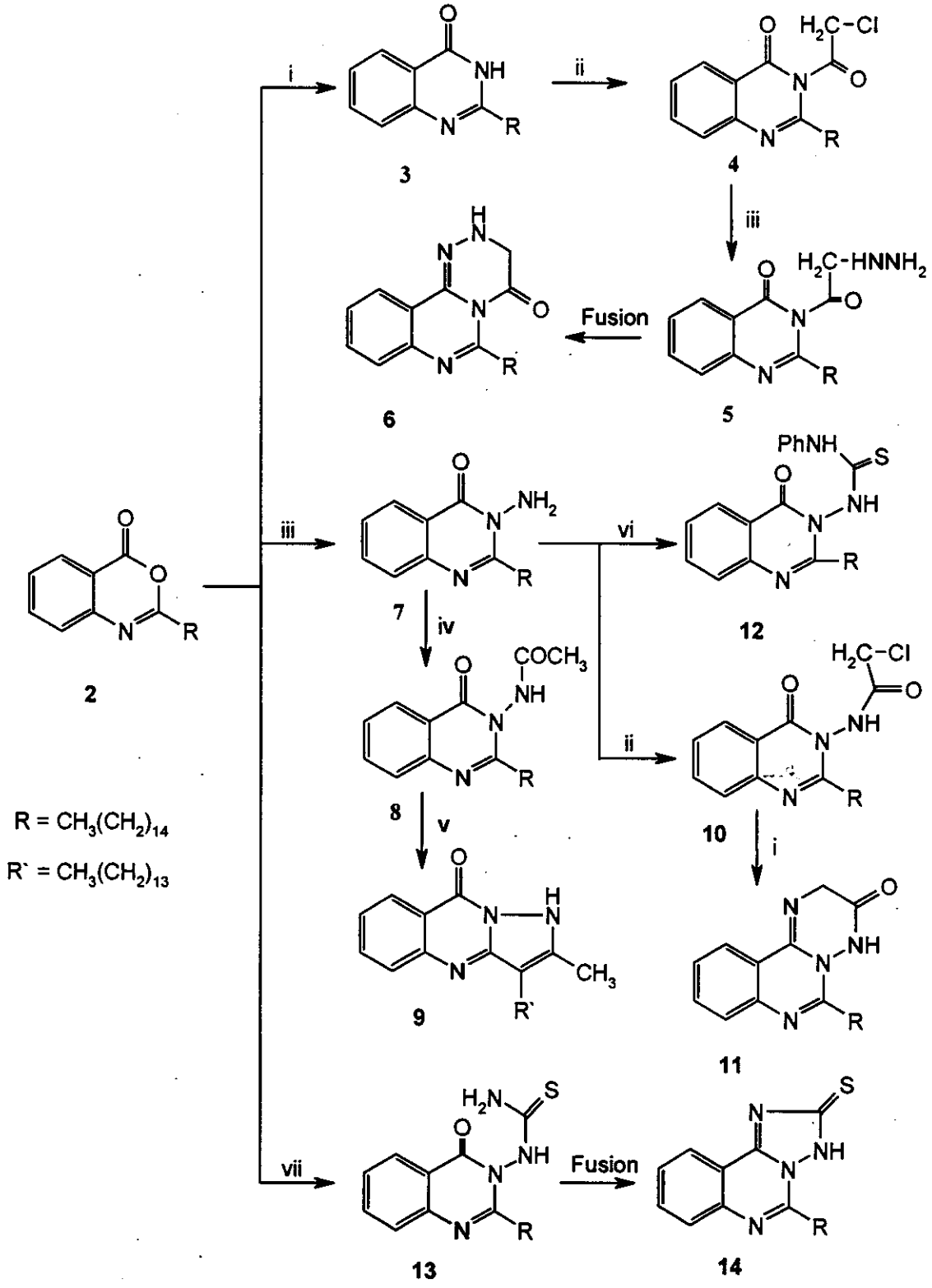
PART (1)

Synthesis of some new heterocyclic compounds from palmitic acid

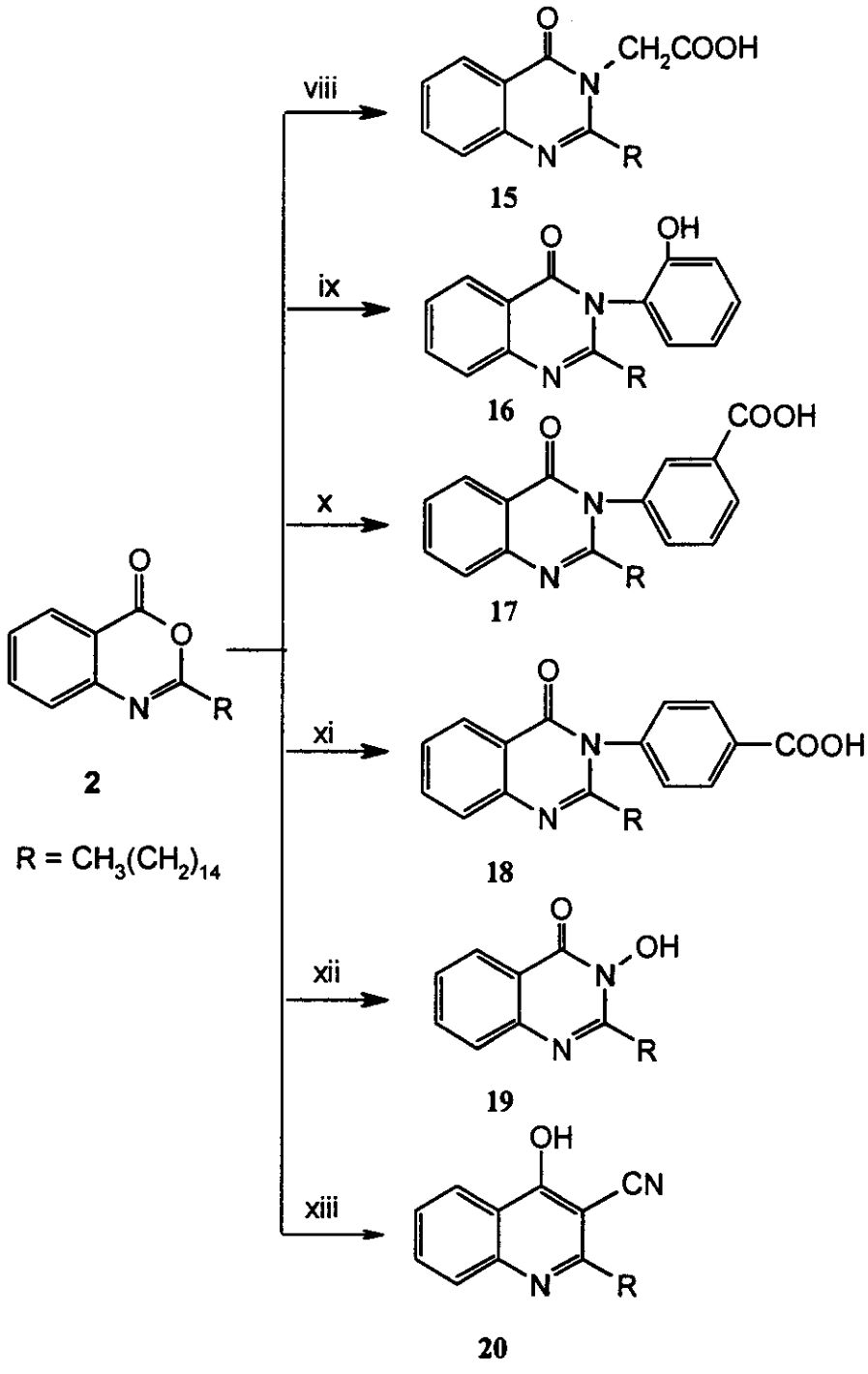
Varied biological activities have been attributed to 2-substituted-3,1-benzoxazin-4-ones [182] as well as the corresponding quinazoline derivatives were found to posses antipyretic [183], antiinflammatory [184], antimitotic, anticancer activity [185] and also have a good storage stability in detergents [186].This leds us to synthesis a new series of 4*H*-3,1-benzoxazin-4-one and corresponding quinazolinone derivatives **(2-20)** of pharmaceutical and industrial applications as the scheme **1,2**.

This also encouraged us to the synthesis a novel group of nonionic surface active agents containing this nucleus from long chain fatty acid (palmitic acid). Some of the synthesized compounds which have active hydrogen atom were subjected to react with propylene oxide by different moles (n = 5, 10 and 15) to produce a novel group of nonionic compounds **(21a-c-38a-c)** having a double function as antimicrobial and surface active agents which can be serve in the manufacture of drugs, cosmetics, pesticides or can be use as antibacterial and/or antifungal agents. The antimicrobial activity, the physical properties as surface and interfacial tension, cloud point, foaming height, wetting time and emulsification power were determined. The biodegradability was also screened.



Scheme 1

Reagents: i) CH_3COONH_4; ii) $ClCOCH_2Cl$ iii) NH_2NH_2; iv) CH_3COCl; v) CH_3CH_2ONa; vi) PhNCS; vii) $H_2NCSNHNH_2$.



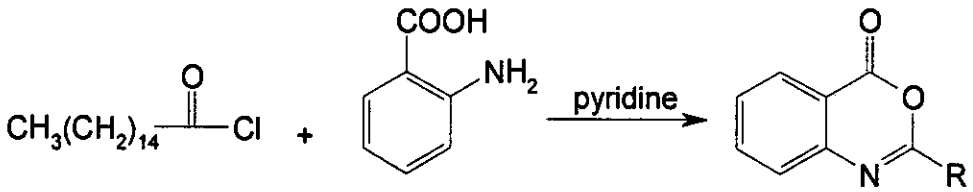
Scheme 2

Reagents: viii) H_2NCH_2COOH; ix) $H_2NC_6H_4OH$ (*o*); x) $H_2NC_6H_4COOH$ (*m*); xi) $H_2NC_6H_4COOH$ (*p*); xii) $H_2NOH.HCl$; xiii) $CH_2(CN)_2$

Synthesis of some new heterocyclic compounds from palmitic acid

1. Synthesis of 3,1-benzoxazinone derivative (2) by the reaction of palmitoyl chloride (1) with anthranilic acid

The starting compound 2-pentadecyl-*4H*-3,1-benzoxazin-4-one **2** was prepared from the reaction of palmitoyl chloride **1** with anthranilic acid in boiling pyridine.



1 **2**

R = $CH_3(CH_2)_{14}$

IR spectrum of (**2**) shows the following bands in cm^{-1}, band's at 1644 and 1763 for νC≡N, νC=O and 3005, 2920, 2850 for νCH of aromatic and aliphatic of alkyl chain beside the characteristic band of the linear structure of the compound at 2342, 2359 and characteristic band for aromatic νC=C at 1606 and 1580 cm^{-1} (*cf.* fig. 1).

^{1}HNMR spectrum of (**2**) shows signals at δ 0.9 (t, 3H, terminal CH_3), δ 1.2-1.6 (m, 28H, CH_2 of alkyl chain), δ 6.7-8.3 (m, 4H, ArH). (*cf.* fig. 23).

Mass spectrum of (**2**) shows molecular ion peak at $M^{\cdot+}$ = 257, 24.99 %, base peak at m/z =161, 100 % and the benzoxazone moiety at m/z = 146, 21.99 % its also shows that the sum of the three main fragments equal to the molecular ion peak (*cf.* fig. 31, chart 1).

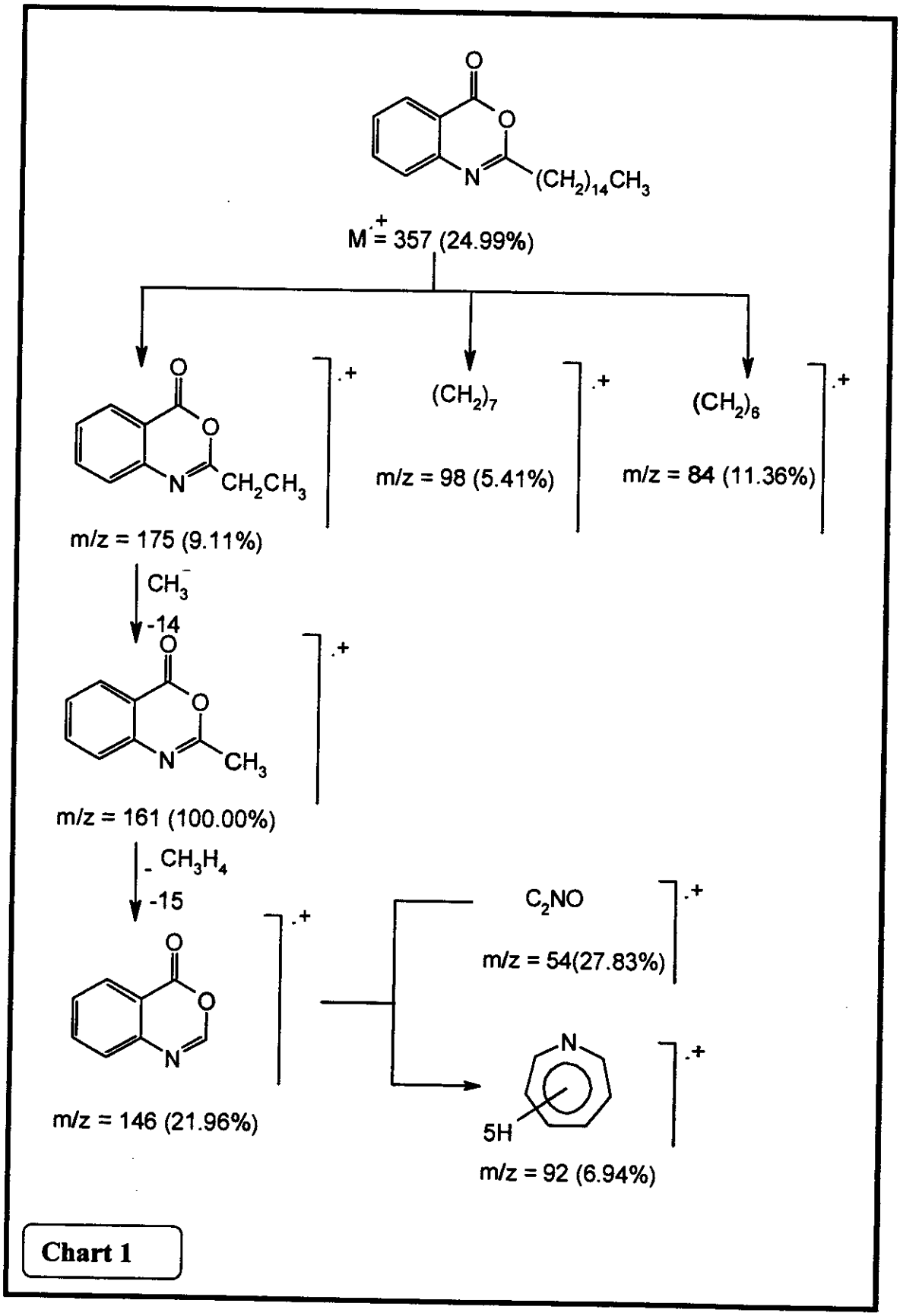
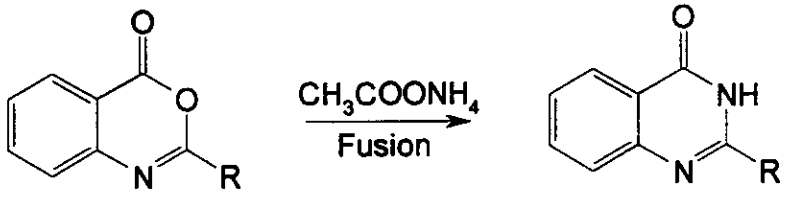

M.+ = 357 (24.99%)
(CH2)14CH3
CH2CH3
m/z = 175 (9.11%)
(CH2)7
m/z = 98 (5.41%)
(CH2)6
m/z = 84 (11.36%)
CH3
-14
m/z = 161 (100.00%)
- CH3H4
-15
m/z = 146 (21.96%)
C2NO
m/z = 54(27.83%)
5H
m/z = 92 (6.94%)

Chart 1

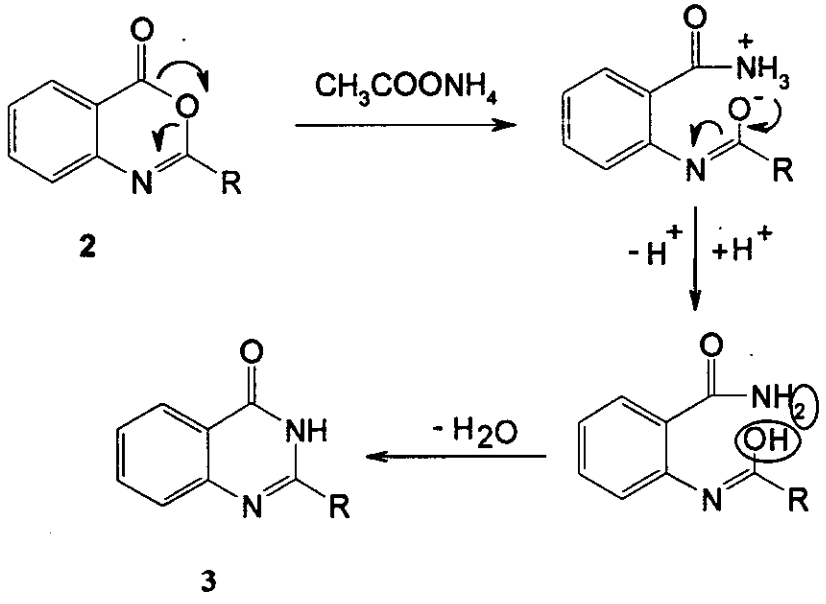
2. Synthesis of quinazolinone (3) by the reaction of benzoxazone (2) with ammonium acetate

Treatment of benzoxazin-4-one 2 with ammonium acetate by fusion produced 2-pentadecylquinazolin-4(*3H*)-one (**3**).



2 R = $CH_3(CH_2)_{14}$ **11**

The reaction may takes place as the following mechanism:



3

IR spectrum of (**3**) shows bands at 3341 for ν(NH,OH), 1676 for νC=O and 2920, 2850 for νCH aliphatic of alkyl chain, beside the characteristic bands of quinazoline moiety [189] at 1585, 1530 and 1470 cm^{-1}.(*cf.* fig.2).

Mass spectrum of (**3**) shows a molecular ion peak at M^++1= 357, **8.73** % and the base peak at m/z = 161, 100 % (*cf.* fig.32 , chart 2).

