Introduction

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- First of a Kind, One of a Kind Custom Equipment Design
- ASME NQA-1 and B&PV Quality Programs
- ASME B&PV Code
- cGMP Pharmaceutical Equipment
- Hazard and Safety Analysis
- Installation and Start-Up
- Factory and Site Testing

Prior experience in chemical, petrochemical, nuclear, and pharmaceutical industries.
Introduction

- **Pressure Safety Inspectors**
  - Performed ~100 engineering peer reviews (3rd party design verifications) on extraction equipment
  - Reviewed 300+ extraction facilities in California, Colorado, Nevada, Oregon, and Washington
  - Performed hundreds of equipment field verifications
  - Started working in this industry in February of 2014

- **Headquartered in Colorado**
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OBJECTIVES
Course Objectives

- Understand the following:
  - Hazards Associated with Extraction
  - Codes & Standards
  - Equipment Approval Process
PROCESSING HAZARDS
Hazards

- Extraction Equipment
  - Water Based (no significant hazards)
  - Solvent Based Extraction
    - Butane (LPG)
    - Propane (LPG)
    - Carbon Dioxide (CO$_2$)
    - Ethanol
    - Purge Gases
    - Carbon Dioxide (CO$_2$) in use other than solvent
    - Others
Hazards – Solvent Based Extraction Equipment

- Butane (LPG)
  - Could be n-butane or iso-butane
  - Vapor pressure up to 100 psig @100°F (piping designed for 350 psig)
  - Temperature typically from -20°F to 125°F
    - Could be as low as -110°F
  - Typical Quantity 10 lbs to 50 lbs of LPG
Hazards – Solvent Based Extraction Equipment

- Propane (LPG)
  - Vapor pressure up to 250 psig @100°F (piping designed for 350 psig)
  - Can also run Butane in the system
  - Temperature typically from -20°F to 125°F
    - Could be as low as -110°F
  - Quantity 10 lbs to 50 lbs of LPG
Hazards – Solvent Based Extraction Equipment

- LPG
  - Low Pressure
  - Asphyxiation
  - Explosion/Fire
  - Contact with Eyes/Skin
  - Low Temperature (freezes skin on contact)
  - Unscented
Hazards – Solvent Based Extraction Equipment

- Butane only Equipment
  - Significantly lower vapor pressure therefore system is designed to operate below 100 psig @100°F with pressure reliefs set at around 130 psig
  - Propane and Propane mixtures can not be used with this equipment due to higher vapor pressures
    - The vapor pressure of Propane at 100°F is 172 psig
    - While the equipment is protected by pressure relief valves, hydrocarbons will be released into the room if propane is used
Hazards – Solvent Based Extraction Equipment

- Carbon Dioxide (CO₂)
  - Very High Pressure
  - Asphyxiation
  - Contact with Eyes/Skin
  - Temperatures
Hazards – Solvent Based Extraction Equipment

- Carbon Dioxide (CO$_2$) (not as a solvent)
  - May be used to cool through Joule-Thomson effect
    - The Joule-Thomson effect describes the temperature change of a liquid when it is forced through a valve or porous plug while kept insulated so that no heat is exchanged with the environment. This procedure is called a throttling process or Joule–Thomson process. The throttling process is commonly exploited in thermal machines such as refrigerators, air conditioners, heat pumps, and liquefiers.

- Dry Ice baths (Cooling Baths)
  - Dry Ice in a solvent bath
  - Dry Ice with Acetone
    - As low as -78°C (-109°F)
Hazards – Solvent Based Extraction Equipment

- Purge Gases (Displace Oxygen)
  - Argon
  - Nitrogen
  - Helium
Hazards – Solvent Based Extraction Equipment

- Ethanol
  - AKA: Ethyl Alcohol; Grain Alcohol
  - Typically not pressurized may be in vacuum
  - -100°F to 212°F
  - Up to 100 gallons
Hazards – Solvent Based Extraction Equipment

- Ethanol
  - External Pressure (Vacuum) or atmospheric
  - Pressure (must ensure pressure relief)
  - Explosion/Fire
  - Contact with Eyes/Skin
Hazards – Other Chemicals

- Other chemicals that may be present
  - Isopropanol
  - Acetone
  - Heptane
  - Hexane
  - Pentane
UNDERSTANDING THE EXTRACTION PROCESS
What is this Process and Which Codes Apply?

