDC industrial area].

• There is no doubt in the fact that presence of VOCs in the ambient air are only due to industrial processes, raw materials, products and by-products.

Observation on Fugitive Emission Monitoring:

• The probable places of loss of VOCs through fugitive emissions are Centrifuging, filtration, glands, charging material into the reactors, solvent storage area, distillation and ETP area.

• Volatile organic compound are monitored at following three industrial fugitive emission sources covering total seven locations.

M/s Rallis India ltd,

1. Between centrifuge area; 2. Between solvent storage area.

M/S Kansai Nerolac paints ltd,

1. Grinding area of plant; 2.Down wind/back side of the unit.

M/s Gharda chemicals ltd,

1.Centrifuge area; 2.Down wind of 1,2,3 Production block; 3.Down wind of 4,5,6 production block.

• In Rallis India ltd detected VOCs with maximum concentrations are Naphthalene $(1087.0\mu g/m^3)$ and 1,2,4- Trimethyl benzene $(210.9.0\mu g/m^3)$.

• In Kansai Nerolac paints detected VOCs with maximum concentrations are Ethyl benzene (4155.1 μ g/m³), Prppylbenzene (1945.3 μ g/m³), o- Xylene (2021.2 μ g/m³), m&pXylene(1239.9 μ g/m³) and Toluene 558.2 μ g/m³.

• In Gharda chemicals ltd, detected VOCs are Carbontetrachloride, Chlorobenzene, Ethylbenzene, Dichloromethane, Napthalene, Toluene, 1,2,4-Trimethylbenzene, 1,3,5-Trimethylbenzene, o-Xylene, m&p xylene and Styrene. The VOC detected with maximum concentration is 1,2-Dichloroethane (9464µg/m³).

It is learnt from literature review that exposure to low levels of ethylene dichloride can occur from breathing ambient or workplace air. Inhalation of concentrated ethylene dichloride vapor can induce effects on the human nervous system, liver, and

kidneys, as well as respiratory distress, cardiac arrhythmia, nausea, and vomiting. Chronic (long-term) inhalation exposure to ethylene dichloride produced effects on the liver and kidneys in animals. EPA has classified ethylene dichloride as a Group B2, probable human carcinogen.



1,2-Dichloroethane

• The numbers and types of VOCs found at few locations during fugitive emission monitoring are in contradiction to the name & number of solvent used provided by the industry concerned.

• The concentration of VOCs in fugitive emissions are primary cause of concern for workers but equally important from ambient air quality of the industrial estate as whole, as many proven adverse health effects including increase in ozone level concentration to trigger formation of other secondary pollutants. It can also be concluded that fugitive sources are predominant contributors of VOCs in ambient through the means like leakages, effluent discharge etc.

Observation on Wastewater Monitoring:

• The presence of solvents (VOCs) in the wastewater samples occur mainly due to improper separation of intermediates/products/solvents at different unit

process operations, which result in to high organic load in wastewater and difficulty in treatment.

- Normally the wastewater generated by the industries contains high VOCs and the industries are doing only primary treatment. During these processes some percentage of VOCs are dispersed into the atmosphere. After primary treatment the industries are pumping their effluent to CETP for further treatments. In CETP, during aeration process, most of the VOCs are vaporized and dispersed into the atmosphere. These VOCs are directly impacting the Ambient VOCs concentration.
- Presence of VOCs in wastewater affects the ambient air quality, efficiency of the effluent treatment system and the quality of the final receiving body. VOCs (Solvents) are emitted in to the environment during various treatment unit operations particularly during aeration in equalization tanks and aeration tanks.
- Effluent samples collected and analyzed from following Locations 1. Rallis O&G Trap Collection Tank, 2.USV Collection sump, 3. Kansai Nerolac paints Collection sump, 4.Gharda chemical Collection sump and 5.Common effluent treatment plant.
- The 4-Isopropyltoluene(247.3µg/l) and Chloromethane(159.9µg/l) are found with maximum concentration in collection tank after O & G trap of M/s Rallies India Ltd.
- The exceptionally high concentration of Dichloromethane (380636.2µg/l) is detected in USV collection sump which is much higher than any other locations in LOTE industrial estate. It clearly indicates that huge amount of Dichloromethane is used by this industry and in turn the presence in effluent is a threat to the efficient operation of CETP as well as to the environment.
- The VOCs detected with maximum concentrations in M/s Kansai Nerolac Paints are Ethyl benzene (1450.6µg/l), Dichloromethane (3646.5µg/l), o-Xylene(1741.1µg/l), m &p-Xylene(926.5µg/l) and Toluene (588.6µg/l). In

addition to these presence of Benzene, Chloroform, Chloromethane, Styrene, Naphthalene, Isopropyl benzene and 4-Isopropyl toluene also found in effluent sample.

- The exceptionally high concentration of Dichloromethane (35480.2 μ g/l) is detected in collection sump-1 of M/s Gharda Chemicals. The concentration of Dichloromethane in collection sump-2 of the unit also found to be very high i.e. 23155 μ g/l. It clearly indicates that huge amount of Dichloromethane is used by this industry and in turn the presence in effluent is a threat to the efficient operation of CETP as well as to the environment.
- High concentration of Dichloromethane is found in all locations except one in M/s Rallies India Ltd..
- Chlorobenzene, 1,2-Dichlorobenzene, 1,2-Dichloroethane, Dichloromethane, 1,2,4 Trimethylbenzene and Xylene are predominant in effluent sample of M/s Gharda chemicals.
- 1,2 -Dichlorobenzene, Xylene, 1,2,4 Trimethylbenzene and Naphthalene are predominant in effluent sample of common effluent treatment plant (CETP).
- The presence of VOCs in effluent not only causes great adverse impact on the efficient operation of biological treatment system of the Common effluent Treatment Plant but also a cause of great concern for adverse environmental impact.
- The higher concentrations of VOCs in effluent shows the less concern of the industries in control & recovery of solvents used in the process.

13.0 RECOMMENDATIONS

The CPCB-Zonal Office suggested following recommendations during deliberation on the findings of the monitoring. The aspect of VOCs emissions, control and presence in

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ambient is still at primary stage in the country. There is a need for development of common protocol of sampling and analysis based on Indian conditions. So that uniform database can be generated for further course of action on development of standard & technology as well.

More laboratories should be encouraged to come up with advanced facilities for sampling and analysis of VOCs which, may help in increasing the capabilities and thereby cost of monitoring will reduce.

Buffer zone with green belt around the industrial estates should be ensured to minimize the impact on the surroundings.

The workers in the industrial units should be made aware about effects of VOCs of environment and human health. They should also be properly trained to handle the solvents so as to minimize the escape of VOCs in to atmosphere.

Strategic road-map is required to be prepared for development of standards/guidelines, LDAR programmes for various industrial sectors and creation of awareness on VOCs.

The industries can look more closely in to following aspects for control of VOCs:

- Closed handling system for chemicals;
- Improved solvent recovery by the use of some special condensers and sub cooling system.
- Mechanical seal for chemical handling pumps;
- LDAR system;
- Venting of storages with trap receiver & condenser; and
- Training to the labors and staff.
- Proper system of loading and unloading of solvents
- Proper solvent recovery systems
- Work environment monitoring with respect to VOC has to conduct and compare with Factories act's occupational health standards.

All the emissions emitting sources are to be channelized through ducts to a common conduit, after advanced condensers and/ or scrubbing with relevant or proper scrubbing, the treated emissions to be let into the air.

14.0 CONCLUSIONS

Following conclusions are drawn from the field observations, monitoring results and above discussions:

- There is escape of VOCs from industries through fugitive emissions, effluent discharge and hazardous waste generation but units normally pay least attention to identify & quantify such losses & discharge of VOCs. Limited available analysis facility, absence of emission/discharge standards, no mandatory LDAR programme and cost involved in assessment & control are the major factors on part of the emissions of VOCs from industrial units.
- Companies change the products with respect to demand in the market accordingly the solvent type and quantity will also vary. Due to these variations the concentrations of VOC in AAQ, Fugitive, wastewater and solid waste may vary.
- There are cases, where number of VOCs detected in the samples collected during fugitive emission and wastewater sampling found to be more than what units reported in their consumption details. Units have to understand the importance of transparency in information sharing as their employees are first to get the adverse health impact of VOCs if not controlled properly.
- The probable reasons for escape of solvent in to atmosphere are:
 - Inadequate storage facilities for material. (e.g. leakages from store.
 - Use of small carboys for temporary storages of solvents and residues.
 - Inadequate closure of reactor vessels.
 - Crude temperature control methods for process.
 - Evaporation from filtration, centrifuge, layer separation due to improper equipment specifications.

