from the "norleucine-region" concentrated. Aliquots of the concentrate were chromatographed in butanol–benzyl alcohol and failed to show any trace of norleucine. The sensitivity of this approach is limited but our results indicate that norleucine, if present, occurs in concentrations of less than 1.6 mg./100 g. casein. Norleucine from casein samples of Trial I were identified on the chromatogram both from the radioactivity and from the color reaction with ninhydrin spray. These samples contained considerably more norleucine than the maximum that could have been present in the control samples and from Carbon-14 measurements we have estimated that the minimum concentration of norleucine in the three hour casein sample of Trial I was 4.73 mg. of norleucine/100 g. casein.

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SODIUM FLUOBORATE AS A FLUORINATING AGENT

Sir:

We treated triphenylchlorosilane with sodium fluoborate in acetone solution, in an attempt to prepare triphenylsilyl fluoborate, but the resulting product was triphenylfluorosilane which was obtained in fair yield. The reaction may be described by the equation

$$(C_6H_5)_3SiCl + NaBF_4 + (CH_3)_2CO \longrightarrow (C_6H_5)_3SiF + NaCl + (CH_3)_2CO{:}BF_3$$

This is believed to be the first reported use of sodium fluoborate as a direct fluorinating agent without the intervening preparation of a diazonium salt. It is the most convenient method known for replacing chlorine on silicon by a fluorine atom. No special apparatus is needed and acid solutions are avoided.

The Swarts reaction, using SbF_3,[1] has been most commonly used in carrying out these reactions; other methods employ anhydrous zinc fluoride,[2] or anhydrous hydrogen fluoride.[3] The most convenient method previously reported involved the reaction of the chlorocompounds with aqueous HF at 0°.[4]

Sodium fluoroborate was found to be insoluble in diethyl ether, petroleum ether and dioxane. It was slightly soluble in alcohol and soluble to the extent of 1.0 g. per 100 ml. in acetone.

Materials.—The triphenylchlorosilane was Dow–Corning purified grade. The sodium fluoborate was commercial grade which had been recrystallized once from water (m.p. 364–367°).

Preparation of Triphenylfluorosilane.—In a typical preparation, triphenylchlorosilane (6.32 g., 0.0215 mole) was dissolved in dry acetone and the solution poured into a clear acetone solution of sodium fluoborate (2.23 g., 0.0203 mole). A precipitate appeared almost immediately. After two hours the solution was filtered, with recovery of 76% of the calculated NaCl. The filtrate was concentrated, and on crystallization a trace of tetraphenylsilane, triphenylfluorosilane and an intractable tar were obtained in three different fractions.

On recrystallization, the triphenylfluorosilane (2.9 g., 54% yield) had a m.p. 59–60° (lit. value 61.5°).[5] A cryoscopic determination in benzene gave a molecular weight of 274 (calcd. 278). Qualitative tests confirmed the presence of silicon and fluorine and the absence of boron.

The tar contained material melting slightly above room temperature which gave a positive test for boron and fluorine but not for BF_4^- (by nitron test). The solid could not be easily isolated, as it decomposed on attempted recrystallization. This was assumed to be impure $(CH_3)_2CO{:}BF_3$.

In another reaction performed similarly, a 66% yield of triphenylfluorosilane was obtained. No attempt was made to determine optimum conditions for maximum yield.

One attempt was made to prepare triphenylsilyl fluoborate by passing BF_3 over a benzene solution of the fluorosilane, in the manner of Witschonke and Kraus,[6] but without success.

The generality of the fluorination is being investigated.

(1) H. S. Booth and P. H. Carnell, THIS JOURNAL, **68**, 2736 (1946), and subsequent papers.

(2) J. J. Emeleus and C. J. Wilkins, *J. Chem. Soc.*, 454 (1944); A. E. Newkirk, THIS JOURNAL, **68**, 2736 (1946).

(3) W. H. Pearson, T. J. Brice and J. H. Simons, *ibid.*, **67**, 1769 (1947).

