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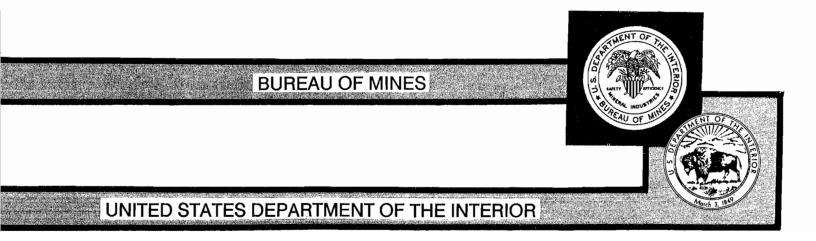
Primary Gas Toxicities and Smoke Particle Characteristics During Combustion of Mine Ventilation Ducts

Development of a Test Parameter

9284

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By Maria I. De Rosa and Charles D. Litton



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UNITED STATES DEPARTMENT OF THE INTERIOR Manuel Lujan, Jr., Secretary

BUREAU OF MINES T S Ary, Director

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		EASURE ABBREVIATIONS	USED IN TH	IIS REPORT
o	C	degree Celsius	L	liter
с	em	centimeter	L/min	liter per minute
с	cm²/p	square centimeter per particle	min	minute
	$cm^2/(p \cdot g)$	square centimeter per	pct	percent
C	.m / (p•g)	particle per gram	p/cm ²	particle per square centimeter
f	ť	foot	ppm	part per million
g	S	gram	ppm/g	part per million
iı	n	inch	ЪЪш\ <i>В</i>	per gram

PRIMARY GAS TOXICITIES AND SMOKE PARTICLE CHARACTERISTICS DURING COMBUSTION OF MINE VENTILATION DUCTS

Development of a Test Parameter

By Maria I. De Rosa¹ and Charles D. Litton²

ABSTRACT

The U. S. Bureau of Mines performed experiments to determine if the primary gas toxicities evolved during the early (~300° C) and later (\geq 400° C) combustion stages of chlorinated polyester-reinforced mine ventilation ducts could be predicted by a smoke particle characteristic, for the development of a test parameter. The experiments were conducted in an approximately 20-L furnace, at 14-min durations at set furnace temperatures of 250° and 1,000° C, with an airflow through the furnace of 10 L/min. The variables studied, as a function of time, were the hydrogen chloride (HCl), carbon monoxide (CO), carbon dioxide (CO₂), and oxygen (O₂) concentrations, furnace temperatures, the sample mass weight loss, the average smoke particle diameter (d_g) and average number and concentration (n_o), and the product of the average smoke particle diameter and concentration (d_gn_o). Results show that the inverse of the smoke particle diameter-concentration product (1/d_gn_o), correlates directly and significantly with the primary gas toxicities evolved during both early (HCl) and later (CO) stages of combustion.

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INTRODUCTION

In 1984, the U.S. Bureau of Mines, within its mission to create and maintain safety in the mines and, specifically, to assess the hazards of mine materials during fire, initiated a series of experiments to study smoke particle characteristics measured during the early (~300° C) combustion stage of mine combustibles. It was found $(1)^3$ that smoke particles produced by different combustibles differed in average diameter (d_g), average number concentration (n_o), and the product of the smoke particle average diameter and concentration (d_gn_o).

A direct correlation was established (2) between the inverse of the smoke particle diameter-concentration product $(1/d_g n_o)$, measured during the combustion of various materials, and the toxicity data obtained with similar materials and reported by Anderson and Alarie (3) (animal study) and Paciorek (4) (gas analyses). In subsequent studies (5-8), simultaneous measurements were

