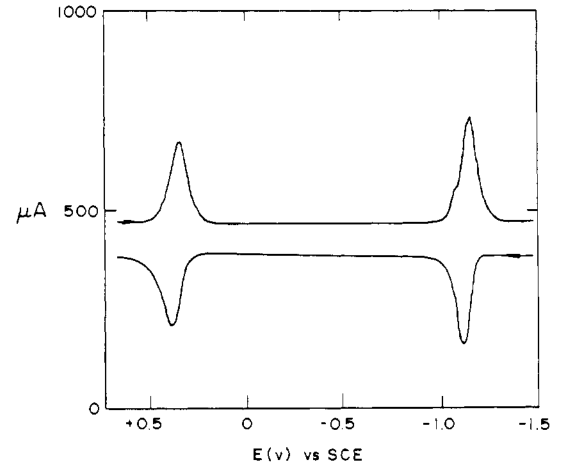

Figure 1. Differential pulse polarogram of DTT (10^{-5} M in CH_3CN) at glassy carbon electrode.

attachment. Ultraviolet spectra were recorded on a Cary 14 recording spectrophotometer in acetonitrile (Burdick & Jackson) solutions. Infrared spectra (IR) were recorded on a Perkin-Elmer 225 spectrophotometer in KBr. Nuclear magnetic resonance (NMR) spectra were run in $CDCl_3$ solution containing Me_4Si as internal standard using a Varian 220-MHz instrument. Mass spectra were obtained using a Hitachi/Perkin-Elmer RMH-2 instrument. Polarographic measurements were performed in MeCN on a Princeton Applied Research Model 174A polarographic analyzer using a glassy carbon working electrode and a Ag|Ag$^+$ reference electrode. Microanalyses were performed by Galbraith Laboratories, Knoxville, Tenn.

***Naphthaceno[5,6-cd]-1,2-dithiole* (*2*).** A mixture of 1 (0.50 g) and sublimed sulfur (0.14 g) in anhydrous dimethylformamide (25 mL) was refluxed for 24 h under argon. The reaction mixture was cooled and filtered and filtrate was concentrated. The combined solid obtained above (0.56 g) was gradient sublimed (195 °C, 3 × 10^{-5} Torr) three times with only successive middle fractions retained for subsequent sublimations to give 0.2 g (31%) of **2** as a violet crystalline solid: mp 212.7–214.6 °C; IR 1610, 1580, 1485, 1303, 1290, 938, 860, 829, 770, 750, 729, and 559 cm^{-1}; λ_{max} 214 nm (log ϵ 6.47), 236 (6.60) 297 (6.99), 402 (sh) (5.29), 425 (5.63), and 561 (5.63); NMR δ 8.00 (2 H, s, 11- and 12H_2), 7.82 (2 H, d, J = 2 Hz, 4- and 7H_2), 7.55 (2 H, d, J = 8 Hz, 1- and 10H_2), and 7.48–7.23 (4 H, m, 2-, 3-, 8-, and 9H_2); mass spectrum *m/e* (relative intensity) 290 (M, 100), 145 (M/2, 7).

Anal. Calcd for $C_{18}H_{10}S_2$: C, 74.44; H, 3.48; S, 22.08. Found: C, 74.43; H, 3.43; S, 22.29.

***Naphthaceno[5,6-c,d]-1,2-dithiole* (*tetracyanoquinodimethane*) (*4*).** A hot saturated solution of **2** in dichlorobenzene (Burdick & Jackson) was added to a hot equimolar solution of TCNQ in acetonitrile (Burdick & Jackson). The resulting mixture was allowed to cool, filtered, and vacuum dried to give **4** as black needles: mp 219.3 °C dec; IR 2210, 1615, 1575, 1530, 1345, 1285, 1115, 929, 865, 835, 768, 730, 612, 468, and 458 cm^{-1}.

Anal. Calcd for $C_{30}H_{14}N_4S_4$: C, 72.85; H, 2.86; N, 11.83; S, 12.96. Found: C, 72.73; H, 2.99; N, 11.60; S, 13.02.

