with alcoholic HCl and cooled. The precipitate was recrystallized from alcohol. Obtained: 1.25 g of the bis(hydrochloride) of (IX), 75.7% yield, mp 233-234°C. Found, %: C 47.66; H 6.22. $C_{13}H_{20}N_4O \cdot 2HCl \cdot {}^1/_2H_2O$. Calculated, %: C 47.57; H 6.15.

CONCLUSIONS

The behavior of 5(6)-hydroxybenzimidazole in aminomethylation was studied. 4,6-Bis-substituted Mannich bases were obtained for the first time by selection of the conditions and reagents.

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HETEROARYLADAMANTANES.

COMMUNICATION 2.* STRUCTURE OF 3-(1-ADAMANTYL)-3-CHLOROPROPENAL AND THE SYNTHESIS OF RELATED ADAMANTYL-SUBSTITUTED NITROGEN HETEROCYCLES

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UDC 541.6:547.518:547.446:
542.91:547.73:547.85

We have previously described [1, 2] a method of synthesis of 3-(1-adamantyl)-3-chloropropenal (I), which is a useful synthon for preparing adamantyl-substituted thiophenes and isoxazoles. PMR and IR spectroscopic methods [1] established the trans-Z-configuration of (I). In this paper we present the results of an x-ray investigation of I and further study its reaction properties.

X-ray structural analysis of I is in agreement with the trans configuration of the aldehyde and adamantyl substituents on the $C^2=C^3$ double bond. It has near C_s symmetry with the approximate mirror plane passing through atoms O, C^1, C^2, C^3, Cl, C^4, C^7, C^{11}, and C^{10} (Fig. 1).

Exact C_s symmetry for the adamantyl fragment has previously been observed in the crystal structure of the bis-acid chloride of adamantane-1,3-dicarboxylic acid [3]. Table 1 shows the torsion angles, the configuration of the side chain, and its orientation relative to the adamantyl fragment.

The bond lengths for $O=C^1$ 1.216(3), C^1-C^2 1.462(3), $C^2=C^3$ 1.325(3), and C^3-C^4 1.511(3) Å are close to the corresponding standard values 1.215(5), 1.44(1), 1.339(5), and 1.510(5) Å [4]. The average C–C distance in the adamantane nucleus is 1.532(9) Å, near to that observed in unsubstituted adamantane [1.536(5) Å] [5]. A small increase [to 1.756(2) Å] in the length of the C^3-Cl bond (standard value for $C_{sp^2}-Cl$ 1.736 Å [6]) is probably due to a weak inter-

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*For Communication 1, see [1].

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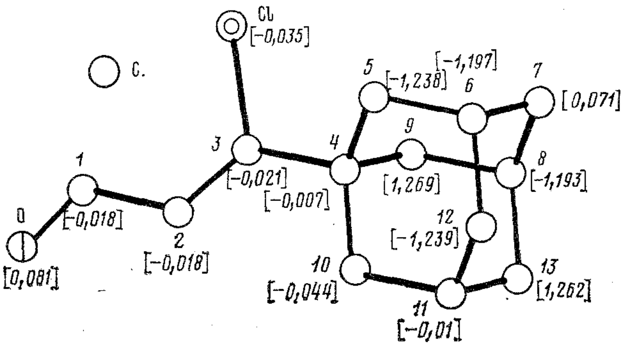


Fig. 1. The geometry of I (H atoms not shown). Deviations (Å) of atoms from the approximate mirror plane of symmetry −0.226x + 109y − 0.958z + 2.489 = 0 (passing through atoms O, C^1, C^2, C^3, Cl, C^4, C^7, C^{11}, and C^{10}) are given in square brackets.

