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concentration detection device for calibration
for and monitoring of materials in use at
various work areas of Naval Air Rework
Facility at North Island, California

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Monterey, California. Naval Postgraduate School

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USE OF PORTABLE INFRARED AIRBORNE VAPOR
CONCENTRATION DETECTION DEVICE FOR CALIBRATION
FOR AND MONITORING OF MATERIALS IN USE AT VARIOUS
WORK AREAS OF NAVAL AIR REWORK FACILITY AT
NORTH ISLAND, CALIFORNIA

J. H. Duffin

December 1978

Approved for public release: distribution unlimited

Prepared for: AIRCRAFT ENVIRONMENTAL SUPPORT OFFICE (Code 64270)
NAVAL AIR REWORK FACILITY, NORTH ISLAND, CALIFORNIA

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER NPS-62Dn78-010	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Use of Portable Infrared Airborne Vapor Concentration Detection Device for Calibration for and Monitoring of Materials in use at Various Work Areas of Naval Air Rework Facility at North Island, California		5. TYPE OF REPORT & PERIOD COVERED Final Report-AY78
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) J. H. Duffin Naval Postgraduate School Monterey, California		8. CONTRACT OR GRANT NUMBER(s) N6852079WR20097-Fund Authoriz 1791804.6289 Approp. Also: N 6588878WR00142; 1781804.6289
9. PERFORMING ORGANIZATION NAME AND ADDRESS Aircraft Environmental Support Office Code 64270, San Diego, CA 92135 (Naval Air Rework Facility, North Island)		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS Memo NC4(0021)rg 2 Nov 78 and WR NAVCOMPT 140 attached.
11. CONTROLLING OFFICE NAME AND ADDRESS Naval Aviation Logistics Center Patuxent River, MD 20670 F. B. Garris, Comptroller (Mr. A. Jones)		12. REPORT DATE December 1978
		13. NUMBER OF PAGES 45
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution unlimited		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)		
Infrared	Chemicals	Chemical Cleaning
Toxic Vapors	Calibration	Modeling
Airborne Vapor Concentrations	Industrial Atmospheres	Computer
Solvents	Paint-Apply/Strip	Production
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)		
<p>This report covers work done at the Naval Postgraduate School in April, May, June 1978 and at the Naval Air Rework Facility, North Island in October-December 1978.</p> <p>A Foxboro/Wilks Portable Infrared Gas Analyzer (MIRAN-IA) was calibrated for about 50 materials (either pure or in mixtures) in use in general plant operations at a naval air rework facility. All successful calibrations are reported here.</p> <p>The MIRAN-IA was used to monitor airborne vapor concentrations in the Paint</p>		

Block 20

Shop, Building 472, NAVAIREWORKFAC North Island. Its potential use in the Chemical Cleaning Shop, Building 472 and the Airplane Paint/Strip Hangars, Buildings 464-468 was considered but not done due to lack of time.

Building 472 Paint Shop results indicate that, in general, concentrations of various chemical vapors are below Occupational Safety and Health Act limits for human exposure. The Paint Shop operation is judged to be a careful and safe operation. The MIRAN-1A was updated by installing silver bromide cell windows and an improved detector/preamplifier assembly.

ABSTRACT

This report covers work done at the Naval Postgraduate School (NAVPGSCOL) in April, May, June 1978 and at the Naval Air Rework Facility (NAVAIREWORKFAC) North Island, in October-December 1978.

A Foxboro/Wilks Portable Infrared Gas Analyzer (MIRAN-IA) was calibrated for about 50 materials (either pure or in mixtures) in use in general plant operations at a naval air rework facility. All successful calibrations are reported.

The MIRAN-IA was used to monitor airborne vapor concentrations in the Paint Shop, Building 472, NAVAIREWORKFAC North Island. Its potential use in the Chemical Cleaning Shop, Building 472 and the Airplane Paint/Strip Hangars, Buildings 464-468 was considered but not done due to lack of time.

Building 472 Paint Shop results indicate that, in general, concentrations of various chemical vapors are below Occupational Safety and Health Act (OSHA) limits for human exposure. The Paint Shop operation is judged to be a careful and safe operation. It was not possible to "model" the Paint Shop operation because of the difficulty of defining a production rate.

The MIRAN-IA was updated by installing silver bromide cell windows, and an improved detector/preamplifier assembly.

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I. INTRODUCTION

This report covers work done as a continuing effort along the lines reported on in reference 1. The work was done at both the NAVPGSCOL Monterey, California and at the NAVAIREWORKFAC North Island, California. The Monterey work covers the time period April-May-June, 1978, and the North Island work covers the time period October-November-December, 1978.

A broad range of hazardous (or toxic) gases was investigated. The materials to be investigated were decided upon by scanning the materials listed in reference 2, and by personal consultation with various members of the Materials Laboratory at North Island. Mr. K. Hillman, chemist, has been particularly helpful in providing information, materials, and supplies.

A MIRAN-IA was used for all work. A description of this instrument and its use in calibrating materials is given in reference 1. There has been a deterioration of the sodium chloride cell windows with time due to moisture in air and calibration materials. The windows were replaced with silver bromide windows which pass a wider band of infrared radiant energy and are less susceptible to moisture deterioration. Other updating work on the instrument is also covered in this report.

Some 43 pure materials were calibrated in either a rough or detailed manner. Two mixtures of materials were also studied as it is quite common in plant work to use mixtures.

Certain materials gave signals which although weak and/or noisy, were definitely detectable. Such signals cannot be used for calibration. However, they can indicate, in a general way, whether or not the concentration of the material complies with OSHA limits.

For those materials that were calibratable, calibration data were plotted and then fitted with second order polynomials using least squares regression analysis. The method of doing this is described in reference 1.

The report also presents the results of taking the MIRAN-IA into certain plant areas to monitor the ambient air for its content of specified materials for which it had been calibrated. Attempts to correlate concentration levels with productivity levels are reported.

II. MATERIALS CALIBRATED

A. General

A very broad range of materials is used in day-to-day operations of a NAVAIREWORKFAC. Many of these materials are potentially or actually hazardous because of their toxic nature. The effects of exposure of humans to these materials are of increasing concern to the medical community and to Navy managers. Instantaneous and cumulative, or long-range, exposure effects should be constantly monitored and studied. Knowledge gained through study and research will provide proper handling procedures for these materials, as well as exposure limits for both airborne and skin contact. The formal setting of these limits is the province of such organizations as OSHA, Toxic Substances Control Act (TOSCA), and National Institute for Occupational Safety and Health (NIOSH), as well as many others. It is the Navy's immediate responsibility to be aware of all hazardous materials in use, their exposure limits, and their proper handling.

B. Monitoring for Hazardous Materials

In an effort to help provide information concerning airborne concentration levels of many materials, the Aircraft Environmental Support Office (AESO) acquired a MIRAN-IA. A thorough description of the instrument and its use is reported on in reference 1.

The MIRAN-IA can be taken into the plant to continuously monitor the air that a human breathes. The intake to the instrument can literally be placed alongside the nose of an individual. In this manner, rapid, accurate determination of airborne concentration levels can be made and recorded for short or long periods. The use of the instrument requires that it be calibrated for a given material to be studied.

Calibration for a given material is in the form of absorbance versus concentration in parts-per-million (ppm). Absorbance is output as a voltage (0-1 volt) and is the variable recorded. In an actual plant run, recorder output is labeled in terms of ppm using known calibration curves. In this manner, time histories of concentration levels in various work areas can be obtained.

C. List of Materials to be Calibrated

An initial list of materials that were candidates for calibration was compiled using reference 2. This initial list is shown in Table 1. Of the materials appearing in this list, five had been calibrated and reported upon in reference 1. They were acetone, methyl ethyl ketone (MEK), ethanol, toluene, and 1,1,1-trichloroethane (methyl chloroform).

Location (Bldg. or Shop No.)	Description / Action	Chemicals Used
472(B)	degreaser	1,1,1 trichlorethane(methyl chloroform)
97322(S)	chemical cleaning	stoddard solvent; epoxy and acrylic paint removers(strippers
472(B)	oil spray booth	preservative oil
472(B)	sonic cleaner	Turco, Caviclean No. 2
472(B)	still	Freons
97322(S)	electroplating - MEK spray booth	methyl ethyl ketone(MEK)
97333(S)	painting - apply/strip	epoxy coating; phenolic resins; acrylic lacquers; enamels; polyurethane coatings; lacquer/acrylic/polyurethane thinners acetone; MEK
96242(S)	machine shop - spray booth	ethyl alcohol
379(B)	fluro-inspection spray booth	WP-167 penetrant
379(B)	chemical cleaning	Turco Carb; methylene chloride
65(B)	old bearing cleaning system	PC-111 solution; fingerprint cleaner
65(B)	magnaflux inspection system	kerosene
65(B)	vapor degreaser	Freon-113
341(B)	graphic arts - bake oven	methyl isobutyl ketone; toulene; various thinners
341(B)	paint spray booth	rubber solvent; naphtha
341(B)	etch booth	xylene
378(B); 94144(S)	solvents spray booths	isopropyl alcohol; methylene chloride; tetrachloroethane; perchloroethylene(tetrachloroethylene)
378(B)	bake oven	dry lube; varnish
463(B)	avionics - plating unit	tin/lead fluoroborates; formaldehyde; nickel; sulfamate

NOTE: This is a selection from reference 1 to give some idea of variety of materials used.

For hydrogen cyanide (HCN), calibration had been attempted but was unsuccessful. Further study of the results of the previous work plus information obtained from Foxboro Analytical indicates that the reason for the unsuccessful calibration attempt was faulty technique on the part of the instrument operator. The MIRAN-IA subsequently was calibrated for HCN.

Of the remaining materials on the list, several are mixtures of chemicals. Such things as thinners, lacquers, coatings, cleaners, etc. are complex mixtures of liquids, or solids and liquids. The liquid portions of these materials contain many hazardous materials, and an attempt was made to indentify them. This was done by obtaining, from the Materials Lab, a list of materials used in plant operations. This list is shown in Table 2. Note that annual gallon usage in 1976 is shown, as well as Military Specification Number (MIL-SPEC) and Naval Specification Number (NSN). The MIL-SPEC number proved quite useful in identifying materials and their content, while physical properties were obtained for the most part from reference 4 and reference 10.

A microfilm library of MIL-SPEC's is located in the Materials Lab, and it was used to look up certain materials appearing in Table 2. Contents of various mixtures were obtained from this library and are shown in Table 3.

Based on all information available, a "Master List" of materials to be calibrated or investigated was prepared. This list is shown in Table 4. The list is not exhaustive, but does cover the majority of materials in general use in plant production areas. It is to be noted that a certain few chemicals represent by far the largest mass usage. Even small concentrations of these and other materials could prove hazardous in airborne or skin contact and, as a result, some of the smaller mass usage materials were also investigated and calibrated. Not every material listed is a "pure" material but rather a mixture of other materials appearing on the list. Some materials are to be considered for later calibration. They appear as ingredients or are used "as is" in plant work and are not shown on any charts as having a concentration limit or Threshold Limit Value (TLV).