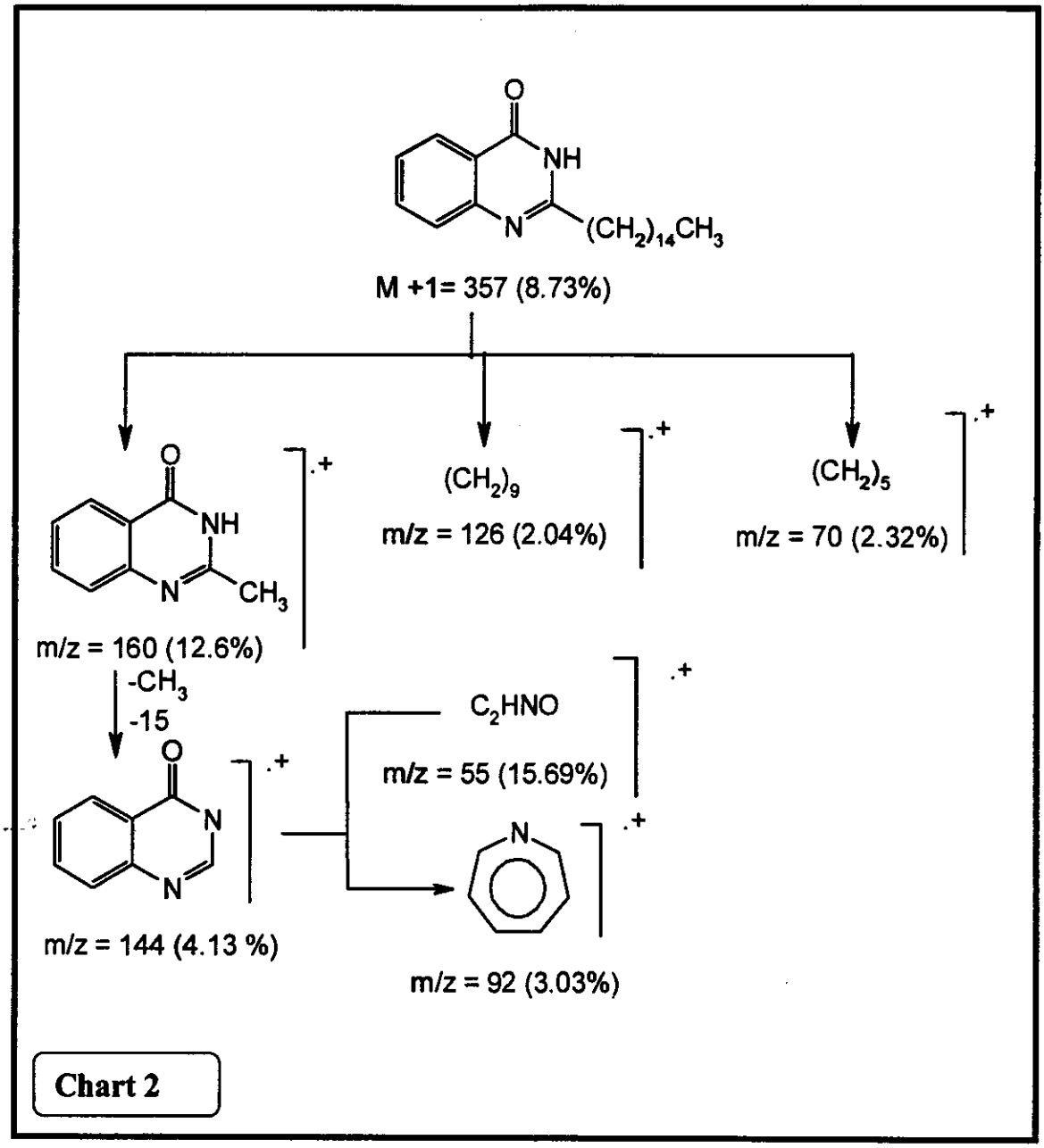
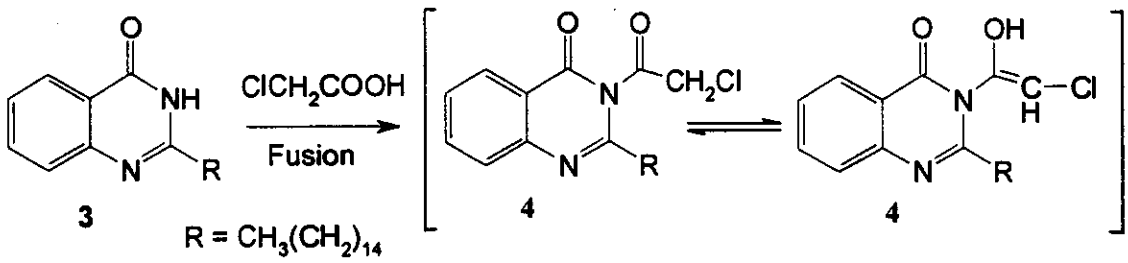


Chart 2

The structure of quinazolinone **(3)** chemically was confirmed from the reaction of quinazolinone **(3)** with chloroacetic acid

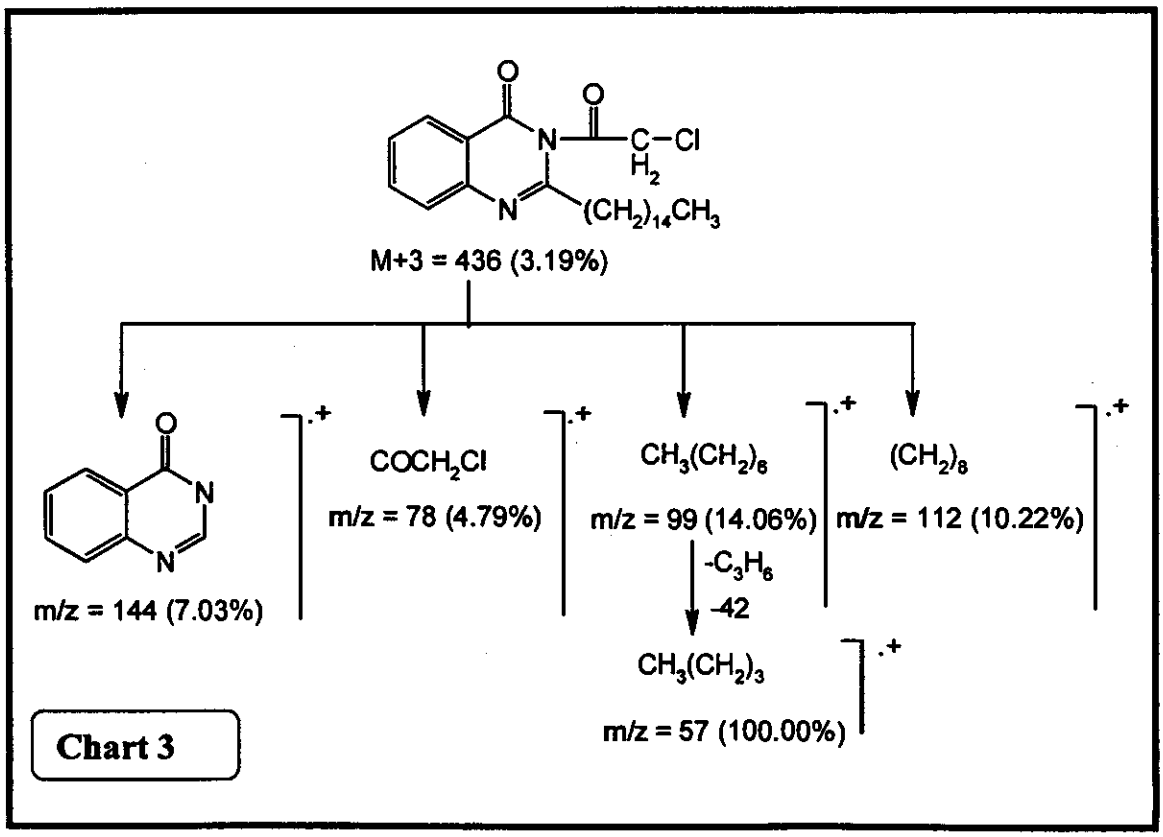
Addition of chloroacetic acid to quinazolinone **(3)** by fusion gives 3-(2-chloroacetyl)-2-pentadecylquinazolin-4(*3H*)-one **(4)**.



IR spectrum of **(4)** shows band at 3342 for ket-enol form of νOH, 1688 and 1678 for νC=O and 2921, 2851 for νCH aliphatic of alkyl chain, beside the characteristic bands of quinazoline moiety at 1586, 1530 and 1468 cm^{-1} (*cf.* fig.3).

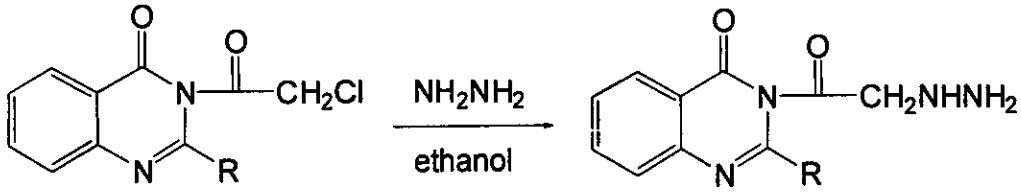
^{1}HNMR spectrum of **(4)** shows signals at δ 0.95 (t, 3H, terminal CH_3), δ 1.2-1.4 (m, 28H, CH_2 of alkyl chain), δ 7.2-8.4 (m, 4H, ArH), δ 11.1 (s, 1H, OH) which appear as keto-enol form (*cf.* fig. 24).

Mass spectrum of **(4)** shows a molecular ion peak at M^++3= 436, 3.19 % and the base peak at m/z = 57, 100 % (*cf.* fig.33 , chart 3).



Also, compound (**4**) was confirmed by formation of hydrazide compound (**5**) by its reaction with hydrazine hydrate.

Quinazolinone (**4**) was treated by hydrazine hydrate in ethanol lead to formation of 3-(2-hydrazino-acetyl)-2-pentadecyl-*3H*-quinazolin-4-one (**5**)

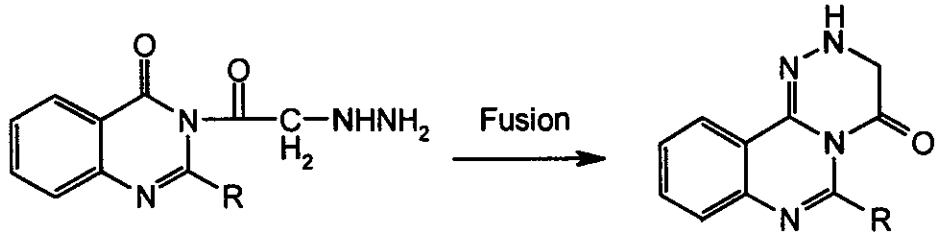


4 R = $CH_3(CH_2)_{14}$ **5**

IR spectrum of (**5**) shows band for 3342 and 3232 for νNH and NH_2, 2919, 2850 cm^{-1} for νCH aliphatic of alkyl chain, 1701 and 1679 for νC=O and 1530, 1469, 1414 cm^{-1} for the characteristic band of quinazoline moiety (*cf.* fig.4).

^{1}HNMR spectrum of (**5**) shows signals at δ 0.95 (t, 3H, terminal CH_3), δ 1.2-1.5 (m, 28H, CH_2 of alkyl chain), δ 3.54 (s, 2H, $\underline{CH_2}NHNH_2$), δ 7.2-7.9 (m, 4H, ArH) and δ 8.4 (s, 3H, NH and NH_2) which disappeared by addition D_2O (*cf.* fig. 25).

The structure of compound (**5**) chemically was confirmed by its cyclyzation by fusion to yield 6-pentadecyl-2,3-dihydro-[1,2,4]triazino[4,3-c]quinazolin-4-one (**6**)

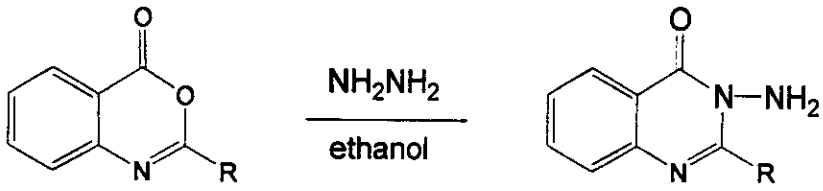


5 R = $CH_3(CH_2)_{14}$ **6**

IR spectrum of (**6**) shows band at 3233 for νNH, 2918, 2849 for νCH aliphatic of alkyl chain, 1677 for νC=O and 1507, 1594 $cm^{-1}$for νC=N (*cf.* fig.5).

3. Synthesis of 3-aminoquinazolinone (7) by the reaction of benzoxazone (2) with hydrazine hydrate

Treatment of benzoxazinone **2** with hydrazine hydrate in boiling ethanol produced 3-amino-2-pentadecylquinazolin-4(*3H*)-one (**7**)



2 R = $CH_3(CH_2)_{14}$ **7**

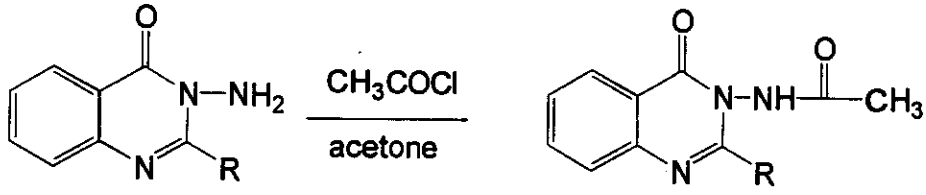
IR spectrum of (**7**) exhibits, νNH's centered at 3177 and bands at 2918, 2849 for νCH aliphatic of alkyl chain, 1688 for νC=O, beside the characteristic bands of quinazoline moiety at 1585, 1529 and 1470 cm^{-1} (*cf.* fig.6).

Mass spectrum of (**7**) shows a molecular ion peak at $M^+ + 4 = 375$, 4.12 %. The molecular ion peak of quinazoline moiety [187] at m/z = 146, 9.88 % and the base peak at m/z = 137, 100 % (*cf.* fig. 34).

The structure of quinazolinone (**7**) was confirmed chemically from the following reaction:

a- Acylation followed by cyclyzation

Addition of acetyl chloride to quinazoline (**7**) in dry acetone leads to formation of *N*-(4-oxo-2-pentadecylquinazolin-3(*4H*)-yl)acetamide (**8**).



7 R = $CH_3(CH_2)_{14}$ **8**

IR spectrum of **(8)** exhibits bands at 3448, 3226 for ν(OH and NH toutomerism), 2920, 2850 for νCH aliphatic of alkyl chain, 1698, 1651 for νC=O and 1598, 1510, 1449 cm^{-1} for the characteristic band of quinazoline moiety (*cf.* fig.7).

Mass spectrum of **(8)** shows a molecular ion peak at M^{+}-1= 412, 4.12 %. The ion peak of quinazoline at m/z = 144, 5.19 % and the base peak at m/z = 55, 100 % (*cf.* fig. 35, chart 4).

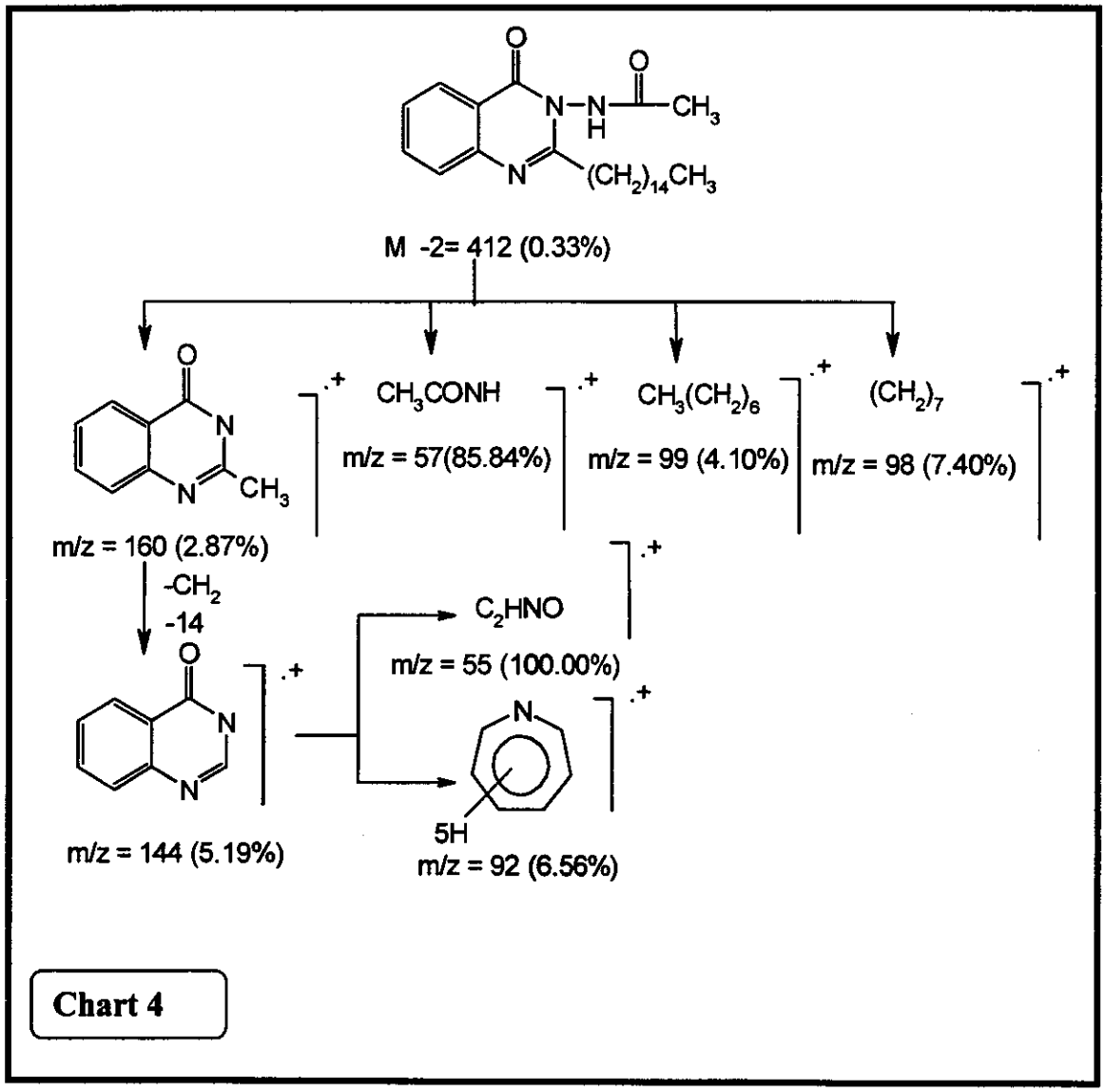
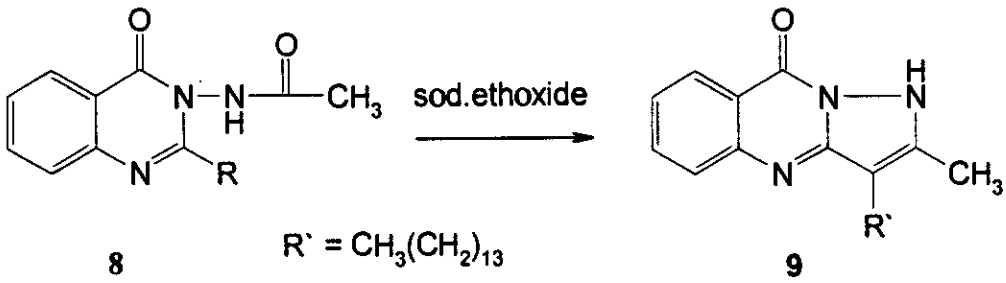


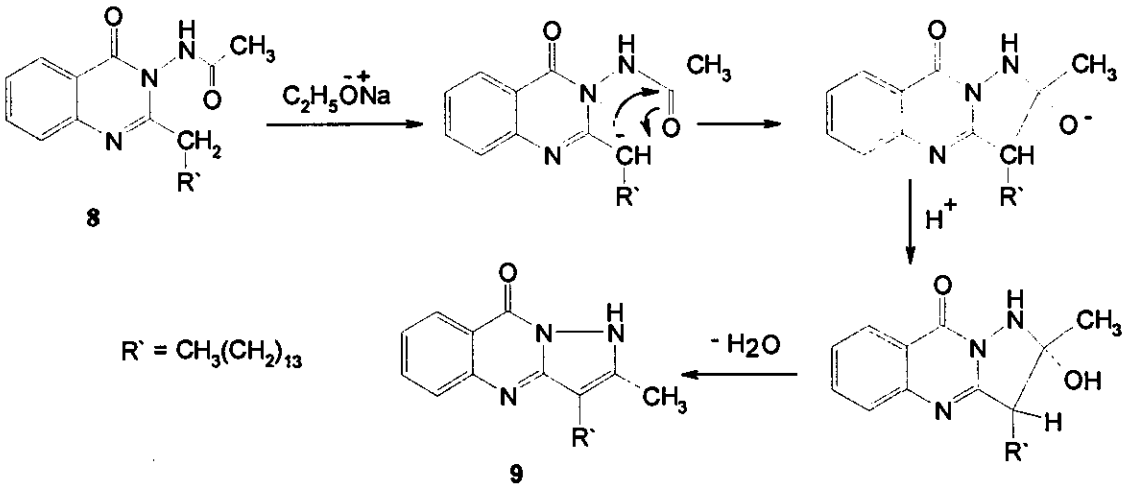
Chart 4

Quinazolinone (**8**) was cyclized by sod.ethoxide [188] to yield 2-methyl-3-tetradecylpyrazolo[5,1-b]quinazolin-9(*1H*)-one (**9**)



8 **9**

The reaction may takes place as the following mechanism:



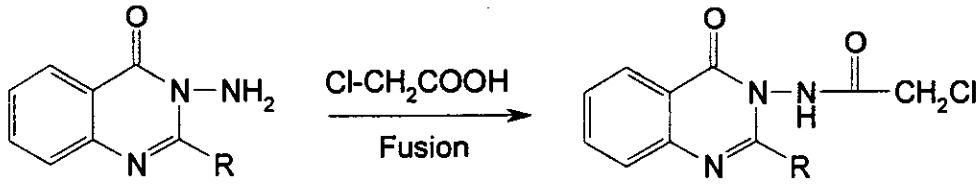
8 **9**

IR spectrum of (**9**) exhibits bands at 3417 for νNH, 2918, 2849 for νCH aliphatic of alkyl chain, 1653 for νC=O and 1559, 1469, 1439 cm^{-1} for the characteristic band of quinazoline moiety (*cf.* fig.8).

