dialkylaminostilbazole methiodides, which reduce to colorless products, persisted during the hydrogenation until the hydrogen uptake was practically complete. This was taken to mean that some of the unreacted material was still present right up to the end. From this it was inferred that once reduction of a particular molecule had started it was carried to completion before another of the colored molecules was attacked. For if either the pyridine ring alone *or* the ethylenic side chain alone had been saturated the resulting molecule would be colorless. If either all pyridine rings or all side chains were preferentially hydrogenated then the reaction mixture should become practically colorless by the time three-fourths of the total hydrogen uptake had been accomplished in the first case or by the time one-fourth had been done in the second. As high color persisted significantly beyond the three quarters point both of these latter possibilities seem to be eliminated. In contrast, the ethylenic side chain of 2- or 4-stilbazole hydrochloride was reduced preferentially, under the same conditions, and the reduction could be stopped conveniently at that stage to yield the 2- or 4-phenethylpyridines.[3]

Experimental

Hydrogenation of the Stilbazole Methiodides.—The pure stilbazole methiodide (0.02–0.05 *M*) was dissolved or suspended in absolute methanol (50–100 cc.), approximately 0.2 g. of Adams catalyst was added, and the hydrogenation was carried out in a Burgess–Parr catalytic hydrogenation apparatus with shaking at two to three atmospheres overpressure of hydrogen. Hydrogen uptake proceeded rapidly (usually complete in one to two hours) and came to a stop at or near the calculated value. Platinum was removed by filtration, washed with methanol, and the methanol filtrates were evaporated to dryness. The residue stirred up with ether gave usually a white crystalline product, which was recrystallized from combinations of alcohol, ethyl acetate and ether. For details see Table I.

Acknowledgment.—The author wishes to thank Mr. Samuel W. Blackman for the microanalytical results included here.

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(3) Phillips, *J. Org. Chem.*, **13**, 622 (1948).

WELLCOME RESEARCH LABORATORIES
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The Alpha Phase of Sodium Dodecyl Sulfate

BY FLOYD RAWLINGS, JR.,[1] AND E. C. LINGAFELTER

We have thought it of interest to compare the solid phases of the sodium alkyl sulfates with those of the sodium alkane sulfonates which have been under investigation in this Laboratory for some time.[1a,2,3,4] We have accordingly started an X-ray crystallographic investigation of the series, sodium hexyl, heptyl, octyl, nonyl, decyl, undecyl, dodecyl, tetradecyl, hexadecyl and octadecyl sulfates.

From a solution of sodium dodecyl sulfate in 95% ethanol, slow evaporation at room temperature yielded a crop of well-formed crystals, some of which were used for X-ray investigation. The crystals are very similar in habit to the alpha phase of the sodium alkane sulfonates.[1a] They are quite thin, tabular on (001), and elongated parallel to the *a* axis. In most cases the tablet is outlined by (01*l*) and (11*l*).

X-Ray diffraction data were obtained from rotation, Weissenberg, and precession photographs using copper radiation. Table I contains the constants of the monoclinic unit cell, and the data for two sodium 1-alkane sulfonates for comparison.

TABLE I

	a_0, Å.	b_0, Å.	c_0, Å.	β
$C_{12}H_{25}SO_4Na \cdot xH_2O$	16.47	10.35	77.70	93° 18′
$C_{13}H_{27}SO_3Na \cdot {}^1/_8H_2O$[4]	16.76	10.04	78.21	91° 40′
$C_{12}H_{25}SO_3Na \cdot {}^1/_8H_2O$[1a]	16.80	10.14	76.07	92° 3′

The space group (Aa or A2/a) and the number of molecules in the unit cell (32) is the same for all three of the substances. The amount of hydration of the sodium dodecyl sulfate has not yet been determined. However, the assumption of $^1/_8$ H_2O gives a calculated density of 1.166 g./cc. (observed by flotation method, 1.165 g./cc.).

The sodium dodecyl sulfate differs from the sodium tridecane sulfonate by the substitution of an oxygen atom for a methylene group. Taking the covalent radii to be 0.66 and 0.77 Å., respectively,[5] and assuming the angle of tilt of the chains from the normal to (001), one calculates an expected decrease in d_{001} of 0.68 Å. compared with the observed decrease of 0.51 Å. There is also a small, but real, decrease in a_0 and increase in b_0.

Two other solid phases of sodium dodecyl sulfate have been found, one from 95% alcohol at 25–26° and one from water at 25–26°, both of which are also similar in habit to known phases of the sodium alkane sulfonates. We are continuing the investigation of these phases and others which may be discovered for the long-chain sodium alkyl sulfates.

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[1] Proctei and Gamble Research Fellow, 1949–1950.

(1a) Jensen and Lingafelter, THIS JOURNAL, **66**, 1946 (1944).

(2) Jensen and Lingafelter, *ibid.*, **68**, 1729, 2730 (1946).

(3) Lingafelter and Jensen, *Am. Mineral.*, **32**, 691 (1947).

(4) Wilcox and Lingafelter, to be published.

(5) Pauling, "The Nature of the Chemical Bond," 2nd edition, Cornell University Press, Ithaca, N. Y., 1948, p. 164.

DEPARTMENT OF CHEMISTRY
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Synthesis of 2-(4′-Diethylamino-1′-methylbutylamino)-4-phenylquinoline and a 6-Chloro Derivative

BY GEORGE A. REYNOLDS AND CHARLES R. HAUSER

The recent synthesis of 4-phenyl-2-chloroquinoline (I, X = H)[1] from aniline and ethyl benzoylacetate followed by treatment with phosphorous oxychloride has made possible the convenient

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(1) Hauser and Reynolds, THIS JOURNAL, **70**, 2402 (1948).