CONOCO INC.
CONOCO CHEMICALS
LAKE CHARLES CHEMICAL PLANT LABORATORY

Benzene in Air

Analysis for Benzene from Personnel Dosimetry Charcoal Badges

Safety Precautions

Both benzene and carbon disulfide are extremely toxic chemicals. All transfers of these materials must be made in a fume hood with the blower running. Rubber gloves must be worn whenever any contact with benzene or carbon disulfide is possible. Badge caps or plugs must be tight enough not to leak CS_2 fumes.

Explanation: Note 1

Procedure

1. From a calibrated solvent dispenser add 1.5 ml (± .05 ml) carbon disulfide containing 0.05 mg/ml undecane (C_{11} Paraffin, 99%) to each badge and cap tightly. Allow 30 minutes for desorption while swirling occasionally. (Note 2) After desorption attach short piece of tubing to side part on badge and pour contents into an auto sampler vial and cap.

2. The new Perkin-Elmer Sigma I chromatograph on your left when facing the console has been designated and set up to run Benzene and certain other personnel dosimetry analyses. This GC has a self-contained computer. Following is the step by step procedure for setting up the GC system for the first analysis in a series.

   A. The 20' 1/8" 20% FFAP column is to be connected to the upper injector and the lower (B) detector. On top of the GC: Be sure B detector button is depressed and the H_2 to A flame is off. GC must be set to Range 10 and Flows to 20. On the Keyboard: Push in the exact order listed.

1. Void
2. 2
3. Enter
4. Set-up
5. 2 (Refers to GC)
6. Enter
7. 1 (Refers to Detector)
8. Enter
9. 211 (Refers to Method)
10. Enter
11. Enter
12. Enter
13. Sample Number (Your own sample #)
14. Enter
15. Sample Name (From tag by Safety Dept
16. Enter

The GC is now set up to run. Sample can be injected when light goes on in button by injection port. Samples may also be run using Auto

The above procedure will only provide a printout. In order to have a trace (chromatogram) the plotter may be linked to the GC as in "B" below, or a strip chart recorder may be connected to the chromatograph.

B. On the Keyboard: Push in the exact order listed.

1. Plot
2. Void
3. Plot
4. 2 (Refers to GC)
5. Enter
6. 1 (Refers to Detector)
7. Enter
8. 0
9. Enter

Both the GC and recorder are now ready to go. Inject 1 microliter into the lower injection port on the GC and push the lighted button just below to start the computer and recorder.

In order to run another sample in the series, only Section A above has to be repeated. (Assuming no one uses the instrument between your runs).

In order to rerun the same sample nothing has to be done except wait for the start button to relight, indicating the system is ready. Simply inject again and push the start botton.

3. Results will be printed out in mg (milligrams) benzene and logged on a sheet designated by the Control Section. All traces and printouts are to be turned into the Control Chemist for forwarding to the Safety Department. No calculations are required by the Analyst. If no area is detected for benzene, report <0.0025 mg.

Notes:

1. Charcoal badges will be tagged with a sample number. A blank badge will be submitted with each series of samples and should be handled and logged the same as samples.

2. Carbon disulfide containing 0.05 mg (milligram)/ml undecane: Fill 1 100 ml volumetric flask equipped with a glass stopper to the mark with reagent grade carbon disulfide (CS_2). With a 10 microliter syringe add exactly 6.75 microliters of high purity 99%+) undecane (C_{11} paraffin). Stopper and mix by shaking vigorously for at least 1 minute. (Caution: Must be done in a fume hood with the blower on while wearing rubber gloves.

3. Reagents:

   1. Carbon disulfide (CS_2) Reagent Grade
   2. N-Undecane (C_{11} Paraffin) 99% or Better Purity

Revised: 2-14-80 by DDP
Approved by FJW

Note 5 To verify Benzene Personal Dosimetry Method

1. Prepare carbon disulfide containing 0.05 mg/ml undecane as explained in Note 2 of LC-PCDA-4 (Benzene in air) method.

2. Prepare a stock solution of 5 mg/ml benzene by weighing 0.5000 grams of benzene into 100 ml Volumetric flask. Dilute to the mark with desorbing solution described in #1 above.

3. Add 1.50 ml desorbing solution described in #1 to as many Sigma I sample vials as needed and cap.

4. To simulate in a 10 liter air sample:

$\frac{0.030 \text{ mg}}{10 \text{ liters}}$	= 1 ppm	Add 6 microliters stock solution from #2 to vial containing 1.5 ml D.S.
$\frac{0.015 \text{ mg}}{10 \text{ liters}}$	= 0.5 ppm	Add 3 microliters stock solution from #2 to vial containing 1.5 ml D.S.
$\frac{0.003 \text{ mg}}{10 \text{ liters}}$	= 0.1 ppm	Add 0.6 microliters stock solution from #2 to vial containing 1.5 ml D.S.