Mass spectrum of (**9**) shows a molecular ion peak at M^+-2= 397, 63.64 % and the base peak at m/z = 80, 100 % (*cf.* fig.36).

b- Reaction with chloroacetic acid followed by cyclyzation

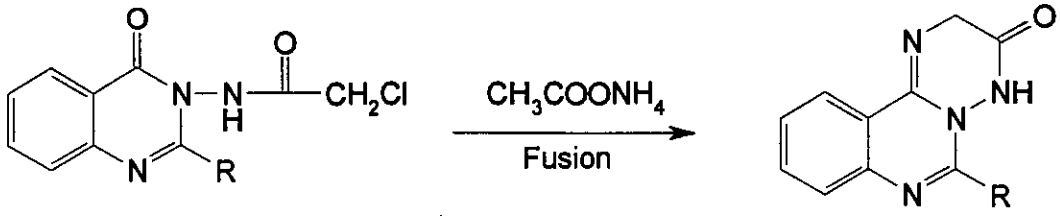
Treatment of quinazolinone (**7**) with chloroacetic acid by fusion gave 2-chloro-*N*-(4-oxo-2-pentadecylquinazolin-3(*4H*)-yl)-acetamide (**10**)



7 10

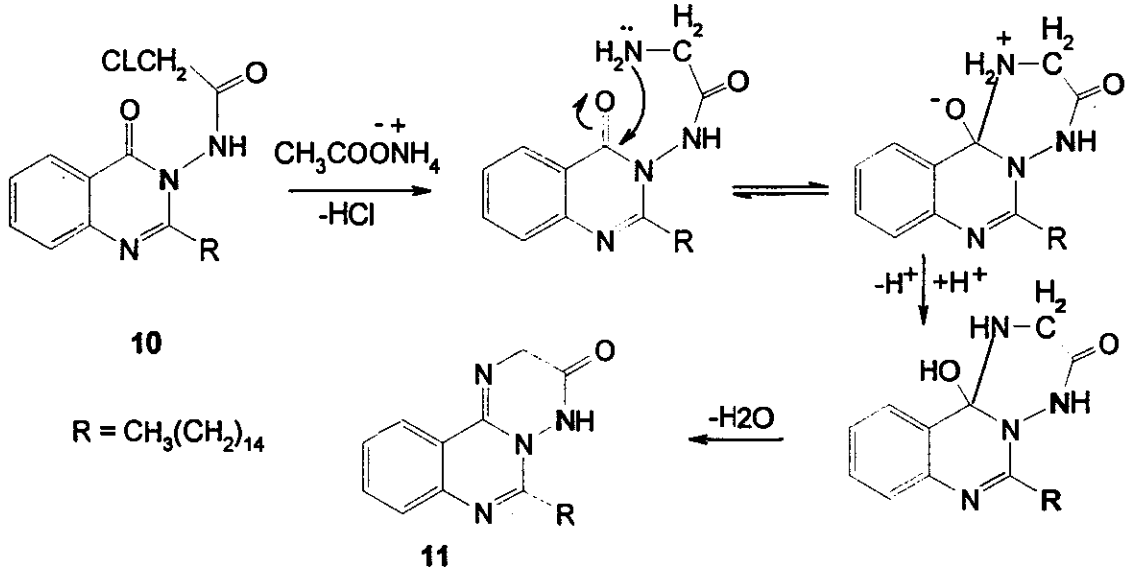
IR spectrum of **(10)** shows band at 3336 for νNH, 2918, 2849 for νCH aliphatic of alkyl chain, 1697 for νC=O and 1675 and 1546, 1498, 1436 cm^{-1} for the characteristic band of quinazoline moiety (*cf.* fig.9).

Quinazolinone **(10)** was cyclized by fusion with ammonium acetate to yield 6-pentadecyl-*2H*-[1,2,4]triazino[2,3-c]quinazolin-3(*4H*)-one **(11)**



10 11

The reaction may takes place as the following mechanism:



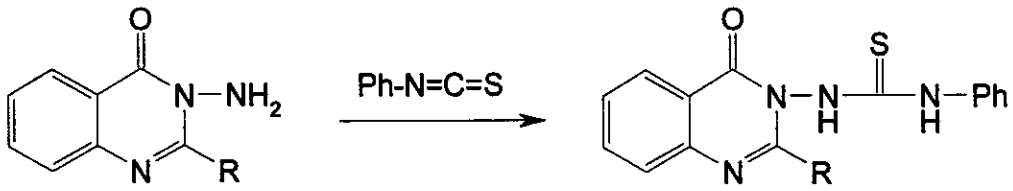
10 11

IR spectrum of **(11)** shows band at 3343 and 3422 for ν (OH and NH tuotumerism), 2923, 2852 for νCH aliphatic of alkyl chain, 1587, 1528 for νC=N and 1679 cm^{-1} for νC=O (*cf.* fig.10).

^{1}HNMR spectrum of **(11)** shows signals at δ 0.85 (t, 3H, terminal CH_3), δ 1.2-1.6 (m, 28H, CH_2 of alkyl chain), 4.4 (s, 2H, CH_2 of triazino ring), δ 8.0 (s, 1H, NH), δ 7.3-7.6 (m, 4H, ArH).

c-Reaction of quinazolinone (7) with phenyl isothiocyanate

Aminoquinazolinone **(7)** was attacked by phenyl isothiocyanate in benzene to yield 1-(4-oxo-2-pentadecylquinazolin-3(*4H*)-yl)-3-phenylthiourea **(12)**



7 R = $CH_3(CH_2)_{14}$ **12**

IR spectrum of **(12)** exhibits bands at 3275 and 3197 for νNH's, 2918, 2849 for νCH aliphatic of alkyl chain, 1681 for νC=O, 1583, 1498, 1436 for the characteristic band of quinazoline moiety and 1350 cm^{-1} for νC=S (*cf.* fig.11).

4- Synthesis of quinazolinyl thiourea (13) by the reaction of benzoxazone (2) with thiosemicarbazide

The reaction of benzoxazone **(2)** with thiosemicarbazide in boiling pyridine afforded (2-pentadecyl-4-oxo-*4H*-quinazolin-3-yl)thiourea **(13)**.

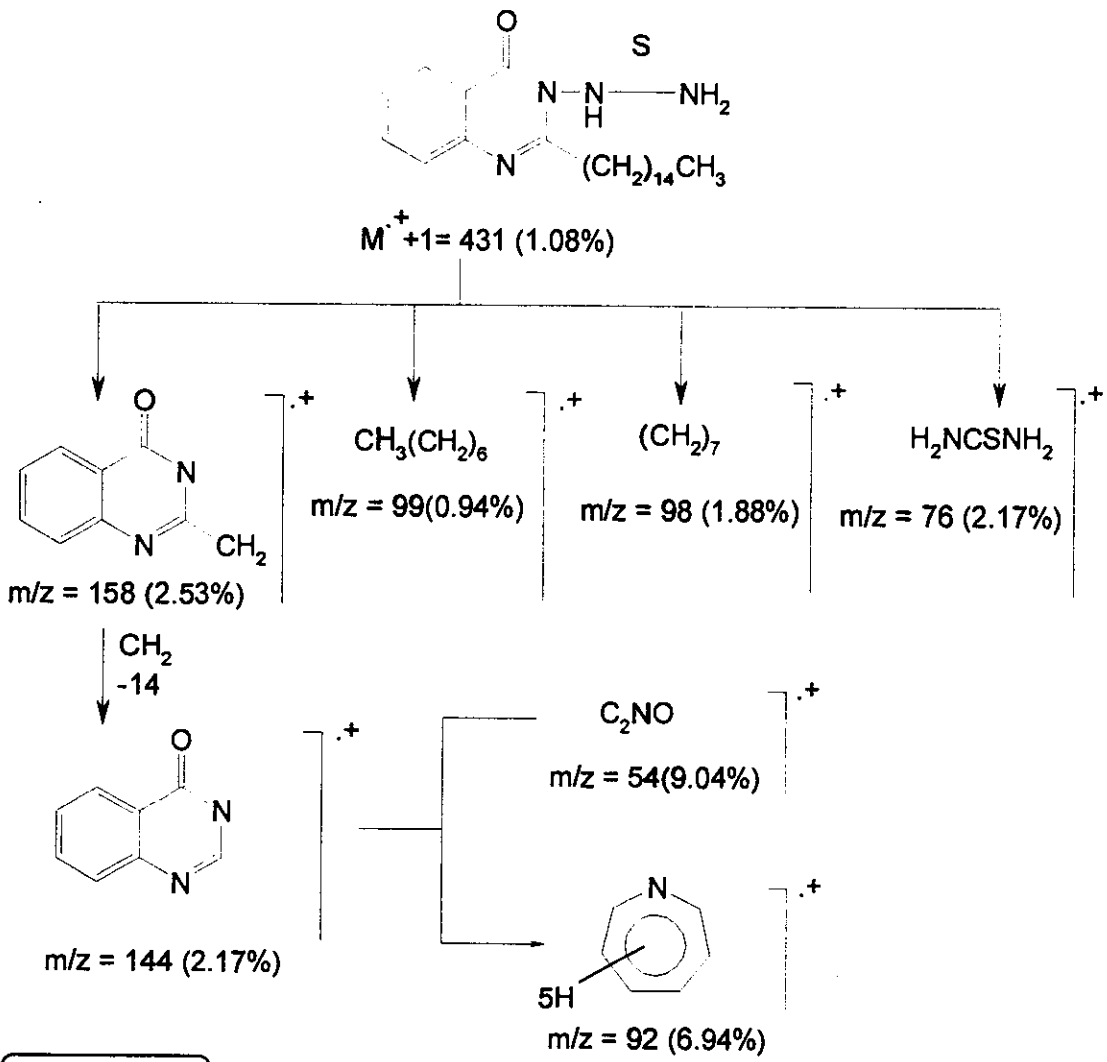
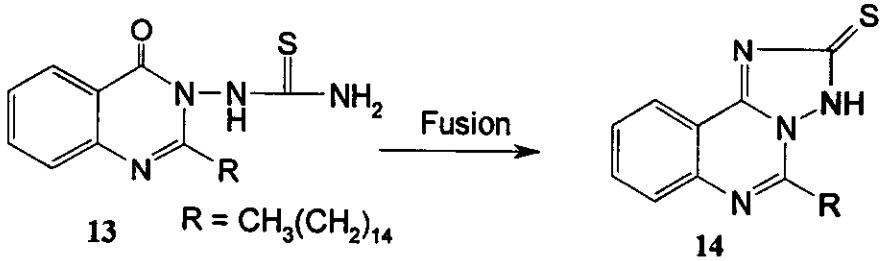
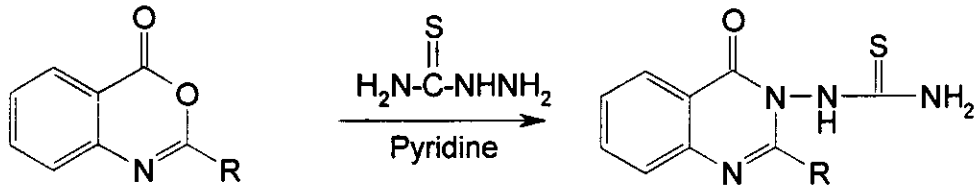


Chart 5

The structure of quinazolinyl thiourea **(13)** was confirmed chemically from its cylcyzation by fusion above its melting point and yielded 5-pentadecyl-[1,2,4]triazolo[1,5-c]quinazoline-2(*3H*)-thione **(14)**



13 R = $CH_3(CH_2)_{14}$ **14**



2 R = $CH_3(CH_2)_{14}$ **13**

IR spectrum of **(13)** exhibits, the presence of (NH and OH) toutomerism thus there are two bands centered at 3425 and 3250, νCH's of aromatic and aliphatic chain at 3050, 2918, 2849, νC=O at 1655, beside νC=N and νC=C of aromatic at 1590, 1557, the characteristic band of quinazoline moiety at 1525, 1467, 1421 and νC=S at 1367 cm^{-1} (*cf.* fig.12).

^{1}HNMR spectrum of **(13)** shows signals at δ 0.9 (t, 3H, terminal CH_3), δ 1.2-1.6 (m, 28H, CH_2 of alkyl chain), δ 6.7-8.3 (m, 4H, ArH), δ 10.3, 11.1 and 12.0 (s, 3H, exchangeable for 3NH's). (*cf.* fig. 26).

Mass spectrum of **(13)** shows molecular ion peak at M^++1 = 431,1.08 %, the base peak at m/z =137, 100 % and the other corresponding to quinazolinone nucleus at m/z = 144, 2.17 % (*cf.* fig.37, Chart 5).

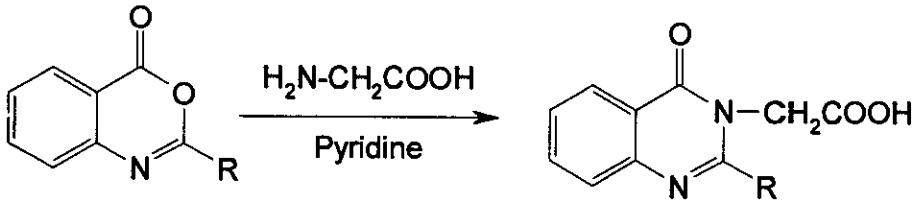
IR spectrum of **(14)** shows νNH at 3240, νCH of alkyl chain 2919, 2850 and characteristic band for triazol ring skeletal vibration [189] in a range (1515-1480) and νC=S at 1438, beside the characteristic bands of quinazoline nucleus (1630-1620), (1580-1570) cm^{-1}. (*cf.* fig. 13).

^{1}HNMR spectrum of **(14)** shows signals at δ 0.85 (t, 3H, terminal CH_3), δ 1.2-1.5 (m, 28H, CH_2 of alkyl chain), δ 6.7-8.4 (m, 4H, ArH), δ 11.0 (s, 1H, exchangeable NH). (*cf.* fig. 27).

5- Reaction with aliphatic and aromatic amino acids.

a-Synthesis of quinazolinyl acetic acid (15) by the reaction of benzoxazone (2) with glycine

Glycine reacts with benzoxazin-4-one **(2)** in the presence of pyridine as a base to produce 2-(4-oxo-2-pentadecylquinazolin-3(*4H*)-yl) acetic acid **(15)**.



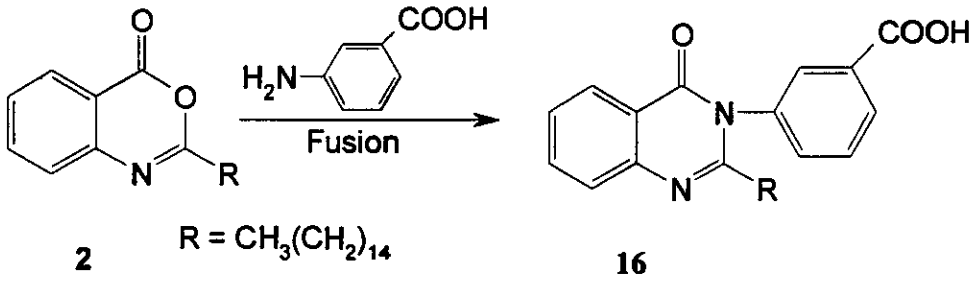
2 R = $CH_3(CH_2)_{14}$ **15**

IR spectrum of **(15)** shows band at 3341 for νOH , 2919, 2850 for νCH aliphatic of alkyl chain, 1700, 1678 for νC=O and the characteristic band of quinazoline moiety at 1585, 1469, 1413 cm^{-1} (*cf.* fig.14).

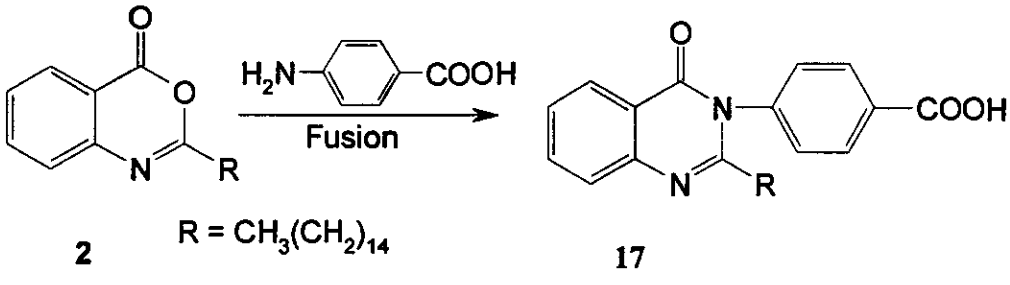
b- Synthesis of quinazolinone (16 and 17) by the reaction of benzoxazone (2) with m-aminobenzoic acid and/or p-aminobenzoic acid

Addition of *m*-aminobenzoic acid and/or p-aminobenzoic acid to benzoxazone **(2)** by fusion gives 3-(4-oxo-2-pentadecylquinazolin-3(*4H*)-yl)-

benzoic acid (**16**) and/or 4-(4-oxo-2-pentadecylquinazolin-3(*4H*)-yl)benzoic acid (**17**).



2 R = $CH_3(CH_2)_{14}$ **16**

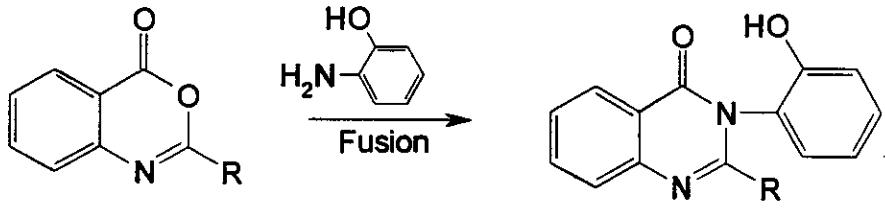


2 R = $CH_3(CH_2)_{14}$ **17**

IR spectrum of (**16**) shows band at 3341 for νOH, 2920, 2850 for νCH aliphatic of alkyl chain, 1585 for νC=N, 1678 and 1699 cm^{-1} for νC=O (*cf.* fig.15).

6- Synthesis of quinazolinone (18) by the reaction of benzoxazone (2) with o-aminophenol

Benzoxazone (**2**) was attacked by the oxygen-atom of *o*-aminophenol to form 3-(2-hydroxyphenyl)-2-pentadecylquinazolin-4(*3H*)-one (**18**).

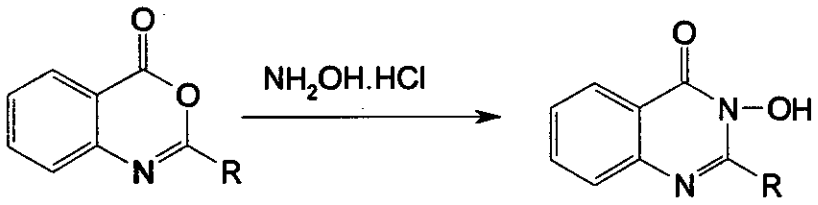


2 R = $CH_3(CH_2)_{14}$ **18**

IR spectrum of **(18)** shows band at 3342 for νOH, 2921, 2851 for νCH aliphatic of alkyl chain, 1530 for νC=N and 1678 cm^{-1} for νC=O (*cf.* fig.16).

7. Synthesis of hydroxyl quinazolinone derivative (19) via the reaction of benzoxazone (2) with Hydroxylamine hydrochloride

Hydroxylamine hydrochloride reacts with benzoxazin-4-one **(2)** in presence of pyridine to produce 3-hydroxy-2-pentadecylquinazolin-4(*3H*)-one **(19)**

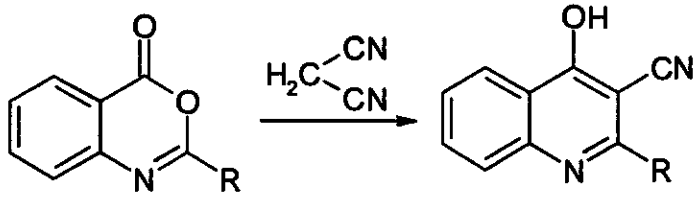


2 R = $CH_3(CH_2)_{14}$ **19**

IR spectrum of **(19)** shows band at 3422 for νOH, 2919, 2850 for νCH aliphatic of alkyl chain, 1600 for νC=N and 1647 cm^{-1} for νC=O (*cf.* fig.17).

8- Synthesis of quinoline derivative (20) via the reaction of benzoxazone (2) with malononitrile

Addition of malononitrile to benzoxazone **(2)** in ethanol [190] gave 4-hydroxy-2-pentadecylquinoline-3-carbonitrile **(20)**.



