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Study of Contamination Sensors

Volume I: Executive Summary Report

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STUDY OF CONTAMINATION SENSORS

VOLUME I: EXECUTIVE SUMMARY REPORT

April 1966

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FOREWORD

The Martin Company submits this final report of the contamination sensors study, as required by paragraph III.B.4, Contract NAS10-2693. This report is submitted in two volumes:

Volume I - Executive Summary Report;

Volume II - Final Technical Report.

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I. INTRODUCTION

The purpose of this study is to provide the required information to evaluate and develop automatic contamination sensors and monitors capable of being installed in a fluid distribution system and remotely indicating particle-contamination count, amount of moisture present, purity of the fluid, or a combination of these. The study also provides the required information for the development of a mechanical sampling procedure and technique, and to some extent, the equipment necessary to perform this sampling.

Specific objectives are to:

- 1) Establish design criteria for automatic remotely-indicating fluid contamination sensors, monitors, counters, and recorders based on the requirements of the facilities at Launch Complexes 34, 37, and 39, Kennedy Space Center (KSC);
- 2) Provide data and information from launch facilities, other centers, and manufacturers required for the evaluation and development of such equipment;
- 3) Evaluate existing equipment (without resorting to hardware testing) and make specific recommendations as to the suitability of each and where future development efforts might best be expended;
- 4) Investigate possible use of other tried and untried development methods for this equipment;
- 5) Establish design criteria for sampling equipment, procedures, and techniques based on the fluid-sampling requirements at Launch Complexes 34, 37, and 39;
- 6) Investigate sampling techniques at these facilities and, by surveying NASA prime contractors and other manufacturers, determine the techniques providing the optimum results, compare these techniques with those in use at KSC, and make recommendations for improvement.

SAMPLING EQUIPMENT AND

TEST SITES PARAMETERS SAMPLED FOR	DAC-VAFB	MMC-VAFB	LAC-VAFB	GD/C-VAFB	GE-VAFB	KSC	P&W	A.F. PLANT 74	GD/C-S.D.C.	EAFB-RPL	RD-C.P.C.	RD-S.S.C.	RD-RENO	AGC-SACTO	DAC-R.C.C.
GH ₂ PURITY & PARTICLES	O	O	O	O	O	F ₁ F ₂ H U	O	H ₁ E ₁ X Y	O	O	N	R	O	O	O
RP-1 PURITY	C	O	O	C	O	C ₃	O	O	N	CC ₁	C ₄ L	C ₄ L	O	O	O
RP-1 PARTICLES	C	O	O	C	O	C ₃	O	O	C	CC ₁	C ₄ L	C ₄ L	O	O	O
LOX PURITY	A ₃	O	O	A	O	A	AD	A	A	AB	A	A	O	A	A
LOX PARTICLES	N	O	O	B	O	BC ₂	N	U ₆	K ₁	B	K	K	O	B	B
LN ₂ PURITY	A ₃	O	O	A	O	A	A	A	A	AB	A	A	O	A	A
LN ₂ PARTICLES	N	O	O	B	O	B	N	U ₆	K ₁	B	K	K	O	B	B
LH ₂ PURITY	O	O	O	O	O	A ₁	N	A ₁	N	N	A ₁	A ₁	O	N	N
LH ₂ PARTICLES	O	O	O	O	O	C ₃	N	N	K ₁	N	N	N	O	N	N
GN ₂ PURITY	N	D	D	D	N	N	H ₁ D	H ₁ Z	D	D	DR	DR	N	D	D
GN ₂ HYDROCARBON	T	D	D	D	N	F	D	X ₁	D	D	DR	DR	N	D	T
GN ₂ PARTICLES	U ₃	U ₁	U	U	U	U	U ₅	N	U ₂	U	U	U	U	N	U ₁
GN ₂ MOISTURE	E	D	E	E	E	EE ₁	E ₃	E ₁	EE ₂	E	DR	DR	N	D	E ₁
HELIUM PURITY	D	O	D	D	N	N	D	O	D	D	DR	DR	O	D	D
HELIUM HYDROCARBON	D T	O	D	D	N	F	D	O	D	D	DR	DR	O	D	T
HELIUM MOISTURE	E	O	E	E	E	EE ₁	E ₃	O	EE ₂	EE ₂	DR	DR	O	D	E ₁
HELIUM PARTICLES	U ₃	O	U	U	U	U	N	O	U ₂	U	U	U	O	N	U ₁
GO ₂ PURITY	O	O	O	O	O	D	O	H ₁	D	D	DR	DR	O	O	O
GO ₂ HYDROCARBON	O	O	O	O	O	D	O	X ₁	D	D	DR	DR	O	O	O
GO ₂ MOISTURE	O	O	O	O	O	D	O	E ₁	EE ₂	E	DR	DR	O	O	O
GO ₂ PARTICLES	O	O	O	O	O	U	O	N	U ₂	U	U	U	O	O	O
N ₂ O ₄ PURITY	O	D ₁	O	O	D	D	O	O	O	D	DJ M	DJ M	M	P	C ₄
N ₂ O ₄ PARTICLES	O	D ₁	O	O	D	D	O	O	O	D	DJ M	DJ M	S	P	N
A-50 PURITY	O	D ₁	D*	O	O	C	O	O	O	D	DJ M	DJ M	O	C	O
A-50 PARTICLES	O	D ₁	D*	O	O	C	O	O	O	D	DJ M	DJ M	O	C	O
MMH PURITY	O	O	O	O	D*	C	O	O	O	O	DJ M	DJ M	M	O	C ₄
MMH PARTICLES	O	O	O	O	D*	C	O	O	O	O	DJ M	DJ M	S	O	N

