ANALYSIS OF NATURAL GAS BY FT-IR; CALIBRATIONS & VALIDATIONS

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A fast experimental protocol was established for the simultaneous determination of methane, ethane, propane, and butane in nitrogen from Fourier transform infrared (FT-IR) spectra. The method is based on correlations established between known gas compositions and their FT-IR spectra. The spectra were collected in the region of 4,000 to 400 cm^{-1} through KBr windows in a 100-mm pathlength gas sample cell operating at 100, 300, 500, 700, and 1,000 mm Hg pressure. Correlation between blending partial pressure or GC-based analysis and FT-IR data produced squared correlation coefficients (R^2) in excess of 0.98. Total required analytical time is less than 2 minutes.

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EXECUTIVE SUMMARY

Problem: Traditionally, natural gas has been analyzed by relatively cumbersome and time-consuming gas chromatographic methods that are not conveniently adaptable to field use.

Objective: The objective of this program was to investigate and define the use of midband Fourier transform infrared (FT-IR) spectroscopy as a rapid and reliable means of estimating natural gas composition and derived properties in the field.

Importance of Project: This method provides a quick and simple way of estimating the concentrations of the major natural gas components and their derived properties simultaneously. Compared to the gas chromatographic procedure, the spectroscopic method offers advantages in cost, time required per analysis, and adaptability to use in remote locations.

Technical Approach: Since methane, ethane, propane, and butane each have distinct infrared spectra, the measurement of their individual concentration can be performed. Using standards, calibration models were constructed to correlate actual concentration of components with FT-IR spectra. The calibration models were validated using a set of independently procured and analyzed natural gas samples.

Accomplishments: A fast experimental protocol was established for the simultaneous determination of methane, ethane, propane, and butane in nitrogen using FT-IR spectroscopy. The spectra were collected in the region of 4,000 - 400 cm^{-1} wavenumbers in a 100 mm pathlength gas sample cell at absolute pressures between 100 and 1,000 mm Hg. Correlation between blending partial pressure, or GC-based analysis, and FT-IR data produced squared correlation coefficients (R^2) in excess of 0.98. Limited validation experiments indicate that FT-IR spectra, taken at 500 mm Hg absolute sample cell pressure provides favorable analysis for the C_1 - C_4 natural gas components down to one mole percent concentration. Computer output of compositional data may also include values for various composition related properties, *e.g.*, heat of combustion, carbon-to-hydrogen ratio, and density. Using commercially available equipment, these analyses may be performed in the laboratory or in the field.

Military Impact: Improved analytical chemical speed and convenience facilitates obtaining compositional data on natural gas in the laboratory or in the field. The method may also be adopted as an on-line analyzer for natural gas pipelines.

FOREWORD/ACKNOWLEDGMENTS

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This report is a continuation of Interim Report TFLRF No. 319, entitled "Analysis of Natural Gas by Fourier Transform Infrared Spectroscopy," in which results of only the calibration experiments were included. The current report includes the development of both the calibration models and the validation of these models.

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I. INTRODUCTION

Composition of natural gas in pipelines is not constant.[1] King demonstrated that, as expected, the composition of natural gas will have a marked effect on the operational characteristics of engines burning natural gas as the fuel.[2] Kubesh and coworkers showed that gas composition has a significant effect on the octane number of natural gas blends used as fuel in internal combustion engines.[3] For these reasons and for determining values in product custody transfers, a reliable, quick, and accurate method was needed to determine gas composition that is more convenient and timely to perform than the commonly used gas chromatographic method.

Between 85 and 95 volume percent (vol%) of pipeline quality natural gas is methane. Generally, the balance of the gas is ethane, propane, butane, and inert gases. Although some studies have found measurable amounts of higher molecular weight hydrocarbons, it was shown that the components present in natural gas at concentrations of less than 0.2 mole percent (mol%) do not contribute significantly to the calorific value of the natural gas.[4] For this reason, a natural gas may be adequately described by the concentrations of the four lightest saturated hydrocarbons, i.e., methane through butane. From the concentrations of the major active ingredients, several pertinent gas properties, e.g., heating value, density, etc., may be easily calculated by built-in computers.

FT-IR and near-IR spectroscopies have the potential to meet the requirements of an on-line or a transportable natural gas analyzer. Near-infrared (near-IR) spectroscopy generally operates in the 900 to 2,000 nanometer (nm), or 0.9 to 2.0 micrometer (μm), wavelength region. Midband Fourier transform infrared (FT-IR) spectroscopy is normally used in the 2.0 to 50 μm region. Due to limitations of optical window materials, FT-IR is usually used in the restricted spectral region from about 2.5 to 25 μm, corresponding to 4,000 to 400 cm^{-1} wavenumbers. In comparing near-IR with FT-IR, it may be noted that near-IR is the result of second and third overtones and combination bands of the fundamental frequencies that produce the directly measurable FT-IR region of the infrared spectrum. Since FT-IR spectroscopy is based on the measurement of characteristic fundamental resonances, it produces specific, usually sharp, well-defined peaks at substantially increased extinction coefficients. Potentially, these facts lead to higher analytical specificity, accuracy, and sensitivity. The higher sensitivity manifested by FT-IR allows accurate measurement of the various natural gas components at substantially reduced pressures.

Brown and Lo demonstrated the feasibility of near-IR in monitoring the energy content of natural gas.[5] The quartz optics and fiber-optic probes typically used with near-IR instruments allow the analyzer to be positioned remote to the measurement site, making it adaptable to field use.[6] Westbrook[7] used near-IR to analyze natural gas at 207 kPa (30 psig) in a flowing line. During the FT-IR analysis of gasolines and turbine and diesel fuels, Fodor et al. demonstrated that the use of infrared spectroscopic analyses, combined with multivariate calibration techniques, allowed the estimation of several pertinent fuel properties. In these experiments the accuracy of measurements was comparable to measurements from techniques that were designed to directly measure the desired fuel properties.[8-10]

II. OBJECTIVE

The objective of this program was to explore the use of midband FT-IR spectroscopy as a rapid and reliable laboratory or field method to estimate natural gas composition and derived properties and to demonstrate the feasibility of FT-IR as an on-line natural gas analyzer.

III. APPROACH

Reference or calibration gases were blended according to compositions determined by statistical treatment of the natural gas compositional limits to maximize the expected correlational output using a minimum number of samples. FT-IR spectra were generated from each of these quantitatively blended reference gas mixtures. The same blends were also analyzed by gas chromatography (GC) to substantiate their composition. Using these data, calibration models were developed for methane, ethane, propane, and butane in nitrogen.

IV. EXPERIMENTAL

Compositions of calibration gas mixtures were designed using the E-CHIP statistical program. The experimental design included the four C_1–C_4 saturated hydrocarbons in nitrogen to provide the minimum number of standard samples, allowing calibration within the widest expected concentration ranges for each component:

methane	50 to 100 vol%
ethane	0 to 10 vol%
propane	0 to 30 vol%
butane	0 to 5 vol%
nitrogen	0 to 35 vol%

To properly blend the various gas components, a four-port mixing manifold was fabricated, and regulating needle valves were installed at each port. Quantitative blending of calibration gas standards was performed using a precision vernier "pressure volume controller" and a pressure regulator and monitor (Heise Models PTE-1 and HBC-1000). A schematic diagram of this sampling system is shown in Fig. 1. The calculated compositional data on these calibration standard mixtures are given in TABLE A-1 of Appendix A. Concentrations of components of the calibration gas standards were confirmed by GC using an instrument (Hewlett-Packard Model 5890 Series II) equipped with a gas sampling valve and a thermal conductivity detector. A GC method, based on the procedure described in ASTM D 1945-91 (Standard Method for the Analysis of Natural Gas by Gas Chromatography), was used as the benchmark method for the analysis of all natural gas samples. Matheson Gas Co. primary standards were used to calibrate the GC. The operating conditions of the GC are summarized in TABLE A-2. To facilitate quantitative FT-IR analysis, pressure control was provided by the same precision pressure regulator that was used for the blending operations. Temperature control relied on the constant temperature environment of the spectrometer's internal sample compartment.

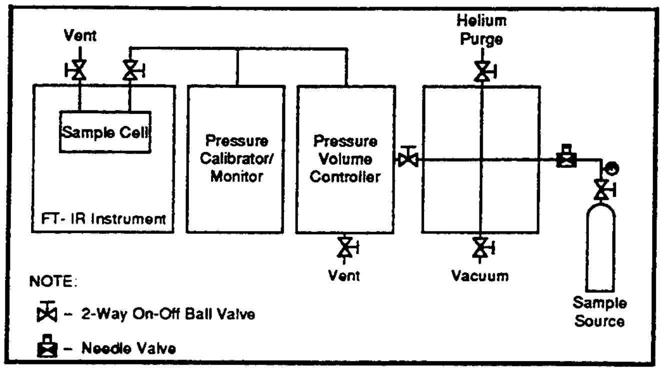

Figure 1. Schematic diagram of gas sampling system

The 100 mm pathlength gas cell was equipped with KBr windows. Spectra of the average of 32 FT-IR scans of each gas sample were collected on all 44 calibration gas mixtures at a resolution of 2 cm^{-1} within the 4,000 to 400 cm^{-1} wavenumber spectral region at 100, 300, 500, 700, and 1,000 mm Hg absolute cell pressure. The various applied gas pressures served to optimize FT-IR response to the vastly different concentration ranges of the natural gas components.

V. CHEMOMETRICS

Spectroscopic data were correlated to fuel property values using Galactic Industries' PLSplus chemometric software package within the GRAMS/386 program. Since all gas components will exhibit only carbon-hydrogen bonds in their IR spectra, no regions were excluded from building the correlation models.

The PLS method creates a simplified representation of the spectroscopic data by a process known as spectral decomposition. Good summary treatises of PLS were published by Martens and Naes[11] and by Haaland and Thomas.[12,13] The PLS approach is based on a bilinear modeling method. A precursor to the PLS technique, which is closely linked to the bilinear framework used in PLS, is the latent root regression analysis, formulated in the 1970's by Webster, Gunst, and Mason.[14] The PLS algorithm initially calculates the concentration, or property value, weighted average spectrum of all the spectra of the fuels in the calibration matrix. This calculation is followed by a computationally intensive procedure, accomplished by performing cross-validation calculations for all samples in the training set. In the cross-validation procedure, a given number of samples (in this study, two) are removed from the calibration data set, and a calibration model, calculated from the remaining samples in the training set, is used to predict the concentration (property value) of the removed samples. The residual errors, or the difference between the predicted and known concentration values, are squared and summed to determine the prediction error. Repeating this cross-validation process for the other samples in the training set results in a refined regression model that is useful in predicting the properties of unknown fuels. The results of spectral decomposition give one set of scores and one set of factors (loading vectors) for calibration for each component of interest. After a calibration model is established, it must be tested by validation experiments in which the calibration model is applied to similar fuels that were not part of the calibration training set. The predicted property values may then be compared with those derived by established ASTM procedures.

It is critical to establish the correct number of factors to be used in the correlation files, because the predicted fuel property values calculated from the model depend upon the number of factors used in the model. Too few factors will not adequately model the system, while too many factors will introduce noise vectors in the calibration, resulting in less than optimum prediction for samples outside the calibration set. The PLS program by Galactic Industries provides data for selecting the appropriate factor by plotting the prediction residual error sum of squares (PRESS) versus the factor. The factor may be selected for (a) the point at which the PRESS value is at a minimum, normally corresponding to a maximum in the value of the squared correlation coefficient, R^2, (b) the point at which the curve indicates that further increase in factors should have negligible effects (a rather arbitrary choice), or (c) a compromise as recommended by Haaland and Thomas. These authors advise the use of an *F*-statistic to arrive at the best compromise in factors. The *F*-statistic can be calculated as the ratio of the minimum PRESS value to all PRESS values corresponding to fewer factors. As the difference between the minimum PRESS and other PRESS values become smaller, the probability, *p*, that each additional factor will provide significant improvement to the model becomes smaller. Haaland and Thomas empirically determined that the optimum number of factors should be at the first PRESS value, where the F-statistic probability drops below 0.75. Initially, we used factors corresponding to an F-statistic probability of 0.5, coinciding with the maximum for the squared correlation coefficients. However, to avoid possible overfitting, we standardized by using the compromise value of $p \leq 0.75$, as suggested by Haaland and Thomas.

VI. RESULTS AND DISCUSSION

A. Calibrations

The synthetic blends of pure components were mixed following the E-CHIP statistical design guidelines. Concentrations of the individual components, expressed in mole percent (mol%), were available by calculation from the blending partial pressure data and the supporting GC data. The FT-IR spectra were collected at ambient temperature at gas sample cell absolute pressures of 100, 300, 500, 700, and 1,000 mm Hg.

The FT-IR spectra of the pure calibration gas components (methane, ethane, propane, butane, and nitrogen), obtained at absolute gas cell pressure of 100 mm Hg, are shown in Figs. B-1 through B-5 in Appendix B. Nitrogen has no active infrared resonance band.

The raw, unmodified spectroscopic data were correlated to both sets of concentration data (*i.e.*, those derived from blending partial pressures and those obtained from GC analysis) for methane, ethane, propane, butane, and the IR-inactive inert diluting nitrogen, using Galactic Industries' PLSplus chemometric software package. Since all gas components exhibit only carbon-hydrogen bonds in their IR spectra, no spectral regions were excluded from building the correlation models. To maintain simplicity of operation without adverse effects on measurement capabilities, no baseline segments were excluded from the calibration files.

Data derived from both the blending partial pressure information and from gas chromatographic data were used for the calibrations. A summary of the data from the pressure and GC-derived calibration experiments is summarized in TABLES A-3 and A-4, respectively. As expected, these two data sets gave essentially identical results, therefore further illustrations show only the pressure-derived data.

Figures B-6 and B-7 in Appendix B are barcharts derived from TABLES A-3 and A-4 using the factors obtained at minimum PRESS and at $p \leq 0.75$, respectively. These figures show that R-squared values for of all of the calibrations are above 0.95, and if the 700 mm Hg data for butane are excluded, all R-squared values are above 0.98.

The excellent agreement between the calibration standards and FT-IR derived concentration data is illustrated in Figs. B-8 through B-12 for methane, ethane, propane, butane, and nitrogen, respectively, using factors at the compromise $p \leq 0.75$. Further information is given in the bracketed area of each figure for (a) the number of factors, F, (b) the sum of the absolute value of the differences between the known and FT-IR derived concentrations for all the samples, or total error, TE, (c) squared correlation coefficient, R^2, and (d) root mean squared difference, RMSD, an indication of the average error in the analysis. Figures B-13 through B-17 show the relationship between the standard error of cross-validation, SECV, and the sample cell pressures for the five components using factors corresponding to (a) minimum PRESS and (b) $p \leq 0.75$ values. In the case of methane (and the inert, inactive diluent nitrogen), the SECV decreases with increasing pressure while remaining fairly constant with the C_2–C_4 hydrocarbons. This suggests that improved analytical data may be obtained for methane at elevated pressures. However, using a 100 mm gas cell at elevated pressures, all of these hydrocarbons displayed spectra with some peaks in the nonlinear range of the absorbance, indicating detector overload. For instance, such detector overload may be observed in case of methane for each of the two characteristic resonance bands, *i.e.*, at 3,014 cm^{-1} due to asymmetric stretching and at 1,303 cm^{-1}, the result of asymmetric bending. Note, however, the reduced error (SECV) for methane at the higher calibration pressures (concentrations) in comparison with those of the other components.

As discussed earlier, to avoid problems caused by overfitting, it is desirable to use the smallest number of factors in the model that yields acceptable data. During our calibrations, we used up to 20 factors. Factors associated with minimum in PRESS values, or at $p \leq 0.75$, were lowest for all five components at sample cell pressures of 100 mm Hg. These data are shown in Figs. B-18 and B-19.

It should be noted that computer output of measurement results may also show composition related properties, *i.e.,* properties that may be calculated from compositional data, *e.g.*, the heat of combustion, carbon-to-hydrogen ratio, density, etc. These are key properties used for air/fuel ratio management in engines, or for gas custody transfers.

B. Validations

Following the calibration studies on synthetic natural gas compositions, the method was validated by applying the developed calibration models to FT-IR spectra of independently obtained and analyzed natural gas samples.

Prior to comparing the GC and FT-IR derived data, experiments were carried out to establish the realistic precision of the benchmark GC method. A natural gas standard, prepared and analyzed by Scott Specialty Gas Co., was used to establish GC analytical repeatability. The Scott gas sample was analyzed by the GC method 15 times, and the sample standard deviation was calculated. TABLE A-5 provides a summary of the GC analyses, including the applicable ASTM repeatability values, the known concentration values, the average of the GC data, and a measure of error in the GC analysis. Results indicate that the average residual error, *i.e.,* the average of the arithmetic difference between the average GC data and the known data, is -0.13 mol% for methane, 0.02 mol% for ethane, and 0.01 mol% for propane and butane. When percent error is calculated, the resultant figures are 0.15, 0.57, 1.00, and 2.50 percent for methane through butane, respectively.

The FT-IR derived analytical results were evaluated by comparing them to the GC data. As practiced during the calibration experiments, an FT-IR spectrum was taken for each sample at absolute pressures of 100, 300, 500, 700, and 1,000 mm Hg. Compositional analyses of these samples were derived from these spectra by using the appropriate calibration models.

Two methods were used to evaluate correlations between the GC and the FT-IR methods. A simple way of assessing the agreement between the benchmark and new measuring techniques considers the residual errors (arithmetic differences) between the established and new methods. Another procedure uses the "limits of agreement."[15] This method is recommended in cases where the results of the benchmark method may be uncertain. To generate the limits of agreement between the generally accepted (GC) and new (FT-IR) methods, the residual error is plotted against the average value of the two methods, and the results are evaluated at one and two standard deviations, *e.g.*, Difference Value ± 2 standard deviation (Difference Values).

