A Facile Synthesis of Perhydropyrimidines through the Michael Addition of Some Active Methylene Compounds to *N,N'*-Dibenzylidenephenylmethanediamines and Some Related Reactions

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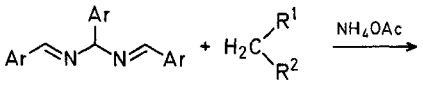
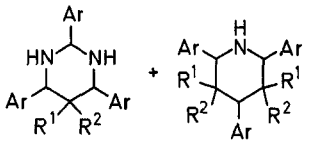
Since its first preparation in 1837[1], *N,N'*-dibenzylidenephenylmethanediamine **(1a)** and analogs have been little used synthetically, except for the isomerization to amarine by thermal[2] or electrocyclic[3] ring closure, hydrogenation[4], complex-formation[4], and synthesis of Δ^3-pyrroline-2-one derivatives[5,6].

Recently, we reported simple syntheses of pyrimidobenzoxazines[7], 1,3,7-triazabicyclo[3.3.1]-3-nonenes, and 2,3,4,5-tetrahydropyrimidines[8] from **1** and carbonyl compounds. The initial step of these reactions is a Michael addition of the carbonyl compounds to one azomethine group of **1**, followed by elimination of an aldehyde from the other azomethine group of the adducts. Herein, we report that the synthesis of hexahydropyrimidine derivatives by Michael addition of active methylene compounds **2** to both azomethine groups of **1**, is achieved by using malononitrile, nitromethane, nitroethane, methyl cyanoacetate, or α-cyanoacetamide.



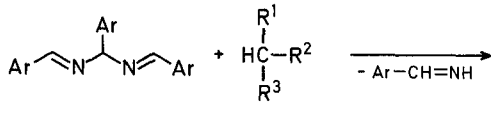
1 **2**



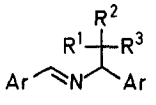
3 a-h **4 a, b, f, g**

The reactions are carried out by stirring an equimolar mixture of **1**, **2**, and ammonium acetate in ethanol at ambient temperature or under reflux. When the reaction is carried under reflux, the yield of **3** sometimes decreases and the yield of another product, identified as triarylpiperidine **4**, increases.

Similar treatment of 2-nitropropane or dimethyl malonate **5** with **1a** or *N,N'*-bis[2-hydroxybenzylidene]-2-hydroxyphenylmethanediamine **(1c)** gives the *N*-arylidenearylmethylamine **6**, which is assumed to be an intermediate in the formation of **4**.



1 **5**



6 a-d

Moreover, when methyl cyanoacetate is employed as the methylene reactant, crystalline 3,5-dicyano-4,6-diphenyl-5-

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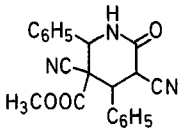
Table 1. Compounds **3**, **4**, **6**, and **7** prepared

Product No.	R^1	R^2	R^3	Ar	Method/ time [h]	Yield[a] [%]	m.p. [°C] (solvent)	Molecular formula[b]	
3a	NC—	NC—	—	C_6H_5	A/2	41	130–131° (C_2H_5OH)	$C_{24}H_{20}N_4$	(364.4)
3b	NC—	H_3COOC—	—	C_6H_5	C/4	31	150–151° (THF/CH_3OH)	$C_{25}H_{23}N_3O_2$	(397.4)
3c	NC—	H_2N—CO—	—	C_6H_5	A/10	58	180–181° (THF/CH_3OH)	$C_{24}H_{22}N_4O$	(382.5)
3d	H	O_2N—	—	C_6H_5	B/1	37	190–191° (THF/CH_3OH)	$C_{22}H_{21}N_3O_2$	(359.4)
3e	H_3C	O_2N—	—	C_6H_5	A/16	67	155–156° (THF/CH_3OH)	$C_{23}H_{23}N_3O_2$	(373.4)
3f	NC—	NC—	—	4-H_3C—C_6H_4	A/2	59	121–122° (CH_3OH)	$C_{27}H_{26}N_4$	(406.5)
3g	NC—	H_3COOC—	—	4-H_3C—C_6H_4	C/0.5	84	146–147° (CH_3OAc/*c*-C_6H_{12})	$C_{28}H_{29}N_3O_2$	(439.5)
3h	NC—	H_2N—CO—	—	4-H_3C—C_6H_4	C/20	71	158–159° (THF/CH_3OH)	$C_{27}H_{28}N_4O$	(424.5)
4a	NC—	NC—	—	C_6H_5	B/1.5	53	178–179° (THF)	$C_{27}H_{19}N_5$	(413.4)
4b	NC—	H_3COOC—	—	C_6H_5	B/2.5	66	195–196° (THF/CH_3OH)	$C_{29}H_{25}N_3O_4$	(479.5)
4c	NC—	NC—	—	4-H_3C—C_6H_4	B/0.5	60	159–160° (THF/CH_3OH)	$C_{30}H_{25}N_5$	(455.5)
4d	NC—	H_3COOC—	—	4-H_3C—C_6H_4	B/1	30	196–197° (THF/CH_3OH)	$C_{32}H_{31}N_3O_4$	(521.6)
6a	H_3C	H_3C	O_2N—	C_6H_5	A/10	80	104–105° (CH_3OH)	$C_{17}H_{18}N_2O_2$	(282.3)
6b	H	H_3COOC—	H_3COOC—	C_6H_5	A/30	89	95–96° (CH_3OH)	$C_{19}H_{19}NO_4$	(325.3)
6c	H_3C	H_3C	O_2N—	2-HO C_6H_4	B/2	79	164–165° (C_2H_5OH)	$C_{17}H_{18}N_2O_4$	(314.3)
6d	H	H_3COOC—	H_3COOC—	2-HO—C_6H_4	A/12	72	147–148° (THF/CH_3OH)	$C_{19}H_{19}NO_6$	(357.4)
7	—	—	—	—	—/48 h	6	174–175° (CH_3OH)	$C_{21}H_{17}N_3O_3$	(359.4)

