

Outgas Analysis for Hard Disk Industries

1. Hard Disk Drive and Necessity of its Outgas Analysis

The cross section and the inside view of the hard disk drive (HDD) 3.5" are illustrated in Fig. 5.

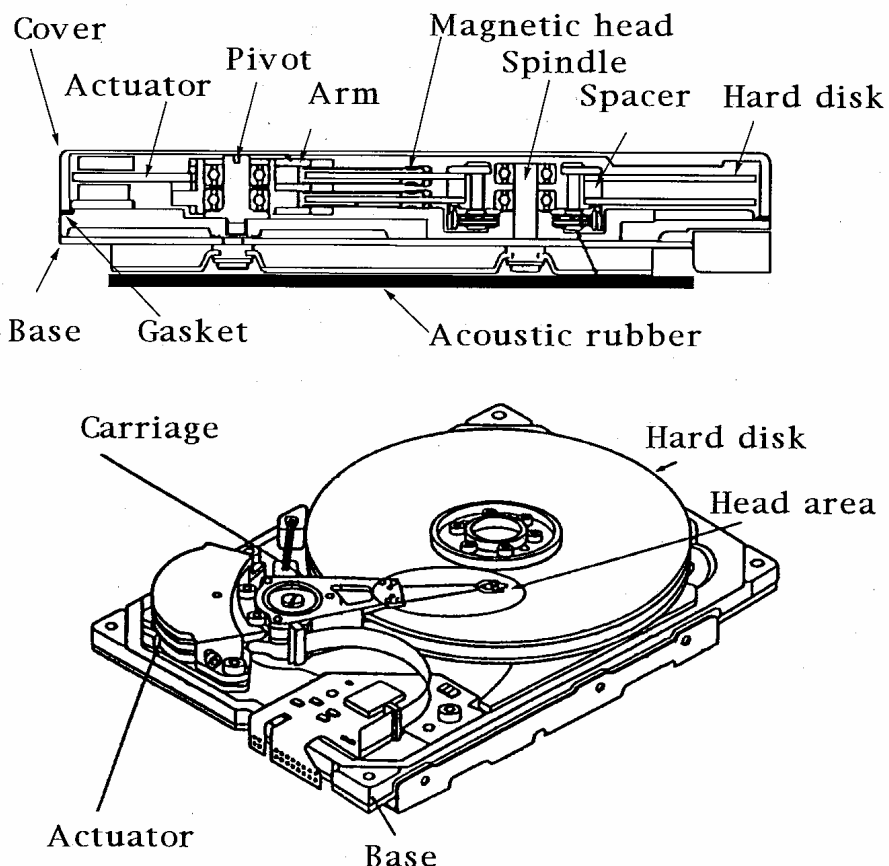


Figure 5 Cross section and the inside view of the hard disk drive

Prior to P&T-GC/MS analysis, the outgas from HDD, hard disk (HD) or major parts for HDD should be collected into the PAT by using exclusive outgas collector which were manufactured according with size of devices.

The collection method and its application are described in this chapter.

The HDD is consisting of a lot of parts as indicated in Fig. 5. The outgas is emitted from the adhesive or compositional parts in the HDD. There are wrong outgas for HDD, for instance, polydimethyl silixane (DSi), organic phosphorous compounds and/ or higher molecular weight of organic components (HMO). It was told that if DSi presence 1 ppb in 2.5" HDD, the SDSi will be made $0.1 \text{ f}\hat{\text{m}}^2$ of SiO_2 with its oxidation, also organic phosphorous compounds make solid, after that they will act as the dust. On the other hand, HMO condenses on the HD after turn off the power supply of HDD, because inside temperature of the HDD goes to lower temperature, never the condensed HD turn, or the HD will be scratched by the head at next power on time.

From such facts, necessity of the measurement of outgas is needed.

2. Comparison between solvent extraction and P&T method for characterization of the organic contaminants on the hard disk

The organic contaminants had been measured by solvent extraction method by using chloromethylene. Figure 6 and Fig. 7 are typical chromatograms obtained by solvent extraction and P&T method respectively.

