INFRARED SPECTRA OF THIOPHENE ANALOGS OF CHALCONES AND THEIR VINYLOGS

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The IR spectra of 45 thiophene analogs of chalcones and vinylogs, and (2-thienyl)phenylketone are determined in the 1700–650 cm^{-1} region, and frequency assignments made. It is confirmed that the carbonyl group and aliphatic double bond are so situated with respect to one another that there is approximation to the S-cis form. It is shown that in the systems studied, 2-thienyl has an electron-donating effect, exceeding that of 2-furyl. It is also found that for 1-(2-thienyl)-3-arylpropen-1-ones the carbonyl group frequency shift due to substituents tends to correlate with the Hammett σ parameters.

Previous communications have dealt with results of studies of UV and visible region absorption spectra [1] and dipole moments [2] of a regular series of α, β unsaturated ketones containing the thiophene ring. Continuing work on elucidating relationships between properties and structures of these compounds, we decided to measure their IR spectra.

The literature information on IR spectra of thiophene derivative carbonyl compounds is rather limited. There are relevant data only for thiophene aldehyde, 2-acetothienone, thiophene carboxylic acids, esters of these, and some other compounds [3–8]. Hitherto the vibration spectra of thiophene analogs of chalcone and their vinylogs have not been investigated.

We have carried out measurements, generally on solids, but in some cases on carbon tetrachloride and chloroform solutions, of the IR spectra of 45 thiophene analogs of chalcones and of their nearest vinylogs, as well as, for comparison, (2-thienyl)phenylketone. Tables 1 and 2 give the characteristic vibration frequencies of the individual groups, and some absorption plots are given in Fig. 1.

All the α, β unsaturated ketones investigated showed an absorption band whose intensity varied from medium to high, characteristic of out-of-plane deformation vibrations of hydrogens of the vinylene group trans to the substituents. Generally the band was at 965 cm^{-1} [9], but here, as a result of conjugation with the carbonyl group, or with the aromatic and thiophene rings it is displaced in the higher frequency direction, and lies at 970–1000 cm^{-1}. With compounds containing two (XXVIII–XLII) and three (XLIII–XLV) double bonds, this absorption is particularly intense; it lies in the 990–1015 cm^{-1} region, and sometimes consists of two bands. It is noteworthy that, as a rule, the intensity of a carbonyl group valence vibrations band is considerably less than the intensity of absorption of an aliphatic double bond (see Fig. 1). It is known that this kind of intensity relationship for the C=O and C=C bonds bands is an analytical indicator of their being S-trans to one another [10–13]. Thus the IR spectra data confirm the

conclusions previously drawn from studies of dipole moments [2], that the thiophene analogs of chalcones and their vinylogs studied by us are trans isomers as
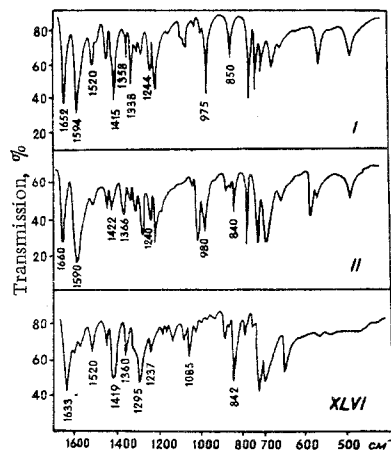

Fig. 1. IR spectra of compounds I, II, and XLVI.

far as the relative position of substituents and aliphatic double bond is concerned, while the relative position of the carbonyl and vinyl groups corresponds to S-cis conformations. It is of interest that the conclusion drawn [10] that $\Delta\nu = \nu_{C=O} - \nu_{C=C}$ should be over 60 cm^{-1} for the S-cis form, is not in accord with what is found for many of our ketones (see Table 1), while the figure of 75 cm^{-1} [14,15] is in even poorer agreement.

