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ORO-5197-8

MASTER

STUDY OF HYDROCARBON - SHALE INTERACTION

PROGRESS REPORT #8

January 1 - March 31, 1978

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Prepared for the U. S. Energy Research and Development Administration

under Contract No. EY-76-S-05-5197

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QUARTERLY PROGRESS REPORT

January 1, 1978 - March 31, 1978

Contract No. EY-76-S-05-5197

1. Executive Summary: Work was directed at getting the DOE computer operational. Toward the end of the period some data was collected. This effort was accomplished primarily by Paul Schettler on a $\frac{1}{2}$ released time basis. (Appendix A)

2. Technical Progress

2.1.1: Current Status

- a) DOE computer up and operational except for plotting capability. Detailed report in Appendix B.
- b) Apparatus calibrated to ascertain repeatability and absolute accuracy. Detailed report in Appendices C and D.
- c) Data gathering initiated on CH_4 and He isotherms and CH_4 degasibilities on particles. Detailed report in Appendix E.

2.1.2: No serious problems. The lack of plotter capability caused some questions of interpretation of a few measurements.

2.1.3: Plans

- a) Finish construction/conversion of software to new computer.
 - 1) Obtain plotting capability.
 - 2) Finish slab and BET programs.
 - 3) Incorporate permanent storage of detailed data on diskette.
- b) Continue data collection program as follows:
 - 1) N1 RAR #1 well (finish 1 of six)
 - 2) EGSP WV #5 well (10 samples)
 - 3) R-109 Washington Co. (1 sample)
 - 4) Illinois T₁ well (6 samples)
 - 5) Other samples as received.
 - 6) LC 20403 well (100 samples)
- c) Construction of high pressure apparatus.

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WORK SCHEDULES

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WORK SCHEDULES

The principal investigator worked 1/2 time during the contract period except for term break during which time he worked full time for one week. For the most part, 1/2 time means all afternoons, evenings, and weekends. Students, Jay Nelson, Douglas Eadline, and Jill Thayer, collectively spent a total of 130 hours getting the computer operational (building the programmable clock, the I/O patch board, and grinding samples). Dale Wampler spent some time determining how to modify the plotter driver routines (from the departmental computer) so that they would work on the DOE computer.

AUTOMATED ISOTHERM/DIFFUSION APPARATUS

STATUS/CAPABILITY REPORT

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AUTOMATED ISOTHERM/DIFFUSION APPARATUS
STATUS/CAPABILITY REPORT

Work on the automated device and the associated computer was a major component of the previous quarterly report (1). This report is an update of those efforts.

Automated Isotherm/Diffusion Constant Device

This part of the apparatus continues to work well in all regards as discussed in the last report (1).

Computer

This section is an update of the "items to be accomplished" listed in the "Status Report: Computer Capability" section of the last quarterly report. Those items and progress and completion dates are listed.

- 1) Missing cable. Received mid-March.
- 2) Real time programmable clock. This modification of the computer hardware was necessary to operate our software which demands a highly flexible clock. Clock was completed and debugged before the end of January.
- 3) Circuit patch board. This board allows communication between the computer and the isotherm/diffusion apparatus itself. It involved some 40 fused connections plus 2 power supplies to operate the solenoid valves of the apparatus. High impedance connections were protected in addition by a zener diode circuit. Completion occurred before the end of January with final checkout occurring in February.
- 4) Copy software from the cassette tapes to the diskettes. This was accomplished on January 28-29 by contract agreement with Data General in Pittsburgh. Due to differences between the NOVA 3 DOS BASIC and the NOVA 1220 SOS BASIC,

most software did not work initially. Debugging occupied the first part of February.

Improvements obtained by this effort include:

- 1) It is no longer necessary to interface our isotherm and diffusion measurements with a large number of educational demands of computer time.
- 2) Faster and more reliable terminal response.
- 3) Increase in speed, ease of operation, and data storage capacity by virtue of a disk(ette) operating system.

Software Status Report

This report is an update of the report on this subject included in the last quarterly report (1).

