An Improved Synthesis of 1,3-Diphenyl-2-buten-1-ones (β-Methylchalcones)*

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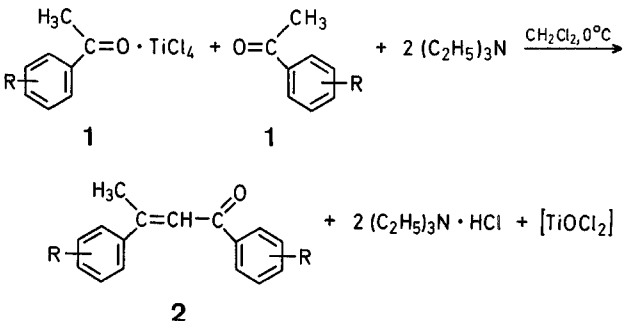
1,3-Diphenyl-2-buten-1-ones (**2**, β-methylchalcones) have been prepared by various condensation methods[1-7]. Some of these methods are of general applicability[1,7] but require long and rather complicated procedures. Moreover, only in few cases have details been given concerning the stereoisomerism of the products[3,8]. We report here a convenient and general synthesis of ketones **2** by the self-condensation of acetophenones (**1**) in the presence of titanium(IV) chloride and triethylamine. The use of titanium(IV) chloride and pyridine has been reported[9] for Knoevenagel condensations; however, only unidentified products were obtained from acetophenone.

The reaction conditions (reagents ratio and sequence of mixing, reaction temperature) have been found to be critical; good results were obtained by slowly adding a solution of ketone **1** (0.05 mol) and triethylamine (0.1 mol) in dichloromethane to a previously prepared cold solution of ketone **1** (0.05 mol) and titanium(IV) chloride (0.05 mol) in hexane/dichloromethane.



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The reaction in dichloromethane as solvent ultimately leads to a clear solution, from which the titanium complexes are easily removed by water extraction. By using pyridine instead of triethylamine, self-condensations of ketones **1a** and **1e** are slowed down while the desired reaction does not take place with ketone **1d**.

The reaction time required for optimum yields is ~20 h in most cases. After a reaction time of only 5 h, the yields are usually 10–15% lower than those reported in Table 1; for ketone **2d**, any appreciable increase in yield is not observed after a 5 h reaction time. The products were obtained either as mixtures of (*E*)- and (*Z*)-isomers **(2a, b, c)**, or exclusively as the (*E*)-isomer **(2d, e, f)** (see Table 1). The relative amount of (*Z*)-isomer could be increased in some cases **(2a, b, e)** by irradiation in solution: isomerization was unsuccessful for compounds **2d** and **2f**, and was not needed for product **2c**. Satisfactory separation of (*E*)- and (*Z*)-isomers was achieved for products **2a** (by preparative G.L.C. or T.L.C.)[10] and **2c** (by column chromatography); the spectra of (*E*)-**2b** and (*Z*)-**2b** were studied for different isomer mixtures.

(*E*)- and (*Z*)-isomers exhibit remarkable spectroscopic differences which allow the reported assignments of structure (Table 2). In fact, coplanarity of the aromatic rings causes a red-shift in the U.V. spectrum of the (*E*)-isomers as compared to that of the corresponding (*Z*)-isomers. Moreover, methyl protons bound to the ethylenic system are more deshielded in the (*E*)-isomers by the carbonyl groups than in the (*Z*)-isomers: differences of 0.2–0.3 ppm are observed in their ^{1}H-N.M.R. chemical shift. Differences between the U.V. spectra of stereoisomers had been previously pointed out for ketone **2a**[8,10].

The present method leads to the formation of minor amounts of by-products: 1,3,5-triphenylbenzene (besides **2a**); 1,3,5-tris[4-methoxyphenyl]-benzene (besides **2f**); polycondensation products (average mol weight 1300), 5-hydroxy-1-oxo-1,3,5-tris[4-nitrophenyl]-2-hexene, and 2-methyl-2,4,6-tris[4-nitrophenyl]-2*H*-pyran (besides **2e**).

