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A PRELIMINARY COMPARISON OF THERMAL DECOMPOSITION PRODUCTS OF AIRCRAFT INTERIOR MATERIALS USING THE NATIONAL BUREAU OF STANDARDS SMOKE CHAMBER AND THE COMBUSTION TUBE FURNACE

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METRIC CONVERSION FACTORS

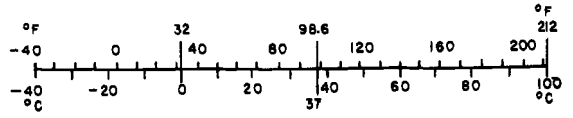
Approximate Conversions to Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
in	inches	*2.5	centimeters	cm
ft	feet	30	centimeters	cm
yd	yards	0.9	meters	m
mi	miles	1.6	kilometers	km
		AREA		
in^2	square inches	6.5	square centimeters	cm^2
ft^2	square feet	0.09	square meters	m^2
yd^2	square yards	0.8	square meters	m^2
mi^2	square miles	2.6	square kilometers	km^2
	acres	0.4	hectares	ha
		MASS (weight)		
oz	ounces	28	grams	g
lb	pounds	0.45	kilograms	kg
	short tons (2000 lb)	0.9	tonnes	t
		VOLUME		
tsp	teaspoons	5	milliliters	ml
Tbsp	tablespoons	15	milliliters	ml
fl oz	fluid ounces	30	milliliters	ml
c	cups	0.24	liters	l
pt	pints	0.47	liters	l
qt	quarts	0.95	liters	l
gal	gallons	3.8	liters	l
ft^3	cubic feet	0.03	cubic meters	m^3
yd^3	cubic yards	0.76	cubic meters	m^3
		TEMPERATURE (exact)		
°F	Fahrenheit temperature	5/9 (after subtracting 32)	Celsius tamperature	°C

*1 in = 2.54 (exactly). For other exact conversions and more detailed tables, see NBS Misc. Publ. 286, Units of Weights and Measures, Price $2.25, SD Catalog No. C13.10:286.

Approximate Conversions from Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
mm	millimeters	0.04	inches	in
cm	centimeters	0.4	inches	in
m	meters	3.3	feet	ft
m	meters	1.1	yards	yd
km	kilometers	0.6	miles	mi
		AREA		
cm^2	square centimeters	0.16	square inches	in^2
m^2	square meters	1.2	square yards	yd^2
km^2	square kilometers	0.4	square miles	mi^2
ha	hectares (10,000 m^2)	2.5	acres	
		MASS (weight)		
g	grams	0.035	ounces	oz
kg	kilograms	2.2	pounds	lb
t	tonnes (1000 kg)	1.1	short tons	
		VOLUME		
ml	milliliters	0.03	fluid ounces	fl oz
l	liters	2.1	pints	pt
l	liters	1.06	quarts	qt
l	liters	0.26	gallons	gal
m^3	cubic meters	35	cubic feet	ft^3
m^3	cubic meters	1.3	cubic yards	yd^3
		TEMPERATURE (exact)		
°C	Celsius temperature	9/5 (then add 32)	Fahrenheit temperature	°F



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16. Abstract

Twelve aircraft interior materials were burned under standard flaming combustion conditions in the National Bureau of Standards (NBS) smoke chamber. Each material was also exposed to 600° Celsius (C) in a combustion tube furnace under conditions of oxidative pyrolysis. The combustion products were collected in liquid-filled impingers, and the contents were analyzed for hydrogen cyanide, hydrogen sulfide, hydrogen chloride, hydrogen bromide, and formaldehyde by differential pulse polarography, nitrogen dioxide and sulfur dioxide by visible spectrophotometry, and hydrogen fluoride by ion-selective electrode. Carbon monoxide was measured directly by a nondispersive infrared analyzer. The yields of the nine gases are reported in terms of milligrams per gram of material. The toxic gas yields were obtained for each material in the NBS smoke chamber and were compared to the yields obtained in the combustion tube furnace. The yields were also compared to those obtained using colorimetric detector tubes in the NBS smoke chamber. The extent of the correlation of the various procedures is reported. The relative yields of $HC\ell$, HCN, and H_2S were found to be somewhat independent of the exposure conditions. The relative yields of the oxidized gases, CO, HCHO, NO_2, and SO_2, are much more dependent on the exposure conditions.

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PREFACE

The authors would like to acknowledge the assistance provided by Mr. Ross Glidewell and Mr. Stanley Sternik in acquiring laboratory data. We would also like to thank Mr. Louis Allen for reviewing the statistics in this report.

The materials utilized in this study were obtained through the cooperation of the Aerospace Industries Association of America and leading aircraft industry seat material suppliers.

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INTRODUCTION

PURPOSE.

The primary purpose of this report is to compare selected toxic gas yields of aircraft interior materials using the National Bureau of Standards (NBS) smoke chamber and the combustion tube furnace. A secondary objective is to compare selected toxic gas yields obtained with colorimetric detector tubes in the NBS smoke chamber with the yields obtained using instrumental methods of analysis.

BACKGROUND.

Two previous studies conducted at the National Aviation Facilities Experimental Center (NAFEC) have involved the analysis of toxic gases generated by thermally decomposing aircraft interior materials. These tests have included 66 materials and nine material components. In the first study (reference 1), the materials were exposed to flaming conditions in the NBS smoke chamber, and selected toxic gases were measured with colorimetric detector tubes. A more recent study (reference 2) involved the thermal decomposition of the same materials in a combustion tube furnace at 600° Celsius (C). Instrumental methods of analysis were employed in the latter study to examine the various thermal decomposition products. In addition, the Civil Aeromedical Institute (CAMI) has conducted animal toxicity tests by thermally decomposing these materials in a combustion tube furnace (reference 3). Both the recent NAFEC toxic gas program and the animal toxicity studies at CAMI employed similar experimental procedures to generate thermal decomposition products.

It is generally accepted that exposure conditions play a significant role in determining both the qualitative and quantitative aspects of combustion products (references 1 and 4). It can be argued that a combustion tube furnace is not an appropriate means of generating combustion products, since the thermal environment is assumed to differ rather significantly from that encountered during flaming combustion. The purpose of this study is to compare the thermal decomposition products that are produced using a combustion tube furnace with those obtained under flaming exposure conditions in the NBS smoke chamber. This comparison is based on the qualitative and quantitative analysis of eight thermal decomposition products produced by 12 randomly selected aircraft interior materials.

EXPERIMENTAL SECTION

AIRCRAFT INTERIOR MATERIALS.

The materials utilized in this study were chosen from among those interior materials which were used in wide-bodied aircraft during 1972/1973. Many of these materials are still in current use. The 12 test materials were selected

from materials that were analyzed in previous studies (references 1, 2, and 3), and include panels (4), fabrics (6), and carpets (2). The chemical and physical characteristics of the materials, including their usage categories, are described in table 1.

TABLE 1. DESCRIPTION OF MATERIALS

No.	Chemical Composition	Thickness (inch)	Unit Weight (oz/yd^2)	Designation	Cabin Use
1	PVF/Epoxy-Fiberglas/ Aramid Honeycomb/ Epoxy-Fiberglas	0.388	48.5	Panel	Ceiling panel
33	Wool Pile/Polyester Backing/ Latex Coating	0.265	51.8	Flooring	Carpet
34	Wool Pile/Polyester Backing/ Latex Coating/Urethane Pad	0.345	51.3	Flooring	Carpet
43	PVF/Phenolic-Fiberglas Screen/Aramid Honeycomb/ Aramid Honeycomb filled with Phenolic-Fiberglas Batt/Phenolic-Fiberglas	0.732	85.8	Panel	Drop ceiling panel
61	PVF/PVC/Phenolic-Fiberglas/ Epoxy Adhesive/Aramid Honeycomb/Epoxy Adhesive/ Phenolic-Fiberglas	0.500	60.1	Panel	Overhead stowage panel
70	FR Wool (90 percent)/ Nylon (10 percent)	0.037	11.3	Fabric	Upholstery
78	Aromatic Polyamide	0.046	12.1	Fabric	Upholstery
82	FR Wool (76 percent)/PVC (24 percent)	0.039	12.6	Fabric	Upholstery
88	FR Wool	0.055	17.2	Fabric	Upholstery
92	Aromatic Polyamide	0.036	11.8	Fabric	Upholstery
142	FR Wool (90 percent)/ Nylon (10 percent)	0.035	10.3	Fabric	Upholstery
144	PVF/Epoxy-Fiberglas/ Aramid Honeycomb/ Epoxy-Fiberglas	0.276	43.3	Panel	Wall panel

FR - Flame Retardant Treated
PVC - Polyvinyl Chloride
PVF - Polyvinyl Fluoride

All materials were cut to approximate size and placed in a conditioning chamber at 50-percent relative humidity and 70° Fahrenheit (F) (21.1° C) for at least 24 hours. The materials were then reweighed prior to testing. Sample weights for the combustion tube were 250 ±5 milligrams (mg). The samples for the NBS smoke chamber were cut to fit a standard 2 9/16-inch-square sample holder.

COMBUSTION TUBE FURNACE.

Since details of the experimental procedures have been described in a previous report (reference 2), only a brief summary of the procedure is presented. A 250-mg sample of material was exposed to 600° C in a combustion tube furnace. The material was heated in a 2/3-inch (1.7 centimeters (cm)) diameter Vycor® tube for 5 minutes while ambient air was drawn through the combustion tube at a rate of 2 liters per minute (ℓpm) with a laboratory vacuum pump. The combustion gases were collected in liquid-filled bubblers, each containing an absorbing solution appropriate for the gases to be analyzed. Carbon monoxide (CO) was collected for analysis by replacing the liquid-filled bubbler with a 12-liter Saran® sample bag while maintaining the airflow with a tank of purified air. Three replicate tests were made on each material, and the reported gas yields are the average of the three tests.

NBS SMOKE CHAMBER.

The materials were tested under standard flaming exposure conditions (reference 5) (radiant heat plus flamlets) in order to more closely simulate flaming combustion. The glass in the chamber door was covered with a transparent Teflon® film to prevent etching of the glass by hydrogen fluoride (HF). The gas sampling apparatus consisted of four impingers placed inside the NBS smoke chamber.

