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DETERMINATION OF POSSIBLE INTERFERENT COMPOUNDS TO THE DETECTION AND MONITORING SYSTEM FOR THE QL PRODUCTION FACILITY

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July 1989

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Based on the list of chemicals at the QL production facility, the potential interferents have been identical. The use of Fourier transform, infrared vapor-phase, spectrum library searches, and the analysis of compounds not found in the library have resulted in the identification of compounds that could trigger a false alarm for the MIRAN detection system. All phosphorus compounds provide a positive response, triggering the alarm on the PA260 phosphorus analyzer. Keywords: Bin[illegible] Chem[illegible]

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PREFACE

The work described in this report was authorized under Project No. QPJM05400, Bigeye Fill/Close Monitoring System. This work was started in May 1988 and completed in October 1988.

The use of trade names or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

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DETERMINATION OF POSSIBLE INTERFERENT COMPOUNDS TO THE DETECTION AND MONITORING SYSTEM FOR THE QL PRODUCTION FACILITY

1. INTRODUCTION

The proposed detection and monitoring system for the Bigeye QL* production facility consists of using the MIRAN/1B and MIRAN 80 infrared (IR) analyzers supplemented by the Hydrogen Flame Emission Detector (HYFED) PA260 phosphorus analyzer. Basically, the IR analyzers would be calibrated at selected wavelengths to detect the leakage of compounds at designated sites throughout the facility. At these wavelengths, the target compounds QL, KB, and YL would be detected. However, other compounds used at the facility may absorb at some of these selected wavelengths. These interferent compounds must be identified to minimize the frequency of false alarms of the detection system. The PA260 analyzer would be used to detect only the organophosphates, QL, and YL. Although this analyzer responds to all phosphorus compounds, it will be used in conjunction with the MIRAN analyzers to provide a complete detection system.

The selection of the monitoring wavelengths is extremely critical in achieving a monitoring system with reliable detection capabilities for target compounds while minimizing false responses due to interferent compounds. Therefore, it would be necessary to obtain an IR spectrum of all of the compounds (identified in Table 1) used in the QL production process. The commpounds used in the QL production process would also include any construction materials (i.e., paints, primers, cleaning solvents, etc.) that are used in fabricating the munition and shipping containers. A list of these compounds is shown in Table 1. The resulting spectra would then be compared with the monitoring wavelengths to determine the degree of interference.

2. EXPERIMENTAL PROCEDURES

Whenever possible, the IR spectrum of a compound was obtained through commercially available or in-house libraries. The spectrum for compounds that do not exist in any library were obtained through analysis on a DIGILAB Model FTS-20-E/D Fourier Transform Infrared Spectrometer (FTIR) interfaced to a Hewlett-Packard Model 5880 Gas Chromatograph. A fused silica megabore column (30 m by 0.53 mm) with a 1.5-μm thick liquid phase of 95% dimethyl-(5%) diphenylpolysiloxane was used in all separations.

Compounds were analyzed neat whenever possible. Some samples such as paints, primers, solvents, and the degradation products of QL samples were analyzed as shown in the following sections.

2.1 Paints and Primers.

Approximately 4 mL of paint or primer sample was placed in a test tube and centrifuged at 2500 rpm for 1.5 hr to remove pigments. The supernatent was removed and analyzed (neat or mixed 50:50 with acetone) under the following conditions:

*All chemical names are identified in Table 1.

- Temperature Program: 50 °C for 2.5 min, then 8 °C/min to 240 °C

- Carrier Type: Helium at 8 cm^3/min

- Sample Size: 0.7 µL

2.2 Solvents.

Both solvents (Nature Sol 100 and Nature Sol Emulsion) were analyzed neat under the following conditions:

- Temperature Program: 120 °C Isothermal

- Carrier Type: Helium at 10 cm^3/min

- Sample Size: 1 µL

2.3 Degradation Products in QL Sample.

A degraded QL sample was analyzed neat under the following conditions.

- Temperature Program: 80 °C for 1 min, then 12 °C/min to 240 °C

- Carrier Type: Helium at 10 cm^3/min

- Sample Size: 1 µL

For all analyses, the temperature of the light pipe was maintained at 250 °C; light pipe make-up flow was 6 cm^3/min, and light pipe vent was 0.5 cm^3/min.