TABLE 2 - TYPICAL MATERIALS USE LIST, NAVAIREWORKFAC NORTH ISLAND

Item	MIL-SPEC	Appears on Wilks Chart	NSN	Properties Needed		Annual Gallon Usage FY-76
				Molecular Weight	Liquid Density	
cleaning solvent(stoddard)	Fed-PD-680 Type 1	Yes	6850-00-264-9039	100	0.7	279,940
calibrating fluid	Mil-F-7024 Type 2	No	6850-00-656-0810	100.2	0.710	15,994
1,1,1,-trichloroethane	Mil-T-81533	Yes	None	133.42	1.3249	84,242
methyl ethyl ketone(2-butanone)	Fed-TT-M-261	Yes	6810-00-985-7098	72.10	0.805	42,108
toulene, tech.	Fed-TT-M-261	Yes	6810-00-290-0046	92.13	0.8669	7,585
methyl isobutyl ketone	Fed-TT-M-268	Yes	6810-00-281-2761	100.16	0.7978	605
naptha aliphatic	Fed-TT-N-95 Type 2	Yes	6810-00-238-8117	86.18	0.6603	1,030
deoderized kerosene	None	No	9140-00-965-2396	--	--	4,455
dichlormethane(methylene chloride)	Mil-D-6998	Yes	6810-00-616-9188	84.93	1.3266	8,635
trichlorotrifluoroethane(Freon 113)	Mil-C-81302 Type 2	Yes	6850-00-983-0282	187.4	1.5635	9,340
isopropyl alcohol	Fed-TT-A-735 Grade B	Yes	6810-00-855-1158	60.11	0.7855	1,665
epoxy resins and adhesives	Various	--	Various	--	--	1,223
epoxy paint remover	Mil-R-81294	†	8010-00-926-1490	--	--	25,755
epoxy primer	Mil-P-23377C	†	8010-00-082-2450	--	--	7,244
epoxy coatings	Mil-C-22750	†	Various	--	--	1,224
epoxy thinner(TMIK)	Mil-T-19588	†	6810-00-286-0458	--	--	980
acrylic paint remover	Fed-TT-R-248	†	8010-00-782-3928	--	--	1,100
acrylic lacquer	Mil-L-19538	†	Various	--	--	1,012

†Contains components which do appear on Wilks chart (reference 3)

TABLE 2 - TYPICAL MATERIALS USE LIST, NAVAIWORKFAC NORTH ISLAND (Cont'd)

Item	MIL-SPEC	Appears on Wilks Chart	NSN	Properties Needed		Annual Gallon Usage FY-76
				Molecular Weight	Liquid Density	
acrylic lacquer	Mil-L-81532	†	8010-00-935-6608	--	--	80
acrylic lacquer thinner	Mil-T-19544	†	8010-00-527-2896	--	--	6,255
polyurethane coatings	Mil-C-81773	†	Various	--	--	4,032
polyurethane thinner	Mil-T-81722	†	8010-00-181-8079	--	--	879
phenolic resins	Mil-R-7575		8030-00-200-3793			745
phenolic resins	Mil-R-3043		8030-00-200-6946			210
nubelon coating	None		8010-00-200-3259			56
polyester resin	Epon 828		None			300
enamel coating	Fed-TT-E-489		8010-00-616-7488			600
red oxide primer	Fed-TT-P-664		8010-00-292-1127			390
lacquer thinner	Fed-TT-T-266	†	8010-00-160-5789	--	--	6,575
varnish remover	Fed-TT-R-251	†	8010-00-160-5800	--	--	169
acetone	Fed-A-0-51	Yes	6810-00-281-1864	58.08	0.792	1,325
vinyl thinner	3M-3911		8010-00-506-1983			30
KEL-F coating	E.C. 1981		8030-LL-L03-0498			265
lubricating oil	Mil-O-6081 Grade 1010		9150-00-231-6676			16,500
lubricating oil	Mil-L-23699		9150-00-681-5999			9,552
Turco Caviclean-2	None		7930-LL-L03-0399			715
rust veto 4214	None		6859-LL-L03-0392			220
Turco Carb - Plain	None		6850-LL-L03-0466			706

† Contains components which do appear on Wilks chart (reference 3)

TABLE 2 - TYPICAL MATERIAL USE LIST, NAVAIREWORKFAC NORTH ISLAND (Cont'd)

Item	MIL-SPEC	Appears on Wilks Chart	NSN	Properties Needed		Annual Gallon Usage FY-76
				Molecular Weight	Liquid Density	
carbon remover	Mil-C-19853 Type 2	†	6850-00-550-7453	--	--	4,070
Surjex	Mil-C-6864		6850-00-551-3694			385
corrosion preventative	Mil-C-6529 Type 3		6850-00-209-7230			605
corrosion preventative	Mil-C-6529 Type 2		6850-00-209-7234			550
watch cleaner	L&R 222		6850-LL-L03-0326			660
cleaning compound	B&B 2020		6810-LL-L05-0002			670
cleaning compound	Fed-PC111 Type 2		6850-00-281-3042			880
finger print remover	Mil-C-15074	†	8030-00-252-8300	--	--	750
walkway coatings	Mil-W-5044		Various			1,450
Sermetal-W	Mil-C-81751		8030-00-145-0039			342
brush plating solutions	Dalic & Selectron		Various			1,995
		† Contains components which do appear on Wilks Chart (reference 3)				
Item	MIL-SPEC		NSN			Quantity Usage FY-76
preimpregnated fiberglass	PGLA181		None			14 rolls
plastic coating (Maskote 2)	Mil-P-23242		8030-LL-L03-0385			11,250 lbs
bonding adhesive film	AF-13, AF-30, AF-6, AF-126-2, AF-126-3		None			300 lbs
bonding adhesive film	AF110 & FMI000		None			80 rolls

TABLE 3 - SOME APPROXIMATE MIX COMPOSITIONS

ingredient	percent (%) by weight in a given formula mixture												
	epoxy paint remover	epoxy primer	epoxy thinner	acrylic paint remover	acrylic lacquer	acrylic lacquer thinner	poly-urethane thinner	lacquer thinner	varnish remover	carbon remover	epoxy coatings	poly-urethane coatings	finger print remover
butyl acetate	--	--	--	--	--	--	10.0	--	--	--	--	10.0	--
n-butyl alcohol	--	8.2	--	--	--	--	--	--	--	--	3.4	--	--
carbon tetrachloride	--	--	--	--	--	--	--	--	✓	--	--	--	--
cellosolve acetate	--	--	--	--	--	--	40.0	--	--	--	--	40.0	--
cresol	12.0	--	--	--	--	--	--	--	--	33.6	--	--	--
dicholomethane (methylene chloride)	48.8	--	--	44.2	--	--	--	--	✓	54.9	--	--	--
dimethylformamide	0.9	--	--	--	--	--	--	--	--	--	--	--	--
dioctyl phthalate	--	--	--	--	4.65	--	--	--	--	--	--	--	--
ethanolamines	1.7	--	--	0.8	--	--	--	--	--	--	--	--	--
ethyl acetate	--	--	--	1.0	--	--	--	✓	--	--	--	--	--
ethyl alcohol (ethanol)	--	--	--	1.7	--	--	--	✓	--	--	--	--	--
ethylene glycol mono-butyl ether	--	--	--	--	✓(?)	5.0	--	--	--	--	--	--	--
isopropyl alcohol	--	8.2	--	--	--	--	--	11.0	--	--	2.5	--	--
methyl alcohol (methanol)	3.6	--	--	3.4	--	--	--	--	--	--	--	--	✓
methyl cellosolve	--	--	--	--	--	--	--	--	--	--	--	--	--
methyl ethyl ketone (MEK)	2.0	--	--	4.0	17.7	--	30.0	✓	--	--	--	30.0	--
methyl isobutyl ketone (MIBK)	--	12.6	50.0	--	17.7	45.0	--	31.0	--	--	16.9	--	--
4-methyl-2-pentanol	--	--	--	--	--	--	--	8.0	--	--	--	--	--
monoethylamine	--	--	--	5.1	--	--	--	--	--	--	--	--	--
monoisopropanolamine	--	--	--	2.2	--	--	--	--	--	--	--	--	--
naphtha	--	--	--	--	--	--	--	25.0	--	--	--	--	✓
phenol	11.4	--	--	--	--	--	--	--	--	1.0	--	--	--
solids - various	✓	✓	--	✓	✓	--	--	--	--	✓	✓	--	--
toulene	--	19.5	50.0	7.7	2.79	45.0	12.0	25.0	--	--	✓(?)	12.0	--
toulene diisocyanate	--	--	--	--	--	--	✓	--	--	--	--	✓	--
xylene	--	3.2	--	--	--	5.0	8.0	✓	--	--	8.0	8.0	--

III. CALIBRATION RESULTS

A. General

Calibration procedure details are given in reference 1. These procedures were followed to obtain calibration on materials appearing in Table 4, including the mixtures. Most calibrations resulted in calibration curves of absorbance versus ppm concentration. These curves are always second order polynomials of the form

$$\text{Concentration (ppm)} = \alpha + \beta \cdot A + \gamma \cdot A^2$$

where A = absorbance read from MIRAN-IA meter

$$\left. \begin{array}{l} \alpha \\ \beta \\ \gamma \end{array} \right\} \text{equation coefficients}$$

The coefficients are obtained by a least squares regression analysis program using a Wang 2200C computer. A typical curve is shown in Figure 1.

Certain materials gave such a weak, noisy signal at the allowed concentration limits that a calibration was not possible. The best that could be said for most of these materials is that the concentration is or is not below the limiting value. This type information is quite valuable even though an actual concentration is not measured.

Mixtures of materials are quite commonly used in plant production work. Attempts at calibration and analysis on two frequently used mixtures are reported. In general, it would appear that detection of components in mixtures can be quantitatively made.

B. Calibration Curves - Pure Materials

The "curves" for materials successfully calibrated are reported in the form of α ; β ; γ constants rather than as graphical curves. The constants obtained to the time of the report are shown in Table 5.

Additional data are reported in this table. The column labeled "ppm per μl " should be read as "parts-per-million per microliter". This is a key number in all calibration work and is obtained by injecting microliter samples into the MIRAN-IA using Hamilton liquid or gas syringes. For all work reported here, liquids were used. The calculation of this number is done as follows:

$$\frac{C_{\text{ppm}}}{V_{\text{LIQ}}} \left(\frac{\text{ppm}}{\mu\text{l}} \right) = \frac{\rho \cdot R \cdot T \cdot 10^3}{5.64 \cdot \text{MOL WT} \cdot P_{\text{ATM}}}$$

TABLE 4 - MATERIALS CALIBRATED OR CONSIDERED FOR CALIBRATION USING FOXBORO/WILKS ANALYTICAL MIRAN-IA

1. acetic acid (glacial)	27. hexane
2. acetonitrile	28. hydrogen cyanide
3. acetone	29. isoamyl alcohol
4. benzene	30. isopropyl alcohol
5. <u>n</u> -butyl acetate	31. 2,2,4-trimethylpentane
6. <u>n</u> -butyl alcohol	32. methyl alcohol (methanol)
7. calibrating fluid (as heptane)	33. methyl isobutyl ketone (MIBK)
8. carbon tetrachloride	34. methyl cellosolve
9. chloroform	35. methyl cellosolve acetate
10. cresol (commercial isomer mix)	36. 4-methyl-2-pentanol
11. <u>m</u> -cresol	37. monoethanolamine
12. <u>p</u> -cresol	38. methyl ethyl ketone (MEK)
13. cyclohexane	39. naphtha (various boiling ranges)
14. decane (†)	40. octane
15. dichloromethane (methylene chloride)	41. pentane
16. diethylamine	42. phenol (88% in water solution)
17. N,N-dimethylformamide	43. stoddard solvent (as hexane)
18. dioctyl phthalate (†)	44. tetrachloroethane
19. <u>p</u> -dioxane	45. tetrachloroethylene (perchloroethylene)
20. epoxy primer (*)	46. 1,1,1-trichloroethane
21. epoxy thinner (*)	47. trichloroethylene
22. ethyl alcohol (ethanol)	48. 1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113)
23. ethyl acetate	49. triethylamine
24. ethylene glycol monobutyl ether (butyl cellosolve)	50. toluene
25. formaldehyde (37% water solution) (†)	51. <u>p</u> -xylene
26. heptane	52. xylene (commercial mix of isomers)

FIGURE 1

TYPICAL CALIBRATION CURVE
- FOR HYDROGEN CYANIDE (HCN)

WAVELENGTH - 3.04 μ

COARSE ZERO - X10

PATH LENGTH - 20.25 m

TIME CONSTANT - 2.5

SLIT - 2 mm

(0-0.1) ABSORBANCE

ABSORBANCE

0.07

0.06

0.05

0.04

0.03

0.02

0.01

LEAST SQUARES CURVE EQUATION:

$$C_{ppm} = -0.0403 + 597.707 A + 4293.45 A^2$$

10

20

30

40

50

60

CONCENTRATION (ppm)