Organic materials when heated release gaseous species whose toxicity and rates of release depend on the nature of the material, on temperature, and on material ease of thermal decomposition (5). Synthetic polymeric materials, because of resin composition, release toxic gases earlier, and at faster rates, because the cohesive and covalent bonds of the polymer chain break easily under heat penetration (9). Polyester-reinforced mine ventilation ducts are made of polyester resin, derived from the reaction of a dibasic acid (maleic or phthalic anhydride) with a dibasic dihydric alcohol (propylene or ethylene glycol), blended with a reactive vinyl monomer (styrene, vinyl benzene, or vinyl toluene), and reinforced (>80 pct) with fiberglass, treated with inhibitors (phenolic compounds) and flame retardants (chlorine, phosphorous, and antimony trioxide). However, even the finished products can be ignited by sufficient heat sources (10), releasing at lower temperatures hydrogen chloride (HCl), and at higher temperatures, carbon monoxide (CO). Table 1 reports the toxicity of pyrolysis products of polyester fibers (11). HCl, with a short-term exposure limit (STEL) of 25 ppm and immediately dangerous to life and health (IDLH) at 100 ppm as hydrochloric acid, deposits on soot particles, which are transported to the mucous membranes, causing irritation and severe damage to the eyes and to the lower respiratory tract, with ensuing cough, dizziness, heaviness of the legs, impaired vision, and disorientation (12). The pulmonary damage from polymers containing HCl has been shown (13-14) to be more severe than that from HCl alone, probably because the particulates may be drawn into and deposited into the lungs, where the absorbed and/or

made of primary gas concentrations and smoke particle characteristics during the combustion of polyvinyl chloride (PVC) and other chlorinated mine conveyor belts, PVC and other chlorinated mine brattices, wood, and coal; it was found that the inverse of the smoke particle diameter-concentration product $(1/d_gn_o)$, correlated directly and significantly with those toxicities.

In this study, the Bureau set out to correlate the smoke particle characteristic $1/d_{gn_0}$ with the primary gas toxicities evolved during the early (~300° C) and later (≥400° C) combustion stages of chlorinated polyester, fiberglass-reinforced ventilation duct samples. These correlations would allow the development of a test parameter for assessing simply, and inexpensively, the toxic hazard of ducts during stages of fire when miners must plan and prepare for escape.

BACKGROUND

dissolved HCl enhances the toxic effects. The HCl LC50 (lethal concentration at which 50 pct of laboratory animals die) has been found (15) to decrease from 5,600 ppm for pure HCl gas to 1,900 ppm for PVC products, and the HCl LC50 for cannulated animals (simulating human breathing habits) has been found (16) to be 1,000 ppm as opposed to 10,000 ppm for noncannulated animals; humans tend to breathe through the mouth in the presence of such irritants. Furthermore, the HCl may also be condensed by moist air to form a corrosive hydrochloric acid mist that, together with smoke, can obscure vision, interfering with escape and rescue.

At higher temperatures ($\geq 400^{\circ}$), which during fire may follow in a matter of minutes (10), the dehydrochlorinated residue yields large quantities of carbon monoxide (CO) (STEL = 400 ppm; IDLH = 1,500 ppm). CO, easily absorbed through the lungs into the blood to form. carboxyhemoglobin (COHb), blocks the oxygen carriage to the brain, causing disorientation, vertigo, and cardiovascular and neurological complications (17). The COHb content of laboratory animals dead in PVC fires was found to be 13 to 30 pct compared with the expected 85 pct for pure CO (18). The CO LC50 in PVC fires was found to be 900 ppm compared with 4,600 ppm for pure CO (19).

Table 1.-Toxicity of pyrolysis products of 10-g polyester fibers (11)

Pyrolysis temp	500
Maximum CO ppm	2,900-5,300
COHb ¹ pct	20-30
Minimum O ₂	15.6-19.7
Time to incapacitation min	15-19

¹Carboxyhemoglobin of 1 male rat placed in a 13-L exposure chamber connected by a hose to a 45-L combustion chamber.

³Italic numbers in parentheses refer to items in the list of references at the end of this report.