METHODS



LAC-S.C.	LAC-S.C.C.	BOEING-B.O.	BOEING-S.W.	DAC-S.M.C.	NAA-D.C.	JPL-P.C.	LEC-WSTF	GAEC-WSTF	ZIA-WSTF	NAA-WSTF	MSFC	RMD, TCC-DNJ	MMC - DENVER
O	O	O	O	O	O	O	O	O	O	O	N	O	N
O	O	O	O	C	O	O	O	O	O	O	C	O	C
O	O	O	O	C	O	O	O	O	O	O	C	O	C
O	N	A	A	A A ₃	A	O	A G	O	A	A	A B	N	B
O	N	B	B	B	B	O	B	O	U ₃ B	N	N	N	B
O	N	N	A	A A ₃	A	O	A	O	A	A	A B	N	B
O	N	N	B	B	B	O	B	O	U ₃ B	N	N	N	B
O	N	N	A ₁	N	A ₁	O	A ₂	O	A ₂	A ₂	A ₁ B	N	N
Q	N	N	N	N	B	O	N	O	N	N	N	N	N
D	N	N	N	D	D ₃	D	D D ₁	D D ₁	DD ₁	D ₃ D ₁	N	N	D
D	N	N	N	T	D ₃	D	DD ₁	DD ₁	DD ₁	D ₃ D ₁	N	N	D
U	U	N	N	N	N	U	U ₁	W	U ₁	N	U	N	U ₁ U ₇
E ₁	E ₁	N	N	E E ₁	D ₃	E	E ₂	E ₂	E ₂	D ₃ D ₁	N	N	E E ₂
D	N	N	N	D	D ₃	O	DD ₁	DD ₁	DD ₁	D ₃ D ₁	N	N	D
D	N	N	N	D	D ₃	O	DD ₁	DD ₁	DD ₁	D ₃ D ₁	N	N	D
E ₁	E ₁	N	N	D	D ₃	O	E ₂	E ₂	E ₂	D ₃ D ₁	N	N	E E ₂
U	U	N	N	N	N	O	U ₁	W	U ₁	N	N	N	U ₁ U ₇
O	O	O	O	O	D ₃	O	O	O	O	O	N	N	O
O	O	O	O	O	D ₃	O	O	O	O	O	N	N	O
O	O	O	O	O	D ₃	O	O	O	O	O	N	N	O
O	O	O	O	O	N	O	O	O	O	O	N	N	O
O	D*	O	C ₂	C ₄	I	D	D ₁	D ₁	D ₁	D ₁	O	D	D
O	D*	O	C ₂	N	I	D	D ₁	D ₁	D ₁	D ₁	O	D	D
O	C*	O	C ₂	O	I	D	D ₁	D ₁	D ₁	D ₁	O	D	D
O	C*	O	C ₂	O	I	D	D ₁	D ₁	D ₁	D ₁	O	D	D
O	N	O	O	C	I	O	O	O	O	O	O	D	O
O	N	O	O	C	I	O	O	O	O	O	O	D	O

Legend:

- A Cosmodyne Cryogenic Sampler
- A₁ Cosmodyne, Cryogenic Sampler Modified
- A₂ Vacuum-Jacketed Cosmodyne Cryogenic
- A₃ Douglas Cryogenic Sampler
- B Dewar
- C Glass Bottle
- C₁ Pharmaceutical Graduate (200 ml)
- C₂ Erlenmeyer Flask
- C₃ Glass Beaker
- C₄ Teflon Bottle
- D Double-Ended, Double-Valved Bottle
- D₁ Double-Ended, Double-Valved Bottle with Millipore Filter Holder Attached, (O
- D₂ Single-Ended, Single-Valved Bottle
- D₃ K Bottle
- E CEC Moisture Monitor
- E₁ Beckman Electrolytic Hygrometer
- E₂ Alnor Dewpointer
- E₃ Dewpoint Cup
- F Beckman Hydrocarbon Analyzer
- G Cryenco Liquid-to-Gas Vaporizer Sampler
- H Lockwood-McLorie O₂ Analyzer
- H₁ Analytic Systems O₂ Analyzer
- H₂ Beckman Oxygen Analyzer
- I Cosmodyne Storable Fuels Sampler
- J Polyethylene Bomb Sampler with Millipore
- K Remote Cryogenic Dewar Sampler
- K₁ Modified Stanley Bottle
- L Remote RP-1 Sampler
- M NTO or Fuel Panel and Flanged Sample
- N Not Sampled
- O Not Used
- P AGC N₂O₄ Sample Container
- R Flanged Gas Sampler (3000 psi)
- S Glass Millipore Funnel with OH Membrane
- T 1/2-in. Stainless Steel Tube Filled with
- U High-Pressure Millipore Membrane Holder
- U₁ High-Pressure Millipore Membrane Holder
- U₂ High-Pressure Millipore Membrane Holder
- U₃ High-Pressure Millipore Membrane Holder
- U₄ High-Pressure Millipore Membrane Holder
- U₅ Millipore Aerosol Monitor Holder with