All the thiophene ketones investigated show a sharply defined absorption maximum in the 1635–1670 cm^{-1} region characteristic of valence vibrations of the carbonyl group. In the case of measurements made in chloroform solution (see Table 2), $\nu_{C=O}$ is 5–11 cm^{-1} lower than the corresponding ones in carbon tetrachloride, in agreement with reported inferences regarding the effect of the hydrogen bond formed (see [16], p. 393). This confirms a view which three of us put forward from a study of electronic spectra, about hydrogen bonding of α, β unsaturated ketones of the thiophene series in ethanol and chloroform solution [1]. In comparison with solutions in carbon tetrachloride, $\nu_{C=O}$ in the solid phase is lower by 3–8 cm^{-1}, which can be put down to a change in the C=O bond force constant due to the action of the crystal field, and to weakening of the dipole orientation (see [16], p. 380).

Replacement of the benzene ring in chalcone ($\nu_{C=O}$ = 1669 cm^{-1}; $\nu_{C=C}$ = 1610 cm^{-1}) [17] by a

Table 1

Characteristic Frequencies of Thiophene Analogs of Chalcones and Their Vinylogs

Compound	Name	Formula	C=O	C=C	Ring vibrations			Out-of-plane C–H		Trans CH=CH
I	1-(2-Thienyl)-3-phenylpropen-1-one	C_4H_3S—CO—CH=CH—C_6H_5*	1652	1594	1415	1358	1244	850	860	975
II	1-(2-Thienyl)-3-phenylpropen-3-one	C_4H_3S—CH=CH—CO—C_6H_5	1660	1590	1422	1366	1240	840	870	980
III	1,3-Bis(2-thienyl)propen-1-one	C_4H_3S—CO—CH=CH—C_4H_3S	1640	1575	1412	1353	1246	830	860	975
IV	1-(2-Thienyl)-3-(4-methylphenyl)propen-1-one	C_4H_3S—CO—CH=CH—C_6H_4—CH_3	1650	1590	1415	1354	1240	845	860	990
V	1-(2-Thienyl)-3-(4-methylphenyl)propen-3-one	C_4H_3S—CH=CH—CO—C_6H_4—CH_3	1661	1595	1425	1366	1240	840	850	965
VI	1-(2-Thienyl)-3-(4-methoxyphenyl)propen-1-one	C_4H_3S—CO—CH=CH—C_6H_4—OCH_3	1652	1603	1420	1360	1238	—	860	985
VII	1-(2-Thienyl)-3-(4-methoxyphenyl)propen-3-one	C_4H_3S—CH=CH—CO—C_6H_4—OCH_3	1660	1595	1423	1374	1242	850	860	978
VIII	1-(2-Thienyl)-3-(2,4-dimethoxyphenyl)propen-1-one	C_4H_3S—CO—CH=CH—$C_6H_3(OCH_3)_2$	1640	1610	1419	1365	1245	828	860	982
