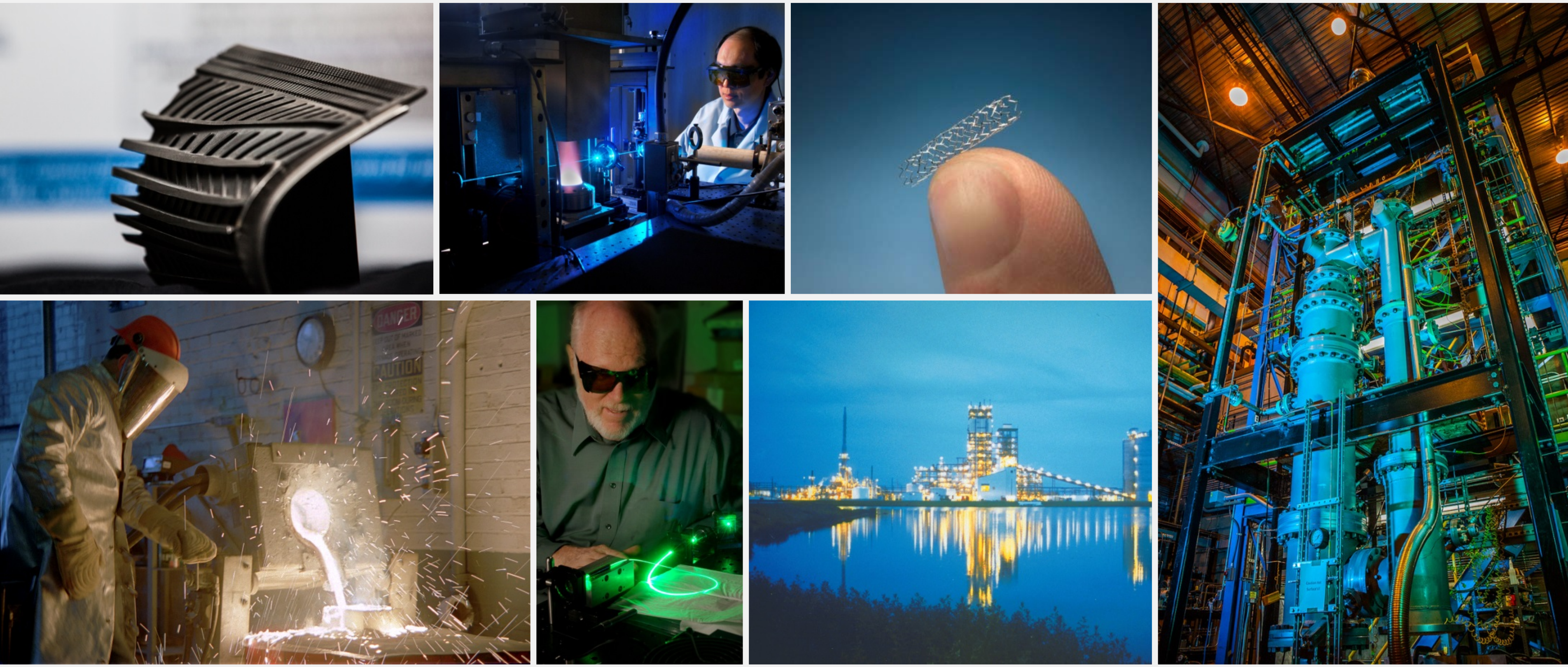
Driving Innovation ♦ Delivering Results



Guidelines for Parameter Measurements in Laboratory-Scale Post-Combustion Research Efforts

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NETL, Systems Engineering and Analysis
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August 8, 2016

U.S. DEPARTMENT OF ENERGY | National Energy Technology Laboratory

Objectives

- **Identify the minimal requisite parameters that should be measured by a technology developer through laboratory investigations to effectively support post-combustion CO_2 capture system studies**

The focus of this work is on laboratory-scale testing; however, the information identified relates to bench-scale, prototype-scale, and pilot-scale testing

Systems Analysis Levels

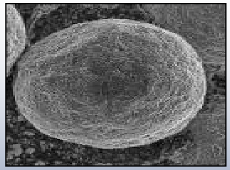
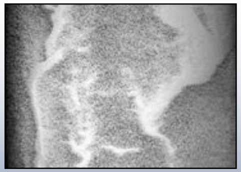
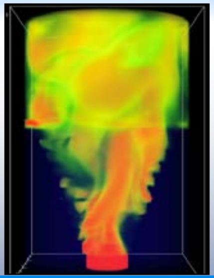
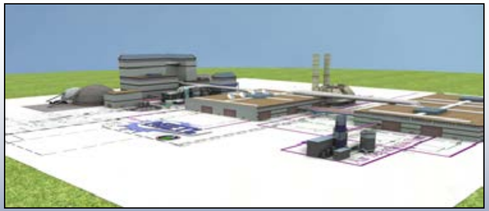
- **Pre-screening**
  - Initial engineering judgement as to availability of sufficient information to conduct a screening analysis; Identify data gaps and feasibility issues
  - Results:
    - Performance feasibility issues with the proposed process concept
    - Identities of missing test parameter data
- **Screening**
  - Engineering judgement regarding potential of subject technology to meet relevant NETL R&D goals with further development
  - Results:
    - List of key performance parameters having large sensitivity
    - Estimated commercial operating conditions (informs continued testing)
    - Current technology PC plant efficiency, and comparison to conventional technology and judgement of cost feasibility

Systems Analysis Levels (con't)

- **Commercial TEA**
  - More rigorous and comprehensive engineering judgement of potential to meet relevant NETL R&D goals with further development
  - Scope:
    - Commercial process performance and cost data are assumed to be available including estimates of Contact Material life and makeup cost
    - Develop independent plant M&E balances and plant performance projections
    - Review available commercial equipment cost information and cost projections for commercial Contact Material
  - Results:
    - Develop independent plant performance and cost estimates
    - Identities of any technology barriers to achieve the relevant NETL technology goals
    - Recommend options for addressing the barriers

Computational Tools for Accelerating Development

CCSI
Carbon Capture Simulation Initiative

Rapidly synthesize optimized processes to identify promising concepts → Better understand internal behavior to reduce time for troubleshooting → Quantify sources and effects of uncertainty to guide testing & reach larger scales faster → Stabilize the cost during commercial deployment

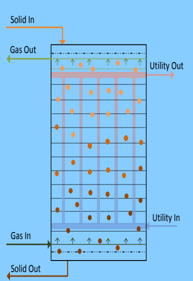
National Labs

Academia

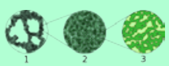
Industry

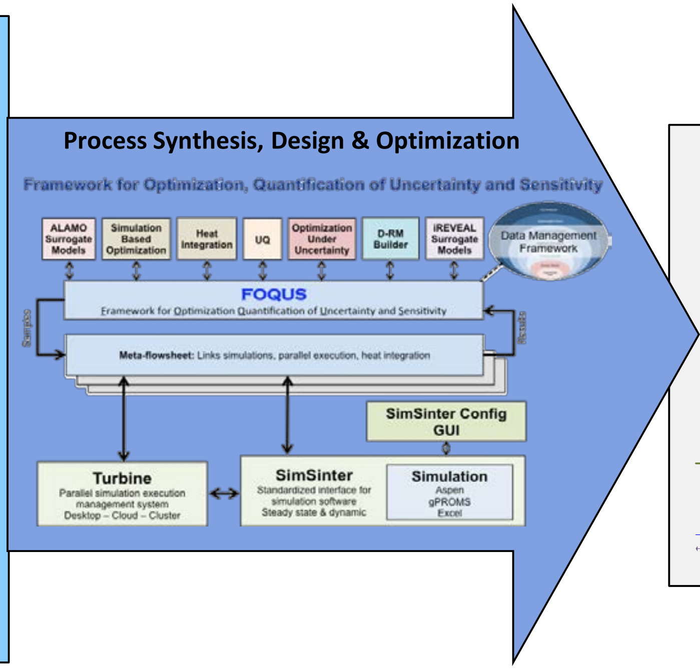
Computational toolset to accelerate development and scale-up

