

NETL TEST PROTOCOL TESTING OF HYDROGEN SEPARATION MEMBRANES

DOE/NETL-2008/1335



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NETL Test Protocol – Testing of Hydrogen Separation Membranes

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Objective

The overall objective of this testing is to develop membrane technologies that achieve target performance of hydrogen separation membranes for use in a gasification system process. NETL needs (1) to know whether developers are approaching/achieving Hydrogen Program Technical Targets, (2) to be able to compare results on an “apples to apples” basis, and (3) to clearly state expectations to contractors. Results may be of value in deciding on down-selections. Another objective is to determine the suitability of each membrane and to assess the compatibility of each membrane’s optimum operating conditions for use in plant hardware as it currently exists.

This test protocol provides background information on gasification systems, descriptions of membrane tests, the prescribed procedures for tests, and the analyses and reporting to be completed. Very briefly, a series of tests is described beginning with no gasification contaminants to determine ideal membrane performance, followed by tests with sulfur, corresponding to sulfur levels for membranes aimed at different process configurations. Should any membrane not perform satisfactorily during the first two tests, the membrane shall be considered to be disqualified from further testing at larger scales and/or more stringent conditions. Future tests will be considered at higher contaminant levels and/or raw syngas streams.

Background

Advancements in hydrogen membrane separation technologies have the potential to reduce costs, improve efficiency, and simplify hydrogen production systems. Desirable characteristics of separation membranes are high hydrogen flux at low pressure drops; tolerance to contaminants, including sulfur species, CO, NH₃, chlorides, As, and Hg; mechanical strength; low cost; and operation at a range of system temperatures to provide versatility in location of membrane separators in the sequence of process operations. Many current hydrogen membrane technologies are at the research stage, but some have the potential to provide hydrogen purity above 99.99 %.

The DOE Hydrogen from Coal RD&D Plan¹ establishes Technical Targets for Hydrogen Separation Membranes that are necessary to meet the performance and cost goals of the program. The reader is referred to the RD&D Plan for rationale and more detailed descriptions of the Targets. Note: For the convenience of readers, this document will quote temperatures in both °C and °F, pressures in common engineering units of psia, psig, and atmospheres, fluxes in standard cubic centimeters per minute per square centimeter of membrane area (sccm/cm²) and standard

¹ Hydrogen from Coal RD&D Plan, External Draft, Sept. 2007

cubic feet per hour per square foot of membrane area (SCFH/ft²), and gas compositions in molar percent and/or parts-per-million (ppm). The Technical Targets are as follows:

- **2010 Target**
 - Hydrogen flux – 200 SCFH/ft² (~ 100 sccm/cm²) @ 100 psi ΔP H₂ partial pressure. Standard conditions are 150 psia hydrogen feed pressure and 50 psia hydrogen sweep pressure.
 - Temperature – 300 to 600 °C (572 to 1112 °F).
 - Pressure performance – ΔP – Up to 400 psi.
 - Sulfur tolerance – 20 ppm.
 - CO tolerance – Yes.
 - Water-gas-shift (WGS) activity – Yes.
 - Hydrogen purity – 99.5 %.
- **2015 Target**
 - Hydrogen flux – 300 SCFH/ft² (~ 150 sccm/cm²) @ 100 psi ΔP H₂ partial pressure. Standard conditions are 150 psia hydrogen feed pressure and 50 psia hydrogen sweep pressure.
 - Temperature – 250 to 500 °C (482 to 932 °F).
 - Pressure performance – ΔP – 800 to 1000 psi.
 - Sulfur tolerance – >100 ppm.
 - CO tolerance – Yes.
 - WGS activity – Yes.
 - Hydrogen purity – 99.99 %.

Gasifier Process Conditions and Syngas Compositions

Hydrogen separation membranes may be used in a variety of locations in a gasification-based coal-to-hydrogen production process, depending on the capability of the membrane to withstand temperature and pressure conditions as well as variations in gas composition. The following discussion briefly outlines some of those configurations. The coal gasifiers currently under consideration for coal-to-hydrogen or co-production plants that produce electricity and hydrogen are listed in Table 1, along with typical temperature and pressure conditions and gas compositions at a point in the process downstream of the first stages of gas cooling and particulate cleanup. Figure 1 shows the main process units that the gas stream encounters prior to this point.

