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BREATH-COLLECTION DEVICE FOR DELAYED BREATH-ALCOHOL ANALYSIS

Part 2

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16. Abstract

This report includes the details of a study to develop, evaluate, and validate a breath collection device (BCD) for delayed breath-alcohol analysis. Primary applications of the BCD include collection of breath-alcohol samples for field surveys or for later laboratory analysis for independent verification of evidential breath-alcohol tests for traffic law enforcement purposes, and research uses.

The work involved development of a BCD consisting of a molecular sieve 13X column as the alcohol sorbent for 250 ml. samples, development of a method of analysis for the stored alcohol, and both in-vitro and in-vivo evaluation of the delayed analysis system. Alcohol analysis was performed by automated gas chromatographic headspace analysis on the aqueous alcohol eluate from the molecular sieve. Evaluations included studies with precision-simulator effluents which showed essentially complete recovery of the alcohol, and studies with 32 alcohol-consuming human subjects. Results of direct breath-alcohol analysis with a Gas Chromatographic Intoximeter and of delayed gas chromatographic analyses after intervening alcohol sorption on molecular sieve 13X, on 369 series-sampled split breath specimens, were essentially identical with a mean difference of 0.00032 g/210 L. Complete alcohol retention for at least 11 months was shown to be feasible.

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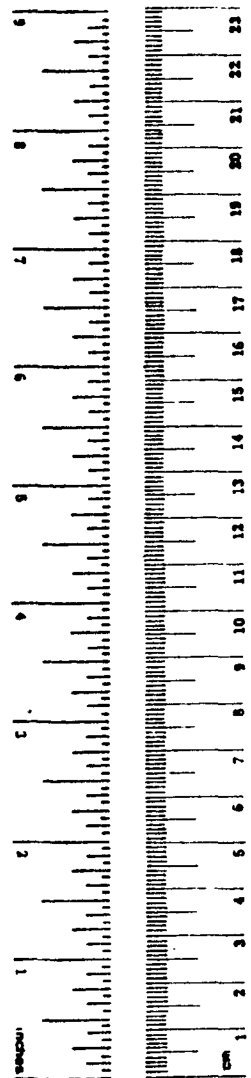
METRIC CONVERSION FACTORS

Approximate Conversions to Metric Measures

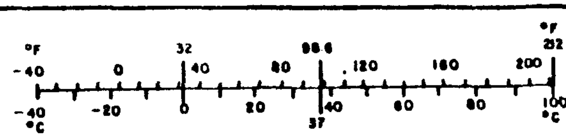
Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
in	inches	*2.5	centimeters	cm
ft	feet	30	centimeters	cm
yd	yards	0.9	meters	m
mi	miles	1.6	kilometers	km
		AREA		
in^2	square inches	6.5	square centimeters	cm^2
ft^2	square feet	0.09	square meters	m^2
yd^2	square yards	0.8	square meters	m^2
mi^2	square miles	2.6	square kilometers	km^2
	acres	0.4	hectares	ha
		MASS (weight)		
oz	ounces	28	grams	g
lb	pounds	0.45	kilograms	kg
	short tons (2000 lb)	0.9	tonnes	t
		VOLUME		
tsp	teaspoons	5	milliliters	ml
Tbsp	tablespoons	15	milliliters	ml
fl oz	fluid ounces	30	milliliters	ml
c	cups	0.24	liters	l
pt	pints	0.47	liters	l
qt	quarts	0.95	liters	l
gal	gallons	3.8	liters	l
ft^3	cubic feet	0.03	cubic meters	m^3
yd^3	cubic yards	0.76	cubic meters	m^3
		TEMPERATURE (exact)		
°F	Fahrenheit temperature	5/9 (after subtracting 32)	Celsius temperature	°C

*1 in ≡ 2.54 (exactly). For other exact conversions and more detailed tables, see NBS Misc. Publ. 286, Units of Weights and Measures, Price $2.25, SD Catalog No. C13.10:286.



Approximate Conversions from Metric Measures

Symbol	When You Know	Multiply by	To Find	Symbol
		LENGTH		
mm	millimeters	0.04	inches	in
cm	centimeters	0.4	inches	in
m	meters	3.3	feet	ft
m	meters	1.1	yards	yd
km	kilometers	0.6	miles	mi
		AREA		
cm^2	square centimeters	0.16	square inches	in^2
m^2	square meters	1.2	square yards	yd^2
km^2	square kilometers	0.4	square miles	mi^2
ha	hectares (10,000 m^2)	2.5	acres	
		MASS (weight)		
g	grams	0.035	ounces	oz
kg	kilograms	2.2	pounds	lb
t	tonnes (1000 kg)	1.1	short tons	
		VOLUME		
ml	milliliters	0.03	fluid ounces	fl oz
l	liters	2.1	pints	pt
l	liters	1.06	quarts	qt
l	liters	0.26	gallons	gal
m^3	cubic meters	35	cubic feet	ft^3
m^3	cubic meters	1.3	cubic yards	yd^3
		TEMPERATURE (exact)		
°C	Celsius temperature	9/5 (then add 32)	Fahrenheit temperature	°F



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INTRODUCTION

Since the earliest applications of breath-alcohol[1] analysis in traffic law enforcement in the United States, attempts have been made to collect the alcohol contents of breath specimens in the field for subsequent laboratory analysis at a remote site. In part, those efforts reflected the belief of the law enforcement community and of forensic scientists that constitutional due process and equal protection considerations required the retention and preservation of aliquots (or their functional equivalents) of officially-analyzed specimens whose analysis results were to be introduced in evidence.

The legal principle involved was formally set forth in 1963[2], and there has been an accelerating trend to urge application of that principle to quantitative evidential breath-alcohol analyses, especially since the 1974 *Hitch* decision (1). The problem has become urgent and widespread. At least one state - Vermont - statutorily requires retention of a breath specimen for "no more than 30 days" from the date of sampling when breath is the specimen taken under its Implied Consent law (2); and no trial involving chemical test evidence may be commenced in Vermont before the 30-day sample retention period has expired, absent an express waiver by the defense (3). Elsewhere, a growing body of recent appellate court decisions holds that a defendant charged with an alcohol-related traffic offense is constitutionally entitled to the opportunity for an independent scientific laboratory evaluation of a breath-alcohol test to be

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[1] In this report, the unmodified term *alcohol* refers to ethanol; *BrAC* refers to breath-alcohol concentration.

[2] *Brady vs. Maryland* (373 U.S. S. Ct., 1963): Suppression by the prosecution of material evidence favorable to an accused who has requested it violates due process, irrespective of the good or bad faith of the prosecution.

introduced in evidence against him. Leading cases to that effect in state courts of last resort are *Garcia vs. District Court* in Colorado, 1979 (4) and *Baca vs. Smith* in Arizona, 1980 (5), both involving the right to a retained breath specimen, or its equivalent, under defined conditions. The trend toward adopting the doctrine of breath preservation as a duty will clearly grow rapidly once such preservation has been authoritatively shown to be feasible and reliable. Routine retention of breath-alcohol specimens in forensic practice was proposed as follows by Mason and Dubowski (6):

> "It is strongly recommended that the federal requirements for approval of an evidential breath tester be amended to provide that a portion of the breath specimen, substantially identical with that actually analyzed, be saved (for example, trapped by an adsorbent) for later confirmatory analysis if such is desirable or necessary."

There are, of course, many actual and potential nonforensic applications for delayed analysis of breath-alcohol specimens including anonymous roadside screening, other mass testing, and biomedical research uses.

The two principal variants of breath-alcohol sampling for delayed analysis are: 1) Preservation of a whole-breath specimen[3], or 2) retention of the alcohol content of a measured or fixed volume of breath of specified characteristics (7). However, none of the dozens of methods or procedures in either category, including various patented approaches, have proven entirely satisfactory. The major obstacle to satisfactory long-term retention of whole-breath specimens has been the loss of alcohol through container walls or closures or by chemical or

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[3] In this report, the term *specimen* is generally intended to apply to the entire quantity of a biological material obtained for analysis, while the term *sample* is generally applied to that portion of a specimen which is subjected to analysis.

microbial action during storage. Satisfactory use of solid sorbents, on the other hand, has been hindered by failure of the sorbents to trap or later to yield the alcohol quantitatively, or to retain it completely during storage; as well as such problems as deliquescence of the sorbent or difficulties in desorption and analysis of the stored alcohol. An additional problem militating against widespread practical application of delayed breath-alcohol analyses has been the practice of manufacturers to market breath preservation systems compatible only with proprietary quantitative evidential breath analyzers from the same source.

To meet these several needs and overcome the prior problems, the research and development activities summarized in this Final Report were planned and carried out, under contract with the National Highway Traffic Safety Administration of the U. S. Department of Transportation, by the Toxicology Laboratories of the University of Oklahoma under the direction of Kurt M. Dubowski, Ph.D., Professor of Medicine & Director of Toxicology Laboratories, as the Principal Investigator and project director.

Reduced to essentials, the objectives of this study were (1) to consider alternative approaches to breath sampling for delayed breath-alcohol analysis and to select the most promising, broadly-compatible scheme; (2) to develop the indicated features and techniques of breath-sampling, alcohol retention and storage, and alcohol recovery and analysis; (3) to couple the sampling procedure with typical quantitative evidential breath-alcohol analyzers meeting the NHTSA's Standard for Devices to Measure Breath Alcohol (8, 9); and (4) to evaluate and validate the resultant system. These objectives have been met successfully and fully.

MATERIALS AND METHODS

Apparatus, Methods, and Procedures

Reference Alcohol Analyses. Analyses for alcohol in simulator solutions and for other purposes were carried out by automated gas chromatographic head-space analysis with the Model F-45 Vapor Space Chromatograph (Perkin-Elmer Corp., Norwalk, CT 06852) as described by Dubowski (10). Results were calculated by least-squares linear regression analysis of the GC output, employing either peak height measurements of the potentiometric strip-chart recorder chromatograms or, usually, automatic integration with a Model 3380A Advanced Reporting Integrator (Hewlett-Packard Co., Palo Alto, CA 04304).

Breath-Alcohol Analyses - Gas Chromatographic Intoximeter. Breath-alcohol was measured in human subjects by direct analysis with a Mark IV Gas Chromatographic Intoximeter ("GCI") (Intoximeters, Inc., St. Louis, MO 63103). Results were obtained from the built-in digital readout, in 3-decimal figures (0.XXX g./210 L). In-vitro vapor alcohol concentrations of simulator effluents were measured comparably. All series of subject analyses were preceded, followed, and accompanied by contemporaneous control analyses of simulator effluent. The GCI was calibrated to reflect vapor ethanol concentrations in grams/210 liters[4].

Breath-Alcohol Analysis - Molecular Sieve. Breath-alcohol was measured in human subjects by indirect (or delayed) analysis involving (1) initial breath

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[4] This practice reflects and is in keeping with recent recommendations of the National Safety Council's Committee on Alcohol & Drugs, adopting the suggestion of Mason and Dubowski for direct reporting of breath-alcohol analyses (6, 11), and the August 1979 revision of §11-902.1(a)5 of the Uniform Vehicle Code adopting the definition "Alcohol concentration shall mean either grams of alcohol per 100 milliliters of blood or grams of alcohol per 210 liters of breath" (12).

collection with a modified DPC Intoximeter breath collection device, (2) immediate delivery of the fixed 250 ml. breath sample into and through a Molecular Sieve 13X column, (3) intervening storage of the molecular sieve tube, and (4) subsequent analysis for alcohol.

The breath sampling arrangement is illustrated schematically in Figure 1.

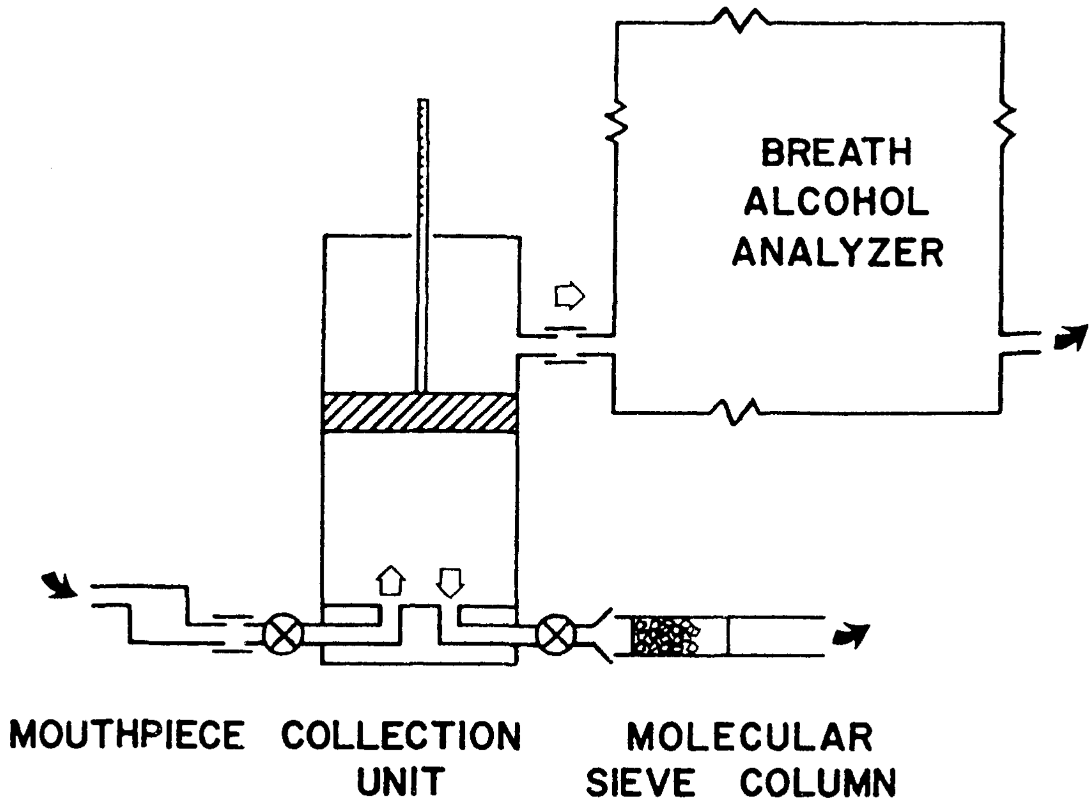

Figure 1. Scheme of Breath-Sampling for Breath-Collection Device

The in-series flow-through arrangement sequencing collection unit to breath-alcohol analyzer to discard was adopted as standard for this project after trials involving both in-parallel and in-series arrangements.

The molecular sieve 13X breath-alcohol collection tube is illustrated schematically in Figure 2. It consists of a borosilicate glass tube measuring

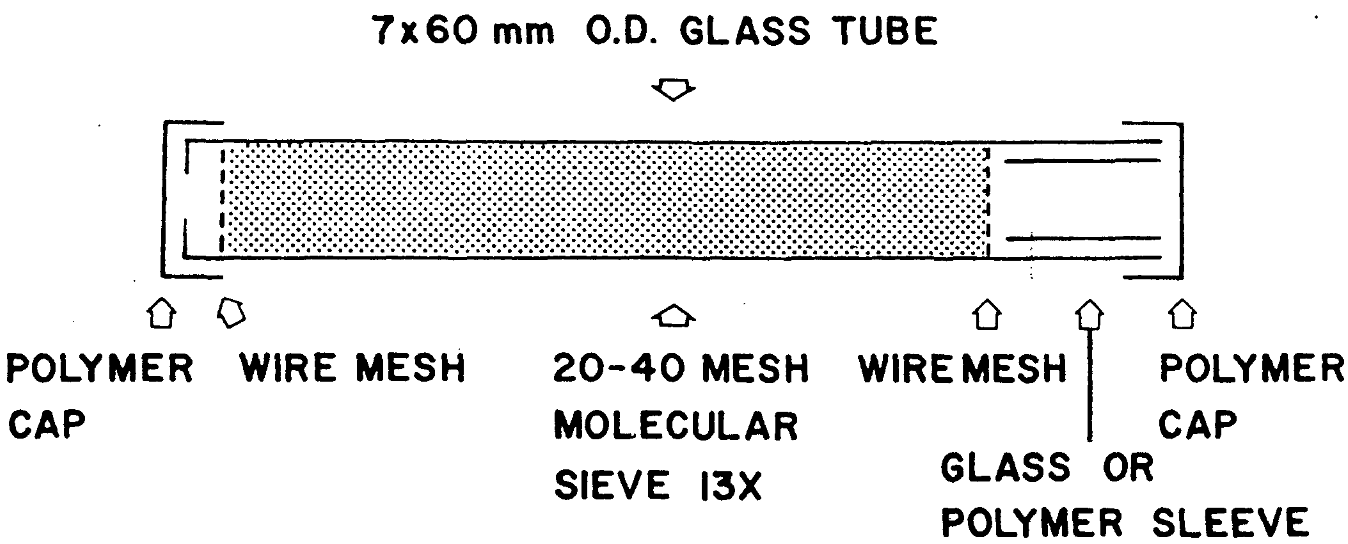

Figure 2. Scheme of Molecular Sieve Breath-Collection Device

approximately 7x60 mm OD which contains 0.4 gram of 20-40 mesh essentially anhydrous molecular sieve 13X retained between filter paper discs. The sorbent material (13) has a nominal pore diameter of 10 Angstroms and is a crystalline sodium alumino-silicate with a unit cell formula of

$$Na_{86}[(AlO_2)_{86}(SiO_2)_{106}]\cdot 276\ H_2O$$

which is activated for alcohol sorption by removal of the water of hydration (Union Carbide Corporation, New York, NY 10017).

Analysis of breath-alcohol sorbed on molecular sieve 13X was carried out by desorption of alcohol in a sealed 25 ml. glass headspace vial, containing the molecular sieve charge plus 1.0 g. of crystalline sodium chloride, with 1.00 ml. of aqueous acetonitrile solution (110 microliters/liter) as desorbent/diluent/

internal standard[5], followed by automated gas chromatographic headspace analysis using a Perkin-Elmer F-45 Vapor Space Chromatograph, after equilibration at 50°C for 45 minutes. This constitutes a modification of the procedure described by Dubowski (14) for alcohol analysis after calcium sulfate sorption.

Simultaneous calibrators (i.e., "standards"), prepared by delivering into molecular sieve tubes 250 ml. of appropriate simulator effluents prepared by equilibrium alcohol distribution between air and water at 34°C, employing the precision simulator equilibration technique developed by Dubowski and described in Part 1 of this Final Report (15) were chromatographed with each turntable load of 30 vials (or less) automated headspace GC run. At least 3 calibrators of different alcohol concentrations (generally 0, 50, 100, 200, 300 mg. alcohol/210 liters nominal values) were employed, and the final analysis results obtained by least-squares linear regression analysis of the GC responses, as described above.

<u>In-Vitro Vapor-Alcohol Analyses - Molecular Sieve</u>. Analyses of molecular sieve 13X tubes, after charging with simulator effluent, or when employed as reagent blanks, were carried out as described above for breath-alcohol analyses, but substituting for breath the in-vitro specimens prepared by precision simulator equilibration techniques at 34°C.

Break-through tests of the alcohol capacity of the molecular sieve tubes containing a standard 0.4 g charge of molecular sieve 13X were conducted with consecutive, incremental 250 ml. simulator-effluent loads delivered into sets of

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[5] This internal standard concentration of acetonitrile was chosen in order to optimize the chromatographic peak ratio of acetonitrile and ethanol for our breath collection and instrumental GC analysis conditions. Other collection or analysis conditions may require different acetonitrile concentrations.

two identical m/s tubes connected in tandem for vapor alcohol concentrations up to 1.0 g/210 liters.

Analysis of molecular sieve 13X tubes, after charging with simulator effluent at a target value of 0.20 g/210 liters, were carried out, in triplicate in-vitro, after long-term storage at room temperature for intervals from 0 to 11 months. Similar in-vitro studies were conducted after subjecting charged tubes in triplicate to accelerated environmental stresses by storing them for 24, 48, and 72 hours at 0, 25, and 40°C, respectively before analysis.

Human Subject Studies. These studies were conducted on healthy adult subjects (20 males and 12 females) who volunteered to participate in this study and were paid for their services. Experimental activities were carried out in full accordance with all applicable national standards for investigations involving human subjects (16-17), and only after initial approval and periodic review and reapproval of experimental protocols and of these human subject studies by the OUHSC Institutional Review Board.