- Before we get to which codes are relevant, we must understand what the process is.
  - Industrial Process or Refrigeration Cycle?
  - Is NFPA 36 Standard for Solvent Extraction Plants relevant?
A Refrigeration Cycle this is NOT

- Butane and Propane may be used as a refrigerant in some applications (more prevalent outside of US)
- This process is NOT a refrigeration cycle
- This process may be achieved without a compressor (thermal cycle only)
- Represented to AHJs as a refrigeration cycle (false)
  - Only needs to comply with IMC Chapter 11 (false)
  - Containers are routinely opened
  - Refrigeration Cycles do not include plant material
A Refrigeration Cycle this is NOT

BASIC REFRIGERATION CYCLE

HEAT IN

LOW PRESSURE GAS

LOW PRESSURE LIQUID & GAS

COOLER

CONTROL DEVICE

LOW PRESSURE

HIGH PRESSURE

HIGH PRESSURE GAS

HEAT OUT
Industrial Process

- This is an industrial chemical process
- The solvent is condensed and evaporated in order to introduce or remove the solvent from the product, not to provide cooling
Industrial Process
Industrial Process

Flow Line Diagram

1. Liquid solvent is injected from the operating tank into the extraction column via the red safety hose where it permeates the source material and removes the desired constituents. The soak time and pressure will vary depending on the solvent used.

2. The solvent remains fluid under pressure, contained within the extraction column between valves 1 and 2. When valve 2 is opened, the pressure forces the liquid solvent through the slotted-screen and perforated gasket into the separator vessel. The gauges should reflect pressure equalizing a few moments after opening valve 2.

3. The extract pools at the bottom of the collector vessel and the solvent begins converting to vapor. Applying heat to the collector vessel via the water jacket speeds this process. Opening valve 3 releases pressurized solvent into the expansion filter.

4. The top connector of the expansion filter receives solvent from the collector vessel at the terminus of valve 3 via the first green safety hose. The vapor drops to the bottom of the vessel and is passed through a molecular sieve before being drawn out by the recovery pump through the front connector. Applying heat to the expansion filter via the water jacket speeds this process.

5. The solvent vapor exits the front of the expansion filter and is drawn into the inlet side of the recovery pump via the second green safety hose. Before entering the recovery pump the vapor passes through a desiccant filter and spot glass connected to the pump inlet.

6. The scrubbed solvent vapor is drawn into the recovery pump in pulses and stabilized in the internal compressor. The solvent is then released from the discharge side of the recovery pump, back into the operating tank via the blue safety hose.
EXTRACTION EQUIPMENT HISTORY
A brief History of Extraction Equipment

- From illegal operations to legal – it’s a struggle
  - No regulation
  - No procedures
  - No training
- Evolved from a PVC pipe in a backyard...
A brief History of Extraction Equipment
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A brief History of Extraction Equipment
CODES AND STANDARDS
Standard for Solvent Extraction Plants

Chapter 1 Scope:

1.1.1 This standard shall apply to the commercial scale extraction processing of animal and vegetable oils and fats by the use of Class I flammable hydrocarbon liquids, hereinafter referred to as “solvents.”

1.1.9 This standard shall not apply to extraction processes that use flammable gases, liquefied petroleum gases, or nonflammable gases.
NFPA 36

- Scale or the processing plant:
  - Intent is for a large facility
  - Thousands of gallons of solvent
  - Outdoor processing
  - Conveyor systems
  - Cooling towers
  - Massive throughput – think about how much coffee needs to be decaffeinated...
  - Does not take into account security concerns specific to this industry
Solvent Extraction Plant - Example
Solvent Extraction Plant - Example
NFPA 36