D.S. - Desorbing Solution from #1

6-20-78
DDH

4. GC Parameters

GC North P & E Sigma I
Method: #11

Conditions:	Injection Port Temp	250°C
	Detector Temp	275°C
	Carrier Flow	30 cc/min
	Initial Oven Temp	50°C 9 min. Hold
	Program Rate	5°C/min to 160°C
	Range	10
Column:	20' x 1/8" 10% FFAP on Chrom. W-AW-DMCS 80/100 Mesh	

Issued: 9/9/77 by DDP
Revised: 2/1/78 by LBD & DDH
Approved by FBN

CONTINENTAL OIL COMPANY
CONOCO CHEMICALS
Lake Charles Chemical Plant Laboratory

Methyl Chloride in Air
(Personnel Dosimetry via Charcoal Tubes and Bendix Flasher)

GC Parameters

IP-203, Option 39, S

Conditions as listed on Instrument

Carrier Flow 50 cc/min

Column - 10', 1/8" Poropak QS, 80/100 Mesh

Order of Elution - See Reference Trace

Flasher Parameters

Temperature 275°C

Check for leaks using an empty charcoal tube in flasher.

Calibration of Instrument

Syringe inject 0.25 cc pure methyl chloride from a LCCP shipment into GC several times to obtain an average Area, record. Instrument sensitivity (range 10^{-11}) should be such that an attentuation of 128, or more, is required to keep methyl chloride peak on scale. "Methyl Chloride should be handled in a hood".

Procedure

1. Place Flasher mode switch in by-pass position.
2. Insert charcoal tube (ringed end down) and allow to heat for one minute. (Be sure angel hair is not protruding outside tube ends as this contributes to leaks).
3. Place Flasher mode switch in Inject Position, start recorder and computer. Record area of methyl chloride peak.
4. Analyze both Primary (2-rings) and Secondary (3-rings) tubes.

Calculation:

$$\frac{\text{Area of Sample}}{\text{Area of Standard}} \times 25 = \text{ml Methyl Chloride}$$

Reporting Results:

Report ml Methyl Chloride on
1. Primary Tube
2. Secondary Tube
3. Total (Primary + Secondary)

Issued: 9/15/77 by DDP
Approved by EBN

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ANALYSIS OF METHYL CHLORIDE

Safety Precautions

Methylene chloride is highly toxic by inhalation, and a strong eye irritant. Avoid contact with eyes, skin and clothing. All transfers must be made in a fume hood with the blower running.

Procedure

1. The Analyzer 2 is used.

   A. The FFAP column is to be connected to the upper injector and the lower detector (B).

   On top of the GC:

   Be sure detector B button is depressed and H_2 to A flame is off.

   On the Keyboard:

   Push in the exact order listed.

1.	Void	9.	250 (method no.)
2.	2	10.	Enter
3.	Enter	11.	Enter
4.	Set-up	12.	Enter
5.	2 (refers to GC)	13.	Sample no. (your sample no.)
6.	Enter	14.	Enter
7.	1 (refers to detector)	15.	Enter
8.	Enter		

The above procedure will only provide a printout. In order to have a trace (chromatogram) the recorder must be turned on using 0.25 in/min.

2. Results will be printed out in mg (milligrams) of methyl chloride and logged on a sheet designated by the Control Section. All traces and printouts are to be forwarded to the Control Chemist.

   NOTE: Since the desorption solution may contain a small amount of methyl chloride, it may be necessary to subtract this value (obtained from blank) from milligrams reported by instrument.

3. GC Parameters

   GC: Analyzer 2
   Method 250

   Conditions:

Injection Port Temp	250°C
Detector Temp	275°C
Carrier Flow	30 cc/min
Initial Oven Temp	50°C, 8 min
Program Rate	20°C/min to 180°C
Range	10

   Column: 20' x 1/8" 10% FFAP on Chrom. W-AW-DMCS 80/100 mesh

   Sample Size: 1.5 ul

   Recorder: 0.25 in/min, ATTN-4

Auto-Sampler Use in Sampling Methyl Chloride for Analysis

Safety Precautions:

Methylene chloride is highly toxic by inhalation and a strong eye irritant. Avoid contact with eyes, skin and clothing.

All transfers should be made in a fume hood with the blower running.

1. Take a 10 ml serum vial which contains the charcoal to be desorbed. Using a 5 ml gas tight syringe, add 4 ml of desorption solution. Gently, shake solution. Allow solution to set for 10 minutes before proceeding to Step 2.