Breath pressures against a standard resistance (Model 900 Breathalyzer) were measured with a Model 2050C "Magnehelic" direct-reading differential pressure gauge (Dwyer Instruments, Inc., Michigan City, IN 46340). Breath temperatures were measured with a Heath/Schlumberger Model EU-200-41/EU-200-62 digital thermometer (Heath Co., Benton Harbor, MI 49022) and a Model 705 thermolinear thermistor probe (Yellow Springs Instrument Co., Yellow Springs, OH 45387). Breath volumes were measured with a Model 06001 9-liter water-displacement respirometer (Warren E. Collins, Inc., Braintree, MA 02184) modified by us with a Model 20013 rotary motion potentiometer (Warren E. Collins, Inc.)

for direct electronic volume readout via a digital voltmeter. The characteristics of these respiratory measurements procedures have been reported by Dubowski (18).

Alcohol Intake Sessions. After appropriate briefing, preparation, and human experimentation consent procedures, the alcohol-free status of each subject was established by breath-alcohol measurement by infrared absorptiometry with a Model 4011A Intoxilyzer (CMI, Inc., Minturn, CO 81645). Following respiratory parameter measurements, the subjects ingested alcohol doses individually adjusted to produce target BrACs between 0.05 and 0.15 g/210 L in accordance with prior alcohol pharmacokinetics experience in our laboratories (19, 20). Subjects were kept under continuous observation before, during, and after alcohol intake, until they again were alcohol-free. In these sessions, we employed as laboratory assistants, under direct supervision, specially trained off-duty police officers who perform drinking-driving enforcement and breath-alcohol analyses as normal on-duty activities and who possess State of Oklahoma permits as breath-alcohol analysis supervisors and operators.

RESULTS AND FINDINGS

The positive information developed by the in-vitro and in-vivo studies in this phase of the project are reported under 3 categories encompassing related results and findings. In most instances, these reported results are not the "raw" data or values as observed, but calculated or computed "final" values.

I. In-Vitro (Simulator) Studies of Vapor Alcohol Analysis with Molecular Sieve.

Accuracy and Precision Studies. Results of tests of the accuracy and precision of vapor-alcohol analyses after intervening molecular sieve (m/s) sorption, on precision simulator-generated specimens are shown in Tables 1 and 2. The former results were obtained with gas chromatographic peak height quantitation, while the latter reflect GC peak area quantitation. Results of initial experiments on the accuracy and precision of such analyses, conducted with both in-series and in-parallel specimen collection arrangements, are shown in Table 3. A partial statistical summary of the in-vitro accuracy and precision data, in Tables 1 and 2 in comparison with applicable portions of the U. S. Department of Transportation "Standard for Devices to Measure Breath Alcohol" (8), is given in Table 4 for the GC peak height and in Table 5 for the GC peak area methods of result calculation.

Alcohol Capacity Tests. The results of the break-through tests of alcohol capacity of the m/s 13X tubes for incremental 250 ml. simulator-effluent loads are given in Table 6. None of the second m/s columns contained detectible alcohol; hence the alcohol capacity of a standard 0.4 g m/s 13X column was found to exceed 1.0 g alcohol/210 liters of specimens saturated with water vapor.

TABLE 1

Breath-Alcohol Simulator Studies of Accuracy & Precision

of Molecular Sieve 13X Sorption Tubes

(Tandem Simulators, Internal Standard GC, Peak Heights Quantitation)

TEST	Results of Molecular Sieve 13X Tube Analyses, g/210 L[1] For Target Values of 0.05	0.15	0.25
1	0.047	0.149	0.249
2	0.047	0.146	0.253
3	0.047	0.149	0.252
4	0.047	0.147	0.246
5	0.047	0.149	0.248
6	0.047	0.146	0.253
7	0.047	0.148	0.253
8	0.047	0.147	0.249
9	0.046	0.146	0.243
10	0.046	0.146	0.247
11	0.049	0.147	0.246
12	0.046	0.144	0.245
13	0.045	0.144	0.246
14	0.046	0.147	0.245
15	0.048	0.143	0.252
16	0.046	0.144	0.248
17	0.046	0.144	0.251
18	0.045	0.145	0.248
19	0.046	0.145	0.248
20	0.047	0.145	
21		0.145	
22		0.144	
Mean	0.047	0.146	0.248
S.D.	±0.0009	±0.0018	±0.0031
C.V.	2.01%	1.23%	1.25%
Range	0.045-0.049	0.143-0.149	0.243-0.253

Notes: [1]Target values and results are stated in grams of ethanol per 210 liters of Simulator effluent

TABLE 2

Breath-Alcohol Simulator Studies of Accuracy & Precision of Molecular Sieve 13X Sorption Tubes

(Tandem Simulators, Internal Standard GC, Area Integration Quantitation)

TEST	Results of Molecular Sieve 13X Tube Analyses, g/210 L[1] For Target Values of 0.05	0.10	0.15	0.20	0.25	0.30
1	0.048	0.101	0.148	0.189	0.247	0.311
2	0.048	0.100	0.146	0.198	0.255	0.302
3	0.049	0.102	0.148	0.197	0.256	0.316
4	0.050	0.102	0.150	0.198	0.243	0.308
5	0.050	0.099	0.154	0.192	0.249	0.297
6	0.049	0.103	0.149	0.200	0.245	0.301
7	0.050	0.102	0.150	0.197	0.248	0.315
8	0.049	0.101	0.148	0.196	0.249	0.287
9	0.048	0.102	0.149	0.198	0.251	0.316
10	0.050	0.102	0.151	0.199	0.254	0.285
11	0.047	0.102	0.146	0.197	0.252	0.302
12	0.046	0.102	0.149	0.197	0.249	0.288
13	0.049	0.102	0.150	0.198	0.249	0.306
14	0.049	0.101	0.150	0.198	0.250	0.293
15	0.051	0.101	0.148	0.197	0.252	0.297
16	0.050	0.101	0.149	0.192	0.251	0.286
17	0.050	0.098	0.151	0.193	0.255	0.274
18	0.050	0.101	0.148	0.195	0.256	0.308
19	0.050	0.098	0.148	0.198	0.253	0.303
20	0.045	0.101	0.148	0.198		0.314
21	0.051	0.100	0.145	0.199		0.308
22		0.096	0.147			
Mean	0.049	0.100	0.149	0.196	0.251	0.300
S.D.	±0.0015	±0.0017	±0.0020	±0.0028	±0.0036	±0.0118
C.V.	3.06%	1.67%	1.32%	1.41%	1.45%	3.91%
Range	0.045-0.051	0.096-0.103	0.145-0.154	0.189-0.200	0.243-0.256	0.274-0.316

Notes: [1]Target values and results are stated in grams of ethanol per 210 liters of Simulator effluent

TABLE 3

Studies of Breath-Alcohol in Human Subjects

Control Measurements[1] of Simulator-Effluent Vapor-Alcohol[2] by Gas Chromatography and Molecular Sieve 13X Sorption Tubes

Vapor-Alcohol Concentration, g/210 Liters							
Series Sampling				Parallel-Split Sampling			
No.	GCI	M/S	Difference GCI-M/S	No.	GCI	M/S	Difference GCI-M/S
01	0.101	0.095	0.006	01	0.102	0.096	0.006
02	0.099	0.096	0.003	02	0.098	0.091	0.007
03	0.099	0.094	0.005	03	0.095	0.090	0.005
04	0.103	0.094	0.009	04	0.095	0.098	-0.003
05	0.099	0.096	0.003	05	0.097	0.094	0.003
06	0.101	0.097	0.004	06	0.095	0.093	0.002
07	0.105	0.097	0.008	07	0.102	0.091	0.009
08	0.101	0.096	0.005	08	0.099	0.090	0.009
09	0.102	0.096	0.006	09	0.098	0.090	0.008
10	0.102	0.098	0.004	10	0.099	0.095	0.004
11	0.103	0.098	0.005				
12	0.105	0.099	0.006				
13	0.105	0.100	0.005				
14	0.102	0.099	0.003				
15	0.106	0.100	0.006				
16	0.101	0.096	0.005				
17	0.106	0.100	0.006				
18	0.099	0.093	0.006				
19	0.099	0.098	0.001				
20	0.102	0.102	0.000				
Mean	0.102	0.097	+0.0048		0.098	0.095	+0.0050
S.D.	±0.0024	±0.0024	±0.0021		±0.0026	±0.0067	±0.0037
C.V.	2.4%	2.4%	44.2%		2.6%	7.05%	74.2%

NOTES: [1]Between-run individual measurements

[2]Target value = 0.100 g/210 Liters

TABLE 4

Summary of Results of Breath-Alcohol Simulator Studies of Accuracy & Precision of Molecular Sieve 13X Sorption Tubes

(Compared with DOT "Standard for Devices to Measure Breath Alcohol"[1])

Item	DOT Requirements	Results of Molecular Sieve 13X Analyses, g/210 L[2,3,4] for Target Value (g/210 L) of		
		0.05	0.15	0.25
Mean Measured Value	N/A	0.047	0.146	0.248
S.D. of Mean Measured Value	N/A	±0.0009	±0.0018	±0.0031
Accuracy (As Systematic Error)	≦ 10% @ 0.05 g/210 L ≦ 5% @ 0.1 & 0.15 g/210 L	-6.0%	-2.7%	-0.8%
Precision (As Ave. Std. Deviation)	≦ 0.004 g/210 L @ 0.05, 0.10, 0.15 g/210 L	← 0.0014 g/210 L →		

NOTES: [1]Federal Register 34:30459-30463 (Nov. 5, 1973)

[2]Automated GC Headspace Analysis with internal standard; quantitation by peak height measurement

[3]Vapor Specimens = 250ml; tandem simulators at 34°C

[4]Target values and results are stated in grams of ethanol per 210 liters of simulator effluent, unless other units are shown

TABLE 5

Summary of Results of Breath-Alcohol Simulator Studies of Accuracy & Precision

of Molecular Sieve 13X Sorption Tubes

(Compared with DOT "Standard for Devices to Measure Breath Alcohol"[1])

Item	DOT Requirements	Results of Molecular Sieve 13X Analyses, g/210 L[2,3,4] for Target Value (g/210 L) of					
		0.05	0.10	0.15	0.20	0.25	0.30
Mean Measured Value	N/A	0.049	0.096	0.149	0.197	0.251	0.301
S.D. of Mean Measured Value	N/A	±0.0015	±0.0017	±0.0020	±0.0028	±0.0036	±0.0118
Accuracy (As Systematic Error)	≦ 10% @ 0.05 g/210 L ≦ 5% @ 0.1 & 0.15g/210 L	-2.0%	-4.0%	-0.7%	-1.5%	+0.4%	+0.3%
Precision (As Ave. Std. Deviation)	≦ 0.004 g/210 L @ 0.05, 0.10, 0.15 g/210 L	← 0.0017 g/210 L →					

NOTES: [1]Federal Register 34:30459-30463 (Nov. 5, 1973)

[2]Automated GC Headspace Analysis with internal stardard; quantitation by peak area integration

[3]Vapor Specimens = 250 ml; tandem simulators at 34°C

[4]Target values and results are stated in grams of ethanol per 210 liters of simulator effluent, unless other units are shown

TABLE 6

STUDIES IN VAPOR-ALCOHOL ANALYSIS

Capacity of Molecular Sieve 13X Traps for Alcohol

Test	Vapor-Alcohol Concentration,[1,2] g/210 L		
	Target	M/S Trap 1	M/S Trap 2[3]
1	0.05	0.047	0
2	0.10	0.099	0
3	0.15	0.152	0
4	0.20	0.199	0
5	0.40	0.382	0
6	0.60	0.603	0
7	0.80	0.787	0
8	1.00	0.999	0

NOTES: [1] In-vitro vapor specimens: 250 ml. effluent from tandem simulators at 34°C

[2] Results obtained by automated GC headspace analysis with internal standard against EtOH standards trapped on M/S 13X; quantitation by peak area integration

[3] Traps 1 and 2 were connected in tandem

Storage and Stress Tests. Results of long-term in-vitro storage tests of m/s 13X tubes are given in Table 7. All specimens were stored, in the dark, at normal room temperature with various tube sealing techniques in 3 different series of tests. The respective tube closure methods reflected in the table are: (1) unsealed glass m/s columns retained in 9 ml. capacity 17x60 mm OD glass vials with vinylite screw-caps, (2) glass m/s columns with ends occluded by single thickness Saran wrap and 1/4-inch polymer GC column end caps, and (3) glass columns with ends occluded by Teflon-faced polymer septum material. The results of short-term environmental stress tests are given in Table 8 for m/s tubes stored in screw-cap vials.

Accuracy and Precision of Gas Chromatographic Intoximeter. Since the results of delayed breath-alcohol analyses after molecular sieve sorption were to be compared with those obtained on the same breath specimens by direct analysis with a Mark IV Gas Chromatographic Intoximeter (GCI), we performed a brief study of the accuracy and precision of vapor-alcohol measurements with the GCI, using precision simulator techniques to produce the in-vitro alcohol specimens for one series and employing a reference (anhydrous) ethanol-in-nitrogen gas mixture manufactured to our specifications for another series of tests. Results of the replicate analyses of simulator effluents are given in Table 9, while those for the replicate analyses of the gas mixture are given in Table 10.

II. Human Subject Demographic and Respiratory Parameter Data

Demographic Data. The age and sex statistics for the volunteer human subjects who participated in the alcohol-consumption phase of this project are given in Table 11.

TABLE 7

Studies of Vapor Alcohol Analysis

Stability of Alcohol Trapped[1,2] on Molecular Sieve 13X After Long-Term Storage

Elapsed Time Months	Vapor-Alcohol Concentration of Duplicates[3], g/210 L		
	Series 1[4]	Series 2[4]	Series 3[4]
1	0.199, 0	0.178, 0.184	0.197, 0.203
2	0.185, 0	0.208, 0.184	0.199, 0.197
3	0.167, 0	0.151, 0.127	0.209, 0.201
4	0.184, 0	0.210, 0.164	
5	0, 0	0.188, 0.118	
6	0, 0	0.214, 0.215	
7	0.099, 0	0.186, 0.215	
8	0.160, 0	0.197, 0.099	
9	0.166, 0	0.157, 0.096	
10	0.166, 0	0.215, 0.181	
11	0, 0	0.194, 0.037	

NOTES: [1]Target Value = 0.20 g/210 L

[2]In-vitro vapor specimens: 250 ml of effluent from tandem simulators at 34°C

[3]Results obtained by automated GC headspace analysis with internal standard; quantitation by peak area integration

[4]M/S columns stored, respectively, in (1) open tubes in screw-cap vials, (2) tubes with Saran and polymer-cap closures, and (3) tubes with polymer septum closures

TABLE 8

Studies in Vapor-Alcohol Analysis

Stability of Alcohol Trapped[1,2] on Molecular Sieve 13X Columns after Storage under Various Environmental Conditions

Test	Storage Condition	Vapor-Alcohol Concentration[3], g/210 L, after			
		0 Hours	24 Hours	48 Hours	72 Hours
1	0°C		0.196	0.204	0.200
2			0.204	0.201	0.204
3			0.195	0.203	0.195
Mean			0.198	0.203	0.200
4	(Blank)		0.000	0.000	
5	25°C	0.200	0.194	0.190	0.197
6		0.201	0.202	0.199	0.187
7		0.200	0.200	0.196	0.202
Mean		0.200	0.199	0.195	0.195
8	(Blank)	0.000	0.000	0.000	
9	40°C		0.197	0.194	0.201
10			0.200	0.202	0.202
11			0.184	0.197	0.202
Mean			0.194	0.198	0.202
12	(Blank)		0.000	0.000	

NOTES: [1]Target value = 0.20 g/210 L

[2]In vitro vapor specimens: 250 ml. effluent from tandem simulators at 34°C

[3]Results obtained by automated GC headspace analysis with internal standard against EtOH standards trapped on M/S 13X; quantitation by peak area integration

TABLE 9

Studies of Vapor-Alcohol Analysis

In-Vitro (Simulator) Studies of Accuracy & Precision

of Gas Chromatographic Analyses[1] for Target Value

of 0.100 g/210 Liters[2,3]

Test	GCI Result	Test	GCI Result
1	0.101	19	0.100
2	0.101	20	0.101
3	0.101	21	0.101
4	0.101	22	0.100
5	0.097	23	0.100
6	0.098	24	0.101
7	0.097	25	0.101
8	0.097	26	0.100
9	0.099	27	0.101
10	0.098	28	0.102
11	0.099	29	0.100
12	0.099	30	0.100
13	0.099	31	0.100
14	0.100	32	0.101
15	0.100	33	0.101
16	0.100	34	0.100
17	0.099	35	0.100
18	0.101	36	0.101
Mean			0.0999
S.D.			±0.0013
C.V.			1.27%
Range			0.097-0.102

Notes: [1]Alcohol analyses were performed with a Mark IV Gas Chromatographic Intoximeter

[2]Tandem simulators at 34°C

[3]Target values and results are stated in grams of ethanol per 210 Liters of Simulator effluent

TABLE 10

Studies of Vapor-Alcohol Analysis

In-Vitro Studies of Accuracy and Precision

of Gas Chromatographic Analyses[1] for Target Value

of 0.115 g/210 Liters[2,3]

Test	GCI Result[3]	Test	GCI Result[3]
1	0.112	13	0.114
2	0.112	14	0.115
3	0.112	15	0.115
4	0.113	16	0.115
5	0.114	17	0.115
6	0.113	18	0.115
7	0.114	19	0.115
8	0.114	20	0.115
9	0.113	21	0.115
10	0.114	22	0.115
11	0.116	23	0.114
12	0.115	24	0.115
		25	0.115

Mean	0.1142
S.D.	±0.00112
C.V.	0.98%
Min./Max.	0.112 to 0.116

Notes: [1]Alcohol analyses were performed with a MK IV Gas Chromatographic Intoximeter

[2]Reference ethanol-in-nitrogen gas mixture

[3]Target values and results are stated in nominal units of grams of ethanol per 210 Liters of gas

TABLE 11

In-Vivo Studies of Breath-Alcohol in Human Subjects

Demographic Data on Human Experimental Subjects

Age and Sex of Subjects

Males	Age, Yrs.	Females	Age, Yrs.
01-	27	01-	32
02-	36	02-	32
03-	24	03-	26
04-	35	04-	23
05-	31	05-	26
06-	30	06-	24
07-	23	07-	36
08-	22	08-	27
09-	22	09-	32
10-	39	10-	26
11-	41	11-	24
12-	31	12-	26
13-	21		
14-	24		
15-	27		
16-	24		
17-	24		
18-	24		
19-	33		
20-	39		

	RANGE	MEAN
MALES (N=20)	21 - 41	29
FEMALES (N=12)	23 - 36	28
TOTAL (N=32)	21 - 41	28

Respiratory Parameter Data. The individual experimental data on breath exit temperature, breath volume (both maximum expiratory, i.e., vital capacity, and normal exhalation after a normal inspiration), and breath pressures at a Breathalyzer Model 900 inlet tube distal to the mouthpiece during normal breath sampling are given in Table 12 for those subjects who participated in the alcohol consumption phase of this project. The same table contains a statistical summary of these data for the 32 subjects. A respiratory data summary for the same experimental findings is given in Table 13.

III. In-Vivo Comparison Studies of Breath-Alcohol Measurement, on Divided Breath Specimens, by Direct Analysis with the GCI and Delayed Analysis after Molecular Sieve Sorption.

The following results were all obtained by in-vivo studies on human subjects, in the alcohol-free state (for "blank" values) and after alcohol consumption during the absorptive and postabsorptive phases unless otherwise specified. For each paired set of Gas Chromatographic Intoximeter (GCI) and molecular sieve 13X (m/s) results, the breath specimen was directed through a standard plastic subject mouthpiece in a continuous, uninterrupted single exhalation into and through the DPC collection device and into the GCI breath inlet as shown in Figure 1. In two subset experiments, parallel division of the breath specimens was used; i.e., the breath specimen was divided at the distal (outlet) side of a standard plastic subject mouthpiece and simultaneously directed to the breath inlet of the GCI and the breath inlet of the DPC collection device.

