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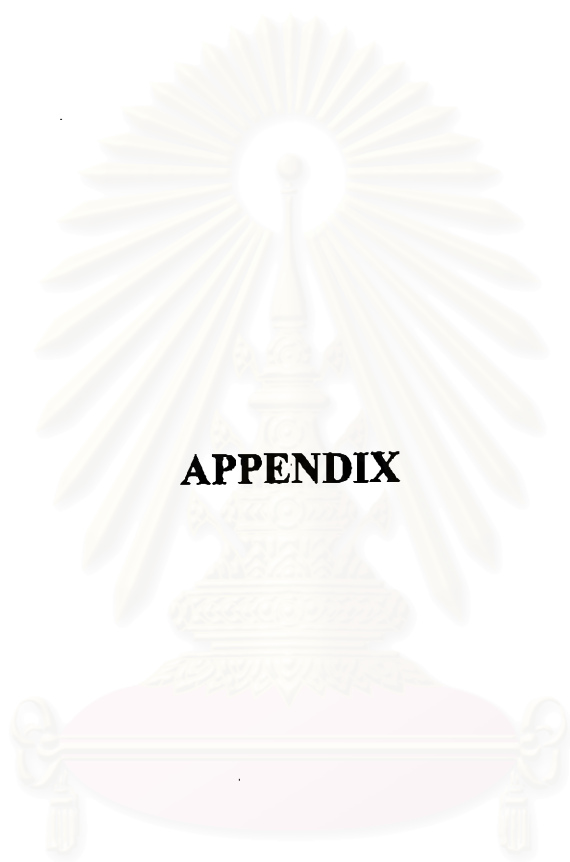
APPENDIX

APPENDIX A

1. DETERMINATION OF SILICONE CONTENT ON VARIOUS RELEASE LINERS AND LABEL ADHESIVE SITE USING FT-IR ANALYSIS.

1.1 PROCEDURE IN DETAIL (52)

1.1.1 Tools, Equipment and Chemical

1. Externally exhausted laminar flow hood

2. Fourier Transform Infrared Spectrometer with MCT detector. For this experiment uses Nicolet Magna 760 E.S.P with MCT detector see Figure A1.

Figure A1. Nicolet Magna 760 E.S.P. with MCT detector FT-IR

3. Attenuated Total Reflectance (ATR) Attachment, Vertical type as variable ATR Spectra Tech, #300, P/N#0012-010 see Figure A2.

4. Crystal type used as KRS-5 (thallous bromide-iodide) and 50x10x3 mm, 45°, parallelogram, for VATR P/N #0050-703

5. Micro syringe, 1-10 µl range, all glass and stainless steel

Note: Material of the syringe has to be inert to solvents and especially, a syringe should be cone tip type

6. Hexane, HPLC grade

The solvent currently used in developing this procedure is hexane. Therefore, sample deposition on the crystal will be affected by different solvent evaporation rates and surface tension differences between the solvent and the crystal.

7. Evaporating dishes

1.2 GENERAL REQUIREMENTS

1. Sample Deposition

When depositing a sample on the ATR crystal, it should be deposited along the entire length of the crystal in a drop wise fashion. If the sample is deposited on a single point, be aware that the sample thickness could exceed the IR beam penetration. Therefore, it is necessary to determine the concentration level at which linearity is lost (Absorbance vs. concentration plot becomes flat at the high end). In general, single point deposition is more useful for very small samples to prevent the signal loss that would occur if the sample were spread out over the entire crystal surface.

2. ATR KRS-5 Crystal Cleaning

2.1   Rinse the crystal using hexane or the solvent being used, a minimum of three times, i.e., until visually clean prior to sample deposition and between samples

2.2   Assure that the VATR crystal is dry and any excess solvent has evaporated

2.3   Place the VATR crystal onto the assembly and record a background spectrum

2.4   Wait approximately 2 minutes and record a sample spectrum

2.5   If one notices any solvent (hexane) peaks in the background, then repeat steps 4 through 6 until no solvent peaks (positive or negative) are present in the background spectrum

2.6   If the crystal is clean and free of solvent residue as well as other contaminants, resume operation

3. FT-IR Sample Recovery

Definition: Sample recovery is defined as the percent sample transferred from the evaporating dish to the ATR crystal.