IX	1-(2-Thienyl)-3-(2,4-dimethoxyphenyl)propen-3-one	C_4H_3S—CH=CH—CO—$C_6H_3(OCH_3)_2$	1650	1585	1422	1370	1240	830	870	975
X	1-(2-Thienyl)-3-(2,4,6-trimethoxyphenyl)propen-1-one	C_4H_3S—CO—CH=CH—$C_6H_2(OCH_3)_3$	1648	1575	1425	1345	1238	840	860	990
XI	1-(2-Thienyl)-3-(2,4,6-trimethoxyphenyl)propen-3-one	C_4H_3S—CH=CH—CO—$C_6H_2(OCH_3)_3$	1642	1612	1425	1360	1235	830	860	970
XII	1-(2-Thienyl)-3-(4-dimethylaminophenyl)propen-1-ohe	C_4H_3S—CO—CH=CH—C_6H_4—$N(CH_3)_2$	1635	1615	1420	1375	1235	847	860	986
XIII	1-(2-Thienyl)-3-(4-dimethylaminophenyl)propen-3-one	C_4H_3S—CH=CH—CO—C_6H_4—$N(CH_3)_2$	1640	1610	1420	1385	1242	—	860	982
XIV	1-(2-Thienyl)-3-(4-chlorophenyl)propen-1-one	C_4H_3S—CO—CH=CH—C_6H_4—Cl	1660	1608	1422	1360	1242	—	860	980
XV	1-(2-Thienyl)-3-(4-chlorophenyl)propen-3-one	C_4H_3S—CH=CH—CO—C_6H_4—Cl	1660	1597	1425	1365	1242	860	870	975
XVI	1-(2-Thienyl)-3-(4-diphenylyl)propen-1-one	C_4H_3S—CO—CH=CH—C_6H_4—C_6H_5	1650	1593	1416	1365	1237	—	860	975
XVII	1-(2-Thienyl)-3-(4-diphenylyl)propen-3-one	C_4H_3S—CH=CH—CO—C_6H_4—C_6H_5	1665	1593	1410	1370	1245	845	873	984
XVIII	1-(2-Thienyl)-3-(4-nitrophenyl)propen-1-one	C_4H_3S—CO—CH=CH—C_6H_4—NO_2	1665	1585	1430	1373	1246	832	860	980
XIX	1-(2-Thienyl)-3-(4-nitrophenyl)propen-3-one	C_4H_3S—CH=CH—CO—C_6H_4—NO_2	1653	1600	1413	—	—	843	865	987
XX	1-(5-Nitro-2-thienyl)-3-phenylpropen-1-one	O_2N—C_4H_2S—CO—CH=CH—C_6H_5	1657	1600	1455	—	1240	820	865	982
XXI	1-(5-Nitro-2-thienyl)-3-phenylpropen-3-one	O_2N—C_4H_2S—CH=CH—CO—C_6H_5	1665	1600	1432	1373	1238	822	858	987
XXII	1-(5-Nitro-2-thienyl)-3-(4-nitrophenyl)propen-1-one	O_2N—C_4H_2S—CO—CH=CH—C_6H_4—NO_2	1650	1610	1433	—	1233	822	850	988
XXIII	1-(5-Nitro-2-thienyl)-3-(4-nitrophenyl)propen-3-one	O_2N—C_4H_2S—CH=CH—CO—C_6H_4—NO_2	1669	1595	1430	1370	1243	819	845	990
XXIV	1-(5-Nitro-2-thienyl)-3-(4-methoxyphenyl)propen-1-one	O_2N—C_4H_2S—CO—CH=CH—C_6H_4—OCH_3	1650	1613	1445	—	1245	827	843	990
XXV	1-(5-Nitro-2-thienyl)-3-(4-methoxyphenyl)propen-3-one	O_2N—C_4H_2S—CH=CH—CO—C_6H_4—OCH_3	1666	1610	1432	—	1245	823	—	970