Breath-Alcohol Analyses in Alcohol-Free Subjects. The results of BrAC

TABLE 12

DOT HUMAN SUBJECT SESSIONS MAY/JUNE 1979

Respiratory Parameter Data

Subject	Sex & Age		Breath Exit Temp. °C	Breath Expiratory Volume, ml. Vital Capacity	Breath Expiratory Volume, ml. Normal Exhalation	Breath Pressure (Breathalyzer) inches H_2O
1	M	27	34.50	5048	3852	16
2	M	36	34.57	4622	2205	21
3	M	24	35.18	5217	2720	15
4	M	35	34.82	4040	2223	22
5	M	31	35.09	5036	2167	12
6	M	30	34.17	4731	1758	38
7	F	32	34.95	4130	2692	7
8	M	23	35.07	5316	3131	23
9	F	32	34.54	3311	2815	9
10	M	22	34.88	3953	2813	17
11	F	26	33.81	3961	3575	17
12	F	23	35.07	3088	2016	9
13	M	22	34.50	4696	2475	23
14	M	39	33.98	3974	2554	17
15	F	26	35.11	2656	1579	14
16	F	24	34.55	3763	1243	10
17	M	41	34.01	4196	2566	38
18	F	36	34.23	3240	1612	21
19	F	27	33.75	2751	1425	15
20	M	31	34.32	4717	3311	23
21	M	21	34.50	5210	3230	25
22	M	24	34.80	5848	2729	18
23	F	32	35.77	3429	2348	8
24	F	26	34.92	2420	1786	10
25	M	27	34.66	4923	2432	14
26	M	24	35.06	5000	2836	21
27	M	24	34.67	3982	1830	12
28	M	24	34.28	4406	3340	14
29	F	24	33.96	4935	3210	14
30	M	33	34.85	3866	2568	17
31	F	26	34.82	3778	3326	10
32	M	39	33.72	3841	2240	15
Mean		28.5	34.60	4190	2519	17.0
S.D.		±5.6	±0.48	±846.3	±657.4	±7.4
C.V.		19.7%	1.4%	20.2%	26.1%	43.5%
Range		21-41	33.72-35.77	2420-5848	1243-3852	7-38

TABLE 13

DOT HUMAN SUBJECT SESSIONS MAY/JUNE 1979

Respiratory Data Summary

End-Expiratory Breath Temperatures in Human Subjects, Measured at the Mouth

Subjects	N	End-Expiratory Temperature, °C Range	Mean	SD	CV
Men	20	33.72-35.18	34.89	+0.40	1.15%
Women	12	33.75-35.77	34.62	±0.60	1.73%
Total	32	32.72-35.77	34.60	±0.48	1.39%

Expiratory Breath Volumes in Human Subjects - Forced Vital Capacity

Subjects	N	Forced Vital Capacity, ml. Range	Mean	SD	CV
Men	20	3841-5848	4631	±577	12.5%
Women	12	2420-4935	3455	±708	20.5%
Total	32	2420-5848	4190	±846	20.2%

Maximum Expiratory Breath Volumes in Human Subjects after Normal Inhalation

Subjects	N	Maximum Exhalation Volume, ml. Range	Mean	SD	CV
Men	20	1758-3852	2649	±531	20.0%
Women	12	1243-3575	2302	±806	35.0%
Total	32	1243-3852	2519	±657	26.1%

Breath Pressures in Human Subjects Into a Model 900 Breathalyzer

Subjects	N	Breath Pressure, inches H_2O Range	Mean	SD	CV
Men	20	12-38	20.1	±7.3	36.3%
Women	12	7-21	12.0	±4.2	35.0%
Total	32	7-38	17.0	±7.4	43.5%

measurements on alcohol-free subjects by means of the GCI and simultaneous sorption on m/s columns, in divided breath specimens, are shown in Table 14 for both 28 alcohol-consuming subjects prior to alcohol intake and 28 additional non-consumers as control subjects. No BrAC value exeeded 0.001 g/210 L by either method of analysis, and no analysis by means of m/s sorption and subsequent GC analysis of the eluate yielded a positive result at any concentration.

Breath-Alcohol Measurements in Subjects by Parallel-Split Sampling. The results of these two subseries of tests are segregated for ease in recognizing the differences in paired breath-alcohol measurements in breath specimens divided by in-parallel and in-series schemes. Table 15 contains paired analysis results from 22 different subjects for both parallel and series sampling shortly after oral alcohol intake, reflecting presence of "mouth-alcohol." These "mouth-alcohol" results are represented graphically in Figure 3 for the series portion of the data in Table 15.

The parallel specimen correlation data in Table 15 were analyzed by least-squares linear regression analysis, and yielded

$$y = 0.7813x + 0.03709 \qquad \text{(I)}$$

$$R^2 = 0.830; \; R = 0.911$$

$$N = 4 \text{ result pairs}$$

where y = BrAC by molecular sieve delayed analysis, g/210 L

x = BrAC by GCI direct analysis, g/210 L

R^2 = Coefficient of Determination

R = (Pearson) Correlation Coefficient

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurements by Gas Chromatographic Intoximeter and Molecular Sieve Sorption Tubes in Alcohol-Free Subjects on Single Breath Specimens

Breath-Alcohol Concentration, g/210 Liters					
Experimental Subjects			Control Subjects		
No.	GCI	M/S	No.	GCI	M/S
01-	0.001	0.000	01-	0.001	0.000
02-	0.001	0.000	02-	0.001	0.000
03-	0.001	0.000	03-	0.001	0.000
04-	0.001	0.000	04-	0.001	0.000
05-	0.001	0.000	05-	0.001	0.000
06-	0.001	0.000	06-	0.001	0.000
07-	0.001	0.000	07-	0.001	0.000
08-	0.000	0.000	08-	0.001	0.000
09-	0.000	0.000	09-	0.000	0.000
10-	0.000	0.000	10-	0.000	0.000
11-	0.000	0.000	11-	0.000	0.000
12-	0.000	0.000	12-	0.000	0.000
13-	0.000	0.000	13-	0.001	0.000
14-	0.000	0.000	14-	0.001	0.000
15-	0.000	0.000	15-	0.000	0.000
16-	0.000	0.000	16-	0.000	0.000
17-	0.000	0.000	17-	0.000	0.000
18-	0.000	0.000	18-	0.000	0.000
19-	0.000	0.000	19-	0.000	0.000
20-	0.000	0.000	20-	0.000	0.000
21-	0.000	0.000	21-	0.000	0.000
22-	0.000	0.000	22-	0.000	0.000
23-	0.000	0.000	23-	0.000	0.000
24-	0.001	0.000	24-	0.000	0.000
25-	0.000	0.000	25-	0.000	0.000
26-	0.000	0.000	26-	0.001	0.000
27-	0.000	0.000	27-	0.000	0.000
28-	0.000	0.000	28-	0.000	0.000
Mean	0.0003	0.000		0.0004	0.000
S.D.	±0.0005	±0.0000		±0.0005	±0.0000
C.V.	160%	0%		126%	0%

TABLE 15

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurements of BrAC by Gas Chromatography and Molecular

Sieve 13X Sorption Tubes

Shortly After Oral Intake (Residual Mouth Alcohol),

on Single Breath Specimens[1]

Breath-Alcohol Concentration, g/210 Liters			
No.	GCI	M/S	Difference GCI-M/S
	Parallel Sampling		
01	0.281	0.263	0.018
02	0.174	0.158	0.016
03	0.378	0.311	0.067
04	0.092	0.082	0.010
	Series Sampling		
01	0.133	0.133	0.000
02	0.229	0.216	0.013
03	0.136	0.134	0.002
04	0.207	0.210	-0.003
05	0.140	0.134	0.006
06	0.136	0.130	0.006
07	0.136	0.129	0.007
08	0.096	0.090	0.006
09	0.082	0.082	0.000
10	0.207	0.194	0.013
11	0.078	0.072	0.006
12	0.090	0.087	0.003
13	0.146	0.162	-0.016
14	0.288	0.277	0.011
15	0.191	0.192	-0.001
16	0.132	0.121	0.011
17	0.080	0.088	-0.008
18	0.099	0.101	-0.002
Mean Difference - Parallel			+0.0278
Mean Difference - Series			+0.0024

NOTES: [1]Each pair of results is from a different subject

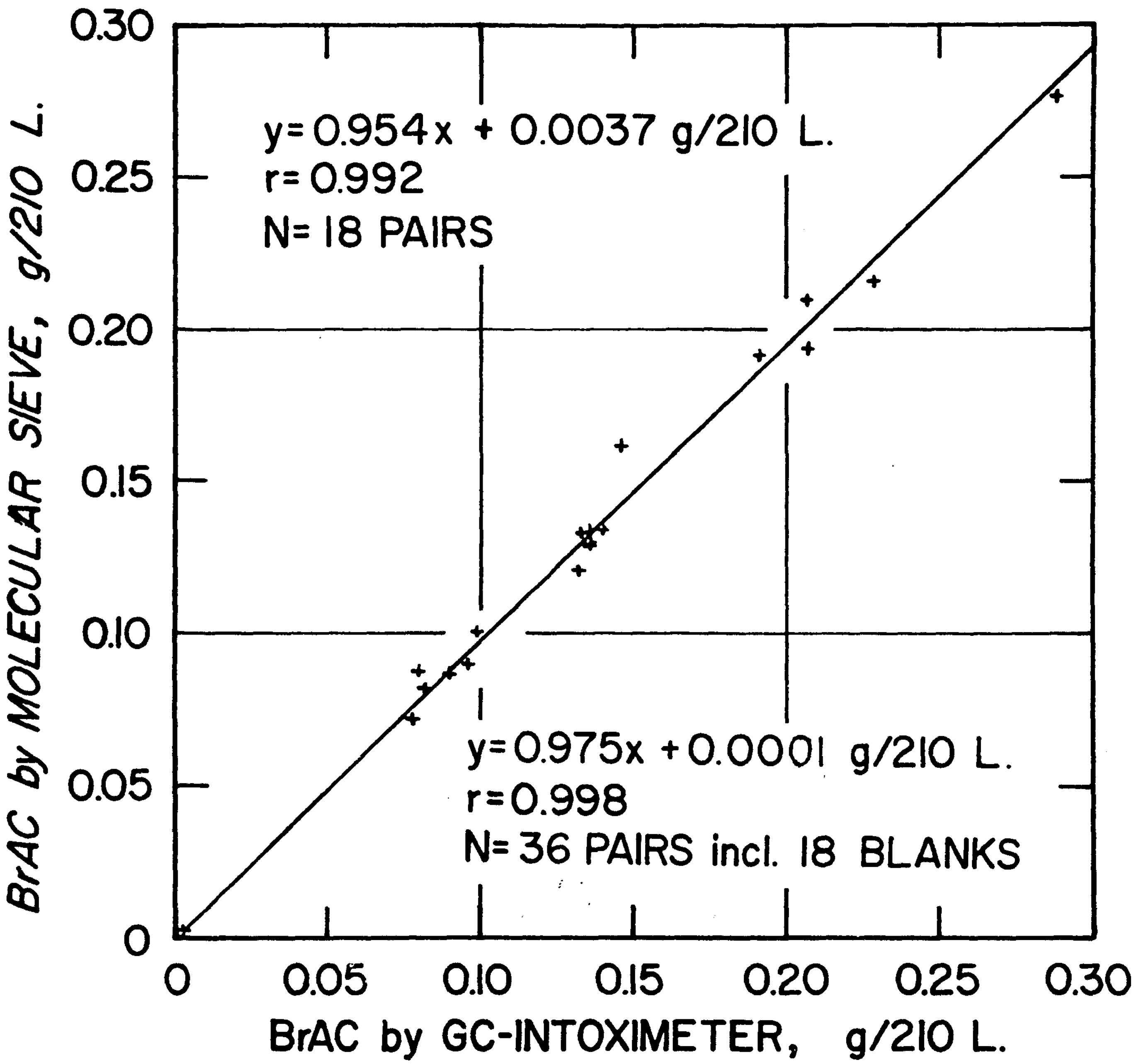

Figure 3. Correlation of Breath-Alcohol Concentrations in Series-Sampled Divided Specimens by GC-Intoximeter and Molecular Sieve, Shortly After Alcohol Ingestion

When the 4 "blank" result pairs for this subject group were included in the data and the same computation was repeated, the regression analysis yielded

$$y = 0.8990x + 0.00600 \qquad \text{(II)}$$
$$R^2 = 0.945;\ R = 0.972$$
$$N = 8 \text{ result pairs including 4 blank pairs}$$

where y, x, R^2, and R have the significance defined immediately above.

For the series sampling correlation data of Table 15, the same regression analysis yielded

$$y = 0.9539x + 0.00367 \qquad \text{(III)}$$
$$R^2 = 0.984;\ R = 0.992$$
$$N = 18 \text{ result pairs}$$

where y, x, R^2, and R have the significance defined on p. 33.

When the 18 "blank" result pairs for this subject group were included in the data and the same computation was repeated, the regression analysis yielded

$$y = 0.9753x + 0.00010 \qquad \text{(IV)}$$
$$R^2 = 0.996;\ R = 0.998$$
$$N = 36 \text{ result pairs, including 18 blank pairs}$$

where y, x, R^2, and R have the significance defined on p. 33.

The result of paired analyses for parallel breath sampling in 7 subjects after elimination of any possible contamination by retained mouth-alcohol are given in Tables 16 and 17; the former contains the actual paired direct (GCI) and delayed (m/s) breath-alcohol analysis results, while the latter reflects the differences of these 124 result pairs, together with a statistical summary thereof. For the data in Table 17, a nonparametric positive/negative binomial Sign Test for paired data yielded

		N	%
Data Pairs	=	124	100.0
-Δ Frequency	=	102	82.3
+Δ Frequency	=	17	13.7
0Δ Frequency	=	5	4.0

For the same set of 124 parallel sampling BrAC data pairs, the differences are plotted in frequency histogram form in Figure 4 with 8 low and 1 high values omitted from the graph, but included in the statistical calculations.

The parallel-sampling paired BrAC correlation data in Table 16 were analyzed by least-squares linear regression analysis, and yielded

$$y = 0.8225x + 0.00387 \qquad \text{(V)}$$

$$R^2 = 0.620; \; R = 0.787$$

$$SE = 0.01494$$

$$N = 124 \text{ result pairs}$$

where y, x, R^2, and R have the significance defined on p. 33, and SE is the standard error of the estimate.

When the 7 "blank" result pairs for this subject group were included in the data sets and the same computation was repeated, the regression analysis yielded

TABLE 16

In-Vivo Studies of Breath-Alcohol in Human Subjects

Correlation of BrAC Measurements by Gas Chromatography and Molecular Sieve 13X Sorption Tubes on Divided Breath Specimens[1,2]

(7 Subjects; Parallel Split Sampling Technique)

Breath-Alcohol Concentration, g/210 Liters								
No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S
01	0.104	0.108	42	0.103	0.105	83	0.105	0.085
02	0.107	0.079	43	0.109	0.101	84	0.106	0.089
03	0.115	0.109	44	0.111	0.110	85	0.109	0.116
04	0.120	0.137	45	0.102	0.108	86	0.111	0.115
05	0.109	0.103	46	0.108	0.118	87	0.100	0.072
06	0.090	0.086	47	0.091	0.018	88	0.094	0.102
07	0.094	0.103	48	0.100	0.039	89	0.095	0.091
08	0.100	0.093	49	0.078	0.083	90	0.078	0.084
09	0.066	0.062	50	0.063	0.057	91	0.056	0.055
10	0.057	0.054	51	0.092	0.089	92	0.089	0.082
11	0.093	0.093	52	0.104	0.112	93	0.105	0.075
12	0.104	0.034	53	0.103	0.112	94	0.104	0.022
13	0.107	0.070	54	0.087	0.065	95	0.085	0.057
14	0.087	0.039	55	0.083	0.074	96	0.083	0.073
15	0.080	0.076	56	0.073	0.070	97	0.081	0.074
16	0.079	0.025	57	0.070	0.073	98	0.065	0.067
17	0.060	0.060	58	0.057	0.058	99	0.055	0.055
18	0.051	0.047	59	0.076	0.060	100	0.083	0.072
19	0.088	0.075	60	0.093	0.091	101	0.093	0.085
20	0.075	0.063	61	0.072	0.062	102	0.068	0.063
21	0.070	0.059	62	0.065	0.054	103	0.066	0.056
22	0.063	0.054	63	0.059	0.050	104	0.058	0.049
23	0.060	0.052	64	0.058	0.056	105	0.052	0.044
24	0.052	0.045	65	0.052	0.045	106	0.064	0.054
25	0.072	0.063	66	0.087	0.071	107	0.079	0.066
26	0.081	0.069	67	0.058	0.051	108	0.052	0.044
27	0.049	0.045	68	0.048	0.044	109	0.048	0.048
28	0.045	0.041	69	0.040	0.036	110	0.040	0.037
29	0.033	0.032	70	0.032	0.033	111	0.111	0.097
30	0.103	0.103	71	0.105	0.088	112	0.107	0.098
31	0.103	0.094	72	0.099	0.085	113	0.097	0.082
32	0.094	0.078	73	0.086	0.074	114	0.082	0.074
33	0.080	0.072	74	0.134	0.108	115	0.135	0.118
34	0.130	0.107	75	0.131	0.110	116	0.119	0.108
35	0.121	0.098	76	0.115	0.094	117	0.096	0.084
36	0.095	0.078	77	0.087	0.073	118	0.083	0.071
37	0.082	0.073	78	0.077	0.060	119	0.077	0.066
38	0.071	0.067	79	0.085	0.071	120	0.083	0.070
39	0.078	0.071	80	0.077	0.066	121	0.066	0.058
40	0.067	0.056	81	0.063	0.053	122	0.056	0.052
41	0.054	0.048	82	0.051	0.046	123	0.047	0.045
						124	0.110	0.075

NOTES: [1]Automated GC headspace analysis with internal standard; quantitation by automatic electronic peak area integration

[2]Molecular Sieve 13X-sorbed alcohol as simultaneous calibrators

TABLE 17

In-Vivo Studies of Breath-Alcohol in Human Subjects

BrAC Measurements by Gas Chromatography and Molecular Sieve 13X Sorption Tubes

Differences Between Results: GCI-Molecular Sieve 13X Tube Result

(7 Subjects; Parallel Split Sampling Technique)

Breath-Alcohol Concentration, g/210 Liters

No.	Difference	No.	Difference	No.	Difference
01	+0.004	42	+0.002	83	-0.020
02	-0.028	43	-0.008	84	-0.017
03	-0.006	44	-0.001	85	+0.007
04	+0.017	45	+0.006	86	+0.004
05	-0.006	46	+0.010	87	-0.028
06	-0.004	47	-0.073	88	+0.008
07	+0.009	48	-0.061	89	-0.004
08	-0.007	49	+0.005	90	+0.006
09	-0.004	50	-0.006	91	-0.001
10	-0.003	51	-0.003	92	-0.007
11	0.000	52	+0.008	93	-0.030
12	-0.070	53	+0.009	94	-0.082
13	-0.037	54	-0.022	95	-0.028
14	-0.048	55	-0.009	96	-0.010
15	-0.004	56	-0.003	97	-0.007
16	-0.054	57	+0.003	98	+0.002
17	0.000	58	+0.001	99	0.000
18	-0.004	59	-0.016	100	-0.011
19	-0.013	60	-0.002	101	-0.008
20	-0.012	61	-0.010	102	-0.005
21	-0.011	62	-0.011	103	-0.010
22	-0.009	63	-0.009	104	-0.009
23	-0.008	64	-0.002	105	-0.008
24	-0.007	65	-0.007	106	-0.010
25	-0.009	66	-0.016	107	-0.013
26	-0.012	67	-0.007	108	-0.008
27	-0.004	68	-0.004	109	0.000
28	-0.004	69	-0.004	110	-0.003
29	-0.001	70	+0.001	111	-0.014
30	0.000	71	-0.017	112	-0.009
31	-0.009	72	-0.014	113	-0.015
32	-0.016	73	-0.012	114	-0.008
33	-0.008	74	-0.026	115	-0.017
34	-0.023	75	-0.021	116	-0.011
35	-0.023	76	-0.021	117	-0.012
36	-0.017	77	-0.014	118	-0.012
37	-0.009	78	-0.017	119	-0.011
38	-0.004	79	-0.014	120	-0.013
39	-0.007	80	-0.011	121	-0.008
40	-0.011	81	-0.010	122	-0.004
41	-0.006	82	-0.005	123	-0.002
				124	-0.035

N = 124
Mean Difference in Results = -0.01109
S.D. of Mean = ±0.015240
Range of Differences = -0.082 to +0.010