for LH ₂ Service sampler	U ₆	High-Pressure Millipore Membrane Holder with a Gelman Type A Glass Fiber Filter
	U ₇	Martin-Designed High Pressure Screen-Filter Holder, with a 50-mesh Screen
	W	Series of 3 High-Pressure Membrane Holders with 0.8 μ Membranes
	X	MSA Infrared Analyzer, Model MG3823
	X ₁	Liston Becker Infrared Analyzer
	Y	Gow-Mac Ortho-Para Meter
	Z	Arnold O. Beckman Oxygen Analyzer (for N ₂)
	*	The commodity that the organization samples is not the true commodity shown. However it is close enough that the sampling procedure used would be applicable to the subject fluid. Example: Lockheed does not use or sample A-50, but they do use and sample UDMH. It is felt that sampling of UDMH is the same as sampling A-50.
with High-Pressure 0.45 μ Membrane).	DAC-VAFB	Douglas Aircraft Co., Vandenberg Air Force Base
	MMC-VAFB	Martin Marietta Corp., Vandenberg Air Force Base
	LAC-VAFB	Lockheed Aircraft Corp., Vandenberg Air Force Base
	GD/C-VAFB	General Dynamics/Convair, Vandenberg Air Force Base
	GE-VAFB	General Electric Corp., Vandenberg Air Force Base
	KSC	Kennedy Space Center
ler	P&W	Pratt and Whitney Aircraft, West Palm Beach, Florida
	A.F. Plant 74	A. F. Plant #74, Stearns - Rogers Corp., West Palm Beach, Florida
	GD/C-S.D.C.	General Dynamics/Convair, San Diego, Calif.
	EAFB-RPL	Edwards Air Force Base Rocket Propulsion Laboratory
pore Holder Attached	RD-C.P.C.	Rocketdyne, Canoga Park, California
	RD-S.S.C.	Rocketdyne, Santa Susana, California
	RD-Reno	Rocketdyne, Reno, Nevada
	AGC-Sacto	Aerojet - General Corp., Sacramento, Calif.
r	DAC-R.C.C.	Douglas Aircraft Corp., Rancho Cordova, Calif.
	LAC-S.C.	Lockheed Aircraft Corp., Sunnyvale, Calif.
	LAC-S.C.C.	Lockheed Aircraft Corp., Santa Cruz, Calif.
	Boeing-B.O.	Boeing Co., Boardman, Oregon
ane	Boeing-S.W.	Boeing Co., Seattle, Washington
	DAC-S.M.C.	Douglas Aircraft Corp, Santa Monica, Calif.
with Crushed Fire Brick	NAA-D.C.	North American Aviation, Downey, California
der with a 0.45 μ	JPL-P.C.	Jet Propulsion Laboratory, Pasadena, Calif.
der with a 0.8 μ	LEC-WSTF	Lockheed Electronics Co., White Sands Test Facility
der with a 1.2 μ	GAEC-WSTF	Grumman Aircraft Engineering Corp., White Sands Test Facility
der with a 5.0 μ	ZIA-WSTF	Zia Company, White Sands Test Facility
der with a 10.0 μ	NAA-WSTF	North American Aviation, White Sands Test Facility
h a 0.8 μ Membrane	MSFC	Marshall Space Flight Center
	RMD,TCC-D.N.J.	Reaction Motors Div, Thiokol Chemical Corp., Denville, New Jersey
	MMC-Denver	Martin-Marietta Corporation, Denver Division, Denver, Colorado

Fig. 1 Sampling Equipment and Methods

The second portion of the survey was devoted to visiting manufacturers of instrumentation and sampling equipment to obtain complete information concerning the instrumentation and sampling equipment readily available, possible modifications to existing equipment, and conceptual ideas. Thirty-five such organizations were visited. The equipment and philosophies of these organizations are believed to present the most complete composite available at the present time.

III. SUMMARY OF DETERMINATION OF APPLICABLE CONTAMINATION CONTROL CRITERIA

A. PURITY/IMPURITY CRITERIA

Table 1 outlines the fluid purity criteria for the fluids of interest and is a compilation of the following specifications:

- 1) MSFC-SPEC-234 - Nitrogen; Space Vehicle Grade (Type I Gaseous, Type II, Liquid);
- 2) MSFC-SPEC-364A - Helium;
- 3) MSFC-SPEC-399 - Oxygen (Type I Gaseous, Type II, Liquid);
- 4) MSFC-SPEC-356 - Hydrogen, Liquid;
- 5) MIL-P-27402 (USAF) - Hydrazine-uns-Dimethylhydrazine (50% N_2H_4 - 50% UDMH);*
- 6) MIL-P-27404 - Monomethylhydrazine;
- 7) MIL-P-26539A - Nitrogen Tetroxide;
- 8) MSFC-SPEC-342 - Rocket Fuel, RP-1.

B. PARTICLE CRITERIA

The particle criteria listed in Table 2 were extracted from Appendix A, Media Specifications, from MSFC DWG 13M20097 for the Saturn IB, and from Appendix A, Media Specifications, from MSFC DWG 13M50099 for the Saturn V. These criteria are the most complete criteria available for this program. Correspondence from Propulsion Division, Propulsion and Vehicle Engineering Laboratory, MSFC, dated 9 March 1966, indicates that additional particle criteria are forthcoming for all Saturn fluids and should be completed by 1 June 1966. It is not anticipated that these criteria will include parameters that cannot be sampled or monitored by the methods offered in this report.

*Aerzine-50 (Abbreviated A-50 in this report).