Results of the validation experiments are given in TABLES A-6a and A-6b. In these tables the results of analysis for each of nine validation samples are presented for methane, ethane, propane, butane, and for the IR-inactive, diluent "nitrogen." The presented data include (a) the GC data, (b) the FT-IR derived data as measured at five pressures, and (c) the residual error. The residual error in the validation experiments are shown in Figure 20, and the sample standard deviation of the residual errors are shown in Figure 21. The limits of agreement data are provided in Figs. B-22 through B-26 for methane, ethane, propane, butane, and diluting, inert, IR-inactive "nitrogen."

It may be concluded that 500 mm Hg absolute sample cell pressure provides a good overall compromise to achieve favorable analysis for the C_1 - C_4 natural gas components down to one mole percent concentration using FT-IR spectroscopy.

VII. CONCLUSIONS

A fast experimental protocol was established for the simultaneous determination of methane, ethane, propane, and butane in nitrogen using FT-IR spectroscopy. The spectra were collected in the region of 4,000 - 400 cm^{-1} wavenumbers in a 100 mm pathlength gas sample cell at absolute pressures between 100 and 1,000 mm Hg. Correlation between blending partial pressure, or GC-based analysis, and FT-IR data produced squared correlation coefficients (R^2) in excess of 0.98.

Limited validation experiments indicate that FT-IR spectra, taken at 500 mm Hg absolute sample cell pressure provides favorable analysis for the C_1 - C_4 natural gas components down to one mole percent concentration. Computer output of compositional data may also include values for composition related properties, e.g., the heat of combustion, carbon-to-hydrogen ration, density.

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APPENDIX A

Tables

Table A-1. Calibration Gas Mixture Composition (mol%)

Methane	Ethane	Propane	Butane	Nitrogen
61.13	2.77	1.42	0.00	34.68
100.00	0.00	0.00	0.00	0.00
74.66	0.00	0.00	0.00	25.34
63.88	0.00	21.69	0.00	14.50
61.13	9.97	3.61	0.00	25.29
49.98	10.65	0.00	5.02	34.36
54.86	10.17	0.00	0.00	34.97
86.58	0.00	8.30	5.11	0.00
49.60	9.90	8.23	3.63	28.64
75.10	11.58	8.37	4.95	0.00
49.04	0.00	25.97	3.91	21.08
82.99	7.31	0.00	0.00	9.71
89.98	10.02	0.00	0.00	0.00
49.87	2.92	30.04	0.00	17.18
59.19	6.25	12.60	5.00	16.96
49.96	9.77	22.97	0.00	17.30
57.69	9.47	28.11	4.73	0.00
100.00	0.00	0.00	0.00	0.00
53.57	5.03	3.77	2.55	35.90
49.73	0.00	15.18	0.00	35.08
96.26	0.00	0.00	3.74	0.00
86.21	10.12	0.00	3.66	0.00
49.84	7.32	12.09	0.00	30.75
69.70	0.00	30.30	0.00	0.00
53.70	0.00	30.12	1.41	14.76
71.31	0.00	0.00	3.61	25.08
49.67	0.00	30.40	5.10	14.96
49.80	0.00	10.03	5.10	35.07
86.13	0.00	8.44	0.00	5.43
92.19	2.76	0.00	5.06	0.00
75.12	0.00	0.00	0.00	24.88
63.87	5.02	14.02	0.00	17.08
68.10	10.36	21.54	0.00	0.00
85.03	0.00	0.00	5.09	9.87
59.79	0.00	0.00	5.25	34.96
52.84	10.01	29.94	0.00	7.21
77.50	4.74	15.25	2.52	0.00
66.11	0.00	29.07	4.82	0.00
66.83	3.01	0.00	4.99	25.16
49.67	10.25	0.00	5.07	35.01
54.72	10.06	30.21	5.02	0.00
74.38	10.14	0.00	4.97	10.52
63.14	10.16	0.00	1.39	25.30
49.87	7.24	29.81	3.66	9.42

TABLE A-2. Gas Chromatographic Conditions

Instrument:	Hewlett-Packard model 5890 Series II	
Detector:	Thermal conductivity, 250°C	
Injector:	Gas sampling valve, 0.25 mL sample loop, 125°C	
Column:	Porapak QS, 40/60 mesh, 4.6 m × 3.2 mm (15 ft × 1/8 in.) stainless steel packed column	
Carrier Gas:	Helium	
Flow Rates:	Analytical: 20 mL/min Reference: 30 mL/min	
Column Program:	Initial temperature:	100°C
	Initial hold:	0 min
	Rate:	20°C/min
	Final temperature:	225°C
	Final hold:	4 min
Total Analytical Time Required:	Approx. 15 min	

TABLE A-3. FT-IR Calibration for Natural Gas Components Based on Blending Partial Pressures of Synthetic Standards

	P^* (abs)	at min. PRESS			at $p \le 0.75$		
Compound	mm Hg	F	SECV	R^2	F	SECV	R^2
methane	100	4	1.652	0.9891	3	1.804	0.9872
	300	7	1.350	0.9928	6	1.410	0.9922
	500	11	1.184	0.9944	8	1.301	0.9932
	700	11	1.138	0.9948	8	1.234	0.9940
	1000	13	0.664	0.9982	11	0.714	0.9980
ethane	100	8	0.179	0.9985	7	0.198	0.9981
	300	9	0.254	0.9969	7	0.268	0.9966
	500	9	0.193	0.9982	8	0.214	0.9978
	700	12	0.200	0.9981	10	0.218	0.9977
	1000	20	0.138	0.9991	18	0.151	0.9989
propane	100	11	0.350	0.9992	5	0.365	0.9991
	300	20	0.479	0.9984	19	0.522	0.9981
	500	20	0.376	0.9990	17	0.414	0.9988
	700	18	0.650	0.9971	15	0.711	0.9965
	1000	20	0.469	0.9985	17	0.509	0.9982
butane	100	13	0.152	0.9956	11	0.158	0.9952
	300	18	0.223	0.9906	17	0.228	0.9903
	500	20	0.257	0.9881	18	0.280	0.9861
	700	20	0.442	0.9627	17	0.477	0.9565
	1000	20	0.228	0.9906	18	0.251	0.9887
nitrogen	100	4	1.808	0.9818	4	1.808	0.9818
	300	7	1.400	0.9890	6	1.453	0.9882
	500	9	1.333	0.9901	8	1.405	0.9890
	700	10	1.077	0.9935	8	1.143	0.9929
	1000	8	0.856	0.9960	7	0.898	0.9958

Notes:

P^*	Absolute pressure, mm Hg
PRESS	Predicted Residual Error Sum of Squares
p	F-statistic probability
F	Number of factors in calibration model
SECV	Standard Error of Cross Validation
R^2	Squared correlation coefficient

TABLE A-4. FT-IR Calibration on Natural Gas Components Based on GC Analysis of Synthetic Blend of Standards

	P^* (abs)	at min. PRESS			at $p \leq 0.75$		
Compound	mm Hg	F	SECV	R^2	F	SECV	R^2
methane	100	4	1.545	0.9905	4	1.545	0.9905
	300	7	1.350	0.9928	6	1.410	0.9922
	500	10	1.230	0.9940	8	1.319	0.9931
	700	10	0.962	0.9964	8	1.032	0.9960
	1000	13	0.823	0.9973	8	0.899	0.9969
ethane	100	15	0.183	0.9984	12	0.202	0.9980
	300	9	0.254	0.9969	7	0.268	0.9966
	500	20	0.193	0.9982	16	0.205	0.9980
	700	16	0.224	0.9976	14	0.246	0.9971
	1000	10	0.227	0.9975	8	0.238	0.9973
propane	100	5	0.488	0.9983	4	0.524	0.9981
	300	20	0.479	0.9984	19	0.522	0.9981
	500	20	0.432	0.9987	17	0.467	0.9985
	700	20	0.709	0.9965	16	0.758	0.9960
	1000	19	0.498	0.9983	16	0.524	0.9981
butane	100	14	0.152	0.9957	11	0.158	0.9953
	300	18	0.223	0.9906	17	0.228	0.9903
	500	20	0.305	0.9828	14	0.335	0.9798
	700	20	0.470	0.9582	17	0.501	0.9526
	1000	19	0.282	0.9855	16	0.300	0.9836
nitrogen	100	4	1.775	0.9823	4	1.775	0.9823
	300	7	1.400	0.9890	6	1.453	0.9882
	500	10	1.263	0.9910	8	1.364	0.9895
	700	10	1.040	0.9940	8	1.075	0.9937
	1000	10	0.912	0.9953	7	0.959	0.9952

Notes:

P^*	Absolute pressure, mm Hg
PRESS	Predicted Residual Error Sum of Squares
p	F-statistic probability
F	Number of Factors in calibration model
SECV	Standard Error of Cross Validation
R^2	Squared correlation coefficient

TABLE A-5. GC Analysis of a Scott Natural Gas Standard

compound	known mol %	GC mol %	ASTM repeatability	GC stds	residual error (known-GC)	error %
methane	88.68	88.81	0.30	0.30	-0.13	-0.15
ethane	3.50	3.48	0.05	0.05	0.02	0.57
propane	1.00	0.99	0.03	0.03	0.01	1.00
butane	0.40	0.38	0.03	0.38	0.01	2.50

GC = average result of 15 measurements
stds = sample standard deviation of 15 measurements
error % = 100*(arithmetic difference / known)

TABLE A-6a. Validation of Calibration Models for Natural Gas Components

FT-IR Cell Pressure = 100 mm Hg, abs

Sample	Methane				Ethane				Propane				n-Butane				"Nitrogen"			
FTIR ID	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2
val04p1	94.74	93.92	0.82	94.33	2.74	3.09	-0.34	2.91	0.37	0.49	-0.12	0.43	0.04	0.95	-0.91	0.49	1.85	2.74	-0.89	2.29
val05p1	94.55	94.94	-0.39	94.75	2.95	3.14	-0.19	3.04	0.40	0.21	0.19	0.30	0.06	0.83	-0.77	0.45	1.91	1.52	0.39	1.72
val06p1	94.73	95.76	-1.03	95.24	2.74	2.89	-0.14	2.81	0.37	0.65	-0.28	0.51	0.04	0.90	-0.86	0.47	1.76	1.23	0.53	1.50
val07p1	88.78	89.34	-0.56	89.06	3.53	3.57	-0.04	3.55	1.02	0.52	0.50	0.77	0.40	1.62	-1.22	1.01	3.04	4.93	-1.89	3.99
val08p1	90.10	91.36	-1.26	90.73	5.98	6.29	-0.31	6.14	0.25	0.60	-0.35	0.43	0.10	0.74	-0.64	0.42	2.49	0.95	1.54	1.72
val09p1	96.37	97.87	-1.49	97.12	1.94	2.12	-0.18	2.03	0.20	0.23	-0.02	0.21	0.00	0.12	-0.12	0.06	0.23	0.00	0.23	0.12
val10p1	88.78	89.88	-1.10	89.33	3.53	3.54	-0.01	3.54	1.02	0.69	0.33	0.86	0.40	1.21	-0.81	0.81	2.35	4.24	-1.89	3.30
val11p1	84.98	86.33	-1.35	85.66	6.50	6.81	-0.31	6.66	3.00	2.66	0.34	2.83	0.30	1.09	-0.79	0.70	3.51	2.82	0.69	3.17
val12p1	88.68	90.05	-1.37	89.36	3.50	3.54	-0.04	3.52	1.00	0.78	0.22	0.89	0.40	1.22	-0.82	0.81	2.49	4.12	-1.63	3.31
average	-----	-----	-0.86	-----	-----	-----	-0.17	-----	-----	-----	0.09	-----	-----	-----	-0.77	-----	-----	-----	-0.32	-----
stds	-----	-----	0.73	-----	-----	-----	0.13	-----	-----	-----	0.30	-----	-----	-----	0.29	-----	-----	-----	1.27	-----

FT-IR Cell Pressure = 300 mm Hg, abs

Sample	Methane				Ethane				Propane				n-Butane				"Nitrogen"			
FTIR ID	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2
val04p2	94.74	95.04	-0.30	94.89	2.74	2.99	-0.25	2.86	0.37	0.00	0.37	0.19	0.04	0.17	-0.13	0.10	1.85	1.11	0.74	1.48
val05p2	94.55	95.61	-1.06	95.08	2.95	2.95	-0.00	2.95	0.40	0.00	0.40	0.20	0.06	0.16	-0.10	0.11	1.91	0.46	1.45	1.18
val06p2	94.73	94.95	-0.22	94.84	2.74	2.75	-0.01	2.75	0.37	0.00	0.37	0.19	0.04	0.21	-0.17	0.12	1.76	1.52	0.24	1.64
val07p2	88.78	ND	ND	44.39	3.53	ND	ND	1.77	1.02	ND	ND	0.51	0.40	ND	ND	0.20	3.04	ND	ND	1.52
val08p2	90.10	90.57	-0.47	90.34	5.98	6.14	-0.16	6.06	0.25	0.00	0.25	0.13	0.10	0.87	-0.77	0.49	2.49	1.58	0.91	2.04
val09p2	96.37	96.11	0.27	96.24	1.94	2.18	-0.24	2.06	0.20	0.00	0.20	0.10	0.00	0.07	-0.07	0.04	0.23	1.30	-1.07	0.77
val10p2	88.78	89.41	-0.63	89.09	3.53	3.45	0.08	3.49	1.02	0.00	1.02	0.51	0.40	1.23	-0.83	0.82	2.35	4.47	-2.12	3.41
val11p2	84.98	85.82	-0.84	85.40	6.50	6.59	-0.09	6.55	3.00	1.94	1.06	2.47	0.30	1.14	-0.84	0.72	3.51	3.12	0.39	3.32
val12p2	88.68	89.27	-0.59	88.97	3.50	3.43	0.07	3.47	1.00	0.00	1.00	0.50	0.40	1.28	-0.88	0.84	2.49	4.63	-2.14	3.56
average	-----	-----	-0.43	-----	-----	-----	-0.07	-----	-----	-----	0.52	-----	-----	-----	-0.42	-----	-----	-----	-0.18	-----
stds	-----	-----	0.41	-----	-----	-----	0.12	-----	-----	-----	0.40	-----	-----	-----	0.39	-----	-----	-----	1.31	-----

FT-IR Cell Pressure = 500 mm Hg, abs

Sample	Methane				Ethane				Propane				n-Butane				"Nitrogen"			
FTIR ID	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2
val04p3	94.74	95.08	-0.34	94.91	2.74	2.99	-0.25	2.87	0.37	0.40	-0.03	0.38	0.04	0.12	-0.08	0.08	1.85	1.46	0.39	1.66
val05p3	94.55	94.76	-0.21	94.65	2.95	2.89	0.06	2.92	0.40	0.55	-0.15	0.47	0.06	0.00	0.06	0.03	1.91	2.00	-0.09	1.96
val06p3	94.73	95.41	-0.68	95.07	2.74	2.93	-0.19	2.83	0.37	0.51	-0.14	0.44	0.04	0.00	0.04	0.02	1.76	1.29	0.47	1.53
val07p3	88.78	88.22	0.56	88.50	3.53	3.49	0.05	3.51	1.02	1.82	-0.80	1.42	0.40	1.10	-0.70	0.75	3.04	4.92	-1.87	3.98
val08p3	90.10	90.21	-0.11	90.16	5.98	6.16	-0.18	6.07	0.25	0.77	-0.52	0.51	0.10	0.87	-0.77	0.48	2.49	1.81	0.69	2.15
val09p3	96.37	96.76	-0.39	96.57	1.94	2.11	-0.17	2.02	0.20	0.00	0.20	0.10	0.00	0.23	-0.23	0.12	0.23	0.89	-0.66	0.56
val10p3	88.78	88.51	0.27	88.64	3.53	3.43	0.10	3.48	1.02	0.98	0.04	1.00	0.40	1.46	-1.06	0.93	2.35	5.10	-2.75	3.73
val11p3	84.98	84.84	0.14	84.91	6.50	6.72	-0.22	6.61	3.00	3.42	-0.42	3.21	0.30	1.21	-0.91	0.76	3.51	3.87	-0.36	3.69
val12p3	88.68	88.71	-0.03	88.69	3.50	3.41	0.09	3.46	1.00	1.04	-0.04	1.02	0.40	1.38	-0.98	0.89	2.49	5.05	-2.56	3.77
average	-----	-----	-0.09	-----	-----	-----	-0.08	-----	-----	-----	-0.21	-----	-----	-----	-0.51	-----	-----	-----	-0.75	-----
stds	-----	-----	0.3735	-----	-----	-----	0.1474	-----	-----	-----	0.3149	-----	-----	-----	0.4564	-----	-----	-----	1.3210	-----

Notes:
GC = GC calibration data
FTIR = FT-IR derived validation data
GC - FTIR = arithmetic difference = residual error
stds = sample standard deviation
ND = no data
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TABLE A-6b. Validation of Calibration Models for Natural Gas Components