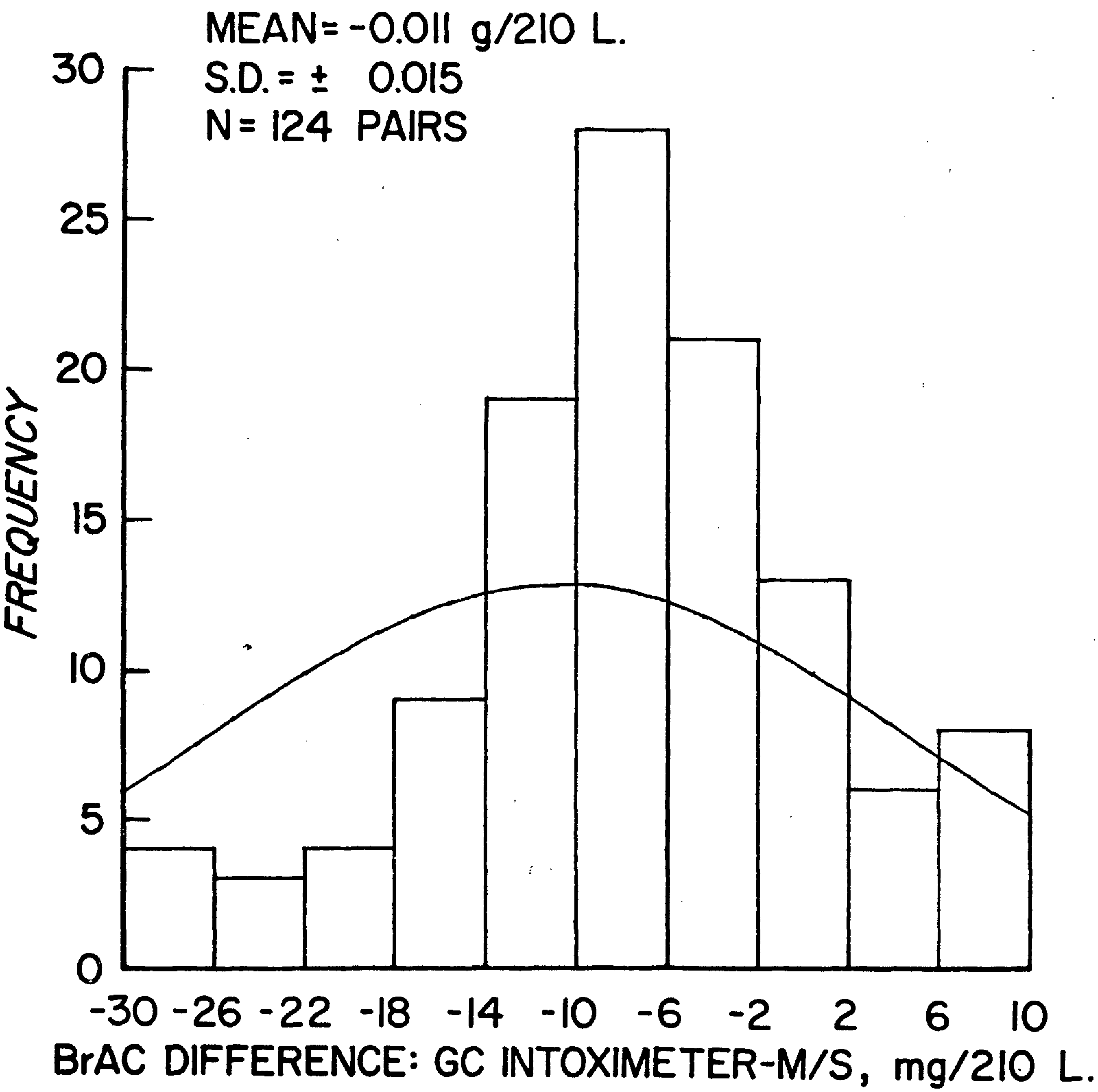

Figure 4. Differences Between Breath-Alcohol Concentrations in Parallel-Sampled Divided Specimens by GC-Intoximeter and Molecular Sieve (8 Low and 1 High Values are Omitted from the Graph)

$$y = 0.8416x + 0.00217 \quad \text{(VI)}$$

$$R^2 = 0.742;\ R = 0.862$$

$$SE = 0.01455$$

$$N = 131 \text{ result pairs, including 7 blanks}$$

where y, x, R^2, R, and SE have the significance defined immediately above.

Breath-Alcohol Measurements in Subjects by In-Series Sampling. A brief experimental study of direct BrAC measurement by GCI and delayed BrAC measure- jects is summarized in Table 18. For the 18 pairs of GCI and m/s BrAC results, the GCI-m/s differences were (mean±SD) -0.0020±0.0031 g/210 L. For the six triplicate direct BrAC measurements on consecutive breaths by means of the GCI, the mean difference between each BrAC result and the mean for that set of three consecutive breath BrAC results was -0.000006 g/210 L. For the six triplicate delayed BrAC measurements on consecutive breaths by means of m/s, the mean difference between each BrAC result and the mean for that set of three consecutive breath BrAC results was -0.0004 g/210 L. The time interval between the three successive breath samplings for each subject was the minimum required for the successive GCI tests, i.e., approximately two minutes.

The principal and key data compilations for the in-vivo correlation studies of breath-alcohol analysis by paired GCI and m/s tests is given in Table 19. That table contains 369 paired test results on individual divided breath specimens from 25 subjects, after alcohol consumption, collected by the in-series breath collection technique. GC analysis of the m/s eluates for this series was by automatic electronic peak area integration. Least-squares linear regression analysis for these 369 data pairs yielded

TABLE 18

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement of BrAC by Gas Chromatography and Molecular Sieve 13X Sorption Tubes on Three Separate Successive Breath Specimens (6 Subjects: Series Sampling Technique)

Subject	Breath No.	BrAC, g/210 L. GCI	BrAC, g/210 L. M/S
	01-	0.071	0.074
A	02-	0.077	0.080
	03-	0.075	0.070
	04-	0.084	-
B	05-	0.085	0.090
	06-	0.081	0.084
	07-	0.048	0.052
C	08-	0.049	0.058
	09-	0.049	0.050
	10-	0.045	0.048
D	11-	0.046	0.046
	12-	0.047	0.049
	13-	0.056	0.057
E	14-	0.057	0.058
	15-	0.057	0.058
	16-	0.047	0.048
F	17-	0.047	0.052
	18-	0.045	0.048

TABLE 19

In-Vivo Studies of Breath-Alcohol in Human Subjects

Correlation of BrAC Measurements by Gas Chromatography and Molecular Sieve 13X Sorption Tubes on Single Breath Specimens[1,2]

(25 Subjects; Series-Sampling Technique)

Breath-Alcohol Concentration, g/210 Liters											
No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S
01	0.101	0.100	45	0.112	0.111	89	0.111	0.110	133	0.113	0.11
02	0.104	0.101	46	0.095	0.096	90	0.090	0.090	134	0.071	0.07
03	0.078	0.072	47	0.071	0.072	91	0.070	0.067	135	0.067	0.06
04	0.066	0.061	48	0.061	0.064	92	0.061	0.058	136	0.135	0.13
05	0.123	0.125	49	0.119	0.120	93	0.116	0.115	137	0.115	0.11
06	0.113	0.110	50	0.095	0.095	94	0.115	0.110	138	0.104	0.10
07	0.106	0.104	51	0.104	0.105	95	0.105	0.107	139	0.099	0.10
08	0.106	0.107	52	0.091	0.093	96	0.089	0.091	140	0.082	0.07
09	0.071	0.074	53	0.077	0.080	97	0.075	0.070	141	0.111	0.10
10	0.109	0.106	54	0.111	0.110	98	0.108	0.100	142	0.104	0.10
11	0.102	0.101	55	0.096	0.096	99	0.098	0.087	143	0.096	0.09
12	0.098	0.094	56	0.094	0.093	100	0.096	0.093	144	0.085	0.07
13	0.081	0.077	57	0.074	0.072	101	0.065	0.058	145	0.100	0.10
14	0.090	0.089	58	0.095	0.095	102	0.089	0.089	146	0.088	0.086
15	0.081	0.081	59	0.076	0.077	103	0.059	0.063	147	0.057	0.054
16	0.054	0.055	60	0.054	0.056	104	0.048	0.051	148	0.047	0.04
17	0.046	0.049	61	0.043	0.047	105	0.041	0.045	149	0.040	0.04
18	0.144	0.143	62	0.144	0.141	106	0.131	0.133	150	0.132	0.132
19	0.131	0.132	63	0.110	0.107	107	0.107	0.101	151	0.110	0.099
20	0.107	0.112	64	0.102	0.102	108	0.102	0.099	152	0.101	0.099
21	0.095	0.097	65	0.095	0.091	109	0.128	0.130	153	0.135	0.131
22	0.132	0.133	66	0.133	0.135	110	0.131	0.132	154	0.129	0.125
23	0.120	0.123	67	0.124	0.124	111	0.104	0.098	155	0.101	0.096
24	0.095	0.095	68	0.093	0.092	112	0.090	0.093	156	0.084	0.079
25	0.078	0.078	69	0.082	0.084	113	0.093	0.091	157	0.087	0.083
26	0.081	0.078	70	0.079	0.080	114	0.076	0.079	158	0.077	0.078
27	0.073	0.074	71	0.069	0.074	115	0.065	0.069	159	0.085	0.086
28	0.096	0.094	72	0.103	0.101	116	0.113	0.109	160	0.119	0.112
29	0.116	0.113	73	0.126	0.118	117	0.129	0.124	161	0.107	0.109
30	0.085	0.085	74	0.083	0.083	118	0.080	0.082	162	0.095	0.091
31	0.107	0.106	75	0.098	0.095	119	0.102	0.103	163	0.087	0.088
32	0.091	0.091	76	0.073	0.077	120	0.075	0.075	164	0.070	0.071
33	0.067	0.068	77	0.063	0.067	121	0.064	0.068	165	0.060	0.064
34	0.062	0.063	78	0.060	0.062	122	0.063	0.065	166	0.052	0.056
35	0.047	0.051	79	0.060	0.060	123	0.078	0.071	167	0.084	0.084
36	0.105	0.102	80	0.115	0.111	124	0.130	0.127	168	0.137	0.135
37	0.147	0.141	81	0.154	0.149	125	0.134	0.135	169	0.130	0.131
38	0.145	0.136	82	0.121	0.122	126	0.135	0.133	170	0.126	0.125
39	0.126	0.126	83	0.107	0.109	127	0.079	0.078	171	0.094	0.092
40	0.097	0.095	84	0.090	0.083	128	0.100	0.095	172	0.095	0.093
41	0.088	0.087	85	0.082	0.082	129	0.080	0.080	173	0.072	0.075
42	0.068	0.071	86	0.065	0.067	130	0.058	0.064	174	0.058	0.061
43	0.050	0.053	87	0.080	0.076	131	0.095	0.105	175	0.110	0.121
44	0.107	0.096	88	0.094	0.095	132	0.095	0.094	176	0.092	0.099

TABLE 19, CONT'D.

Breath-Alcohol Concentration, g/210 Liters											
No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S
177	0.077	0.076	227	0.077	0.071	277	0.093	0.093	327	0.053	0.055
178	0.085	0.090	228	0.081	0.084	278	0.099	0.092	328	0.047	0.048
179	0.095	0.097	229	0.081	0.086	279	0.090	0.083	329	0.047	0.052
180	0.130	0.130	230	0.073	0.071	280	0.091	0.094	330	0.045	0.048
181	0.059	0.075	231	0.055	0.058	281	0.090	0.094	331	0.042	0.043
182	0.048	0.052	232	0.049	0.058	282	0.146	0.144	332	0.053	0.055
183	0.049	0.050	233	0.040	0.045	283	0.081	0.083	333	0.067	0.062
184	0.048	0.055	234	0.052	0.050	284	0.082	0.075	334	0.081	0.076
185	0.054	0.056	235	0.056	0.060	285	0.089	0.092	335	0.087	0.094
186	0.058	0.057	236	0.056	0.070	286	0.082	0.078	336	0.096	0.099
187	0.054	0.056	237	0.056	0.052	287	0.087	0.086	337	0.100	0.101
188	0.056	0.058	238	0.040	0.038	288	0.090	0.092	338	0.106	0.100
189	0.039	0.040	239	0.079	0.077	289	0.077	0.083	339	0.110	0.110
190	0.069	0.067	240	0.071	0.070	290	0.064	0.061	340	0.112	0.108
191	0.084	0.084	241	0.080	0.079	291	0.061	0.058	341	0.111	0.117
192	0.089	0.087	242	0.077	0.077	292	0.058	0.042	342	0.104	0.092
193	0.075	0.073	243	0.075	0.075	293	0.057	0.063	343	0.106	0.108
194	0.060	0.064	244	0.058	0.060	294	0.055	0.054	344	0.099	0.103
195	0.053	0.048	245	0.055	0.055	295	0.054	0.057	345	0.093	0.099
196	0.055	0.054	246	0.052	0.056	296	0.051	0.051	346	0.097	0.101
197	0.051	0.054	247	0.104	0.103	297	0.046	0.045	347	0.084	0.087
198	0.104	0.110	248	0.103	0.107	298	0.046	0.044	348	0.052	0.041
199	0.095	0.105	249	0.095	0.097	299	0.043	0.037	349	0.070	0.064
200	0.090	0.096	250	0.085	0.087	300	0.095	0.100	350	0.083	0.084
201	0.065	0.066	251	0.062	0.065	301	0.113	0.111	351	0.088	0.093
202	0.054	0.055	252	0.055	0.057	302	0.102	0.106	352	0.097	0.094
203	0.049	0.050	253	0.045	0.048	303	0.109	0.107	353	0.079	0.072
204	0.046	0.046	254	0.047	0.049	304	0.106	0.107	354	0.078	0.077
205	0.043	0.047	255	0.042	0.047	305	0.091	0.079	355	0.075	0.075
206	0.042	0.040	256	0.087	0.084	306	0.088	0.081	356	0.073	0.064
207	0.059	0.062	257	0.057	0.059	307	0.080	0.068	357	0.068	0.060
208	0.063	0.065	258	0.067	0.068	308	0.081	0.081	358	0.061	0.058
209	0.067	0.067	259	0.073	0.073	309	0.078	0.080	359	0.060	0.052
210	0.073	0.069	260	0.074	0.072	310	0.072	0.069	360	0.061	0.055
211	0.074	0.075	261	0.067	0.068	311	0.072	0.068	361	0.048	0.049
212	0.067	0.068	262	0.060	0.063	312	0.068	0.064	362	0.096	0.098
213	0.057	0.060	263	0.060	0.062	313	0.063	0.058	363	0.091	0.093
214	0.056	0.057	264	0.057	0.058	314	0.063	0.061	364	0.116	0.115
215	0.057	0.058	265	0.055	0.057	315	0.058	0.053	365	0.095	0.097
216	0.088	0.072	266	0.094	0.092	316	0.089	0.093	366	0.096	0.101
217	0.096	0.096	267	0.106	0.106	317	0.087	0.085	367	0.076	0.076
218	0.105	0.106	268	0.108	0.108	318	0.085	0.083	368	0.113	0.108
219	0.115	0.122	269	0.109	0.108	319	0.086	0.086	369	0.083	0.077
220	0.105	0.107	270	0.105	0.103	320	0.074	0.076			
221	0.100	0.102	271	0.095	0.095	321	0.066	0.063			
222	0.093	0.094	272	0.090	0.079	322	0.067	0.069			
223	0.090	0.092	273	0.077	0.078	323	0.060	0.057			
224	0.140	0.142	274	0.120	0.118	324	0.056	0.058			
225	0.133	0.131	275	0.117	0.120	325	0.150	0.149			
226	0.119	0.116	276	0.116	0.112	326	0.053	0.052			

NOTES: [1]Automated GC headspace analysis with internal standard; quantitation by automatic electronic peak area integration

[2]Molecular Sieve 13X-sorbed alcohol as simultaneous calibrators

$$y = 0.9675x + 0.00244 \quad \text{(VII)}$$
$$R^2 = 0.974;\ R = 0.987$$
$$N = 369 \text{ result pairs}$$

where y, x, R^2, and R have the significance defined on p. 33.

When the 25 pre-alcohol consumption "blank" result pairs for the 25 subjects were included in the data base and the computation then repeated, the regression analysis yielded

$$y = 0.9810x + 0.00119 \quad \text{(VIII)}$$
$$R^2 = 0.985;\ R = 0.992$$
$$N = 394 \text{ result pairs including 25 blanks}$$

where y, x, R^2, and R have the significance defined on p. 33.

The correlation between the GCI and the m/s BrAC results for the 369 data is also illustrated in Figure 5 with the least-squares best-fit linear regression line shown for equation VII, above. A Student's paired t test on the data contained in Table 19 combined with the 25 "blank" result pairs for the subjects yielded

t-paired value, tP = 1.589

Degrees of Freedom, DF = 393.

For DF = ∞ and a probability level of P = 0.01, the $tP_{0.01}$ table value for Student's t distribution = 2.567 (21). Since $|t_{table}| \geq |t_{calc.}|$ the null hypothesis is sustained, i.e., there is no real difference in the two result populations and hence no real difference in the two breath-alcohol analysis methods.

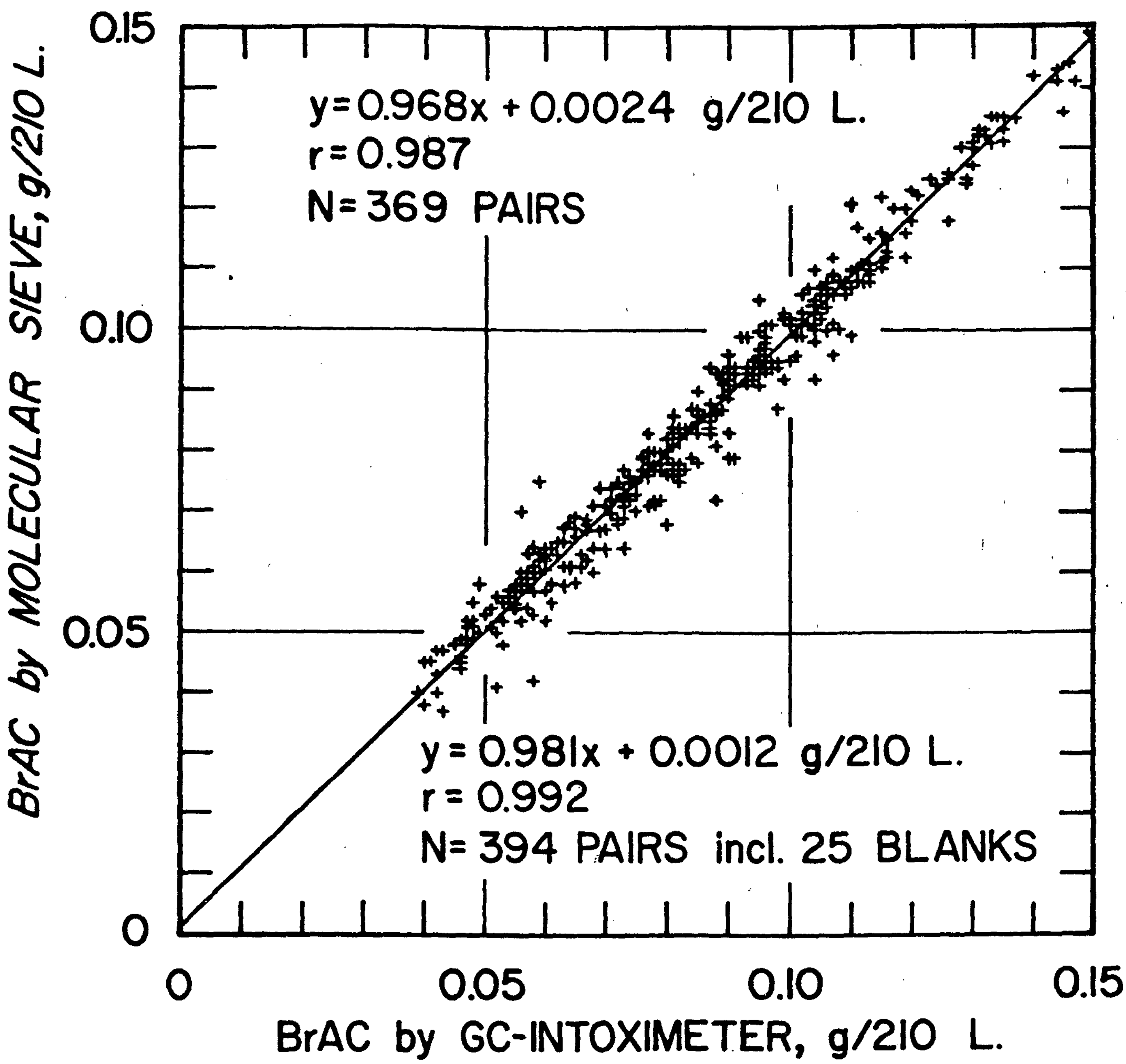

Figure 5. Correlation of Breath-Alcohol Concentrations in Series-Sampled Divided Specimens by GC-Intoximeter and Molecular Sieve

For the same set of 369 data pairs of BrAC measurements by means of GCI and m/s on series-collected breath specimens, the (GCI-m/s) differences between the respective paired results are given in Table 20, together with the applicable statistical summary. When the data in Table 20 were subjected to the nonparametric positive/negative binomial Sign Test for paired data, the following results were obtained:

		N	%
Data Pairs	=	369	100.0
-Δ Frequency	=	172	46.6
+Δ Frequency	=	156	42.3
0Δ Frequency	=	41	11.1

where Δ = BrAC by GCI-BrAC by m/s, g/210 L.