Process Models

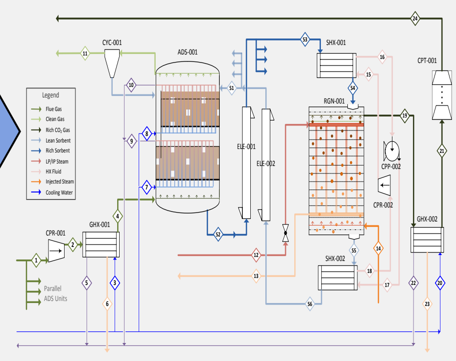


Basic Data Submodels

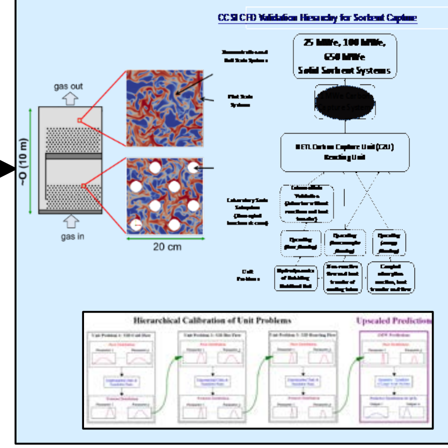




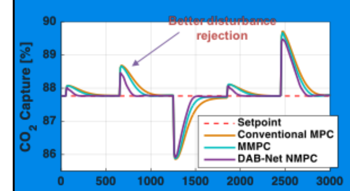
Carbon Capture Process



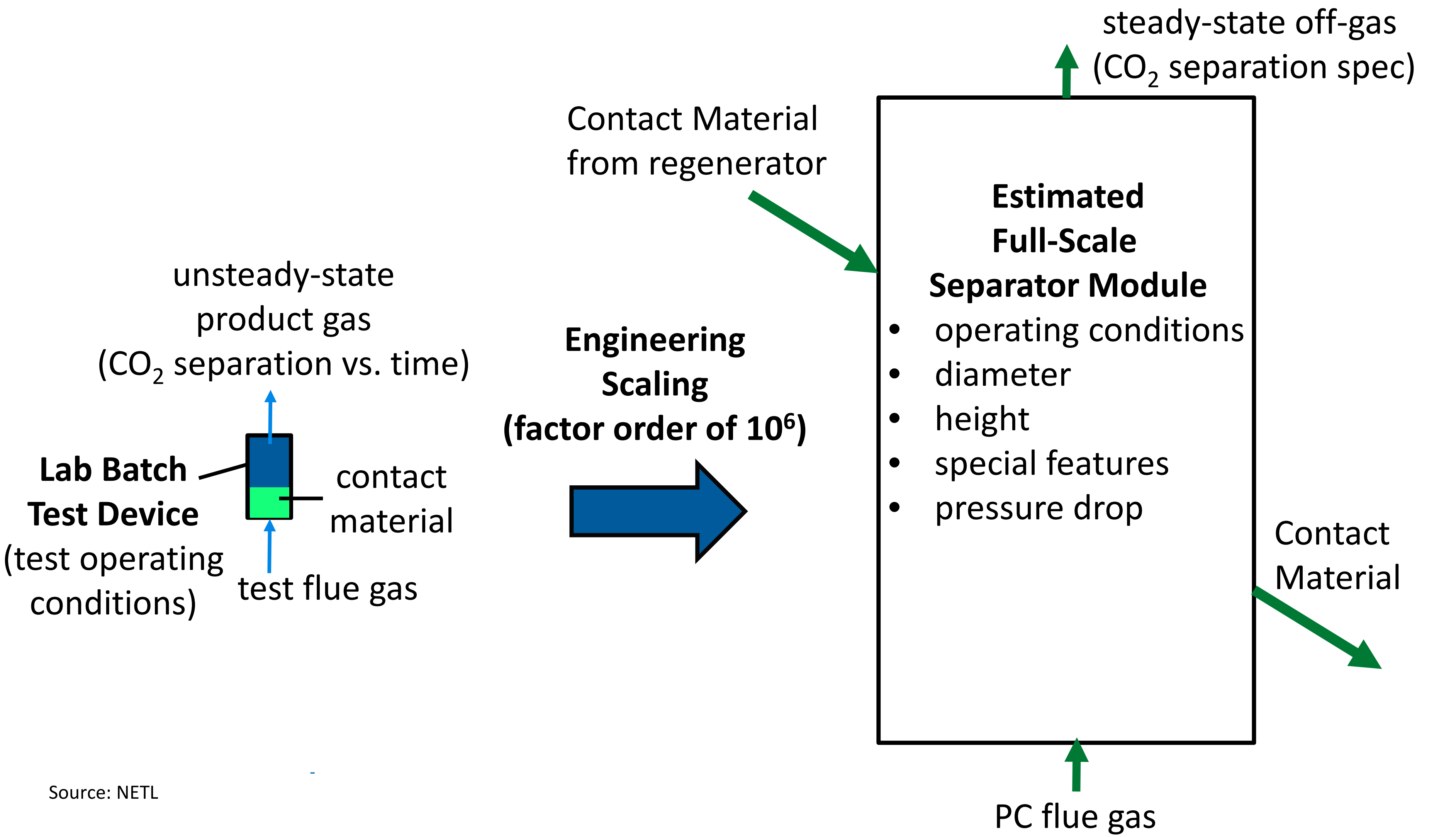
CFD Device Models



Process Dynamics and Control



Transformation of Lab Information to Commercial Estimates



Source: NETL

Contact Material Alternatives

- **Physical Adsorbent**
- **Chemical Sorbent**
- **Solvent** ★
- **Membrane**
- **Refrigerated Medium**

