Microwave-Assisted Stereoselective 1,3-Dipolar Cycloaddition of *C*,*N*-Diarylnitrone (*i.e.*, *N*-(Arylmethylidene)benzenamine *N*-Oxide) and Bis(arylmethylidene)acetone (=1,5-Diarylpenta-1,4-dien-3-one): NMR and Crystal Analysis of Diastereoisomeric Bis(isoxazolidines)

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Microwave-assisted stereoselective 1,3-dipolar cycloaddition of *C*,*N*-diarylnitrones (*i.e.*, *N*-(arylmethylidene)benzenamine *N*-oxides) **2** to substituted bis(arylmethylidene)acetones (=1,5-diarylpenta-1,4-dien-3-ones) **1** leading to diastereoisomer pairs of bis-isoxazolidines **3** and **4** in good to excellent yield is described (*Scheme 2* and *Table 2*). The configuration outcome of the reaction is discussed based on the NMR and X-ray data of the products.

Introduction. – The 1,3-dipolar cycloaddition of nitrones to alkenes affords isoxazolidines through a concerted C–C and C–O bond forming mechanism with generation of as many as three new contiguous stereogenic centers in a single step [1]. Isoxazolidines exhibit antibacterial and antifungal properties [2], and the possibility of their transformation *via* ring opening to open-chain derivatives [3] makes these compounds valuable synthons for the synthesis of natural and biologically important compounds, such as amino sugars, amino alcohols, alkaloids, β-lactams, and amino acids [4]. The stereochemical course of the 1,3-dipolar cycloaddition reaction has been well explained in terms of secondary orbital interactions, steric factors, H-atom bonding and/or dipole–dipole electrostatic interactions [2b][5].

The use of microwaves for carrying out organic reactions is a well established procedure and has emerged as a promising synthetic technique, since the reactions are clean, fast, and economical with easy workup. The technique of microwave irradiation enables organic reactions to occur expeditiously at ambient pressure and provides unique reaction processes with special attributes such as accelerated reaction rate and relatively good yields [1g][2b][6] with selectivity in certain cases.

Results and Discussion. – We have already indicated that with 1:1 and 1:2 ratios of dibenzylideneacetone (=1,5-diphenylpenta-1,4-dien-3-one) and nitrone, the cycloadditon under reflux leads to a complex mixture of products, the major product being the mono-cycloadduct indicating that the second cycloaddition seems to be difficult under the reaction conditions studied [1b]. In the present work, in an attempt to prepare exclusively the bis-adduct having two isoxazolidine rings connected by a C=O group from dibenzylideneacetone and to deliberate the stereochemical features due to the generation of six new stereogenic centers, we carried out the cycloaddition under

different harsh conditions. The reaction of bis(arylmethylidene)acetone **1** with excess nitrone **2** (1:4), even after refluxing in toluene for two days, led to a mixture of three products; two of them, **3** and **4**, were identified as the expected isomeric bis-adducts, while the third one was the mono-isoxazolidine **5** with a free cinnamoyl moiety (*Scheme 1*). Thus, this conventional procedure resulted in a nearly 90% overall yield and a considerable amount of **5** (*Table 1*).

Scheme 1. *Reaction of* **1** *and* **2** *at Reflux Temperature in Toluene*

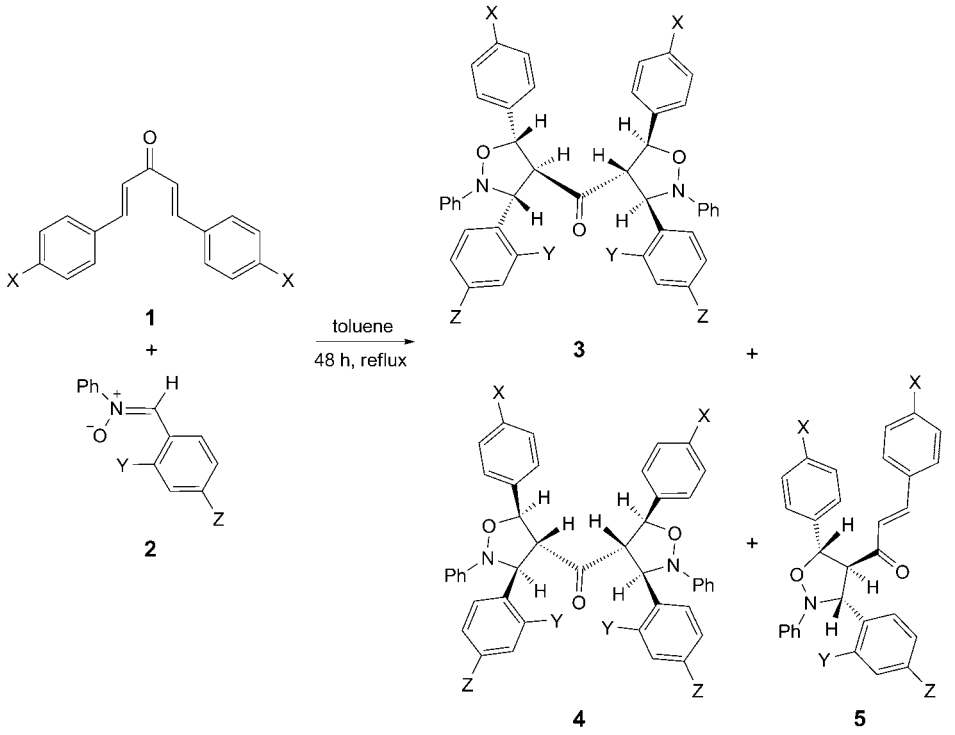


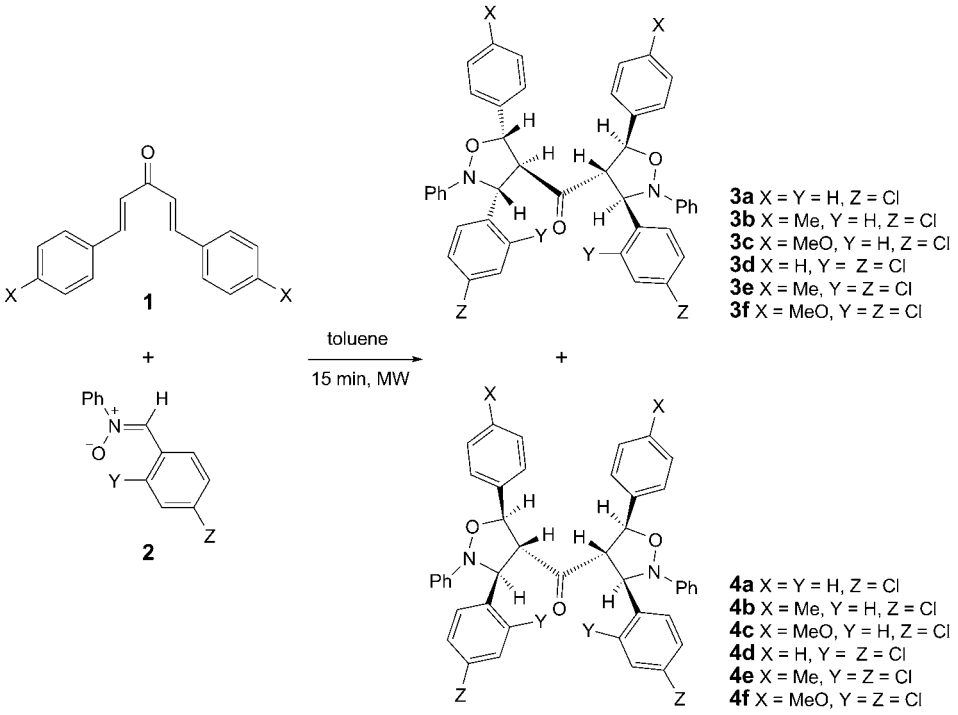
Table 1. *Yields of* **3**–**5** *by the Conventional-Heating and Microwave-Irradiation Method*