The inlet of each impinger was positioned near the geometric center of the chamber. Glass open-tip impingers were used for the collection of all gases except HF. A fritted polypropylene bubbler was used for the collection of HF. The airflow through each impinger was maintained at 1 ℓpm for 10 minutes by four rotameters equipped with high-accuracy needle values. Each rotameter was protected from tars by a cold trap and an absorption tube containing a layer each of Drierite® and activated charcoal. A description of the collection medium in each impinger and the combustion gases that were collected in it is contained in table 2. Carbon monoxide concentrations were monitored continuously with a nondispersive infrared analyzer (NDIR). The CO sampling probe was also located near the geometric center of the chamber. The combustion products passed through a particulate filter and a cold trap at -8° C before entering the CO analyzer. Three replicate tests were performed on each material, and the reported gas yields are the averages of the three tests.

In addition, CO yields were obtained under nonflaming conditions (radiant heat only) for six of the materials.

TABLE 2. GAS SAMPLING PROCEDURES FOR NBS SMOKE CHAMBER

Impinger	Collection Medium	Toxic Gases
1	25-mℓ modified Griess-Saltzman Reagent +0.25-mℓ acetone (reference 6)	NO_2
2	25 mℓ of .05 M NaOH	HCN, H_2S, HCℓ, HBr
3	25 mℓ of .04 M tetrachloromercurate (reference 6)	SO_2
4	(a) 10 mℓ of 1-percent $NaHSO_3$ (b) 100 mℓ of .05 M NaOH (fluoride containing materials)	HCHO HF

Details of the experimental procedure in which colorimetric detector tubes were employed have been described in a previous report (reference 1). Only a brief summary of the procedure is presented. Bag samples were taken periodically from the chamber, and gas analysis was performed after the test. This procedure was followed for CO, hydrogen cyanide (HCN), hydrogen sulfide (H_2S), formaldehyde (HCHO), sulfur dioxide (SO_2), and the nitrogen oxides (NO_x). Hydrogen chloride (HCℓ) and HF were measured directly inside the chamber. The HCℓ and HF detector tubes were placed at the geometric center of the chamber attached to plastic tubing passing through the ceiling to a hand pump. Samples were taken once a minute for a duration of 7 minutes after the initiation of the test.

METHODS OF ANALYSIS.

The contents of the impingers were analyzed for HCN, HCℓ, hydrogen bromide (HBr), and HCHO by differential pulse polarography; NO_2 and SO_2 by visible spectrophotometry; and HF by ion-selective electrode. The instrumental methods of analysis employed for each of the nine gases are summarized in table 3.

A Princeton Applied Research (PAR) model 174A polarographic analyzer, equipped with a model 172A drop timer and a model 315 Automated Electroanalysis Controller was used for the determination of HCN, H_2S, HCℓ, HBr, and HCHO concentrations. The polarograph was operated in the differential pulse mode using a three electrode configuration which included a dropping-mercury working electrode, a platinum ribbon counter electrode, and a saturated calomel reference electrode (SCE). The SCE was isolated from the sample solution by a 1.0 molar (M) sodium nitrate salt bridge. Instrumental parameters include a scan rate of 1 millivolt per second (mV/s) for HCN, H_2S, HCℓ, and HBr; 2 mV/s for HCHO; a drop time of 1 second; and pulse amplitude of 10 mV.

Infrared analyzers were used for the analysis of CO. A Beckman model 864 nondispersive CO Infrared Analyzer was used for the combustion tube furnace,

TABLE 3. ANALYTICAL PROCEDURES

Instrumentation	Combustion Method	Toxic Gas	Analytical Procedure
PAR model 174A Polarographic Analyzer (differential pulse mode)	Combustion Tube Furnace and NBS Smoke Chamber	HCN, H_2S	Add 1-mℓ sample to 9-mℓ deaerated 0.05 M NaOH; Scan from -0.90 V to -0.15 V vs. SCE.
		HCℓ, HBr	Add 1-mℓ sample to 9-mℓ deaerated 0.05 M NaOH; Acidify with 0.10 mℓ of 10.0 M HNO_3; Scan from 0.0 V to +0.40 V vs. SCE.
		HCHO	Add 1-mℓ sample to 9-mℓ deaerated 0.05 M NaOH; Scan from -1.50 V to -1.80 V vs. SCE.
Coleman model 124 Scanning UV/VIS Spectrophotometer	Combustion Tube Furnace and NBS Smoke Chamber	SO_2	Modified West-Gaeke Procedure (reference 6).
		NO_2	Griess-Saltzman Procedure (reference 6).
Orion model 801 pH/millivolt meter with Solid State Fluoride Electrode	Combustion Tube Furnace	HF	(a) Calibration curve for concentrations less than 5×10^{-4} M in acetate buffer (pH5). (b) $La(NO_3)_3$ titration in mixed alcohol acetate buffer.
	NBS Smoke Chamber		(a) Calibration curve in acetate buffer (pH5).
MSA Model 303 infrared analyzer	NBS Smoke Chamber	CO	Continuous monitoring of CO levels.
Beckman model 864 non-dispersive infrared analyzer	Combustion Tube Furnace		Bag sampling prior to analysis.

and a Mine Safety Appliances Model 303 continuous infrared CO analyzer was used for the NBS smoke chamber. An Orion model 801 pH/mV meter with an Orion solid state fluoride electrode and Orion double junction reference electrode were used for the analysis of HF. A Coleman model 124 Scanning UV/VIS spectrophotometer was used for the analysis of SO_2 and NO_2. Colorimetric detector tubes were also employed for measuring gases in the NBS smoke chamber (reference 1).

STATISTICS.

The ability of an equation to describe the test data is best measured by the coefficient of correlation (R) or the coefficient of determination (R^2). In the case of a linear relationship, R^2 is the ratio of the explained variation to the total variation. R^2 is the same regardless of whether X or Y is considered the independent variable. The possible values of R^2 range from 0 to 1.0. A value of 1.0 represents a perfectly correlated data set, while a value of zero represents no linear correlation. However, a high R^2 value (i.e., near 1.0) does not necessarily indicate a direct interdependence of the variables. One also needs to decide whether to regard an observed value of R as a safe indication that the true value of R for the universe is different from zero. This is done by testing to determine if the value of R which was obtained is significantly different from zero at a given probability level ($P\ell$) (reference 7). One can determine confidence limits for estimates obtained from the regression line by using the standard error of estimate (s_{yx}) which is the standard deviation of the errors of estimation. If lines are constructed parallel to the regression line of y on x at the vertical distance of $2s_{yx}$, and if the number of data sets, N, is large enough, 95 percent of the sample points would be included between these lines (reference 8).

The relative yields for each gas are compared between the combustion tube furnace and the NBS smoke chamber. Other conditions which are compared are flaming versus nonflaming conditions in the NBS smoke chamber, and colorimetric detector tube analyses versus instrumental methods of analysis in the NBS smoke chamber. Only nonzero yields are compared to avoid biasing the results. The "least squares best fit" to a straight line was obtained for each comparison. The coefficient of correlation, slope, y-intercept (y_o), and s_{yx} were determined for each regression line. All standard errors of estimates in this report are corrected for the small number of data points employed in the calculations (reference 8). The 95-percent ($2s_{yx}$) confidence bands are indicated as dashed lines in figures 1 to 11.

The R value corresponding to the best fit was determined for each comparison. A Textronix 4051 with the Statistical Package Volume 1 was used to calculate the least squares best fit, the coefficient of correlation, the slope, and the y-intercept. The corrected standard error of estimate was computed from a separate least squares program using a NOVA 3/12 minicomputer.

DISCUSSION OF RESULTS

REPRODUCIBILITY.

The yields of the nine gases are reported in terms of milligrams per gram (mg/g) of material for the 12 materials tested. Peak concentrations reported for the colorimetric detector tubes and continuous CO analyses have been used to calculate equivalent yields in terms of mg/g. This was necessary in order to compare the NBS smoke chamber data with the combustion tube furnace. All other reported yields are average yields, measured from samples taken continuously over the test duration. These data are contained in tables 4 and 5. Although HBr yields are reported in table 4, they have not been utilized for purposes of comparison since this gas was primarily detected only when using the combustion tube furnace.

Data on the average percent-weight-loss of each material are also included in tables 4 and 5. The relative standard deviation (RSD) in the weight loss was determined for the three replicate tests of each material. The RSD is the standard deviation of a set of values divided by the average value. In the combustion tube furnace, small variations in sample weight loss occurred between the three sets of tests. The average of the relative standard deviations (ARSD) of weight loss for the 12 materials tested is 5 percent. In the NBS smoke chamber a greater variation in sample weight loss occurred during the three replicate tests, with an ARSD of 15 percent. This variation could be due to the loss of charred material and dripping of the sample. It could also be due to variations in the combustion process itself for some materials.

Reproducibility is important for regulatory purposes because it is directly related to our ability to differentiate between closely ranked materials. Although NAFEC is not directly involved in the regulatory process, the work we do often contributes to regulatory actions.

A rough determination of the reproducibility of gas yields can be made by looking at the ARSD for each gas. Table 6 contains the ARSD for each of the eight gases in addition to the minimum and maximum RSD for each gas. In the NBS smoke chamber, the ARSD's ranged from 30 to 52 percent for all eight gases. In the combustion tube, the ARSD's range from 9 to 24 percent for CO, HCℓ, HCN, HF, and H_2S; and 52 to 69 percent for NO_2, HCHO, and SO_2. The reproducibilities for CO, HCN, H_2S, and HF were found to be at least twice as good in the combustion tube furnace as in the NBS smoke chamber. Carbon monoxide and HCN are considered to be of primary importance in regards to the hazards in combustion atmospheres.

