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Analysis of Organic Contaminants from Silicon Wafer and Disk Surfaces by Thermal Desorption GC-MS

by

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ABSTRACT

Organic contaminants can affect semiconductor wafer processing including gate oxide integrity, polysilicon growth, deep ultraviolet photoresist line-width, and cleaning & etching steps. Organophosphates are known to counter dope silicon wafers.[1] Organic contaminants in disk drives can cause failures due to stiction or buildup on the heads.[2-6] Therefore, it is important to identify organic contaminants adsorbed on wafer or disk surfaces and find their sources so they can be either completely eliminated or at least controlled. Dynamic headspace TD-GC-MS (Thermal Desorption-Gas Chromatography-Mass Spectrometry) methods are very sensitive and can be used to identify organic contaminants on disks and wafers, in air, or outgassing from running drives or their individual components.

Keywords: Organic, contamination, silicon, wafer, disk drive, GC-MS, thermal desorption, dynamic headspace, gas chromatography, outgassing

1.0 INTRODUCTION

Surfaces on sensitive products can get contaminated during manufacture, assembly or use. Contamination by particles is routinely monitored on smooth surfaces by various techniques including light scattering. Non-particulate contaminants also can contribute to yield loss. Oily films or residues can be determined by NVR (non-volatile residue) or surface analysis methods, but exact identifications of complex mixtures of compounds can be difficult. Outgassing of materials and adsorption onto substrates, even at sub-monolayer levels, can also affect yields, reliability or properties of finished products. We have been studying both the effects and methods for analysis of outgassed organic compounds on disk drive, semiconductor, aerospace and optical applications. We will describe how thermal desorption GC-MS may be applied to assessing what compounds get into air, which come from outgassing of materials and how much adheres to the substrates. Examples of applications for the disk drive and semiconductor industries will be given.

For the semiconductor industry, we will describe an example of a fab with yield losses caused by organophosphates. The organophosphates were detected in air at sub-ppbv (parts per billion by volume) levels, and affected yields by adsorption onto silicon wafers. The source of the organophosphate was determined by TD-GC-MS outgassing methods.[1]

The disk drive industry is sensitive to airborne molecular contamination during manufacturing and in use.[2-6] Airborne contaminants in the cleanroom can condense onto disk surfaces before sputtering processes and cause delamination. Contaminants can adsorb onto carbon overcoats on disks to affect the fluorolube application and can also adsorb onto finished disks. During head manufacturing and drive assembly, organic contaminants can adsorb onto surfaces and affect adhesion or electrical conductivity. The assembled drive is a closed environment where molecules outgassing from internal components such as seals, adhesives or coatings can adsorb onto the disk, cause media corrosion, get picked up by the heads, and cause disk and head smears, stiction and fly height problems.[2-6] The net result can be read/write errors and partial or total disk drive failures. We will describe tests for the outgassing of integrated hard disk drives while running, outgassing of individual drive components, and assessing which compounds are detected by desorbing the disk itself.

While few standard test methods for organic outgassing exist, a variety of standards organizations are attempting to develop consensus test methods, and these efforts will be noted.

2.0 DESCRIPTIONS OF TEST METHODS (FIGURE 1)

2.1. Thermal desorption GC-MS analysis method. All analyses were performed using the same instrumentation. Perkin Elmer ATD-400 automated thermal desorbers were used to desorb 3.5" long x 0.25" OD stainless steel sampling tubes. The sampling tubes contained either a test polymer for in-instrument outgassing analysis or an adsorbent used to trap compounds collected using air sampling or off-line thermal desorption or outgassing methods. The sampling methods are described in sections 2.2 – 2.7 below. The ATD-400's were connected via a heated transfer line to GC-MS systems consisting of Hewlett Packard HP 5890 or 6890 GC's connected to HP 5970B or 5972 Quadrupole Mass Selective Detectors respectively.

2.2. Cleanroom Air sampling (Figure 1a). Air was pumped through sampling tubes containing multiple adsorbent beds that selectively trap hydrocarbons with between six and twenty-eight carbon atoms, and most other volatile organics within a similar boiling point or retention time range. The air was sampled for 6 hours, then the tube was capped and returned to the lab for analysis.

Each tube was analyzed using a PE ATD-400. An internal standard (deuterated toluene d_8, labeled ISTD in all figures) was added to every run to ensure the instrument was functioning correctly. On the PE ATD-400, the adsorbent tubes were purged with helium, leak tested and thermally desorbed into a packed cold trap where compounds were refocused to improve the chromatography. The cold trap was then heated to desorb organic compounds into the GC-MS system. Individual compounds were separated on a non-polar poly(dimethylsiloxane) column, then were ionized by electron impact (70eV) ionization and detected using a quadrapole mass spectrometer. Compounds were identified by comparison with a library of 275,000 reference spectra and proprietary libraries, or by analyst interpretations when the library searches were unsuccessful or unreasonable. Details of the GC-MS instrumentation and methods have been published elsewhere.[1,7] The same instrumentation was used for all of the GC-MS analyses described in this report. Quantitation was based on the TIC (total ion count) response factor of an n-decane external standard. For comparison of the boiling ranges of compounds detected, compounds were summed into 3 ranges based on comparison with the retention times of n-alkane external standards. The ranges were defined as low (C6-C10), medium (>C10-C20) and high (>C20 to C30 or above).

2.3. Wafer desorption (Figure 1b)
Full wafers that have been exposed to cleanroom air for 24 hours, or to other environments or processes, were broken using a scribe in a cleanroom. Pieces were then placed into an inert chamber and thermally desorbed under a nitrogen purge at 300°C for 1 hour. The desorbed compounds were collected onto a sampling tube and analyzed as described in 2.2 above.

2.4. Cleanroom materials outgassing method (in-instrument, 100°C screening test method, Figure 1c)
This outgassing test has been described in detail elsewhere.[7] Small samples (typically 0.2 grams) of cleanroom materials such as HEPA filter potting compounds, were added to a stainless steel tube. The samples were heated at 100°C for 30 minutes while continuously purging with dry helium. The outgassing compounds were trapped on a cold trap for refocusing. This cold trap was then thermally desorbed into the GC-MS for analysis as described in 2.2 above.

2.5. Full disk drive outgassing while running (Figure 1d)
Drive A was a > 1 year old 3.5" drive (with Al substrate) removed from a personal computer at our company due to failure.

Drive B was a new unused laptop drive with two glass platters that had been through reliability testing. The drive B disks were considerably smaller than for drive A above. This work was performed for a drive company that chose to remain anonymous, but did approve the publication of the results in writing.

Inlet and outlet holes were opened on opposite sides of the disk drives. A whole drive was placed into a stainless steel chamber. The chamber was purged with 1 slpm of dry nitrogen. An air pump was used to pull 100 mL/minute of the ambient nitrogen from the stainless steel chamber, into the drive, through the sampling tube where organic compounds outgassed from the drive are trapped. The air pump was outside of the stainless steel chamber. The total sampling period was 24 hours. The tube and the analysis methods were as described in 2.2 above. The running drive was kept in a room temperature ambient, but the internal drive temperatures were expected to increase due to use. During these tests, the disk was spinning, but the actuator was not cycled. Each of the drives tested had two disks. Similar test have been described in the literature.[6]

2.6. Disk drive component outgassing (Off-line outgassing method, Figure 1e)
After completing the whole running drive outgas tests described above, each drive was disassembled and various parts removed for outgas testing. Samples of each material to be tested were placed into inert chambers of approximate volume 40 mL. Gas was passed through the containers at 50 mL/minute for 4 hours while the chambers were heated in an 85°C heater block. The gas exiting the chambers was collected on sampling tubes. The tube analysis method was described in 2.2 above.

2.7. Media thermal desorption (Figure 1b)
Media were removed from the drives. Each drive had two platters, but only one disk from each drive was tested. The media were cut or broken into small pieces, e.g. by using cleaned and baked metals shears. The samples were placed into an inert chamber and thermally desorbed at 200°C for 1hr using 100 mL/minute nitrogen dynamic headspace conditions. The desorbed compounds were trapped on a sampling tube and analyzed as described in 2.2 above.

3.0. RESULTS

3.1. Air analysis in a semiconductor fab. The analysis of organic contaminants in cleanroom air using adsorbent-containing tubes can be extremely sensitive. Figure 2 shows a GC-MS analysis for organic compounds in a cleanroom that was undergoing yield losses due to organophosphate compounds.[1] A variety of compounds were detected including trace (0.07 ppbv, parts per billion by volume) of tris(chloropropyl) phosphate (TCPP, an organophosphate flame retardant). Some siloxanes and some compounds that may be used in lithography (PGMEA, diethylaminoethanol) were also detected.

3.2. Witness wafer analysis. The main compound found on the witness wafer exposed 24 hours to the same fab air was TCPP, a common flame retardant (Figure 3). The amount on the wafer corresponded to 3E13 P atoms/cm^2 or about 0.01 monolayer. The estimated detection limit for this analysis was about 1E12 P atoms/cm^2. Most of the lower boiling compounds found in air (3.1 above) did not adsorb onto the wafer. Some DOP was detected on the wafer, but not in the air.

3.3. In-instrument outgassing analysis of cleanroom components. Outgas testing of most cleanroom components did not find any TCPP. A sample of the HEPA filter potting compound that was used to seal the pleated edges of the media into the HEPA frame was outgassed at 100°C for ½ hour. The main compound detected outgassing from the potting compound was tris(chloropropyl) phosphate (Figure 4, 530 ppmw).

3.4. Running drive outgassing

3.4.1. PC Drive A. The chromatogram showing the compounds detected outgassing from drive A while running for 24 hours is shown in Figure 5. Compounds detected included phenol, alcohols, cresol, aldehydes, 2-ethylhexanoic acid, BHT, diethyl-, dibutyl- and dioctyl phthalates. Note that this drive had been used for over one year prior to failure.