All solvents and reagents used were reagent grade; melting points were taken by a Büchi Model 510 apparatus and are uncorrected. I.R. and U.V. spectra were recorded with Perkin-Elmer 457 and Cary 14 spectrophotometers, respectively, and ^{1}H-N.M.R. spectra with a Perkin-Elmer R-32 (90 MHz) spectrometer. Mass spectra were recorded on a Perkin-Elmer 270 Mass-spectrometer at 70 eV. Microanalyses were carried out with a Perkin-Elmer 240 Elemental Analyzer, and molecular weights were measured with a Hitachi-Perkin-Elmer model 115 apparatus (isopiestic method). G.L.C. analyses were carried out on a Perkin-Elmer 800 gas chromatograph equipped with a F.I.D. and with a Perkin-Elmer SIP 1 data system for peak integrations. A 2 m steel column (1/8 in. I.D.) packed with 5% OV-17 on Anakrom ABS (80–100 mesh) was used. For the components analysed by G.L.C., t'_R (retention time, min) and R.D. (response factor at the detector) are reported; octadecane was used as internal standard (I.S.). Silica gel (Merck 70–230 mesh) was used for chromatographic columns, and silica gel F_{254} (Merck) plates (0.25 thickness) for T.L.C. analyses. The irradiations were carried out with a Madza Lamp, 250 W, $\lambda = 365$ nm at 40 cm distance from the sample, in the air.

1,3-Diphenyl-2-buten-1-ones (2, β-Methylchalcones); General Procedure:

A solution of ketone **1** (0.05 mol) in anhydrous dichloromethane (100 ml) is added to a stirred solution of titanium(IV) chloride (9.46 g, 0.05 mol) in hexane (20 ml) at −5 to 0 °C; then a solution of ketone **1** (0.05 mol) and of anhydrous triethylamine (10.12 g, 0.1 mol) in anhydrous dichloromethane (100 ml) is added dropwise, the reaction temperature being kept at −5 °C (for ~1 h). The mixture is then allowed to reach room temperature, in the dark under stirring, and set aside overnight (~18 h). Then, cold water (100 ml) is added, the organic layer is separated, washed with water (5 × 50 ml). The aqueous washings are extracted with dichloromethane (2 × 25 ml) and the combined organic layers are dried with sodium sulfate. The solvent is removed in vacuo. Procedures for isolation of the products **2** and their isomers are reported in detail herein after.

Table 1. Ketones **2** Present in the Reaction Mixtures after 20 h

2[a]	R	Yield [%][b]	*E*/*Z* Ratio [%][c]	Residual **1** [%][b]
a	H	87	93/7	8
b	2-CH_3	82	88/12	7
c	2-Cl	88	70/30	10
d	2-NO_2	50	100/0	25
e	4-NO_2	70	100/0	11
f	4-OCH_3	63	100/0	17

[a] Previous synthetic methods: **2a**, Ref. [1,2,5,7]; **2c**, Ref. [7]; **2e**, Ref. [3,4]; **2f**, Ref. [3,6,14].

[b] Determined by G.L.C. with octadecane as internal standard, the response factors at the detector (R.D.) having been measured on the pure compounds. The yield of **2e** was calculated from the I.R. absorption of $\bar{\nu}_{C=O} = 1660$ cm^{-1} ($\varepsilon = 36.4$). Compound **2d** was in part isolated (30%) in dichloromethane and in part obtained as mixture with **1d**.

[c] Evaluated from G.L.C. area ratios.

1,3-Diphenyl-2-buten-1-one **(2a,** *Dypnone*); G.L.C. data (t'_R and R.D.) for the components of the residue: **1a** (2.08, 1), I.S. (7.37, –), (*Z*)-**2a** (12.14, 1.1), (*E*)-**2a** (14.79, 1.1) [G.L.C. conditions: Injector 200 °C, column 150–235 °C at 5 °C/min, N_2 20 ml/min]. Distillation (6 cm Vigreux column) of the crude product gave **2a** (*E*/*Z* = 93/7); yield: 7.10 g (64%); purity: 98%; b.p. 145–150 °C/1.5 torr. From a fraction (0.5 g) at b.p. 200–210 °C/1 torr, 1,3,5-triphenylbenzene was isolated by crystallization from ethanol at −10 °C. Separation and spectroscopic data of (*E*)-**2a** and (*Z*)-**2a** have been previously reported[8,10].

1,3-Bis[2-methylphenyl]-2-buten-1-one **(2b);** G.L.C. data (t'_R, R.D.): **1b** (3.77, 1.1), I.S. (9.09, –), (*Z*)-**2b** (24.16, 1.23), (*E*)-**2b** (33.56, 1.23) [G.L.C. conditions: Injector 220 °C, column 150–220 °C at 10 °C/min, N_2 10 ml/min]. Mass spectra of both isomers gave molecular peaks at $m/e = 250$ and parent peaks at $m/e = 235$ ($M^+ - CH_3$). T.L.C. with xylene/acetone (5:1) gave the following R_f values: **1b** (0.78), (*Z*)-**2b** (0.85), (*E*)-**2b** (0.90), and an unknown product (0.6). Distillation of the crude product gave a fraction (10 g, purity 80%) which was rectified to give **2b** (*E*/*Z* = 80/20); yield: 7.4 g (59%); purity: 99%; b.p. 155–159 °C/1 torr.

