neutral oxalate by adding a solution of 1 g. anhydrous oxalic acid in 10 cc. absolute alcohol. Yield 1.65 g. (93%). It was recrystallized from 80% alcohol and had a m.p. 201–202°.

Anal. Calcd. for $C_{10}H_{24}O_8N_2$: C, 40.0; H, 8.0; N, 9.3. Found: C, 39.9; H, 8.1; N, 9.3.

DL-*Threoninol.* Three and one half g. of raw *N*-benzylthreonine was debenzylated as above and converted into the neutral oxalate. Yield 2.55 g. (94%). After recrystallization from 80% alcohol, it had m.p. 211.5° (dec.).

Anal. Calcd. for $C_{10}H_{24}O_8N_2$: C, 40.0; H, 8.0; N, 9.3. Found: C, 40.0; H, 7.6; N, 9.3.

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C=N Stretching Frequency in Infrared Spectra of Aromatic Azomethines

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In conjunction with a study of the stereochemistry of aromatic Schiff-bases, 17 compounds of this type have been synthesized by standard techniques[1] and their infrared spectra determined. Each compound was found to exhibit an absorption band of medium intensity in the double-bond stretching region at 1613–1631 cm.$^{-1}$, as shown in Table I. This band, which occurs in the spectra just to the high frequency side of the normal aromatic band near 1600 cm.$^{-1}$, appears to be of considerable diagnostic value.

TABLE I

C=N STRETCHING FREQUENCIES IN AROMATIC SCHIFF BASES. (Measurements made on Beckman IR-3 spectrophotometer, NaCl optics.)

Compound	Frequency (cm^{-1})[a]
N-benzylidineaniline*	1631
N-(2-hydroxy)benzylidineaniline*	1622
N-(4-hydroxy)benzylidineaniline	1629[b]
N-(4-methoxy)benzylidineaniline*	1630
N-(2-nitro)benzylidineaniline	1621[b]
N-(4-acetylamino)benzylidineaniline	1629[b]
N-(4-dimethylamino)benzylidineaniline*	1626
N-benzylidine-2-aminophenol	1629
N-benzylidine-2-anisidine	1631[c]
N-(4-methoxy)benzylidine-2-anisidine	1627[c]
N-benzylidine-4-anisidine*	1629
N-(4-methoxy)benzylidine-4-anisidine	1626[b]
N-benzylidine-4-toluidine	1628[c]
N-benzylidine-*N*′-dimethyl-4-phenylenediamine*	1627
N-(2-hydroxy)benzylidine-2-aminophenol	1624[b]
N-(4-dimethylamino)benzylidine-2-aminophenol	1613
N,*N*′-dibenzylidine-4-phenylenediamine	1628

[a] in CCl_4 solution; [b] in $CHCl_3$ solution; [c] as KBr pellets; * indicates the compounds reduced using $NaBH_4$.

The absorption frequency characteristic of the stretching mode of the C=N group in compounds of this type has not been known with any degree of certainty.[2] Therefore, in order to ascertain whether the absorption band in this region was attributable to the C=N group, it was necessary to compare the infrared spectra of the Schiff-bases with those of similar compounds that do not contain this grouping. Consequently, six of the azomethines listed in Table I were reduced to the corresponding substituted *N*-benzylanilines by means of sodium borohydride, using a modification of the procedure of Chaikin and Brown.[3] Since this band is absent in the infrared spectra of these amines, this indicates that the absorption bands shown by *N*-benzylidine aniline and its ring-substituted derivatives are indeed due to the stretching mode of the C=N group.

It is also worth noting that the sodium borohydride reduction of aromatic azomethines usually proceeds very smoothly and results in fair yields (60–80% of theoretical). Consequently this reaction may find application in the synthesis of secondary amines of this type.

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(1) Cf. G. Smets and A. Delvaux, *Bull. soc. chim. Belg.*, **56**, 106 (1947).

(2) L. J. Bellamy, *The Infrared Spectra of Complex Molecules*, John Wiley and Sons, New York, N. Y. (1954), pp. 226–229.

(3) S. W. Chaikin and W. G. Brown, *J. Am. Chem. Soc.* **71**, 122 (1949).

Chloroalkyl and Chloroaryl Chloromethylphosphonates

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In 1950, Kabachnik and Medved reported the preparation of chloromethylphosphonic dichloride by the reaction of paraformaldehyde with phosphorus trichloride.[1] We have prepared and characterized several chlorinated esters from this acid chloride.

In order to prevent alkyl halide formation when an alcohol reacts with a phosphonic chloride, it is generally necessary to use a tertiary amine to absorb the liberated hydrogen chloride. We found, however, that 2-chloroethanol and 2,2,2-trichloroethanol could be successfully treated with chloromethylphosphonic dichloride at reflux tempera-

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(1) M. I. Kabachnik and T. Ya. Medved, *Izvest. Akad. Nauk S.S.S.R., Otdel Khim Nauk*, 635 (1950); [*Chem. Abstr.*, **45**, 8444 (1951)].