Table 1 Fluid Purity Criteria

Constituent	Fluid										
	Gaseous Nitrogen	Helium	Gaseous Oxygen (Grade C)	Gaseous Hydrogen	Liquid N ₂ , O ₂ , H ₂ as gases	A-50	POH	N ₂ O ₂	RP-1	Gaseous Oxygen (Grade A)	Gaseous Oxygen (Grade B)
Oxygen	50 ppm	-	99.60%	-	"	-	-	-	-	99.99%	99.6%
Total Hydrocarbons	5 ppm as methane	5 ppm as propane	50 ppm as methane	9 ppm Total max	"	-	-	-	-	14.0 ppm as methane	29.0 ppm as methane
Moisture Content	11.5 ppm	9 ppm	3 ppm								
Nitrogen	99.99%	14 ppm	-	99.99%	"	-	-	-	-	3.0 ppm	3.0 ppm
Hydrogen	-	1 ppm	-	-	"	-	-	99.5% by weight min	-	-	-
N ₂ O ₂	-	-	-	-	-	-	-	-	-	-	-
N ₂ O	-	-	-	-	"	-	-	0.08% by weight	-	1.0 ppm	1.0 ppm
Chloride (as NOC-)	-	-	-	-	-	-	-	-	-	-	-
Helium	-	99.99%	-	39 ppm	"	-	-	-	-	-	-
O ₂ + Argon	-	1 ppm	-	1 ppm	"	-	-	-	-	-	-
Total Impurities	100 ppm	50 ppm	-	50 ppm	"	-	-	-	-	-	-
Carbon-Bearing Gases	-	-	-	1 ppm	"	-	-	-	-	1.0 ppm	5.0 ppm CO 5.0 ppm CO ₂
Hydrazine	-	-	-	-	-	51.0, ± 0.8% by weight	-	-	-	-	-
UDMH + Amine	-	-	-	-	-	47% min by weight	-	-	-	-	-
Monomethyl Hydrazine	-	-	-	-	-	-	98% min by weight	-	-	-	-
Existing Gum	-	-	-	-	-	-	-	-	7 mg/100 ml	-	-
Potential Gum	-	-	-	-	-	-	-	-	14 mg/100 ml	-	-
Sulphur	-	-	-	-	-	-	-	-	0.05% by weight	-	-
Mercaptan-Sulphur	-	-	-	-	-	-	-	-	0.005% by weight	-	-
Aromatics	-	-	-	-	-	-	-	-	5.0%	-	-
Olefins	-	-	-	-	-	-	-	-	1.0%	-	-
Methane, Ethane	-	-	-	-	"	-	-	-	-	10 ppm methane 2 ppm ethane	25.0 ppm methane 2.0 ppm ethane
Propane and Higher Hydrocarbons	-	-	-	-	"	-	-	-	-	1 ppm	1 ppm
Alkyne and Acetylene Hydrocarbons	-	-	0.05 ppm as acetylene	-	"	-	-	-	-	0.05 ppm as acetylene	0.05 ppm as acetylene
Halogenated Hydrocarbons	-	-	-	-	"	-	-	-	-	1.0 ppm	1.0 ppm
All Other Impurities	-	-	-	-	"	-	-	-	-	30 ppm	-

Notes: 1. Concentrations are based on volume except where indicated.
2. Purity values are minimums; impurity values are maximums.

Note: 1. Concentrations are based on volume except where indicated.

2. Purity values are minimums; impurity values are maximums.

Table 2 Particle and TFS Criteria for Saturn IB and Saturn V

Fluid	Basic Specification	Total Filterable Solids Maximum	Solid Particles Maximum Allowable		Fibers Maximum Allowable	
			Size (μ)	Number	Size (μ)	Number
LO ₂	MSFC-SPEC-399	1.0 mg/liter	<175 >175	No Limit None	<175 >175	No Limit None
LH ₂	MSFC-SPEC-356	**	<175 >175	No Limit None	<175 >175	No Limit None
LN ₂	MSFC-SPEC-234	**	**	**	**	**
MMH	MIL-P-27404	1.0 mg/liter (MIL-SPEC)	**	**	**	**
N ₂ O ₄	MIL-P-26539A, Amendment 1	10.0 mg/liter (MIL-SPEC)	**	**	**	**
A-50	MIL-P-27402, Amendment 1	10.0 mg/liter (MIL-SPEC)	**	**	**	**
GO ₂ GN ₂	MSFC-SPEC-399 MSFC-SPEC-234	**	**	**	**	**
He	MSFC-SPEC-364A	0.01 mg/liter	<20 >20	No Limit None	<20 >20	No Limit None
CH ₂	MSFC-SPEC-356	**	**	**	**	**
RP-1	MSFC-SPEC-342	**	10 to 25 26 to 50 51 to 100 >100 & Fibers*	6500 1600 300 40		

*A fiber is defined as any particle measuring over 100 μ in length and having a length-to-width ratio of 10 to 1, or higher; **No limit established.

