WEB OF SCIENTIST: INTERNATIONAL SCIENTIFIC RESEARCH JOURNAL
ISSN: 2776-0979, Volume 7, Issue 3, March- 2026

IR AND PMR - SPECTRUM - ANALYSIS OF 1-BENZOYL-3-(4-BROMPHENYL) -5-HYDROXY-5-TRIFLUOROMETHYL-2-PYRAZOLINE LIGAND

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Abstract

This paper presents an analysis of IR and PMR spectra 1-benzoyl-3-(4-bromophenyl)-5-hydroxy-5-trifluoromethyl-2-pyrazoline, obtained as a result of scientific research.

Keyword: Aroyltrifluoroacetylmethanes, NMR, IR spectrum, 1-benzoyl-3-(4-bromophenyl)-5-hydroxy-5-trifluoromethyl-2-pyrazoline, cis-enol structure, intrachelate migration. carbonyl aroyl group.

INTRODUCTION

Aroyltrifluoroacetylmethanes exist in solutions as conjugated enols. In the PMR spectra of these diketones, which we specially recorded, in full agreement with the literature data [2], there are no signals that could be attributed to the diketone form. According to previously performed spectroscopic studies, an equilibrium between two cis-enol structures occurs for these 1,3-diketones, the mutual transition of which occurs due to rapid intrachelate migration of a proton from the carbon atom to the oxygen of the carbonyl group and the redistribution of bonds, with the cis-enol tautomers present in fairly comparable quantities [3].

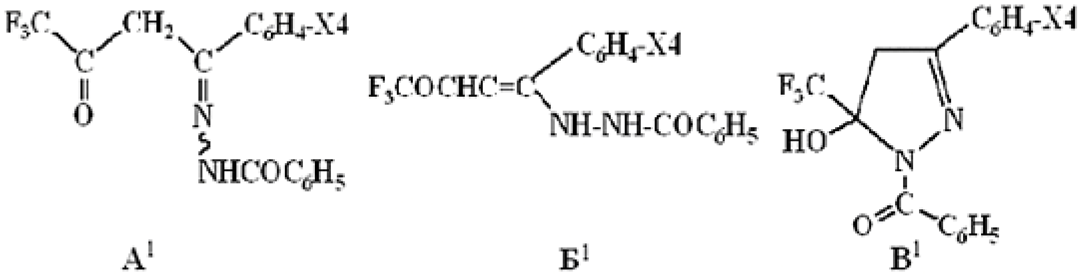
LITERATURE

According to the data, aroyltrifluoroacetylmethanes can react with nucleophilic reagents either as cis-enols or as dicarbonyl compounds, even if the concentration of the diketone form is not detectable by NMR. In [5], using a special jet NMR method, it was shown that almost completely enolized 1,3-diketones (including trifluoroacetylacetone $CH_3COCH_2COCF_3$) react with hydrazine, methyl- and phenylhydrazines in the diketone form. The low concentration of the reacting form is more than compensated by its high reactivity compared to the cis-enol form, which can be considered as a kind of vinyl analogue of the acidic structure [9].

The interaction of 1,3-dicarbonyl compounds with acylhydrazines has not been studied in this context. Therefore, it remains unclear in what form the fluorinated 1,3-diketones we selected will react with acylhydrazines. However, given the above data, it seems more likely that the diketone form is involved in the reaction. Based on this, we will discuss the obtained experimental data in the context of possible competition between condensation processes in the direction of trifluoroacetyl and aroyl carbonyls [4].

RESEARCH METHODOLOGY

When conducting the interaction of benzoylhydrazine with a series of aroyltrifluoroacetylmethanes $CF_3COCH_2COC_6H_4X$-4 (X = H, CH_3, CH_3O, Cl, Br, NO_2), where the substituent in the aromatic ring of the 1,3-dicarbonyl component was varied, under mild conditions (mixing alcohol solutions of reagents at room temperature without heating and the absence of any catalysts), condensation products at the aroyl carbonyl (compounds H_2L^1–H_2L^6 structure I) were obtained, the structure of which was proven by IR and NMR methods ^1H and With spectroscopy.



A^1 $Б^1$ $В^1$

X = H (H_2L^1), CH_3 (H_2L^2), CH_30 (H_2L^3), Cl (H_2L^4), Br (H_2L^5), NO_2 (H_2L^6)

ANALYSIS RESULTS

A study of the IR spectrum of the compound H2L5 in a pressed KBr tablet revealed a number of bands in the region of stretching vibrations of multiple bonds, including an absorption band at approximately 1730 cm^{-1} (Fig. 2). This indicates that the obtained compound H2L5 in the solid state has a cyclic 5-hydroxypyrazoline structure and that condensation of the nucleophile occurs at the carbonyl of the aroyl group .

