Letters to the Editor

Robert E. Peary's Polar Journal

Sir:

The paper "90° North?—Examination of Robert E. Peary's Polar Journal No. 1, 1909" by Arthur T. Anthony in the September 1991 issue of the *Journal* provides us with a document-examiner's fresh insight into an 80-year-old question: did Peary ever reach the North Pole? The author's interpretation of data surrounding Peary's journal is noteworthy, but casual readers may be misled by the author when he questions the Navigation Foundation's interpretation of data obtained by means of close-range photogrammetry.

The author's misunderstanding of the accuracy of the photogrammetric data is understandable through his later admission that he had not read the full report [*1*] issued by the Navigation Foundation, which formed the basis for the *National Geographic* article [*2*] published in January 1990.

The photogrammetric analyses performed by The Navigation Foundation and reported in the above references were based upon measurements of shadows produced by the sun in a total of 13 photographs in which the sun was outside of the camera's field of view. This photographic evidence is very powerful in establishing the distance the photographer was away from the Pole when each image was exposed if there are several well-placed shadows in each photograph, the horizon line is visible and the approximate time each picture was made are available to the analyst. (Only *approximate* time estimates are required at or near the North Pole in April because the elevation of the sun varies only 22 min of arc over a 24-h period.)

With regard to the application of this photogrammetric method to the Peary photographs, the author is quite correct on one count: the underlying technique is sound. After suggesting the application of this shadow method to Adm. Davies of The Navigation Foundation as an objective means of testing Peary's claim, I conducted a series of experiments under controlled conditions to assess the accuracy of the basic technique. It was found to be accurate to within 6 min of arc in determining the sun's elevation above the horizon, as reported in the *National Geographic* [*3*]. This corresponds to 6 nautical miles on the earth's surface.

Unless the author had direct access to Peary's negatives and the photographic enlargements made from them by National Geographic photographic laboratory personnel, it is presuming of him to classify them as "poor-quality prints and negatives." More importantly, it is presumptuous of the author to impugn the validity of the photogrammetric results because he disagrees with the Navigation Foundation's speculation on a matter totally unrelated to the photogrammetric analysis of the Peary photographs; namely, the Foundation's theory that Peary may have carried more than one notebook. This theory was offered by the Foundation to explain the "anecdotal evidence" of the loose notebook page inscribed *Pole at last!!!* in Peary's journal.

On 23 February 1990 at the Academy's 42nd annual meeting in Cincinnati, I presented fresh photogrammetric evidence based upon two newly discovered Peary photographs in which the sun and horizon are both clearly visible. The discovery of these photographs then made it possible to measure the sun's elevation directly, as opposed to indirectly by the shadow method. This new information was presented publicly for the first time as part of my oral presentation entitled "Gleaning the Maximum Information from Evidentiary Photographs." It was pointed out in this presentation that measurements I personally performed on these two photographs confirmed the earlier conclusions reported by The Navigation Foundation. The elevation of the sun at the North Pole at 10 P.M. on April 6, 1909, when these photographs were reported by Peary to have been

exposed, was 6° 42′ of arc. My measurements from the two photographs were 6° 41′ and 6° 49′ respectively, with an average value of 6° 45′. The 3′ difference between this average value and the sun's true elevation at the North Pole corresponds to 3 nautical miles of ground distance from the Pole. The results of my analyses were included in a supplemental report [4] issued by The Navigation Foundation two months later in April 1990.

Peary's claim to the North Pole continues to be challenged; and rightly so, as new and important evidence is brought to light. But it is wrong to condemn the close-range photogrammetric evidence for reasons unrelated to photogrammetry or because the magazine half-tone reproductions of Peary's photographs appear to be of poor quality. The most recent evidence based upon my independent photogrammetric analyses clearly supports The Navigation Foundation's findings and Peary's claim.

References

[1] Davies, E. E., Editor, *Robert E. Peary At The North Pole*, The Foundation for the Promotion of the Art of Navigation, Rockville, MD, 11 December 1989, 181 pages plus appendices.
[2] Davies, T. E., "New Evidence Places Peary At The Pole," *National Geographic*, Vol. 177, No. 1, January 1990, pp. 44–61.
[3] Garrett, Wilbur E., *National Geographic*, Vol. 177, No. 1, January 1990, Editor's page.
[4] Davies, Thomas E., *Robert E. Peary At The North Pole, A Report to the National Geographic Society by The Foundation for the Promotion of the Art of Navigation, Supplemental Report*, 16 April 1990.

William G. Hyzer PE,
Certified Photogrammetrist
136 South Garfield Avenue
Janesville, WI 53545

Author's Response

Dear Sir:

I read with interest William G. Hyzer's letter concerning my Last Word Society article (Journal Forensic Sciences).

In an attempt to make a Last Word Society article of interest to the general AAFS membership, I felt the necessity to offer some historical background, personal speculations, as well as the views of an author and an author/explorer who recently commented on the so called "anecdotal evidence."