IV. PHILOSOPHY FOR ESTABLISHING SAMPLING AND MONITORING POINTS

The following philosophy was used to establish sampling and monitoring points:

- 1) Procurement sampling, performed on the operational fluid while in the vendor container at the point of delivery, or while in a bonded storage tank prior to delivery, will be considered adequate to establish the acceptability of the fluid for introduction into the NASA system;
- 2) All operational fluid sampling performed on the NASA system, therefore, will be designed to detect primarily those contaminants that are a degradation product of the operational fluid or which are system-generated both by proper and improper control of system conditions;
- 3) No sampling of the operational system upstream of the storage tanks is recommended unless the operational fluid is uniquely sensitive to contaminants that cannot be completely kept from the system (e.g., atmospheric gases in an LH_2 system);
- 4) Liquid storage tanks will be sampled only while the contents are agitated by a recirculating sampling stream. The recommendations, therefore, provide for pump-operated recirculation loops for each liquid storage tank that does not have one as part of its operational configuration. As an alternative, tank contents can be agitated by bubbling the blanketing gas through them. A dynamic sample can then be taken after homogenization;
- 5) In general, two principal monitoring points are recommended in each main fluid line downstream of the storage containers, one at the outlet into the transfer system (but downstream of storage-area filters) and the second as near to the airborne interface as is practical;
- 6) In addition to the principal monitoring points, a number of sampling points are necessary. These sampling points are not intended for continuous

monitoring, but rather for taking fluid samples before actual booster loading. These points would be used for checking system cleanliness and dryness during blowdown of the system or during other exercises in which sampling would be indicated;

- 7) The recommended monitoring points are based on the ultimate in reliability and on the assumption that some points will be sufficiently close that one analyzer will be shared by more than one monitoring point. Based on this concept, it is possible that some sampling points might actually become monitoring points in the ultimate design. The final selection of the number and exact locations of monitoring points will depend on many factors, among which are the following:
 - a) Relative costs of the possible combinations,
 - b) Weight,
 - c) Available locations and proximity to one another,
 - d) Minimum modification to system,
 - e) Length of sampling lines required,
 - f) Length of time for switching,
 - g) Ability to use flow sampling wedges for multiple flow rates and two-directional flow.

V. RECOMMENDATIONS FOR SAMPLING AND MONITORING

A. SAMPLING

In many instances the final design criteria and specific equipment recommendations resulting from this study are equivalent to or identical to existing or planned methods, separate from this study, for the same fluids, systems and complexes. Mechanical sampling for purity parameters (as distinguished from particulate sampling) as it exists or is planned for these facilities cannot practicably be improved on for the majority of these fluids, especially for the noncryogenics.

In the case of RP-1, a one-quart, glass bottle with screw-on-type bakelite cap and conical polyethylene seal is recommended. For the cryogenic liquids a Cosmodyne Cryogenic Sampler is recommended. For N_2O_4 , A-50, MMH, and the gases, a Hoke double-valved stainless steel pressure bomb is recommended. For determining moisture content in gases, an electrolytic-type moisture indicator attached directly to the system, is recommended. Table 3 summarizes present practice and recommendations for sampling for purity-impurity parameters.

For particle sampling, design criteria for conceptual equipment and methods are presented to provide for:

- 1) Performing all filtrations for particle count criteria at the site of sampling;
- 2) Precounting the membrane after the sampling tool is connected to the sampling point and an initial operational fluid flow has occurred through it;
- 3) Performing the sample flow and particle count without disturbing or transporting the sampling device between these operations;
- 4) Taking other precautions that will minimize or account for the contribution to the sample from sources other than the operational fluid. This represents a significant departure from previous technology.

Table 3 Purity-Impurity Sampling Summary

Fluid	Present Practice	Recommendation	Cost (\$)
LO ₂ LN ₂	Cryogenic Sampler (MIL-S-27626) Cryogenic Sampler (MIL-S-27626)	Cosmodyne CS 4.4* Cryogenic Sampler; used with Wyle Dynamic Fluid Sampler.	1015**
LH ₂	Cryogenic Sampler (MIL-S-27626)	Cosmodyne CS 4.4H* Cryogenic Sampler used with Wyle Dynamic Fluid Sampler.	1220**
RP-1	Screw-top glass bottle with conical polyethylene seal.	One-quart glass bottle with screw-on type bakelite cap and conical poly- ethylene seal.	
N ₂ O ₄ A-50 MMH	Stainless steel, double-valved pressure bomb. Some sample A-50 and MMH in a glass or polyethyl- ene bottle.	Hoke Model 8D1488-2250 double-valved stainless steel pressure bomb.	210
GO ₂ GN ₂ GH ₂ Helium	Stainless steel, double valved pressure bomb. Some use avi- ator's breathing oxygen bottle.		
Moisture in gases	Electrolytic-type moisture indica- tor attached directly to system. Some take moisture readings from bomb samples.	CEC Moisture Monitor No. 26-304 Beckman "Hygromite" No. 17902	700 1000

*These units will be replaced by Cosmodyne CS-5.0 used with Wyle Dynamic Fluid Sampler when available. Costs will be comparable to those shown for the existing equipment.

**Costs given are for the Cosmodyne unit only. Costs of the Wyle DFS are not available because the design of the specific units depends on such application requirements as line size and type, system pressure, type of end connections, and automatic or manual control.

No company is taking the above fundamental steps toward sample validity although all users with whom the concept was discussed agreed that it appears to answer the problem. Instead, the industry still uses one of two general procedures. The first is to take a fluid sample in a container, filter this sample through a membrane which has a counting grid imprinted on it, rinse the membrane and transfer it to a microscope stage for counting. In the second procedure, the membrane is inserted into a sampling tool in the laboratory. The tool is then transported to the sampling point and connected to it. The particle sample is taken from the fluid directly on the membrane. The tool is then disconnected from the sampling point, and transported back to the laboratory (in most instances in uncontrolled orientation with respect to the vertical) where the sampling device is disassembled and the membrane is transferred to the microscope stage for counting.