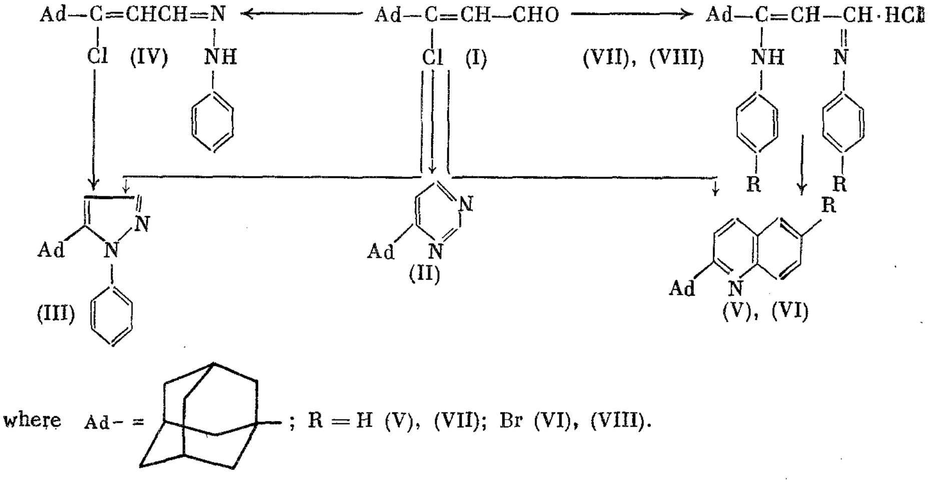
molecular C=O...Cl interaction (see below); this effect on the C–Cl bond length has been proposed, for example, for crystals of terephthaloyl chloride [7].

Deformations in the series of bond angles are observed due to the short intramolecular nonvalence contacts. Angle OC^1C^2 is increased to 122.9(2)° (O...H^2, 2.49(2) Å, sum of van der Waals radii Σ = 2.72 Å [8]), $C^1C^2C^3$ to 126.4(2)° [C^1...Cl 3.047(2) Å, Σ = 3.45 Å], $C^2C^3C^4$ to 127.4(2)°, $C^3C^4C^{10}$ to 110.0(2)° [C^2...C^{10} 2.887(3) Å, Σ = 3.40 Å]. The steric interaction of Cl with C^1 in I is evidently greater than with the adamantane fragment [C^1...Cl 3.047(2) Å, C^5...Cl 3.146(2) Å, C^9...Cl 3.174(2) Å, valence angles C^2C^3Cl 118.3(2)° being greater than ClC^3C^4 114.3(2)°]

There is no shortening of the intermolecular contacts in crystalline I. In the direction of the *a* axis of the molecule, the distance Cl...O [3.447(2) Å] exceeds the sum of the van der Walls radii of Cl and O (3.27 Å [8]). However, the mutual configuration of the groups O=C^1 and ClC^3 [angles C^3–Cl...O 151.8(2)° and Cl...O–C^1 114.9(2)°] is reminiscent of crystalline terephthaloyl chloride (angles CCl...O and Cl...OC 167.5 and 114.5°) in which an attractive C...O interaction is proposed at a distance of 3.32 Å [7]. Such an attraction is also possible for I.

A study of the reaction of I with formamide, phenylhydrazine hydrochloride, aniline, and p-bromoaniline has pointed to the possibility of preparing various adamantyl-substituted nitrogen heterocycles (Scheme 1).

Scheme 1



Compound I reacts with formamide at 190°C to give 4-(1-adamantyl)pyrimidine (II) in 32% yield.

TABLE 1. Torsion Angles τ and Substituent Conformation and Orientation Relative to the Adamantane Nucleus for Molecule I

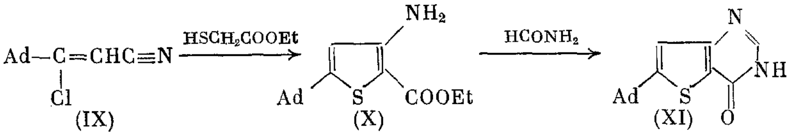
Angle	τ, deg
$OC^1C^2C^3$	174,6(4)
$C^1C^2C^3C^4$	−179,3(4)
$C^2C^3C^4C^{10}$	−2,3(3)
$C^2C^3C^4C^5$	−122,5(3)
$C^2C^3C^4C^9$	118,4(3)

The reaction of I with phenylhydrazine hydrochloride in refluxing alcohols is accompanied by much tarring and leads to the formation of 1-phenyl-5-(1-adamantyl)pyrazole III, isolated in only 18% yield by column chromatography. The intermediate 3-(1-adamantyl)-3-chloropropenal hydrazone (IV) is formed when I is treated with phenylhydrazine hydrochloride in ethanol at ~20°C in the presence of sodium acetate. It yields pyrazole III in 40% yield when heated in H_3PO_4.