Laboratory-Scale Test Equipment Features

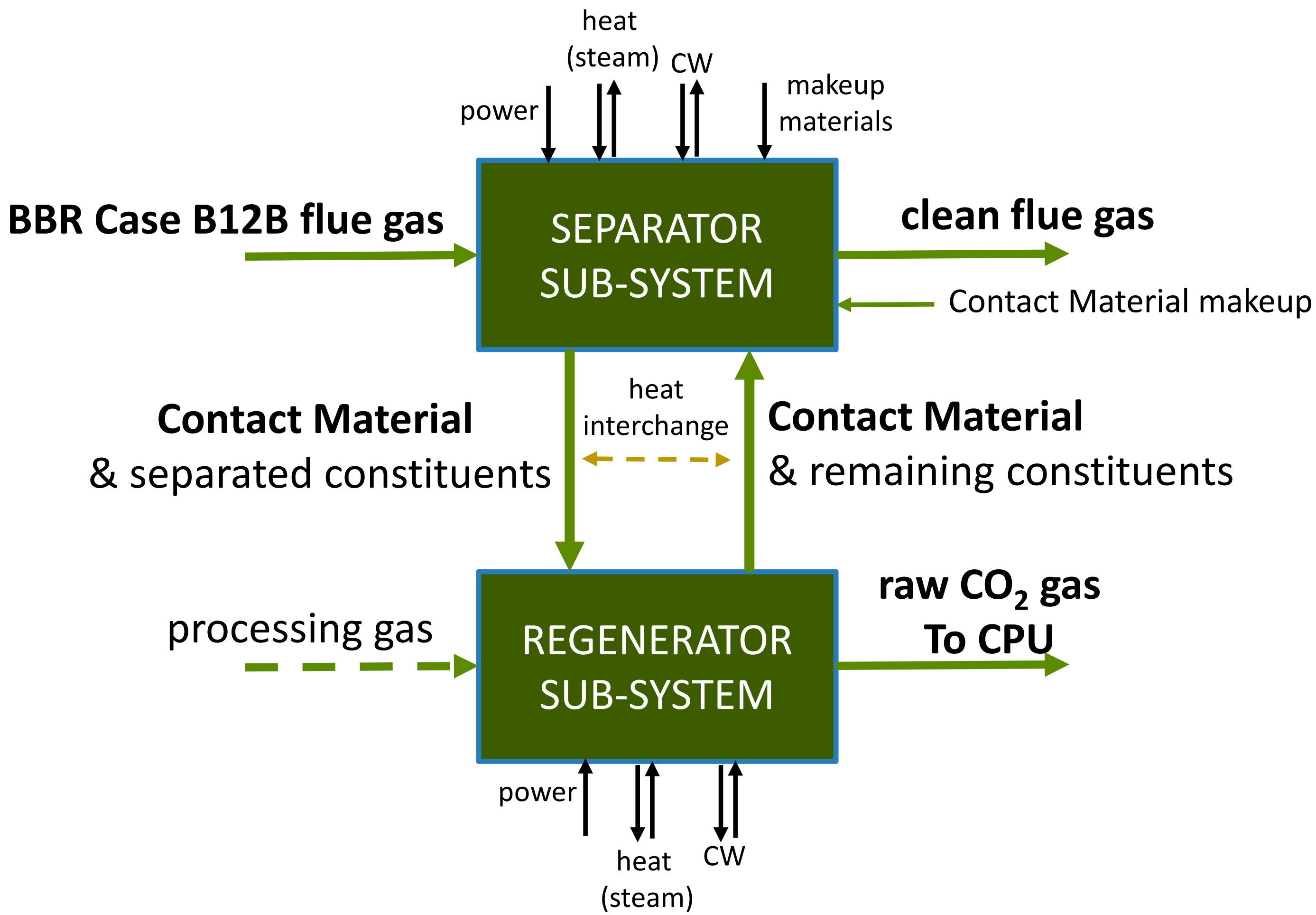
- **Small sample of Contact Material and simulated flue gas applied**
- **Unsteady state, batch, or semi-batch operations**
- **Equipment has large Surface Area/Volume ratio (large wall-effects)**
- **Heat losses large and complicate understanding of results**
- **Tests revolve step-wise between CO_2 separation and Contact Material regeneration**
- **Test equipment not integrated with other process systems**
- **Selection of best Contact Material characteristics, separation and regeneration operating conditions, and equipment features not resolved**
- **No meaningful information on commercial Contact Material makeup rate or replacement cost generated**
- **No meaningful information on commercial equipment costs generated**

Note: Most of these characteristics will persist at bench-scale, prototype-scale, and even pilot-scale development phases

Required Lab-Test Contactor Characteristics

- **The lab-test contactor can have any configuration that provides a contact environment between the Contact Material and the flue gas (separator) or processing gas (regenerator) that attempts to represent the local contact environment of a full-scale, commercial contactor**
- **The closer the lab-contactor operating conditions, contacting environment, and Contact Material characteristics are to the full-scale situation, the less uncertainty will exist in the engineering scaling of the contactor performance and cost to full scale**
- **Even at best, lab-testing data is likely to result in great uncertainties in engineering performance estimates, and equipment design and cost scaling**
- **These uncertainties will be reduced, but will still be significant in subsequent bench-scale, prototype-scale and pilot-scale testing**

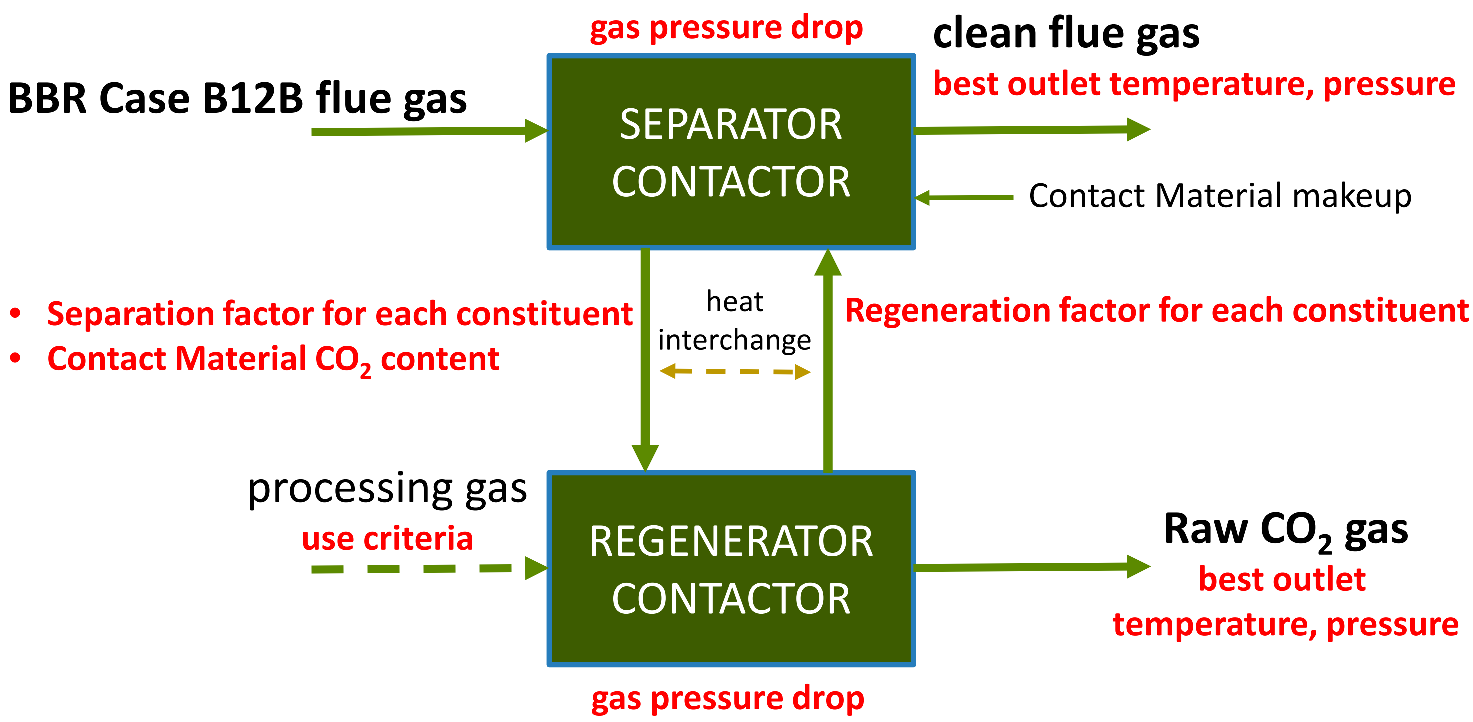
General Structure of CO_2 Separation System