2 R = $CH_3(CH_2)_{14}$ **20**

IR spectrum of **(20)** shows band at 3342 for νOH, 2920, 2851 for νCH aliphatic of alkyl chain, 1678 for νC=O and 1533 cm^{-1} for νC=N.

Mass spectrum of **(20)** shows a molecular ion peak at M^{+}= 380, 3.19 % and the base peak at m/z = 196, 100 % (*cf.* fig. 36, chart 6).

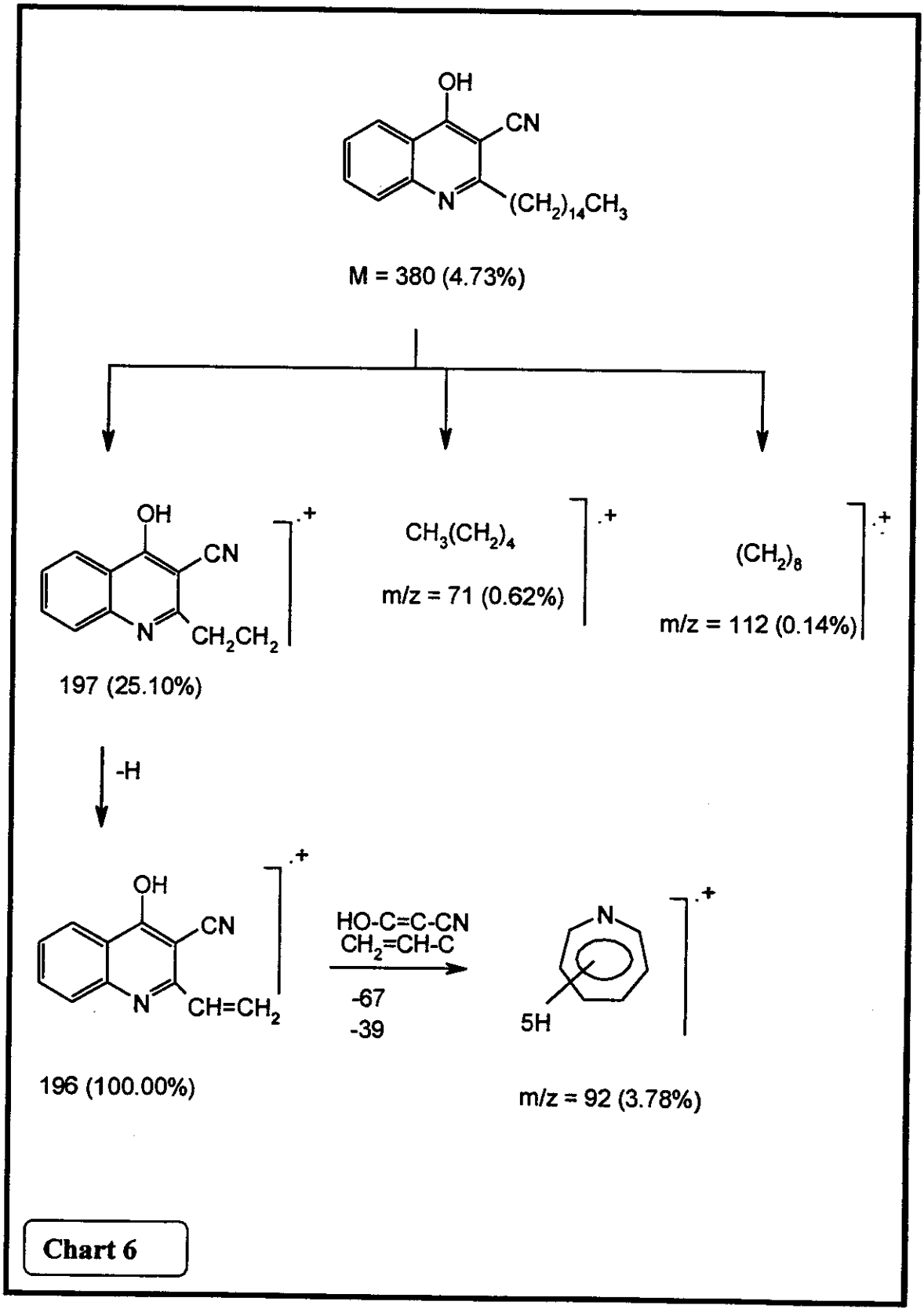

OH
CN
N
$(CH_2)_{14}CH_3$
M = 380 (4.73%)
OH
CN
N
CH_2CH_2
.+
197 (25.10%)
$CH_3(CH_2)_4$
.+
m/z = 71 (0.62%)
$(CH_2)_8$
.+
m/z = 112 (0.14%)
-H
OH
CN
N
$CH=CH_2$
.+
196 (100.00%)
HO-C=C-CN
CH_2=CH-C
-67
-39
N
5H
.+
m/z = 92 (3.78%)

Chart 6

Table (1): physical properties of prepared compounds

Compd	M.F.	M. wt	m. p °C	Yield % Solvent	Color	Analysis data calc./Found %			
						C	*H*	*N*	*S*
2	$C_{23}H_{35}NO_2$	357.27	46.	62 Benz	Yellow	77.27 77.35	9.87 9.35	3.92 3.87	
3	$C_{23}H_{36}N_2O$	356.54	80	65 EtOH	Brown	77.48 77.45	10.18 10.41	7.86 7.52	
4	$C_{25}H_{37}ClN_2O_2$	433.03	135	63 EtOH	Brown	69.34 69.41	8.61 8.83	6.47 6.91	
5	$C_{25}H_{40}N_4O_2$	428.61	124	65 Xyl	Brown	70.06 70.45	9.41 9.41	13.07 13.52	
6	$C_{25}H_{38}N_4O$	410.6	99	68 Tol	Yellow	73.13 73.41	9.33 9.59	13.65 13.82	
7	$C_{23}H_{37}N_3O$	371.56	98	55 EtOH	Pale Yellow	74.35 74.06	10.04 10.55	11.31 11.35	
8	$C_{25}H_{40}N_3O_2$	414.62	124	58 Benz	Pale Yellow	72.42 72.67	9.72 9.83	10.13 10.82	
9	$C_{25}H_{37}N_3O$	395.58	100	52 But	Pale Yellow	75.91 75.41	9.43 9.22	10.62 10.25	
10	$C_{25}H_{38}ClN_3O_2$	448.04	130	65 EtOH	Yellow	67.02 67.30	8.55 8.62	9.38 9.03	
11	$C_{25}H_{38}N_4O$	410.6	102	68 EtOH	Yellow	73.13 73.81	9.33 9.29	13.65 13.22	
12	$C_{30}H_{42}N_4OS$	506.75	84.	52 Xyl	Pale Yellow	71.10 71.41	8.35 8.22	11.06 11.25	6.63 6.87
13	$C_{24}H_{38}N_4OS$	430.66	108	65 EtOH	Yellow	66.94 66.56	8.89 9.82	13.01 13.04	7.45 7.22
14	$C_{24}H_{36}N_4S$	412.36	68	68 Xyl	Yellow	69.86 69.66	8.79 8.51	13.58 13.37	7.77 7.37
15	$C_{25}H_{39}N_2O_3$	415.60	70	75 Xyl	Red brown	72.25 72.43	9.46 9.88	6.74 6.73	
16	$C_{29}H_{40}N_2O_2$	448.64	151	73 EtOH	Red brown	77.64 77.20	8.99 8.65	6.24 6.08	
17	$C_{30}H_{40}N_2O_3$	476.66	68	70 EtOH	Red brown	75.59 75.12	8.46 8.22	5.88 5.66	
18	$C_{30}H_{40}N_2O_3$	476.66	85	70 EtOH	Red brown	75.59 75.34	8.46 8.64	5.88 5.35	
19	$C_{23}H_{36}N_2O_2$	372.54	72	65 EtOH	Brown	74.15 74.45	9.74 9.41	7.52 7.52	
20	$C_{24}H_{37}N_3O$	383.58	93.	75 EtOH	Red brown	75.15 75.43	9.72 9.88	10.95 10.73	

Biological activity.

All the prepared compounds were screened for their activity against Gram-positive bacteria (*Stophyloccus aureus, Bacillus subtilis, Bacillus cereus*), Gram-negative bacteria (*Pseudomonas aurignosa, Echerichia coli, Enterobacter aerogenes*), as well as fungi (*Aspergillus niger, Penicillium italicum, Fusarium oxysporum*). The results are listed in Tables **2** and **3**.

Table 2: Antibacterial activity of the prepared compounds.

Compds.	*Stophyloccus aureus s*	*Bacillus subtilis*	*Bacillus cereus*	*Pseudomonas aurignosa*	*Escherichia coli*	*Enterobacter aerogenes*
2	+	+	+	+	+	+
3	+	++	+	+	+	++
4	++	++	++	++	+	++
5	+	+	+	+	+	+
6	+++	++	+	++	+	+++
7	+	+	+	+	+	+
8	+	+	+	+	+	+
9	++	++	+	+	+	+
10	+	+	+	++	+	++
11	++	+++	+	+	+	+
12	++	+++	++	++	+	+++
13	++	+++	++	+++	+	+++
14	++	+++	+	++	+	++
15	++	++	+	++	+	++
16	+	+	+	++	+	+
17	+	+	+	+	+	+
18	+	+	+	+	+	+
19	++	+	+	++	+	++
20	++	++	+	+	+	+
Amoxicillin	++	+++	+	++	+	++

It is apparent from the data listed in Table **2** that some of the synthesized compounds showed antibacterial activity. However, concerning the activity against Gram-positive bacteria (*Bacillus subtilis*), compounds **6, 11, 12, 13,** and **14** showed excellent activity, compounds **4, 9, 15, 19** and **20** exhibit good activity, whereas compounds **2, 3, 5, 7, 8 , 10** and **18** showed moderate activity. On the other hand, the Gram-negative bacteria (*Pseudomonas aurignosa*) showed high responses to five of the prepared products. Compound **13** showed the maximum activity higher than that of

amoxicillin [191]. Compound **6** exhibits excellent antibacterial activity towards (*Entrobacter aerogenes*).

Concerning the data of antifungal activity in Table 3, compounds **6** and **11** showed excellent activity against (*Aspergillus niger*), comparable to mycostatin, while compounds **8, 9, 10, 15** and **19** exhibit good activity. Also, compound **2** displays good activity toward (*Penicillium italicum*). In general, the data obtained from the microbiological screening showed that the activity of the most some synthesized compounds showed moderately activity.

Table 3: Antifungal activity of the prepared compounds.

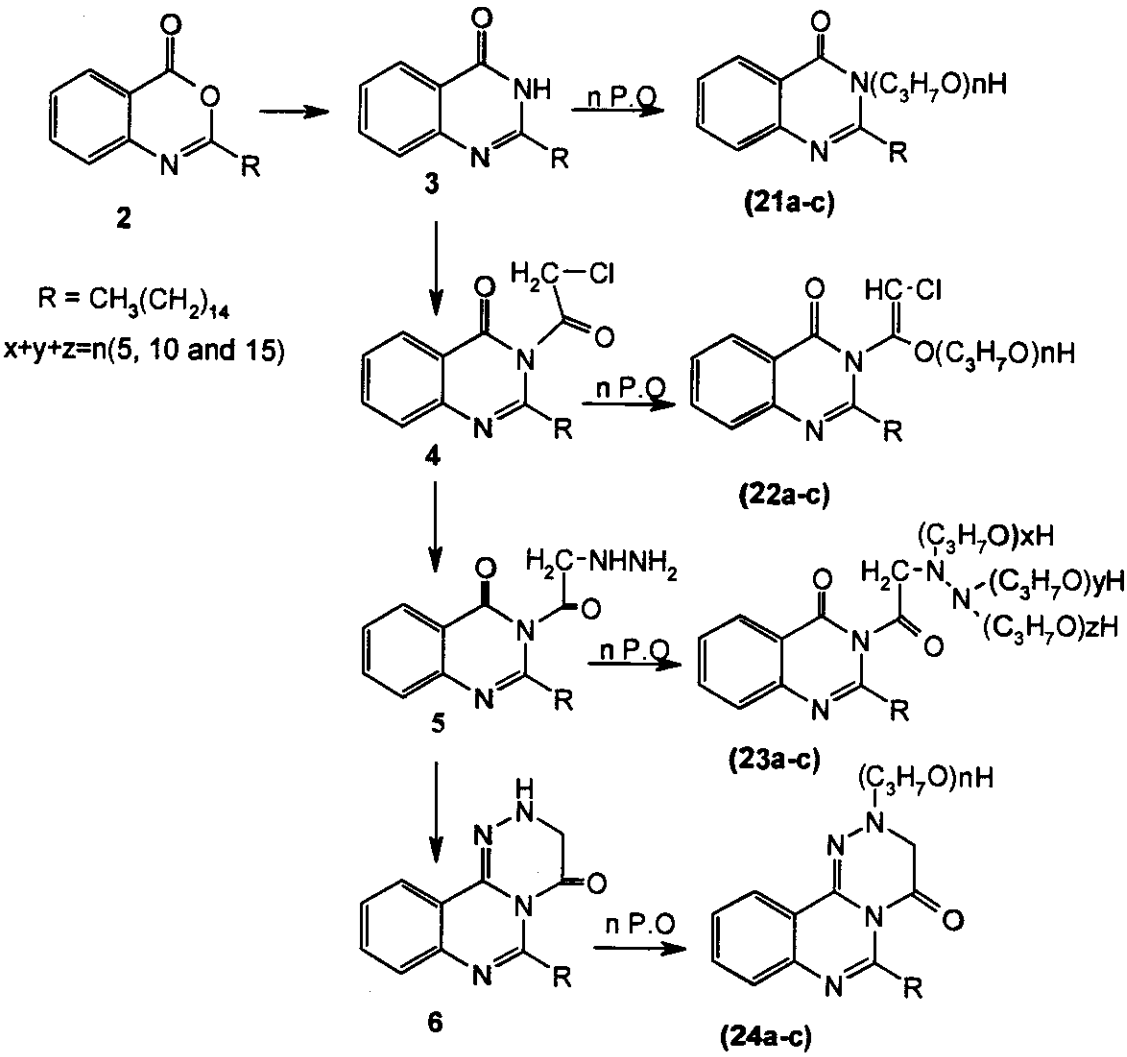
Compds.	*Aspergillus niger*	*Penicillium italicum*	*Fusarium oxysporum*
2	+	++	+
3	++	+	+
4	++	+	+
5	+	+	+
6	+++	+	+
7	++	+	+
8	+	+	+
9	++	+	+
10	++	+	+
11	+++	+	+
12	+	+	+
13	++	+	+
14	++	+	+
15	+	+	+
16	++	+	+
17	++	+	+
18	+	+	+
19	+	+	+
20	++	+	+
Mycostatin	++	+	+

PART (2)

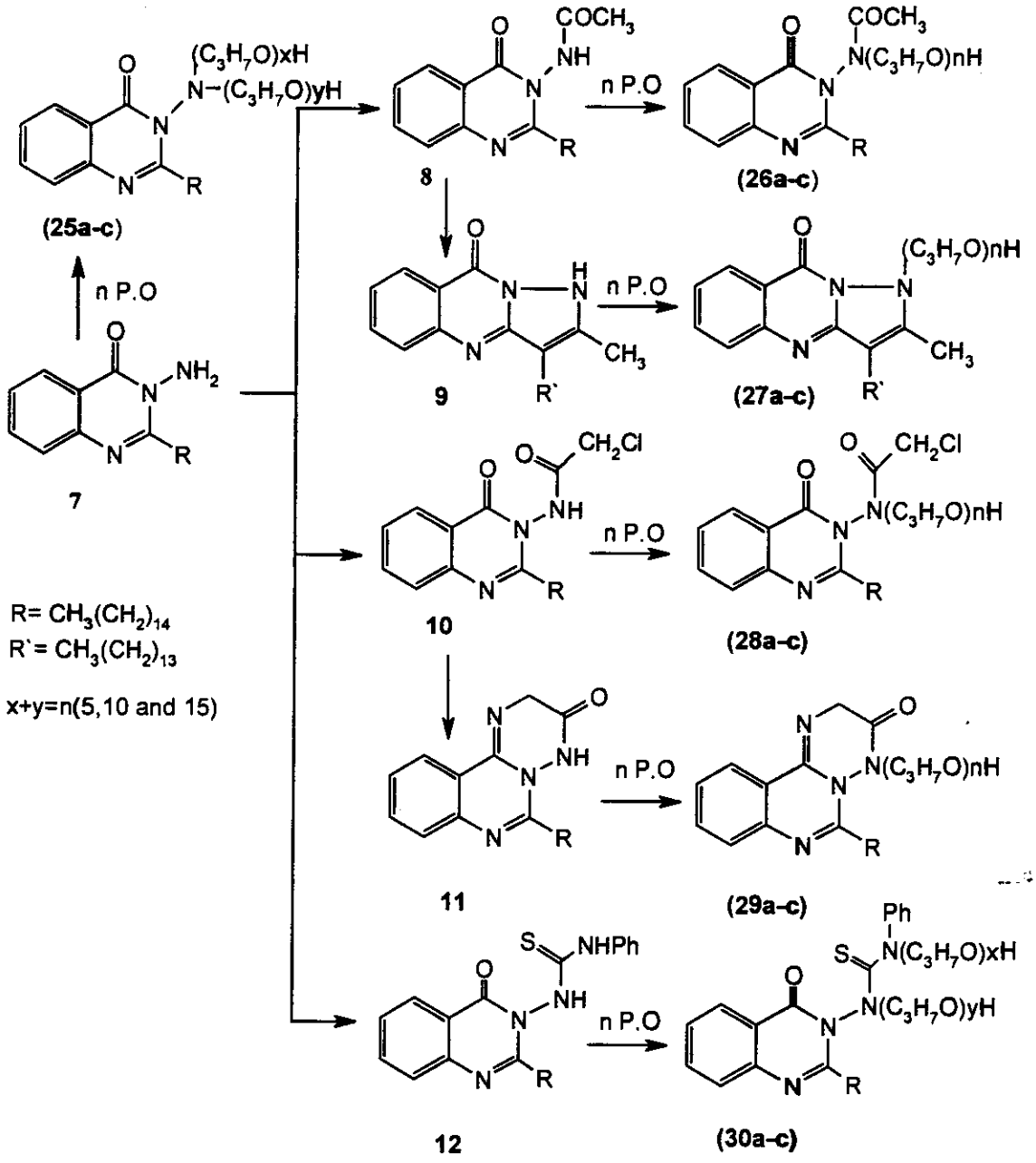
Preparation of nonionic surface active agents from the synthesized compounds.

The terms of nonionic surfactants refers chiefly to polyoxypropylene derivatives, they are usually prepared by the addition of different moles (n) of propylene oxide (n ≅ 5, 10 and 15) to synthesized products **(3-20)** which contain one or more active hydrogen atoms (NH, OH, SH COOH) to yield polypropenoxylation products **(21a-c-38a-c)**. This reaction is one of the principal processes used to introduce hydrophilic functional groups into an hydrophobic organic moiety (scheme **3**, **4**, **5**).

