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HAZARDS CONTROL PROGRESS REPORT NO. 57, October through March 1979

R. V. Griffith: Scientific Editor

August 20, 1979

MASTER

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DISCLAIMER

There is a growing body of research on the effectiveness of various types of drug treatment programs. This research has found that the most effective programs are those that are based on the principles of behavior modification. These programs typically involve a combination of individual counseling, group therapy, and self-help materials. The research also suggests that the most effective programs are those that are tailored to the needs of the individual client. This means that the program should take into account the client's level of motivation, their social support, and their access to resources. Finally, the research indicates that the most effective programs are those that are monitored and evaluated regularly. This allows the program to be adjusted as needed to ensure that it is meeting the needs of the client.

MASTER

FOREWORD

This report is divided into three major sections. The first section, Progress Reports, covers the status of activities undertaken or continuing during the period; additional reports or separate publications will cover the final results of these activities. The second section, Technical Notes, contains reports on interesting activities of a more limited scope on which further reporting is not anticipated. The third section lists recent publications.

Readers who are interested in more detail regarding any item may contact the authors of the reports listed in the Contents.

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HAZARDS CONTROL PROGRESS REPORT NO. 57

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PROGRESS REPORTS

FIRE SAFETY

Gas Chromatographic/Mass Spectrometric Analysis of Thermal Degradation Products from Wood and Composite Burns

Introduction. In fiscal year 1979 we chemically analyzed the combustion products from wood and composite burns to determine the cause of HEPA filter plugging.¹ Our preliminary results suggested that filter plugging might be caused by the concentration of thermal degradation products rather than by the production of specific chemicals. This year, using a more advanced method, we reanalyzed the combustion products to verify the 1979 findings.

Experimental Method. Wood cribs and cribs constructed of polyvinylchloride, wood, polymethylmethacrylate, polycarbonate, and fiber-reinforced plastic (composite cribs) were burned in a large fire test cell, and the combustion products were directed

into the ductwork, which houses a rolling prefilter and a HEPA filter (Fig. 1). For a better understanding of the combustion products and their effect on the filters, we sampled products upstream and downstream of the HEPA filter and upstream of the rolling prefilter. The various sampling points are shown in Fig. 1.

We used a vacuum pump to pull the combustion products into glass traps set in a Dewar flask filled with liquid nitrogen. The products were sampled at 5-min intervals, beginning 5 min after the onset of the burn. Each sampling period lasted 5 min. This approach allowed us to include any possible changes in either the quantity or kinds of combustion products generated during the burn. After the samples were trapped, they were brought to room temperature, extracted with chloroform, and stored in a refrigerator until gas chromatographic/mass spectrometric (GC/MS) analysis.

FIG. 1. Ductwork housing the rolling prefilter and the HEPA filter. Sampling points are shown.

For a better understanding of the types of products coating the HEPA filter and the prefilter, we extracted a concentrated 2-g sample of each filter with 60 ml of chloroform in a Soxhlet extractor. In this process hot solvent is vaporized, condensed, and dropped on the solid substance contained in the thimble. The extracted compounds contained in the liquid fill the body of the extractor and automatically siphon into the flask. The process continues repeatedly as the solvent is vaporized and condensed.

The degradation products were analyzed on two capillary glass columns, the SP 2100 and the SE-30. Both columns were 30 m long and had approximately 60,000 theoretical plates. Column temperature was programmed from 90 to 225°C at a rate of 5°C/min, and injection port temperature was set at 300°C. The flame ionization detector oven, its transfer line, and the transfer line between the gas chromatograph (GC) and the ion source of the mass spectrometer (MS) were maintained at 250°C. Nitrogen, our make up gas, was set at a flow rate of 60 ml/min at the detector's inlet. A platinum-iridium line split the effluent between our

quadrupole MS and the flame ionization detector, allowing simultaneous recording of the chromatographic peaks and total ion counts as a function of time. We used the computerized Wiley library containing the mass spectra* to find the dominant peaks in the chromatograms and then compared the spectra of suspected compounds with the spectra of each chromatographic peak to further identify the components.

Results. The results of the GC/MS analysis are summarized in Tables 1-5. Each table shows the burn parameters; the compounds collected on, near, and extracted from the HEPA filter or the prefilter; the retention time for each compound (i.e., the time required for it to elute off the column from the time of injection); and the concentration of each compound. We have expressed the concentrations in terms of peak heights; keep in mind, however, that these are not absolute concentrations because we did not use internal standards in our analysis.

*Compiled by Hewlett-Packard.

Table 1. Degradation products from wood burn 41.

Burn parameters	Retention time, min ^a	Products upstream of HEPA filter	HEPA filter extracts	Products downstream of HEPA filter	Semi-quantitative comparison ^b
Moisture content of wood (Douglas fir) was 8-11%.	1.70		phenol		3
	1.90	<i>o</i> -phenol, benzaldehyde acetophenone			1
Prefilter was not modified.	2.20	2,2-ar-methylstyrene, <i>o</i> -methylstyrene, <i>a</i> -methylstyrene			2
	2.38			<i>o,m,p</i> -cresols	3
Temp max in cell, 740°C	2.57		<i>o,m,p</i> -cresols		3
	2.70	<i>o,m,p</i> -cresols, indene methylphenylacetylene			3
in cell at exhaust, 70°C	3.30	2-methylbenzofuran			2
	3.47		3,4-dimethylphenol	3,4-dimethylphenol	2.1 ^c
in duct at HEPA dry, 60°C wet, 60°C	3.72		methylphenol	methylphenol	2.1
	4.05	1-methyl-1H-indene			3
Ventilation initial, 250 l/s final, 125 l/s	4.25		naphthalene or azulene	naphthalene or naphthalene	3.5
	4.50	naphthalene or azulene			5
Time to plugging, 810 s	5.62		indane-1-one		1
	6.20		1-methylnaphthalene	1-methylnaphthalene	2
	6.32		2-methylnaphthalene or 5,6-benzobicyclo (2,2,1)-hept-2-ene	2-methylnaphthalene or 5,6-benzobicyclo (2,2,1)-hept-2-ene	2
	6.70	2-methylnaphthalene or 5,6-benzobicyclo (2,2,1)-hept-2-ene			3
	6.90		trans-cinnamic acid		1
	7.47		biphenyl	biphenyl	2

Table 1. Continued.

Burn parameters	Retention time, min ^a	Products upstream of HEPA filter	HEPA filter extracts	Products downstream of HEPA filter	Semi-quantitative comparison ^b
	7.87		benzo (c) cinoline		1
	7.90	biphenyl			2
	8.30	1,2-dimethylnaphthalene			1
	8.80		acenaphthalene	acenaphthalene	4
	9.00	unidentified			2
	9.40	acenaphthalene or biphenylene			3
	9.60			6-methyl-6-phenylfulvene	1
	10.10	unidentified			1
	10.23		dibenzofuran	dibenzofuran	2
	10.70	unidentified			1
	11.90		4-methyl fluorene		2
	12.10	unidentified			1
	12.30		fluorene or diphenyl diazomethane		2
	12.50	unidentified			1
	15.40		phenanthrene or anthracene	phenanthrene or anthracene	5,3
	16.17	phenanthrene			2
	16.90		1-methyl-3-phenylindane	1-methyl-3-phenylindane	1
	17.70		9,10-phenanthraquinone or 9,10-anthraquinone	9,10-phenanthraquinone or 9,10-anthraquinone	2
	19.00		dibenzooheptafulvene or 2-phenylnaphthalene	dibenzooheptafulvene or 2-phenylnaphthalene	1
	20.70		pyrene or fluoranthene	pyrene or fluoranthene	3,1
	21.00		1,2,3,10B-tetrahydro fluoranthene		2
	21.57		pyrene	pyrene	3,1

^aThe amount of time required for a compound to elute off the column from the time of injection.

^bConcentrations are expressed in terms of peak heights: 5 indicates a very prominent peak (a very high concentration); 4, a prominent peak (a high concentration); 3, a medium size peak (a medium concentration); 2, a small peak (a low concentration); and 1, a very small peak (a very low concentration).

^cTwo numbers signify that (1) the same compounds have emerged from the GC column at the same time or (2) that a peak is comprised of two different compounds.

We found that the pyrolysis and combustion products from the wood burns and the composite burns were similar, probably because wood was a major component of the composite cribs. Both types of burns generated a number of aromatic and polynuclear components (e.g., cresol, phenanthrene, naphthalene), some of which are the dominant compounds formed during the first 5 min of the burns. Most of the dominant compounds have high molecular weights and high boiling points (above 200°C) and are heavier than water, solid in their pure form, and soluble in organic solvents (Table 6).

We detected substantial amounts of polycyclic compounds upstream of the HEPA filter, downstream of the prefilter, on the HEPA filter, and on the prefilter but found only insubstantial amounts downstream of the HEPA filter.

In comparing the smoke constituents from wood and composite burns, we find that composite burns generate more aerosol isomers and more phenols than wood burns and that wood burns form more polynuclear components but fewer simple aromatics than composite burns. In addition, composite burns generate HCl, HBr, and probably a number of halogenated organics. The halogenated

Table 2. Degradation products from wood burn 42.