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Aldol-Condensation Products from the Multiple Anions of Oximes, Benzanilides, a Benzamide, and *o*-Tolylanilides

Charles F. Beam,* Charles A. Park, and David C. Reames

Department of Chemistry, Newberry College, Newberry, South Carolina 29108

Scott A. Miller and Charles R. Hauser[†]

Paul M. Gross Chemical Laboratory, Duke University, Durham, North Carolina 27706

Several new β-hydroxyoximes, γ-hydroxyamides and δ-hydroxyamides were prepared from respective multiple anions by an aldol-type condensation with an aldehyde or ketones.

In connection with our work with $C(\alpha),O$-dilithiooximes (*2–4, 7–10*), several new β-hydroxyoximes were prepared by condensation of the dianion with ketones, such as 2-benzoyl-

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[†] Deceased.

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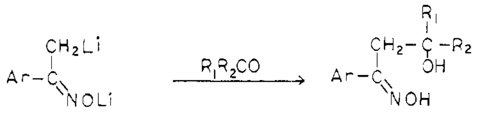
Table I. β-Hydroxyoximes and Hydroxyamides

Compd no.	Parent amide or oxime	Electrophilic reagent (aldehyde or ketone)	Empirical formula of hydroxyoxime or hydroxyamide[a]	% yield	Mp, °C	Recryst solv
1	Acetophenone oxime	2-Benzoylthiophene	$C_{19}H_{17}NO_2S$	40	149-151	Ethanol-water
2	4-Methylacetophenone oxime	2-Benzoylthiophene	$C_{20}H_{19}NO_2S$	16	153-155	Ethanol-water
3	4-Methylacetophenone oxime	4-Methylbenzophenone	$C_{23}H_{23}NO_2$	17	172-174	Ethanol-water
4	4-Methylacetophenone oxime	Benzophenone	$C_{22}H_{21}NO_2$	74	154-156	Ethanol-water
5	Benzanilide	4-Methylbenzophenone	$C_{27}H_{23}NO_2$	61	180-182	Acetonitrile
6	*N*-Methylbenzamide	4-Methoxybenzaldehyde	$C_{16}H_{17}NO_3$	55	123-125	Acetonitrile
7	Benzanilide	4-Chlorobenzophenone	$C_{26}H_{20}ClNO_2$	75	170-173	Acetonitrile
8	Benzanilide	4-Methoxybenzaldehyde	$C_{21}H_{19}NO_3$	67	147-148	Acetonitrile
9	*N*-(*o*-Chlorophenyl)-*o*-tolylanilide	Benzophenone	$C_{27}H_{22}ClNO_2$	51	151-152	Acetonitrile
10	*N*-(*p*-Chlorophenyl)-*o*-tolylanilide	Benzophenone	$C_{27}H_{22}ClNO_2 \cdot CH_3CN$	43	200-203	Acetonitrile

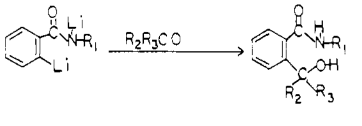
[a] Elemental analyses for C, H, and N in agreement with theoretical values obtained and submitted for review. They were performed by M-H-W Laboratories, Garden City, Mich., and Robertson's Laboratory, Florham Park, N.J.

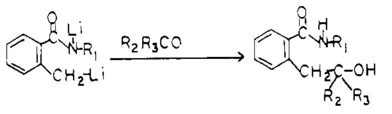
thiophene and 4-methylbenzophenone. The synthetic method is straight-forward (*7*) and requires readily available starting materials, and the β-hydroxyoximes prepared appear to be new. The results are listed in Table I.



C(α),*O*-dilithiooxime β-hydroxyoxime

The dianions of *N*-alkylbenzamides (*6*) and *N*-alkyl-*o*-toluamides (*5*) have been condensed with ketones to give γ- and δ-hydroxyamides; however, their cyclization under acid-catalyzed conditions has led to questionable results (*1*), which do not invalidate the value of the aldol condensation to give the hydroxyamide. The diversity of the reaction was shown by condensation of several of these anions with an aldehyde and ketones to give new materials which are listed in the table.





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