The system (fig. 1) consists of an approximately 20-L furnace whose temperature during experiments rises automatically from ambient at a rate depending on the set temperature, which ranges from 100° to 1,200° C (fig. 2). A universal load cell, located under the furnace floor, contacts a sample cup, which transmits sample weight loss (voltages) via a bridge amplifier to a strip chart recorder. A vacuum pump draws ambient air (10 L/min) continuously into the furnace, via an opening at the center of the furnace door, and pumps combustion air into the CO, CO_2 , and O_2 gas analyzers, and into the submicrometer particle detector analyzer (SPDA) (1.6 L/min) (20). The SPDA voltages are used to compute the average smoke particle diameter (d_g) , the average number and concentration (n_o) , and the product of the average smoke particle diameter and concentration (d_gn_o) . A data acquisition system continuously acquires, computes, and stores data of sample and furnace temperatures, airflows, gas concentrations, weight losses, and the SPDA initial and experimental voltages. The stored data are then plotted.

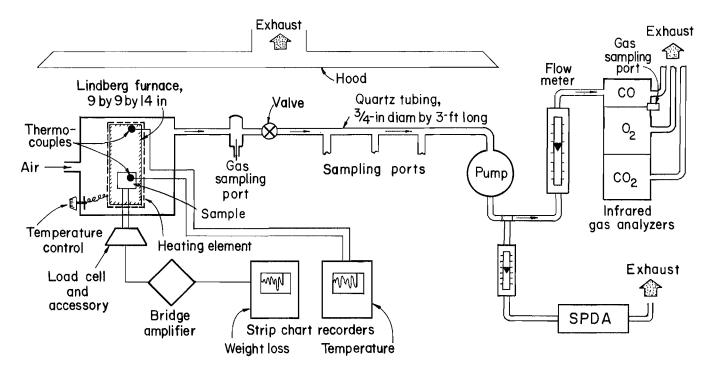


Figure 1.-Experimental system.

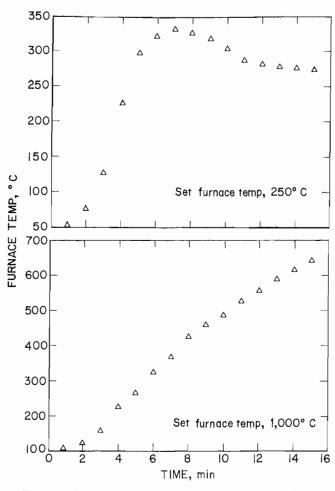


Figure 2.-Furnace temperature versus time at set furnace temperatures of 250° and 1,000° C.

DATA REDUCTION AND ANALYSES

HCl and CO concentrations were measured at 250° C set furnace temperature and 14-min test duration (representing an early stage of combustion, at which most of the HCl evolves with no gross degradation of the sample). The HCl and CO concentrations observed at 0.1 g of sample weight loss, approximately at the 10th minute, adjusted to 1 g of weight loss, yielded the HCl and CO load values (in parts per million per gram). The load value, derived at a specific time and airflow, is important toxicological parameter, for it establishes a priori the total toxic gas concentrations expected to evolve at each stage of combustion for each material: HCl evolving during the early stage (~300° C) of combustion, because of an early and fast dehydrochlorination process (CO concentrations are insignificant at this stage); CO evolving at a later stage $(\geq 400^{\circ} \text{ C})$ of combustion, because of gross degradation of the sample. Therefore, the HCl load value is predictive of the toxic hazard that the polyester chlorinated ducts pose during the early stage of combustion; the combined HCl and CO load values are

predictive of the total toxic hazard that the ducts pose during the later stages of combustion. At 250° C conditions, the average smoke particle diameter (d_g) , average number and concentration (n_o) , and the product of the average smoke particle diameter and concentration $(d_g n_o)$ were measured. The $d_g n_o$ is obtained from the ratio of the SPDA experimental and initial current output (I_e/I_o) following the relationship in equation 1. See also figure 3.