[a] Yield of isolated product.

[b] The microanalysis showed the following maximum deviations from the calculated values: C, ±0.30; H, ±0.26; N, ±0.27.

methoxycarbonyl-2-oxopiperidine[9] (**7**) is formed after a few days in the filtrate from which **3** and **4** have been removed.



7

5,5-Dicyano-2,4,6-triphenylhexahydropyrimidine (3a) and 3,3,5,5-Tetracyano-2,4,6-triphenylpiperidine (4a); Typical Procedures:

Method A: A mixture of **1a** (2.98 g, 10 mmol), malononitrile (0.66 g, 10 mmol), and ammonium acetate (0.77 g, 10 mmol) in ethanol (15 ml) is magnetically stirred at ambient temperature for 2 h. The resultant white precipitate is collected by vacuum filtration, washed with ethanol (2 × 5 ml), and air-dried at room temperature. The crude material (3.2 g) is heated under reflux in ethanol (50 ml) for 30 min, the undissolved white substance (0.6 g) filtered off while the solution is hot, and recrystallized from 1:1 tetrahydrofuran/methanol to give **4a**; yield: 0.25 g (12%); m.p. 178–179 °C (tetrahydrofuran).

$C_{27}H_{19}N_5$	calc.	C 78.43	H 4.63	N 16.94
(413.4)	found	78.70	4.58	16.98

White, crystalline **3a** is isolated from the cooled filtrate; yield: 1.51 g (41%); m.p. 130–131 °C (ethanol).

$C_{24}H_{20}N_4$	calc.	C 79.09	H 5.53	N 15.38
(364.4)	found	79.39	5.54	15.56

Method B: The above mixture of **1a**, malononitrile, and ammonium acetate in ethanol is stirred and heated under reflux for 1.5 h. The resultant white precipitate is filtered while hot and recrystallized to give **4a**; yield: 1.1 g. A mixture (0.9 g) of **3a** and **4a** is obtained from the cooled filtrate.

5-Cyano-5-methoxycarbonyl-2,4,6-triphenylhexahydropyrimidine (3b), 3,5-Dicyano-3,5-dimethoxycarbonyl-2,4,6-triphenylpiperidine (4b), and 3,5-Dicyano-4,6-diphenyl-5-methoxycarbonyl-α-piperidone (7); Typical Procedure:

Method C: A mixture of **1a** (10 mmol) and methyl cyanoacetate (12 mmol) in methanol (5 ml) is stirred at ambient temperature for 4 h. The resultant white precipitate is collected by filtration, washed with water (10 ml) and methanol (10 ml), and dried in vacuo to give a mixture of **3b** and **4b** (9:1 molar ratio); yield: 3.3 g. Repeated recrystallization of the mixture from tetrahydrofuran/methanol gives pure **3b**; yield: 1.22 g (31%); m.p. 150–151 °C.