Absorption, cm^{-1}

a) C_4H_3S = 2-thienyl, C_6H_5 = phenyl.

Table 1
(cont'd)

Com-pound	Name	Formula	C=O	C=C	Ring vibrations			Out-of-plane C—H		Trans CH=CH
XXVI	1-(5-Nitro-2-thienyl)-3-(2,4-dimethoxyphenyl)propen-1-one	$O_2N—C_4H_2S—CO—CH=CH—C_6H_3(OCH_3)_2$	1650	1584	1430	—	—	820	—	990
XXVII	1-(5-Nitro-2-thienyl)-3-(2,4,6-trimethoxyphenyl)propen-3-one	$O_2N—C_4H_2S—CO—CH=CH—C_6H_2(OCH_3)_3$	1648	1570	1455	—	1243	820	848	990
XXVIII	1-(2-Thienyl)-5-phenylpentadien-1-one	$C_4H_3S—CO—(CH=CH)_2—C_6H_5$	1642	1586	1417	1370	1260	835	862	984
XXIX	1-(2-Thienyl)-5-phenylpentadien-3-one	$C_4H_3S—CH=CH—CO—CH=CH—C_6H_5$	1653	1595	1415	1365	1245	860	884	970
XXX	1-(2-Thienyl)-5-(4-methoxyphenyl)pentadien-1-one	$C_4H_3S—CO—(CH=CH)_2—C_6H_4—OCH_3$	1652	1595	1423	1364	1239	840	865	985
XXXI	1-(2-Thienyl)-5-(4-methoxyphenyl)pentadien-3-one	$C_4H_3S—CH=CH—CO—CH=CH—C_6H_4—OCH_3$	1653	1610	1430	1372	1232	840	860	985
XXXII	1-(2-Thienyl)-5-(4-methoxyphenyl)pentadien-5-one	$C_4H_3S—(CH=CH)_2—CO—C_6H_4—OCH_3$	1658	1608	1430	1370	1232	840	855	1000
XXXIII	1-(2-Thienyl)-5-(2,4-dimethoxyphenyl)pentadien-1-one	$C_4H_3S—CO—(CH=CH)_2—C_6H_3(OCH_3)_2$	1640	1612	1415	1370	1255	840	865	995
XXXIV	1-(2-Thienyl)-5-(2,4-dimethoxyphenyl)pentadienon-5-one	$C_4H_3S—(CH=CH)_2—CO—C_6H_3(OCH_3)_2$	1656	1610	1427	1370	1230	840	865	995
XXXV	1-(2-Thienyl)-5-(2,4,6-trimethoxyphenyl)pentadien-3-one	$C_4H_3S—CH=CH—CO—CH=CH—C_6H_2—(OCH_3)_3$	1664	1613	1425	1375	1240	—	880	970
XXXVI	1-(2-Thienyl)-5-(2,4,6-trimethoxyphenyl)pentadien-5-one	$C_4H_3S—(CH=CH)_2—CO—C_6H_2(OCH_3)_3$	1670	1595	1422	1370	1235	835	860	1000
XXXVII	1-(2-Thienyl)-5-(4-nitrophenyl)pentadien-1-one	$C_4H_3S—CO—(CH=CH)_2—C_6H_4—NO_2$	1643	1590	1420	—	1238	830	855	995
XXXVIII	1-(2-Thienyl)-5-(4-nitrophenyl)pentadien-3-one	$C_4H_3S—CH=CH—CO—CH=CH—C_6H_4—NO_2$	1653	1597	1418	—	1235	—	845	987
XXXIX	1-(2-Thienyl)-5-(4-nitrophenyl)pentadien-5-one	$C_4H_3S—(CH=CH)_2—CO—C_6H_4—NO_2$	1663	1590	1423	1370	1260	835	860	1015
XL	1-(5-Nitro-2-thienyl)-5-phenylpentadien-1-one	$O_2N—C_4H_2S—CO—(CH=CH)_2—C_6H_5$	1652	1583	1440	—	1248	818	—	980
XLI	1-(5-Nitro-2-thienyl)-5-phenylpentadien-3-one	$O_2N—C_4H_2S—CH=CH—CO—CH=CH—C_6H_5$	1655	1625	1433	1374	1230	823	—	980
XLII	1-(5-Nitro-2-thienyl)-5-phenylpentadien-5-one	$O_2N—C_4H_2S—(CH=CH)_2—CO—C_6H_5$	1650	1588	1426	—	1263	818	840	990
XLIII	1-(2-Thienyl)-7-phenylheptatrien-1-one	$C_4H_3S—CO—(CH=CH)_3—C_6H_5$	1640	1616	1415	1362	1240	865	890	1010
XLIV	1-(2-Thienyl)-7-phenylheptatrien-3-one	$C_4H_3S—CH=CH—CO—(CH=CH)_2—C_6H_5$	1664	1615	1425	1370	1232	840	860	1010
XLV	1-(2-Thienyl)-7-phenylheptatrien-5-one	$C_4H_3S—(CH=CH)_2—CO—CH=CH—C_6H_5$	1670	1623	1430	1370	1248	—	860	995
XLVI	(2-Thienyl)phenylketone	$C_4H_3S—CO—C_6H_5$	1633	—	1419	1360	1237	842	870	—

thiophene one results in decrease in valence vibrations frequency of the carbonyl group and of the aliphatic double bond (cf. I–III). This decrease is greater when thienyl replaces a phenyl substitutent immediately adjacent to the carbonyl group, and it is particularly great when both phenyls are replaced by thienyl.

Table 2

Carbonyl Group Characteristic Frequencies

Com-pound	$\nu_{C=O}$, cm^{-1}		
	KBr tables	CCl$_4$	CHCl$_3$
I	1652	1655	1650
II	1660	1663	1652
III	1640	1648	1640
XXVIII	1642	1645	1639

Hence in these systems the thiophene ring exhibits a greater electron-donating effect than phenyl, causing a decrease in electron density at the carbonyl oxygen, and consequent lowering of $\nu_{C=O}$; here there is greater conjugation of thienyl with the carbonyl group when they are next to one another. This latter fact is also confirmed by the spectrum of I having more intense absorption bands in the 1200–1600 cm^{-1} region than II (see Fig. 1).

