Process to Remove Heavy-End Contaminants from Commercial LP Gas Streams

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Project Objectives

- Design and test an adsorption column / filtration process to remove “heavy-end” contaminants from LP Gas
- Achieve average heavy residues content below 25 ppm

Background

- Higher purity LP Gas is necessary for present and future applications.
- LP Gas residues are a world-wide problem causing failures in:
  - Vaporizers
  - Meters
  - Regulators
  - Fuel Cells
  - Engine Fuel Systems
  - Microturbine Fuel Systems and Injectors

Higher Purity = Higher Cost

- Average U.S. West Coast market premium over HD-5 prices for low-residues LP Gas is ~10¢/gallon.
- Low residue LP Gas from natural gas processing may be costlier than LP Gas from closer refineries.
- Farther LP Gas sources $\Rightarrow$ Higher LP Gas transportation costs.

LP Gas Residues: Comparison of International Standards

- International LP Gas residue limits provide a basis to determine to what level the decontamination unit should reduce residues

Nation	LP Gas Specification	Residues Limit (ppm)	Test Method	Evap. T (°C/°F)
US California	HD-5 (ASTM D 1835, GPA 2140) HD-10 (CCR §2292.6)	500 (and oil stain) “	ASTM D 2158 “	38/100
Japan	Utility Grade	12	JLPGA-S-05T	-
Australia	ALPGA Automotive Spec. 2000	20	JLPGA-S-05T/86	105/221
Europe	EN-589	100 (proposed reduction to 50 ppm)	EN ISO 13757	105/221

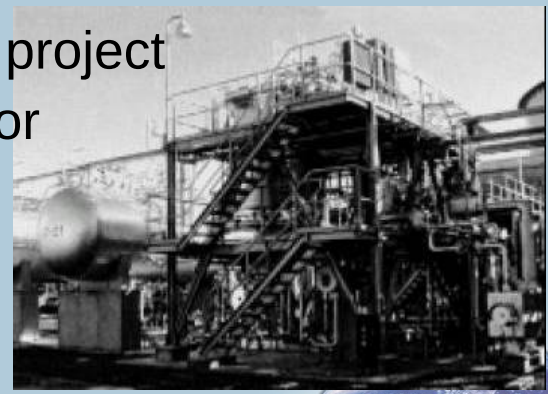
Prior LP Gas Decontamination Work

John O'Connor (formerly of Phillips Petroleum Co.)

- U.S. Patent No. 5,474,671, a *Process for Removing Oil from Liquefied Petroleum Gas*. (December 1995)
- Activated carbon packed column to effectively remove residual oil
- Method to retain mercaptans during the process
- Mr. O'Connor is an ASCENT project advisor

Cosmo Engineering Co. (CEC)

- Multi-million dollar, Japanese government funded project
- Resulted in a 2 ton/h plant with activated carbon for residue removal
- CEC provided preliminary engineering support
- Dr. Quan Zhuang (a former principal CEC project principal) is an ASCENT project advisor



1st Pilot Scale Test Objectives

Determine:

- Effect of activated carbon on selected compounds found in LP Gas residues
- Amount and size of activated carbon fines lost from column
- Changes in process temperatures and pressures during process

Experimental Setup







Contaminated Supply Preparation

- Selected pure contaminants were measured and mixed with Propellant-Grade LP Gas

Contaminant	Description
n-Octane:	representative compound of gasoline contamination
Pentadecane	representative compound of diesel contamination
Methyl linoleate	plasticizer and known LP Gas contaminant
Dioctyl adipate	plasticizer and known LP Gas contaminant
Butyl benzyl phthalate	plasticizer and known LP Gas contaminant

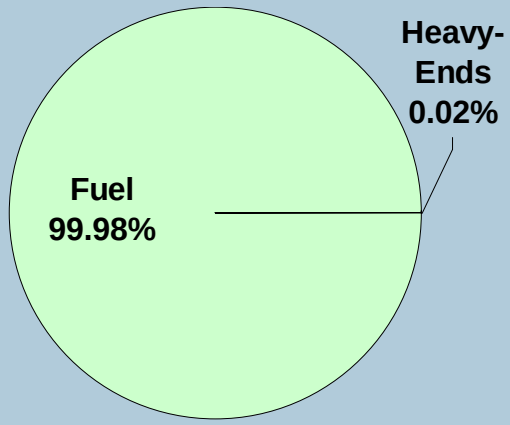
Results from 1st Pilot Scale Tests

Contaminants	Concentration (mass ppm)				
	Base	Feedstock	Sample #4	Sample #5	Sample #6
n-Octane	0	19	11	21	20
Pentadecane	0	42	0	0	0
Methyl linoleate	0	19	0	0	0
Dioctyl adipate	0	27	0	0	0
Butyl benzyl phthalate	0	32	0	0	0
TOTAL	**1**	**141**	**12**	**22**	**24**
Sample Conditions					
LP Gas Processed Prior to Sample (gal)	N/A	0	8.4	11.7	13.0
Average Flowrate During Sampling (gal/min/ft²)	N/A	N/A	1.9	10.7	7.5