2. Using a different 5 ml gas tight syringe, withdraw about 1 ml of liquid from 10 ml serum vial in Step 1. Prior to removal from the vial, the syringe must be closed. This is accomplished by holding the barrel firm and turning the tip in a clockwise direction two full revolutions. Take care to align dot on tip with dot on barrel. NOTE 1

NOTES:

1. This syringe must be rinsed in a small vial of desorption solution between each sample. This is to minimize cross-contamination.

3. Remove the syringe from the serum vial. Inject into a capped auto-sampler vial until about 3/4 full. Auto sampler vials caps should be tight enough not to turn.

4. Make a list as you proceed, starting with Lab #1 and correlating this number to the date, sample no., and whether the vial contains primary or secondary sample.

   EXAMPLE:

	Date	Sample #	
Lab #1	1/1/79	MC-700	Primary
Lab #2	1/1/79	MC-101	Secondary

   Included in the auto-sampler tray should be at least one vial of desorption solution (neat) to be used as a blank.

5. Load the circular auto-sampler rack in numerical order with the Lab # for each vial corresponding to numbers inscribed around the upper section of the rack.

   Make two copies of the list prepared in Step 4 and keep the original in the work area to attach to the data printouts from the Sigma 1 printer.

   Before releasing this data, it must be checked by the Shift Supervisor/or Control Group Leader.

6. Make sure the auto-sampler is turned off (on the control unit). Turn the mode selector (on the control unit) to EXTERNAL. After checking the injector unit alignment, insert the sample rack into the injector unit and tighten with the big Teflon screw. Slide the rack holder into the unit and lock in place by means of a screw on the right side rear of the injector unit.

7. Adjust blank knob on pressure gauge (labeled 7/20) to a reading of 1-2 pounds. Turn to right to increase and to left to decrease.

8. Follow the procedures in Step I of the specific method for the analysis involved to set up the Sigma I GC and recorder.

9. Only after everything is ready, turn the switch on the auto-sampler control unit to the ON position. The unit should then process all the samples in the sample rack. There must be no empty spaces between samples in the rack that are to be run. Always leave position #30 (upper rack section #) empty (no vial) or the instrument will continue to cycle.

Preparation of Desorption Solution for Methyl Chloride Analysis

Caution: Methylene Chloride is highly toxic by inhalation; moderately by injestion and skin absorption. Strong eye irritant.

1. Weigh a 100 ml volumetric flask with stopper on a 4 place balance. Record weight.
2. Weigh 1 g ± 0.01 of n-octane into the flask (replacing stopper to record weight).
3. Dilute to 100 ml mark with methylene chloride that is methyl chloride free. Record weight. This is Solution "A".
4. Calculation:
   a. Subtract wt. in Step 2 from wt. in Step 1.

$$\begin{array}{l} \text{Step 1 weight} \\ -\ \underline{\text{Step 2 weight}} \\ \text{Gram of n-octane} \end{array}$$

   b. Divide wt. of n-octane by wt. in Step 3.

$$\frac{\text{n-octane}}{\text{(Step 3 wt. - Step 1 wt.) or wt. of total solution}} = \text{factor}$$

5. Weigh a second 100 ml volumetric flask with stopper on a 4 place balance. Record weight
6. Calculate the amount of Solution "A" to add to the volumetric flask by this equation:

$$X = \frac{.01540}{\text{factor (Step 4b)}}$$

   = number of grams (Sol. "A") to add to volumetric flask (B).

7. Weigh in exactly this amount of Solution "A" to the nearest ± 0.005 g (replacing stopper to record wt.).
8. Dilute to exactly 100 ml with methylene chloride.

   Calculation:

$$\frac{\text{(wt. from Step 7)(factor from Step 4b)}}{\text{100 ml}} \text{ 1000 mg/g} = \underline{\quad X \quad} \text{ mg}$$

9. Transfer to a septum capped 4 oz bottle.

Selection of sample size to be injected

1. Set-up injector for a "dummy" injection.

   a. Unscrew thumbscrew which holds sample tray base plate in position (right rear corner).

   b. Pull out the plate which holds the sample tray.

   c. Place an empty vial (no cap, no septum) in Position 1 (read lower row of numbers). Rotate the tray until the number 1 on the upper ring of numbers is aligned with the arrow on the base plate. This insures that vial No. 1 is in position under the filter probe, ready for sampling.

   d. Make sure the sample filler probe is all the way up, and that the needle and syringe are out of and away from the GC injection port.

   e. Turn rotary selector switch on programmer to manual.

   f. Turnpower switch "on".

   g. Turn volume adjustment knob (smaller nut on the end of the syringe) clockwise as far as it will go. (Approximately 10 turns) Do Not exert excess force to turn the knob.

   h. Push and hold the lighted "Inject" button until the light goes out. This will start a sample cycle, which you will interrupt to set sample size.

   i. As the cycle proceeds, watch volume adjustment knob as it moves out approximately one inch. After a few seconds the knob will spring back in. Immediately turn off the power switch.

   j. Observe the plunger tip of the syringe. It will be located at some volume calibration mark on the scale of the syringe barrel.