The software package in that report has been adapted to the NOVA-3. This process took about a week to ten days because of differences in Assembler and Basic between the two computers. At the present time all software described in the last report (1) works on the NOVA-3 with the following exceptions:

- 1) The full operating system that will save all data on diskettes for later (possible) retrieval is not operative. A subset of this extended system is operating however.
- 2) The 2nd generation "Slab" diffusion constant program is not operative.
- 3) The 2nd generation BET program is not operative.

The lack of 2nd generation "SLAB" and "BET" programs is not curtailing present operations. The reduced operating system is satisfactory in all regards except for permanent data storage.

Reference

1. Schettler, P. D., Progress Report #7, Study of Hydrocarbon/Shale Interaction, ORO-5197-7.

ORO-5197-8
Appendix C

CALIBRATION REPORT

AUTOMATED ISOTHERM/DIFFUSION APPARATUS

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CALIBRATION REPORT
AUTOMATED ISOTHERM/DIFFUSION APPARATUS

Introduction

Careful checks of procedures and calibration is important to most experimental measurements. This report documents the calibration and performance checks that we have made upon the isotherm/diffusion apparatus and related software. The accuracy of all our results depends upon the accuracy of calibration of the internal volumes of the apparatus because the number of moles of gas adsorbed or desorbed from the sample is determined by differences of pressure of the gas within a known volume connected to the sample. As the equation of state of a gas is highly temperature dependent, close monitoring must be kept on this parameter as well.

Pressure Measurement

Overall accuracy of the Datametrics pressure transducer is reported by the manufacturer to be .25% for our configuration. Of more importance to our application is the measurement of pressure differences which are often small. The stability and resolution necessary to maximize the precision of these difference measurements are obtained by:

1. Data averaging. In general points are gathered at a rate of 25,000 points/sec. and averaged in groups. An equilibrium pressure reading consists of 1.5×10^6 individual readings; for gas release versus time measurements each recorded point is an average of 250-7000 individual measurements depending upon the actual point gathering rate. Theoretically the resolution achieved for an equilibrium point should be one part in 10^7 . Actual practice seems to suggest a resolution of 1 part in 1.6×10^5 due to slow drifts that are not averaged out over the time of each measurement.

2. Thermostating. The pressure transducer and some of its electronics is held at a fixed temperature during a run.

3. Software zeroing and full-scale procedures. The electronic null and full-scale readings of the pressure transducer electronics is determined automatically before each run and if necessary before each measurement.

Routines for the data averaging and zeroing have been discussed before (1).

Volume Calibration

Volume calibrations are made using the precisely known volumes of the gas buret (Fig. I) in conjunction with equations of state for He, CH₄, and N₂ with non ideal gas corrections (Beattie-Bridgman or Benedict-Webb-Rubin equations of state). The first volume measurement is taken in the tubing terminated by V₁, V₂, mark one of the gas buret, and the non-pressure dependent part of the Barocel. The second volume has the same limits but with V₂ open so as to include the tubing to M₁. The third volume represents the linear part of the Barocel volume change with pressure.

Table I summarizes the results obtained by least squares analysis of 8 points (obtained from adjusting the buret between all 8 of its possible positions. The error limits on each measurement are an output of the least squares routine and represent the scatter of data between each of the eight points.

TABLE I

	<u>CH₄</u>	<u>N₂</u>	<u>He</u>
Volume One (ml)	15.428±.02	15.519±.026	15.488±.022
Volume Two (ml)	---	19.60±.04	19.67±.026
Datametrics volume change (volume/1000 torr)	.3004±.02	.2117±.025 .277±.04	.2787±.021 .2750±.02

Figure I.

The accuracy of these results depends upon the accuracy of calibration of the gas buret. This had been done by the PI in 1974 by mercury weight with a precision of .03%.

Temperature Stability

It was attempted to measure the effect of changes of room temperature upon the volumes measured in the previous section. An effect here may occur, not because of expansions and contractions (which are negligible at "constant" temperature) but because the actual gas temperatures in various parts of the apparatus may not be exactly at the bath temperature. The design of the temperature regulating part of the isotherm/diffusion device has been discussed previously wherein the short term temperature regulation of the circulating bath is reported as $\pm .002^{\circ}\text{C}$ (1). Clearly, however, the measured stability of the bath is not the only factor involved in maintaining the temperature stability of the system wherein the effect of the insulation and the room temperature also contribute.