It seems that clarification of several points made by Mr. Hyzer are necessary. First, I never asserted that the thrust of my article was an attempt to establish whether Robert E. Peary reached the North Pole; my goals and objectives are clearly outlined in the abstract. Additionally, it was never my intention to mislead anyone, even "casual readers."

Mr. Hyzer states "the author is quite correct on one count: the underlying technique is sound." I would like to point out that I actually wrote, "I do not question the techniques," in reference to the close-range photogrammetry. I am not qualified to question the principles of such an esoteric field.

Additionally, Mr. Hyzer claims the quality of prints reproduced in the *National Geographic* [1] are possible due to "half-tone reproductions of Peary's photographs." I am certain that it was not the intention of the *National Geographic* to mislead. However, Mr. Hyzer must admit the quality of the photographic reproductions are somewhat deceiving, especially the two page reproduction depicting Peary's 1906 expedition.

A caption states, in part, "photogrammetric analysis" confirms Peary's "farthest north" claim. There is no horizon visible and no visible shadows, two of the essential criteria Mr. Hyzer states are necessary in applying his method.

Because of the foregoing, I did not find the photographic evidence convincing. *National Geographic* did a disservice to its readers for allowing such poor reproductions to be published.

I mentioned my personal opinion regarding the above, in response to Rear Admiral Thomas D. Davies', comments about the so-called "anecdotal evidence," "These are not matters that a foundation such as ours is designed to examine, belonging to historians or psychologists rather than navigators" [1]. The Foundation ventured outside their area of expertise in commenting about the documentary evidence involving the "Pole at last!!!" loose sheet in the 1909 Journal. Therefore, it seemed appropriate for me to comment about the photogrammetric analysis conclusions that I did not find convincing.

I would encourage Mr. Hyzer to publish his initial interpretation of data, as well as, the data based on "newly discovered Peary photographs in which the sun and horizon are both clearly visible" in a scientific journal such as the *Journal of Forensic Sciences*.

Arthur T. Anthony
Certified Forensic Document Examiner
P.O. Box 620400
Atlanta, GA 30362

Reference

[1] "New Evidence Places Peary at the Pole," *National Geographic*, Vol. 177, No. 1, Jan. 1990.

Remains Identification by Frontal Sinus Radiographs

Dear Sir:

The article in the November 1991 issue of the Journal of Forensic Sciences by Marlin, Clark, and Standish (Identification of Human Remains by Comparison of Frontal Sinus Radiographs: A Series of Four Cases; Journal of Forensic Sciences 1991;36(6):1765–1772) describes techniques of skeletal identification that I had assumed to be quite well-known and frequently used throughout the forensic pathologic and anthropologic communities. I was distressed that the references did not cite what is probably the most extensive modern frontal sinus pattern analysis for identification purposes, that of Douglas Ubelaker, who compared 35 crania with a total of 595 comparison points to establish that the sinus patterns in no two crania were alike (Positive Identification from the Radiographic Comparison of Frontal Sinus Patterns, in *Human Identification*, edited by Rathbun and Buikstra, Charles C Thomas, publisher, 1984).

Kris Sperry, M.D.
Associate Medical Examiner
Fulton County
50 Coca Cola Place, S.E.
Atlanta, GA 30303

Author's Response

Dear Sir:

Dr. Sperry's statement regarding Douglas Ubelaker's chapter in *Human Identification*, edited by Rathbun and Buikstra, Charles C Thomas, publisher, 1984, is entirely correct.

I was unaware of this reference at the time our article was written. I have since reviewed it and agree it should have been cited in the references.

Donnell C. Marlin, D.D.S.
Assistant Professor
Indiana University
School of Dentistry
Dental Diagnostic Sciences
1121 W. Michigan St.
Indianapolis, IN 46202-5186

Remains Identification by Frontal Sinus Radiographs

Dear Sir:

In their article (Identification of Human Remains by Comparison of Frontal Sinus Radiographs: A Series of Four Cases, Vol. 36, No. 6, November 1991, pp. 1765–1772) Marlin et al. omitted from their references a most relevant book: N. Asherson, "Identification by Frontal Sinus Prints: A Forensic Medical Pilot Survey," H. K. Lewis & Company, LTD, London, 1965.

Asherson examined 74 sets of twins, both identical and nonidentical, and verified that the frontal sinus pattern was discordant in all individuals over the age of 14. Even with identical twins, the frontal sinus pattern was different as the twins developed from birth through the age 14. "Of the 74 twins in the series examined in children, there was no identity between any of them. Each displayed the same random development exhibited in the remainder of cases examined, and in the general population."

We have recently had a difficult case of identification and have attempted to make positive comparison between antemortem and postmortem computerized tomography (CT) scans. This was made even more difficult by the absence of soft tissue in the postmortem specimens. With increased reliance on CT scans rather than plain films of the skull, I believe that this is a problem to be addressed by radiology and forensic pathology. Is there anyone out there with enough money and time to still do research?