(4) N. S. Marans, L. H. Sommer and F. C. Whitmore, *ibid.*, **73**, 5127 (1951).

(5) C. Curran, R. W. Witucki and P. A. McCusker, *ibid.*, **75**, 4471 (1953).

(6) C. R. Witschonke and C. A. Kraus, *ibid.*, **69**, 2472 (1947).

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INCORPORATION OF ADENOSINE-5′-PHOSPHATE INTO RIBONUCLEIC ACID

Sir:

Since we have shown that cell-free preparations of pigeon liver that incorporate adenine into ribonucleic acid (RNA)[1] can also convert added adenine into adenosine-5′-phosphate (AMP),[2] it was pertinent to determine whether the mononucleotide was a precursor of the polynucleotide. Previous work indicated that mononucleotides were not as efficient as adenine in RNA formation by intact animals,[3] and that surviving tissue slices did not incorporate 5′ nucleotides into RNA.[4] A recent report, however, seems to implicate nucleoside-5′-diphosphates in RNA biosynthesis by extracts of micro-organisms.[5]

AMP labeled with C^{14} in the 4 and 6 positions of the adenine moiety was isolated from the pooled acid-soluble nucleotides derived from the viscera of mice that had been injected with adenine-4,6-

(1) E. Goldwasser, *J. Biol. Chem.*, **202**, 751 (1953).

(2) E. Goldwasser, *Biochem. Biophys. Acta*, **13**, 341 (1954)

(3) H. Weinfeld, P. M. Roll and G. B. Brown, *J. Biol. Chem.*, **213**, 523 (1955).

(4) K. C. Leibman and C. Heidelberger, *ibid.*, **216**, 823 (1955).

(5) M. Grunberg-Manago and S. Ochoa, THIS JOURNAL, **77**, 3165 (1955).

C^{14}.[6] The purified AMP had no detectable contaminant by paper chromatography and assayed 99–100% AMP by enzymic deamination. Its specific activity was 46000 c.p.m./μM.

Incubation of pigeon liver homogenate with the test compound was done under the conditions previously described, except that the homogenate was centrifuged for 5 minutes at 500 *g* and only the supernatant was used.[1] Comparison of AMP and adenine as precursors of RNA yielded the results summarized in Table I.

TABLE I

INCORPORATION OF AMP-4,6-C^{14} INTO RNA

Each flask had 6 ml. of a 20% homogenate, incubated for 2 hours under air at 36° with labeled precursors. RNA was obtained as a mixture of mononucleotides after alkaline hydrolysis.

Precursor	μM.	% of added c.p.m. in RNA	RNA (c.p.m./mg.)	R.S.A.[a] $\times 10^4$
Adenine-8-C^{14}	0.8	0.26	1900	3.1
AMP-4,6-C^{14}	2.1[b]	0.24	107	8.2

[a] Relative specific activity = (specific activity RNA adenine)/(specific activity precursor adenine). [b] Includes endogenous AMP.

From these data AMP may be considered to be at least as effective a precursor of RNA as is adenine in this system.

Evidence that AMP is incorporated into RNA with the ribose-phosphate bond intact was obtained using P^{32}-labeled AMP prepared by Eggleston's method.[7] The resulting AMP, which was chromatographically homogeneous, had a specific activity of 5×10^6 c.p.m./μM. and assayed 99–101% AMP enzymically. The experiment was carried out in the same manner as that described in Table I, but the alkaline hydrolyzate was separated into its constituent mononucleotides by ion-exchange chromatography.[8] The results are summarized in Table II.

TABLE II

INCORPORATION OF AMP-P^{32} INTO RNA MONONUCLEOTIDES

Incubations as in Table I, with 6 μM. of adenine-4,6-C^{14} (A) or with 2 μM. of AMP-P^{32} (B).