The next significant departure from previous technology involves the taking of all samples from cryogenic fluid streams and all particulate samples from the other fluid streams. It is recommended that such samples be taken through a Wyle Laboratories Dynamic Fluid Sampler or a Maledco Engineering Company Turbulent Flow Sampling valve, depending upon the line sizes and fluids involved, instead of through a simple side-tap. The Wyle unit incorporates two basic principles: (1) inclusion of a mechanism which permits the withdrawal of a "slice" of fluid from the full cross section of the fluid system; (2) maintenance of isokinetic flow conditions both inside and outside of the sample withdrawal mechanism. The Maledco unit is designed with a "Z" section flow path which creates turbulence, and the sample is withdrawn directly from the turbulent section. These are the only suitable continuous representative particulate sampling devices disclosed by the study.

Because this is a very controversial parameter, previous studies pertaining to it were investigated thoroughly. It was concluded that, for particles larger than 100 μ in fluids less viscous than MIL-H-5606 hydraulic oil, isokinetic sampling is necessary for adequate representation in the sample of the system particulate content regardless of whether the flow is laminar or turbulent.

Table 4 summarizes present practice and recommendations for sampling for particulate contamination.

Table 4 Particle Content Sampling Summary

Fluid	Present Practice	Recommendation
LO ₂ LN ₂ LH ₂	Membrane filtration into or from a dewar flask.	In-line filter-holder and counter (ILFHC) used with Wyle Dynamic Fluid Sampler.
RP-1	Screw-top glass bottle with conical polyethylene seal.	ILFHC used with Wyle Dynamic Sampler (Maledco Turbulent Flow Sampling Valve may be used with some flow conditions and line sizes).
N ₂ O ₄ A-50 MMH	Stainless steel double-valved pressure bomb, with or without a high-pressure membrane holder between sampling point and bomb. In the latter case particle count is taken from bombs. One contractor samples N ₂ O ₄ in Millipore bomb sampler. One contractor samples N ₂ O ₄ and A-50 directly into glass Millipore filtration assembly.	
GO ₂ GN ₂ GH ₂ Helium	Millipore high-pressure membrane holder attached directly to system; gas is purged to atmosphere. Some sample for particles from pressure bomb used to take purity sample.	ILFHC used with Wyle Dynamic Fluid Sampler.

B. PARTICLE MONITORING

The survey of launch sites and static test facilities using propellants and pressurants identical or similar to the operational fluids in this study disclosed no facilities using any of the zone-sensing-type automatic particle counters for inspection of the fluids.

None of the automatic particle counters investigated can detect or report the longest dimension of the particle or fiber. All criteria defining allowable particulate contamination in fluids, including those of NASA, refer to particles and fibers by the longest dimension. Therefore, the use of automatic particle counters for monitoring particulate contamination in the fluids of interest to this study is not feasible unless:

- 1) The criteria are changed to define particles in terms of some parameter other than the longest dimension, e.g., projected cross-sectional area, which can be detected by automatic particle counters;
- 2) A correlation can be demonstrated between particle size-and-count, determined by the automatic counter, with size-and-count determined by the light-microscope method of SAE Aeronautical Recommended Procedure (ARP) 598;
- 3) An automatic instrument can be developed to actually measure longest particle dimension.

Attempts to demonstrate correlations as indicated by the second possibility have been made by a number of organizations. Results of these programs are presented in reports published by NASA-MSFC, Douglas Aircraft Corporation, and AF Materials Laboratory, Wright-Patterson AFB. All of the evaluations have been performed on hydraulic fluids in which nearly all particles were $<100\ \mu$ and most were $<50\ \mu$ in size. The results indicate that the use of automatic particle counters for monitoring particulate contamination in hydraulic fluids appears feasible. However for particles larger than $100\ \mu$ including fibers, the relationship between microscopic determination of the longest particle dimension, and that reported by the automatic counting devices, deteriorates with increasing particle size.

No successful evaluations have been performed in the fluids of interest to this study with the types and sizes of particles of interest to this study. Since, in addition, the available particle counting techniques do not measure particulate contamination in terms of existing criteria, recommendations for the use of automatic particle counters are not indicated at this time.

C. PURITY MONITORING

During the survey of launch and static test facilities only a few instances of on-stream monitoring of operational systems for the fluids of interest were found. These were:

- 1) Flame-ionization hydrocarbon analyzers for total hydrocarbons. These served both as continuous analyzers and periodic analyzers;
- 2) Moisture meters that read by the hydrolysis of absorbed water on hygroscopic plates. These are used for both continuous and periodic monitoring;
- 3) The fritted silver -- KOH type O_2 analyzer for oxygen content in gases. These all were used as continuous on-stream analyzers;
- 4) A gas chromatograph that was programed by electronic tape for on-stream measurement of trace gas contaminants in hydrogen;
- 5) An instrument designed to measure the ortho-para ratio of hydrogen continuously on-stream;
- 6) A liquid process moisture meter that measured moisture content in petroleum products. This device is used continuously on-stream.

All the organizations visited used various means of instrumental analysis in a laboratory for some contamination parameters in the fluids of interest, with the exception of N_2O_4 . However, a number of these organizations still depended quite heavily upon wet chemical analysis.

At the present time there are some specific instruments that appear promising for adaption to on-line use for the contamination parameters in the fluids of interest. These have maximum applicability to the gases.