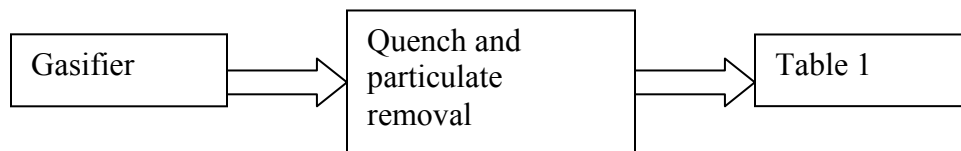


Figure 1 – Block Diagram of Syngas Process Units Corresponding to Table 1

The primary contaminants of the syngas are sulfur (H₂S and COS) and nitrogen compounds (NH₃), but there also may be small amounts of chlorides (such as HCl) and mercury. Total sulfur concentrations (typically most of the sulfur is H₂S) in gasifier raw gases are usually in the 1% range. Ammonia (NH₃) generally is in the 0.1 to 0.2% range. Mercury concentrations are difficult to measure, but are estimated to be approximately 50 parts-per-million by weight (ppbw) based on mercury content in most coals². Hydrogen chloride (HCl) concentrations may range from 50 to 500 ppm³, and these are a concern regarding corrosion in heat exchangers. The pressure and temperature conditions here would represent the harshest conditions to which hydrogen separation membranes would likely be exposed if used in conjunction with advanced shift reactor processes in the raw gas stream.

Table 1 – Gasifier Syngas Conditions and Compositions Upstream of Shift Reactors

Vendor/Gasifier	GE Energy Radiant ⁴	Conoco-Phillips E-Gas ⁵	KBR Transport Gasifier (TRIG) ⁶	Shell ⁷
Syngas Pressure (psia)	800	614	400+	565
Syngas Temperature [°C (°F)]	210 (410)	927 (1700)	260-371 (500-700)	260 (500)
Syngas Composition (mole %)	H ₂ 25.9 CO 26.7 CO ₂ 11.6 H ₂ O 33.6 CH ₄ 0.08 H ₂ S 0.56 COS 0.01 NH ₃ 0.13	H ₂ 26 CO 37 CO ₂ 14 H ₂ O 15 CH ₄ 4 H ₂ S 0.51 COS 0.00 NH ₃ 0.19	H ₂ 29.2 CO 34.3 CO ₂ 13.6 H ₂ O 18.9 CH ₄ 2.5 H ₂ S 0.056 COS ? NH ₃ 0.28 HCN 0.032	H ₂ 15.6 CO 30.7 CO ₂ 1.13 H ₂ O 48.3 CH ₄ 0.02 H ₂ S 0.43 COS 0.04 NH ₃ 0.18

² The Cost of Mercury Removal in an IGCC Plant, NETL Final Report, Sept. 2002, Parsons Corporation.

³ High Temperature Corrosion in Gasifiers, Bakker, W., Materials Research, 2004, v. 7, No. 1, pp. 53-59.

⁴ Data from NETL Cost and Performance Baseline for Fossil Energy Plants, Volume 1: Bituminous Coal and Natural Gas to Electricity Final Report (Bituminous Baseline [BB] Report), DOE/NETL-2007/1281, May 2007, Exhibit 3-33, Stream 9 upstream of shift reactor.

⁵ Pressure, temperature, and major gas species (H₂-CH₄) from NETL Gasification Overview Presentation, July 2007. Sulfur and ammonia values from Cost and Performance Baseline for Fossil Energy Plants, Volume 1: Bituminous Coal and Natural Gas to Electricity Final Report, DOE/NETL-2007/1281, May 2007, Exhibit 3-66, Stream 9 after shift reactors. Note – BB report does not show stream composition before shift reactor.

⁶ Data from Power Systems Development Facility Update on Six TRIG™ Studies, presented at the International Pittsburgh Coal Conference 2006.

⁷ Data from NETL Cost and Performance Baseline for Fossil Energy Plants, Volume 1: Bituminous Coal and Natural Gas to Electricity Final Report (BB Report), DOE/NETL-2007/1281, May 2007, Exhibit 3-99, Stream 13 upstream of water scrubber.

