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LNT Standard Characterization Protocol

General Specifications:

- Measurements made with degreened monolith core sample a minimum of 1 inch in length and diameter ≥ ½ inch; exact sample length, diameter, and cell density must be provided with data.
- Sample degreened for 16 hours in 10%H_2O/air at 700°C.
- .02-inch-diameter (20-mil-diameter) thermocouples installed in gas stream at core inlet and outlet and one in contact with the wall of a central channel at the catalyst midpoint.
- Unless otherwise noted, measurement rates for NO, NO_2, and UEGO should be 10 samples/s. Measurement rates for H_2, CO, N_2O, and NH_3 should be ≥ 2 samples/s.

Part 1: Instrument and reactor calibration matrix
Nominal fixed conditions: 500°C temperature, 30,000 1/hr SV.
Objective: Establish gas analyzer instrument times and repeatability of measurements for standard sample.
Frequency: Run at the beginning and end of protocol testing for each material (as a minimum).

Run No.	Reactor mode*	Gas Mix Sequence+	Period 1 (s)	Period 2 (s)	Period 3 (s)	No. of cycles (repeats of each period)
C1	Bypass or empty	M1/M2/M3	100	100	100	5
C2	Normal with std. reference sample++	M4	900	–	–	1
C3	Normal with std. reference sample++	M1/M2/M3	100	100	100	5

Footnotes:
* For calibration, the reactor is run empty or with all gas bypassed directly to the gas analyzers (Run C1) to determine the instrument response by itself and then with a standard LNT sample (Run C3) to ensure consistency over time. Run C2 is an initialization condition for the standard sample case.

+ Calibration data consists of measured post-reactor values for NO, NO_2, H_2, CO, N_2O, NH_3, and UEGO output for runs C1 and C3 as the inlet gas mixture is switched (in steps) among the 3 different conditions.
Inlet gas composition summary:
M1- Calibration gas mix 1= 5% H_2O, 5% CO_2, balance N_2
M2- Calibration gas mix 2= 5% H_2O, 5% CO_2, 10% O_2, 300 PPM NO, balance N_2
M3- Calibration gas mix 3= 5% H_2O, 5% CO_2, 1%CO, balance N_2
M4- Calibration gas mix 4= 5% H_2O, 5% CO_2, 1%H_2, balance N_2
The inlet condition sequences are repeated cyclically as specified for runs C1 and C3. The transition between conditions should be as fast as possible.

++ Std. reference sample should be degreened according to above procedure and maintained under Argon when not in use.

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Part 2: Oxygen storage matrix
Nominal fixed conditions: 30,000 1/hr SV.
Objective: Establish inherent oxygen storage (as reflected in reductant demand) exclusive of NOx.
Frequency: Run at the beginning of protocol testing for each material.

Run No.*	Temp (deg C)[+]	Gas Mix[++]	Lean period (s)	Injected Reductant	Regen peak (ppm)	Regen period (s)	No. of cycles
C1	T_{max}	M1	0	H2	10,000	900	1
C2	T_{max}	M2	60	CO	10,000	30	30
C3	$.75(T_{max}-T_{min})+T_{min}$	M2	60	CO	10,000	30	30
C4	$.5(T_{max}-T_{min})+T_{min}$	M2	60	CO	10,000	30	30
C5	$.5(T_{max}-T_{min})+T_{min}$	M3	60	CO	10,000	30	30
C6	$.5(T_{max}-T_{min})+T_{min}$	M4	60	CO	10,000	30	30
C7	$.25(T_{max}-T_{min})+T_{min}$	M2	60	CO	10,000	30	40
C8	T_{min}	M2	60	CO	10,000	30	50

Footnotes:
* Calibration data consists of measured post-reactor values for CO and UEGO output for runs C2-C8 as the inlet gas mixture is cycled between rich and lean conditions.

[+] T_{max} = 500°C and T_{min} = 150°C.

++ Gas inlet mix M1= 5% H2O, 5% CO2, balance N2
Gas inlet mix M2= 5% H2O, 5% CO2, 10% O2, balance N2
Gas inlet mix M3= 5% H2O, 5% CO2, 5% O2, balance N2
Gas inlet mix M4= 5% H2O, 5% CO2, 1% O2, balance N2

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Part 3. Short-cycle matrix, POX reductant
Objective: Establish short cycle characteristics in presence of typical exhaust and POx reductant.
Frequency: Run once for each material (after oxygen storage measurements).