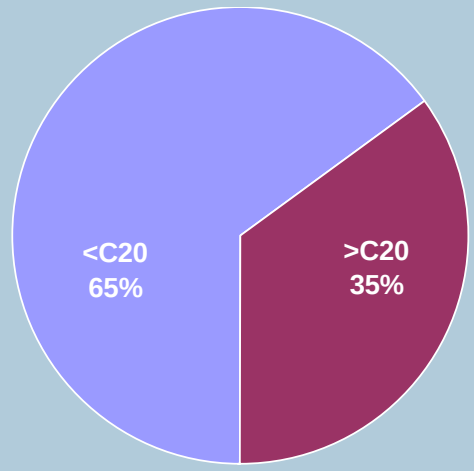
Full Removal of All Contaminants Except Octane

Residues Causing Fuel System Deposits

LP Gas Composition



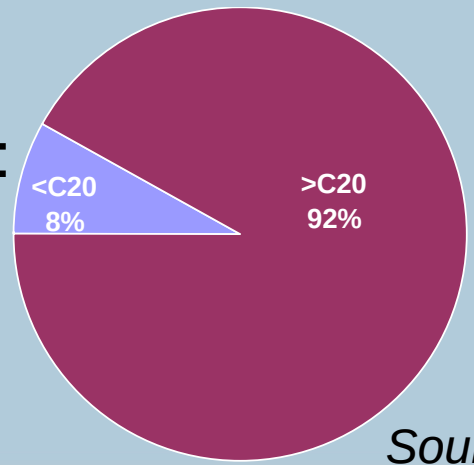
Heavy Ends in Fuel



Recent SwRI Research Shows:

- >C20 Form Most Deposits
- <C20 Are Carried Through Combustion

Heavy Ends in Deposits



Source: SwRI

2nd Pilot Scale Test Objectives

- Determine effect of activated carbon on actual LP Gas residues and C20+ compounds
- Determine effect on ethyl mercaptan (odorant)
- Use shorter bed length
- See breakthrough trends for all contaminants
- Achieve better flow control

2nd Test Contaminated Supply

- Contaminants representative of heavier residues were mixed with HD-5 LP Gas

Contaminant	Description
n-Octane (C_8H_{18})	a representative compound of gasoline contamination
Dioctyl adipate ($C_{22}H_{42}O_4$)	a plasticizer and known LP Gas contaminant
American Welding & Tank residues	a blend of many contaminants from LP Gas tanks (95% recovered below n-C20 boiling point
Mobil Rarus 427 compressor oil	ISO 100 (viscosity) with 100% recovered above n-C20 boiling point