$C_{18}H_{18}O$	calc.	C 86.36	H 7.25
(250.4)	found	86.20	7.11

Mol Weight (in $CHCl_3$): 257.

The same *E*/*Z* ratio was determined from the ^{1}H-N.M.R. spectra (β-methyl signals). Irradiation for 48 h of the $CDCl_3$ solution used for N.M.R. converted the *E* isomer into the *Z* isomer until the ratio *E*/*Z* was 25/75. U.V. spectra were taken in dichloromethane on the above isomers mixture (*E*/*Z* = 80/20) and after 2 h irradiation (*E*/*Z* = 29/71). The ε values of the two isomers (*Z*)-**2b** and (*E*)-**2b** (reported in Table 2) were evaluated from the optical densities of the two above mixtures at 243 and 263 nm.

1,3-Bis[2-chlorophenyl]-2-buten-1-one **(2c);** G.L.C. data (t'_R R.D.): **1c** (3.41, 1.5), I.S. (7.21, –), (*Z*)-**2c** (28.94, 1.6), (*E*)-**2c** (35.80, 1.6) [G.L.C. conditions: Injector 230 °C, column 150–220 °C at 10 °C/min, N_2 20 ml/min]. The brown oily residue afforded **2c** (*E*/*Z* = 70/30) by simple distillation; yield: 9.2 g (66%); purity: 99%;

Table 2. Characteristic Data of the *E* and *Z* Isomers of Ketones **2**

2	R	Yield of isolated *E/Z* mixture [%]	b.p./torr or m.p.	Isomer	I.R.[c] $\tilde{\nu}_{C=O}$ [cm^{-1}]	U.V.[d] λ_{max} [nm] (log ε)	^{1}H-N.M.R. ($CDCl_3$/TMS) δ [ppm] β-CH_3	H_{olefin}
a[a]	H	64	145–150 °C/1.5	*E*	1660	296 (4.21), 265 (4.03)	2.55 (d)	7.02 (q)
			(*E/Z* = 93/7)	*Z*	1660	251 (4.14), 280 (4.06)	2.18 (d)	6.50 (q)
b	2-CH_3	59	156–159 °C/1	*E*	1665	263 (4.22),	2.45 (d)	6.45 (q)
			(*E/Z* = 80/20)	*Z*	1670	243 (4.05)	2.08 (d)	6.58 (q)
c	2-Cl	66	170–172 °C/0.5	*E*	1670	266 (4.07)	2.51 (d)	6.56 (q)
			(*E/Z* = 70/30)	*Z*	1670, 1655	240 (4.14)	2.18 (d)	6.62 (q)[f]
d	2-NO_2	12	175 °C	*E*	1695, 1660	257 (4.23)	2.71 (s)	
e[b]	4-NO_2	41	154 °C	*E*	1660	280 (4.31), 308 (4.40)	2.60 (d)	7.20 (q)
			178 °C	*Z*	1670	264 (4.35)	2.40 (d)	7.00 (q)
f[c]	4-OCH_3	41	95 °C	*E*	1645	227 (4.23), 324 (4.37)	2.61 (d)	7.16 (q)

[a] Previously characterized, Ref. [8,10].

[b] Ref. [3], m.p. 153 °C; I.R. (KBr); $\tilde{\nu}_{C=O}$ = 1660 cm^{-1}; ^{1}H-N.M.R. ($CDCl_3$): δ_{CH_3} = 2.70 ppm.

[c] Ref. [12], m.p. 83 °C; Ref. [13], b.p. 190 °C/0.1 torr; I.R. (KBr): $\tilde{\nu}_{C=O}$ = 1652 cm^{-1}; Ref. [3], ^{1}H-N.M.R. ($CDCl_3$): δ_{CH_3} = 2.51 ppm.

[d] **2a** in ethanol; **2b, c, f** in dichloromethane; **2d, e** in chloroform.

[e] **2a, b, c,** liquid phase; **2d, e, f,** KBr pellets.