      To reduce the volume to be injected, turn the volume adjustment screw counter-clockwise until the plunger tip aligns with the desired volume mark. The unit is now ready to operate.

   k. To enlarge the sample injection volume, start at Step e and proceed through step i.

   l. Repeat Step e through Step i until desire volume is obtained.

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Methanol in Air

Analysis for Methanol from Personal Dosimetry Silica Gel Tubes

Safety Precautions

Reasonable care should be exercised when handling methanol and 2-butanol which have TLV's of 200 ppm and 150 ppm in air respectively.

Explanation Note 1

Procedure

1. Using a 2.5 cc gas tight syringe equipped with a 21 ga 1 1/2 inch needle, accurately ± 0.05 ml add 1.5 ml (1.5 cc) deionized water containing 3.0 mg/ml 2-butanol to each vial and cap tightly. Allow 4 hours for desorption while swirling occasionally. Note 2

2. The new Perkin-Elmer Sigma I chromatograph on your left when facing the console has been designated and set up to run methanol and certain other personal dosimetry analyses. This GC has a self-contained computer.

   The step-by-step procedure for setting up the GC and its computer system is as follows for setting up the first analysis in a series. Note: If the auto-sampler is to be used see the special procedure.

   A. The OV-1 Column is to be connected to upper injection port and the upper (A) detector.

      On Top of the GC: Be sure that "A" detector button is depressed and the H_2 to "B" detector is "off" and H_2 to "A" detector is "on".
      On Front of the GC: Range 100

3. On the Keyboard: Push designated keys in the exact order listed.

1. Void	9. 12 (Refers to Method)
2. 1	10. Enter
3. Enter	11. Enter
4. Set-up	12. Enter
5. 1 (Refers to GC)	13. Sample Number (Your Own Spl #)
6. Enter	14. Enter
7. 1	15. Sample Name (From Tag by Safety Department)
8. Enter	16. Enter
	15-30 second delay-wait till light goes off

The GC is now set-up to run. Sample can be injected when light goes on in back by injection port.

The above procedure will only provide a printout. In order to have a trace (chromatogram) the plotter must be linked to the GC as follows:

B. On the Keyboard: Push in the exact order listed

1. Plot
2. Void
3. Plot
4. 1 (Refers to GC)
5. Enter
6. 1 (Refers to Detector)
7. Enter
8. 0
9. Enter

Both the GC and the recorder are now ready to run. Inject 1.5 microliters into the upper injection port on the GC and push the lighted button just below to start the computer and recorder.

In order to run another sample in the series, only section "A" above has to be repeated. (Assuming no one used the instrument between your runs).

In order to rerun the same sample nothing has to be done except wait for the start button to light up indicating the system is ready. Then inject again and push the start button.

3. Results will be printed out in mg (milligrams) methanol and logged on a sheet designated by the Control Section. All traces and printouts are to be turned in to the Control Chemist for forwarding to the Safety Department. No calculations are required by the Analyst.

Notes:

1. One dram vials containing silica gel from tubes used in personal dosimetry sampling will be prepared and tagged by the Safety Department. Two vials will be submitted for each tube (sample). One vial will contain silica gel from the primary section of the tube and will be designated by the letter "P" on the tag (Example: Me-4-P) and the other vial will contain silica gel from the secondary section and be designated by the letter "S" (Example Me-4-S) The contents of each vial will be analyzed exactly the same way.

2. Deionized water containing 3 mg/ml 2-butanol: Weigh exactly 0.3000 grams ± 0.001 gram 2-butanol into a 100 ml volumetric flask equipped with a ground glass stopper. Fill carefully to the mark with deionized water. Stopper and shake vigorously for 30 seconds three times over a 10 minute period.

3. Reagents: 2-butanol 99.0+%
   Deionized water (Lab supply)

4. GC Parameters
   GC: North P & E Sigma I IP# 327
   Method: #12
   Detector Temp: 275°C
   Injection Port Temp: 250°C
   Carrier Flow: 16 cc/min
   Initial Oven Temp: 40°C - 4 min Hold
   Program Rate: 10°C/min to 170°C
   Range: 100

   Column 12' by 1/8" 3% OV 1 on Chromsorb HP 80/100 Mesh

Note 5 To verify method for Methanol

1. Prepare deionized water containing 3 mg/ml 2-butanol desorbing solution as described in Note 2.

2. Prepare a stock solution of 130 mg/ml methanol by weighing 13.000 grams (4 place balance) methanol into a 100 ml Volumetric flask and diluting to the mark with desorbing solution from #1 above.