Harry J. Bonnell, M.D.
Chief Deputy Medical Examiner
5555 Overland Ave. Bldg. 14
San Diego, CA 92123

Author's Response

Dear Sir:

As you may recall, our original manuscript for the article "Identification of Human Remains by Comparison of Frontal Sinus Radiographs: A Series of Four Cases," Donnell C. Marlin, et al., *Journal of Forensic Sciences 36(6):1765–1772*, was much longer and was edited to the final published version. The Asherson book was referenced in the original version; this reference and several others did not appear in the final version. I agree with Dr. Bonnell that it is good material in this area.

Donnell C. Marlin, D.D.S.
Assistant Professor
Indiana University
School of Dentistry
Dental Diagnostic Sciences
1121 W. Michigan St.
Indianapolis, IN 46202-5186

Remains Identification by Frontal Sinus Radiographs

Dear Sir:

I would like to call attention to the statement contained in the abstract and discussion of the article by Donnell C. Marlin and coauthors titled "Identification of Human Remains by Comparison of Frontal Sinus Radiographs: A Series of Four Cases," that appeared in the *Journal of Forensic Sciences*, (Vol. 36, No. 6, Nov. 1991, pp. 1765–1772). The authors state that the frontal sinus outline remains constant throughout an individual's lifetime. This is erroneous and misleading. To quote from a reference cited as reference 8 by the above authors:

> "The frontal sinuses are absent at birth, but begin to pneumatize at approximately the second year of life. Pneumatization progresses slowly through the sixth to eight year, at which time the chambers are fairly well developed. Over the next several years, until puberty, the frontal sinuses continue to grow and may change considerably in their configuration, reaching adult proportions only after puberty [*1*]."

At the other extreme of life, with advancing age, absorption of bone from the inner walls of the sinuses may occur as an atrophic change leading to further enlargement [*2*,*3*].

With these developments in mind, it is well for investigators to take into consideration the caution that when individuals are younger than 18 years of age some normal growth changes may have occurred between the dates the radiographs were taken [*4*]. The same caution should be observed when postmortem radiographs of individuals of advanced age are compared with antemortem radiographs taken at earlier ages.

William D. Haglund, Ph.D.
King County Medical Examiner's Office
325 9th Avenue
Seattle, WA 98104

References

[*1*] Messmer, J. M. and Fierro, M. F., "Personal Identification by Radiographic Comparison of Vascular Groove Patterns of the Calvarium, "*The American Journal of Forensic Medicine and Pathology*," Vol. 7, No. 2, June 1986, pp. 159–162.

[*2*] Hauser, G. and De Stephano, G. F., *Epigenetic Variants of the Human Skull*, Schweizerbart'che Verlagsbuchhandlung, Stutgart, 1989, p. 31.

[*3*] Ubelaker, D. H., "Positive Identification from the Radiographic Comparison of Frontal Sinus Patterns," *Human Identification*, T. A. Rathbun and J. Buikstra, Eds., Charles C Thomas, Springfield, IL, 1984, pp. 399–411.

[*4*] Warwick, R. and Williams, P. L., *Gray's Anatomy*, 35th British Edition, W. B. Saunders, Philadelphia, PA, 1973, p. 299.

Author's Response

Dear Sir:

We have reviewed the cautions cited by Dr. Haglund and agree that they should be taken into account. It should be remembered that the original manuscript was of much greater length and contained some of these statements.

Donnell C. Marlin, D.D.S.
Assistant Professor
Indiana University
School of Dentistry
Dental Diagnostic Sciences
1121 W. Michigan St.
Indianapolis, IN 46202-5186

Determining Total Method Level of Detection and Level of Quantitation for Breath Alcohol Analysis Programs

Dear Sir:

The importance of establishing the limit of detection (LOD) and limit of quantitation (LOQ) for the total analytical method was recently emphasized by Jones [*1*]. The same type of analysis can be performed with duplicate breath alcohol data and is the focus of the present letter.

When ethanol exists in the matrix of human breath the question of distinguishing between true ethanol and measurement "noise" becomes important at very low concentrations. The LOD is that breath alcohol concentration (BrAC) at which one can conclude with some statistical confidence that it resulted from the analyte (alcohol) being present and not just due to a high blank value. A distribution of blank (no analyte present) values theoretically exists and it is the standard deviation (S_0) of this distribution that is important [*2*]. The LOD is typically estimated then from some multiple (usually 3) of the standard deviation (S_0) of blank values [*3*]. S_0 is usually estimated from the intercept of the linear model that regresses standard deviation upon concentration values. S_0 then becomes an estimate of the standard deviation of blank values. The LOQ is then typically estimated to be 10 S_0 and is the level at which quantifiable results should be reported [*3*].

