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INTERACTION OF 1-ACETYL-2-PHENYL-5-HYDROXYPYRAZOLIDINES WITH β-SUBSTITUTED ESTERS ON THE SURFACE OF ADSORBENTS

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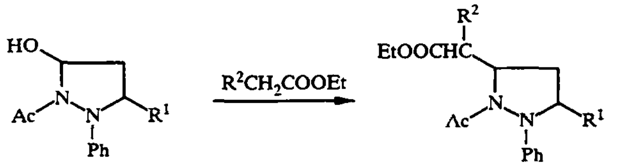
The interaction of 1-acetyl-2-phenyl-5-hydroxypyrazolidines with esters of carboxylic acids, having an electron-acceptor group in the β-position, proceeds under neutral conditions on the surface of adsorbents without preliminary activation. The diastereomeric composition of the isomers obtained was determined for derivatives of cyanacetic and nitroacetic esters; the derivative of acetoacetic ester shows keto–enol tautomerism in solution. The possibility of activation of the reactions on the surface of the adsorbent with uhf irradiation was shown.

We showed previously that the hydroxyl group of 5-hydroxypyrazolidines (I) readily undergoes nucleophilic substitution [1], including that in reactions with ketones as the C-nucleophiles. In accomplishing the last conversion, the application of an unusual method for carrying out the process — in the adsorbed state without a solvent [2], not requiring preliminary activation of the CH-acid — was shown to be very successful. The adsorbent aluminum oxide, employed by us in reactions with ketones and being one of the most common for surface reactions [3], showed unsatisfactory results in the synthesis of derivatives of the esters (II), whereby the reactions had a poor yield or, for example in the case of cyanacetic ester, did not proceed at all [2]. The search, undertaken by us in the present work, for the best conditions in the synthesis of the derivatives (II) showed that there is no universal adsorbent to perform reactions of the pyrazolidines (I) with CH-acid compounds: the individual search for optimal conditions of the adsorbent and the temperature was undertaken in practically every case. Moreover, we showed that the irradiation of the reaction mixture in a microwave oven instead of the usual heating was found to be effective in some cases. The reaction time was thereby shortened in all cases, and the composition of the reaction mixture was changed in the reaction of the pyrazolidine (Ib) with carbethoxycyclopentanone. The optimal reaction conditions found as well as the characteristics of the compounds synthesized are presented in Table 1.



Ia, b IIa-d

I a R^1 = H; b R^1 = Me; II a R^1 = Me; R^2 = COMe; b R^1 = H; R^2 = COOEt;
c R^1 = H; R^2 = NO_2; d R^1 = H; R^2 = CN

Compound (IIa), which we obtained previously on the surface of aluminum oxide [2], is an α-substituted β-ketoester having the capability of keto–enol tautomerism. The spectral data for compound (IIa) (see Tables 1-3), which we synthesized on the surface of polyamide, confirmed its identity with the sample obtained on aluminum oxide. In the same way as previously [2], the spectral characteristics only indicate the presence of the ketonic tautomer — the IR spectrum of the solution in CCl_4 does not contain absorption bands of the enol isomer. According to the data of [2], compound (IIa) has the

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TABLE 1. Characteristics of the Synthesized Compounds (IIa-e) and (IIIe)

Compound	Adsorbent	Reaction time, h	Reaction temperature °C	mp, ° C (R_f)	Found, % / Calculated, % C	Found, % / Calculated, % H	Empirical formula	IR spectrum, cm^{-1}	Yield, %
IIa	Al_2O_3 Polyamide	72 72	60 60	129			$C_{18}H_{24}N_2O_4$	1750, 1725 1680	9 [2] 18
IIb	Al_2O_3 $MgSiO_3$	240 72	60 20	84	62,2 62,7	6,9 6,9	$C_{18}H_{24}N_2O_5$	1740, 1685	7 [2] 52
IIc	Al_2O_3 DEAE	240 72	60 20	87	54,7 55,6	6,1 6,1	$C_{15}H_{19}N_3O_5$	1760, 1690, 1535	Traces 11
IId	$MgSiO_3$	72	20	74	65,4 64,7	7,1 6,8	$C_{16}H_{19}N_3O_3$	2270, 1730, 1680	51
IIe	Gudri catalyst	12 0,5	60 uhf	(0,75)	63,4 67,0	7,8 7,2	$C_{20}H_{26}N_2O_4$	1750, 1720, 1680	0 10
IIIe	Gudri catalyst	12 13	60 uhf	(0,6)	70,7 71,3	8,2 7,7	$C_{17}H_{22}N_2O_2$	1720, 1680	8 18