TABLE 5 - CALIBRATION DATA

Material	properties		ppm per μ l	calibration					parameters			OSHA limit (ppm)	regression coefficients			remarks
	MOL WT	density (g/cc)		wave length (μ)	slit (mm)	dial-path	course zero	time const	absorb scale	α	β		γ			
acetic acid (glacial)	60.05	1.0492	74.48	8.34	1	13.31	X10	2.5	0.250	0.22400	33.3300	246.8200	difficult to work with			
acetonitrile	41.05	0.7857	81.59	9.60	1	13.31	X10	10.0	0.025	*	*	*	weak, noisy signal			
acetone	58.08	0.7920	58.13	8.10	1	1.40	X10	2.5	1.000	-18.56000	1731.0800	1017.4800	good signal			
benzene	78.12	0.8787	47.94	3.19	1	13.24	X10	10.0	0.025	*	*	*	difficult to work with			
n-butyl acetane	116.16	0.8820	32.37	8.03	1	2.18	X10	2.5	1.000	3.96150	218.9400	486.5900	good signal			
n-butyl alcohol	74.12	0.8098	46.57	9.50	1	3.50	X10	2.5	0.250	0.12840	812.3900	247.4100	good signal			
calibrating fluid	100.20	0.7100	29.09	3.37	1	0.25	X10	2.5	1.000	3.18510	622.0200	509.0100	treat as heptane			
carbon tetrachloride	153.82	1.5940	44.17	12.49	1	12.80	X10	2.5	1.000	0.07500	3.5377	42.9340	good signal			
chloroform	119.38	1.4832	52.96	12.85	1	3.50	X10	2.5	1.000	1.60620	54.1050	66.7001	good signal			
cresol (commercial mix)	108.13	1.0350	40.80	8.60	1	13.36	X10	10.0	0.025	*	*	*	(s)=skin			
cyclohexane	84.16	0.7789	39.43	3.38	1	1.30	X10	2.5	1.000	6.07830	219.2800	555.2600	good signal			
dichloromethane	84.93	1.3266	66.58	13.22	1	0.30	X10	2.5	1.000	-17.54000	1196.6800	959.7500	good signal			
diethylamine	73.14	0.7108	41.42	8.65	1	13.34	X10	2.5	0.250	-0.75040	293.2110	244.7890	good signal			
dimethylformamide	73.10	0.9487	55.32	9.19	1	12.80	X10	2.5	0.100	0.69623	74.2730	1069.8700	poor signal			
p-dioxane	88.12	1.0337	50.00	8.72	1	3.55	X10	2.5	1.000	8.87800	267.3700	-7.3123	good signal			
ethyl alcohol	46.07	0.7893	73.03	9.40	1	1.32	X10	2.5	1.000	3.56140	1226.1600	750.9270	good signal			
ethyl acetate	88.10	0.9010	43.59	7.92	1	0.46	X10	2.5	1.000	3.34450	411.7980	526.2790	good signal			
butyl cellosolve	118.17	0.9027	32.56	8.75	1	6.85	X10	2.5	0.250	1.02530	195.8140	619.6450	good signal			
heptane	100.20	0.7100	29.09	3.37	1	0.25	X10	2.5	1.000	2.38520	598.2280	544.9010	good signal			
hexane	86.18	0.6603	32.63	3.35	1	0.30	X10	2.5	1.000	0.65232	662.1490	395.9140	good signal			

* calibration curve unobtainable - can only say limit is or is not exceeded

Material	properties		calibration						parameters				regression coefficients			remarks
	MOL WT	density (g/cc)	ppm per μ l	wave length (μ)	slit (mm)	dial-path	course zero	time const	absorb scale	OSHA limit (ppm)	α	β	γ			
hydrogen cyanide	27.01	0.6880	108.58	3.04	2	13.30	X10	10.0	0.100	10	-0.04300	597.7070	4293.4500	good signal-Z _p		
isoamyl alcohol	88.15	0.8092	39.13	9.31	1	6.35	X10	2.5	1.000	100	-0.96790	369.2950	92.6287	good signal		
isopropyl alcohol	60.11	0.7855	55.70	8.58	1	3.51	X10	2.5	1.000	400	4.33890	899.8960	489.0010	good signal		
methyl alcohol	32.04	0.7914	105.29	9.39	1	3.50	X10	2.5	1.000	200	-12.40200	785.3150	-351.7700	good signal		
methyl isobutyl ketone	100.16	0.7978	33.95	8.42	1	13.27	X10	2.5	1.000	100	-1.43710	240.7110	69.2331	good signal		
methyl cellosolve	76.10	0.9647	54.03	8.70	1	13.34	X10	2.5	0.250	25	-0.33492	106.4290	75.2436	good signal		
methyl cellosolve acetate	118.31	1.0054	36.28	7.95	1	13.34	X10	2.5	1.000	25(s)	-0.34161	50.0459	19.5929	skin-good signal		
4-methyl-2-pentanol	102.18	0.8025	33.48	8.70	1	13.31	X10	10.0	0.100	25	-0.40436	312.3210	1503.8800	good signal		
methyl ethyl ketone	72.10	0.8050	47.59	8.40	1	13.02	X10	2.5	1.000	200	0.93115	212.2830	103.1660	good signal		
monoethanolamine	61.08	1.0180	71.04	13.00	2	13.30	X10	10.0	0.100	3	0.32273	849.8490	-10757.600	weak signal-poor data		
naphtha	86.18	0.6603	32.63	3.35	1	1.65	X10	2.5	1.000	500	-0.97334	370.1420	88.5547	treat as hexane		
octane	114.23	0.7025	26.21	3.35	1	0.35	X110	2.5	1.000	500	3.81390	456.5510	466.6390	good signal		
pentane	72.15	0.6262	37.00	3.35	1	0.30	X10	2.5	1.000	1000	3.16380	708.3650	576.7360	good signal		
phenol (88% in water)	94.11	1.0720	48.55	8.42	1	13.34	X10	2.5	0.250	5(s)	-0.16348	149.6410	130.2790	skin-good signal-Z _p		
stoddard solvent	100.00	0.7000	29.84	3.33	1	0.43	X10	2.5	1.000	500	3.45630	686.1030	1163.5100	treat as naphtha		
tetrachloroethane	167.85	1.5953	40.52	8.43	1	13.57	X10	10.0	0.025	5	*	*	*	weak signal-poor data		
tetrachloroethylene	165.85	1.5311	41.92	10.80	1	4.06	X10	2.5	1.000	100	5.57200	70.3504	232.3660	good signal		
trichloroethane	133.42	1.3390	42.78	9.2/13.9	1	2.64	X10	2.5	1.000	350	1.02790	371.5840	381.4150	good signal		
trichloroethylene	131.39	1.4558	47.23	11.70	1	3.00	X10	2.5	1.000	100	2.53190	274.2320	170.9020	good signal		

* calibration curve unobtainable - can only say limit is or is not exceeded.

μ l = microliter Z_p -zero point included in regression

TABLE 5 - CALIBRATION DATA (Cont'd)

Material	properties		ppm per μ l	calibration					parameters			OSHA limit (ppm)	regression coefficients			remarks
	MOL WT	density (g/cc)		wave length (μ)	slit (mm)	dial-path	course zero	time const	absorb scale	α	β		γ			
Freon 113	187.40	1.5635	35.57	8.36	1	0.30	X10	2.5	1.000	45.34800	251.4660	1257.3900	1000	251.4660	1257.3900	good signal
triethylamine	101.19	0.7229	30.45	9.23	1	13.34	X10	2.5	0.250	-0.66904	309.9940	223.8040	25	309.9940	223.8040	good signal
toluene	92.13	0.8669	40.11	13.67	2	5.91	X10	2.5	1.000	-0.76239	343.1910	160.5340	200	343.1910	160.5340	good signal
p-xylene	106.13	0.8611	34.58	12.45	1	12.75	X10	2.5	1.000	-1.28750	237.9490	110.1340	100	237.9490	110.1340	good signal
xylene (commercial mix)	106.13	0.8611(?)	34.58	12.60	2	13.28	X10	10.0	0.250	-0.52443	569.6900	758.5050	100	569.6900	758.5050	good signal-Z _p

* calibration curve unobtainable - can only say limit is or is not exceeded.

μ l = microliter μ = microns Z_p-zero point included in regression

where:

V_{LIQ} = Liquid volume in microliters

C_{ppm} = Concentration in parts-per-million

P_{ATM} = Pressure in atmospheres (assume 1 atm)

ρ = Liquid density (grams per milliliter)

MOL WT = Molecular weight (grams per mole)

R = Gas constant (0.08205 liter atmospheres per mole °K)

T = Absolute temperature (°K) (use 293 °K = 20 °C)

5.64 = Volume of gas cell plus calibrating pump (liters)

with actual numerical values used being:

$$\frac{\text{ppm}}{\mu\text{l}} = \frac{\rho \cdot (0.08205) \cdot 293 \cdot 10^3}{5.64 \cdot \text{MOL WT} \cdot 1} = 4262.5 \cdot \frac{\rho}{\text{MOL WT}}$$

A typical calibration curve is obtained as follows using methyl isobutyl ketone. Using

MOL WT = 100.16 and Density ρ = 0.7978

calculate

$$\frac{\text{ppm}}{\mu\text{l}} = \frac{0.7978}{100.16} (4262.5) = \underline{\underline{33.95}}$$

From the Foxboro Analytical Chart (reference 3) the suggested MIRAN-IA settings are:

Wavelength-8.5 μ Path-20.25 meters Absorbance-0.31

OSHA limit-100 ppm

The amount of liquid sample needed to provide a concentration of 100 ppm is:

$$\frac{100}{33.95} = 2.95 \mu\text{l}$$

therefore, 3 μl are used. In addition, it is decided that a slit opening of 1 mm, a time constant of 2.5, and a coarse zero setting of X10 will be used. The path length of 20.25 meters is about equivalent to a dial setting of 13.00 on the instrument. With all these parameters set, a 3 μl liquid sample is injected into the system and the wavelength wheel

and path length dial are varied until a maximum signal is obtained. The best signal is found at a wavelength of 8.42 μ and a path dial setting of 13.27 with an absorbance reading of about 0.38 on unit absorbance scale. With all instrument parameters set, the calibration proceeds. It is done by injecting 0.5 μ l increments up to a total of 3 μ l. After each 0.5 μ l increment is injected, approximately 30 to 60 seconds are allowed for the instrument to settle to a steady output value before the next injection. This procedure is repeated, possibly with variations, for checking purposes.

An average absorbance reading is obtained at each concentration level. This data is then fed to the Wang 2200C least squares regression analysis program, which provides the "best" (in the least squares sense) second order polynomial to fit the data. This fit is in the form of the α ; β ; γ constants shown in Table 5 under methyl isobutyl ketone.

A calibration is specific for the material used. Many of the materials used were obtained in a pure or spectro-grade form from the NAVPGSCOL chemistry laboratories. It was found the same chemical material obtained from the plant or laboratory of NAVAIWORKFAC North Island might give a similar but different calibration. This is probably due to commercial grade material which is not pure, or to mixtures of isomers for a material having isomeric forms. Care should be taken to calibrate with the same material that will actually be used in plant work so that correct plant concentrations will be reported.

The slit opening generally was set at 1 mm. If a signal was weak, slit opening could be increased, as well as path length, to try to improve the signal. Only a few materials required this action. The time constant was varied depending on noisiness of signal and rate of signal response. This generally was a problem for weak signal materials requiring 0.1 or 0.025 absorbance scales. All calibrations reported were made with the original detector/preamplifier supplied with the instrument. This device tended to be noisy. A new detector/preamplifier was purchased and installed before going into the plant with the instrument. The new device appears to give signals with appreciably attenuated noisiness.

The complete calibration results for methyl isobutyl ketone are shown in Table 6. The regression analysis was done with and without a zero point. Theoretically, all calibration curves should go through zero. However, when a curve is "forced" to go through zero by inputting a '0' absorbance point, the resulting polynomial fit is generally relatively good near zero and not so good at concentrations near the OSHA limit. If the zero point is left out of the regression analysis, the fit near zero is not too good, while that near the OSHA limit is generally better than for the zero point case. The α ; β ; γ constants of Table 5 are for the non-zero point case with a few noted exceptions.

TABLE 6 - TYPICAL CALIBRATION RESULTS - METHYL ISOBUTYL KETONE (MIBK)
(Source - Pure from NAVPGSCOL Chemical Labs)

μ l liquid injected	A = absorb- ance	C_{ppm} = conc (ppm)	regression analysis			
			with '0' point		w/o '0' point	
			value	% E	value	% E
0.0	0	0	∞	-0.29	-1.44	∞
0.5	.073	16.98	.06	16.99	16.50	2.83
1.0	.146	33.95	3.77	35.23	35.18	2.27
1.5	.203	50.93	1.55	50.14	50.28	1.28
2.0	.267	67.90	0.47	67.58	67.77	0.19
2.5	.327	84.88	0.34	84.59	84.68	0.24
3.0	.387	101.86	0.38	102.25	102.09	0.23
		α		-0.29001848		-1.4371272
		β		230.18757600		240.7109490
		γ		89.87631500		69.2331060

Polynomial fit (with '0' point) equation:

$$C_{ppm} = -0.29001848 + 230.187576A + 89.876315A^2$$

Polynomial fit (without '0' point) equation:

$$C_{ppm} = -1.4371272 + 240.710949A + 69.233106A^2$$

A = measured variable

C_{ppm} = calculated variable

MADE USING:

Wavelength - 8.42 μ

slit - 1mm

path set - 13.27 (\approx 20.25 meters)

coarse zero - X10 (gain)

time constant - 2.5

absorbance scale - (0-1)

C. Calibration Curves-Mixtures

In many areas of the NAVAIREWORKFAC North Island plant, mixtures of materials are used in daily operations. If the MIRAN-IA is to be used for sampling in these areas, it is necessary to give some consideration to the effect of one or more other materials being present when sampling for a specified material. Many questions arise. Assuming the composition of a mixture is known, it is possible to set the instrument at the calibration settings for one of the ingredients and to sample for that ingredient and to try to determine the effect on absorbance readings due to other ingredients present. If the other ingredients have absorbance at the wavelength, path length, etc. set for a specified ingredient, then absorbance readings should be different than those for the pure material. Will the measured absorbance be more or less than that for the pure material? How is the actual concentration for a given material determined when absorbance readings come from a mixture of two or more materials? In an attempt to answer these and other questions, two common mixtures were chosen to work with.

