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A Comparison of Three Prospective Analytical Methods for Benzene Analysis in Jet Fuel Environments

MOHAMMAD A. HOSSAIN, Major, USAF, BSC

August 1990

Final Report

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AF Occupational and Environmental Health Laboratory (AFSC)
Human Systems Division
Brooks Air Force Base, Texas 78235-5501

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MOHAMMAD A. HOSSAIN, Major, USAF, BSC
Chief, Occupational Chemistry Branch

THOMAS C. THOMAS, GM-15
Chief, Analytical Services Division

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13. ABSTRACT (Maximum 200 words)
Accurate analysis of benzene in jet fuel has been a concern over the past several years. The method we have been using to analyze benzene in jet fuel is the NIOSH 1501 method, a method specifically designed for aromatic hydrocarbons including benzene. However, the method is not designed for analysis of benzene in jet fuel environments. At the present time there is no approved (either by NIOSH or OSHA) method for analysis of benzene in fuel environments. At the request of HQ AAC/SGPB, we recently conducted a study to compare three prospective analytical methods (NIOSH method 1501 [GC/FID with packed column], modified NIOSH 1501 method [GC/FID with capillary column], and High Pressure Liquid Chromatography with Ultraviolet Detection [HPLC/UV]). In this study spiked charcoal tube samples as well as air samples of known concentrations of benzene in JP-4 and Stoddard Solvents were analyzed by all three methods. The test results showed that modified NIOSH 1501 and HPLC methods had good correlation between spiked and measured amount of benzene in JP-4 and Stoddard Solvent mixtures. The NIOSH 1501 method utilizing packed column over estimated the test benzene concentration indicating positive interference from other hydrocarbons present in JP-4 and Stoddard Solvents. KW

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Contents

I. INTRODUCTION

A. Background: HQ AAC/SGPB letter dated 20 Oct 89 requested the Air Force Occupational and Environmental Health Laboratory (AFOEHL) review and update the current analytical method for benzene in jet fuel environments to ensure the analytical results provided are representative. The letter also indicated that the National Institute for Occupational Safety and Health (NIOSH) method 1501 used for analysis of benzene in jet fuels identified unusually high workplace concentrations of benzene. The NIOSH 1501 method, specifically designed for aromatic hydrocarbons including benzene, utilizes a glass, 3.0 m x 2 mm, 10% OV-275 on 100/120 mesh Chromosorb W-AW or equivalent column in conjunction with the gas chromatography/flame ionization detector (GC/FID). This method is not designed for analysis of benzene in jet fuel environments. At the present time there is no approved (either by NIOSH or OSHA) method for analysis of benzene in fuel environments. However, the Occupational Safety and Health Administration (OSHA) laboratory at Salt Lake ity, Utah utilizes a capillary column instead of a packed column as specified in the NIOSH method to analyze benzene in complex mixtures.

B. Our Consultative Letter, CL 89-131SA0687LAE dated 1 Dec 89 discussed the validity of the NIOSH method 1501 and other methods for analysis of benzene in complex mixtures such as jet fuel. The letter also stated that a study to compare three prospective methods (NIOSH method 1501 [Gas chromatography/flame ionization detector with packed column], OSHA method [GC/FID with capillary column], and high-pressure liquid chromatography with ultraviolet detection) would be accomplished.

II. STUDY PROCEDURES

A. Between December 1989 and June 1990 we conducted a study to compare three prospective analytical methods: NIOSH method 1501 (GC/FID with packed column), OSHA method (GC/FID with capillary column), and High Pressure Liquid Chromatography with Ultraviolet Detection (HPLC/UV). In this study, spiked charcoal tube samples with known amounts of benzene in JP-4 and in Stoddard solvents were analyzed by all three methods. Air samples with known concentrations of benzene in JP-4 vapor, generated in a dynamic flow dilution system at the industrial hygiene laboratory, were also collected in charcoal tubes and analyzed by the same methods. However, our air sampling study was somewhat limited.

B. For each test, two or more samples were prepared for each analytical method at the same concentrations.

III. ANALYTICAL METHODS

A. For HPLC analysis, Hewlett-Packard model 1084B liquid chromatograph equipped with a 254 nm UV detector was used. A Supelco 25 cm x 4.6 mm column of 5 um LC-PAH was used for separations. The mobile phase used was a mixture of water and acetonitrile. All spiked charcoal tubes were desorbed for 30 minutes in a 2 mL portion of 40% ethyl acetate in methylene chloride.