TABLE 2. PMR Spectra of the Synthesized Compounds (IIa-e) and (IIIe) (δ, ppm)

Compound	Ac (3H)	3-Me (3H)	3-H	4-H (2H)	5-H (1H)	Ph (5H)	α-H (1H)	EtOOC (5H)	R^2
IIa	2,06 s	1,21 d	4,15 m	1,69 m 2,28 d.d	4,97 m	6,96 m, 7,17 m, 7,29 m	3,38 d	1,34, 4,25	2,13 s
IIb	2,09 s	—	3,87 m 3,31 m	1,82 m 2,54 m	4,95 d.d.	7,15 m, 7,35 m	3,45 m	1,20, 4,05	1,31, 4,32
IIc	2,12 s 2,07 s	—	3,45 m 3,95 m	1,94 m 2,59 m	5,15 d.d 5,31 d.d	7,01 m, 7,43 m	4,35 m	1,26, 1,38 4,22, 4,23	
IId	2,10 s 2,15 s	—	3,39 m 3,95 m	2,05 m, 2,45 m	4,77 d.d 5,02 d.d	6,95 m, 7,41 m	3,92 m	1,28, 1,38 4,23, 4,24	
IIe	2,05 s	1,45 d	3,82 m	1,92 m 2,19 m	5,42 d.d	6,67 m 7,18 m	—	1,32, 4,19	1,51 t 1,82 m 2,22 t
IIIe	2,05 s	1,39 d	3,69 m	1,89 m, 2,29 m	5,41 d.d	6,40 m, 7,21 m	4,12 m	—	2,32 m 1,81 m

trans configuration of the pyrazolidine ring. At the same time, we do not even see the doubling of signals in the ^1H and ^{13}C NMR spectra of compound (IIa), having three chiral centers, in connection with the appearance of the new chiral center at the α-carbon atom. It should therefore be assumed that we are seeing the averaged NMR spectrum due to the keto–enol tautomerism in the solution of (IIa) with the virtually complete predominance of the ketonic isomer. Such a predominance is encountered in derivatives of acetoacetic ester having a bulky substituent in the α-position [4]. The X-ray structure analysis of compound (IIa) (see Fig. 1 and Tables 4-7) also indicates the trans configuration of the ring and the ketonic structure of the compound in the crystalline state. The conformation of the heterocycle and the orientation of the substituents are close to those previously established for the initial pyrazolidine (Ib) [5] — the pyrazolidine ring of compound (IIa) occurs in the conformation of the twisted envelope (see Table 4) whereby the deviation of the $C_{(5)}$ atom from the plane $N_{(1)}-N_{(2)}-C_{(3)}-C_{(4)}$ (the plane is satisfied with the accuracy of ± 0.043 Å) comprises -0.526 Å, and the dihedral angle between the plane under consideration and the plane traced through the atoms $N_{(1)}-C_{(5)}-C_{(4)}$ equals 34.8°. The bond lengths and bond angles in the molecule of (IIa) are close to the standard values [6], and no inter- and intramolecular interactions were found (the distances are less than the sums of the van der Waals radii of the atoms [7]).

TABLE 3. ^{13}C NMR Spectra of the Synthesized Compounds (IIb-e) and (IIIe) (δ, ppm)

Compound	Ac	3-Me	$C_{(3)}$	$C_{(4)}$	$C_{(5)}$	Ph	Cα	COOEt	Remaining signals
IIb	175,091 21,571		52,625	31,948	55,957	149,091 *i* 114,558 *o* 129,091 *m* 121,423 *p*	61,692	167,1 60, 167,1 26, 61,178, 13,646	
IIc	177,010 21,103 21,019		52,941	29,661	56,522 56,811	149,292 150,123 *i* 114,402 114,710 *o* 129,557 129,571 *m* 122,021 *p*	63,569	160,0 66 162,7 98 63,135 63,569 13,719 13,659	
IId	176,130 21,148 21,385		53,701	29,655 31,976	56,144	149,433 *i* 115,504 115,474 *o* 129,342 129,473 *m* 122,012 *p*	57,010	164,2 28 63,147 13,646	114,732 (CN)
IIe	174,518 20,845	15,002	52,367	37,683	57,784	148,831 *i* 112,817 *o* 125,443 *m* 117,015 *p*	62,811	174,1 19 57,506 19,355	210,147 (CO) 37,119 26,101 25,634 20,084
IIIe	172,926 23,111	14,661	50,118	36,015	56,271	150,456 *i* 112,352 *o* 125,536 *m* 117,243 *p*	53,356		212,913 (CO) 38,156 25,315 · 23,111