3.1 Add 0.5 μg of silicone from the standard solution into a precleaned petri dish

3.2 Add 2 ml of hexane into the evaporating dish and swirl in the dish to insure mixing

3.3 Evaporate to approximately 1 ml using low heat. (Do Not Boil!)

3.4 Transfer the residue from the evaporating dish onto the ATR crystal using a precleaned syringe, in a drop-wise fashion. (ONE SHOULD RINSE THE EVAPORATING DISH 3 TIMES WITH HEXANE IN ORDER TO ASSURE A HIGH SAMPLE RECOVERY)

Note: Multiple rinsing beyond three times may be necessary if a high concentration of contaminants are present. In general, one should not notice additional residue on the ATR crystal if a high recovery has been attained.

3.5 Collect the IR spectrum and calculate the concentration from the measured Absorbance using the standard curve (0-1 μg range) generated earlier

Example: For line equation, $Y = mX + b$

Y = measured mAbs

m = line slope

b = Y-intercept

$$X = \text{concentration of unknown in micrograms}$$

if the measured mAbs is 9.6, then, unknown, $X = \dfrac{9.6 - b}{m}$

3.6 Calculate % Recovered as follows:

$$\% \text{ Recovered} = \dfrac{\text{measured (}\mu g\text{)}}{0.5} \times 100$$

3.7 Repeat the above for the 1.0 µg standard for high concentration calibration curve

Note: Repeat step 1-6 three times for each concentration.

4. FT-IR Optical Bench Parameter

Normally, FT-IR optical benches are extremely stable, and variations in mirror and beam splitter position and IR emittance are very small. However, good control at the bench level is necessary to minimize variations once the ATR accessory is in place. Any bench variation will be multiplied as the IR beam reflects and reflect through the various optical elements of the accessory.

4.1 Remove any accessories and establish proper bench purge level. Cool the detector with liquid nitrogen for about 15 to 30 minutes prior to use

4.2 Verify that the proper collection parameters have been

set for the empty bench. For example, the empty bench parameters used on the Magna 760 E.S.P with an MCT-A are;

Velocity = 1.8988 Aperture = 32 Gain = 1 NSS = 64

NSB = 64 Resolution = 4 cm^{-1} Filter = Screen B

5. Crystal Position

Follow the manufacturer's instructions and diagrams to properly position the ATR crystal in the metal holder. The crystal face need to bisect the pin positioning hole (Figure A3). The analyst should be able to look down on it and see a semi-circular hole, the crystal should be covering the other haft of the hole. A poorly positioned crystal will prevent the IR beam from being properly centered on the crystal face. This would reduce the IR throughput, signal-to-noise ratio, and sensitivity.

5.1 Adjust the crystal holder position angle to 45° on the scale (Figure A3 and A4)

5.2 Adjust both flat mirrors (M1 & M2 to approximately 45° relative to the sample slide mount and parallel to each other. (Figure A5)

5.3 Place the ATR accessory in the slide mount

5.4 If applicable, adjust the slide mount position left or right to position the crystal face as close to the focal plane as possible

5.5 Adjust the height and mirror angle of the ART accessory so that the crystal face is centered at the instrument IR beam height, usually measured up from the base plate

6. Precaution

6.1 ATR crystal replacement should more careful because it is easily broken

6.2 Do not touch or catch KRS-5 crystal with free hand

7. Methods for Creating Standard Curve in Detail

7.1   Silicone Standard Curve (Absorbance vs Concentration)

7.1.1 Weigh 25 mg (0.1 mg) of 500 cs viscosity, Dow Corning 200 Fluid, a dimethyl polysiloxane, in a clean pre-weighed 25ml volumetric flask. Dilute to 25 ml (to meniscus level) with spectroscopic grade hexane. This creates a 1 mg/ml standard solution. Label this solution with the concentration, the date it was prepared, and the analyst's initials.

7.1.2 Preparation of standard solutions for:

0-10.0 µg curve Pipette 1 ml of the 1 mg/ml standard silicone solution into a 10 ml volumetric flask. Dilute to the meniscus level with clean hexane. This creates a 0.1 mg/ml standard solution.