[f] Signals covered by signals of aromatic protons; J_{H,CH_3} = 1.2–1.5 Hz, identical for each couple of isomers.

b.p. 169–172°/0.5 torr. T.L.C. (xylene) of this product gave (R_f): (*Z*)-**2c** (0.34), (*E*)-**2c** (0.56), and an unknown product (0.92), fluorescent at λ = 356 nm. A part of this fraction (1.6 g) was chromatographed on a silica gel column (xylene) to afford the (*E*)-isomer (0.4 g, 25%) and the (*Z*)-isomer (0.3 g, 19%), both as pale yellow oils. The isomers appeared to be stable if stored in the dark.

$C_{16}H_{12}Cl_2O$	calc.	C 66.00	H 4.15	Cl 24.35
(291.2)	*E* found	65.85	4.01	24.08
	Z found	66.11	4.10	24.60

Mol Weight (in $CHCl_3$): *E*, 296; *Z*, 294.

Irradiation of dilute or concentrated solutions of the pure isomers in chloroform did not lead to interconversion but produced unidentified brown products.

1,3-Bis[2-nitrophenyl]-2-buten-1-one [(*E*)-**2d**]; by G.L.C. analysis of the residue of preparation only **1d** (24%) (t_R' 6.15, R.D. 1.80), was detected (Injection at 200 °C, column 170 °C, N_2 15 ml/min). T.L.C. (xylene/acetone 5/1) of the crude product gave (R_f): (*E*)-**2d** (0.55), **1d** (0.48), and unknown products with R_f values: 0.28, 0.19, 0.13, 0. By crystallization of the crude product from acetone (30 ml) at −10 °C, (*E*)-**2d** (2.8 g, 18%) was obtained as a yellow solid. By addition of diethyl ether (250 ml) to the mother liquor, a green polycondensation product (0.4 g, mol weight 1290) was isolated. The solvent was removed from the solution, and the residue was purified by column chromatography on silica gel (benzene/dichloromethane 7/3): compounds **1d** and (*E*)-**2d** were obtained as a mixture, wherefrom the ketone (*E*)-**2d** was in part separated by crystallization from ether/hexane (7/4) at −10 °C; yield: 1.9 g (12%); m.p. 175.5–176 °C (toluene or acetone).

$C_{16}H_{12}N_2O_5$	calc.	C 61.54	H 3.87	N 8.97
(312.3)	found	61.33	3.95	9.16

Mol Weight (in $CHCl_3$): 317.

The mother solution containing **1d** (3.2 g, as determined by G.L.C. analysis) and (*E*)-**2d** was concentrated to give 6.8 g of a mixture of **1d** and (*E*)-**2d**.

1,3-Bis[4-nitrophenyl]-2-buten-1-one **(2e);** by G.L.C. analysis (Injector 170 °C, column 90–170 °C at 5 °C/min, N_2 15 ml/min) of the reaction mixture only **1e** (11%, t_R' = 18.24, R.D. 1.82) was detected. By T.L.C. (xylene/acetone 9/1), the following compounds were found (R_f): (*Z*)-**2e** (0.59 traces), **1e** (0.64), (*E*)-**2e** (0.81), and additional unknown products with the following R_f values: 0.71, 0.16, 0. The amounts of **1e** (11%) and (*E*)-**2e** (70%) were calculated from the I.R. absorptions in dichloromethane at 1710 cm^{-1} (**1e**, ε = 36.4) and 1660 cm^{-1} [(*E*)-**2e**, ε = 33.0] (concentrations below 0.15 molar and 0.07 molar, respectively). From the reaction solution, product (*E*)-**2e** separated on concentration; m.p. 153–154 °C (Ref. [4], m.p. 153 °C), recrystallized twice from benzene; yield: 6.4 g (41%). Sun-irradiation for 20 h of a saturated solution of (*E*)-**2e** in chloroform, produced (*Z*)-**2e** which was separated at −5 °C as a pale yellow solid; m.p. 177–179 °C (washed with cold chloroform).

$C_{16}H_{12}N_2O_5$	calc.	C 61.54	H 3.87	N 8.97
(312.2)	found	61.30	3.94	8.76

Mol Weight (in $CHCl_3$): 315.

During the crystallization of (*E*)-**2e**, an insoluble product (T.L.C., R_f = 0.16; 0.1 g) was isolated and identified as 5-hydroxy-1-oxo-1,3,5-tris[4-nitrophenyl]-2-hexene; m.p. 201–202 °C (methanol).

$C_{24}H_{19}N_3O_8$	calc.	C 60.38	H 4.01	N 8.80
(477.4)	found	60.29	4.17	8.69

Mol Weight (in butanone): 468.