- Improper maintenance of pumps, flanges, valves, compressors, condensers, coolers.
- Limited efficiency of reflux condensers.
- Lack of awareness among labors/workers is also responsible on part of escape of VOCs in atmosphere due to human error or negligence.
- Fume extraction systems of all the possible emission sources and that can be treated.

For developing the standard protocol, standards and guidelines for control of VOCs in ambient environment, it is required to generate data-base for substantial time-span in different industrial regions by similar type of studies.

Annexure-I

S.NO	VOC	S.NO	VOC
1	Benzene	31	Trans-1,3-Dichloropropene
2	Bromobenzene	32	Ethyl Benzene
3	Bromochloromethane	33	Hexachloro-1,3-butadiene
4	Bromodichloromethane	34	Isopropylbenzene
5	Chloroform	35	Para-Isopropyltoluene
6	Bromoform	36	Methylenechloride
7	n-Butylbenzene	37	Naphthalene
8	Sec-Butylbenzene	38	2-Propylbenzene
9	Ter-Butylbenzene	39	Styrene
10	Carbon Tetra chloride	40	1,1,1,2-Tetrachloroethane
11	Chlorobenzene	41	1,1,2,2-Tetrachloroethane
12	2-Chlorotoluene	42	Tetrachloroethene
13	4-Chlorotoluene	43	Toluene
14	Dibromochloromethane	44	1,2,3-Trichlorobenzene
15	1,2-Dibromo-3-chloropropane	45	1,2,4-Trichlorobenzene
16	1,2-Dibromoethane	46	1,1,1-Trichloroethane
17	Dibromomethane	47	1,1,2-Trichloroethane
18	1,2-Dichlorobenzene	48	Trichloroethylene
19	1,3-Dichlorobenzene	49	1,2,3-Trichloropropane
20	1,4-Dichlorobenzene	50	1,2,4-Trimethylbenzene
21	1,1-Dichloroethane	51	1,3,5-Trimethylbenzene
22	1,2-Dichloroethane	52	Xylene
23	1,1-Dichloroethene	53	Meta-Xylene
24	Cis-1,2-Dichloroethene	54	Para-Xylene
25	Trians-1,2-Dichloroethene	55	Chloroethane
26	1,2-Dichloropropane	56	Chloromethane
27	1,3-Dichloropropane	57	Trichlorofluoromethane
28	2,2-Dichloropropane	58	Bromomethane
29	1,1-Dichloropropene	59	Vinyl Chloride
30	Cis-1,3-Dichloropropene	60	Dichlorofluoromethane

List of 60 Volatile Organic Compounds (VOC s)

Annexure-II

PHYSICAL & CHEMICAL PROPERTIES OF VOCs

			Solubility	Boiling
S.No.	Name of VOC	Mol.Wt.	water.	Point D
			at 20 (ml /100 ml)	
1	Dichlorofluoromethane	102		
	Name: Fluorodichloromethane			
	Formula: CHCl2F			
	MW: 102			
2	Chloromethane	50	303	-23.7
	Name: Methane, chloro-		(Slightly soluble)	
	Formula: CH3Cl			
_	MW: 50	(2)		40.07
3	Vinyl chloride	62	Slightly soluble	-13.37
l	Name: Ethene, chloro-			
I				
4	MW: 62	0.1	1 7E a / 100 a water	2.57
4	Name: Methane, bromo	94	1.75 g / 100 g water	3.30
	Formula: CH3Br			
	MW· 94			
5	Fthylchloride	64	0.574 g / 100 ml	12.3
5	Name: Ethyl Chloride	04	0.374 g / 100 mil	12.5
	Formula: C2H5Cl			
	MW: 64			
6	Name: Trichloromonofluoromethane	136	insol. In water	23.7
-	Formula: CCI3F			
	MW: 136			
7	Name: Ethene, 1,1-dichloro-	96	Practically insoluble	31.7
	Formula: C2H2Cl2		in water.	
	MW: 96			
8	Name: Methylene Chloride	84	~ 50 parts water.	39.75
	Formula: CH2Cl2			
	MW: 84			
9	Trans 1,2 Dichloroethene	96	Insol. In water.	59.6
	Name: Ethene, 1,2-dichloro-, (E)-			
	Formula: C2H2Cl2			
	MW: 96			
10	1,1 Dichloroethane	98	Soluble in about	57.3
	Name: Ethane, 1,1-dichloro-		200 parts water.	
	Formula: C2H4Cl2			
4.4	MW: 98			F0 (
11	Cis 1,2 - Dichloroethene	96	insol.in water.	59.6
	Name: Etnene, 1,2-dichloro-, (2)-			
10	MW, 90	420		
12	DI OINOCHIOFOMETNANE	128		1

	Name: Methane, bromochloro- Formula: CH2BrCl			
	MW: 128			
13	Chloroform Name: Trichloromethane Formula: CHCl3 MW: 118	118	1 ml / 200 ml water	61-62
14	Name: Propane, 2,2-dichloro- Formula: C3H6Cl2 MW: 112	112	Slightly sol. In water	95-96
15	Name: Ethane, 1,2-dichloro- Formula: C2H4Cl2 MW: 98	98	sol. In 120 parts Water	83-84
16	Name: Ethane, 1,1,1-trichloro- Formula: C2H3Cl3 MW: 132	132	insol. In water	74.1
17	Name: 1-Propene, 1,1-dichloro- Formula: C3H4Cl2 MW: 110	110		108
18	Carbon Tetrachloride Name: Carbon Tetrachloride Formula: CCl4 MW: 152	152	1 ml / 2000 ml water	76.7
19	Name: Benzene Formula: C6H6 MW: 78	78	0.188%	80.1
20	Name: Methane, dibromo- Formula: CH2Br2 MW: 172	172	11.93 g / 1000g water	97
21	1,2 dichloropropane Name: Propane, 1,2-dichloro- Formula: C3H6Cl2 MW: 112	112	Slightly soluble in water.	95 - 96
22	Trichloroethylene Name: Trichloroethylene Formula: C2HCl3 MW: 130	130	0.11 g / 100 g.	86.9
23	Bromodichloromethane Name: Methane, bromodichloro- Formula: CHBrCl2 MW: 162	162		91-92
24	1,3 dichloropropene Name: 1-Propene, 1,3-dichloro- Formula: C3H4Cl2 MW: 110	110		108
25	Trans 1,3 dichloropropene Name: 1-Propene, 1,3-dichloro-, (E)- Formula: C3H4Cl2	110		112

	MW: 110			
26	1,1,2-Trichloroethane Name: Ethane, 1,1,2-trichloro- Formula: C2H3Cl3 MW: 132	132	In soluble in Water	113-114
27	Name: Toluene Formula: C7H8 MW: 92	92	0.067% very slightly sol. in water.	110.6
28	Name: Propane, 1,3-dichloro- Formula: C3H6Cl2 MW: 112	112		
29	Dibromochloromethane Name: Methane, dibromochloro- Formula: CHBr2Cl MW: 206	206		121.3-121.8
30	1,2 Dibromomethane Name: Ethane, 1,2-dibromo- Formula: C2H4Br2 MW: 186	186	11.93 g / 1000 g water	97
31	Tetrachloroethylene Name: Tetrachloroethylene Formula: C2Cl4 MW: 164	164	10000 vol Water	121
32	1,1,1,2-Tetrachloroethane Name: Ethane, 1,1,1,2-tetrachloro- Formula: C2H2Cl4 MW: 166	166	1 g in 350 ml water (sparingly sol. In water)	146.5
33	Chlorobenzene Name: Benzene, chloro- Formula: C6H5Cl MW: 112	112	Insol. In water.	131-132
34	Ethyl Benzene Name: Ethylbenzene Formula: C8H10 MW: 106	106	Practically Insoluble in water.	136.25
35	Bromoform Name: Methane, tribromo- Formula: CHBr3 MW: 250	250	sol. In about 800 parts water.	149-150
36	p-Xylene Name: p-Xylene Formula: C8H10 MW: 106	106	insol. In water.	137-138
37	M-Xylene Name: Benzene, 1,3-dimethyl- Formula: C8H10	106	insol. In water.	139.3
	MW: 106			