Burn parameters	Retention time, min ^a	Products upstream of HEPA filter	Products downstream of HEPA filter	Semiquantitative comparison ^b
Moisture content of wood (Douglas fir) was 7-11%.	1.90	<i>o</i> -phenol	No significant peaks	2
	2.80	<i>o</i> , <i>p</i> -cresols		2
	4.00	<i>m</i> , <i>p</i> -ethylphenols		1
Prefilter consisted of a single layer of 50% efficiency media backed by a plastic grid.	4.60	azulene or naphthalene		2
	6.00	indian-1-one		1
	6.40	styrene		1
	6.80	2-methylnaphthalene		1
Temp max in cell, 750°C	9.40	acenaphthalene		3
in cell at exhaust, 250°C	10.40	2-naphthol		3
in duct at HEPA dry, 80°C	10.80	dibenzofuran		3
wet, 68°C	11.80	phenalene		2
Ventilation initial, 260 l/s	12.23	fluorene		2
final, 198 l/s	12.67	4-methylfluorene		2
Time to plugging, 510 s	13.50	unidentified		2
	14.60	<i>m</i> -phenylphenetole		2
	15.20	unidentified		2
	16.30	phenanthrene or anthracene		5
	17.70	dibenzosheptulvene		2
	18.50	9-fluorenone		3
	19.00	4,5-methylene-phenanthrene		3
	19.60	dibutylphthalate		2
	19.90	unidentified		2
	20.60	unidentified		1
	21.60	fluoranthene		4
	22.10	1,3,3,10- β -tetrahydro-fluoranthene		2
	22.70	pyrene		4
	25.15	2,3-benzofluorene		1
	25.17	4-methylpyrene		1
	26.90	2,3-benzofluorene		1
	31.33	2,13-benzofluoranthene		2
	34.30	3,4-benzophenanthrene		2

^aThe amount of time required for a compound to elute off the column from the time of injection.

^bConcentrations are expressed in terms of peak heights: 5 indicates a very prominent peak (a very high concentration); 4, a prominent peak (a high concentration); 3, a medium size peak (a medium concentration); 2, a small peak (a low concentration); and 1, a very small peak (a very low concentration).

organics, however, were not detected on the columns we used.

If we were to analyze the physical properties of the main constituents of both types of burns, we would probably find that the dominant compounds dissolve in some of the organic solvents and become airborne through the ductwork in the form of a fine mist that condenses on the walls of the ductwork and the HEPA filter. Furthermore, because the temperature near the HEPA filter is between 50 and 60°C, we could probably expect compounds with

higher boiling points to deposit on the filter.

Conclusion and Recommendation. The overall results of this study suggest that filter plugging is not caused by the unique chemical makeup of one compound or group of compounds but rather by a concentration effect.

Because our analytical method was aimed at detecting only the predominant differences among the degradation products of each burn, we did not look at trace components or examine how the various compounds detected were formed. The GC

Table 3. Degradation products from composite burn 43.

Burn parameters	Retention time, min ^b	Products upstream of HEPA filter	Products downstream of HEPA filter	Semiquantitative comparison ^c
Composite crib made of wood (Douglas fir)	1.22	<i>o</i> -xylene	No significant peaks	5
with a moisture content of 7-8%, polyvinylchloride, polymethylmethacrylate, polycarbonate, and fiber-reinforced plastic. ^a	1.30	cyclooctatetraene		4
	1.67	phenol		3
	1.83	isopropylbenzene		2
	1.93	cyclopropylbenzene or allylbenzene		2
	2.30	methylphenylacetylene		3
	3.28	1,2-dihydronaphthalene		2
Prefilter consisted of two layers of 50% efficiency media and one layer of 10% efficiency media.	3.60	naphthalene		4
	5.67	biphenyl		2
	10.22	phenanthrene		2
Temp max in cell, 640°C				
in cell at exhaust, 160°C				
in duct at HEPA dry, 58°C				
wet, 48°C				
Ventilation initial, 250 l/s				
final, 220 l/s				
Pressure drop across filter initial, 200 Pa				
final, 540 Pa				
Time to plugging, 410 s				

^aNot fire retarded.^bThe amount of time required for a compound to elute off the column from the time of injection.^cConcentrations are expressed in terms of peak heights: 5, indicates a very prominent peak (a very high concentration); 4, a prominent peak (a high concentration); 3, a medium size peak (a medium concentration); 2, a small peak (a low concentration); and 1, a very small peak (a very low concentration).

columns used in this analysis were not specific for halogenated compounds, and therefore, a number of the compounds that may have formed in the composite burns may have gone undetected. These could have been irreversibly absorbed on the columns or perhaps formed in such low quantities that they were masked by other products. In the latter case, they would not have been detected by the MS because of background interference.

A more detailed analysis of the degradation products and their mechanism of formation would require that we:

1. Use capillary columns specific for halogenated compounds and polynuclear compounds.

2. Pyrolyze wood and plastic samples separately and introduce their degradation products directly onto a GC column before they have a chance to react and form secondary and tertiary reaction products.

3. Develop a sampling system that traps pyrolysis and combustion products as well as trace components.

4. Devise a sample work-up technique to use before we inject the samples onto the analytical column.

However, we believe that we have found the major cause of HEPA filter plugging and that further research in this area would be of secondary importance to filtration chemistry.

Table 4. Degradation products from composite burn 45.

Burn parameters	Retention time, min ^b	Products upstream of HEPA filter	Products behind prefilter (sampled in the first 5 min)	Semiquantitative comparison ^c
Composite crib made of wood (Douglas fir) with a moisture content of 8.5%, fiber-reinforced plastic, polycarbonate, and polymethylmethacrylate.	1.20	<i>o,p</i> -xylenes		5
		ethylbenzene		
	1.27	cyclooctatetraene		3
	1.57	phenyl-N-methylcarbonate		3
Prefilter consisted of one layer of 10% efficiency media, one layer of 90% efficiency media, and one layer of 50% efficiency media.	1.92	11-ethoxytricyclo (4,3,2,0)		2
		10-azo-2,4,10-undecatriene		
	2.37	<i>o</i> -cresol		4
		methylphenylacetylene		
Temp max in cell, 670°C	3.45	<i>p</i> -ethylphenol		3
	4.07	naphthalene		4
	4.35	<i>m</i> -isopropylphenol or <i>o</i> -isopropylphenol or <i>p</i> -isopropylphenol		2
		1-methylnaphthalene		
in cell at exhaust, 155°C	5.80	5,6-benzobicyclo (2,2,1)		2
	6.07	hept-2-ene		
	7.20	biphenyl		2
	8.58	biphenylene		2
in duct at HEPA dry, 50°C	11.27	hydroxy-3,6-dimethyl benzaldehyde		2
wet, 60°C	11.70	1,1-bicycloheptatriene		1
Ventilation initial, 250 l/s	12.07	diphenyldiazomethane		1
final, 230 l/s	12.65	diphenylpropane		1
Pressure drop across filter initial, 200 Pa	12.97	<i>p</i> -alpha-amyphenol		1
	13.67	1,1-diphenylethane		1
final, 500 Pa	13.67	1,1-diphenylethane		1
		anthraquinone		1
	14.20	phenanthrene		2
	15.20		<i>di-N</i> -propylnaphthalate	1
	19.12		<i>di</i> -2-ethylhexylnaphthalate	5
	23.73		butyl-butoxyethylnaphthalate	3
Time to plugging, 880 s	26.98			

^aNot fire retarded.^bThe amount of time required for a compound to elute off the column from the time of injection.^cConcentrations are expressed in terms of peak heights: 5 indicates a very prominent peak (a very high concentration); 4, a prominent peak (a high concentration); 3, a medium size peak (a medium concentration); 2, a small peak (a low concentration); and 1, a very small peak (a very low concentration).

Corrosion in the Experimental Ducting of the Fire Test Cell

In January of 1979 we completed replacement of the experimental ducting connected to the fire test cell (Fig. 2) operated by the Fire Science Group of Hazards Control, Special Projects Division. The ducting is constructed of 16-gauge carbon steel sheeting, material similar to that used in most commercial applications. The temperature of the gases entering the ducting varies; it can be as high as 250°C, though it averages about 140°C.

Since January 1976, when the first experimental fire was ignited, the ducting has been exposed to

smoke and combustion products of approximately 75 fires. Typical fuels tested were Douglas fir wood, polyvinylchloride (PVC) sheet, fiberglass-reinforced polyester resin, polycarbonate sheet, and acrylic sheet (PMMA-FR). Fuel arrangements were from simple crib to complex full-scale laboratory mockup configuration.

Chemical analysis of the smoke showed a large concentration of hydrochloric acid and an indication of hydrobromic acid. Hydrochloric acid can be generated by the combustion of PVC, hydrobromic acid by the combustion of PMMA-FR. Both caused corrosion in the ducting. Coating the inside of the

Table 5. Degradation products from composite burn 46.