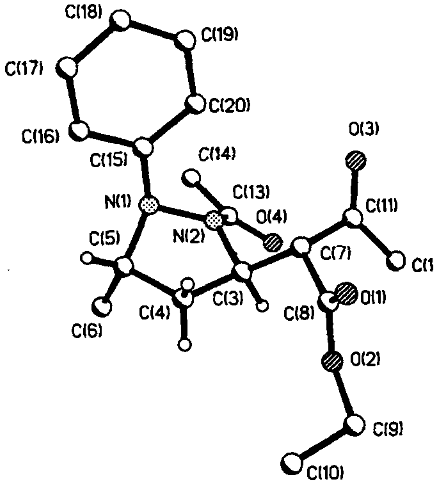


Fig. 1. General view of the molecule of (IIa). Only hydrogen atoms connected to the heterocycle are shown.

The compounds (IIc) and (IId) contain two chiral centers, and therefore we see signals of two diastereomeric pairs in the ^{1}H and ^{13}C NMR spectra. However, their ratio changes, depending on the solvent, from 1:3 in $CDCl_3$ to 1:6 in acetone-D_6. The enolization is probably possible in such systems on account of the high CH-acidity of the ester, and its low rate allows each of the isomers to be seen.

The interaction of the pyrazolidine (IIb) with carbethoxycyclopentanone proceeds more slowly and with more difficulty than the remaining reactions. The formation of only a small amount of the α-pyrazolidinylcyclopentanone (IIIe) was

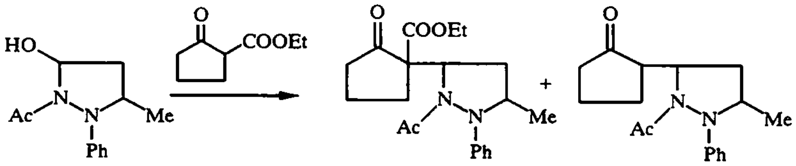
TABLE 4. Bond Lengths in the Molecule of (IIa)

Bond	*l*, Å	Bond	*l*, Å	Bond	*l*, Å
O$_{(1)}$—C$_{(8)}$	1,189(5)	N$_{(2)}$—C$_{(13)}$	1,371(5)	C$_{(11)}$—C$_{(12)}$	1,491(7)
O$_{(2)}$—C$_{(8)}$	1,333(5)	C$_{(3)}$—C$_{(4)}$	1,543(7)	C$_{(13)}$—C$_{(14)}$	1,492(6)
O$_{(2)}$—C$_{(9)}$	1,470(5)	C$_{(3)}$—C$_{(7)}$	1,539(6)	C$_{(15)}$—C$_{(16)}$	1,394(6)
O$_{(3)}$—C$_{(11)}$	1,207(5)	C$_{(4)}$—C$_{(5)}$	1,524(6)	C$_{(15)}$—C$_{(20)}$	1,391(6)
O$_{(4)}$—C$_{(13)}$	1,223(5)	C$_{(5)}$—C$_{(6)}$	1,515(7)	C$_{(16)}$—C$_{(17)}$	1,375(6)
N$_{(1)}$—N$_{(2)}$	1,429(5)	C$_{(7)}$—C$_{(8)}$	1,509(5)	C$_{(17)}$—C$_{(18)}$	1,381(7)
N$_{(1)}$—C$_{(5)}$	1,487(5)	C$_{(7)}$—C$_{(11)}$	1,533(6)	C$_{(18)}$—C$_{(19)}$	1,384(7)
N$_{(1)}$—C$_{(15)}$	1,430(5)	C$_{(9)}$—C$_{(10)}$	1,490(8)	C$_{(19)}$—C$_{(20)}$	1,382(6)
N$_{(2)}$—C$_{(3)}$	1,469(5)				

TABLE 5. Bond Angles ω (deg) in the Molecule of (IIa)