Our first attempt at measuring the effect of room temperature was to look at calibrated volumes as a function of a variety of room temperatures. There is a paucity of data, but the data that is available suggests the effect of room temperature is small (as expected).

Our second attempt was to make direct thermocouple measurements of various parts of the apparatus during a period when there was significant difference between the temperature of the regulating bath and the room. The temperature measurements were made by puncturing the insulation and inserting the thermocouple against the tube. Table II shows the results of these measurements along with the volume (crude) to be associated with each temperature. From these measurements, it is estimated that

$$\text{Vol (1)} = 15.433 + .0077 (T_B - T_R)$$

$$\text{Vol (2)} = 19.59 + .010 (T_B - T_R)$$

$$\text{Vol (Datametrics)} = .27 \pm .02$$

The constant terms in these later equations are obtained by weighted least square analysis of available data corrected to $T_R = T_B$. The uncertainty in Vol (1) and Vol (2) is approximately $\pm .02$ and $\pm .03$ ml respectively.

TABLE II

	<u>T (°C)</u>	<u>V (~ ml)</u>	$\alpha = \frac{T_B - T}{T_B - T_R}$
Circulating Bath	27.1° C	----	---
Room Temperature	20.7	----	---
H ₂ O Return to Bath	27.1	----	---
Capillary to Cell	24.0	1.6	.48
Return Line from Buret	26.9	14.0 (mark #3)	.03
Capillary to Buret	26.0	.83	.17
Chamber (Valves & Datametrics)	26.2	15.4	.14

T_B = Circulating Bath temperature

T_R = Room temperature

T = Measured temperature

In short, the effect of room temperature fluctuations can be ignored for small (typical) variations. For variations of 5 degrees or more, the variations may be expected to be noticeable but should be easily correctable pending verification of the above equations by direct calibration during times of unusual temperature conditions.

System Time Constant

For any kinetics apparatus that measures transients, it is important that the time constant of the apparatus be short in comparison to the transient being measured. In our case, degasibility measurements or diffusion constants are made by equilibrating the sample at one pressure then discontinuously changing the pressure to a new (lower) value by opening valve V_1 . Initially gas rushes out of the cell. Then, responding to the lowered pressure, the shale particles degas, approximately at least according to the equation

$$1) \quad M_T = A \sqrt{t} + \dots$$

where A is a fitted constant that includes within it the diffusion constant, or degasibility. To make the fit we follow pressure measurements after the first outrush of gas from the cell. Within this constraint, values at low values of t are more important than large values of t. It is therefore important to know the time involved in initial outrush of gas from the cell. Pressure measurements as a function of time for an inert (non-sorbing) material of the same amount and mesh size should provide this net "instrumental" time constant.

Figure II shows an example of several pressure versus time measurements that have been made for a sample of 700-1000 μ glass beads of similar weight and in a cell similar to that used in standard shale runs. The shape of the curve is reminiscent of some results of some numerical work the PI did on transient flow through a packed tube some 13 years ago (2). It seems clear that equilibrium is established within about .1 sec. A rock diffusion constant greater than $\sim .1 \text{ cm}^2/\text{sec}$ would be indistinguishable from the flow of air from an inert sample.

We have no results for inert samples of 500-700 microns, but it seems safe to say that the instrumental time constant is less than 1 sec for such samples. Using these numbers, a diffusion constant greater than 4×10^{-3} would be indistinguishable from mass air flow.

FIGURE II

From the results we conclude that the direct mass flow of air from the sample is distinguishable from diffusion from the particle for any diffusion constant that we are liable to find in practice.

Measurements on An Empty Cell

"Isotherm" measurements were made on an empty (but calibrated) cell as a final check to insure that

- 1) the calibration procedures were accurate.
- 2) the isotherm software is "bug" free.

Table III shows the results.

TABLE III

<u>Gas</u>	<u>Measurement Error (milliliters)</u>
Helium	-.0070
Methane	-.0076
Helium	-.0063

For the usual 5 gram sample of shale, this would correspond to an error in the isotherm slope of 4×10^{-6} V/V/torr or about 1% of the value usually observed for methane and about 5% of the values observed for helium.