Burn parameters	Retention time, min ^b	Soxhlet extract of prefilter	Products sampled behind prefilter			Semi-quantitative comparison ^c
			5 min	10 min	15 min	
Composite crib made of wood (Douglas fir)	1.37				ethylbenzene	3
	1.40			ethylbenzene		
	1.48				styrene, cyclooctatetraene	5
with a moisture content of 8.5%, fiber-reinforced plastic, a polycarbonate, and polymethylmethacrylate.	1.50			styrene, cyclooctatetraene		3
	1.60	phenol				3
	1.97			phenol		3
	2.00				phenyl methylcarbonate	4
Prefilter consisted of one layer of 10% efficiency media,	2.22				isopropyl benzene	2
one layer of 90% efficiency media,	2.25			4-methylstyrene		2
and one layer of 50% efficiency media.	2.37		1-(2-ethoxypropoxy) propanol-2	4-methylstyrene, ar-methylstyrene, allylbenzene cyclopropylbenzene		1, 2 ^d
					allylbenzene phenylcyclopropane, ar-methylstyrene	2
						2
Temp max in cell, 800°C	2.90				<i>o,p</i> -cresols	4
in cell at exhaust, 150°C	2.95			<i>o,p</i> -cresols		5
in duct at HEPA dry, 50°C	3.70	<i>p</i> -ethylphenol				3
wet, 50°C	4.30	naphthalene				3
	4.32			<i>m,p</i> -ethylphenols		2
	4.45			1,2-dihydronaphthalene		2
Ventilation initial, 250 l/s final, 30 l/s	4.57		naphthalene or azulene			2
	4.68	<i>m</i> -isopropylphenol				2
Pressure drop across filter initial, 250 Pa final, 2950 Pa	4.93			naphthalene, azulene	naphthalene	4, 4
	5.32			<i>o</i> -isopropylphenol or 2,4,5-trimethylphenol or <i>m</i> -isopropylphenol		1
Time to plugging, 1320 s	6.20	1-methylnaphthalene			3-isopropylphenyl-N-methylcarbonate	1, 1
	6.52	1,2,3-trimethyl-5-pyrazolone				3, 1
	6.92			1-methylnaphthalene or 2-methylnaphthalene		2
	7.72	biphenyl or acenaphthalene				2
	8.50			acenaphthalene or biphenyl		2
	9.13	acenaphthalene	acenaphthalene			2, 2
	9.87	diphenylmethane, allylnaphthalene, acenaphthalene				1
	10.00			acenaphthalene or biphenylene		2
	10.57	dibenzofuran				1

Table 5. Continued.

Burn parameters	Retention time, min ^b	Soxhlet extract of prefilter	Products sampled behind prefilter			Semi-quantitative comparison ^c
			5 min	10 min	15 min	
	12.42	4-methylfluorene, 1,1-bicycloheptatriene				1
	12.80	diphenyldiazomethane				1
	15.37			fluorene	fluorene	2,1
	15.52		phenanthrene	4-methylfluorene		2
	15.92	phenanthrene				3
	16.88	anthracene			phenanthrene	1,1
	16.93			phenanthrene		2
	17.32	para-diphenylphenol				2
	18.38			unidentified		1
	19.27			1-methylphenanthrene		1
	19.58	dibenz(a,h)fluorene				1
	19.67			4-methylphenanthrene		1
	20.65			2-phenylnaphthalene		1
	21.25	fluoranthene or pyrene				2
	22.28			fluoranthene		1
	22.93	pyrene		1,2,3,4-tetrahydrofluoranthene		5,1
	23.45					
	34.40	naphthalene		pyrene		1

^aNot fire retarded.^bThe amount of time required for a compound to elute off the column from the time of injection.^cConcentrations are expressed in terms of peak heights: 5 indicates a very prominent peak (a very high concentration); 4, a prominent peak (a high concentration); 3, a medium size peak (a medium concentration); 2, a small peak (a low concentration); and 1, a very small peak (a very low concentration).^dTwo numbers signify that (1) the same compounds have emerged from the column at the same time or (2) that a peak is comprised of two different compounds.

ducting with a special corrosion-resistant paint or resin—which we did not do when the duct was initially installed—would have prevented this.

The major areas of corrosion are shown in Fig. 3a-g. The most severe corrosion took place at the weld locations (Fig. 3b), probably because of the stress areas set up during the welding of the sheet metal to the angled iron frame. Large flakes of rust found in the bottom of the ducting corroborate this. Other points of severe corrosion are indicated in Fig. 3d, f, and g. Very little corrosion was noted on the slanted section connecting the high- and low-exhaust points (Fig. 3c).

When we installed the new ducting, we noticed that in the early morning there was a large amount of moisture on the upper inside surfaces. This

moisture would accelerate corrosion and account for corrosion on the upper inside surfaces. In an attempt to reduce this problem, we coated the inside surfaces with an epoxy paint recommended by our paint shop. This coating should provide a high resistance to corrosion caused by hydrochloric acid.

ENVIRONMENTAL PROTECTION

On-Line X-Ray Fluorescence Analysis of Transition Metals in Waste Water: Phase II

Introduction. Previously we reported the encouraging results of a feasibility study on the use of x-ray fluorescence analysis (XRFA) in an on-line monitor for metals in waste water.² We have subsequently built a prototype monitoring system utilizing a dedicated model LSI-11 microprocessor

Table 6. Physical constants of the main organic compounds.

Compound	Physical appearance	bp, °C	mp, °C	Density	Solubility
<i>o,p,m</i> -xylenes	liquid, solid	138-144	-47-13	0.8684	alcohol, ether, organic solvents
phenanthrene	blue fluorescent (in solution)	340	101	1.1820	sol in alcohol; slightly CS ₂
ethyl benzene	liquid	136	-94	0.8672	alcohol, ether
<i>o,m,p</i> -cresols	liquid, but solid in the case of <i>p</i>	191-201	11-34.8	1.0460	alcohol, ether, organic solvents
ethyl phenol (para)	solid needles	219	45-46		alcohol, ether, benzene, CS ₂
naphthalene	solid monoclinic	210	80.22	1.1450	ether, alcohol, benzene, CS ₂
fluoranthene	pale yellow needles	375	111	1.2520	ether, alcohol (slightly), benzene, CS ₂
pyrene	pale yellow	260	149-150	1.2710	sol in alcohol
acenaphthalene	solid (platelet (alcohol); prism (ether))	265-275	92	0.8988	alcohol, ether, benzene
2-naphthol	monoclinic leaflet	295	122	1.2800	alcohol, ether, benzene, slightly SO ₂
dibenzofuran	leaflet or scales (alcohol);	287	86-87	1.0886	alcohol, ether, benzene, acetic acid
anthracene	solid	340	216	1.2500	benzene, chloroform, toluene

FIG. 2. Experimental ducting of the fire test cell.

FIG. 3. Major areas of corrosion in the experimental ducting of the fire test cell.

(Digital Equipment Company, Maynard, Mass.) for data collection and analysis. We have tested and calibrated the prototype in the laboratory, installed it on-line, and are now in the process of "debugging" it.

Equipment. Our earlier work showed that the best results were obtained when a sample of the waste water stream flowed under the detector assembly in the form of a thin (1 mm) layer, backed by a polyethylene film and air. These measures are necessary to optimize signal-to-background ratios. "Background" in this instance is the signal from pure water, which is caused primarily by scattered photons from the exciting source. The geometry shown in Fig. 4 was found to be best. The sample flume, complete with detector-source holder, is shown in Figs. 5, 6, and 7. The exciting source is about 70 mCi of ^{109}Cd in an approximately 1-cm-diam disk configuration. The detector is an inexpensive Xe CO_2 -filled proportional counter. Signals from it are routed via an ORTEC 109PC pream-

plifier to a Tenelec TC-202BLR amplifier and an analog-to-digital adjustor (ADC), designed and built by R. Kiefer of our Electrical Engineering Group. The original ADC had severe differential linearity problems so we eventually replaced it with one of higher resolution and "threw away" the least significant data bits. The digital data are stored as a 256-channel pulse-height spectrum in the LSI-11 microprocessor.

Data Reduction. Twelve spectral regions of interest are defined by software: one centered on the predominant x-ray line for each of eleven elements,* and one centered on the large peak from the Compton-scattered photons from the source. Counts are accumulated for some moderate period (currently set at 500 s) and the integrals for each region are found. Each x-ray region integral is divided by the scatter integral. Using this ratio eliminates errors caused by source decay, moderate

*Cr, Mn, Fe, Co, Ni, Zn, As, Se, Hg, and Pb.

FIG. 4. Cross section of source-detector geometry for the XFRA monitor.

FIG. 5. Overall view of the XRFA flume and detector assembly.

FIG. 6. The XRFA flume and detector assembly with covers installed.