3. Add 1.50 ml desorbing solution described in #1 above to as many Sigma I sample vials as needed and cap.

4. To simulate as in a 10 liter air sample.

$\frac{1.3\ mg}{10\ liters}$	=	100 ppm	Add 10 microliters stock solution from #2 to vial containing 1.50 ml D.S.
$\frac{0.65\ mg}{10\ liters}$	=	50 ppm	Add 5 microliters stock solution from #2 to vial containing 1.50 ml D.S.

D.S. - Desorbing Solution from #1

6-20-78
DH

CONTINENTAL OIL COMPANY
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Ethanol, Butanol, and Hexanol in Air

Analysis for C_2OH, C_4OH and C_6OH from Personal Dosimetry Charcoal Tubes

Safety Precautions:

Carbon disulfide is an extremely toxic chemical. All transfers of material containing carbon disulfide must be made in a fume hood with the blower running. Rubber gloves must be worn whenever any contact with carbon disulfide is possible.

Explanation: Note 1

Procedure:

1. Using a special 2.5 cc syringe equipped with a 21 ga. 1 1/2 needle, accurately (± 0.05 ml) add 1.5 ml (1.5 cc) carbon disulfide containing 1.0% by wt., 2 butanol and 1.52 mg/ml N dodecane (C_{12} paraffin, 99%+) to each vial and cap tightly. Allow 30 minutes for desorption while swirling occasionally. Note 2

2. The new Perkin-Elmer Sigma I Chromatograph (IP-327), on your left when facing the console, has been designated and set up to run ethanol, butanol, and hexanol as well as certain other personal dosimetry analyses. This GC has a self-contained computer. Following is the step by step procedure for setting up the GC system for the first analysis in a series.

   A. On top of the GC: Be sure B detector button is depressed.
   On the Keyboard: Push keys in the exact order listed.
   On Front of GC: Range switch 100 depressed

1.	Void	9.	26 (Refers to Method)
2.	1	10.	Enter
3.	Enter	11.	Enter
4.	Set up	12.	Enter
5.	1 (Refers to GC)	13.	Sample Number (Your Own Spl. #)
6.	Enter	14.	Enter
7.	1 (Refers to Detector)	15.	Sample Name and/or Number (From Tag ...
8.	Enter	16.	Enter

The GC is now set-up to run. Sample can be injected when light co... on in button by the injection port.

The above procedure will only provide a printout. In order to hav... trace (chromatogram) the plotter must be linked to the GC as follo...

B. On the Keyboard: Push keys in the exact order listed.

1. Plot
2. Void
3. Plot
4. 1 (Refers to GC)
5. Enter
6. 1 (Refers to Detector)
7. Enter
8. 0
9. Enter

Both the GC and recorder are now ready to run. Inject 2 µl microliter sample into the lower injection port on the GC and push the lighted button just below to start the computer and recorder.

In order to run another sample in the series (analysis for the same compounds), only section A above has to be repeated. (Assuming no one uses the instrument between your runs)

In order to rerun the same sample, nothing has to be done except wait for the start button to relight, indicating the system is ready. Simply inject again and push the start button.

3. Results will be printed out in mg (milligrams) C_2OH, C_4OH, and/or C_6OH and are to be logged on a sheet designated by the Control Section. All traces and printouts are to be turned in to the Control Chemist for forwarding to the Safety Department. No calculations are required by the Analyst.

Notes:

1. Explanation - One dram vials containing charcoal from tubes used in personal dosimetry sampling will be prepared and tagged by the Safety Department. Two vials will be submitted for each sample tube. One vial will contain charcoal from the primary section of the tuba and will be designated by the letter "P" on the tag (Example: Alc-2-P) and the other vial will contain charcoal from the secondary section and be designated by the letter "S" (Example: Alc-2-S). The contents of each vial will be analyzed exactly the same way. Blanks (unused tube's charcoal) will be submitted with each series of samples and will also be analyzed and logged like the samples.

2. Carbon disulfide desorbent preparation. Fresh carbon disulfide desorbent must be prepared monthly or with each series of samples (which are scheduled several weeks apart). For C_2OH, C_4OH, and C_6OH determinations prepare desorbent as follows: Weigh exactly 1 gram ± .05 2-butanol into a glass stoppered 100 ml volumetric flask. Then weigh in exactly 0.1523 ± 0.001 grams dodecane (99%+ NC_{12} paraffin) on a 4 place balance. Fill the flask carefully to the mark with reagent grade (approved by Control Section as being acceptable for use) carbon disulfide. Stopper and mix by shaking vigorously for at least 1 minute.