Comparison of $\nu_{C=O}$ for thiophene series α, β unsaturated ketones with $\nu_{C=O}$ for analogous thiophene derivative compounds [17], shows that in the in the former case $\nu_{C=O}$ is always lower than in the latter (by 8–10 cm^{-1}). When investigating electronic absorption spectra of α, β unsaturated ketones containing furan and thiophene rings [1,18,19], it was established that λ_{max} for the long wave band differed little in the two cases, so that the effects of conjugation of these two heterocyclic rings is approximately equal. From a study of bond lengths and dipole moments of furan and thiophene, Braude and Fawcett [20] concluded that 2-furyl and 2-thienyl have approximately equal static conjugation effects. Hence it follows that the appreciably lower value of $\nu_{C=O}$ for thiophene ketones in comparison with furan ones, must not be ascribed to a difference in conjugation effects. It is best explained as being due to the negative inductive effect of furyl being greater than that of thienyl [21], so that the total electron-donating effect of thienyl exceeds that of furyl. It is of interest that, on the contrary the total dynamic electrondonating effect of 2-furyl is greater than that of 2-thienyl [21,22]. A comparatively low valence vibrations frequency is found for (2-thienyl)-phenylketone (XLVI) though it lacks a conjugated double bond, which latter usually lowers $\nu_{C=O}$ by about 40 cm^{-1} (cf. [9], p. 426). **Possibly it arises, as a result of an increase in valence angle at the carbonyl group, due to steric effects, which are known to lead to lowering of the frequency [23,24].**

In most cases introduction of electron-donating groups (CH$_3$, CH$_3$O, (CH$_3$)$_2$N) into the molecule of the thiophene analog of chalcone results in appreciable

lowering of $\nu_{C=O}$ (see IV–XIII), while on the other hand electron-accepting groups raise $\nu_{C=O}$ (see **XIV, XV, XVIII, XIX**). This change in $\nu_{C=O}$ under the action of substituents of different electronic nature tends to run parallel to the Hammett equation σ constants, as can be seen, for example, from Fig. 2, which shows how $\Delta\nu_{C=O}$ is a function of the McDaniel and Brown σ parameters [25] in the case of 1-(2-thienyl)-3-arylpropen-1-ones. The lowering of $\nu_{C=O}$ is ascribed to the accumulation of vinylene groups in the ketone molecule, i.e. the transformation from propenones to pentadienones and heptatrienones is not accompanied by removal of carbonyl groups from the heterocyclic ring, e.g. (I, XVIII, XLIII etc.).

The characteristic vibrations of the aliphatic double bond for the compounds investigated due to conjugation with the carbonyl group, are considerably lowered, in comparison with the usual ones, by the thiophene and aromatic rings, and occur in the 1575–1615 cm^{-1} region. Replacement of phenyls in the chalcone by thienyls, also gives rise to a 16–35 cm^{-1} $\nu_{C=C}$ lowering. An even greater $\nu_{C=C}$ lowering is brought about by transition from propenones to pentadienones, then further to heptatrienones, and is sometimes accompanied by band splitting. The effects of the various substituents in the aromatic ring on $\nu_{C=C}$ is less pronounced than on $\nu_{C=O}$; however here too there is a tendency to lowering of $\nu_{C=C}$ through the action of neighboring electrons, and to an increase due to electron-acceptors (cf. IV-XIX).
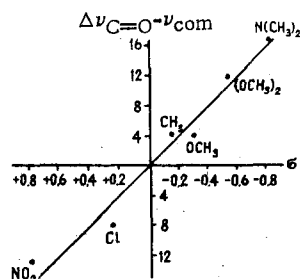

Fig. 2. $\Delta\nu_{C=O}$ as a function of the McDaniel and Brown σ parameter.