$$I_e/I_o = 1/(K d_g n_o) (1 - exp(-K d_g n_o)),$$
 (1)

where $K = charging constant, 0.012 cm^2/p.$

The total $d_{g}n_{o}$ values observed during each experiment for each material, adjusted to 1 g of sample mass weight loss, yielded the $d_{g}n_{o}$ load value (in particles per square centimeter per gram). Its inverse, $1/d_{g}n_{o}$ load (in square centimeters per particle per gram), has been correlated with the HCl load and with the combined HCl and CO

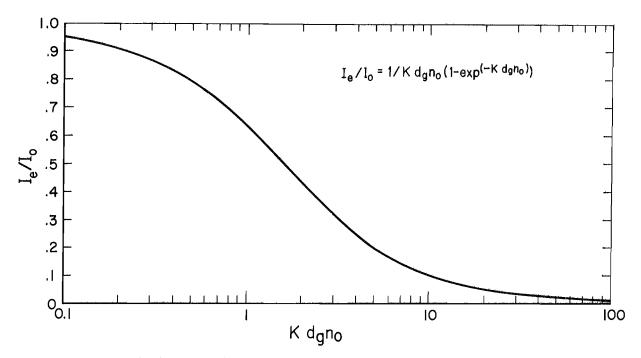


Figure 3.-SPDA current ratio as function of smoke particle diameter-concentration product.

combined HCl and CO loads (the CO load values having been previously normalized to HCl values by scaling them down by a factor of 15, because CO is at least 15 times less toxic than HCl). The $1/d_g n_o$ load values have been also correlated with the combined HCl and CO load values scaled to their STEL and IDLH values. At 1,000 °C set furnace temperature and 14-min test duration (representing the later stage of combustion at which most of the CO evolves, because of gross degradation of the sample) CO and other gas concentrations, sample weight loss, and furnace temperatures were measured.

EXPERIMENTAL PROCEDURE

Two sets of experiments (five experiments in each set, each experiment repeated three times) were performed at set furnace temperatures of 250° C (maximum furnace temperature, 300° C at the 7th minute) and 1,000° C (maximum furnace temperature, 600° C at the 14th minute) for a 14-min duration, with samples of polyesterreinforced mine ventilation ducts. A list of the materials is reported in table 2. A 1-g sample was placed in a 2.5-cm-diam cup, the furnace was set at the desired temperature, and the furnace (10 L/min) and the SPDA (1.6 L/min) airflows were also set. Syringe grab samples, stored in vacuum containers, were analyzed for CO, CO₂, and O_2 by gas chromatography; HCl concentrations were determined by short-term exposure tubes. The chlorine (Cl₂) content of the material was determined by the bombcombustion, selective-electrode method; the antimony trioxide (Sb_2O_3) content of the material was obtained from the manufacturer. Other variables measured, as a function

of time, were the sample and furnace temperatures, sample mass weight loss, and the SPDA initial voltages (without smoke) and experimental voltages (with smoke).

At $1,000^{\circ}$ C set furnace temperature and 14-min duration, CO, CO₂, and O₂ concentrations (by syringe grab samples), and sample mass loss were measured.

Table 2.-Materials investigated

Polyester-reinforced ventilation duct ¹	Cl ₂ , pct
D1	10.5
D2	4.7
D3	4.3
D4	3.8
D5	4.7
¹ Delymost component is polycostor recip reinforced (00 not) wit

¹Polymer component is polyester resin reinforced (>80 pct) with fiberglass fibers, treated for flame retardancy with chlorinated additives and antimony trioxide. All materials contained 5 pct Sb₂O₃.

At 250° C set furnace temperature, HCl was found to be the primary gas released for all ducts, depending on the percentage of chlorinated additives of the material; the time and rates of release depended on the ease of thermal material decomposition. At this stage of combustion, small CO concentrations were released by all ducts because of low thermal decomposition of the samples. The higher the HCl values, the higher the $1/d_n$ values.