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TABLE 4. COMPARISON OF TOXIC GAS YIELDS IN THE NBS SMOKE CHAMBER AND THE COMBUSTION TUBE FURNACE

Material		% Wt. Loss	TOXIC GAS YIELDS (mg/g)								
			CO	HCℓ	HCN	HF	H_2S	HBr	HCHO	SO_2	NO_2
1	CDT	27.0	29.9	46.7	1.2	6.4	0	-	0.5	0	0.88
	IMA	22.3	21.8	13.5	0.6	3.9	-	0	-	-	0.03
	CTF	61.4	95.7	32.8	4.7	8.3	0	4.8	-	0	0.08
33	CDT	60.0	32.3	17.5	3.2	0	0	-	1.7	24.6	9.72
	IMA	64.7	27.2	7.1	3.0	-	3.0	0	0	17.0	0.14
	CTF	90.9	55.2	21.9	14.9	-	5.3	0	T	2.2	0
34	CDT	62.0	38.0	31.8	2.6	0	0	-	2.9	22.4	8.92
	IMA	65.0	36.0	11.8	2.6	-	0.5	0	0	15.8	0.13
	CTF	91.3	45.7	24.9	13.5	-	6.1	0	1.0	2.5	0
43	CDT	19.0	19.9	13.0	0.8	3.6	0	-	0.7	0	0.33
	IMA	15.8	19.3	5.0	0.4	4.0	-	T	-	-	0.01
	CTF	57.8	147.0	11.3	5.2	8.5	0	T	-	0	0.37
61	CDT	25.0	25.5	27.7	0.6	4.6	0.2	-	0.5	0	1.39
	IMA	17.1	15.0	3.8	0.2	2.4	-	T	-	-	0.12
	CTF	62.9	142.0	27.6	6.8	5.5	0	0	-	0	0.25
70	CDT	50.0	48.7	0	8.2	0	11.8	-	0	33.4	4.00
	IMA	53.2	48.0	0	12.1	-	18.8	T	0.3	6.2	0.45
	CTF	80.3	78.2	0	33.8	-	13.9	0	0.8	0	0
78	CDT	26.0	35.2	21.4	1.1	0	0	-	0	10.7	1.15
	IMA	35.3	74.5	25.1	1.4	-	T	0	0.8	7.6	0.26
	CTF	90.7	95.6	43.1	7.0	-	0	0	1.2	11.2	0.53
82	CDT	66.0	57.2	134.0	5.5	0	9.7	-	0.5	18.3	9.39
	IMA	57.7	55.1	40.0	3.6	-	9.1	0	0.8	16.2	0.22
	CTF	97.0	112.0	87.8	19.5	-	10.7	0	0.8	4.8	0.03
88	CDT	46.0	33.5	0	6.5	0	9.2	-	0	28.7	3.58
	IMA	54.3	35.6	T	10.3	-	15.2	T	0.5	6.4	0.45
	CTF	82.8	88.8	0	41.7	-	13.4	0	1.2	0.3	0
92	CDT	21.0	40.5	T	0.7	0	0	-	0	5.8	1.25
	IMA	23.0	46.1	0	0.9	-	-	2.1	0.6	2.3	0.27
	CTF	80.1	63.4	0	14.9	-	0	9.6	T	8.5	1.60
142	CDT	64.0	74.3	0	12.9	0	10.8	-	0	10.2	4.88
	IMA	55.5	69.0	0	9.2	-	15.5	T	0.4	3.1	0.63
	CTF	92.1	112.0	0	37.2	-	14.2	20.5	0	1.47	0
144	CDT	11.0	16.0	5.2	0.1	4.8	0	-	0.4	0	0.16
	IMA	12.8	23.4	0.6	0.2	3.1	-	0	-	-	0.04
	CTF	59.0	143.0	0	8.2	4.1	T	5.5	-	0	0.33

CDT - NBS Smoke Chamber with Colorimetric Detector Tube Analysis
IMA - NBS Smoke Chamber with Instrumental Methods of Analysis
CTF - Combustion Tube Furnace with Instrumental Methods of Analysis
T - Trace Amount
- - No Data

TABLE 5. CARBON MONOXIDE YIELDS FOR NONFLAMING CONDITIONS IN THE NBS SMOKE CHAMBER

Material	Percent Weight Loss	CO Yield (mg/g)
33	40.6	4.9
34	34.1	8.0
78	15.8	9.6
82	51.5	14.1
92	14.2	9.2
142	38.5	12.0
144	13.1	4.3

TABLE 6. REPRODUCIBILITY OF GAS YIELDS

	Toxic Gases							
Reproducibility	CO	HCℓ	HCN	HF	H_2S	HCHO	NO_2	SO_2
Combustion Tube Furnace								
Average RSD (%)	9	24	19	15	15	64	52	69
Minimum RSD (%)	0.3	5	10	2	9	10	11	12
Maximum RSD (%)	18	30	33	22	28	166	162	177
NBS Smoke Chamber								
Average RSD (%)	30	42	52	50	48	48	33	43
Minimum RSD (%)	15	25	9	36	22	35	4	16
Maximum RSD (%)	48	119	183	84	86	60	65	86

COMPARISON OF GAS YIELDS FOR THE COMBUSTION TUBE FURNACE AND THE NBS SMOKE CHAMBER.

Comparisons of gas yields in terms of mg/g for the combustion tube furnace and the NBS smoke chamber are illustrated in figures 1 through 4 for CO, HCℓ, HCN, and H_2S, respectively. The least squares best fit to a straight line was obtained for each significant correlation. The corresponding R values were calculated for each gas and are reported in table 7. The corresponding slope, y_o, and standard error are also reported for all gases.

TABLE 7. REGRESSION ANALYSIS OF TOXIC GAS YIELDS (mg/g): COMBUSTION TUBE FURNACE VERSUS FLAMING CONDITIONS IN THE NBS SMOKE CHAMBER

GAS	N	R	Pℓ(%)	Slope	Y_o	Standard Error
CO	12	0.25	>10	--	--	--
CO*	8	0.74	5	1.1	27.0	18.0
HCℓ	8	0.99	1	1.9	5.4	8.6
HCN	12	0.95	1	2.9	6.6	4.3
H_2S	6	0.96	1	0.52	5.3	1.1
HCHO	5	0.14	>10	--	--	--
HF	4	0.79	>10	--	--	--
NO_2	12	0.14	>10	--	--	--
SO_2	7	0.25	>10	--	--	--

*Excluding panels 1, 43, 61, and 144.

The R values for HCN, H_2S, and HCℓ ($0.95 \leq R \leq 0.99$) are significant at the 1-percent probability level. The yields of these gases are significantly correlated for rather diverse exposure conditions. This suggests that the relative yields of these gases may be somewhat independent of exposure conditions. The ratios of gas yields in the combustion tube furnace to those obtained in the NBS smoke chamber are approximately 3:1 for HCN, 1:2 for H_2S, 2:1 for HCℓ, and 1:1 for CO with the panels deleted. The CO yields for the panels 1, 43, 61, and 144 are substantially higher in the combustion tube furnace than in the NBS smoke chamber. Further studies are required to determine how the yields of these gases vary with exposure conditions.

The observed yields of the oxidized gases such as CO, SO_2, NO_2, and HCHO are not significantly correlated. The R values for these gases are not significantly different from zero, even at the 10-percent probability level. The formation of these gases is apparently much more sensitive to exposure conditions. Although a significant correlation was not obtained for HF, this result is less meaningful since only four materials were used in the comparison.

Although the correlation for CO is not significant, the data are plotted in figure 1. As indicated, CO yields for the panels, materials 1, 43, 61, and 144, are substantially higher in the combustion tube furnace and tend to form

a distinct group. This is due to the total immersion of the sample in the combustion tube furnace, whereas only the front face of the material is exposed in the NBS smoke chamber. This factor substantially affects the results for composite materials. Therefore, the use of one-dimensional heating in the combustion tube furnace should be explored. Deleting the panels does improve the correlation for CO to a point where the R-value (0.74) is significant at the 5-percent probability level. The correlation illustrated in figure 1 is with the panels deleted.

Nonflaming conditions in the NBS smoke chamber were also used to measure CO yields for the seven materials listed in table 5. The CO yields obtained under flaming conditions are correlated with these obtained under nonflaming conditions in the NBS smoke chamber as illustrated in figure 5. The R-value of 0.77 is significant at the 5-percent probability level. The data relevant to figure 5 include a slope of 4.4, a y-intercept of 8.8 and a standard error of estimate of 14. The slope indicates that flaming CO yields are significantly higher than the yields obtained with nonflaming conditions.

However, the correlation for CO is greatly improved if the CO yields are compared in terms of milligrams per gram weight loss of material (mg/Δg), as illustrated in figure 6. Comparing CO yields on the basis of sample weight loss, rather than initial weight, increases the R-value to 0.98, which is significant at the 1-percent probability level. This would tend to indicate that the degree of combustion of the sample varies considerably from one test to the next. The relevant data for figure 6 includes a slope of 3.3, a y-intercept of 1.1, and a standard error of estimate of 16.

Carbon monoxide yields for nonflaming conditions in the NBS smoke chamber are compared to those obtained with the combustion tube furnace in figure 7. The data for figure 7, deleting panel number 144, include a slope of 7.9, a y-intercept of 5.1, and a standard error of estimate of 18. The R-value of 0.85 is significant at the 5-percent probability level. The ratio of CO yields in the combustion tube furnace to those obtained in the NBS smoke chamber with nonflaming conditions is approximately 8:1. The degree of correlation is similar to that obtained for flaming conditions (figure 1), and does not represent an improved correlation for CO.

A COMPARISON OF COLORIMETRIC DETECTOR TUBES AND INSTRUMENTAL METHODS OF ANALYSIS IN THE NBS SMOKE CHAMBER.

Gas yields obtained in the NBS smoke chamber with colorimetric detector tubes (reference 1) have been compared with gas yields obtained using instrumental methods of analysis. The statistical data for each correlation, on a mg/g basis, are contained in table 8. The R-values for CO, HCℓ, HCN, and H_2S ($0.76 \leq R \leq 0.93$) are significant at the 1-percent probability level and are illustrated in figures 8 through 11. The corresponding slopes of 0.63, 2.7, 0.81, and 0.69 differ from a slope of unity. This suggests that colorimetric detector tubes may provide a rapid method for obtaining relative yields for these gases. However, the R-values for NO_2, HCHO, and SO_2 are not significantly different from zero, even at the 10-percent probability level. Although a significant correlation was not obtained for HF, this result is less meaningful since only four materials were used in the comparison.

TABLE 8. REGRESSION ANALYSIS OF TOXIC GAS YIELDS (mg/g) IN THE NBS SMOKE CHAMBER: COLORIMETRIC DETECTOR TUBES VERSUS INSTRUMENTAL METHODS OF ANALYSIS

GAS	N	R	Pℓ(%)	Slope	Y_o	Standard Error
CO	12	0.76	1	0.63	13.0	11.0
HCℓ	8	0.85	1	2.7	1.6	23.0
HCN	12	0.88	1	0.81	0.65	2.0
H_2S	6	0.93	1	0.69	-0.17	2.2
HCHO	7	0	>10	--	--	--
HF	4	0.11	>10	--	--	--
NO_2	12	0.20	>10	--	--	--
NO_2*	9	0.95	1	7.4	0.11	0.59
SO_2	8	0.36	>10	--	--	--

*Excluding materials 33, 34, and 82.