B. A Hewlett-Packard model 5880A gas chromatograph equipped with flame ionization detector was used for all GC analysis. Two separate columns (packed and capillary) were used to compare the separation of benzene in a complex mixture such as JP-4 & Stoddard solvents. The packed column was a Supelco 6.1 m (20 ft) x 3.2 mm (1/8 inch) stainless steel column of 10% FFAP on acid washed, 100/120 mesh Chromosorb W. The capillary column was a Supelco 30 m x 0.53 mm ID, DB-1 Wide Bore column with a 5 μm film thickness.

C. An Acculab model 8 Infrared Spectrometer (Beckman Instruments) with a 0.1 mm thickness Potassium Bromide (KBr) cell was used to determine benzene concentrations in JP-4 and Stoddard solvents. The percentage of benzene in bulk JP-4 and Stoddard solvents were measured as 0.53% and none detected, respectively.

IV. RESULTS AND DISCUSSION

The test data and the analytical results are presented in Appendix A, Table I. The average, standard deviation, and coefficient of variation were calculated for each test and summarized in Appendix A, Table II. Linear regression lines and the 95% confidence limits for the predicted means were calculated for the relationship between the spiked amount and the measured amount of benzene for each analytical method and plotted in Figures 1, 2 & 3. The slopes, intercepts and correlation coefficients of regression lines between the spiked amount and measured amount of benzene for three analytical methods were: (a) slopes (0.73 for HPLC, 1.68 for GC/FID [packed column], and 1.10 for GC/FID [capillary column]); (b) intercepts (-5.80 for HPLC, 266 for GC/FID [packed column], and -4.47 for GC/FID [capillary column]); and (c) correlation coefficients (0.96 for HPLC, 0.43 for GC/FID [packed column], and 0.88 for GC/FID [capillary column]). These results indicated that HPLC and GC/FID (capillary column) methods had good correlation between the spiked and measured amount of benzene. The overall performances for GC/FID (packed column) was unsatisfactory. As is clear from Figure 2, the GC/FID (packed column) analytical data are scattered and well above the spiked amount over the range. This method overestimated the test benzene concentration indicating positive interference from other hydrocarbons present in the jet fuel environments. In fact, the packed column method reported large amounts (averaged 688.3 micrograms) of benzene for the spiked samples of Stoddard solvents (with no benzene) and failed to differentiate between benzene and other compounds of similar retention time as benzene.

Figure 4 presents regression lines for all three methods. The points shown in this figure are the spiked and measured amounts of benzene averaged for each set of tests.

Analysis of variance (ANOVA) and student's t distribution (t-test) were performed to test the hypothesis concerning the linearity, slope and intercept of regression line. All tests were performed at 95% confidence interval (i.e. inference of $x = 0.05$). Summaries of ANOVA, t-test and regression analysis for all three methods are shown in Appendix A, Table III. The GC/FID (packed column) data while accepting the null hypotheses of linearity and slope equals to one ($b = 1$) rejected hypothesis of intercept equals to zero ($a = 0$). The intercept of 266 and correlation coefficient of 0.43 clearly indicated poor correlation and is unacceptable for analytical method of benzene in jet fuel environments. The HPLC and GC/FID (capillary column) data accepted the null hypothesis of linearity and correlated well ($r = 0.96$ & 0.88, respectively) with the spiked data. The HPLC data also accepted the hypothesis of intercept equals to zero ($a = 0$), but rejected the hypothesis of slope equals to one ($b = 1$). On the other hand, the GC/FID (capillary column) data accepted both hypotheses of intercept equals to zero ($a = 0$) and slope equals to one ($b = 1$). We accept both HPLC and GC/FID (capillary column) methods as valid for the analysis of benzene in jet fuel environments. However, it is clearly evident that the GC/FID (capillary column) method provided better accuracy than the HPLC method.

V. CONCLUSIONS

On the basis of the test results, the following was concluded:

1. The NIOSH method 1501 utilizing GC/FID with packed column showed positive interference in analyzing benzene in jet fuel environments and thereby could lead to incorrect exposure assessments for workers.

2. Both HPLC and modified NIOSH method 1501 utilizing capillary column provided reasonable accuracy in determining benzene concentration in a JP-4 environment.

3. The GC/FID (capillary column) method showed a better accuracy than the HPLC method while comparing the test and the measured concentrations of benzene in jet fuel environments.