$C_{25}H_{23}N_3O_2$	calc.	C 75.54	H 5.83	N 10.57
(397.4)	found	75.45	5.73	10.45

The mixture of reaction filtrate and wash solution (~20 ml) is allowed to stand in a refrigerator for 2 days, and the resulting solid gives white crystals of **7**; yield: 0.13 g (6%); m.p. 174–175 °C (methanol).

$C_{21}H_{17}N_3O_3$	calc.	C 70.18	H 4.77	N 11.69
(359.4)	found	70.26	4.84	11.50

Method B: A mixture of **1a** (10 mmol), methyl cyanoacetate (12 mmol), and ammonium acetate (10 mmol) in methanol (10 ml) is heated under reflux with stirring for 2.5 h. The solid product which deposits during the reaction is collected, washed with water and methanol, and dried. Recrystallization from tetrahydrofuran/methanol gives **4b**; yield: 1.9 g (66%); m.p. 195–196 °C.

$C_{29}H_{25}N_3O_5$	calc.	C 72.63	H 5.26	N 8.76
(479.5)	found	72.45	5.23	8.89

***N*-Benzylidene-2-methyl-2-nitro-1-phenylpropanamine (6a); Typical Procedure:**

Method A: A mixture of **1a** (6.7 mmol), 2-nitropropane (10 mmol), and ammonium acetate (10 mmol) in methanol (5 ml) is stirred at ambient temperature for 10 h, and worked up as described above to give white crystalline **6a**; yield: 2.26 g (80%); m.p. 104–105 °C (methanol).

$C_{17}H_{18}N_2O_2$	calc.	C 72.32	H 6.43	N	9.92
(282.3)	found	72.11	6.69		10.06

***N*-(2-Hydroxybenzylidene)-1-(2-hydroxyphenyl)-2-methyl-2-nitropropanamine (6c); Typical Procedure:**

Method B: A mixture of *N*,*N*′-bis[2-hydroxybenzylidene]-2-hydroxyphenylmethanediamine (**1c**; 6.7 mmol), 2-nitropropane (10 mmol), and ammonium acetate (20 mmol) in methanol (5 ml) is heated under reflux for 2 h and then stirred at room temperature for ~1 h until no more precipitate is produced. The pale yellow