Results were also obtained on glass beads which are a bit puzzling as we expected to get no adsorption. The first isotherm (methane) gave 1.1 Vol/Vol/torr isotherm slope. A repeat at a later time gave 2.55×10^{-4} V/V/torr and 2.41×10^{-4} V/V/torr for helium and methane respectively. We cannot reconcile these results in terms of the high degree of consistency obtained with the rest of the calibration procedure. We thus question the validity of the "inert" assumption. In particular the beads were from an unknown source and not washed, degreased, or otherwise pre-treated except for sieving as further treatment did not seem to be necessary at the time as judged by visual inspection.

Repeatability of Measurement

(Isotherms)

Repeat runs were made on several samples so as to compare the actual scatter of measurements with the scatter predicted by the least squares subroutine on the basis of each individual run as shown in Table IV.

TABLE IV

<u>Identification</u>	<u>Run</u>	<u>Result</u>	
LC 20403 2986'2"	He Iso	3.627×10^{-4} V/V/torr $\pm .5\%$	
		3.601×10^{-4}	
N1RAR#1 562.30'	He Iso	2.71×10^{-4} V/V/torr $\pm .5\%$	
		2.67×10^{-4}	$\pm .4\%$
	CH ₄ Iso	6.32×10^{-4}	$\pm .64\%$
		6.31×10^{-4}	$\pm .64\%$
N1RAR #1 368.55'	CH ₄ Iso	9.26×10^{-4}	$\pm .6\%$
		9.10×10^{-4}	$\pm .7\%$
		9.08×10^{-4}	$\pm .8\%$
N1RAR#1 418.33'	CH ₄ Iso	4.33×10^{-3}	$\pm .22\%$
		4.36×10^{-3}	$\pm .23\%$

Conclusion

The calibration measurements made form a highly consistent set that suggests that the least square error printed out with each isotherm slope is in fact a good measure of the absolute accuracy of the isotherm.

It has been established that the time constant associated with the apparatus is appropriate to the measurement of diffusion constants of gases through shale.

References

1. Schettler, P. D., Progress Report #7, Study of Hydrocarbon/Shale Interaction, ORO-5197-7.
2. Schettler, P. D., and Giddings, J. C., "Pressure Profiles, Fluctuations and Pulses in Gas Thromatography," Anal. Chem. 37, 835 (1965).

EXPERIMENTAL MEASUREMENTS OF DEGASIBILITY/DIFFUSION

STATUS REPORT

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EXPERIMENTAL MEASUREMENTS OF DEGASIBILITY/DIFFUSION

STATUS REPORT

Introduction

The software package described in the last report (1) was designed to obtain degasibilities and diffusion constants without the need for human intervention or judgment-making. We now believe that this effort must be supplemented by checks upon the computerized data analysis on a routine basis. There are two reasons for this:

- 1) While many samples exhibit very ideal behavior, in that a diffusion constant can be measured with considerable certainty, nevertheless some samples do not seem to exhibit ideal behavior.

- 2) The process of least square fit (see Ref. 2) to a polynomial involves a decision as to how many terms of the polynomial to fit and a decision as to what points of the data run to include in the least squares analysis. These two points are discussed in turn, followed by a description of the present status.

Sample Nonidealities

Figure I is a reproduction of a typical good plot of experimental pressure versus time as compared to the exact function calculated for spheres.

On the other hand Figure II is a reproduction of a plot with evidence of nonidealities. In these cases a diffusion constant approximately fits the data but nevertheless the detailed degassing curve exhibits behavior that is not easily described by a single parameter. In the case of nonidealities, it is typical that the experimental curve should exhibit more curvature than the theoretical curve. Possible sources of nonidealities include the heterogeneous structure of the shale, the kinetics of the sorption process itself, et cetera.