FT-IR Cell Pressure = 700 mm Hg, abs

Sample	Methane				Ethane				Propane				n-Butane				"Nitrogen"			
FTIR ID	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2
val04p4	94.74	95.57	-0.83	95.15	2.74	3.08	-0.34	2.91	0.37	0.79	-0.42	0.58	0.04	0.08	-0.04	0.06	1.85	0.57	1.28	1.21
val05p4	94.55	95.82	-1.27	95.19	2.95	3.11	-0.16	3.03	0.40	0.60	-0.20	0.50	0.06	0.24	-0.18	0.15	1.91	0.29	1.63	1.10
val06p4	94.73	95.59	-0.85	95.16	2.74	2.94	-0.20	2.84	0.37	0.68	-0.31	0.52	0.04	0.09	-0.05	0.06	1.76	0.78	0.98	1.27
val07p4	88.78	88.77	0.01	88.77	3.53	3.62	-0.09	3.58	1.02	2.21	-1.19	1.61	0.40	0.44	-0.04	0.42	3.04	4.09	-1.04	3.56
val08p4	90.10	91.95	-1.85	91.03	5.98	6.24	-0.25	6.11	0.25	2.06	-1.81	1.16	0.10	0.43	-0.33	0.26	2.49	0.00	2.49	1.25
val09p4	96.37	97.27	-0.90	96.82	1.94	2.10	-0.16	2.02	0.20	0.80	-0.60	0.50	0.00	0.00	0.00	0.00	0.23	0.05	0.18	0.14
val10p4	88.78	90.45	-1.67	89.61	3.53	3.54	-0.01	3.54	1.02	2.18	-1.16	1.60	0.40	0.81	-0.41	0.61	2.35	2.90	-0.55	2.62
val11p4	84.98	86.61	-1.63	85.79	6.50	6.52	-0.02	6.51	3.00	4.66	-1.66	3.83	0.30	0.50	-0.20	0.40	3.51	2.00	1.51	2.76
val12p4	88.68	90.56	-1.89	89.62	3.50	3.59	-0.09	3.55	1.00	1.89	-0.89	1.45	0.40	0.88	-0.48	0.64	2.49	2.72	-0.23	2.61
average	-----	-----	-1.21	-----	-----	-----	-0.15	-----	-----	-----	-0.91	-----	-----	-----	-0.19	-----	-----	-----	0.69	-----
stds	-----	-----	0.62	-----	-----	-----	0.11	-----	-----	-----	0.58	-----	-----	-----	0.18	-----	-----	-----	1.16	-----

FT-IR Cell Pressure = 1,000 mm Hg, abs

Sample	Methane				Ethane				Propane				n-Butane				"Nitrogen"			
FTIR ID	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2	GC	FTIR	GC-FTIR	(GC+FTIR)/2
val04p5	94.74	94.22	0.52	94.48	2.74	3.26	-0.52	3.00	0.37	0.26	0.11	0.32	0.04	0.30	-0.26	0.17	1.85	2.09	-0.24	1.97
val05p5	94.55	95.28	-0.73	94.92	2.95	3.26	-0.31	3.10	0.40	0.07	0.33	0.24	0.06	0.32	-0.26	0.19	1.91	0.62	1.29	1.26
val06p5	94.73	94.65	0.08	94.69	2.74	2.96	-0.22	2.85	0.37	0.23	0.14	0.30	0.04	0.22	-0.18	0.13	1.76	1.58	0.18	1.67
val07p5	88.78	87.19	1.59	87.98	3.53	3.85	-0.32	3.69	1.02	2.10	-1.08	1.56	0.40	1.22	-0.82	0.81	3.04	5.84	-2.80	4.44
val08p5	90.10	89.60	0.50	89.85	5.98	6.57	-0.59	6.27	0.25	0.48	-0.23	0.37	0.10	0.84	-0.74	0.47	2.49	3.13	-0.64	2.81
val09p5	96.37	96.17	0.20	96.27	1.94	2.19	-0.25	2.07	0.20	0.05	0.15	0.13	0.00	0.38	-0.38	0.19	0.23	1.46	-1.23	0.85
val10p5	88.78	87.88	0.90	88.33	3.53	4.02	-0.49	3.77	1.02	1.24	-0.22	1.13	0.40	1.56	-1.16	0.98	2.35	5.77	-3.42	4.06
val11p5	84.98	80.46	4.53	82.72	6.50	7.26	-0.76	6.88	3.00	4.48	-1.48	3.74	0.30	0.35	-0.05	0.33	3.51	9.18	-5.67	6.35
val12p5	88.68	89.24	-0.56	88.96	3.50	3.87	-0.37	3.69	1.00	1.44	-0.44	1.22	0.40	1.42	-1.02	0.91	2.49	3.26	-0.77	2.88
average	-----	-----	0.78	-----	-----	-----	-0.42	-----	-----	-----	-0.30	-----	-----	-----	-0.54	-----	-----	-----	-1.48	-----
stds	-----	-----	1.57	-----	-----	-----	0.18	-----	-----	-----	0.61	-----	-----	-----	0.40	-----	-----	-----	2.13	-----

Notes:

GC = GC calibration data
FTIR = FT-IR derived validation data

GC - FTIR = arithmetic difference = residual error
stds = sample standard deviation

ND = no data
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APPENDIX B

Figures

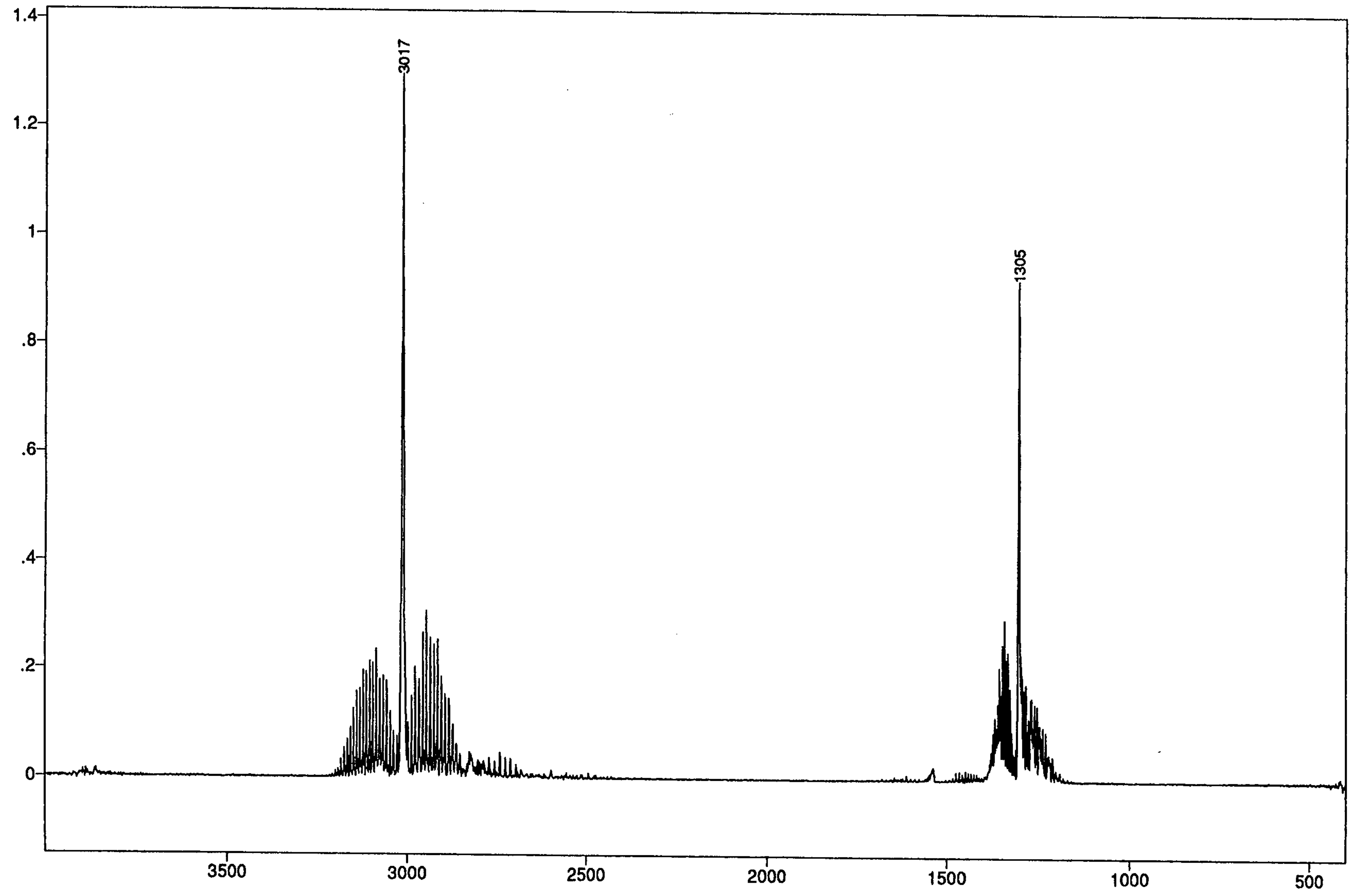

Figure B-1. FT-IR spectrum of methane (P=100 mm Hg)

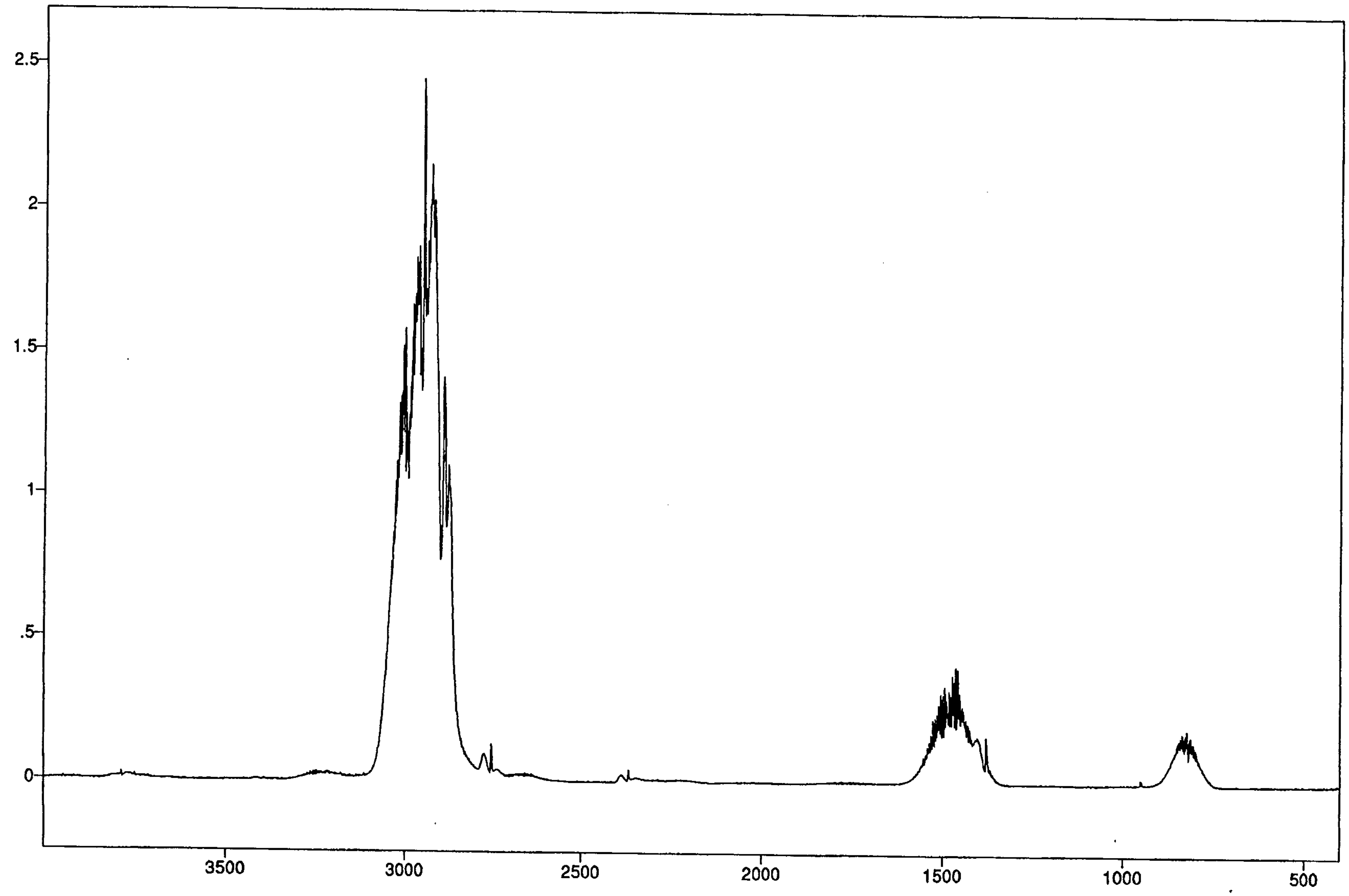

Figure B-2. <u>FT-IR spectrum of ethane (P=100 mm Hg)</u>

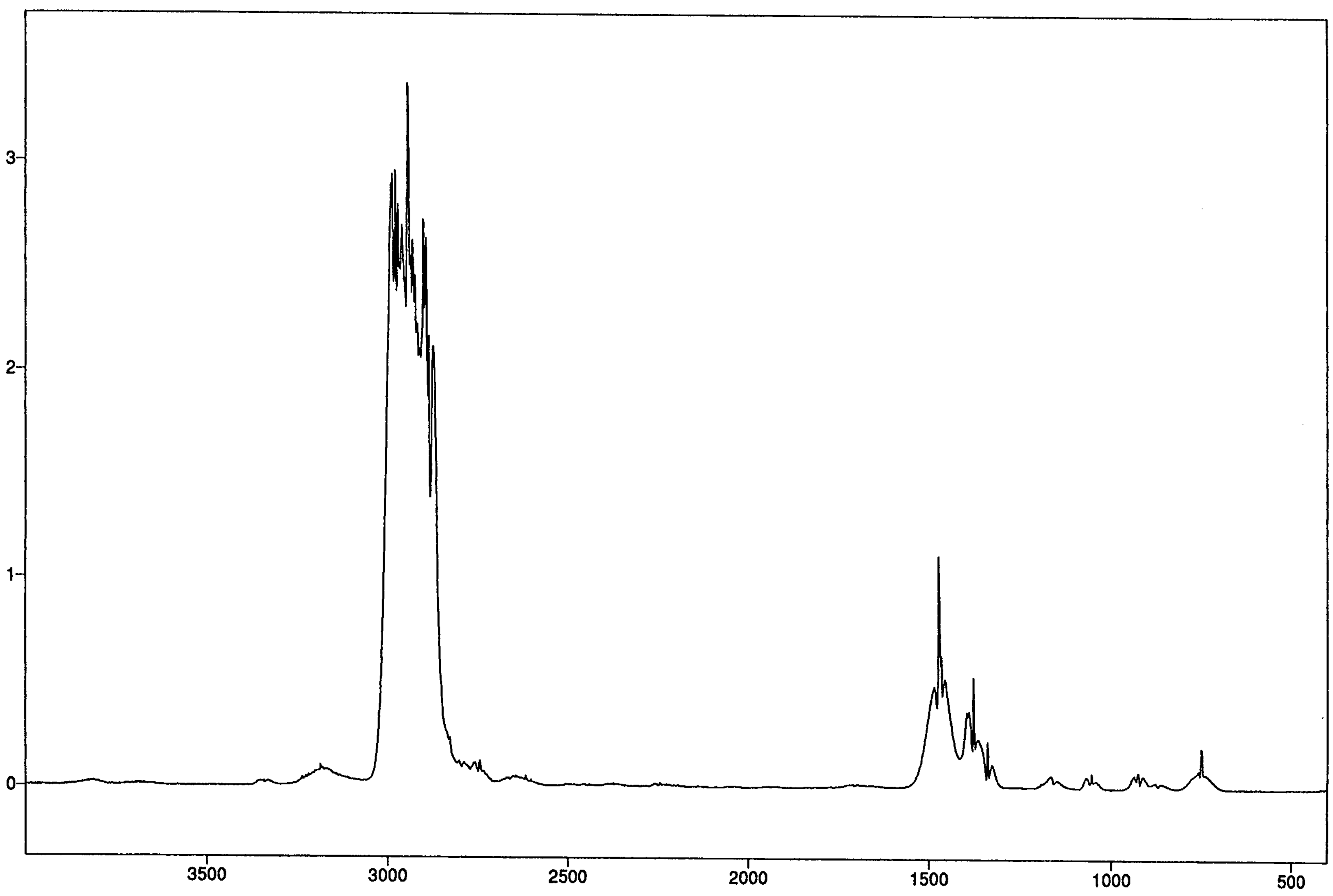

Figure B-3. FT-IR spectrum of propane (P=100 mm Hg)