Operation of the PA260 was reported earlier.*

3. RESULTS AND DISCUSSION

The target compounds to be monitored at the QL production facility are QL, YL, and KB. Table 2 lists the preliminary monitoring wavelengths for these compounds on the MIRAN analyzers. The IR spectrum of every compound used in the QL production process was examined for IR absorption at these particular wavelengths, and the results are summarized in Table 3. Many of the compounds tested show IR absorbance at one of the selected wavelengths. Seven compounds (TEP, TEPO, HAC, LT, MD, MPA, and trisodium phosphate) absorbed at two of the wavelegths.

Some of the chemicals used in the QL process, which are not commercially available, are still in the process of being obtained. Many of these compounds structures are very similar to compounds in which IR spectra have already been obtained. For example, MR, MP, and ML are identical in structure to TR, TEP, and QL, respectively, except for the presence of a methoxy group

*Ferguson R.E., Ellzy, M.W., Lovrich, J., Stozzle, T., Janes, L.G., and Lindsay, T.J., U.S. Army Chemical Research, Development and Engineering Center, July 1988, unpublished data.

(OCH_3) instead of an ethoxy group (OC_2H_5). Similarly, TRX is identical to TRO in structure except TRX has an ethyl group instead of an ethoxy group. Such small differences in structure may not effect the IR spectrum enough to warrant their synthesis. It may be possible to determine their interference from already obtained spectra. In addition, the possible interference of some of the hyphenated compounds, MR-YL for example, may be inferred from the spectra of their individual componets (MR and YL). Each of these compounds is being examined for properties and possible methods of synthesis due to its relationship to the other compounds investigated.

Table 4 lists the basic components of the paints, primers, and solvents used at the QL production facility and their possible interference at the selected wavelengths.

3.1 Paints and Primers.

Many of the individual components found in the paint and primer samples interfered at one of the selected wavelenghts. Acetone and methyl ethyl ketone absorbed at two of the selected wavelengths. Both of these compounds have high vapor pressures and are typically found in high concentrations in many paints and primers. Some of the minor components in Primer TT-P-664 could not be positively identified but showed no absorption in the areas of interest. In addition, many compounds with halogen moiety (i.e., methylene chloride) or substituted benzene compound (toluene or xylene) may interfere at the QL wavelength.

3.2 Solvents.

There were only two major components found in the solvents, dipropylene glycol methyl ether and limonene. One solvent seems to be a watered down version of the other. The ether absorbed at the KB wavelength. Limonene, which gives the solvents their citrus odor, did not absorb at any of the wavelengths.

Any compound that absorbed at any of the selected wavelengths could be a possible interferent to the detection system. However, the compound's concentration and strength of IR absorbance at the selected wavelength will greatly determine whether the presence of the compound will initiate a false alarm. The MIRAN analyzer's MDL is, on the average, 0.5 ppm and will respond to any compound that absorbs IR radiation at a given wavelength. Because of this, a test of all of the possible interferences on the MIRAN analyzer may not be useful.

The PA260 phosphorus analyzer will respond to any phosphorus-containing compounds used at the QL facility in concentrations down to 0.5 ppb or less. In theory, the PA260 response should be directly related to the amount of phosphorus present in the molecule. The response should be twice as much for a compound with two phosphorus atoms than for a compound with one phosphorus atom at the same concentration. However, there are small differences in the PA260 response between different organophosphates with the same number of phosphorus atoms per molecule. For example, the Figure shows the PA260 response to QL, YL, TEPO, and TR. The response is slightly different for each compound tested, resulting in a family of linear calibration curves. However, at any given response to the PA260, equivalent concentrations for

QL, YL, TEPO, and TR could easily be determined from such a group of calibration curves. Therefore, a PA260 response of 5 x 10^{-9} amps would result in concentrations of 0.028 mg/m^3, 0.140 m/m^3, 0.024 mg/m^3, and 0.008 mg/m^3 for QL, YL, TEPO, and TR, respectively.

4. CONCLUSIONS

Over 88% of the compounds in the QL production process have been analyzed for IR absorption at the selected wavelengths. The remaining compounds are in the process of synthesis or being obtained through commercial sources. All construction materials (i.e., paints, primer, and solvents) have been analyzed. Many of the compounds absorbed at one or two of the selected wavelengths.