At 1,000° C, large CO concentrations were released by all duct samples because of complete and fast combustion of the samples (only fibers remained).

Correlations (correlation coefficient (r) = 0.85) were established between the $1/d_g n_o$ load and the HCl load (fig. 4), between the $1/d_g n_o$ load and the combined HCl and CO loads (fig. 5), between the $1/d_g n_o$ load, the combined loads, and the HCl load (fig. 6), and between the $1/d_g n_o$ load and the combined loads scaled to their STEL and IDLH values (fig. 7).

Figure 4 shows that sample duct D1, with a chlorine content of approximately 10 pct, yielded, by far, the highest HCl load values and the highest $1/d_{\sigma}n_{o}$ load values (see

also table 3); also, duct D1 yielded the highest combined CO and HCl load values (fig. 5). Figure 6 shows that the CO load values, normalized to HCl values, did not significantly alter the HCl load values for all samples. Finally, as expected, duct D1 contained by far the highest number of STEL and IDLH values (fig. 7). Table 4 shows total sample weight losses and CO concentrations. At 250° C set furnace temperature, all ducts underwent little (>15 pct) thermal decomposition, accompanied by low CO concentrations. Maximum CO concentrations of approximately 60 ppm were released at the 10th minute by ducts D1 and D2.

At 1,000° C set furnace temperature, all samples underwent total thermal decomposition; the percentage of sample mass not decomposed contained only glass fibers, yielding large CO concentrations. The largest CO concentrations (approximately 1,500 ppm), however, were released at the 10th minute by ducts D1 and D3. Figure 8 shows smoke particle diameter-concentration product $(d_g n_o)$ versus time at 250° C. Figure 9 shows CO concentrations versus time at 250° and 1,000° C.

Table 3.-Oxidative thermal degradation data at 250° C with furnace airflow of 10 L/min

RESULTS AND DISCUSSION

Polyester-reinforced							Loa	ad		
ventilation duct ¹	HCI,	CO,	WL,	Time,	² HCl,	² CO,	³ 1/d _g n _o ,	⁴ HCI and CO,	⁵ HCI_a	nd CO
	ppm	ppm	g	min	ppm/g	ppm/g	$cm^2/(p \cdot g)$	ppm/g	STEL	IDLH
D1, 10.5 pct Cl ₂	40	130	0.1	10	400	1,300	0.0009	487	19.5	5
D2, 4.7 pct Cl ₂ [*]	15	84	.1	10	150	840	.0003	165	7	2
D3, 4.3 pct Cl ₂	11	120	.1	10	110	1,200	.00025	123	5	1.2
D4, 3.8 pct Cl_2^2	10	88	.1	10	100	880	.0002	119	5	1.2
$\underline{D5}, 4.7 \text{ pct } Cl_2 \cdots$	15	83	.1	9	150	830	.0003	165	7	1.7

IDLH Immediately dangerous to life and health.

STEL Short-term exposure limit.

WL Weight loss.

¹1-g sample.

²Each load value derives from the concentration observed at 0.1 g of sample weight loss per gram of weight loss, at a specific time and dilution air.

اًnverse of d_an_o load. Each d_an_o load is derived from total d_an_o values per gram of sample weight loss.

⁴Each value derives from the combined HCI and CO load values. Each CO load value has been previously normalized to HCl values by scaling it down by a factor of 15, because CO is at least 15 times less toxic than HCl.