Compound I reacts with aniline or p-bromoaniline to give 2-(1-adamantyl)quinoline (V) or 2-(1-adamantyl)-6-bromoquinoline (VI) in good yields. Heterocyclization of the enaminimine salts VII and VIII (obtained by treating I with aniline or p-bromoaniline in ethanol at 20°C) also gives good yields of V or VI.

We have previously prepared 3-(1-adamantyl)-3-chloroacrylonitrile (IX) from I [1]. Condensation of IX with ethyl trioglycolate in the presence of sodium ethoxide gives X. The latter can react with formamide at 190-200°C to give 6-(1-adamantyl)thieno[2,3]pyrimidin-4-one (XI) (Scheme 2).

Scheme 2



The basic course of fragmentation of the synthesized heteroaryladamantanes under electron impact consists of the formation of $[M - C_3H_7]^{+\cdot}$ and $[M - C_4H_9]^{+\cdot}$ ions as a result of breaking of the adamantane portion.

EXPERIMENTAL

GLC analysis of the reaction mixtures and separated compounds was carried out on an LKhM-8MD chromatograph using helium as the carrier gas, flame-ionization detector, and a 1.5 m × 3 mm column consisting of Dexsil-300 on Chromosorb W. IR spectra were measured on a UR-20 spectrophotometer as KBr disks, UV spectra on a Specord UV-VIS spectrophotometer, PMR spectra (δ, ppm) on a Varian DA-60 instrument with HMDS as internal standard, and mass spectra on a Varian MAT CH-6.

4-(1-Adamantyl)pyrimidine (II). Finely divided I (2.24 g) was added to formamide (15 ml) at 190°C over 10 min; after 1.5 h at 190-200°C the mixture was cooled, poured into water (100 ml), and extracted with ether. Ether was evaporated off and the residue was steam distilled, filtered, and sublimed at 60°C (2 mm). Compound II was obtained in 0.7 g (32%) yield, mp 72.5-73°C. PMR spectrum ($CDCl_3$): 1.6-2.4 m (15H, Ad), 7.17 dd (1H, H^5, J = 5.5, 1.5 Hz), 8.55 d (1H, H^6, J = 5.5 Hz), 9.08 br s (1H, H^2). Found, %: C 78.0; H 8.6; N 13.1, $M^{+\cdot}$ m/z 214. $C_{14}H_{18}N_2$. Calculated, %: C 78.5; H 8.5; N 13.1, M 214.3.

3-(1-Adamantyl)-3-chloropropenal Phenylhydrazone (IV). A solution of sodium acetate (0.9 g, 11 mmoles) and phenylhydrazine hydrochloride (1.59 g, 11 mmoles) in a mixture of EtOH (10 ml) and water (10 ml) was added to 2.24 g (10 mmoles) of I in EtOH (30 ml). After 24 h

TABLE 2. Atomic Coordinates ($\cdot 10^5$ for Cl, 10^4 for O and C, and 10^3 for H)

Atom	X	Y	Z	Atom	X	Y	Z
Cl	42164(10)	17378(6)	23264(8)	H^1	117(4)	349(3)	313(3)
O	−1498(3)	3585(2)	3543(2)	H^2	−31(3)	97(2)	322(3)
C^1	156(4)	2999(2)	3267(3)	$H^{5.1}$	490(4)	−125(3)	435(3)
C^2	611(3)	1441(2)	3047(3)	$H^{5.2}$	592(4)	−96(3)	321(3)
C^3	2298(3)	738(2)	2638(3)	H^6	669(4)	−339(3)	371(3)
C^4	2794(3)	−867(2)	2388(2)	$H^{7.1}$	589(4)	−444(2)	134(3)
C^5	4974(3)	−1465(2)	3383(2)	$H^{7.2}$	667(4)	−291(3)	122(3)
C^6	5469(3)	−3087(2)	3099(3)	H^8	344(3)	−299(2)	−46(2)
C^7	5572(3)	−3372(2)	1505(3)	$H^{9.1}$	391(4)	−69(3)	54(3)
C^8	3414(3)	−2813(2)	515(3)	$H^{9.2}$	170(4)	−75(3)	20(3)
C^9	2918(3)	−1189(2)	778(3)	$H^{10.1}$	−28(4)	−128(3)	210(3)
C^{10}	1087(3)	−1660(2)	2733(3)	$H^{10.2}$	91(4)	−141(2)	370(3)
C^{11}	1595(3)	−3289(2)	2455(3)	H^{11}	60(4)	−366(3)	268(3)
C^{12}	3759(4)	−3859(2)	3452(3)	$H^{12.1}$	362(4)	−368(3)	450(3)
C^{13}	1685(3)	−3575(2)	859(3)	$H^{12.2}$	410(3)	−491(2)	325(3)
				$H^{13.1}$	34(5)	−318(3)	20(3)
				$H^{13.2}$	203(4)	−461(2)	71(3)