	X	Y	Z	Yield [%] after MW[a])		Yield [%] after reflux[a])		
				3	**4**	**3**	**4**	**5**
a	H	H	Cl	48	43	24	21	44
b	Me	H	Cl	46	44	27	23	45
c	MeO	H	Cl	47	45	27	24	45
d	H	Cl	Cl	45	40	24	23	43
e	Me	Cl	Cl	48	44	29	26	41
f	MeO	Cl	Cl	46	42	26	24	42

[a]) Yield of isolated product after purification by column chromatography.

However, the reaction of acetone derivative **1** with nitrone **2** showed remarkable selectivity when carried out under microwave irradiation. The yield of **5** was considerably reduced, and by optimizing the microwave parameters, only the bis-adducts **3** and **4** with no trace of **5** were formed. The influence of microwave power, temperature, and irradiation time on the reaction was then systematically investigated for the reaction of **1** (X = MeO) and **2** (Y = H, Z = Cl) (*Table 2*). In the microwave assisted reaction (*Scheme 2*), the highest yield of isomeric bis-adducts was observed when the temperature of the reaction medium was maintained at 125° (*Table 2*); a decrease in the yield was noticed while increasing or decreasing the temperature. The data (*Tables 1* and *2*) show that the reaction was stereoselective, as the yield of isomer **3** was always slightly higher than that of **4**. It must be mentioned that both isomers were successfully separated, in spite of the fact that they have close R_f values. The low stereoselectivity observed suggests that the energies of activation for the formation of these compounds do not differ much.

Scheme 2. *Reaction of* **1** *and* **2** *under Microwave Irradiation*



The cycloaddition of dibenzylideneacetones **1** with nitrones **2** proceeds regioselectively through the addition of the O-atom of the nitrone to the C(β) of the benzylidene moiety affording the two diastereoisomeric bis-isoxazolidines. The regioselectivity is in accord with the polarization of the C=C bond enabling the more electron-deficient C(β) to preferentially react with the electron-rich O-atom of the

Table 2. *Optimization of the Microwave Assisted Reaction of* **1** (X = MeO) *and* **2** (Y = H, Z = Cl)

Temp. [°]	Power [watt]	Time [min]	Yield [%]	
			overall	**3c**/**4c**/**5c**
70	100	12	36	43 : 40 : 17
80	100	10	45	44 : 41 : 15
80	120	15	48	47 : 42 : 11
90	120	20	60	45 : 43 : 12
110	120	10	72	49 : 47 : 4
115	125	15	80	50 : 48 : 2
125	130	15	92	51 : 49 : 0
125	130	5	83	50 : 47 : 3
125	140	25	90	52 : 48 : 0
140	150	15	85	47 : 47 : 6
165	150	10	54	42 : 40 : 18
186	200	10	8	–[a])

[a]) Trace amounts of all three products.

nitrone. Assigning the configuration of these two isomers is interesting. The NMR spectra indicated that both **3** and **4** had only one set of signals for their isoxazolidine rings showing that the bis-adducts were symmetrical with respect to the center. Based on one- and two-dimensional NMR data, the complete assignments of the position of the H- and C-atoms for **3** and **4** could be achieved (as detailed for **3f** and **4f** in *Figs. 1* and *2*).

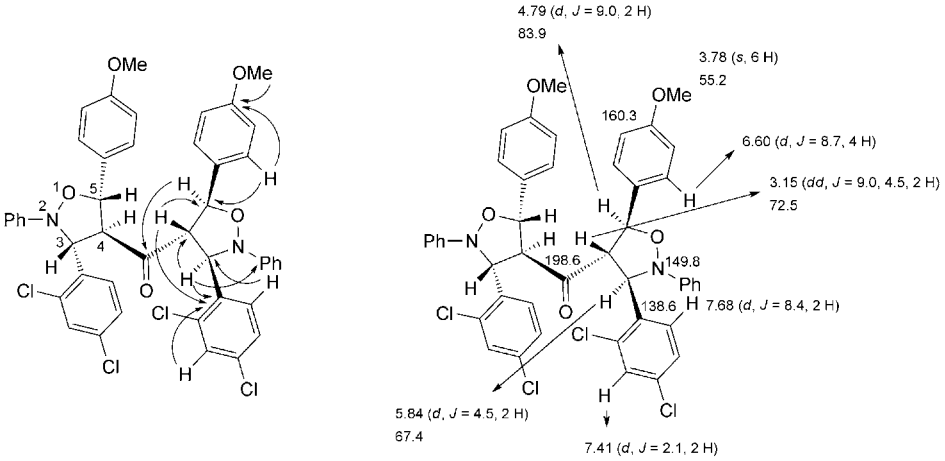


Fig. 1. *Selected HMBCs and* ^{1}H- *and* ^{13}C-*NMR chemical shifts of* **3f**. δ in ppm, J in Hz.

The spatial arrangement of the isoxazolidine rings is again similar for both **3** and **4**, the H-atoms all being in *trans* relation to each other. These details restricted the structures of the two diastereoisomeric bis-adducts to the ones shown in *Schemes 1* and *2* as well as in *Figs. 1* and *2*. One of them is a racemate and the other a *meso* form. The close similarity of the configurational features makes the energy associated with these

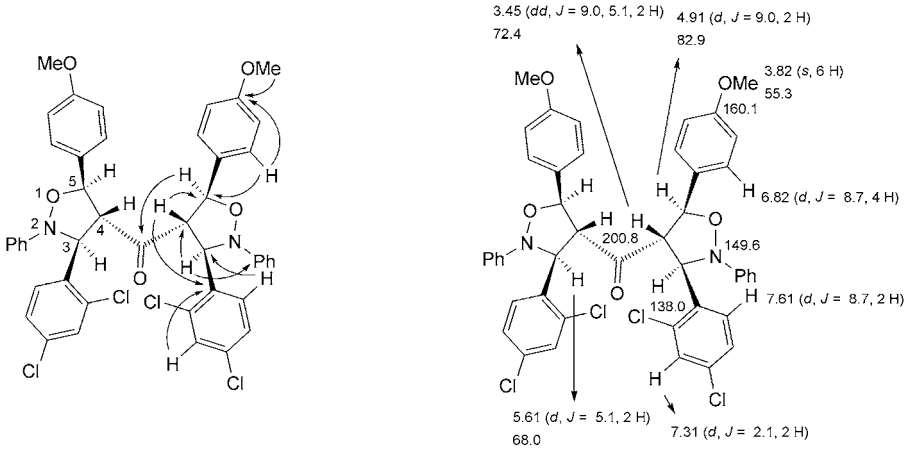


Fig. 2. *Selected HMBCs and* ^{1}H- *and* ^{13}C-*NMR chemical shifts of* **4f**. δ in ppm, J in Hz.