Angle	ω, deg	Angle	ω, deg
C$_{(8)}$—O$_{(2)}$—C$_{(9)}$	117,5(4)	O(1)—C$_{(8)}$—C$_{(7)}$	123,4(4)
N$_{(2)}$—N$_{(1)}$—C$_{(5)}$	103,3(3)	O$_{(2)}$—C$_{(8)}$—C$_{(7)}$	112,1(3)
N$_{(2)}$—N$_{(1)}$—C$_{(15)}$	114,5(3)	O$_{(2)}$—C$_{(9)}$—C$_{(10)}$	110,0(4)
C$_{(5)}$—N$_{(1)}$—C$_{(15)}$	117,7(3)	O$_{(3)}$—C$_{(11)}$—C$_{(7)}$	118,1(4)
N$_{(1)}$—N$_{(2)}$—C$_{(3)}$	111,4(3)	O$_{(3)}$—C$_{(11)}$—C$_{(12)}$	122,1(4)
N$_{(1)}$—N$_{(2)}$—C$_{(13)}$	119,0(3)	C$_{(7)}$—C$_{(11)}$—C$_{(12)}$	119,8(4)
C$_{(3)}$—N$_{(2)}$—C$_{(13)}$	120,8(3)	O$_{(4)}$—C$_{(13)}$—N$_{(2)}$	119,5(4)
N$_{(2)}$—C$_{(3)}$—C$_{(4)}$	103,7(3)	O$_{(4)}$—C$_{(13)}$—C$_{(14)}$	123,2(4)
N$_{(2)}$—C$_{(3)}$—C$_{(7)}$	111,9(3)	N$_{(2)}$—C$_{(13)}$—C$_{(14)}$	117,2(4)
C$_{(4)}$—C$_{(3)}$—C$_{(7)}$	112,1(3)	N$_{(1)}$—C$_{(15)}$—C$_{(16)}$	118,6(4)
C$_{(3)}$—C$_{(4)}$—C$_{(5)}$	104,0(3)	N$_{(1)}$—C$_{(15)}$—C$_{(20)}$	122,3(4)
N$_{(1)}$—C$_{(5)}$—C$_{(4)}$	104,0(4)	C$_{(16)}$—C$_{(15)}$—C$_{(20)}$	118,8(4)
N$_{(1)}$—C$_{(5)}$—C$_{(6)}$	109,9(3)	C$_{(15)}$—C$_{(16)}$—C$_{(17)}$	120,7(4)
C$_{(4)}$—C$_{(5)}$—C$_{(6)}$	113,2(4)	C$_{(16)}$—C$_{(17)}$—C$_{(18)}$	120,8(5)
C$_{(3)}$—C$_{(7)}$—C$_{(8)}$	111,5(3)	C$_{(17)}$—C$_{(18)}$—C$_{(19)}$	118,4(4)
C$_{(3)}$—C$_{(7)}$—C$_{(11)}$	112,0(3)	C$_{(18)}$—C$_{(19)}$—C$_{(20)}$	121,7(4)
C$_{(8)}$—C$_{(7)}$—C$_{(11)}$	112,8(4)	C$_{(15)}$—C$_{(20)}$—C$_{(19)}$	119,5(4)
O$_{(1)}$—C$_{(8)}$—O$_{(2)}$	124,5(3)		

observed on the surface of the Gudri catalyst at 60°C after 12 h — (IIIe) is the product of the decarboxylation of the desired compound (IIe) in the absence of the ester (IIe) itself. We observed such processes previously in reactions on the surface of aluminum oxide [2]. The same interaction on the surface of the same catalyst using irradiation in a microwave oven for 25 min only led to the formation of compound (IIe) having low stability (see Tables 1-3); subsequent irradiation led to the appearance of the decarboxylated product (IIIe), which becomes the sole product after 13.5 h, in the reaction mixture.



Ib IIe IIIe

Compound (IIIe) was also synthesized by the direct interaction of the pyrazolidine (Ib) with cyclopentanone on the surface of the Gudri catalyst (see Tables 1-3).