The thiophene ring shows up in the spectrograms of ring in-phase and out-of-phase vibrations in the ~1420, ~1350 and ~1240 cm^{-1} regions. The thiophene ring vibration band, given in [3], and lying at 1523 ± ± 9 cm^{-1} can in most cases be observed only in compounds where the carbonyl groups are directly joined to the heterocyclic ring; furthermore, in a number of cases it is hard to differentiate between it and the absorption due to aromatic systems. However, in the case of III, where aryl is absent, it can easily be seen at 1520 cm^{-1} (medium intensity). It should be noted that chalcone itself shows absorption at the 1340 cm^{-1} region (above medium intensity). All this goes to show that the thiophene ring vibrations in α, β unsaturated ketones can be followed most clearly by bands at 1420 and 1240 cm^{-1}.

Thienyl C—H planar deformation vibrations at 1081 ± 3 cm^{-1} [3] are clear only for those of the

present ketones where the carbonyl group is beside the heterocyclic ring. Such a band, then, as that at 1043 cm^{-1} cannot serve to identify the thiophene ring, since in the majority of cases it is very weak, and furthermore in the case of chalcone there is absorption in the 1035 cm^{-1} region.

2-Substituted thiophenes can further be identified by heterocyclic ring C—H out-of-plane deformation vibrations, lying in the 830—890 cm^{-1} region (see Table 1). There, however, it must be remembered that not far away are C—H deformation vibrations of 1,4-disubstituted benzenes at 810—832 cm^{-1} (see [16], p. 316), of 1,2,4-trisubstitued benzenes at 805—825 cm^{-1}, and of 1,2,4,6-tetrasubstituted benzenes, which here appeared at 820—830 cm^{-1}. For these reasons it was difficult to identify with our compounds, the thiophene ring "breathing" vibrations band, which according to published [3] data is at 823 ± 20 cm^{-1}. A paper [3] gives yet another band characteristic of C—H out-of-plane vibrations of 2-substituted thiophenes, at 925 ± 8 cm^{-1}. We, like Hidalgo [26], did not find this absorption. 5-Nitro-2-thienyl derivatives (XX-XXVII) have a high-intensity band at 818—830 cm^{-1}, probably owing their origin to out-of-plane cophase vibrations of two adjacent C—H bonds. Here there is also often observed a weak absorption at 880—890 cm^{-1}, which can be assigned to C—N valence vibrations (see [16], p. 432).

A nitro group at position 5 in the thiophene ring (XXI-XXVII, XL-XLII) is characterized by highly intense bands, where the frequency of the antisymmetric vibrations is appreciably lowered, and lies in the 1492—1510 cm^{-1} region. This is most probably bound up with the electron-donating nature of the thiophene ring, it being known that conjugation with donor groups decreases the corresponding frequency (see [9], p. 426). In this connection it is characteristic that the nitro group frequency decreases, when the carbonyl group moves away from the thiophene ring. With compounds XVIII, XIX, XXXVII-XXXIX, where the nitro group is in the benzene ring, ν_{as} is in the usual region at 1515—1533 cm^{-1}. With regard to the nitro group symmetric vibrations band, it is usually combined with the absorption due to thiophene ring vibrations (1354 ± 7 cm^{-1} [3]) and the absorption of the chalcone at 1340 cm^{-1}, and is distinguished by an altogether high integral intensity in the 1328—1360 cm^{-1} region.

Many of the compounds investigated contained a methoxyl group joined to an aromatic ring (VI-X, XXIV-XXVII, XXX-XXXVI). Its characteristic vibration frequencies are, agreeing with the results of other authors [27] in the 1260—1330 cm^{-1} region; methyl-oxygen valence vibrations lie at 1020—1040 cm^{-1}. The latter band can be identified quite well, and with accumulation of methoxyl groups in the molecule, its frequency is appreciably raised. Thus when one methoxyl group is present it is at 1020—1028 cm^{-1} (VI-VII), in the case of 1,3-dimethoxy derivatives at 1027—1033 cm^{-1} (VIII, IX), and with 1,3,5-trimethoxy groups, at 1030—1040 cm^{-1} (X, XI). It should be noted that with (2-thienyl)phenylketone band intensities,

as compared with the chalcone anaologs and the vinylogs, are distinctly lower, and that the number of bands is less (see Fig. 1).

EXPERIMENTAL

IR spectra were determined, using an IKS-14 instrument, in CCl$_4$ and CHCl$_3$, and with a UR-10 instrument tabletted with KBr (1.5 mg compound per 100 mg KBr), over the region 1800—650 cm^{-1}.

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