4. The lower detection limits of benzene using HPLC and GC/FID (capillary column) methods were noted as four and two micrograms, respectively. Based on these detection limits, the minimum sample volumes required to detect one-tenth of PEL for benzene (3.0 mg/m^3) were calculated as 13.3 and 6.7 liters (L), respectively.

5. Since the HPLC utilizing a UV detector set at 254 nm will not detect aliphatic or alicyclic hydrocarbons, the measurement of total hydrocarbons in jet fuel environments need to be performed by ancther method such as GC/FID (capillary column) method.

6. The GC/FID (capillary column) method will be able to measure both benzene and total hydrocarbons in jet fuel environments.

VI. RECOMMENDATIONS

A. Based on the results of this study, we recommend the use of the modified NIOSH 1501 method (GC/FID with capillary column) for analyzing benzene in jet fuel environments or other complex mixtures. The minimum recommended sample volume specified in the NIOSH method 1501 should be increased to 6.7 liters instead of 2 liters for a detection limit of one-tenth of benzene PEL (i.e., detection limit of 0.3 mg/m^3). If lower sensitivity is accepted, the following sample volumes will be required:

Minimum Sample Volume	Detection Limit
3.3 liters	20% PEL (i.e. 0.60 mg/m^3)
2.7 liters	25% PEL (i.e. 0.75 mg/m^3)
1.4 lites	50% PEL (i.e. 1.50 mg/m^3)

B. Modify the 1989 Sampling Guide (pages III-10, 11) with the following information:

NAME	COLLECTION METHOD	SAMP RATE MIN - MAX		RECMD VOL MIN - MAX	NOTES	REMARKS	REFERENCE
Benzene	CTXX-101	0.02	0.2	6.7 - 30	J	6.7L sample volume is required for a detection limit of 0.3 mg/m^3. For lower sensitivity, such as 0.75 mg/m^3 detection limit (25% PEL), 2.7L sample volume is required.	Modified N1501C