The first was epoxy thinner used in paint shops. It is known to be a mixture of equal parts by weight of toluene and methyl isobutyl ketone (MIBK) as follows:

	<u>Weight Fraction</u>	<u>Mole Fraction</u>	<u>Volume Fraction</u>
Toluene	0.5	0.52088	0.47916
MIBK	0.5	0.47912	0.52084

If the instrument is set at calibrate settings for toluene and 10 μ l of epoxy thinner mix are injected, an absorbance of about 0.46 might be expected. This figure was obtained by noting that 10 μ l of mixture contains 4.8 μ l of toluene which, if injected as pure material, should give a 0.46 absorbance reading. The actual reading was about 0.40.

On the other hand, if the instrument is set at MIBK calibrated settings and 5.75 μ l of epoxy thinner mix are injected, an absorbance of about 0.35 might be expected. In 5.75 μ l of mix, there are 3 μ l of MIBK which should give an absorbance of about 0.35, if injected as pure material. The actual reading for 5.75 μ l of mix was about 0.34.

These readings are close to those for pure materials and suggest that it might be possible to sample for toluene or MIBK as pure materials even though they are present in a mixture. This would be the case if there was absolutely no interference from the presence of other materials. Alternatively, the mixture could be treated as a "new" material for which a calibration could be run and then an attempt could be made to get data for C_{ppm} of toluene and MIBK. The results could be used for plant sampling in areas where epoxy thinner is used.

The alternative procedure was followed and the results are shown in Table 7 and Table 8. Thus, it should be possible to take the instrument into a plant area where epoxy thinner mix is being used, set

TABLE 7 - CALIBRATION RESULTS-EPOXY THINNER MIX-TOLUENE CALIBRATION SETTINGS

μl mix injected	in mix		MIBK C _{ppm}	mix absorb- ance	toluene C _{ppm}	regression analysis				pure to luene C _{ppm}
	μl to luene	μl MIBK				with '0' point		w/o '0' point		
						value	% E	value	% E	
0	0	0	0	0	0	-0.52	∞	-1.60	∞	-0.76
1	0.47916	0.52084	17.68	.063	19.22	20.77	2.86	20.20	5.10	21.50
2	0.95832	1.04168	35.37	.108	38.44	37.36	2.81	37.07	3.56	38.18
3	1.43748	1.56252	53.05	.158	57.66	57.13	0.92	57.07	1.02	57.47
4	1.91664	2.08336	70.73	.206	76.88	77.44	0.73	117.53	0.85	76.75
5	2.39580	2.60420	88.42	.249	96.10	96.74	0.67	96.90	0.83	94.68
6	2.87496	3.12504	106.10	.288	115.31	115.15	0.14	115.33	0.02	111.39
7	3.35412	3.64588	123.79	.326	134.53	133.91	0.46	134.06	0.35	128.18
8	3.83328	4.16672	141.47	.364	153.75	153.49	0.17	153.56	0.12	145.43
9	4.31244	4.68756	159.15	.401	172.97	173.33	0.21	173.28	0.18	162.67
10	4.79160	5.20840	176.84	.435	192.19	192.76	0.24	192.05	0.07	178.91
					α	-0.5204405		-1.599366		
					β	320.2164500		329.234010		
					γ	282.6383100		266.528040		

The last column obtained by using mix absorbance value in pure toluene calibration equation. Indication is that for a given C_{ppm} of toluene, the absorbance in the mix will be lower than pure toluene absorbance.

TABLE 8 - CALIBRATION RESULTS-EPOXY THINNER MIX-MIBK CALIBRATION SETTINGS

μl mix injected	in mix		to luene C_{ppm}	mix absorb- ance	MIBK C_{ppm}	regression analysis				pure MIBK C_{ppm}
	μl to luene	μl MIBK				with '0' point		w/o '0' point		
						value	% E	value	% E	
0	0	0	0	0	0	-0.17	∞	-0.99	∞	-0.29
1	0.47916	0.52084	19.22	.074	17.68	18.35	3.79	18.04	2.04	17.24
2	0.95832	1.04168	38.44	.135	35.37	35.19	0.51	35.17	0.57	32.42
3	1.43748	1.56252	57.66	.190	53.05	51.61	2.71	51.71	2.52	46.69
4	1.91664	2.08336	76.88	.255	70.73	72.50	2.50	72.62	2.67	64.25
5	2.39580	2.60420	96.10	.300	88.42	87.91	0.57	87.96	0.51	76.86
6	2.87496	3.12504	115.31	.350	106.10	105.95	0.14	105.83	0.25	91.29
					α	-0.1681649		-0.988332		
					β	236.0371100		244.323980		
					γ	191.8752900		173.931220		

The last column obtained by using mix absorbance value in pure MIBK calibration equation. Indication is that for a given C_{ppm} of MIBK, the absorbance in the mix will be lower than pure MIBK absorbance.

it for either toluene or MIBK, take readings, and resolve them into concentration of both materials. The tables also show the data for pure material and indicate that at a given C_{ppm} , the absorbance for a material present in the mix will be lower than if the same material were present as pure material.

The other mixture studied was epoxy primer, which is a mixture of solids and liquids as follows:

	<u>% by weight of total mix</u>	<u>% by weight of liquid part</u>
solids	48.29	--
MIBK	12.60	24.37
toluene	19.50	37.70
xylene	3.21	6.21
isopropyl alcohol	8.20	15.86
<u>n-butyl alcohol</u>	<u>8.20</u>	<u>15.86</u>
	<u>100.00</u>	<u>100.00</u>

It is the liquid part of this mix that is of interest since it contains five ingredients. The liquid portion was injected into the MIRAN-IA in 3 and 15 μ l amounts at the calibration settings for each of the five pure materials. The 15 μ l amounts contained the following amounts of the various ingredients:

	l of Mix Ingredient	Equivalent C
MIBK	3.66	128.33*
toluene	5.65	215.99*
xylene	0.93	31.13
isopropyl alcohol	2.38	139.53
<u>n-butyl alcohol</u>	<u>2.38</u>	<u>113.16*</u>

15.00

*OSHA limit exceeded.

The results at the various pure component calibration settings are shown in Table 9. In general, for a given C_{ppm} , the absorbance of the mix is higher than it would be for the pure material. This behavior is the opposite of that experienced for the epoxy thinner mix and indicates that mixture behavior is unpredictable. To be sure of mixture behavior, it should be calibrated before using the MIRAN-IA to sample for material in a work area.

In any case, if sampling is done in areas where mixtures are being used, any absorbance reading obtained that is less than OSHA limit indicates that a safe level of concentration for the given material exists. What may not be readily available is the exact concentration of the given material. The problem lies in what other materials present are contributing to an absorbance reading. It is known that absorbance readings are additive. Thus, if the contributions of other materials is known, these corrections can be made so that an exact concentration can be determined. However, the first question that must always be answered is "Is the OSHA limit for the concerned material being exceeded?" The

TABLE 9 - CALIBRATION RESULTS - EPOXY PRIMER MIX - VARIOUS CALIBRATION SETTINGS

μl mix injected	toluene calibrate set			pure toluene C_{ppm}	MIBK calibrate set			pure MIBK C_{ppm}
	μl	absorb- ance	C_{ppm}		μl	absorb- ance	C_{ppm}	
0	0	0	0	-0.76	0	0	0	-0.29
2	0.718	.102	28.80	35.91	0.504	.135	17.11	32.42
4	1.436	.181	57.60	66.61	1.008	.259	34.22	65.36
6	2.154	.262	86.40	100.17	1.512	.371	51.33	97.48
8	2.872	.330	115.20	129.97	2.016	.486	68.44	132.81
10	3.590	.395	143.99	159.85	2.520	.568	85.55	159.45
12	4.308	.453	172.79	187.65	3.024	.669	102.66	193.93
14	5.026	.514	201.59	218.95	3.528	.763	119.78	227.67
15	5.385	.525	215.99	223.66	3.780	.790	128.33	237.65

μl mix injected	xylene calibrate set			pure xylene C_{ppm}	isopropyl alcohol calibrate set			isopropyl alcohol C_{ppm}
	μl	absorb- ance	C_{ppm}		μl	absorb- ance	C_{ppm}	
0	0	0	0	-0.52	0	0	0	1.32
3	.18	.0188	6.22	10.46	0.501	.050	27.91	48.79
6	.36	.0344	12.45	19.97	1.002	.104	55.81	102.49
9	.54	.0506	18.67	30.25	1.503	.149	83.72	149.19
12	.72	.0681	24.90	41.79	2.004	.192	111.62	195.46
15	.90	.0831	31.12	52.06	2.505	.230	139.53	237.69

μl mix injected	n-butyl alcohol calibrate set			pure n-butyl alcohol C_{ppm}
	μl	absorb- ance	C_{ppm}	
0	0	0	0	2.88
2	0.324	.0293	15.09	26.95
4	0.648	.0572	30.18	50.25
6	0.972	.0822	45.27	71.44
8	1.296	.1080	60.35	93.62
10	1.620	.1325	75.44	114.98
12	1.944	.1548	90.53	134.66
14	2.268	.1760	105.62	153.59
15	2.430	.1873	113.17	163.77

second question is "What is the exact concentration of the concerned material?" The first question can always be answered by the use of the MIRAN-IA, and it may well be possible to answer the second question also.

IV. UPDATING THE MIRAN-IA

A. General

The MIRAN-IA owned by the AESO was obtained in January of 1973. It was used extensively in 1976, for a period covering two months, and again in 1976 for some work at NAVPGSCOL, covering a period of one month. Prior to these periods, it had been little used. In anticipation of some further work to be done using the instrument during 1978, updating the instrument was desirable. Accordingly, letters of inquiry were sent to

Dr. Donald S. Lavery
Consultant in Analytical Chemistry
13 Betts Place
East Norwalk, CT 06855

and to

Foxboro Analytical
Suite 301
151 Callan Ave.
San Leandro, CA 94577

asking about the possibility and desirability of updating the MIRAN-IA.

Dr. Lavery replied to the effect that the original performance tests should be repeated and results compared to original results. If a degradation of performance was noted, then the following should be done:

1. Inspect windows and replace if surfaces are deteriorated.
2. Replace detector/preamplifier assembly.
3. Rebuild long-path cell.

Mr. Charles Salisbury of the Foxboro San Leandro office visited NAVPGSCOL to observe and check the instrument. It was his opinion that the sodium chloride windows had deteriorated due to exposure to moisture. Even though the instrument was still usable, the infrared energy reaching the long-path cell was substantially reduced. It was recommended the windows be replaced by silver bromide windows, which are less susceptible to moisture deterioration and pass a wider band of infrared energy. Mr. Salisbury also recommended that the detector/preamplifier assembly be replaced with a current model, which is much less susceptible to the effect of airborne acoustical interference (i.e., microphonic interference).

As a result of the recommendations of Dr. Lavery and Mr. Salisbury, the following materials were ordered:

1. Zero gas filter for removal of organic vapors.
2. Replacement cartridges for the zero gas filter.
3. New sodium chloride windows.
4. Silver bromide windows.
5. Silver bromide window holders.
6. Complete detector/preamplifier assembly.
7. Book on "Hazardous Gases and Vapors" (reference 4).
8. Particulate filter replacement cartridges.
9. Septa membranes for use with calibration circulating pump.
10. Slide/tape presentation on the detection and measurement of toxic gases and vapors.