The NO_2 detector tube is responsive to total NO_x, whereas the Griess-Saltzman procedure primarily responds to NO_2 concentrations. Therefore, based on figure 12, it appears that relatively large concentrations of nitrogen oxides other than NO_2 may be produced during the combustion of the wool carpets, materials 33 and 34, and the wool/PVC blend, material 82. The carpets exhibited unusually high flames during a substantial portion of the test period. The correlation for NO_2 improves considerably from an R-value of 0.20 to an R-value of 0.95 when these three materials are excluded from the regression analysis.

SUMMARY OF RESULTS

1. The ARSD for toxic gas yields in the NBS smoke chamber was approximately 43 percent for CO, HCℓ, HCN, HF, H_2S, HCHO, NO_2, and SO_2. In comparison, the ARSD for toxic gas yields in the combustion tube furnace was 9 percent for CO, while it was approximately 18 percent for HCℓ, HCN, HF, and H_2S. The ARSD in the combustion tube furnace was roughly 62 percent for NO_2, SO_2, and HCHO.

2. The variation in sample weight loss for three replicate tests was significantly greater in the NBS smoke chamber (ARSD = 15 percent) than the combustion tube (ARSD = 5 percent) for the 12 materials tested.

3. The gas yields of materials in mg/g using the combustion tube furnace correlated well with the gas yields of materials in the NBS smoke chamber for HCℓ, HCN, and H_2S ($0.95 \leq R \leq 0.99$). The R values are significant at the 1-percent probability level. No significant correlation was obtained for CO, HF, HCHO, NO_2, and SO_2. Deleting the panels improves the correlation for CO to a point where the R value (0.74) is significant at the 5-percent probability level.

4. The ratios of gas yields in the combustion tube furnace to those obtained in the NBS smoke chamber are approximately 3:1 for HCN, 1:2 for H_2S, 2:1 for HCℓ, and 1:1 for CO with the panels deleted. The CO yields for the panels, materials 1, 43, 61, and 144 are substantially higher in the combustion tube furnace than in the NBS smoke chamber.

5. In the NBS smoke chamber, when mg/g ranking is used, some correlation exists for CO yields obtained under flaming and nonflaming conditions. The R-value of 0.77 is significant at the 5-percent probability level. However, when a mg/Δg basis is used, the coefficient of correlation improves (R=0.98) and is significant at a 1-percent probability level.

6. In the NBS smoke chamber, flaming CO yields are approximately four times higher than the yields obtained with nonflaming conditions.

7. There is good correlation between the colorimetric detector tubes and the instrumental methods of analysis for CO, HCN, H_2S, and HCℓ when mg/g ranking is used ($0.76 \leq R \leq 0.93$). The R values are significant at the 1-percent probability level. The corresponding slopes for CO, HCℓ, HCN, and H_2S are 0.63, 2.7, 0.81, and 0.69, respectively. The R values obtained for HF, HCHO, NO_x, and SO_2 are not significantly different from zero even at the 10-percent probability level.

CONCLUSIONS

1. The reproducibilities of combustion gas yields are dependent upon the method used to thermally decompose the test material.

2. The conditions in the NBS smoke chamber are more oxidative than in the combustion tube furnace for the experimental conditions chosen for these tests.

3. The relative gas yields are somewhat independent of the exposure conditions for $HC\ell$, HCN, and H_2S. However, CO, HCHO, NO_2, and SO_2 relative yields are much more dependent on the exposure conditions.

4. Colorimetric detector tubes for HCHO and SO_2 are less reliable for combustion gas analysis than those for CO, $HC\ell$, HCN and H_2S.

5. Colorimetric detector tubes provide a rapid method of obtaining relative yields for CO, $HC\ell$, HCN, and H_2S.

RECOMMENDATIONS

From the conclusions, it is recommended that:

1. Additional laboratory tests be conducted using the combustion tube furnace to determine whether or not a specific set of conditions will simulate the results obtained under flaming combustion conditions in the NBS smoke chamber for both reduced and oxidized combustion gases.

2. Intermediate and full-scale material tests be conducted with the objective of developing and parameterizing a laboratory test which simulates a full-scale fire.

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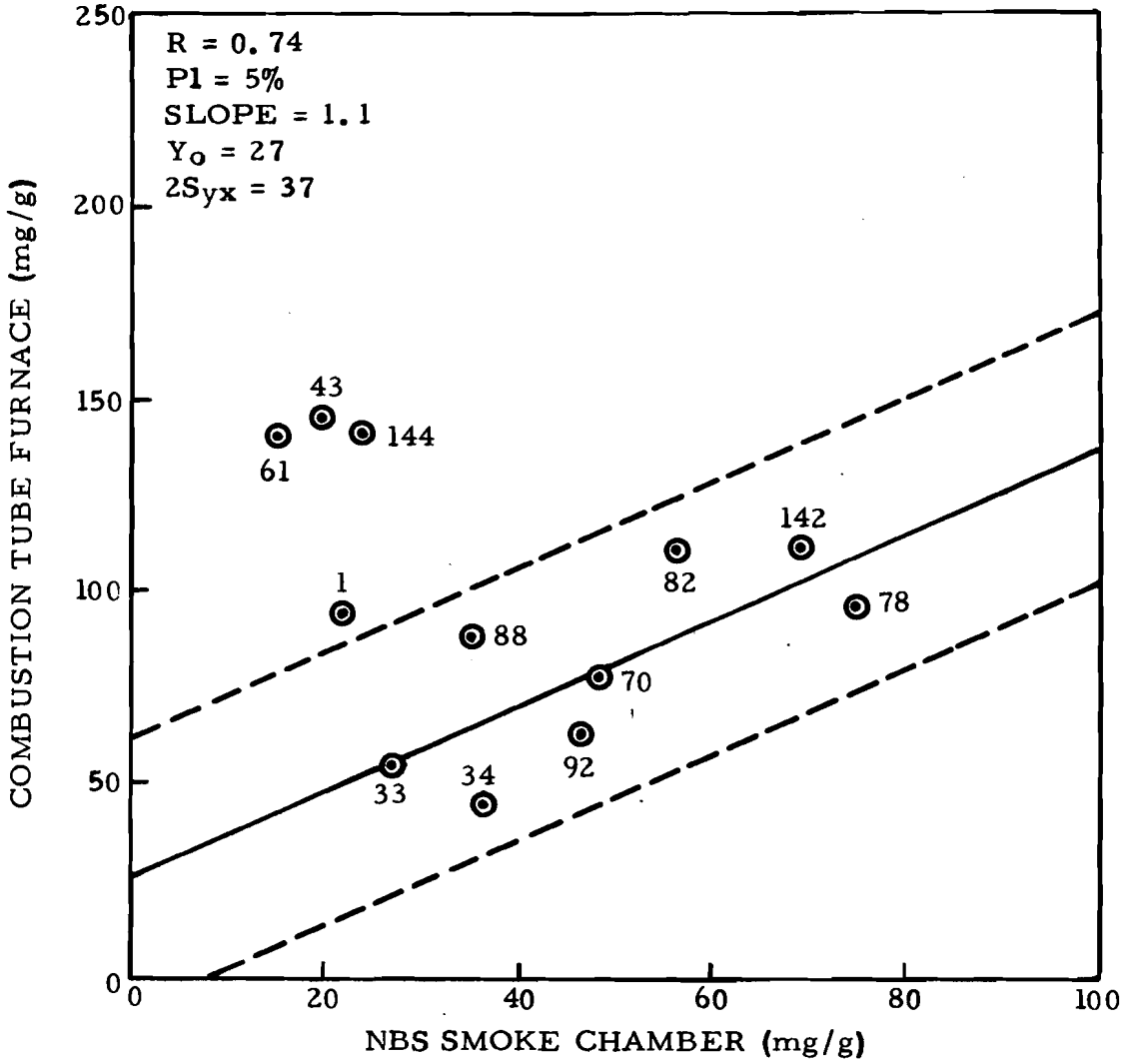

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FIGURE 1. COMPARISON OF CO YIELDS FOR FLAMING CONDITIONS IN THE NBS SMOKE CHAMBER WITH YIELDS FROM THE COMBUSTION TUBE FURNACE