SPIKED VS MEASURED BENZENE BY HPLC

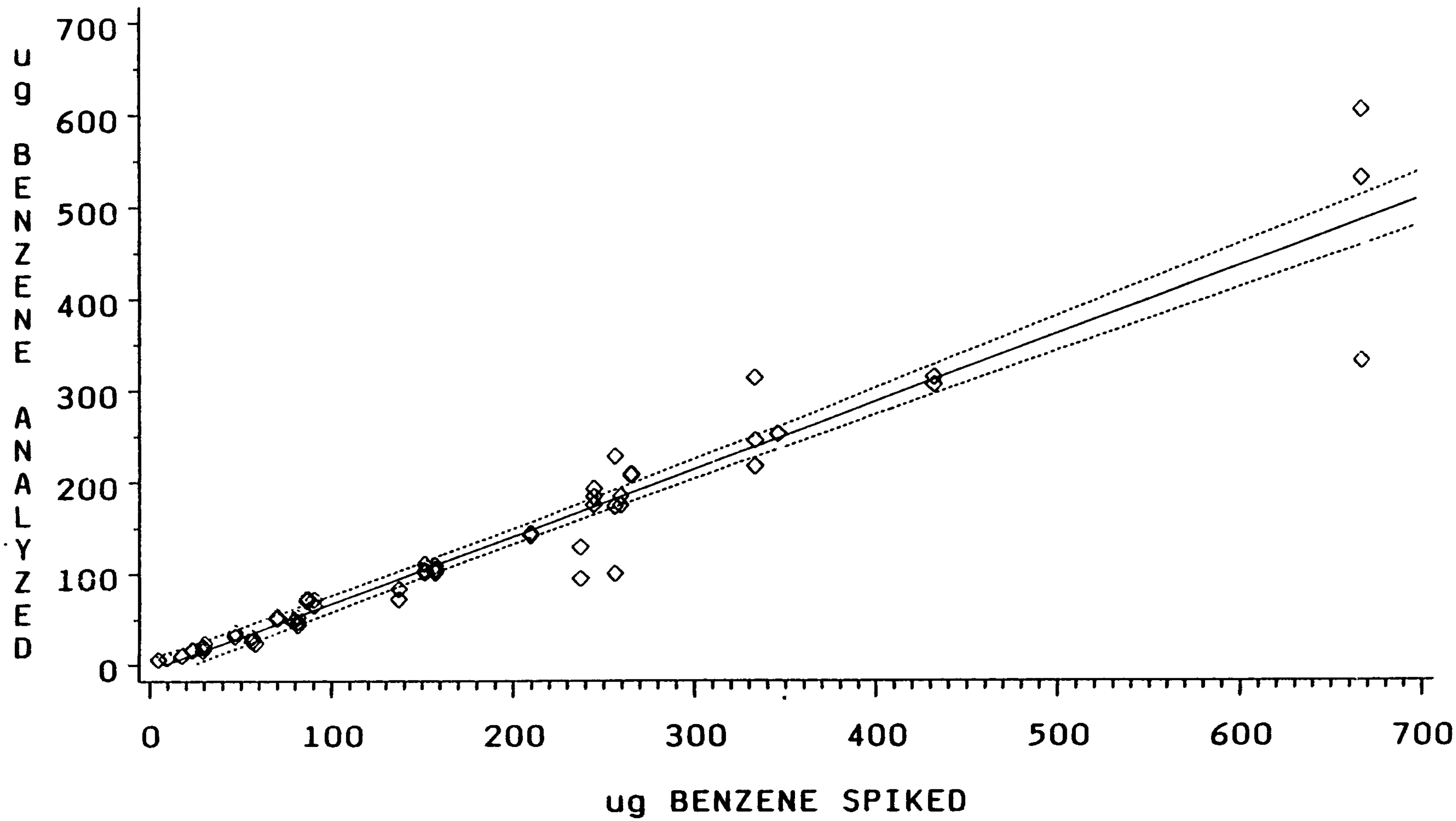

Figure 1. Spiked vs measured benzene by HPLC. The solid line is the linear regression line and the broken lines are the 95% confidence limits for the predicted means.

SPIKED VS MEASURED BENZENE BY GC/FID [PACKED COLUMN]

