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Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/lsyc20

The Reaction of 1-Aryl-2-(4-nitrophenylsulfonyl)ethanones with α,β-Unsaturated Esters

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Published online: 22 Aug 2006.

To cite this article: Yue Guoren & Zhang Zheng (1997) The Reaction of 1-Aryl-2-(4-nitrophenylsulfonyl)ethanones with α,β-Unsaturated Esters, Synthetic Communications: An International Journal for Rapid Communication of Synthetic Organic Chemistry, 27:8, 1455-1463, DOI: 10.1080/00397919708006077

To link to this article: http://dx.doi.org/10.1080/00397919708006077

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SYNTHETIC COMMUNICATIONS, 27(9), 1455-1463 (1997)

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THE REACTION OF 1-ARYL-2-(4-NITROPHENYLSULFONYL)ETHANONES WITH α,β-UNSATURATED ESTERS

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Abstracts; 1-Aryl-2-(4-nitrophenylsulfonyl)ethanones (**1**) reacted with one equiv. of α,β-unsaturated esters at 60 °C to afford the tandem addition-rearrangement products (**3**), while at 75 °C **1a** reacted with two equiv. of methyl acrylate and gave an unexpected addition-rearrangement-addition-annulation product which is dimethyl 4-hydroxyl-1-(4-nitrophenyl)-4-phenyl-1,3-cyclohexyldicarboxylate (**4**). A mechanism is suggested.

Many reports[1-5] have appeared on using sulfur compounds for organic synthesis, since Seebach and Corey[6] reported the utilization of 1,3-dithiane for the preparation of aldehydes and ketones. In general, an organic sulfur compound can be used as a synthetic agent according to the following scheme: (1) the formation of a C-C bond between a sulfur agent and an organic compound to afford an intermediate and (2) the intermediate's desulfurization accompanied with the creation of a new functional group.

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A novel type of tandem addition-rearrangement reaction was found when *p*-nitrophenylsulfonylacetonitrile[7], *p*-nitrophenylsulfonylacetates[8] or 1-nitro-4-(4-nitrophenylsulfonylmethyl)benzene[9] reacted with α,β-unsaturated esters in the system of K_2CO_3/DFM/DEBAR. The formation of two C-C bonds and the elimination of sulfur dioxide proceeded at the same time. But when other arylsulfonyl compounds such as p-methylphenylsulfonylacetates[10], p-methylphenylsulfonylacetonitriles[11], and p-methylphenylsulfonylmethylbenzenes[12] were used, only normal Michael addition was observed. As a new type of reaction, it should or could be applied widely. On this consideration, we have investigated the reaction of 1-aryl-2-(4-nitrophenylsulfonyl)ethanones (**1**) with α,β-unsaturated esters (**2**). Although the preparation[13] and physical properties such as spectra data[14,15] of **1** have appeared, the reaction has not been reported. Michael addition[16] where the normal Michael addition products were obtained, alkylations[17] and Knoevenagel reaction[18] of other arylsulfonylethanones such as 1-(4-methylphenylsulfonyl)-2-phenylethanone have appeared in the literatures. Our studies show that, under solid-liquid PTC conditions, **1** reacted with one equiv. of α,β-unsaturated esters at 60 °C to afford the tandem addition-rearrangement products (Scheme 1).

While at a higher temperature of 75 °C **1a** reacted with two equiv. of methyl acrylate and gave an unexpected addition-rearrangement-addition-annulation product which is dimethyl 4-hydroxyl-1-(4-nitrophenyl)-4-phenyl-1,3-cyclohexyldicarboxylate (**4**) (scheme2)

Results and Discussion

1. Under solid-liquid PTC conditions, **1** reacted with acrylates and methacrylates and addition-rearrangement products were obtained; but probably due to larger steric hindrances **1** didn't react with itaconate and cinnamate and of course no addition-rearrangement products were obtained. The data from our studies (Table 1) showed that the reaction of **1** with acrylates is faster than that with methacrylates.