Determining LOD and LOQ are best accomplished by considering the total analytic method, which includes the multiple instruments used, actual field operators, arrested subjects, between-day intervals, etc. This provides the best estimate from actual conditions under which the total measurement algorithm is expected to perform. The same computational approach can be performed, however, using simulators and thus evaluating the analytical LOD and LOQ only which is also informative. In this approach the standard deviations of replicate analyses are plotted against their means throughout the range (i.e. 0.01 to 0.40 g/210 L) with appropriate regression analysis.

Duplicate breath alcohol data (n = 15 493) were selected from the BAC Verifier DataMaster instrument employed in the State of Washington. Only cases where both the first and second sample were ≥ 0.01 g/210 L were selected. The data was received from 173 different DataMaster instruments from April 1, 1991 through August 31, 1991. The duplicate results were truncated to three decimal places with a resulting mean also computed to three decimal places and reported as g/210 L. The means were then grouped into 0.01 g/210 L intervals and ranged from 0.01 to 0.34 g/210 L. Within each interval the standard deviation of a single determination (SD_i) was determined according to: $SD_i = \sqrt{\Sigma d^2/2n}$, where d = difference between duplicates and n = number of duplicate pairs for that interval [*4,5*]. This method was the same used by Jones [*1*] based on difference sum of squares.

The resulting SD_i were then plotted against the concentration interval as seen in Fig. 1. One observes the SD_i computed for the 0.34 g/210 L interval is quite large, which is partially explained by the smaller n (n = 18) at that concentration. This may explain why some higher concentration values deviate from linearity as also seen by Jones [*1*]. Figure 1 also shows the best fit line resulting from simple linear regression. The regression analysis was performed using SPSS/PC + (SPSS Inc., Chicago) and generated the regression coefficient (slope) along with the intercept term. From Fig. 1 we see the intercept term is 0.0025 g/210 L with a standard error (SE) of 0.000 28 g/210 L. From this determination of S_0 we would estimate LOD = 0.0075 g/210 L and LOQ = 0.025 g/210 L.

Comparing the data in Fig. 1 with that of Jones we see a general linear trend throughout. Jones found his method variability (for blood alcohol analysis by gas chromatography) remained fairly constant up to around 0.08 g/210 L equivalent and thereafter increased proportional to concentration. The computed LOD and LOQ values are also considerably larger than those found by Jones and probably reflects the greater biological variability