For this binomial sign test, the critical value of r (=the less frequent sign frequency) is 140 at a probability level of P = 0.01 (21). Since the experimental value of r (=156) is not less than the critical value of r (=140) there is no reason to believe that the two methods of analysis yield significantly different results on the same specimens. The BrAC differences data in Table 20 for the 369 result pairs are shown in histogram cell statistical form in Table 21. The corresponding histogram of these (GCI-m/s) BrAC differences for divided breath specimens is illustrated in Figure 6 together with normal curve overlay; one low and two high values are omitted from the graph because the values are beyond the x-axis scale limits, but are included in the calculations. The (GCI-m/s) BrAC difference for these 369 paired results (mean±SD) = +0.00039±0.00413 g/210 L; the span of differences is 0.032 g/210 L for the overall differences range of -0.016 to +0.016 g/210 L. Further to document the

TABLE 20

In-Vivo Studies of Breath-Alcohol in Human Subjects

BrAC Measurements by Gas Chromatography and Molecular Sieve 13X Sorption Tubes

Differences Between Results: GCI-Molecular Sieve 13X Tube Result

(25 subjects; Series Sampling Technique)

Breath-Alcohol Concentration, g/210 Liters							
No.	Difference	No.	Difference	No.	Difference	No.	Difference
01	+0.001	45	+0.001	89	+0.001	133	-0.002
02	+0.003	46	-0.001	90	0.000	134	-0.001
03	+0.006	47	-0.001	91	+0.003	135	+0.005
04	+0.005	48	-0.003	92	+0.003	136	0.000
05	-0.002	49	-0.001	93	+0.001	137	-0.001
06	+0.003	50	0.000	94	+0.005	138	0.000
07	+0.002	51	-0.001	95	-0.002	139	-0.003
08	-0.001	52	-0.002	96	-0.002	140	+0.006
09	-0.003	53	-0.003	97	+0.005	141	+0.003
10	+0.003	54	+0.001	98	+0.008	142	+0.004
11	+0.001	55	0.000	99	+0.011	143	-0.001
12	+0.004	56	+0.001	100	+0.003	144	+0.007
13	+0.004	57	+0.002	101	+0.007	145	-0.002
14	+0.001	58	0.000	102	0.000	146	+0.002
15	0.000	59	-0.001	103	-0.004	147	+0.003
16	-0.001	60	-0.002	104	-0.003	148	-0.002
17	-0.003	61	-0.004	105	-0.004	149	-0.005
18	+0.001	62	+0.003	106	-0.002	150	0.000
19	-0.001	63	+0.003	107	+0.006	151	+0.011
20	-0.005	64	0.000	108	+0.003	152	+0.002
21	-0.002	65	+0.004	109	-0.002	153	+0.004
22	-0.001	66	-0.002	110	-0.001	154	+0.004
23	-0.003	67	0.000	111	+0.006	155	+0.005
24	0.000	68	+0.001	112	-0.003	156	+0.005
25	0.000	69	-0.002	113	+0.002	157	+0.004
26	+0.003	70	-0.001	114	-0.003	158	-0.001
27	-0.001	71	-0.005	115	-0.004	159	-0.001
28	+0.002	72	+0.002	116	+0.004	160	+0.007
29	+0.003	73	+0.008	117	+0.005	161	-0.002
30	0.000	74	0.000	118	-0.002	162	+0.004
31	+0.001	75	+0.003	119	-0.001	163	-0.001
32	0.000	76	-0.004	120	0.000	164	-0.001
33	-0.001	77	-0.004	121	-0.004	165	-0.004
34	-0.001	78	-0.002	122	-0.001	166	-0.004
35	-0.004	79	0.000	123	+0.007	167	0.000
36	+0.003	80	+0.004	124	+0.003	168	+0.002
37	+0.006	81	+0.005	125	-0.001	169	-0.001
38	+0.009	82	-0.001	126	+0.002	170	+0.001
39	0.000	83	-0.002	127	+0.001	171	+0.002
40	+0.002	84	+0.007	128	+0.005	172	+0.002
41	+0.001	85	0.000	129	0.000	173	-0.003
42	-0.003	86	-0.002	130	-0.006	174	-0.003
43	-0.003	87	+0.004	131	-0.010	175	-0.011
44	+0.011	88	-0.001	132	+0.001	176	-0.007

TABLE 20, CONT'D.

Breath-Alcohol Concentration, g/210 L.

No.	Difference	No.	Difference	No.	Difference	No.	Difference
177	+0.001	227	+0.006	277	0.000	327	-0.002
178	-0.005	228	-0.003	278	+0.007	328	-0.001
179	-0.002	229	-0.005	279	+0.007	329	-0.005
180	0.000	230	+0.002	280	-0.003	330	-0.003
181	-0.016	231	-0.003	281	-0.004	331	-0.001
182	-0.004	232	-0.009	282	+0.002	332	-0.002
183	-0.001	233	-0.005	283	-0.002	333	+0.005
184	-0.007	234	+0.002	284	+0.007	334	+0.005
185	-0.002	235	-0.004	285	-0.003	335	-0.007
186	+0.001	236	-0.014	286	+0.004	336	-0.003
187	-0.002	237	+0.004	287	+0.001	337	-0.001
188	-0.002	238	+0.002	288	-0.002	338	+0.006
189	-0.001	239	+0.002	289	-0.006	339	0.000
190	+0.002	240	+0.001	290	+0.003	340	+0.004
191	0.000	241	+0.001	291	+0.003	341	-0.006
192	+0.002	242	0.000	292	+0.016	342	+0.012
193	+0.002	243	0.000	293	-0.006	343	-0.002
194	-0.004	244	-0.002	294	+0.001	344	-0.004
195	+0.005	245	0.000	295	-0.003	345	-0.006
196	+0.001	246	-0.004	296	0.000	346	-0.004
197	-0.003	247	+0.001	297	+0.001	347	-0.003
198	-0.006	248	-0.004	298	+0.002	348	+0.011
199	-0.010	249	-0.002	299	+0.006	349	+0.006
200	-0.006	250	-0.002	300	-0.005	350	-0.001
201	-0.001	251	-0.003	301	+0.002	351	-0.005
202	-0.001	252	-0.002	302	-0.004	352	+0.003
203	-0.001	253	-0.003	303	+0.002	353	+0.007
204	0.000	254	-0.002	304	-0.001	354	+0.001
205	-0.004	255	-0.005	305	+0.012	355	0.000
206	+0.002	256	+0.003	306	+0.007	356	+0.009
207	-0.003	257	-0.002	307	+0.012	357	+0.008
208	-0.002	258	-0.001	308	0.000	358	+0.003
209	0.000	259	0.000	309	-0.002	359	+0.008
210	+0.004	260	+0.002	310	+0.003	360	+0.006
211	-0.001	261	-0.001	311	+0.004	361	-0.001
212	-0.001	262	-0.003	312	+0.004	362	-0.002
213	-0.003	263	-0.002	313	+0.005	363	-0.002
214	-0.001	264	-0.001	314	+0.002	364	+0.001
215	-0.001	265	-0.002	315	+0.005	365	-0.002
216	+0.016	266	+0.002	316	-0.004	366	-0.005
217	0.000	267	0.000	317	+0.002	367	0.000
218	-0.001	268	0.000	318	+0.002	368	+0.005
219	-0.007	269	+0.001	319	0.000	369	+0.006
220	-0.002	270	+0.002	320	-0.002		
221	-0.002	271	0.000	321	+0.003		
222	-0.001	272	+0.011	322	-0.002		
223	-0.002	273	-0.001	323	+0.003		
224	-0.002	274	+0.002	324	-0.002		
225	+0.002	275	-0.003	325	+0.001		
226	+0.003	276	+0.004	326	+0.001		

N = 369
Mean Difference in Results = +0.00032 g/210 L
S.D. of Mean = ±0.004126 g/210 L
Range of Differences = -0.016 to +0.016 g/210 L.

TABLE 21

In-Vivo Studies of Breath-Alcohol in Human Subjects

BrAC Measurements, in Divided Breath Specimens, by
Gas Chromatographic Intoximeter and Molecular Sieve 13X Tubes

Differences Between Results: GCI Result-Molecular Sieve 13X Tube Result

Histogram Cell Statistics

BrAC Difference (GCI-M/S 13X), g/210 Liters				
Cell	Lower & Upper Bounds		Δ Frequency in Cell	% Rel. Δ Frequency
1	-0.017	-0.015	1	0.27
2	-0.015	-0.013	1	0.27
3	-0.013	-0.011	0	0
4	-0.011	-0.009	3	0.81
5	-0.009	-0.007	1	0.27
6	-0.007	-0.005	11	2.98
7	-0.005	-0.003	32	8.67
8	-0.003	-0.001	75	20.33
9	-0.001	+0.001	89	24.12
10	+0.001	+0.003	61	16.53
11	+0.003	+0.005	43	11.65
12	+0.005	+0.007	26	7.05
13	+0.007	+0.009	14	3.80
14	+0.009	+0.011	3	0.81
15	+0.011	+0.013	7	1.90
16	+0.013	+0.015	0	0
17	+0.015	+0.017	2	0.54
	Range= -0.016 to +0.016		N=369	100.0%

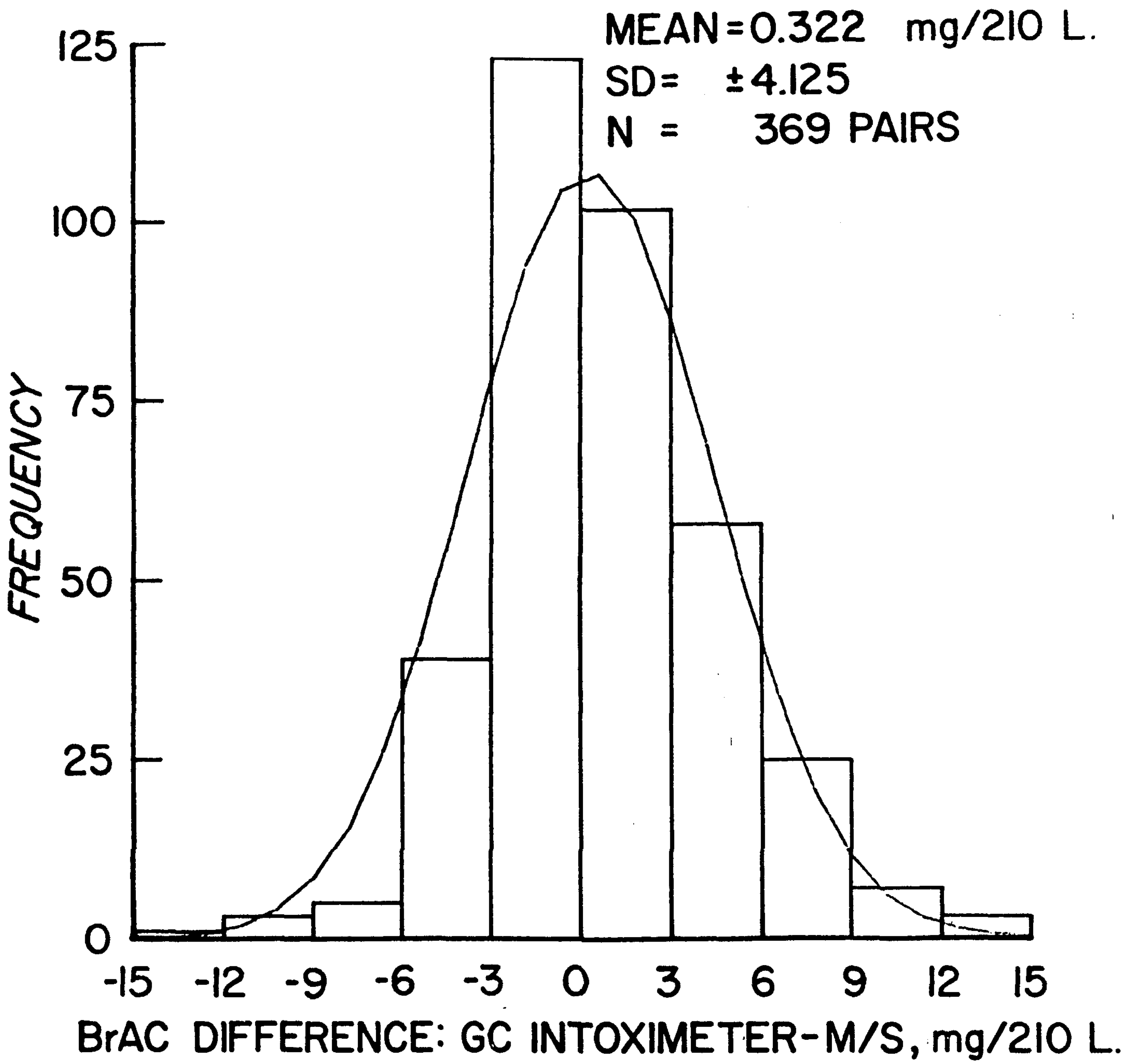

Figure 6. Differences Between Breath-Alcohol Concentrations in Series-Sampled Divided Specimens by GC-Intoximeter and Molecular Sieve (1 Low and 2 High Values are Omitted from the Graph)

essentially Gaussian distribution of the differences data, Figure 7 illustrates them in the form of a cumulative per cent frequency plot employing rectangular coordinates, and Figure 8 presents these data in a cumulative frequency per cent plot employing cumulative probability coordinates. On both plots, the mean BrAC difference determined graphically as equivalent to a cumulative percentage frequency of 50 is near zero g/210 L, in agreement with the calculated mean difference of +0.00032 g/210 L.

To isolate comparative data for BrACs on divided breath specimens by GCI and m/s analysis near two critical concentrations, subsets of the correlation data for the 369 data pairs (Table 19) were abstracted and are reported in Tables 22-25. These tables contain correlation data, and statistical summaries, for the pairs of results obtained when the Gas Chromatographic Intoximeter results were 0.080-0.089 and 0.100-0.109 g/210 L, respectively, (Tables 22 and 23) and when the molecular sieve 13X delayed analysis results were 0.080-0.089 and 0.100-0.109 g/210 L, respectively, (Tables 24 and 25).

Experimental Data and Breath-Alcohol Concentration Differences Reported in Conventional Two-Place BrAC Units. The BrAC results of the foregoing in-vivo studies in this project are reported in 3-decimal place concentration units (0.XXX g/210 L) in the tablulations cited so far, for purposes of statistical analysis and data treatment. Since the results of the BrAC measurements with the molecular sieve tubes will generally be reported only in 2-decimal place form (0.XX g/210 L) for law enforcement applications, the basic results of these subject studies have also been restated in truncated 2-decimal place form, with the third decimal dropped and all calculations performed after such truncation

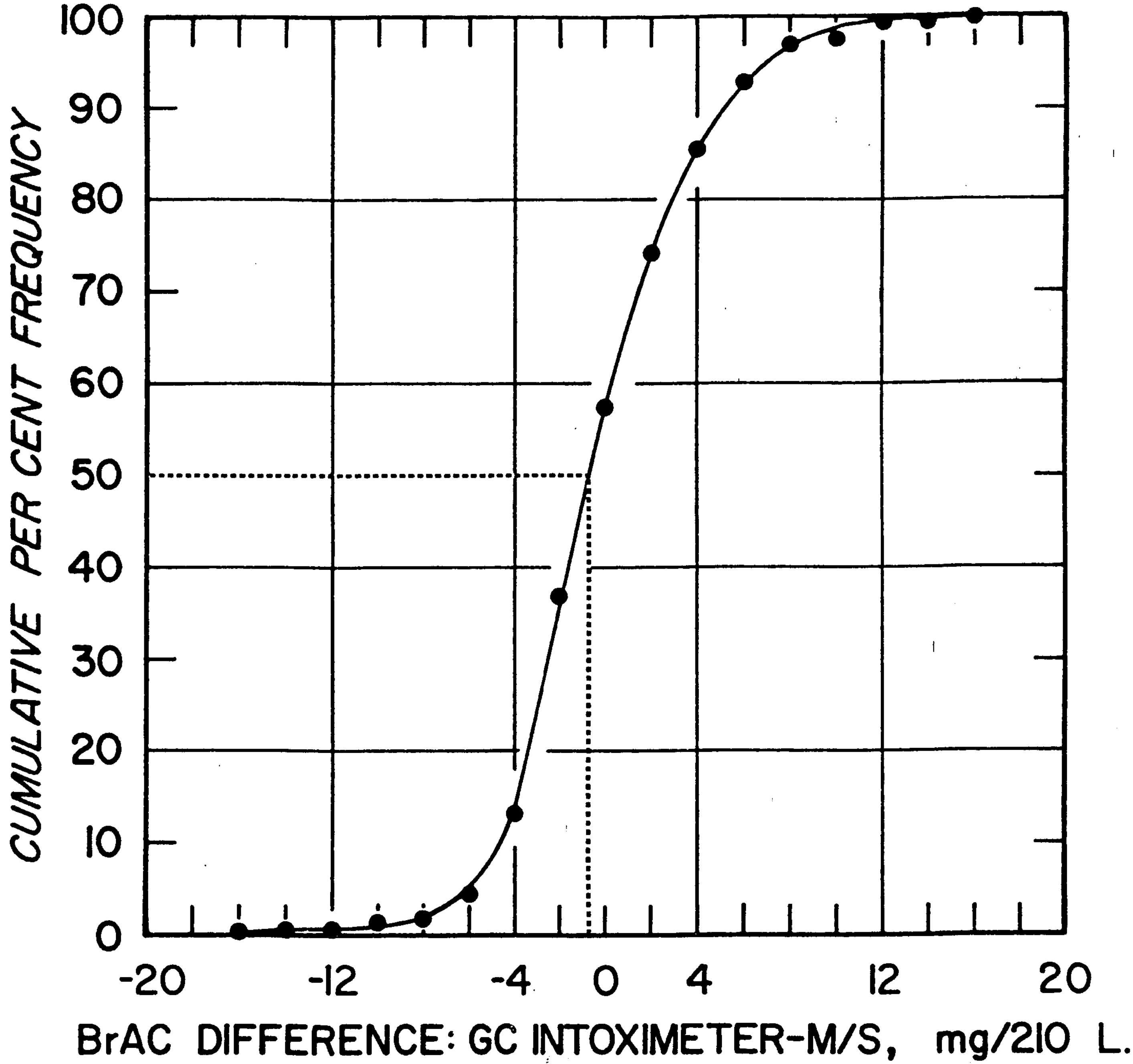

Figure 7. Cumulative Differences Between Breath-Alcohol Concentrations in Series-Sampled Divided Specimens by GC-Intoximeter and Molecular Sieve