two isomers to be close accounting for the observed near-equal yield of them. Thus, out of the twenty possible diastereoisomers due to the presence of six degenerate chiral centers, only two diastereoisomers were selectively produced during this cycloaddition. It should be mentioned that NMR data of **3** and **4** are not helpful to distinguish between the racemic and *meso* forms. However, the single-crystal X-ray analyses of **3f** and **4f** revealed unambiguously that the former is racemic and the latter is *meso* (*Fig. 3*). The crystal structures of **3c**, **3f**, and **4f** (*Figs. 4–6*) indicated that the isoxazolidine rings are all in an envelope conformation. It is pertinent that the mono-isoxazolidine has also the central ring in an envelope conformation with the O-atom placed out-of-plane [1a][1b]. It may be highlighted that H–C(3) and H–C(3′) (and also H–C(4) and H–C(4′) and H–C(5) and H–C(5′)) of **3** are homotopic, while in **4**, these pairs are in an enantiotopic relationship (*Fig 3*). Had they been diastereoisotopic, they would have become nonequivalent in the NMR spectra, resulting in two sets of signals for the isoxazolidine rings. The HR-MS of the compounds **3f** and **4f** had signals

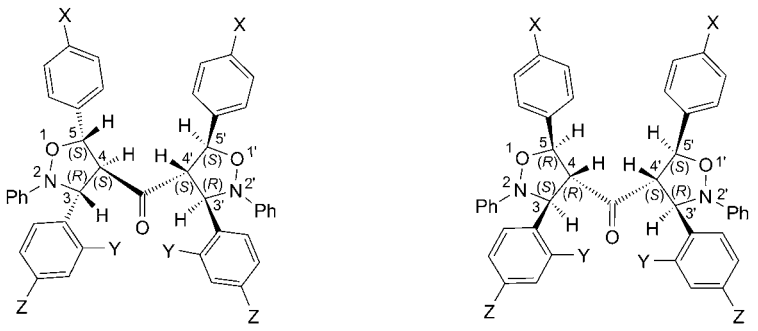


Fig. 3. *Configurations of bis-isoxazolidines* **3** *and* **4** *from crystal structures*

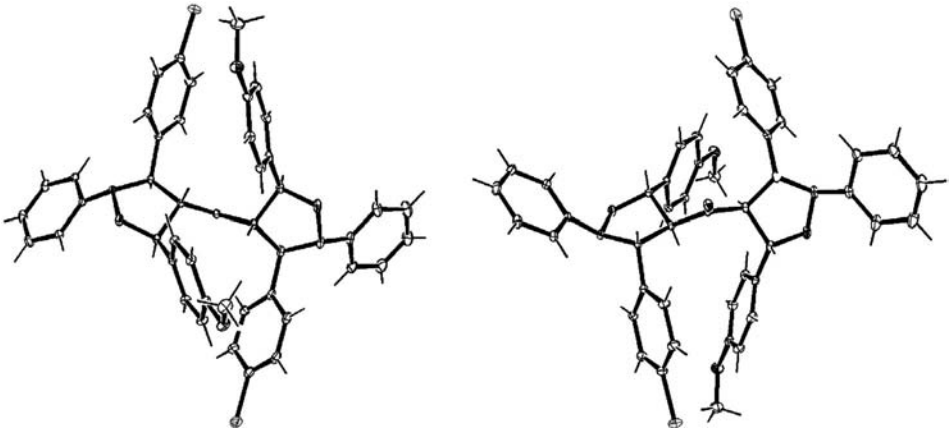


Fig. 4. *ORTEP Diagram of* **3c**

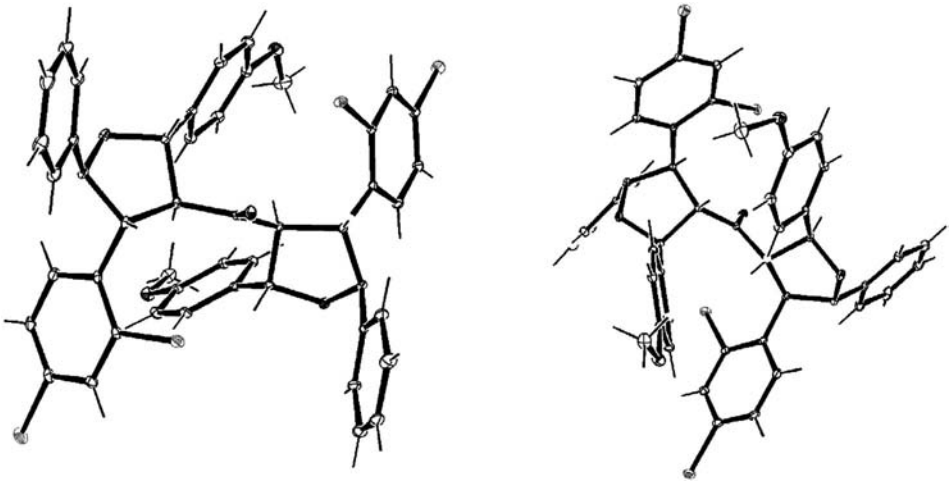


Fig. 5. *ORTEP Diagram of* **3f**

at *m*/*z* 847.12705 ([*M* + 23]) and 823.13056 ([*M* – 1]), respectively; moreover, all isomers **3** and **4** showed elemental analyses compatible with their structures.

A comparison of the chemical shifts and coupling constants of isomeric bis-isoxazolidines **3** and **4** and the mono-adduct **5** is illustrated in *Table 3*. Thus, between the diastereoisomers (**3c** and **4c**, or **3f** and **4f**), the variations of values were not very important. But a change in the substitution pattern of the aryl ring at C(3) of the isoxazolidine ring altered the chemical shifts and coupling constants considerably. In the case of the 2,4-dichloro-substituted systems (see **3f**, **4f**, and **5f**), the isoxoazolidine-ring H-atoms were deshielded, while the corresponding C-atoms were shielded compared to the 4-chloro-substituted systems (see **3c**, **4c**, and **5c**). The coupling constant between H–C(3) and H–C(4) decreased substantially in the 2,4-dichloro-