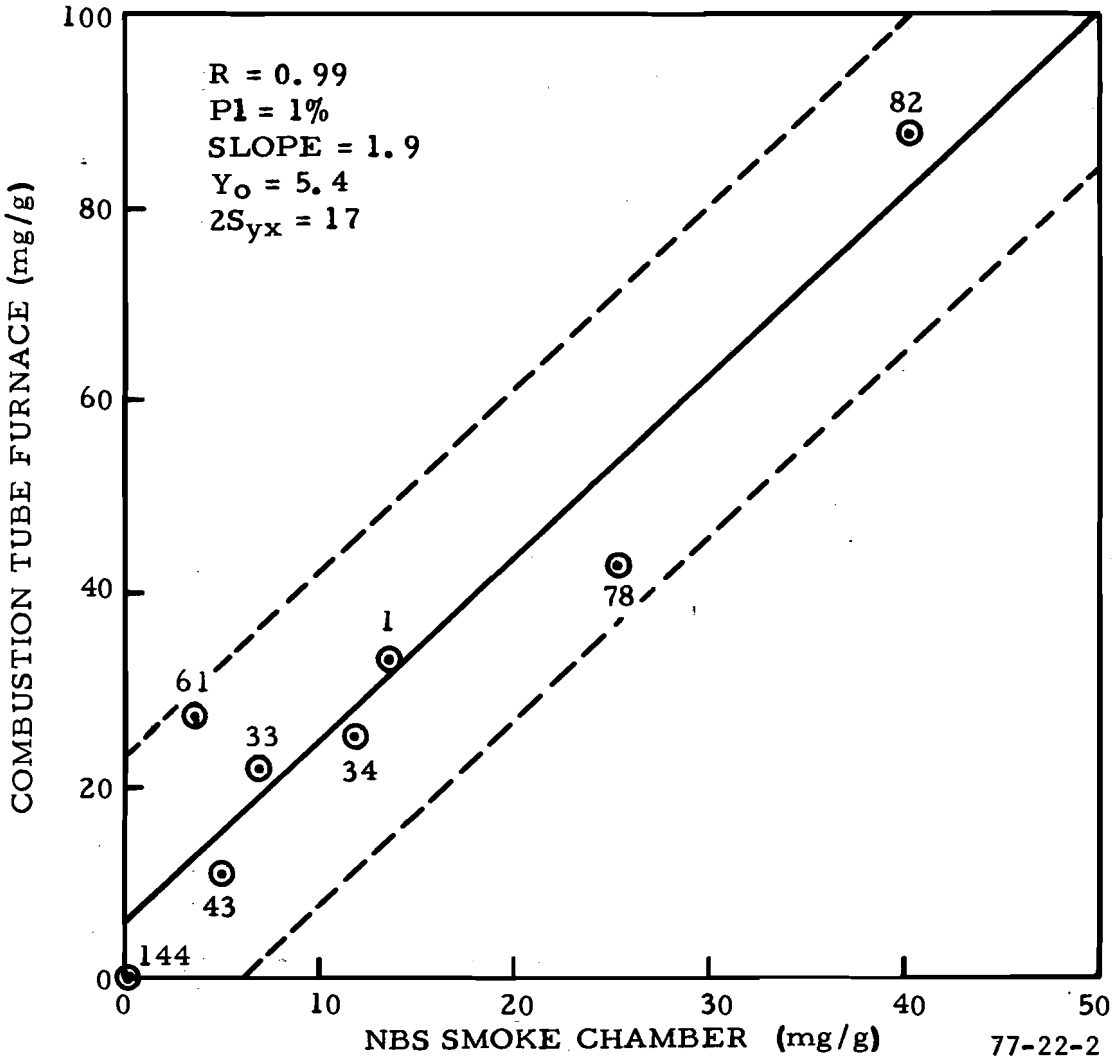

FIGURE 2. COMPARISON OF HCℓ YIELDS FOR FLAMING CONDITIONS IN THE NBS SMOKE CHAMBER WITH YIELDS FROM THE COMBUSTION TUBE FURNACE

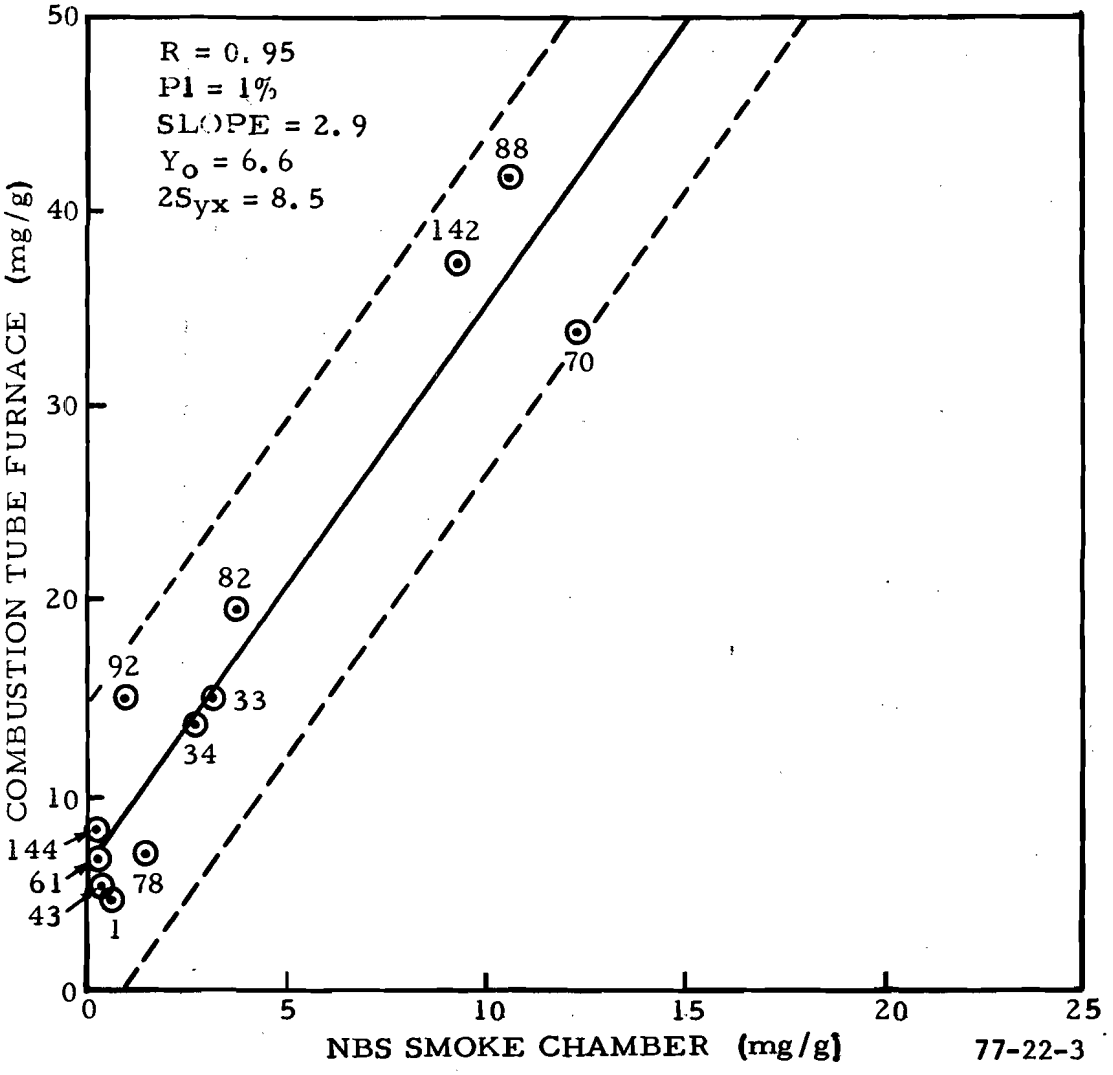

FIGURE 3. COMPARISON OF HCN YIELDS FOR FLAMING CONDITIONS IN THE NBS SMOKE CHAMBER WITH YIELDS FROM THE COMBUSTION TUBE FURNACE

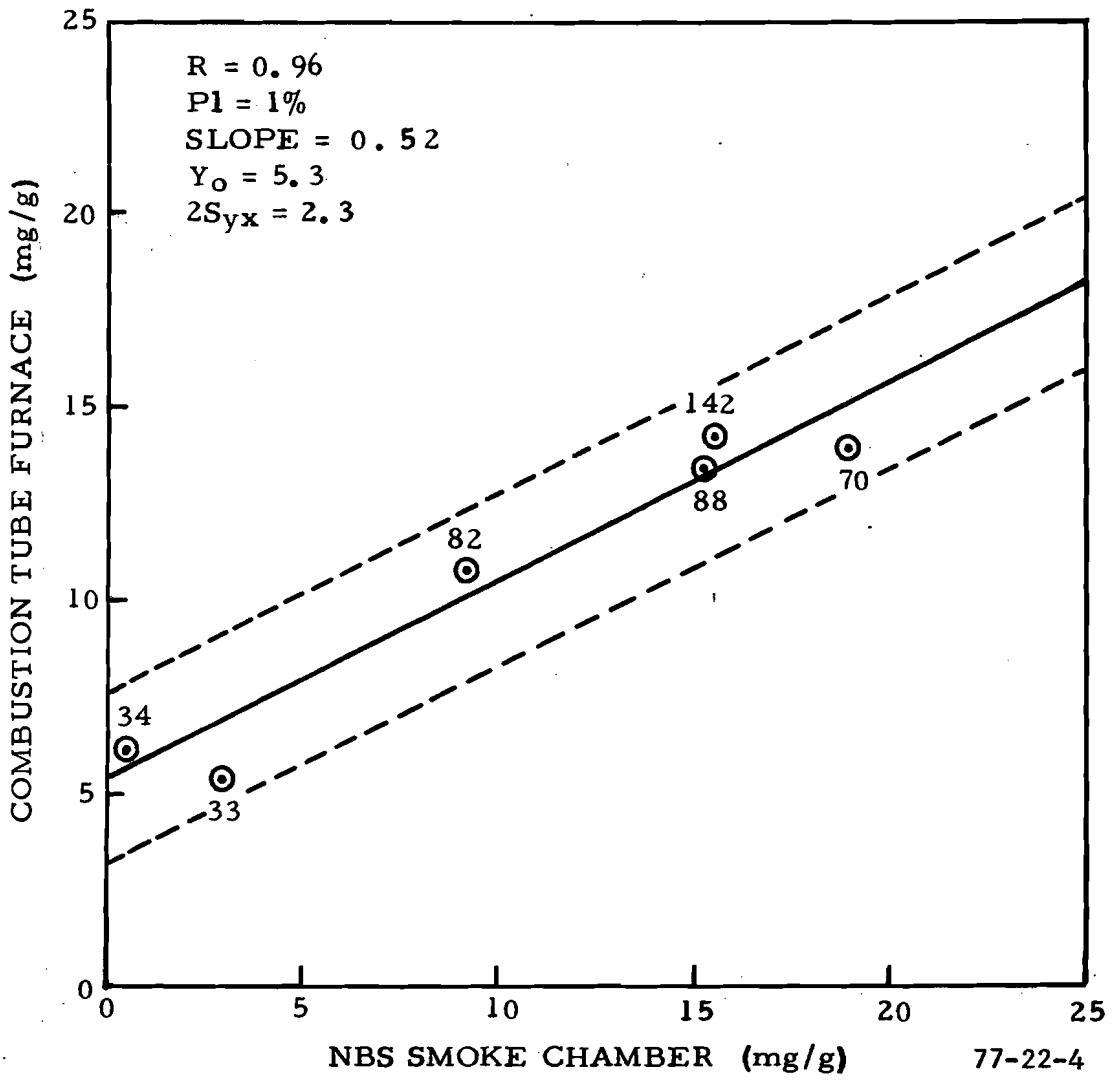

FIGURE 4. COMPARISON OF H_2S YIELDS FOR FLAMING CONDITIONS IN THE NBS SMOKE CHAMBER WITH YIELDS FROM THE COMBUSTION TUBE FURNACE

