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Mesomorphic Schiff's Bases Derived from *p*-Acetoxy-, *p*-*n*-Butyl-, and *p*-*n*-Butoxy-Anilines

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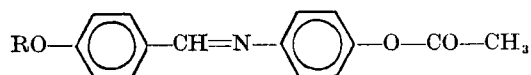
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This note describes thirty-three new Schiff's bases which for structural reasons⁽¹⁾ were expected to show nematic phases of low thermal stability. In summary, the following trends seem apparent from the data listed in Tables 1, 2, and 3 for the *p*'-acetoxy-, *p*'-*n*-butyl- and *p*'-*n*-butoxy anils, respectively.

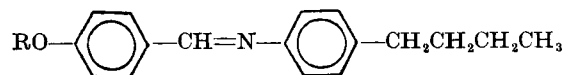
- (1) The lowest transitions were observed within *p*'-*n*-butylanils for analogous benzylidene compounds. The transitions of *p*'-*n*-butoxy-anils were higher than those of *p*'-acetoxy-anils in five out of eight cases.
- (2) For a given anil, the transitions were lowest for *p*-hydroxy-benzylidene groups with oxa-alkyl-oxy-substituents (4, 5, 6 and Ref. 1) at a sacrifice in the incidence of mesomorphism. Those with alkyl- and butenyl-oxy substituents (2, 3, 12, 13, 29, 30) were among the highest melting compounds studied and showed transitions which were up to 20° above those for the saturated analogues of identical chain length.⁽¹⁾ An acyl oxy substituent (7, 16, 31) caused all transitions to be higher than an alkylcarbonate group of the same total chain length (8, 17, 32), both melting around or below the alkyl oxy analogues. An increase in the alkyl chain of the latter caused a further reduction in transition temperatures (9, 19, 33). Acyl-ester or carbonate groups within substituent chains resulted in a loss of mesomorphism (10, 21, 22, 23, 24) except in one (20) case. The melting points of these bases were well below their alkyl analogues⁽¹⁾ but above those of the oxa-alkyl analogues.⁽¹⁾ Aromatic substituents (11, 27, 28) caused the melting points to

TABLE 1 Transition Temperatures °C



	R	Smectic Range		Nematic Range		MP
1	C ₂ H ₅ —			104.6	132.8	—
2	CH ₂ =CH—CH ₂ —	84.6	110.8	110.8	111.4	—
3	CH ₃ —CH=CH—CH ₂ —			127	134.5	—
4	C ₂ H ₅ O—(CH ₂) ₂ —					81
5	<i>n</i> -C ₄ H ₉ O—(CH ₂) ₂ —					62.8
6	C ₂ H ₅ (OCH ₂ CH ₂) ₂ —					70.3
7	<i>n</i> -C ₃ H ₇ —CO—	92.3	135.1			
8	C ₂ H ₅ O—CO—			115.1	123.9	
9	<i>n</i> -C ₄ H ₉ O—CO—	66.7–78.6–94.9				
10	C ₂ H ₅ O—CO—CH ₂ —					93.4
11	C ₆ H ₅ CH ₂ —					160.4

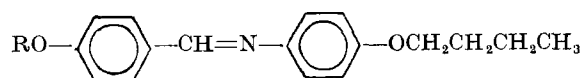
TABLE 2 Transition Temperatures °C



	R	Nematic Range		%	MP
12	CH ₂ =CH—CH ₂ —	49.7	68.5	—	—
13	CH ₃ —CH=CH—CH ₂ —	62.7	94.2	—	—
14	CH ₃ —CO—				57.9
15	C ₂ H ₅ —CO—	65	71.2	—	—
16	<i>n</i> -C ₃ H ₇ —CO—	67.7	77.9	—	—
17	C ₂ H ₅ —O—CO—	52	60	—	—
18	C ₂ H ₅ —S—CO—				65.3
19	<i>n</i> -C ₄ H ₉ —O—CO—				76.1
20	C ₂ H ₅ O—CO—CH ₂ —	49 (monotr.)			63.9
21	<i>n</i> -C ₄ H ₉ O—CO—CH ₂ —				35.5
22	C ₂ H ₅ O—CO—(CH ₂) ₃ —				49
23	C ₂ H ₅ O—CO—O—(CH ₂) ₃ —				57.4
24	CH ₃ —CO—O—(CH ₂) ₂ —				57.9
25	CN—CH ₂ —				66.4
26	CH ₃ —(CH ₂) ₅ —O—CO—				57.4
27	C ₆ H ₅ —CH ₂ —				102.2
† 28	C ₆ H ₅ —	120.2	131.1		