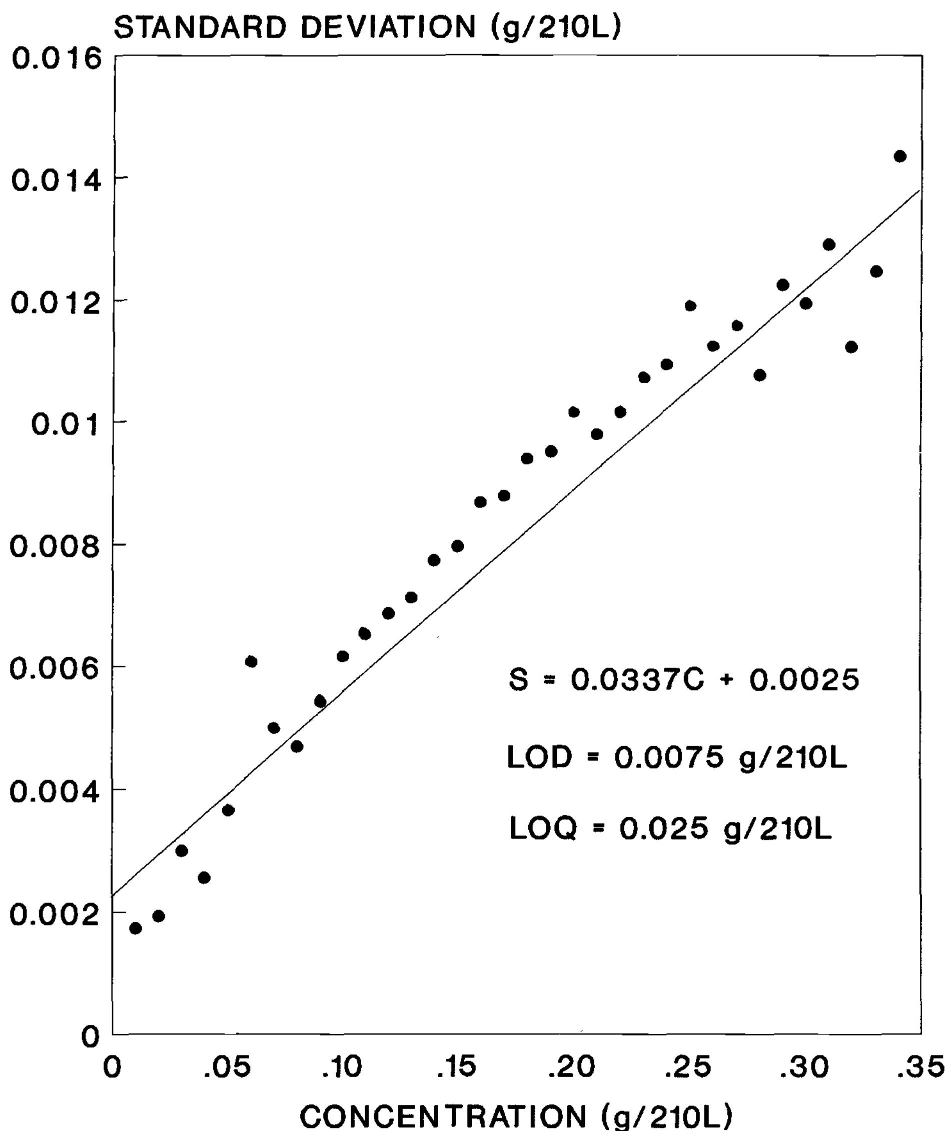

FIG. 1—*Plot of standard deviation against concentration and resulting linear regression analysis.*

found in replicate breath alcohol analyses. It is clearly evident that measurement variability is proportional to concentration in breath alcohol analysis.

One important application of this information (as noted by Jones) is in regards to decreasing "per se" legal standards. The method capabilities (LOD and LOQ) should be considered when establishing very low "per se" concentrations. Another important application is seen in establishing confidence intervals around the means of duplicate breath alcohol results. Figure 1 reveals that the variability (SD_i) is proportional to concentration and thus the standard error employed in a confidence interval calculation should employ the value that corresponds to the particular BrAC being considered. The methods described here could also be applied in comparing different agencies, instruments, protocols, etc. in order to better assess total program performance. Other juris-

dictions employing duplicate breath alcohol analyses are encouraged to compute relevant LOD and LOQ values for their particular programs as well. Finally, manufacturers of breath alcohol instruments should be familiar with these concepts and able to report their instrument's capabilities in regard to LOD and LOQ.

Rod G. Gullberg
Washington State Patrol
Breath Test Section
6431 Corson Avenue South
Seattle, WA 98108-3462

References

[1] Jones, A. W., "Limits of Detection and Quantitation of Ethanol in Specimens of Whole Blood From Drinking Drivers Analyzed by Headspace Gas Chromatography," Letter to the Editor, *Journal of Forensic Sciences*, Vol. 36, No. 4, 1991, pp. 1277–1279.
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[3] Taylor, J. K., *Quality Assurance of Chemical Measurements*, Chelsea, Michigan: Lewis Publishers, Inc., 1987, pp. 79–82.
[4] Thompson, M. and Howarth, R. J., "The Rapid Estimation and Control of Precision by Duplicate Determination," *The Analyst*, Vol. 98, No. 1164, March 1973, pp. 153–160.
[5] Bennett, C. A. and Franklin, N. L., *Statistical Analysis in Chemistry and the Chemical Industry*, New York, John Wiley and Sons, Inc., 1954, p. 654.

Author's Response

Dear Sir:

The letter from Sgt. Gullberg [1] demonstrates that the standard deviation (S) of measurements of breath-alcohol increase as a function of the concentration (C) in the breath sample. The linear regression relationship was $S = 0.0025 + 0.0337C$, and this equation was established over a concentration interval (x-variate) from 0.01 g/210 L to 0.34 g/210 L. The breath-test instrument evaluated (Datamaster) has been used for law enforcement purposes in the State of Washington for a number of years. Sgt. Gullberg suggests that one might use the relationship between standard deviation and concentration of alcohol in the breath to estimate the limit of detection (LOD) and limit of quantitation (LOQ) of the method.

Sgt. Gullberg cites my recent letter to the editor [2] that dealt with LOD and LOQ of the method used in Sweden for forensic blood-alcohol analysis using headspace gas chromatography. Indeed, the statistical technique used to determine LOD and LOQ is well established in analytical chemistry [3]. However, the total variance of a breath-alcohol measurement is made up of two components, namely, analytic variation and biologic variation. The biologic source of variation, owing to the breath-to-breath physiologic variations, dominates the total variability. Elsewhere, Sgt. Gullberg has estimated that biologic variation (the person) accounts for about 95.6% of the total variance whereas analytic variation (the instrument) accounts for the remaining 4.4% [4]. I am not certain whether the methods developed to measure LOD and LOQ in analytical chemistry are applicable when biologic variation makes such a major contribution to total variability. The biologic sources of variation swamp the true instrumental variation when measurements are made at low concentrations necessary to estimate LOD and LOQ. Moreover, the magnitude of the biological variation varies from subject to subject depending on, among other things, their breathing technique and a multitude of other factors. The variance derived from breath-tests with drunk drivers (Sgt. Gullberg's material) will not

be the same as the variance in tests with healthy subjects in controlled drinking experiments.