3.4.2. Laptop drive B. This drive had been used for reliability testing and had not failed. Drive B was first outgassed for 24 hours at room temperature without being powered on. The outgassing results are not shown here. The drive was then powered on and the outgassing compounds were collected for 24 hr (Figure 6). Solvents, organic acids, epoxy byproducts & unknowns were found. Overall, the compounds outgassed were similar for the running drive vs. the static drive, except the total outgassing amount increased about 3 times. The increased outgassing may be due to the added heat and turbulence of the running drive.

3.5. Drive component outgassing

After the full drive outgassing tests, most of the drive components were removed and outgassed individually at 85°C for 30 minutes to assess which are the major contributors to the outgassing. Only components with the highest levels of outgassing are discussed.

3.5.1. For drive A, the component with the highest amount of total detectable outgassing compounds (35 µg) was the gasket used to seal the cover to the base plate. The gasket outgassed compounds including: phenol, alcohols, 2-ethyl hexanoic acid, benzothiazole, silicones, phthalates, 2,2-dimethoxy-1,2-diphenyl ethanone (sometimes called DMAP), and tris(chloropropyl) phosphate (TCPP), along with many other unresolved or unidentified compounds (Figure 7).

3.5.2. For drive B, the gasket outgassed a variety of compounds totaling 105 µg. The most abundant compounds were not identified and are probably polar, based on the broad peaks. They may be proprietary compounds. Organic acids, alcohol, phenols, BHT & 1-chloro-3-phenoxypropanol were identified (Figure 8).

3.5.3. For drive A, the preamp with attached adhesive showed the 2nd highest level of outgassing for drive B components (22 µg, Figure 9). Compounds outgassed included cyclic siloxanes, alcohols, ethyl hexanoic acid, benzophenone, tri(ethylene glycol) dimethacrylate, 2,2-dimethoxy-1,2-diphenyl ethanone (DMAP), & dibutyl phthalate. Dimethylformamide, sometimes used as a solvent, e.g. for epoxies, was detected. Small amounts of tributylamine were found.

3.5.4. For drive B, other components including sealants, preamps, and the recirculation air filter outgassed relatively little compared with the gasket above (Figure 10 shows adhesive from preamp).

3.6. Disk media analysis by thermal desorption

When the media were thermally desorbed at 200°C for 1 hour, only trace amounts of organic compounds were detected.

3.6.1. Disk media A (Figure 11, 6.8 μg total outgassed). Compounds detected included hydroxyethyl methacrylate (HEMA), triethylene glycol dimethacrylate (TRIGMA or TGDMA a common crosslinker in adhesives),[2] tributylamine which may be a catalyst or primer for adhesives, DMAP a photoinitiator, and DBP & DEHP (also called DOP) which are plasticizers.

3.6.2. Disk media B (Figure 12, 0.97 μg total). This media had less total outgassing than A above, but this laptop disk drive media had about 4 times less area than the media in A. Compounds detected outgassing included phenol, 2-ethylhexanol, tributylamine, antioxidants, organic esters, dibutyl- and dioctyl phthalates.

4.0 DISCUSSION

4.1 Semiconductor applications

4.1.1. Cleanroom air analysis. GC-MS analysis of the cleanroom air was able to detect sub-ppb levels of organic contaminants, and some sensitive substrates such as semiconductors can be affected by specific contaminants even at these low levels.[1] The same procedures can be used for cleanroom air in disk drive, optical, aerospace or other sensitive industries.

4.1.2. Witness wafers. Cleanroom air can also be sampled using witness wafers. Many of the compounds in the air do not adsorb and do not affect the wafer. The wafer sampling method is most sensitive to those high boiling compounds that tend to adhere and affect processing. It may not detect many lower boiling compounds. The same method can be applied to witness disks, or other witness surfaces. The detection limits are on the order of 25 ng per wafer, about 0.1 ng/cm^2, or <0.001 monolayer for any one compound on a 150-mm wafer. In the test here, the wafers were heated at 300°C to maximize desorption of high boiling compounds such as DOP. Use of temperatures significantly above 300°C can cause decomposition of many organic compounds.[8] ASTM (Am. Soc. for Testing & Materials, www.astm.org) committee F1 on electronics is drafting a standard test method for organic contamination on wafers by thermal desorption GC-MS methods.

4.1.3. The outgassing test readily identified the flame retardant TCPP in the HEPA filter potting compound as the source of the contamination. Flame retardants are often used at percent levels and may outgas for many years. The in-instrument outgassing screening methods provide a relatively simple method to rapidly screen for organic contaminants outgassing from cleanroom construction materials. By using elevated temperatures such as 100°C, for a short time such as ½ hour, many materials can be rapidly screened to assess the worst materials. The worst materials should be improved first. Once a database is developed, specification for specific materials can be made to reduce the chance that a new fab may be affected. The use of 100°C is a compromise. Higher temperature would be make the test more sensitive to outgassing of high boiling compounds, but many materials may start to melt or decompose at higher temperatures. Ideally, lower temperature mimicking the actual use conditions would be used to assess what will come out during actual use. However, larger samples and longer sample times are typically needed to have adequate sensitivity to see trace contaminants outgassing at low rates. This would require larger sample chambers and offline methods, which are more time consuming and complicated to perform. IEST (Institute of Environmental Sciences and Technology, Mt. Prospect IL. http://www.iest.org) working group 31 is drafting methods for the standardized organic outgassing analyses of cleanroom materials by thermal desorption GC-MS. Detection limits for this method are about 1 ppm by weight. We have not encountered any components with outgassing < 10 ppm at 100°C that have caused problems in a cleanroom due to outgassing. Problem materials generally have over 100 ppm total outgassing, but the impact is highly dependent on both the process technologies and the compounds outgassed.

4.1.4. For the semiconductor industry, phthalates, silicones, organophosphates and amides are known to cause wafer processing defects. Silicon wafers are especially susceptible to contamination by organophosphates since they can dope the silicon. In the example given, 0.07 ppbv of an organo phosphate in the cleanroom air deposited roughly 0.01 monolayer of P on the wafer surface, and this was enough to cause up to 15% yield losses at the fab. DOP was also detected on the wafer, but not in the air. Hence, we recommend the wafer sampling method for trace high boiling compounds in cleanroom air.

4.1.5. All three tests can be applied for QC or to baseline existing fabs and set specifications of maximum levels for specific contaminants. We recommend using the outgassing method to qualify incoming materials used to make the cleanroom, doing the witness wafers to test the quality of the cleanroom air for wafer production, and using the air sampling methods to detect solvents and compounds such as amides that may affect lithography processes.

4.2 Disk drive applications

IDEMA has published a contaminant catalogue that includes compounds that are known to cause disk drive failures. Some of the organic compounds known to cause failures include amines, amides, siloxanes, benzoic acid, organic acids and plasticizers such as phthalates.[5] Examples of all of these types of compounds were found either outgassing from the running

drive or the components. In addition, a variety of radical initiators, monomers, stabilizers, crosslinking agents, solvents, reaction by-products and catalysts were detected. Many of these compounds have been documented to either outgas from drives and components, or affect drive performance.[2-6]

4.2.1. Static vs. running drives. When new drive B was studied, the overall outgassing with the drive off (17 μg/day) increased to 56 μg/day with the drive running. The increase was presumably due to the heating and more dynamic airflow when the drive was spinning. Comparing the two drives while running, the outgassing compounds were very different in both ID and amounts. Drive outgassing is expected to vary significantly with time since manufacture and with use. In addition, each drive is expected to have very different compounds outgassing if different adhesives, seals and other components are used. Two different manufacturers made the two drives in this paper. Neither drive had C adsorbers.

4.2.2. Running drive A. For drive A, the phthalates and polymerization catalysts could affect the drive performance. The effects of the other contaminants at the levels detected are not known.

4.2.3. Running drive B. The organic acids could affect corrosion, and the 1-chloro-3-phenoxy-2-propanol could hydrolyze to form HCl, which could corrode the media or heads. This compound could come from an epoxy adhesive or coating. Ideally, all hydrolyzable chloride compounds would be kept out of disk drives. Other high boiling organic esters, possibly adipates, could affect the fluorolube or head buildup.

4.2.4. Comparison of running drives. The total amount of compounds detected for running drive A (2.2 μg/day) was much less than for drive B (56 μg/day). This may be in part due to A being an older drive that had much time to outgas during its lifetime. Outgassing clearly continues for a long time. Studies of the time dependent outgassing of running drives throughout their lifetime are needed. Most studies emphasize looking at the early life, but long-term outgassing of high boiling compounds may accumulate on the disk or heads very slowly over long periods.

4.2.5. The component outgassing experiments detected a variety of compounds, especially from the gaskets & the preamps or their adhesives. The drive A gasket outgassed compounds including benzothiazole, 2-ethylhexanoic acid, DMAP, TCPP & dibutyl phthalate. The benzothiazole has been documented to affect the tribology of disks when purposely spiked into running disk drives[4]. The chlorinated organophosphate TCPP might thermally decompose or hydrolyze to form HCl, which could cause corrosion. The total outgassed from gasket A at 85°C for 4 hours was 35 μg/whole gasket.