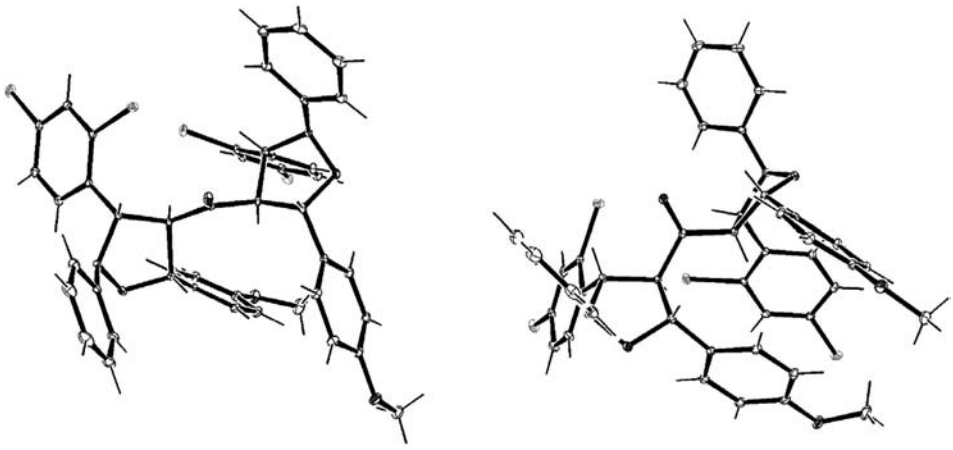


Fig. 6. *ORTEP Diagram of* **4f**

phenyl systems, suggesting a change in the conformation, with probably more deformation in this case due to steric hindrance. A comparison of the coupling constants indicated a similar configuration for the isoxazolidine ring in the mono- and bis-adducts in the respective series, with all H-atoms *trans* to each other at the ring. The H_o of the 2,4-dichlorophenyl residue in **3f** and **4f** appeared at δ(H) 7.68 (d, $J = 8.4$ Hz) and at δ(H) 7.61 (d, $J = 8.7$ Hz), respectively, while that for **5f** appeared at δ(H) 8.02 (d, $J = 9.0$ Hz). This deshielding in the latter can be attributed to the spatial proximity of the C=O group with the isoxazolidine ring in the mono-adduct as compared to the situation in the bis-adduct.

Table 3. *Comparison of NMR Chemical Shifts and Coupling Constants of Isomeric Bis-isoxazolidines and Mono-isoxazolidines.* δ in ppm, J in Hz.

	δ(H)			δ(C)		
	H–C(3)	H–C(4)	H–C(5)	C(3)	C(4)	C(5)
3c	5.20 ($J = 7.5$)	3.12 ($J = 9.6, 7.5$)	4.56 ($J = 9.6$)	71.4	73.6	83.5
3f	5.84 ($J = 4.5$)	3.15 ($J = 9.0, 4.5$)	4.79 ($J = 9.0$)	67.4	72.5	83.9
4c	4.95 ($J = 9.3$)	3.22 ($J = 9.3, 8.1$)	4.73 ($J = 8.1$)	72.7	73.7	82.2
4f	5.61 ($J = 5.1$)	3.45 ($J = 9.0, 5.1$)	4.91 ($J = 9.0$)	68.0	72.4	82.9
5c	5.37 ($J = 7.2$)	3.97 ($J = 9.3, 7.2$)	5.22 ($J = 9.3$)	72.4	73.1	83.9
5f	5.85 ($J = 4.5$)	3.86 ($J = 8.7, 4.5$)	5.16 ($J = 8.7$)	70.1	71.9	84.9

Conclusions. – We optimized a method for the preparation of diastereoisomeric bis-isoxazolidines exclusively achieving the selectivity through microwave irradiation. The diastereoisomers were separated and well characterized by ^{1}H-, ^{13}C-, and 2D-NMR, MS, and single-crystal X-ray analyses.

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Experimental Part

General. A *CEM-Discover* microwave synthesizer (model No. 908010) operating at 180/264 V and 50/60 Hz with a microwave-power maximum level of 300 W and microwave frequency of 2455 MHz was employed for the microwave-assisted experiments. TLC: silica gel *G* plates (SiO_2; *Merck*), petroleum ether (60–80°) and AcOEt as eluents. M.p.: open capillary tubes; uncorrected. IR Spectra: *Jasco* FT-IR instrument; in KBr; $\tilde{\nu}$ in cm^{-1}.^{1}H-, ^{13}C-, and 2D-NMR: *Bruker-Avance-300* NMR instrument; in $CDCl_3$; δ in ppm rel. to Me_4Si as internal standard, *J* in Hz. Elemental analyses: *Perkin–Elmer-2400* series *II* elemental CHNS analyzer.

Conventional-Heating Method. A mixture of dibenzylideneacetone **1** (1 mmol) and nitrone **2** (4 mmol) in toluene (7 ml) was heated under reflux for 48 h. Then the mixture was concentrated under vacuum and the crude residue subjected to NMR analysis before separation. The separation by column chromatography (CC; SiO_2 petroleum ether/AcOEt 9 : 1) afforded the pure diastereoisomers **3** and **4** and the mono-adduct **5**.

Microwave-Irradiation Method. A mixture of dibenzylideneacetone **1** (1 mmol) and nitrone **2** (4 mmol) in toluene (3 ml) in a 10-ml microwave tube was placed in the microwave synthesizer and irradiated at 125° with 130 watts for 15 min. After completion of the reaction (TLC monitoring), the mixture was concentrated under vacuum, and the residue was subjected to CC (SiO_2 petroleum ether/AcOEt 9 : 1): pure diastereoisomers **3** and **4**. Spectroscopic data for all the compounds are given below.

(±)-rel-*Bis[(3*R*,4*S*,5*S*)-3-(4-chlorophenyl)-2,5-diphenylisoxazolidin-4-yl]methanone* (**3a**). Colorless solid. M.p. 192°. IR: 2931 (arom. H), 1705 (C=O). ^{1}H-NMR: 3.11 (*dd*, *J* = 9.6, 7.5, 2 H, H–C(4)); 4.66 (*d*, *J* = 9.6, 2 H, H–C(5)); 5.14 (*d*, *J* = 7.5, 2 H, H–C(3)); 6.70 (*d*, *J* = 7.5, 4 arom. H); 6.84 (*d*, *J* = 7.8, 4 arom. H); 6.95 (*t*, *J* = 7.2, 2 arom. H); 7.10–7.38 (*m*, 18 arom. H). ^{13}C-NMR: 71.6; 74.0; 83.4; 114.0; 122.1; 127.1; 127.6; 128.9; 129.0; 129.2; 129.4; 133.7; 134.5; 139.3; 151.2; 200.9. Anal. calc. for $C_{43}H_{34}Cl_2N_2O_3$: C 74.03, H 4.91, N 4.02; found: C 74.00, H 4.87, N 4.07.

(±)-rel-*Bis[(3*R*,4*S*,5*S*)-3-(4-chlorophenyl)-5-(4-methylphenyl)-2-phenylisoxazolidin-4-yl]methanone* (**3b**): Colorless solid. M.p. 187°. IR: 2931 (arom. H), 1703 (C=O). ^{1}H-NMR: 2.38 (*s*, 2 Me); 3.15 (*dd*, *J* = 9.6, 7.5, 2 H, H–C(4)); 4.60 (*d*, *J* = 9.6, 2 H, H–C(5)); 5.15 (*d*, *J* = 7.5, 2 H, H–C(3)); 6.61 (*d*, *J* = 7.8, 4 arom. H); 6.84 (*d*, *J* = 7.8, 4 arom. H); 6.97 (*d*, *J* = 7.8, 4 arom. H); 7.13 (*d*, *J* = 8.4, 4 arom. H); 7.21 (*d*, *J* = 8.4, 4 arom. H); 7.25–7.28 (*m*, 6 arom. H). ^{13}C-NMR: 21.3; 71.5; 73.6; 83.6; 113.9; 114.4; 121.9; 127.2; 127.6; 129.0; 129.1; 129.6; 131.0; 133.6; 139.5; 151.3; 201.1. Anal. calc. for $C_{45}H_{38}Cl_2N_2O_3$: C 74.48, H 5.28, N 3.86; found: C 74.44, H 5.25, N 3.92.