Source: NETL

Information Needed to Estimate PC Plant Efficiency/Capture Process Power Factor

Items in red need to be estimated from lab-test information

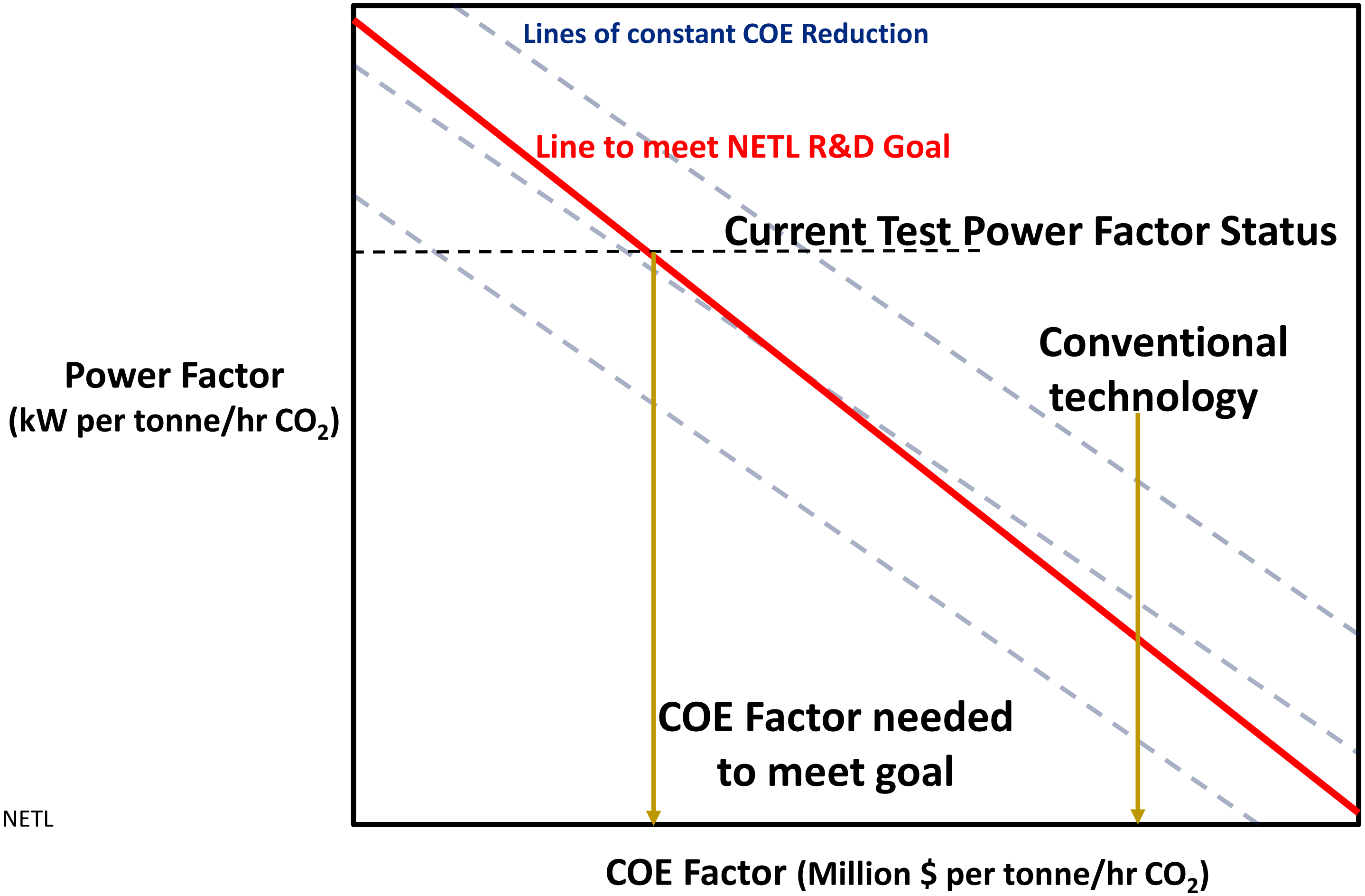


Source: NETL

Information Needed for PC Plant Performance and Cost Estimation

- **For PC Plant Efficiency: Need to estimate the <u>CO_2 separation process Power Factor</u>**
    1. Auxiliary power
    2. Heating duty and heating temperature
    3. Cooling water duty
    4. Raw CO_2 gas composition and pressure
- **For PC Plant COE: Need to estimate the <u>CO_2 separation process COE Factor</u>**
    1. Materials consumption rates and materials replacement prices
    2. Capital investment for process equipment

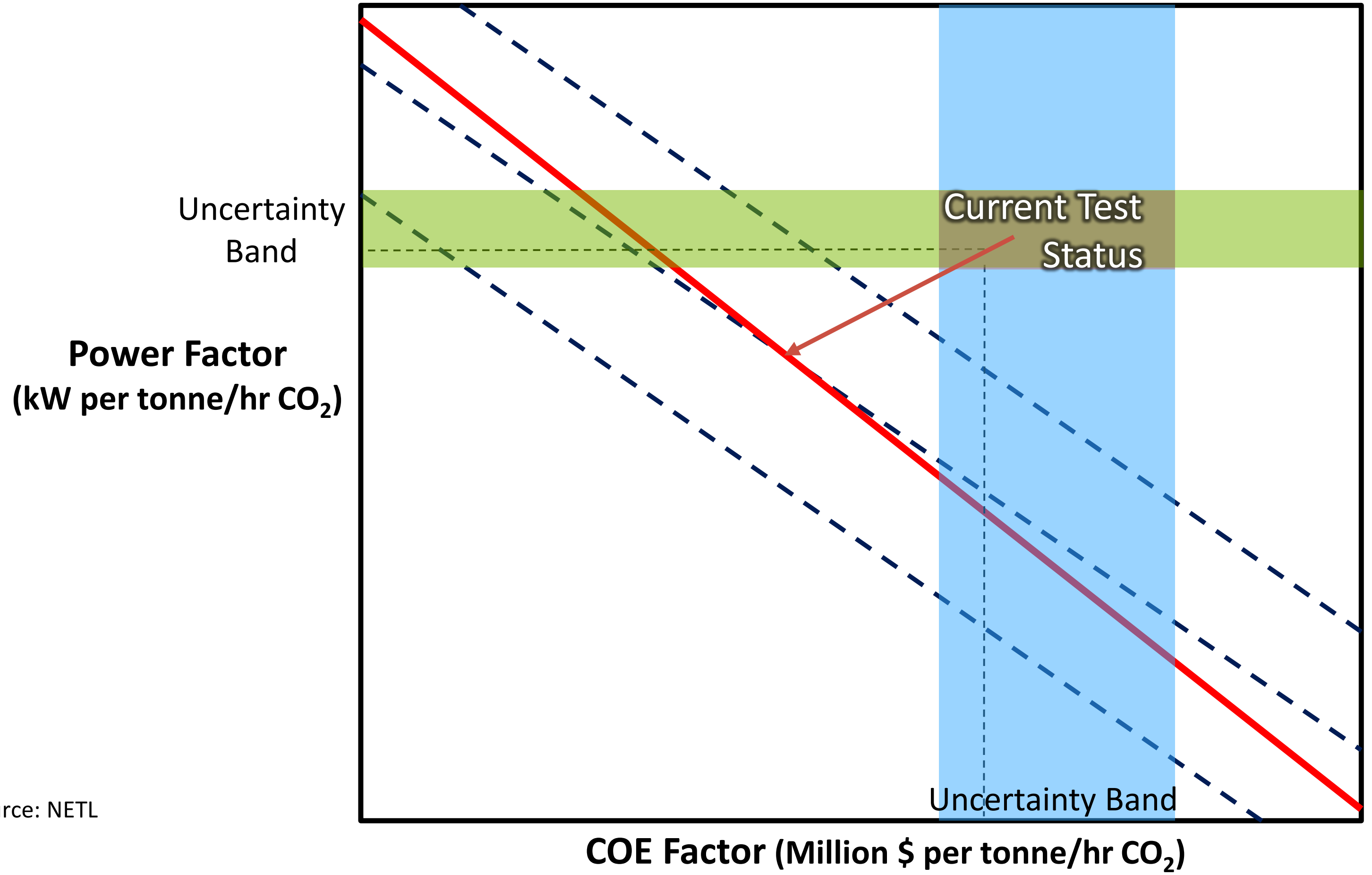
Lab-Test Data Screening Assessment Approach: Methodology Plot