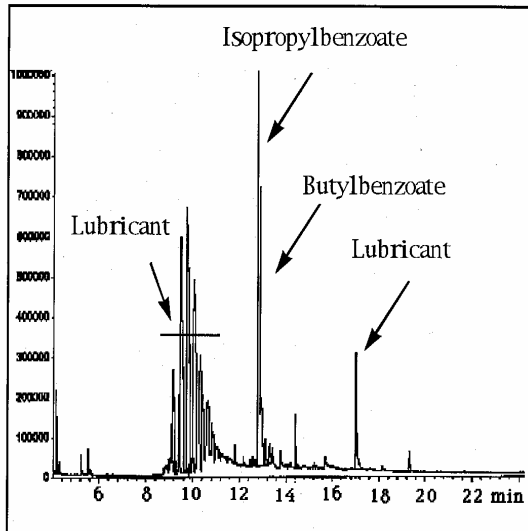


Fig. 6 Organic chromatogram obtained from HD with solvent extraction method.

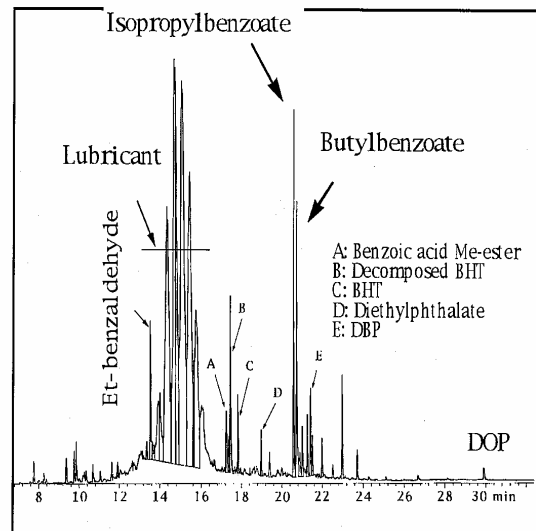


Fig. 7 Organic chromatogram obtained from HD with P & T-GC/MS.

In the case of solvent extraction method, the lubricant peak in Fig. 6 is clearly detected at Rt 10 min and RT 17.1 min, however the organic contaminants are only detected two peaks (isopropylbenzoate and butylbenzoate) at RT 12.8 min.

On the other hand, in the case of P&T method, the lubricant peak in Fig. 7 is clearly detected at Rt 15 min, also the above mentioned contaminants peak are detected. Moreover, lot of peaks are observed on Fig. 7 than that of Fig. 6.

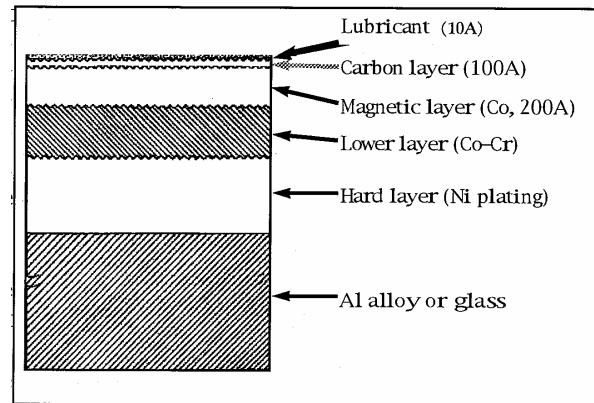


Fig. 8 Cross section view of HD

Those facts told that by use of extraction method is not enough characterization method for the contaminants analysis. It is considerable to postulate that solvent extraction method can not extract so much contaminants, i.e. contaminants in the carbon layer (refer to Fig.8) were not extracted, because the contaminants were more strongly adsorbed in the carbon layer than the extraction power of chloromethylene.

Analytical Condition :

Instrument : GC/MS : HP5973, EI, 70 eV; Outgas collector : HD-3.5"; P&T : JHS-100A

Purging : 60ml/min, He; Thermal extraction : 180 for 30 min; PAT : Tenax GR;