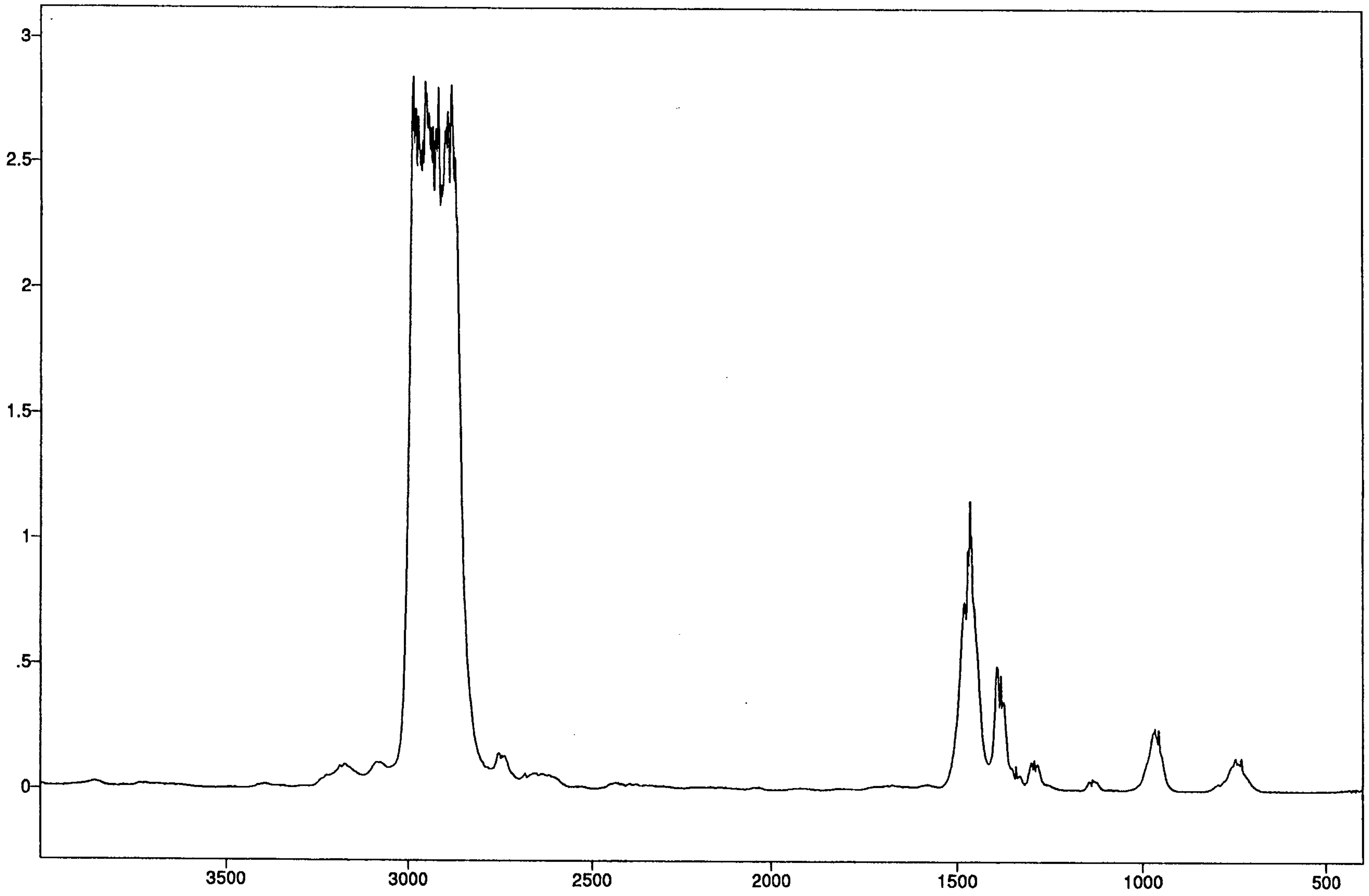

Figure B-4. FT-IR spectrum of butane (P=100 mm Hg)

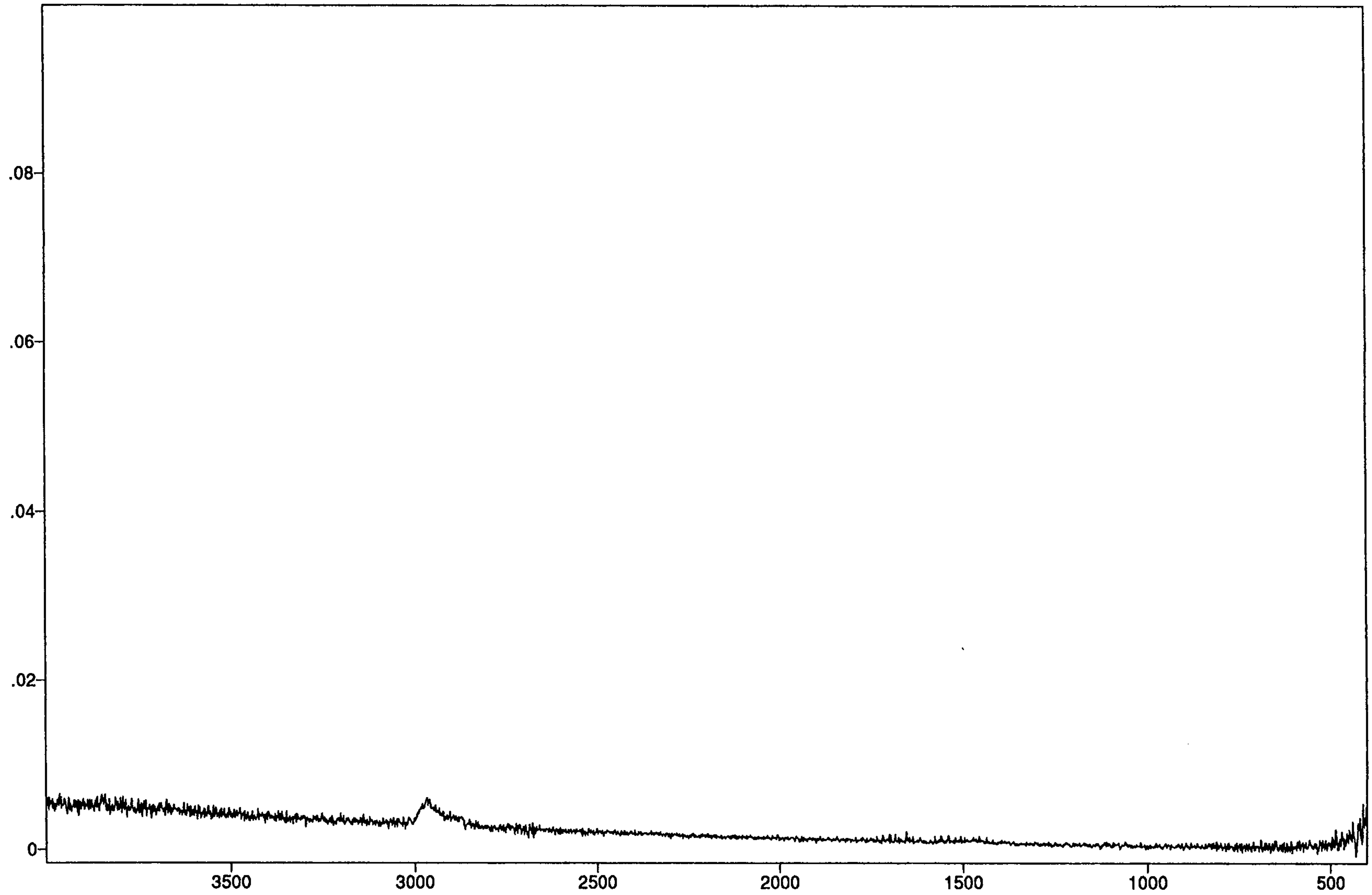

Figure B-5. <u>FT-IR spectrum of nitrogen (P=100 mm Hg)</u>

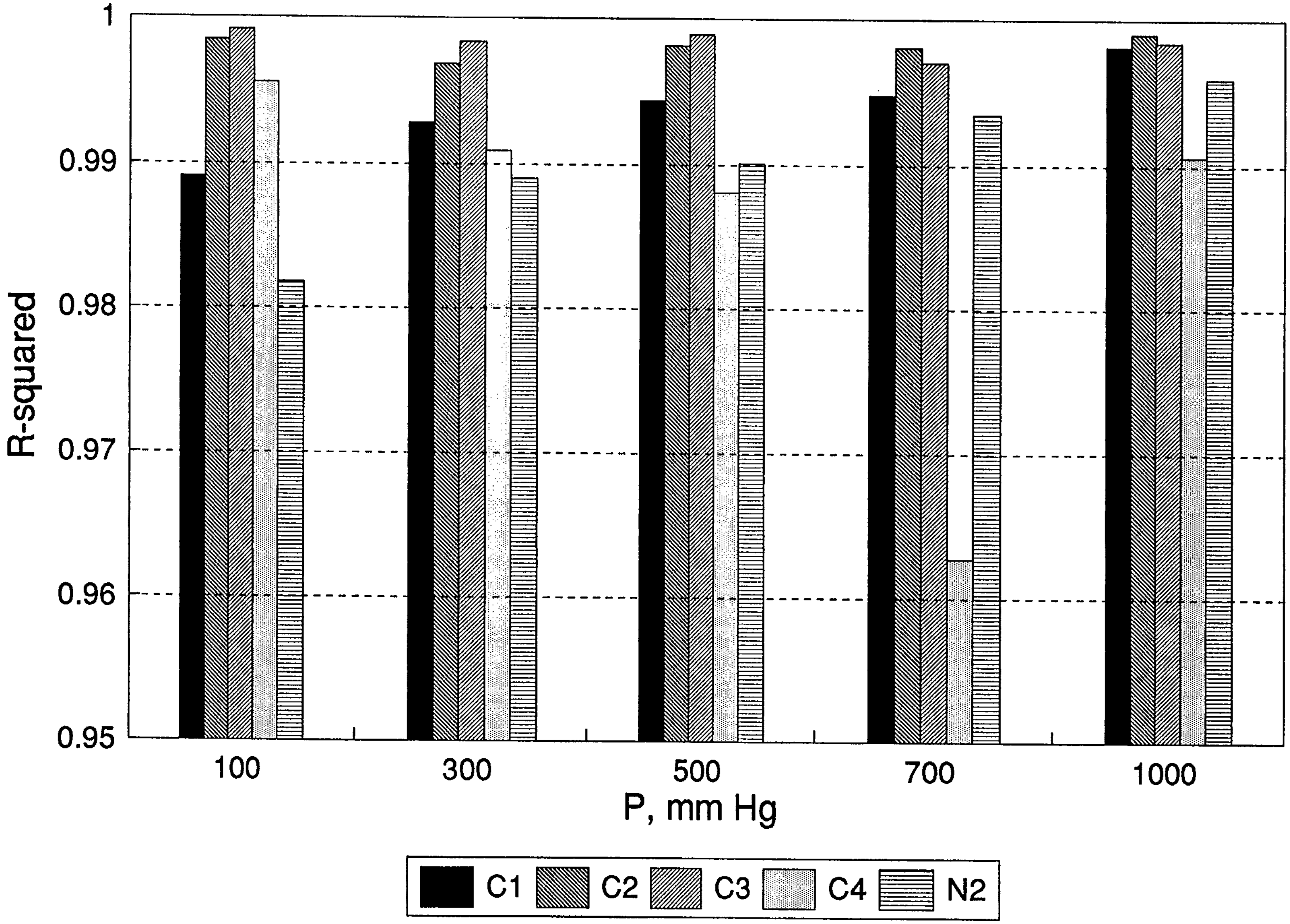

Figure B-6. Effect of cell pressure on R^2 of calibration (F at min. PRESS; pressure-based analysis)

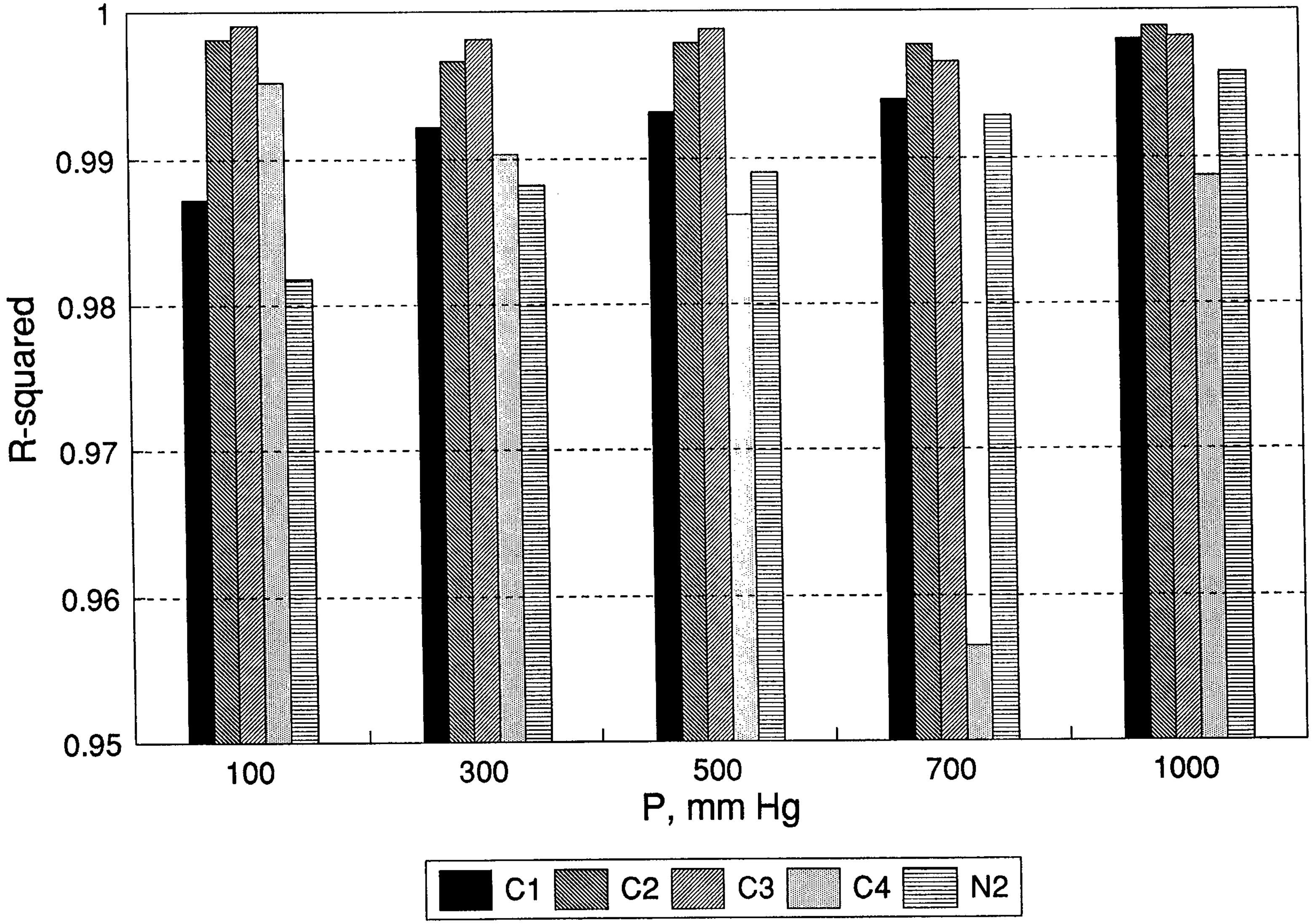

Figure B-7. Effect of cell pressure on R^2 of Calibration (F at $p \leq 0.75$; pressure-based analysis)

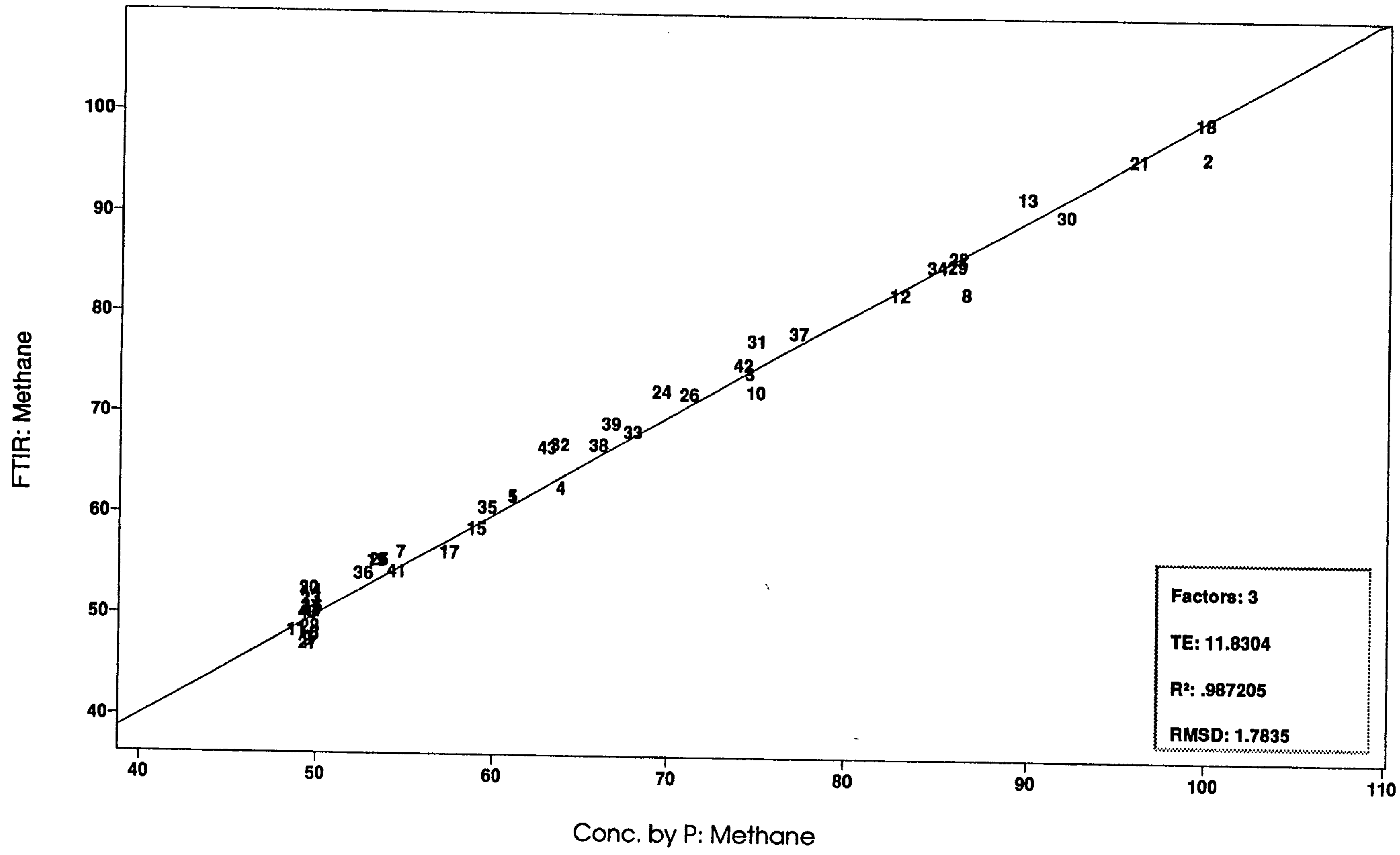

Figure B-8. Calibration for methane at cell P=100 mm Hg (pressure-based analysis)