The drive B gasket outgassed BHT, an antioxidant, & other compounds. The main two compounds outgassed were not identified. The total outgassed from this gasket at 85°C for 4 hours was 105 μg, roughly an order of magnitude higher than gasket A above, despite drive B being much smaller. The preamp with adhesive A outgassed a variety of compounds including benzoic acid, silicones, phthalates, acrylates, photoinitiators & tributylamine (22 μg total). Gaskets are often a major contributor to outgassing of the drive, & different gaskets can have orders of magnitude variations in outgassing rates.[6]

4.2.6. Media desorption tests. The disks were heated to 200°C to drive off most of the volatile contaminants. Higher temperatures can be used to increase recovery of high boiling compounds such as DOP, but some outgassing of the fluorolube layer may also occur. The outgassed fluorolubricant can contaminate the instrument and cause carryover and recovery problems. DOP was found on the media for both drives, but was not detected outgassing from the running drives. Either the disks were contaminated prior to or during assembly, or the DOP was outgassed from the running drive at extremely low rates, yet concentrated over time onto the media to eventually integrate to detectable levels. The main compound detected from the drive A media was benzodioxole carboxaldehyde (also called piperonal). The source or function of this compound is unknown, but it has been reported to outgas from other drives in the literature.[6] It was not detected outgassing from the gasket, preamp, or other drive components tested. The presence of amines is a concern since they may react with acidic vapors to form hygroscopic salts that can aid corrosion and build up on heads. If alkylammonium salts are formed, they may not be recovered well by GC-MS. The majority of outgassed compounds do not end up on the media, but must go elsewhere or partially equilibrate throughout the drive. Neither of these drives had a C adsorber, which would be expected to adsorb many of the compounds detected.

4.3 Limitations of thermal desorption GC-MS test methods.

For the test methods described, the instrument sensitivity was not a limiting factor to the applications of these methods, with the exception of the disk media outgassing method. Overall, only small amounts of organics were thermally desorbed from the disk media. This may be due to the relatively smooth disk surface and the low affinity of organic compounds for the

fluorolube layer vs. the higher activity of other surfaces in the drives, making organic compounds less likely to equilibrate onto the disk. This hypothesis deserves more experimentation to assess what is on the disk vs. other surface of the drive, e.g. the base plate, etc. Outgassing multiple disks together when needed can increase the sensitivity.

Some of the lower-boiling compounds might not adhere to the disk alone, but when monomers and radical initiators co-condense onto the disk, they can polymerize to leave behind non-volatile contaminants. Any non-volatile contaminants would not be detected by GC-MS, and other methods would be needed for their detection or identification.

Some compounds are thermally unstable and can decompose prior to, or during analysis. Some low boiling compounds will not be trapped by these methods, but they usually have little effect on the products unless they are reactive.

For the outgassing of the disk drive components in used drives, some cross contamination between parts was possible. For this reason, it is important to test both materials before they go into the drive, after they go into the drive, and for failure analysis after use to get the clearest picture of the contaminant sources.

5.0 CONCLUSIONS

Combining all three types of methods: 1) air or gas sampling; 2) on-substrate analysis by thermal desorption; and 3) outgassing of components; can improve the ability to solve organic contamination problems.

Air sampling is useful especially for new fabs and for baselining existing fabs. The wafer sampling is very sensitive to high boiling compounds that may affect adhesion, conduction, or high temperature processing steps. If baseline airborne and witness substrate data for a running fab are collected, analysis during an upset can be used to set specifications for specific problem compounds.

Outgassing of running drives can be used to QC the whole drive to assess whether it meets a specification, either for the drive manufacturer or OEM user (i.e. laptop, PC or server manufacturers). The IDEMA (Intl Disk Drive Equipment & Materials Association, www.dema.org) microcontamination committee is developing standard dynamic headspace GC-MS methods for the outgassing of disk drive components. Outgassing of individual drive components can be used to QC incoming components for either vendors or drive companies, or for failure analysis to ID contaminant sources. Outgas testing of the media can assess which compounds are sticking to the media, which is especially useful for failure analysis or qualifying incoming disks. Witness disks can be used to monitor the media manufacturing environment to reduce delamination and fluorolubing problems, or to QC disks as they are received to ensure they did not get contaminated during shipping.

To make the most use of outgassing tests, standard methods need to be developed that can be used by suppliers, manufacturers, labs and end users. For many of the above methods, IEST, IDEMA, ASTM and other organizations are developing standards. This should facilitate making consistent databases where products can be compared on an equivalent basis, leading to improved reliability and yields for affected industries. Similar procedures can be applied to other surfaces or products in the aerospace, flat panel display, optical and other sensitive industries.

6.0 ACKNOWLEDGMENTS

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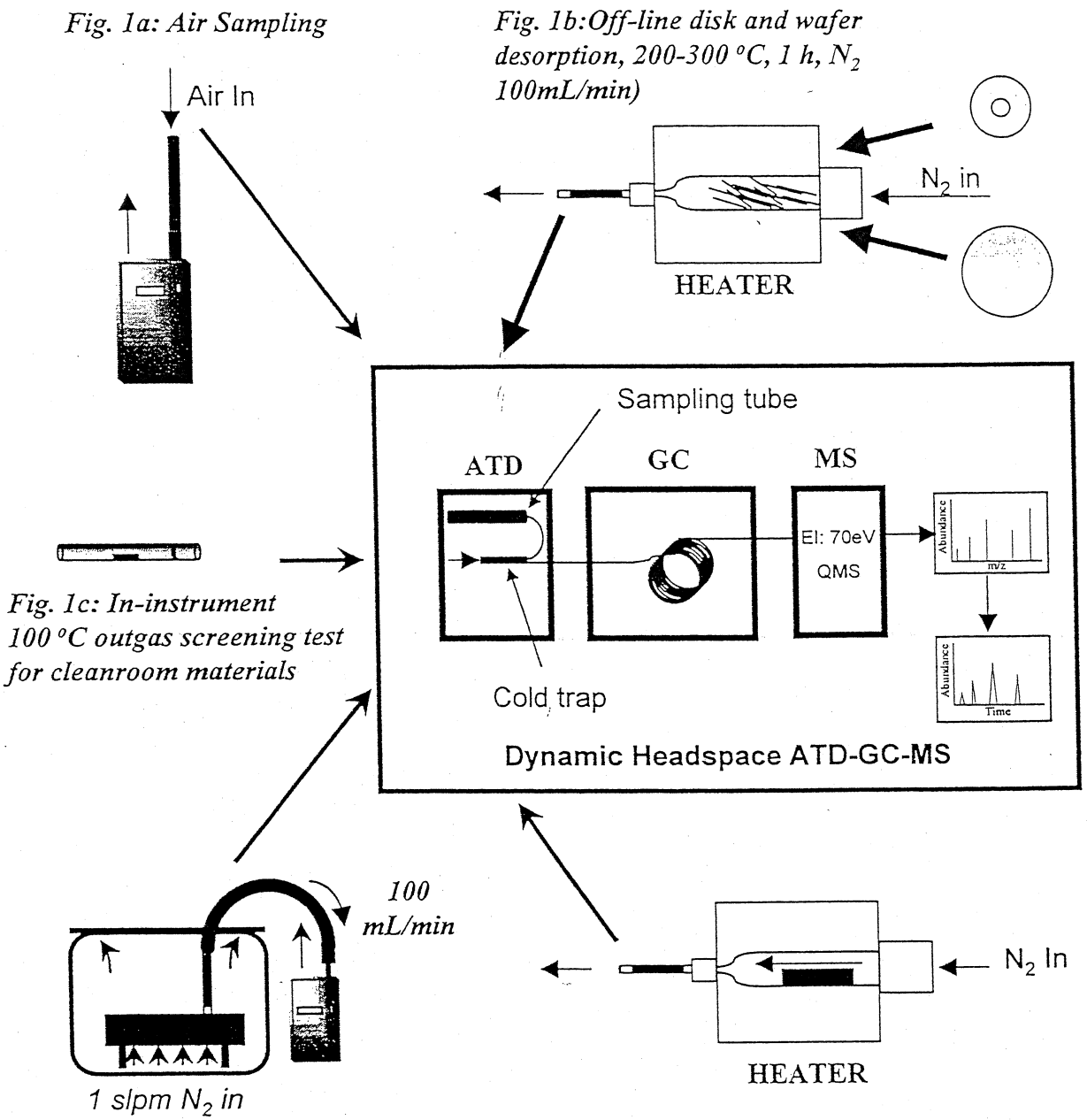
FIGURE 1: Dynamic headspace TD-GC-MS analyses for organic compounds.



Tracking down organophosphate contamination sources in semiconductor fab

FIGURE 2: AIR SAMPLING FOR ORGANICS: TD-GC-MS showing 0.07 ppbv of organophosphate* in cleanroom air.

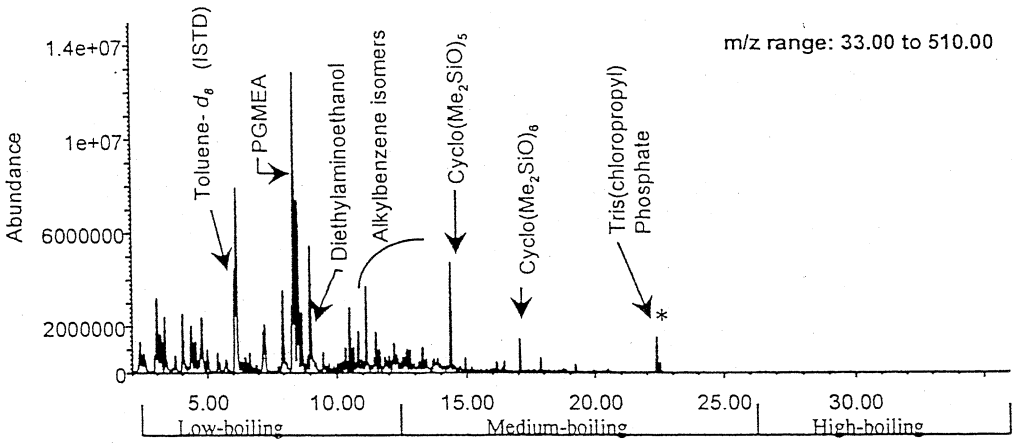


FIGURE 3: WAFER SAMPLING FOR ORGANICS: TD-GC-MS of a bare silicon wafer exposed to cleanroom air; showing organophosphate* (3E13-P/cm^2 estimated) is dominant contaminant on wafer.