The PA260 analyzer will respond to any phosphorus compound at a concentration level of 0.5 ppb or better. Some differences have been observed in calibration curves when challenging the PA260 with QL, YL, TEPO, and TR. Equivalent concentrations for these or other organophosphate compounds can be determined for any given response of an individual PA260 analyzer.

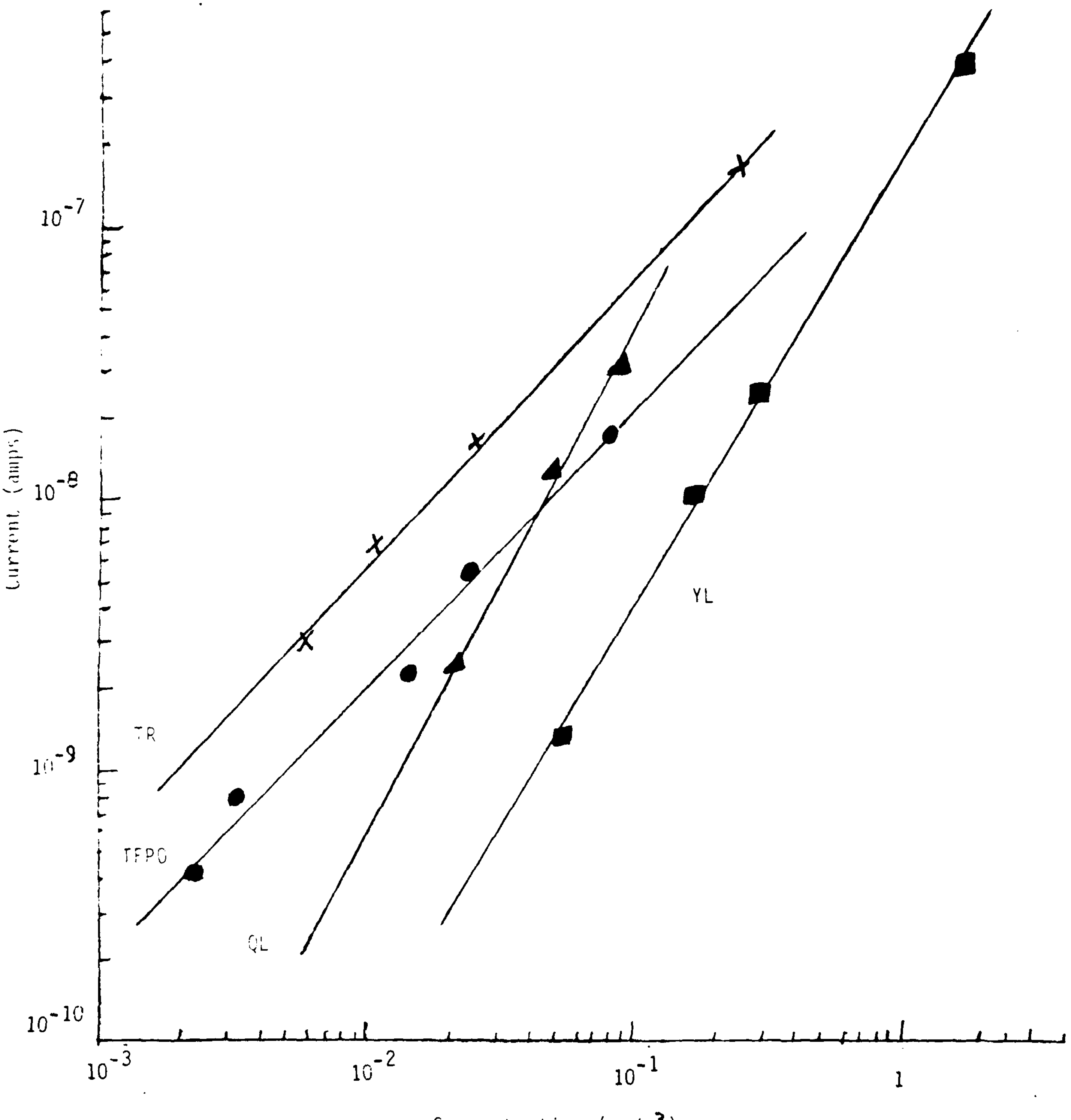

Figure. PA26C Response to QL, YL, TEPO, and TR

Table 1. QL Process Components and Construction Methods

CODE	NAME	FORMULA
NW	Sodium Hydroxide	$NaOH$
TH	Phosphorus Trichloride	PCl_3
SW	Methyl Dichlorophosphite	CH_3PCl_2
MD	Methoxydichlorophosphine	CH_3OPCl_2
XT	Oxygen	O_2
IG	Inert Gas	-
LP	Methane	CH_4
CxHy	Ethane	C_3H_5
ZT	Hydrogen Chloride	HCI
HB	High Boilers	-
BT	Lean Oil	NUJOL
LO	Mineral Oil (Vac pumps)	NUJOL
RM	Isobutane	$(CH_3)_3CH$
ZK	Ammonia	NH_3
ZS	Ethanol	C_2H_5OH
TR	O,O'-Diethylmethylphosphonite	$CH_3P(OC_2H_5)_2$
THO	Phosphorous Oxychloride	$POCl_3$
JO	Chloroethane	C_2H_5Cl
YL	O-Ethylmethylphosphonate	$CH_3P(O)H(OC_2H_5)$
MR	Methylethylmethylphosphonite	$CH_3P(OCH^3)(OC_2H_5)$
MP	Diethylmethylphosphite	$CH_3OP(OC_2H_5)_2$
TEP	Triethylphosphite	$(C_2H_5O)_3P$
RX	Ammonium Chloride	NH_4Cl
TRO	O'O'-diethylmethylphosphonate	$CH_3P(O)(OC_2H_5)_2$
DE	Sodium Chloride	$NaCl$
MPA-NaOP		$CH_3P(O)(H_2)(ONa)$
W	Water	H_2O
TRX,TEPO		$(C_2H_5O)_3P(O)$[TEPO]
MP-YL	Unknown	-