<u>0-1.0 µg curve</u> Pipette 1 ml of the above standard solution (0.1 mg/ml) into a 10 ml volumetric flask. Dilute to meniscus level with clean hexane. This creates a 0.01 mg/ml standard solution.

7.1.3 Generation of the 0-10 µg curve

7.1.3.1 Step 1

Draw 5 µl of the 0.1 mg/ml standard silicone oil solution into the cleaned syringe and deposit the solution near the **first deposition point** of the ATR crystal. This volume equates to 0.5 µg of silicone. Take care not to touch the ATR crystal with the syringe, and try to keep the droplet centered on the ATR crystal and away from the edges while the hexane evaporates. **Do not blow on the droplet to facilitate evaporation of the solvent.** An array of concentric circles results if this procedure has been performed correctly. Replace the ATR crystal in the FTIR. Collect an FTIR spectrum of this sample and ratio it against the background. Repeat the above procedure until an appropriate baseline is obtained. If all is satisfactory, then record the baseline corrected peak height of the 800 cm^{-1} band associated with this concentration of silicone.

7.1.3.2 Step 2

7.1.3.2.1 Deposit an additional 5 µl of the solution (as described above) on the **next deposition point.** Overall, this is the second point of deposition. This volume equates to an additional 0.5 µg of silicone for a combined total of 1.0 µg of silicone on the crystal and

collect an FTIR spectrum of this sample and ratio it against the background. Record the baseline corrected peak height of the 800 cm^{-1} band associated with this concentration of silicone.

7.1.3.3 Step 3

7.1.3.3.1 Deposit an additional 10 μl of the solution (as described above) on **the next deposition point.** Overall, this is the third point of deposition. This volume equates to a total of 2.0 μg of silicone on the crystal and collect an FTIR spectrum of this sample and ratio it against the background. Record the baseline corrected peak height of the 800 cm^{-1} band associated with this concentration of silicone.

Note: Check the peak heights of the 800 cm^{-1} silicone band for the three (3) concentrations (0.5 μg, 1.0 μg, and 2.0 μg) at this time. If the peak heights have not doubled (within 10%) from the first to the second deposition (0.5 μg to 1.0 μg) and from the second to the third deposition (1.0 μg to 2.0 μg). Then you must stop data acquisition, clean the ATR crystal, and begin a new from section 6.1.3.1.

7.1.3.4 Step 4

Repeat Step 3 incrementing the deposition point by one each time and depositing 10 μl of solution per increment until a total of 10 μg (11 deposition points) of silicone has been deposited onto the ATR crystal.

Note: Solution depositions are a critical aspect of this procedure.

Acceptable depositions exhibit the following characteristics.

a) The depositions should be circular in shape

b) The circles should be concentric

c) The circles should be centered near the deposition point

d) The circles should not overlap with each other

e) The circles should not be concentrated in the middle of the crystal

g) The circles should not touch the sides of the crystal or veer off to the edge

h) The circles should not be oval in shape

7.1.3.5 Step 5

After completion of Step 4, there should be a total of 11 data points ranging from 0.5-10 µg of silicone generated. From each spectrum obtained, calculate the baseline corrected peak height for silicone. The following baseline points are suggested:

Compound	Peak (cm^{-1})	Starting Point (cm^{-1})	End Point (cm^{-1})
Silicone	800 ± 10 cm^{-1}	925-900	750-730

Plot "mAbs vs. Concentration (µg silicone)" and determine the equation of the line that best represents the data (Linear Regression). This is the standard linear plot for the 0-10 µg range. A linear regression program will generate the slope, y-intercept, and correlation coefficient in the form of $y = mX + b$ where

y = measured mAbs

m = line slope

b = y-intercept

X = concentration in μg

7.1.4 Generation of 0-1 μg curve

Repeat the above procedure to create the standard curve for 0-1.0 μg range using the 0.01 mg/ml standard solution. (Refer to 7.1.2)

7.1.5 Standard Curve Criteria

In addition, it is suggested that the following criterion be used in determining the validity of the regression equations.