38	Styrene	104		
	Name: Styrene			
	Formula: C8H8			
	MW: 104			
39	Name: Ethane, 1,1,2,2-tetrachloro-	166	1 g / 350 ml water.	146.5
	Formula: C2H2Cl4		(sparingly soluble)	
	MW: 166			
40	0 - Xylene	106	insol. In water.	144
	Name: o-Xylene			
	Formula: C8H10			
	MW: 106			
41	1 2 3-Trichloropropane	146		
••	Name: Propane 1 2 3-trichloro-	110		
	Formula: C3H5Cl3			
42		120		452 452
42	Names Benzene (1 methylethyl)	120	insol. in water.	152-155
	Name: Benzene, (1-methylethyl)-			
	MW: 120			(5(0
43	Bromobenzene	156	0.045 g / 100 g water	156.2
	Name: Benzene, bromo-		Practically insoluble	
	Formula: C6H5Br		in water.	
	MW: 156			
44	n-Propylbenzene	120	0.06 g / L water	159.2
	Name: Benzene, propyl-		Very slightly sol.	
	Formula: C9H12		in water.	
	MW: 120			
45	2-chlorotoluene	126	Slighly sol. In water	158.97
	Name: Benzene, 1-chloro-2-methyl-			
	Formula: C7H7Cl			
	MW: 126			
46	4-chlorotoluene	126	Slightly sol.In water	161.75
	Name: Benzene, 1-chloro-4-methyl-			
	Formula: C7H7Cl			
	MW: 126			
47	1.3.5-Trimethylbenzene	120	BDI 2 g / 100g water	164.7
77	Name: Benzene 1 3 5-trimethyl-	120	Practically	101.7
	Formula: C9H12		Insoluble in water.	
	MW· 120			
48	Tert-butyl benzene	13/	Insoluble in water	168 5
70	Name: Benzene tert-butul-	154		100.5
	Formula: C10H14			
40		400	Due at the U	1(0.171
49	1,2,4 - Irimetnyibenzene	120	Practically	169-1/1
	Name: Benzene, 1,2,4-trimetnyl-		insoluble in water.	
	Formula: C9H12			
	MW: 120			
50	Sec-Butylbenzene	134	Insolu. In water.	173.5
	Name: Benzene, (1-methylpropyl)-			

	Formula: C10H14			
	MW: 134			
51	1,3-Dichlorobenzene Name: Benzene, 1,3-dichloro- Formula: C6H4Cl2	146	Practically insoluble in water.	173
52	1 4 Dichlorobonzono	146	Practically insoluble	17/ 12
52	Name: Benzene, 1,4-dichloro- Formula: C6H4Cl2 MW: 146	140	in water.	174.12
53	4-Isopropyltoluene Name: Benzene, 1-methyl- 4-(1-methylethyl)- Formula: C10H14 MW: 134	134	Practically insoluble in water.	175.14
54	1,2-Dichlorobenzene Name: Benzene, 1,2-dichloro- Formula: C6H4Cl2 MW: 146	146	Practically Insoluble in water.	180.5
55	Name: Benzene, butyl- Formula: C10H14 MW: 134	134	Insol. In water.	183.1
56	1,2-dibromo 3, chloropropane Name: Propane, 1,2-dibromo-3-chloro- Formula: C3H5Br2Cl MW: 234	234	Slightly sol. In water.	196
57	1,2,4-trichlorobenzene Name: Benzene, 1,2,4-trichloro- Formula: C6H3Cl3 MW: 180	180	insol. In water.	213
58	Naphthalene Name: Naphthalene Formula: C10H8 MW: 128	128	Insol. In water	217.9
59	Hexachlorobutadiene Name: 1,3-Butadiene, 1,1,2,3,4,4-hexachloro- Formula: C4Cl6 MW: 258	258		
60	1,2,3-trichlorobenzene Name: Benzene, 1,2,3-trichloro- Formula: C6H3Cl3; MW: 180	180	Insol. In water.	221

Annexure - III A



Annexure - III B

EVALUATION SCHEME FOR VOC MONITORING AND ANALYSIS



Annexure - III C

FLOW CHART OF METHOD OF ANALYSIS AS PER VOC METHOD TO-17




Annexure-VA

	VOC Sampling Details – LOTE INDUSTRIAL ESTATE, MAHARASHTRA (Round-I)					
S.No	Date of Sampling	Time of Sampling	Sampling Location	Flow rate	Sampling Duration	Volume of air Sampled
		Am	bient Air Quality Monitoring – 1 st Cycle			
1	28/12/2010	13.00 - 21.00 Hrs	Telecom Exchange – Ratnagiri	500ml/min	8 hours	240Litres
2	28/12/2010	13.50 - 21.50 Hrs	Ganesh Ice Factory, GIDC – B 69/2	500ml/min	8 hours	240Litres
3	28/12/2010	15.30 - 23.30 Hrs	Aimco Pesticides, GIDC – B1/1	500ml/min	8 hours	240Litres
4	28/12/2010	16.30 - 00.30 Hrs	Nerolac Paints, MIDC, F-3 – Top of Admn. Building	500ml/min	8 hours	240Litres
5	28/12/2010	17.45 - 01.45 Hrs	CETP – Opp to Admn. Building	500ml/min	8 hours	240Litres
Ambient Air Quality Monitoring – 2 nd Cycle						
1	28/12/2010 – 29/12	21.10 - 05.10 Hrs	Telecom Exchange – Ratnagiri	500ml/min	8 hours	240Litres
2	28/12/2010 – 29/12	22.00 - 06.00 Hrs	Ganesh Ice Factory, GIDC – B 69/2	500ml/min	8 hours	240Litres
3	28/12/2010 – 29/12	23.40 - 07.40 Hrs	Aimco Pesticides, GIDC – B1/1	500ml/min	8 hours	240Litres
4	28/12/2010 – 29/12	00.40 - 08.40 Hrs	Nerolac Paints, MIDC, F-3 – Top of Admn. Building	500ml/min	8 hours	240Litres
5	28/12/2010 – 29/12	01.50 - 09.50 Hrs	CETP – Opp to Admn. Building	500ml/min	8 hours	240Litres
		Am	bient Air Quality Monitoring – 3 rd Cycle		· · · · · · · · · · · · · · · · · · ·	
1	29/12/2010	05.20 - 13.20 Hrs	Telecom Exchange – Ratnagiri	500ml/min	8 hours	240Litres
2	29/12/2010	06.10 - 14.10 Hrs	Ganesh Ice Factory, GIDC – B 69/2	500ml/min	8 hours	240Litres
3	29/12/2010	07.50 - 15.50 Hrs	Aimco Pesticides, GIDC – B1/1	500ml/min	8 hours	240Litres
4	29/12/2010	08.50 - 16.50 Hrs	Nerolac Paints, MIDC, F-3 – Top of Admn. Building	500ml/min	8 hours	240Litres
5	29/12/2010	10.00 - 18.00 Hrs	CETP – Opp to Admn. Building	500ml/min	8 hours	240Litres

Annexure-VB

	VOC Sampling Details – LOTE INDUSTRIAL ESTATE, MAHARASHTRA (Round-II)					
S.No	Date of Sampling	Time of Sampling	Sampling Location	Flow rate	Sampling Duration	Volume of air Sampled
		Am	bient Air Quality Monitoring – 1 st Cycle			
1	29/12/2010	16.45 - 00.45 Hrs	Telecom Exchange – Ratnagiri	500ml/min	8 hours	240Litres
2	29/12/2010	16.55 - 00.55 Hrs	Ganesh Ice Factory, GIDC – B 69/2	500ml/min	8 hours	240Litres
3	29/12/2010	17.00 - 01.00 Hrs	Aimco Pesticides, GIDC – B1/1	500ml/min	8 hours	240Litres
4	29/12/2010	17.20 - 01.20 Hrs	Nerolac Paints, MIDC, F-3 – Top of Admn. Building	500ml/min	8 hours	240Litres
5	29/12/2010	17.30 - 01.30 Hrs	CETP – Opp to Admn. Building	500ml/min	8 hours	240Litres
Ambient Air Quality Monitoring – 2 nd Cycle						
1	30/12/2010	00.50 - 08.50 Hrs	Telecom Exchange – Ratnagiri	500ml/min	8 hours	240Litres
2	30/12/2010	01.00 - 09.00 Hrs	Ganesh Ice Factory, GIDC – B 69/2	500ml/min	8 hours	240Litres
3	30/12/2010	01.05 - 09.05 Hrs	Aimco Pesticides, GIDC – B1/1	500ml/min	8 hours	240Litres
4	30/12/2010	01.25 - 09.25 Hrs	Nerolac Paints, MIDC, F-3 – Top of Admn. Building	500ml/min	8 hours	240Litres
5	30/12/2010	01.35 - 09.35 Hrs	CETP – Opp to Admn. Building	500ml/min	8 hours	240Litres
		Aml	bient Air Quality Monitoring – 3 rd Cycle			
1	30/12/2010	08.55 - 16.55 Hrs	Telecom Exchange – Ratnagiri	500ml/min	8 hours	240Litres
2	30/12/2010	09.05 - 17.05 Hrs	Ganesh Ice Factory, GIDC – B 69/2	500ml/min	8 hours	240Litres
3	30/12/2010	09.10 - 17.10 Hrs	Aimco Pesticides, GIDC – B1/1	500ml/min	8 hours	240Litres
4	30/12/2010	09.30 - 17.30 Hrs	Nerolac Paints, MIDC, F-3 – Top of Admn. Building	500ml/min	8 hours	240Litres
5	30/12/2010	09.40 - 17.40 Hrs	CETP – Opp to Admn. Building	500ml/min	8 hours	240Litres

Annexure - VI

<u>METHOD OF ANALYSIS</u> <u>Ambient Air Quality & Fugitive Emission Samples</u> <u>Method - EPA TO - 17</u> <u>Method Validation Document</u>

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1.0 Purpose

To validate the GC-MS(P&T) and GC-MS-ATD method for the determination of VOC's. in Air Samples.