The membrane must be structurally capable of withstanding the expected pressure drop across the system. In general, current coal gasification systems operate around 40 atmospheres of pressure; therefore if the hydrogen product from the membrane is at 5–10 atmospheres, the differential pressure across the membrane would be about 30–35 atmospheres or 450–525 psi. Future coal gasification systems for hydrogen may operate at 80 atmospheres, so that the system pressure differential across the membrane could be as high as 800–1,000 psi. The Program Targets require membranes to withstand a pressure differential of 400 psi (2010 Target) or 800–1000 psi (2015 Target). Corresponding to the total pressure conditions and hydrogen contents in the tables above, implied maximum available hydrogen partial pressures in these systems would be as follows:

- GE – 334 psi.
- Shell – 266 psi.
- E-Gas – 212 psi.

According to DOE’s H2A analysis protocol⁸, the desired hydrogen delivery pressure to the plant gate of a central hydrogen production plant is 300 psi. Clearly, considering additional membrane separation pressure drops, in some cases additional end-stage or inter-stage compression may be necessary to achieve this delivery pressure. Process designs for hydrogen plants might try to maximize gasifier pressure to achieve high ΔP and hydrogen flux, and/or minimize the need to recompress hydrogen. However, higher pressure gasifiers produce more methane, so a tradeoff study might be necessary to optimize pressure selection. In any case, the feed pressures and maximum pressures listed in the Program Targets above are reasonably consistent with available hydrogen partial pressures for gasification systems.

Water-Gas-Shift (WGS) Reactor Process Conditions

Shift reactors must be used to convert water and CO in the gas into additional H₂ and CO₂ in order to maximize the H₂ content for separation and make CO₂ available for sequestration. Raw gas shift reactors using sulfur resistant catalysts can be applied to the gas before desulfurization. Alternatively, clean synthesis gas after sulfur removal can be reheated to 315–371 °C (600–700 °F) for WGS reactors that produce additional hydrogen through the catalytic reaction of CO with H₂O to form CO₂ and H₂.

As mentioned earlier, there are several possible strategies for placement of hydrogen separation membranes in a coal gasification process. One strategy is to position the membrane downstream of the shift reactors; another is to place membrane separators between shift reactors, while a third is to integrate shift reaction with hydrogen separation to use removal of hydrogen from the syngas as a means to drive the shift reaction to completion at higher temperatures than would normally be utilized in a shift reactor. The latter innovative design is referred to as a water-gas-

⁸ http://www.hydrogen.energy.gov/h2a_analysis.html

shift membrane reactor (WGSMR). Because the WGS reaction is exothermic and a large amount of shift is expected to occur within the membrane reactor, the membrane reactor also should operate in a temperature range compatible for the WGS to occur. WGS activity is an essential function of the membrane reactor for coal-to-hydrogen applications. In addition, for metallic membranes where catalytic activity for hydrogen dissociation is important, tolerance to sulfur compounds such as H₂S and COS is desirable. Failure to achieve sulfur tolerance would require an additional sulfur polishing step in the coal-to-hydrogen plant configuration.

The following information on shift reactor conditions is taken from the DOE Hydrogen from Coal RD&D Plan1. Typically shift reactors may be designed as multiple stages, with the first stages operating at higher average temperatures to take advantage of better kinetics, and later stages operating at lower average temperatures because of equilibrium chemistry that favors higher hydrogen contents (driving the shift reaction to the right). Because of the exothermic nature of the shift reaction, temperature will increase at the reactor exit unless internal cooling is provided. The gas is cooled between stages by heat exchange and/or steam addition. Typical reactor conditions and catalyst properties for the various types of shift reactors are as follows:

- Raw or sour gas (RG) shift
 - Temperature range – 250 to 550 °C (482 to 1022 °F).
 - Catalyst – Co/Mo (the catalyst is actually a sulfided form of Co and Mo).
 - Sulfur tolerance – > 100 ppmv (sulfur is required for the catalyst).
- High temperature (HT) shift
 - Temperature range – 300 to 500 °C (572 to 932 °F).
 - Catalyst – Fe/Cr.
 - Sulfur tolerance – < 100 ppmv .
- Low Temperature (LT) shift
 - Temperature range – 200 to 300 °C (392 to 572 °F).
 - Catalyst – Cu/Zn or Cu/Zn/Al.
 - Sulfur tolerance – < 1 ppmv.