Source: NETL

The estimated Power Factor is used to estimate the COE Factor needed to meet the NETL COE reduction goal; judged relative to conventional technology COE Factors

Lab Test Data Screening: Methodology Plot with Uncertainties



Source: NETL

Iterative testing and evolution to larger test scales reduces the uncertainty bands and may provide a trajectory to meet the NETL COE reduction goal

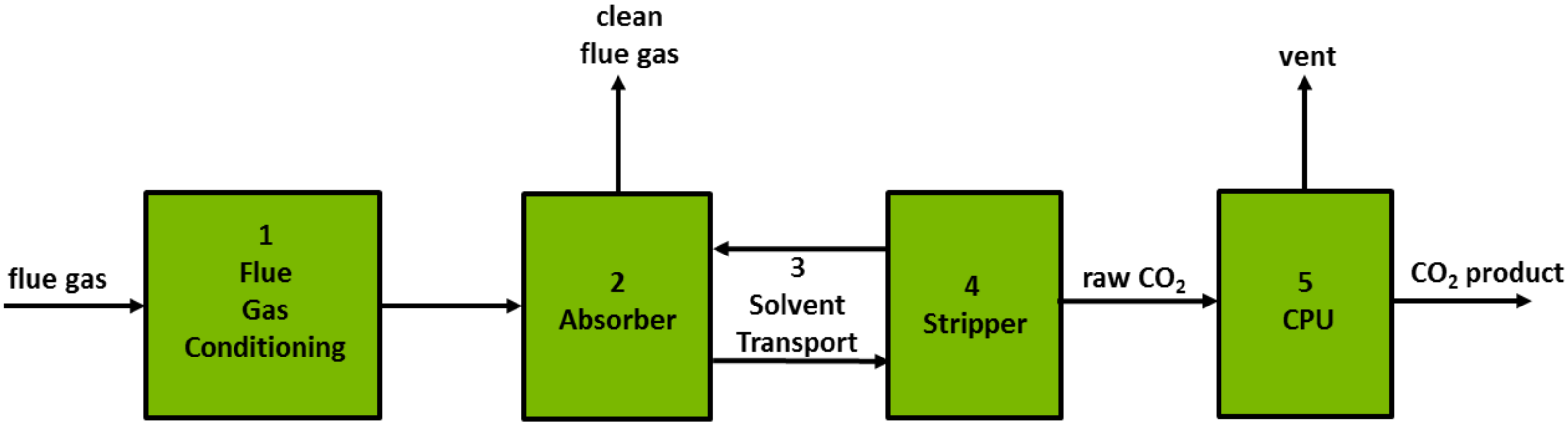
Background and Minimum Information for Solvent-Based Process

Characteristics of CO_2 Solvents

- May be physical solvent (physical absorption), chemical solvent (chemical absorption), or combination
- Chemical solvent is normal needed for atmospheric-pressure applications
- May be an aqueous solution or non-aqueous solution
- Solvent stripping can be promoted by temperature increase and/or pressure reduction
- Solvents generally absorb CO_2 with small amounts of other flue gas constituents
- Solvent performance may degrade with extended flue gas constituent interaction or thermal exposure
- Rate of CO_2 separation decreases as solvent CO_2-loading increases
- Solvent-CO_2 equilibrium behavior should not limit the CO_2 separation efficiency, but may limit absorption and stripping rates
- Effective gas-liquid contacting can be achieved in a variety of contactor types: packed column, tray column, spray column, bubble column, stirred tank, jet loop tank, venture ejector tank, etc.

Primary CO_2 Absorption Process Steps

1. **Flue gas conditioning (e.g., temperature, pressure, composition)**
2. **CO_2 Absorption**
3. **Solvent transport between absorber and stripper**
4. **CO_2 stripping**
5. **Raw CO_2 compression and purification (CPU)**



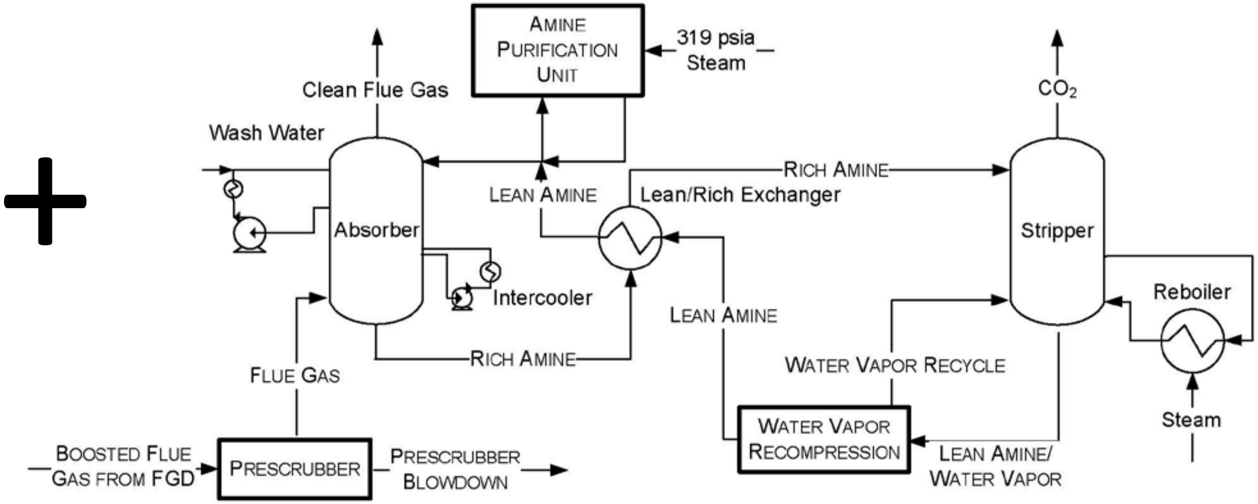
Source: NETL

Solvent Minimum Information for Screening

A. Development focus

+

Conventional process



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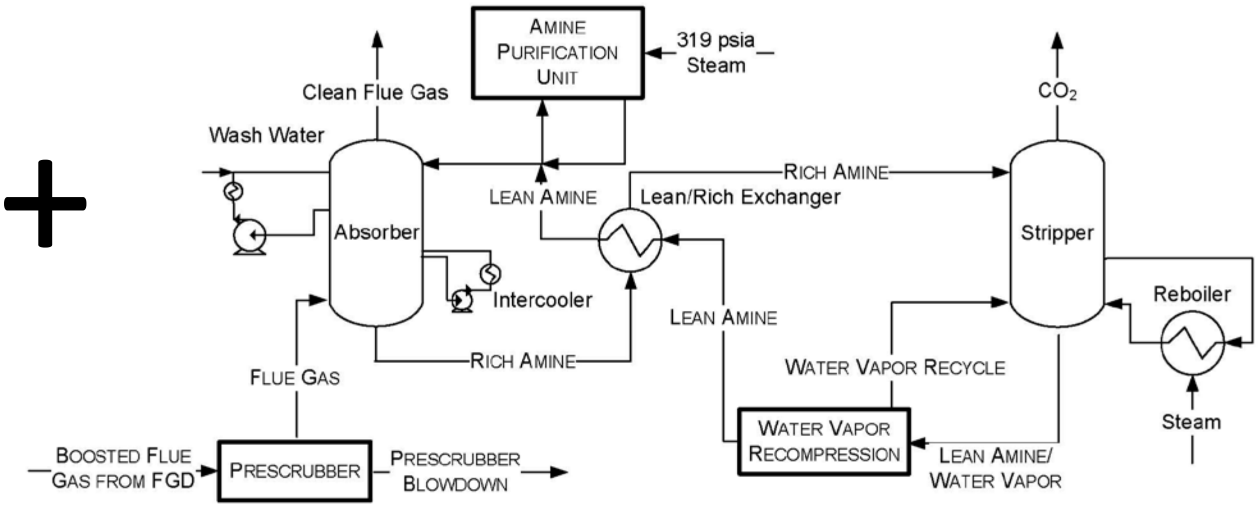
Conventional contactor equipment