EXPERIMENTAL

The IR spectra were measured on the UR-20 instrument and the Specord IR-75 in mineral oil or methylene chloride. The ^{1}H and ^{13}C NMR spectra were obtained on the Varian VXR-400 and Bruker WM-250 instruments in CDCl$_3$ with HMDS as the internal standard. The monitoring of the course of reactions and the purity of the compounds obtained was ac-

TABLE 6. Coordinates ($\times 10^4$, and $\times 10^3$ for H) and Isotropic Equivalent Thermal Parameters of Nonhydrogen Atoms (H — Isotropic) in the Molecule of (IIa)

Atom	x	y	z	$U_{(eq)}$
$O_{(1)}$	647(3)	4169(3)	7563(2)	52(1)
$O_{(2)}$	352(3)	2321(3)	7092(2)	44(1)
$O_{(3)}$	2626(3)	2409(3)	9598(2)	45(1)
$O_{(4)}$	3700(3)	-150(2)	8766(2)	40(1)
$N_{(1)}$	5976(4)	2010(3)	8204(2)	28(1)
$N_{(2)}$	4699(3)	1509(3)	8408(2)	27(1)
$C_{(3)}$	3311(5)	1859(4)	7832(2)	29(1)
$C_{(4)}$	3856(5)	2462(4)	7150(3)	33(2)
$C_{(5)}$	5497(5)	2116(4)	7315(2)	32(2)
$C_{(6)}$	5746(6)	953(5)	6929(3)	44(4)
$C_{(7)}$	2364(5)	2696(4)	8211(2)	31(2)
$C_{(8)}$	1036(5)	3165(4)	7591(2)	33(2)
$C_{(9)}$	-888(5)	2680(5)	6420(3)	47(2)
$C_{(10)}$	-316(6)	2968(6)	5704(3)	49(2)
$C_{(11)}$	1932(5)	2128(4)	8932(3)	33(2)
$C_{(12)}$	668(6)	1272(5)	8797(3)	44(2)
$C_{(13)}$	4826(5)	422(3)	8773(2)	28(1)
$C_{(14)}$	6354(6)	34(4)	9210(3)	39(2)
$C_{(15)}$	6557(4)	3044(3)	8653(2)	25(1)
$C_{(16)}$	7687(5)	3674(4)	8431(3)	33(2)
$C_{(17)}$	8345(5)	4632(4)	8875(3)	37(2)
$C_{(18)}$	7913(5)	4982(4)	9554(3)	39(2)
$C_{(19)}$	6813(5)	4339(4)	9783(3)	35(2)
$C_{(20)}$	6134(5)	3377(4)	9344(2)	28(1)
$H_{(3)}$	264(4)	113(4)	766(2)	4(1)
$H_{(41)}$	328(4)	216(3)	667(2)	3(1)
$H_{(42)}$	369(5)	336(4)	717(3)	6(1)
$H_{(5)}$	605(4)	278(4)	714(2)	3(1)
$H_{(61)}$	540(4)	102(3)	635(2)	3(1)
$H_{(62)}$	519(6)	29(5)	709(3)	7(2)
$H_{(63)}$	679(5)	70(4)	707(2)	4(1)
$H_{(7)}$	297(4)	342(3)	839(2)	1(1)
$H_{(91)}$	-146(5)	200(4)	629(2)	4(1)
$H_{(92)}$	-146(6)	336(5)	653(3)	7(2)
$H_{(101)}$	-113(5)	314(4)	524(3)	5(1)
$H_{(102)}$	31(5)	369(4)	579(3)	6(2)
$H_{(103)}$	19(5)	232(5)	554(3)	7(2)
$H_{(121)}$	57(5)	81(4)	836(3)	7(2)
$H_{(122)}$	-42(6)	170(5)	866(3)	7(2)
$H_{(123)}$	71(5)	84(4)	927(3)	6(1)
$H_{(141)}$	630(4)	-73(4)	934(2)	4(1)
$H_{(142)}$	670(5)	49(4)	969(3)	5(1)
$H_{(143)}$	715(6)	28(5)	889(3)	7(2)
$H_{(16)}$	797(4)	349(3)	797(2)	2(1)
$H_{(17)}$	909(4)	505(3)	873(2)	3(1)
$H_{(18)}$	839(4)	559(4)	989(2)	4(1)
$H_{(19)}$	652(4)	455(3)	1024(2)	3(1)
$H_{(20)}$	536(4)	294(3)	950(2)	3(1)

complished by the method of TLC on plates of Silufol UV-254 in the 1:1 system of benzene−ethyl acetate; development was performed by iodine vapor and the alcoholic solution of ferric chloride. Purification of the compounds obtained was carried out by the method of flash-chromatography on silica gel L 40/100 in a gradient of benzene−ethyl acetate.

The following adsorbents were utilized: Al_2O_3 of the firm Reanal, Florisil of the firm Fluka, polyamide of the firm Woelm, and the Gudri catalyst of the Groznyi Chemical Plant (with the Al_2O_3 content of ~15%).