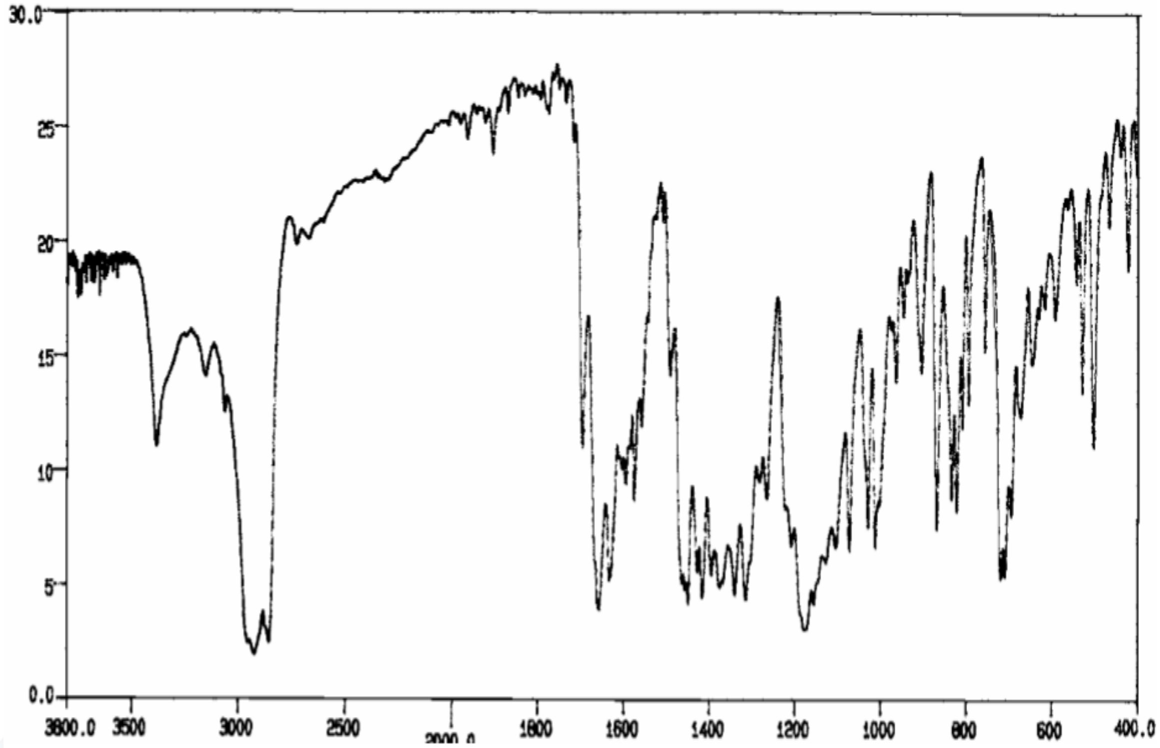


Fig. 1. IR spectrum of the ligand 1-benzoyl-3-(4-bromophenyl)-5-hydroxy-5-trifluoromethyl-2-pyrazoline H_2L^5 structure I

This conclusion is also confirmed by the ν(O–H) absorption bands in the region of ~3400–3500 cm^{-1} and 1660 cm^{-1}, which we attributed to the stretching vibrations of the hydroxyl group bound to the fifth carbon atom of the heterocyclic pyrazoline ring in the H_2L^5 molecule of structure (I) and the amide fragment ν(C=O) of the hydrazone part of the molecule. In the IR spectra of the XLII-type ligands under consideration, intense absorption bands were recorded at 1633, 1594, 1574, 1558 and 1490 cm^{-1} in the region of stretching vibrations of multiple bonds. The absorption band at 1633 cm^{-1} indicates the presence of the C=N bond, while the remaining absorption bands belong to vibrations of the sesquivalent bonds of the aromatic ring and to deformation vibrations of the NH bond. The spectrum (Fig. 2) also contains absorption bands characteristic of C–F bonds. The bands of medium and strong intensity at 1238–1277, 1119–1131, and 1039–1059 cm^{-1} have been assigned by us to the νs and νas C–F bonds. The bands of medium intensity at 755 cm^{-1} correspond to fan vibrations, the weak absorption bands at 528 cm^{-1} are caused by deformation vibrations, and 541 cm^{-1} is due to torsional vibrations of these bonds [7, 8]. The IR spectra of the remaining compounds of type I largely coincide with the IR spectrum of H_2L^1, with a slight difference [9, 10, 14].

