N-Methyl-(6-dimethylamino-3-acetoxy-1,3,5-hexatrienyl)-formimine Methiodide.—Furylacrolein (1.0 g., 0.0082 mole) (Eastman Kodak Co., once recrystallized from ligroin), dissolved in 10 cc. of commercial isopropyl alcohol, was cooled to approximately −25°. A solution containing 1.4 g. (0.0079 mole) dimethylammonium iodide, 1.0 g. (0.0075 mole) of a 33% methanol solution of dimethylamine, and 5 cc. of isopropyl alcohol at room temperature was added to the furylacrolein suspension immediately upon its removal from the cooling bath. The addition, with shaking, required thirty seconds. The mixture was cooled immediately to −25° and kept at this temperature except for brief removal periods for stirring (at five-minute intervals). After thirty minutes the reaction mixture was removed from the cooling bath, 10 cc. of dry ether at 0° added, the solution stirred and filtered. The reaction flask and residue were washed with another cold 10-cc. ether portion. The residue was dried three minutes at the pump, and then transferred to a solution containing nitrobenzene (commercial) 100 cc., dry pyridine 10 cc., and acetic anhydride 10 cc. The solution was swirled until all of the crude material dissolved. The reaction required an hour at room temperature. The product was then thrown out with ether to give 1.2 g. (44%) of crude dye. Several recrystallizations from n-butyl alcohol gave an analytical sample with m. p. 191° (dec.). The crystals are a deep purple and possess a metallic luster.

Anal. Calcd. for $C_{13}H_{21}N_2O_2I$: C, 42.86; H, 5.81. Found: C, 42.89; H, 5.79.

I should like to thank Dr. L. G. S. Brooker for showing me an unpublished modification of reference 8, and Professors W. G. Dauben and G. E. K. Branch for much helpful advice.

DEPARTMENT OF CHEMISTRY
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NEW COMPOUNDS

Some Heterocyclic-Substituted Hydrazines

The condensation of a 2- or 4-chloroquinoline or of 2-amino-4-chloropyrimidine with benzenesulfonhydrazide was carried out in two ways. Refluxing a mixture of one mole of the halogen compound and two moles of benzenesulfonhydrazide in alcohol for two to six hours gave 40–50% yields of condensation product as the hydrochloride. In confirmation of the observations of Banks[1] it was found that the condensation of molal proportions of halogen compound and benzenesulfonhydrazide in the presence of one mole of hydrogen chloride (added as concentrated hydrochloric acid) increased the yields to 80–90%; the rate of condensation was also appreciably increased by this modification. The hydrochlorides were purified by crystallization from dilute or glacial acetic acid; excepting the pyrimidine derivative, the products were almost insoluble in water or dilute acids. Dilute bases produced the expected decomposition.[2]

1-Benzenesulfonyl-2-(7-chloroquinolyl-4)-hydrazine hydrochloride was also prepared, in 35% yield, by the action of benzenesulfonyl chloride on 7-chloro-4-hydrazinoquinoline in dry pyridine in the usual manner, followed by treatment of the product with dry hydrogen chloride in alcohol. The latter compound was prepared in 88.6% yield from 4,7-dichloroquinoline essentially by the general procedure of Koenigs and Loesch.[3] The compound formed white needles from alcohol, m. p. 231–232° (cor.) (dec.).

Anal. Calcd. for $C_9H_8ClN_3$: N, 21.70. Found: N, 21.68.

Surrey and Cutler[4] reported m. p. 220–221°.

1-Benzenesulfonyl-2-(5-nitropyridyl-2)-hydrazine was prepared from 2-hydrazino-5-nitropyridine[5] and benzenesulfonyl chloride in dry pyridine. This compound could not be prepared by the direct condensation of 2-chloro-5-nitropyridine with benzenesulfonhydrazide in either the presence or absence of hydrochloric acid.

The substituted hydrazines are listed in the accompanying table.

TABLE I

N^1-BENZENESULFONYL-N^2-SUBSTITUTED HYDRAZINE HYDROCHLORIDES

N^2-Substituent	M. p., °C.	Formula	Analyses, % Sulfur Calcd.	Sulfur Found	Chlorine Calcd.	Chlorine Found
Quinolyl-2-	207–209[a]	$C_{15}H_{14}ClN_3O_2S$	9.55	9.60	10.56	10.58
4-Methylquinolyl-2-[b]	171–172	$C_{16}H_{18}ClN_3O_3S$	8.72	8.69	9.64	9.65
5-Chloroquinolyl-4-	219.5–220.0[a]	$C_{15}H_{13}Cl_2N_3O_2S$	8.66	8.35	9.58[e]	9.56
7-Chloroquinolyl-4-	203–204[a]	$C_{15}H_{13}Cl_2N_3O_2S$	8.66	8.36	9.58[e]	9.66
7-Chloro-3-methylquinolyl-4-[c]	196–197[a]	$C_{18}H_{19}Cl_2N_3O_4S$	7.22	7.27	15.96	15.98
7-Phenoxyquinolyl-4-	209–210[a]	$C_{21}H_{18}ClN_3O_3S$	7.49	7.51	8.29	8.20
2-Aminopyrimidyl-4-	237–239[a]	$C_{10}H_{12}ClN_5O_2S$	10.63	10.52	11.75	11.61
5-Nitropyridyl-2-[d]	196–197[a]	$C_{11}H_{10}N_4O_4S$	10.89	11.00	...	...

[a] With decomposition. [b] As the monohydrate. [c] With one mole of acetic acid of crystallization. [d] Free base. [e] Ionic halogen only.

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[1] Banks, THIS JOURNAL, 66, 1127 (1944).

(2) 1-Benzenesulfonyl-2-(7-chloroquinolyl-4)-hydrazine hydrochloride gave a 43.2% yield of 7-chloroquinoline, m. p. 31–32°, when steam distilled from excess sodium carbonate solution. 1-Benzenesulfonyl-2-(5-nitropyridyl-2)-hydrazine similarly gave a 32% yield of 3-nitropyridine, m. p. 39.5–40.5°. *Cf.* McFadyen and Stevens, *J. Chem. Soc.*, 584 (1936).

(3) Koenigs and Loesch, *J. prakt. Chem.*, **143**, 59 (1935); *cf.* Perkin and Robinson, *J. Chem. Soc.*, **103**, 1978 (1913).

(4) Surrey and Cutler, THIS JOURNAL, **68**, 2570 (1946).

(5) Rath, U. S. Patent 1,733,695.

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Preparation of Organic Silicon Chlorides[1]

The general method of synthesis was to add the appropriate Grignard reagent dropwise into an excess of silicon

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(1) This work was performed in 1945 as part of the research program of the Research and Development Branch, Military Planning Division, of the Office of the Quartermaster General. The opinions and conclusions contained in this report are those of the authors. They are not to be construed as necessarily reflecting the views or endorsement of the Department of the Army. Article not copyrighted. This work was performed with the assistance of Charles A. Miller, Joseph Rynasiewicz, Nelda Gulbransen, Esther Nielson and Eleanor Swenson.