Technical Papers

One-Step Preparation of C^{14}-Cyanide from Barium Carbonate-C^{14}

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The importance of cyanide in the synthesis of organic compounds labeled with isotopic carbon requires that it be made available in high yields by simple and inexpensive procedures starting with barium carbonate. Methods (*1–5*) have been described for the conversion of alkali carbonates to cyanide but each has its own limitation. A procedure has been developed in this laboratory based on the findings of Hood and Salamon (*6*) and J. A. McCarter (*7*) whereby barium carbonate can be converted to sodium cyanide in one step. This was accomplished by heating barium carbonate, zinc dust, and metallic sodium in a stream of anhydrous ammonia which had passed over hot iron. Iron seemed to have an important role in that it catalyzed the decomposition of ammonia to give nitrogen gas and other "active" forms of nitrogen which were essential in the reaction. The yields in this laboratory were quantitative as described in the following experimental procedure.

Powdered anhydrous C^{14} barium carbonate (1.0 millimole) was mixed with 1.0 g of zinc dust and 0.2000 g of metallic sodium in pea size chunks, and this mixture was transferred to a porcelain combustion boat of suitable size. The boat was placed in a Vycor combustion tube (600 mm in length and 19 mm inside diameter) in an atmosphere of anhydrous ammonia and containing a 5.0-g ball of iron wire or 5.0 g of powdered iron distributed throughout a plug of Pyrex glass wool occupying the mid-portion of the tube. The boat was pushed into the tube until it touched, or almost touched, the iron plug.

The end of the tube nearest the iron was connected to a cylinder of anhydrous ammonia. A stream of ammonia was allowed to flow through a gas bubbler at the rate of 3 bubbles/second in and through the tube containing the mixture. The portion of the tube containing the mixture and iron was brought to and maintained at approximately 650° C for a period of 4 hr. The flow of ammonia was continued until the tube was cool.

The boat and all contents of the tube, except the iron ball, were washed into a 250-ml flask attached to a Kjeldahl distillation head for subsequent distillation of hydrogen cyanide. The solution in the flask was acidified with dilute (2 *N*) sulfuric acid and 20–30 ml of distillate were collected in an Erlenmeyer flask containing a 20% excess of the theortical amount of 1 *N* sodium or potassium hydroxide.

Analysis by the argentimetric method (*8*) of the distillate obtained in three experiments showed a yield of cyanide which was quantitative. The specific activity of the labeled cyanide was unchanged from that of the barium carbonate used as the starting material.

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Manuscript received October 8, 1953.

Evaluation of Oral Temperature Readings

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Many studies have dealt with the variability of body temperature as reflected in inter-individual differences, day-to-day changes, diurnal variations, and other comparisons (*1*). Such studies, while yielding considerable information as to the extent of variability to be expected under different conditions, have failed to provide any systematic method for evaluating the significance of a given temperature reading, or a difference between two readings taken under specified circumstances. One possible solution to this problem is presented in the following paragraphs; it involves an approach which has been used in connection with a similar question regarding metabolism measurements (*2*).

The data for this analysis were taken from 29 ostensibly normal male university students, ranging in age from 17 to 27 years. Twelve readings of oral temperature were made upon each subject, the complete series consisting of records taken at 8:00 A.M. (before breakfast), 12:00 M., 6:00 P.M., and 10:00 P.M. on each of 3 different days, approximately a week apart. Only 1 subject was observed on any given day. Every session was preceded by a 30-min rest, the subject lying upon a cot throughout this period and during the actual observation of his temperature. All readings were made with the same thermometer, a MICH 3 Oral (Serial J 1273).[1] The thermometer always was inserted under the subject's tongue and left in place a full 3 min.

The 348 temperature readings thus obtained ranged from 95.4° to 99.1°, and averaged 98.0° F, a value very close to Ivy's mean of 98.1° for his 276 medical students, tested in class between 8:00 and 9.00 A.M. (*3*). Individual means for our 29 subjects ranged

[1] Manufactured by E. Kessling, New York, N. Y.