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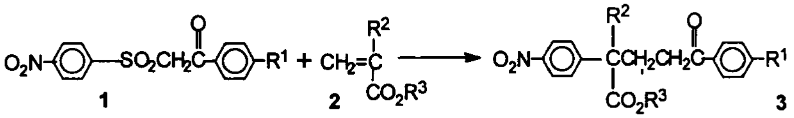
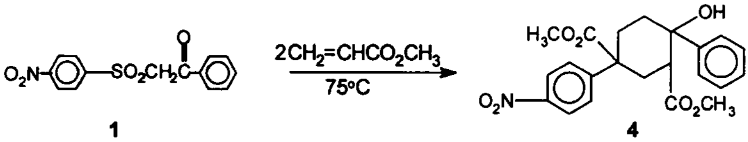


1a; R^1 =H,	2a; R^2 =H,	R^3 =CH_3,	3aa; R^1 =H,	R^2 =H,	R^3 =CH_3
1a; R^1 =H,	2c; R^2 =CH_3,	R^3 =CH_3,	3ac; R^1 =H,	R^2 =CH_3,	R^3 =CH_3
1a; R^1 =H,	2d; R^2 =CH_3,	R^3 =C_2H_5,	3ad; R^1 =H,	R^2 =CH_3,	R^3 =C_2H_5
1b; R^1 =CH_3,	2a; R^2 =H,	R^3 =CH_3,	3ba; R^1 =CH_3,	R^2 =H,	R^3 =CH_3
1b; R^1 =CH_3,	2b; R^2 =H,	R^3 =C_2H_5,	3bb; R^1 =CH_3,	R^2 =H,	R^3 =C_2H_5
1b; R^1 =CH_3,	2c; R^2 =CH_3,	R^3 =CH_3,	3bc; R^1 =CH_3,	R^2 =CH_3,	R^3 =CH_3
1b; R^1 =CH_3,	2d; R^2 =CH_3,	R^3 =C_2H_5,	3bd; R^1 =CH_3,	R^2 =CH_3,	R^3 =C_2H_5
1c; R^1 =Cl,	2a; R^2 =H,	R^3 =CH_3,	3ca; R^1 =Cl,	R^2 =H,	R^3 =CH_3
1c; R^1 =Cl,	2c; R^2 =CH_3,	R^3 =CH_3,	3cc; R^1 =Cl,	R^2 =CH_3,	R^3 =CH_3
1c; R^1 =Cl,	2d; R^2 =CH_3,	R^3 =C_2H_5,	3cd; R^1 =Cl,	R^2 =CH_3,	R^3 =C_2H_5

Scheme 1



Scheme 2

2. Normal Michael addition products were not obtained under the same conditions, which suggests that rearrangement is faster than addition and controlling the outcome of the reaction.

3. In the presence of K_2CO_3 /DFM /DEBAR and at a temperature of 60 °C, **1a** reacted with methyl acrylate (**2a**) to give a desired addition-rearrangement compound (**3aa**). However, at a higher temperature of 75 °C, the unexpected product (**4**) was obtained. On the basis of the spectra analytical data of **4**, we assert that the molecular structure is as follows;

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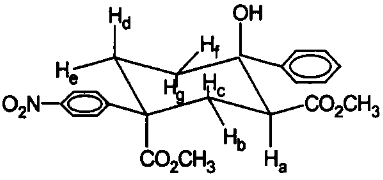
Table 1 Physical properties and data of elementary analysis and MS for products