7.1.5.1 The y-intercept value should not be greater than 10% of the slope value

7.1.5.2 If the value of the y-intercept is greater than 10% of the slope value, determine the change in the slope if the linear regression line was forced through the origin (0,0)

7.1.5.3 If this change is within 5% of the original slope value, then the original equation with the y-intercept value can be used

7.2 Sample Extraction for FT-IR analysis

7.2.1 Cut a sample minimum at least 4 x 4 sq.cm.

7.2.2 Rinse the sample with Hexane into an evaporating dish

7.2.3 Evaporate the extracted solution at 65°C until the solution remain a few μl

7.2.4 Use a clean syringe draw solution remaining and deposit onto KRS-5

7.2.5 Rinse the evaporating dish with a few dropper of hexane

7.2.6 Repeat step.3-4 and then collect an FT-IR spectrum.

Note: A positive identification of a silicone oil compound requires the presence of four (4) absorption bands at the following wave numbers:

$$800 \text{ cm}^{-1} \pm 10 \text{ cm}^{-1}$$
$$1020 \text{ cm}^{-1} \pm 10 \text{ cm}^{-1}$$
$$1090 \text{ cm}^{-1} \pm 10 \text{ cm}^{-1}$$
$$1260 \text{ cm}^{-1} \pm 10 \text{ cm}^{-1}$$

7.3   Calculation

7.3.1 Collect the IR spectrum and calculate the concentration from the measured Absorbance using the standard curve generate earlier

Example: For line equation, Y= m X+ b

Y = measured Absorbance

m = line slope

b = Y-intercept

X = unknown in μg

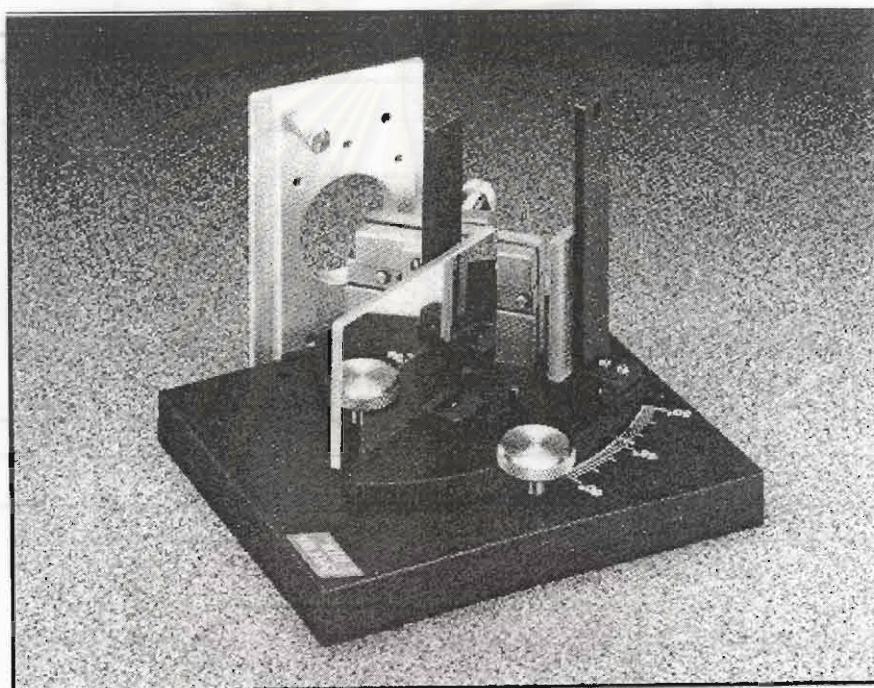if the measured milli absorbance is 9.6, then, unknown, $X = \dfrac{9.6 - b}{m}$

7.3.2 So, Absolute silicone on a piece part, consumable material, or release liner is XK/SA

Where X = Amount of silicone (ester or aliphatic hydrocarbon)