Item 10 is the first step in a plan to provide further instruction and insight to the use of infrared techniques for determining compliance with OSHA regulations. Both Dr. Lavery and Foxboro Analytical have been consulted about the possibility of a short course presentation at AESO San Diego, concerning the use of infrared techniques and the MIRAN-IA in particular. It is hoped that a more intelligent and wider use of such instruments can be made if a continuing education aspect is introduced.

Dr. Lavery is quite willing to provide this service at a rather substantial cost. Foxboro Analytical says it will consider providing such a service, but is not yet organized to do so. However, they indicated they might arrange some sort of training program in the near future. Accordingly, no decision has been made on what to do at the moment. A sort of "pressure" is being kept on Foxboro Analytical to do something since it may be more desirable for the manufacturer to give such a presentation.

The local (i.e., San Diego) Foxboro representative

Mr. James Steele
Foxboro Analytical
Flair Business Park
Suite 100 - 9420 Telstar Ave.
El Monte, CA 91731

has been contacted concerning updating the MIRAN-IA and a course presentation. He has visited the AESO group for consultation on changes to the instrument, and has indicated that our request for continuing education has been passed on to the company for consideration.

All materials ordered have arrived, the last parts reaching San Diego on 22 November 1978.

B. Window Replacement

The AESO version of the MIRAN-IA has a 7.5 cm cell built in for possible use with strongly absorbing materials. This short cell provided some concern as to just how to replace the cell windows. The Operation, Maintenance, and Service Manual (reference 11) was written for an instrument without the short cell. After consultation with Mr. Salisbury and Mr. Steele of Foxboro Analytical and thoroughly studying the instrument mechanical assembly, the replacing of the sodium chloride windows with silver bromide windows was done with no trouble. The details of the replacement procedure are written up in reference 12, page 43.

It was not necessary to get an instrument reference reading before separating the cell body from the infrared source module. The mechanical assembly was such that when the separated bodies were re-joined, there was no optical alignment shifting that would require peaking the instrument response. The short cell was put back even though it never had been used. At some future time there might be a need for it.

After installation of the silver bromide windows, the instrument was set for MIBK calibration and 3 μ l of MIBK were injected. A good response was obtained, which checked earlier results using the sodium chloride windows. This response can be seen in reference 12, page 43.

C. Detector/Preamplifier Replacement

It is possible for optical misalignment to occur when changing detector/preamplifier assemblies. Thus, before starting the change, the instrument was set to the following settings:

slit - 1 mm time constant - 1 coarse zero - X10
wavelength - 3.5 μ path dial - 0.3 (i.e., '0' meters)

and the fine zero was adjusted to its minimum point, which gives about 0.0 on the percent Transmittance (T) scale.

After installing the new assembly, it was necessary to move it in X, Y, and Z directions to try to maximize a reading of about 60 percent T, which was set using fine zero control. This was accomplished with little or no trouble. The details of the change appear in reference 12, page 41.

Finally, the instrument was set for MIBK calibration and 3 μ l of MIBK were injected. A good response was obtained, which checked with that obtained after window installation and with the sodium chloride windows. It was noted that the recorded response gave a much "quieter" signal, which is the behavior expected. The response can be seen in reference 12, page 43, and can be compared to that for window replacement only.

V. ADDITIONAL MATERIALS

A. General

The commercial mixture of isomers used for cresol proved to be quite difficult to work with in that a poor signal was obtained and no calibration was possible. Samples of the para and meta cresol isomers were available at NAVPGSCOL and were run for calibration.

Three additional materials were candidates for calibration and were run. They are:

1. Dioctyl phthalate (a plasticizer)
2. Isooctane
3. Formaldehyde (37% water solution)

All three are used in varying amounts at NAVAIROWORKFAC North Island. Only one, formaldehyde, appears on the Foxboro Analytical chart, with an OSHA limit of 3 ppm.

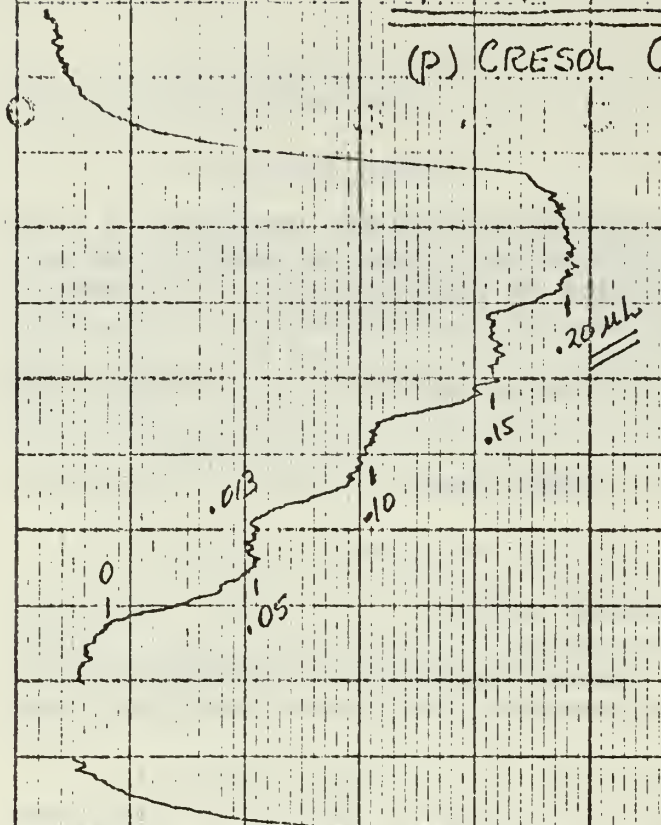
B. p-Cresol and m-Cresol

Samples of these isomers were successfully calibrated. The calibration data is shown in Figures 2 and 3. The calibration results are:

	<u>p-Cresol</u>	<u>m-Cresol</u>
MOL WT	108.13	108.13
density (g/ml)	1.0347	1.0336
ppm/ μ l	40.79	40.74
wavelength (μ)	8.48	8.6
slit (mm)	1	1
dial path	13.29	13.29
coarse zero	X10	X10
time const.	1	1
absorb scale	(0-.1)	(0-.1)
OSHA limit (ppm)	5 (skin)	5 (skin)
α	-1.41704×10^{-3}	1.81125×10^{-2}

FIGURE 2

(P) CRESOL CALIBRATION DATA



P-CRESOL

$\lambda = 8.48$ DIAL-13.29 X10

1mm $\Sigma = 1$ (0-.1) AB
20mm/m

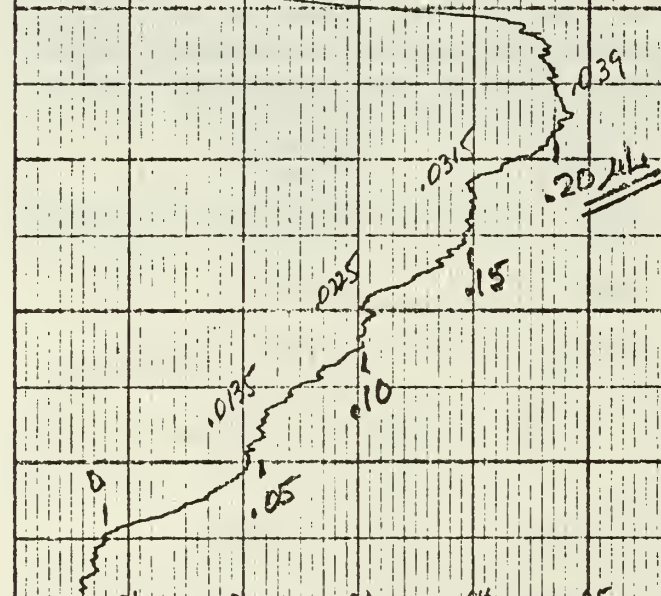
AMT IN3	AB		AUG
.05	.0135	.013	.0133
.10	.0225	.023	.0228
.15	.0315	.0335	.0325
.20	.0390	.0400	.0395

CHART 10.9-21193

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ESTIMATED ANGLE

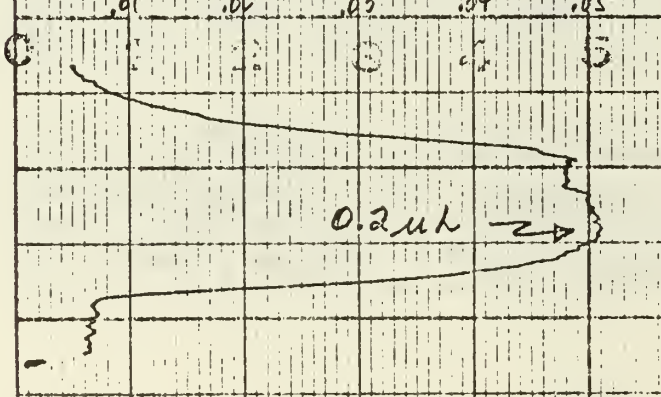
CHART 10.9-1.193



(P) CRESOL

$\lambda = 8.48$ $\Sigma = 1$ X10 (0-.1) AB

SLIT-1mm DIAL-13.29 20mm/m



(P) CRESOL

$\lambda = 8.48$ $\Sigma = 1$ X10 (0-.1) AB

SLIT-1mm DIAL-13.29

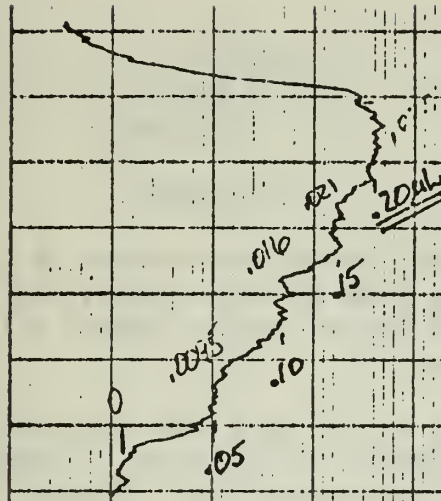
CHART SPEED - 20mm/m

CHART VOLTAGE - (0-1)V.

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1 DEC. 78

FIGURE 3

(m) CRESOL CALIBRATION DATA

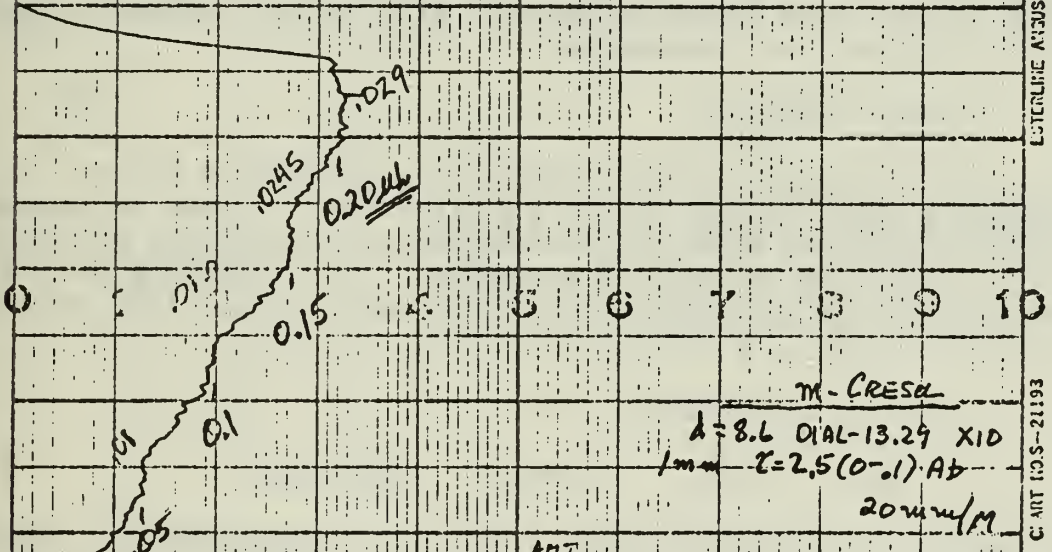


m-CRESOL

$$\lambda = 8.6 \text{ DIAL-13.29 X10}$$

$$1 \text{ mm } \gamma = 1.0 \text{ (0-.1) AB}$$

20 mm/M



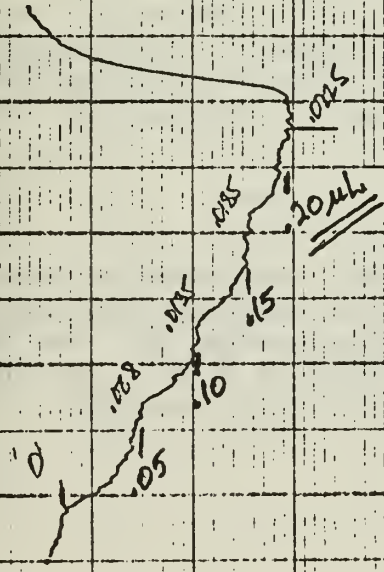
m-CRESOL

$$\lambda = 8.6 \text{ DIAL-13.29 X10}$$

$$1 \text{ mm } \gamma = 2.5 \text{ (0-.1) AB}$$

20 mm/M

AMT [µM]				AMIS
.05	.0080	.0100	.0095	.0092
.10	.0135	.0160	.0160	.0152
.15	.0185	.0245	.0210	.0213
.20	.0275	.0290	.0250	.0255



m-CRESOL

$$\lambda = 8.6 \text{ DIAL-13.29 X10}$$

$$1 \text{ mm } \gamma = 2.5 \text{ (0-.1) AB}$$

ESTERLINE / CHART SPEED - 20 mm/M
ANALYS / CHART VOLTAGE - (0-1) V

1 DEC. 78

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ESTERLINE ANALYS

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ESTERLINE ANALYS

	<u>p-Cresol</u>	<u>m-Cresol</u>
β	131.62263	177.17585
γ	1864.81075	5513.8483

Both materials gave good relatively noise-free signals at a time constant of unity. This work was done with the new windows and new detector/preamplifier, and indicates that a better quality signal will generally be available with the new equipments.