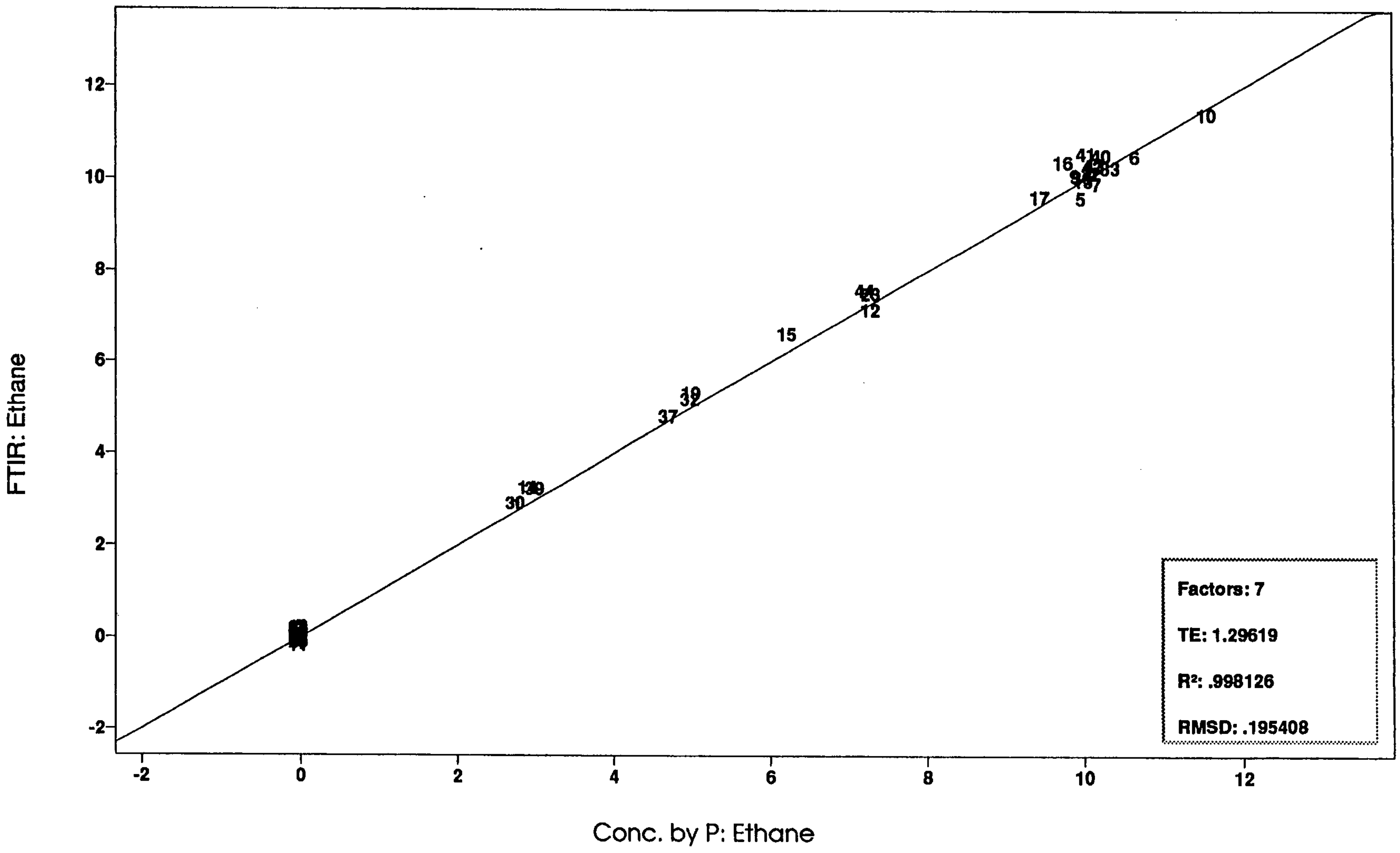

Figure B-9. Calibration for ethane at cell P=100 mm Hg (pressure-based analysis)

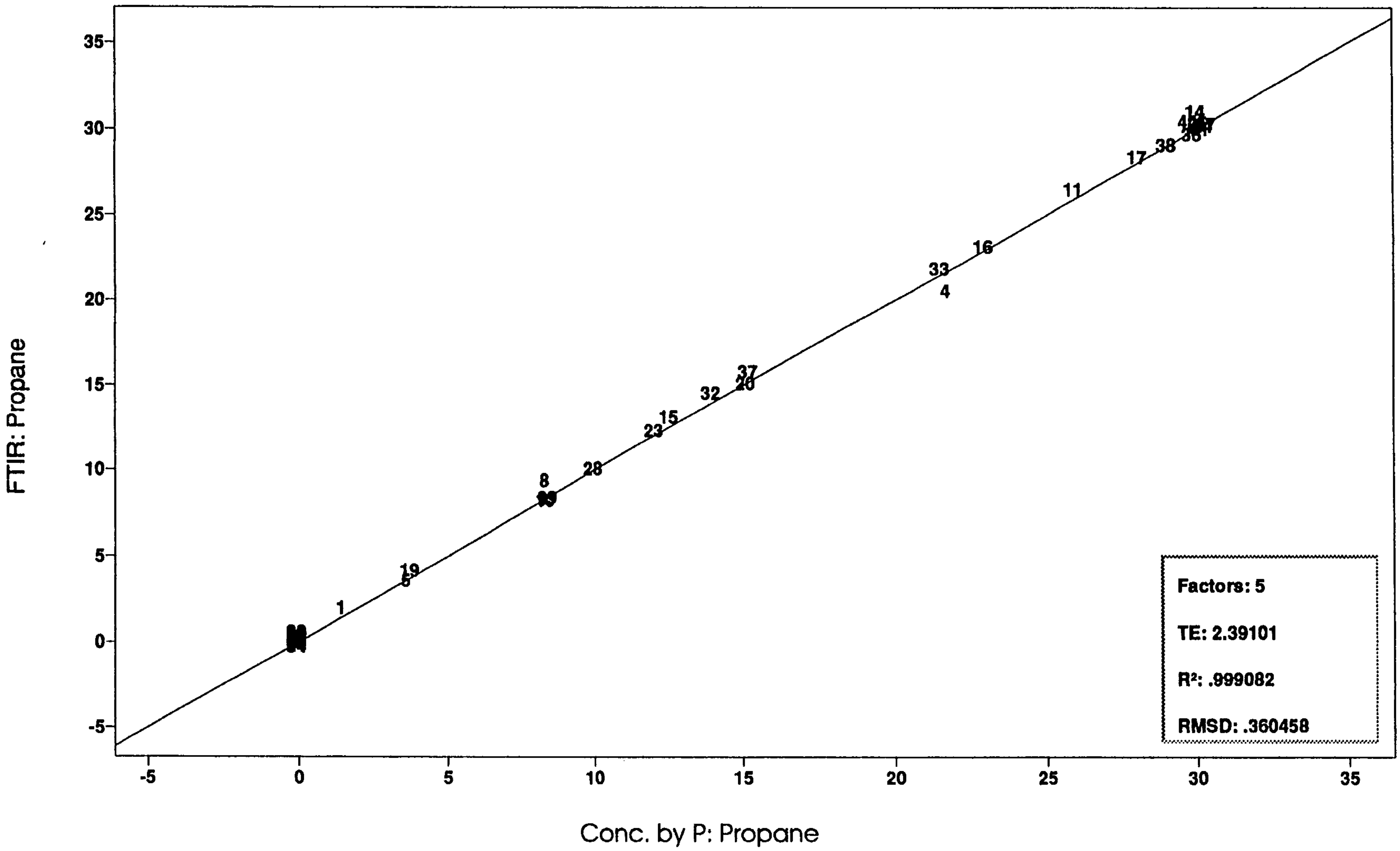

Figure B-10. Calibration for propane at cell P=100 mm Hg (pressure-based analysis)

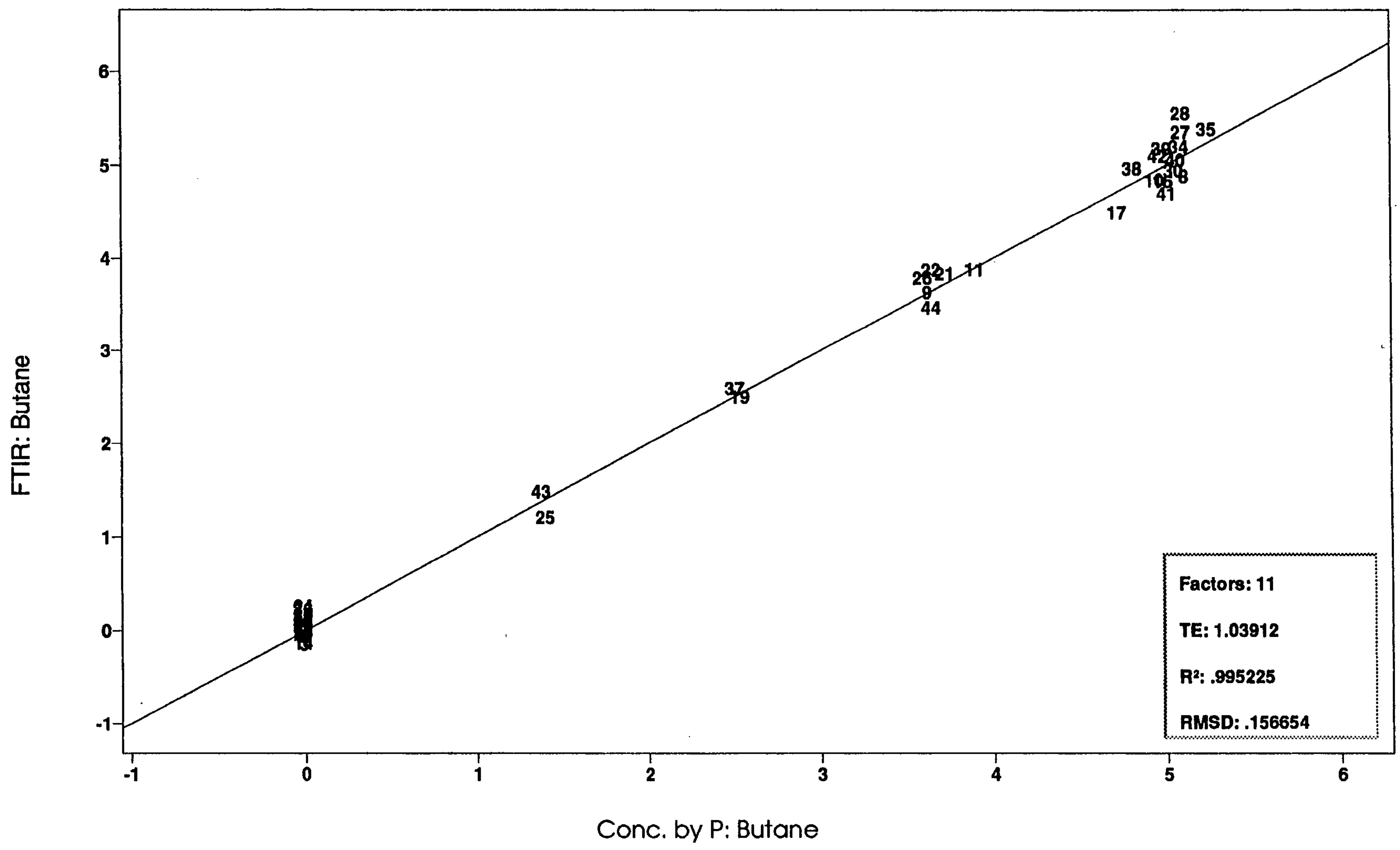

Figure B-11. Calibration for butane at cell P=100 mm Hg (pressure-based analysis)

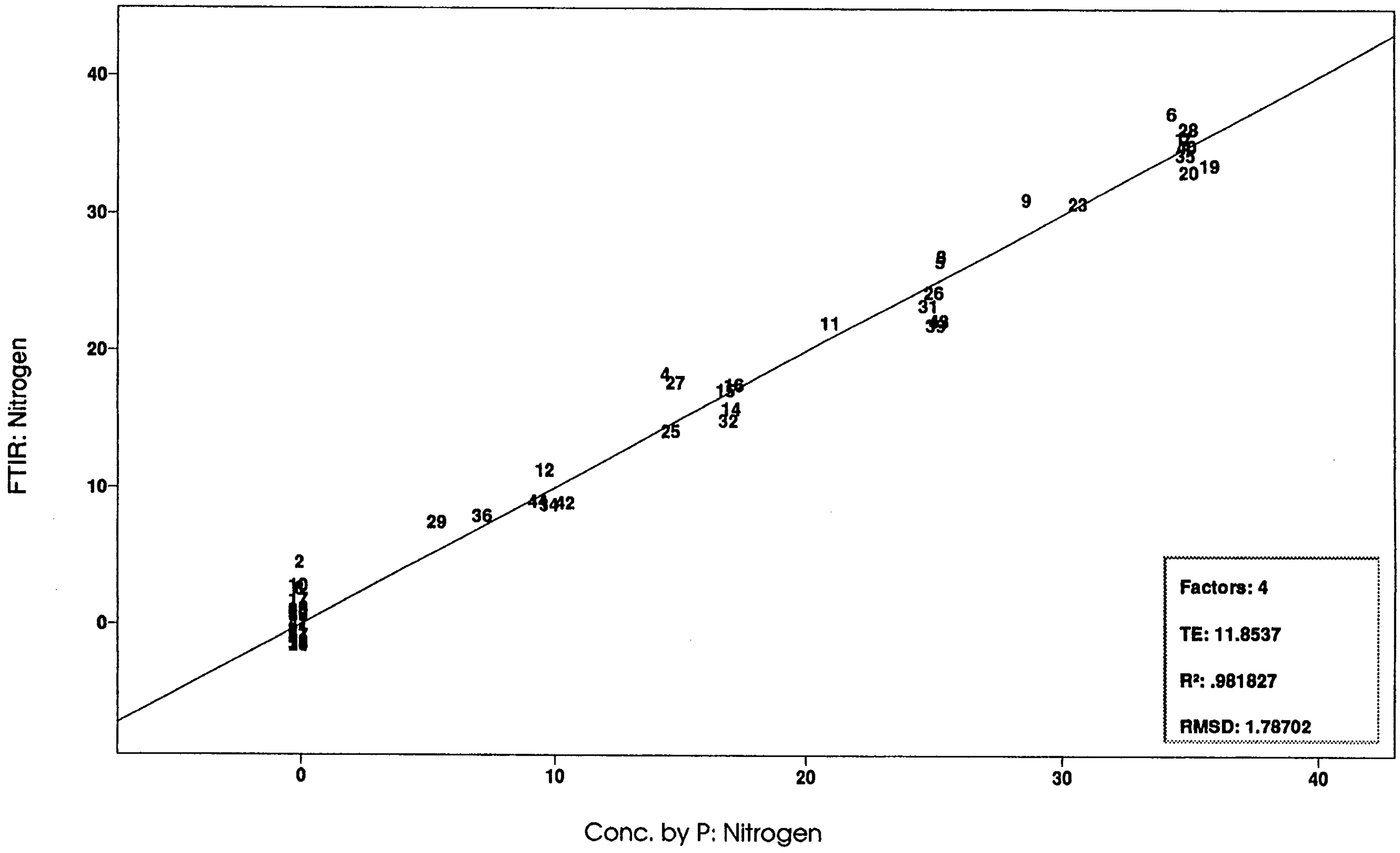

Figure B-12. Calibration for "nitrogen" at cell P=100 mm Hg (pressure-based analysis)