The NETL IGCC studies discussed above use water gas shift before cold desulfurization, which implies that raw gas shift is used in these systems. The Eastman Chemical/RTI warm gas cleanup system has desulfurization before water gas shift, which implies that HT shift is used.

Since hydrogen separation membranes may alternatively be installed in the gasification system process stream following shift reactors, gas conditions and hydrogen content following shift reactors are listed in Table 2. (This information is taken from reference 4.) Figure 2 shows the

main process units that produce these compositions.

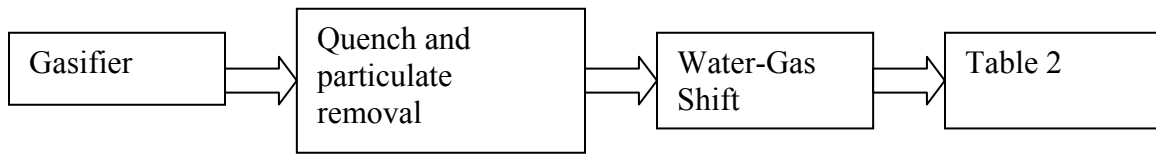


Figure 2 – Block Diagram of Syngas Process Units Corresponding to Table 2

The primary changes from Table 1 are that H₂ and CO₂ levels are higher and CO levels are significantly lower. Steam levels are considered to be variable. All cases use raw or sour gas shift, so sulfur and nitrogen species will be similar to the values in Table 1. This report does not consider the TRIG gasifier, but these compositions would be expected to be similar.

Table 2 – Gasifier Syngas Conditions and Compositions Downstream of Shift Reactors

Vendor/Gasifier	GE Energy Radiant	Conoco- Phillips E-Gas	Shell
Syngas Pressure (psia)	777	516	483
Syngas Temperature [°C (°F)]	270 (519)	236 (457)	35 (95) ⁹
Syngas Composition (only major gases are shown) (mole %)	H ₂ 43% CO 0.9 CO ₂ 31 H ₂ O 24	H ₂ 41% CO 0.5 CO ₂ 32 H ₂ O 22	H ₂ 55% CO 1.7 CO ₂ 38 H ₂ O 0.1

Warm Gas Cleanup Process Conditions

Warm gas cleanup¹⁰ can improve the efficiency of the gasification process by eliminating the need to cool the gas all the way to ambient temperature before separation and/or utilization. A typical process sequence design for warm gas cleanup in a hydrogen production process is particulate removal, gas cleanup, shift reactor, and hydrogen separation. Figure 3 shows the

⁹ Shift and additional gas cooling are combined in this system.

¹⁰ Comparison of a New Warm-Gas Desulfurization Process versus Traditional Scrubbers for a Commercial IGCC Power Plant, presented at Gasification Technologies Conference, Oct 17, 2007 by Jerry Schlather and Brian Turk (RTI/Eastman Chemical).

main process units in this system.

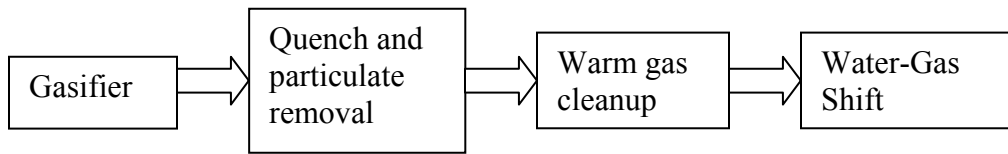


Figure 3 – Block Diagram of Warm Gas Cleanup System

Detailed gas compositions are not available for this system. However, typical warm gas desulfurization conditions are as follows:

- Desulfurization temperature range – 315 to 538 °C (600 to 1000 °F).
- Desulfurization pressure range – 300 to 600 psig.
- Inlet sulfur concentration – 7000 to 8661 ppmv (parts-per-million-by-volume).
- Outlet sulfur concentration – 0.4 to 20.6 ppmv.

For warm gas contaminant removal (other than sulfur), these conditions generally apply:

- Temperature range – > 250 °C (482 °F).
- Ammonia – regenerable – removal to < 50 ppmv.
- Mercury, other – disposable sorbents.