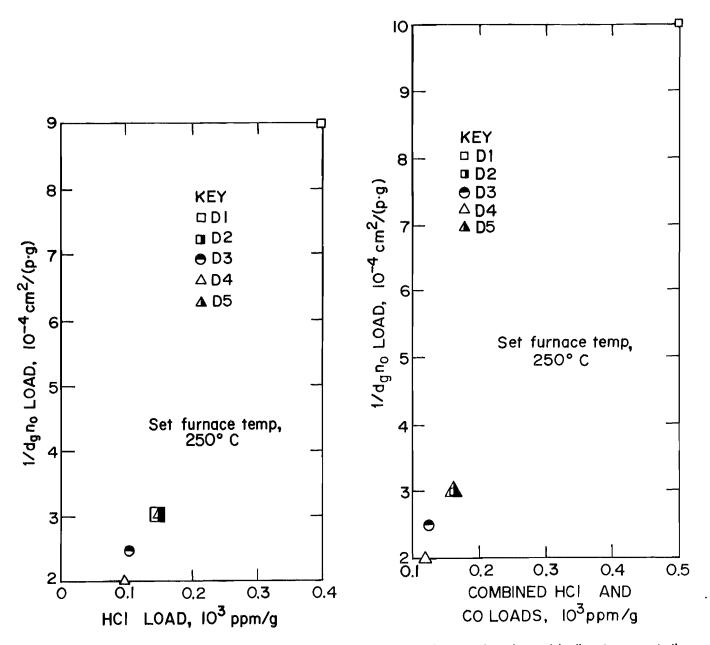
 $^\circ$ STEL and IDLH values contained in each combined load (STEL, 25 ppm ; IDLH, 100 ppm).

· · _ ·				250° C					1,000	° C		
Polyester-reinforced ventilation duct ¹	CO, ppm	Time, min	0 ₂ , pct	CO ₂ , ppm	Total WL, g	Furnace temp, °C	CO, ppm	Time, min	0 ₂ , pct	CO₂, ppm	Total WL, g	Furnace temp, °C
D1	6 9 25 60 18 12	6 8 9 10 12 14	21 20.8 20.8 20.8 20.8 21 20.8	500 500 550 550 520 500	0.13	<pre>266 320 318 311 295 280</pre>	10 50 600 1,100 520 320	6 8 9 10 12 14	20.8 20.7 20.7 20.7 20.7 20.7 20.7	700 710 990 1,200 1,100 1,100	0.41	280 380 430 450 530 600
D2	6 8 33 64 14 12	6 8 9 10 12 14	21 21 21 21 20.8 21	500 520 550 520 500 500	.12	273 323 318 311 295 281	10 49 616 948 417 303	6 9 10 12 14	20.8 20.8 20.7 20.7 20.8 20.7	690 670 960 1,190 1,010 1,020	.33	270 370 420 460 535 600
D3	7 8 7 20 14 8	6 8 9 10 12 14	21 20.8 20.8 21 20.8 20.8	520 550 580 600 580 530	.11	269 323 320 313 298 285	5 219 646 1,340 577 343	6 9 10 12 14	21 20.8 20.7 20.7 20.7 20.7	560 750 920 1,200 930 1,360	.41	273 373 417 459 534 599
D4	4 10 13 28 20 12	6 8 9 10 12 14	21 21 21 21 21 21	660 660 640 640 600 600	.14	266 321 318 311 296 281	8 48 369 764 478 369	6 9 10 12 14	20.8 20.8 20.7 20.7 20.7	620 700 910 1,130 6,150 1,980	.64	275 376 420 460 540 605
D5	5 18 23 13 7 5	6 8 9 10 12 14	21 21 21 21 21 21	550 550 580 560 600 530	.10	260 323 319 312 297 282	19 500 739 837 503 510	6 8 9 10 12 14	20.9 20.8 20.7 20.5 20.6 20.6	570 600 1,160 2,500 1,770 1,670	.41	265 263 406 447 526 558

Table 4.--Comparison of oxidative thermal degradation data at 250° and 1000° C, with furnace airflow of 10 L/min

WL Weight loss. ¹1-g sample.

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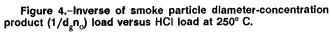


Figure 5.–Inverse of smoke particle diameter-concentration product $(1/d_g n_c)$ load versus combined HCl and CO loads at 250° C.