It should be noted that the first cresol work done was with a commercial isomer mix obtained at NAVAIWORKFAC North Island. This material had an unknown but bad effect on two one-microliter syringes in that they were both completely unusable after working with this material. New syringes were purchased and used with the para and meta isomers and no difficulties were encountered.

C. Diocetyl Phthalate

This material is one of several plasticizers in use at NAVAIWORKFAC North Island, but is a typical one. It does not appear on the Foxboro Analytical chart, so no information is available about its potential toxicity or calibration parameters. It is possible to find infrared spectra for this material and they indicated possible absorbance bands around 7.8-7.9 μ or 9 μ .

Using the properties

MOL WT - 390.3 Density - 0.978 g/ml.

it was possible to calculate ppm/ μ l = 10.68. It was decided to inject 5 μ l of this viscous (oily) material and look for the best signal possible. It was not possible to find a recordable signal in the 7.8-7.9 μ wave-length region.

Upon trying the 9 μ region and using slit opening of 2 mm, a recordable signal resulted. This is shown in Figure 4. A calibration was not attempted even though the results indicated it could be done. The objective of showing that this material was calibratable was accomplished.

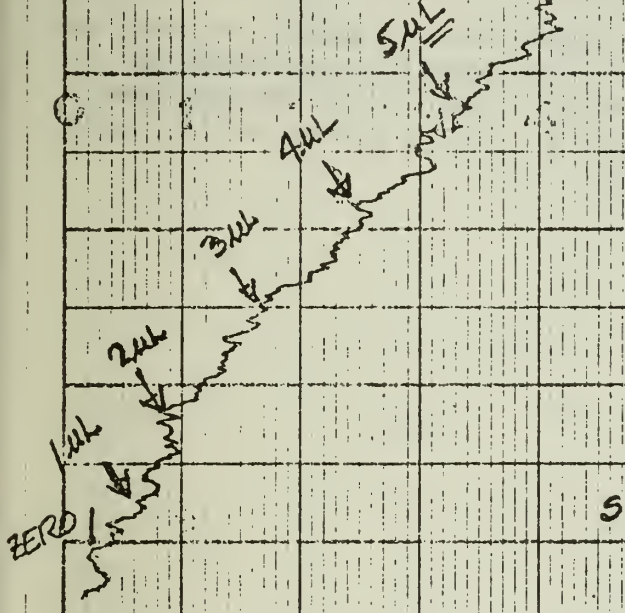
D. Formaldehyde

This material is highly toxic and can cause death in high concentrations. The OSHA standards are: 3 ppm for an 8-hour time-weighted average and 5 ppm maximum exposure for any 30-minute period. The Foxboro Analytical chart reports an analytical wavelength of 3.58 μ . No other calibration parameters were known so, as with diocetyl phthalate, a trial-and-error approach was necessary.

FIGURE 4

FLUSH WITH "ZERO" AIR

DIOCTYL PHTHALATE -
INJECT 1 μ L AT A TIME
UP TO 5 μ L TOTAL.



$\lambda = 9.15$ SLIT-2mm X10 $\gamma = 2.5$
(0-.025) AB DIAL-13.29 20mm/M

SIGNAL DEFINITELY PRESENT!!

DIOCTYL PHTHALATE

$\lambda = 9.15$ SLIT-2mm X10 $\gamma = 2.5$
DIAL-13.29 CHART-20mm/M
(0-.025) AB CHART VOLTS-(0-1)

g. W. Duffin
1 DEC. 78

ESTERLINE P-505

CHART HQ.5-21193

ESTERLINE ANGUS

CHART HQ.5-21193

An original wavelength setting of about 3.58 μ was varied upon injection of a 0.2 μ l sample. A unique maximum was not found so 3.58 μ was set on the wavelength wheel. The signal was rather weak - (0-.025) absorbance scale - so the slit was opened to 2 mm. As can be seen from Figure 5, a reasonable signal was obtained with a time constant of one. Some sort of calibration appears to be possible.

E. Isooctane (2,2,4-Trimethylpentane)

This is a strictly try-it-for-the-fun-of-it material! Data was available on physical properties and an absorbance peak was shown at about 3.4 μ . As can be seen from Figure 6, a good signal was obtained and calibration, although not done, could be easily carried out. Concentrations up to about 77 ppm was checked.

FIGURE 5

FORMALDEHYDE
SOLUTION INJECTIONS.

~ 0.2 μL

FORMALDEHYDE

CHECK RUN

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~ 0.2 μL

FORMALDEHYDE

(37.4 WEIGHT % - 40 VOL. % H₂O SOLN.)
 $\lambda = 3.52$ SLIT - 2 mm $\chi = 1$
(0-.025) AB DIAL - 13.29 20 mm/M

CUT 103-21

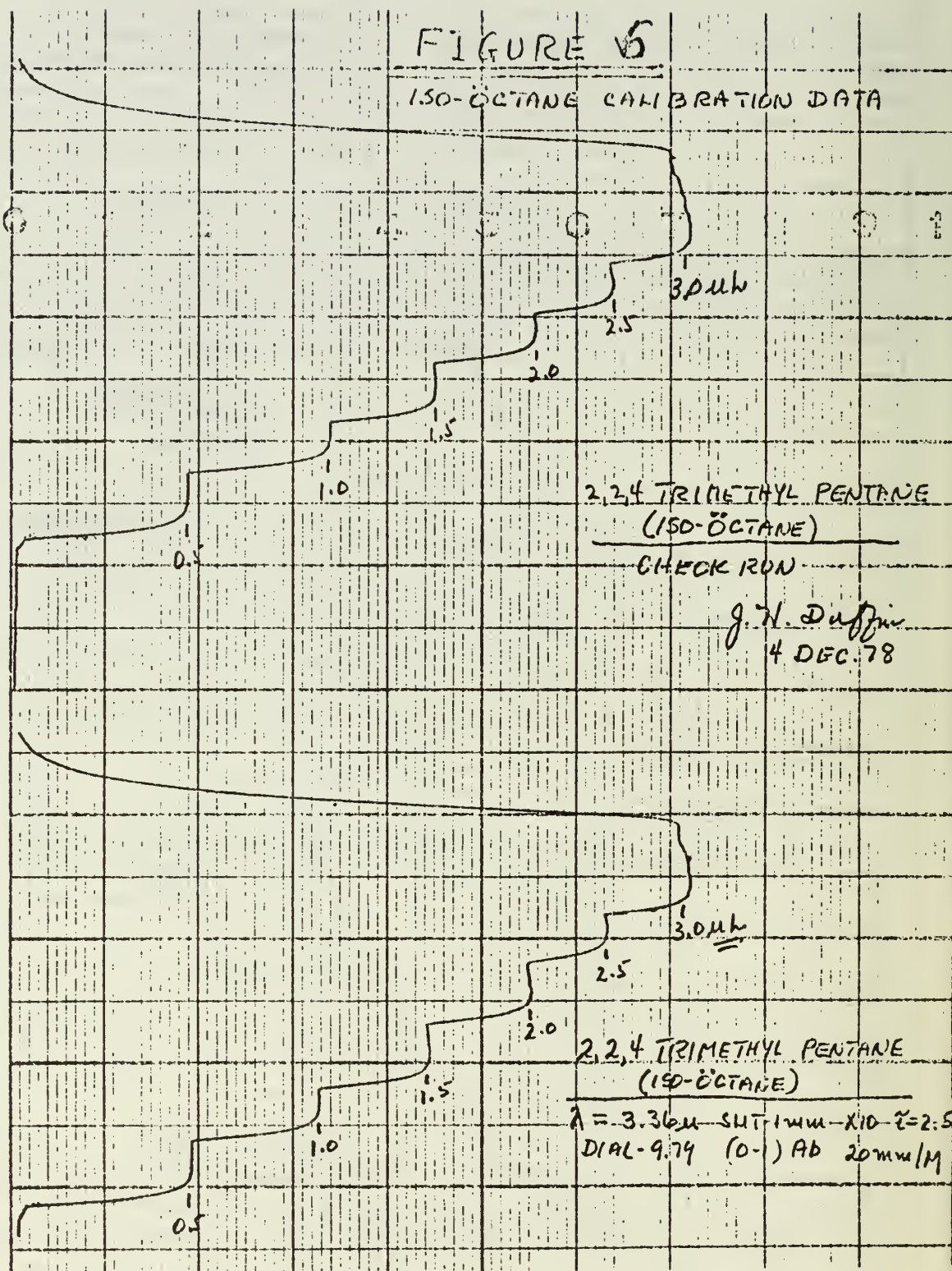
INDIANAPOLIS IND U.S.A.

ESTERLINE ANGUS

CUT 103-2193

FIGURE 5

ISO-OCTANE CALIBRATION DATA



J. N. Duffin
4 DEC. 78

VI. PAINT SHOP MONITORING

A. General

One of the original goals of the work reported upon here was to use the MIRAN-IA to monitor in various locations at NAVAIREWORKFAC North Island. Accordingly, three areas were investigated. They were: (1) Paint Shop, Bldg. 472; (2) Chemical Cleaning Shop, Building 472; and (3) Airplane Paint/Strip Shops, Buildings 464, 465, 466, 467, and 468. It was hoped that the "production" in these areas could be correlated in the form of a model, which would predict expected hazardous gas air concentration levels as a function of production rates.

In each of the three areas above, production consists of pieces of hardware moving into and out of an area where a series of services are performed. The pieces can vary from something the size of a small screw to a full-sized airplane. In view of this, production is somewhat hard to define and measure. It would appear that one possibility is to define production in terms of total number of pieces moving through an area per unit of time. Such a figure would mask other properties such as mass movement, area movement (in the sense of surface area), etc. How to define production so that it can be used in correlation work would appear to be an interesting study in its own right.

The areas chosen were all users of fairly large amounts of potentially hazardous materials. Thus, some correlation between airborne concentration levels of these materials and some defined production rate might be expected.

B. Paint Shop, Building 472

About two weeks were spent in this area. The first week was spent in observation of the kinds of pieces that moved through the shop and the rate of movement of total pieces. It turned out that such a wide variety of pieces moved through at such a random rate that any attempt to define a production rate was difficult and time consuming. Because of this, it was decided to monitor the spray painting of pieces, both large and small, with a variety of paints to see what could be expected in the way of readings on the MIRAN-IA. The instrument was set up on a portable cart with about 60 feet of power cord so that it could be moved to various paint stations.