QL	Ethyl-2-diisopropylamino-ethylmethylphosphonite	$CH_3P(OCH_2H_5N(iC_3H_7)_2$
KB	2-Diisopropylaminoethanol	$(iC_3H_7)_2NC_2H_4OH$

Table 1. QL Process Components and Construction Methods (continued)

CODE	NAME	FORMULA
KX	2-Diisopropylamine	$(iC_3H_7)_2NH$
HAc	Acetic Acid	CH_3COOH
LT	Bis(2-Diisopropylaminoethyl) methylphosphonite	$CH_3P(OC_2H_4N(iC_3H_7)_2)_2$
QB	O'-O'-Ethyl-2-diisopropyl-aminoethylmethylphos-phinate	$CH_3P(O)(OC_2H_5)(OC_2H_5N(iC_3H_7)_2)$
QC	O-(2-Diisopropylaminoethyl) methylethylphosphonate	$CH_3P(O)(C_2H_5)(OC_2H_4N(iC_3H_7)_2$
TEPO	Triethylphosphate	$(C_2H_5O)_3P(O)$
TRX	O-Ethylmethylethylphosphonate	$CH_3P(O)(C_2H_5)(OC_2H_5)$
ML	O-O-Methyl-2-Diisopropyl aminoethylmethylphos-phonite	$CH_3P(OCH_3)OC_2H_4N(iC_3H_7)_2$
KBA	Diisopropylaminoethylacetate	$(iC_3H_7)_2NC_2H_4OC(O)CH_3$
QE	Unknown	
QA	O-(2-Diisopropylaminoethyl)-methylphosphinate	$CH_3P(O)(H)(OC_2H_4N(iC_3H_7)_2)$
QD	Bis(2-Diisopropylaminoethyl)-ether	$(iC_3H_7)_2NC_2H_4OC_2H_4N(iC_3H_7)_2$
-	Methyl Ethyl Phosphinic Acid	$CH_3P(O)(OH)(OC_2H_5)$
MPA	Methyl Phosphinic Acid	$CH_3P(O)(H)(OH)$
-	Acetic Acid Sodium Salt	CH_3COONa
-	-	$HP(O)(ONa)_2$
-	Trisodium Phosphate	Na_3PO_4
-	Sodium Carbonate	Na_2CO_3
WF	Carbon Dioxide	CO_2
-	Oxides of Nitrogen	NOx
RP	Nitrogen	N_2
MR-YL	Unknown	-
-	Freon 22	$ClCHF_2$
-	Carbon Monoxide	CO
-	Vehicular Exhaust	P_2O_5
-	Cigarette Smoke	
-	Ethylene Glycol	$OHCH_2CH_2OH$