X-Ray Structural Investigation. Crystals of compound (IIa) are monoclinic, and the following apply at −80°C: $a = 9.248(3)$ Å, $b = 11.305(4)$ Å, $c = 17.175(8)$ Å, $\beta = 104.29(3)°$, $V = 1740(2)$ Å^3, $d_{calc} = 1.269$ g/cm^3, $Z = 4$, and the space group is $P2_1/C$. The cell parameters and intensities of the 2528 independent reflections were measured on the Syntex

TABLE 7. Main Torsion Angles τ in the Molecule of (IIa)

Angle	τ	Angle	τ
$N_{(1)}—N_{(2)}—C_{(3)}—C_{(4)}$	9,7	$C_{(15)}—N_{(1)}—C_{(5)}—C_{(6)}$	148,4
$N_{(1)}—N_{(2)}—C_{(13)}—O_{(4)}$	-161,4	$C_{(13)}—N_{(2)}—C_{(3)}—C_{(7)}$	101,6
$N_{(1)}—N_{(2)}—C_{(13)}—C_{(14)}$	22,6	$C_{(9)}—O_{(2)}—C_{(8)}—O_{(1)}$	5,5
$N_{(2)}—N_{(1)}—C_{(5)}—C_{(4)}$	37,1	$C_{(8)}—O_{(2)}—C_{(9)}—C_{(10)}$	90,5
$N_{(2)}—N_{(1)}—C_{(15)}—C_{(16)}$	-172,5	$C_{(9)}—O_{(2)}—C_{(8)}—C_{(7)}$	-175.2
$N_{(2)}—C_{(3)}—C_{(4)}—C_{(5)}$	13,8	$C_{(3)}—C_{(7)}—C_{(8)}—O_{(1)}$	-136,8
$C_{(3)}—C_{(4)}—C_{(5)}—N_{(1)}$	-31,4	$C_{(11)}—C_{(7)}—C_{(8)}—O_{(1)}$	96,2
$C_{(5)}—N_{(1)}—N_{(2)}—C_{(3)}$	-29,6	$C_{(3)}—C_{(7)}—C_{(8)}—O_{(2)}$	43,9
$C_{(7)}—C_{(3)}—C_{(4)}—C_{(5)}$	134,7	$C_{(3)}—C_{(7)}—C_{(11)}—O_{(3)}$	101,4
$C_{(3)}—C_{(4)}—C_{(5)}—C_{(6)}$	87,8	$C_{(8)}—C_{(7)}—C_{(11)}—O_{(3)}$	-131,9
$C_{(15)}—N_{(1)}—N_{(2)}—C_{(3)}$	99,6	$C_{(3)}—C_{(7)}—C_{(11)}—C_{(12)}$	-79,6
$C_{(15)}—N_{(1)}—N_{(2)}—C_{(13)}$	-112,7	$C_{(8)}—C_{(7)}—C_{(11)}—C_{(12)}$	47,1

$P2_1$ four-circle automatic diffractometer (λ MoKα, graphite monochromator, and $\theta/2\theta$-scanning up to the θ_{max} = 26°). The structure was interpreted by the direct method, which showed all the nonhydrogen atoms, and was specified by the full-matrix MLS with the anisotropic approximation for the nonhydrogen atoms by the 1450 reflections with the $I > 3\sigma(I)$. All atoms of hydrogen were shown objectively by difference Fourier syntheses, and were specified isotropically. The final values of the divergence factors are $R = 0.043$ and $R_W = 0.043$ ($S = 1.137$). All calculations were performed using the SHELXTL PLUS program [8] (version PC). Coordinates and isotropic equivalent (isotropic for H) thermal parameters of nonhydrogen atoms are given in Table 6.

General Method for the Synthesis of Compound (II). The initial pyrazolidine and ester are dissolved in the minimum amount of benzene prior to the gradual introduction of the tenfold amount, by weight, of the calcined adsorbent with continuous shaking, which is continued for about 5 min more. The solvent is evaporated to dryness *in vacuo*. The mixture is left at room temperature. It is heated in a thermostat or irradiated in a microwave oven (power 170 W; irradiation is performed for 2-5 min with 5 min intervals; the total irradiation time is presented in Table 1). Monitoring is accomplished by the TLC of samples taken. The compounds obtained are extracted with chloroform. The solvent is evaporated, and purification is by chromatography. The characteristics of the compounds obtained are presented in Table 1. The NMR spectra are presented in Tables 2 and 3. The data of x-ray structural analysis are presented in Tables 4-7.

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