FIG. 7. Close-up of the XRFA source-detector and window.

sample thickness changes, dead time losses, and timing inaccuracy. The ratios for a "blank" sample (distilled water), stored in the program, are subtracted and the net signal, if any, is compared with the standard deviation (σ) of the blank spectrum. If the net signal is less than 3σ above the blank, no action is taken. If it is between 3 and 10σ , the concentration for that element is calculated and printed out as an advisory. If it is greater than 10σ above the blank, the alarm circuit is activated as well. After this action, the most recent 500-s count (for each element) is placed into an array containing the nine previous 500-s counts. These are then summed to give an effective "running" 5000-s count. Then, the same series of inspections is performed, and advisories or alarms are initiated from the running total data. This dual register system allows both a rapid response to a high concentration and a more sensitive response to low concentrations in a time frame 10 times longer. The times, incidentally, along with many other system parameters, are easily changed by teletype inputs to the program. Data acquisition and timing are accomplished with assembly language interrupt routines; data-handling routines are written in FORTRAN for easy modification.

Results. We calibrated the instrument in the laboratory using standard solutions of each metal that ranged from 60 to 1000 ppm. The solutions were recirculated through the flume, and data were collected for at least 5000 s per run. The calibration curves thus obtained (Fig. 8) were stored in the microprocessor. From these data, we calculated

sensitivities and minimum detectable concentrations for the 11 elements of interest. These figures, for both 500- and 5000-s counting times, are shown in Table 7.

Table 8 shows the L.L. maximum permissible discharge concentrations for all elements with established limits. If we compare Tables 7 and 8, we find that, at maximum permissible concentration, only zinc will alarm in 500 s. However, all the metals will alarm at 5000 s.

It should be noted that sensitivity changes linearly with source strength and as the square root of counting time. Consequently, the source has a limited useful life (half-life for ^{109}Cd is 453 days), but it can be extended somewhat by lengthening the counting periods. The source is nearing the end of its life now; replacement will be required in approximately 6 months.

FIG. 8. Calibration curves obtained with the XRFA monitor.

Table 7. Minimum detectable and alarm levels for the XRFA monitor.

Element	500-s counting time		5000-s counting time	
	Minimum detectable (3σ above blank), ppm	Alarm (10σ), ppm	Minimum detectable (3σ), ppm	Alarm (10σ), ppm
Cr	35.6	118.7	11.2	37.3
Mn	23.4	78.0	7.4	24.7
Fe	14.9	49.7	4.7	15.7
Co	9.8	32.6	3.1	10.3
Ni	6.3	21.0	2.0	6.7
Cu	4.9	16.3	1.6	5.3
Zn	4.1	13.7	1.3	4.3
As	2.2	7.3	0.7	2.3
Se	2.2	7.3	0.7	2.3
Hg	5.0	16.7	1.6	5.3
Pb	3.6	12.0	1.1	3.7

Table 8. LLL maximum permissible discharge concentrations for metals in sewage.

Metal	Maximum permissible discharge concentration, ppm
Cr	100
Cu	10
Zn	50
Ni	10
Combinations	Total <100

Problems. In order to provide the thin sample layer, the sample must be free of large particulates. We purchased a 1-1/2 BHP macerator allegedly capable of grinding any solids to <100- μ m diam. This unit has given us a great deal of trouble: it becomes clogged with the very materials it is supposed to grind. Currently we are redesigning the plumbing with the hope of removing this problem.

Future Plans. The system has been installed on-line, and with the exception of the plugging problem, seems to be operating properly. We plan to gather data and experience for weeks or even months before hooking up the alarm circuits; numerous false alarms tend to generate a credibility gap among operational personnel that is difficult to eradicate! We anticipate that minor software changes will be required and that other plumbing problems will arise. If all goes well with this installation, an even more sensitive system utilizing a gas scintillation proportional detector* will be considered.

*Not built yet.

INDUSTRIAL HYGIENE

Fire Environmental Tests for Self-Contained Breathing Apparatus

We are concerned that National Institute for Occupational Safety and Health Certification tests for self-contained breathing apparatus (SCBA) do not adequately evaluate the performance of SCBA under fire conditions. We know that the environment to which a fire fighter and his equipment are exposed is the result of many variables such as high air temperature, high humidity, water droplets in the air, steam, fire decomposition products, fire-extinguishing agents, and radiant heat. We think a fire fighter's protective gear should not only give him the protection he needs for hazardous work,

but also should provide added protection in the case of unforeseen emergencies such as flarups.

We centered our studies on evaluating existing equipment so SCBA could be selected from devices that offer the best protection against conditions that could exist during a fire at the Laboratory. Because a literature search revealed very little information on such evaluations, we developed test parameters and criteria to evaluate various makes and models of SCBA.

The results of our subjective tests to high and low air temperatures, our instrumented tests to high and low air temperatures, and our thermal shock tests have all been published previously.^{3,5} This report covers the results of the moisture test.

Parameters and Equipment for Moisture Tests.

The water present at a fire scene may be in various forms—from a fine mist similar to water from a hose nozzle overspray to a deluge of run-off water from an upper level. A fine mist would appear to create only visual problems. However, large volumes of water might interfere with the operation of SCBA. To determine what effects large volumes of water would have on the operational characteristics of SCBA, we tested various makes or models of SCBA beneath a large shower head. A solenoid was placed in the waterline to provide an instant on or off. We tested two of each make or model of SCBA, all of which were either new or maintained in top condition by manufacturer-certified personnel. The facepiece for each SCBA was mounted on a dummy head; the entire SCBA was then connected to the LLL mechanical breathing machine located outside the shower chamber. The breathing machine was set on a moderate to heavy work rate of 52.5 l of air/min to simulate a fire fighter at work. The difference between atmospheric and internal facepiece pressure was measured with a calibrated pressure transducer and monitored on a strip-chart recorder.

The SCBA unit, including the facepiece, was thoroughly inspected and tested for leaks before we set it up in the shower chamber. We operated it for 1 min to ensure that it was working properly and to measure its normal operating pressures. The shower was then turned on and the SCBA was deluged with water (Fig. 9). The unit was exposed to this water flow for the length of time it took to empty the air cylinder. The total operating time and the time at which the low air pressure alarm went off were noted.

FIG. 9. Moisture test setup for SCBA. The breathing machine was located outside the test chamber.

Table 9. Moisture test for pressure-demand SCBA.

Make of SCBA	Facepiece pressure before shower, H ₂ O"		Facepiece pressure during shower, H ₂ O"	
	Inhalation	Exhalation	Inhalation	Exhalation
Globe	0.10	1.21	0.10	1.20
MSA	0.00	1.95	0.00	1.95
Scott 6000	-0.20	2.46	-0.20	2.46
Scott 9000	0.80	2.39	0.80	2.39
Scott 4.5	0.13	1.76	0.13	1.76
Survivair	0.20	1.80	0.20	1.80

Test Results. Two each of the following makes and models were tested under the water stream:

- Scott 6000
- Scott 9000
- Scott 4.5
- Globe Guardsman
- MSA 401
- Survivair

The operational characteristics of all units tested were unaffected by the large amount of water, as shown in Table 9.

INSTRUMENT DEVELOPMENT

Developments in Neutron Spectrometry

A program to develop neutron spectrometry systems for radiation protection measurements has existed in Hazards Control for several years.⁶⁻¹⁴ This effort resulted in the system described by Slaughter *et al.*¹⁵ Recently the emphasis of the program has shifted towards optimizing the operating parameters of the system and improving our data reduction techniques. Thus, in this report, we briefly discuss (1) the energy range required for neutron spectroscopy measurements used in radiation protection, (2) the means of estimating the resolution required, (3) some advances in pulse-shape discrimination, (4) the light output of the NE213 organic liquid scintillator for protons near 1 MeV, and (5) the means of estimating a proportional counter's response to neutrons with energies greater than its upper detection limit.

Energy Range. Neutron spectroscopy has many applications in radiation protection. We use it (1) to obtain information for correcting the response of dosimeters; (2) to provide data for shielding calculations

and to determine the efficacy of shields; (3) to make direct measurements of dosimetric quantities, particularly in regions poorly covered by existing dosimeters; and (4) to characterize calibration sources.

Although we can measure the neutron energy spectrum from a few kiloelectron volts to several tens of megaelectron volts by using a combination of detectors,¹⁶⁻¹⁸ we find that the actual energy range may be considerably less. Thus, to determine what energy range to consider, we assume that, for radiation protection purposes, an adequate measurement of the neutron spectrum is one for which neither the neutrons below the low-energy limit nor those above the high-energy limit produce more than 5% of the total dose equivalent.

Based on this assumption, we calculated the energies below which 5 and 95% of the total dose equivalent occurred for 121 of the neutron spectra compiled by Ing and Makra.¹⁹ The cases considered were the ²³⁵U Maxwellian and Cranberg fission spectra; the Jezebel, Godiva IV, and Flattop critical assemblies; the HPRR, VIPER, and IBR reactors; homogeneous H₂O and D₂O fissile solutions; and the Cranberg spectrum transported through H₂O, D₂O, concrete, Fe, Cu, Pb, and ²³⁸U. Also considered were H₂O and D₂O moderated spectra transported through concrete, Fe, Cu, and Pb; and 14.6-MeV neutrons transported through concrete, Fe, Cu, Pb, and ²³⁸U. The factors required to convert the neutron spectra to dose-equivalent spectra were obtained by linear interpolation between the maximum dose-equivalent-per-neutron-fluence values given by Auxier *et al.*²⁰

The results of the calculations are summarized in Table 10, which shows the percentages of spectra requiring measurements below some typical low-energy thresholds. The majority of the spectra, even those from 14.6-MeV neutrons, require measurements below 500 keV, which is often the practical lower limit for measurements taken with organic

Table 10. The percentage of neutron spectra requiring measurements below the given threshold energies.