Table 1. QL Process Components and Construction Methods (continued)

CODE	NAME	FORMULA
Paints	(1) Label: Trans Officer MIL-L-81352 #17875 BA. 29419 DA 4/87 12887	Type: Spray Can Mfg: Koppers, Inc. Pittsburgh, PA
	(2) Label: P-860 Laquer L Acrylic Comp L. Spec MIL-L-81352A Gray 36231	Type: 1 gal Mfg: Unknown
	(3) Label: From Material Safety Data Sheet	Type: Spray can Mfg: Spra_on Products Bedford Heights, OH
Primers	(1) Label: Trans. Office TT.P.664 Primer BA 28922 DA 3/87	Type: Spray Can Mfg: Koppers, Inc. Pittsburgh, PA
	(2) Label: P-441 Iron Oxide Primer Spec TT-P-664C Contains Lead	Type: 1 gal can Mfg: Unknown
Solvents	(1) Label: Nature Sol 100	Type: 1 gal can Mfg: Brulin & Co. Indianapolis, IN
	(2) Label: Nature Sol Emulsion Lot No. NE007	Type: 1 gal con Mfg: Brulin & Co. Indianapolis, IN

Table 2. Preliminary Monitoring Wavelengths for MIRAN Analyzers

Compound	Wavelength (cm^{-1})
KB	1176 (8.54 μm)
YL	957 (10.45 μm)
QL	758 (13.20 μm)

Table 3. QL Process Components and Possible Wavelength Interferences

		Wavelength (cm^{-1})		
Code	Name	KB(1176)	YL(957)	QL(758)
NW	Sodium Hydroxide	?	?	?
TH	Phosphorus Trichloride		ordered	
SW	Methyldichlorophosphite		in-house	
MD	Methoxydichlorophosphine	X	-	X
XT	Oxygen			
IG	Inert Gas			
LP	Methane	-	-	-
CxHy	Ethane	-	-	-
ZT	Hydrogen Chloride	-	-	-
HB	High Boilers	-	-	-
BT	Lean Oil	-	-	-
LO	Mineral Oil	-	-	-
RM	Isobutane	X	-	-
ZK	Ammonia	-	-	-
ZS	Ethanol	-	-	-
TR	0,0-Diethylmethyl-phosphonite	-	?	X
THO	Phosphorus Oxychloride		ordered	
JO	Chloroethane	-	X	-
YL	Ethylmethylphos-phonate	-	X	-
MR	Methylethylmethyl-phosphonite	Spectra not obtained to date		
MP	Diethylmethylphosphite	Spectra not obtained to date		
TEP	Triethylphosphite	?	X	X
RX	Ammonium Chloride	-	-	-
TRO	0'-0'-Diethylmethyl	-	X	-
DE	Sodium Chloride	-	-	-
MPA-NaOP	Unknown		Not obtained to date	
W	Water	-	-	-
TRX-TEPO	Unknown		Not obtained to date	
HP-YL	Unknown		Not obtained to date	
QL	Ethyl-2-diisopropylamino-ethylmethylphosphonite	-	X	X
KB	2-Diisopropylaminoethanol	X	-	-
KX	2- Diisopropylamine	X	-	-
HAc	2-Acetic Acid	X	X	-
LT	Bis(2-diisopropylamino-ethyl)methylphosphonate	-	X	X
QB	0'0'-Ethyl-2-diisopropyl-aminoethylmethylphosphonate	-	?	-
QC	0-(2-Diisopropylaminoethyl) methylethylphosphonate	-	X	-
TEPO	Triethyl Phosphate	?	X	X
TRX	0-Ethylmethylethylphosphonate		Not obtained to date	
ML	0-0-Methyl-2-Diisopropyl aminoethylmethylphosphonite		Not obtained to date	
KBA	Diisopropylaminoethyl acetate		Not obtained to date	

TABLE 3. QL Process Components and Possible Wavelength Interferences (continued)