(±)-rel-*Bis[(3*R*,4*S*,5*S*)-3-(4-chlorophenyl)-5-(4-methoxyphenyl)-2-phenylisoxazolidin-4-yl]methanone* (**3c**): Colorless solid. M.p. 184°. IR: 2930 (arom. H), 1702 (C=O). ^{1}H-NMR: 3.12 (*dd*, *J* = 9.6, 7.5, 2 H, H–C(4)); 3.84 (*s*, 2 MeO); 4.56 (*d*, *J* = 9.6, 2 H, H–C(5)); 5.20 (*d*, *J* = 7.5, 2 H, H–C(3)); 6.63 (*d*, *J* = 8.4, 4 arom. H); 6.68 (*d*, *J* = 9.0, 4 arom. H); 6.84 (*d*, *J* = 8.4, 4 arom. H); 6.94 (*t*, *J* = 7.2, 2 arom. H); 7.15–7.30 (*m*, 12 arom. H). ^{13}C-NMR: 55.3; 71.4; 73.6; 83.5; 113.8; 114.2; 121.8; 125.7; 127.6; 128.6; 129.0; 129.1; 133.5; 139.7; 151.4; 160.4; 201.0. Anal. calc. for $C_{45}H_{38}Cl_2N_2O_5$: C 71.33, H 5.06, N 3.70; found: C 71.27, H 5.01, N 3.73.

(±)-rel-*Bis[(3*R*,4*S*,5*S*)-3-(2,4-dichlorophenyl)-2,5-diphenylisoxazolidin-4-yl]methanone* (**3d**): Colorless solid. M.p. 161°. IR: 2934 (arom. H), 1705 (C=O). ^{1}H-NMR: 3.18 (*dd*, *J* = 8.7, 4.5, 2 H, H–C(4)); 4.84 (*d*, *J* = 8.7, 2 H, H–C(5)); 5.83 (*d*, *J* = 4.5, 2 H, H–C(3)); 6.66 (*d*, *J* = 7.5, 4 arom. H); 6.97–7.05 (*m*, 10 arom. H); 7.19–7.31 (*m*, 8 arom. H); 7.40 (*s*, 2 arom. H); 7.66 (*d*, *J* = 8.4, 2 arom. H). ^{13}C-NMR: 67.4; 72.7; 84.0; 113.9; 122.1; 127.3; 127.9; 128.9; 129.1; 129.2; 129.3; 129.8; 132.3; 133.9; 134.5; 138.3; 149.6; 198.5. Anal. calc. for $C_{43}H_{32}Cl_4N_2O_3$: C 67.38, H 4.21, N 3.65; found: C 67.35, H 4.17, N 3.70.

(±)-rel-*Bis[(3*R*,4*S*,5*S*)-3-(2,4-dichlorophenyl)-5-(4-methylphenyl)-2-phenylisoxazolidin-4-yl]methanone* (**3e**): Colorless solid. M.p. 188°. IR: 2928 (arom. H), 1704 (C=O). ^{1}H-NMR: 2.30 (*s*, 2 Me); 3.19 (*dd*, *J* = 9.0, 4.5, 2 H, H–C(4)); 4.79 (*d*, *J* = 9.0, 2 H, H–C(5)); 5.82 (*d*, *J* = 4.5, 2 H, H–C(3)); 6.56 (*d*,

$J = 8.1$, 4 arom. H); 6.82 (*d*, $J = 7.8$, 4 arom. H); 6.95 – 6.98 (*m*, 6 arom. H); 7.23 – 7.30 (*m*, 6 arom. H); 7.40 (*d*, $J = 2.1$, 2 arom. H); 7.65 (*d*, $J = 8.7$, 2 arom. H). ^{13}C-NMR: 21.2; 67.4; 72.5; 84.0; 113.9; 122.0; 127.3; 127.8; 129.0; 129.2; 129.4; 129.8; 131.2; 133.3; 133.8; 138.4; 139.2; 149.8; 198.6. Anal. calc. for $C_{45}H_{36}Cl_4N_2O_3$: C 68.02, H 4.57, N 3.53; found: C 67.99, H 4.54, N 3.56.

(±)-rel-*Bis[(3*R*,4*S*,5*S*)-3-(2,4-dichlorophenyl)-5-(4-methoxyphenyl)-2-phenylisoxazolidin-4-yl]methanone* (**3f**): Colorless solid. M.p. 165°. IR: 2932 (arom. H), 1702 (C=O). ^{1}H-NMR: 3.15 (*dd*, $J = 9.0$, 4.5, 2 H); 3.78 (*s*, 2 MeO); 4.79 (*d*, $J = 9.0$, 2 H); 5.84 (*d*, $J = 4.5$, 2 H); 6.54 (*d*, $J = 8.7$, 4 H); 6.60 (*d*, $J = 8.7$, 4 H); 6.95 – 7.00 (*m*, 6 arom. H); 7.25 – 7.31 (*m*, 6 arom. H); 7.41 (*d*, $J = 2.1$, 2 arom. H); 7.68 (*d*, $J = 8.4$, 2 arom. H). ^{13}C-NMR: 55.2; 67.4; 72.5; 83.9; 113.9; 114.2; 122.0; 126.2; 127.8; 128.8; 129.1; 129.2; 129.9; 132.4; 133.8; 138.6; 149.8; 160.3; 198.6. HR-MS: 847.12705 ([$M + 23$]; calc. 847.13783). Anal. calc. for $C_{45}H_{36}Cl_4N_2O_5$: C 65.39, H 4.39, N, 3.39; found: C 65.34, H 4.35, N 3.43.

meso-*Bis[3-(4-chlorophenyl)-2,5-diphenylisoxazolidin-4-yl]methanone* (= *[(3*R*,4*S*,5*S*)-3-(4-Chlorophenyl)-2,5-diphenylisoxazolidin-4-yl][(3*S*,4*R*,5*R*)-3-(4-chlorophenyl)-2,5-diphenylisoxazolidin-4-yl]-methanone*; **4a**): Colorless solid. M.p. 161°. IR: 2933 (arom. H), 1714 (C=O). ^{1}H-NMR: 3.19 (*dd*, $J = 9.3$, 8.1, 2 H, H–C(4)); 4.72 (*d*, $J = 8.1$, 2 H, H–C(5)); 5.00 (*d*, $J = 9.3$, 2 H, H–C(3)); 6.83 (*d*, $J = 8.4$, 4 arom. H); 6.89 (*d*, $J = 7.5$, 4 arom. H); 6.96 (*t*, $J = 7.2$, 2 arom. H); 7.02 (*d*, $J = 8.4$, 4 arom. H); 7.19 – 7.30 (*m*, 14 arom. H). ^{13}C-NMR: 72.8; 74.0; 82.2; 114.6; 122.5; 126.7; 128.0; 128.8; 128.9; 129.0; 129.4; 134.1; 135.7; 139.1; 150.9; 201.7. Anal. calc. for $C_{43}H_{34}Cl_2N_2O_3$: C 74.03, H 4.91, N 4.02; found: C 74.01, H 4.89, N 4.07.