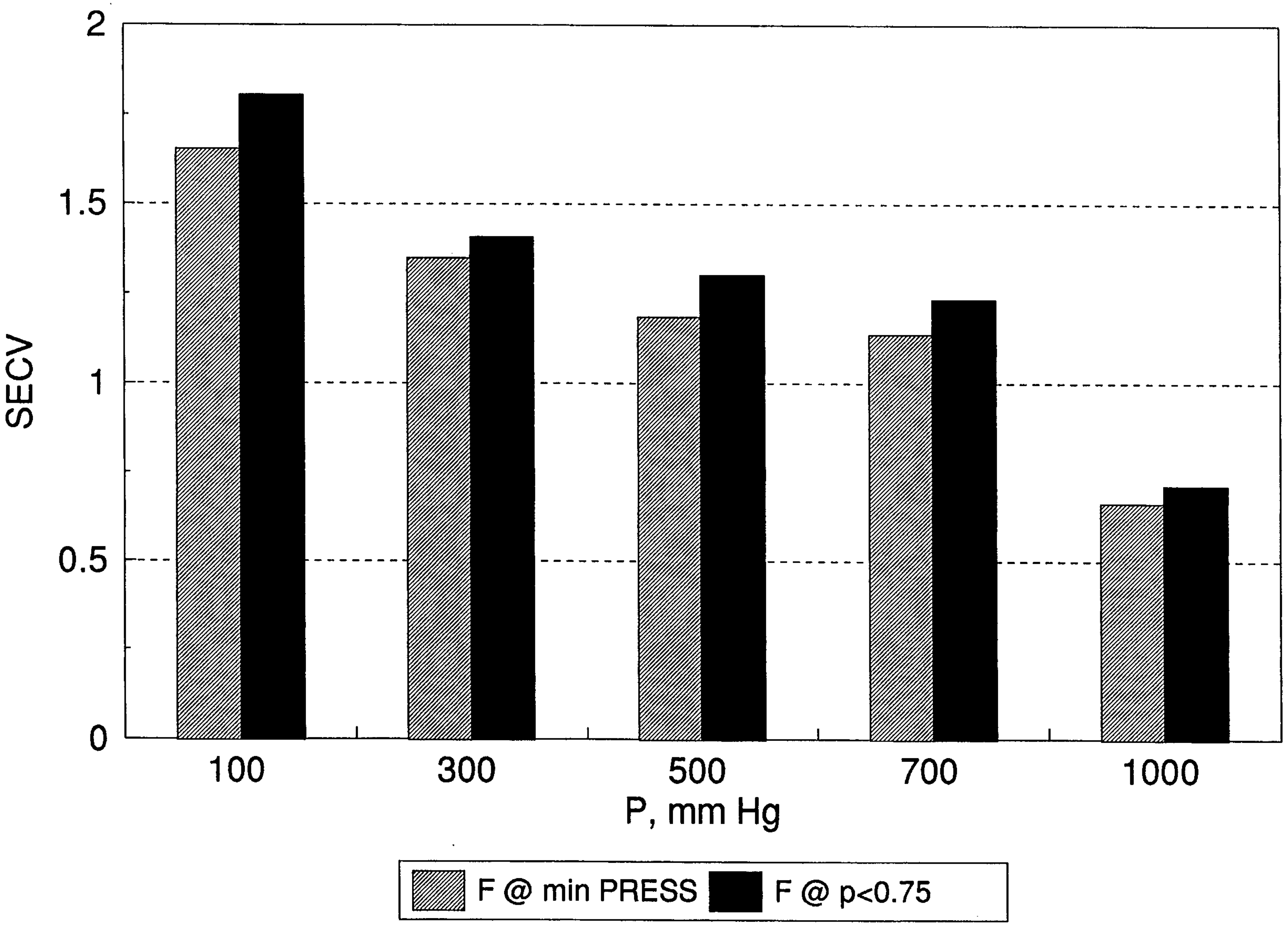

Figure B-13. Effect of cell pressure on SECV for methane (pressure-based analysis)

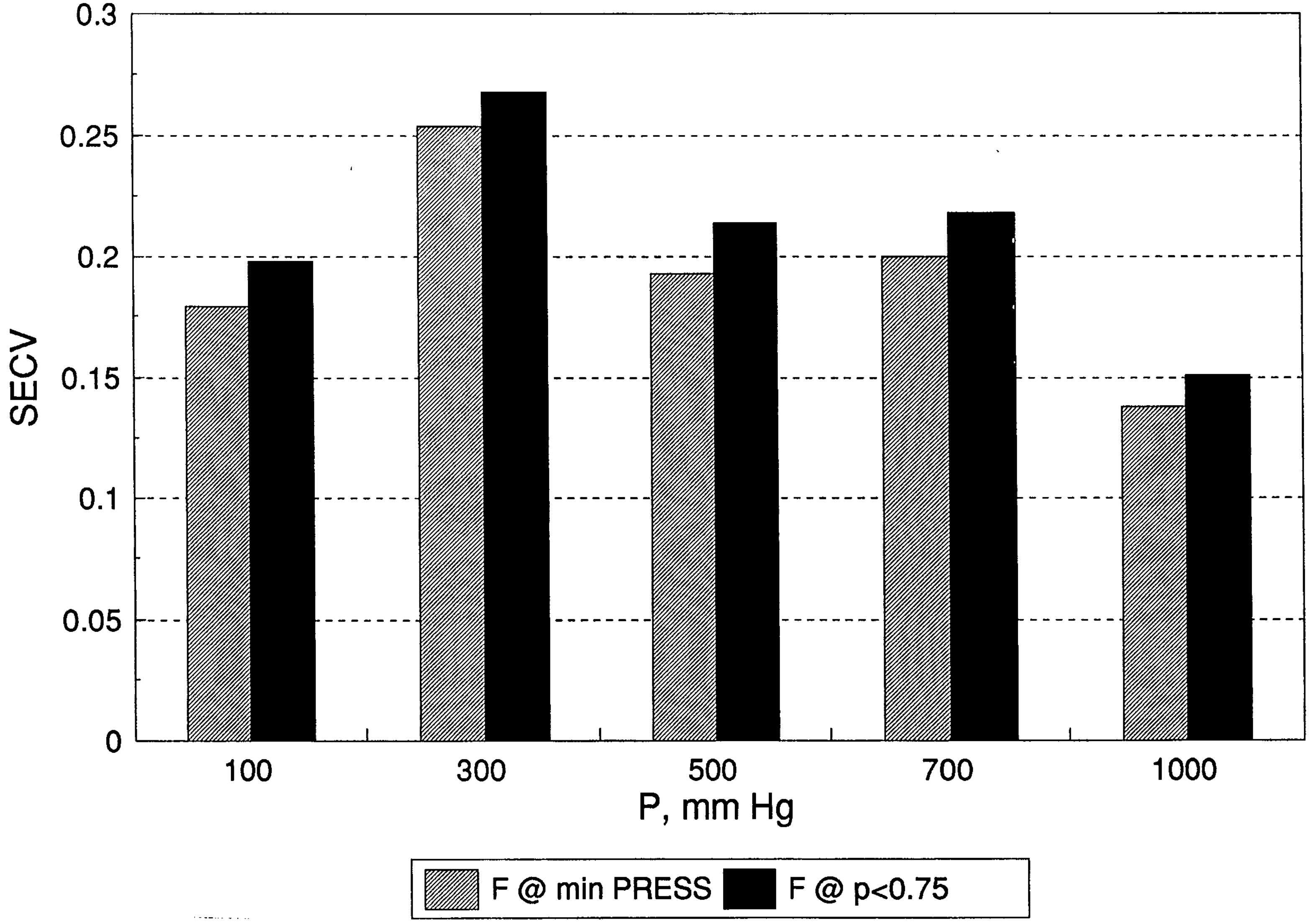

Figure B-14. Effect of cell pressure on SECV for ethane (pressure-based analysis)

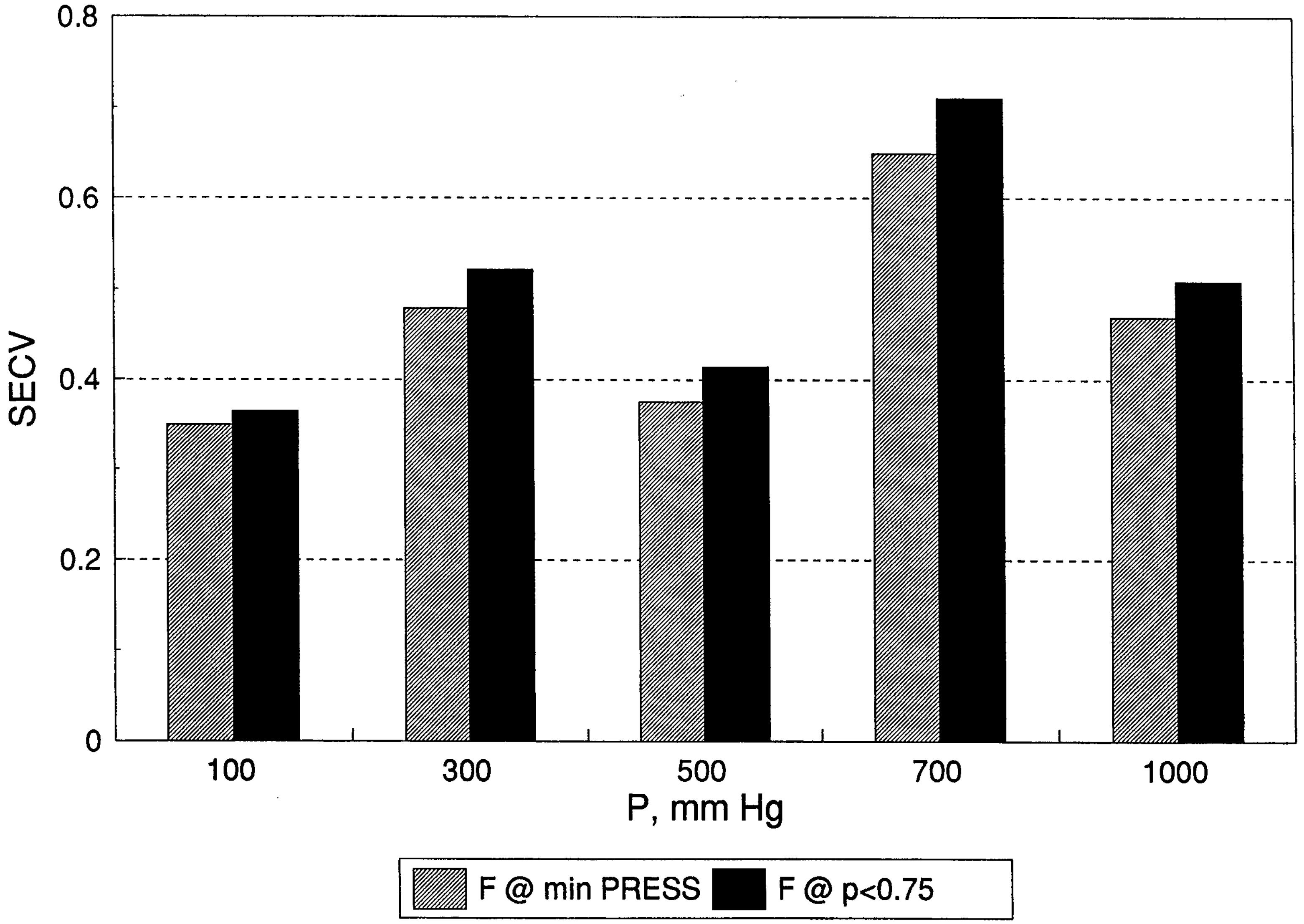

Figure B-15. <u>Effect of cell pressure on SECV for propane (pressure-based analysis)</u>

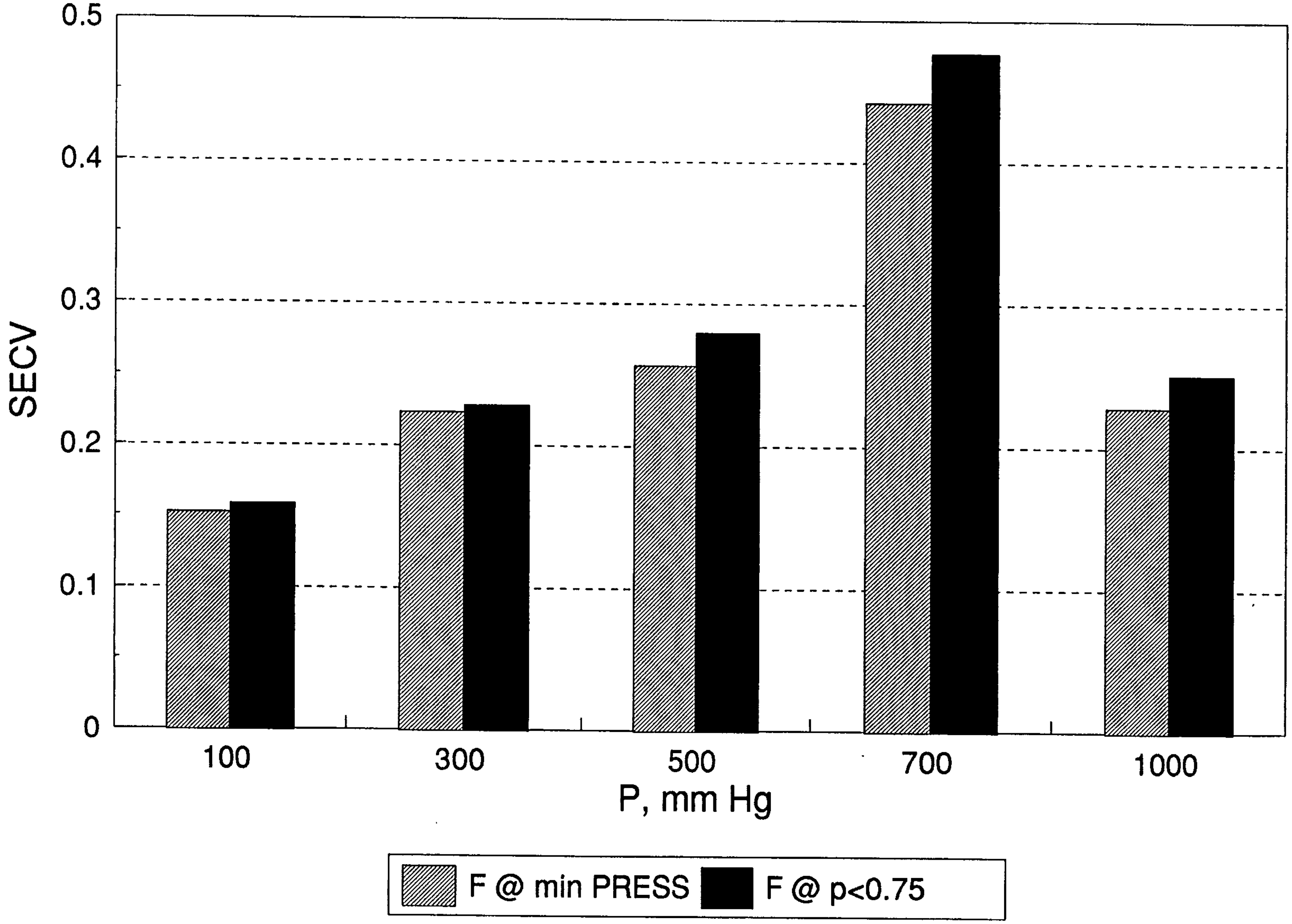

Figure B-16. Effect of cell pressure on SECV for butane (pressure-based analysis)

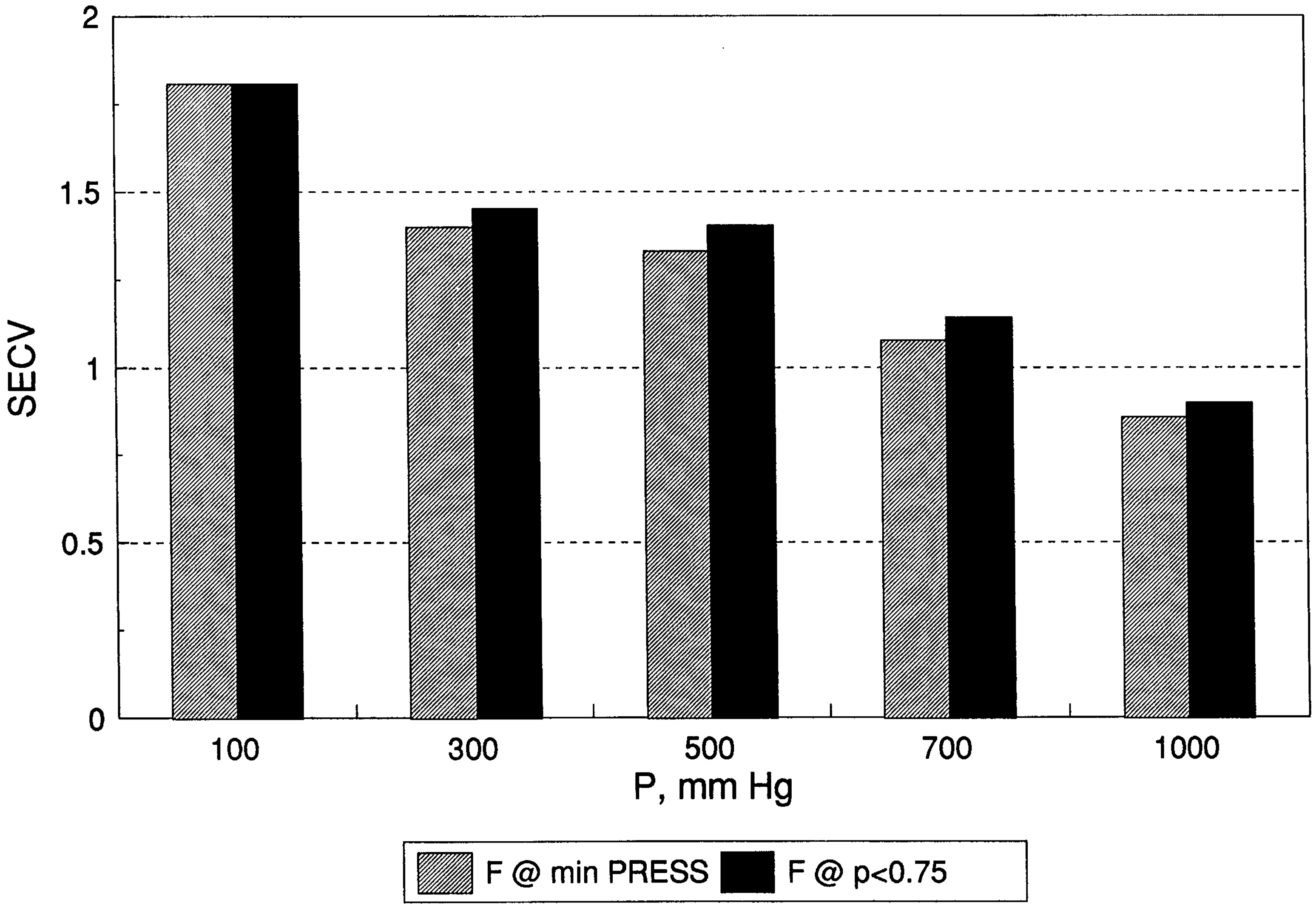

Figure B-17. <u>Effect of cell pressure on SECV for “nitrogen” (pressure-based analysis)</u>