2.0 Scope

This protocol provides guidance to validate the method of analysis to evaluate its reliability and suitability for its intended purpose.

3.0 Terms and abbreviations

The following table lists terms and abbreviations and their descriptions which are used in the protocol.

Terms	Abbreviations
GC-MS	Gas Chromatography with Mass Spectrometer
RSD	Relative Standard Deviation
ATD	Automated Thermal desorber
P&T	Purge and Trap
LOQ	Limit of quantification

4.0 Validation Program and Acceptance Criteria

Validation parameters: Following performance parameters will be assessed during validation.

- Selectivity/Specificity
- Sensitivity (LOQ)
- Linearity
- Precision & Accuracy
- Recovery
- Measurement of Uncertainty

4.1 Selectivity/Specificity

The selectivity/specificity of the present method will be established by checking the reagent blank and matrix blank (without spiking VOC Standard) and comparing the interference at the retention time or mass transition (m/z) of analytes against solvent LOQ.

Acceptance Criteria:

If the reagent blank and Matrix blank contains significant interference at the retention time or mass transition (m/z) of analytes in question, that will be rejected. (Criteria \leq 30% of LOQ).

4.2 Sensitivity

Sensitivity of the method will be expressed as limit of quantification.

Acceptance Criteria:

Lowest level for which it has been demonstrated that criteria for accuracy and precision have been met .

4.3 Recovery

Recovery of an analyte in an assay is the instrument responses obtained from the amount of analyte added to and recovered from sample matrix, compared to detector response obtained from pure authentic reference standard. The percentage recoveries are determined by measuring the peak area of recovery samples at concentrations of LOQ. Comparing with the responses of the un-extracted solvent standards.

Acceptance criteria:

Accuracy should be 70 to 120% at LOQ level (As per the FDA Guidelines)

CV % of the recovery of analyte at each concentration level shall be ≤ 20 .

If the above criteria are not met, repeat the recovery experiment.

4.4 Linearity

Determination of the relationship between the observed signal (response produced by the detection system) from the target analyte in the sample extract and known quantities of the analyte prepared as standard solutions is called as calibration curve. The no. of standards used in constructing a calibration curve will be a function of the anticipated range of analytical values. Concentrations of standards should be chosen on the basis of the concentration range expected in the samples.

A calibration curve consists of reagent and matrix blank and 7 non-zero calibration standards covering the expected range (coded as CC1 (LLOQ) through to CC7 (~20xLOQ)). A linear equation will be determined to produce the best fit for the concentration /response relationship.

Acceptance criteria

Maximum allowed deviation of the CC1 from actual concentration shall be $\pm 20\%$ and standards other than CC1 from actual concentration shall be $\pm 15\%$

'r' shall be ≥ 0.99 (or) 'r2' shall be ≥ 0.98

At least 3 out of 7 non-zero standards shall meet the above criteria, including the CC1 and the calibration standards at the highest concentration (CC7). Any two consecutive points shall not be excluded. (As per FDA Guidelines)

4.5 Precision and Accuracy

Precision:

Under each calibration curve analyze 6 sets of each of LOQ,~3Xloq and ~5xLOQ samples.

The precision of the method is represented by % Coefficient of variation of analyzed samples under recovery experiment and calculated as follows.

CV % = (SD/Mean) x 100,

Where CV% = coefficient of variation and SD = standard deviation

Acceptance criteria:

The precision denoted by CV % at each concentration level should be $\leq 15\%$ expect for LLOQ where it should not be $\leq 20\%$.

Accuracy:

The accuracy of method is represented by % recovery of the analytes and will be calculated as follows:

% Accuracy = (Mean of obtained concentrations /Actual concentration) x 100

Acceptance criteria:

The % accuracy obtained should be within 70-120%

4.6 Measurement of Uncertainty :

A range around the reported result within which the true value can be expected to lie with a specified probability (confidence level, usually 95%). Uncertainty data should encompass trueness (bias) and reproducibility

Measurand : Determination of Pesticide Residues in Grape Fruit

- Instrument used:
- Balance ID :
- Volumetric flask ID :.....
- Method of testing: In house method
- Standard used: All neat standards procured from known source
- Micropipettes used:

- Sources of Uncertainty:
 - 1. Uncertainty due to Standard Preparation
 - a) Reference standard purity
 - b) Balance uncertainty
 - c) Glassware uncertainty
 - d) Micropipette uncertainty (10-100µL)
 - e) Micropipette uncertainty (100-1000µL)
 - 2. Repeatability uncertainty (Type-A): Sample preparation

Calculations:

As per EURACHEM/CITAC Guide CG4

5.0 Analytical Procedure GC-MS(P&T):

- 5.1 Instrument & equipment
 - GC –MS
 - Data system Chem Station
 - VRX, length 20m x ID 0.18mm, Film thickness 1.0µm
 - Micropipette
 - Analytical Balance

5.2 Reagents

- Methanol AR grade
- Reagent Water Milli-Q water (with reference to the specification of Water for Laboratory ISO3696:1987 Electrical conductivity and TOC are within the specifications)

5.3 Standards

Reference standards procure from Sharalu Chem or Equivalent

5.4 Preparation of Standards for calibration curve:

Standards for calibration curve were prepared from stock of 2000µg/mL (AccuStandard)

Conc. Of Stock	Volume of	Volume of	Made up	Final Conc.
Solution (mg/L)	Stock (mL)	Diluent* (mL)	Volume (mL)	(mg/L)
2000	1	9	10	200
200	2.5	7.5	10	50
200	1.25	8.75	10	25
50	4	6	10	20
20	5	5	10	10
10	5	5	10	5
10	1	9	1	1

Prepration of Stock Solutions

<u>Preparation of pesticide residues for calibration curves</u>: Prepare CC1 toCC7 daily ranging from 1 to 20 ng/mL with Water.

Conc. Of Stock Solution (µg/mL	Volume of Stock (µL)	Made up Volume (mL)	Final Conc(µg/L	Lable
20	43	43	20	CC7
15	43	43	15	CC6
20	43	43	10	CC5
10	43	43	8	CC4
5	43	43	5	CC3
1	43	43	1	CC2
0.1	43	43	0.1	CC1

5.5 **Preparation of Stock Solution other than 60compounds:**

Preparation of stock dilutions of mixed standard solution.10,000 ppm mix standard prepared based on the specific gravity (g/cc) mentioned on the solvent bottles.

For eg : Acetone 1L - 0.79Kg

1000ml - 790gm

10ml - 7.9gm

? - 1gm

1.2658ml for 1gm $\Rightarrow 1265.82$ µL for 1 gm

1265.8µL for 1000mg For 10mg = 12.65μ L \Rightarrow 1000ppm

126.5µL for 10,000ppm

Similarly all the remaining solvents were calculated and mix of 10,000ppm was prepared.by adding DCM-75.75 μ L(1L - 1.32Kg), Chloroform- 67.1141 μ L(1L - 1.49Kg), Ethyl acetate – 111.23 μ L, Tetrahydrofuran-112.36 μ L.(1L - 0.89Kg) in a 10ml volumetric flask.

6.0 Analytical Procedure GC-MS(ATD)

6.1 Instrument & equipment

- GC –MS
- Data system Chem Station
- ATD
- Data system Unity
- VF-624, length 60m x ID 0.25mm, Film thickness 1.4µm
- Micropipettes
- Analytical Balance

6.2 Preperation of Calibratin Curve Standards

For	TDS
-----	-----

Conc. Of Stock	Volume of	Volume of	Made up	Final Conc.
Solution (mg/L)	Stock (mL)	Diluent* (mL)	Volume (mL)	(mg/L)
2000	1	9	10	200
200	5	5	10	100
100	5	5	10	50
50	5	5	10	25
100	1	9	10	10
10	5	5	10	5
10	1	9	10	1

Depending on this relation for getting 5ng concentration inject 5µL of 1ppm std.

Similarly			
5ppm	-	5µL	- 5ng
10ppm	-	5µL	- 50ng
25ppm	-	5µL	- 125ng
50ppm	-	5µL	- 250ng
100ppm	-	5µL	- 500ng
200ppm	-	5µL	- 1000ng

6.3 Sample Preservation

On receipt of samples, store them at about 4°C.