Table 2. Spectral Data of Compounds **3**, **4**, **6**, and **7**

Product No.	I.R. (nujol) ν [cm^{-1}]	^{1}H-N.M.R. ($CDCl_3$) δ [ppm]	M.S. (70 eV, 200 °C) *m/e* (per cent total inoization, %$\geq$15)
3a	3350, 3330 (NH); 2240 (C≡N); 1340; 745, 708 (δ_{C-H})	7.8–7.3 (m, 15 H_{arom}); 4.93 (t, 1 H, 999—N)[a]; 4.42 (d, 2 H, N—CH—C)[b]; 2.35 (t, 2 H, C—NH—C)[c]	194 (4), 154 (10), 127 (7), 106 (3), 105 (4), 104 (2), 103 (5), 77 (4), 51 (3), 50 (2)
3b	3350 (NH); 2240 (C≡N); 1731 (C=O); 1264 (C—O); 1137; 768, 707 (δ_{C-H})	7.8–7.2 (m, 15 H_{arom}); 5.01 (t, 1 H, N—CH—N)[a]; 4.64 (d, 2 H, N—CH—C)[b]; 3.33 (s, 3 H, $COOCH_3$); 2.23 (q, 2 H, C—NH—C)[c]	397 (M^+, 0.1), 195 (3), 194 (16), 187 (5), 186 (3), 156 (5), 128 (3), 116 (2), 105 (5), 104 (6), 77 (4)
3c	3490, 3340, 3320 (NH); 2230 (C≡N); 1687 (C=O); 1600; 763, 702 (δ_{C-H})	8.0–7.0 (m, 15 H_{arom}); 7.1 (br, 2 H, NH_2); 5.0 (br, 1 H, N—CH—N)[a]; 4.70 (d, 2 H, N—CH—C)[b]; 2.9 (br, 2 H, C—NH—C)[c,d]	194 (7), 172 (4), 171 (8), 128 (3), 105 (8), 104 (10), 102 (3), 78 (3), 77 (6)
3d	3325 (NH); 1545 (NO_2); 1315; 744, 700 (δ_{C-H})	7.8–7.2 (m, 15 H_{arom}); 5.06 (s, 1 H, N—CH—N)[a]; 4.6 (m, 3 H, N—CH—CH—CH); 2.1 (br, 2 H, C—NH—C)[c]	359 (M^+, 0.5), 358 (2), 28 (7), 194 (3), 106 (6), 105 (5), 104 (11), 103 (2), 91 (3), 78 (2), 77 (5), 51 (2)
3e	3340, 3250 (NH); 1530 (NO_2); 1115; 740, 698 (δ_{C-H})	7.8–7.2 (m, 15 H_{arom}); 5.05 (s, 1 H, N—CH—N)[a]; 4.44 (s, 2 H, N—CH—C)[b]; 2.1 (br, 2 H, C—NH—C)[c], 1.37 (s, 3 H, CH_3)	373 (M^+, 0.1); 372 (0.1); 268 (2), 222 (3), 194 (10), 117 (3), 115 (3), 105 (6), 104 (6), 91 (3), 77 (4)
3f	3320 (NH); 2240, 2220 (C≡N); 1605; 814, 786 (δ_{C-H})	7.8–7.0 (m, 12 H_{arom}); 4.88 (t, 1 H, N—CH—N)[a]; 4.38 (d, 2 H, N—CH—C)[b]; 2.34 (s, 9 H, CH_3); 2.3 (br, 2 H, C—NH—C)[c]	—
3g	3345 (NH); 2230 (C≡N); 1731 (C=O); 1511; 1270 (C—O); 821, 789 (δ_{C-H})	7.7–7.1 (m, 12 H_{arom}); 5.01 (t, 1 H, N—CH—N)[a]; 4.64 (d, 2 H, N—CH—C)[b]; 3.40 (s, 3 CH, $COOCH_3$); 2.37 (s, 3 H, CH_3); 2.33 (s, 6 H, CH_3); 2.18 (q, 2 H, C—NH—C)[c]	—
3h	3470, 3360, 3300 (NH); 2220 (C≡N); 1682 (C=O); 1600; 799, 786 (δ_{C-H})	7.7–7.1 (m, 12 H_{arom}); 5.5 (br, 1 H, NH); 5.2 (br, 1 H, NH); 5.02 (s, 1 H, N—CH—N)[a]; 4.71 (s, 2 H, N—CH—C)[b]; 2.38 (s, 3 H, CH_3); 2.33 (s, 6 H, CH_3); 2.1 (br, 2 H, C—NH—C)[c]	—
4a	3330 (NH); 2250 (vw, C≡N); 703 (δ_{C-H})	8.2–7.4 (m, 15 H_{arom}); 4.92 (s, 2 H, N—CH—C); 4.74 (s, 1 H, C—CH—C); 4.1 (br, 1 H, NH)[e]	—
4b	3330 (NH); 2230 (C≡N); 1741, 1730 (C=O); 1269, 1256 (C—O); 768, 705 (δ_{C-H})	7.8–7.2 (m, 15 H_{arom}); 4.59 (s, 2 H, N—CH—C); 4.14 (s, 1 H, C—CH—C); 3.30 (s, 6 H, $COOCH_3$); 2.4 (br, 1 H, NH)	—
4c	3350 (NH); 2250 (C≡N); 1605; 817, 733 (δ_{C-H})	7.9–7.2 (m, 12 H_{arom}); 4.39 (s, 2 H, N—CH—C); 3.61 (s, 1 H, C—CH—C); 2.40 (s, 9 H, CH_3); 1.6 (br, 1 H, NH)	—
4d	3350 (NH); 2250 (C≡N); 1740 (C=O) 1513; 1250 (C—O); 784, 720 (δ_{C-H})	7.7–7.1 (m, 12 H_{arom}); 4.56 (s, 2 H, N—CH—C); 4.11 (s, 1 H, C—CH—C); 3.55 (s, 6 H, $COOCH_3$); 2.34 (s, 6 H, CH_3); 2.30 (s, 3 H, CH_3); 2.1 (br, 1 H, NH)	—
6a	1648 (C=N); 1528 (NO_2); 1357; 760, 709 (δ_{C-H})	8.25 (s, 1 H, CH=N); 7.9–7.2 (m, 10 H_{arom}); 4.92 (s, 1 H, N—CH—C); 1.70 (s, 3 H, CH_3); 1.57 (s, 3 H, CH_3)	—
6b	1757, 1744 (C=O); 1632 (C=N); 1315, 1285 (C—O); 758, 694 (δ_{C-H})	8.39 (s, 1 H, CH=N); 7.9–7.2 (m, 10 H_{arom}); 5.08, 4.18 (2 d, 1 H each, N—CH—CH, J = 10.5 Hz); 3.66 (s, 3 H, $COOCH_3$); 3.50 (s, 3 H, $COOCH_3$)	—
6c	3320 (OH); 1629 (C=N); 1546 (NO_2); 1350; 771, 754 (δ_{C-H})	12.9 (br s, 1 H, OH); 10.0 (br, 1 H, OH); 8.53 (s, 1 H, CH=N); 7.6–6.7 (m, 8 H_{arom}); 5.51 (s, 1 H, N—CH—C); 1.66 (s, 3 H, CH_3); 1.56 (s, 3 H, CH_3)[d]	—
6d	1748, 1730 (C=O); 1629 (C=N); 1525, 1270 (C—O); 755, 742 (δ_{C-H})	13.5–10.3 (br, 2 H, OH); 8.56 (s, 1 H, CH=N); 7.5–6.7 (m, 8 H_{arom}); 5.38, 4.50 (2 d, 1 H each, N—CH—CH, J = 9.0 Hz); 3.64 (s, 3 H, $COOCH_3$); 3.54 (s, 3 H, $COOCH_3$)[d]	—
7	3280 (NH); 2230 (C≡N); 1750, 1667 (C=O); 1285, 1265 (C—O); 713, 706 (δ_{C-H})	8.92 (s, 1 H, NH); 7.4 (s, 5 H_{arom}); 7.3 (s, 5 H_{arom}); 5.37 (s, 1 H, N—CH—C); 4.83 (d, 1 H, CHCH—CN, J = 13.4 Hz); 4.50 (d, 1 H, CH—CH—CN, J = 13.4 Hz)[f]; 3.34 (s, 3 H, $COOCH_3$)	