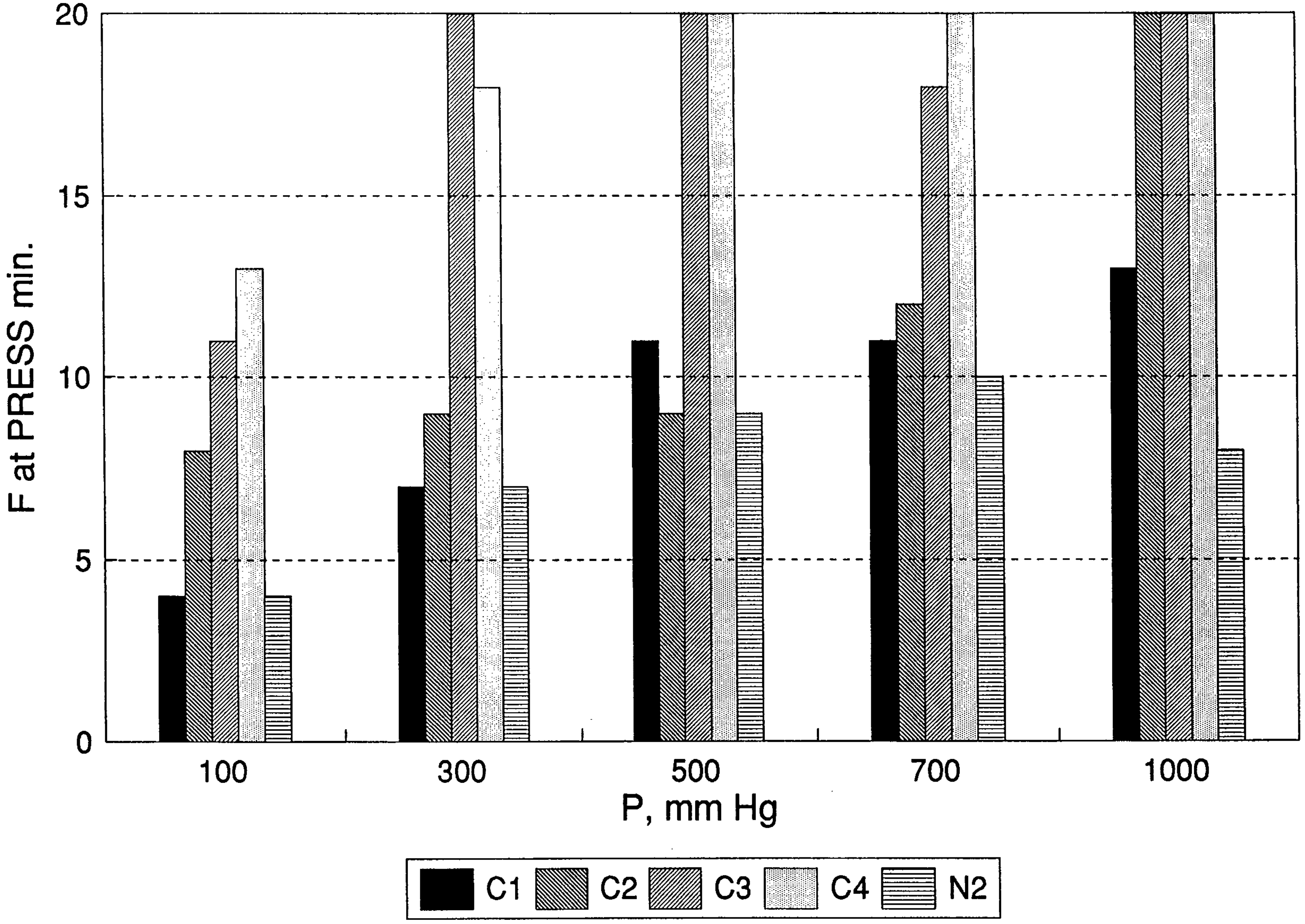

Figure B-18. Effect of cell pressure on the number of factors (F min, PRESS; pressure-based analysis)

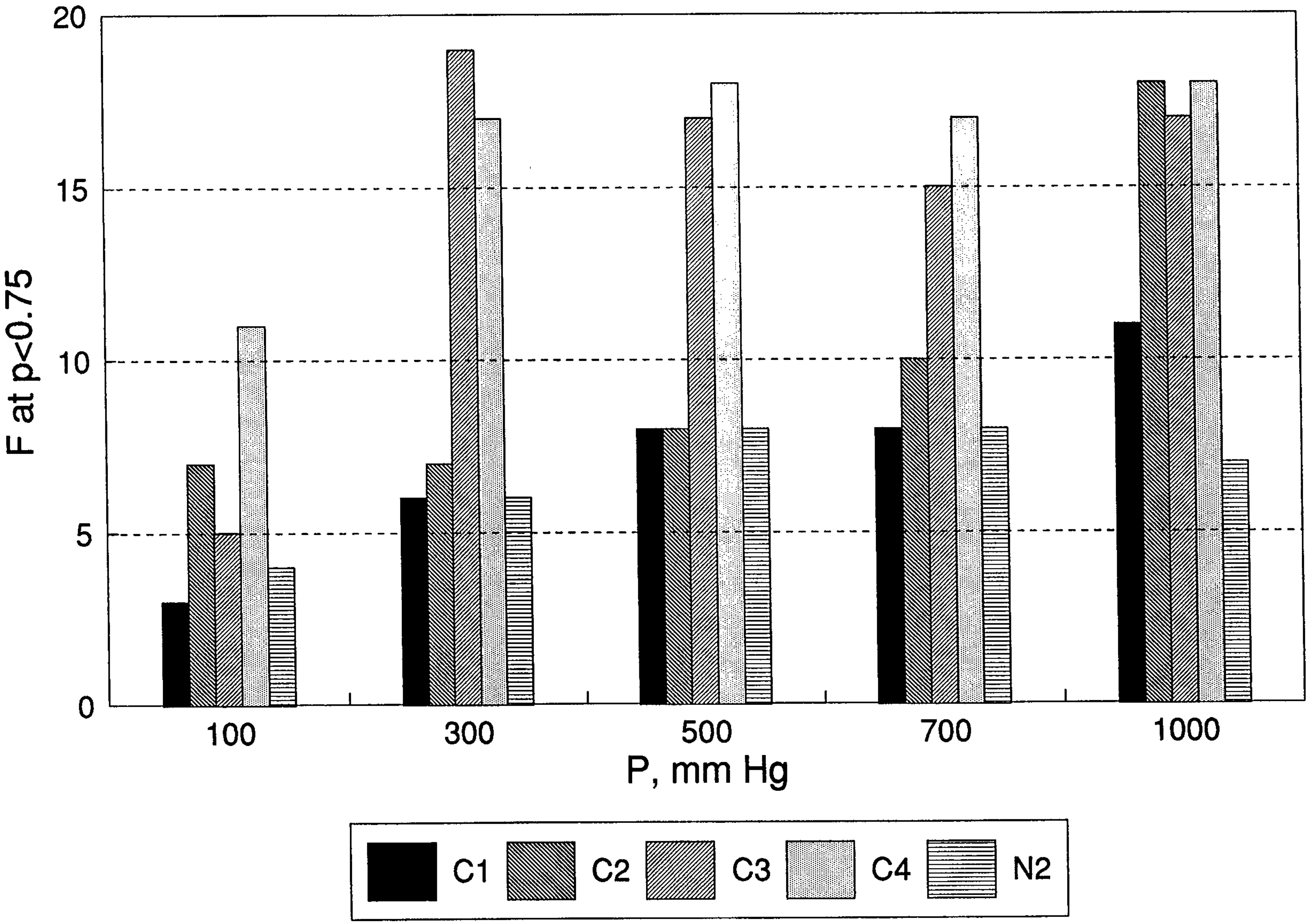

Figure B-19. Effect of cell pressure on the number of factors (F at $p \leq 0.75$; pressure-based analysis)

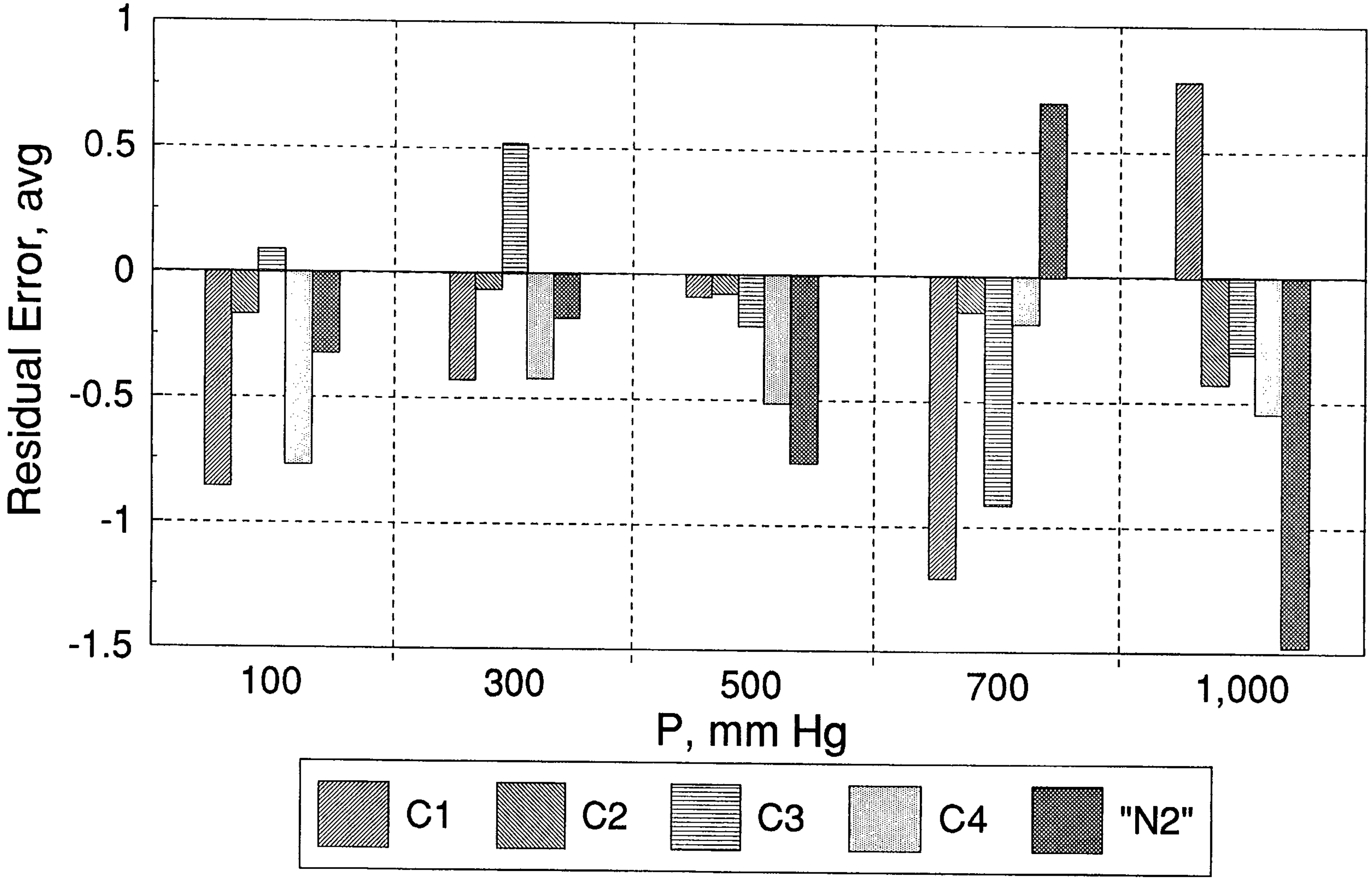

Figure B-20. Effect of cell pressure on the residual error in validation

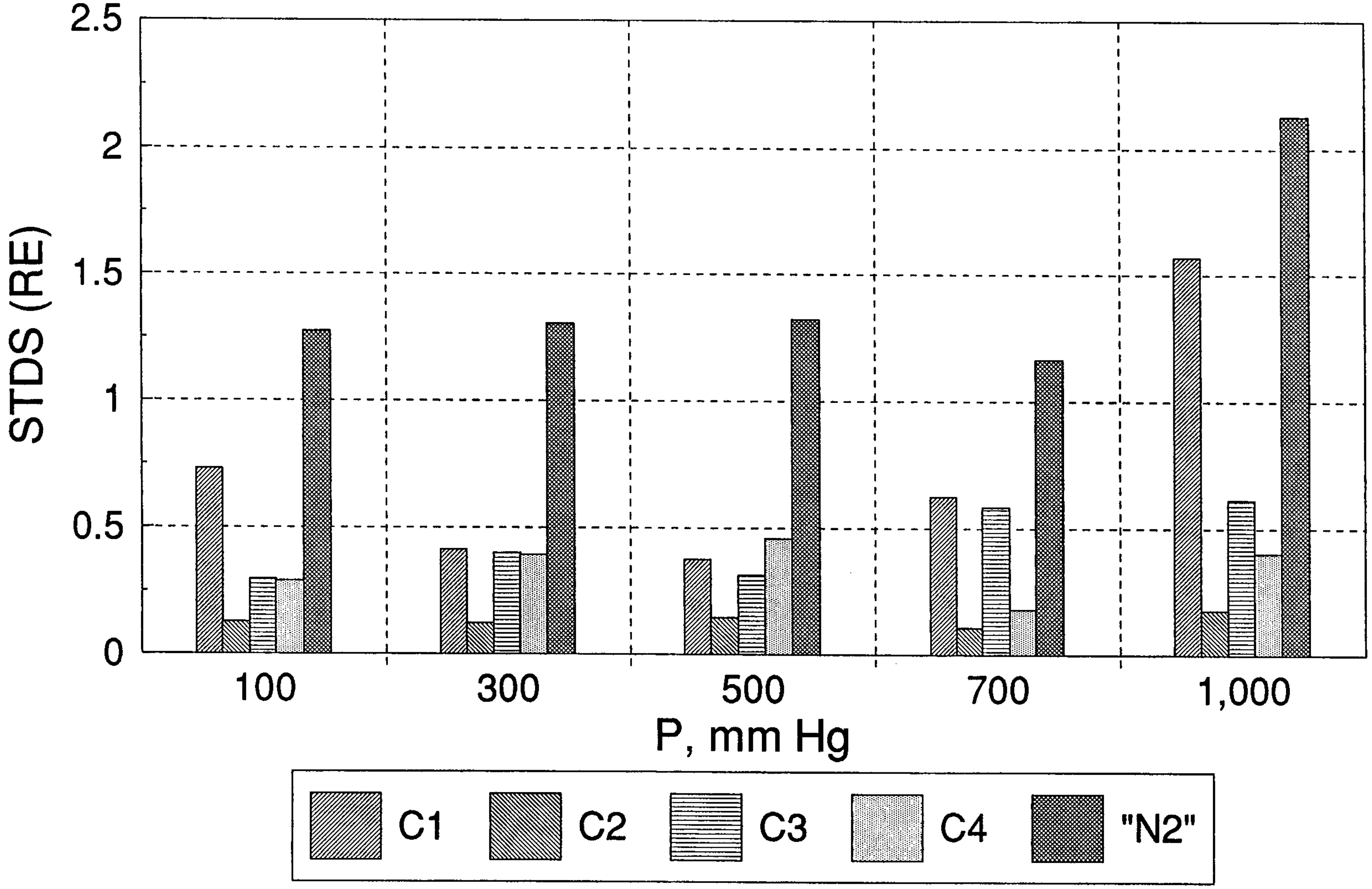

Figure B-21. <u>Effect of cell pressure on the standard deviation of the residual error</u>