FIGURE I

FIGURE II

Least Square Fits

Fits are made to curves such as Figure II so as to maximize certainty of evaluation of the A term in

$$1) \quad P = P_0 + A\sqrt{t} + Bt + Ct^{3/2}$$

The parameter A is dependent only upon external particle surface area, the pressure conditions of the apparatus, and the diffusion constant. In particular it is shape independent, at least for a number of common shapes (cylinders, spheres, slabs, etc.). Thus we often fit only over that portion of the curve wherein a P versus $\sqrt{t}$ curve is fairly linear and cutoff points at large times. On the other hand, points gathered at very low times will still show the influence of gas flow from the cell originating from the expansion of nonsorbed gas in the cell in response to the pressure pulse.

In order to minimize the effect of the particular choice of beginning and ending points on the parameter A, it is useful to least square the polynomial shown in Equation 1) or Equation 1) excluding the C term.

No difficulty is obtained with this procedure in fitting ideal curves. Nonideal curves, however, have been fitted by us in the past by interactive operation between the operator armed with plotting routines and the least square program. In particular the operator will evaluate A for a choice of parameters, then evaluate the rate of degassing for the ideal case (exact function) and compare the exact function, the polynomial fit, and the data on the same graph.

Status

The second generation software automates this least square fitting procedure by using the data points taken after the first 10 seconds but before the pressure has reached the halfway point in its drift towards equilibrium. A third order fit is used and the computer prints out the results automatically. These

parameters were chosen on the basis of interactive experience with some 157 measurements wherein least square parameters were determined so as to give the best fit of the exact function (for spheres) with the data. As such we expect it to work well in the majority of cases wherein ideal fits are observed. Nevertheless, a plotter needs to be incorporated into the computer facility to ascertain the degree and type of departure from ideality (if any is present) and, if nonideality is present, whether the best fit was actually obtained. To this end, Dale Wampler is presently building/modifying plotter drivers that will allow us to make oscilloscope traces as in the past. We wonder, though, if a digital plotter would present worthwhile advantages in terms of permanence, presentation, and storage of results.

References

1. Schettler, P. D., Progress Report #7, Study of Hydrocarbon/Shale Interaction, ORO-5197-7.
2. Schettler, P. D., Progress Report #1, Study of Hydrocarbon/Shale Interaction, ORO-5197-1.

SUMMARY OF ISOTHERM AND DEGASIBILITY MEASUREMENTS

MADE BETWEEN 1 JANUARY AND 31 MARCH 1978

N1RAR #1 AND LC 20403 WELLS

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SUMMARY OF ISOTHERM AND DEGASIBILITY MEASUREMENTS
MADE BETWEEN 1 JANUARY AND 31 MARCH 1978
N1RAR#1 AND LC 20403 WELLS

Tables I and II of this report contain a summary of all samples run during the contract period. Those measurements mentioned elsewhere in the report (for other purposes) are also reported here.

LC 20403

Three depths are reported. Most of the features of these results are similar to those discussed elsewhere for the same well.

N1RAR#1

These samples are quite unusual, particularly in regard to their isotherm properties. In particular, the samples at 320.3' and 418.3' display isotherm slopes that are an order of magnitude larger than has been heretofore observed. Further, the specific degasibilities of all of these samples tend to be very nonideal.

The 320' sample is a light greenish-grey shale with a fine grained, almost homogeneous, appearance. The 418' sample is a rather light grey color with some occurrence of fine bonding. Neither sample has a particularly distinctive appearance in respect to the appearance of "normal" shales. The large values of the isotherms do not in themselves suggest the existence of gas in the well. However, for a given well pressure the amount of gas in these formations ought to be about an order of magnitude greater than is usual.

It should be noted that the amount of helium sorbed by these samples is greater than the volume of the sample (which leads, among other things, to a negative helium density). One concludes that for these samples (at least),

helium adsorption must be an important component. This order of magnitude change can be understood if one assumes that the type of clay minerals in the unusual samples permit interstitial sorption.

The degasibilities for this set of samples was very "nonideal" leading to the uncertainty reported.

LC 20403 - 3446'

Dry versus Dry-Wet-Dry

Significant increases in both the isotherm parameters and specific degasibility occur as a result of the wetting and drying cycle. We have suggested that some (perhaps) small swelling occurs upon wetting. Shrinkage back to the original volume may not occur upon drying leading to an increased openness in the shale structure.