TABLE 3. Bond Lengths

Bond	d, Å	Bond	d, Å
C^1-O^1	1,216(3)	C^6-C^{12}	1,524(3)
C^1-C^2	1,462(3)	C^7-C^8	1,532(3)
C^2-C^3	1,325(3)	C^8-C^9	1,529(3)
C^3-Cl	1,756(2)	C^8-C^{13}	1,525(3)
C^3-C^4	1,511(3)	C^9-C^4	1,546(3)
C^4-C^5	1,553(3)	$C^{10}-C^{11}$	1,537(3)
C^4-C^{10}	1,531(3)	$C^{11}-C^{12}$	1,540(4)
C^5-C^6	1,532(3)	$C^{11}-C^{13}$	1,525(4)
C^6-C^7	1,524(4)		

the precipitated solid was filtered off and recrystallized from EtOH. Compound IV was obtained in a yield of 2.29 g (72.8%), mp 149-151°C. PMR spectrum ($CDCl_3$): 1.5-2.2 m (15H, Ad), 6.21 d (1H, C=CH, J = 9 Hz), 6.55-7.6 m (6H, H_{arom} + NH), 7.43 d (1H, CH=N, J = 9 Hz). Found, %: C 72.7; H 7.5; N 8.9; Cl 11.3, $M^{+\cdot}$ m/z 314, 316. $C_{19}H_{23}N_2Cl$. Calculated, %: C 72.5; H 7.4; N 8.9; Cl 11.3, M 314.9.

1-Phenyl-5-(1-adamantyl)pyrazole (III). a) A mixture of I (2.24 g, 10 mmoles), phenylhydrazine hydrochloride (1.54 g, 11 mmoles), and EtOH (30 ml) was refluxed (4 h) and then cooled, poured into 50 ml of saturated Na_2CO_3 solution, and extracted with ether. The ether extracts were dried ($MgSO_4$), the ether distilled off, and the residue column chromatographed on silica gel (L 40/100, 25 × 1.5 cm) using CCl_4—$MeCO_2Et$ (10:1). Removal of the solvent gave 0.5 g (18%) of III, mp 135-136°C.

b) A mixture of IV (0.94 g, 3 mmoles) in H_3PO_4 (20 ml) was heated for 2 h at 160°C, cooled, poured into 100 ml of a 10% solution of KOH, and extracted with ether. The extract was dried ($MgSO_4$). Workup as in (a) gave 0.4 g (48%) of III, mp 135-136°C. PMR spectrum: (C_6D_6): 1.3-2.0 m (15H, Ad), 6.03 d (1H, H^4, J = 1.5 Hz), 6.8-7.3 m (5H, H_{arom}), and 7.53 d (1H, H^3, J = 1.5 Hz). Found, %: C 81.8; H 8.0; N 10.1%. $M^{+\cdot}$ m/z 278. $C_{19}H_{22}N_2$. Calculated, %: C 82.0; H 8.0; N 10.1%. M 278.4.

Enaminimine Salt (VII). Aniline (0.93 g, 10 mmoles) in ethanol (10 ml) was added to I (1.12 g, 5 mmoles) in C_6H_6 (30 ml) and the mixture allowed to stand at 20°C for 3 days. The crystals produced were filtered off, washed with 50 ml of ether, and dried in a desiccator over P_2O_5; 1.6 g (81.1%) of VII was obtained with mp 199-221°C (decomp). UV spectrum [EtOH, λ_{max}, nm (log ε)]: 248(4.17), 276(4.46). Found, %: C 76.9; H 7.3; N 7.0. $[M-HCl]^{+\cdot}$ m/z 356. $C_{25}H_{29}N_2Cl$. Calculated, %: C 76.4; H 7.4; N 7.1. M 392.98.

Enaminimine Salt (VIII). p-Bromoaniline (2.58 g, 15 mmoles) in 20 ml EtOH was added to I (1.69 g, 7.5 mmoles) in C_6H_6 (20 ml) and the mixture allowed to stand for 3 days at 20°C.