On a given day, a specific material was monitored. The first thing done in the morning was an instrument calibration for that material. Then the spray painting of various pieces moving through the paint stations was monitored. In many cases, the paint mixtures had to be prepared and this process was also monitored. For the most part, small to medium sized pieces, up to several square feet of surface area, were painted. Occasionally, large pieces such as portions of airplane landing gear or the inner assembly of helicopter blade connectors (not the blades themselves) were spray painted. The small pieces usually arrived

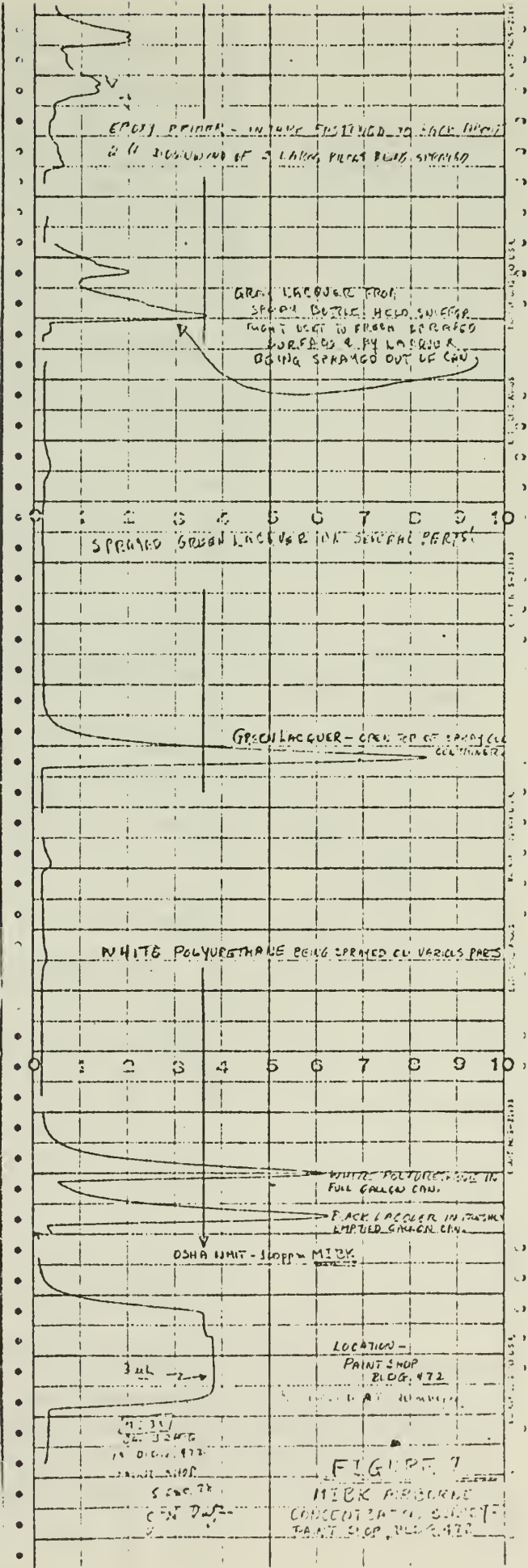
in a "pack" of six or eight with each piece suspended by hooks from a moving overhead conveyor. The hook suspension allowed the spray operator to easily rotate the piece and thus keep the paint spray directed toward the spray wash/ collector walls. The spray operator wore a mask which primarily filtered out paint particulate matter and helped somewhat in reducing the amount of vapors reaching his respiratory system.

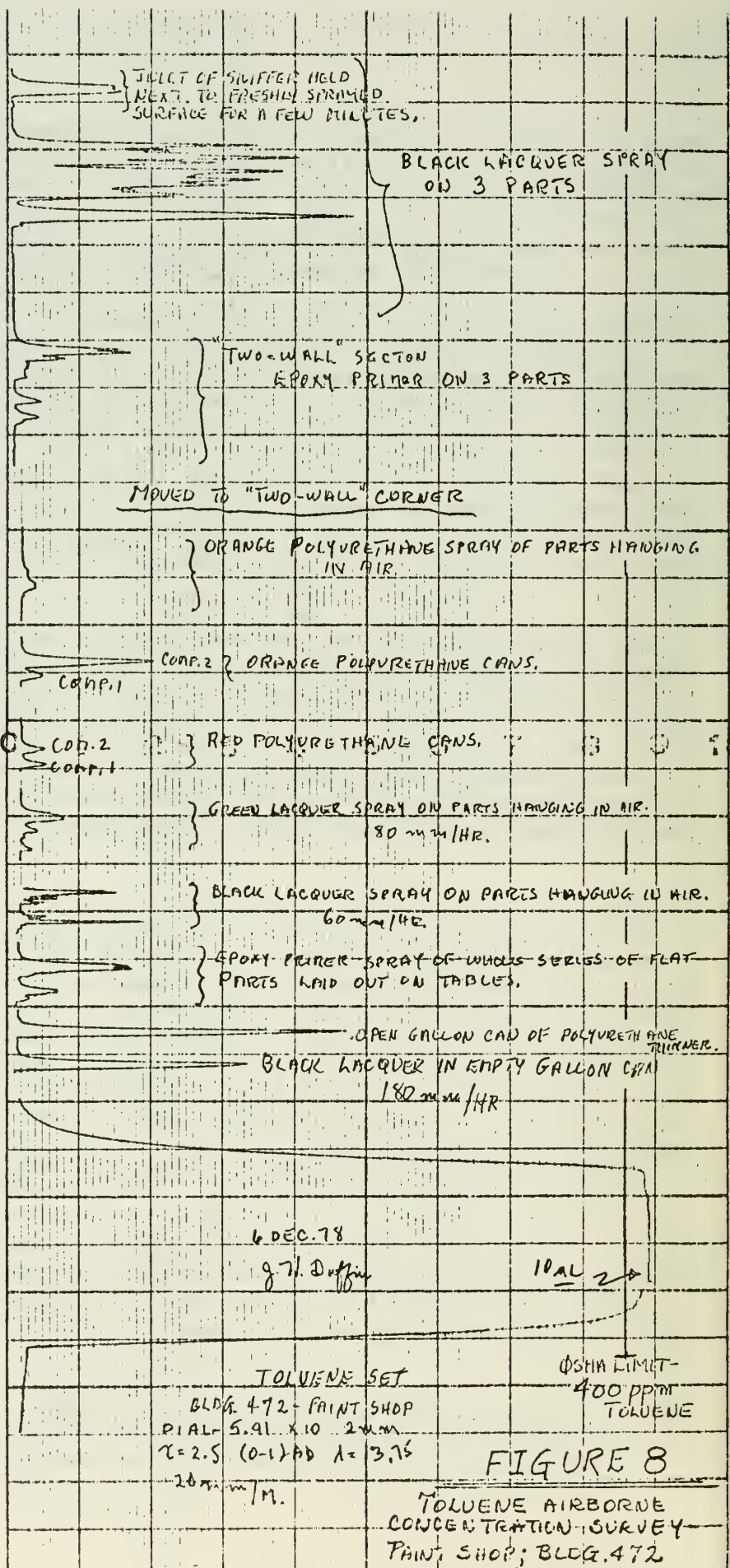
The process of spraying a piece appeared to be highly inefficient in the sense that only a small fraction of atomized paint ever reached a piece surface. The majority of the atomized material was drawn by the exhaust air against a wall down which a film of water cascaded. These were the wash/collector walls. It was imperative that the spray operator always try to spray paint in such a manner that the atomized material was in general directed toward the wall. The operator should avoid situations where he places himself between the atomized spray and the wash walls as under these conditions he would receive the full effect of vapors directly into his face and onto his skin. It was this procedure which was monitored.

The monitoring technique used was one of trying to place the instrument sampler at a point in space which would be indicative of what the spray operator's respiratory system was "seeing" in the way of airborne vapor concentrations. Obviously it was not practical to place the sampler right at an operator's nose. In general, the sampler was placed a foot or two from the operator's face and moved around as the operator moved. Also, after a piece had been spray painted, the sampler would be placed next to the surface to monitor vapor concentration levels during the drying process.

Typical results are indicated in a series of figures. Figure 7 shows results for MIBK, which is an ingredient in many paints and thinners. Various lacquer and polyurethane paint sprayings were monitored. Figure 8 shows results for toluene. This figure also shows results for a special area of the paint shop where one of the wash/collector walls was out of service. In this area, there were two such walls so that one was working and one was not. The pieces sprayed were large pieces which required a lot of spraying activity. Figure 9 shows results for MEK. Figure 10 shows an interesting result. When air movement and water to the walls is shut off, it would be expected the local airborne concentrations of vapors would be high. They were, in fact, high initially, and then tailed off as diffusion occurred. Also shown is the general background concentration level of MEK when everything is shut down during lunch break. It is quite obvious that it went up but was still well within safe limits. Momentary pockets of higher concentration were detected, probably due to many open containers of various solvents lying about. Finally, Figure 11 shows some results for methyl cellosolve acetate ("cellosolve acetate").

What is quite apparent from all these results is that the paint spray operation in the Paint Shop, Building 472 is, for the most part, a safe operation in the sense that vapor concentration levels stay well





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ESTIMATING

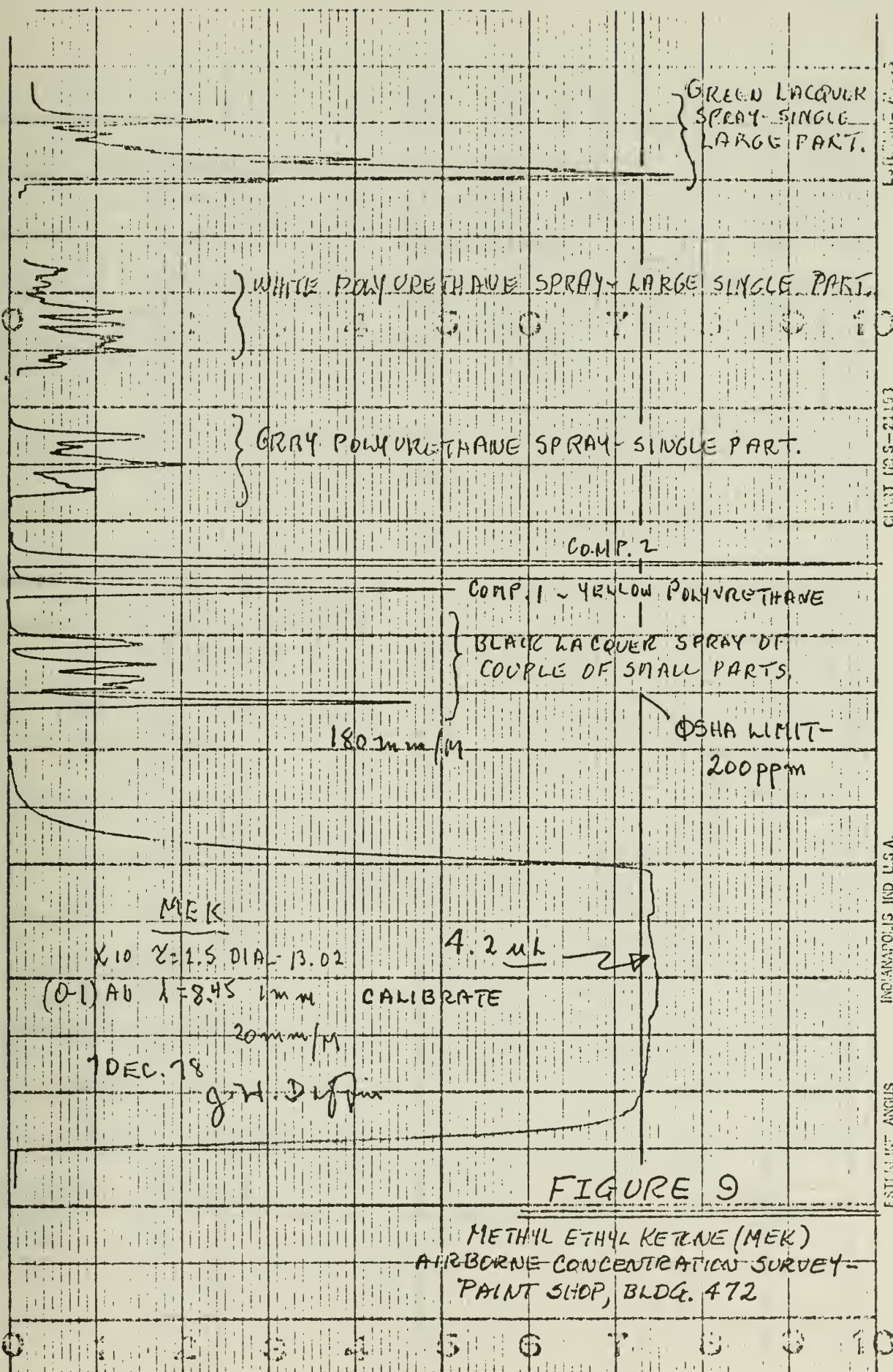


FIGURE 9

METHYL ETHYL KETONE (MEK)
AIRBORNE CONCENTRATION SURVEY -
PAINT SHOP, BLDG. 472

LUNCH PERIOD - ALL AIR/WATER FLOW SHUT
 DOWN. SNIFFER SETTING ON TABLE AT
 CENTER OF PAINT STATION.
 SOMETHING CAUSED MEK PULSE 0 20