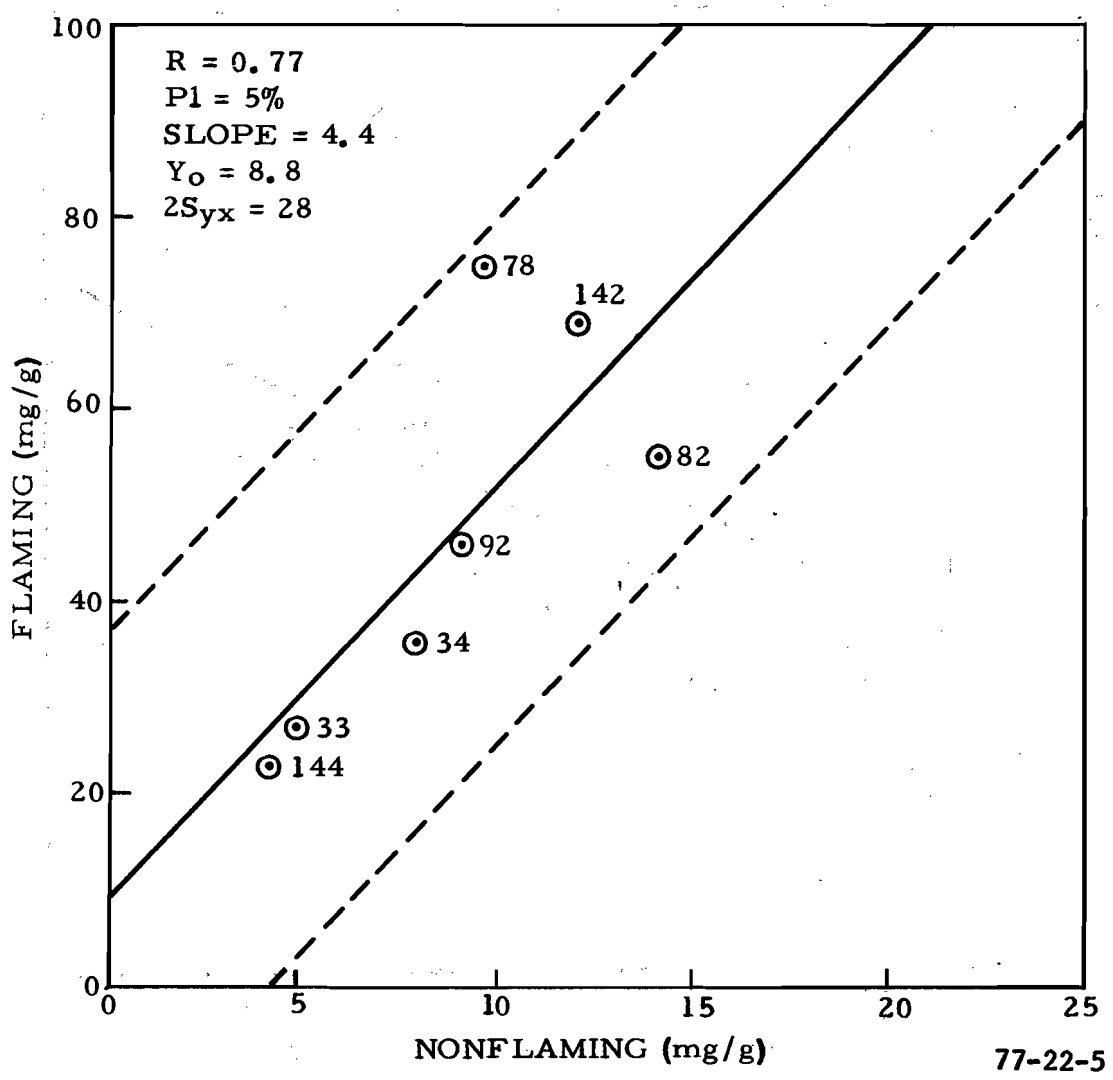

FIGURE 5. COMPARISON OF CO YIELDS FOR FLAMING AND NONFLAMING CONDITIONS IN THE NBS SMOKE CHAMBER

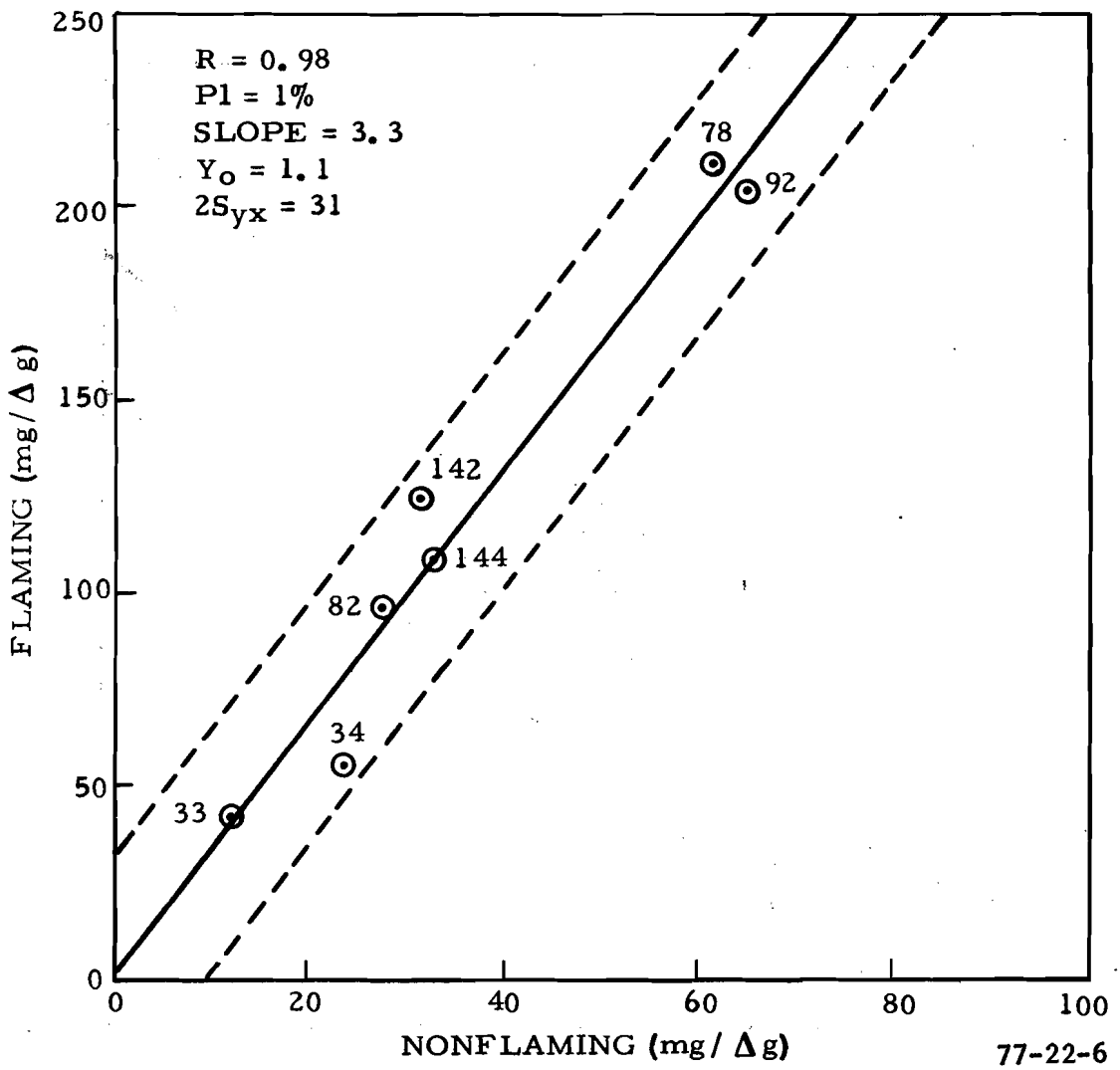

FIGURE 6. COMPARISON OF CO YIELDS FOR FLAMING AND NONFLAMING CONDITIONS IN THE NBS SMOKE CHAMBER ON A WEIGHT-LOSS BASIS

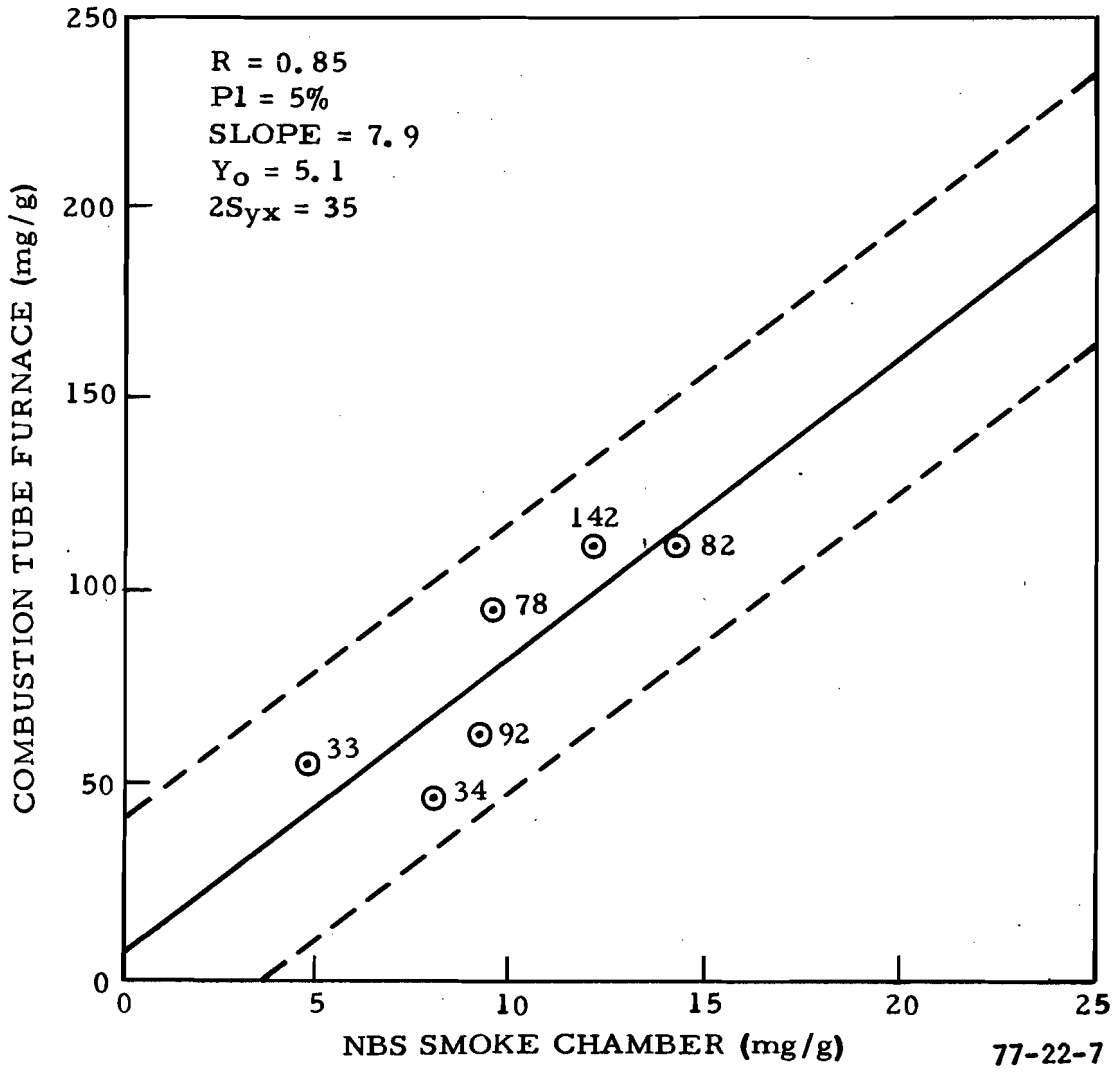

FIGURE 7. COMPARISON OF CO YIELDS FOR NONFLAMING CONDITIONS IN THE NBS SMOKE CHAMBER WITH YIELDS FROM THE COMBUSTION TUBE FURNACE