		Wavelength (cm^{-1})		
Code	Name	KB(1176)	YL(957)	QL(758)
QE	Unknown	Not obtained to date		
QA	O-(2-Diisopropylaminoethyl) methylphosphinate	-	-	X
QD	Bis(2-Diisopropylaminoethyl) ether	-	-	-
-	Methyl Ethyl Phosphinic Acid	X	?	-
-	Methyl Phosphinic Acid	X	X	?
-	Acetic Acid Sodium Salt	-	-	-
-	$HP(O)(ONC)_2$	Not obtained to date		
-	Trisodium Phosphate	-	X	X
-	Sodium Carbonate	-	-	-
WF	Carbon Dioxide	-	-	-
-	N_2O	-	-	-
-	NO	-	-	-
-	NO_2	-	-	-
RP	Nitrogen	-	-	-
MR.YL	Unknown	Not obtained to date		
	Freon.22	X	-	-
	Helium	-	-	-
	Carbon Monoxide	-	-	-
	Phosphorus Pentoxide		Ordered	
	Ethylene Glycol	?	-	-

Legend: X = interference at wavelength
? = possible interference at wavelength
\- = no interference at wavelength

Table 4. Construction Materials and Possible Interferences

Paint: MIL-L-81352, Spray Can

Compound (% Composition)	KB	YL	QL
Iso-Octane (1.6)	-	-	-
Acetone (44.4)	X	X	-
Methylene Chloride (8 75)	-	-	X
Methyl Ethyl Ketone (15.1)	X	X	-
Acetic Acid, Propyl Ester (5.81)	X	-	-
4-Methyl Pentanone (3.14)	-	-	-
Toluene (7.97)	-	-	X
Isobutyric Acid, isobutylester (0.6)	X	-	-
Ethyl Ethoxypropionate (9.91)	X	-	-

Paint: MIL-L-81352-A, 1 gal

Compound (% Composition)	KB	YL	QL
Methyl Ethyl Ketone (42.75)	X	X	-
1-Butanol (6.06)	?	-	-
Toluene (30.69)	-	-	X
Butyl Acetate (15.35)	X	-	-
Methoxy Butyl Acetate (4.44)	X	-	-
2-Ethoxy Ethyl Acetate (.21)	-	-	-

Paint: ID Code, LA16 SP, spray can

Compound (% Composition)	KB	YL	QL
Methyl Ethyl Ketone (25%)	X	X	-
Acetone (20%)	X	X	-
Ethyl-ethoxypropionate (5%)	X	-	-
Methylene Chloride (20%)	-	-	X
Isobutane (15%)	X	-	-
Propane (15%)	-	-	-

Primer: TT-P-664

Compound (% Composition)	KB	YL	QL
Isooctane (2.04)	-	-	-
Acetone (16.55)	X	X	-
Methylene Chloride (8.09)	-	-	X
Methyl Ethyl Ketone (30.97)	X	X	-
Isobutanol (3.68)	-	-	-
1-Butanol (2.81)	-	-	-
Aliphatic Hydrocarbon (0.56)	-	-	-
Aliphatic Hydrocarbon (<.1)	-	-	-
Toluene (6.34)	-	-	X
Octane or Heptane (1.92)	-	-	-
Aliphatic H.C.(5.13)	-	-	-
Aliphatic Hydrocarbon (1.82)	-	-	-
Aliphatic Hydrocarbon (1.18)	-	-	-
Aliphatic Hydrocarbon (3.16)	-	-	-
Aliphatic Hydrocarbon (2.14)	-	-	-
Aliphatic Hydrocarbon (<.1)	-	-	-

Table 4. Construction Materials and Possible Interferences (continued)

Compound (% Composition)	KB	YL	QL
Isobutyric Acid (Isobutyl ester) (1.80)	X	-	-
Ethyl Etoxy Propionate (4.58)	X	-	-
Phthalic Acid Benzy Ester (.1)	-	X	-
Primer: TTP664.C			
Methyl Ethyl Ketone	X	X	-
Methyl Propyl Ketone	-	-	-
Toluene	-	-	X
M-Xylene	-	-	-
O-Xylene	?	-	X
Phthalic Acid Butyl Ester	?	X	-
Solvent: Nature Scl 100			
Dipropylene Glycol Methyl Ester (2.5)	X	-	-
Limonene (96.8)	-	-	-
Solvent: Nature Sol Emulsion			
Water (>50%)	-	-	-
Dipropylene Glycol Methyl	X	-	-
Este (20%)	-	-	-
Limonene	-	-	-

Legend: X = interference at wavelength
? = possible interference at wavelength
\- = no interference at wavelength