

$S_2O_8^{2-}$ ,  $F^-$ ,  $C_2O_4^{2-}$  and  $ClO_4^-$  showed that only  $CrO_4^{2-}$  and  $MnO_4^-$  interfered seriously when present in equal amounts and in 10 fold excess to that of the bismuth.  $S_2O_8^{2-}$ ,  $AuCl_4^-$  and  $CNS^-$  ions in 10 fold excess interfered, the latter two interfered only slightly. All other anions did not interfere even when present in 10 fold excess.

Interference due to iron (III) could be removed by using oxalic acid and ammonium fluoride as the masking reagents and a good recovery of bismuth (error within 1%) was achieved. These reagents did not interfere in the determination of bismuth. Interference due to chromium (VI) could be removed by prior separation of chromium by extraction with isobutyl methyl ketone from solutions above 1 N with respect to hydrochloric or sulphuric acid, a method that has been used in the determination of chromium<sup>11,12</sup>. Recovery of bismuth was within 2% error. The application of the common analytical practice of reducing potassium permanganate with oxalic acid in acidic medium proved to be successful in the removal of interference due to  $MnO_4^-$  ion. Recovery of bismuth was within 3% error.

Thus the method proved to be a simple and rapid one providing a good recovery of bismuth in presence of equal amounts of most of the common ions. Besides, it also provides simple techniques for the removal of interferences due to these interfering seriously.

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Department of Chemistry, N. K. BAISHYA.  
University of Gauhati, G. BARUAH.  
Gauhati 781 014, Assam, July 31, 1975.

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## MESOMORPHIC BEHAVIOUR OF SOME SCHIFF BASE COMPOUNDS COMPRISING NAPHTHALENE MOIETY

A NUMBER of homologous series comprising schiff base compounds exhibiting mesomorphism are known. Generally a linear structure favours the development of mesomorphism. It is observed that when molecular structure is varied within a group of suitable compounds the liquid crystalline properties change markedly. The introduction of a 1,4 substituted naphthalene nucleus in place of a 1,4 substituted phenylene ring usually reduces the thermal stabilities of the mesophase.

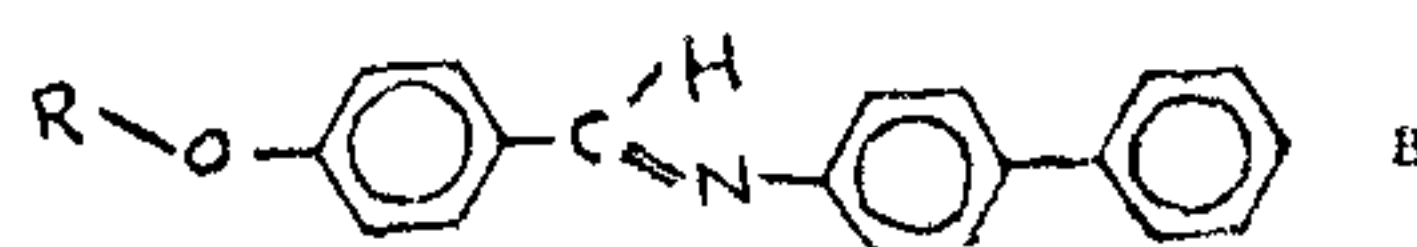
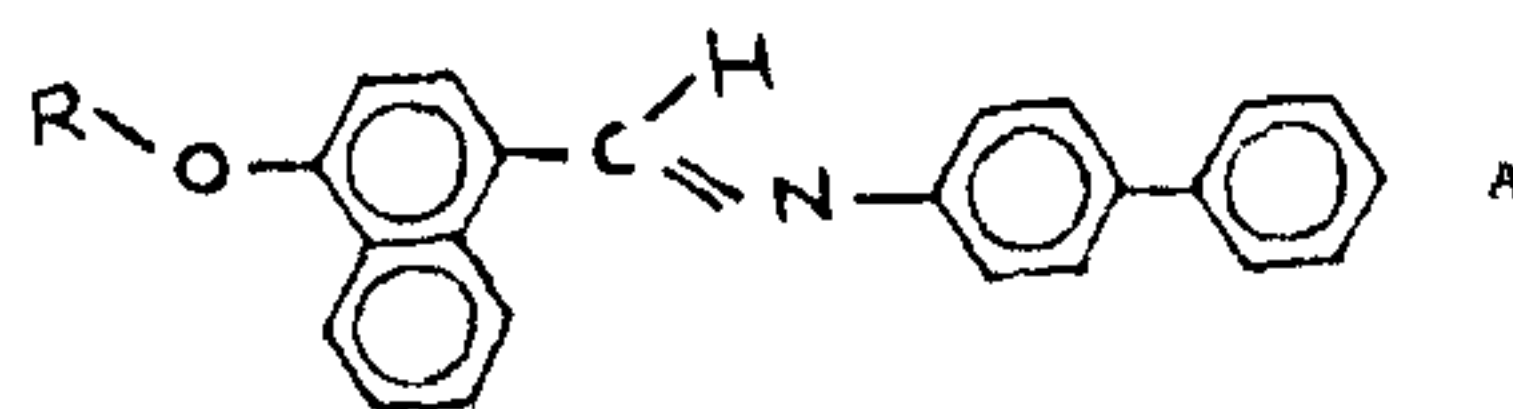
Dave *et al.*<sup>1-5</sup>, have studied a number of schiff base series comprising a naphthalene moiety. As a part of our programme of the study of the effect of breadth on mesomorphism we describe here the mesomorphic behaviour of the four compounds of the series 4-*n*-alkoxy-1-naphthalidene-4'-aminobiphenyls. The melting points and transition temperatures are summarized in Table I.