Prod No.	Formula (M.W)	React. Time (h)	Yield (%)	M.P (°C)	Elementary Anal. found (cald.)%			M/z (%)
					C	H	N	
3aa	$C_{18}H_{17}NO_5$ (327.36)	8	92	108-109	66.20 (66.04)	5.25 (5.24)	4.21 (4.28)	327(M^+,0.3),296(3.8),295(9.9),268(16.2),267(54.9),133(6.7),120(78.7),105(100),77(67.8)
3ac	$C_{19}H_{19}NO_5$ (341.39)	60	75	110-111	66.64 (66.84)	5.71 (5.62)	3.82 (4.10)	341(M^+,1.5),311(2.1),282(4.2),252(7.4),133(13.4),120(6.7),105(100),77(40.2).
3ad	$C_{20}H_{21}NO_5$ (355.42)	60	70	76-77	67.67 (67.58)	5.98 (5.97)	3.88 (3.94)	355(M^+,0.2),339(1.4),325(4.8),310(1.9),282(31.8),252(21.5),133(44.4),120(28.1),105(100),77(95.6).
3ba	$C_{19}H_{19}NO_5$ (341.39)	2	96	114-115	67.09 (66.84)	5.69 (5.62)	4.06 (4.10)	341(M^+,1.0),311(0.4),310(2.4),281(16.4),147(2.7),134(64.5),119(100).
3bb	$C_{20}H_{21}NO_5$ (355.42)	1.5	98	76-77	67.82 (67.58)	6.02 (5.97)	3.81 (3.94)	355(M^+,1.1),310(3.2),281(17.6),147(3.7),134(58.8),119(100),91(41.9)
3bc	$C_{20}H_{21}NO_5$ (355.42)	15	85	108-109	67.60 (67.58)	6.02 (5.97)	3.90 (3.94)	355(M^+,1.5),296(2.5),147(10.7),134(7.4),119(100),91(26.2).
3bd	$C_{21}H_{23}NO_5$ (369.45)	24	87	66-67	68.50 (68.27)	6.38 (6.29)	3.77 (3.79)	369(M^+,2.3),339(0.3),296(15.9),147(44.6),134(22.2)119(100),91(79.0)
3ca	$C_{18}H_{16}ClNO_5$ (361.80)	1.5	92	99-100	60.02 (59.75)	4.63 (4.47)	3.80 (3.87)	363,361(M^+,0.3,0.7),331,329(3.3,8.3),304,302(3.8,11.5),156,154(21.5,67.4),141,139(41,100),113,111(12.2, 37.5)
3cc	$C_{19}H_{18}ClNO_5$ (375.83)	55	78	98-99	60.55 (60.72)	4.90 (4.84)	3.55 (3.73)	377,375(M^+,0.1,0.4),318,316(5.1,14.3),236(4.7),209(14.7),169,167(10.5,32.5),156,154(5.9,17.7),141,139(69.7,100),113,111(12.8, 39.2)
3cd	$C_{20}H_{20}ClNO_5$ (389.86)	50	77	58-59	61.68 (61.61)	5.19 (5.18)	3.56 (3.59)	391,389(M^+,0.2,0.7),318,316(5.3,15.3),264(30.0),223(13.2)169,167(3.9,14.1),156,154(5.7,17.7),141,139(48.6,100),113,111(11.4, 33.5)
4	$C_{22}H_{23}NO_5$ (413.46)	15	85	148-149	63.99 (63.90)	5.76 (5.62)	3.14 (3.39)	413(M^+,0.2),397(0.1),383(2.6),381(6.2),327(13.6),281(46.6),268(8.7),267(49.1),249(39.0),178(12.8),133(66.9),120(80.2)105(100),77(40.4)

Except for product 4 given at 75°C, the other products(3) were obtained at 60°C.



Besides the addition-rearrangement reaction, the addition-rearrangement-addition reaction was also reported in the literature[9]. Depending on our experimental results, a reaction mechanism via addition, rearrangement, addition, and then annulation is suggested (Scheme 3).

Experimental Section

Unless noted materials were obtained from commercial supplies and used without further purification. Melting points were determined with a Yancco MP-500 apparatus and uncorrected. Microanalysis were performed with a Perkin-Elmer 240C instrument. IR spectra were recorded on a Nicolet 170SX FT-IR spectrometer (in KBr pellet). ^{1}HNMR spectra were determined with a JEOL PMX 60 SI spectrometer or a AM 500 spectrometer using TMS as an internal standard(chemical shifts in d values). Mass spectra were obtained from a ZAB-HS spectrometer at 70ev (EI).

1; 2-(4-Nitrophenylsulfonyl)-1-phenylethanone (**1a**) was prepared by the method described in the literature[16].