K = Recovery factor for each curve range of each standard

SA = Surface Area (cm^2) of sample
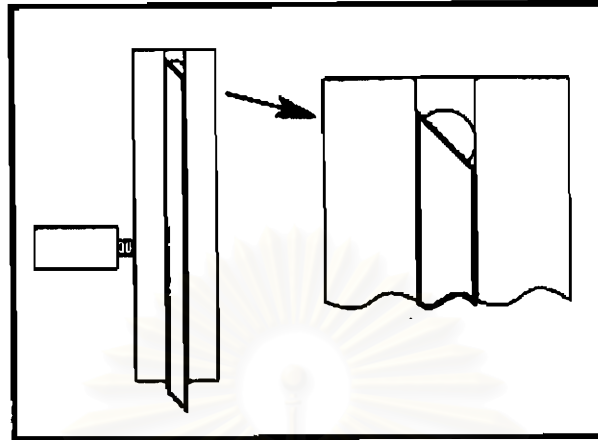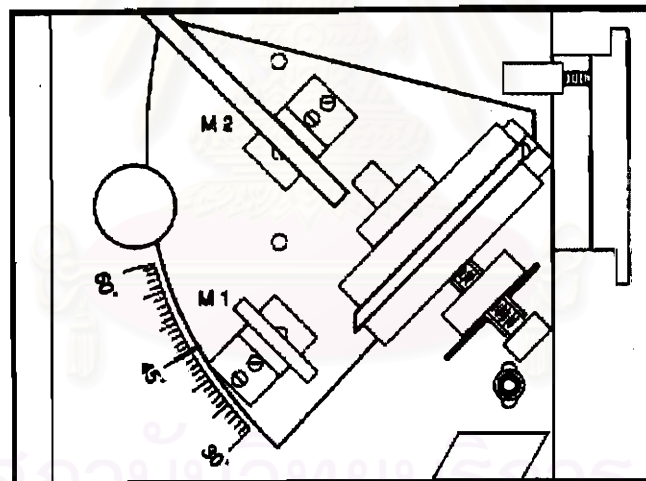
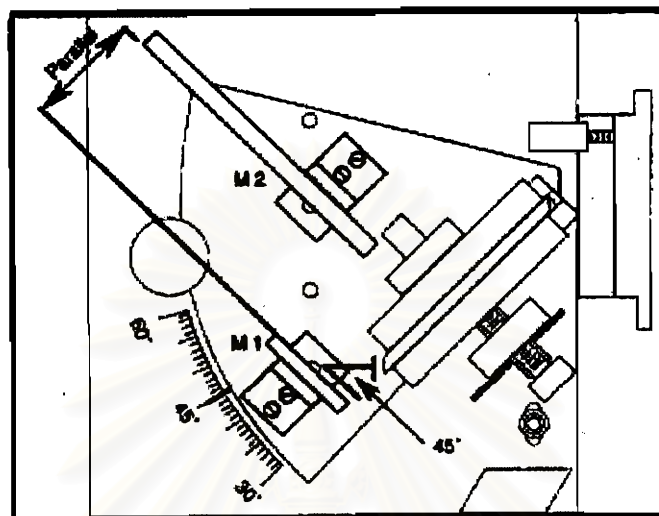**Figure A2. Vertical ATR**

