method of Whitmore, Revukas, and Smith.[2] The method consists, essentially, of treating nitroaminoguanidine with the aldehyde or ketone in an aqueous or dilute alcoholic medium to which a very small amount of acetic acid has been added. The crystals formed upon cooling the mixture were recrystallized from 50% ethyl alcohol and their melting points determined.

Aldehyde or ketone	Appearance	M. p., °C.	Dumas nitrogen analyses, % Calcd.	Found
Crotonaldehyde	White crystals	177.5	40.93	40.90
Hexone	White flakes	112.5	34.82	35.01
Octyl aldehyde	White crystals	118	31.95	32.43
Acetaldehyde	White crystals	234	48.24	48.35
Heptaldehyde	White crystals	93	32.53	32.72
Butyraldehyde	White crystals	95	40.20	40.00
Acetoacetanilide	White crystals	184	31.46	31.66
Methyl *n*-amyl ketone	White crystals	109.5	32.53	32.64
Veratric aldehyde	Yellow crystals	195	26.22	26.43

The nitroaminoguanidine required was prepared by the hydrazinolysis of nitroguanidine as first suggested by Phillips and Williams.[3]

Nitroaminoguanidine is a very useful addition to the aldehyde and ketone condensing agents. It may be employed in an aqueous medium and yields a crystalline product in less than five minutes, in all cases which we have reported.

BROOKLYN, NEW YORK RECEIVED JULY 10, 1937

---

(2) Whitmore, Revukas and Smith, THIS JOURNAL, **57**, 706 (1935).

(3) Phillips and Williams, *ibid.*, **50**, 2465 (1928).

COMMUNICATIONS TO THE EDITOR

THE ATOMIC WEIGHTS OF SODIUM AND CARBON

Sir:

The results of recent chemical determinations of the atomic weight of carbon by the combustion of hydrocarbons [Baxter and Hale, THIS JOURNAL, **59,** 506 (1937)] and by the titration of benzoyl chloride with silver [Scott and Hurley, *ibid.*, **59,** 1905 (1937)] indicate that the atomic weight of carbon is very close to 12.010. It is of interest to use this value for carbon in the calculation of the atomic weight of sodium from existing measurements of mass ratios involving sodium carbonate.

Four such ratios have been determined accurately: $Na_2CO_3:I_2O_5$ [Baxter and Hale, *ibid.*, **56,** 615 (1934)], $Na_2CO_3:2Ag$, $Na_2CO_3:2AgBr$, and $Na_2CO_3:Na_2SO_4$ [Richards and Hoover, *ibid.*, **37,** 95, 108 (1915)]. The last ratio is, however, unsuitable for this calculation since it requires the use of values for carbon and sulfur known to four decimals in order to obtain sodium to three. On the other hand, if there is no experimental error inherent in the determination of the other three ratios, they should prove to be the best chemical determinations of the atomic weight of sodium yet performed, since the weight of two gram atoms of sodium is directly calculable from the experimental ratios. Employing the value 12.010 for carbon and the current International values for silver, bromine, and iodine, the following values for the atomic weight of sodium are obtained from the three ratios

$Na_2CO_3:2Ag$	22.993
$Na_2CO_3:2AgBr$	22.993
$Na_2CO_3:I_2O_5$	22.994

These values are in good agreement, but are somewhat lower than the present accepted value for sodium, 22.997. They are, however, in agreement with the value 22.994 obtained by Johnson [*J. Phys. Chem.*, **37,** 923 (1933)] from a determination of the ratio NaCl:Ag.

Since no determination of the atomic weight of sodium by the mass-spectrograph method has been made, it is impossible to seek verification of these low values from this source. A physical value, 22.9972, has, however, been obtained recently by Pollard and Brasefield [*Phys. Rev.*, **51,** 8 (1937)] from a study of the nuclear reaction $Ne^{20} + He^4 \longrightarrow Na^{23} + H^1$. When converted to the chemical scale with the factor 1.00025 the value 2.9915 is obtained for sodium. While this value may not be as accurate as the number of significant figures would indicate, it is near enough to those obtained from the sodium carbonate ratios and to Johnson's value to be looked upon as confirming a lower value for sodium. Although any final judgment at this time is probably premature, this concordance suggests that the present International value for the atomic weight of sodium is 0.003–0.004 unit too high.