[a] J = 6–7 Hz in the cases in which a triplet was observed, and a singlet was observed for ND—CH—ND (D_2O exchanged).
[b] J = 6–8 Hz in the cases in which a doublet was observed, and a singlet was observed for ND—CH—C (D_2O exchanged).
[c] J = 6–8 Hz in the cases in which a triplet or quartet was observed.
[d] DMSO-d_6 solution.
[e] Acetone-d_6 solution.
[f] A singlet was observed for CH—CD—CN (D_2O exchanged).

crystalline matter is worked up as described above to give **6c**; yield: 2.5 g (79%); m.p. 164–165 °C (ethanol).

$C_{17}H_{18}N_2O_4$	calc.	C 64.95	H 5.77	N 8.91
(314.3)	found	64.71	5.68	8.88

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[1] M. A. Laurent, *Justus Liebigs Ann. Chem.* **21**, 130 (1837).
[2] M. A. Laurent, *C. R. Acad. Sci., Paris* **19**, 353 (1844).
[3] D. H. Hunter, S. K. Shim, *J. Am. Chem. Soc.* **91**, 6202 (1969).
[4] T. Tsumaki et al., *J. Chem. Soc. Jpn.* **74**, 161 (1953).
[5] C. Shin, J. Yoshimura, *Tetrahedron Lett.* **1973**, 2615.
[6] C. Shin et al., *Nippon Kagaku Kaishi* **1976**, 1100; *C. A.* **86**, 5250 (1977).
[7] S. Kambe et al., *Synthesis* **1975**, 802.
[8] T. Takajo et al., *Synthesis* **1977**, 647.
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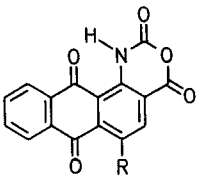
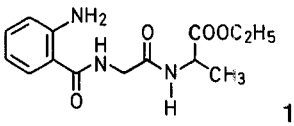
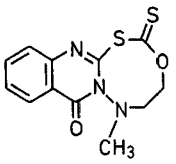
G. M. Coppola, *Synthesis* **1980** (7), 505–536;
The structures of compounds **43** (p. 511), **122** (p. 520), and **241** (p. 533) should be as shown below:



43

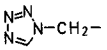


122



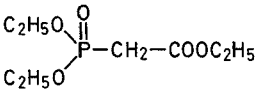
241

J. Diago-Meseguer, A. L. Palomo-Coll, J. R. Fernández-Lizarbe, A. Zugaza-Bilbao, *Synthesis* **1980** (7), 547–551;
The substitutent R^1 in Table 1 entries 2 and 20 and Table 2, entry 1 should be:



A more correct name for reagent **4** (as used in index) is **3,3'-(Chlorophosphinylidene)-bis[2-oxo-1,3-oxazolidine].**