Sgt. Gullberg fails to make it clear whether outlying values, if any, were excluded from the material of 15 493 cases that he examined. The presence of outlying values can seriously inflate the estimated standard deviation because the absolute differences between each pair of duplicates is squared. Sgt. Gullberg suggests that instrument manufactures should report the LOD and LOQ of their product. However, if LOD and LOQ are calculated as described in his letter, the results will depend on the magnitude of breath-to-breath variation for the particular group of subjects tested. The LOD and LOQ of the method derived from controlled experiments will not necessarily be the same when the same instrument is used for law enforcement purposes.

In my letter [2], which seemed to have inspired Sgt. Gullberg to write his letter, the standard deviation of blood-alcohol analysis is not influenced by biologic sources of variation inherent in the method. However, the LOD and LOQ that I reported might depend to some extent on the specimen matrix, that is, whether water, whole blood, or plasma solutions were being analyzed. The papers on statistics cited by Gullberg were taken from the field of industrial chemistry for which the variance of the instrumental methods of analysis used are not influenced by physiologic variations. Indeed, when the SD of an analytical method is plotted against the concentration of analyte in the sample, one almost always obtains a hockey stick curve as shown in reference [2]. Moreover, because the standard deviations close to the limit of detection are important when LOD and LOQ of a method are calculated, Sgt. Gullberg might have restricted his regression analysis to the interval 0.005 to 0.05 g/210 L; then the slope and intercept of the regression equation might have been different.

If the LOD and LOQ of a breath-alcohol instrument are of concern, then I suggest making experiments in vitro, such as, by analyzing air-alcohol-vapor standards generated from a breath simulator device. The target concentrations might include levels of 0.005 g/210 L, 0.015 g/210 L, 0.025 g/210 L, 0.035 g/210 L, and 0.05 g/210 L. With this approach, it seems more realistic to compare LOD and LOQ for breath-alcohol analyzers from different manufactures or instruments using different analytical principles. I am not convinced that the results from duplicate breath tests with drunk drivers is a valid method to assess LOD and LOQ for a breath-alcohol instrument. On the other hand, the relationship between standard deviation and breath-alcohol concentration is important if a confidence interval for the analytic result in a given subject is required. The confidence band is obviously going to be wider for those subjects with higher concentrations of ethanol in their breath.

A.W. Jones, Ph.D.
Department of Alcohol Toxicology
National Laboratory of Forensic Chemistry
University Hospital
581 85 Linköping
Sweden

References

[1] Gullberg, R. G., "Determining Total Method Level of Detection and Level of Quantitation for Breath Alcohol Analysis Programs," *Journal of Forensic Sciences*, Vol. 37, No. 5, September 1992, pp. 1208–1210.

[2] Jones, A. W., "Limits of Detection and Quantitation of Ethanol in Specimens of Whole Blood from Drinking Drivers Analyzed by Headspace Gas Chromatography," *Journal of Forensic Sciences*, Vol. 36, No. 5, September 1991, pp. 1277–1279.

[3] Taylor, J. K., "Quality Assurance of Chemical Measurements," Lewis Publishers Inc., Chelsea, MI, 1987, pp. 1–328.

[4] Gullberg, R. G., "Breath Alcohol Test Precision: An in vivo vs In Vitro Evaluation," *Forensic Science International*, Vol. 43, 1989, pp. 247–255.

Fingerprint Age Determination by Fluorescence

Dear Sir:

When laser fingerprint detection began to be explored in 1976, the initial focus was on fluorescence inherent to fingerprint residue. It was noted early on that fresh fingerprints exhibit a greenish yellow fluorescence whereas old fingerprints (on the order of a year or older) show orange fluorescence. It was proposed at the time that inherent fingerprint fluorescence might offer a prospect for dating latent prints [1]. In a subsequent preliminary study [2], thin layer chromatography of fingerprint residue was performed with the aim of isolating and identifying fluorescent components of palmer sweat. Riboflavin was tentatively identified as a key fluorescer in fingerprint residue. Between 1980 and 1982, I undertook a comprehensive fingerprint aging study, utilizing fluorescence spectroscopy as the technique via which to attempt dating latent prints. The outcome of this study was negative. The results of the study were reported to the sponsoring agency (National Science Foundation) and at a conference of the International Association for Identification, but not in the forensic science literature. On retrospect, this is a regrettable oversight because forensic scientists unaware of this study might embark on similar (very time consuming) ones, to no avail. I report here on my earlier study in hopes of preventing this. Indeed, I very recently received (courtesy of M. Carrick, Lightning Powder Co., Inc.) a copy of an article (in Russian) by M. E. Dzakishev (Ministry of Interior, Moscow, Russia) which describes an effort very much along the lines of my earlier one, that is, the prospects for wasted research efforts are real. Dzakishev, incidentally, independently arrived at the conclusion reached by us in 1978 that riboflavin is a key fluorescent component of fingerprint residue [3]. Although it is recognized from the outset that environmental variations (presence or absence of ambient light, temperature, humidity) as well as contaminants on fingers may well pose insurmountable obstacles to the dating of latent prints, fingerprint aging studies (by whatever means) are worthwhile, even if high risk, given the potential value of fingerprint age determination in criminal investigation.