Run No.$^{\alpha}$	Temp (deg C)$^{+}$	Gas Mix^{++}	SV (1/hr)	Lean period (s)	Reductant*	Regen peak (ppm)**	Regen period (s)#	No. of cycles
1	T_{max}	1	30,000	0	H2	1,000	900	1
2	T_{max}	2	30,000	60	CO/H2	PMAX	5	30
3	T_{max}	2	30,000	60	CO/H2	.5PMAX	5	30
4	T_{max}	1	30,000	60	H2	1,000	900	1
5	$.75(T_{max}-T_{min})+T_{min}$	2	30,000	60	CO/H2	PMAX	5	30
6	$.75(T_{max}-T_{min})+T_{min}$	2	30,000	60	CO/H2	.5PMAX	5	30
7	T_{max}	1	30,000	0	H2	1,000	900	1
8	$.5(T_{max}-T_{min})+T_{min}$	2	30,000	60	CO/H2	PMAX	5	30
9	$.5(T_{max}-T_{min})+T_{min}$	2	30,000	60	CO/H2	.5PMAX	5	30
10	T_{max}	1	30,000	0	H2	1,000	900	1
11	$.25(T_{max}-T_{min})+T_{min}$	2	30,000	60	CO/H2	PMAX	5	40
12	$.25(T_{max}-T_{min})+T_{min}$	2	30,000	60	CO/H2	.5PMAX	5	40
13	T_{max}	1	30,000	0	H2	1,000	900	1
14	T_{min}	2	30,000	60	CO/H2	PMAX	5	50
15	T_{min}	2	30,000	60	CO/H2	.5PMAX	5	50
16	T_{max}	1	30,000	0	H2	1,000	900	1
17	$.5(T_{max}-T_{min})+T_{min}$	2	15,000	60	CO/H2	PMAX	5	30
18	T_{max}	1	30,000	0	H2	1,000	900	1
19	$.5(T_{max}-T_{min})+T_{min}$	2	50,000	60	CO/H2	PMAX	5	30

Footnotes:
$^{\alpha}$ Characterization data consists of measured post-reactor values for NO, NO2, H2, CO, N2O, NH3, and UEGO output.

$^{+}$ T_{max} = 500°C and T_{min} = 150°C.

++ Gas mix 1= 5% H2O, 5% CO2, 1% H2, balance N2
Gas mix 2= 5% H2O, 5% CO2, 10% O2, 300 PPM NO, balance N2

* Reductant= H2 for re-cleaning surface, CO/H2 mixture is 5/8 CO and 3/8 H2

** Regen peak= PMAX = (2*stored NOx equivalent + stored oxygen requirement) as PPM for 5s. The stored oxygen requirement is determined from results of Part 2.

For regen period, transition time should be as fast as possible and consistent with Part 1.

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Part 4. Short-cycle matrix, diesel or gasoline reductant
Objective: Establish short cycle characteristics in presence of typical exhaust and fuel reductant.
Frequency: Run once for each material (after oxygen storage measurements).

Run No. $^{\alpha}$	Temp (deg C)$^{+}$	Gas Mix^{++}	SV (1/hr)	Lean period (s)	Reductant*	Regen peak (ppm)**	Regen period (s)	No. of cycles
1	T_{max}	1	30,000	0	H2	1,000	900	1
2	T_{max}	2	30,000	60	L	PMAX	5	30
3	T_{max}	2	30,000	60	L	.5PMAX	5	30
4	T_{max}	1	30,000	60	H2	1,000	900	1
5	$.75(T_{max}-T_{min})+T_{min}$	2	30,000	60	L	PMAX	5	30
6	$.75(T_{max}-T_{min})+T_{min}$	2	30,000	60	L	.5PMAX	5	30
7	T_{max}	1	30,000	0	H2	1,000	900	1
8	$.5(T_{max}-T_{min})+T_{min}$	2	30,000	60	L	PMAX	5	30
9	$.5(T_{max}-T_{min})+T_{min}$	2	30,000	60	L	.5PMAX	5	30
10	T_{max}	1	30,000	0	H2	1,000	900	1
11	$.25(T_{max}-T_{min})+T_{min}$	2	30,000	60	L	PMAX	5	40
12	$.25(T_{max}-T_{min})+T_{min}$	2	30,000	60	L	.5PMAX	5	40
13	T_{max}	1	30,000	0	H2	1,000	900	1
14	T_{min}	2	30,000	60	L	PMAX	5	50
15	T_{min}	2	30,000	60	L	.5PMAX	5	50
16	T_{max}	1	30,000	0	H2	1,000	900	1
17	$.5(T_{max}-T_{min})+T_{min}$	2	15,000	60	L	PMAX	5	30
18	T_{max}	1	30,000	0	H2	1,000	900	1
19	$.5(T_{max}-T_{min})+T_{min}$	2	50,000	60	L	PMAX	5	30

Footnotes:
$^{\alpha}$ Characterization data consists of measured post-reactor values for NO, NO2, H2, CO, N2O, NH3, and UEGO output.

$^{+}$ T_{max} = 500°C and T_{min} = 150°C.

++ Gas mix 1= 5% H2O, 5% CO2, 1% H2, balance N2
Gas mix 2= 5% H2O, 5% CO2, 10% O2, 300 PPM NO, balance N2

* Reductant= H2 for re-cleaning surface, L mixture is 40wt% toluene and 60wt% dodecane for diesel or 40wt% toluene and 60wt% iso-octane for gasoline

** Regen peak= PMAX = (2*stored NOx equivalent + stored oxygen requirement) as PPM for 5s. The stored oxygen requirement is determined from results of Part 2.