Source: NETL

Solvent
Minimum Information for Screening

A. Development focus

Conventional process



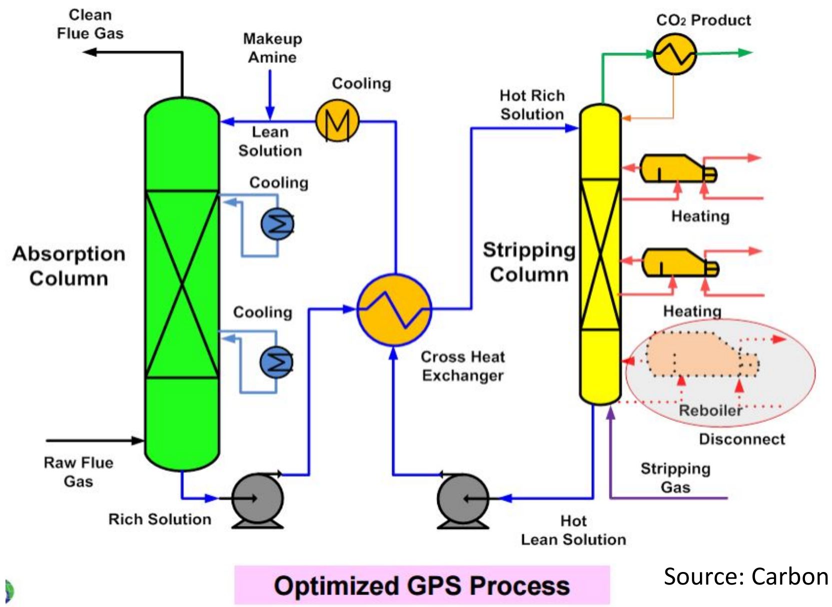
Novel contactor equipment

Source: NETL

Solvent
Minimum Information for Screening

A. Development focus

+



Source: Carbon Capture Scientific

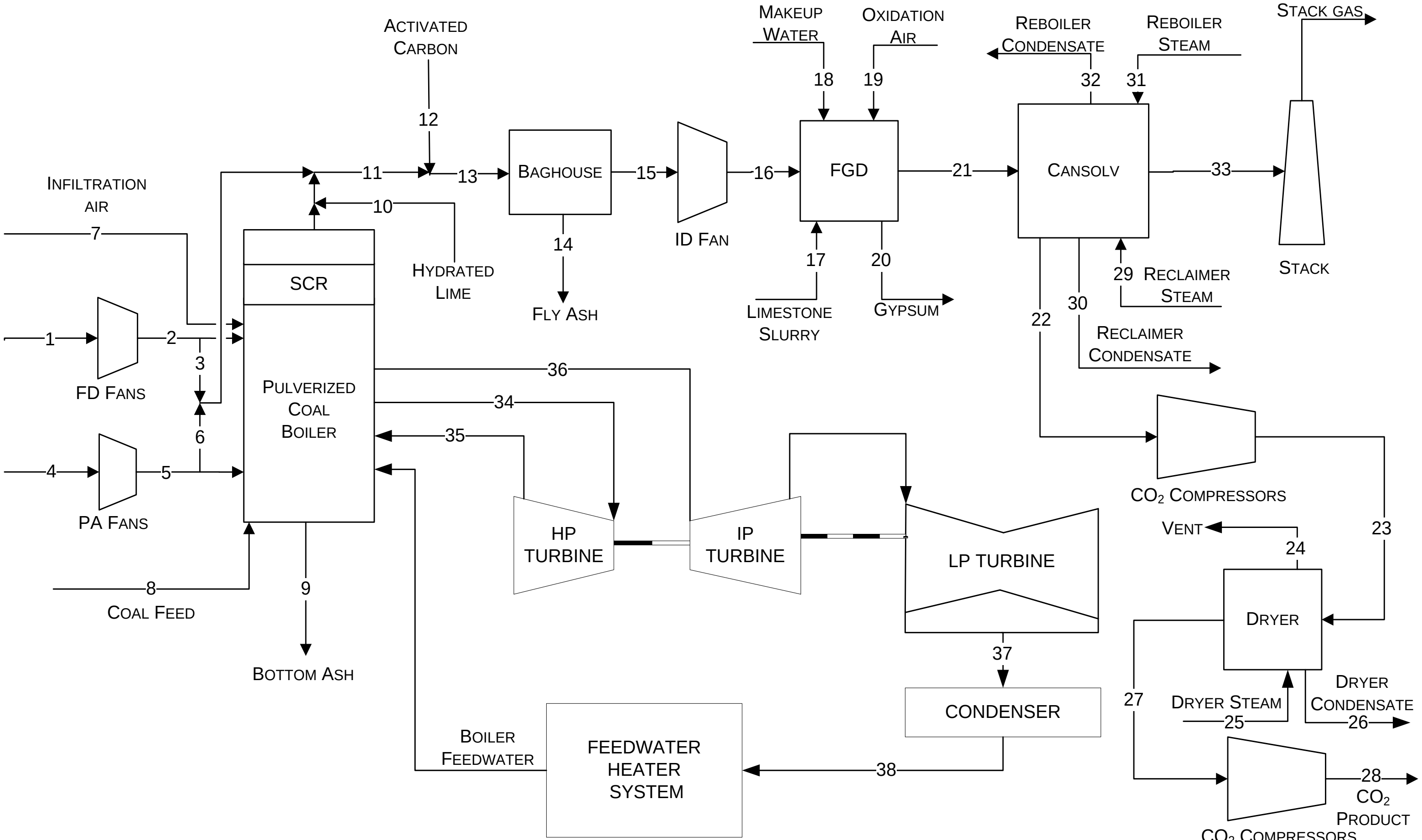
+

Novel contactor equipment

Source: NETL

Solvent
Minimum Information for Screening

B. Proposed commercial process flow diagram



Note: Block Flow Diagram is not intended to represent a complete material balance. Only major process streams and equipment are shown.

Source: NETL

Solvent
Minimum Information for Screening

C. Proposed commercial absorber-stripper process description

- Considerations for inclusions:
  - Absorber-stripper process concept description
  - Vessel temperature control scheme description
  - Solvent circulation system description
  - Solvent CO_2 separation system conventional equipment types and functions

Solvent
Minimum Information for Screening

D. Proposed absorber and stripper contacting descriptions

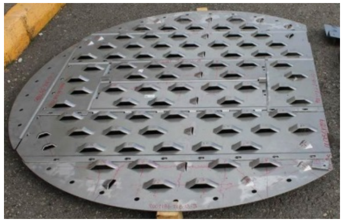
Packed Column

Random

Structured

Staged, Tray Column

Sieve



Valve

Bubble Cap

Other Approaches/Special Features

- Spray Tower
- Bubble Column
- Others

Source: Sulzer

Solvent
Minimum Information for Screening

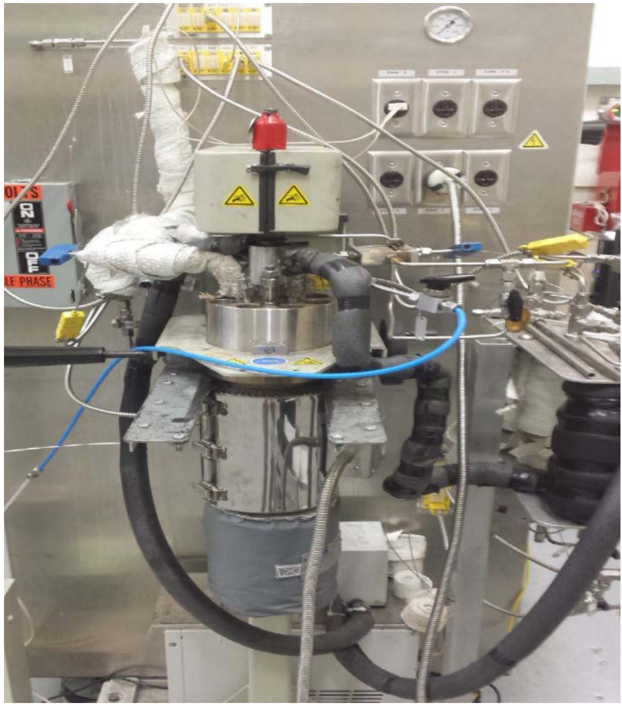
E. Test-solvent properties (based on laboratory measurement or literature data)