6.4 Sample preparation

Before preparing the sample allow the charcoal tube to get room temperature, then break the charcoal tube just above the glass wool plug. Carefully transfer the charcoal into a vial, add 5ml of chromatographic pure methanol, cap the container or vial tightly such that no volatilization occur, sonicate it for 30sec settle for 20-30min.

Then take 43μ L of supernatant methanol into a VOC vial containing 43ml of Milli-Q water, swirl 2-3 times and place the vial in auto sampler of purge and trap system. Same manner do the blank without charcoal.

6.5 Analytes :

Dichlorodifluoromethane, Chloromethane, Vinylchloride, Bromomethane, Ethylchloride, Trichloromonofluromethane, 1,1-Dichloroethane, Methylenechloride, trans-1,2dichloroethane. 1-1.dichloroethane. cis-1,2-dichloroethene, Bromochloromethane, Chloroform, 2,2-dichloropropane, 1,2-dichloroethane, 1,1,1-trichloroethane, 1.1dichloropropene, carbontetrachloride, Benzene, Dibrmomethane, 1,2-dichloropropane, Trichloroethylene, Bromodichloromethane, 1,3-dichloropropene, trans-1,3dichloropropene, 1, 1, 2-trichloroethane, Toluene, dichlorpropane, Dibromochlormethane, 1,2-dibromoethane, Tetrachloroethylene, 1,1,1,2-tetrachloroethane, Chlorobenzene, ethylbenzene, Bromoform, m&p-xylene, Styrene, 1,1,2,2-tetrachloroethane, o-xylene, dichloropropane, isopropylbenzene, bromobenzene, n-propylbenzene, 2chlorotoluene, chlorotoluene, 1,3,5trimethylbenzene, tert-butylbenzene, 1,2,4trimethylbenzene, sec-butlbenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, 4isopropyltoluene, 1,2-dichlorobenzene, butylbenzene, 1,2-dibromo-3,-chloropropane, 1,2,4-trichlorobenzene, Naphthalene, Hexachlorobutadiene, 1,2,3-trichlorobenzene.

6.6 Details of GC/MS Method :

Instrument Details: Model Used:- 6890 GC-MSD (Make:- Agilent Technologies Ltd.)

GC conditions:-

Oven Initial Temperature Initial Time Ramps:		: 40 °C (On) : 3.00 Min.	Maximum Equilibriu	Temp m Time	: 260°C : 0.50 min.
Rate Final	Temp.	Final Time			
1 10 2 25 3 0.0 (c	100 225 off)	0.00 3.00			
Post Temp Post Time Run Time	: 0 °C : 0.00 : 17.00	min.) min.			
Oven Initial Tempe Initial Time Ramps:	erature	: 40 °C (On) : 3.00 Min.	Maximum Temp Equilibrium Time	: 300 : 0.50	°C) min.
Rate Final 1 8.00 2 6.00 3 0.0 (c)	Temp. 90 240 off)	Final Time 2.00 3.00			
Post Temp Post Time Run Time	: 0 °C : 0.00 : 39.2:	min. 5 min.			
Front Inlet (Split / S	Split less)			
Mode Inlet Temp Pressure Split Split flow	: Split : 250 ° : 23.20 : 50:1 : 49.6	°C (On) 0 psi (On) mL / min.			

Total flow	: 53.1 mL / min.
Gas saver	: ON
Gas Type	: Helium

Column 1

Column 2

Capillary Column Model Number: J&W US5270225H DB-VRX,20m x 0.18mm x 1µm Intial flow : 1.0mL/min Inlet : Front Inlet Outlet : MSD Outlet pressure : vacuum

Model No.: J&W US5215021H DB-624,60m x 0.25mm x 1.4µm Intial flow : 1.5mL/min Inlet : Back Outlet : MSD Outlet pressure : Vaccum

Thermal Aux 2 (Transfer line)

Use	: MSD Transfer Line Heater
Initial temp	: 260 °C
Initial time	: 0.00 min.

MS Acquisition Parameters

General Information

Tune File	: atune.u
Acquisition Mode	: Scan

	Quantification
Name	ion
Dichlorodifluoromethane	85
Chloromethane	50
Vinyl chloride	62
Bromomethane	94
Ethylchloride	64
Trichloromonofluoromethane	101
1,1-dichloroethene	96
Methylene chloride	84
Trans-1,2-Dichloroethene	96
1,1-dichloroethane	63
Cis-1,2-Dichloroethene	96
Bromochloromethane	128
Chloroform	83
2,2-dichloropropane	77
1,2-dichloroethane	62
1,1,1-Trichloroethane	97
1,1-dichloropropene	75

Carbon Tetrachloride	117
Benzene	78
Dibromomethane	93
1,2-dichloropropane	63
Trichloroethylene	95
Bromodichloromethane	83
1,3-dichloropropene	75
Trans-1,3-dichloropropene	75
1,1,2-Trichloroethane	83
Toluene	91
1,3-Dichloropropane	76
Dibromochloromethane	129
1,2-Dibromoethane	107
Tetrachloroethylene	166
1,1,1,2-Tetrachloroethane	131
Chlorobenzene	112
Ethylbenzene	91
Bromoform	173
m & p - Xylene	106
Styrene	104
1,1,2,2-Tetrachloroethane	83
O-Xylene	106
1,2,3-Trichloropropane	75
Isopropyl benzene	105
Bromobenzene	156
n-Propylbenzene	91
2-Chlorotoluene	91
4-Chlorotoluene	91
1,3,5-trimethylbenzene	105
Tert-Butylbenzene	119
1,2,4-trimethylbenzene	105
Sec-Butylbenzene	105
1,3-dichlorobenzene	146
1,4-dichlorobenzene	146
4-isopropyltoluene	119
1,2-dichlorobenzene	146
Butylbenzene	91
1,2-dibromo-3,-	
chloropropane	75
1,2,4-trichlorobenzene	118
Naphthalene	128
Hexachlorobutadiene	225
1,2,3-trichlorobenzene	180

Variable	Value	Variable	Value
Valve Oven Temp.	150°C	Dry Purge Temp.	40°C
Transfer Line Temp.	150°C	Dry Purge Flow	200 mL / min.
Sample Mount Temp.	90°C	GC Start	Start of Desorb
Purge Ready Temp.	45°C	Desorb Preheat Temp.	245°C
Dry Flow Standby Temp.	175°C	Desorb Drain	On
Standby Flow	10 mL / min.	Desorb Time	1.00 min.
Pressurize Time	0.25 min.	Desorb Temp.	250°C
Fill I.S. Time	0.00 min.	Desorb Flow	200 mL / min.
Sample Transfer Time	0.25 min.	Bake Rinse	On
Pre-purge Time	0.00 min.	Number of Bake Rinses	3
Pre-Purge Flow	40 mL / min.	Bake Drain Time	0.50 min.
Sample Heater	Off	Bake Drain Flow	400 mL / min.
Sample Preheat Time	1.00 min.	Bake Time	3.00 min.
Preheat Temp.	40°C	Bake Temp.	270°C
Purge Time.	11.00 min.	Dry Flow Bake Temp.	175°C
Purge Temp.	0°C	Bake Flow	400 mL / min.
Purge Flow	40 mL/min.	Focus Temp.	-150°C
Purge Rinse Time	0.25 min.	Inject Time	1 min.
Purge Line Time	0.25 min.	Inject Temp.	180°C
Dry Purge Time	0.00 min.	Standby Temp.	100°C

Purge a	& Tra	p Conditions	- Velocity	V XPT	(with AC	DUATEK 70) method
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Unity Thermal desorber Conditions :

Desorption temp :	275°C
Desorption time :	5 min
Cold trap packing :	Tennax TA
Cold trap focusing temp :	-10°C
Cold trap (secondary) desorption temp :	300°C
Secondary desorption time	3 min
Flow path temp :	120°C
Desorb flow :	4ml/min
Inlet split :	52ml/min
Outlet split :	50ml/min

7.0 Processing & Calculations

The chromatograms have to be acquired using the computer based chem. Station software. The concentration of the unknown has to be calculated from the equation using regression anaaysis of the reciprocal of the pesticide residue concentration as weighing factor (1/x)

Y = mx+c Where y = analyteareas x = concentration of analyte m = slope of the calibration curve c = y-axix intercept value

Inject and analyze the verification Standard solution.

Calculate the concentrations as follows:

Conc. (mg/100mL) = working Std. Soln. Conc. (in mg/100mL) x analyte peak area Verification std. soln. peak area

Analyze the working standard solution after every 15 samples, and at the end of the sample set.