Collection of Fingerprint Samples

Fingerprints were collected in the fall of 1980 from some 50 individuals (Texas Tech University students). About 10 prints were taken from each student. Since in these samples no control could be exerted over finger contamination, a second set of fingerprints was collected from five individuals (students in my laboratory), 10 prints from each, after careful washing of the hands and subsequent waiting (without touching anything) to allow build-up of perspiration on fingers. Finally, fingerprints were collected from these individuals on different days after washing/waiting with the aim of determining what fingerprint fluorescence variations might exist as a result of dietary variations, etc. All prints were placed on glass microscope slides. This substrate was chosen because it exhibits virtually no fluorescence that might interfere with the fingerprint fluorescence of interest.

In order to minimize needless complications in the initial phase of the study, the collected samples were stored in covered boxes (absence of ambient light) in an air conditioned laboratory (reasonably uniform conditions of temperature and humidity).

Spectral Measurements

Fingerprint fluorescence was excited by an argon-ion laser (Spectra Physics 164-05) operating in the all lines blue-green mode at a power of a few tens of milliwatts, with the beam expanded to cover an area of about 0.5 cm^2. The fluorescence was pre-filtered with an orange long-wavelength-pass glass filter (Ealing, OG 550), which essentially is

a standard argon-ion laser safety filter, and was focused onto a 0.3 m monochromator (McPherson 218 equipped with a 1200 lines/mm grating blazed at 500 nm). The fluorescence was detected with a photomultiplier tube (EMI/9785B), processed by photon counting instrumentation (PAR 1120 amplifier/discriminator and 1112 photon counter/processor), and displayed on a chart recorder. Since the fluorescence of fingerprints is broad and featureless, the monitored quantity was the fluorescence maximum. The fluorescence maximum ranges from about 550 nm (fresh prints) to about 580 nm (old prints). Fingerprint spectra were recorded in one-month-intervals over a period of some 18 months. In each measurement, a fingerprint region not previously subjected to laser illumination, or a previously unexposed print, were selected in order to avoid problems associated with photodecomposition (which does occur in fingerprints exposed to laser light).

Results

The earlier noted red-shift of the fluorescence of old prints vs. fresh prints was found, quite reproducibly. However, there were pronounced variations in the initial fluorescence maxima as well as the rates of red-shifts with time, not only from individual to individual (set of 50 prints and also set of 5 prints deposited after washing/waiting), but also for those prints collected from a particular individual on different dates after washing/waiting. These variations were too large to premit any quantitative conclusions.

The project was terminated as a failure.

E. R. Menzel, Ph.D.
Professor of Physics and Chemistry,
and Director,
Center for Forensic Studies,
Texas Tech University, Lubbock, TX 79409-1051.

References

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[2] Duff, J. M. and Menzel, E. R., "Laser-Assisted Thin-Layer Chromatography and Luminescence of Fingerprints: An Approach to Fingerprint Age Determination," *Journal of Forensic Sciences*, Vol. 23, No. 1, Jan. 1978, pp. 129–134.
[3] Dzakishev, M. E., "Photochemical Reactions in Perspiration—Fat Substance. A Method of Fingerprint Age Determination," (translated from Russian). This article is a preprint. It is not known to me whether is has been published in the Russian literature. It describes the initial phase of a study aimed at fingerprint age determination, not a completed study.

Determination of the Concentration of Tetramethylenedisulfotetramine in Human Blood by GC/FPD

Dear Sir:

On May 22, 1991, seven people were poisoned after eating lunch in their company cafeteria. The soup had accidentally contained the rodenticide tetramethylenedisulfotetramine (TDT). Five victims were brought to the First Teaching Hospital, affiliated with the China Medical University, 4 h later. We detected the blood concentration of the TDT by using gas chromatography (GC) with a flame photometric detector (FPD) and obtained significant results.

A SHIMADA4.ZU GC-9A gas chromatograph connected with a CR-3A integrator and column (10% SE-30, 3.1 m × 3 mm, chromosorb W AW DMCS) were used in our

experiments. The conditions were: column temperature 240°C; nitrogen flow rate 40 mL/min.

To quantitate the blood concentration, pure TDT, provided by the Health and Epidemic Prevention Station of Shenyang, China, was dissolved in acetone solution at 200 μg/mL. This solution of 0.1 mL was diluted continually by acetone in multiplication and a 2 μL aliquot of each concentration was injected onto the GC, respectively. Thus, the concentration curve of chromotographic peak height vs. drug concentration was obtained for quantitative analysis.

Two mL of the blood were extracted with 6 mL benzene by shaking a vibrator for 10 min and centrifuged (3500 rpm, 10 min) to obtain a clear supernatant. This procedure was repeated once. The combined supernatant, about 11 mL, was put into a glass syringe, which was connected to a PT-series cartridge of neutral aluminum oxide (Jingyang Filter Material Plant of Hebei Province, China, content: 1 g), and passed through the cartridge at a flow rate not greater than 5 mL/min. After being dried under the stream of nitrogen at room temperature, the residue was dissolved in 100 μL acetone and 2 μL aliquot was subject to GC analysis.