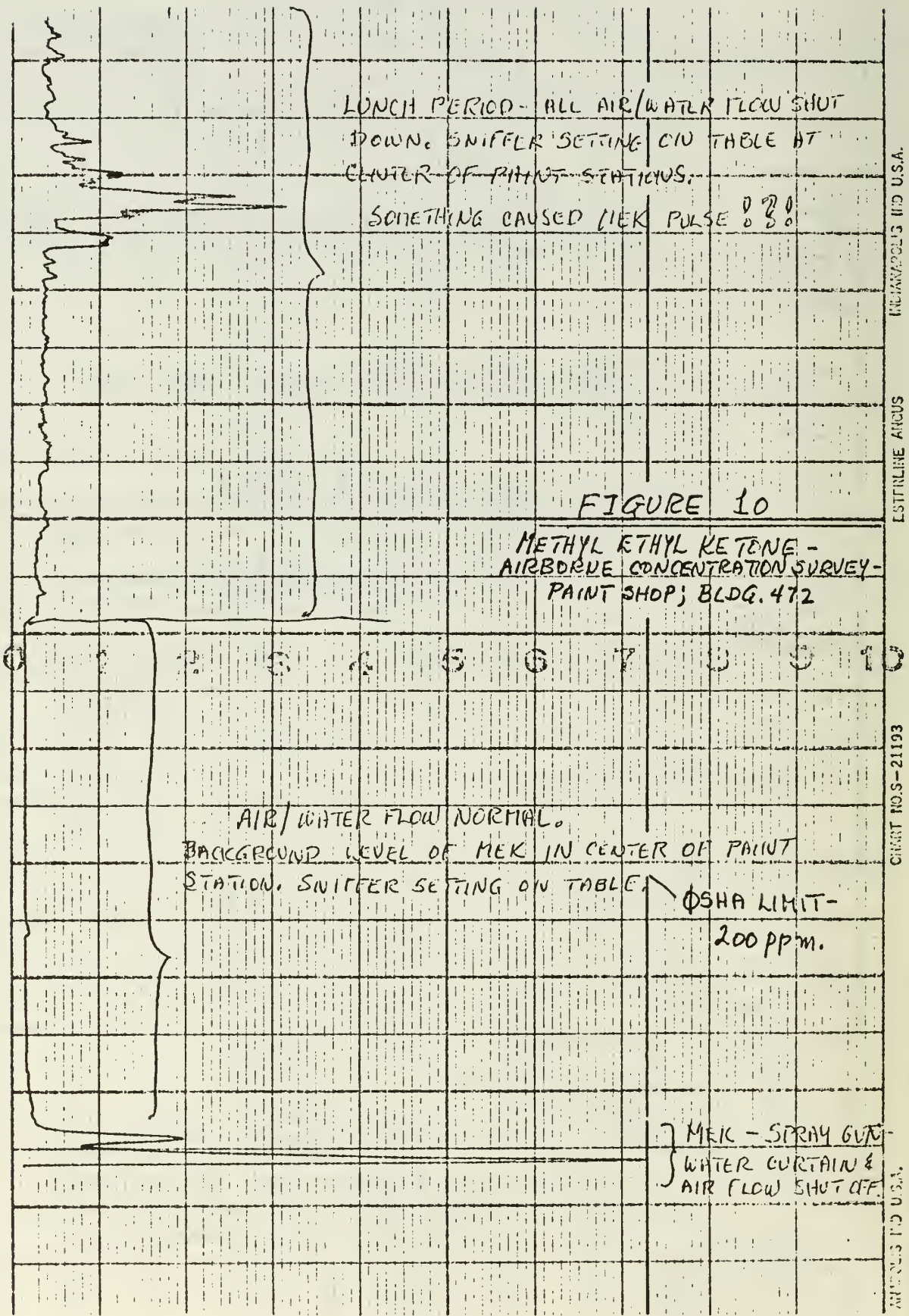
FIGURE 10

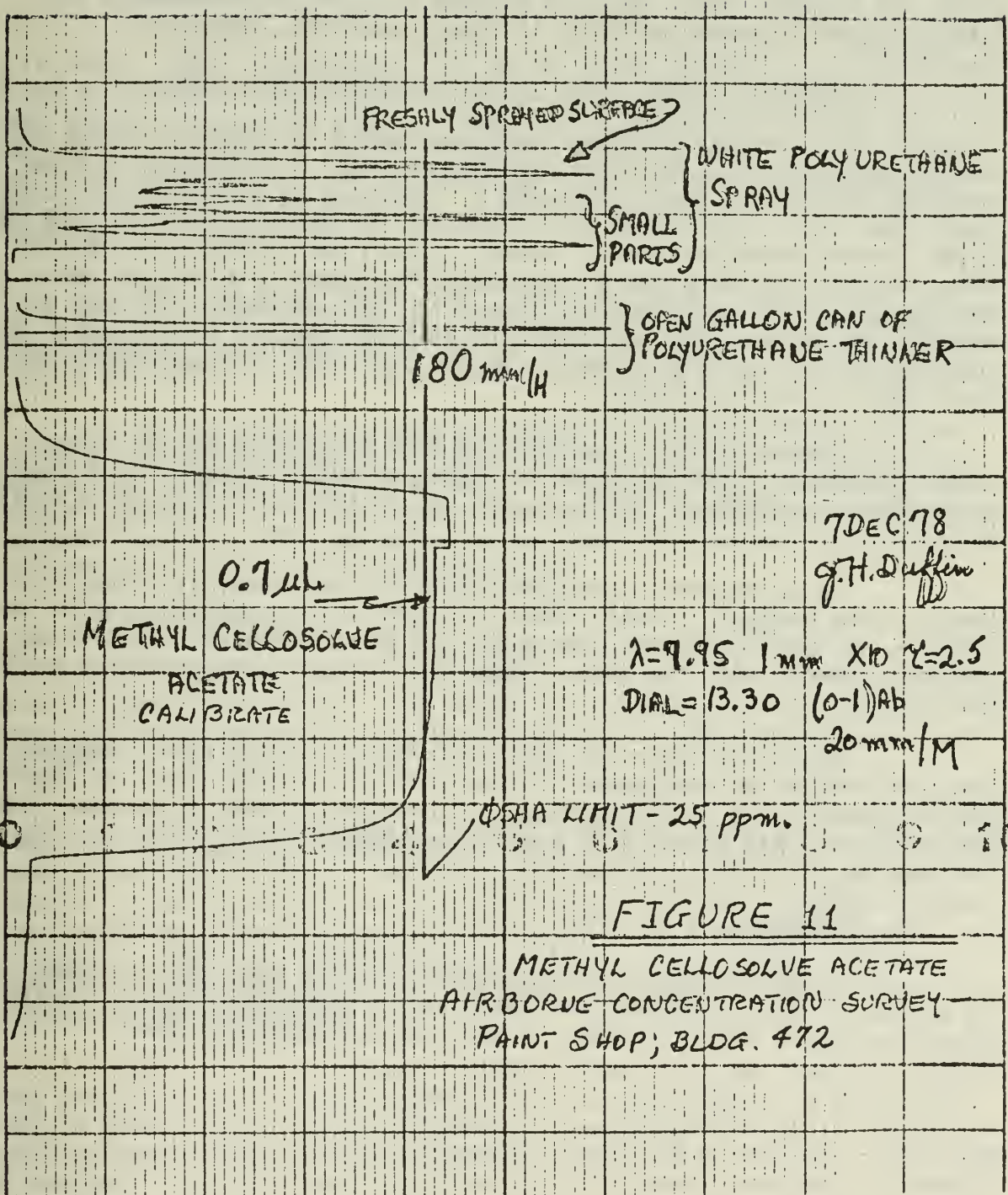
METHYL ETHYL KETONE -
 AIRBORNE CONCENTRATION SURVEY -
 PAINT SHOP; BLDG. 472

AIR/WATER FLOW NORMAL.
 BACKGROUND LEVEL OF MEK IN CENTER OF PAINT
 STATION. SNIFFER SETTING ON TABLE.

OSHA LIMIT -
 200 ppm.

MEK - SPRAY GUN -
 WATER CURTAIN &
 AIR FLOW SHUT OFF.





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CHART NO. S-21193

below OSHA limits. There are isolated peaks which go above limits for short periods of time, but these are measured at two to three feet from the point of human respiration. Concentrations right at the nose/mouth point are probably safe. As long as care is taken and procedures followed, it would appear that this is a well operated, safe area.

C. Chemical Cleaning Shop - Airplane Paint/Strip Shops

The Chemical Cleaning Shop was checked for materials used and operating procedures. The shop contains a number of tanks very similar to those in the Plating Shop (reference 1). Various solutions are in these tanks, but two in particular are candidates for monitoring. One tank contains a water sealed mixture of phenol, cresol, methylene chloride, and other materials. There was a noticeable odor above this tank in spite of the water seal and suction ducts around the tank perimeter. Pieces of material of varying size and shape are lowered into and raised out of this solution using an overhead crane. At the time this dipping process occurs, odor is very noticeable.

A second tank is a degreasing tank containing methyl chloroform. Pieces are lowered in and out using a basket attached to an overhead crane arrangement. Again, vapors in the tank area were quite noticeable.

The paint/strip operation shops involved whole airplanes in large closed hangars. A wide range of materials is used with one in particular, methylene chloride, being used in large amounts. In addition, cresols, phenol, etc. are used. These are all quite objectionable materials.

These shops were not monitored due to lack of time. However, they are both prime candidates for monitoring, and this should be done as soon as possible. The Paint/Strip Shop area was alluded to by many plant personnel as a bad area. This should be checked for the sake of health and morale.

D. Modeling

It was not possible on the basis of this work to do any modeling of airborne vapor concentration levels versus production rates. In the shops monitored, the difficulty is defining a production rate.

VII. CONCLUSION AND RECOMMENDATIONS

A. Conclusions

The Foxboro/Wilks Portable Infrared Gas Analyzer (MIRAN-IA) is a reliable, useful instrument. It can be taken anywhere in a plant operation to monitor for any material for which it can be calibrated. It has been successfully calibrated for about fifty materials, most of which are used in varying amounts at NAVAIREWORKFAC North Island. It can be used to follow concentrations of vapors from such processes as bonding, baking, paint application/stripping, cleaning, plating, etc. which occur throughout the plant area.

Modeling of airborne vapor concentrations versus production rate is a desirable thing to do. Even though this was not successfully done in the work reported here, the concept has value. The biggest difficulty is defining what is a production rate in a given operating area.

The mechanical and optical aspects of MIRAN-IA operation are not difficult to maintain and change. The changing of the cell optical windows was done quite easily as was the changing of the preamplifier/detector assembly. The new windows and detector combination noticeably improved the instrument operation in the sense of less noisy, faster response to detected vapor concentrations.

B. Recommendations

The modeling effort should be continued. The only difficulty is defining production rates. This might best be done with the help of plant production personnel who have knowledge on what they consider production rates to be.

The use of the MIRAN-IA in other operating areas of NAVAIREWORKFAC North Island should be investigated. Over a period of two years the Helicopter Blade Bonding Shop, the Electroplating Shop, Paint Shops, and Chemical Cleaning Shops have been investigated for vapor concentrations. There are other shops and areas that possibly could be investigated. It is possible that other materials are used (i.e., other than the ones reported as being calibrated in this work) and should be investigated for calibration and monitoring.

Work in the Airplane Paint/Strip Shops and the Chemical Cleaning Shop (Building 472) only proceeded as far as identifying the materials used in these areas. Operating procedures were observed to try to get some preliminary idea of what production consisted of and how a rate might be defined. No actual monitoring was done in these areas, but it should be done there.

Finally, a continuing education effort should be made so that AESO personnel learn more about the principles of operation of the MIRAN-IA, its maintenance and possible updating, and its application. In

this way, a more intelligent and wide-spread use of the analyzer can be made. Foxboro Analytical is considering the presentation of a short course. They should be contacted periodically to try to get them to present such a course. Failing this, the following person can give such a course:

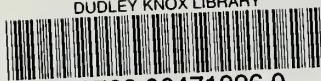
Donald S. Lavery, Ph.D.
Consultant in Analytical Chemistry
13 Betts Place
East Norwalk, CT 06855

Whether it is Foxboro Analytical or Dr. Lavery, a one to three day short course on the MIRAN-IA and infrared monitoring in general is highly recommended.

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