I.R. (KBr): $\tilde{\nu}_{O-H}$ = 3580; $\tilde{\nu}_{C=O}$ = 1670 cm^{-1}.

U.V. ($CHCl_3$): λ_{max} = 275 nm (log ε = 4.49).

^{1}H-N.M.R. (DMSO-d_6): δ = 1.54 (s, 3H, CH_3); 3.22 (s, 2H, CH_2); 5.55 (s, 1H, OH); 6.93 (s, 1H_{olefin}); 7.10–8.37 ppm (m, 12H_{arom}).

From the mother liquor (dichloromethane) of the reaction mixture, the solvent was removed and the residue was crystallized from chloroform: the product (R_f 0.71; 0.5 g) was identified as 2-methyl-2,4,6-tris[4-nitrophenyl]-2*H*-pyran.

$C_{24}H_{17}N_3O_7$	calc.	C 62.75	H 3.73	N 9.15
(459.5)	found	62.72	3.70	9.37

Mol Weight (in $CHCl_3$): 461.

U.V. (CH_2Cl_2): λ_{max} = 274 (log ε = 4.40); 370 nm (4.15).

^{1}H-N.M.R. (DMSO-d_6): δ = 1.8 (s, 3H, CH_3); 6.73 (d, 1H_{olefin}); 6.96 (d, 1H_{olefin}); 8.05 ppm (m, 12H_{arom}).

1,3-Bis[4-methoxyphenyl]-2-buten-1-one **(2f);** G.L.C. data (t_R', R.D.): **1f** (3.20, 1.40), I.S. (5.79, –), (*E*)-**2f** (23.89, 1.77) [G.L.C. conditions: Injector 240 °C; column 190 °C for 7 min, then 190–270 °C at 40 °C/min; N_2 20 ml/min]. By T.L.C. (xylene/acetone 9/1) were detected (R_f): 1,3,5-tris[4-methoxyphenyl]-benzene (0.80), (*E*)-**2f** (0.66), **1f** (0.55), and several products at lower R_f values.

The orange semisolid residue was treated with boiling ether (250 ml) in order to obtain ketone (*E*)-**2f** (1.4 g) which is almost insoluble. The solution was filtered through silica gel. Then, by fraction-

ated crystallizations, more product (*E*)-**2f** was recovered (3.0 g). By column chromatography (silica gel and dichloromethane) of the residue from the mother liquors afforded 1,3,5-tris[4-methoxyphenyl]benzene (0.6 g; m.p. 144–145 °C, Ref. [4], m.p. 143 °C) and further (*E*)-**2f** (2 g). The crude (*E*)-**2f** product (total yield: 6.4 g, 46%) was recrystallized from ethanol (120 ml): m.p. 95 °C (Ref. [12], m.p. 83 °C). Irradiation of $CDCl_3$ or dichloromethane solutions of (*E*)-**2f** did not produce the isomer (*Z*)-**2f**, while the solution became brown.

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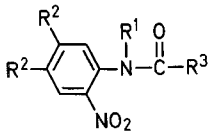
Errata and Addenda 1980

V. N. R. Pillai, *Synthesis* **1980** (1), 1–26;
The structure of compound **86** (p. 12) should be:



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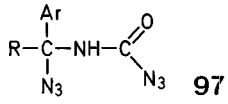
V. I. Cohen, *Synthesis* **1980** (1), 60–63;
The alternative name (in brackets) for compounds **1** (p. 62, first experimental procedure) should be ***S*-Methylpseudothiourea Hydroiodides.**

J. R. Mahajan, H. C. de Araújo, *Synthesis* **1980** (1), 64–66;
The authors have erroneously stated that "exaltolide" is a trivial name for pentadecanolide. In fact "exaltolide" is a trademark registered in the name of Firmenich SA, Geneva and should be designated as Exaltolide®.