- Composition
- Chemical solvent absorber and stripper overall reaction schemes
- Density
- Specific heat
- Thermal stability vs. temperature
- Vapor pressure vs. temperature
- Heats of reaction/absorption & stripping
- Viscosity vs. temperature and CO_2 loading
- Chemical solvent-CO_2 equilibrium conversion vs. temperature and pressure
- CO_2 (and other flue gas constituents) solubility vs. temperature and pressure

Solvent
Minimum Information for Screening

F& G. Test-absorber/stripper vessel characteristics

- Test-absorber/stripper design approach:
  - Mimic commercial absorber stage(s) features at lab scale
  - Use non-commercial features, but provide controllable, intimate gas-solvent contact
- Gas-solvent contacting description (e.g., bubble tank, stirred tank, packed bed)
- Continuous or batch?
- Internals characteristics (e.g., packing type, packing size, tray type)
- Novel contactor features description (e.g., mixing jets)
- Vessel measurements
  - inner cross-sectional area
  - height (gas inlet to gas outlet)
- Vessel temperature-control design description
- Inlet gas distribution design description
- Solvent feed distribution design description



Source: NETL

Solvent
Minimum Information for Screening

H& I. Test absorber/stripper vessel operating conditions

- Gas composition
  - Absorber: Feed Gas (CO_2, H_2O, O_2, N_2, Ar)
  - Stripper: Stripping gas (steam, etc.)
- Gas inlet condition
  - Absorber: Feed volumetric flow rate, temperature, pressure
  - Stripper: Stripping gas volumetric flow rate, temperature, pressure
- Solvent condition
  - Volumetric feed rate
  - Solvent feed/inlet temperature
  - Solvent initial weight or volume in vessel
  - Feed or loaded solvent exposure history (e.g., time, number cycles)
  - Feed or loaded solvent CO_2 loading

Solvent
Minimum Information for Screening

J& K. Test absorber/stripper vessel test results

- Results needed
  - Outlet gas composition, temperature, pressure vs. time
  - Cooling rate vs. time – absorber
  - Heating rate vs. time – stripper
- Test performance parameters extracted from results
  - Absorber/stripper pressure drop
  - CO_2 loading vs time (and all other reacted species)
  - Absorption factor / desorption factor vs. time
  - Fresh solvent overall absorption/desorption rate constants
  - Overall absorption/desorption rate constant degradation with time or cycle number

Solvent
Minimum Information for Screening

E. <u>Test</u>-Solvent SO_2 Tolerance Test Results

- Focus on 400 ppmv SO_2
- Typical concentration of SO_2 in post FGD bituminous flue gas
- Determine if polishing to ~ 1 ppmv is needed

- Test data needed:
  - SO_2 fraction absorbed with 40 ppmv SO_2 gas content
  - SO_2 fraction desorbed after 40 ppmv SO_2 exposure
  - Solvent performance degradation observed after 40 ppmv SO_2 exposure

Basic Data Requirements for CCSI Analysis

- **Solvents**
  - Heat of Absorption, f(T, x_i)
  - Vapor-Liquid Equilibrium Data
    - over relevant $p_{y,i}$,T, x_i ranges
  - Kinetic Data, f($p_{y,i}$, T, x_i)
    - Including speciation
  - Mass Transfer Data
    - from wetted wall column, bench scale system
  - Viscosity, f(T, x_i)
  - Heat Capacity, f(T, x_i)
  - Density, f(T, x_i)
  - Surface Tension, f(T, x_i)
  - Vapor Pressure, f(T, x_i)
  - Thermal Conductivity, f(T, x_i)
  - Hydraulic Data for specific packing

Solvent Capture Technology Check List Information and Data Provided

A	**Development focus**
B	**Proposed commercial process block flow diagram**
C	**Proposed commercial solvent absorber-stripper process description**
D	**Proposed commercial absorber and stripper contacting descriptions**
E	**Test-solvent properties**
1	Composition
2	Chemical solvent absorber and stripper overall reaction schemes
3	Density
4	Specific heat
5	Thermal stability vs. temperature
6	Vapor pressure vs. temperature
7	Heats of reaction/absorption and desorption
8	Viscosity vs. temperature and CO_2 loading
9	Chemical solvent equilibrium CO_2 conversion vs. temperature and pressure
10	CO_2 (and other flue gas constituents) solubility vs. temperature and pressure

Solvent Capture Technology Check List Information and Data Provided

F	**Test-absorber vessel characteristics**
1	Test-absorber design approach
2	Gas-solvent contacting description
3	Operation: continuous or batch
4	Internals characteristics
5	Novel contactor features description
6	Vessel inner cross-sectional area
7	Vessel height (gas inlet to gas outlet)
8	Vessel temperature-control design description
9	Inlet gas distribution design description
10	Solvent feed distribution design description

Solvent Capture Technology Check List Information and Data Provided

G	**Test-stripper vessel characteristics**
1	Test-stripper design approach
2	Gas-solvent contacting description
3	Operation: continuous or batch
4	Internals characteristics
5	Novel contactor features description
6	Vessel inner cross-sectional area
7	Vessel height (gas inlet to gas outlet)
8	Vessel temperature-control design description
9	Inlet gas distribution design description
10	Solvent feed distribution design description

Solvent Capture Technology Check List Information and Data Provided

H	**Test-absorber operating conditions**
1	Feed gas composition (includes all major flue gas species)
2	Feed gas flow rate
3	Feed gas inlet pressure
4	Feed gas inlet temperature
5	Solvent feed rate
6	Solvent initial height or weight in vessel
7	Feed or loaded solvent exposure history
8	Feed or loaded solvent CO_2 loading
I	**Test-stripper operating conditions**
1	Stripper gas composition
2	Stripper gas flow rate
3	Stripper gas inlet pressure
4	Stripper gas inlet temperature
5	Solvent feed rate
6	Solvent initial height or weight in vessel
7	Solvent inlet temperature
8	Fed or loaded solvent exposure history
9	Fed or loaded solvent CO_2 loading

Solvent Capture Technology Check List Information and Data Provided

J	**Test-absorber test results**
1	Outlet gas composition vs. time (for all adsorbed species)
2	Outlet gas temperature vs. time
3	Outlet gas pressure vs. time
4	Cooling rate vs. time
K	**Test-Stripper Test results (for status tests)**
1	Outlet gas composition vs. time (for all adsorbed species)
2	Outlet gas temperature vs. time
3	Outlet gas pressure vs. time
4	Heating rate vs. time
L	Test-solvent SO_2 tolerance test results