- Does NOT apply to small scale cannabis processing
Codes & Standards

- International Fire Code (IFC)
- NFPA
  - NFPA 1 (Fire Code) Chapter 38 (new chapter in development)
    - Will not include details on equipment requirements (very focused on facility requirements)
  - NFPA 58 (Liquefied Petroleum Gas Code)
    - This type of process equipment is not included in scope
  - NFPA 55 (Compressed Gases and Cryogenic Fluids Code)
    - This type of process equipment is not included in scope
  - NFPA 45 (Standard on Fire Protection for Laboratories Using Chemicals)
    - Manufacturing plants not included in scope
  - NFPA 30 (Flammable and Combustible Liquids Code)
  - ASME Boiler and Pressure Vessel Code
NFPA 58

- Liquefied Petroleum Gas Code
  - Code is aimed at storage and use of LPG as a fuel
  - Only code that specifically deals with LPG

Scope

- (1) Containers, piping, and associated equipment, when delivering LP-Gas to a building for use as a fuel gas.
- (2) Highway transportation of LP-Gas.
NFPA 58

Liquefied Petroleum Gas Code

Non-application of Code

(4)* Chemical plants where specific approval of construction and installation plans is obtained from the authority having jurisdiction

A.1.3.2(4) The exclusion of the use of LP-Gas as a chemical reactant (feedstock) or in processes recognizes the unique and complex fire hazard problems that often exist in a chemical plant. The term chemical plant includes all facilities owned by chemical companies where LP-Gas is used primarily as a chemical reactant, process solvent gas, or solvent. However, there is no standard definition of a chemical plant, and facilities in which few or no chemical reactions are carried out may be called chemical plants.
NFPA 58

Relevant Parts of NFPA 58

Parts of Chapter 5: LP-Gas Equipment and Appliances

- Materials of Construction (Chemical Compatibility)
- DOT and ASME requirements
  - Maximum Allowable Working Pressure of ASME containers
- Pressure Relief Requirements
- Warning Labels
- Hose requirements
- Safeguards for 100% liquid LPG (no vapor space) trapped in a piping system (Hydrostatic Pressure)
NFPA 55

- Compressed Gases and Cryogenic Fluids Code
  - Code is aimed at storage, use, and handling of compressed gases and cryogenic fluids in portable and stationary cylinders, containers, equipment, and tanks in all occupancies.
NFPA 55

- Compressed Gases and Cryogenic Fluids Code
  - Non-application of Code
    - (6) Storage, use, and handling of liquefied and nonliquefied compressed gases in laboratory work areas in accordance with NFPA 45, Standard on Fire Protection for Laboratories Using Chemicals
NFPA 55

- Relevant Parts of NFPA 55
  - Parts of Chapter 13: Insulated Liquid Carbon Dioxide Systems
    - Pressure Relief
    - Vent Pipe Systems (PRV exhaust)
    - Burst pressures ** (already covered by ASME for pressure vessels)
NFPA 45

- Fire Protection for Laboratories Using Chemicals
  - Code is aimed laboratory environments
  - Scope
    - 1.1.1 This standard shall apply to laboratory buildings, laboratory units, and laboratory work areas whether located above or below grade in which chemicals, as defined, are handled or stored.
NFPA 45

- Fire Protection for Laboratories Using Chemicals
  - Non-application of Code
    - (4) Laboratories that are primarily manufacturing plants
NFPA 45

- Relevant Parts of NFPA 45
  - In general this is process uses laboratory equipment and is very similar to a laboratory environment.
    - Refrigerator/Freezer requirements
    - Chemical Fume Hood requirements
NFPA 30

- Flammable and Combustible Liquids Code
- Code
  - Scope
    - 1.1.1* This code shall apply to the storage, handling, and use of flammable and combustible liquids, including waste liquids, as herein defined and classified.
NFPA 30

- Flammable and Combustible Liquids Code
  - Ethanol processing does fall into the scope of this document
  - This code does not cover chemical fume hoods
  - This code does not cover freezers/refrigerators
ASME Boiler and Pressure Vessel Code

- Section VIII Division 1
  - Rules for the Construction of Pressure Vessels
  - Scope
    - (2) For the scope of this Division, pressure vessels are containers for the containment of pressure, either internal or external.
    - (3) This Division contains mandatory requirements, specific prohibitions, and nonmandatory guidance for pressure vessel materials, design, fabrication, examination, inspection, testing, certification, and pressure relief.
When is ASME BPVC applicable?