Figure A3. Crystal Position

Figure A4. Angle of VATR Accessory

Figure A5. Mirror Adjustment

2. DHS OUTGASSING TEST ANALYSIS:

2.1   PROCEDURE IN DETAIL (53)

2.1.1 Tools and Permanent Equipment

1. Gas Chromatograph with Detection by Mass Spectroscopy (e.g. HP) (Figure A6)
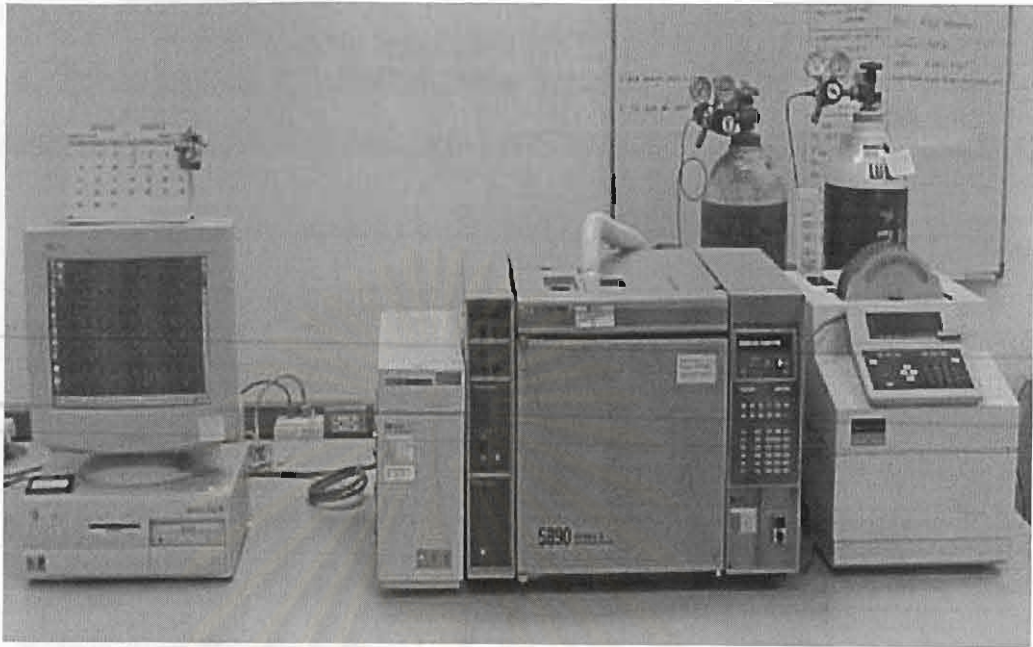
Figure A6. HP GC/MSD attached with Perkin Elmer ATD 400

2. Automatic Thermal Desorption System (PE ATD 400) (Figure A7)

Figure A7. Automated Thermal Desorber ATD 400 Perkin-Elmer

3. Stainless steel adsorbent tubes packed with 200 mg Carbotrap C and 100 mg Carbotrap B

4. Tubes can be purchased from Supelco with the correct packing Construction of tubes: Stainless Steel ATD tubes PE#L4270123

5. Pesticide grade (already silanized) glass wool (Supelco#2-0409)

Carbotrap C (Supelco #2-0309)

Carbotrap B (Supelco #2-0287)

6. Swagelock Connectors

7. Gas Flow Meter (Humonics Optiflow flowmeters Optiflow 520 Digital Flow Meter Supelco Catalogue No. 2-2910)

8. Injection Ports for Standards

Swagelok 1/8" Tube Tee

Septa (Restek) #20374

These are fittings allowing a septum to be placed in-line at the inlet of the chamber or jar for injection of the standard.

9. Dynamic headspace sampler chamber

2.1.2 Expendable Materials

2.1.2.1 Expendable Equipment

1. O-ring, Viton, Ace Seal Part # 2-226, V75 (Note: O-rings must be pre-cleaned by the user in two successive ultrasonic hexane cleaning baths to reduce the chance of introducing contamination)

2. O-ring, Viton, Ace Seal Part # 2-010, V75 (Note: see above note on cleaning)

3. Nitrogen Gas, 99.999% Pure

4. Vials, scintillation, 10 - 20 ml, Preferred VWR PN 66022-130

2.1.2.2 Expendable Chemicals

1. n-Hexadecane standard (e.g. Sigma-Aldrich P/N H-0255)

2. Methylene chloride HPLC grade (e.g. JT-Baker)

2.2   MEASUREMENT METHODS

Operation of Equipment

2.2.1 Calibration Procedure

This section includes the procedure for making standard solutions and using the standard solutions to produce calibration curves. The acceptance criteria for the calibration curves are also outlined.

2.2.2 Calibration Procedure for Semi-Quantitative Analysis

2.2.2.1   Preparation of n-Hexadecane External and Internal Standards

2.2.2.2 Calibration Procedure for Quantitative Analysis (for Semi-Quantitative Analysis)

2.3   PREPARATION OF STANDARDS

2.3.1. Make 10.0 μg/μl stock solutions of the n-hexadecane standards. This should be done by weighing the 0.2500 g of the analyte

(In practice the weight used will depend on the purity of the neat standard) into an empty vial. Using HPLC grade methylene chloride, rinse the material out of the vial and into a 25.0 ml volumetric flask. Cap the flask with a ground glass stopper and turn over/mix the solution at least five times. Carefully adjust the final volume of the flask to the 25.0 ml mark. The concentration in the 25.0 ml volumetric is 10 $\mu g/\mu l$, which is your stock standard. Turn over/mix the solution another five times and then transfer the solution to a scintillation vial. Cap the scintillation vials. Label the vial with the appropriate concentration and content information, along with the analyst's initials and date of mixing. Store the stock solutions in a refrigerator at less than 5°C.