However, IR spectroscopy does not allow us to indicate the regional direction of the condensation reaction at the carbonyl group adjacent to the $–CF_3$ or $–C_6H_4X$-4 fragment, and the results are somewhat contradictory. In order to unambiguously prove the conclusions about the structure of the obtained ligands in the solid state and in solutions, we recorded the PMR spectra of these compounds immediately after preparing the solutions and after some time. For example, in the PMR spectrum of a solution of the compound H_2L^3 in $CDCl_3$, recorded immediately after preparation (Fig. 3) and reflecting the structure of the substance in the solid state, a set of signals corresponding to the 5-hydroxypyrazoline structure is observed. Two asymmetric doublets with a total intensity of two protons, at δ 3.57 and 3.73 ppm and with a spin-spin interaction constant (SSIC) of 20 Hz, belonging to the methylene group of the ring, a singlet signal at δ 3.87 ppm. three protons with an intensity corresponding to the CH_3 group of the aromatic ring of the β-dicarbonyl part of the molecule, a signal at δ 6.82 ppm with an intensity of one proton – the OH group, a set of signals in the region of δ 6.91-8.03 ppm belonging to 9 protons of the aromatic rings (Fig. 1, Table 1).

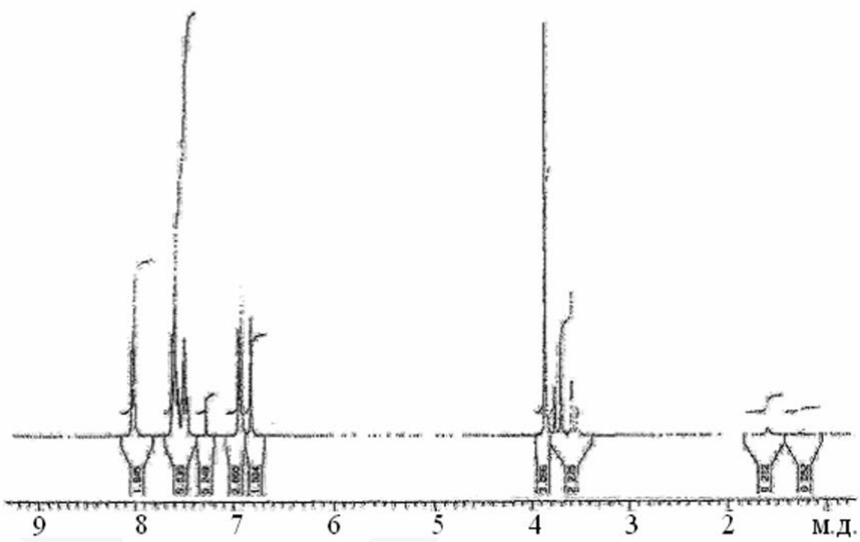


Fig. 2. NMR spectrum of 1-benzoyl-3-(4-methoxyphenyl)-5-hydroxy-5-trifluoromethyl-2-pyrazoline (H_2L^3) structure I in $CDCl_3$ solution

The spectrum does not change over time, indicating the absence of possible tautomeric transitions to the hydrazone A_1 or enhydrazine B_1 forms. These data, of course, do not allow us to conclude that we are dealing with a condensation product specifically at the aroyl carbonyl. This conclusion follows from examining the ^{13}C NMR spectrum .

Its most characteristic feature is the presence of a quartet signal at δ 93.58 ppm (spin coupling constant 32 Hz). It belongs to the carbon atom in position 5 of the ring; the cleavage is due to interaction with the adjacent trifluoromethyl group. The signal of the methoxy carbon group of the aromatic ring is recorded at δ 153.36 ppm, the C=N bond at δ 162.33 ppm, and the C=O bond at δ 171.36 ppm.

Table 1. Parameters of the NMR spectra of 1-benzoyl-3-aryl-5-hydroxy-5-trifluoromethyl-2-pyrazolines (I) in CDCl $_{3\ \text{solution}}$ (δ, ppm)

Соедине-ние	X	CH_2*	OH	C_6H_5	X
H_2L^1	H	3.59; 3.73	6.72	7,45м;7,55м;7,96м	–
H_2L^2	CH_3	3.57; 3.75	6,77	7,25м;7,56м;8,02м	2,44
H_2L^3	OCH_3	3.57; 3.73	6.82	6,91м; 8,03м	3,87
H_2L^4	Cl	3,58;3,76	6.72	7,45м; 8,01м	–
H_2L^5	Br	3,56; 3,75	6,74	7,50м; 7,95м	–
H_2L^6	NO_2	3.61; 3.80	6.60	7,48м;7,63м;8,05м	–

* Note: JAB = 20–22 Hz.

CONCLUSION

1. Using the PMR spectroscopy method, it was established that the synthesized organic ligands based on aroyltrifluoroacetylmethanes in the crystalline state and in CDCl $_{3\ \text{solution}}$ they are in the 5-oxypyrazoline form, and in solution DMSO-d $_6$ cyclic form is in equilibrium with the linear hydrazone form .
2. The presence of aroyltrifluoroacetylmethanes in the enol state in solution was determined based on the results of their NMR spectrum.
3. These ligands are 5- oxo- pyrazoline in the solid state, when the nucleophilic condensation of the aroyl to the carbonyl is studied by IR spectroscopy.

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