TABLE 1

SIGNIFICANT VARIATIONS IN ORAL TEMPERATURE READINGS UNDER SPECIFIED CONDITIONS

Measure evaluated	Source of error estimate	Degrees of freedom	Components of error estimate	Significant variation (°F)	
				5%	1%
Any single reading in comparison with some hypothetical value, e.g., "normal temperature"	S × H × D	168	σ^2 (Sampling error: random trial-to-trial variations of readings after eliminating major sources of variability)	0.5	0.7
Difference between any two single readings	S × H × D	168	Same as above	0.7	0.9
Comparable readings made upon two individuals at same time of day; or two readings made upon one individual at same hour on different days	S × D	56	$\sigma^2 + 4\sigma^2_{m(s \times d)}$ (Sampling error plus variation among 87 means representing 4 observations each)	1.2	1.5
Two readings made upon one individual at different hours, either on same day or on different days	H × D	6	$\sigma^2 + 29\sigma^2_{m(h \times d)}$ (Sampling error plus variation among 12 means representing 29 observations each)	1.6	2.4

from 97.2° to 98.5°; the 4 hourly means ran from a low of 97.5° at 8:00 A.M. to a high of 98.2° at 6:00 P.M., while the 3 daily means varied by less than 0.1°.

A conventional analysis of variance yielded mean squares significant at the 1% level or beyond for "between subjects," "between hours," and 3 interactions, "subjects × hours," "subjects × days," and "hours × days." Significance of the first-order interactions was established by testing them against the residual variance or "subjects × hours × days" interaction; the main variables, "subjects" and "hours," were evaluated in terms of the appropriate first-order interactions. Furthermore, a series of Bartlett tests, applied to different groupings of the original data, justified certain assumptions concerning homogeneity of variance which would permit the use of all the interactions except, possibly, "subjects × hours" in evaluating individual temperature readings. The latter is suspect for this purpose, because the variance "within hours," estimated from the 29 means of 3 daily readings each, appeared to differ significantly from one hour to the next.

Standard deviations representing the several sources of variation may be obtained by extracting the square roots of the mean squares resulting from the analysis of variance. Those derived from the 3 usable interactions were employed to establish the magnitudes of significant variations at the 5% and the 1% levels of confidence for a number of comparisons frequently made in practical work (*4*). These values are presented in Table 1, together with suggestions for their appropriate use and information concerning the components of the error estimate in each case (*5*). If, for example, any two readings differ by 0.9° F or more, the chances are 99 in 100 that a real difference exists between the actual temperatures in question. If, however, the readings are made at different times of day, the difference must equal at least 2.4° for the same level of confidence, since, in this case, a component of the hourly variation must be included in the error estimate. In general, any given reading must deviate from some hypothetical value or from another reading by amounts as great as or greater than those shown in Table 1 before one may safely conclude that there is a significant difference in oral temperature itself.

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Manuscript received October 7, 1953.

Use of the Thymus Gland in Chicks to Elucidate Interrelationships Between Pteroylglutamic Acid and Biologically Related Substances[1]

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Sadun *et al.* (*1*) reported that a pteroylglutamic acid (PGA) deficiency in chicks infected with *Ascaridia galli*, the large roundworm of chickens, fed a highly purified semisynthetic diet resulted in atrophy of the thymus gland. At the time Sadun's studies were carried out, vitamin B_{12} was unavailable and therefore

[1] Journal Article 1553, Michigan Agricultural Experiment Station. Portion of a thesis submitted by Gerald Brody to the Graduate Faculty of Michigan State College in partial fulfillment of the requirements for the Ph.D. Degree. This research was supported by a grant from Lederle Division, American Cyanamid Co., Pearl River, N. Y.

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[3] The author is indebted to William D. Lindquist for his valuable guidance in this project.