Minimum Information for Physical Sorbent Screening Summary

Category	Physical Sorbent
A thru D – Commercial Concept Considerations	Novel technology description (process, sorbent, contactor equipment) Block flow diagram PSA, VSA, TSA, etc Fixed, Moving, Stagged bubbling fluidized beds, etc
E – Test Sorbent Properties	Sorbent composition, particle properties (mean dia, size distribution, shape, bulk density, voidage), adsorption/desorption equilibrium isotherms, heat of adsorption, specific heat
F & G – Test Adsorber/Desorber Vessel	Design approach, Contact Type (bed type), Operating mode (continuous feed & drain, batch), Geometric measurements, cooling & heating control designs
H & I – Test Adsorber/Desorber Operating Conditions	Gas compositions (feed or purge), volumetric flow rates, pressure, temperature, sorbent feed rate, initial bed height or weight, exposure history (number of cycles), adsorbed gas species content (absorber and desorber)
J & K – Test Adsorber/Desorber Test Results	Required Test Results: Gas composition (all adsorbed species), outlet gas temp & pressure, cooling/heating rate all as a function of time, final vessel adsorbent weight Extracted Parameters: pressure drop, adsorbent CO_2 content and gas species adsorption/desorption factors vs. time, Rate constants
L – Test Adsorbent SO_2 Tolerance	SO_2 absorbed/desorbed with 40 ppmv exposure, performance degradation after exposure

Minimum Information for Chemical Sorbent Screening Summary

Category	Physical Sorbent
A thru D – Commercial Concept Considerations	Novel technology description (process, sorbent, contactor equipment) Block flow diagram Regeneration, circulation schemes Bubbling/circulating fluidized beds, etc
E – Test Sorbent Properties	Sorbent composition, particle properties (mean dia, size distribution, shape, bulk density), sorbent-CO_2 (and other reacting species) equilibrium gas composition as a function of operating conditions, heat of reaction, specific heat
F & G – Test Reactor/Regenerator Vessel	Design approach, Contact Type (bed type), Operating mode (continuous feed & drain, batch), novel features (internal stirring, etc), Geometric measurements, cooling/heating/ heat loss control designs
H & I – Test Reactor/Regenerator Operating Conditions	Gas compositions (feed or reaction gas), volumetric flow rates, pressure, temperature, sorbent feed rate, initial bed height or weight, exposure history (number of cycles), adsorbed gas species content (or extent utilization)
J & K – Test Reactor/Regenerator Test Results	Required Test Results: Gas composition (all reactive/adsorbed species), outlet gas temp & pressure, cooling/heating rate all as a function of time, final vessel adsorbent weight Extracted Parameters: pressure drop, adsorbent CO_2 content and gas species separation factors vs. time, Rate constants
L – Test Adsorbent SO_2 Tolerance	SO_2 absorbed/desorbed with 40 ppmv exposure, performance degradation after exposure

Basic Data Requirements for CCSI Analyses

- **Sorbents**
  - Adsorption equilibrium, $f(p_{y,i}, T, x_i)$
    - All species over relevant conditions
  - Heat of Adsorption for all species, $f(T, x_i)$
    - CO_2 and H_2O minimum
  - Heat Capacity, $f(T, x_i)$
  - Adsorption/Desorption Kinetics, $f(p_{y,i}, T, x_i)$
    - All species over relevant conditions
  - Thermal Conductivity, $f(T, x_i)$
  - Density, $f(T, x_i)$
  - Particle Size Distribution
  - Sphericity

Minimum Information for Membrane Screening Summary

Category	Physical Sorbent
A thru C – Commercial Process Considerations	Novel technology description (process, membrane material, membrane geometry, flow configuration) Block flow diagram, Driving force scheme
D – Commercial Membrane Material and Geometry	Material description Geometry (tubular, spiral wound, planar, hollow fibers, other) Other special features
E – Commercial Membrane Flue Gas-Permeate Configuration	Co-current / Counter-current / Cross-flow Flue gas-permeate flow location (i.e. flue gas flow inside membrane tubes)
F – Test Membrane Characteristics and Properties	Test membrane design approach Geometry tested, dimensions Material bulk density, nature of material permeation driving force
G – Test Membrane Vessel Characteristics	Vessel dimensions and Flow configuration (counter-, co-, cross-) # of parallel sections and their arrangement
H – Test membrane operating conditions	Feed gas composition, volumetric flow, pressure, temperature Sweep gas composition, volumetric flow; Permeate gas pressure, # hours operated
I – Test Membrane Vessel Test Results	Flue gas outlet (and/or permeate) composition and volumetric flow vs. time Determines gas species time-averaged permeance and permeance degradation
J – Test Membrane SO_2 Tolerance	SO_2 absorbed/desorbed with 40 ppmv exposure, performance degradation after exposure

Minimum Information for Refrigerated Medium Screening Summary

Category	Physical Sorbent
A thru D – Commercial Concept Considerations	Novel technology description (process, refrigeration medium, contactor equipment) Block flow diagram Flue gas conditioning, refrigerated medium generation, condensation process (liquid or solid CO_2)
E – Test Device Characteristics	Description of vessels and refrigerated medium use for all process components (condenser, separator, melter)
F – Test Device Operating Conditions	For each device component: Feed composition (gas/liquid/solid), pressure, temperature, flow rate Solid CO_2 characteristics (particle size, density) Refrigerated medium flow rate Companion gas flow rate, temperature, composition
G – Test Results	Data lists for each device component includes: CO_2 solid particle/droplet size distribution and mean diameter, shape, density Condensed-flue gas multicomponent equilibrium vapor/liquid/solid vapor pressures over range of operating conditions
H – Test Adsorbent SO_2 Tolerance	SO_2 condensed with 40 ppmv exposure, performance degradation after exposure

Summary Minimum General Information Needed for Screening Analysis

- **Development focus**
- **Proposed commercial process flow diagram**
- **Proposed commercial process concept description**
- **Proposed commercial contacting concept description**
- **Test-Contact Material properties (for status Contact Material)***
- **Test-separator and -regenerator characteristics**
- **Test-separator and -regenerator operating conditions (for status tests)****
- **Test-separator and -regenerator test results (for status tests)****
- **Test-separator and -regenerator Contact Material SO_2 tolerance test results****

* *Requires property measurements and/or literature information*

** *Requires process measurements*

Notes:

- ***Status* represents the current state of development of the technology**
- ***The information needed to conduct a screening analysis differs significantly from the information needed to assess the quality of test data (test equipment and test procedures, analytical equipment, data reproducibility, etc.)***

Lab-Test Measurement Selection Considerations

- **Applicability (will it function at given stream conditions?)**
- **Accuracy**
- **Precision**
- **Stability (i.e., measured steady-state value changes with time)**
- **Availability (i.e., periodically not operational)**
- **Response time (e.g., time-fluctuating composition cannot use chromatograph effectively)**
- **Multicomponent capability (e.g., multiple gas species in flue gas may need separate instrument for each constituent)**
- **Operational difficulties (e.g., frequent calibration/cleaning needed)**
- **Expense (investment, operating cost)**

Summary and Conclusions

- Minimum information lists for TEA-like screening analyses have been developed for five types of CO_2 separation Contact Materials
- The lists focus on laboratory-scale testing, but also relate directly to bench-scale, prototype-scale, and pilot-scale testing
- The minimum information list provides a concise way to identify technology data gaps
- Few CO_2 capture technology development projects report/generate the minimum information needed
- Analyses should be applied iteratively with testing to provide rational and more rapid go/no-go assessment of technology prospects

Next Steps

- **Further integration/development of data requirements for CCSI-like systems analysis**
- **Incorporate input from laboratory researchers on information requested**
  - Perspectives on
    - Appropriate/best techniques to obtain data
    - Difficulty/cost of some data acquisition
    - Technology types not covered
  - Other thoughts/concepts to make documents more comprehensive
- **Finalize report with checklists for posting on NETL website**
- **Possibility to expand to other areas of R&D**