Compressed gas containers, cylinders and tanks shall be designed, fabricated, tested, marked with the specifications of manufacture and maintained in accordance with the regulations of the ASME Boiler and Pressure Vessel Code, Section VIII (IFC 5303.2).

Non-application of code

(-1) vessels having an internal or external pressure not exceeding 15 psi (100 kPa)

(-i) vessels having an inside diameter, width, height, or cross section diagonal not exceeding 6 in. (152 mm), with no limitation on length of vessel or pressure

Therefore all vessels containing pressure in excess of 15 psi with an internal diameter greater than 6” falls into the scope
ASME Boiler and Pressure Vessel Code

- UG-116 Required Markings
  - Official Certification Mark
  - Name of the manufacturer
  - MAWP
  - MDMT
  - Manufacturer’s serial number
  - Year built

- Most States require registration with National Board
Counterfeit ASME Nameplates – Imported from China
Equipment Code Path

- International Fire Code invokes:
  - NFPA 55 (CO₂ systems)
  - NFPA 58 (LPG systems)
    - ASME BPVC
  - ASME BPVC
State Specific Rules

- Some States have developed rule for the processing of marijuana
  - Typically written by attorneys or politicians with no technical knowledge
  - Add requirements above and beyond adopted codes and standards
  - Processors must comply with state rules and local rules
State Specific Rules

- What about hemp?
  - Uses the same equipment!
  - Usually not required to follow the same State rules!
California

- AB-2679
  - Equipment must:
    - Be closed loop
    - (iii) A licensed engineer certifies that the system was commercially manufactured, safe for its intended use, and built to codes of recognized and generally accepted good engineering practices, including, but not limited to, the American Society of Mechanical Engineers (ASME), the American National Standards Institute (ANSI), Underwriters Laboratories (UL), the American Society for Testing and Materials (ASTM), or OSHA Nationally Recognized Testing Laboratories (NRTLs).
Other State Rules

- Existing State rules for equipment and facility design may be misleading
- As new States develop rules they tend to use other State rules as a template
- Errors are repeated
“equipment shall be listed or approved”
LISTINGS
NRTL (Nationally Recognized Testing Lab)

- UL, ETL, etc. (OSHA approved)

- Cons
  - No procedure for this type of equipment
  - Not Code based (no procedure)
  - No pressure vessel analysis
  - No Process Hazard Analysis
  - No chemical compatibility test
  - No field verification

- Pros
  - Addresses electrical systems (will not list equipment as a whole)
ETL Listing of a LPG Extractor

- Issued in 2012
- UL Standard for Safety Motor-Operated Appliances (ANSI/UL 73)

Tests Performed:
- Stability Test
- Leakage Current Test
- Starting Current Test
- Input Test
- Dielectric Voltage Withstand Test
- Operation Test

- Listing was not renewed by Intertek in 2013
ETL Listed Extractor

4.0 Critical Components

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<tr>
<th>Photo no.</th>
<th>Item no.</th>
<th>Name</th>
<th>Manufacturer/trademark</th>
<th>Type / model</th>
<th>Technical data and securement means</th>
<th>Mark(s) of conformity</th>
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<td>1</td>
<td>Accessory</td>
<td>Various</td>
<td>Stainless</td>
<td>Stainless Steel</td>
<td>NR</td>
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<td>1</td>
<td>2</td>
<td>Recovery Pump</td>
<td>Various</td>
<td>Various</td>
<td>115V, 60Hz, 7.1A, 1/2HP</td>
<td>cULus</td>
</tr>
</tbody>
</table>

NOTES:
1) Not all item numbers are indicated (called out) in the photos, as their location is obvious.
2) “Various” means any type, from any manufacturer that complies with the “Technical data and securement means” and meets the “Mark(s) of conformity” can be used.
3) Indicates specific marks to be verified, which assures the agreed level of surveillance for the component. “NR” - indicates Unlisted and only visual examination is necessary. “See 5.0” indicates Unlisted components or assemblies to be evaluated periodically refer to section 5.0 for details.
ENGINEERING PEER REVIEWS
Peer Reviewed Equipment

- Value of a Peer Review
  - Code Based Review
  - Pressure Safety Evaluation
    - Pressure Vessel Analysis
    - Pressure Relief Evaluation
  - Chemical Compatibility of Materials
  - Process Hazard Analysis
  - Operation & Maintenance Manual Review
    - Equipment Safety
    - Minimum Facility Requirements

- Allows for Meaningful Field Verification
Why are Peer Reviews a good idea?