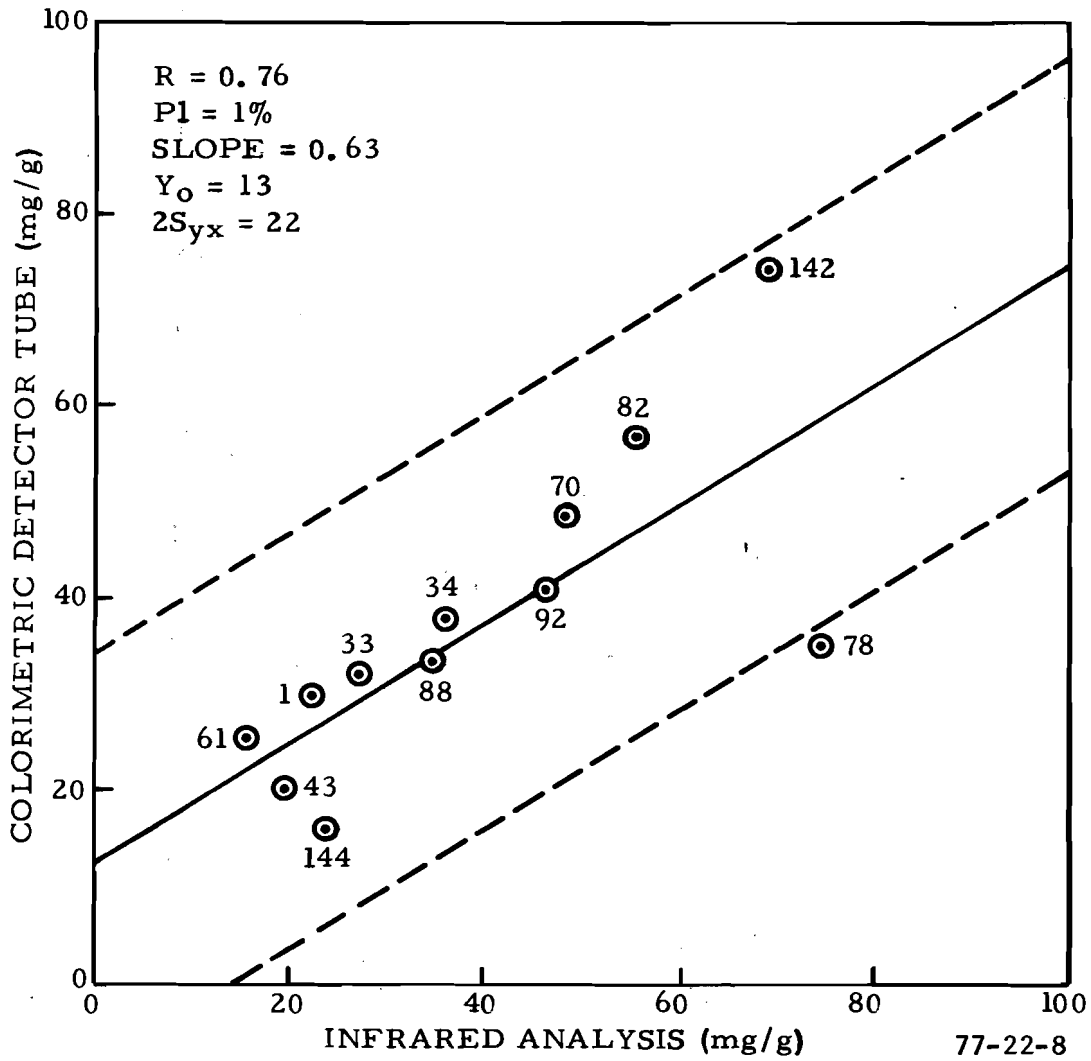

FIGURE 8. COMPARISON OF CO YIELDS IN THE NBS SMOKE CHAMBER OBTAINED USING COLORIMETRIC DETECTOR TUBES WITH YIELDS OBTAINED USING INFRARED ANALYSIS

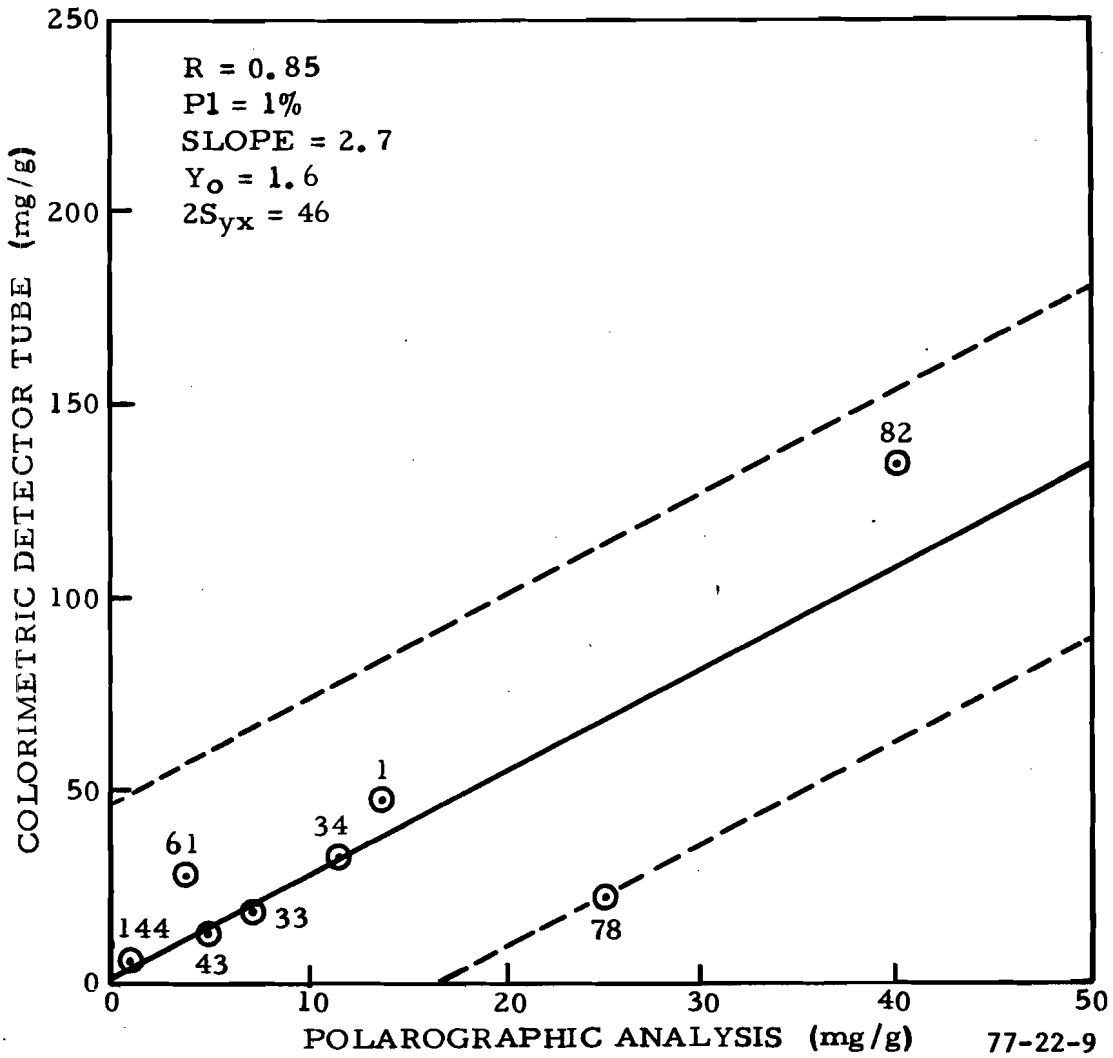

FIGURE 9. COMPARISON OF HCℓ YIELDS IN THE NBS SMOKE CHAMBER OBTAINED USING COLORIMETRIC DETECTOR TUBES WITH YIELDS OBTAINED USING POLAROGRAPHIC ANALYSIS