TABLE 4. Valence Angles

Angle	ω, deg	Angle	ω, deg
$O^1C^1C^2$	122,9(2)	$C^5C^6C^7$	109,9(2)
$C^1C^2C^3$	126,4(2)	$C^5C^6C^{12}$	109,8(2)
C^2C^3Cl	118,3(2)	$C^6C^7C^8$	109,2(2)
$C^2C^3C^4$	127,4(2)	$C^6C^{12}C^{11}$	109,0(2)
ClC^3C^4	114,3(2)	$C^7C^6C^{12}$	109,9(2)
$C^3C^4C^5$	110,4(2)	$C^7C^8C^9$	109,0(2)
$C^3C^4C^9$	110,1(2)	$C^7C^8C^{13}$	109,9(2)
$C^3C^4C^{10}$	111,0(2)	$C^8C^{13}C^{11}$	109,4(2)
$C^4C^5C^6$	109,7(2)	$C^9C^4C^{10}$	109,0(2)
$C^4C^9C^8$	110,4(2)	$C^9C^8C^{13}$	110,8(2)
$C^4C^{10}C^{11}$	110,4(2)	$C^{10}C^{11}C^{12}$	109,2(2)
$C^5C^4C^9$	108,0(2)	$C^{10}C^{11}C^{13}$	109,8(2)
$C^5C^4C^{10}$	108,4(2)	$C^{12}C^{11}C^{13}$	109,5(2)

After workup as for VII, 3.42 g (82.6%) of VIII was obtained with mp 195-198°C (decomp.) UV spectrum [EtOH, λ_{max}, nm (log ε)]: 226(4.0), 245(3.9), 390(4.2). Found, %: C 54.3; H 5.2; N 5.0, $[M - HCl]^{+\cdot}$ m/z 514. $C_{25}H_{27}N_2BrCl$. Calculated, %: C 54.4; H 4.9; N 5.1. M 550.8.

2-(1-Adamantyl)quinoline (V). a) A mixture of I (1.12 g, 5 mmoles), aniline (0.45 g, 5 mmoles), and glacial acetic acid (10 ml) was refluxed for 1.5 h, then cooled, poured into concentrated KOH solution (100 ml), and extracted with ether. After drying ($MgSO_4$), the ether was distilled off and the residue chromatographed on silica gel L 40/100 (25 × 1.5 cm) in CCl_4–C_6H_{14}–$MeCO_2Et$ (5:5:1). The solvent was removed and the residue recrystallized from EtOH to give V (0.57 g, 43.5%), mp 94.5-95.5°C (see [9]).

b) A mixture of VII (1.96 g, 5 mmoles) and 15 ml of glacial acetic acid were refluxed for 1.5 h, cooled, poured into concentrated KOH solution (100 ml), and extracted with ether. The ether extracts were washed with water and dried ($MgSO_4$). The ether was removed and the residue chromatographed on a silica gel column (40/100, 25 × 1.5 cm) using CCl_4–C_6H_{14}–Et_2O (5:5:1). The solvent was distilled off and the residue recrystallized from EtOH. Compound V (0.81 g, 61.1%) was obtained with mp 94.5-95.5°C.

2-(1-Adamantyl)-6-bromoquinoline (VI). a) A mixture of 1.12 g (5 mmoles) of I and p-bromoaniline (1.03 g, 5 mmoles) in glacial acetic acid (10 ml) was refluxed for 1.5 h and worked up as described in the previous experiment. The unpurified product was column chromatographed on silica gel (L 25 × 1.5 cm) using CCl_4; after distillation of the solvent the product was recrystallized from EtOH, and VI (0.73 g, 42.8%) was obtained with mp 138.5-139.5°C.

b) A mixture of VIII (2.75 g, 5 mmoles) and glacial acetic acid (20 ml) was refluxed for 15 h and worked up as in experiment (a). Compound VI (1.04 g, 60.8%) was obtained with mp 138.5-139.5°C. PMR spectrum [$(CD_3)_2CO$]: 1.7-2.3 m (15H, Ad), 7.5-8.3 m (5H, H_{arom}), UV spectrum [EtOH, λ_{max}, nm (log ε)]: 225(3.2), 234(3.5). Found, %: C 66.7; H 6.2; N 4.4; Br 23.3. $M^{+\cdot}$ m/z 341/343. $C_{19}H_{20}NBr$. Calculated, %: C 66.7; H 5.9; N 4.1; Br 23.3. M 342.3.