The test recovery of the described extraction method, 50 μL and 25 uL of the standard solution (containing the 10 μg and 5 μg of the TDT) were added to 2 mL blood from healthy subjects. (This extraction and clean-up procedure was the same as previously described.) Then, each was injected onto the GC five times. After its peak height was compared with the concentration curve, a recovery of 94.6% was obtained (n = 10, SD = 0.017).

Using this method, the TDT blood concentrations of five patients were 0.64 μg/mL; 0.18 μg/mL; 0.15 μg/mL; 0.10 μg/mL; and 0.10 μg/mL, respectively. Figure 1 shows the gas chromatogram for the TDT (7.1 min) in the blood of one case (0.64 μg/mL). The other peaks were not identified as they are supposed to be some endogenous constituents in the blood and some medicine. The detection limit for TDT was 0.05 ng in 2 μL injection volume under these conditions.

It has been reported that benzene is better than acetone as an extraction solvent for TDT [*1*]. We came to the same conclusion by comparing the two reagents. As acetone is soluble in water, it brought more impurities from the blood samples and prolonged evaporation time.

The PT-series cartridge is the only one commercially available in China for solid-phase extraction. The neutral aluminum oxide used in these experiments possessed evident effect for adsorbing the chromatic impurities and the drug was not lost by this procedure.

In the victims we treated, two mildly poisoned patients (0.10 μg/mL and 0.10 μg/mL concentrations of the TDT) showed only general poisoning symptoms such as lighthead-

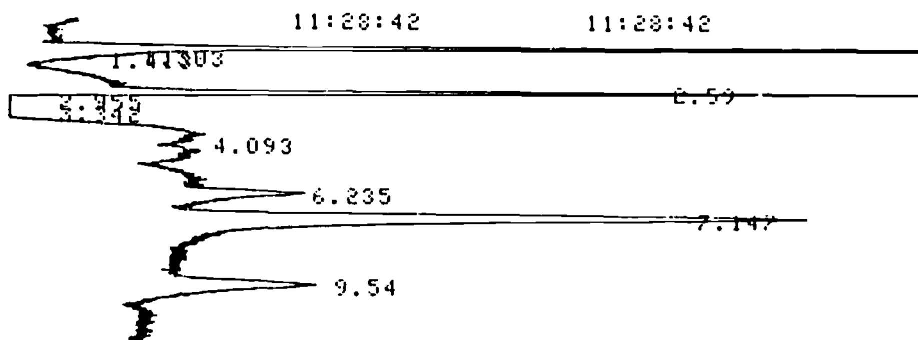

FIG. 1—*The gas chromatogram for tetramethylenedisulfotetramine (7.1 min) in the poisoned blood.*

edness, faintness, nausea, and vomiting; two patients (0.18 μg/mL and 0.15 μg/mL blood concentration of the TDT) showed convulsions and the other one showed tonic convulsions and deep coma as typical symptoms. The three patients whose blood concentrations were higher than 0.15 μg/mL had bleeding symptoms such as vomitus cruentus, nosebleed, melena, hematuria, and subcutaneous hemorrhage. However, in another case in which a 35-year-old woman attempted suicide by ingesting a large amount of TDT-containing rodenticide, no bleeding symptoms were observed. Therefore, we do not know whether the bleeding was caused by the TDT. This needs further research.

Although we have not found data on the lethal concentrations of TDT in human blood, it was reported that the blood concentration was 0.99 μg/mL in a fatal poisoning case [*1*]. In our cases, each individual's concentration was lower than 0.99 μg/mL. They were all out of danger.

Early reports on the toxicity of TDT injected i.p. into mice indicated that it was 5 to 10 times more toxic than strychnine. After local application to a rat's cerebral cortex, its activity was 70 times greater than strychnine [*2*]. As its LD_{50} is very low, we did not find TDT in the blood of the individuals with GC/FID. GC/FTD of GC/FPD was necessary to detect the very small amount of TDT in blood from the poisoning victims.

Tetramethylenedisulfotetramine is an extraordinarily effective rodenticide, but its use is limited because it is toxic to people [*3*]. According to the Merck Index, it is only an experimental poison and is rarely introduced in many pesticide manuals. However, many commercial rodenticides presently use TDT. Some rodenticides are prepared by adulterating the original agent with starch, thus leaving no color. The analysts of clinical and forensic chemistry will be watching anxiously for other poisoning incidents due to TDT.

Wu Guohua
Emergency Ward of The 1st Teaching
Hospital Affiliated to China Medical
University, Shenyang 110001
P.R. China

Huang Meiyi
Beijing Neurosurgical Institute
Beijing 100050
P.R. China

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