The details of the wetting and drying cycle were as follows:

1. Run a sample that had been crushed and sieved in the dry state.
(500-700 μ fraction used)
2. Remove sample and
 - a) Inject H_2O to cover sample with syringe inserted into cell. Leave in contact with water for 15 minutes.
 - b) Suck off H_2O with syringe.
 - c) Vacuum dry by placing cell in hot water 4 hours.
 - d) Dry at $110^\circ C$ under high vacuum for 20 minutes.
 - e) Place on apparatus under high vacuum with hot water in a Dewar overnight.
3. Repeat runs.

EXPLANATION OF COLUMN HEADINGS USED IN TABLE I.

Column 1	Sample I.D. -- Sample #
Column 2	ρ_B -- Density of shale as measured by immersion in toluene (g/cm ³)
Column 3,4	S -- Initial Isotherm slope (vol _{gas} STP/vol _{rock} /torr x 10 ⁻⁷) STP -- 760 torr, 0° C. Measurements made at 0° C Column 3 -- Helium; Column 4 -- Methane
Column 5	D -- Diffusion constant CH ₄ measured at 0° C (cm ² /sec x 10 ⁻⁷)
Column 6	G _{CH₄} -- Specific degassibility (cm ³ _{gas} (STP)/cm ² /torr/sec ^{$\frac{1}{2}$} x 10 ⁻⁷)
Column 7	ρ_{He} -- density (g/cm ³) as obtained from He isotherm
Column 8	S _{CH₄(s)} -- that part of the isotherm slope for CH ₄ that must be due to sorption (Vol _{gas} (STP)/Vol _{rock} /torr x 10 ⁻⁴)

TABLE I. DIFFUSION AND ISOTHERM PARAMETERS

Sample I.D.	ρ_B	S_{He} $\times 10^{-4}$	S_{CH_4} $\times 10^{-4}$	D_{CH_4} $\times 10^{-7}$	G_{CH_4} $\times 10^{-7}$	ρ_{He}	$S_{CH_4(s)}$ $\times 10^{-4}$
LC20403 well, Lincoln County Virginia							
2976'2-4"	2.699	1.593	3.635	7.47	17.73	3.071	2.042
2986'2-4"	2.407	3.614	5.176	91.6	8.84	3.318	1.562
2996'2-4"	2.558	1.817	3.2708	62.2	4.778 4.34	2.968	1.454
N1 RAR #1 well, Illinois (500-700 μ except as noted)							
320.3'	2.515	3x5.08	4x1.29	.0046	.5	*	6.21
368.5	2.568	3.24	9.26 9.1 9.08	.375*	1 *	3.41	5.90
368.5 (700-1000 μ)	2.568	3.19	9.23	.181*	.7*	3.38	6.04
418.33	2.573	38.3	43.6	1.0	7.8	*	5.30
562.3	2.573	2.71 2.67	6.32 6.31	.63*	.9*	3.23	3.62

* these values are uncertain

TABLE II

Dry vs. Dry-Wet-Dry

LC 20403 -- 3446'

500-700 μ

	S_{CH_4} $\times 10^{-4}$	G_{CH_4} $\times 10^{-7}$
Dry	1.66	3.12
	2.01	3.05
	1.96	
	1.93	
	Av. $1.89 \pm 8\%$	Av. $3.09 \pm 1.6\%$
Dry-Wet-Dry	3.262	3.51
	3.264	3.43
	Av. $3.263 \pm .04\%$	Av. $3.47 \pm 1.6\%$

DIFFUSION

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Date 8/17/1978

Sample ID : WU#6-MERC-1X:7267

Gas : Nitrogen

Initials : DLW

Book : 1

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SAMPLE DATA

Weight : 5.322 (grams)

Density : 2.67122 (gm/cc)

Sample Temp : -196 (cent)

Particle Size : .085 (cm)

DATA COLLECTED

P initial : 238.864

P equilibrium : 49.9709

P o : 36.5336

SPECIFIC DEGASIBILITY 8.59232E-06

UNCERTAINTY .113954 %

System d Moles/ dP 8.2694E-06