Threshold, keV	Fission neutrons, %	14.6-MeV neutrons, %	Total, %
10	26.4	3.3	20.7
50	39.6	3.3	30.6
100	49.5	13.3	40.5
500	100.0	56.6	89.3

scintillators. Upper energy limits are shown in Table 11, which gives the percentages of spectra requiring measurements above various high-energy limits. As expected, most spectra produced by 14.6-MeV neutrons require measurements above 10 MeV (actually 14 MeV), but only a few of the fission neutron spectra need go above 7 MeV. Being able to limit the dynamic range is of considerable practical value.

Measuring neutron spectra below 10 keV is too difficult to attempt routinely for radiation protection purposes. However, the dose equivalent per neutron fluence varies so little below 10 keV that detailed neutron spectra in this region may not be necessary. If we assume no measurements will be made below 10 keV, we can reconsider the usefulness of making measurements below 50 keV. Since only 13.2% of the fission spectra and none of the 14.6-MeV spectra require thresholds between 10 and 50 keV, it is not unreasonable to place the lower limit for practical measurements at 50 keV. Of course, some radiation-protection applications of neutron spectroscopy will require broader energy limits than those suggested by these results, and other input spectra, for example, 2.5-MeV neutrons from the $D(d,n)^3\text{He}$ reaction, could modify the conclusions.

Resolution. The results just presented were based on the dose-equivalent spectrum. We also established the resolution for neutron spectroscopy measurements based on this spectrum. We assumed that we needed the best data where the dose equivalent per neutron fluence changed the most rapidly as a function of energy. (A fluence measurement is adequate for regions where the conversion factor does not vary.) Therefore, we calculated the necessary energy intervals between 10 keV and 20 MeV to ensure that the dose conversion factors would always be within 10% of the published values.²⁰ The results, given in Table 12, show that a

resolution on the order of 20% was sufficient over most of the energy range.

Pulse-Shape Discrimination. Neutron spectroscopy detectors, e.g., proportional counters filled with hydrocarbon gas and organic scintillators, usually require discrimination circuits that reject unwanted gamma-ray pulses on the basis of the pulse shape. The pulse-shape discrimination (PSD) circuit we are presently using¹⁵ is a modern version of the zero-cross timing circuit described by Alexander and Goulding.²¹ It has a wide dynamic range and can handle high counting rates but is limited in its low-energy response. Thus, we have studied a number of PSD circuits with the purpose of developing one that will provide an optimum low-energy response for an organic scintillator.

An alternative to the zero-cross timing circuit is the charge comparison circuit described by Gatti and de Martini.²² This circuit compares the charge in the early part of the pulse with that in the late part by using a linear filter. It is also possible to

Table 12. Minimum number of energy intervals for dose-equivalent-per-fluence values to be within 10% of the published values.

Energy range, MeV	Dose equivalent per fluence	$200 \times \left(\frac{E_2 - E_1}{E_2 + E_1} \right)$
0.010 - 0.015	11.9	40.0
0.015 - 0.022	14.4	37.8
0.022 - 0.029	17.3	27.5
0.029 - 0.039	20.9	29.4
0.039 - 0.050	25.2	24.7
0.050 - 0.064	30.5	24.6
0.064 - 0.080	36.8	22.2
0.080 - 0.10	44.4	22.2
0.10 - 0.13	53.8	26.1
0.13 - 0.17	65.2	26.7
0.17 - 0.21	79.2	21.1
0.21 - 0.26	96.1	21.3
0.26 - 0.33	117	23.7
0.33 - 0.41	142	21.6
0.41 - 0.50	172	19.8
0.50 - 0.64	207	24.5
0.64 - 0.80	249	22.2
0.80 - 1.0	299	22.2
1.0 - 2.5	338	85.7
2.5 - 4.0	377	46.2
4.0 - 10.0	422	85.7
10.0 - 12.0	477	18.2
12.0 - 14.0	569	15.4
14.0 - 20.0	624	35.3

Table 11. The percentage of neutron spectra requiring measurements above the given upper energy limits.

Upper energy limit, MeV	Fission neutrons, %	14.5-MeV neutrons, %	Total, %
5	45.1	86.7	55.4
6	5.5	86.7	25.6
7	2.2	86.7	23.1
8	1.1	86.7	22.3
10	0	86.7	21.5

make the comparison by forming a ratio.²³ Sabbah and Suhaimi²⁴ have shown that a charge comparison circuit gives a better low-energy response than a zero-cross timing circuit.

Using the data of Kuchnir and Lynch,²⁵ we compared the relative variance of the differences in neutron and gamma-ray pulses produced by linear filters and ratio measurements. We obtained 4.0 for linear filters and 6.2 for ratio measurements. Based on these results, we have directed our efforts towards designing a circuit that is similar to Sabbah and Suhaimi's charge comparison circuit²⁴ but that more closely approximates Gatti and de Martini's optimum filter.²²

Light Output. The NE213 liquid organic scintillator (Nuclear Enterprises, Inc., San Carlos, Calif.) is widely used for fast-neutron spectroscopy. In this scintillator neutrons are measured via the light produced by recoil protons. Unfortunately, the relationship between the deposited proton energy and the light output is complicated. A number of people have measured this relationship and have obtained a wide variety of results.²⁶ However, since the data of Verbinski *et al.*²⁷ cover the greatest energy range, they are perhaps the most widely used.

We calibrate the pulse-height of the spectrometer using Compton recoil electrons produced by a gamma-ray source such as ²²Na; hence, it is useful to relate the proton energies to the electron energies that produce the same light output in the scintillator. The data of Verbinski *et al.* allow us to do this because of the assumption that the light output, as a function of electron energy, is linear with a zero intercept. Other data²⁸ show that the light output is a linear function of electron energy but that zero light output occurs at a nonzero electron energy.

We have studied this problem by measuring the response of an NE213 scintillator (2.54 cm in diameter by 2.54 cm in height) to gamma-rays from ²⁴¹Am, ⁵⁷Co, ²²Na, ¹³⁷Cs, and ⁵⁴Mn. The 60-keV gamma ray from ²⁴¹Am produces a total energy peak in the scintillator. For the other sources, the half-heights of the Compton electron distributions were found and fitted with a linear function of energy based on the maximum values for the Compton recoil electrons. A straight line fit the data to within 0.3% for all of the points, which illustrates the linearity of the light output as a function of electron energy; the intercept, however, was definitely

nonzero. Using the light units defined by Verbinski *et al.*, we found that electron energy (E_e) = 14.1 keV + 1171.6 keV/light unit. The data of Verbinski *et al.* are plotted in Fig. 10 as proton energy vs electron energy for the assumptions $E_e(0) = 0$ and $E_e(0) = 14.1$.

These data, and their first and second derivatives, play an important part in reducing the recorded pulse-height spectrum to a neutron spectrum.¹⁵ The response function used has its major effect on the reduced data for neutron energies of 1 MeV and lower. This problem is important enough to require further determinations of E_p vs E_e .

A Proportional Counter's Response to High-Energy Neutrons. As we have shown above, most neutron spectra require measurements at energies below the lower detection limit of an organic scintillator. A hydrogen-filled proportional counter can be used to measure neutrons at these low energies,¹⁶ but a problem occurs when there are neutrons present that produce recoil protons with ranges that exceed the dimensions of the counter: these recoil protons produce a pulse-height spectrum that must be subtracted before the data can be reduced to a neutron spectrum. In the past we used a parametric procedure¹⁵ to determine this pulse-height spectrum, but it was not completely satisfactory. As a result, we developed a Monte Carlo procedure similar to that described by Benjamin.¹⁶

In our program random numbers choose (1) where the n, p scattering event will occur in the counter and (2) the energy and direction of the recoil proton. A geometrical calculation determines

FIG. 10. Proton energies and electron energies producing the same light output (L) in the scintillator plotted for the data of Verbinski *et al.* $\odot$, ($L=0$)=0 keV; $\square$, ($L=0$)=14.1 keV.

the distance from the point of interaction to the nearest counter wall, in the direction the proton is going. The proton range vs energy, a relation obtained by integrating the stopping cross sections given by Andersen and Ziegler,²⁹ determines the energy deposited by the proton.

The program assumes that the counter has a cylindrical geometry, but it may have any dimensions and may be filled with any hydrogenous gas or gas mixture. Although the program can assume an isotropic irradiation of the counter, it normally assumes a plane beam, which may be incident on the counter at any angle.