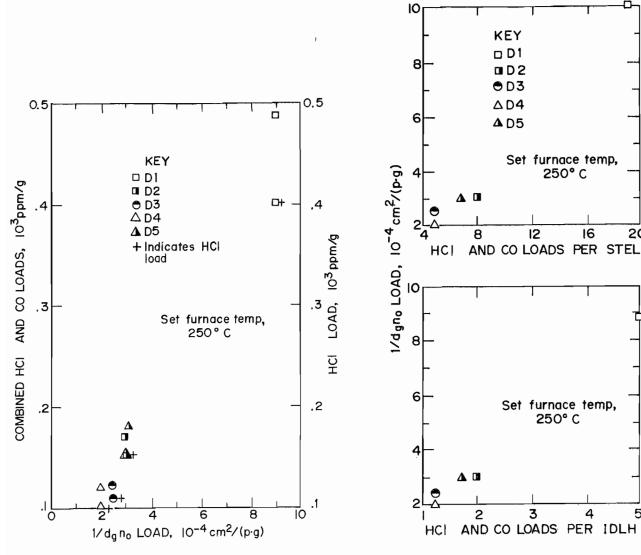


Figure 6.-Inverse of smoke particle diameter-concentration product $(1/d_en_e)$ load versus combined loads and versus HCI load at 250° C.

Figure 7.-Inverse of smoke particle diameter-concentration product $(1/d_gn_o)$ load versus combined HCI and CO loads scaled to STEL and IDLH values at 250° C.

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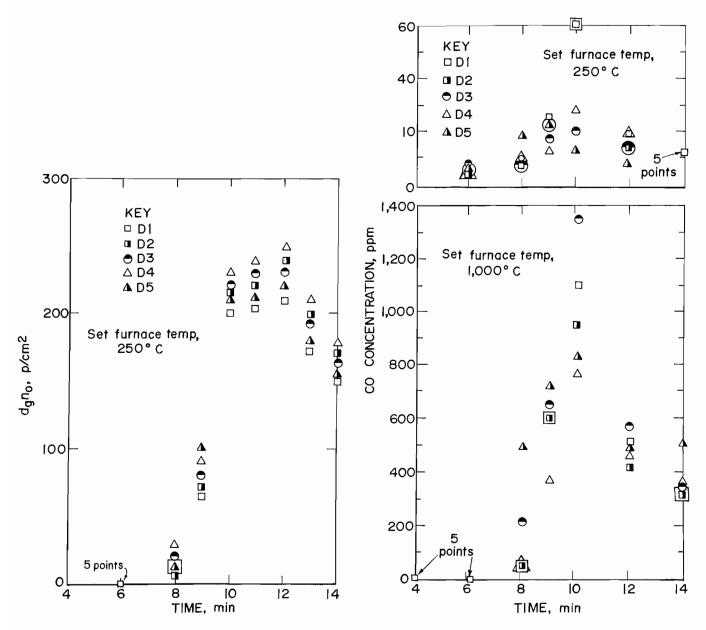


Figure 8.–Smoke particle diameter-concentration product $(d_g n_o)$ versus time at 250° C.

Figure 9.--CO concentrations versus time at 250° and 1,000° C.

CONCLUSIONS

According to the Bureau's findings, the inverse of the smoke particle diameter-concentration product $(1/d_gn_o)$ load correlates directly and significantly with the HCl and CO toxicities; HCl being the primary toxicity evolving during the early (~300° C) stage of combustion depending on the percentage of chlorinated additives of the material (CO concentrations are minimal at this stage), and CO being the primary toxicity evolving in large quantities

during a later (\geq 400 ° C) stage of combustion, which during fire can develop in a matter of minutes.

The excellent correlation of the inverse of the smoke particle diameter-concentration product $(1/d_gn_o)$ with the primary gas toxicities suggests its use as a test parameter to assess simply and inexpensively the toxic hazard that ducts pose during fire.

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