1.40 g (5 mmol) of 1-phenyl-2-(4-nitrophenylthio)ethanone was dissolved in 10 mL of glacial acetic acid at 60 °C. 1.84 g (12 mmol) of sodium perborate tetrahydrate was added. The mixture was continuously stirred at 60 °C for 2 hours. The solvent was removed under reduced pressure. The residue was treated with water. The precipitated product was collected, washed with water and recrys-tallized from dilute acetone to give 1.35 g (89 %) of analytical sample of **1a**. M.P. 136-137 °C. (Lit.[9] M.P.129 °C). IR (in KBr pellet): 1675 (CO), 1533, 1354 (NO_2), 1313, 1155 (SO_2) cm^{-1}. ^{1}HNMR (CD_3COCD_3): 8.40 (2H, d, Ar-H,

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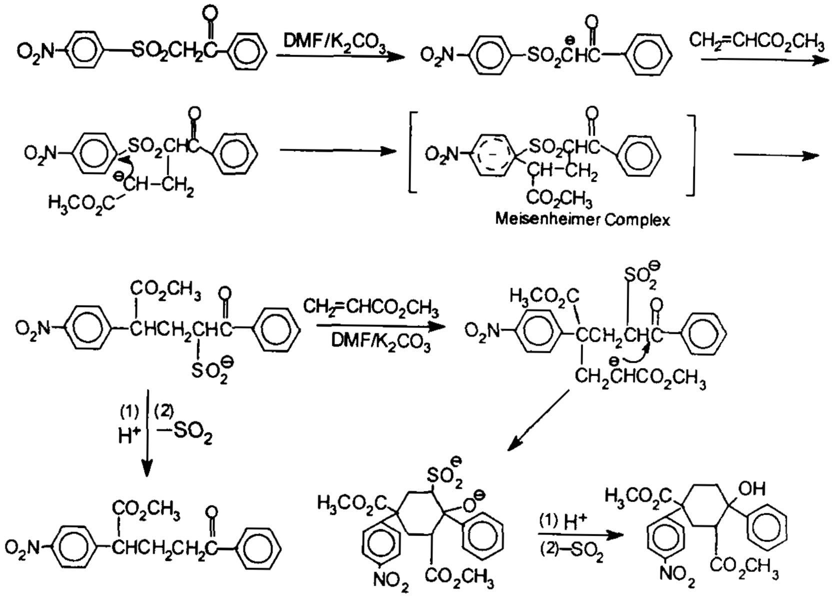

Scheme 3

J=9.0 Hz), 8.16 (2H, d, Ar-H, J=9.0Hz), 8.06-7.30 (5H, m, Ar-H), 3.13 (2H, s, CH_2) ppm.

By the similar method, compounds **1b** and **1c** were synthesized.

The yield of **1b** was 94 %, M.P. 159-160 °C. (lit[4] M.P. 155 °C). IR: 1688 (CO), 1538, 1350 (NO_2), 1305, 1146 (SO_2) cm^{-1}; ^{1}HNMR ($CDCl_3$): 8.30 (2H, d, Ar-H, J=8.5Hz), 8.00 (2H, d, Ar-H, J=8.5Hz), 7.72 (2H, d, Ar-H, J=8.0Hz), 7.20 (2H, d, Ar-H, J=8.0Hz), 4.72 (2H, s, CH_2), 2.44 (3H, s, Ar-CH_3) ppm.

The yield of **1c** was 91 %, M.P. 180-181 °C. (lit[4] M.P.179 °C). IR: 1677 (CO), 1531, 1349 (NO_2), 1300, 1154 (SO_2) cm^{-1}; ^{1}HNMR ($CDCl_3$): 8.36 (2H, d, Ar-H, J=9.0Hz), 8.03 (2H, d, Ar-H, J=9.0Hz), 7.85 (2H, d, Ar-H, J=8.5Hz), 7.20 (2H, d, Ar-H, J=8.5Hz), 4.70(2H, s, CH_2) ppm.