If we ignore multiple scattering and keep the energy-loss calculations as simple as possible, the program will run on a small computer. Moreover, it will run rapidly enough to handle several thousand cases per half-hour, which is comparable to the time required to obtain satisfactory data with the proportional counter. Our tests indicate good agreement between measured and calculated responses to monoenergetic neutrons both within and above the energy limit of the counter. We have also determined that, with smoothing, relatively few (10^4) cases provide a background of sufficient quality for our data reduction techniques. Using a small computer may make it possible to apply these techniques to a portable spectrometer.

TECHNICAL NOTES

RADIATION PROTECTION

Intermediate Energy X-Ray Spectra for General Shielding Calculations

Recent efforts to perform skyshine calculations for standard x-ray units have resulted in the development of a set of intermediate energy (0.5–10 MeV) x-ray spectra* (Figs. 11–16) from a number of literature sources.^{30–40} These are summarized in a form convenient for input to the LLL version of the MORSE code transport code

(Tables 13–18). Each spectrum is generally a numerical average of the spectra found in the references noted. A thick tungsten target was assumed, along with some beam filtration. The electron potential is included for each table and figure. The spectra are, in each case, normalized to 1.0.

FIG. 11. The 0.5-MeV x-ray spectrum.

*These include both experimental and calculated spectra.

FIG. 12. The 1.0-MeV x-ray spectrum.

In the absence of specific spectra, these should provide guidance for initial radiation shielding and scatter estimates in the middle energy range.

FIG. 13. The 2.0-MeV x-ray spectrum.

FIG. 14. The 4.0-MeV x-ray spectrum.

FIG. 15. The 6.0-MeV x-ray spectrum.

FIG. 16. The 10.0-MeV x-ray spectrum.

Table 13. The 0.5-MeV maximum potential x-ray spectrum.³⁰⁻³²

MORSE 76L group number	Average energy, MeV	Number of photons/group	Number of photons/MeV
1-24	-	0	0
25	0.464	3.23×10^{-2}	1.54×10^{-2}
26	0.411	5.54×10^{-2}	2.99×10^{-2}
27	0.362	7.81×10^{-2}	4.77×10^{-2}
28	0.320	9.72×10^{-2}	6.74×10^{-2}
29	0.282	1.09×10^{-1}	8.52×10^{-2}
30	0.249	1.21×10^{-1}	1.08×10^{-1}
31	0.220	1.22×10^{-1}	1.23×10^{-1}
32	0.194	1.16×10^{-1}	1.32×10^{-1}
33	0.171	1.06×10^{-1}	1.38×10^{-1}
34	0.151	9.32×10^{-2}	1.37×10^{-1}
35	0.133	7.09×10^{-2}	1.18×10^{-1}

Table 14. The 1.0-MeV maximum potential x-ray spectrum.³²⁻³⁴

MORSE 76L group number	Average energy, MeV	Number of photons/group	Number of photons/MeV
1-18	-	0	0
19	0.986	9.03×10^{-3}	2.64×10^{-3}
20	0.870	2.07×10^{-2}	6.86×10^{-3}
21	0.768	3.16×10^{-2}	1.18×10^{-2}
22	0.678	4.43×10^{-2}	1.89×10^{-2}
23	0.598	5.26×10^{-2}	2.54×10^{-2}
24	0.528	6.21×10^{-2}	3.39×10^{-2}
25	0.465	6.93×10^{-2}	4.28×10^{-2}
26	0.411	7.65×10^{-2}	5.35×10^{-2}
27	0.362	8.18×10^{-2}	6.49×10^{-2}
28	0.320	8.56×10^{-2}	7.73×10^{-2}
29	0.282	8.84×10^{-2}	9.00×10^{-2}
30	0.249	8.73×10^{-2}	1.01×10^{-1}
31	0.220	8.09×10^{-2}	1.06×10^{-1}
32	0.194	7.20×10^{-2}	1.07×10^{-1}
33	0.171	5.99×10^{-2}	1.01×10^{-1}
34	0.151	4.66×10^{-2}	8.87×10^{-2}
35	0.133	3.13×10^{-2}	6.75×10^{-2}

Table 15. The 2.0-MeV maximum potential x-ray spectrum.³²⁻³⁵

MORSE 76L group number	Average energy, MeV	Number of photons/group	Number of photons/MeV
1-13	-	0	0
14	1.84	1.40×10^{-2}	3.06×10^{-3}
15	1.63	2.05×10^{-2}	5.12×10^{-3}
16	1.44	2.71×10^{-2}	7.64×10^{-3}
17	1.27	3.42×10^{-2}	1.09×10^{-2}
18	1.12	3.93×10^{-2}	1.42×10^{-2}
19	0.986	4.54×10^{-2}	1.86×10^{-2}
20	0.870	4.86×10^{-2}	2.26×10^{-2}
21	0.768	5.29×10^{-2}	2.78×10^{-2}
22	0.678	5.79×10^{-2}	3.46×10^{-2}
23	0.598	6.38×10^{-2}	4.31×10^{-2}
24	0.528	6.68×10^{-2}	5.12×10^{-2}
25	0.465	7.03×10^{-2}	6.10×10^{-2}
26	0.411	7.05×10^{-2}	6.94×10^{-2}
27	0.362	7.15×10^{-2}	7.97×10^{-2}
28	0.320	6.76×10^{-2}	8.56×10^{-2}
29	0.282	6.22×10^{-2}	8.91×10^{-2}
30	0.249	5.61×10^{-2}	9.12×10^{-2}
31	0.220	4.82×10^{-2}	8.88×10^{-2}
32	0.194	3.63×10^{-2}	7.59×10^{-2}
33	0.171	2.33×10^{-2}	5.51×10^{-2}
34	0.151	1.52×10^{-2}	4.05×10^{-2}
35	0.133	8.30×10^{-3}	2.51×10^{-2}

Table 16. The 4.0-MeV maximum potential x-ray spectrum.³⁴⁻³⁶

MORSE 76L group number	Average energy, MeV	Number of photons/group	Number of photons/MeV
1-7	-	0	0
8	3.96	9.92×10^{-4}	2.0×10^{-4}
9	3.49	1.29×10^{-3}	2.0×10^{-3}
10	3.04	1.82×10^{-2}	3.87×10^{-3}
11	2.69	2.84×10^{-2}	6.80×10^{-3}
12	2.37	3.48×10^{-2}	9.45×10^{-3}
13	2.09	4.02×10^{-2}	1.24×10^{-2}
14	1.84	4.28×10^{-2}	1.50×10^{-2}
15	1.63	4.58×10^{-2}	1.81×10^{-2}
16	1.44	4.88×10^{-2}	2.18×10^{-2}
17	1.27	5.51×10^{-2}	2.79×10^{-2}
18	1.12	5.95×10^{-2}	3.42×10^{-2}
19	0.986	6.26×10^{-2}	4.07×10^{-2}
20	0.870	6.18×10^{-2}	4.56×10^{-2}
21	0.768	5.95×10^{-2}	4.98×10^{-2}
22	0.678	5.83×10^{-2}	5.54×10^{-2}
23	0.598	5.71×10^{-2}	6.14×10^{-2}
24	0.528	5.30×10^{-2}	6.46×10^{-2}
25	0.465	4.99×10^{-2}	6.89×10^{-2}
26	0.411	4.71×10^{-2}	7.38×10^{-2}
27	0.362	4.19×10^{-2}	7.45×10^{-2}
28	0.320	3.66×10^{-2}	7.38×10^{-2}
29	0.282	3.00×10^{-2}	6.84×10^{-2}
30	0.249	2.36×10^{-2}	6.09×10^{-2}
31	0.220	1.18×10^{-2}	3.47×10^{-2}
32	0.194	7.32×10^{-3}	2.41×10^{-2}
33	0.171	5.21×10^{-3}	1.96×10^{-2}
34	0.151	3.84×10^{-3}	1.64×10^{-2}
35	0.133	2.98×10^{-3}	1.41×10^{-2}