Ethyl 3-Amino-5-(1-adamantyl)thiophene-2-carboxylate (X). To an alcohol solution of EtONa [prepared from Na (0.39 g, 17 mmoles) and absolute EtOH (20 ml)] there was added dropwise 1.65 g (7.5 mmoles) of 3-(1-adamantyl)-3-chloroacrylonitrile (IX) in absolute EtOH (20 ml). After 1 h (20°C) the solvent was removed and the residue extracted with hot hexane. After cooling, the crystals were filtered off to give X (0.92 g, 40.2%), mp 155-156°C. PMR spectrum ($CDCl_3$): 1.7-2.1 m (15H, Ad), 1.25 t (3H, Me), 4.63 q (2H, CH_2), 6.04 br s (2H, NH_2), 6.40 s (1H, thiophene), IR spectrum (KBr, ν, cm^{-1}): 1670 (CO), 3358 (NH), UV spectrum in EtOH, λ_{max}, nm (log ε): 230(3.62), 270(3.77), 319(3.36). Found, %: C 67.1; H 7.5; N 4.7; S 10.5. $M^{+\cdot}$ m/z 305. $C_{17}H_{23}O_2NS$. Calculated, %: C 66.9; H 7.6; N 4.6; S 10.5. M 305.4.

6-(1-Adamantyl)thieno[2,3-e]pyrimidin-4-one (XI). A mixture of X (0.47 g, 1.5 mmoles) and formamide (15 ml) was refluxed for 2 h, then cooled and poured into water (150 ml). The precipitate was filtered off and recrystallized from alcohol. Compound XI (0.38 g, 86.5%), mp 239-240°C. IR spectrum (KBr, ν, cm^{-1}): 1665 (CO). Found, %: C 67.0; H 6.2; N 9.6; S 11.5. $M^{+\cdot}$ m/z 286. $C_{16}H_{18}ON_2S$. Calculated, %: C 67.1; H 6.0; N 9.8; S 11.2. M 286.4.

X-Ray Analytical Investigation of 3-(1-adamantyl)-3-chloropropenal (I). Triclinic crystals, $a = 6.535(3)$, $b = 9.382(4)$, $c = 9.402(4)$ Å, $\alpha = 92.97(3)$, $\beta = 103.55(3)$, $\gamma = 81.50(3)°$, $d_{calc} = 1.35\ g/cm^3$, Z = 2, $C_{13}H_{17}OCl$, space group $P\bar{1}$.

The unit cell parameters and intensities of 2744 reflections were measured at −120°C on an automatic four-circle Syntex $P2_1$ diffractometer (λMo K_α, graphite monochromator, $\theta/2\theta$ scan to $2\theta_{max}$ = 55°C). After averaging of equivalent reflections and rejection of weak reflections ($I \leqslant 2\sigma$), 2368 reflections were used in subsequent calculations. The structure was solved by the MULTAN program and refined by the block diagonal method of least squares in the anisotropic approximation for atoms Cl, O, and C, and in the isotropic approximation for H (all located in difference Fourier synthesis). The final values of the divergence factors were $R = 0.067$ and $R_W = 0.057$. Atomic coordinates are given in Table 2 and the main bond lengths and valence angles in Tables 3 and 4.*

CONCLUSIONS

1. 3-(1-Adamantyl)-3-chloropropenal is a useful synthon for obtaining adamantyl-substituted nitrogen heterocycles: 2-(1-adamantyl)quinoline, 2-(1-adamantyl)-6-bromoquinoline, 6-(1-adamantyl)pyrimidine, 1-phenyl-5-(1-adamantyl)pyrazole, and 6-(1-adamantyl)thieno[2,3-e]-pyrimidin-4-one.

2. X-ray crystallographic analysis points to the trans configuration of the aldehydic and adamantyl substituents on the C=C double bond in the molecule of 3-(1-adamantyl)-3-chloropropenal which is characterized by near C_s symmetry. A small increase in the C—Cl bond length in this molecule is probably due to a weak intermolecular attractive interaction at a distance of 3.447(2) Å.

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*Equivalent isotropic thermal parameters can be obtained from the authors.