- Codes and Standards
  - No specific standard for this equipment, though portions of existing codes are relevant
- Ensures system is “Closed Loop”
  - “Open Blasting” is illegal and dangerous
- A lot of equipment in this industry is not “engineered or designed” by qualified individuals
- No (or Few) Quality Assurance Programs
- YouTube
Sample BHO Extractor
What’s wrong with that extractor?

- Glass Material Column
  - Is likely fine for normal operation (low pressure)
  - Is NOT ok in an abnormal situation
  - Not per code

- No apparent Pressure Relief

- Pressure Gauges only read to 60 psig

- Vessels over 6” Diameter need to be ASME or DOT vessels

- Recovery Pump is not rated for use with Hydrocarbons (only to be used with non-flammable refrigerants)
Industry Safety Alert: Hydrocarbon Explosion Hazards with Refrigerant Recovery Machines

Explosion hazards always exist when pumping or handling flammable gases or liquids. Refrigerant recovery machines are NOT designed to pump hydrocarbons nor flammable liquids or gases (they are designed for specifically non-flammable refrigerants only). Flammable compounds are being used more frequently in refrigeration systems, and as distillation solvents in other industries. The pumping of these compounds into tanks at high pressures or into tanks creates explosion hazards.

Here’s how:

- Air can be drawn into a system inadvertently at any point due to leaks in fittings, gaskets or seals to create an explosive mixture of air and the flammable compound.
- Any ignition source, including static electrical discharge or heat can cause a sealed tank, vessel or any contained volume with the correct mixture of air and combustible gas to explode.
- Moreover, electrical components and relays produce internal electrical discharges that can ignite any flammable gases that may have leaked into the surrounding air from any discharge of flammable gas into the surrounding environment – Note: This can include the vapors from spilled gasoline or alcohol (any flammable liquid).

The handling of any flammable compounds should only be undertaken by thoroughly trained professionals, and only using certified explosion-proof devices in explosion-proof environments.

Flammable compounds are NOT safe to pump or recover using standard equipment under any circumstances.
Sample Extractor Design
What’s wrong with that extractor?

- No Support Frame
- Attached to a 200 pound Propane Tank
- Attached to a pump that can produce 1450 psig of output pressure
- Collection Tank is NOT ASME or DOT
- No pressure relief
- Equipment is not capable of pressures in excess of 90 psig
Bottom Line

- ISIS would be proud! This company successfully engineered a bomb!
- All parts purchased from a brewery supply store and assembled in a garage.
- Company is no longer in business
Peer Review Process

- PSI analyzes the original configuration of the equipment
- Analysis identifies shortcomings of equipment which is then submitted to the OEM for resolution
- OEM must resolve open safety issues by replacing components, adding components, or redesigning part of the equipment
- OEM then submits updated design to PSI for analysis
- If all items are resolved, PSI issues report stating that the equipment is satisfactory
INSPECTIONS
EPRs, Inspections & Field Verifications

- Performed EPRs for over 50 OEMs
  - [http://www.psinspectors.com/peer-reviews.html](http://www.psinspectors.com/peer-reviews.html)
- Hundreds of facilities across California, Colorado, Nevada, Oregon, & Washington
- Includes facility and equipment inspections
- 100% of the facilities FAIL the first inspection
  - SOP Issues
  - Facility Issues
  - Equipment Issues
  - Training Issues
Installation Issues

- Facility, Equipment Installation, and Procedural issues are rampant!
The Ugly
The Ugly
The Ugly
The Ugly
The Ugly
Equipment Verification

- Colorado was the first state to require an engineering peer review or a NRTL listing of extraction equipment.
- Most extraction equipment manufacturers have had to modify their design because of Colorado extraction rules.
- Older models likely include non-compliant parts.
- Extraction Equipment is not always purchased from the manufacturer (Craigslist is a common resale vehicle).
- Users like to customize equipment or modify with cheaper components.
What is a Field Verification?