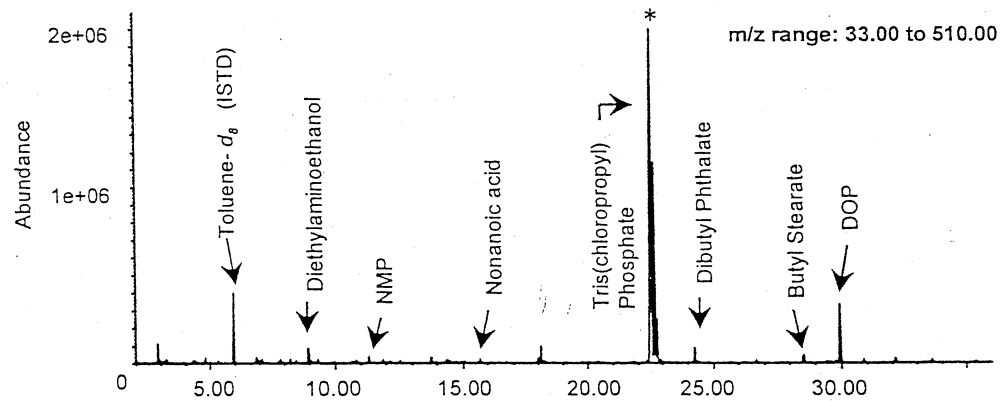
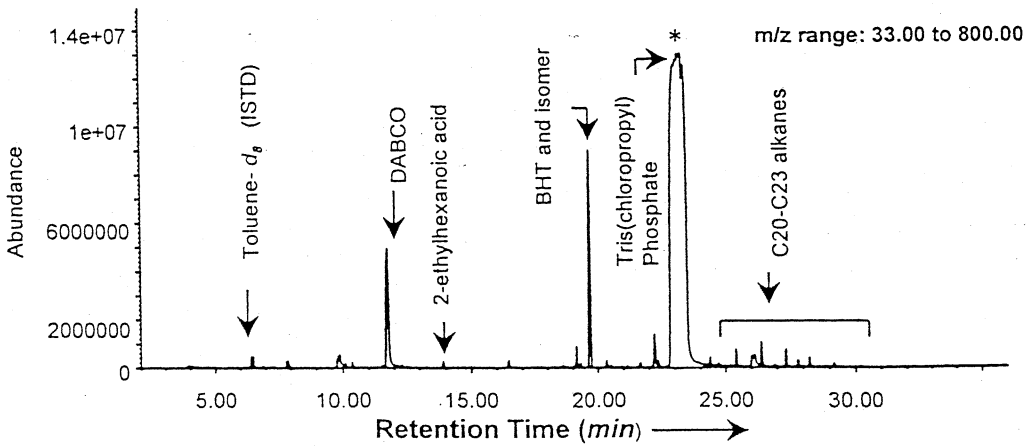


FIGURE 4: FAB MATERIAL ANALYSIS: TD-GC-MS (100°C, DHS): Outgassing from a HEPA filter component identified the source of organophosphate (530 ppmw)* found in air and on wafer.



OUTGASSING OF RUNNING DISK DRIVES

FIGURE 5: Dynamic Headspace GC-MS analysis of organic compounds outgassing from a running old, failed PC disk drive (Drive A). Total outgassing collected running at RT for 24 hours was 2.2 µg.

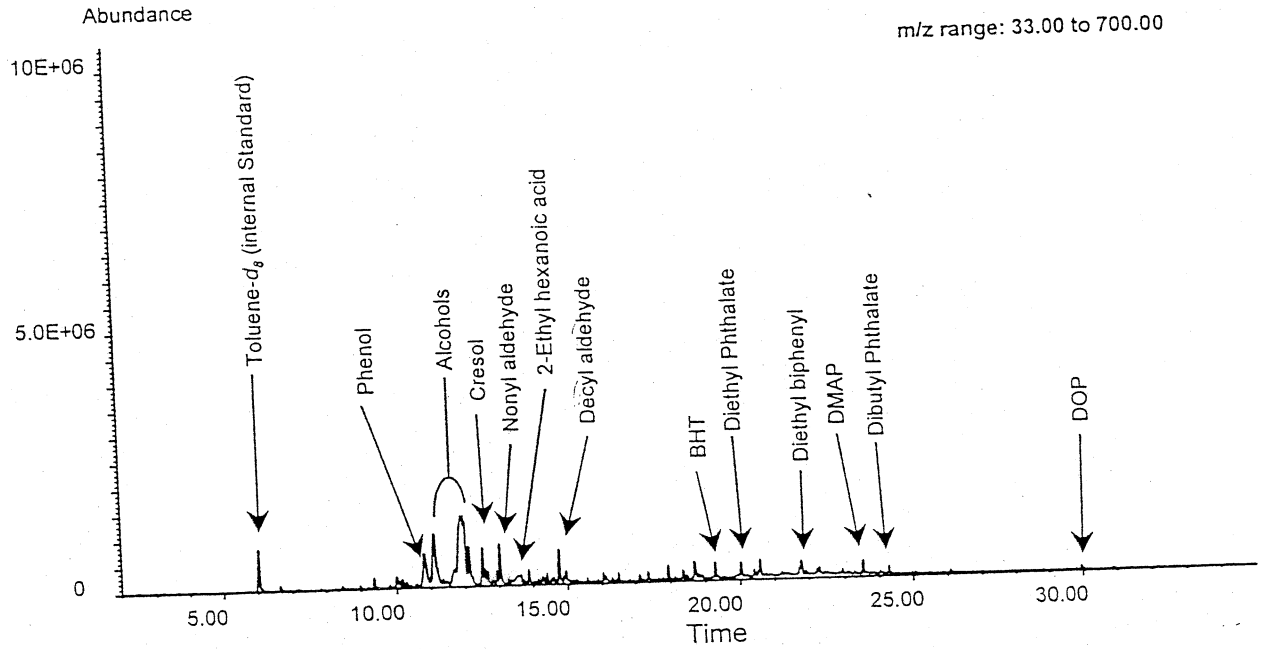
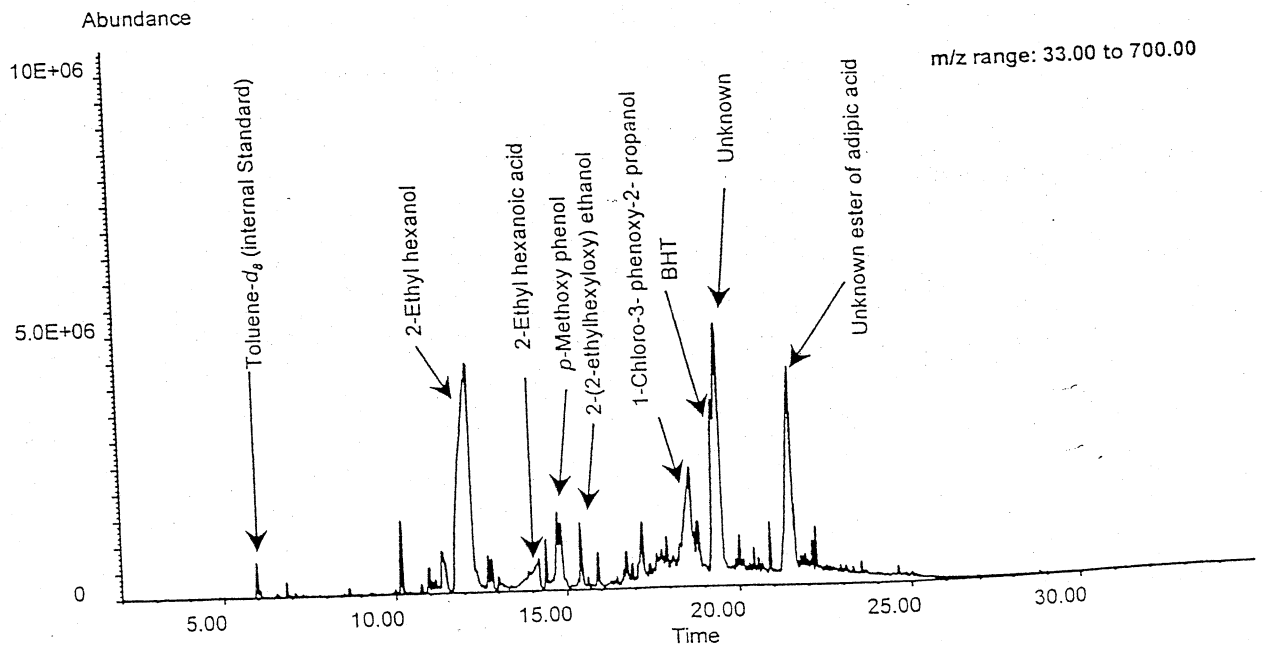


FIGURE 6: Dynamic Headspace GC-MS analysis of organic compounds outgassing from a running new laptop disk drive (Drive B) . Total outgassing collected by running at RT was 56 µg/day



DISK DRIVE GASKET OUTGASSING AT 85°C

FIGURE 7: Dynamic Headspace GC-MS analysis of the gasket from the old, failed PC disk drive (Drive A) at 85°C for 4 hours detected 35 µg organic compounds