TABLE I  
4-*n*-Alkoxy-1-naphthylidene-4'-aminobiphenyls  
 $RO.C_{10}H_6CH : N.C_6H_4.C_6H_5$

R	Transition temperatures (°C)		
	Smectic	Nematic	Isotropic
1. $CH_3$	...	..	201.5
2. $C_{12}H_{25}$	..	(103.0)	107.5
3. $C_{16}H_{33}$	..	92.0	97.0
4. $C_{18}H_{37}$	(69.0)	83.5	93.0

Values in parenthesis indicate monotropy.

The methyl derivative is non-mesomorphic; the dodecyl derivative exhibits a monotropic nematic mesophase and the last two members, i.e., hexadecyl and octadecyl derivatives exhibit enantiotropic nematic mesophases. The octadecyl derivative exhibits a monotropic smectic mesophase also.



The nematic mesophase exhibited by these compounds has the threaded texture and the smectic mesophase has the broken fan shaped texture.

The mesomorphic thermal stabilities of the compounds of the present series (series A) are comparatively less than those of the corresponding compounds of the benzylidene series, *p*-*n*-alkoxybenzylidene-4-aminobiphenyls<sup>6</sup> (series B). This is expected as series (A) contains a 1,4 substituted naphthalene nucleus at one end of the molecule which increases the molecular breadth resulting in the decrease of the thermal stabilities of the mesophases. It is of interest to note that only the last member of the series (A), i.e., the octadecyl derivative exhibits smectic mesophase which is monotropic in nature. In the case of series (B) the smectic phase appears at the pentyl derivative and purely smectic in nature. The reason for this late from the dodecyl derivative onwards the series is commencement of smectic mesophase in series (A) can be attributed to the increased breadth of the molecules of series (A) due to the introduction of the naphthalene moiety which not only reduces the thermal stabilities of the mesophases but also delays the commencement of the smectic mesophase.

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Chemistry Department,  
Faculty of Science,  
M.S. University,  
Baroda, August 1, 1975.

J. S. DAVE.

A. P. PRAJAPATI.

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#### ION-EXCHANGE KINETICS STUDY USING HYDROUS CERIC OXIDE AS AN ION-EXCHANGER

STUDY of the kinetics of ion-exchange is of importance in the use of ion-exchange resins for chemical separations. There is, however, a paucity of such data on inorganic ion-exchangers<sup>1</sup>. The present paper describes an investigation of the kinetics of exchange reactions in hydrous CeO<sub>2</sub> using amines of Co<sup>3+</sup>, Ni<sup>2+</sup>, Cu<sup>2+</sup>, Zn<sup>2+</sup> and Cd<sup>2+</sup> as exchanging ions. The study includes (1) dependence of the exchange rate on the concentra-

tion of the exchanging ion and (2) effect of the size of the exchanger particle and exchanging ion on the rate of exchange.

#### Materials and Method

All the reagents used were of A.R. quality. Hydrous CeO<sub>2</sub> was prepared by adding excess of aqueous ammonia to a saturated solution of ceric ammonium sulphate<sup>2</sup>. The resulting precipitate was washed free of sulphate and dried at 100–110° C. Ammine complexes of Co<sup>3+</sup>, Cu<sup>2+</sup> and Ni<sup>2+</sup> were prepared by the usual methods<sup>3</sup> and those of Zn<sup>2+</sup> and Cd<sup>2+</sup> by dissolving appropriate quantities of the metal salts in water followed by the addition of excess of aqueous ammonia.

**Procedure for the Rate-Study.**—Samples of 20 ml of the metal amines were added to a series of stoppered conical flasks, each containing 100 mg of the exchanger. The resulting mixtures were shaken mechanically up to equilibrium. The solutions were immediately centrifuged and the supernatants (5 ml) were analysed for Co<sup>3+</sup> and Cu<sup>2+</sup> iodometrically<sup>4,5</sup> and Ni<sup>2+</sup>, Cd<sup>2+</sup> and Zn<sup>2+</sup>, complexometrically<sup>6</sup> and the amount of metal ions sorbed, hence their F-values were found out from the relation<sup>7</sup>

$$F = \frac{\text{Amount of the metal ion sorbed at time } t}{\text{Total amount of the metal ion sorbed at equilibrium}}$$

The samples of CeO<sub>2</sub> with an average particle radius  $9.0 \times 10^{-3}$  cm and  $0.85 \times 10^{-3}$  cm were obtained with the help of standard sieves. The particle sizes were further checked by microscopic measurements.

#### Results and Discussion

The results presented in Tables I and II indicate that

- (i) The ion-exchange kinetics exhibit two distinctly separate stages, relatively quick process in the first stage followed by a slower one towards the end.
- (ii) Smaller the size of the exchanger particle as well as exchanging ion faster is the rate of exchange.
- (iii) Amongst the ammine complexes under study, the exchange rate follows the order :  
Co > Cu > Zn > Ni > Cd.

It is well known<sup>8</sup> that sorption of metal ions on hydrous oxides takes place by the mechanism of ion-exchange. It is also known<sup>9</sup> that the overall exchange process is mainly governed by two types of diffusion processes. The first process, i.e., film diffusion depends on (a) stirring characteristics, (b) the external concentration of the exchanging ions and (c) the size of the exchanger particle. The second process, i.e., particle diffusion depends only on the size of the exchanger particle<sup>10</sup>. In very dilute solutions the rate of diffusion of electrolyte across the film barrier about the