With the above considerations in mind, a survey of the instrumentation manufacturers was made. The intent of the survey was to find available instrumentation capable of providing continuous monitoring of one or more of the parameters of interest to the study. Because very few types of continuous monitors were found, an alternative approach was used. This approach was to provide an integrated system of continuous monitors for parameters that could be so monitored, and rapid recycle period monitors for those parameters that are impossible to monitor continuously. This integrated system comes as close to the idealized concept as possible.

One system uses mass spectrometers in conjunction with hydrocarbon analyzers, moisture monitors, and part per million (ppm) oxygen analyzers for the analysis of the contaminants in the cryogenic or gaseous fluids. The other system substitutes process stream gas chromatographs for the mass spectrometer to provide the same analyses. Both systems would use process gas chromatographs for the analysis of the ambient liquids if these parameters are monitored.

The following techniques are recommended for monitoring the specific contaminants in each of the fluids of interest. In some cases an alternative method is indicated. In each case, the primary instrument is listed first.

- 1) GN_2 (converter-compressor and pad storage areas)
Ref: MSFC-SPEC-234:
 - a) Purity will be monitored by instrumental determination of impurities and the automatic subtraction of these from the total;
 - b) Hydrocarbons will be monitored by the flame ionization type total hydrocarbon analyzer;
 - c) Moisture will be monitored by an electrolytic hygrometer than reads hydrolysis of absorbed water;
 - d) Trace O_2 will be monitored by a ppm oxygen analyzer of the type that has lead and fritted silver electrodes immersed in a KOH bath.

2) GN_2 [Mobile Launcher (ML) or AGCS area] Ref: MSFC-SPEC-234:

- a) Purity will be monitored by instrumental determination of impurities and the automatic subtraction of these from the total;
- b) Hydrocarbons will be monitored by the flame ionization type total-hydrocarbon analyzer;
- c) Moisture and trace O_2 will be monitored by a mass spectrographic analyzer.

Alternatives: An electrolytic hygrometer for moisture content and a ppm O_2 analyzer for oxygen content can be used.

3) Helium (ML or AGCS, converter-compressor, pad areas) Ref: MSFC-SPEC-364A:

- a) Purity will be monitored by instrumental determination of impurities and the automatic subtraction of these from the total;
- b) N_2 , H_2 , O_2 and moisture will be monitored by a mass spectrographic analyzer;

Alternatives: N_2 , H_2 can be monitored by a process stream gas chromatograph. O_2 can be monitored by a ppm oxygen analyzer. Moisture can be monitored by an electrolytic hygrometer.

- c) Hydrocarbons will be monitored by a total-hydrocarbon analyzer of the flame ionization type.

4) Gaseous oxygen Ref: MSFC-SPEC 399 (Grade C):

- a) Purity will be monitored by the paramagnetic oxygen analyzer;
- b) Moisture will be monitored by an electrolytic hygrometer;
- c) Total hydrocarbons will be monitored by a flame ionization type total hydrocarbon analyzer;
- d) Acetylene content will be determined by nondispersive infrared spectroscopy.

5) Gaseous hydrogen (ML or AGCS and pad storage areas) Ref: MSFC-SPEC-356:

a) Purity will be monitored by instrumental determination of impurities and the automatic subtraction of these from the total;

b) O_2 , Ar, He, carbon-bearing gases, and moisture will be monitored by a mass spectrographic analyzer;

Alternative: These commodities can be measured by a process stream gas chromatograph.

c) Total hydrocarbons will be monitored by a flame-ionization total-hydrocarbon analyzer.

6) LN_2 (Ref: MSFC-SPEC-234) and propellant grade oxygen (Ref: MSFC-SPEC-399, Grade C):

There are no monitoring devices capable of measuring contamination directly in cryogenic fluids. However, for LN_2 and LO_2 of propellant grade, a continuous cryogenic sampler that used a continuous flash vaporizer can be used with the instrumentation proposed for the gaseous phase of these commodities to perform the required analysis. These cryogenic samplers are commercially available in several forms.

It is not considered necessary that LN_2 be monitored as it flows from the storage dewars to the converter-compressor facility, because N_2 is not used as a liquid. LN_2 is a source of GN_2 , and is monitored as such after vaporization. However, a sampling point to check the condition of LN_2 in the dewars during storage is recommended.

7) LH_2 (Ref: MSFC-SPEC-356):

For LH_2 a continuous vaporizing cryogenic sampler, in conjunction with the instrumentation proposed to monitor GH_2 , will be sufficient to analyze all contaminants.

8) Aerozine-50 (Ref: MIL-P-27402 (USAF):

The N_2H_4 , UDMH, amine, and moisture content of Aerozine-50 can be monitored by a process stream gas chromatograph. However, it is not necessary to monitor A-50 on a continuous basis because the quantities used are not large, the loading time is short enough that monitoring by the gas chromatographic technique appears to be impractical, and the present method of sampling is accurate and reliable enough to ensure that good quality propellant is loaded.

9) MMH (Ref: MIL-P-27404):

Purity and moisture content can be monitored by a process stream gas chromatograph. However, the reasons for not monitoring A-50 apply to MMH.

10) N_2O_4 (Ref: MIL-P-26539A):

At the present time there is no known way of measuring purity or contamination content by instrumental methods. Some manufacturers have proposed the use of a process stream gas chromatograph for these analyses. However, each chromatograph proposed requires an extensive developmental effort, with no guarantee that the method will prove to be really satisfactory. Again, the reasons for not monitoring A-50 apply to N_2O_4 .