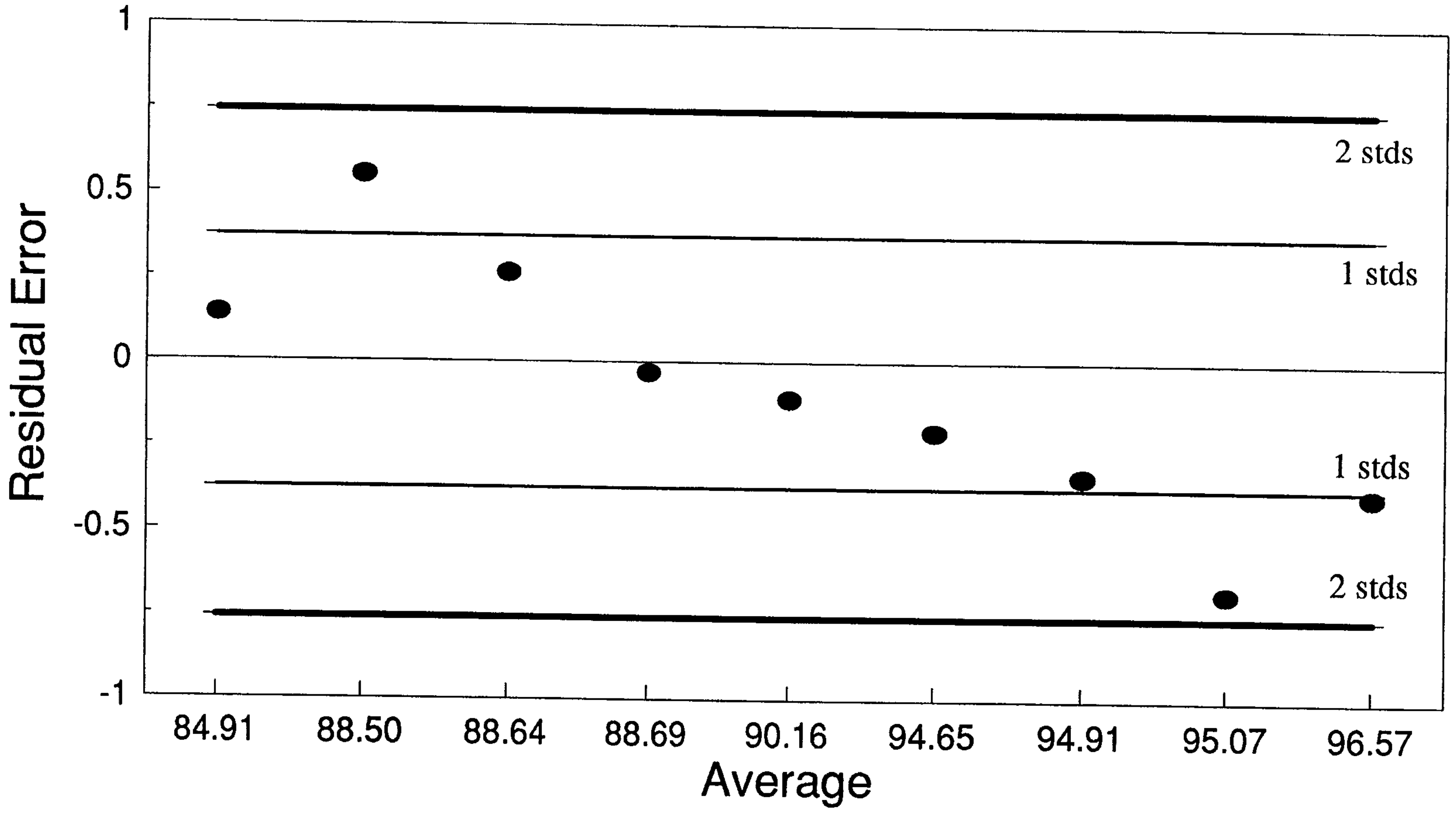

Figure B-22. Limits of agreement for methane validation at cell pressure of 500 mm Hg, abs

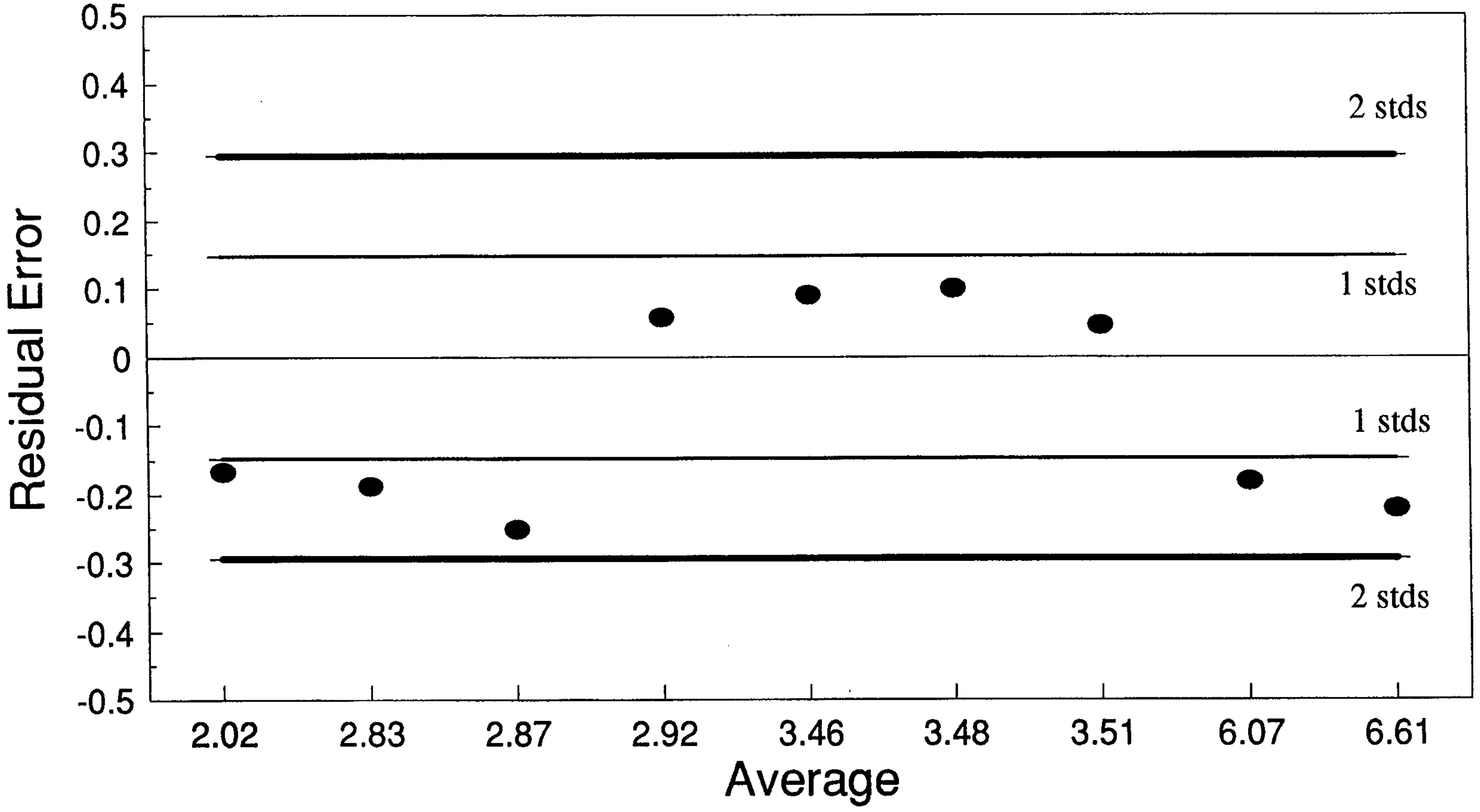

Figure B-23. Limits of agreement for ethane validation at cell pressure of 500 mm Hg, abs

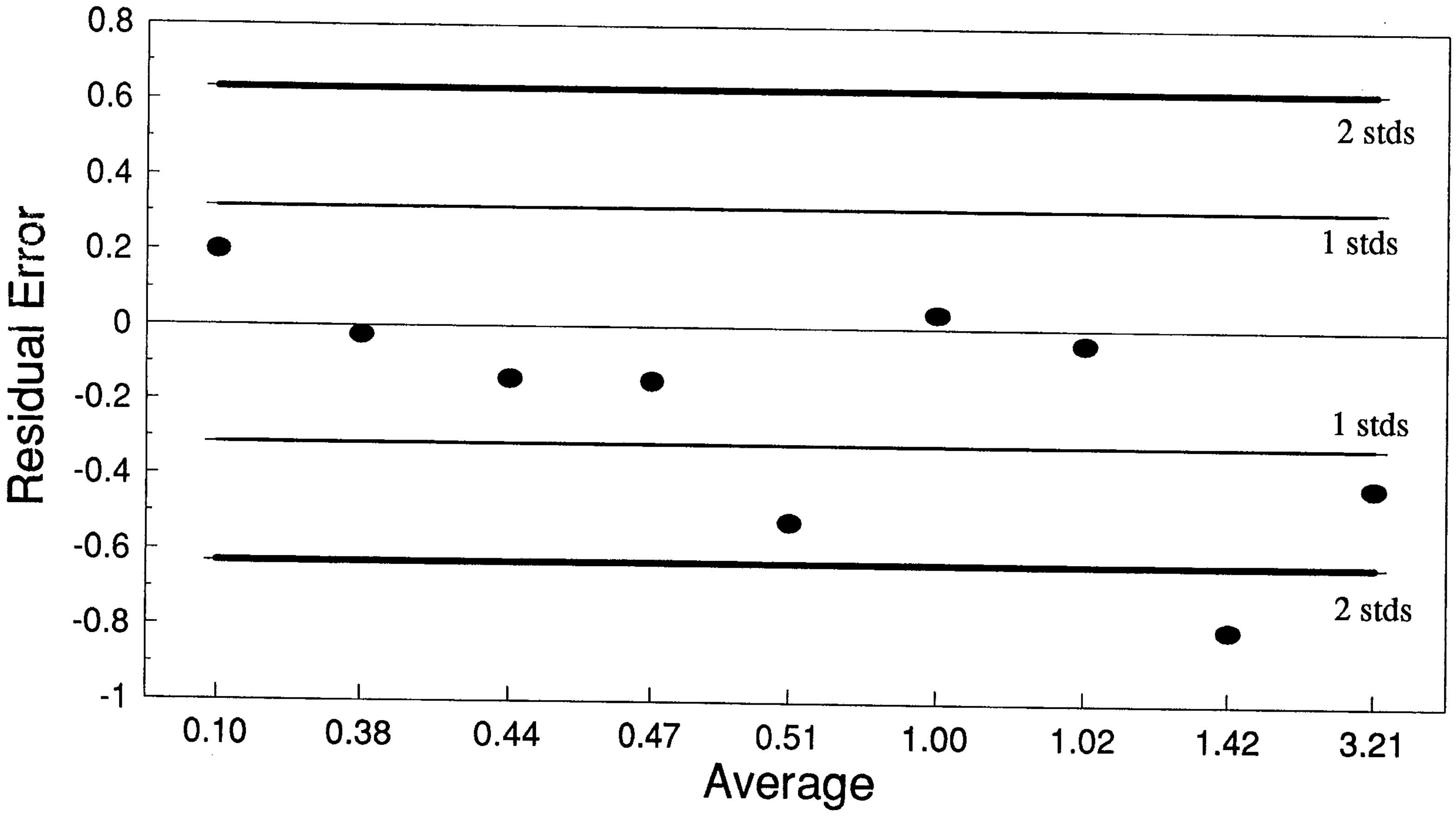

Figure B-24. Limits of agreement for propane validation at cell pressure of 500 mm Hg, abs

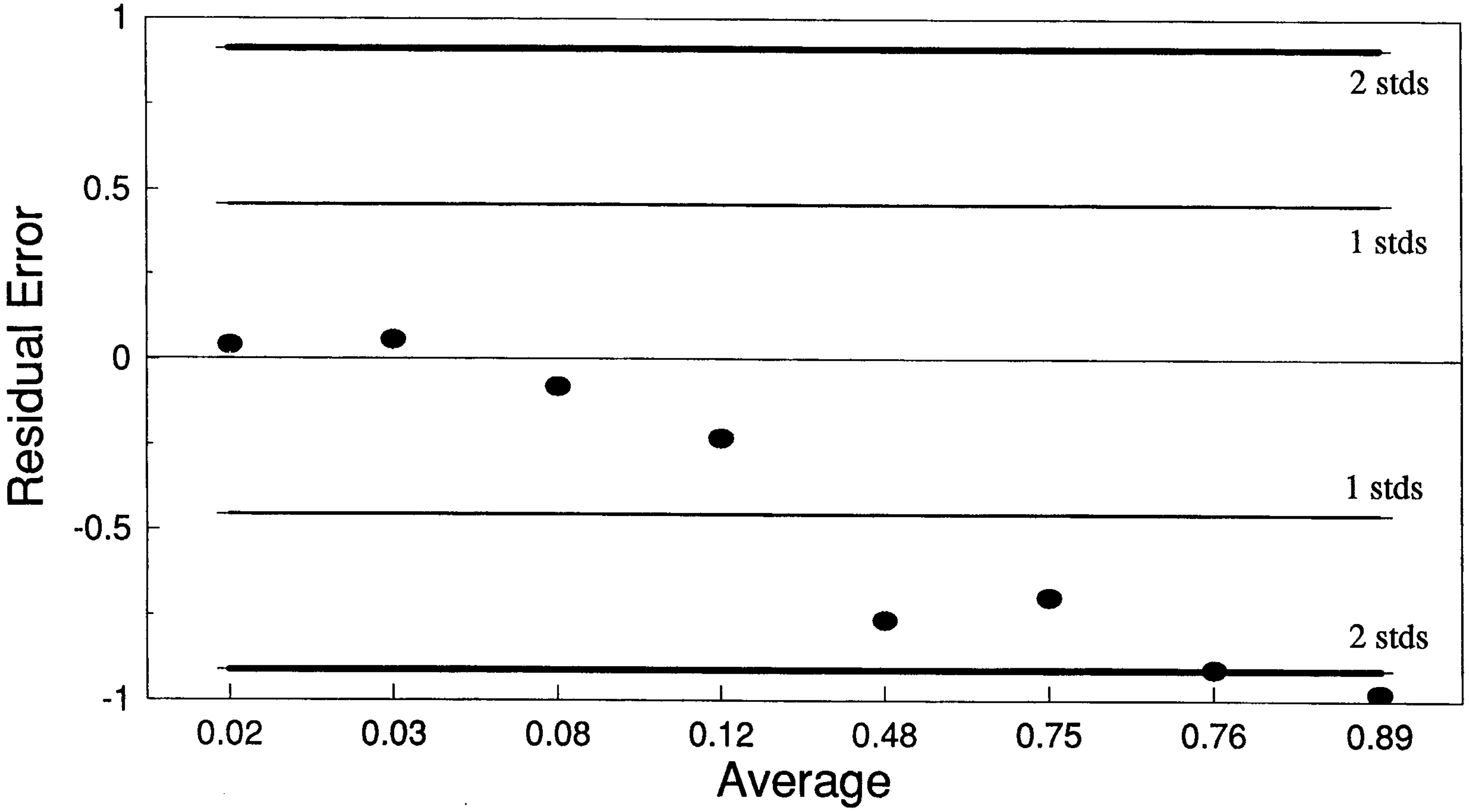

Figure B-25. <u>Limits of agreement for butane validation at cell pressure of 500 mm Hg, abs</u>

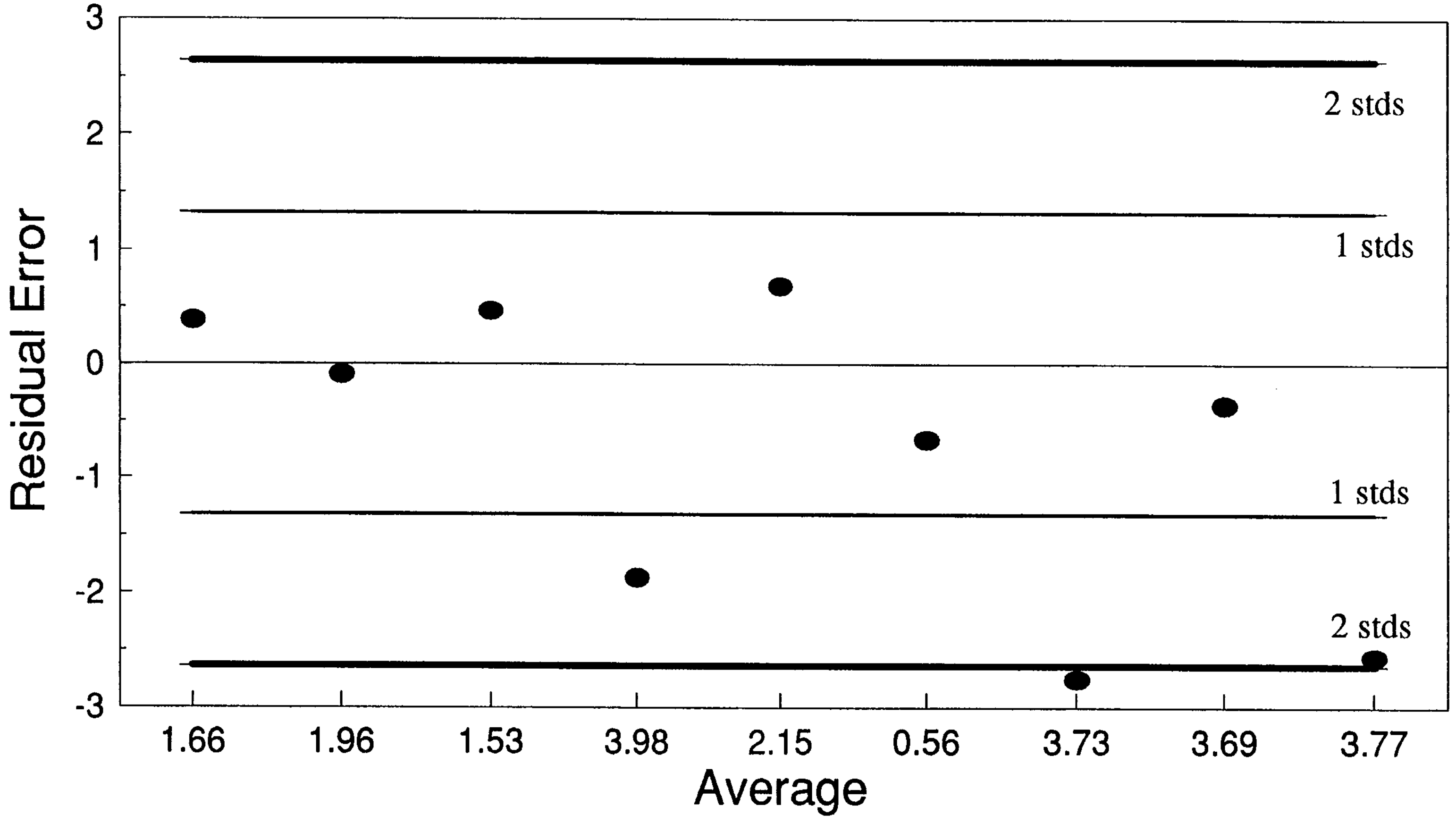

Figure B-26. Limits of agreement for "nitrogen" validation at cell pressure of 500 mm Hg, abs

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