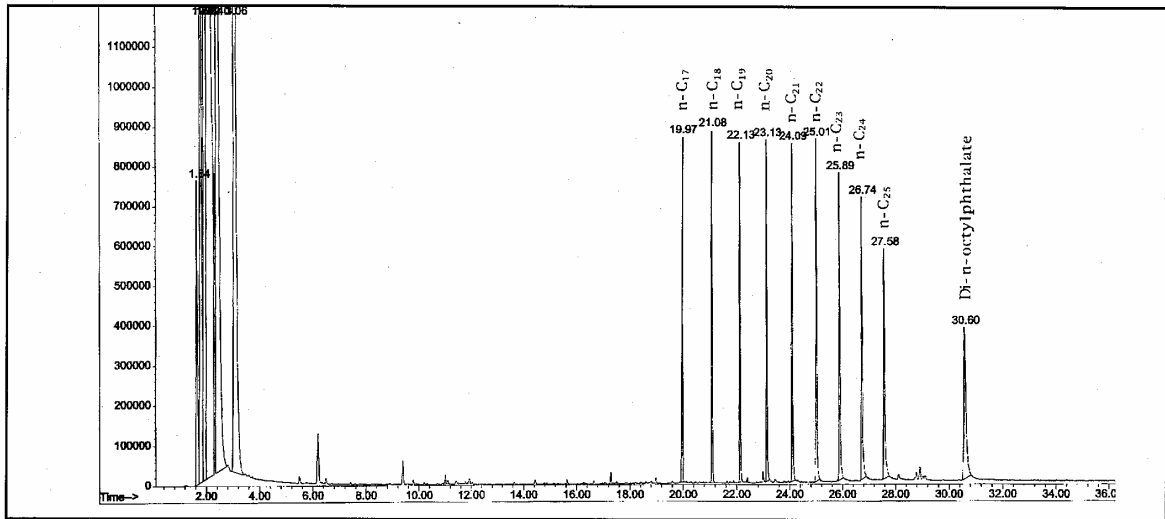
Oven temp. of JHS-100A : 200 ; Secondary adsorption tube : Quartz wool, 255 ;

Column : PTE-%, i.d. 0.25 x 30 m, 40 (hold 3 min)-280 , 10 /min

3. Quantitative Analysis of the Organic Contaminants on the HD

The carbon number 17 to 25 of normal paraffin and 2-ethylhexyl phthalate (DOP) were selected standard sample in order to get calibration curve. Each 10 mg of their compounds are dissolved into 1,000 ml of hexane.

Figure 9 shows typical chromatogram of each 10 ng of the standard samples was left on the HD by using micro syringe. Relative standard deviation (RSD) for eicosane and DOP were measured by 7 continuously repeated sample injection.



conditions are corresponded to Fig. 11.

As a results, RSD of eicosane and DOP were 2.5 and 4.5 respectively.

In the case of outgas analysis for HD, the calibration for DOP is quite straight as shown in Fig. 10. However, that of wafer is curved as mentioned latter.

As a conclusion, the organic compound on the HD can be performed by P&T-GC/MS.