	Specific activity, c.p.m./μM.	
	A	B[a]
	Isolated nucleotide	
Cytidylic acid	26	2780
Adenylic acid	48800	720
Uridylic acid	390	5100
Guanylic acid	1760	3060

[a] There was one, as yet, unidentified fraction isolated that contained a significant amount of P^{32} in experiment B that was not present in experiment A.

These data demonstrate that the AMP was actually incorporated into the framework of the RNA molecule because the P^{32} was recovered, for the most part, in non-adenine containing nucleotides. This is so because after alkaline hydrolysis the phosphorus which was incorporated into RNA from the 5′-nucleotide should be found esterified with the 3′-position of the adjacent nucleoside residue. The differences in specific activities indicate strongly that the P^{32} was not incorporated as inorganic phosphate split off the AMP.

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(6) The author is indebted to Dr. E. L. Bennett of the University of California Radiation Laboratory for generous gifts of the crude acid-soluble nucleotides and a sample of adenine-4,6-C^{14}.

(7) L. V. Eggleston, *Biochem. J.*, **58**, 503 (1954).

(8) W. E. Cohn, THIS JOURNAL, **72**, 1471 (1950).

THE CRYSTALLINE COMPOUND AMMONIA-BORANE,[1] H_3NBH_3

Sir:

The ammonia addition compound of the borane group has been considered anomalous for a long time, since its molecular weight in liquid ammonia corresponds to the formula $B_2H_6 \cdot 2NH_3$[2,3] and since no crystalline forms of the compound have been obtainable for X-ray study.

Very recently the new and long sought monomeric compound ammonia-borane, H_3NBH_3, has been prepared from the "diammoniate of diborane," $B_2H_6 \cdot 2NH_3$, as a white, ether soluble solid which gives a definite X-ray powder pattern. An easier synthesis of the compound is achieved from lithium borohydride. The reactions are carried out in diethyl ether at room temperature in accordance with the following equations.

(a) From Lithium Borohydride.

$$LiBH_4 + NH_4Cl \xrightarrow{\text{Diethyl ether}} LiCl + H_3NBH_3 + H_2$$

$$2LiBH_4 + (NH_4)_2SO_4 \xrightarrow{\text{Diethyl ether}} Li_2SO_4 + 2H_3NBH_3 + 2H_2$$

(b) From the "Diammoniate of Diborane".[4]

$$[H_2B(NH_3)_2^+][BH_4^-] + NH_4Cl \xrightarrow[\text{trace of } NH_3]{\text{Diethyl ether}} [H_2B(NH_3)_2^+][Cl^-] + H_3NBH_3 + H_2$$

Although the theoretical amount of hydrogen is produced in the reactions using lithium borohydride, the yields of ether soluble ammonia-borane have been about 45%; an amorphous ether insoluble compound which appears to be the "diammoniate" is produced also.

Molecular weight measurements in dioxane, by freezing point depression, and in diethyl ether, by vapor pressure depression, indicate that ammonia-borane is a monomer (theory: 30.88; found: 31 ± 4).

Anal. Calcd. for H_3NBH_3: H (hydridic), 9.79; B, 35.0; N, 45.4. Found: H (hydridic), 9.73; B, 35.1; N, 45.6.

The properties of crystalline ammonia-borane contrast sharply with those of the classical "di-

(1) The designation of BH_3 as borane is consistent with the system of nomenclature for boron compounds, which was presented by G. W. Schaeffer and T. Wartik at the 125th meeting of the American Chemical Society, Kansas City Missouri.

(2) A. Stock and E. Pohland, *Ber.*, **58**, 657 (1925); H. I. Schlesinger and A. B. Burg, THIS JOURNAL, **60**, 290 (1938).

(3) D. R. Schultz, S. G. Shore and R. W. Parry, to be published.

(4) This laboratory will soon publish accumulated chemical and physical evidence which indicates that the so-called "diammoniate of diborane" is actually a borohydride with a probable structural formula of $[H_2B(NH_3)_2^+][BH_4^-]$. The formulation is consistent with the experimental observations and is used in analogy to the reaction with $LiBH_4$.