Cold Gas Cleanup

With current commercial syngas cleaning technology, the gas has to be cooled to ambient temperature to remove contaminants. Depending on the system design, a series of scrubbing processes are used to remove particulates, H₂S, COS, and NH₃, and many of these systems operate at low temperatures with synthesis gas leaving the process at about room temperature. The H₂S and COS, once hydrolyzed, are removed by dissolution in, or reaction with, an organic solvent and converted to valuable by-products, such as elemental sulfur or sulfuric acid with 99.8% sulfur recovery. Cold gas desulfurization systems applied to coal gasification can reduce H₂S levels to approximately the parts-per-million (ppm) range. In a design study using the Selexol process,¹¹ syngas is cleaned to less than 1 ppm total sulfur, and the study states that the sulfur level can be further reduced to less than 1 ppb using a zinc oxide sulfur polishing bed.

Potential Sulfur Effects on Metal-based Membranes

Metallic membranes must dissociate molecular hydrogen into hydrogen atoms before diffusion through the separation layer. The presence of trace contaminants, particularly sulfur, can poison the surface sites that are catalytically active for this purpose, diminishing the effectiveness of the

¹¹ Baseline Technical and Economic Assessment of a Commercial Scale Fischer-Tropsch Liquids Facility DOE/NETL-2007/1260.

membrane. The shift reaction and hydrogen extraction by means of a membrane separation system will have a major influence on the driving force for poisoning or corrosion in the membrane. Figure 4 shows a simulation of this effect that was conducted by NETL's Office of Research and Development using the Comsol scientific simulation package.¹² The curves correspond to mole fractions of H₂, CO, CO₂, H₂O, and H₂S in the syngas as it flows in a Water-Gas-Shift Membrane Reactor (WGSMR) that has a cylindrical shape. The general gas flows along the axis of the reactor while the hydrogen (permeate) flows radially. The effect of hydrogen being removed by the membrane as the gas flows axially is to drive the shift reaction towards completion, reducing the CO and H₂O levels, while increasing CO₂, and also increasing the mole fraction of H₂S in the gas, since H₂S is not removed with H₂. The driving force for membrane corrosion is the ratio of H₂S to H₂ in the surface region. Since H₂ is being removed, and the H₂S mole fraction is increasing, the H₂S/H₂ ratio will increase considerably by the time the gas mixture reaches the downstream end of the reactor. Thus levels of H₂S that are non-corrosive at the reactor inlet may become extremely corrosive at the reactor outlet, which could influence the chemical stability of the membrane. This is also the case for oxidation, which is driven by the ratio of CO to CO₂. Membrane testing must be done with cognizance of these issues.