V. I. Gorbatenko, L. I. Samarai, *Synthesis* **1980** (2), 85–110;
The structure of compound **97** (p. 99) should be:



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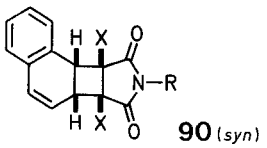
M. Mikołajczyk, P. Bałczewski, S. Grzejszczak, *Synthesis* **1980** (2), 127–129;
The correct name for compound **5a** (first procedure, p. 129) is **Diethyl 1-Phenylthioethanephosphonate.**

G. A. Olah, Y. D. Vankar, M. Arvanaghi, *Synthesis* **1980** (2), 141–142;
The correct name for compound **4** is ***N*-(Chlorosulfonyl)-dimethylsulfilimine.**

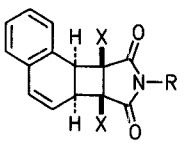
Abstract 5692, *Synthesis* **1980** (2), 159;
The title should be: **Phenols from Aryl Ethyl Ethers.**

Abstract 5698, *Synthesis* **1980** (2) 161;
The title should be: **Enals and Enones from Ketones.**

T. Wagner-Jauregg, *Synthesis* **1980** (3), 165–214;
The structures of compounds **90** (p. 175) should be:



90 (*syn*)



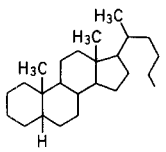
90 (*anti*)

The correct name for compound **251** (p. 188) is **2*H*-Cyclohepta[*gh*]pyrrolizin-Derivat.**

Abstract 5724, *Synthesis* **1980** (3), 254;
The title should be: **Carbamates, Thiocarbamates, and Carbonates from Alcohols or Thiols.**
The first line under the formula scheme should be: Y = O, S.

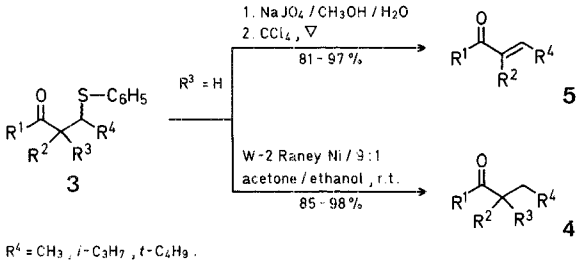
Abstract 5728, *Synthesis* **1980** (3), 256;
The title (and name for compound **3**) should be: ***N*-Sulphenylimines Derived from Amino Acids.**

C. R. Harrison, P. Hodge, *Synthesis* **1980** (4), 299–301;
The 3rd group in the Table, part B (p. 300) should have the structure:



Abstract 5745, *Synthesis* **1980** (4), 334;
The title should be: **Stereocontrolled *cis*-Addition of Organocopper Reagents to 2-Alkynals, 1-Alkynyl Ketones, 2-Alkynoic Acids, and 2-Alkynoic Esters.**

Abstract 5752, *Synthesis* **1980** (4), 336;
The title should be: **α-Alkylation and α-Alkylidenation of Carbonyl Compounds.**
The formula scheme for the conversion **3**→**4** or **5** should be:



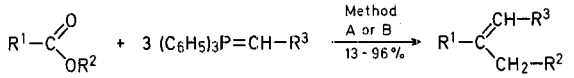
3 **5** **4**

R^4 = CH_3, *i*-C_3H_7, *t*-C_4H_9.

Abstract 5770, *Synthesis* **1980** (4), 342;
The title should be: **Claisen Rearrangement of Ketene Allyl Ethyl Acetals.**

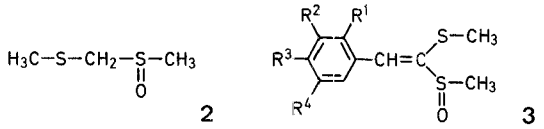
M. A. Alkhader, R. K. Smalley, B. Mohajerani, *Synthesis* **1980** (5), 381–383;
The correct name for compound **6** is **Indazolo[3,2-*b*]naphtho[2,3-*d*]-[1,3] oxazin-6-one.**

Abstract 5782, *Synthesis* **1980** (5), 418;
The formula scheme should be:



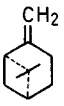
1 **2** **3**

Abstract 5799, *Synthesis* **1980** (5), 424;
The structures of compounds **2** and **3** should be:



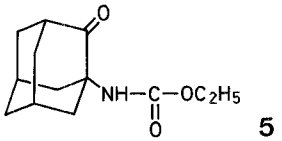
2 **3**

L. M. Harwood, M. Julia, *Synthesis* **1980** (6), 456–457;
The structure of compound (−)-**7** should be:



(−)-**7**

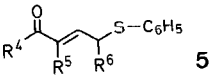
T. Sasaki, S. Eguchi, T. Okano, *Synthesis* **1980** (6), 472–475;
The structure of compound **5** should be:



5

Abstract 5804, *Synthesis* **1980** (6), 498;
The title should be: **Allylic Funtionalisation of Exomethylene Compounds.**

Abstract 5817, *Synthesis* **1980** (6), 503;
The structure of compound **5** should be:



5