Note: Stock solutions must be discarded after 12 weeks.

2.3.2 Preparation n-hexadecane standard of 200 ng/μl take up 100 μl aliquot of the stock solution into a 50 ml volumetric flask for ten times and carefuly adjust the final volume of the flask to the 50 ml mark with HPLC methylene chloride. Turn over and mix the solution another five times and then transfer the solution to a scintillation vial. Cap the scintillation vials. Label the vial with the appropriate concentration and content information, along with the analyst's initials and date of mixing. Store the stock solutions in a refrigerator at less than 5°C.

2.3.3 This will give a working standard of 200 ng/μl. Inject 5 μl of the 200 ng/μl working standard to obtain a total of 1000 ng of n-hexadecane.

n-hexadecane standard should be injected directly into the injection ports or directly into the preheated chamber with nitrogen flowing.

2.4   INSTRUMENT CALIBRATION

Run 6-10 samples chambers using the standard only.   If the standard deviation is greater than 15% of the average, the measurement must be repeated.

2.5   SAMPLING METHOD

2.5.1   Procedure

2.5.2   Sampling Conditions for DHS

Gas:	Nitrogen (99.999%)
Flow:	50 +/-2 ml/min
Temp/Time:	85°C/3 hrs
Adsorption Tubes:	Carbotrap C/Carbotrap B

2.5.3   Conditions for ATD/GC/MS

Gas:	Helium (99.999%)
ATD Flow:	50 +/-2 ml/min
Outlet Split:	1:50 Ratio
Desorb Flow:	50 ml/min
Desorb:	340°C /8 min
ATD Trap:	Carbotrap C packing
Trap Conditions:	350°C/ 30 min

2.5.4 GC Conditions

Column:	Restek XTI-5, 0.25mm ID x 0.25µm, 30 m
MS:	Sample mass 30 to 550
GC Oven Program:	Initial temperature 40°C for 2 min
	Rate 8°C/min
	Final temperature 240°C for 13 min
	Total run time 40 min

If the conditioned adsorbent tubes are to be stored for more than 24 hrs before use, they should be plugged with teflon caps.

2.5.5 Sampling Procedure

2.5.5.1 Condition the adsorbent tubes using the ATD. The ATD should be programmed to condition the tubes for 15 minutes at 350 °C. Attach the conditioned adsorbent tube(s) to the Teflon line(s) from the sample chamber(s) where they exit the oven.

2.5.5.2 Set the laboratory oven to 85°C. Place the sample chambers in the oven at least one hour prior to use to allow them to reach thermal equilibrium. Turn on the nitrogen flow at the bottle. Attach the ATD tubes to teflon exit lines. Using the gas flow meter set the flow rate of the nitrogen of each vessel to 50 +/-2 ml/min.

2.5.5.3 Measure the pressure sensitive adhesive about 3x3 sq.cm. Remove the backing from the adhesive side of the pressure

sensitive adhesive and fold the pressure sensitive adhesive to adhesive. Place the sample to be analyzed into a sample chamber and attach the tops. for outgassing. As for release liner, measure the release liner about 3 x 3 sq.cm. and then place the samples into the chamber for outgassing. Reported as total ng/sq.cm. Place the parts-containing sample chambers into the laboratory oven and attach the incoming gas lines and the gas lines leading to the adsorbent tubes or to the vessel top. Check flow rates of each tubes. Allow the samples to outgas for three hours.

2.5.5.4 Remove the adsorbent tubes from the lines and place them into the ATD. Program the ATD to desorb the tubes as described in Section 2.5.3. Program the gas chromatograph to sample each tube to be analyzed.