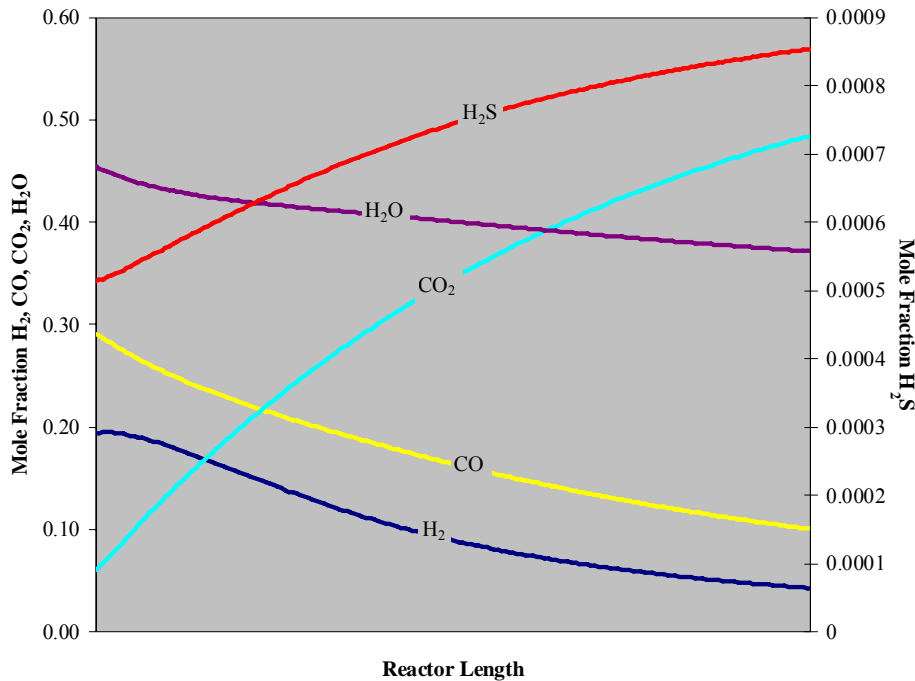


Figure 4 - Comsol® simulated gas composition of a syngas mixture along the length of the Water Gas Shift Membrane Reactor (WGSMR) (permeance used is comparable to a 25 micron Pd-foil).

¹² Bryan Morreale, NETL ORD Hydrogen Separation Group Leader, private communication, December, 2007.
NETL Test Protocol – Testing of Hydrogen Separation Membranes

Testing Protocol Description

General Comments on Testing

The objective of the testing is to qualify the membrane for further consideration in larger scale performance tests of multiple cycles. The membrane formulation and configuration in the bench reactor should be evaluated primarily by:

- Hydrogen separation efficiency and hydrogen flux of the membrane during testing in constant multi-gas loading (calculated at the gas inlet and outlet locations to the membrane unit).
- Measurement of flux and separation factor as a function of gas composition and contaminant level.
- Mechanical and physical properties of the fresh and exposed membranes (e.g., morphology changes and chemical degradation resistance).

For each test, NETL will specify:

- Temperature of membrane and feed gas.
- Feed gas and sweep gas composition.
- Feed and sweep pressure.
- Time of each phase of test.
- Data to be collected during test and data collection frequency.
- Post-test analysis.
- Review of test plan and data analysis.

Membrane Characteristics

The membranes to be tested are mixed metal, metal-ceramic, or pure metal or ceramic formulations which may be thin film or have either metal or ceramic support material. The morphology may vary and may have been produced through various methods. It is anticipated that most of the membranes to be tested in the near term are likely to be constructed of metals that transport hydrogen in the atomic form and generally follow the Sievert's Law pressure dependence exhibited by palladium or palladium-alloy membranes.

Summary of Test Phases

The two near-term test phases will be based on increasing "reality" of gas composition and mechanical stress, and on increasing stringency of hydrogen flux performance. For example, the first test will be with no sulfur or other contaminants, and the first sulfur exposure test will be for sulfur levels comparable to warm gas cleanup. Later tests will use increasing sulfur levels all the

way to raw gas. This outline provides a brief description of the purpose and rationale for the tests; additional detailed descriptions are provided in the next section.

- Test 1 – 2010 Technical Target Screening Test without Sulfur
 - Matches sulfur content for cold gas cleanup with guard bed and LT WGS
- Test 2 – 2010 Technical Target Qualification Tests
 - Matches temperature and sulfur content for warm gas cleanup and HT WGS
- Future Tests – Testing with partially cleaned gas or raw gas from gasifier

Since gas cleanup, WGS, and membrane separation may be closely coupled or integrated in coal-based hydrogen production system designs, it is important to understand these relationships and accommodate them in the testing protocol. Table 3 summarizes the compatibility of hydrogen separation membranes that successfully pass the test phases with the various types of gas cleanup and WGS reactors that might be encountered in a coal gasification-to-hydrogen plant. .

Table 3- Membrane Compatibility with Process Conditions if Successful in Testing

Membrane Compatibility with Coal-to-Hydrogen Plant Process Conditions			
Test Phase	Syngas Cleanup Processes	Water Gas Shift (WGS) Processes	General
Test 1	<ul style="list-style-type: none"> • Requires low temperature desulfurization and possibly zinc oxide guard bed for zero sulfur level 	<ul style="list-style-type: none"> • LT WGS 	<ul style="list-style-type: none"> • Tolerant to syngas CO • Could be placed after LT WGS or staged with LT WGS • Contaminant effects unknown
Test 2	<ul style="list-style-type: none"> • Low temperature desulfurization • Warm gas cleanup that achieves < 20 ppm H₂S 	<ul style="list-style-type: none"> • LT WGS 	<ul style="list-style-type: none"> • Could be placed after LT WGS or staged with LT WGS • Allows tubular membrane operation with 20 ppm H₂S • Other contaminant effects unknown
Future Tests	<ul style="list-style-type: none"> • Partially cleaned gas • Raw gas before desulfurization 	<ul style="list-style-type: none"> • HT WGS • Raw gas WGS 	<ul style="list-style-type: none"> • Suitable for staged WGS or WGS Reactor

