APPENDIX C : NUCLEAR DENSITY GAUGE CALIBRATION

The nuclear density gauge (NDG), which is used to measure the vapor void fraction in the slurry reactor, was calibrated on 25 and 29 April 1991 and recalibrated on 20 May 1991. The derivation of operative equations and the details of standard measurement positions have been previously documented and discussed in detail (*).

The equation used to determine the vapor holdup is defined below:

$$\ln\left(\frac{I_o}{I}\right) = GL((\alpha\rho\varepsilon)_v + (\alpha\rho\varepsilon)_L + (\alpha\rho\varepsilon)_s)$$

where

I_o	=	radiation intensity of source, corrected for absorptance of the empty vessel and insulation.
I	=	radiation intensity as measured at detector
G	=	geometric factor for the system
L	=	path length the beam travels inside the reactor
α	=	absorptance of phase i
ρ	=	density of phase i
ε	=	volume fraction of phase i

$_{v,L,s}$ denotes the vapor, liquid, and solid (catalyst) phases

Calibration of the NDG is performed with nitrogen at different densities (pressures). For the single-phase system, the general equation above reduces to:

$$\ln(I) = \ln(I_o) - GL \bullet (\alpha\rho)_v$$

When the natural log of the measured intensity (I) is plotted against the product $\alpha\rho$, then the slope yields the product GL and the intercept yields the natural log of the corrected source intensity, I_o.

Calibration is carried out at different positions along the height of the reactor. It is found that small variations in the corrected source intensity, I_o, exist at different positions. These variations are due to small differences in wall and insulation thickness as well as the state of the insulation. It is convenient to define a source intensity at the standard position. I_{os},

and relate all other intensities through correction factors, C_R. Thus, at any position, the intensity is given by,

$$I_o = I_{os} \cdot C_R$$

The NDG was initially calibrated on 25 April. The standard position source intensity, I_{os}, appeared low when compared to the previous calibration (June 12, 1988) while the geometric factor, GL, was virtually the same. The decline in I_{os} with time is to be expected since the radiation source intensity decays with time (half-life is 11074 days). However, the observed decline in I_{os} was slightly greater than one would expect from a simple exponential decay. It was concluded that the insulation was probably not completely dried out and, as a consequence of water absoption, the apparent source intensity was being understated.

On 29 April (after the reactor had been heated with syngas and oil for 3 days) a calibration scan was conducted at low pressure (11.5 psig) and is was observed that, indeed, I_{os} had increased to a level more in-line with expectations. Upon completion of operations a three pressure calibration was also conducted (on 20 May). Indeed the source intensity appeared as-expected based on source decay theory. The final calibration to be used for the subsequent analyses was based on the values of GL and C_R determined during from the data collected on 20 May. These calibration constants are presented in Table C1 and compared to the June 12, 1988 and 25 April calibrations.

TABLE C1
CALIBRATION CONSTANTS

Date	(m/d/y)	6/12/88	4/25/91	5/20/91
I_{os}	(cps)	456,900	412,600	431,415
GL	(cm)	47.23	47.41	47.41
Half Life	(days)	11074	11074	11074
C_R	Position (inch)			
	36	1.00	1.00	1.00
	54	1.01	1.01	1.01
	88	1.00	1.04	0.97
	108	0.99	1.05	0.99
	120	0.99	1.01	0.99
	156	1.01	1.03	1.01
	184	1.03	1.07	1.05
	213	1.07	1.09	1.08

The raw calibration data is presented in Table C2; a plot of the NDG calibration is presented in Figure C1. The top line in the figure represents the prior calibration from June 12, 1988; the dashed line represent the calibration from 29 April, 1991.

In order to properly use the holdup equation, the absorptance and densities of the syngas (vapor), slurry oil (liquid), and catalyst (solid) are required. The absorptance of the syngas is calculated from the mass average absorptance of each element (see *); the density is a function of composition, temperature and pressure. For the condensed phases, at reactor conditions (482°F), the absorptance (α) and densities are as follows:

	Absorptance (cm^2/g)	Density (g/cm^3)
Drakeol 10 Oil	0.08776	0.67
BASF S-386 (reduced)	0.07343	5.73
Alumina (dried)	0.07618	3.31
Elements:		
H	0.15370	
N	0.07750	
O	0.07750	

(*) References:

The detailed description and documentation of the nuclear density gauge equations as well as the June 12, 1988 calibration data can be found in - "Liquid Phase Methanol LaPorte Process Development Unit: Modification, Operation, and Support Studies", Topical Report: "Task 2.0: Run E-5, Gas Hold-up and Equipment Evaluation Studies, Appendix A.", 2 January, 1991, No. DE-AC22-87PC90005.

TABLE C2

CALIBRATION DATA FOR THE NUCLEAR DENSITY GAUGE

Date		26-Apr-91		26-Apr-91		26-Apr-91		26-Apr-91			
Gas		N2		N2		N2		N2			
Temp	°F	88		90		90		95			
Pressure	psig	18		103		310		753			
Density	g/cm3	0.0025		0.0090		0.0247		0.0579			
Absorptivit	cm2/g	0.0775		0.0775		0.0775		0.0775			
RAW DATA											
Number	Position in on Tape	Intensity cps/1000	CR	Intensity cps/1000	CR	Intensity cps/1000	CR	Intensity cps/1000	CR		
1	213	437.8	1.09	427.2	1.09	402.7	1.09	358.0	1.09		
2	184	430.2	1.07	421.1	1.08	397.5	1.07	351.4	1.07		
3	156	414.4	1.03	404.2	1.03	381.7	1.03	337.9	1.03		
4	120	404.7	1.01	395.0	1.01	373.6	1.01	329.9	1.01		
5	108	419.3	1.04	409.1	1.04	386.9	1.04	342.0	1.05		
6	88	418.9	1.04	409.0	1.04	386.0	1.04	341.5	1.04		
7	54	406.9	1.01	396.4	1.01	374.4	1.01	331.0	1.01		
8	36	401.8	1.00	391.5	1.00	370.7	1.00	327.1	1.00		
AVG		416.8		406.7		384.2		339.9			

Date		29-Apr-91		20-May-91		26-Apr-91		26-Apr-91			
Gas		N2		N2		N2		N2			
Temp	°F	90		74		66		70			
Pressure	psig	11.5		198		55		6.5			
Density	g/cm3	0.0020		0.0167		0.0055		0.0017			
Absorptivit	cm2/g	0.0775		0.0775		0.0775		0.0775			
RAW DATA											
Number	Position in on Tape	Intensity cps/1000	CR	Intensity cps/1000	CR	Intensity cps/1000	CR	Intensity cps/1000	CR		
1	213	455.7	1.11	437.5	1.08	456.2	1.08	463.8	1.08		
2	184	435.8	1.06	425.5	1.05	441.0	1.04	448.9	1.05		
3	156	416.5	1.02	409.5	1.01	425.4	1.00	431.7	1.01		
4	120	406.4	0.99	403.2	1.00	419.4	0.99	425.5	0.99		
5	108	423.4	1.03	399.5	0.99	418.2	0.99	423.3	0.99		
6	88	425.8	1.04	400.6	0.99	410.6	0.97	415.1	0.97		
7	54	411.7	1.01	409.9	1.01	427.9	1.01	434.0	1.01		
8	36	409.6	1.00	404.8	1.00	423.5	1.00	429.0	1.00		
AVG		423.1		411.3		427.8		433.9			

FIGURE C1

CALIBRATION RESULTS FOR THE NUCLEAR DENSITY GAUGE