8.0 Reference:

- References :
- 1. ASTM D- 3687-01 (Standard practice for Analysis of Organic Compound Vapours Collected by the Activated Charcoal Tube Adsorption Method)
- 2.US-EPA 502.2,524.2 ,8260B, (Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS))
- EURACHEM/CITAC Guide CG4

ANNEXURE - VII

<u>METHOD OF ANALYSIS</u> <u>Wastewater samples</u> <u>Method - EPA 5035A</u>

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- 5.3 Standards
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- 5.6 Sample Preservation
- 5.7 Sample Preparation
- 5.8 Analytes
- 5.9 Chromatographic Conditions of GC-MS/MS
- 6 Processing & Calculations
- 7 Reference

1.0 Purpose

To validate the GC-MS(P&T) method for the determination of VOC's. in Water and Effluent

2.0 Scope

This protocol provides guidance to validate the method of analysis to evaluate its reliability and suitability for its intended purpose.

3.0 Terms and abbreviations

The following table lists terms and abbreviations and their descriptions which are used in the protocol.

Terms	Abbreviations
GC-MS	Gas Chromatography with Mass Spectrometer
RSD	Relative Standard Deviation
LOQ	Limit of quantification

4.0 Validation Program and Acceptance Criteria

Validation parameters: Following performance parameters will be assessed during validation.

- Selectivity/Specificity
- Sensitivity (LOQ)
- Linearity
- Precision & Accuracy
- Recovery
- Measurement of Uncertainty

4.1 Selectivity/Specificity

The selectivity/specificity of the present method will be established by checking the reagent blank and matrix blank (without spiking VOC Standard) and comparing the interference at the retention time or mass transition (m/z) of analytes against solvent LOQ.

Acceptance Criteria:

If the reagent blank and Matrix blank contains significant interference at the retention time or mass transition (m/z) of analytes in question, that will be rejected. (Criteria $\leq 30\%$ of LOQ).

4.2 Sensitivity

Sensitivity of the method will be expressed as limit of quantification.

Acceptance Criteria:

Lowest level for which it has been demonstrated that criteria for accuracy and precision have been met $% \left({{\mathbf{r}}_{i}} \right)$.

4.3 Recovery

Recovery of an analyte in an assay is the instrument responses obtained from the amount of analyte added to and recovered from sample matrix, compared to detector response obtained from pure authentic reference standard. The percentage recoveries are determined by measuring the peak area of recovery samples at concentrations of LOQ. Comparing with the responses of the unextracted solvent standards.

Acceptance criteria:

Accuracy should be 70 to 120% at LOQ level (As per the FDA Guidelines)

CV % of the recovery of analyte at each concentration level shall be \leq 20.

If the above criteria are not met, repeat the recovery experiment.

4.4 Linearity

Determination of the relationship between the observed signal (response produced by the detection system) from the target analyte in the sample extract and known quantities of the analyte prepared as standard solutions is called as calibration curve. The no. of standards used in constructing a calibration curve will be a function of the anticipated range of analytical values. Concentrations of standards should be chosen on the basis of the concentration range expected in the samples.

A calibration curve consists of reagent and matrix blank and 7 non-zero calibration standards covering the expected range (coded as CC1 (LLOQ) through to CC7 (~20xLOQ)). A linear equation will be determined to produce the best fit for the concentration /response relationship.

Acceptance criteria

Maximum allowed deviation of the CC1 from actual concentration shall be $\pm 20\%$ and standards other than CC1 from actual concentration shall be $\pm 15\%$

'r' shall be ≥ 0.99 (or) 'r2' shall be ≥ 0.98

At least 3 out of 7 non-zero standards shall meet the above criteria, including the CC1 and the calibration standards at the highest concentration (CC7). Any two consecutive points shall not be excluded. (As per FDA Guidelines)

4.5 Precision and Accuracy

Precision:

Under each calibration curve analyze 6 sets of each of LOQ,~3Xloq and ~5xLOQ samples.

The precision of the method is represented by % Coefficient of variation of analyzed samples under recovery experiment and calculated as follows.

CV % = (SD/Mean) x 100,

Where CV% = coefficient of variation and SD = standard deviation

Acceptance criteria:

The precision denoted by CV % at each concentration level should be \leq 15% expect for LLOQ where it should not be \leq 20%.

Accuracy:

The accuracy of method is represented by % recovery of the analytes and will be calculated as follows:

% Accuracy = (Mean of obtained concentrations /Actual concentration) x 100

Acceptance criteria:

The % accuracy obtained should be within 70-120%

4.6 Measurement of Uncertainty :

A range around the reported result within which the true value can be expected to lie with a specified probability (confidence level, usually 95%). Uncertainty data should encompass trueness (bias) and reproducibility Measurand : Determination of Pesticide Residues in Grape Fruit

- Instrument used:
- Balance ID :
- Volumetric flask ID :.....
- Method of testing: In house method
- Standard used: All neat standards procured from known source
- Micropipettes used:
- Sources of Uncertainty:
 - 1. Uncertainty due to Standard Preparation
 - f) Reference standard purity
 - g) Balance uncertainty
 - h) Glassware uncertainty
 - i) Micropipette uncertainty (10-100µL)
 - j) Micropipette uncertainty (100-1000µL)
 - 2. Repeatability uncertainty (Type-A): Sample preparation

Calculations:

As per EURACHEM/CITAC Guide CG4

5.0 Analytical Procedure GC-MS(P&T):

- 5.1 Instrument & equipment
 - GC -MS
 - Data system Chem Station
 - VRX, length 20m x ID 0.18mm, Film thickness 1.0µm
 - Micropipette
 - Analytical Balance

5.2 Reagents

• Methanol - AR grade

Reagent Water - Milli-Q water (with reference to the specification of Water for Laboratory ISO3696:1987 Electrical conductivity and TOC are within the specifications)

5.3 Standards

Reference standards procure from Sharalu Chem or Equivalent

5.4 Preparation of Standards for calibration curve: Standards for calibration curve were prepared from stock of 2000µg/mL (AccuStandard)

Conc. Of Stock	Volume of	Volume of	Made up	Final Conc.
Solution (mg/L)	Stock (mL)	Diluent* (mL)	Volume (mL)	(mg/L)
2000	1	9	10	200
200	2.5	7.5	10	50
200	1.25	8.75	10	25
50	4	6	10	20
20	5	5	10	10
10	5	5	10	5
10	1	9	1	1

Prepration of Stock Solutions

<u>Preparation of pesticide residues for calibration curves</u>: Prepare CC1 toCC7 daily ranging from 1 to 20 ng/mL with Water.

Conc. Of Stock	Volume of	Made up	Final	Lable
Solution (µg/mi	Stock (μL)	volume (mL)	Conc(µg/L	
20	43	43	20	C
15	43	43	15	C
20	43	43	10	C
10	43	43	8	C
5	43	43	5	C
1	43	43	1	C
0.1	43	43	0.1	C

5.6 Sample Preservation

On receipt of samples, store them at about 4° C.

5.7 Sample preparation

Sampling:

Reservoir water samples for all analyses were collected using a 1.2-L, Teflon Kemmerer sampler samples collected by submerging the sample bottle, removing the cap, filling the bottle, and recapping the bottle while still submerged.

The finished (treated) drinking-water samples and imported raw-water samples were collected from spigots located at the distribution points. The water lines were flushed for 5 minutes before the sample bottles were filled

5.8 Effluent and water samples: load the effluent sample directly, in case it is turbid or oily dilute the sample with organic free water. Water samples can be loaded directly in to the purge and trap system.

5.8 Analytes :

Chloromethane, Dichlorodifluoromethane, Vinylchloride, Bromomethane, Trichloromonofluromethane, 1.1-Dichloroethane. Ethylchloride, Methylenechloride, trans-1,2-dichloroethane,, 1-1,dichloroethane, cis-1,2dichloroethene, Bromochloromethane, Chloroform, 2,2-dichloropropane, 1,2dichloroethane, 1,1,1-trichloroethane, 1,1-dichloropropene, carbontetrachloride, Benzene. Dibrmomethane, 1,2-dichloropropane, Trichloroethylene, Bromodichloromethane, 1,3-dichloropropene, trans-1,3dichloropropene, 1,1,2-trichloroethane, Toluene. dichlorpropane, Dibromochlormethane, 1,2-dibromoethane, Tetrachloroethylene, 1,1,1,2tetrachloroethane, Chlorobenzene, ethylbenzene, Bromoform, m&p-xylene, Styrene, 1,1,2,2-tetrachloroethane, o-xylene, dichloropropane, isopropylbenzene, bromobenzene, npropylbenzene, 2-chlorotoluene, chlorotoluene, 1,3,5trimethylbenzene, tert-butylbenzene, 1,2,4-trimethylbenzene, sec-butlbenzene, 1.3dichlorobenzene, 1,4-dichlorobenzene, 4-isopropyltoluene, 1,2dichlorobenzene, butylbenzene, 1,2-dibromo-3,-chloropropane, 1,2,4-trichlorobenzene, Naphthalene, Hexachlorobutadiene, 1,2,3trichlorobenzene.