Test Specifications

Test 1 – 2010 Technical Target Screening Test Without Sulfur

The objective of this test stage is to determine whether the proposed membrane design, composition, and construction are capable of meeting the Technical Targets of the program with the exception of resistance to contaminants such as sulfur. The lack of sulfur compounds in the stream is potentially achievable by cold gas cleanup with a zinc oxide guard bed and LT WGS.

The gas composition specified is similar to that produced by the coal gasifiers described above with the exception of contaminant species. The conditions generally correspond to hydrogen separation after cold gas desulfurization including zinc guard bed for final polishing, and LT shift. See Table 4 for detailed specifications of gas composition, feed and sweep pressures, and temperatures. The test will be conducted under the following conditions:

- Temperature – Selected by contractor in range of 300 to 600 °C (572 to 1112 °F).
- Feed gas composition – Syngas after shift using simulated gas (bottled gases).
 - 5–10% He will be added to the feed gas for leak detection and the compositions in Table 4 adjusted to keep the same ratios of other gases.
- Sulfur content – Zero.
- Feed hydrogen differential partial pressure relative to sweep gas – ~100 psi.
- Sweep gas – Tests can be run without sweep, but if sweep gas is used it is recommended that argon be used.

Please note that for these tests

- A leak test will be conducted on each membrane to be tested.
- The target for hydrogen purity is 99.5%.
- Hydrogen flux will be determined at above conditions at constant temperature and flow conditions over an 8 hour period, as a minimum, and reported as an hourly average.
- It is recommended that each membrane be tested to 120 hours to determine whether there are long term performance or structure changes.

Test 2 – 2010 Technical Target Qualification Tests

The objective of this series of tests is to determine whether the targeted membrane hydrogen separation efficiency and flux can be maintained with at least 99.5% H₂ purity in the presence of sulfur contamination up to the 2010 Technical Target level. This series of tests will also determine the potential effects of increasing H₂S/H₂ ratio due to removal of H₂ through the membrane as the feed gas passes along the membrane from inlet to outlet.

Baseline Test

Each test phase in Test 2 will begin with a new membrane. Each membrane used in Test 2 will be subjected to a baseline test at the conditions specified in Test 1 to ensure that its performance is reasonably consistent with the original membrane tested in Test 1. The duration of the baseline test will be decided by the contractor as long as it is sufficient to make a flux determination and determine that the membrane is not leaking. Flux with each new membrane should be reproducible within plus or minus 20% of the flux achieved during the original Test 1.

Test Conditions

The tests in Test 2 will be conducted at the conditions specified below. These conditions generally correspond to those that would be encountered by hydrogen separation membranes installed downstream of warm gas desulfurization and HT shift. See Table 4 for detailed specifications of gas composition, feed and sweep pressures, and temperatures for the following three tests (2A, 2B, and 2C):

- Temperature – Selected by manufacturer in range of 300 to 600 °C (572 to 1112 °F).
- Sulfur (H₂S) content in feed gas – 20 ppm.
- Feed hydrogen differential partial pressure relative to sweep gas – ~100 psi.
- Sweep gas – Tests can be run without sweep, but if sweep gas is used it is recommended that Argon be used.

Please note that for these tests

- A leak test will be conducted on each membrane to be tested.
- The target for hydrogen purity is 99.5%.
- Hydrogen flux will be determined at constant temperature and flow conditions over an 8 hour period, and reported as an hourly average.
- It is recommended that each membrane be tested to 120 hours to determine whether there are long term performance or structure changes.