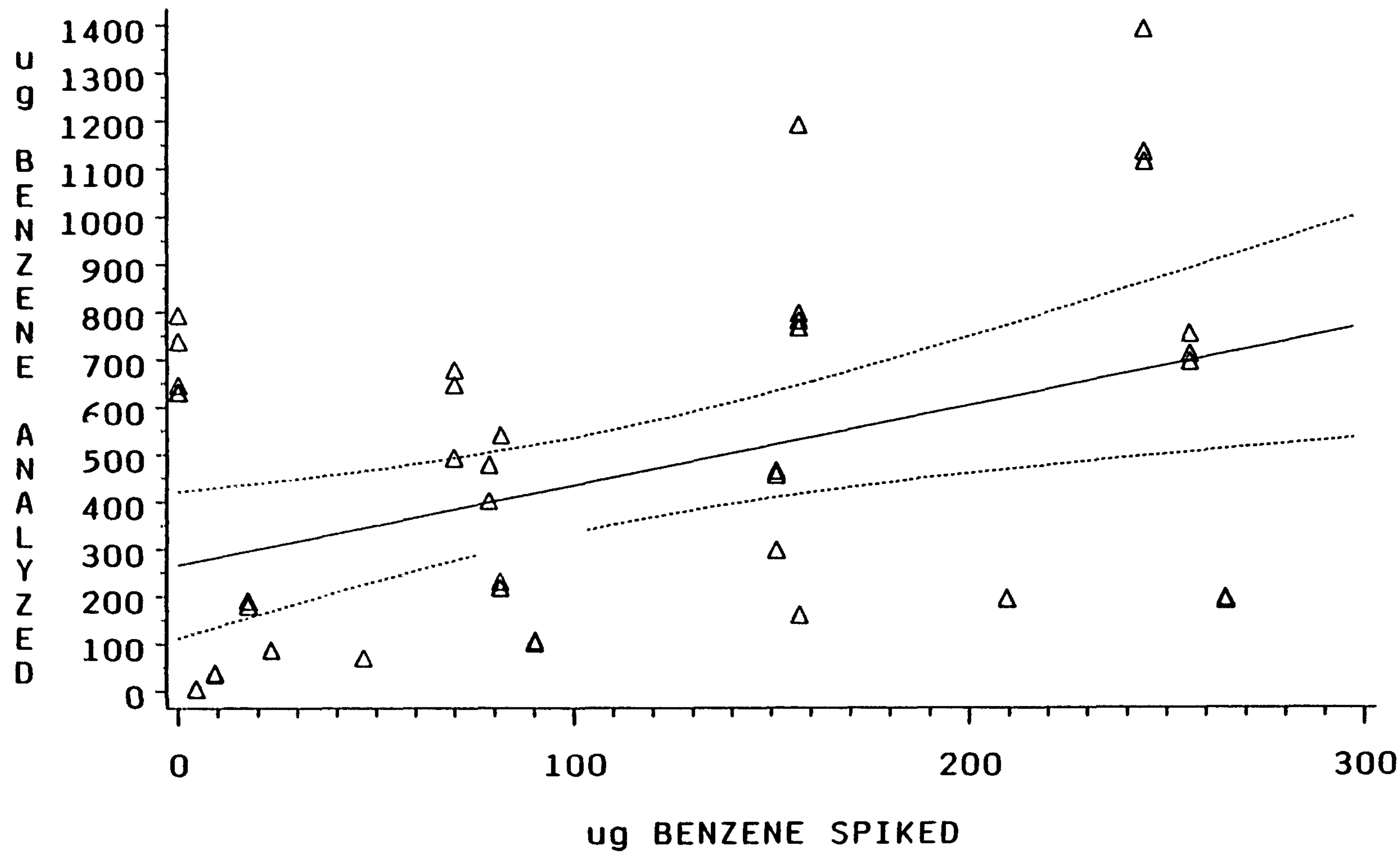

Figure 2. Spiked vs measured benzene by GC/FID [packed column]. The solid line is the linear regression line and the broken lines are the 95% confidence limits for the predicted means.