2.6 CALCULATION

2.6.1 Identification and Verification

Identification of outgassed compounds is performed automatically by the operating GC/MS software. These results are then verified manually by the operator.

2.6.2 Calculation Equation

The concentrations of the outgassed compounds are then calculated according to the following equation:

[S] = (area sample x (standard concentration ng/area std))/SA

[S] is concentration of the compound in the sample in ng,

Area sample is the area of peak of the sample

Area standard is the area of the 1000 ng hexadecane peak

SA = Surface area of sample was used

If more than one part per sample chamber is outgassed, [S] obtained by this equation must be divided by the number of parts used.

3. GC/MSD PROCEDURE IN DETAIL (54)

3.1   TOOLS AND PERMANENT EQUIPMENTS

3.1.1 Gas chromatograph with attachments for split flow capillary column analysis with helium carrier gas (e.g., Hewlett Packard Model 5890 Series II Plus)

3.1.2 Detector sensitive to general organic compounds such as mass selective detector (e.g., Hewlett Packard model No.5972) see Figure A8.
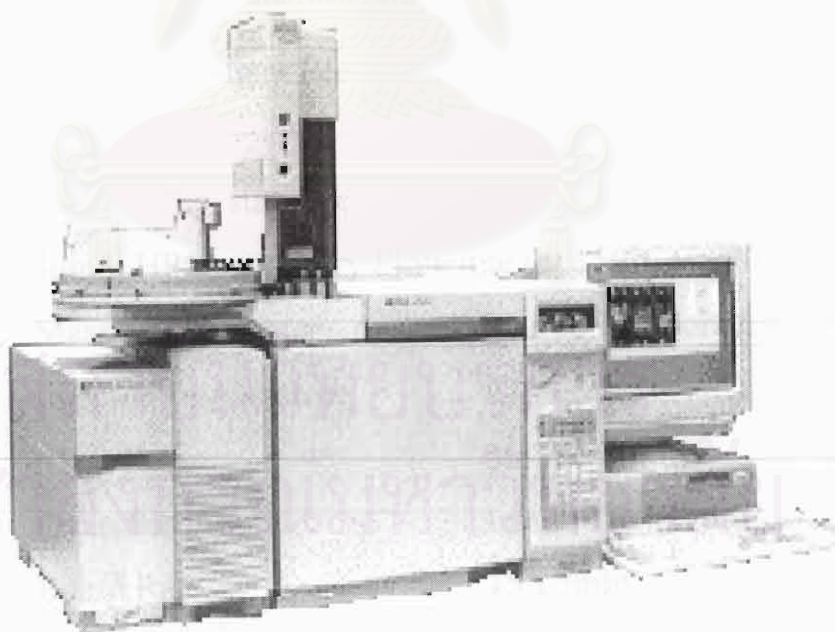
Figure A8. Gas Chromatography/Mass Spectrometre Detector GC/MSD HP5890/5972

3.1.3 Capillary column suitable for separation of general organic compounds; select for consistency of retention times, sensitivity and resolution. A 30 m x 0.25 mm diameter column packed with 5% diphenylpolysiloxane and 95% dimethylpolysiloxane, e.g. Hewlett Packard model 5 is highly recommended.

3.2 CHEMICAL

3.2.1 Methylene chloride, PR grade (e.g. JT-Baker)

3.2.2 Latex rubber gloves

3.3 SAMPLING METHODS

3.3.1 Equipment Preparation

3.3.1.1 The standard instrument settings are shown in the following Column, HP-5, capillary Flow rate (at 150°C) is 0.68 ml/min

Mass Spectrometer	Source Temperature 280°C
Injector Temperature	270°C
Mass Range	30-550 amu
Oven Settings	Initial Temperature 35°C
Ramp Rate	15°C/min
Final Temperature	260°C (15 min)

3.3.2 Sample Preparation

3.3.2.1 Use syringe with methylene chloride 10 µl to localized extract on release liner until left methylene chloride about 2 µl

3.3.2.2 Inject the 2 µl to GC/MSD for analysis

3.3.2.3 Identification and Verification

Identification of extracted compounds is performed automatically by the operating GC/MS software. These results are then verified manually by the operator.

VITA

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