- Conditions for Test 2A:
 - Feed gas composition – Syngas after shift.
 - 5–10% He will be added to the feed gas for leak detection and the compositions in Table 4 adjusted keeping the same ratios of other gases.
- Conditions for Test 2B:
 - Feed gas composition – Syngas after 50% of H₂ has been removed from gas specified in Test 2A.
 - 5–10% He will be added to the feed gas for leak detection and the compositions in Table 4 adjusted keeping the same ratios of other gases.
- Conditions for Test 2C:
 - Feed gas composition – Syngas after 95% of H₂ has been removed from gas specified in Test 2A.
 - 5–10% He will be added to the feed gas for leak detection and the compositions in Table 4 adjusted keeping the same ratios of other gases.

Detailed Test Conditions

Table 4 below details the test conditions for Tests 1 and 2.

Table 4 – Summary of Test Conditions for Tests 1 and 2

	Test Phase			
	Test 1	Test 2A	Test 2B	Test 2C
Feed Gas Composition (Before correction for addition of He)				
H ₂ (%)	50.0	50.0	33.0	4.8
CO (%)	1.0	1.0	1.3	2.0
CO ₂ (%)	30.0	30.0	40.0	57.0
H ₂ O (%)	19.0	19.0	25.0	36.2
H ₂ S (%)	0.000	0.002	0.003	0.004
Sweep Gas Inlet Composition				
Ar (%)	100	100	100	100
Total Feed Pressure (psia)	200	200	200	200
H₂ Feed Partial Pressure (psia)	100	100	66	9.6
Total Sweep Pressure (psia)	<30	<30	<30	<30
(P_{H₂S}/P_{H₂})_{Feed}	0.00E+00	4.00E-05	9.09E-05	8.33E-04
Temperature (°C)	300-600	300-600	300-600	300-600

Future Tests with Partially Cleaned or Raw Syngas

NETL is contemplating future tests to determine hydrogen membrane separation efficiency and flux in the presence of sulfur contamination up to and above the 2015 Technical Target level, extending to raw gas conditions of sulfur levels and other gasifier syngas contaminants. These tests will be further defined as the earlier testing (Tests 1 and 2) are completed. Generally this testing will be intended to address conditions corresponding to use of a hydrogen separation membrane in a Water Gas Shift Reactor configuration in partially cleaned or raw gas from one of the selected gasifiers. The test protocol for these tests will be specified by the NETL COR.

- Feed gas composition would be selected corresponding to syngas after quench and particulate removal but before shift reactors or other gas cleanup systems.
- Sulfur content and other contaminants would be equivalent to those in gasifier raw gas.
- Total pressure would be in the 500 – 800 psi range or possibly higher, depending on the status of gasifier development and the results of systems studies for hydrogen production.
- Exposure to actual gasifier raw gas is desirable if sidestream test facilities are available.

Data Collection and Analysis

The following measurements will be made and reported as a minimum for each test condition.

Note - Standard temperature and pressure (STP) = 60 °F (15.6 °C), 14.7 psia.

- Membrane temperature (°C).
- Time required for heat-up and conditioning.
- Membrane area and thickness (cm² and cm).
- Retentate Pressure (kPa and psia).
- Retentate Flow reported at STP.
- Retentate composition (before and after reactor).
- Sweep Pressure (kPa and psia).
- Sweep Flow reported at STP.
- Sweep composition (before and after reactor).
- Measurements of gas composition by gas chromatograph.

Please note that

- Gas compositions and flows will be reported as hourly averages for an 8 hour test period and for long-term tests (120 hours), if conducted.
- Flows will be reported in scfm, temperatures in °C, pressures in kPa and psia.

Analyses will include the following and will be described in detail sufficient to allow the reader to recalculate the results from experimental data:

- Hydrogen Flux at STP for test conditions – hourly averages for the 8 hour test period. Hydrogen flux will be reported both in sccm/cm² and in SCFH/ft² of membrane area.
- Calculations of hydrogen fluxes at STP for “standard” conditions of 150 psi feed hydrogen partial pressure and 50 psi hydrogen sweep partial pressure for each of the hourly averages.
- Calculations of hydrogen purity.

Reporting

A comprehensive test topical report shall be completed and provided to DOE within two weeks of the completion of each test. All experimental data and calculations will be made accessible to NETL personnel for review.

The minimal outline of this report shall include:

1. Introduction.
2. Test Protocol Modifications (if any).
3. Experimental Procedures.
4. Test Results for Separation.
5. Post-Test Characterization.
6. Summary and Conclusions.