3. (a) Carbon disulfide (CS_2) - Reagent Grade
(each lot needs to be approved by Control Section for use)

(b) N-dodecane (C_{12} paraffin) - 99% or better purity

(c) 2 Butanol - Reagent Grade

4. GC Parameters (conditions)

GC: North P&E Sigma I IP# 327
Method: #26
Injection Port Temp: 250°C
Detector Temp: 275°C
Carrier Flow: 30 cc/min
Initial Oven Temp: 65°C
Program Rate: 5°C/min
Max Temp: 220°/C
Column 20' x 1/8" 10% FFAP on Chrom. WAW-DMCS 80/100 mesh

Issued: 2-15-78 by LBD & DDH
Approved by _____

Note 5 To verify method for n-Butanol

1. Prepare carbon disulfide desorbent solution containing 1% 2-butanol and 1.52 mg/ml n dodecane as explained in Note 2.
2. Prepare a stock solution of 75 mg/ml n-butanol by weighing 7.5000 grams (4 place balance) n-butanol into 100 ml Volumetric flask. Dilute to the mark with desorbing solution described in #1 above and mix thoroughly.
3. Add 1.50 ml desorbing solution described in #1 above to as many Sigma I sample vials as needed and cap.
4. To simulate as in a 10 liter air sample.

$\frac{0.75 \text{ mg}}{10 \text{ liters}}$ = 25 ppm — Add 10 microliters stock solution from #2 to vial containing 1.5 ml D.S.

$\frac{0.375 \text{ mg}}{10 \text{ liters}}$ = 12.5 ppm — Add 5 microliters stock solution from #2 to vial containing 1.5 ml D.S.

D.S. - Desorbing Solution from #1

6-20-78
DDH

CONTINENTAL OIL COMPANY
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Ethylene Oxide in Air

Analysis for Ethylene Oxide from Personal Dosimetry Charcoal Tubes

Safety Precautions

Both carbon disulfide and ethylene oxide must be considered extremely toxic chemicals. All transfers of these chemicals must be made in a fume hood with the blower running. Rubber gloves must be worn whenever any contact with ethylene oxide or carbon disulfide is possible.

I. Sample Preparation: (See Note 1 for Sample Explanation)

1. Put one dram sample vials in a shallow ice bath for at least 10 minutes.

2. Keeping the vial on ice, add 3.0 ± 0.05 ml desorbing solution (carbon disulfide containing 0.0930 mg/ul 2 methyl pentane) to each vial and cap tightly immediately. Note 2

3. After 30 to 45 minutes transfer enough of the solution from each vial to an auto sampler vial to fill 2/3 to 3/4 full. Cap each one tightly as soon as it is filled.

4. Hold vials in ice bath until they are run or until they are put in the auto sampler rack just before running.

II. Set up the P&E Sigma I GC (IP 237) as follows:

1. Make sure the Poropak QS column is hooked up to "A" detector (top right connection in oven)

2. If auto-sampler is to be used make sure the Poropak QS column is hooked up to "A" injection port (top left connection in oven)

3. Set both carrier flows on 20 cc/min.

4. Switch "B" detector hydrogen valve lever to "off" position.

5. Switch "A" detector hydrogen valve to the "on" position.

6. Depress "A" detector button.

7. Check "A" detector flame to be sure it is lit.

8. Depress range 10 switch on front of CC

III. Set up the Sigma I Computer by pushing keyboard buttons as follows:

1. Void
2. 1
3. Enter
4. Set up
5. 1 (Refers to GC)
6. Enter
7. 1 (Refers to Detector)
8. Enter
9. 43 (Refers to Method)
10. Enter
11. Enter
12. Enter
13. Sample # (Your Own Sample #)
14. Enter
15. Sample Name (From Tag by Safety Dept.)
16. Enter (10-20 second delay till light goes out)

The GC system is now set up to run. Sample may be injected when the light by the injection port comes on.

IV. The above procedure will only provide a printout. In order to have a trace (chromatogram), the platter must be linked to the GC as follows:

On the keyboard: Push keys in the exact order listed:

1. Plot
2. Void
3. Plot
4. 1 (Refers to GC)
5. Enter
6. 1 (Refers to Detector)
7. Enter
8. 0
9. Enter

Both GC and Recorder are now ready to run. Inject a 2 microliter sample into the correct injection port (the one the correct column is connected with) and press the lighted button just below the injection ports.