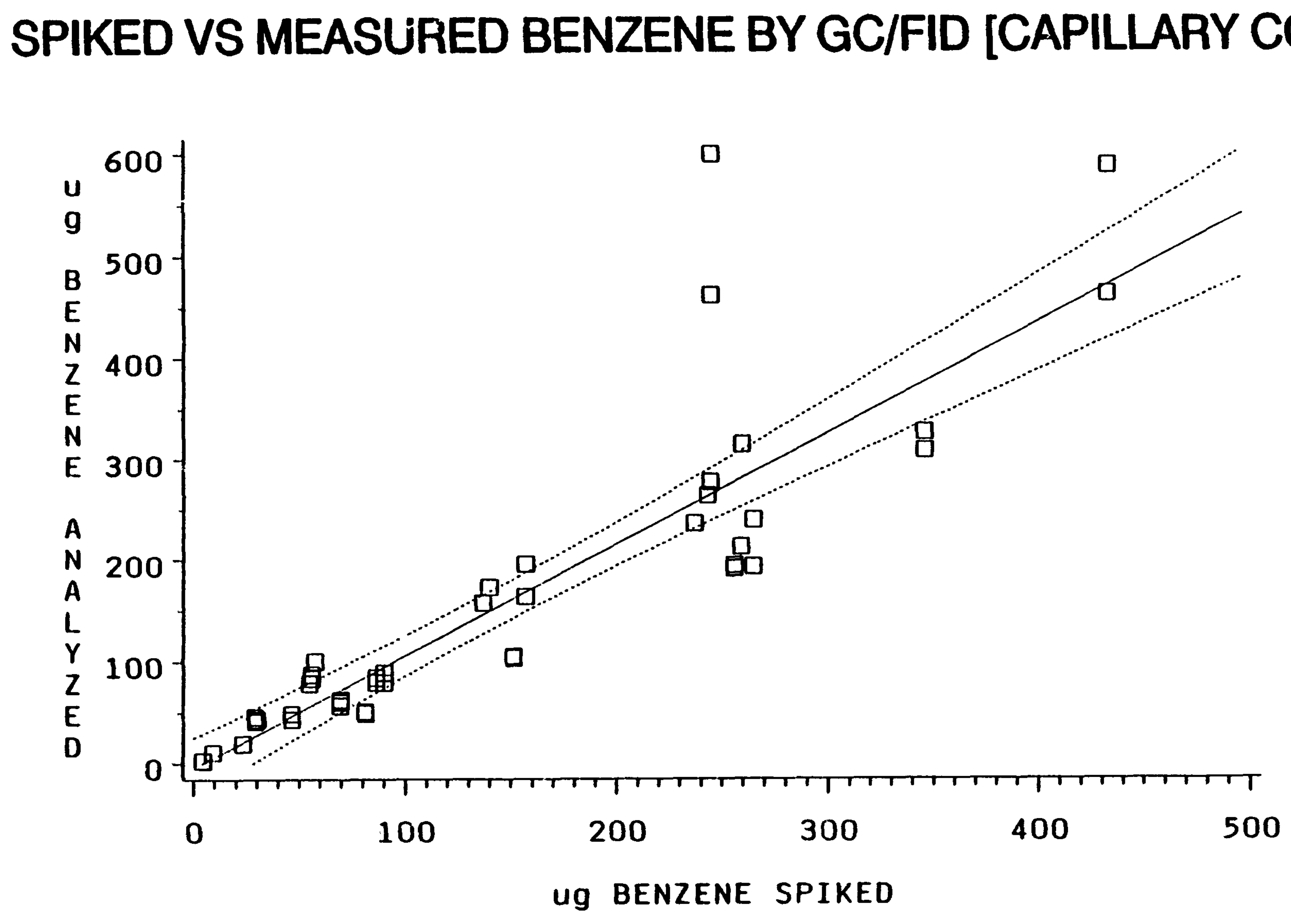

Figure 3. Spiked vs measured benzene by GC/FID [capillary column]. The solid lire is the linear regression line and the broken lines are the 95% confidence limits for the predicted means.

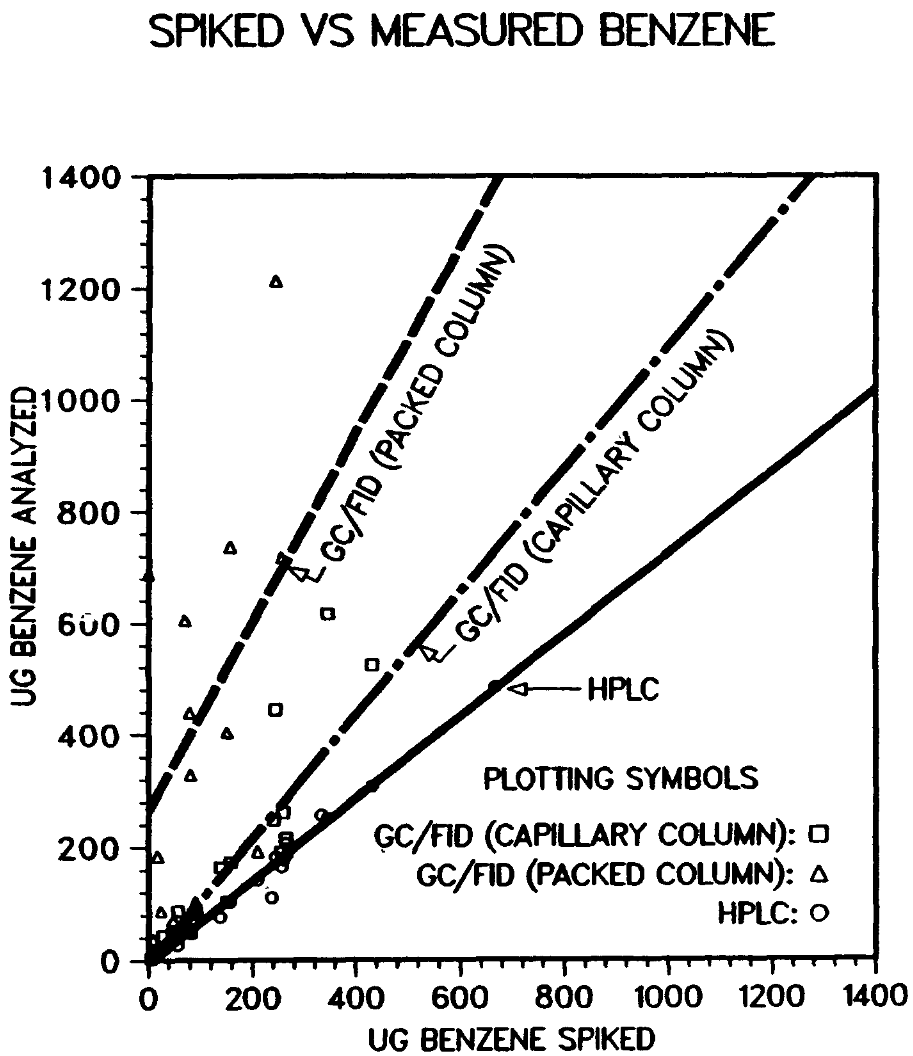
SPIKED VS MEASURED BENZENE
UG BENZENE ANALYZED
UG BENZENE SPIKED
0
200
400
600
800
1000
1200
1400
GC/FID (PACKED COLUMN)
GC/FID (CAPILLARY COLUMN)
HPLC
PLOTTING SYMBOLS
GC/FID (CAPILLARY COLUMN): □
GC/FID (PACKED COLUMN): △
HPLC: ○