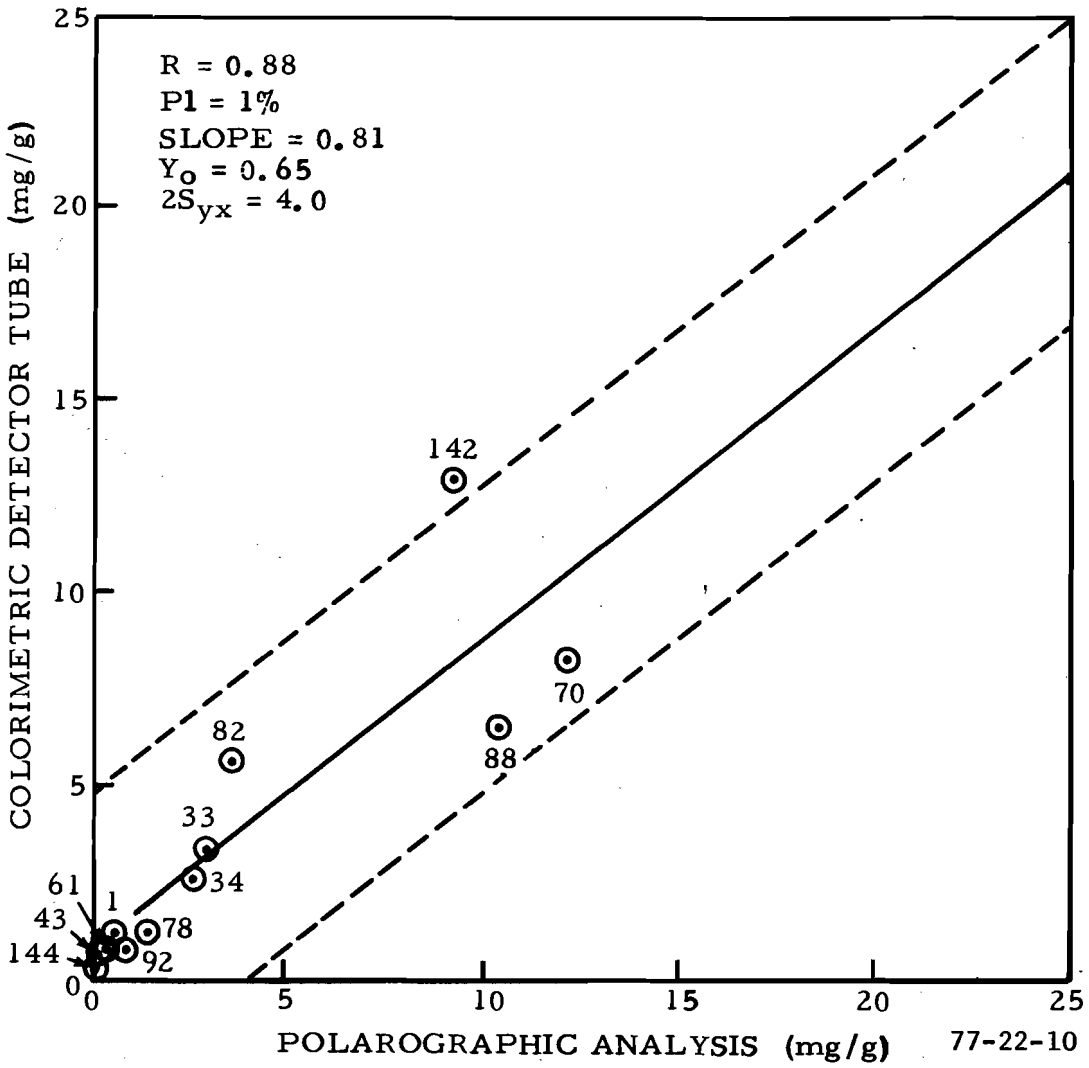

FIGURE 10. COMPARISON OF HCN YIELDS IN THE NBS SMOKE CHAMBER OBTAINED USING COLORIMETRIC DETECTOR TUBES WITH YIELDS OBTAINED USING POLAROGRAPHIC ANALYSIS

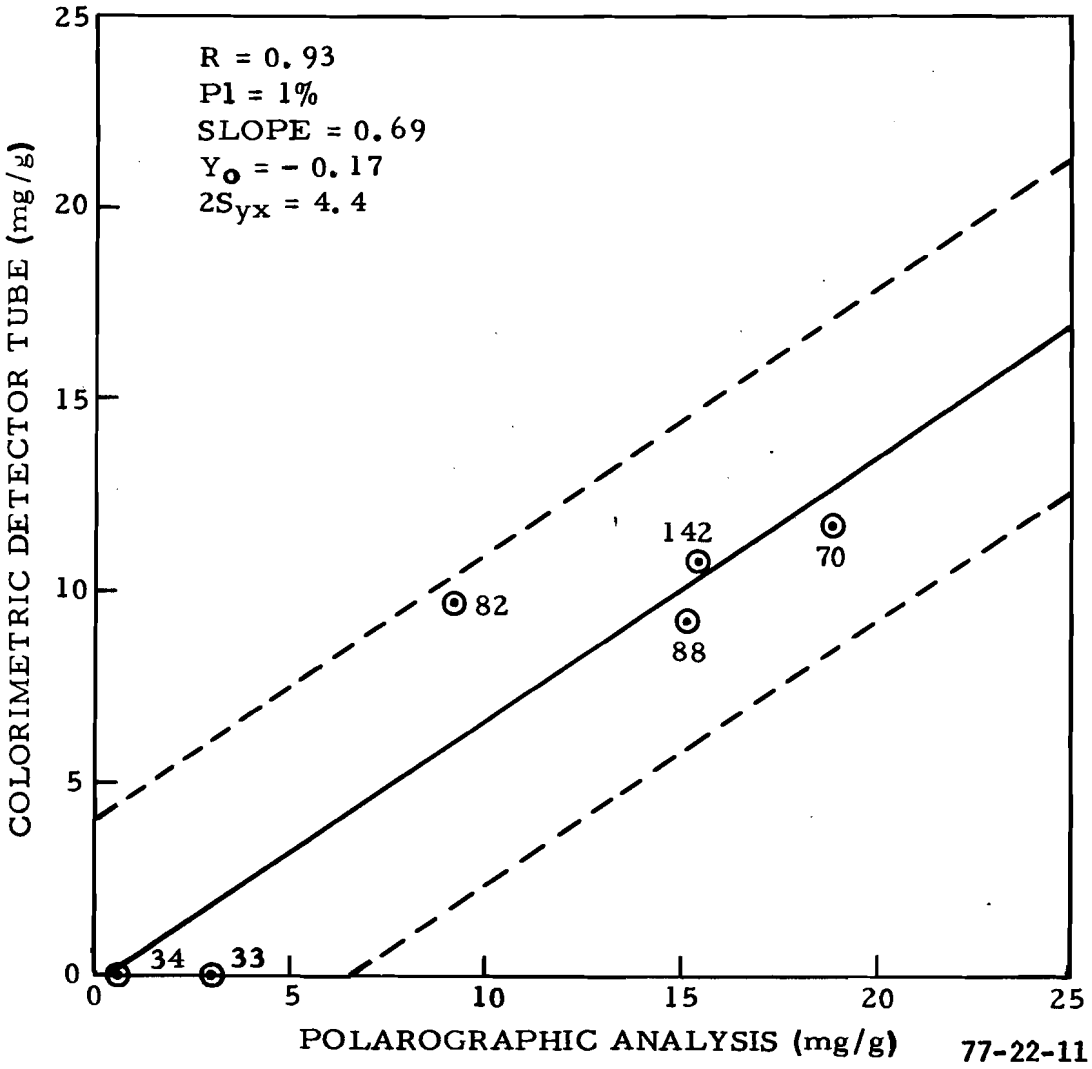

FIGURE 11. COMPARISON OF H_2S YIELDS IN THE NBS SMOKE CHAMBER OBTAINED USING COLORIMETRIC DETECTOR TUBES WITH YIELDS OBTAINED USING POLAROGRAPHIC ANALYSIS

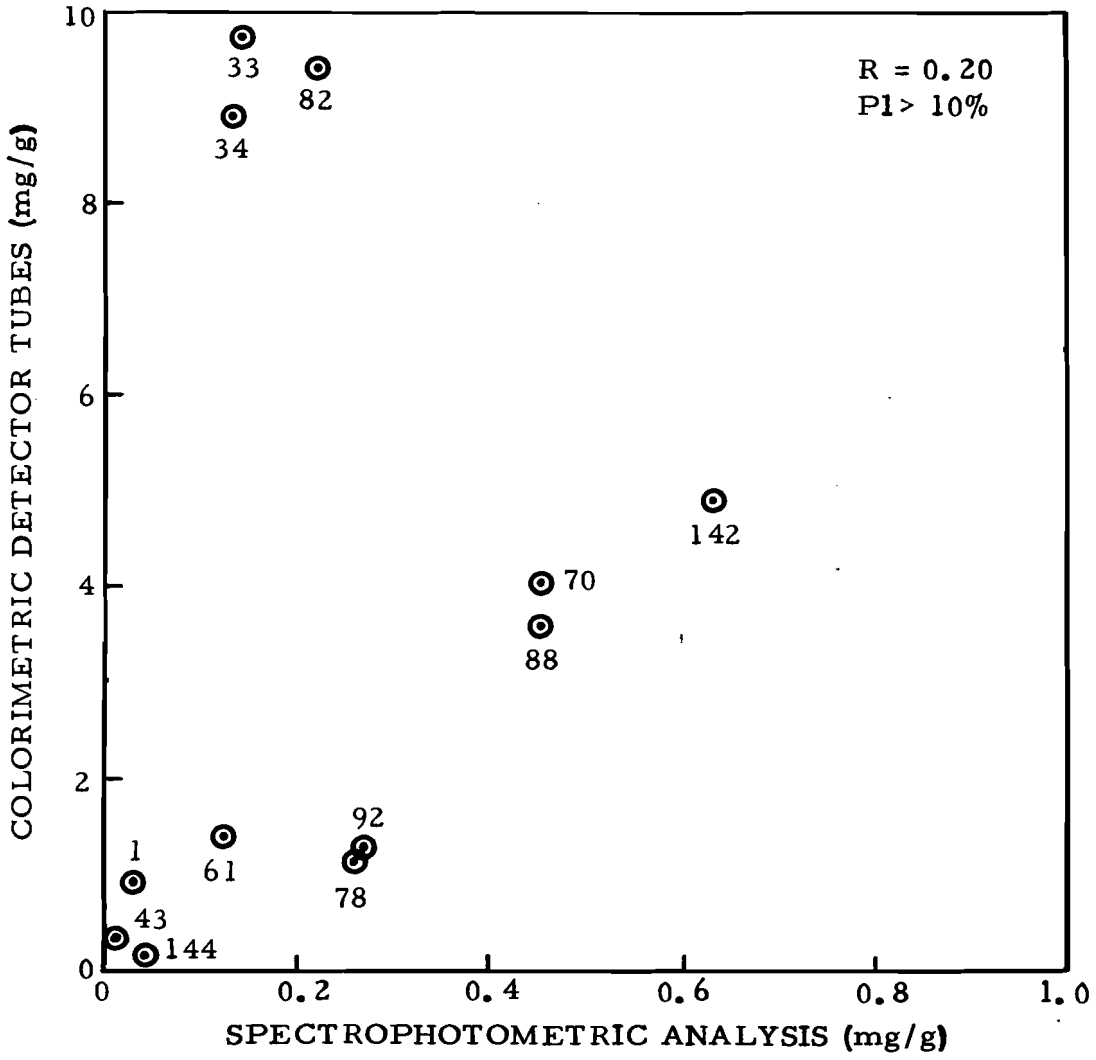

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FIGURE 12. COMPARISON OF NO_x YIELDS IN THE NBS SMOKE CHAMBER OBTAINED USING COLORIMETRIC DETECTOR TUBES WITH NO_2 YIELDS OBTAINED USING SPECTROPHOTOMETRIC ANALYSIS