J. Becher, *Synthesis* **1980** (8), 589–612;
The structure of compound **36** (p. 593) should be:



36

H. Paulsen, F. R. Heiker, J. Feldmann, K. Heyns, *Synthesis* **1980** (8), 636–638;
The correct name for reagent **1** is **3-methyl-2-selenoxo-2,3-dihydro-1,3-benzothiazole.**

G. Sosnovsky, J. A. Krogh, *Synthesis* **1980** (8), 654–656;
The first line of the text should read:
In 1978, Olah and Vankar reported[1] the conversion of

D. A. Walsh, *Synthesis* 1980 (9), 677–688;
The correct name for compound **39** (p. 680) is ***N'*-(2-Carboxyphenyl)-*N,N*-dimethylformamidine.**

M. A. Smoczkiewicz, J. Jasiczak, *Synthesis* **1980** (9), 739–740;
Compounds **2** should be named as **20,21-dioxo derivatives**; the name for compound **1a** (p. 740, Table 1) should be **21-hydroxy-3,20-dioxopregn-4-ene.**

Abstract 5878, *Synthesis* **1980** (9), 759;
The title should be: **Hydrofluorination, Halofluorination, and Nitrofluorination of Alkenes and Alkynes by Pyridinium Poly(Hydrogen Fluoride).**

Abstract 5885, *Synthesis* **1980** (9), 761;
The title should be: **Alkylation of *S*-Methyl 3-Oxoalkanethioates.**

T. Wagner-Jauregg, *Synthesis* **1980** (10), 769–798;
The name of compounds **552a** and **b** (p. 772) should be ***cis*-** and ***trans*-1-methyl-3-phenylindan.**
The heading for Table 2 (p. 784) should be:
Tabelle 2. Herstellung von 1-Arylacenaphthen-Derivaten durch Photocyclisierung von 1-(1-Arylethenyl)-naphthalin-Derivaten in Abwesenheit von Oxidationsmitteln[441].
The structures of the products in this Table should be of the type:

and not

The first paragraph on p. 785 (right-hand side) should read:
Aus den konjugierten 1,2-Diiminen **667** und Phenyl-isocyanaten oder Benzoyl-isocyanat entstehen criss-cross-Addukte (**668**, Schema **2.2.1.-E**)[480,481].

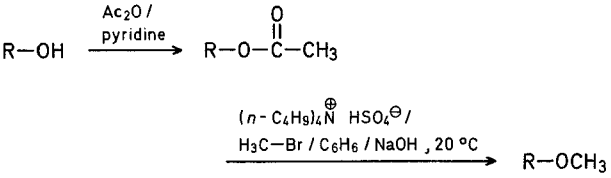
The last line on p. 794 should read:
und der Hydroxamsäuren[552] deutlich gesteigert[553].

Reference 441 (p. 796) should be:
[441] R. Lapougade, R. Koussini, H. Bouas-Laurent, *J. Am. Chem. Soc.* **99**, 7374 (1977).

H. Alper, D. E. Laycock, *Synthesis* **1980** (10), 799;
The last structure for R^1-R^2 in the Table should be:

T. Takajo, S. Kambe, *Synthesis* **1980** (10), 833–836;
Products designated as **4a,b,c,d** in Table 1 (p. 834) and Table 2 (p. 835) should be designated as **4a,b,f,g,** respectively.

P. Di Cesare, P. Duchaussoy, B. Gross, *Synthesis* **1980** (11), 953–954;
The first formula scheme (p. 954) should be:



Z. H. Kudzin, W. J. Stec, *Synthesis* **1980** (12), 1032–1034;
The heading for the first procedure (p. 1033) should be: **3-(Tris-[*t*-butoxy]silylthio)-propanal [3; R = (*t*-$C_4H_9O)_3Si$].**

R. E. Zipkin, N. R. Natale, I. M. Taffer, R. O. Hutchins, *Synthesis* **1980** (12), 1035–1036;
The substituents R^1-R^2 in the Table for product **4e** should be:
$-(CH_2)_2-C[=C(CH_3)_2]-CH_2-$

Abstract 5948, *Synthesis* **1980** (12), 1040;
Compounds **2** should be named **carboximidium dichlorides.**

Abstract 5963, *Synthesis* **1980** (12), 1045;
The title should be: **Acyl Fluorides, Chlorides, Bromides, and Iodides from Carboxylic Acids.**

Abstract 5973, *Synthesis* **1980** (12), 1047;
The title should be: **Acetoxylation-Arylselenylation of Alkenes.**