Figure 4

Appendix A

Test Results and Statistical Analysis

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Table I

Test Results
for
JP-4/Benzene Study

Methods	Actual Amount of Benzene, μg	Measured Amount of Benzene, μg — Analytical Methods		
		HPLC	GC/FID (packed column)	GC/FID (capillary column
JP-4 (@ 0.53% Benzene) Spiked in Charcoal Tube	4.7	5.8	5.5	3.0
	4.7	5.2	5.7	2.5
	4.7	<4*	5.2	2.8
	9.3	6.9	39.4	11.0
	9.3	6.7	35.9	11.0
	23.3	17.0	88.7	19.4
	23.3	15.6	86.2	19.5
	46.6	31.6	70.9	49.0
	46.6	35.6	70.0	43.3
JP-4 + Known Amount of Benzene Spiked in Charcoal Tube	81.5	47.1	231.1	51.2
	81.5	52.4	540.6	50.1
	81.5	43.7	217.2	49.2
	90.2	65.3	101.9	88.9
	90.2	71.7	106.4	79.2
	151.4	103.0	296.0	104.2
	151.4	101.2	464.0	105.1
	151.4	110.0	455.3	103.2
	256.1	99.5	710.1	194.2
	256.1	172.9	752.0	191.5
	256.1	227.0	693.4	190.8
	264.9	206.6	194.6	239.0
	264.9	208.2	190.0	192.6

Table I Cont'd

Methods	Actual Amount of Benzene, µg	Measured Amount of Benzene, µg — Analytical Methods: HPLC	GC/FID (packed column)	GC/FID (capillary column)
Stoddard Solvent	0	<4*	632.6	<2*
(No Benzene) Spiked	0	<4*	630.7	<2*
in Charcoal Tube	0	<4*	646.0	<2*
	0	<4*	738.4	-
	0	<4*	793.8	-
Stoddard Solvent	17.5	10.1	179.7	-
(th Known Amount	17.5	9.5	190.1	-
of Benzene) Spiked				
in Charcoal Tube	69.8	50.6	646.9	60.4
	69.8	51.5	492.6	62.6
	69.8	53.3	677.5	56.9
	78.6	48.4	477.7	-
	78.6	50.2	401.7	-
	86.4	70.4	-	79.7
	86.4	73.4	-	84.2
	157.1	101.0	794.7	-
	157.1	102.1	159.4	-
	157.1	104.8	765.3	163.5
	157.1	108.3	1191.0	163.7
	157.1	103.9	780.0	195.3
	209.5	141.4	193.7	-
	209.5	143.7	192.4	-
	244.4	192.1	1113.0	599.3
	244.4	183.3	1134.0	461.3
	244.4	174.6	1391.0	276.4
	259.2	183.8	-	313.8
	259.2	174.3	-	212.4
	345.6	251.5	-	325.7
	345.6	250.9	-	307.7
	432.1	303.8	-	461.7
	432.1	312.3	-	587.8

Table I Cont'd

Methods	Actual Amount of Benzene, µg	Measured Amount of Benzene, µg — Analytical Methods: HPLC	GC/FID (packed column)	GC/FID (capillary column)
Blank	0	<4*	11.1*	-
	0	<4*	10.6*	-
	0	<4*	<2*	<2*
Known Amount of Benzene were collected in Charcoal Tubes using Vapor Generation System	333	215.7	-	-
	333	244.2	-	-
	333	311.9	-	-
	667	600.7	-	-
	667	328.4	-	-
	667	527.0	-	-
	30	23.6	-	43.8
	29	20.1	-	43.7
	29	19.2	-	41.4
	29	15.7	-	42.1
	29	-	-	46.2
	58	23.6	-	100.4
	56	25.8	-	87.3
	56	30.6	-	83.5
	55	-	-	78.6
	137	82.9	-	157.2
	137	71.6	-	-
	140	-	-	172.8
	237	94.3	-	235.7
	237	128.3	-	-
	243	-	-	263.3

* sample results not included in the statistical analysis.