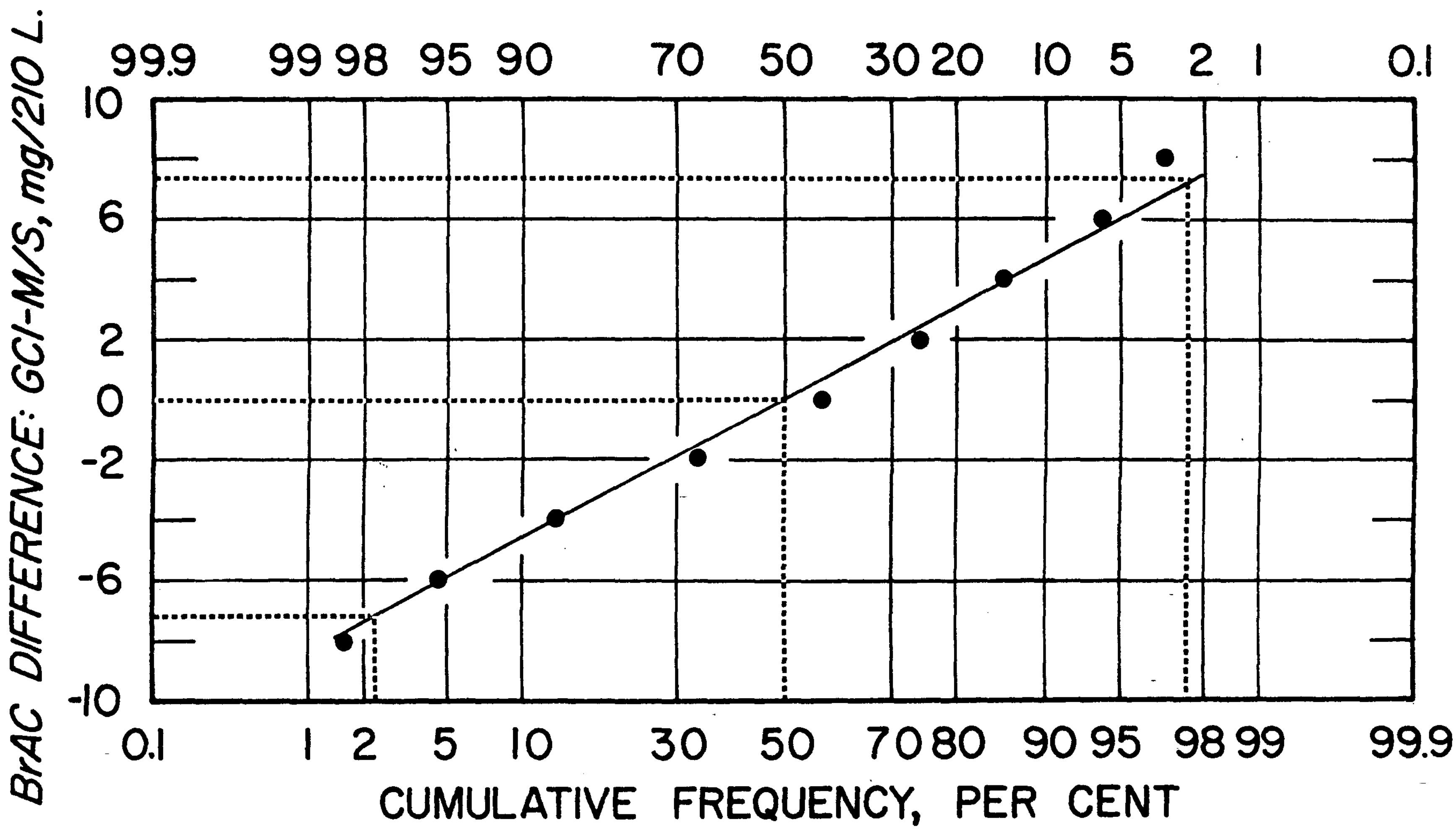

Figure 8. Cumulative Differences Between Breath-Alcohol Concentrations in Series-Sampled Divided Specimens by GC-Intoximeter and Molecular Sieve Plotted on Probability Scale

TABLE 22

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement of BrAC, on Single Breath Specimens, by Gas Chromatographic Intoximeter and Molecular Sieve 13X Sorption Tubes

Subset of Paired Results[1] when GC-Intoximeter Results = 0.08 g/210 Liters

Intox-imeter Result	Breath-Alcohol Concentration, g/210 Liters 0.080	0.081	0.082	0.083	0.084	0.085	0.086	0.087	0.088	0.089
01-	0.076	0.077	0.084	0.083	0.079	0.078	0.086	0.083	0.087	0.091
02-	0.082	0.081	0.082	0.084	0.084	0.086		0.088	0.086	0.089
03-	0.080	0.078	0.076	0.077	0.084	0.090		0.084	0.072	0.087
04-	0.079	0.085	0.075		0.087	0.087		0.086	0.081	0.092
05-	0.068	0.084	0.078			0.083		0.085	0.093	0.093
06-		0.086						0.094		
07-		0.083								
08-		0.081								
09-		0.076								

Statistical Summary

Mean Difference	=	+0.00108 g/210 L
SD of Mean	=	±0.00442 g/210 L
Range	=	-0.007 to +0.016
N	=	48

[1]Horizontal Values = GC Intoximeter Results;
Vertical Column Values = Molecular Sieve 13X Tube Results

TABLE 23

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement of BrAC, on Single Breath Specimens, by Gas Chromatographic Intoximeter

and Molecular Sieve 13X Sorption Tubes

Subset of Paired Results[1] when GC-Intoximeter Results = 0.10 g/210 Liters

Intox-imeter Result	Breath-Alcohol Concentration, g/210 Liters									
	0.100	0.101	0.102	0.103	0.104	0.105	0.106	0.107	0.108	0.109
01-	0.095	0.100	0.101	0.101	0.105	0.102	0.104	0.112	0.100	0.106
02-	0.102	0.099	0.102	0.107	0.098	0.107	0.107	0.106	0.108	0.108
03-	0.102	0.096	0.099		0.104	0.106	0.106	0.096		0.107
04-	0.101		0.103		0.100	0.107	0.107	0.109		
05-			0.106		0.101	0.103	0.100	0.101		
06-					0.110		0.108	0.109		
07-					0.103					
08-					0.092					

Statistical Summary

Mean Difference = +0.00116 g/210 L

SD of Mean = ±0.00383 g/210 L

Range = -0.006 to +0.012 g/210 L

N = 44 Pairs

[1]Horizontal Values = GC Intoximeter Results:

Vertical Column Values = Molecular Sieve 13X Tube Results

TABLE 24

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement of BrAC, on Single Breath Specimens, by Gas Chromatographic Intoximeter and Molecular Sieve 13X Sorption Tubes

Subset of Paired Results[1] when Molecular Sieve 13X Tube Results = 0.08 g/210 Liters

Mol. Sieve 13X Result	Breath-Alcohol Concentration, g/210 Liters									
	0.080	0.081	0.082	0.083	0.084	0.085	0.086	0.087	0.088	0.089
01-	0.077	0.081	0.082	0.083	0.082	0.085	0.088	0.088	0.087	0.090
02-	0.079	0.088	0.080	0.090	0.084	0.087	0.085	0.098		0.089
03-	0.080	0.081		0.087	0.084		0.081	0.089		
04-	0.078			0.090	0.081		0.087	0.085		
05-				0.081	0.087		0.086	0.084		
06-				0.077	0.083					
07-				0.085						

Statistical Summary

Mean Difference	=	-0.00043 g/210 L
SD of Mean	=	±0.00340 g/210 L
Range	=	-0.011 to +0.006 g/210 L
N	=	37 Pairs

[1]Horizontal Values = Molecular Sieve 13X Tube Results
Vertical Column Values = GC-Intoximeter

TABLE 25

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement of BrAC, on Single Breath Specimens, by Gas Chromatographic Intoximeter and Molecular Sieve 13X Sorption Tubes

Subset of Paired Results[1] when Molecular Sieve 13X Tube Results = 0.10 g/210 Liters

Mol. Sieve 13X Result	Breath-Alcohol Concentration, g/210 Liters									
	0.100	0.101	0.102	0.103	0.104	0.105	0.106	0.107	0.108	0.109
01-	0.101	0.104	0.105	0.102	0.106	0.104	0.109	0.106	0.111	0.107
02-	0.108	0.102	0.102	0.104	0.104	0.095	0.107	0.110	0.108	0.113
03-	0.104	0.103	0.099	0.105			0.105	0.105	0.109	0.107
04-	0.095	0.107	0.100	0.099			0.106	0.105	0.112	
05-	0.106	0.100	0.100				0.102	0.103	0.106	
06-		0.097						0.109	0.113	
07-		0.096						0.106		

Statistical Summary

Mean Difference = -0.00020 g/210 L

SD of Mean = ±0.00348 g/210 L

Range = -0.008 to +0.010 g/210 L

N = 46 Pairs

[1]Horizontal Values = Molecular Sieve 13X Tube Results

Vertical Column Values = GC-Intoximeter

of individual results. These compilations are found in Tables 26-37. A comparison of the tabulated data follows:

3-Decimal Place	2-Decimal Place
Table 3	Table 26
Table 14	Table 27
Table 15	Table 28
Table 16	Table 29
Table 17	Table 30
Table 18	Table 31
Table 19	Table 32
Table 20	Table 33
Table 22	Table 34
Table 23	Table 35
Table 24	Table 36
Table 25	Table 37

The statistical summaries of the truncated results naturally differ, on occasion significantly so, from those reflecting the untruncated results.

TABLE 26

Studies of Breath-Alcohol in Human Subjects

Control Measurements[1] of Simulator-Effluent Vapor-Alcohol[2] by Gas Chromatography and Molecular Sieve 13X Sorption Tubes

Vapor-Alcohol Concentration, g/210 Liters							
Series Sampling				Parallel-Split Sampling			
No.	GCI	M/S	Difference GCI-M/S	No.	GCI	M/S	Difference GCI-M/S
01	0.10	0.09	0.00	01	0.10	0.09	0.00
02	0.09	0.09	0.00	02	0.09	0.09	0.00
03	0.09	0.09	0.00	03	0.09	0.09	0.00
04	0.10	0.09	0.00	04	0.09	0.09	0.00
05	0.09	0.09	0.00	05	0.09	0.09	0.00
06	0.10	0.09	0.00	06	0.09	0.09	0.00
07	0.10	0.09	0.00	07	0.10	0.09	0.00
08	0.10	0.09	0.00	08	0.09	0.09	0.00
09	0.10	0.09	0.00	09	0.09	0.09	0.00
10	0.10	0.09	0.00	10	0.09	0.09	0.00
11	0.10	0.09	0.00				
12	0.10	0.09	0.00				
13	0.10	0.10	0.00				
14	0.10	0.09	0.00				
15	0.10	0.10	0.00				
16	0.10	0.09	0.00				
17	0.10	0.10	0.00				
18	0.09	0.09	0.00				
19	0.09	0.09	0.00				
20	0.10	0.10	0.00				
Mean	0.10	0.09	0.00		0.09	0.09	0.00
S.D.	±0.004	±0.004	±0.00		±0.004	±0.000	±0.000
C.V.	4.0%	4.4%	0.0%		4.4%	0.0%	0.0%

NOTES: [1]Between-run individual measurements

[2]Target value = 0.100 g/210 Liters

TABLE 27

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurements[1] by Gas Chromatographic Intoximeter and Molecular Sieve 13X Sorption Tubes in Alcohol-Free Subjects on Single Breath Specimens

Breath-Alcohol Concentration, g/210 Liters					
Experimental Subjects			Control Subjects		
No.	GCI	M/S	No.	GCI	M/S
01-	0.00	0.00	01-	0.00	0.00
02-	0.00	0.00	02-	0.00	0.00
03-	0.00	0.00	03-	0.00	0.00
04-	0.00	0.00	04-	0.00	0.00
05-	0.00	0.00	05-	0.00	0.00
06-	0.00	0.00	06-	0.00	0.00
07-	0.00	0.00	07-	0.00	0.00
08-	0.00	0.00	08-	0.00	0.00
09-	0.00	0.00	09-	0.00	0.00
10-	0.00	0.00	10-	0.00	0.00
11-	0.00	0.00	11-	0.00	0.00
12-	0.00	0.00	12-	0.00	0.00
13-	0.00	0.00	13-	0.00	0.00
14-	0.00	0.00	14-	0.00	0.00
15-	0.00	0.00	15-	0.00	0.00
16-	0.00	0.00	16-	0.00	0.00
17-	0.00	0.00	17-	0.00	0.00
18-	0.00	0.00	18-	0.00	0.00
19-	0.00	0.00	19-	0.00	0.00
20-	0.00	0.00	20-	0.00	0.00
21-	0.00	0.00	21-	0.00	0.00
22-	0.00	0.00	22-	0.00	0.00
23-	0.00	0.00	23-	0.00	0.00
24-	0.00	0.00	24-	0.00	0.00
25-	0.00	0.00	25-	0.00	0.00
26-	0.00	0.00	26-	0.00	0.00
27-	0.00	0.00	27-	0.00	0.00
28-	0.00	0.00	28-	0.00	0.00

NOTES: [1] The results shown are a truncated version of those appearing in Table 14 (with third decimal dropped)

TABLE 28

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurements[1] of BrAC by Gas Chromatography and Molecular

Sieve 13X Sorption Tubes

Shortly After Oral Intake (Residual Mouth Alcohol).

on Single Breath Specimens[2]

Breath-Alcohol Concentration, g/210 Liters			
No.	GCI	M/S	Difference GCI-M/S
	Parallel-Sampling		
01	0.28	0.26	0.02
02	0.17	0.15	0.02
03	0.37	0.31	0.06
04	0.09	0.08	0.01
Mean Difference:	Parallel =		+0.028
	Series-Sampling		
01	0.13	0.13	0.00
02	0.22	0.21	0.01
03	0.13	0.13	0.00
04	0.20	0.21	-0.00
05	0.14	0.13	0.01
06	0.13	0.13	0.00
07	0.13	0.12	0.01
08	0.09	0.09	0.00
09	0.08	0.08	0.00
10	0.20	0.19	0.01
11	0.07	0.07	0.00
12	0.09	0.08	0.01
13	0.14	0.16	-0.02
14	0.28	0.27	0.01
15	0.19	0.19	0.00
16	0.13	0.12	0.01
17	0.08	0.08	0.00
18	0.09	0.10	0.01
Mean Difference:	Series =		+0.002

NOTES: [1]The results shown are a truncated version of those appearing in Table 15 (with third decimal dropped)

[2]Each pair of results is from a different subject

TABLE 29

In-Vivo Studies of Breath-Alcohol in Human Subjects

Correlation of BrAC Measurements[1] by Gas Chromatography and Molecular Sieve 13X Sorption Tubes on Divided Breath Specimens[2,3]

(7 Subjects; Parallel Split Sampling Technique)

Breath-Alcohol Concentration, g/210 Liters								
No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S
01	0.10	0.10	42	0.10	0.10	83	0.10	0.08
02	0.10	0.07	43	0.10	0.10	84	0.10	0.08
03	0.11	0.10	44	0.11	0.11	85	0.10	0.11
04	0.12	0.13	45	0.10	0.10	86	0.11	0.11
05	0.10	0.10	46	0.10	0.11	87	0.10	0.07
06	0.09	0.08	47	0.09	0.01	88	0.09	0.10
07	0.09	0.10	48	0.10	0.03	89	0.09	0.09
08	0.10	0.09	49	0.07	0.08	90	0.07	0.08
09	0.06	0.06	50	0.06	0.05	91	0.05	0.05
10	0.05	0.05	51	0.09	0.08	92	0.08	0.08
11	0.09	0.09	52	0.10	0.11	93	0.10	0.07
12	0.10	0.03	53	0.10	0.11	94	0.10	0.02
13	0.10	0.07	54	0.08	0.06	95	0.08	0.05
14	0.08	0.03	55	0.08	0.07	96	0.08	0.07
15	0.08	0.07	56	0.07	0.07	97	0.08	0.07
16	0.07	0.02	57	0.07	0.07	98	0.06	0.06
17	0.06	0.06	58	0.05	0.05	99	0.05	0.05
18	0.05	0.04	59	0.07	0.06	100	0.08	0.07
19	0.08	0.07	60	0.09	0.09	101	0.09	0.08
20	0.07	0.06	61	0.07	0.06	102	0.06	0.06
21	0.07	0.05	62	0.06	0.05	103	0.06	0.05
22	0.06	0.05	63	0.05	0.05	104	0.05	0.04
23	0.06	0.05	64	0.05	0.05	105	0.05	0.04
24	0.05	0.04	65	0.05	0.04	106	0.06	0.05
25	0.07	0.06	66	0.08	0.07	107	0.07	0.06
26	0.08	0.06	67	0.05	0.05	108	0.05	0.04
27	0.04	0.04	68	0.04	0.04	109	0.04	0.04
28	0.04	0.04	69	0.04	0.03	110	0.04	0.03
29	0.03	0.03	70	0.03	0.03	111	0.11	0.09
30	0.10	0.10	71	0.10	0.08	112	0.10	0.09
31	0.10	0.09	72	0.09	0.08	113	0.09	0.08
32	0.09	0.07	73	0.08	0.07	114	0.08	0.07
33	0.08	0.07	74	0.13	0.10	115	0.13	0.11
34	0.13	0.10	75	0.13	0.11	116	0.11	0.10
35	0.12	0.09	76	0.11	0.09	117	0.09	0.08
36	0.09	0.07	77	0.08	0.07	118	0.08	0.07
37	0.08	0.07	78	0.07	0.06	119	0.07	0.06
38	0.07	0.06	79	0.08	0.07	120	0.08	0.07
39	0.07	0.07	80	0.07	0.06	121	0.06	0.05
40	0.06	0.05	81	0.06	0.05	122	0.05	0.05
41	0.05	0.04	82	0.05	0.04	123	0.04	0.04
						124	0.11	0.07

NOTES: [1]The results shown are a truncated version of those appearing in Table 16 (with third decimal dropped)

[2]Automated GC headspace analysis with internal standard; quantitation by automatic electronic peak area integration

[3]Molecular Sieve 13X-sorbed alcohol as simultaneous calibrators

TABLE 30

In-Vivo Studies of Breath-Alcohol in Human Subjects

BrAC Measurements by Gas Chromatography and Molecular Sieve 13X Sorption Tubes

Differences Between Results: GCI-Molecular Sieve 13X Tube Result

(7 Subjects; Parallel Split-Sampling Technique)

Breath-Alcohol Concentration, g/210 Liters					
No.	Difference	No.	Difference	No.	Difference
01	0.00	42	0.00	83	-0.02
02	-0.02	43	0.00	84	-0.01
03	0.00	44	0.00	85	0.00
04	+0.01	45	0.00	86	0.00
05	0.00	46	+0.01	87	-0.02
06	0.00	47	-0.07	88	0.00
07	0.00	48	-0.06	89	0.00
08	0.00	49	0.00	90	0.00
09	0.00	50	0.00	91	0.00
10	0.00	51	0.00	92	0.00
11	0.00	52	0.00	93	-0.03
12	-0.07	53	0.00	94	-0.08
13	-0.03	54	-0.02	95	-0.02
14	-0.04	55	0.00	96	-0.01
15	0.00	56	0.00	97	0.00
16	-0.05	57	0.00	98	0.00
17	0.00	58	0.00	99	0.00
18	0.00	59	-0.01	100	-0.01
19	-0.01	60	0.00	101	0.00
20	-0.01	61	-0.01	102	0.00
21	-0.01	62	-0.01	103	-0.01
22	0.00	63	0.00	104	0.00
23	0.00	64	0.00	105	0.00
24	0.00	65	0.00	106	-0.01
25	0.00	66	-0.01	107	-0.01
26	-0.01	67	0.00	108	0.00
27	0.00	68	0.00	109	0.00
28	0.00	69	0.00	110	0.00
29	0.00	70	0.00	111	-0.01
30	0.00	71	-0.01	112	0.00
31	0.00	72	-0.01	113	-0.01
32	-0.01	73	-0.01	114	0.00
33	0.00	74	-0.02	115	-0.01
34	-0.02	75	-0.02	116	-0.01
35	-0.02	76	-0.02	117	-0.01
36	-0.01	77	-0.01	118	-0.01
37	0.00	78	-0.01	119	-0.01
38	0.00	79	-0.01	120	-0.01
39	0.00	80	-0.01	121	0.00
40	-0.01	81	-0.01	122	0.00
41	0.00	82	0.00	123	0.00
				124	-0.03

N = 124
Mean Difference in Results = -0.0078 g/210 L
S.D. of Mean = ±0.0147 g/210 L
Range of Differences = -0.08 to +0.01 g/210 L

TABLE 31

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement[1] of BrAC by Gas Chromatography and Molecular Sieve 13X Sorption Tubes on Three Separate Successive Breath Specimens (6 Subjects: Series-Sampling Techniques)

		BrAC, g/210 L.	
Subject	No.	GCI	M/S
	01-	0.07	0.07
A	02-	0.07	0.08
	03-	0.07	0.07
	04-	0.08	-
B	05-	0.08	0.09
	06-	0.08	0.08
	07-	0.04	0.05
C	08-	0.04	0.05
	09-	0.04	0.05
	10-	0.04	0.04
D	11-	0.04	0.04
	12-	0.04	0.04
	13-	0.05	0.05
E	14-	0.05	0.05
	15-	0.05	0.05
	16-	0.04	0.04
F	17-	0.04	0.05
	18-	0.04	0.04

NOTES: [1]The results shown are a truncated version of those appearing in Table 18 (with third decimal dropped)

TABLE 32

In-Vivo Studies of Breath-Alcohol in Human Subjects

Correlation of BrAC Measurements[1] by Gas Chromatography and Molecular Sieve 13X Sorption Tubes on Single Breath Specimens [2,3]

(25 Subjects; Series Sampling Technique)