meso-*Bis[3-(4-chlorophenyl)-5-(4-methylphenyl)-2-phenylisoxazolidin-4-yl]methanone* (= *[(3*R*,4*S*,5*S*)-3-(4-Chlorophenyl)-5-(4-methylphenyl)-2-phenylisoxazolidin-4-yl][(3*S*,4*R*,5*R*)-3-(4-chlorophenyl)-5-(4-methylphenyl)-2-phenyl)-2-phenylisoxazolidin-4-yl]methanone;* **4b**): Colorless solid. M.p. 185°. IR: 2931 (arom. H), 1716 (C=O). ^{1}H-NMR: 2.37 (*s*, 2 Me); 3.23 (*dd*, $J = 9.3$, 8.1, 2 H, H–C(4)); 4.73 (*d*, $J = 8.1$, 2 H, H–C(5)); 4.50 (*d*, $J = 9.3$, 2 H, H–C(3)); 6.81 (*t*, $J = 7.5$, 4 arom. H); 6.96 – 7.10 (*m*, 6 arom. H); 7.18 – 7.30 (*m*, 10 arom. H); 7.36 (*d*, $J = 8.4$, 4 arom. H); 7.52 (*d*, $J = 8.4$, 2 arom. H). ^{13}C-NMR: 21.2; 71.5; 72.7; 82.3; 114.2; 122.3; 126.7; 127.9; 129.0; 129.1; 129.3; 129.5; 132.2; 133.9; 138.9; 151.0; 201.9. Anal. calc. for $C_{45}H_{38}Cl_2N_2O_3$: C 74.48, H 5.28, N 3.86; found: C 74.45, H 5.26, N 3.90.

meso-*Bis[3-(4-chlorophenyl)-5-(4-methoxyphenyl)-2-phenylisoxazolidin-4-yl]methanone* (= *[(3*R*,4*S*,5*S*)-3-(4-Chlorophenyl)-5-(4-methoxyphenyl)-2-phenylisoxazolidin-4-yl][(3*S*,4*R*,5*R*)-3-(4-chlorophenyl)-5-(4-methoxyphenyl)-2-phenyl)-2-phenylisoxazolidin-4-yl]methanone*; **4c**). Colorless solid. M.p. 182°. IR: 2929 (arom. H), 1714 (C=O). ^{1}H-NMR: 3.22 (*dd*, $J = 9.3$, 8.1, 2 H, H–C(4)); 3.84 (*s*, 2 MeO); 4.73 (*d*, $J = 8.1$, 2 H, H–C(5)); 4.95 (*d*, $J = 9.3$, 2 H, H–C(3)); 6.74 (*d*, $J = 8.7$, 4 arom. H); 6.81 – 6.85 (*m*, 6 arom. H); 6.90 – 6.97 (*m*, 4 arom. H); 7.06 (*d*, $J = 8.4$, 4 arom. H); 7.18 – 7.25 (*m*, 4 arom. H); 7.30 (*d*, $J = 8.4$, 4 arom. H). ^{13}C-NMR: 55.3; 72.7; 73.7; 82.2; 114.2; 114.4; 122.3; 125.5; 128.0; 128.2; 129.0; 129.3; 134.0; 139.4; 151.0; 160.2; 202.0. Anal. calc. for $C_{45}H_{38}Cl_2N_2O_5$: C 71.33, H 5.06, N 3.70; found: C 71.28, H 5.01, N 3.75.

meso-*Bis[3-(2,4-dichlorophenyl)-2,5-diphenylisoxazolidin-4-yl]methanone* (= *[(3*R*,4*S*,5*S*)-3-(4-Dichlorophenyl)-2,5-diphenylisoxazolidin-4-yl][(3*S*,4*R*,5*R*)-3-(2,4-dichlorophenyl)-2,5-diphenylisoxazolidin-4-yl]methanone*; **4d**): Colorless solid. M.p. 180°. IR: 2931 (arom. H), 1713 (C=O). ^{1}H-NMR: 3.35 (*dd*, $J = 8.7$, 5.1, 2 H, H–C(4)); 4.96 (*d*, $J = 8.7$, 2 H, H–C(5)); 5.62 (*d*, $J = 5.1$, 2 H, H–C(3)); 6.86 (*d*, $J = 8.4$, 4 arom. H); 6.94 (*d*, $J = 8.4$, 4 arom. H); 7.16 – 7.40 (*m*, 16 arom. H); 7.57 (*d*, $J = 8.4$, 2 arom. H). ^{13}C-NMR: 68.0; 72.7; 82.9; 114.1 (2C); 122.3; 126.9; 128.2; 128.9; 129.1; 129.2; 129.9; 132.4; 134.1; 135.4; 137.7; 149.5; 200.5. Anal. calc. for $C_{43}H_{32}Cl_4N_2O_3$: C 67.38, H 4.21, N 3.65; found: C 67.34, H 4.16, N 3.71.

meso-*Bis[3-(2,4-dichlorophenyl)-5-(4-methylphenyl)-2-phenylisoxazolidin-4-yl]methanone* (= *[(3*R*,4*S*,5*S*)-3-(4-Dichlorophenyl)-5-(4-methylphenyl)-2-phenylisoxazolidin-4-yl][(3*S*,4*R*,5*R*)-3-(2,4-dichlorophenyl)-5-(4-methylphenyl)-2-phenylisoxazolidin-4-yl]methanone*; **4e**): Colorless solid. M.p. 175°. IR: 2931 (arom. H), 1715 (C=O). ^{1}H-NMR: 2.36 (*s*, 2 Me); 3.45 (*dd*, $J = 9.0$, 5.1, 2 H, H–C(4)); 4.93 (*d*, $J = 9.0$, 2 H, H–C(5)); 5.62 (*d*, $J = 5.1$, 2 H, H–C(3)); 6.79 (*d*, $J = 8.1$, 4 arom. H); 6.93 (*d*, $J = 7.8$, 4 arom. H); 7.00 (*d*, $J = 8.1$, 4 arom. H); 7.18 (*dd*, $J = 8.7$, 1.8, 2 arom. H); 7.22 – 7.28 (*m*, 6 arom. H); 7.30 (*d*, $J = 1.8$, 2 arom. H); 7.58 (*d*, $J = 8.7$, 2 arom. H). ^{13}C-NMR: 21.2; 68.0; 72.6; 83.0; 114.2; 122.2; 127.0; 128.0; 129.0; 129.1; 129.5; 130.1; 132.3; 132.5; 134.1; 138.1; 139.1; 149.7; 200.7. Anal. calc. for $C_{45}H_{36}Cl_4N_2O_3$: C 68.02, H 4.57, N 3.53; found: C 67.97, H 4.52, N 3.58.