Other Changes from 1st to 2nd Test

Addition of Contaminants

Transfer hose

Modified inlet:
- Shorter
- Steel

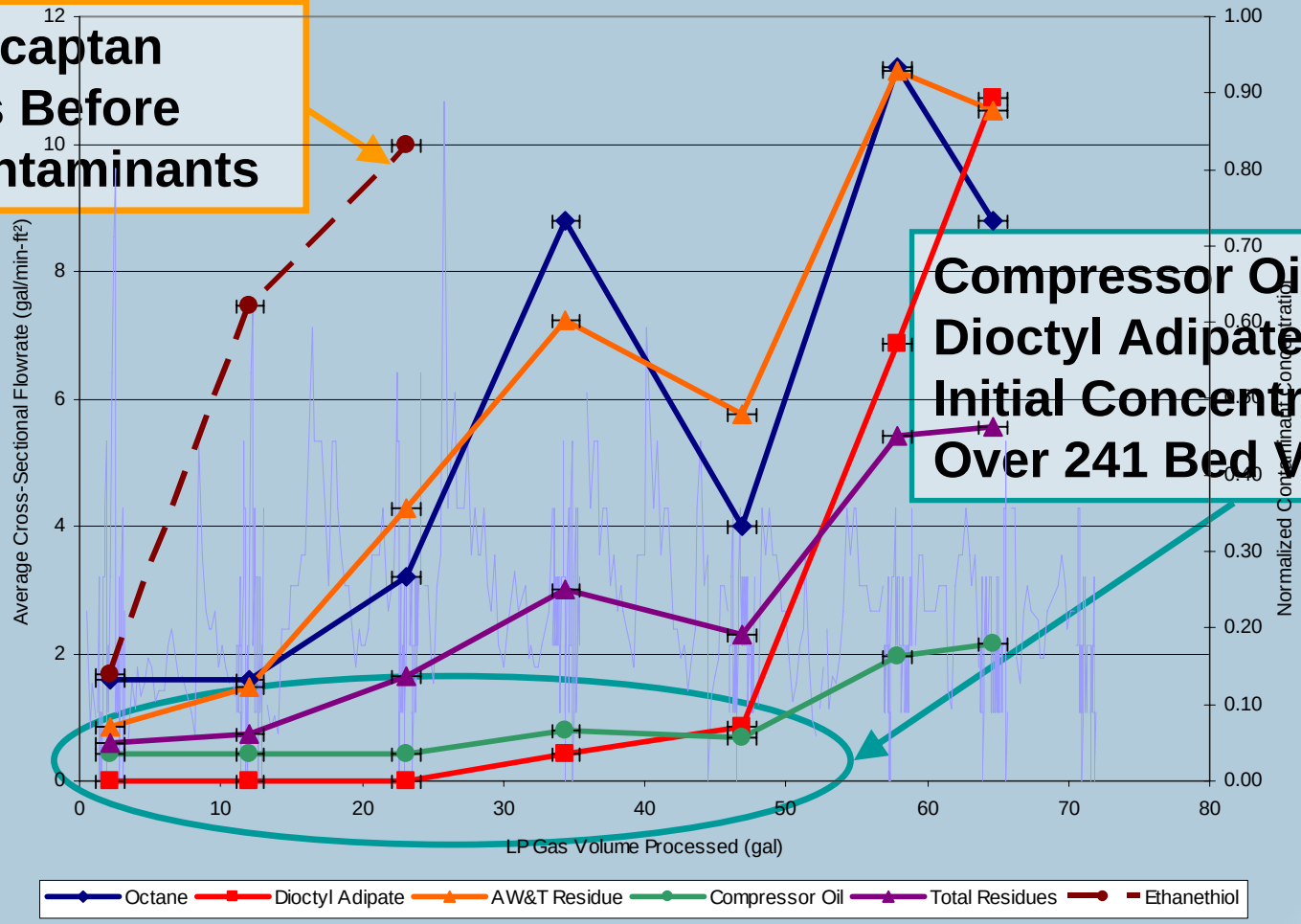
Activated Carbon Bed Length

Long bed

Shorter bed proportional to full-scale column

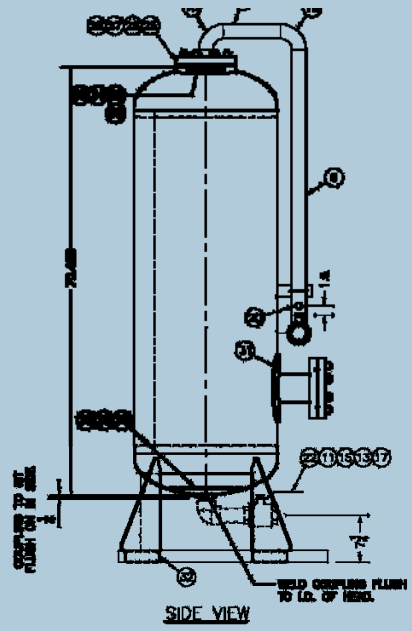
2nd Pilot Scale Test Results

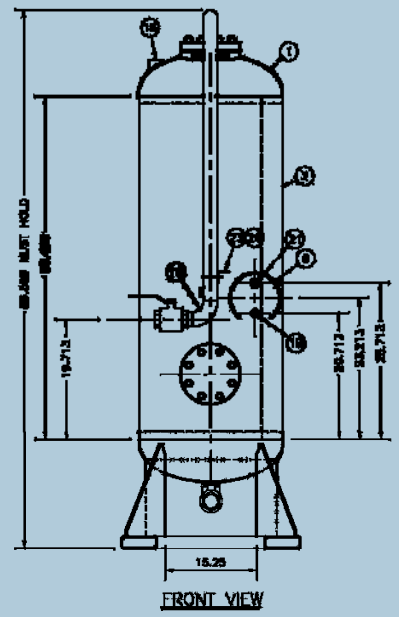
Ethyl Mercaptan Increases Before Other Contaminants



Compressor Oil & Dioctyl Adipate <10% of Initial Concentration for Over 241 Bed Volumes

Full-Scale Vessel Design





Design Constraints:

- Footprint: 48” x 48” (1.2 m x 1.2 m)
- Weight: <3,200 lbs. (1,450 kg)
- Length: 72” (1.83 m)
- Pressure: 250 psi (17.2 bar) rating at 650 °F (343 °C)

Economic Viability

Economic Analysis Summary

Annual Production (gal)	1,057,536
Total Capital Cost	$ 98,862
Annual Manufacturing Cost	$ 64,948
Annual Revenues	$105,754
Payout Time (years)	2.74

- Manufacturing cost per gallon of LP Gas is $0.061
- Revenues are based on $0.10 per gallon of processed LP Gas
- Capital costs include purchase and installation of a new storage tank and gas chromatograph

Immediate Next Steps

- Further Pilot-Scale Tests
  - More effective flow control
  - Increased flowrate
  - Lower contaminant concentrations
- Full-Scale Column Tests

Future Work

- Decontamination Process Optimization
- Regeneration Tests

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