Breath-Alcohol Concentration, g/210 Liters

No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S
01	0.10	0.10	46	0.11	0.11	89	0.11	0.11	133	0.11	0.11
02	0.10	0.10	46	0.09	0.09	90	0.09	0.09	134	0.07	0.07
03	0.07	0.07	47	0.07	0.07	91	0.07	0.06	135	0.06	0.06
04	0.06	0.06	48	0.06	0.06	92	0.06	0.05	136	0.13	0.13
05	0.12	0.12	49	0.11	0.12	93	0.11	0.11	137	0.11	0.11
06	0.11	0.11	50	0.09	0.09	94	0.11	0.11	138	0.10	0.10
07	0.10	0.10	51	0.10	0.10	95	0.10	0.10	139	0.09	0.10
08	0.10	0.10	52	0.09	0.09	96	0.08	0.09	140	0.08	0.07
09	0.07	0.07	53	0.07	0.08	97	0.07	0.07	141	0.11	0.10
10	0.10	0.10	54	0.11	0.11	98	0.10	0.10	142	0.10	0.10
11	0.10	0.10	55	0.09	0.09	99	0.09	0.08	143	0.09	0.09
12	0.09	0.09	56	0.09	0.09	100	0.09	0.09	144	0.08	0.07
13	0.08	0.07	57	0.07	0.07	101	0.06	0.05	145	0.10	0.10
14	0.09	0.08	58	0.09	0.09	102	0.08	0.08	146	0.08	0.08
15	0.08	0.08	59	0.07	0.07	103	0.05	0.06	147	0.05	0.05
16	0.05	0.05	60	0.05	0.05	104	0.04	0.05	148	0.04	0.04
17	0.04	0.04	61	0.04	0.04	105	0.04	0.04	149	0.04	0.04
18	0.14	0.14	62	0.14	0.14	106	0.13	0.13	150	0.13	0.13
19	0.13	0.13	63	0.11	0.10	107	0.10	0.10	151	0.11	0.09
20	0.10	0.11	64	0.10	0.10	108	0.10	0.09	152	0.10	0.09
21	0.09	0.09	65	0.09	0.09	109	0.12	0.13	153	0.13	0.13
22	0.13	0.13	66	0.13	0.13	110	0.13	0.13	154	0.12	0.12
23	0.12	0.12	67	0.12	0.12	111	0.10	0.09	155	0.10	0.09
24	0.09	0.09	68	0.09	0.09	112	0.09	0.09	156	0.08	0.07
25	0.07	0.07	69	0.08	0.08	113	0.09	0.09	157	0.08	0.08
26	0.08	0.07	70	0.07	0.08	114	0.07	0.07	158	0.07	0.07
27	0.07	0.07	71	0.06	0.07	115	0.06	0.06	159	0.08	0.08
28	0.09	0.09	72	0.10	0.10	116	0.11	0.10	160	0.11	0.11
29	0.11	0.11	73	0.12	0.11	117	0.12	0.12	161	0.10	0.10
30	0.08	0.08	74	0.08	0.08	118	0.08	0.08	162	0.09	0.09
31	0.10	0.10	75	0.09	0.09	119	0.10	0.10	163	0.08	0.08
32	0.09	0.09	76	0.07	0.07	120	0.07	0.07	164	0.07	0.07
33	0.06	0.06	77	0.06	0.06	121	0.06	0.06	165	0.06	0.06
34	0.06	0.06	78	0.06	0.06	122	0.06	0.06	166	0.05	0.05
35	0.04	0.05	79	0.06	0.06	123	0.07	0.07	167	0.08	0.08
36	0.10	0.10	80	0.11	0.11	124	0.13	0.12	168	0.13	0.13
37	0.14	0.14	81	0.15	0.14	125	0.13	0.13	169	0.13	0.13
38	0.14	0.13	82	0.12	0.12	126	0.13	0.13	170	0.12	0.12
39	0.12	0.12	83	0.10	0.10	127	0.07	0.07	171	0.09	0.09
40	0.09	0.09	84	0.09	0.08	128	0.10	0.09	172	0.09	0.09
41	0.08	0.08	85	0.08	0.08	129	0.08	0.08	173	0.07	0.07
42	0.06	0.07	86	0.06	0.06	130	0.05	0.06	174	0.05	0.06
43	0.05	0.05	87	0.08	0.07	131	0.09	0.10	175	0.11	0.12
44	0.10	0.09	88	0.09	0.09	132	0.09	0.09	176	0.09	0.09

TABLE 32, CONT'D.

Breath-Alcohol Concentration, g/210 Liters											
No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S	No.	GCI	M/S
177	0.07	0.07	227	0.07	0.07	277	0.09	0.09	327	0.05	0.05
178	0.08	0.09	228	0.08	0.08	278	0.09	0.09	328	0.04	0.04
179	0.09	0.09	229	0.08	0.08	279	0.09	0.08	329	0.04	0.05
180	0.13	0.13	230	0.07	0.07	280	0.09	0.09	330	0.04	0.04
181	0.05	0.07	231	0.05	0.05	281	0.09	0.09	331	0.04	0.04
182	0.04	0.05	232	0.04	0.05	282	0.14	0.14	332	0.05	0.05
183	0.04	0.05	233	0.04	0.04	283	0.08	0.08	333	0.06	0.06
184	0.04	0.05	234	0.05	0.05	284	0.08	0.07	334	0.08	0.07
185	0.05	0.05	235	0.05	0.06	285	0.08	0.09	335	0.08	0.09
186	0.05	0.05	236	0.05	0.07	286	0.08	0.07	336	0.09	0.09
187	0.05	0.05	237	0.05	0.05	287	0.08	0.08	337	0.10	0.10
188	0.05	0.05	238	0.04	0.03	288	0.09	0.09	338	0.10	0.10
189	0.03	0.04	239	0.07	0.07	289	0.07	0.08	339	0.11	0.11
190	0.06	0.06	240	0.07	0.07	290	0.06	0.06	340	0.11	0.10
191	0.08	0.08	241	0.08	0.07	291	0.06	0.05	341	0.11	0.11
192	0.08	0.08	242	0.07	0.07	292	0.05	0.04	342	0.10	0.09
193	0.07	0.07	243	0.07	0.07	293	0.05	0.06	343	0.10	0.10
194	0.06	0.06	244	0.05	0.06	294	0.05	0.05	344	0.09	0.10
195	0.05	0.04	245	0.05	0.05	295	0.05	0.05	345	0.09	0.09
196	0.05	0.05	246	0.05	0.05	296	0.05	0.05	346	0.09	0.10
197	0.05	0.05	247	0.10	0.10	297	0.04	0.04	347	0.08	0.08
198	0.10	0.11	248	0.10	0.10	298	0.04	0.04	348	0.05	0.04
199	0.09	0.10	249	0.09	0.09	299	0.04	0.03	349	0.07	0.06
200	0.09	0.09	250	0.08	0.08	300	0.09	0.10	350	0.08	0.08
201	0.06	0.06	251	0.06	0.06	301	0.11	0.11	351	0.08	0.09
202	0.05	0.05	252	0.05	0.05	302	0.10	0.10	352	0.09	0.09
203	0.04	0.05	253	0.04	0.04	303	0.10	0.10	353	0.07	0.07
204	0.04	0.04	254	0.04	0.04	304	0.10	0.10	354	0.07	0.07
205	0.04	0.04	255	0.04	0.04	305	0.09	0.07	355	0.07	0.07
206	0.04	0.04	256	0.08	0.08	306	0.08	0.08	356	0.07	0.06
207	0.05	0.06	257	0.05	0.05	307	0.08	0.06	357	0.06	0.06
208	0.06	0.06	258	0.06	0.06	308	0.08	0.08	358	0.06	0.05
209	0.06	0.06	259	0.07	0.07	309	0.07	0.08	359	0.06	0.05
210	0.07	0.06	260	0.07	0.07	310	0.07	0.06	360	0.06	0.05
211	0.07	0.07	261	0.06	0.06	311	0.07	0.06	361	0.04	0.04
212	0.06	0.06	262	0.06	0.06	312	0.06	0.06	362	0.09	0.09
213	0.05	0.06	263	0.06	0.06	313	0.06	0.05	363	0.09	0.09
214	0.05	0.05	264	0.05	0.05	314	0.06	0.06	364	0.11	0.11
215	0.05	0.05	265	0.05	0.05	315	0.05	0.05	365	0.09	0.09
216	0.08	0.07	266	0.09	0.09	316	0.08	0.09	366	0.09	0.10
217	0.09	0.09	267	0.10	0.10	317	0.08	0.08	367	0.07	0.07
218	0.10	0.10	268	0.10	0.10	318	0.08	0.08	368	0.11	0.10
219	0.11	0.12	269	0.10	0.10	319	0.08	0.08	369	0.08	0.07
220	0.10	0.10	270	0.10	0.10	320	0.07	0.07			
221	0.10	0.10	271	0.09	0.09	321	0.06	0.06			
222	0.09	0.09	272	0.09	0.07	322	0.06	0.06			
223	0.09	0.09	273	0.07	0.07	323	0.06	0.05			
224	0.14	0.14	274	0.12	0.11	324	0.05	0.05			
225	0.13	0.13	275	0.11	0.12	325	0.15	0.14			
226	0.11	0.11	276	0.11	0.11	326	0.05	0.05			

NOTES: [1]The results shown are a truncated version of those appearing in Table 19 (with third decimal dropped)

[2]Automated GC headspace analysis with internal standard; quantitation by automatic electronic peak area integration

[3]Molecular Sieve 13X-sorbed alcohol as simultaneous calibrators

TABLE 33

In-Vivo Studies of Breath-Alcohol in Human Subjects

BrAC Measurements[1] by Gas Chromatography and Molecular Sieve 13X Sorption Tubes

Differences Between Results: GCI-Molecular Sieve 13X Tube Result

(25 subjects; Series Sampling Technique)

Breath-Alcohol Concentration, g/210 Liters							
No.	Difference	No.	Difference	No.	Difference	No.	Difference
01	0.00	45	0.00	89	0.00	133	0.00
02	0.00	46	0.00	90	0.00	134	0.00
03	0.00	47	0.00	91	0.00	135	0.00
04	0.00	48	0.00	92	0.00	136	0.00
05	0.00	49	0.00	93	0.00	137	0.00
06	0.00	50	0.00	94	0.00	138	0.00
07	0.00	51	0.00	95	0.00	139	0.00
08	0.00	52	0.00	96	0.00	140	0.00
09	0.00	53	0.00	97	0.00	141	0.00
10	0.00	54	0.00	98	0.00	142	0.00
11	0.00	55	0.00	99	+0.01	143	0.00
12	0.00	56	0.00	100	0.00	144	0.00
13	0.00	57	0.00	101	0.00	145	0.00
14	0.00	58	0.00	102	0.00	146	0.00
15	0.00	59	0.00	103	0.00	147	0.00
16	0.00	60	0.00	104	0.00	148	0.00
17	0.00	61	0.00	105	0.00	149	0.00
18	0.00	62	0.00	106	0.00	150	0.00
19	0.00	63	0.00	107	0.00	151	+0.01
20	0.00	64	0.00	108	0.00	152	0.00
21	0.00	65	0.00	109	0.00	153	0.00
22	0.00	66	0.00	110	0.00	154	0.00
23	0.00	67	0.00	111	0.00	155	0.00
24	0.00	68	0.00	112	0.00	156	0.00
25	0.00	69	0.00	113	0.00	157	0.00
26	0.00	70	0.00	114	0.00	158	0.00
27	0.00	71	0.00	115	0.00	159	0.00
28	0.00	72	0.00	116	0.00	160	0.00
[illegible]	0.00	73	0.00	117	0.00	161	0.00
[illegible]	0.00	74	0.00	118	0.00	162	0.00
	0.00	75	0.00	119	0.00	163	0.00
	0.00	76	0.00	120	0.00	164	0.00
	0.00	77	0.00	121	0.00	165	0.00
	0.00	78	0.00	122	0.00	166	0.00
	0.00	79	0.00	123	0.00	167	0.00
	0.00	80	0.00	124	0.00	168	0.00
	0.00	81	0.00	125	0.00	169	0.00
	0.00	82	0.00	126	0.00	170	0.00
	0.00	83	0.00	127	0.00	171	0.00
	0.00	84	0.00	128	0.00	172	0.00
	0.00	85	0.00	129	0.00	173	0.00
	0.00	86	0.00	130	0.00	174	0.00
	0.00	87	0.00	131	-0.01	175	-0.01
	+0.01	88	0.00	132	0.00	176	0.00

S: [1]The results shown are a truncated version of those appearing in Table 20 (with third decimal dropped)

TABLE 33, CONT'D.

	Breath-Alcohol Concentration, g/210 L.						
No.	Difference	No.	Difference	No.	Difference	No.	Difference
177	0.00	227	0.00	277	0.00	327	0.00
178	0.00	228	0.00	278	0.00	328	0.00
179	0.00	229	0.00	279	0.00	329	0.00
180	0.00	230	0.00	280	0.00	330	0.00
181	-0.01	231	0.00	281	0.00	331	0.00
182	0.00	232	0.00	282	0.00	332	0.00
183	0.00	233	0.00	283	0.00	333	0.00
184	0.00	234	0.00	284	0.00	334	0.00
185	0.00	235	0.00	285	0.00	335	0.00
186	0.00	236	-0.01	286	0.00	336	0.00
187	0.00	237	0.00	287	0.00	337	0.00
188	0.00	238	0.00	288	0.00	338	0.00
189	0.00	239	0.00	289	0.00	339	0.00
190	0.00	240	0.00	290	0.00	340	0.00
191	0.00	241	0.00	291	0.00	341	0.00
192	0.00	242	0.00	292	+0.01	342	+0.01
193	0.00	243	0.00	293	0.00	343	0.00
194	0.00	244	0.00	294	0.00	344	0.00
195	0.00	245	0.00	295	0.00	345	0.00
196	0.00	246	0.00	296	0.00	346	0.00
197	0.00	247	0.00	297	0.00	347	0.00
198	0.00	248	0.00	298	0.00	348	+0.01
199	-0.01	249	0.00	299	0.00	349	0.00
200	0.00	250	0.00	300	0.00	350	0.00
201	0.00	251	0.00	301	0.00	351	0.00
202	0.00	252	0.00	302	0.00	352	0.00
203	0.00	253	0.00	303	0.00	353	0.00
204	0.00	254	0.00	304	0.00	354	0.00
205	0.00	255	0.00	305	+0.01	355	0.00
206	0.00	256	0.00	306	0.00	356	0.00
207	0.00	257	0.00	307	+0.01	357	0.00
208	0.00	258	0.00	308	0.00	358	0.00
209	0.00	259	0.00	309	0.00	359	0.00
210	0.00	260	0.00	310	0.00	360	0.00
211	0.00	261	0.00	311	0.00	361	0.00
212	0.00	262	0.00	312	0.00	362	0.00
213	0.00	263	0.00	313	0.00	363	0.00
214	0.00	264	0.00	314	0.00	364	0.00
215	0.00	265	0.00	315	0.00	365	0.00
216	+0.01	266	0.00	316	0.00	366	0.00
217	0.00	267	0.00	317	0.00	367	0.00
218	0.00	268	0.00	318	0.00	368	0.00
219	0.00	269	0.00	319	0.00	369	0.00
220	0.00	270	0.00	320	0.00		
221	0.00	271	0.00	321	0.00		
222	0.00	272	+0.01	322	0.00		
223	0.00	273	0.00	323	0.00		
224	0.00	274	0.00	324	0.00		
225	0.00	275	0.00	325	0.00		
226	0.00	276	0.00	326	0.00		

N = 369
Mean Difference in Results = +0.00014 g/210 L
S.D. of Mean = ±0.00201 g/210 L
Range of Differences = -0.01 to +0.01 g/210 L

TABLE 34

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement[1] of BrAC, on Single Breath Specimens, by Gas Chromatographic Intoximeter

and Molecular Sieve 13X Sorption Tubes

Subset of Paired Results when GC-Intoximeter Results = 0.08 g/210 Liters

Intox-imeter Result	Breath-Alcohol Concentration, g/210 Liters									
	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08
01-	0.07	0.07	0.08	0.08	0.07	0.07	0.08	0.08	0.08	0.09
02-	0.08	0.08	0.08	0.08	0.08	0.08		0.08	0.08	0.08
03-	0.08	0.07	0.07	0.07	0.08	0.09		0.08	0.07	0.08
04-	0.07	0.08	0.07		0.08	0.08		0.08	0.08	0.09
05-	0.06	0.08	0.07			0.08		0.08	0.09	0.09
06-		0.08						0.09		
07-		0.08								
08-		0.08								
09-		0.07								

<u>Statistical Summary</u>

Mean Difference	=	+0.0017 g/210 L
SD of Mean	=	±0.0066 g/210 L
Range	=	-0.01 to +0.02 g/210 L
N	=	48 pairs

NOTES: [1]The results shown are a truncated version of those appearing in Table 23 (with third decimal dropped)

[2]Horizontal Values = GC-Intoximeter Results;

Vertical Column Values = Molecular Sieve 13X Tube Results

TABLE 35

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement[1] of BrAC, on Single Breath Specimens, by Gas Chromatographic Intoximeter and Molecular Sieve 13X Sorption Tubes

Subset of Paired Results[2] when GC-Intoximeter Results = 0.10 g/210 Liters

Intox-imeter Result	Breath-Alcohol Concentration, g/210 Liters									
	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
01-	0.09	0.10	0.10	0.10	0.10	0.10	0.10	0.11	0.10	0.10
02-	0.10	0.09	0.10	0.10	0.09	0.10	0.10	0.10	0.10	0.10
03-	0.10	0.09	0.09		0.10	0.10	0.10	0.09		0.10
04-	0.10		0.10		0.10	0.10	0.10	0.10		
05-			0.10		0.10	0.10	0.10	0.10		
06-					0.11		0.10	0.10		
07-					0.10					
08-					0.09					

Statistical Summary

Mean Difference	=	+0.0011 g/210 L
SD of Mean	=	±0.0044 g/210 L
Range	=	-0.01 to +0.01
N	=	44

NOTES: [1]The results shown are a truncated version of those appearing in Table 22 (with third decimal dropped)

[2]Horizontal Values = GC-Intoximeter Results;

Vertical Column Values = Molecular Sieve 13X Tube Results

TABLE 36

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement[1] of BrAC, on Single Breath Specimens, by Gas Chromatographic Intoximeter and Molecular Sieve 13X Sorption Tubes

Subset of Paired Results[2] when Molecular Sieve 13X Tube Results = 0.08 g/210 Liters

Mol. Sieve 13X Result	Breath-Alcohol Concentration, g/210 Liters									
	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08
01-	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.08	0.09
02-	0.07	0.08	0.08	0.09	0.08	0.08	0.08	0.09		0.08
03-	0.08	0.08		0.08	0.08		0.08	0.08		
04-	0.07			0.09	0.08		0.08	0.08		
05-				0.08	0.08		0.08	0.08		
06-				0.07	0.08					
07-				0.08						

Statistical Summary

Mean Difference	=	0.0000 g/210 L
SD of Mean	=	±0.0047 g/210 L
Range	=	-0.01 to +0.02
N	=	37 pairs

NOTES: [1]The results shown are a truncated version of those appearing in Table 24 (with third decimal dropped)

[2]Horizontal Values = Molecular Sieve 13X Tube Results

Vertical Column Values = GC-Intoximeter

TABLE 37

In-Vivo Studies of Breath-Alcohol in Human Subjects

Measurement[1] of BrAC, on Single Breath Specimens, by Gas Chromatographic Intoximeter and Molecular Sieve 13X Sorption Tubes

Subset of Paired Results[2] when Molecular Sieve 13X Tube Results = 0.10 g/210 Liters

Mol. Sieve 13X Result	Breath-Alcohol Concentration, g/210 Liters									
	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
01-	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.11	0.10
02-	0.10	0.10	0.10	0.10	0.10	0.09	0.10	0.11	0.10	0.11
03-	0.10	0.10	0.09	0.10			0.10	0.10	0.10	0.10
04-	0.09	0.10	0.10	0.09			0.10	0.10	0.11	
05-	0.10	0.10	0.10				0.10	0.10	0.10	
06-		0.09						0.10	0.11	
07-		0.09						0.10		

<u>Statistical Summary</u>

Mean Difference	=	+0.0002 g/210 L
SD of Mean	=	±0.0049 g/210 L
Range	=	-0.01 to +0.01
N	=	46 pairs

NOTES: [1] The results shown are a truncated version of those appearing in Table 25 (with third decimal dropped)

[2] Horizontal Values = Molecular Sieve 13X Tube Results

Vertical Column Values = GC-Intoximeter

DISCUSSION AND CONCLUSIONS

Methodology

The methodological and procedural information was kept brief in this report since all key aspects have been previously reported by this author in the accessible scientific literature. It should be recognized, however, that we conducted this project as a research study and, therefore, employed research approaches, research-grade instrumentation and methods, as well as necessary improvisations and artifices to accomplish our immediate objectives. Our procedures were, therefore, not necessarily optimal or appropriate for field use.