For regen period, transition time should be as fast as possible and consistent with step times in part 1.

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Part 5. Long-cycle matrix, POx or fuel reductant
Objective: Establish long cycle characteristics in presence of typical exhaust and POx or fuel reductant.
Frequency: Run once for each material.

Run No. $^{\alpha}$	Temp (deg C)$^{+}$	Gas Mix^{++}	SV (1/hr)	Lean period (s)	Reductant*	Regen peak (ppm)**	Regen period (s)#	No. of cycles
1	T_{max}	1	30,000	0	H2	1,000	600	1
2	T_{max}	2/3	30,000	900	CO/H2	1,000	600	3
3	T_{max}	2/4	30,000	900	None	0	600	1
4	T_{max}	1	30,000	0	H2	1,000	600	1
5	$.75(T_{max}-T_{min})+T_{min}$	2/3	30,000	900	CO/H2	1,000	600	3
6	$.75(T_{max}-T_{min})+T_{min}$	2/4	30,000	900	None	0	600	1
7	T_{max}	1	30,000	0	H2	1,000	600	1
8	$.5(T_{max}-T_{min})+T_{min}$	2/3	30,000	900	CO/H2	1,000	600	3
9	$.5(T_{max}-T_{min})+T_{min}$	2/4	30,000	900	None	0	600	1
10	T_{max}	1	30,000	0	H2	1,000	600	1
11	$.25(T_{max}-T_{min})+T_{min}$	2/3	30,000	900	CO/H2	1,000	900	5
12	$.25(T_{max}-T_{min})+T_{min}$	2/4	30,000	900	None	0	600	1
13	T_{max}	1	30,000	0	H2	1,000	600	1
14	$.25(T_{max}-T_{min})+T_{min}$	2A/3	30,000	900	CO/H2	1,000	900	5
15	$.25(T_{max}-T_{min})+T_{min}$	2A/4	30,000	900	None	0	600	1
16	T_{max}	1	30,000	0	H2	1,000	600	1
17	T_{min}	2/3	30,000	900	CO/H2	1,000	900	5
18	T_{min}	2/4	30,000	900	None	0	600	1
19	T_{max}	1	30,000	0	H2	1,000	600	1
20	T_{min}	2A/3	30,000	900	CO/H2	1,000	900	5
21	T_{min}	2A/4	30,000	60	None	0	600	1
22	T_{max}	1	30,000	0	H2	1,000	600	1
23	T_{max}	2/3	30,000	900	L	1,000	600	3
24	T_{max}	2/4	30,000	900	None	0	600	1
25	T_{max}	1	30,000	0	H2	1,000	600	1
26	$.75(T_{max}-T_{min})+T_{min}$	2/3	30,000	900	L	1,000	600	3
27	$.75(T_{max}-T_{min})+T_{min}$	2/4	30,000	900	None	0	600	1
28	T_{max}	1	30,000	0	H2	1,000	600	1
29	$.5(T_{max}-T_{min})+T_{min}$	2/3	30,000	900	L	1,000	600	3
30	$.5(T_{max}-T_{min})+T_{min}$	2/4	30,000	900	None	0	600	1
31	T_{max}	1	30,000	0	H2	1,000	600	1
32	$.25(T_{max}-T_{min})+T_{min}$	2/3	30,000	900	L	1,000	900	5
33	$.25(T_{max}-T_{min})+T_{min}$	2/4	30,000	900	None	0	600	1
34	T_{max}	1	30,000	0	H2	1,000	600	1
35	T_{min}	2/3	30,000	900	L	1,000	900	5
36	T_{min}	2/4	30,000	900	None	0	600	1

Footnotes:
$^{\alpha}$ Characterization data consists of measured post-reactor values for NO, NO2, H2, CO, N2O, NH3, and UEGO output.

$^{++}$ T_{max} = 500°C and T_{min} = 150°C.

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++ Inlet gas mix 1= 5% H_2O, 5% CO_2, 1% H_2, balance N_2
Inlet gas mix 2= 5% H_2O, 5% CO_2, 10% O_2, 300 PPM NO, balance N_2
Inlet gas mix 2A= 5% H_2O, 5% CO_2, 10% O_2, 300 PPM NO_2, balance N_2
Inlet gas mix 3= 5% H_2O, 5% CO_2, CO/H_2 or L at specified level, balance N_2
Inlet gas mix 4= 5% H_2O, 5% CO_2, 90% N_2

* Reductant= H_2 for re-cleaning surface, CO/H_2 mixture is 5/8 CO and 3/8 H_2, L is 40wt% toluene and 60wt% dodecane for diesel or 40wt% toluene and 60wt% iso-octane for gasoline

** Regen peak= Magnitude of reductant step input in ppm at inlet of LNT monolith

For regen period, transition time should be as fast as possible and consistent with step times in part 1.
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