To run another sample, wait until the button below the inection ports is relighted-inject sample and press button. Section III, above, only has to be repeated if you wish to change the name of the sample.

V. Results will be printed in mg (milligrams) ethylene oxide and are to be logged on a sheet designated by the control section. All traces and printouts are to be turned in to the Control Chemist for forwarding to the Safety Department. No calculations are required by the Analyst.

Notes:

1. Explanation of the Procedure - One dram vials containing charcoal from tubes used in personal dosimetry sampling will be prepared and tagged by the Safety Department. Two vials will be submitted for each tube. One vial will contain charcoal from the primary section and will be designated by the letter "P" on the tag (Example EO-2-P) and the other vial will contain charcoal from the secondary section to be designated by the letter "S" (Example EO-2-S). The contents of vial will be analyzed exactly the same way. Blanks (charcoal from Tubes) will be submitted with each series of samples and will also [illegible] and logged like the samples.

2. Carbon Disulfide Desorption Solution Preparation - Fresh carbon disulfide desorption solution must be prepared weekly or with each series of samples (which are scheduled several weeks apart). Sigma method internal standard values must be corrected for each new batch of solution. Note 5

Prepare as Follows:

1. Tare an unsealed 10 ml sealable sample vial along with the cap and seal to be used.

2. By pipette add 5 ml carbon disulfide to above vial; seal and reweigh. (in hood

3. Using a gastight syringe add 0.25 ± .01 cc 2-methyl pentane through septum cap to above vial and reweigh.

4. Calculate mg/gram 2 methyl pentane as

$$\frac{\text{wt. of 2 methyl pentane} \times 1000}{\text{wt. of } CS_2 + \text{wt. of 2 methyl pentane}} = \text{mg/gr 2 methyl pentane}$$

5. Calculate the amount of stock solution above needed to prepare 100 ml desorption solution with a concentration of 0.09 mg/ml 2 methyl pentane.

$$100 \times 0.09 = 9 \text{ mg}$$

Sample Calculation:

If the stock solution contains 6.5 grams total and 0.2200 grams 2 methyl pentane:

Example:

$$\frac{0.2200 \times 1000}{6.5000} = 33.5 \text{ mg/gr 2 methyl pentane}$$

Wt. of stock solution required is

$$\frac{9 \text{ mg}}{33.5 \text{ mg}} = .2690 \text{ grams}$$

Note: density of solution is 1.24

a. To a 100 ml volumetric flask add 30 ml carbon disulfide in the hood, stopper and weigh.

b. Using a gastight syringe add 0.22 cc stock solution from the sealed vial from 3 above (in the hood) to the 100 ml volumetric flask from "a" above, stopper and reweigh.

c. Calculate actual mg/ml as in desorption solution as follows:

$$\text{mg/ml 2 methyl pentane} = \frac{(\text{Wt. of Stock Solution}) \times (\text{mg/gr 2 methyl pentane in stock solution})}{100}$$

3. To verify the method.

   a. Prepare carbon disulfide desorption solution containing 0.09 mg/ml 2 methyl pentane per procedure in Note 2.

   b. Prepare a stock solution of ethylene oxide in carbon disulfide as follows:

      1. Weigh a sealable 10 ml sample vial with a seal and septum on an analytical balance.

      2. Add 5 ml carbon disulfide desorption solution (containing 0.09 mg/ml 2 methyl pentane) to the vial, seal securely and reweigh.

      3. Using a gastight syringe inject 0.25 cc ethylene oxide liquid through the septum into the vial and reweigh.

      Calculate the mg/gr ethylene oxide in the solution as follows:

$$\text{mg/gr. E.O.} \quad \frac{\text{Weight of E.O. in gr.} \times 1000}{\text{Weight of E.O.} + \text{Weight of Desorption Solution}}$$

      Each microliter of this solution will then contain

$$\frac{1.24 \times \text{mg/gr E.O.}}{1000} = \text{mg E.O./}\mu\text{l}$$

      4. Determine the range of E.O. concentration to be validated.

      5. Determine the number of microliters of the solution needed to contain the desired weight of E.O. to be validated by the following.

$$\mu\text{l of Sol.} = \frac{\text{Weight of E.O. desired}}{\text{mg/}\mu\text{l E.O. in solution from 3 above}}$$

      6. Add the volume (in μl) determined (in 5 above) to 3 ml (± 0.05) desorption solution in a sealed vial. Caution: Syringe must be chilled solution will flash out during transfer.