Table II

Statistical Analysis
JP-4/Benzene Study

Methods	Actual Amount of Benzene (μg)	Measured Amount of Benzene, μg — Analytical Methods: HPLC Avg. ± S.D.	HPLC CV (%)	GC/FID (packed column) Avg. ± S.D.	GC/FID (packed column) CV (%)	GC/FID (capillary column) Avg. ± S.D.	GC/FID (capillary column) CV (%)
JP-4 (@ 0.53% Behzene) Spiked in Charcoal Tube	4.7	5.5 ± 0.4	7.7	5.5 ± 0.3	4.6	2.8 ± 0.3	9.1
	9.3	6.8 ± 0.1	2.1	37.7 ± 2.5	6.6	11.0 ± 0	0
	23.3	16.3 ± 1.0	6.1	87.5 ± 1.8	2.0	19.5 ± 0.1	0.4
	46.6	33.6 ± 2.8	8.4	70.5 ± 0.6	0.9	46.2 ± 4.0	8.7
JP-4 + Known Amount of Benzene Spiked in Charcoal Tube	81.5	47.7 ± 4.4	9.2	329.6 ± 183	55.5	50.2 ± 1.0	2.0
	90.2	68.5 ± 4.5	6.6	104.2 ± 3.2	3.1	84.1 ± 6.9	8.2
	151.4	104.7 ± 4.6	4.4	405.1 ± 94.6	23.3	104.2 ± 1.0	0.9
	256.1	166.5 ± 64.0	38.4	718.5 ± 30.2	4.2	192.2 ± 1.8	0.9
	264.9	207.4 ± 1.1	0.5	192.3 ± 3.3	1.7	215.8 ± 32.8	15.2
Stoddard Solvent (No Benzene) Spiked in Charcoal Tube	0	<4		688.3 ± 73.9	10.7	<2	

Table II Cont'd

Methods Methods	Actual Amount of Benzene (μg)	Measured Amount of Benzene, μg — Analytical Methods: HPLC Avg. ± S.D.	HPLC CV (%)	GC/FID (packed column) Avg. ± S.D.	GC/FID (packed column) CV (%)	GC/FID (capillary column) Avg. ± S.D.	GC/FID (capillary column) CV (%)
Stoddard Solvent (with Known Amount of Benzene) Spiked in Charcoal Tube	17.5	9.8 ± 0.4	4.3	184.9 ± 7.4	4.0	-	
	69.8	51.8 ± 1.4	2.7	605.7 ± 99.1	16.4	60.0 ± 2.9	4.8
	78.6	49.3 ± 1.3	2.6	439.7 ± 53.7	12.2	-	
	86.4	71.9 ± 2.1	3.0	-		82.0 ± 3.2	3.9
	157.1	104.0 ± 2.8	2.7	738.1 ± 369	50.0	174.2 ± 18.3	10.5
	209.5	142.6 ± 1.6	1.1	193.1 ± 0.9	0.5	-	
	244.4	183.3 ± 8.8	4.8	1213 ± 155	12.8	445.7 ± 162	36.4
	259.2	179.1 ± 6.7	3.8	-		263.1 ± 71.7	27.3
	345.6	251.2 ± 0.4	0.2	-		616.7 ± 12.7	4.0
	432.1	308.1 ± 6.0	2.0	-		524.8 ± 89.2	17.0
Known Amount of Benzene were collected in charcoal Tubes Using Vapor Generation System	333	257.3 ± 49.4	19.2	-		-	
	667	485.4 ± 141	29.0	-		-	
	29.3	19.7 ± 3.2	16.5	-		-	
	29.2	-		-		43.4 ± 1.9	4.3
	56.7	26.7 ± 3.6	13.4	-		-	
	56.3	-		-		87.5 ± 9.3	10.7
	137.0	77.3 ± 8.0	10.3	-		-	
	138.5	-		-		165.0 ± 11.0	6.7
	237.0	111.3 ± 24.0	21.6	-		-	
	240	-		-		249.5 ± 19.5	7.8

Note: Avg. = Average of two or more spiked samples
S.D. = Standard Deviation
CV = Coefficient of Variation

Table III

Least-Square Fit and Regression Analysis of Test Data

Least-Squares	HPLC vs Spiked (n=63)	GC/FID* (p.c) vs Spiked (n=44)	GC/FID** (c.c) vs Spiked (n=52)
Slope, b	0.73	1.68	1.10
Intercept, a	-5.80	266	-4.47
Correlation Coefficient, r	0.963	0.428	0.881
Inference (x=0.05)			
Linear	Accept	Accept	Accept
b=1 (Slope)	Reject	Accept	Accept
b=0 (Slope)	Reject	Reject	Reject
a=0 (Intercept)	Accept	Reject	Accept

* p.c = packed column
** c.c = capillary column

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