DEPARTMENT OF CHEMISTRY
THE RICE INSTITUTE
HOUSTON, TEXAS
ARTHUR F. SCOTT
FRANK H. HURLEY, JR.

RECEIVED JUNE 4, 1937

SYNTHETICAL EXPERIMENTS IN THE CHELIDONINE-SANGUINARINE GROUP OF ALKALOIDS

Sir:

In a recent article under the above title that has just come to our attention, Richardson, Robinson and Seijo [*J. Chem. Soc.*, 835 (1937)] report a general scheme for the synthesis of bases of the chelerythrine-sanguinarine type. We have been working in this field for the past two years and certain of our experiments have paralleled those reported by Robinson and his co-workers. While our work has not progressed as far as that of Professor Robinson, we should like to continue our efforts in this field and to submit a brief outline of what we have been doing.

Attempts to apply the Pschorr phenanthrene synthesis in the synthesis of phenanthridines were no more successful than those of the above authors. Instead of using 6-nitropiperonal in our trial experiments we used *o*-nitrobenzaldehyde which condensed readily with α-naphthylamine. The Schiff base was reduced with tin and hydrochloric acid to give two products, one of which formed a monohydrochloride and the other a dihydrochloride. Both compounds gave diazonium salts as indicated by coupling with β-naphthol but treatment with copper powder under various conditions gave only tar. Since it is possible that a *trans* configuration of the Schiff base prevents ring closure, the nitro compound was converted to the methochloride, reduced with tin and hydrochloric acid and diazotized, but again treatment with copper powder gave only tar.

Thinking that ring closure in the β-position of the naphthalene nucleus might be difficult, attempts were made to bring about closure from the naphthalene side. Benzaldehyde could not be made to condense with *o*-nitroaniline so no attempt was made to condense it with 2-nitro-α-naphthylamine. On the other hand, piperonylmethylamine condensed with *o*-nitrochlorobenzene. Treatment of the reduced and diazotized compound with copper powder did not give as much tar as previously but none of the desired phenanthridine has yet been isolated.

We also have been attempting to apply the phenylnaphthylenediamine synthesis of Atkinson and Thorpe to the synthesis of benzophenanthridines, our starting material for trial runs being benzyl cyanide. In view, however, of the inability of Robinson and Liguori to bring about ring closure in the piperonyl and veratryl series, we shall probably discontinue our own efforts in this direction.

Our first work in this field was an attempt to apply a modification of the Bischler–Napieralski synthesis of isoquinolines. The N-piperonylamides of γ-piperonylisocrotonic acid and γ-piperonylpropionic acid were prepared but all attempts at ring closure were unsuccessful. Apparently ring closure can be effected only when the nitrogen atom becomes a part of the ring which is formed.

Work on a series of reactions almost identical with those successfully used by Robinson and co-workers has been barely started. It differs in that we have been attempting to synthesize α,γ-dipiperonylbutyric acid through piperonylmalonic ester and piperonylmethyl bromide and we expect to continue with this phase of the synthesis.

It will be noted from the formulas of Bruchhausen and Bersch [*Ber.*, **63,** 2520 (1930)] that orthoveratraldehyde and orthopiperonal will be needed for the syntheses of the alkaloids themselves. We should therefore like to report the successful methylenation, using methylene sulfate, of 2,3-dihydroxybenzaldehyde obtained from the commercially available orthovanillin, a series of reactions that is much simpler than that reported by Perkin and Trikojus [*J. Chem. Soc.*, 2925 (1926)].

DEPARTMENT OF CHEMISTRY
STANFORD UNIVERSITY
STANFORD UNIV., CALIF.
C. R. NOLLER
R. O. DENYES
J. W. GATES
W. L. WASLEY

RECEIVED SEPTEMBER 23, 1937