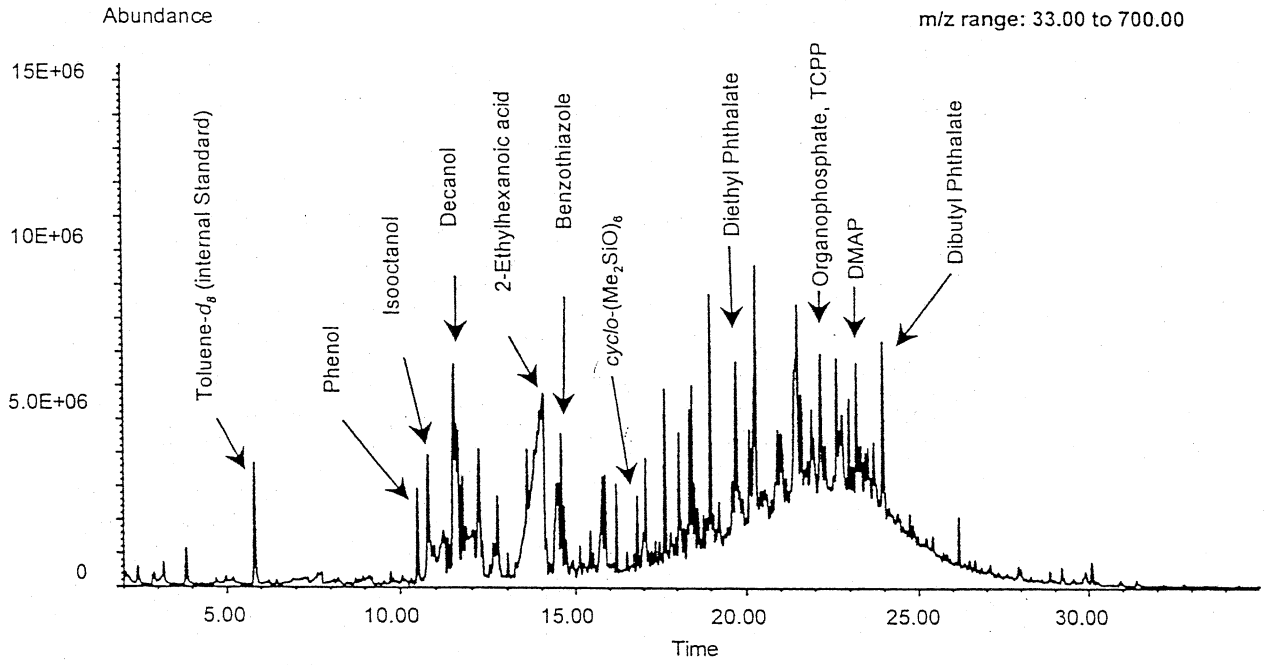
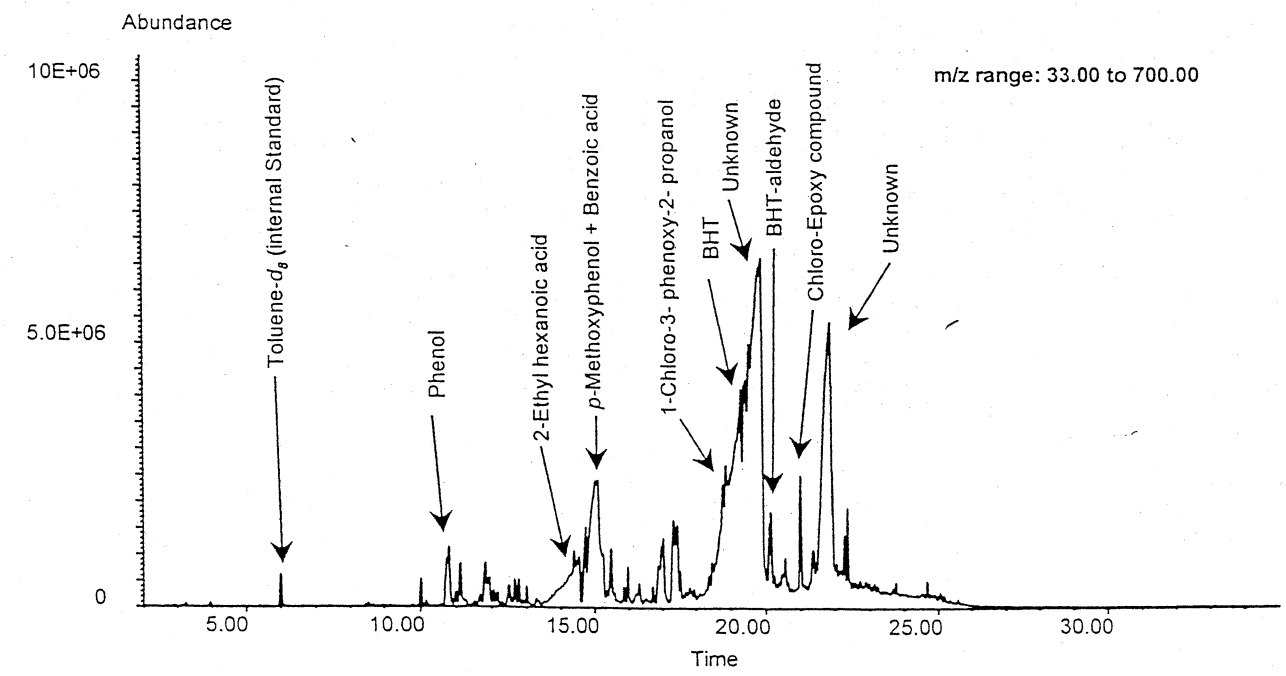


FIGURE 8: Dynamic Headspace GC-MS analysis of the gasket from a new laptop disk drive (Drive B) at 85°C for 4 hours detected outgassing of 105 µg organic compounds.



DISK DRIVE PREAMP AND ADHESIVE OUTGASSING AT 85°C

FIGURE 9: Dynamic Headspace GC-MS analysis of 'Pre-Amp with Adhesive' from the old, failed PC disk drive (Drive A) at 85°C for 4 hours. Total organic compounds detected outgassing was 22 µg .

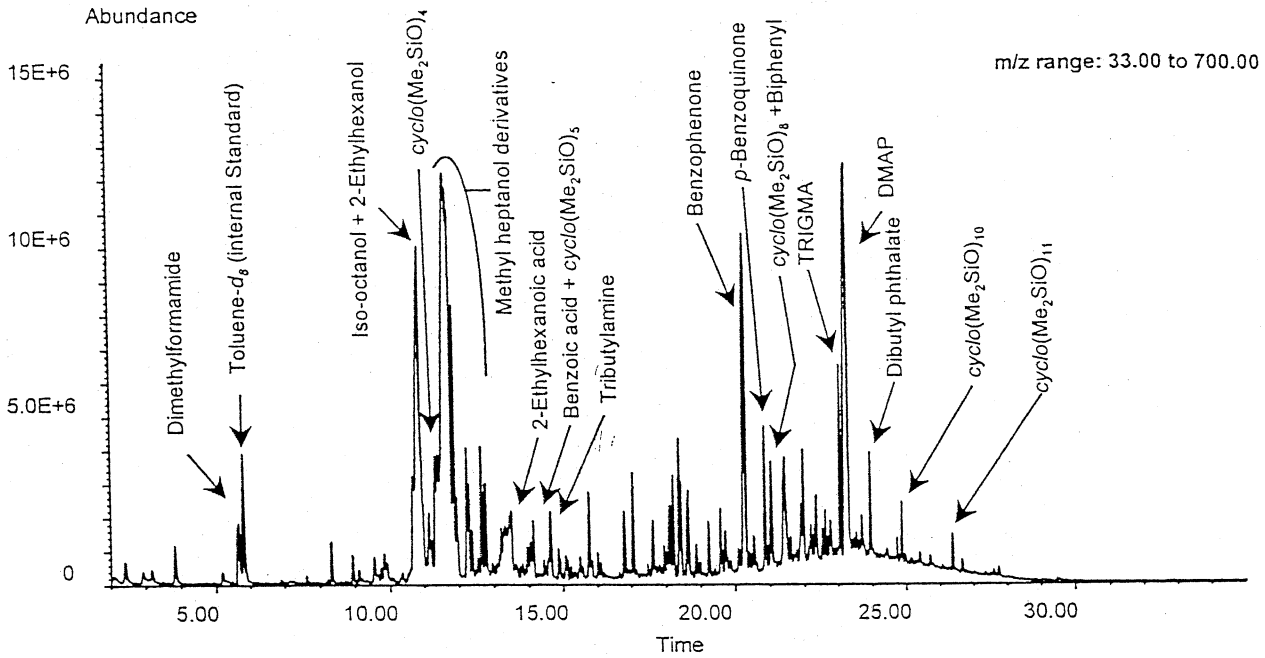
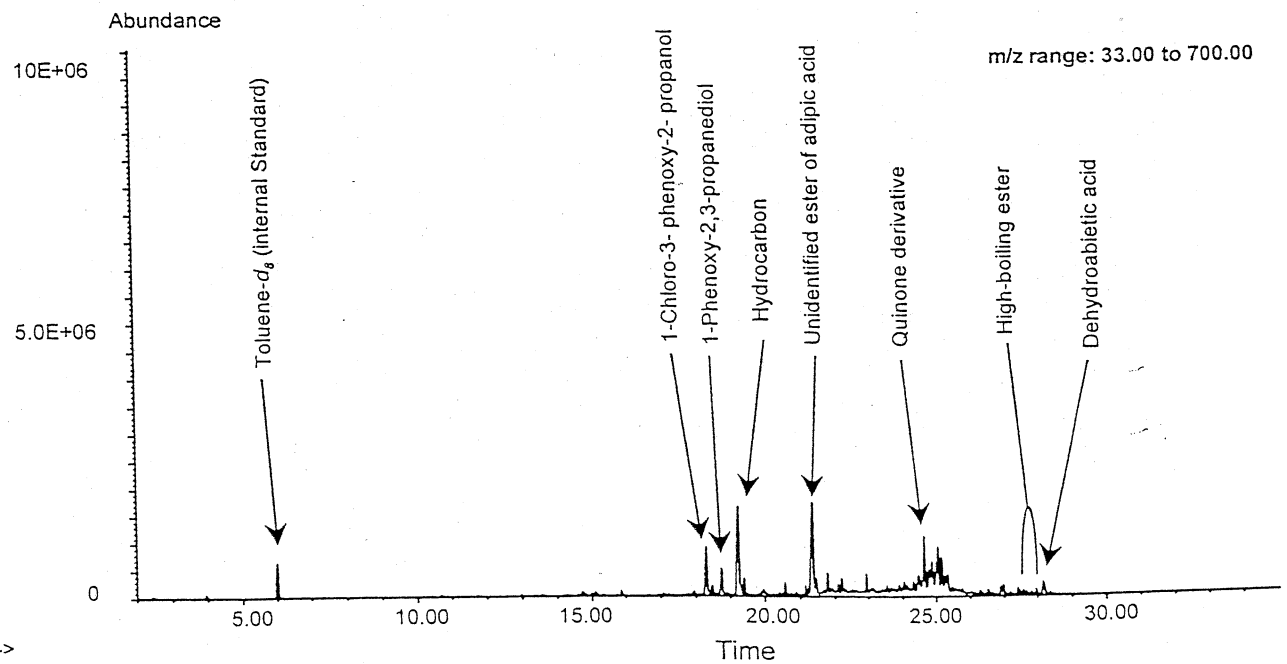