5.9 Details of GC/MS Method :

Instrument Details: Model Used:- 6890 GC-MSD (Make:- Agilent Technologies Ltd.)

GC conditions:-Oven Maximum Temp Initial Temperature : 40 °C (On) : 260°C fhitial Time : 3.00 Min. Equilibrium Time : 0.50 min. Ramps: Rate Final Temp. Final Time 1 10 100 0.00 2 225 3.00 25 3 0.0 (off) Post Temp : 0 °C Post Time : 0.00 min. Run Time : 17.00 min. Front Inlet (Split / Split less) Mode : Split Inlet Temp : 250 °C (On) : 23.20 psi (On) Pressure Split : 50:1 Split flow : 49.6 mL / min. Total flow : 53.1 mL / min. Gas saver : ON Gas Type : Helium Column 1 Capillary Column Model Number: J&W US5270225H DB-VRX,20m x 0.18mm x 1µm Intial flow : 1.0mL/min Inlet : Front Inlet Outlet : MSD Outlet pressure : vacuum Thermal Aux 2 (Transfer line) : MSD Transfer Line Heater Use Initial temp : 260 °C Initial time : 0.00 min. **MS** Acquisition Parameters General Information

Tune File: atune.uAcquisition Mode: Scan

	Quantification
Name	ion
Dichlorodifluoromethane	85
Chloromethane	50
Vinyl chloride	62
Bromomethane	94
Ethylchloride	64
Trichloromonofluoromethane	101
1,1-dichloroethene	96
Methylene chloride	84
Trans-1,2-Dichloroethene	96
1,1-dichloroethane	63
Cis-1,2-Dichloroethene	96
Bromochloromethane	128
Chloroform	83
2,2-dichloropropane	77
1,2-dichloroethane	62
1,1,1-Trichloroethane	97
1,1-dichloropropene	75
Carbon Tetrachloride	117
Benzene	78
Dibromomethane	93
1,2-dichloropropane	63
Trichloroethylene	95
Bromodichloromethane	83
1,3-dichloropropene	75
Trans-1,3-dichloropropene	75
1,1,2-Trichloroethane	83
Toluene	91
1,3-Dichloropropane	76
Dibromochloromethane	129
1,2-Dibromoethane	107
Tetrachloroethylene	166
1,1,1,2-Tetrachloroethane	131
Chlorobenzene	112
Ethylbenzene	91
Bromoform	173
m & p - Xylene	106
Styrene	104
1,1,2,2-Tetrachloroethane	83
0-Xylene	106

1,2,3-Trichloropropane	75
Isopropyl benzene	105
Bromobenzene	156
n-Propylbenzene	91
2-Chlorotoluene	91
4-Chlorotoluene	91
1,3,5-trimethylbenzene	105
Tert-Butylbenzene	119
1,2,4-trimethylbenzene	105
Sec-Butylbenzene	105
1,3-dichlorobenzene	146
1,4-dichlorobenzene	146
4-isopropyltoluene	119
1,2-dichlorobenzene	146
Butylbenzene	91
1,2-dibromo-3,-	
chloropropane	75
1,2,4-trichlorobenzene	118
Naphthalene	128
Hexachlorobutadiene	225
1,2,3-trichlorobenzene	180

Purge & Trap Conditions:-	Velocity	XPT	(with AC	UATEK 70) method
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<u></u>		<u> </u>	
Variable	Value	Variable	Value
Valve Oven Temp.	150°C	Dry Purge Temp.	40°C
Transfer Line Temp.	150°C	Dry Purge Flow	200 mL / min.
Sample Mount Temp.	90°C	GC Start	Start of Desorb
Purge Ready Temp.	45°C	Desorb Preheat Temp.	245°C
Dry Flow Standby Temp.	175°C	Desorb Drain	On
Standby Flow	10 mL / min.	Desorb Time	1.00 min.
Pressurize Time	0.25 min.	Desorb Temp.	250°C
Fill I.S. Time	0.00 min.	Desorb Flow	200 mL / min.
Sample Transfer Time	0.25 min.	Bake Rinse	On
Pre-purge Time	0.00 min.	Number of Bake Rinses	3
Pre-Purge Flow	40 mL / min.	Bake Drain Time	0.50 min.
Sample Heater	Off	Bake Drain Flow	400 mL / min.
Sample Preheat Time	1.00 min.	Bake Time	3.00 min.
Preheat Temp.	40°C	Bake Temp.	270°C
Purge Time.	11.00 min.	Dry Flow Bake Temp.	175°C
Purge Temp.	0°C	Bake Flow	400 mL / min.
Purge Flow	40 mL/min.	Focus Temp.	-150°C
Purge Rinse Time	0.25 min.	Inject Time	1 min.
Purge Line Time	0.25 min.	Inject Temp.	180°C
Dry Purge Time	0.00 min.	Standby Temp.	100°C

6.0 Processing & Calculations

The chromatograms have to be acquired using the computer based chem. Station software. The concentration of the unknown has to be calculated from the equation using regression anaaysis of the reciprocal of the pesticide residue concentration as weighing factor (1/x)

Y = mx+c Where y = analyteareas x = concentration of analyte m = slope of the calibration curve c = y-axix intercept value

Inject and analyze the verification Standard solution.

Calculate the concentrations as follows:

Conc. (mg/100mL) = working Std. Soln. Conc. (in mg/100mL) x analyte peak area

Verification std. soln. peak area

Analyze the working standard solution after every 15 samples, and at the end of the sample set.

7.0 Reference:

- References :
- 1.US-EPA 502.2,524.2 ,8260B, (Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS))
- 2. US-EPA 5035,5035A 5035C (Closed -System Purge and Trap and Extraction for Volatile Organics in soil and waste water)
- EURACHEM/CITAC Guide CG4

ANNEXURE - VIII

CHROMATORGRAMS



Annexure-IX

PROFILE OF THE CHEMICALS

Source: National Institute for Occupational Safety and Health (NIOSH) Pocket Guide to Chemical Hazards, U.S. Department of Health and Human Services, February 2004

Sr. No.	Name of Chemical	Odour	Symptoms	Target organs	Carcinogen
01	Hydrogen sulphide	Rotten eggs	Irritation of eyes, respiratory system, coma, convulsion, conjunctivitis, eye pain, tears to eyes, dizziness, headache, weakness and exhaustion, insomnia, gastrointestinal disturbance	Eyes, respiratory system, Central Nervous System	No
02	Disagreeable odour like garlic or rotten cabbage	Irritation eyes, skin respiratory system	Irritation eyes, skin, respiratory system; convulsion	Eyes, skin, respiratory system, central Nervous System, blood	No
03	Dimethyl Sulphide	NA	Irritation, eyes, skin, respiratory system	Eyes, skin, respiratory system, central nervous system	No
04	Ethanol	Characteristic suffocating odour	Irritation eyes, skin, nose, headache, drowsiness, weakness, exhaustion, cough, liver damage, anemia, reproductive effects.	Eyes, skin, respiratory system, central nervous system, liver, blood, reproductive system	No
05	Methylene Chloride	Faint sweet odour	Irritation eyes, skin, weakness, exhaustion, drowsiness, dizziness, numbness, tingle limbs, nausea [potential occupational carcinogen]	Eyes, respiratory system, cancer site: [in animals: lung, liver, salivary& mammary gland tumours]	Yes
06	Trichloroethane	Chloroform like odour	Irritation eyes, skin, nose, headache, drowsiness, weakness, exhaustion, cough, liver damage, anemia, reproductive effects.	Eyes, skin respiratory system, heart, liver, kidneys cancer site: [in animals: liver and kidney cancer]	Yes
07	Toluene	Sweet pungent benzene like odour	Irritation eyes, nose, weakness, and exhaustion, confusion, dizziness, headache, dilated pupils, tears to eyes, anxiety, muscle fatigue, insomnia dermatitis, liver injury, kidney damage	Eyes, skin, respiratory system, central nervous system liver and kidney	No
08	Dimethyl Disulphide	NA	Irritation eyes, skin, respiratory system	Eyes, skin, respiratory system, central nervous system blood	No
09	Acetone	Fragrant mint like odour	Irritation eyes, nose, throat, headache, dizziness, central nervous system depression, dermatitis	Eyes, skin, respiratory system, central nervous system	No
10	Isopropyl Alchohol	Odour of rubbing	Irritation eyes, nose, throat, drowsiness, dizziness, headache, dry cracking skin, alcohol	Eyes, skin, respiratory system	No
11	n-Hexane	Gasoline like odour	Irritation of eyes, nose, nausea, headache, peripheral neuropathy, numbness, extremities muscle weakness, dermatitis, dizziness, chemical pneumonia	Eyes, skin, respiratory system, central nervous system	No