meso-*Bis[3-(2,4-dichlorophenyl)-5-(4-methoxyphenyl)-2-phenylisoxazolidin-4-yl]methanone* (= *[(3*R*,4*S*,5*S*)-3-(2,4-Dichlorophenyl)-5-(4-methoxyphenyl)-2-phenylisoxazolidin-4-yl][(3*S*,4*R*,5*R*)-3-(2,4-di-

chlorophenyl)-5-(4-methoxyphenyl)-2-phenylisoxazolidin-4-yl]methanone; **4f**). Colorless solid. M.p. 178°. IR: 2930 (arom. H), 1714 (C=O). ^{1}H-NMR: 3.45 (*dd*, *J* = 9.0, 5.1, 2 H); 3.82 (*s*, 2 MeO); 4.91 (*d*, *J* = 9.0, 2 H); 5.61 (*d*, *J* = 5.1, 2 H); 6.71 (*d*, *J* = 8.7, 4 H); 6.82 (*d*, *J* = 8.7, 4 H); 6.91 – 6.98 (*m*, 6 arom. H); 7.20 (*dd*, *J* = 8.7, 2.1, 2 arom. H); 7.23 – 7.28 (*m*, 4 arom. H); 7.31 (*d*, *J* = 2.1, 2 arom. H); 7.61 (*d*, *J* = 8.7, 2 H). ^{13}C-NMR: 55.3; 68.0; 72.4; 82.9; 114.0; 114.1; 122.1; 126.8; 128.0; 128.4; 128.9; 129.1; 129.9; 132.4; 134.0; 138.0; 149.6; 160.1; 200.8. MS: 823.13056 ([*M* – 1]; calc. 823.13783). Anal. calc. for $C_{45}H_{36}Cl_4N_2O_5$: C 65.39, H 4.39, N 3.39; found: C 65.35, H 4.36, N 3.42.

(±)-rel-*(2*E*)-1-[(3*R*,4*S*,5*S*)-3-(4-Chlorophenyl)-2,5-diphenylisoxazolidin-4-yl]-3-phenylprop-2-en-1-one* (**5a**): Colorless solid. M.p. 172°. IR: 2931 (arom. H), 1687 (C=O). ^{1}H-NMR: 4.03 (*dd*, *J* = 9.3, 6.9, 1 H); 5.29 (*d*, *J* = 9.3, 1 H); 5.36 (*d*, *J* = 6.9, 1 H); 6.35 (*d*, *J* = 16.2, 1 H); 6.86 (*d*, *J* = 8.4, 2 arom. H); 6.95 (*d*, *J* = 8.4, 2 arom. H); 7.03 (*t*, *J* = 6.9, 3 arom. H); 7.13 – 7.28 (*m*, 6 arom. H); 7.33 – 7.45 (*m*, 7 arom. H). ^{13}C-NMR: 72.6; 73.1; 83.9; 114.5; 124.9; 125.3; 126.9; 127.7; 127.8; 128.1; 128.9; 129.1; 129.3; 129.8; 130.4; 130.9; 134.7; 139.2; 140.3; 145.1; 151.1; 194.6. Anal. calc. for $C_{30}H_{24}ClNO_2$: C 77.33, H 5.19, N 3.01; found: C 77.29, H 5.16, N 3.06.

(±)-rel-*(2*E*)-1-[(3*R*,4*S*,5*S*)-3-(4-Chlorophenyl)-5-(4-methylphenyl)-2-phenylisoxazolidin-4-yl]-3-(4-methylphenyl)prop-2-en-1-one* (**5b**): Colorless solid. M.p. 185°. IR: 2930 (arom. H), 1686 (C=O). ^{1}H-NMR: 2.28 (*s*, Me); 2.33 (*s*, Me); 4.01 (*dd*, *J* = 9.3, 7.2, 1 H); 5.25 (*d*, *J* = 9.3, 1 H); 5.36 (*d*, *J* = 7.2, 1 H); 6.32 (*d*, *J* = 15.9, 1 H); 6.83 (*d*, *J* = 8.1, 2 arom. H); 6.98 – 7.09 (*m*, 5 arom. H); 7.17 – 7.24 (*m*, 7 arom. H); 7.34 (*d*, *J* = 8.1, 2 arom. H); 7.49 (*d*, *J* = 8.1, 2 arom. H). ^{13}C-NMR: 21.1; 21.2; 72.5; 73.1; 83.9; 114.0; 124.0; 124.4; 127.0; 128.2; 128.5; 128.9; 129.1; 129.4; 129.5; 129.9; 130.0; 130.9; 133.3; 138.8; 140.5; 145.1; 151.2; 194.6. Anal. calc. for $C_{32}H_{28}ClNO_2$: C 77.80, H 5.71, N 2.84; found: C 77.76, H 5.68, N 2.88.

(±)-rel-*(2*E*)-1-[(3*R*,4*S*,5*S*)-3-(4-Chlorophenyl)-5-(4-methoxyphenyl)-2-phenylisoxazolidin-4-yl]-3-(4-methoxyphenyl)prop-2-en-1-one* (**5c**): Colorless solid. M.p. 178°. IR: 2930 (arom. H), 1687 (C=O). ^{1}H-NMR: 3.79 (*s*, MeO); 3.85 (*s*, MeO); 3.97 (*dd*, *J* = 9.3, 7.2, 1 H); 5.22 (*d*, *J* = 9.3, 1 H); 5.37 (*d*, *J* = 7.2, 1 H); 6.25 (*d*, *J* = 16.2, 1 H); 6.81 (*d*, *J* = 8.7, 2 arom. H); 6.96 – 7.09 (*m*, 5 arom. H); 7.13 (*d*, *J* = 6.9, 2 arom. H); 7.19 (*d*, *J* = 6.9, 2 arom. H); 7.25 – 7.30 (*m*, 2 arom. H); 7.38 (*t*, *J* = 8.7, 3 arom. H); 7.52 (*d*, *J* = 8.7, 2 arom. H). ^{13}C-NMR: 55.2 (2 C); 72.4; 73.1; 83.9; 113.9; 114.3 (2 C); 122.7; 123.4; 127.7; 128.5; 128.6; 128.9; 129.0; 129.9; 130.2; 133.3; 140.6; 144.9; 151.5; 160.1; 161.4; 194.5. Anal. calc. for $C_{32}H_{28}ClNO_4$: C 73.07, H 5.37, N 2.66; found: C 73.04, H 5.33, N 2.70.

(±)-rel-*(2*E*)-1-[(3*R*,4*S*,5*S*)-3-(2,4-Dichlorophenyl)-2,5-diphenylisoxazolidin-4-yl]-3-phenylprop-2-en-1-one* (**5d**): Colorless solid. M.p. 178°. IR: 2932 (arom. H), 1687 (C=O). ^{1}H-NMR: 3.92 (*dd*, *J* = 8.7, 4.2, 1 H); 5.22 (*d*, *J* = 8.7, 1 H); 5.84 (*d*, *J* = 4.2, 1 H); 6.49 (*d*, *J* = 15.9, 1 H); 7.06 (*d*, *J* = 8.1, 2 arom. H); 7.13 (*d*, *J* = 8.1, 2 arom. H); 7.18 – 7.25 (*m*, 4 arom. H); 7.29 – 7.46 (*m*, 10 arom. H); 8.03 (*d*, *J* = 9.0, 1 arom. H). ^{13}C-NMR: 70.1; 72.0; 85.0; 113.9; 121.0; 122.3; 126.5; 127.6; 128.3; 128.6; 128.7; 128.9; 129.2; 129.5; 129.6; 130.0; 130.8; 131.8; 133.9; 136.4; 138.8; 145.2; 150.2; 194.9. Anal. calc. for $C_{30}H_{23}Cl_2NO_2$: C 72.00, H 4.63, N 2.80; found: C 71.97, H 4.60, N 2.84.