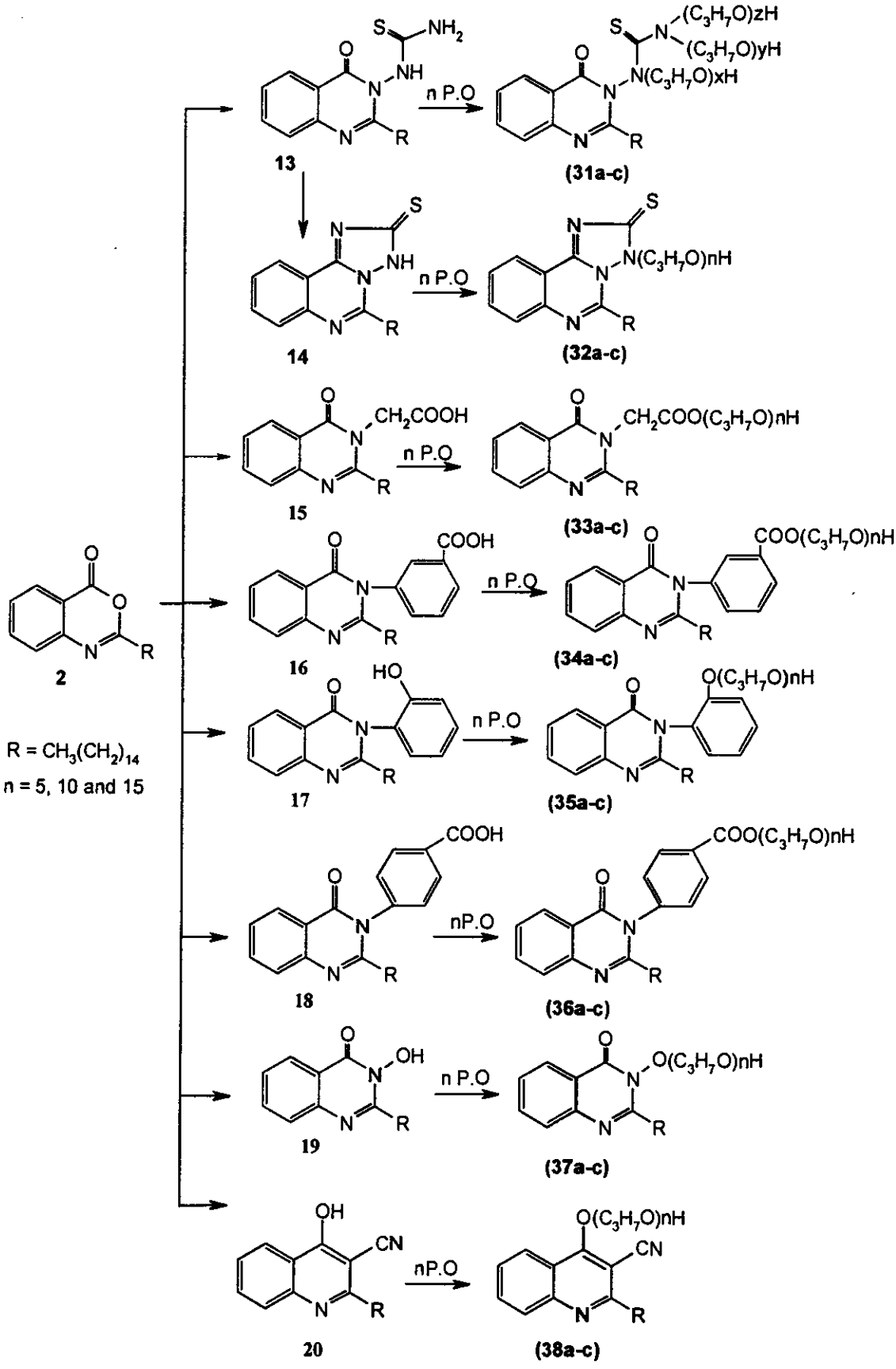
Nonionic surfactants find diverse applications, both in industry and in the home. Their moderate foaming and good detergency are employed in a variety of ways in leather industry. It is used to accelerate soaking, and liming is improved by the addition of wetting agents [192]. Also nonionic surfactants are used extensively because of their good detergency, easy rinsing and low foaming in cleaning of milk and beer bottles.


2
R = $CH_3(CH_2)_{14}$
x+y+z=n(5, 10 and 15)
3
n P.O
(21a-c)
4
H_2C-Cl
HC-Cl
$O(C_3H_7O)nH$
(22a-c)
5
$H_2C-NHNH_2$
$(C_3H_7O)xH$
$(C_3H_7O)yH$
$(C_3H_7O)zH$
(23a-c)
6
$(C_3H_7O)nH$
(24a-c)

Scheme 3


(C3H7O)xH
N–(C3H7O)yH
(25a-c)
n P.O
NH2
7
R= CH3(CH2)14
R`= CH3(CH2)13
x+y=n(5,10 and 15)
COCH3
NH
8
n P.O
N(C3H7O)nH
(26a-c)
9
CH3
R`
(27a-c)
CH2Cl
10
(28a-c)
11
(29a-c)
NHPh
12
Ph
N(C3H7O)xH
N(C3H7O)yH
(30a-c)

Scheme 4


2
R = $CH_3(CH_2)_{14}$
n = 5, 10 and 15
13
(31a-c)
14
(32a-c)
15
(33a-c)
16
(34a-c)
17
(35a-c)
18
(36a-c)
19
(37a-c)
20
(38a-c)
n P.O
nP.O

Scheme 5

The structures of the synthesized nonionic surfactants were confirmed via IR and ^{1}HNMR spectra e.g.

IR spectrum of **(23a)** after the addition of propylene oxide, showed, broad band at 3360 for υ (OH) of alcohol product two and bands at 1097 cm^{-1} and 935 cm^{-1} characteristic for νC-O-C ether linkage of polypropenoxy chain, beside the original bands of the compound (*cf.* fig. 18).

IR spectrum of **(28b)** showed, broad band at 3404 for υ (OH) of alcohol product two and bands at 1093 cm^{-1} and 940 cm^{-1} characteristic for νC-O-C ether linkage of polypropenoxy chain, beside the original bands of the compound (*cf.* fig. 19).

IR spectrum of **(31)** showed, broad band at 3389 for υ (OH) of alcohol product two and bands at 1108 cm^{-1} and 996 cm^{-1} characteristic for νC-O-C ether linkage of polypropenoxy chain, beside the original bands of the compound (*cf.* fig. 20).

IR spectrum of **(32c)** showed, broad band at 3454 for υ (OH) of alcohol product two and bands at 1036 cm^{-1} and 956 cm^{-1} characteristic for νC-O-C ether linkage of polypropenoxy chain, beside the original bands of the compound (*cf.* fig. 21).

IR spectrum of **(36c)** showed, broad band at 3399 for υ (OH) of alcohol product two and bands at 1095 cm^{-1} and 943 cm^{-1} characteristic for νC-O-C ether linkage of polypropenoxy chain, beside the original bands of the compound (*cf.* fig. 22).

^{1}HNMR spectrum of **(32a)** showed, the protons of propenoxy group were assigned as a broad multiple signals in the region (3.0-3.9) ppm, beside the other protons of the compound. (*cf.* fig. 28)

^{1}HNMR spectrum of **(35c)** showed, the protons of propenoxy group were assigned as a broad multiple signals in the region (3.2-3.7) ppm, beside the other protons of the compound. (*cf.* fig. 29).

^{1}HNMR spectrum of **(36b)** showed, the protons of propenoxy group were assigned as a broad multiple signals in the region (3.2-3.7) ppm, beside the other protons of the compound. (*cf.* fig. 30)

1. Surface active properties of nonionic surfactants.

The surface active and related properties, including, surface and interfacial tension, cloud point, wetting, emulsification properties, and foaming were investigated systematically in order to evaluate the possible application of these products in the different industrial fields.

1.1- Surface and interfacial tension.

The surface and interfacial tension of the prepared nonionic surfactants are recorded in **Table 4**. It is evident that, nonionic surfactants with heterocyclic moiety recorded lower values than those prepared from saturated fatty acids, which might be attributed to increasing the hydrophilicity of the molecules. On the other hand, the surface activity is improved by introducing heterocyclic nucleus in the molecules. In generally, these value increases as the mass of hydrophilic groups increase within the range under study.

1.2- Cloud point.

The most efficient use of nonionic surfactants in aqueous systems is by understanding a property called cloud point, which is the temperature at which the aqueous solution of the prepared nonionic surfactants shows turbidity on heating. The cloud points of the synthesized surfactant are shown in **Table 4**. The results indicated that the values of cloud point increases by increasing the number of propylene oxide units.

1.3- Wetting time.

Nonionic surfactants are among the most powerful wetting agents. All the synthesized surfactants are efficient wetting agents **Table 4**. It was reported that nonionic with a low propylene oxide content have been found to be the most efficient wetting promoter.

1.4- Emulsifying properties.

Studies are still being carried out on the utilization of surfactant in emulsion formulation, which is of immense importance to technological development. The data in Table **2** show that greater emulsifying properties were obtained with derivatives containing propylene oxide units incorporated into their structure, where the emulsifying properties increase with decreasing number of propylene oxide units. It is very interest noted that the emulsion stability of the prepared compounds is lower than the corresponding propenoxylated fatty acid which not containing the heterocyclic moiety , these results, might lead to the application of the surfactants of choice in pesticide and cosmetic formulation.

1.5- Foam power.

Low foaming power is the characteristic property of nonionic surfactants, which permits some recent applications for these in dyeing auxiliary textile industry [193]. It was reported that; nonionic surfactants have low foam, on the other hand, the foam height of the prepared surfactants increases with increasing propylene oxide unit per molecule of surfactant.

2. Biodegradability.

Biodegradation die-away test in river water gave good or excellent results **Table 5**. The results of biodegradation reflect that; the biodegradability decreases with increasing the number of propylene oxide units. This leads to the conclusion that a longer propylene oxide chain makes the diffusion of the molecule through the cell membrane and thus also the degradation, more difficult.

3. Biological activity.

Nonionic surfactants which contain heterocyclic moiety afforded a double function as surface-active agents and antimicrobial activities.[194] So, all the prepared surfactants were tested for their bacterocidal activities against the test organisms as represented Gram-positive bacteria (*Stophyloccus aureus, Bacillus*

subtilis, Bacillus cereus) and Gram–negative (*Pseudomonas aurignosa, Echerichia coli, Enterobacter aerogenes*). Antifungal activity (*Aspergillus niger, Penicillium italicum, Fusarium oxysporum*) are given in **Table 6** and 7. The data show that, the presence of heterocyclic moiety in the prepared nonionic surfactant molecule revealed an increase in the biological activity [195]. It is therefore clear that these surfactants were effective and inhibited the growth of all tested microorganism.

Table 4: Surface properties of these compounds.

Compd.	n	Surface Tension (dyne/cm) 0.1 %	Interfacial Tension (dyne/cm) 0.1 %	Cloud Point °C 1%	Wetting time (sec) 0.1%	Emulsion stability (min.)	Foam height (mm) 1%
21a	5	30	9.5	79	35	127	118
21b	10	32	10.5	95	26	111	126
21c	15	34	11.0	>100	20	95	134
22a	5	31	10.0	70	37	128	126
22b	10	33	11.0	90	30	112	135
22c	15	36	11.5	95	22	96	150
23a	5	32	10.5	68	40	130	133
23b	10	35	12.0	87	33	115	140
23c	15	38	13.5	93	25	98	155
24a	5	29	7.5	67	43	117	100
24b	10	31	9.5	77	34	87	125
24c	15	33	10.5	91	27	74	150
25a	5	30	8.5	63	46	120	105
25b	10	32	10.5	72	39	90	128
25c	15	35	12.0	88	30	77	146
26a	5	31	9.5	59	48	125	115
26b	10	34	11.0	66	43	92	147
26c	15	37	12.5	76	32	79	152
27a	5	29	9.0	69	46	118	113
27b	10	31	10.5	74	36	86	127
27c	15	34	11.0	84	27	74	142
28a	5	31	9.0	64	48	120	115
28b	10	34	11.0	79	39	180	130
28c	15	37	11.5	88	30	76	146
29a	5	33	9.5	61	52	123	119
29b	10	36	11.5	76	42	90	133
29c	15	39	12.0	91	32	70	154
30a	5	33	8.5	65	41	117	120
30b	10	35	9.5	74	33	96	125
30b	15	37	10.0	86	28	82	128
31a	5	34	9.0	62	44	118	120
31b	10	37	10.5	70	36	97	127
31b	15	39	11.0	92	30	83	130
32a	5	36	9.5	57	43	120	125
32b	10	39	11.0	66	39	99	130
32b	15	41	12.5	85	33	85	140
33a	5	33	8.5	65	41	117	120
33b	10	35	9.5	74	32	96	125
33b	15	37	10.0	86	28	82	128
34a	5	34	9.0	62	44	118	120
34b	10	37	10.5	70	36	97	127
34b	15	39	11.0	92	30	83	130
35a	5	36	9.5	57	48	120	125
35b	10	39	11.0	66	39	99	130
35b	15	41	12.5	85	32	85	140
36a	5	33	8.5	65	41	117	120
36b	10	35	9.5	74	33	96	125
36b	15	37	10.0	86	28	82	128
37a	5	34	9.0	62	44	118	120
37b	10	37	10.5	70	36	97	127
37b	15	39	11.0	92	30	83	130
38a	5	36	9.5	57	48	120	125
38b	10	39	11.0	66	39	99	130
38b	15	41	12.5	85	32	85	140

Table 5: Biodegradability of the Prepared Surfactants.

Compd.	n	1st day	2nd day	3rd day	4th day	5th day	6th day	7th day
21a	5	54	67	77	86	93	-	-
21b	10	49	60	69	81	92	97	-
21c	15	47	58	64	73	88	94	-
22a	5	52	65	75	84	92	-	-
22b	10	47	58	66	78	90	-	-
22c	15	45	54	61	70	85	94	-
23a	5	50	64	73	82	91	-	-
23b	10	46	57	64	76	87	93	-
23c	15	43	52	59	68	79	87	94
24a	5	57	65	79	88	93	-	-
24b	10	54	62		84	91	-	-
24c	15	51	57	70	75	89	96	-
25a	5	55	64	77	86	92	-	-
25b	10	51	59	72	81	89	93	-
25c	15	49	55	66	75	83	95	-
26a	5	54	63	76	84	91	-	-
26b	10	49	58	69	78	87	92	-
26c	15	46	53	63	72	80	88	96
27a	5	51	61	70	82	93	-	-
27b	10	50	59	68	79	88	97	-
27c	15	47	57	64	75	85	93	-
28a	5	49	59	68	79	90	-	-
28b	10	48	57	65	75	83	93	-
28c	15	45	53	61	71	80	89	95
29a	5	48	57	66	78	89	-	-
29b	10	46	54	62	73	82	93	-
29c	15	43	51	58	68	78	89	-
30a	5	53	63	72	82	92	-	-
30b	10	51	59	68	78	89	95	-
30b	15	48	55	64	76	83	92	-
31a	5	51	61	69	80	89	-	-
31b	10	48	57	65	75	85	91	-
31b	15	45	52	62	72	79	89	97
32a	5	49	58	67	78	87	92	-
32b	10	46	54	62	73	82	89	-
32b	15	43	79	58	68	77	87	96
33a	5	53	63	72	82	92	-	-
33b	10	51	59	68	78	89	95	-
33b	15	48	55	64	76	83	92	-
34a	5	51	61	69	80	89	-	-
34b	10	48	57	65	75	85	91	-
34b	15	45	52	62	72	79	89	97
35a	5	49	58	67	78	87	92	-
35b	10	46	54	62	73	82	89	-
35b	15	43	79	58	68	77	87	96
36a	5	53	63	72	82	92	-	-
36b	10	51	59	68	78	89	95	-
36b	15	48	55	64	76	83	92	-
37a	5	51	61	69	80	89	-	-
37b	10	48	57	65	75	85	91	-
37b	15	45	52	62	72	79	89	97
38a	5	49	58	67	78	87	92	-
38b	10	46	54	62	73	82	89	-
38b	15	43	79	58	68	77	87	96

Table 6: Antibacterial activity of the prepared compounds.

Compd	*n*	*Staphylococcus aureus s*	*Bacillus subtilis*	*Bacillus cereus*	*Pseudomonas aurignosa*	*Escherichia coli*	*Enterobacter aerogenes*
21a	5	+	+++	+	+	+	+
21b	10	++	+++	++	++	+	++
21c	15	++	+++	++	++	++	+++
22a	5	++	++	++	+	+	+
22b	10	++	+++	++	++	+	++
22c	15	+++	+++	+++	++	+	++
23a	5	++	++	++	+	+	+
23b	10	++	++	++	+++	+	++
23c	15	+++	++	+++	++	+	++
24a	5	+	+	+	+	+	++
24b	10	++	++	++	++	+	++
24c	15	++	++	++	++	+	++
25a	5	+	++	+	++	+	+
25b	10	+	+++	+	++	+	++
25c	15	++	+++	++	++	+	++
26a	5	++	+++	++	+	+	++
26b	10	++	++	++	++	+	++
26c	15	++	+++	++	++	+	++
27a	5	+	+	+	+	+	+
27b	10	+	+	+	++	+	++
27c	15	+	+	+	++	++	+++
28a	5	+	++	+	++	+	+
28b	10	+	+++	+	++	+	++
28c	15	+	+++	+	++	++	+++
29a	5	+	+	+	+	+	+
29b	10	++	++	++	++	+	++
29c	15	++	++	++	++	+	++
30a	5	+	+	+	+	+	+
30b	10	+	+	+	++	+	++
30b	15	+	+	+	++	++	++
31a	5	+	+	+	+	+	+
31b	10	+	+	+	++	+	++
31b	15	+	+	+	++	++	++
32a	5	++	++	++	+	+	+
32b	10	++	++	++	+	+	++
32b	15	++	++	++	++	+	++
33a	5	+	+	+	+	+	+
33b	10	+	++	+	+	+	++
33b	15	+	++	+	++	++	++
34a	5	+	+	+	+	+	+
34b	10	+	++	+	+	+	++
34b	15	+	++	+	++	++	++
35a	5	++	++	++	+	+	+
35b	10	+	+	+	++	+	++
35b	15	++	++	++	++	+	++
36a	5	+	+++	+	+	+	+
36b	10	+	++	+	++	+	++
36b	15	+	+++	+	++	+	++
37a	5	+	++	+	+	+	+
37b	10	++	+++	++	++	+	++
37b	15	++	+++	++	++	+	++
38a	5	+	+	+	++	+	+
38b	10	++	++	++	++	+	++
38b	15	++	++	++	++	+	++

(+++) very strong inhibition, (++) strong inhibition, and (+) moderate inhibition.

Table 7: Antifungal activity of the prepared compounds.

Compd	*n*	*Aspergillus niger*	*Penicillium italicum*	*Fusarium oxysporum*
21a	5	+	++	++
21b	10	++	++	++
21c	15	+++	+++	++
22a	5	+	++	++
22b	10	+	++	++
22c	15	+++	++	++
23a	5	++	++	++
23b	10	++	++	++
23c	15	++	++	++
24a	5	+	+	++
24b	10	++	++	++
24c	15	+++	++	++
25a	5	++	++	+
25b	10	++	++	++
25c	15	+++	+	++
26a	5	++	+	++
26b	10	++	++	++
26c	15	+++	++	++
27a	5	+	+	+
27b	10	++	+	+
27c	15	++	+	+
28a	5	++	++	++
28b	10	+++	++	++
28c	15	++	+	++
29a	5	+	+	+
29b	10	++	++	++
29c	15	++	++	++
30a	5	++	+	+
30b	10	++	+	+
30b	15	++	+	+
31a	5	+	+	+
31b	10	++	++	++
31b	15	++	+	+
32a	5	++	++	++
32b	10	++	++	++
32b	15	+++	++	++
33a	5	++	+	+
33b	10	++	++	++
33b	15	++	++	+
34a	5	+	++	+
34b	10	+	++	++
34b	15	+	++	++
35a	5	++	++	++
35b	10	+++	+	+
35b	15	++	+	++
36a	5	+	++	+
36b	10	++	++	+
36b	15	++	+	++
37a	5	+	++	+
37b	10	++	++	++
37b	15	+++	+	++
38a	5	+	+	+
38b	10	++	++	++
38b	15	++	++	++

(+++) very strong inhibition, (++) strong inhibition, and (+) moderate inhibition.