FIGURE 10: Dynamic Headspace GC-MS analysis of organic compounds desorbed from the 'Adhesive of the Pre-Amp' of the new laptop disk drive (Drive B) at 85°C for 4 hours.



DISK MEDIA OUTGASSING AT 200°C

FIGURE 11: Dynamic Headspace GC-MS analysis of organic compounds desorbed from 'Disk Media' of the old, failed PC disk drive (Drive A) at 200°C for 1 hour. Total detected 6.8 µg/disk.

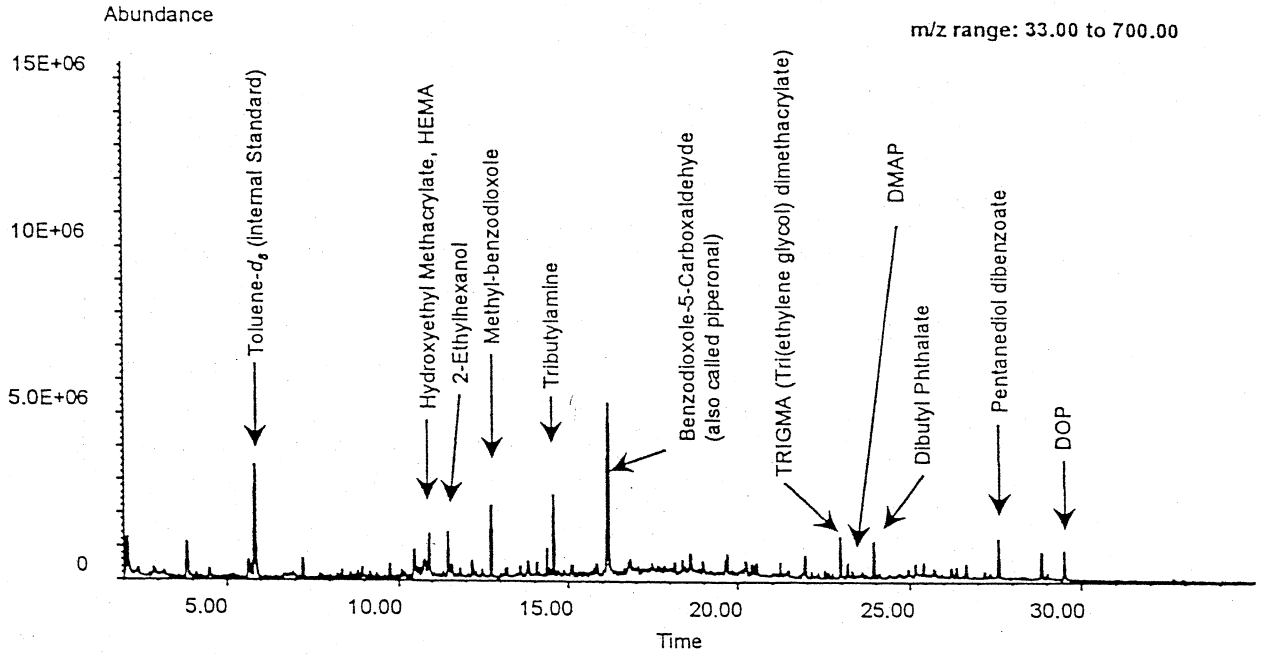
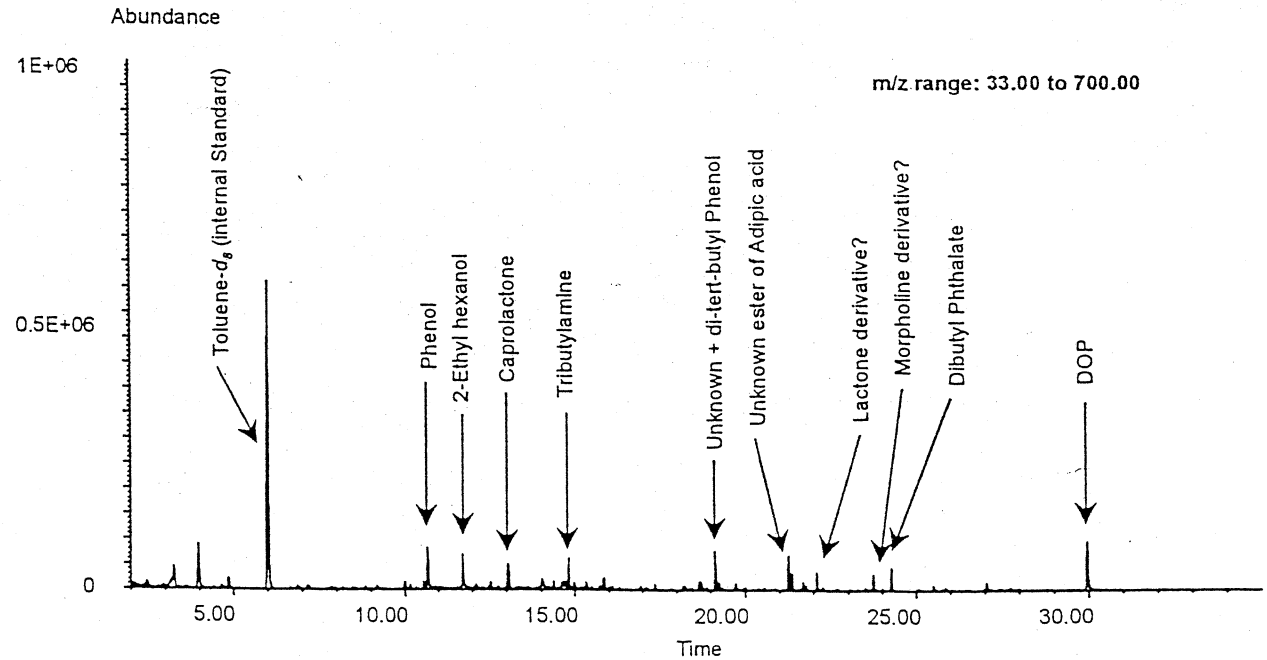


FIGURE 12: Dynamic Headspace GC-MS analysis of organic compounds desorbed from the 'Disk Media' of the new laptop disk drive (Drive B) at 200°C for 1hour. Total detected 0.97 µg/disk.



OUTGASSING OF RUNNING DISK DRIVES

FIGURE 5: Dynamic Headspace GC-MS analysis of organic compounds outgassing from a running old, failed PC disk drive (Drive A). Total outgassing collected running at RT for 24 hours was 2.2 µg.