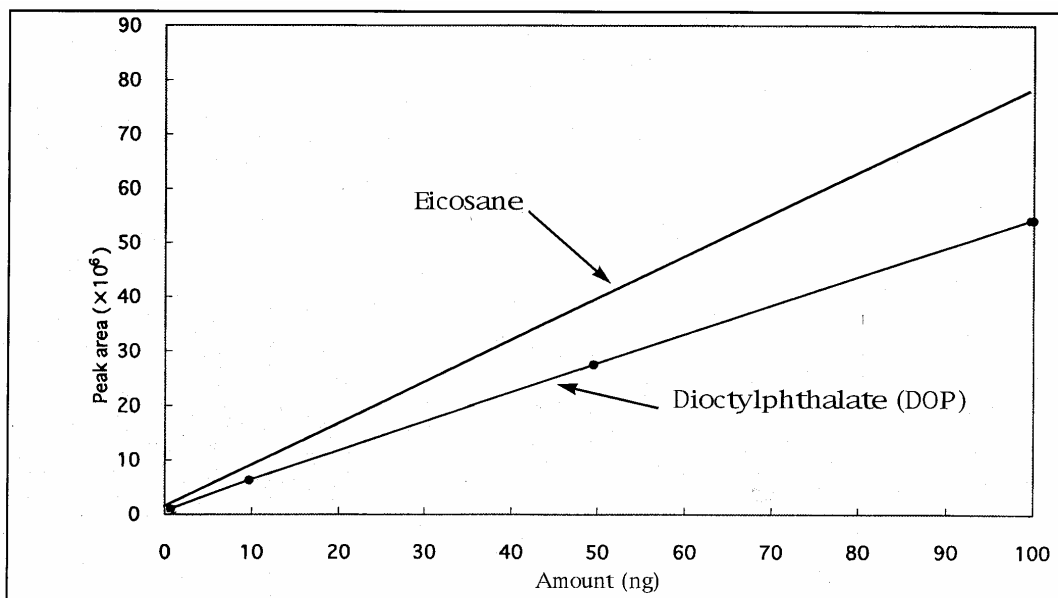


Fig. 10 Calibration for eicosane and DOP peak

4. Contaminants Analysis from Carbon Layer for the HD

Before lubricant coating disk at its manufacturing process was set into the model HD-3.5", The disk was heated at 200 for 20 min with flowing purge gas 60 ml/min. Outgas from the disk was collected into 3.5 g of the Tenax GR packed primary adsorption tube. After pulling out the PAT from HD-3.5", 100 ng of DOP was injected into the PAT by using micro-syringe as standard sample injection. After that the PAT was transferred to JHS-100A, it was then analyzed by P&T-GC/MS.

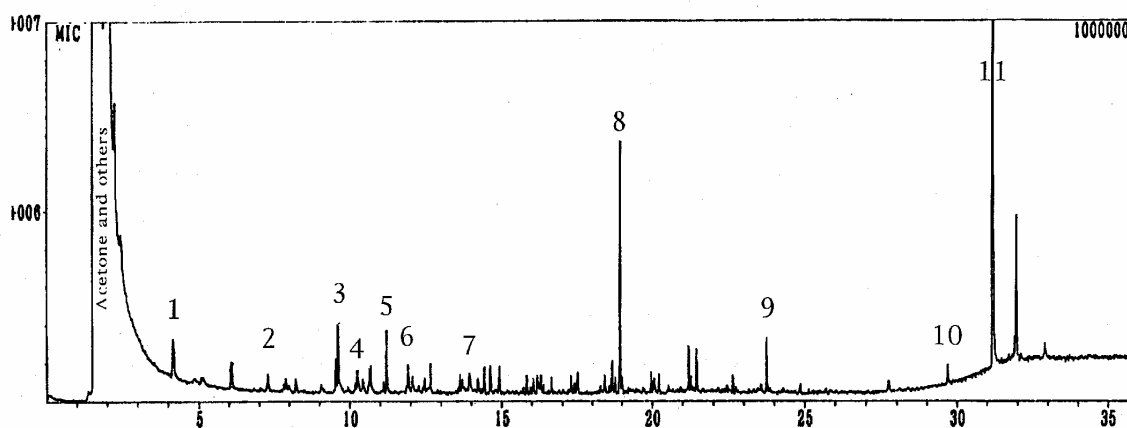


Fig. 11 outgas chromalogram obtained from the before lubricant coating disk

1 : Toluene; 2 : Xylene; 3 : Benzaldehyde; 4 : Phenol; 5 : 2-Ethylhexanol; 6 : Acetophenone; 7 : Benzoic acid; 8 : 2,4-Di-*t*-octylphenol; 9 : *n*-Tetradecanol; 10 : Bis(2-ethylhexyl)phthalate, 11 : Bis(di-*n*-octyl) phthalate 100 ng (internal standard)

Analytical Condition:

Instrument : GC/MS : HP5973, EI, 70 eV; Outgas collector : HD-3.5"; P&T : JHS-100A

Purging : 60ml/min, He; Thermal extraction : 255 for 20 min; PAT : Tenax GR;

Oven temp. of JHS-100A : 200 ; Secondary adsorption tube : Quartz wool,

Column : DB-2, 0.25 f \hat{e} .d. 0.25 x 30 m, 40 (hold 3 min)-280 , 10 /min

Figure 11 shows outgas chromatogram obtained from the before lubricant coating disk. Fundamentally, the organics compounds on the disk was nothing, however, it is clearly observed on the chromatogram. Total organic amount was calculated by the DOP amount of Fig. 10 as 37 ng on the disk.

Toluene and xylene on the chromatogram, they are absorbed airborne compound into the carbon layer after spattering of the carbon, because spattered carbon has higher adsorption power. 2-ethylhexanol and benzoic acid may be decomposed compounds from the DOP during the spattering. Also, it is considerable to postulate that phenol, acetophenone and 2,4-di-*t*-octylphenol were decomposed compounds from oxy-organic compounds or cleaved compounds were reacted each other.