Figures

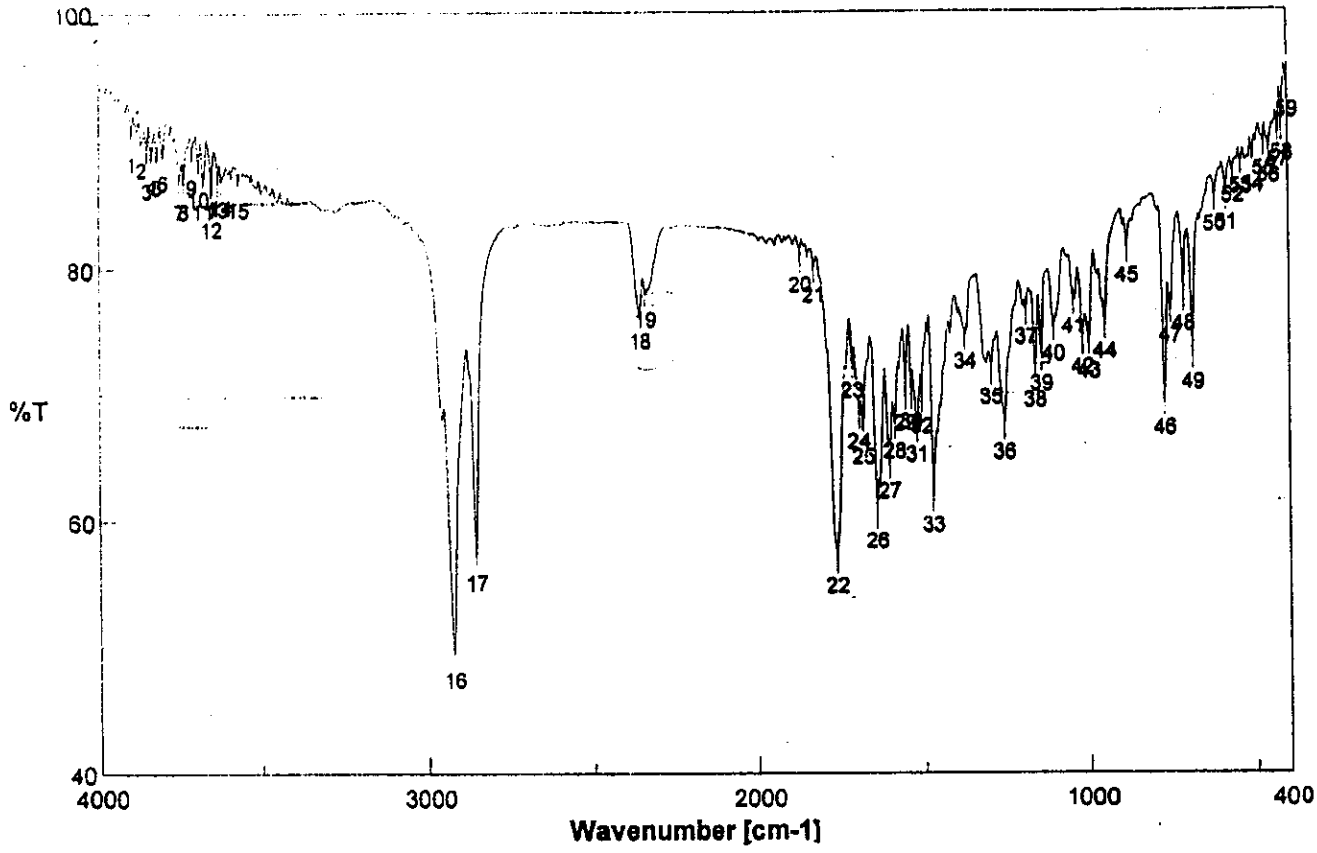
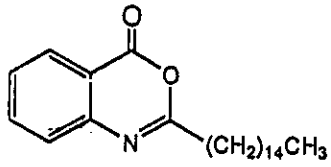


Fig (1)



2

No.	cm-1	%T	No.	cm-1	%T	No.	cm-1	%T
1	3901.29	90.4522	2	3869.47	89.8952	3	3853.08	88.2426
4	3837.65	88.6166	5	3619.33	88.5529	6	3801.01	88.8797
7	3748.94	86.5946	8	3734.48	86.6026	9	3710.37	88.5326
10	3689.16	87.5657	11	3874.69	86.5692	12	3648.66	85.194
13	3628.41	68.7948	14	3617.8	86.9049	15	3565.74	86.6921
16	2920.66	49.437	17	2850.27	57.1384	18	2359.48	76.2266
19	2342.12	77.9187	20	1867.72	80.666	21	1828.19	79.8464
22	1763.58	56.8261	23	1716.34	72.2972	24	1698.02	68.266
25	1684.52	67.0498	26	1644.98	60.4309	27	1606.41	64.365
28	1590.02	67.4877	29	1558.2	69.7097	30	1539.88	89.8382
31	1522.52	67.2153	32	1507.1	69.4723	33	1472.38	61.6932
34	1374.03	74.5022	35	1294.97	71.7404	36	1254.47	67.3941
37	1187.94	76.427	38	1160.94	71.4898	39	1141.65	72.7532
40	1105.01	75.1011	41	1045.23	77.2106	42	1017.27	74.0481
43	998.946	73.7155	44	951.698	75.2657	45	887.095	81.3052
48	777.172	69.2578	47	759.816	76.433	48	718.354	77.4042
49	691.355	72.8335	50	622.895	85.2708	51	588.182	85.4035
52	567.934	87.5409	53	542.863	88.258	54	507.187	88.3665
55	473.439	89.702	56	458.011	89.0877	57	431.012	90.3707
58	419.442	90.9154	59	404.014	94.2966			

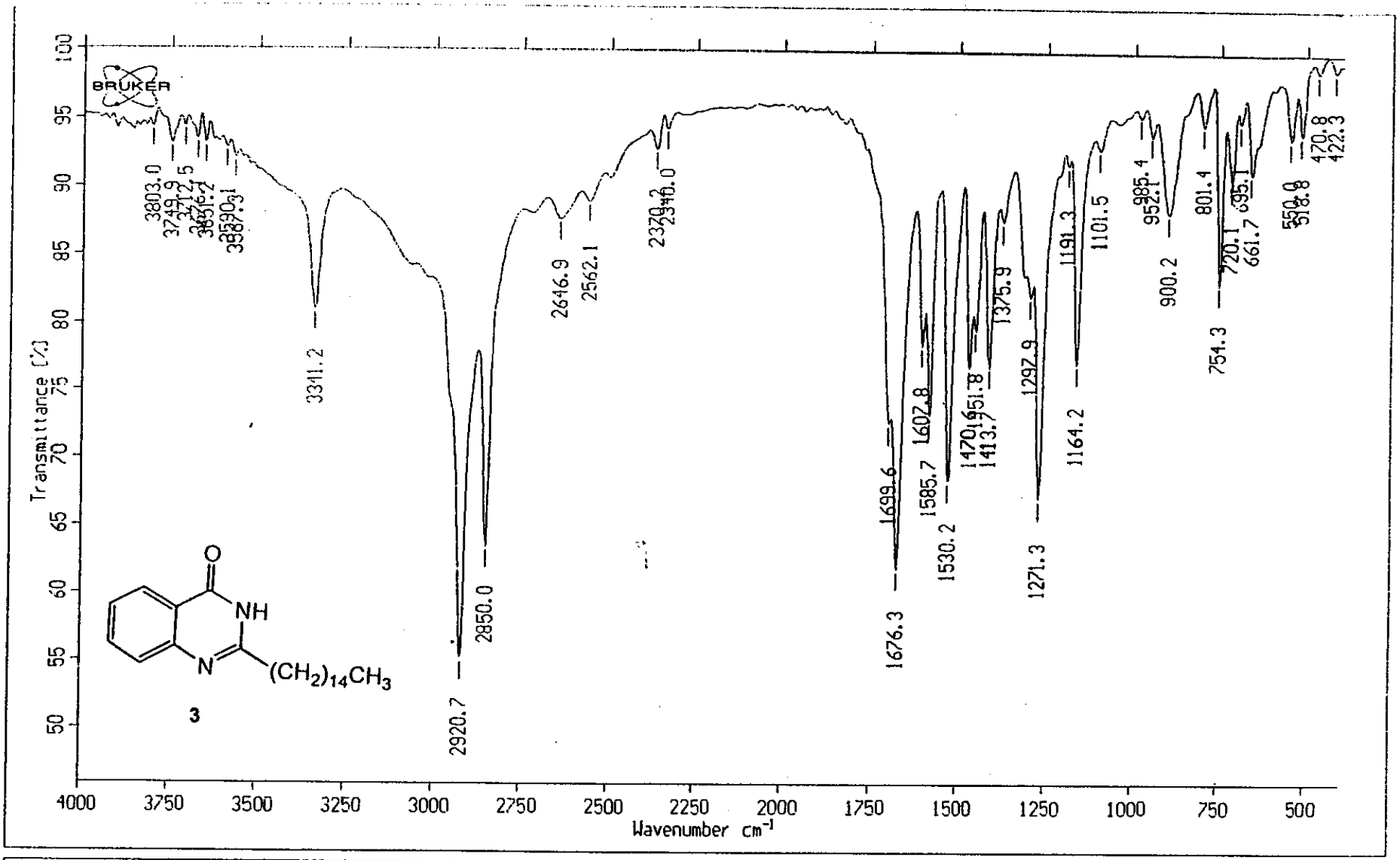


Fig (2)

SAMPLE_N. 32 20/ 5/2005 3:26:37

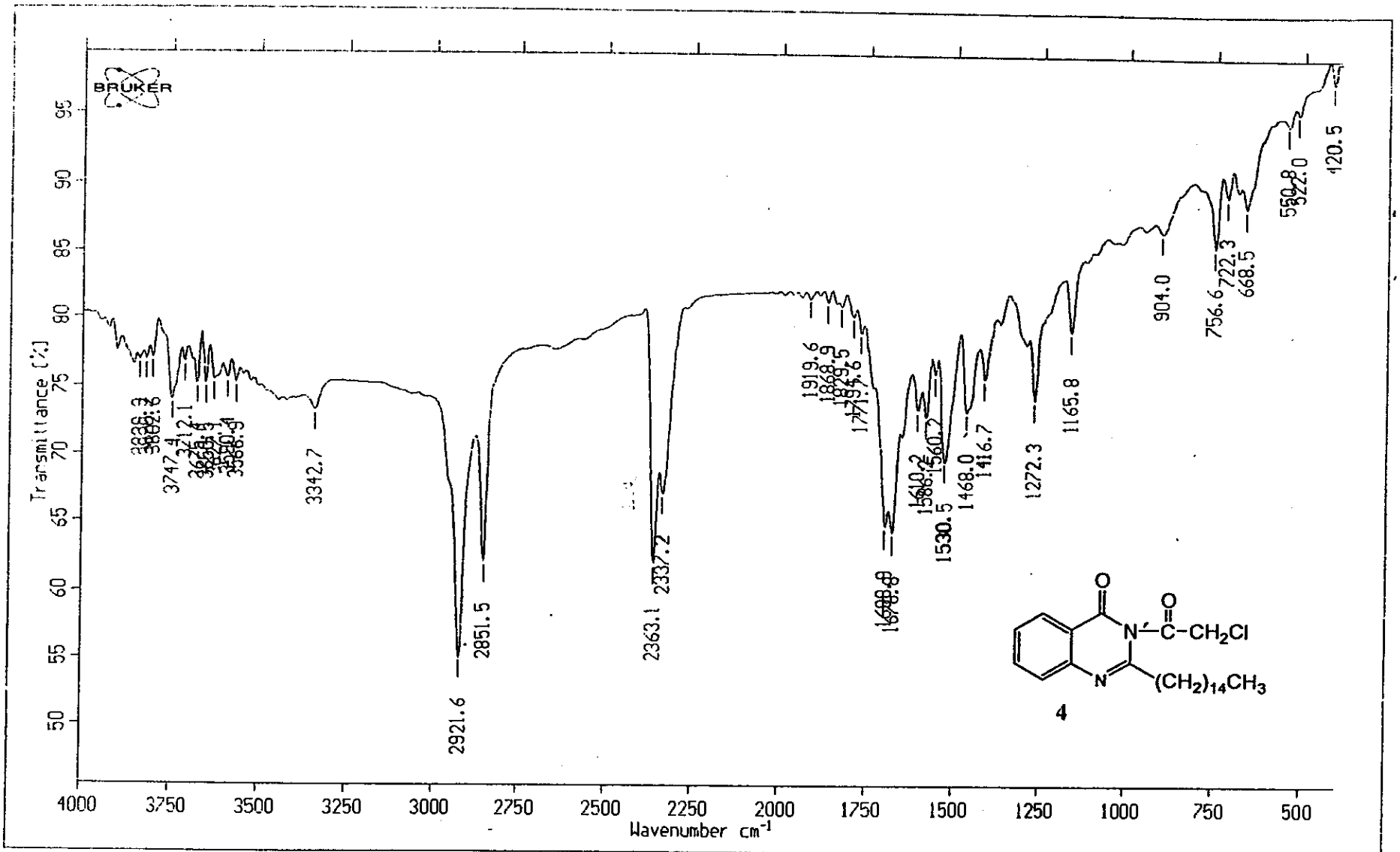
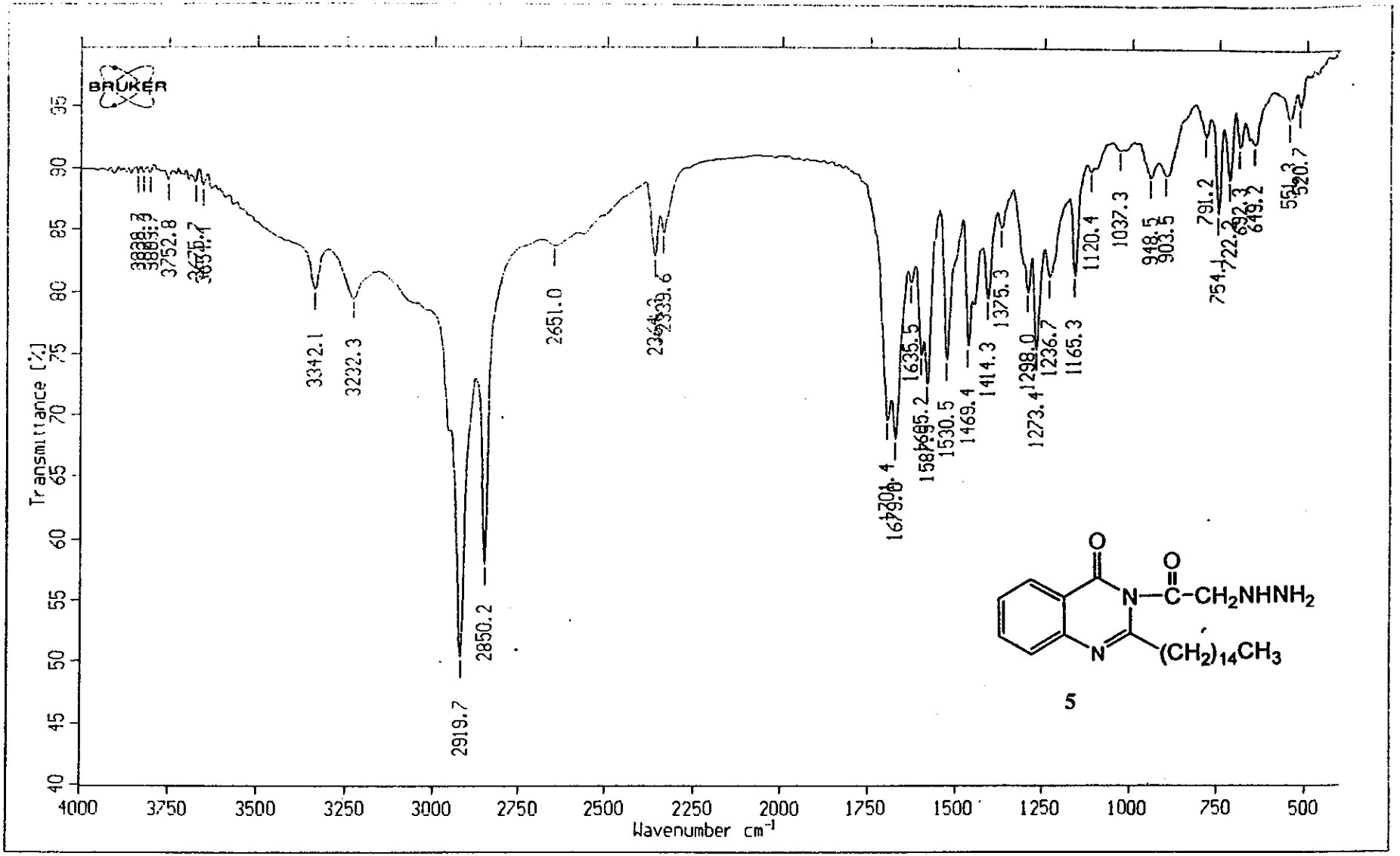


Fig (3)

M. AMIN

SAMPLE_N. 81 20/ 5/2005 14:16:42



5

Fig (4)

SAMPLE_N. 243 -12/ 3/2005 12:48: 8

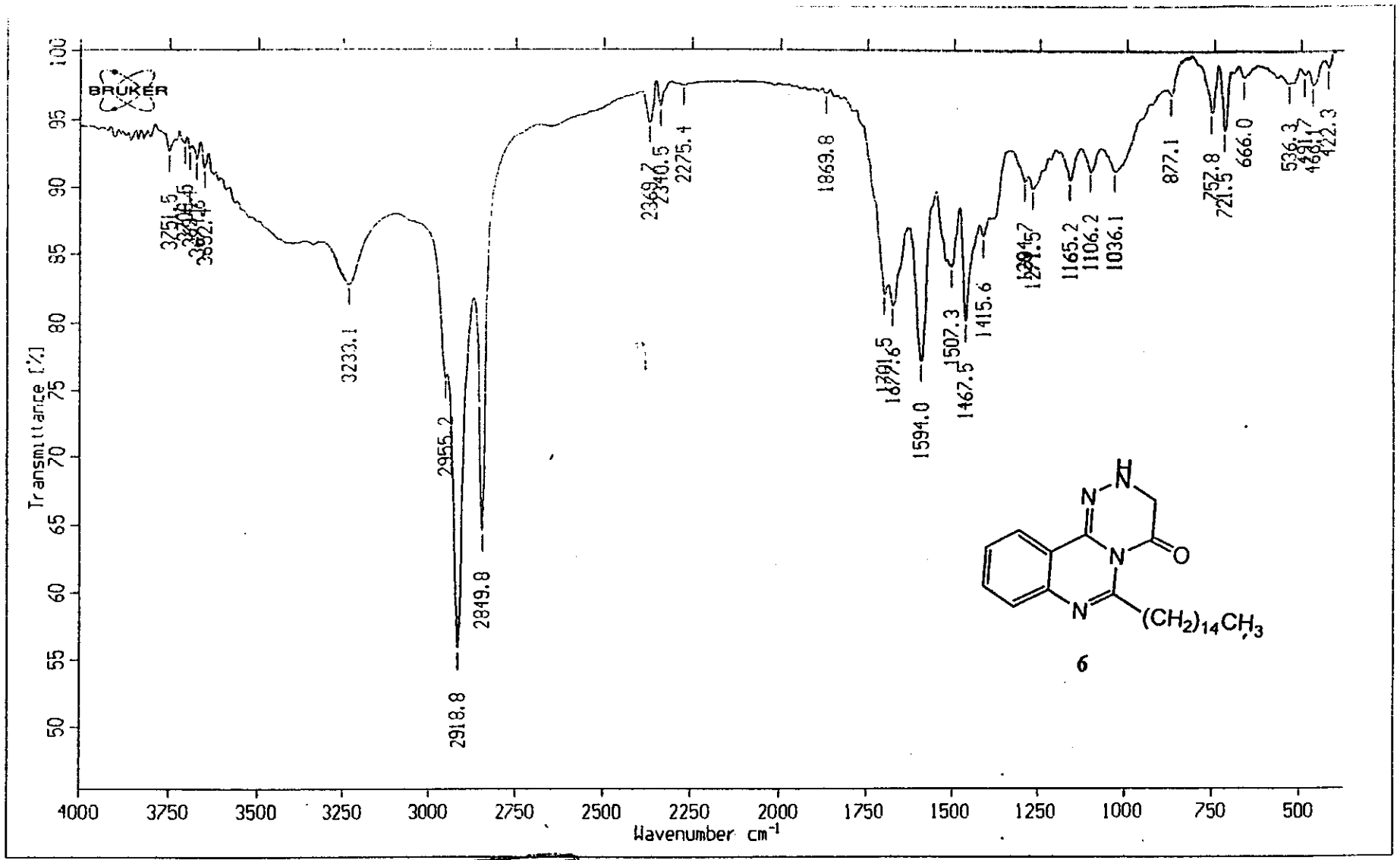


Fig (5)

SAMPLE_N.665 5/12/2004 12:59:

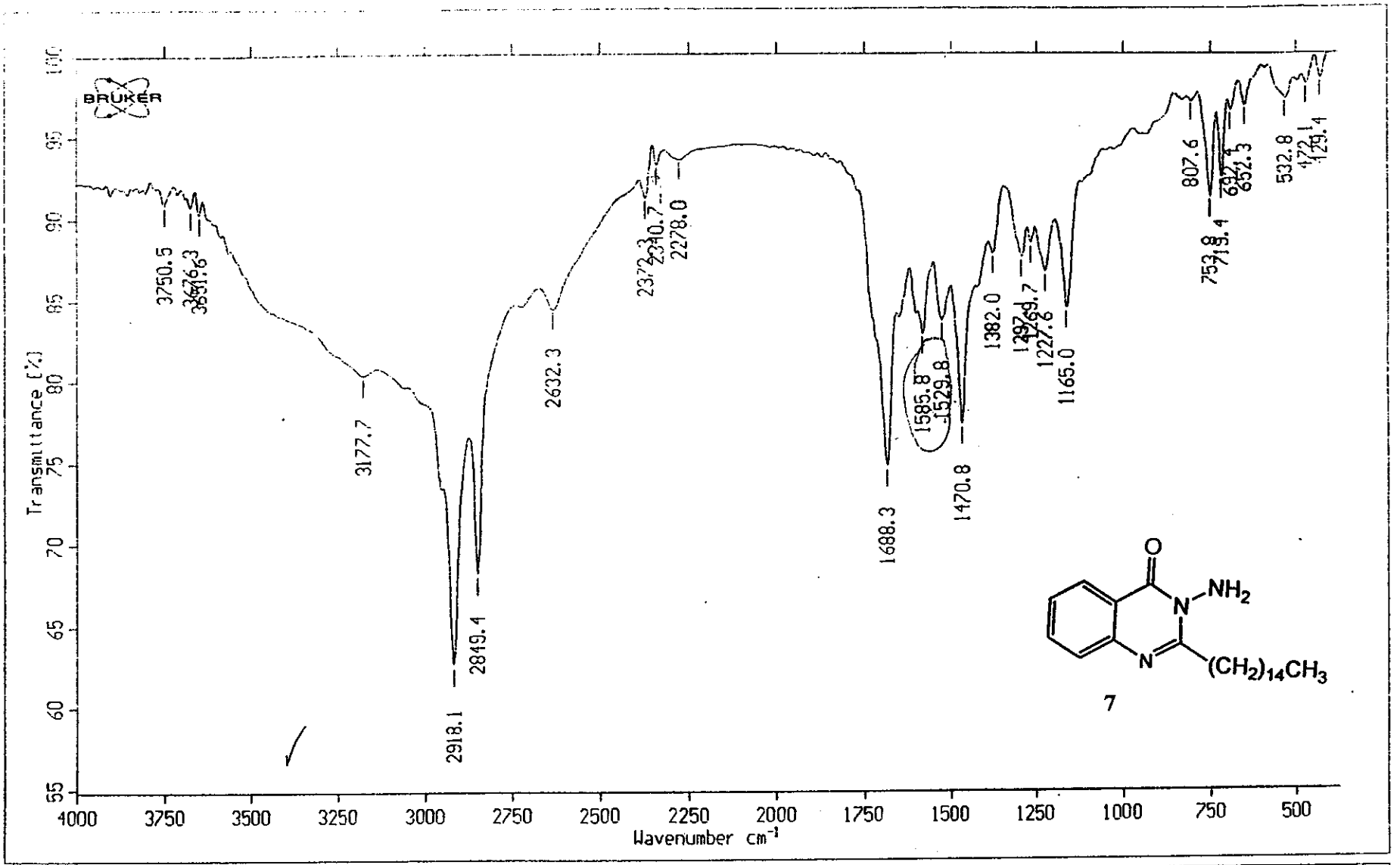
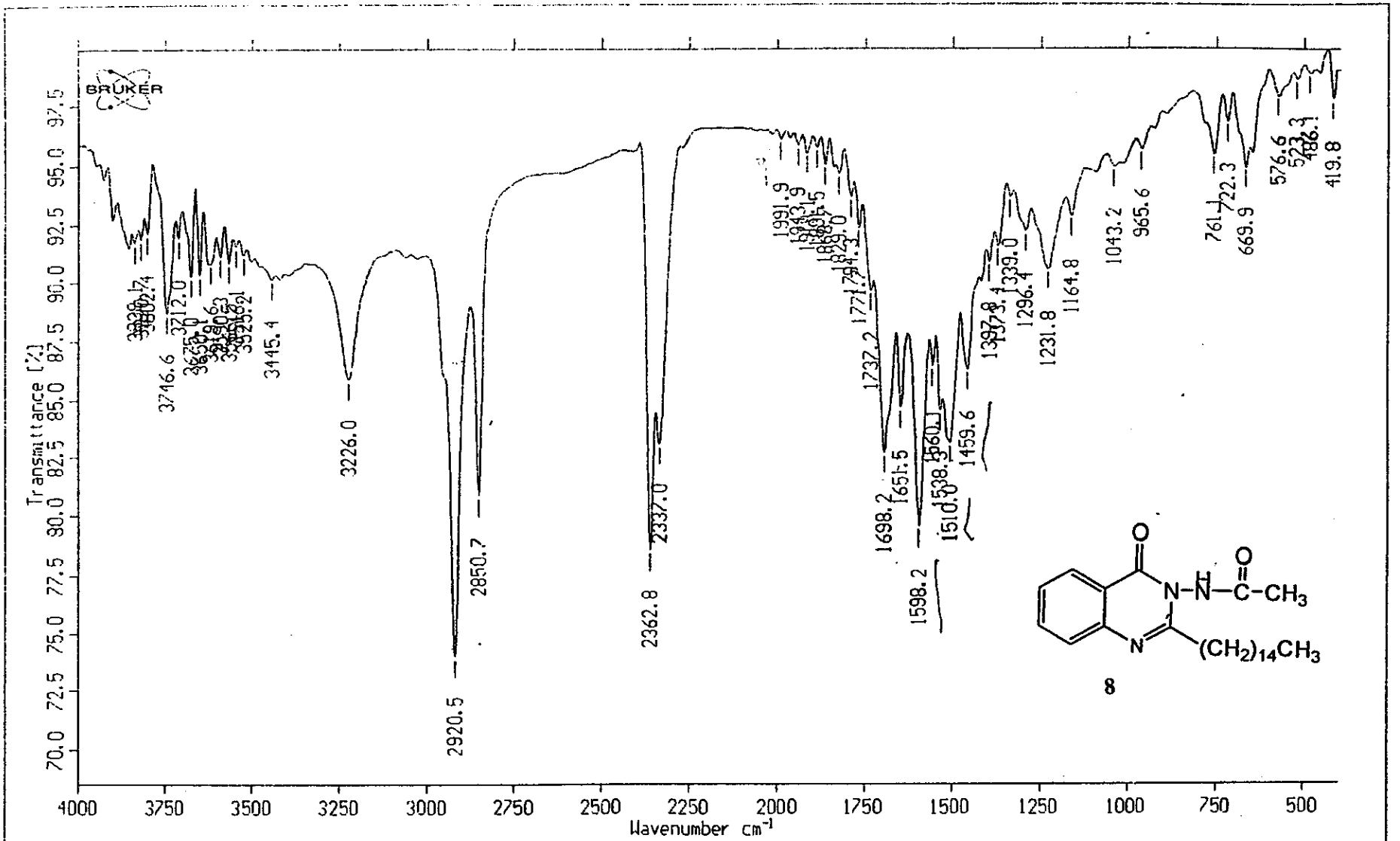


Fig (6)

M. AMIN

SAMPLE_N.182 · 10/ 5/2005 9:30:53



8

Fig (7)

M. AMIN SAMPLE_N.77 20/ 5/2005 14: 5:15

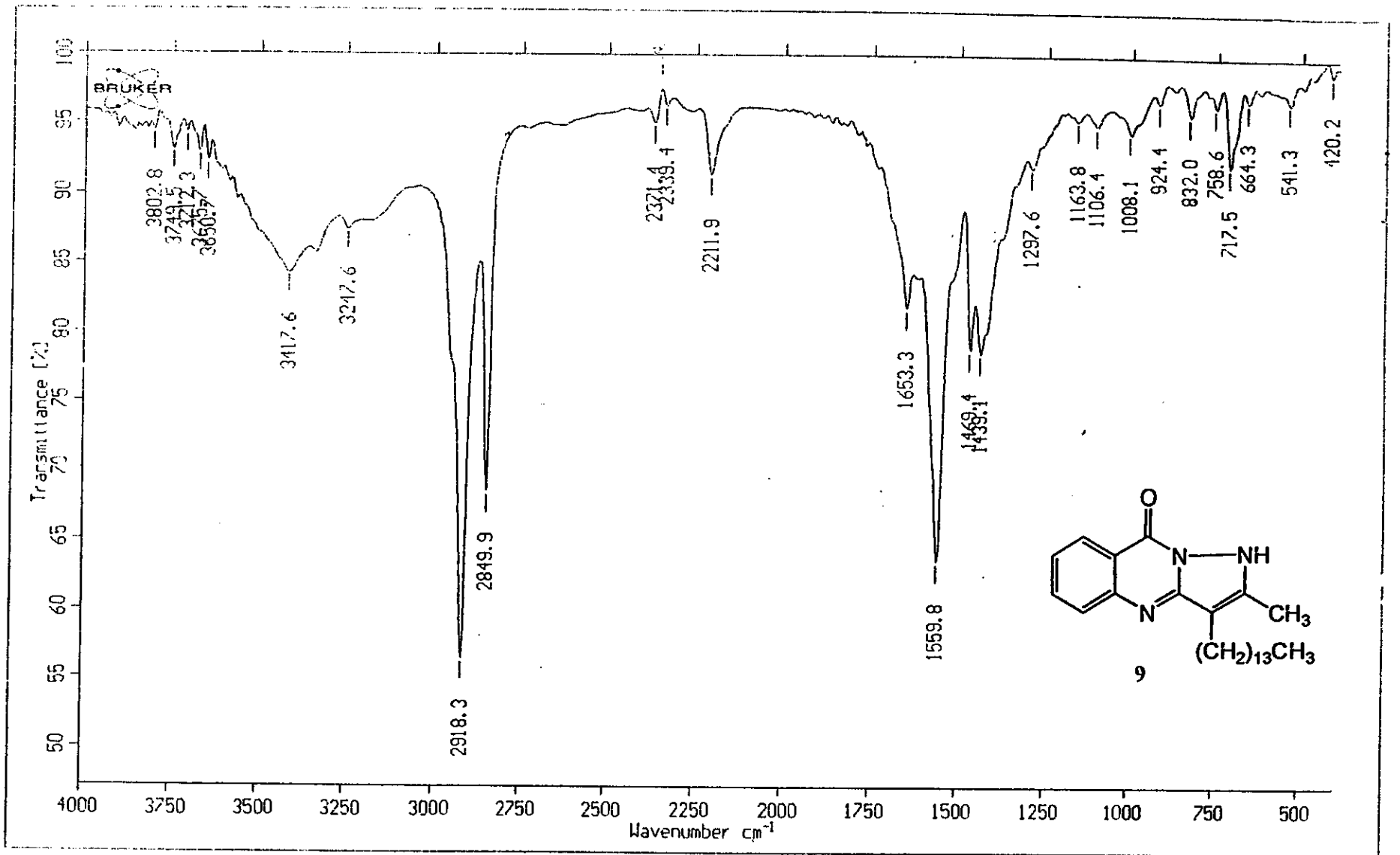


Fig (8) M. AMIN SAMPLE_N.33 20/ 5/2005 3:28: 1

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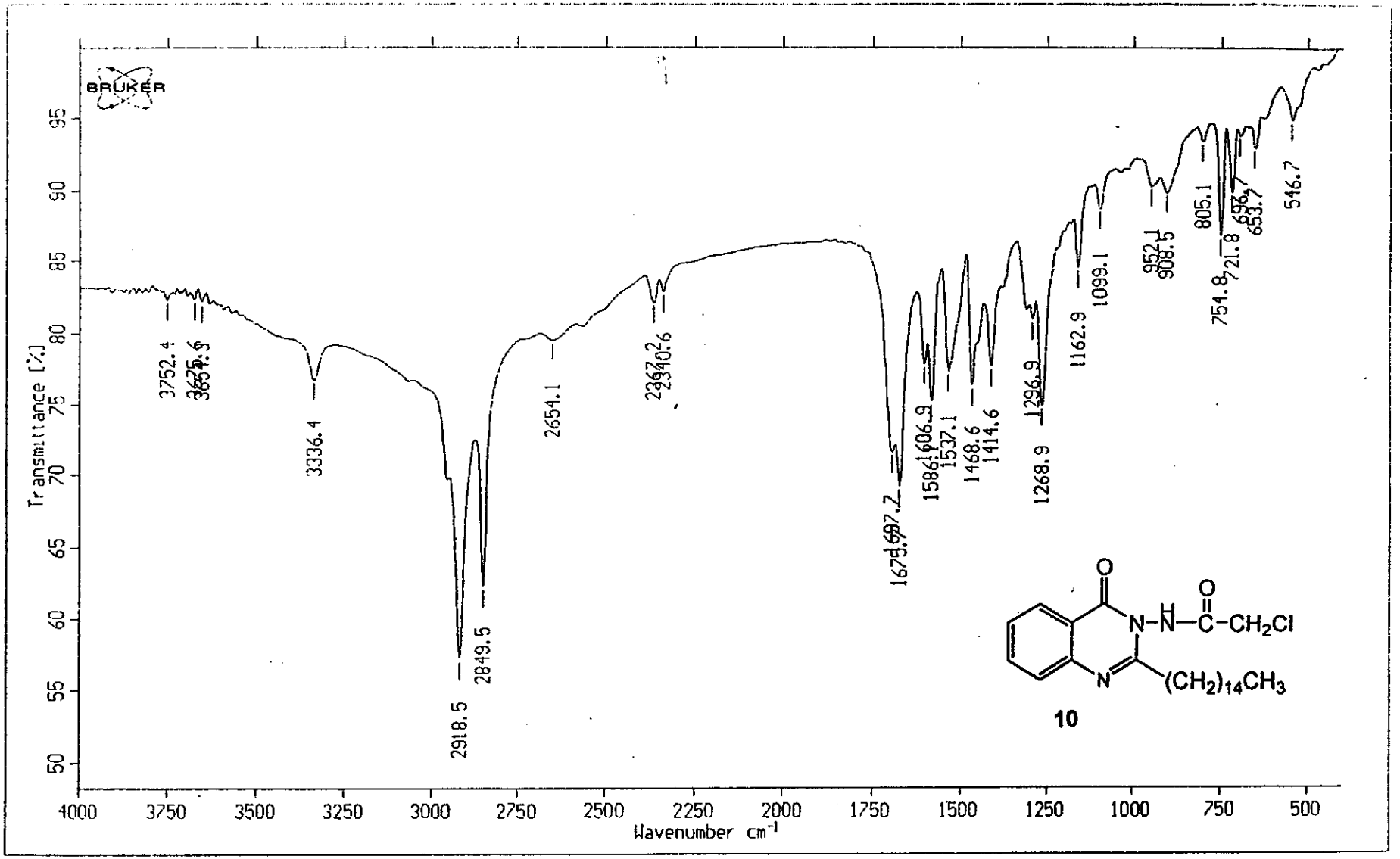


Fig (9) I. AMIN SAMPLE_N. 242 12/ 3/2005 12:38:30

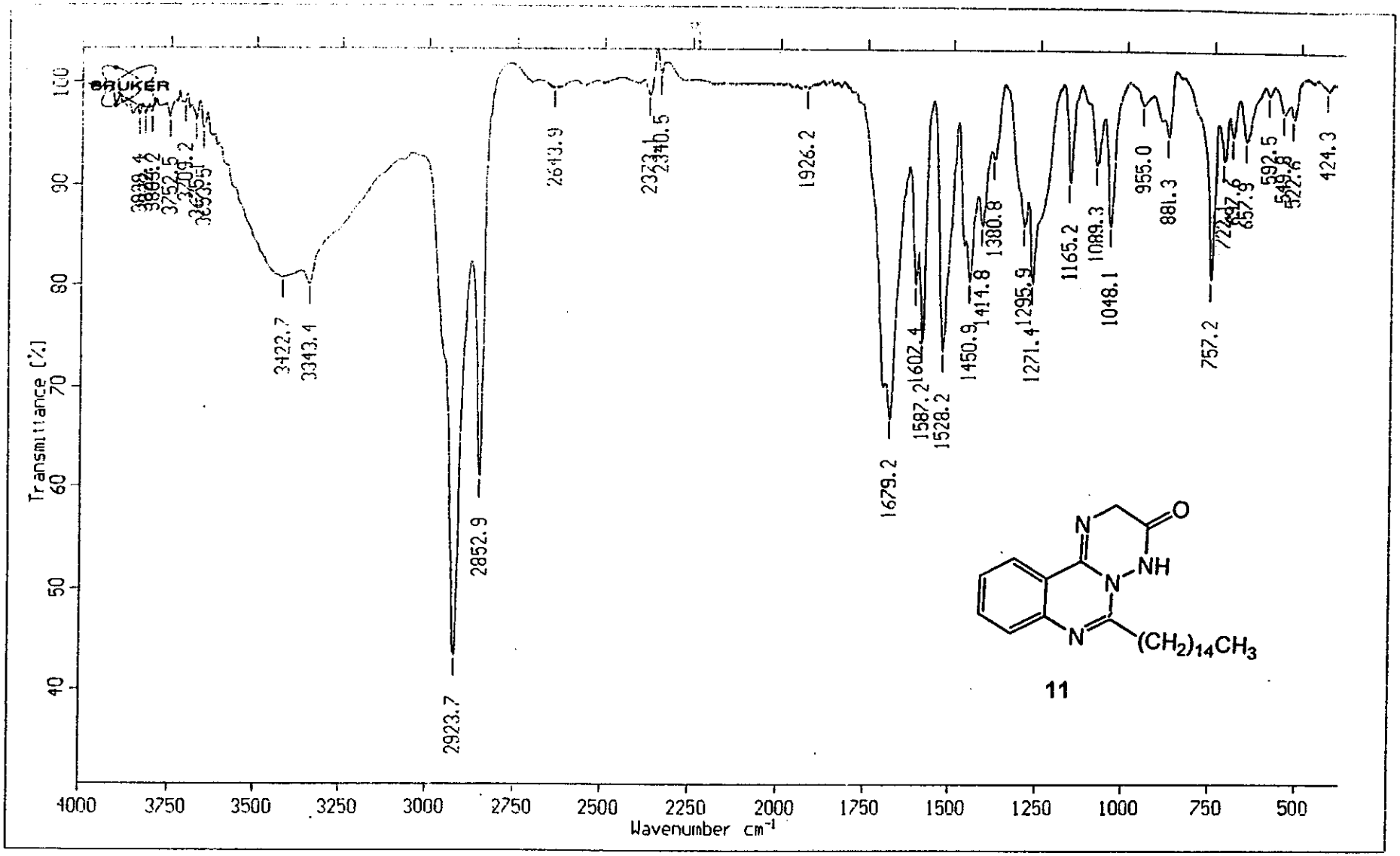


Fig (10)

M. AMIN SAMPLE_N.187 10/ 5/2005 9:40:25

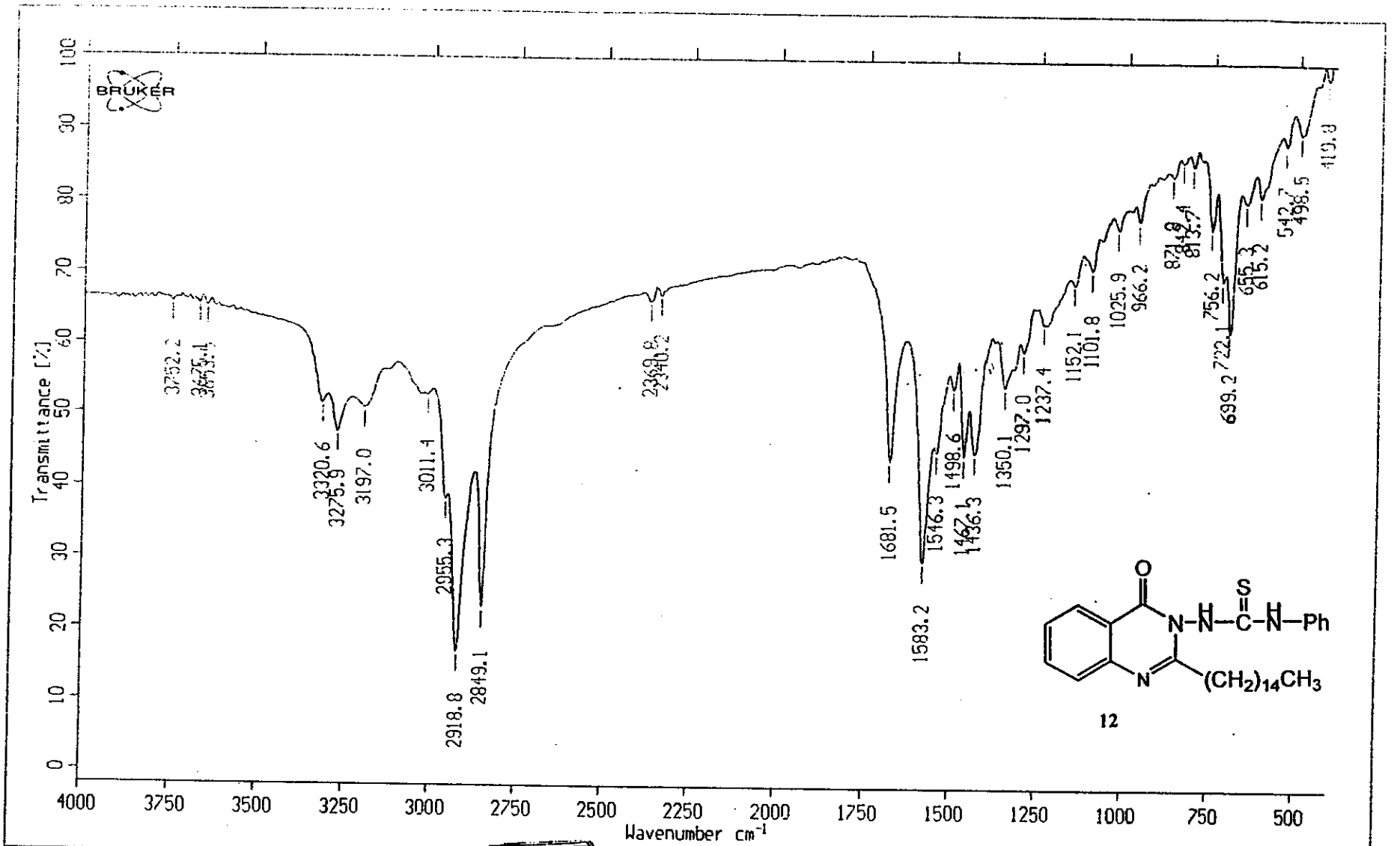


Fig (11)