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Table 2 Data of IR, ^{1}HNMR for products

No.	IR. max(cm^{-1})			^{1}HNMR:δ (ppm)*
	O=C-O	O=C	NO_2	
3aa	1731	1638	1517 1348	8.10(2H,d,Ar-H,J=9.0Hz),7.40(2H,d,Ar-H,J=9.0Hz),7.86-7.30(5H,m,Ar-H) 3.80(1H,t,Ar-CH,J=7.5Hz),3.62(3H,s,OCH_3),3.0-2.03 (4H,m,CH_2CH_2)
3ac	1720	1684	1519 1354	8.22(2H,d,Ar-H,J=9.0Hz),7.56(2H,d,Ar-H,J=9.0Hz),8.03-7.47(5H,m,Ar-H) 3.77(3H,s,OCH_3),3.06-2.76(2H,m,CH_2-C-C=O),2.56-2.30(2H,m, CH_2C=O),1.66(3H,s,C-CH_3)
3ad	1718	1681	1518 1352	8.22(2H,d,Ar-H,J=9.0Hz),7.53(2H,d,Ar-H,J=9.0Hz),8.00-7.40(5H,m,Ar-H) 4.22(2H,q,OCH_2,J=7.0Hz),3.16-2.83(2H,m,CH_2-C-C=O),2.62-2.34(2H,m, CH_2C=O),1.72(3H,s,C-CH_3),1.30(3H,t,O-C-CH_3,J=7.0Hz)
3ba	1732	1681	1516 1344	8.06(2H,d,Ar-H,J=9.0Hz),7.66(2H,d,Ar-H,J=8.0Hz),7.40(2H,d,Ar-H, J=9.0Hz),7.10(2H,d,Ar-H,J=8.0Hz),3.80(1H,t,Ar-CH,J=7.5Hz),3.63(3H,s, OCH_3),3.00-2.76(2H,m,CH_2-C-C=O),2.50-1.96(2H,m,CH_2C=O),2.40(3H, s,Ar-CH_3)
3bb	1722	1676	1518 1351	8.10(2H,d,Ar-H,J=9.0Hz),7.70(2H,d,Ar-H,J=8.0Hz),7.43(2H,d,Ar-H, J=9.0Hz),7.13(2H,d,Ar-H,J=8.0Hz),4.12(2H,q,OCH_2,J=7.0Hz),3.82(1H,t, Ar-CH,J=7.0Hz),3.00-2.76(2H,m,CH_2-C-C=O),2.53-2.10(2H,m, CH_2C=O),2.43(3H, s,Ar-CH_3),1.22(3H,t,O-C-CH_3,J=7.0Hz)
3bc	1724	1678	1518 1353	8.06(2H,d,Ar-H,J=9.0Hz),7.66(2H,d,Ar-H,J=8.0Hz),7.36(2H,d,Ar-H, J=9.0Hz),7.10(2H,d,Ar-H,J=8.0Hz),3.66(3H,s,OCH_3),3.00-2.70(2H,m, CH_2-C-C=O),2.50-2.16(2H,m,CH_2C=O),2.37(3H,s,Ar-CH_3), 1.63(3H,s,C-CH_3)
3bd	1714	1675	1518 1350	8.06(2H,d,Ar-H,J=9.0Hz),7.63(2H,d,Ar-H,J=8.0Hz),7.38(2H,d,Ar-H, J=9.0Hz),7.06(2H,d,Ar-H,J=8.0Hz),4.13(2H,q,OCH_2,J=7.0Hz),3.00-2.70 (2H,m,CH_2-C-C=O),2.50-2.20(2H,m,H_2C=O),2.40(3H,s,Ar-CH_3),1.63 (3H,s,C-CH_3),1.24 (3H,t,O-C-CH_3,J=7.0Hz)
3ca	1735	1679	1525 1345	8.19(2H,d,Ar-H,J=8.5Hz),7.84(2H,d,Ar-H,J=8.3Hz),7.50(2H,d,Ar-H, J=8.5Hz),7.41(2H,d,Ar-H,J=8.3Hz),3.87(1H,t,Ar-CH,J=7.5Hz),3.70(3H, s,OCH_3),2.95(2H,m,CH_2-C-C=O),2.53(1H,dt,CH_aC=O,J_1=21.3Hz, J_2=7.3Hz),2.24(1H,dt, CH_bC=O, J_1=21.3Hz, J_2=6.8Hz)
3cc	1726	1668	1519 1355	8.10(2H,d,Ar-H,J=8.5Hz),7.77(2H,d,Ar-H,J=8.3Hz),7.40(2H,d,Ar-H, J=8.5Hz),7.31(2H,d,Ar-H,J=8.3Hz),3.69(3H,s,OCH_3),3.00-2.70 (2H,m,CH_2-C-C=O), 2.50-2.20(2H,m, CH_2C=O),1.64(3H,s,C-CH_3)
3cd	1727	1687	1521 1348	8.10(2H,d,Ar-H,J=8.5Hz),7.78(2H,d,Ar-H,J=8.3Hz),7.43(2H,d,Ar-H, J=8.5Hz),7.34(2H,d,Ar-H,J=8.3Hz),4.16(2H,q,OCH_2,J=7.0Hz),3.00-2.76 (2H,m,CH_2-C-C=O),2.50-2.20(2H,m,H_2C=O),1.70(3H,s,C-CH_3),1.30 (3H,t,O-C-CH_3,J=7.0Hz)
4	1729 1715	3499 (OH)	1519 1350	8.21(2H,d,Ar-H,J=8.7Hz),7.60(2H,d,Ar-H,J=8.7Hz),7.43-7.23(5H,m,Ar-H) 4.40(1H,s,OH),3.77(3H,s,OCH_3),3.53(3H,s,OCH_3),3.39(1H,dd,H_a,J_{ab}= 3.4Hz,J_{ac}=12.8Hz),2.76(1H,m,H_b),2.51(1H,m,H_e),2.42(1H,dt**,H_d,J_{de}= 13.6Hz,J_{dg}=13.6Hz,J_{df}=3.5Hz),2.41(1H,t,H_c,J_{cb}=12.8Hz,J_{ca}=12.8Hz), 1.96(1H,m,H_f),1.72(1H,dt,H_g,J_{gf}=13.6Hz,J_{gd}=13.6Hz,J_{ge}=3.5Hz).