      7. Follow procedure outlined in Sections I, II, III, and IV for GC determination

4. GC Parameters

GC: IP 237 (North P&E Sigma I)
Method: #43
Column: 7' x 1/8" Poropak QS 80/100 mesh
Conditions: Carrier Flow: 20 cc/min
Injection Port Temp: 100°C
Detector Temp: 275°C
Initial Oven Temp: 110° 6 mm Hold
Program Rate: 6°C/min to 200°C
Range: 10
Method: 43

5. New internal standard values must be entered in Sigma method 43 each time fresh desorption solution is made because the volatility of the reagents make it almost impossible to weigh in exact amounts of 2 methyl pentane. The values must be changed in both the standard weight line and in the data table or results will be in error.

6. Desorption efficiencies of approximately 60% in the range of interest were obtained by this method. A short study using dry ice to cool desorption solution, one dram vials containing charcoal, and the sigma auto sampler vials indicated recoveries of over 80% in the 0.2 to 0.9 mg range

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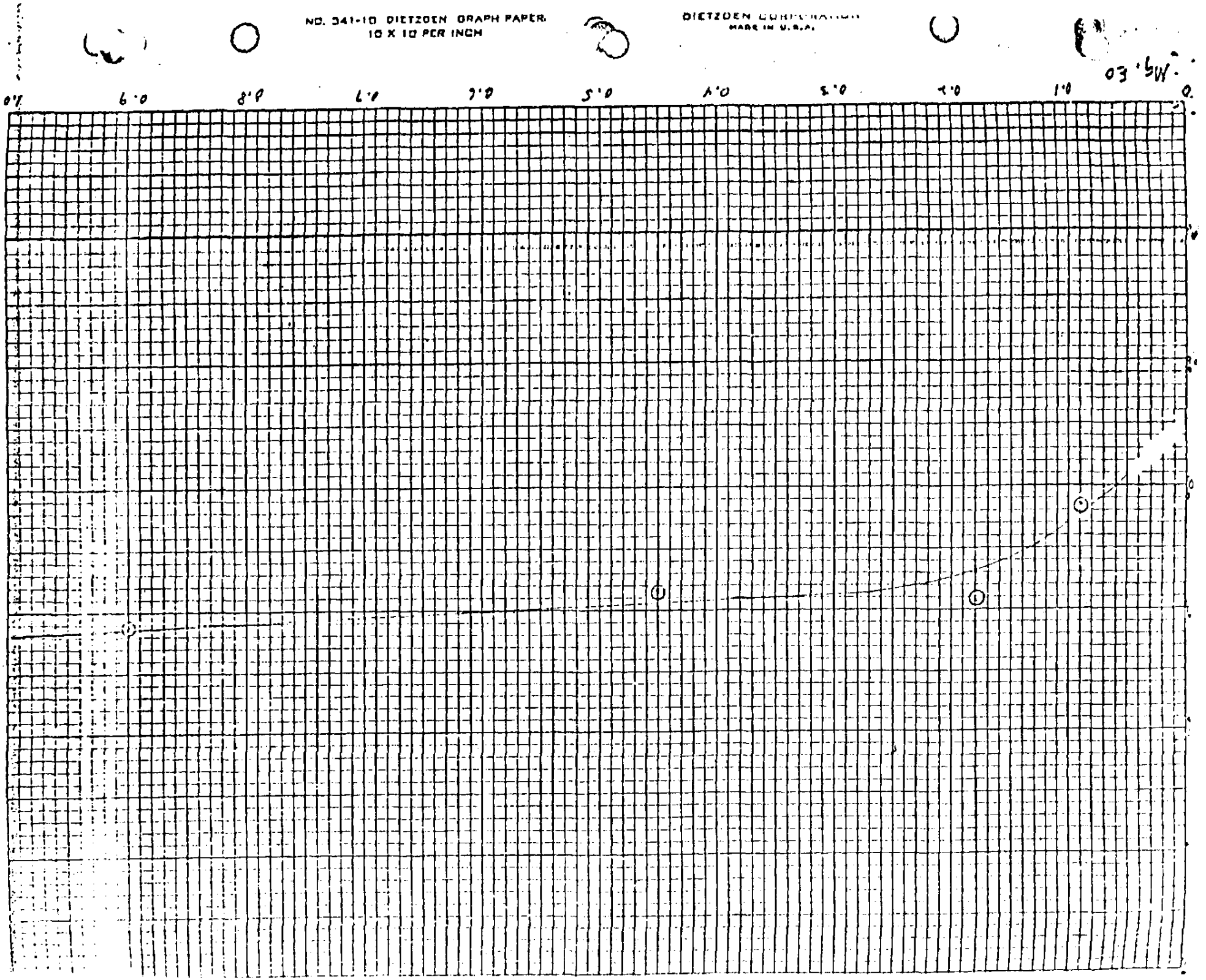

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