- A Field Verification ensures that the equipment is installed in accordance with the engineering peer review.
- Modeled after a NRTL field verification.
Why is this needed?

- No codes/standards for this equipment
- No Listing for this equipment
- Not regulated by the Federal government
- A lot of the equipment manufacturers are start ups
- No Quality Assurance programs
- Some equipment manufacturers do not have the proper engineering knowledge
- Historically only ~40% of the equipment passes the first time.
- Manufacturer may still sell non-certified equipment**
What is a Field Verification?

- Items reviewed during a field verification:
  - P&ID (Piping and Instrumentation Diagram) or PFD (Process Flow Diagram)
  - All Vendor Components are checked (i.e. Valves, PRVs, Gauges, Pumps, etc.)
  - Fabricated Components are verified
  - Warning Labels are reviewed for operating conditions
  - Equipment Nameplate including model and serial number are cataloged
  - Inspection is documented with photographs
What is a Field Verification?

- Additional Benefits
  - A process discussion is conducted with the end user
  - Periodic Maintenance
  - Torqueing
  - Importance of SOPs
  - Hydrocarbon/CO₂ alarms (placement; emergency procedure; etc.)
  - Exhaust system may be discussed
  - Relief Venting
  - Technical discussions (i.e.: Rupture Disks vs Proportional Relief Valves; Cryogenic temperatures; Elastomers; etc.)
Issues with Used Equipment

- Pressure Ratings of Components
- No Pressure Relief
- Chemically incompatible materials (EPDM, silicone)
- Incorrect Assembly
- Hoses not per code
- Customization
Improper CO₂ Venting
Improper Placement of Equipment
Bypassing Safety Features
Pressure Relief Valve Detail

- **Cap**: Provides easy external set pressure adjustment
- **Label**: Identifies set pressure range
- **Lock nut**: Maintains cap position, ensuring set pressure adjustment
- **Lock wire capability**: Secures cap to maintain set pressure adjustment
- **Spring**: Adjusts to provide desired set pressure
- **Quad seal**: Eliminates leakage around stem during relief mode
- **O-ring**: Provides elastomer-to-metal seal for positive shutoff at seat. (Other series use bonded disc. See Materials of Construction)
- **End connections**: Include gaugeable Swagelok® tube fittings and NPT or ISO pipe threads

R3A series valve shown.
Bypassing Safety Features
Subzero Temperatures

- New trend is to super cool the product to separate plant oils to further refine the product
- Most equipment is designed for use from -20°F to 125°F
- Some end users assume they can use dry ice solutions to supercool to temperatures of -100°F
- Equipment must be specially designed for use at these temperatures

Leads to:
- Elastomer Failures
- Valve Failures
- Clamp Failures
Subzero Temperatures
Improper Assembly
Improper Assembly
Operating Procedures

- Some procedural issues can be determined during a field inspection
- SOPs should be reviewed by a third party professional
SOP Review

Ensures that:

- Operator is not deviating from the OEM instructions
- All safety checks are addressed
- Waste Disposal is addressed
- Appropriate detail is included in the SOPs
- Emergency situations are addressed
- Periodic Maintenance is addressed
Facility in Denver, CO
Accident in Denver, CO

- Clamp Nuts were stripped due to overtightening
- Clamp Bolts/Nuts had not been replaced since original purchase
- Extractor lid blew off at 30 psig (12” Diameter lid)
- Hazardous exhaust system not sufficient
- No ignition (whew!)
- SOPs had not been properly reviewed
- Equipment had not been field verified
- OEM did not list torqueing specs in manual
- Operator had not received proper training and didn’t know what a Torque wrench was
- Force of Lid at launch was between 2800 and 3400 LBS
Torqueing Procedures

- All pressure closures are required to be torqued to a specific value in order to properly seal the system
- Includes:
  - Sanitary Clamps
  - Flanges
- Ensures the bolts/nuts are not overtightened (stripped)
Sanitary Clamps
Is it possible to use LPG Safety?

- Yes, of course.
- What’s required:
  - Proper Equipment
  - Proper Operating Procedures
  - Proper Facility Safety
  - Proper Training
Questions?