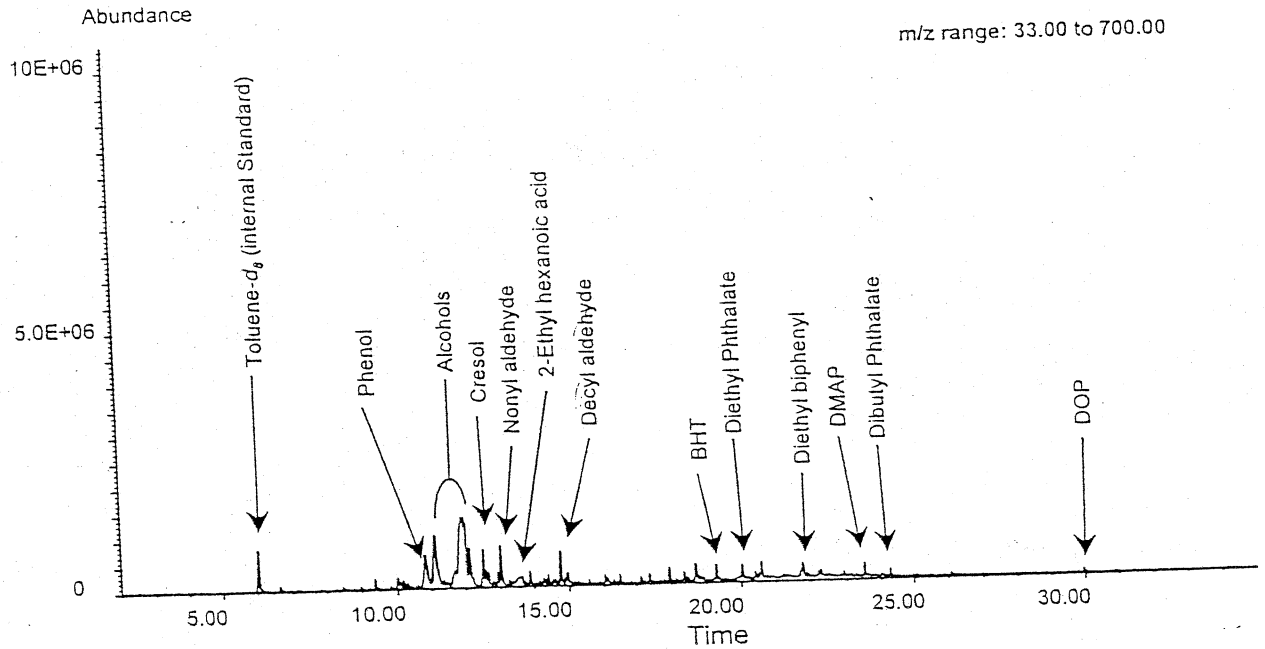
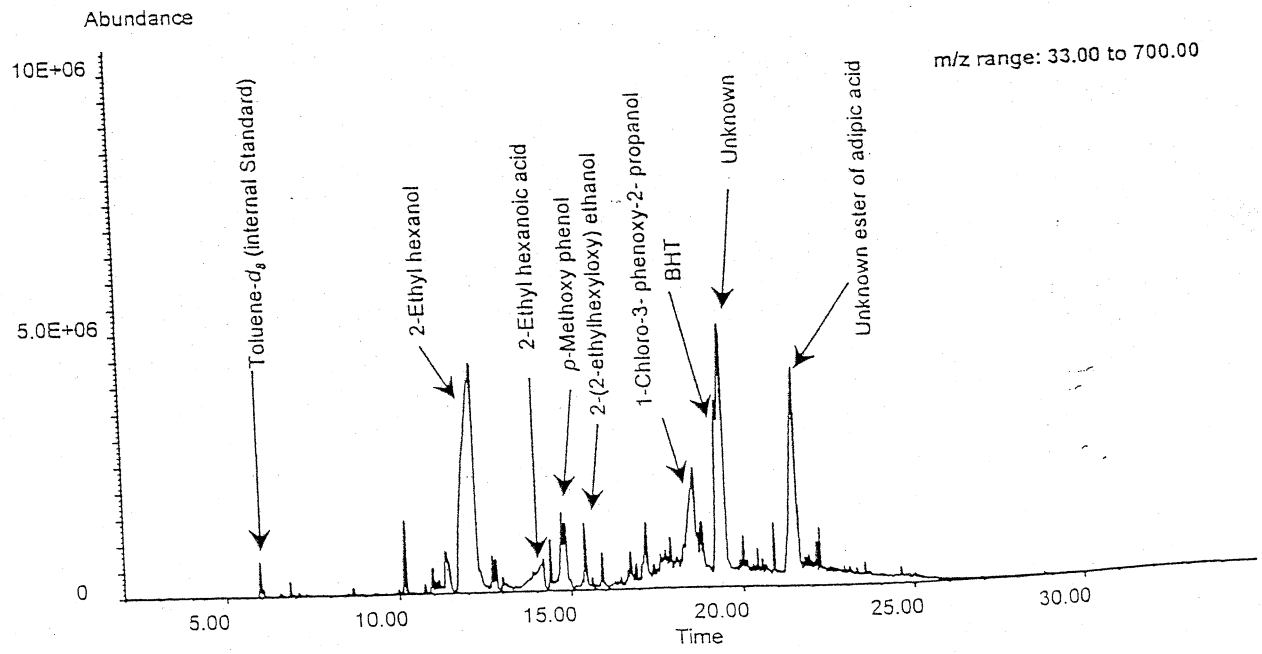


FIGURE 6: Dynamic Headspace GC-MS analysis of organic compounds outgassing from a running new laptop disk drive (Drive B) . Total outgassing collected by running at RT was 56 µg/day



DISK DRIVE GASKET OUTGASSING AT 85°C

FIGURE 7: Dynamic Headspace GC-MS analysis of the gasket from the old, failed PC disk drive (Drive A) at 85°C for 4 hours detected 35 µg organic compounds

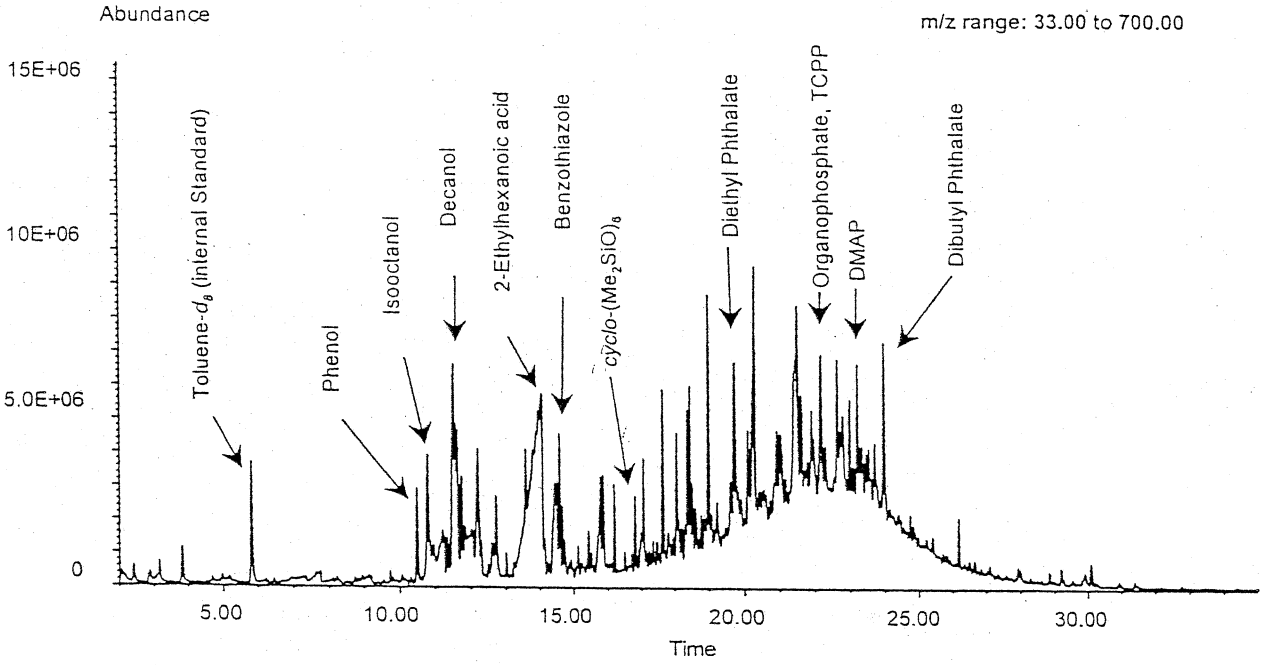
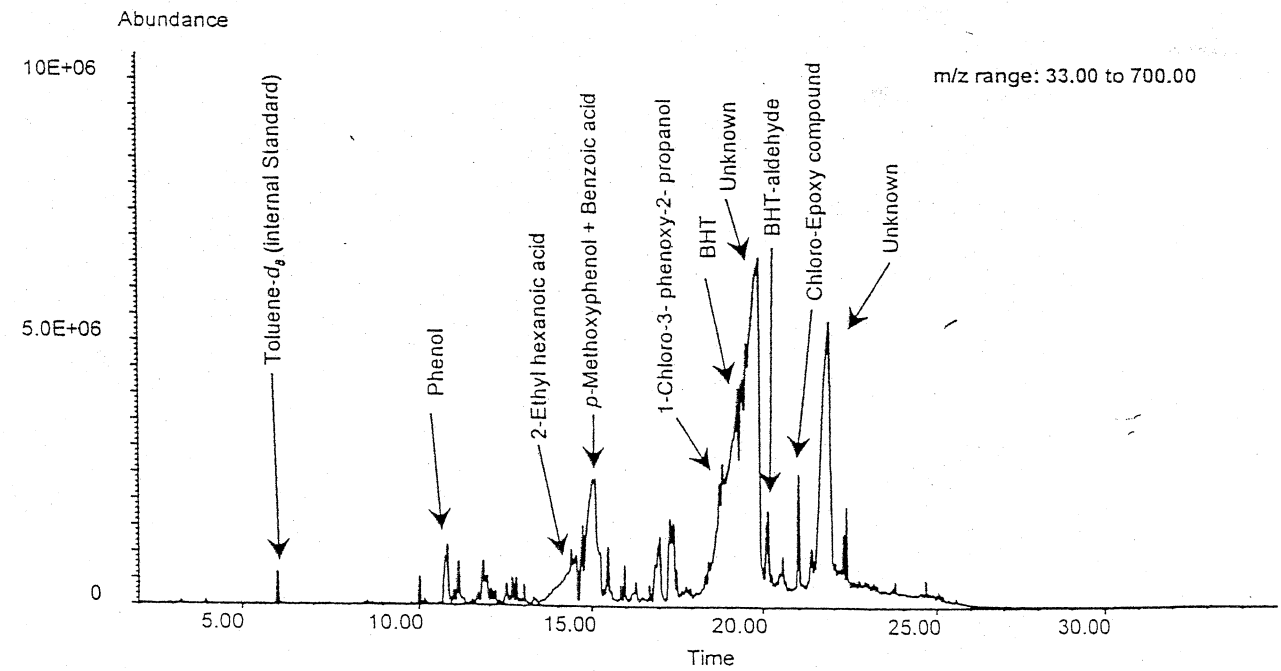


FIGURE 8: Dynamic Headspace GC-MS analysis of the gasket from a new laptop disk drive (Drive B) at 85°C for 4 hours detected outgassing of 105 µg organic compounds.