Table 17. The 6.0-MeV maximum potential x-ray spectrum.^{31,37,38}

MORSE 76L group number	Average energy, MeV	Number of photons/group	Number of photons/MeV
1-4	-	0	0
5	5.69	1.13×10^{-3}	1.42×10^{-4}
6	5.02	7.93×10^{-3}	1.12×10^{-3}
7	4.43	1.77×10^{-2}	2.84×10^{-3}
8	3.96	1.77×10^{-2}	3.94×10^{-3}
9	3.49	3.10×10^{-2}	5.29×10^{-3}
10	3.04	3.02×10^{-2}	7.06×10^{-3}
11	2.69	3.40×10^{-2}	9.03×10^{-3}
12	2.37	3.68×10^{-2}	1.10×10^{-2}
13	2.09	3.92×10^{-2}	1.33×10^{-2}
14	1.84	4.02×10^{-2}	1.55×10^{-2}
15	1.63	4.26×10^{-2}	1.86×10^{-2}
16	1.44	4.32×10^{-2}	2.14×10^{-2}
17	1.27	4.64×10^{-2}	2.59×10^{-2}
18	1.12	5.06×10^{-2}	3.22×10^{-2}
19	0.986	5.16×10^{-2}	3.71×10^{-2}
20	0.870	5.35×10^{-2}	4.38×10^{-2}
21	0.768	6.01×10^{-2}	5.56×10^{-2}
22	0.678	5.87×10^{-2}	6.17×10^{-2}
23	0.598	5.65×10^{-2}	6.72×10^{-2}
24	0.528	5.63×10^{-2}	7.59×10^{-2}
25	0.465	4.84×10^{-2}	7.40×10^{-2}
26	0.411	4.15×10^{-2}	7.19×10^{-2}
27	0.362	3.31×10^{-2}	6.5×10^{-2}
28	0.320	2.65×10^{-2}	5.9×10^{-2}
29	0.282	2.06×10^{-2}	5.2×10^{-2}
30	0.249	1.67×10^{-2}	4.81×10^{-2}
31	0.220	1.36×10^{-2}	4.4×10^{-2}
32	0.194	1.02×10^{-2}	3.73×10^{-2}
33	0.171	6.69×10^{-3}	2.78×10^{-2}
34	0.151	4.54×10^{-3}	2.14×10^{-2}
35	0.133	2.83×10^{-3}	1.51×10^{-2}

Table 18. The 10.0-MeV maximum potential x-ray spectrum.^{31,34,39,40}

MORSE 76L group number	Average energy, MeV	Number of photons/group	Number of photons/MeV
1	9.39	6.46×10^{-3}	5.64×10^{-4}
2	8.29	1.46×10^{-2}	1.44×10^{-3}
3	7.31	1.91×10^{-2}	2.14×10^{-3}
4	6.45	2.31×10^{-2}	2.93×10^{-3}
5	5.69	2.60×10^{-2}	3.74×10^{-3}
6	5.02	2.75×10^{-2}	4.49×10^{-3}
7	4.43	2.95×10^{-2}	5.47×10^{-3}
8	3.96	2.45×10^{-2}	6.30×10^{-3}
9	3.49	3.93×10^{-2}	7.76×10^{-3}
10	3.04	3.42×10^{-2}	9.24×10^{-3}
11	2.69	3.61×10^{-2}	1.10×10^{-2}
12	2.37	3.68×10^{-2}	1.27×10^{-2}
13	2.09	3.76×10^{-2}	1.47×10^{-2}
14	1.84	3.84×10^{-2}	1.71×10^{-2}
15	1.63	3.91×10^{-2}	1.97×10^{-2}
16	1.44	3.99×10^{-2}	2.28×10^{-2}
17	1.27	4.34×10^{-2}	2.81×10^{-2}
18	1.12	4.44×10^{-2}	3.26×10^{-2}
19	0.986	4.56×10^{-2}	3.78×10^{-2}
20	0.870	4.40×10^{-2}	4.15×10^{-2}
21	0.768	4.06×10^{-2}	4.33×10^{-2}
22	0.678	4.06×10^{-2}	4.91×10^{-2}
23	0.598	3.84×10^{-2}	5.26×10^{-2}
24	0.528	3.56×10^{-2}	5.54×10^{-2}
25	0.465	3.32×10^{-2}	5.85×10^{-2}
26	0.411	3.16×10^{-2}	6.31×10^{-2}
27	0.362	2.93×10^{-2}	6.63×10^{-2}
28	0.320	2.68×10^{-2}	6.88×10^{-2}
29	0.282	2.33×10^{-2}	6.8×10^{-2}
30	0.249	1.94×10^{-2}	6.4×10^{-2}
31	0.220	1.70×10^{-2}	6.3×10^{-2}
32	0.194	1.00×10^{-2}	4.2×10^{-2}
33	0.171	2.94×10^{-3}	1.4×10^{-2}
34	0.151	1.30×10^{-3}	7.1×10^{-3}
35	0.133	3.44×10^{-4}	2.1×10^{-3}

PUBLICATIONS

J. H. Elliott, *Tour of the Standards and Calibrations Laboratory*, Lawrence Livermore Laboratory, Livermore, Calif., UCRL-52537 (1978).

This tour of the Standards and Calibrations Laboratory at LLL is a guide to the capabilities of and services offered by this unique laboratory. We explain how to provide radiation fields and measurements for dosimeters and describe survey instruments, spectrometers, sources, and available equipment and facilities. The tour also includes a survey of some health physics and interdepartmental programs supported by the Standards and Calibrations Laboratory and a list of applicable publications.

E. Farley, J. Becker, K. Crase, R. Howe, and D. Selway, *Measurement of Energy Deposited by Charged Particle Beams in Composite Targets*, Lawrence Livermore Laboratory, Livermore, Calif., UCID-79-5 (1979).

No abstract available.

D. A. Fuess, *A Complete System for Portable Gamma Spectroscopy*, Lawrence Livermore Laboratory, Livermore, Calif., UCRL-52611 (1978).

This paper describes a system built around the Computing Gamma Spectrometer (PSA) LEA 74-008. The software primarily supports high-resolution gamma-ray spectroscopy using either a high-purity intrinsic germanium detector (HPGe) or a lithium-drifted germanium detector [Ge(Li)].

D. A. Fuess, D. R. Slaughter, R. E. Strout, and D. W. Rueppel, *Determination of Continuous Gamma-Ray Spectra Over the Energy Range 0.1 to 8 MeV*, Lawrence Livermore Laboratory, Livermore, Calif., UCRL-52513 (1978).

We have established an experimental facility at LLL to characterize radiation fields produced by a variety of sources and transmitted through various shielding materials. We discuss specific techniques for acquiring and reducing continuous gamma-ray energy spectra, including NaI(Tl) detectors. The use of several detector sizes allowed us to study a wide variety of source intensities and to calculate response matrices for a number of collimated detector configurations. We describe a computer

program that performs data reduction by an iterative unfolding process. Our reduction technique discloses the continuous gamma-ray energy spectrum over the range 0.1 to 8 MeV as opposed to the traditional peak-intensity analysis.

R. B. Gammage, T. Vo-Dinh, A. R. Hawthorne, J. H. Thorngate, and W. W. Parkinson, "New Techniques for Measuring Polynuclear Aromatics in the Workplace," in *Advances in Chemistry*, No. 170 (American Chemical Society, Washington, D.C., 1978).

A gap exists between the crude techniques available for measuring polynuclear aromatic compounds in the workplace and the sophisticated analytical tools used in the laboratory for a much fuller characterization of pollutants from synthetic fuel operations such as tar sand and oil shale processing. Real-time or near real-time instruments suitable for use by industrial hygienists are urgently needed to measure fugitive emissions. We describe several new instruments and instrumental techniques that could satisfy some of these needs. They include second-derivative ultraviolet absorption, synchronous luminescence and room-temperature phosphorescence spectrometries, a portable mass spectrometer, differential sublimation, and thermoluminescence. We evaluated the practicality of these approaches and found that they are suitable for monitoring (1) naphthalene and its alkyl derivatives at parts-per-billion concentrations either in the vapor or the solution phase and (2) trace amounts of phenolic compounds in by-product water. They are also suitable for the rapid analysis of samples filtered or spotted on paper adsorbents.

R. B. Gammage, J. H. Thorngate, W. W. Parkinson, A. R. Hawthorne, and T. Vo-Dinh, "On the Desirability of the Health Physics Society Assuming Responsibilities in the Nonnuclear and Nonradiation Fields," *Health Phys.* 35, 711 (1978).

No abstract available.

C. A. Harder and B. J. Held, "Operation of Self-Contained Breathing Apparatus Exposed to Low Air Temperature," *The International Fire Chief* 45(1), 6-10 (1979).

To determine the effect of cold and thermal shock on self-contained breathing apparatus, we have conducted subjective and instrumented low air temperature studies and instrumented thermal shock tests from low to high air temperatures. The results will help Lawrence Livermore Laboratory firefighters and emergency teams select apparatus for use in fires and emergencies at off-site energy projects located in cold climates. In the subjective tests, users evaluated the performance of apparatus that had been cold soaked for 24 h at -25°F . For the instrumented tests, we monitored air pressures in pressure-demand facepieces exposed to temperatures of -25 and -40°F . We also monitored air pressures during a 1-h period in which the air temperature rose from -25 to 200°F . Some models showed subatmospheric inhalation pressures at sub-zero temperatures, others developed problems, and the flow-air pressure alarms on all models failed to sound at -40°F . Thermal shocking of the cold-soaked apparatus did not appear to affect the operating characteristics of any model at either low or high air temperatures.

C. A. Harder and B. J. Held, "Future Research Needs for Fire Fighters' Breathing Apparatus," *The International Fire Chief* 45(3), 26-30 (1979).

In this article we discuss current and planned research to determine how factors such as radiant heat, moisture, ultra-high air temperatures, fire-decomposition products and chemicals, extinguishing agents, and mechanical stress may affect the operation and use of fire fighters' breathing apparatus. We also discuss the need for improved communications between apparatus wearers.