† Note: Compound 28 is not an ether.

TABLE 3 Transition Temperatures °C



	R	Smectic Range		Nematic Range	
29	CH ₂ =CH—CH ₂ —			114	119.6
30	CH ₃ —CH=CH—CH ₂ —	125	128.8	128.8	129.6
31	<i>n</i> -C ₃ H ₇ —CO—	85.5	99.9	99.9	126.8
32	C ₂ H ₅ O—CO—			98.5	118.7
33	<i>n</i> -C ₄ H ₉ O—CO—			88.7	105.5

TABLE 4 Elemental Analyses

	Formula	Calculated %			Found %		
		C	H	N	C	H	N
1	C ₁₇ H ₁₇ NO ₃	72.08	6.05	4.94	71.99	6.13	5.11
2	C ₁₈ H ₁₇ NO ₃	73.20	5.80	4.74	73.18	5.83	4.80
3	C ₁₉ H ₁₉ NO ₃	73.77	6.19	4.53	73.52	6.18	4.83
7	C ₂₀ H ₂₁ NO ₄	70.78	6.24	4.13	70.84	5.89	4.35
8	C ₁₈ H ₁₇ NO ₅	66.05	5.23	4.28	65.76	5.25	4.32
12	C ₂₀ H ₂₃ NO	81.87	7.90	4.77	81.70	7.93	4.89
13	C ₂₁ H ₂₅ NO	82.04	8.19	4.56	82.28	8.24	4.67
15	C ₂₀ H ₂₃ NO ₂	77.64	7.49	4.53	77.40	7.51	4.23
16	C ₂₁ H ₂₅ NO ₂	77.99	7.79	4.33	77.65	7.81	4.46
17	C ₂₀ H ₂₃ NO ₃	73.82	7.12	4.30	73.64	7.17	4.32
28	C ₂₂ H ₂₃ N	87.66	7.69	4.65	87.58	7.47	4.54
29	C ₂₀ H ₂₃ NO ₂	77.64	7.49	4.53	77.37	7.50	4.64
30	C ₂₁ H ₂₅ NO ₂	77.99	7.79	4.33	77.69	7.88	4.42
31	C ₂₁ H ₂₅ NO ₃	74.31	7.42	4.13	74.08	7.30	4.25
32	C ₂₀ H ₂₃ NO ₄	70.36	6.79	4.10	70.57	6.91	4.18
33	C ₂₂ H ₂₇ NO ₄	71.52	7.37	3.79	71.27	7.41	3.98

be relatively high. Similar results have been reported with some of these substituents by Castellano *et al.*⁽²⁾

It is noteworthy that 14, 15, and 16 form the beginning of a homologous series which does not seem to show alternation in transitions when going from odd to even numbered members. Also, the transition temperatures rise with increasing chain length which, if confirmed in continuing the series is somewhat unusual.⁽¹⁾

1. Experimental

The synthesis and characterization procedures including transition points were identical to those described previously.⁽¹⁾ The elemental analyses for compounds which did not show mesomorphism were omitted, others are listed in Table 4. The starting anilines were purchased.

Acknowledgement

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2. Castellano, J. A., Goldmacher, J. E., Barton, L. A. and Kane, J. S., *J. Org. Chem.* **33**, 3501 (1968).
3. See Ref. 13 in Dietrich and Steiger.⁽¹⁾