DISK DRIVE PREAMP AND ADHESIVE OUTGASSING AT 85°C

FIGURE 9: Dynamic Headspace GC-MS analysis of 'Pre-Amp with Adhesive' from the old, failed PC disk drive (Drive A) at 85°C for 4 hours. Total organic compounds detected outgassing was 22 µg .

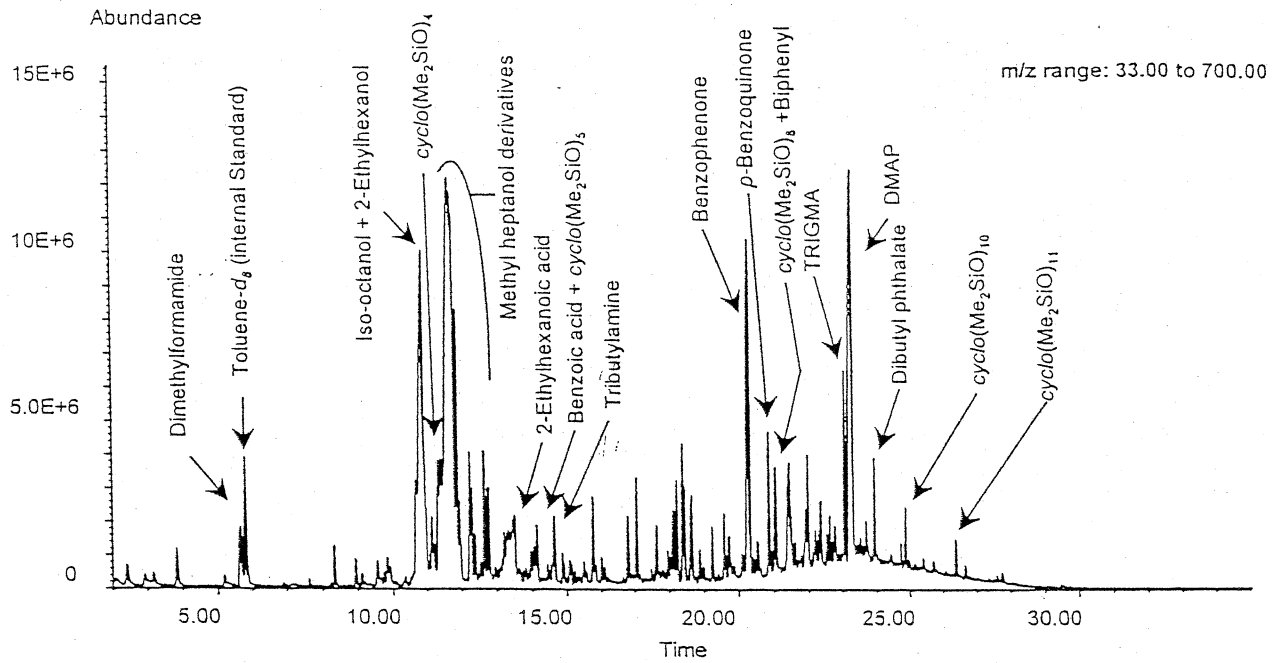
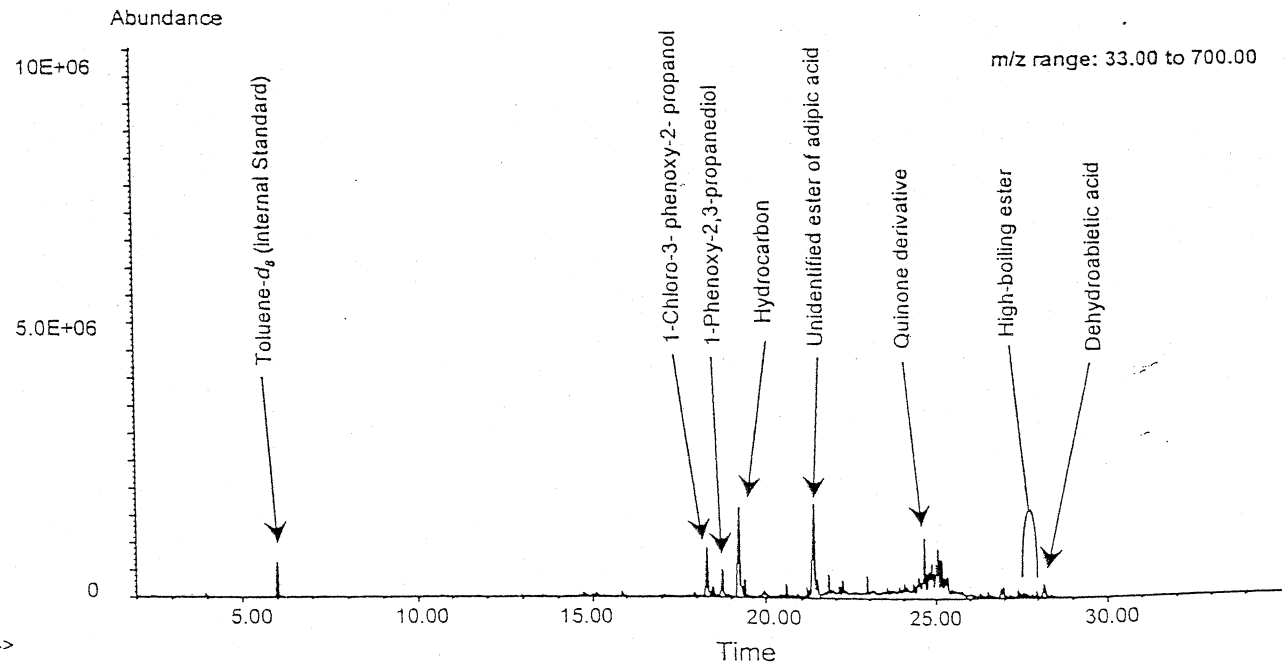


FIGURE 10: Dynamic Headspace GC-MS analysis of organic compounds desorbed from the 'Adhesive of the Pre-Amp' of the new laptop disk drive (Drive B) at 85°C for 4 hours.



Time—>

DISK MEDIA OUTGASSING AT 200°C

FIGURE 11: Dynamic Headspace GC-MS analysis of organic compounds desorbed from 'Disk Media' of the old, failed PC disk drive (Drive A) at 200°C for 1 hour. Total detected 6.8 µg/disk.

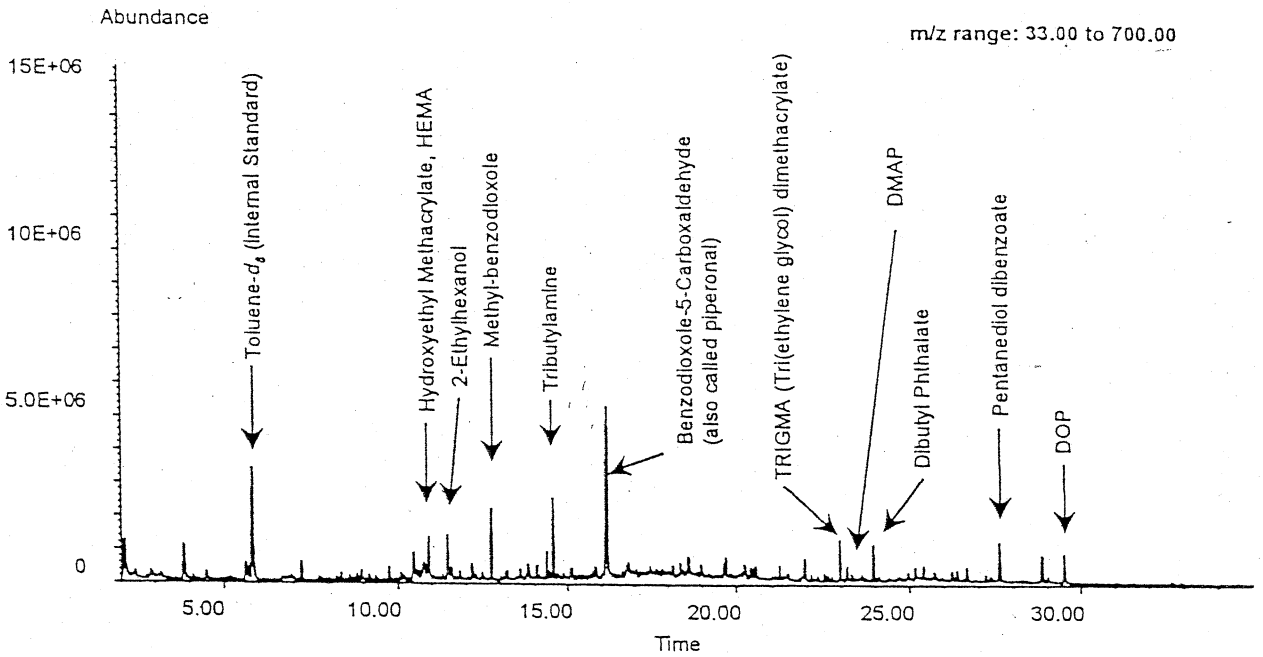


FIGURE 12: Dynamic Headspace GC-MS analysis of organic compounds desorbed from the 'Disk Media' of the new laptop disk drive (Drive B) at 200°C for 1hour. Total detected 0.97 µg/disk.