(±)-rel-*(2*E*)-1-[(3*R*,4*S*,5*S*)-3-(2,4-Dichlorophenyl)-5-(4-methylphenyl)-2-phenylisoxazolidin-4-yl]-3-(4-methylphenyl)prop-2-en-1-one* (**5e**): Colorless solid. M.p. 188°. IR: 2927 (arom. H), 1686 (C=O). ^{1}H-NMR: 2.26 (*s*, 3 H); 2.30 (*s*, 3 H); 3.90 (*dd*, *J* = 8.7, 4.5, 1 H); 5.20 (*d*, *J* = 8.7, 1 H); 5.86 (*d*, *J* = 4.5, 1 H); 6.44 (*d*, *J* = 15.9, 1 H); 6.80 (*d*, *J* = 8.4, 2 arom. H); 6.97 (*d*, *J* = 8.4, 2 arom. H); 7.02 – 7.14 (*m*, 4 arom. H); 7.19 – 7.32 (*m*, 5 arom. H); 7.34 (*d*, *J* = 1.8, 1 arom. H); 7.45 (*d*, *J* = 7.8, 2 arom. H); 8.02 (*d*, *J* = 9.0, 1 arom. H). ^{13}C-NMR: 21.1; 21.3; 69.9; 71.8; 84.9; 113.9; 121.5; 123.7; 126.4; 126.8; 127.6; 128.0; 128.9; 129.1; 129.2; 129.3; 129.5; 132.6; 133.2; 133.6; 138.7; 138.9; 141.3; 145.1; 150.2; 194.8. Anal. calc. for $C_{32}H_{27}Cl_2NO_2$: C 72.73, H 5.15, N 2.65; found: C 72.70, H 5.11, N 2.68.

(±)-rel-*(2*E*)-1-[(3*R*,4*S*,5*S*)-3-(2,4-Dichlorophenyl)-5-(4-methoxyphenyl)-2-phenylisoxazolidin-4-yl]-3-(4-methoxyphenyl)prop-2-en-1-one* (**5f**): Colorless solid. M.p. 178°. IR: 2931 (arom. H), 1684 (C=O). ^{1}H-NMR: 3.79 (*s*, MeO, 6 H); 3.86 (*dd*, *J* = 8.7, 4.5, 1 H); 5.16 (*d*, *J* = 8.7, 1 H); 5.85 (*d*, *J* = 4.5, 1 H); 6.36 (*d*, *J* = 16.2, 1 H); 6.81 (*d*, *J* = 8.7, 2 arom. H); 6.88 (*d*, *J* = 8.7, 2 arom. H); 6.96 – 7.02 (*m*, 2 arom. H); 7.08 – 7.17 (*m*, 4 arom. H); 7.21 – 7.40 (*m*, 6 arom. H); 8.02 (*d*, *J* = 9.0, 1 arom. H). ^{13}C-NMR: 55.3 (2C); 70.1; 71.9; 84.9; 113.9; 114.4 (2 C); 121.8; 122.6; 126.7; 127.8; 128.3; 128.7; 129.2; 129.3; 129.6; 130.2; 132.9; 133.8; 139.1; 144.9; 150.4; 160.2; 161.9; 194.8. Anal. calc. for $C_{32}H_{27}Cl_2NO_4$: C 68.58, H 4.86, N 2.50; found: C 68.54, H 4.83, N 2.55.

Crystallographic Investigations[1]). Single crystals for crystallographic studies were prepared in a AcOEt/CH_2Cl_2. Crystallographic data for compounds **3c**, **3f**, and **4f** are given in *Table 4*.

Table 4. *Crystallographic Data for* **3c**, **3f**, *and* **4f**

	3f	**3c**	**4f**
Empirical formula	$C_{45}H_{36}Cl_4N_2O_5$	$C_{45}H_{38}Cl_2N_2O_5$	$C_{45}H_{36}Cl_4N_2O_5$
M_r	826.56	757.67	826.56
Temperature [K]	293(2)	295(2)	293(2)
Wavelength [Å]	0.71073	0.71073	0.71073
Crystal system	monoclinic	monoclinic	triclinic
Space group	*P*2 1/*n*	*P*2 1/*n*	$P\bar{1}$
Unit cell dimensions:			
a [Å]	17.857(5)	14.7972(4)	10.919(5)
b [Å]	10.882(5)	12.3197(4)	11.831(5)
c [Å]	21.663(5)	21.7046(6)	16.135(5)
α [°]	90.000(5)	90.00	78.632(5)
β [°]	118.364(5)	96.314(2)	81.595(5)
γ [°]	90.000(5)	90.00	80.473(5)
V [Å^3]	3995(2)	3932.7(2)	2001.3(14)
Z	4	4	2
Density (calc.) [Mg/m^3]		1.280	1.372
Absorption coefficient [mm^{-1}]	0.346	0.213	0.345
F(000)	1712	1584	856
Crystal size [mm^3]	$0.4 \times 0.4 \times 0.15$	$0.28 \times 0.19 \times 0.15$	$0.4 \times 0.4 \times 0.15$
θ Range for data collection [°]	2.72–25.00	1.90–24.31	2.78–25.00
Index ranges	$-21 \le h \le 21$, $-7 \le k \le 12$, $-17 \le l \le 25$	$-17 \le h \le 17$, $-14 \le k \le 14$, $-25 \le l \le 25$	$-12 \le h \le 12$, $-14 \le k \le 14$, $-19 \le l \le 19$
Reflections collected	17018	74589	22935
Independent reflections	7021 ($R_{int} = 0.0475$)	6390 ($R_{int} = 0.0335$)	7038 ($R_{int} = 0.0385$)
Completeness to $\theta = 60.00°$	99.8%	99.8%	99.9%
Max. and min. transmission	1.00000; 0.69497	0.97; 0.95	1.00000; 0.71113
Data, restraints, parameters	7021, 0, 507	6390, 0, 487	7038, 0, 507
Goodness-of-fit on F^2	0.840	1.019	0.995
Final *R* indices ($I > 2\sigma(I)$)	$R_1 = 0.0505$, $wR_2 = 0.1322$	$R_1 = 0.0455$, $wR_2 = 0.1130$	$R_1 = 0.0465$, $wR_2 = 0.1342$
R Indices (all data)	$R_1 = 0.0983$, $wR_2 = 0.1738$	$R_1 = 0.0684$, $wR_2 = 0.1305$	$R_1 = 0.0671$, $wR_2 = 0.1508$
Largest diff. peak and hole [e·Å^{-3}]	0.189; −0.290	0.272; −0.378	0.219; −0.342

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[1]) CCDC-819531, -819697, and -825034 contain the supplementary crystallographic data for the bis-isoxazolidines **3f**, **4f**, and **3c**, resp. These data can be obtained, free of charge, *via* http://www.ccdc.cam.ac.uk/data_request/cif.

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