Sr. No.	Name of Chemical	Odour	Symptoms	Target organs	Carcinogen
12	Chloroform	Pleasant odour	Irritation of eyes, skin, dizziness, mental dullness, nausea, confusion; headache, weakness, exhaustion, enlarged liver [potential carcinogen]	Liver, kidneys, heart, eyes, skin, central nervous system cancer site: [in animals; liver and kidney cancer]	Yes
13	Carbon Tetrachloride	Characteristic either like odour	Irritation of eyes, skin, CNS depression, nausea, vomiting, liver, kidney injury, drowsiness, dizziness	Eyes. Respiratory system, lungs, liver kinder, skin cancer site: [in animals: liver cancer]	Yes
14	Benzene	An aromatic odour	Irritation eyes, skin, skin, nose, dizziness, headache, nauseam exhaustion, bone marrow depression [potential occupational carcinogen]	Eyes, skin, respiratory system, blood, central nervous system bone marrow Cancer Site [leukaemia]	Yes
15	2-Butanone (Methyk Ethyl Ketone)	A moderately sharp, fragrant, mint- or acetone like odour	Irritation eyes, skin, nose, headache, dizziness, vomiting, dermatitis	Eyes, skin respiratory system blood, central nervous system	No
16	Carbon Disulphide	A sweet ether like odour	Dizziness, headache, poor sleep, weakness, exhaustion, anxiety, weight loss, gastritis, kidneys, liver injury, eyes burns, dermatitis, reproductive effects	Central nervous system, peripheral nervous system, cardiovascular system, eyes kidneys, liver, skin, reproductive system	No
17	Ethylbenzene	An aromatic odour	Irritation eyes, skin, mucous membrane, headache, coma.	Eyes, skin respiratory system, central nervous system	No
18	m,p Xylenes	An aromatic odour	Irritation eyes, skin, nose, throat, dizziness, excitement, drowsiness, incoordination, staggering, gait, nausea, vomiting, abdominal pain, dermatitis	Eyes, skin, respiratory system, central nervous system gastrointestinal tract, blood, liver, kidneys	No
19	Acetonitrile	An aromatic odour	Irritation nose, throat, nausea, vomiting, chest pain, weakness, exhaustion, convulsion, in animal: liver, kidneys damage	Respiratory system, cardiovascular system, central nervous system, liver, kindneys	No
20	Acrylomitrile	An unpleasant odour	Irritation eyes, skin, headache, sneezing, nausea, vomiting, weakness, exhaustion, dizziness, skin [potential occupational carcinogen]	Eyes, skin, cardiovascular system, liver, kidneys, central nervous system Cancer Site [brain tumours lung & bowel cancer]	Yes
21	1,2-Dichloroethane	Chloroform-like odour	Irritation eyes, central nervous system depression; nausea, vomiting, dermatitis; liver, kidneys, cardiovascular system damage [potential occupational carcinogen]	Eyes, skin, kidneys, liver, central nervous system, cardiovascular system Cancer Site [in animals: forestomach, mammary gland and circulatory system cancer]	Yes
22	Vinyl chloride	Pleasant odour at high concentration	Weakness, exhaustion, abdominal pain, gastrointestinal bleeding, enlarged liver [potential occupational carcinogen]	Liver, Central Nervous System, blood, respiratory system, lymphatic system	Yes
23	I,I Dichlorochane	Chloroform like odour	Irritation skin, central nervous system depression, liver, kidneys, lung damage	Skin, liver, kidneys, lungs, central nervous system	No

24	1,1,2-Trichlorothane	Sweet, chloroform like odour	Irritation eyes, nose, central nervous system depression, liver, kidney damage [potential occupational carcinogen]	Eyes, respiratory system, central nervous system liver, kidneys Cancer Site: [in animal liver cancer]	Yes
25	Clorobenzene	Almond like odour	Irritation eyes, skin, nose, drowsiness, incordination, central nervous system depression; in animals: liver, lung, kidney injury	Eyes, skin, respiratory system, central nervous system, blood.	No
26	o-Xylene	Aromatic odour	Irritation eyes, skin, nose, throat, dizziness, excitement, drowsiness, incordination, anorexia, nausea, vomiting, abdominal pain, dermatitis	Eyes, skin, respiratory system, central nervous system, gastrointestinal tract, blood, liver, kidneys	No
27	1,2,4- Trimethylbenzene		Irritation eyes, skin, nose, throat, respiratory system, bronchitis, headache, drowsiness, fatigue, dizziness, nausea, incordination, vomiting, confusion, chemical pneumonitis	Eyes, skin respiratory system, central nervous system, blood	No
28	Alpha-pinene	A characteristic odour	Irritation eyes, skin, nose, throat, headache, dizziness, convulsion, blood in the urine, kidney damage, abdominal pain, nausea	Eyes, skin respiratory system, central nervous system, kidneys	No
29	d-Limonene	Characteristic citrus odour	Irritation of eyes, nose, lungs, lightness of head, difficulty in breathing, skin irritation, liver injury, kidney damage	Eyes, skin, respiratory system, liver and kidney	No
30	1,3 Butadiene	Mild aromatic and gasoline like odour	Irritation eyes, nose, throat, drosiness, dizziness, reproductive damages, [potential occupational carcinogen]	Eyes, respiratory system, central nervous system, reproductive system Cancer Site [blood cancer]	Yes
31	Acrolin	A piercing disagreeable odour	Irritation eyes, skin, mucous membrane, chronic respiratory disease	Eyes, skin, respiratory system, heart	No
32	Methyl tert-Butyl Ether	NA	NA	NA	NA
33	Styrene	A sweet, floral odour	Irritation eyes, nose, respiratory system, headache, weakness, exhaustion, dissiness, confusion, drowsiness, unsteady gait, possible liver injury, reproductive effects	Eyes, skin, respiratory system, central nervous system, liver, reproductive system	No
34	Nonane	A gasoline like odour	Irritation eyes, skin, nose, throat, headache, drowsiness, dizziness, confusion, nausea, tremor	Eyes. Skin, respiratory system, central nervous	No system
35	Chloromethane	A faint sweet odour	Dizziness, nausea, vomiting, visual, disturbance, stagger, slurred speech, convulsion, coma, liver, kidney damage reproductive [potential occupational carcinogen]	Central nervous system, liver, kidneys, reproductive system Cancer Site [in animals; lungs, kidney & forestomach tumours]	No
36	N-Butyl Acetate	A mild turpentine like odour	Irritation eyes, skin, upper respiratory system, headache, drowsiness	Eyes, skin, respiratory system, central nervous system	No
37	Hexachlorobutadiene	A mild, turpentine like odour	In animals, irritation eyes, skin, respiratory system; kidney damage; [potential occupational carcinogen]	Eyes, skin, respiratory system, kidneys Cancer Site [in animals: kidney tumours]	Yes

38	Carbony Sulphide	NA	NA	NA	NA
39	Chloroethane	A pungent ether- like odour	Incordination abdominal cramps, cardiac arrest; liver, kidney damage	Liver, kidneys, respiratory system, cardiovascular system, central nervous system	No
40	Triclorofluoromethan e	Odourless liquid	Incordination, tremor, dermatitis, cardiac arrest	Skin, respiratory system, cardiovascular system	No
41	4-Methyl-2- Pentanone	A mild odour	Irritation eyes, skin, headache, drowsiness, dermatitis	Eyes, skin central nervous system	No
42	Cumene	A sharp, penentrating aromatic odour	Irritation eyes, skin, mucous membrane; dermatitis, headache, coma	Eyes, skin, respiratory system, central nervous system	No
43	1,3,5-Trimethyl benzene	A distinctive aromatic odour	Irritation eyes, skin, nose, throat, respiratory system, bronchitis, headache, drosiness, fatigue, dizziness, nausea, incordination, vomiting, confusion, chemical pneumonits	Eyes, skin, respiratory system, central nervous system, blood	No
44	Bromomethane	A chloroform like odour at high temperature	Irritation eyes, skin, respiratory system, central Nervous System depression; liver, kidney disease cardiac arrest [potential occupational carcinogen]	Eyes, skin, respiratory system, central nervous system, blood	No
45	Vinyl Acetate	A pleasant fruit odour	Irritation eyes, skin, nose, throat, hoarseness cough, loss of smell; eye burns, skin blisters	Eyes, skin respiratory system	No

ANNEXURE - X

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