11) RP-1 (Ref: MSFC-SPEC-342):

- a) Mercaptans will be monitored by nondispersive infrared at 3.92μ ;
- b) Aromatics will be monitored by nondispersive infrared at 5.1μ ;
- c) Olefins will be monitored by nondispersive infrared at 11μ ;
- d) Existent and potential gums will not be monitored;

- e) Moisture will be monitored by an ultraviolet analyzer;
- f) All of the above instruments require a vaporized sample for proper operation. Therefore, these instruments must be used with a high temperature vaporizer and the analyses made in the vapor phase.
- g) Sulfur in solution could possibly be determined by variations in light transmission monitored by a photocell. This technique will take a long time for development. Sulfur not in solution will probably be filtered out mechanically and will not contribute significantly to the total sulfur content.

The following prices include costs incurred by explosion-proofing and in otherwise making the instruments compatible to NASA specifications.

- 1) Paramagnetic Oxygen Analyzer - The only instrument suitable for the oxygen purity analysis is the Beckman Model G-2 paramagnetic oxygen analyzer. This instrument costs approximately \$3900;
- 2) Total-Hydrocarbon Analyzer - The primary candidate for this instrument is the Beckman total-hydrocarbon analyzer. The Model 108A is suitable for use in nonhazardous areas and costs approximately \$2600. The Model 106E proposed for use in hazardous areas costs approximately \$3800, compared to the Bendix hydrocarbon analyzer for the same use at a cost of \$5000;
- 3) Parts-Per-Million Trace Oxygen Analyzer - The primary candidate for this instrument is the Beckman Model 778. This instrument costs approximately \$4000; the Bendix parts-per-million oxygen monitor also costs \$4000;
- 4) Electrolytic Hygrometer - The primary candidate for this instrument is the Beckman electrolytic hygrometer with a rhodium electrode. This instrument costs approximately \$2200, compared to a CEC moisture monitor at \$2600 and the Bendix electrolytic hygrometer at \$3000;

- 5) Process Gas Chromatographs - The Bendix high-speed gas chromatograph costs approximately \$15,000 with a hydrogen flame detector attached. This instrument costs about \$12,000 without the detector. The Melpar unit costs about \$40,000 for the first unit, including master control panel. Subsequent units with master control panels would cost about \$15,000 each. Additional units without control panels would cost about \$10,000 each. Perkin-Elmer was unable to quote an approximate cost for one of their high-speed chromatographs. The Beckman Model 320-D process gas chromatograph that uses standard chromatographic techniques and equipment costs approximately \$9500. The Bendix chromatograph has the advantage over the others, because it can perform very rapid analyses (15 sec to 2 min), and it is readily available with a minimum of developmental effort. The Beckman model is a standard instrument that performs the analyses in 5 to 10 min. The Perkin-Elmer instrument needs considerable development;
- 6) Mass Spectrograph - The instrument proposed for use as a common mass spectrograph in the base of the ML or AGCS costs between \$150,000 and \$175,000. This instrument would use standard off-the-shelf components. This instrument would cost an additional \$500,000 for developmental effort if it were to use lightweight components. The mass spectrometers proposed for use in all the other areas cost approximately \$25,000 each. CEC was the only instrument manufacturer that proposed use of the mass spectrometer;
- 7) Infrared and Ultraviolet Analyzers - The Perkin-Elmer miniature infrared and ultraviolet analyzers are the instruments proposed. These instruments cost between \$3500 and \$5000 depending on required development effort. Although MSA proposed infrared analyzers, the Perkin-Elmer model was recommended because of the obvious advantage offered by its very small size. MSA did not provide cost data for their equipment.

VI. RECOMMENDATIONS FOR FURTHER STUDIES

To efficiently apply the results of this study to the scope systems at KSC, this study should be continued in the next logical phase, i.e., a detailed analysis of the specific application of the study's recommendations to the specific optimum sampling points for those systems at KSC.

The space, weight, accessibility, safety-hazard, power consumption, data-transmission, operational fluid status and mode, mechanical interference, sampling system adaption, sampling point location, countdown and operational schedule compatibility, display, recording and corrective action limitations and requirements should be investigated and cataloged for each intended sampling point, data transmission point, and display location. The location of each optimum sampling point should be defined together with a comprehensive justification, cognizant of and compatible with the operational schedules.

This study was oriented toward the equipment and method. The next phase should be oriented toward the specific system application. Only after this phase is completed and practicality is shown, should the subsequent logical phases of breadboard design and prototype development be undertaken for monitoring equipment. (Development of sampling equipment may be started before the next program phase because its application is more universal.)

It is recommended that the existing contamination control criteria specifications for particulates be converted to criteria that are oriented toward the area or light-scattering parameters, which are detected, measured, and reported by the zone-sensing automatic particle counters available or conceptual today.

It is recommended that a comprehensive, uniform, contamination control criteria specification document be prepared to contain all the contamination control criteria specifications for all the program system operational fluids and systems (both fluid and hardware criteria) for distribution to all NASA organizations and contractors. This document should provide for a logical progression of criteria from that of the detail parts at component manufacturers facilities, and from the vendor transport vehicles for the fluids to the hardware and fluids in the airborne systems.

A sampling manual should be prepared that identifies sampling points and sampling frequency (both by time interval and by sequence relation to system operation).