C. A. Harder and B. J. Held, "New Standards, Regulations, and Proposals for Self-Contained Breathing Apparatus," *The International Fire Chief* 45(4), 23-27 (1979).

In this article, we look at the changing state and federal standards for self-contained breathing apparatus and their effect on the fire service. We also discuss the current activities of various government and standard-setting organizations and the changes that may result from these activities.

A. R. Hawthorne and J. H. Thorngate, "Improving Analysis from Second-Derivative Ultraviolet-Absorption Spectrometry," *Applied Optics* 17, 724 (1978).

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A. R. Hawthorne and J. H. Thorngate, "Applications of Second-Derivative Ultraviolet-Absorption Spectrometry to Polynuclear Aromatic Analysis," *Applied Spectrometry* (1978).

The increased awareness of the deleterious effects of polynuclear aromatic compounds emphasizes the need for instruments that are capable of monitoring these compounds and suitable for use in health protection programs. Second-derivative ultraviolet-absorption spectrometry with least-squares spectral analysis is a promising method for analyzing vapor phase polynuclear aromatics and solutions containing these compounds. Detection limits of a few parts per billion are attainable for many polynuclear aromatic compounds. The examples we give show the analytical capabilities of this method.

A. R. Hawthorne, J. H. Thorngate, R. B. Gammage, and T. Vo-Dinh, "Development of a Prototype Instrument for Field Monitoring of Polynuclear Aromatic Vapors" (in *Proc. Third Intern. Symp. Polynuclear Aromatic Hydrocarbons, Columbus, Ohio, 25-27 October 1978*).

Coal conversion facilities are a new and potentially large source of man's exposure to polynuclear aromatics. But with the ever dwindling sources of petroleum and the increased dependence on foreign oil, the U.S. Department of Energy is committed to developing coal resources, especially for the production of synthetic fuels. An important part of this commitment is to ensure that there are minimal adverse effects on human health and the environment as a result of new energy technologies. This includes efforts to ensure that adequate industrial hygiene measures are provided to protect workers from exposure to polynuclear aromatics. This paper describes the development of a portable field monitor that can provide real-time measurement of volatile polynuclear aromatics in various work areas.

G. F. Hunt, *A Microprocessor Controlled, Personnel Thermoluminescence Dosimetry Reader*, Lawrence Livermore Laboratory, Livermore, Calif. UCRL-81153 (prepared for submission to the IEEE Nuclear Science Symposium, Washington, D.C., October 18-20, 1978).

Using a DEC LSI-11 microprocessor, we have developed a thermoluminescence dosimetry reader for personnel dosimetry. This microprocessor

provides system control, accumulates data for temporary storage on a cassette, and later outputs the data to a large computer. The microprocessor also provides a human/system interface through a computer terminal. A programmable high-voltage supply and a light-emitting diode are used in the reader system to automatically control photomultiplier tube gain.

J. S. Johnson, *Lawrence Livermore Laboratory's Beryllium Control Program for High-Explosive Test Firing Bunkers and Tables*, Lawrence Livermore Laboratory, Livermore, Calif. UCID-18006 (1978).

This detailed report on Lawrence Livermore Laboratory's control program to minimize beryllium levels in Laboratory workplaces includes an outline of beryllium surface, soil, and air levels and an 11-year summary of sampling results from two high-use, high-explosive test firing bunkers. These sampling data and other studies demonstrate that the beryllium control program is functioning effectively.

J. S. Johnson, "The Acute Toxicology of TATB," *Proc. Symp. Chemistry of 2,2',4,4',6,6'-Hexanitrostilbene (HNS) and 1,3,5-Triamino, 2,4,6-Triaminobenzene (TATB)*, Waltham Abbey, Essex, England, 28 March 1979 (Propellants, Explosives and Rocket Motor Establishment, Essex, England, 1979).

The acute toxicology of TATB and two of its synthetic precursors, 1,3,5-trichlorobenzene and 1,3,5-trichloro-2,4,6-trinitrobenzene, is presented. We describe each acute toxicology test and give typical examples of the industrial hygiene controls used for handling these materials.

J. S. Johnson, *Safe Handling of Chemical Carcinogens in the Research Laboratory*, Lawrence Livermore Laboratory, Livermore, Calif., UCRL-82387 (prepared for presentation at the 177th meeting of the American Chemical Society, held jointly with the Japanese Chemical Society, Honolulu, Hawaii, April 2-6, 1979).

Toxic and corrosive chemicals have been a part of America's development for several decades. Only in the last 30 years, however, has any national regulatory attention been given to the safe handling of such chemicals in the workplace. The use of toxic and corrosive materials in the workplace was formally regulated by the Occupational Safety and

Health Act of 1970; in 1973, the Occupational Health and Safety Administration issued temporary emergency standards for 14 chemical carcinogens. Now this administration is attempting to promulgate a generic carcinogen control standard. Lawrence Livermore Laboratory has established a chemical carcinogen control program on the basis of applicable standards promulgated by the Occupational Safety and Health Administration and has supplemented the program with internal controls for the safe handling of chemical carcinogens in the research laboratory. That control program is outlined here.

G. O. Nelson, *Calibration Techniques*, Lawrence Livermore Laboratory, Livermore, Calif., UCRL-82468 (1979).

This report compares the simple static and the more complex dynamic methods of gas standard generation. A completely automated flow-temperature-humidity-concentration controller is also described.

G. O. Nelson and R. D. Taylor, *Flow-Temperature-Humidity Control System Operating Manual*, Lawrence Livermore Laboratory, Livermore, Calif., UCID-17961 (1978).

This manual contains operating, maintenance, and troubleshooting procedures for the flow-temperature-humidity control system used at the Lawrence Livermore Laboratory to prepare test atmospheres for industrial hygiene and air pollution studies. The system consists of two basic components: a commercially available temperature/humidity indicator unit and a specially built flow-temperature-humidity control module. Procedures are given for using the control system with a vapor generation system or with a trace-gas flowmeter to add vapor or a trace gas to the airstream after it leaves the control module.

D. R. Slaughter, D. W. Rueppel, and D. A. Fuess, *Neutron Spectrum Measurements for Radiation Protection Purposes*, Lawrence Livermore Laboratory, Livermore, Calif., UCRL-52415 (1978).

We have measured the energy spectra of low-intensity neutron sources used for calibrating personnel neutron dose-rate meters and dosimeters and for characterizing the neutron fields to which personnel are exposed. This report describes several detector-analyzer systems that measure in the

energy range 50 keV to 20 MeV at intensities from 10^{-1} to 10^5 n/cm²-s. The systems include NE213 and Stilbene organic scintillators as well as H₂, ³He, and CH₄ proportional counters. Also described are pulse-height analysis and pulse-shape discrimination systems. An unfolding code, NUTSPEC, reduces the pulse-height data to an absolute differential neutron flux $\phi(E)$ for the above detectors. The code uses a derivative unfolding method for the scintillation detectors, and for the proportional counters it calculates a response matrix and uses an iterative unfolding method to determine $\phi(E)$. The unfolded flux distribution, combined with published conversion factors, produces differential neutron dose-equivalent and kerma rates. Spectral segments obtained with different detectors from several measurements merge into a single differential flux spectrum over the range 50 keV to 20 MeV, together with the corresponding differential kerma and neutron dose-equivalent distributions. Also reported are spectrum measurements near ²⁵²Cf and ²³⁸PuBe sources with various moderators.

J. H. Thorngate, *Practical Considerations for the Application of Neutron Spectroscopy to Radiation Protection*, Lawrence Livermore Laboratory, Livermore, Calif., UCRL-81738 (prepared for submission to *Health Physics*, 1978).

Dosimetric considerations control the use of neutron spectroscopy in radiation protection. Detailed information about the energy distribution of the neutron field is required only in regions where

the fluence-to-dose-equivalent conversion factor is a rapidly changing function of energy. Thus, although fluence measurements must be made, spectra are not required for energies below 10 keV and probably are also unnecessary in the energy region from 20 MeV to several hundred MeV. A lower limit of 50 keV and an upper limit of 6 MeV are reasonable for many practical cases. Between 10 keV and 20 MeV, an energy resolution of 20% is sufficient to stay within 10% of the conversion factor. Proportional counters filled with gas and liquid organic scintillators provide adequate sensitivity and resolution. Normally, more than one detector will be required for adequate measurements. The system should also include an on-line computer to simplify the operation of the instrument and to reduce the data rapidly.

T. Vo-Dinh, R. B. Gammage, A. R. Hawthorne, and J. H. Thorngate, "Synchronous Spectroscopy for the Analysis of Polynuclear Aromatic Compounds," *Environ. Sci. and Tech.* **12**, 1297 (1978).
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C. M. Wong, J. L. Cate, and W. L. Pickles, *Preparation of Uranium Standard Solutions for X-Ray Fluorescence Analysis*, Lawrence Livermore Laboratory, Livermore, Calif., UCRL-80778 (prepared for presentation at the American Nuclear Society Topical Conference, Williamsburg, Virginia, May 15-17, 1978).

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