We have demonstrated and documented adequately the feasibility and usefulness of molecular sieve 13X sorption as a temporary breath-alcohol storage procedure, and the validity of breath-alcohol analysis following molecular sieve sorption in comparison with direct breath-alcohol analysis of the same breath specimens by means of the Gas Chromatographic Intoximeter. Certain administrative and technical problems, such as selection and implementation of sample container and identifications system compatible with local legal and operational requirements, need to be dealt with on an individual basis.

Achievement of the degree of accuracy and precision in breath-alcohol analysis after intervening m/s sorption which we obtained requires, among other factors, use of highly standardized contemporaneous calibrators (often called "standards"). We found it necessary to employ calibrators prepared by sorbing known quantities of alcohol on m/s columns in a manner totally analogous to that for breath sampling. Ideally, these calibrators should be prepared, as were ours, with the same breath collection/delivery device used to charge m/s columns with breath-alcohol; thus eliminating the delivered breath volume as a variable. When that practice is not followed, it is necessary to ascertain and take into account the exact breath volume delivered to a m/s column.

Concerning the alcohol analyses, two comments seem pertinent. In recognition of the widespread convention of using a 2100X calibration factor for breath-alcohol analyzers reporting the results in terms of the supposedly corresponding blood-alcohol concentration (6, 23-24), but without thereby endorsing it, we calibrated our breath-alcohol analyzers to reflect results in g/210 liters and reduced our raw experimental data to BrACs in g/210 L units. Our gas chromatographic analyses were performed by headspace analysis with an automated instrument. It was convenient and practical for us to quantitate the instrument responses with a coupled automatic electronic integrator-printer, and we thus quantitated by peak-area measurement for most portions of this project, including all drinking subject BrAC measurements after m/s sorption and elution. When only water vapor, ethanol, and the internal standard acetonitrile are present in the headspace (and the resulting chromatograms), the results thus obtained are identical to those yielded by quantitation by peak-height measurement. The data in Tables 1 and 2, and Tables 4 and 5 demonstrate that essentially identical and entirely satisfactory results are obtainable by either procedure. However, if significant trace contamination with other volatiles is present in sorbed columns which a particular gas chromatograph or electronic peak area integrator cannot resolve and thus distinguish completely from ethanol, the accuracy and precision of the GC analysis will necessarily by decreased by use of peak-area measurements. It is thus important to avoid contamination of m/s tubes with acetone and other volatiles difficult to separate from ethanol by simple gas chromatography.

This project involved generation of large quantities of data and collection and analysis of many specimens and almost innumerable standards, calibrators, and controls. To avoid bias in the comparisons and correlations (e.g., the paired results of GCI tests and breath-alcohol analyses following m/s sorption), we always used appropriate single or double-blind techniques.

The breath collection arrangement shown in Figure 1 requires comment. The Principal Investigator favors on theoretical and experimental grounds a series or tandem scheme of molecular sieve collection unit and quantitative evidential breath-alcohol analyzer. For law enforcement purposes, the former should precede the base unit, e.g., breath → molecular sieve collection unit → breath-analyzer → vent. That arrangement requires, i.a., that the breath-analyzer breath stream be vented to the atmosphere at ambient pressure and that there be no significant flow obstructions or pressure differential between the breath at the m/s collection unit outlet and at the breath-analyzer inlet. That compatibility must be established experimentally. In particular, there are severe limitations to a parallel, breath stream splitting arrangement instead of the in-series scheme. The adverse effect of the parallel arrangement is documented in the preliminary data, obtained with that arrangement, in Tables 3, 15, 16 and 17 and in Figure 4. It is evident from the relatively large BrAC differences for GCI and m/s analyses on breath specimens divided in-parallel and the low correlation coefficients for the regression equations V and VI that parallel splitting of breath specimens is far inferior to series-sampling for the devices employed in this project. That will be a general situation except, perhaps, for collection devices and breath-analyzers completely matched in terms of breath volumes, internal flow rates, and inlet and outlet pressures.

A key feature of this delayed breath-alcohol analysis system with intervening alcohol sorption on molecular sieve 13X is its compatibility with all existing commercial devices for quantitative evidential breath-alcohol analysis. Such combination can be achieved without any modifications of the basic BCD illustrated in Figure 2; and requires either no accessory to or modification of the breath-alcohol analyzer, or only minor attachments or accessories, or use of an auxiliary breath collection unit - depending upon the particular breath-alcohol analyzer. For example, if the m/s column is to be used in conjunction with any current model Intoxilyzer (CMI, Inc., Minturn, CO 81645), the m/s tube is simply inserted into the breath outlet tube of the Intoxilyzer after completion of the direct breath-alcohol analysis, and the breath sample contained in the sample chamber forced through the m/s tube by the internal Intoxilyzer pump as in a normal air-blank step. For use with a Model 900 or 900A Breathalyzer (Smith & Wesson Electronics, Co., Springfield, MA 01101) an additional breath sample is collected within the Breathalyzer after completion of the normal direct analysis, and the breath sample is delivered into the m/s tube which is attached by means of a short silicone rubber sleeve to the delivery tube (instead of the usual bubbler tube). For use with a Gas Chromatographic Intoximeter (Intoximeters, Inc., St. Louis, MO 63103) or other breath-alcohol analyzer which cannot deliver an unaltered breath specimen of known volume, a heated auxiliary breath collection-and-delivery device (such as the DPC Intoximeter used in this study) must be coupled - preferably in series - with the analyzer and the breath specimen aliquot collected with the auxiliary device is delivered into the m/s tube. When desired, such an auxiliary external collection unit can be combined, in series, with any breath-alcohol analyzer by means of simple connections.

Human Subject Aspects

Although the number of experimental subjects employed in this project was necessarily limited and only healthy volunteers in a limited age group were tested, we believe that the data and conclusions are generally applicable to the population at large.

It was again confirmed during this study that the results of breath-alcohol analyses are influenced by individual subject factors, including their breath-providing techniques and the existing BrAC. Despite conscious and consistent efforts to standardize all of the recognized relevant factors subject to our control, some residual variability of unknown extent must be ascribed to this source. Some of those effects become evident in the small test series in Table 18. With much increased BrACs, the effects are greater than those tabulated.

As a matter of interest, we measured BrACs in some subjects shortly after completion of oral alcohol ingestion and before the normal pre-test "deprivation" period of 15 minutes had elapsed. Those data, in Table 15 and Figure 3 include BrACs up to 0.28 g/210 L. The overall correlation of these paired GCI versus m/s results does not vary significantly from that of the paired BrAC data obtained after at least a 15-minute deprivation period, as shown by the mean difference and regression equations III and IV, and it is believed that the two sets of data could properly be merged; however, we did not combine them in our data analysis.

The human subject respiratory parameter measurements found in this project and shown in Tables 12 and 13 are very comparable to those previously found in our laboratory in subject groups of about the same size and male/female composition (18, 25). These data, and our previous observation that an alveolar breath-alcohol plateau is reached only after discard of the initial two-thirds of available breath (7, 18, 25), provided the basis for our decision to employ a 250 ml. breath volume for the retained breath-alcohol sample. Even persons with the lowest exhalation volumes could readily provide that breath quantity within the alveolar plateau region (i.e., from expired alveolar air) by end-expiratory trapping a full exhalation after only normal (not maximal) inhalation. The advantages of this breath sample volume over smaller volumes (i.e., 50 ml.) are significant with respect to greatly improved signal/noise ratio in the subsequent gas chromatographic analysis of alcohol and with respect to the accuracy and precision of the sample volume collection vis-a-vis equipment deadspace and similar factors.

In-Vitro Studies of Alcohol-Vapor Analysis with Molecular Sieve Sorption.

The performance of this system for delayed breath-alcohol analysis following sorption on molecular sieve 13X fully met or exceeded, in all applicable respects, the corresponding requirements of the U. S. Department of Transportation for quantitative evidential breath-alcohol analyzers (8), as shown in Tables 4 and 5, for both peak height and peak area GC quantitation techniques. The in-vitro accuracy and precision results for this delayed analysis involving collection, elution, and subsequent GC analysis of alcohol also compare favorably with those for direct analysis by means of the Mark IV Gas Chromatographic

Intoximeter shown in Tables 9 and 10. (These tables incidentally, document the residual effect in such precision/accuracy studies of breath-alcohol analyzers of the means of producing the target vapor-alcohol specimens; the coefficient of variation for replicate analyses of a homogeneous ethanol-nitrogen gas mixture was only 77% of that for replicate analyses of individually-produced effluents from tandem simulators, with comparable accuracy of the results.) These data allow the conclusion that the two instrumental methods of analysis are not primarily responsible for such variability as was found in the paired analyses in the human subject phases of this project. The in-vitro accuracy and precision of the Mark IV GCI found in this study is very closely comparable to those previously found in our laboratory in similar studies with Model 900A Breathalyzers and Model 4011 Intoxilyzers.

The in-vitro studies also documented that the molecular sieve system employed in this project has ample alcohol capacity well beyond any expectable breath-alcohol concentration, when the breath sample volume is 250 ml.

Short-term storage and retention of alcohol on molecular sieve 13X columns was found to be excellent. Intervening storage stress at 0° and $40^{\circ}C$ for up to 72 hours did not increase the completely negative blank values nor result in any alcohol loss, as shown in Table 8. The prospects for long-term alcohol retention on molecular sieve 13X, for up to 1 year, are also excellent and such long-term retention without significant loss was shown to be possible. The alcohol stability during long-term storage is, however, subject to the effects of mechanical closure of the glass columns, as shown in Table 7. Further work may be required to perfect this aspect of the system.

In-Vivo Studies of Breath-Alcohol Analysis by Means of the GCI and Molecular Sieve 13X Sorption.

These studies of breath-alcohol analysis in human subjects, on split breath specimens, with the m/s tube. and their results, are the heart of this project and, accordingly, these data are the most voluminous and extensive.

Response of the m/s method in alcohol-free subjects was tested in a total of 56 subjects. Alcohol-free status of these subjects was established before conducting these breath "blank" analyses by analyzing their breath directly by several chemically different methods, including infrared absorptiometry and gas chromatography. The resultant data are given in Table 14. It is noteworthy that the m/s method did not yield positive results in any subject. Although not shown in tabular form, room air "blank" analyses by the m/s method were also found to be consistently and completely negative.

The comparative data for GCI and m/s analyses of breath in specimens divided by parallel-sampling (Tables 15, 16, and 17 and Figure 4) are reasonably satisfactory, but much less so than those for the series-sampling technique.

Comparative data for breath-alcohol testing of subjects with residual mouth-alcohol collected by series-sampling (Table 15 and Figure 3) are surprisingly good, considering the nature of these nonhomogeneous samples. These data indicate that no lessening of the high correlation is to be expected at BrACs much higher than the 0.15 g/210 L limit for these in-vivo studies. The brief data in Table 18, and our other observations, document that there is an element of subject-dependency in such correlations, as there is for most if not all breath-alcohol analyses because of the subjects' conscious or unconscious manipulations of breath pressures, flow rates, timing, completeness of expiration, etc. In some subjects, the effects of alcohol stimulate a distinct and often verbalized "gamesmanship" manifested by efforts to manipulate the results.

The great data mass of Tables 19 and 20 constitutes one of the longest correlation series ever reported for breath-alcohol analyses, especially under controlled laboratory conditions. Accordingly, these data were difficult to process and analyze by noncomputer means. The overall agreement or correlation between GCI and m/s results was excellent. The linear regression equation VIII on p. 46 can be considered the "Master Equation" for this correlation. As judged by the coefficients of determination and correlation, as well as by the graphical features of Figure 5, the overall correlation is very high. Equations VII or VIII can, of course, be used to predict the m/s BrAC result expectible for a given BrAC result by means of the GCI, as illustrated for Equation VIII:

	BrAC, g/210 L			
Gas Chromatographic Intoximeter Result:	0.050	0.100	0.150	0.200
Predicted Molecular Sieve 13X Result:	0.050	0.099	0.148	0.197

Since the regression equation VIII indicates a typical, but slight negative bias of 1.9%, or less, for m/s-derived BrACs, the m/s BrAC result can be expected to be slightly lower than a GCI test result on another aliquot of the same breath specimen (e.g., -0.71% at a BrAC of 0.10 g/210 L or -1.31% at 0.20 g/210 L). The validity of the experimentally determined correlation and of the two regression analyses is confirmed by the substantial similarity between equations VII and VIII and the closeness of the slopes and the y-intercepts for the correlation omitting blank values and that including them. These correlations involve the assumptions that the GCI apparatus used in this study was consistently calibrated in g/210 L and that the DPC collection device consistently delivered a 250 ml. breath volume. We believe that those assumptions are valid and in accordance with the experimental confirmations we performed periodically throughout the project.

The closeness of agreement between the paired results, reflected in the mean BrAC difference of close to zero, is quite remarkable when it is considered that at least the following factors enter into the respective accuracy of the two independent analyses:

TABLE 37. Some Factors Affecting the Respective GCI and M/S Results

- Accuracy of GCI Analysis and GCI Calibration
- Division and Aliquoting of Breath Specimen
- Delivery Volume of M/S Collection Unit
- Completeness of EtOH Sorption on M/S
- Completeness of EtOH Retention by M/S
- Completeness of EtOH Elution from M/S
- Accuracy of GC Analysis of M/S Eluate for EtOH
- Absence/Presence of Trace Contaminants in M/S Traps Affecting GC Separation

The "Accuracy of the GC Analysis" factor includes such methodological variables as the accuracy of the external alcohol calibrators ("standards"), the completeness of separation of GC response to acetonitrile and ethanol from those of possible contaminants such as acetone, the accuracy of the instrument response to given quantities of the analytes, the linearity of the instant, individual calibration curve, and the correctness of the result computations. In this project, we conscientiously controlled (or compensated for) these and other variables to the maximum extent feasible. That is a considerable challenge, and cannot be expected to be matched in usual field operations.

Equally as informative as the correlation of GCI and m/s results is examination of the differences in paired results; i.e., the 369 pairs excluding breath blanks and mouth-alcohol specimens. These data in Table 20 follow an essentially Gaussian or normal distribution as shown in Figure 6, 7 and 8. This is confirmed by the Gaussian shape of the normal curve overlay in Figure 6, the sigma-shape of the cumulative per cent difference curve in rectangular coordinates in Figure 7, and the linear relation of the cumulative per cent difference values between 1.5 and 98.5% in the probability coordinates in Figure 8; all tests of the normal or Gaussian distribution of the differences data. Certain statistical predictions based on normal distribution probabilities are therefore warranted. The experimental mean BrAC difference between the two sets of results (+0.00032 g/210 L) is not functionally significant. From it and the experimental standard deviation of this mean, it can be validly predicted that 68.3% of all differences in paired analyses by GCI (or other valid quantitative evidential breath-alcohol analyses) and m/s + GC will lie between -0.0038 and +0.0044 g/210 L, that 95.5% will lie between -0.0079 and +0.0086 g/210 L, and that 99.7% will lie between -0.0120 and +0.0156 g/210 L. In other words, it can be expected that 997 out of 1,000 differences in BrAC measurements on the same breath specimens by GCI (or other valid quantitative evidential breath-alcohol analyzer) and m/s + GC analysis will differ between -0.012 and + 0.015 g/210 L, and that the differences will be in a 0.027 g/210 L zone. When it is recognized that the DOT "Standard for Devices to Measure Breath Alcohol" permits individual 10% accuracy deviations at BrACs of 0.05 g/210 L for each quantitative evidential device (and 5% accuracy deviations at higher BrACs), it is evident in at least 68.3% of all paired tests, there will be no difference greater than the total of the two individual accuracy tolerances for quantitative evidential breath-alcohol analyzers. From the data in Table 21, it can in fact be predicted that the differences will not exceed the maximum allowable DOT total accuracy deviations at BrAC of 0.10 g/210 L for two separate alcohol analyzers (i.e., 2 x 0.005 = 0.10 g/210 L) in 81.3% of all expectible results. The data in Table 21 and Figure 6 also show that the most frequently found BrAC differences in this study lie between -0.005 and +0.005 g/210 L and that there is no marked kurtosis or skewing of the differences distribution curve, which centers about zero.

Subsets of the differences data are given in Tables 22-25 for the 0.08 and 0.10 g/210·L BrAC points which are critical decision points in some jurisdictions. Because the data base in these tables is different from that in Table 20, the statistical summaries are also slightly different, but comparable, as are the statistical projections of likely differences.

In-Vivo Results of Paired Breath-Alcohol Analyses, Reported to Two Decimal Places.

In law enforcement practice, BrACs are universally reported to two decimal places, truncated, i.e., the third-decimal digit is dropped. We therefore converted the results of the in-vivo studies to the same 2-decimal, truncated form (Tables 26-37). The resulting data speak for themselves; the slight differences in statistical summaries from those in the 3-digit form are attributable to recalculations with the new 2-digit data bases. For many purposes, these simpler-looking data compilations are more useful.

Conclusions

From the data and findings of this study, we have reached the following conclusions:

1) Breath-alcohol analysis with intervening alcohol sorption on molecular sieve 13X is a valid and practical method for delayed breath-alcohol analysis for traffic law enforcement, other forensic, field survey, and research applications

2) The analytical characteristics of this method, when it is combined with subsequent gas chromatographic analysis of alcohol, are such that the method can meet or exceed all applicable portions of the U. S. Department of Transportation "Standard for Devices to Measure Breath Alcohol" (34 FR 30459-30463, 5 Nov 73) relating to quantitative evidential breath-alcohol analysis

3) Measurement of breath-alcohol with intervening m/s sorption will yield uniformly negative results in alcohol-free human subjects

4) Breath specimen collection by in-series sampling is preferable to in-parallel arrangements for the devices and breath volumes employed in this study

5) For breath specimens divided by in-series sampling, the results of paired breath-alcohol measurements by means of the Mark IV Gas Chromatographic Intoximeter and molecular 13X sorption will correlate closely in expert hands

6) The results of breath-alcohol analysis with intervening molecular sieve 13X sorption can be validly compared with those yielded by other acceptable quantitative evidential breath-alcohol analysis methods or devices

7) The delayed breath-alcohol analysis method with intervening molecular sieve 13X sorption is capable of measuring the original alcohol concentration of vapor-alcohol specimens stored for periods of up to at least 11 months after initial collection

8) The delayed breath-alcohol analysis method with intervening molecular sieve 13X sorption is capable of and suitable for routine use in the field for traffic law enforcement and similar applications

9) The method is capable of being employed in conjunction with any presently marketed quantitative evidential breath-alcohol analysis device approved by the U. S. Department of Transportation, with only minor individual modifications.

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APPENDIX A: DIRECTIONS FOR FIELD USE OF THE BREATH COLLECTION DEVICE

The Breath Collection Device (BCD) is a simple single-use cartridge for collecting and storing the alcohol content of a fixed-volume breath sample for subsequent laboratory analysis, usually in another location. It contains 0.4 gram of molecular sieve 13X, a material which will retain the alcohol content of a breath sample passed through the cartridge. Among its primary uses are simple and rapid, anonymous collection of breath-alcohol samples at the roadside for survey purposes; and independent verification of an evidential breath-alcohol test result through collection and later analysis of either a portion of the officially-tested breath sample, or an additional breath sample collected immediately before or after an official test.

For use in association with a Model 900-A Breathalyzer[6]:

1) Complete the evidential breath-alcohol test as usual, and purge the Breathalyzer with room air.

2) Attach a fresh mouthpiece-saliva trap to the Breathalyzer inlet tube, and remove the rubber sleeve and bubbler tube from the delivery tube.

3) Collect the last portion of a complete breath exhalation from the test subject by having the subject exhale completely into the Breathalyzer inlet tube with the Breathalyzer control valve in the TAKE position. Verify that the green "PISTON UP" (Full) signal light is on.

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[6] These instructions were drafted for BCD use in conjunction with a Breathalyzer, as an example, since that is the most commonly employed evidential breath-alcohol analyzer. Comparable instructions, with only minor changes, would apply to use with other devices.

4) Attach the 20 mm (3/4 inch) long red silicone rubber sleeve accompanying the BCD to the breath delivery tube of the Breathalyzer (instead of the rubber sleeve and bubbler tube normally used). Insert the end of the BCD marked with the red circle into the silicone rubber sleeve to the red circle mark.

5) Deliver the breath specimen through the BCD by turning the Breathalyzer control valve to the ANALYZE position, and allow the breath to pass through the BCD until the Red "PISTON DOWN" (Empty) signal light illuminates.

6) Remove the BCD and discard the silicone rubber sleeve. Seal the BCD in its storage vial, and identify the content by completing the required information on the storage vial label.

7) Process the sealed BCD unit in accordance with the applicable departmental directives.