* A mixture of $CDCl_3$ and CCl_4 was used as the solvent. **dt represents a doublet of triplet.

2: Methyl 2-(4-nitrophenyl)-4-benzoylbutanate (**3aa**)

To a solution of 0.55 g (2 mmol) of **1a** in 5 mL of DFM was added 0.55 g (4 mmol) of anhydrate potassium carbonate and 0.1g (0.44 mmol) of DEBAR. The solution was stirred 15 minutes at room temperature before 0.52 g (6 mmol) of methyl acrylates(**2a**) was added. The reaction progress was monitored by thin layer chromatography(TLC). After stirred at 60 °C for 8 hours, the reaction mixture was cooled and poured into cold water. The mixture was added with 2N hydrochloric acid to PH=6.5 and extracted with dichloromethane(3×15ml). The mixture was added with 2N hydrochloric acid to PH=6.5 and extracted with dichloromethane(3×15ml). The combined organic phase was dried over anhydrate sodium sulfate and concentrated. The residue was chromatographied on silica. Elution with a mixed solvent of petroleum ether and ethyl acetate (5:1) afforded 0.67 g (92 %) of **3aa** as white needle .M.P. 108-109 °C.

3ac-3cd were synthesized by the method similar to **3aa**. Their principle reaction conditions and analytical data were listed in the table 1 and table 2.

3 dimethyl 4-hydroxyl-1-(4-nitrophenyl)-4-phenyl-1,3-cyclohexyldicarboxylate (**4**).

To a solution of 0.55 g (2 mmol) of **1a** in 5 mL of DFM was added 0.55 g (4 mmol) of anhydrate potassium carbonate and 0.1g (0.44 mmol) of DEBAR. The solution was stirred 15 minutes at room temperature before 0.52 g (6 mmol) of methyl acrylates(**2a**) was added. Then, the solution was stirred at 75°C for 15 hours. It was worked up (*vide supra*) and subjected to column chromatography on silica using a mixed solvent of petroleum ether and ethyl acetate (4:1) as eluent to afford 0.70 g (85 %) of **4** as white needle .M.P. 148-149 °C.

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(Received in the USA 07 November 1996)

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