SAMPLE_N.155 14/10/2004 11:51:24

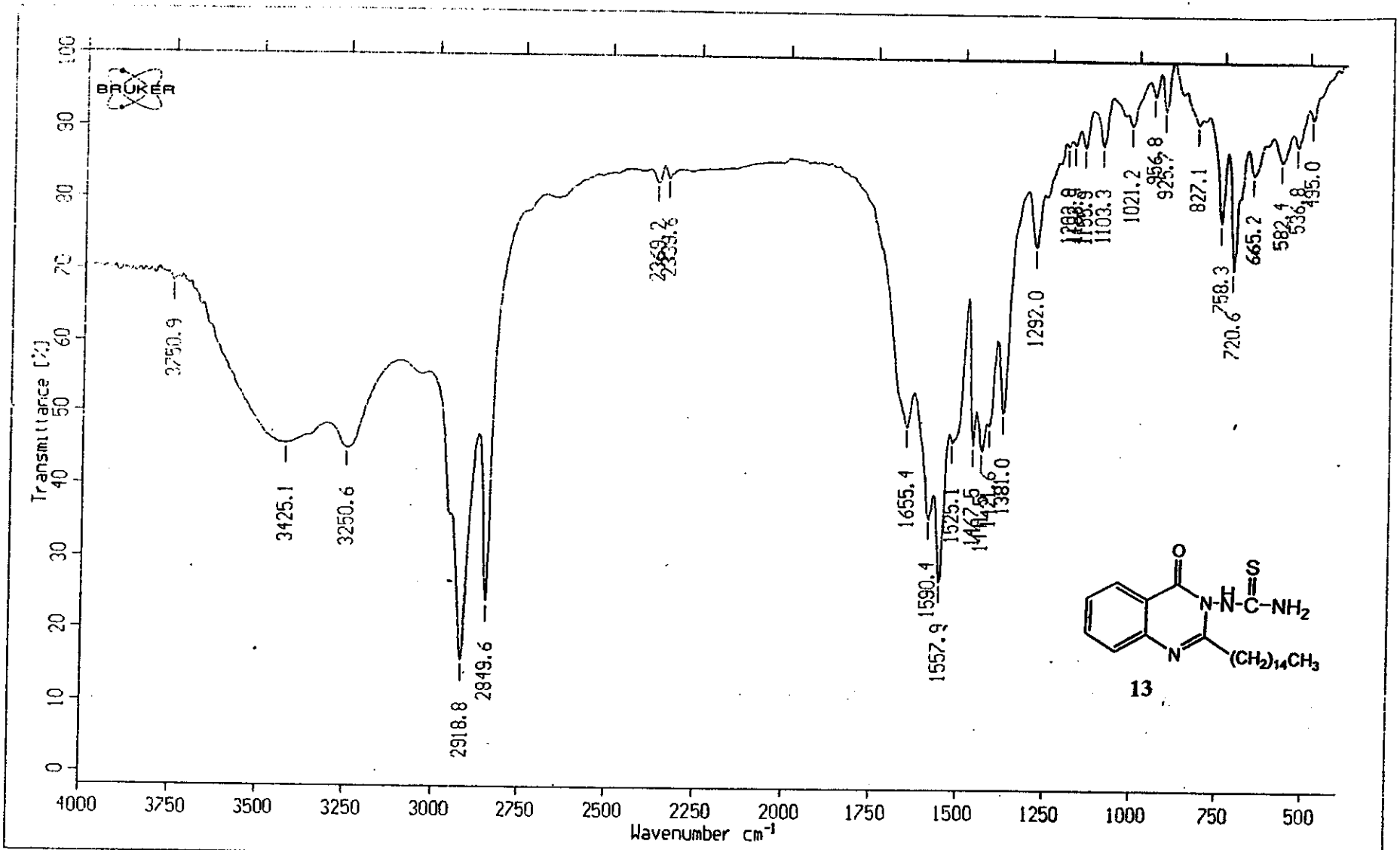
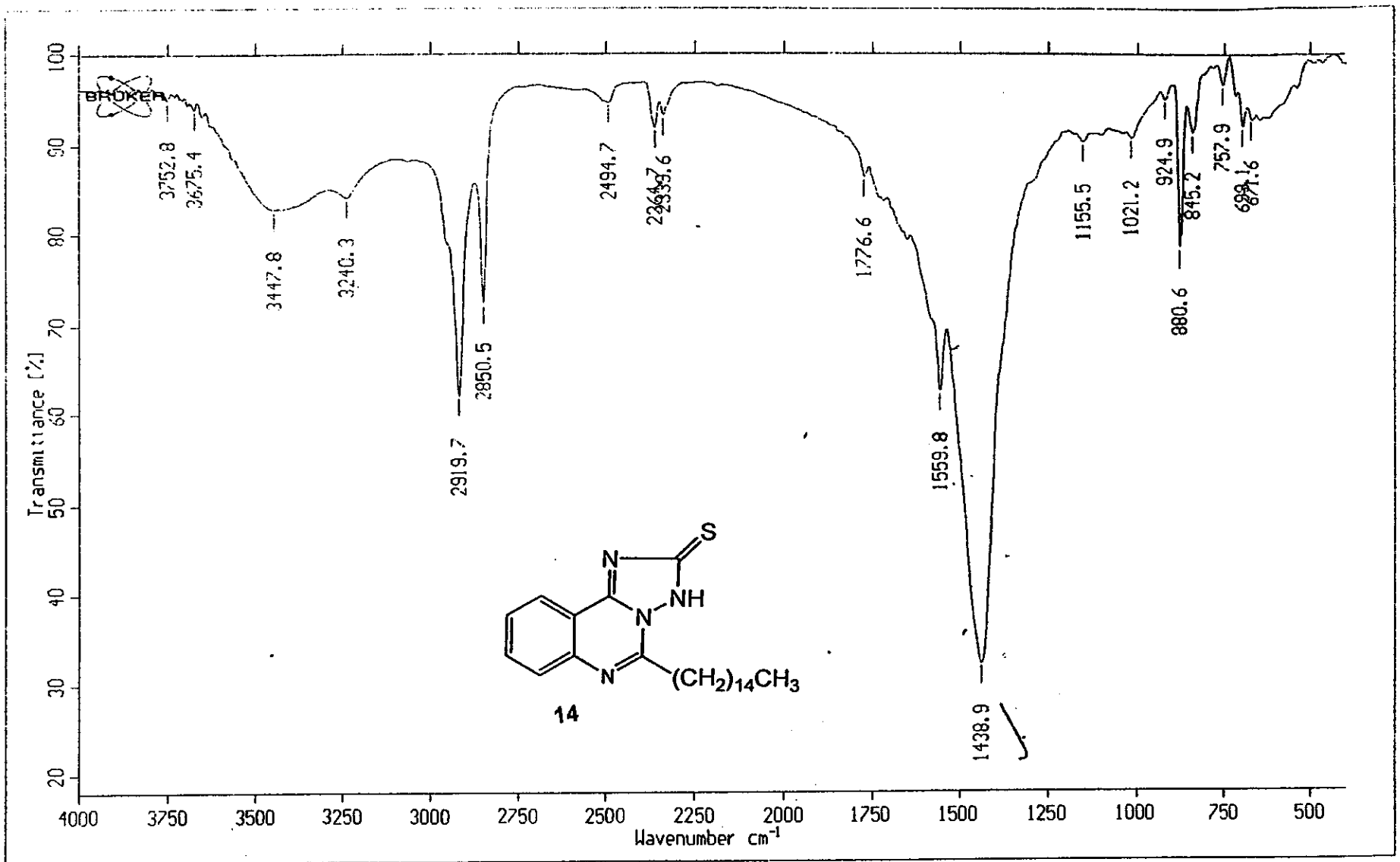


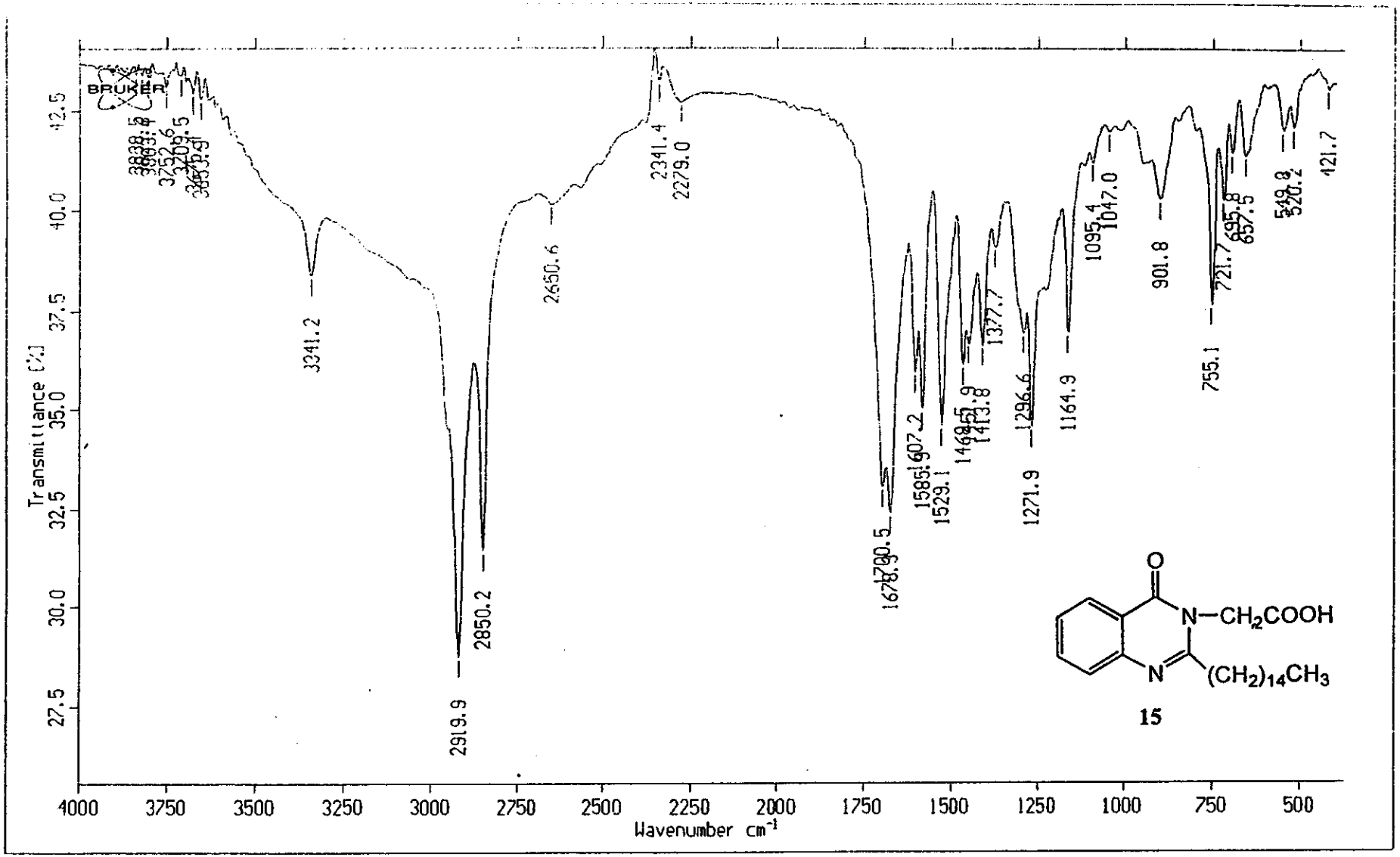
Fig (12) M. AMIN SAMPLE_N.154 14/10/2004 11:47:23



14

Fig (13)

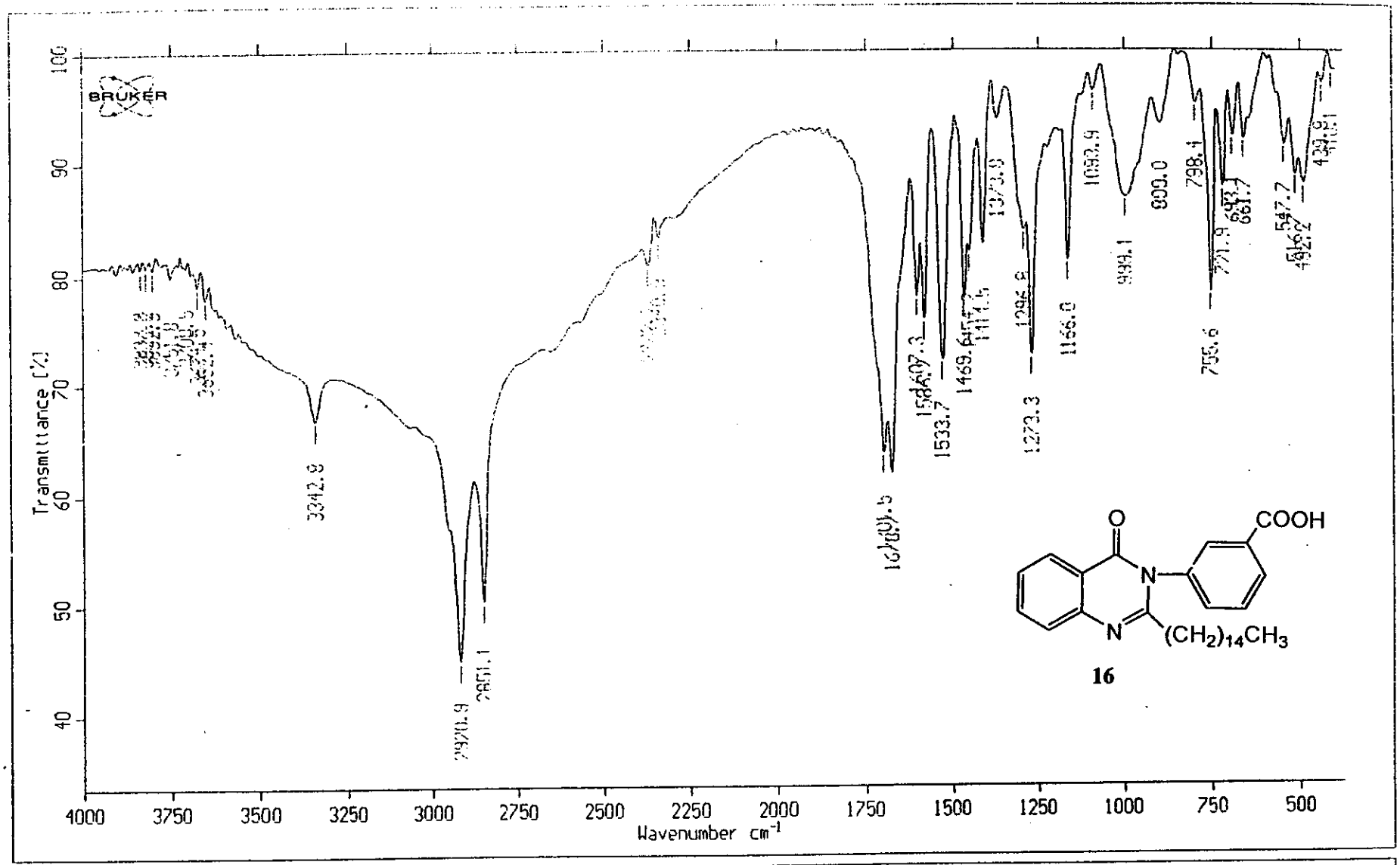
SAMPLE_N. 244 12/ 3/2005 12:52:40



15

Fig (14)

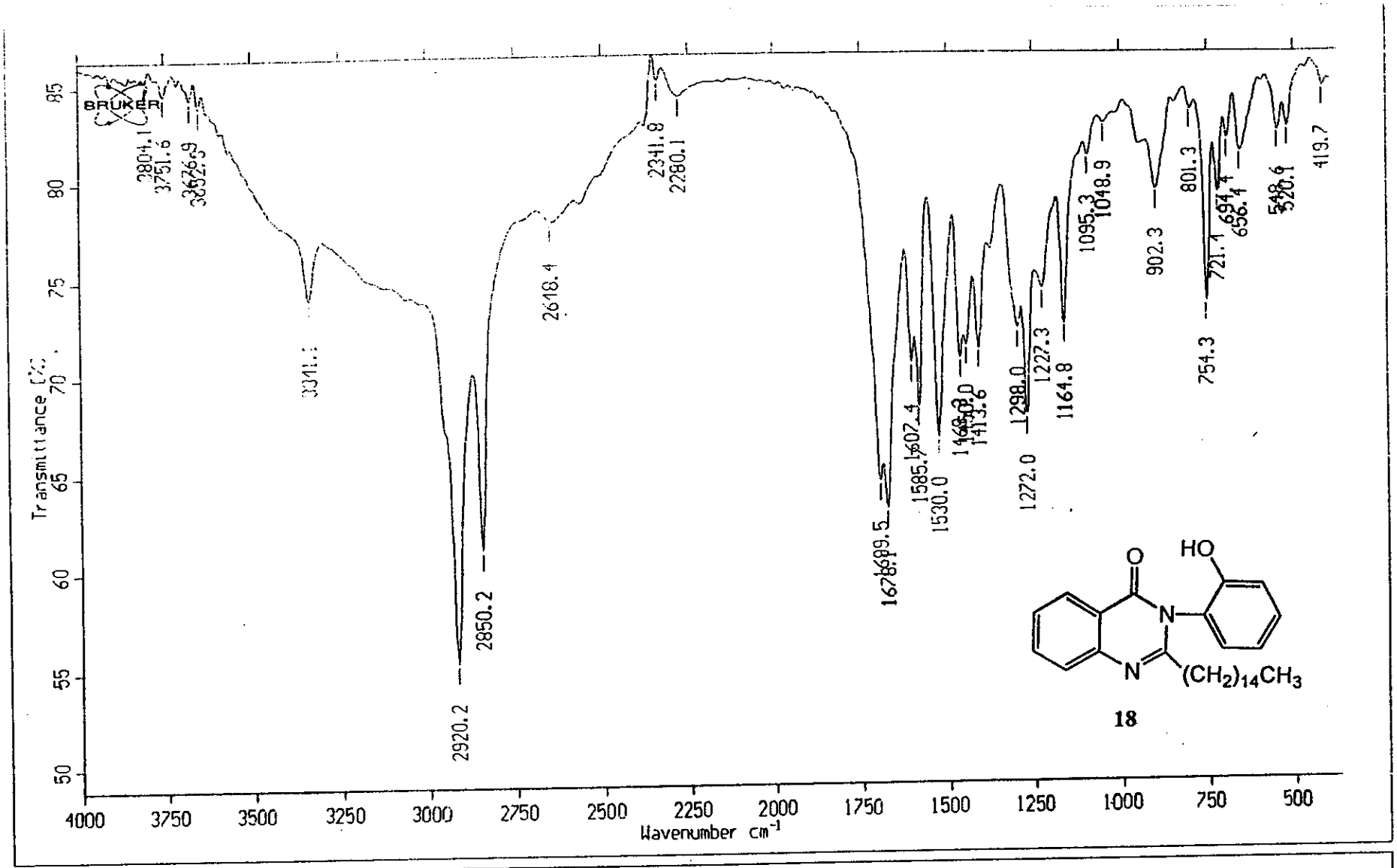
SAMPLE_N.185 10/ 5/2005 9:36:41



16

Fig (15)

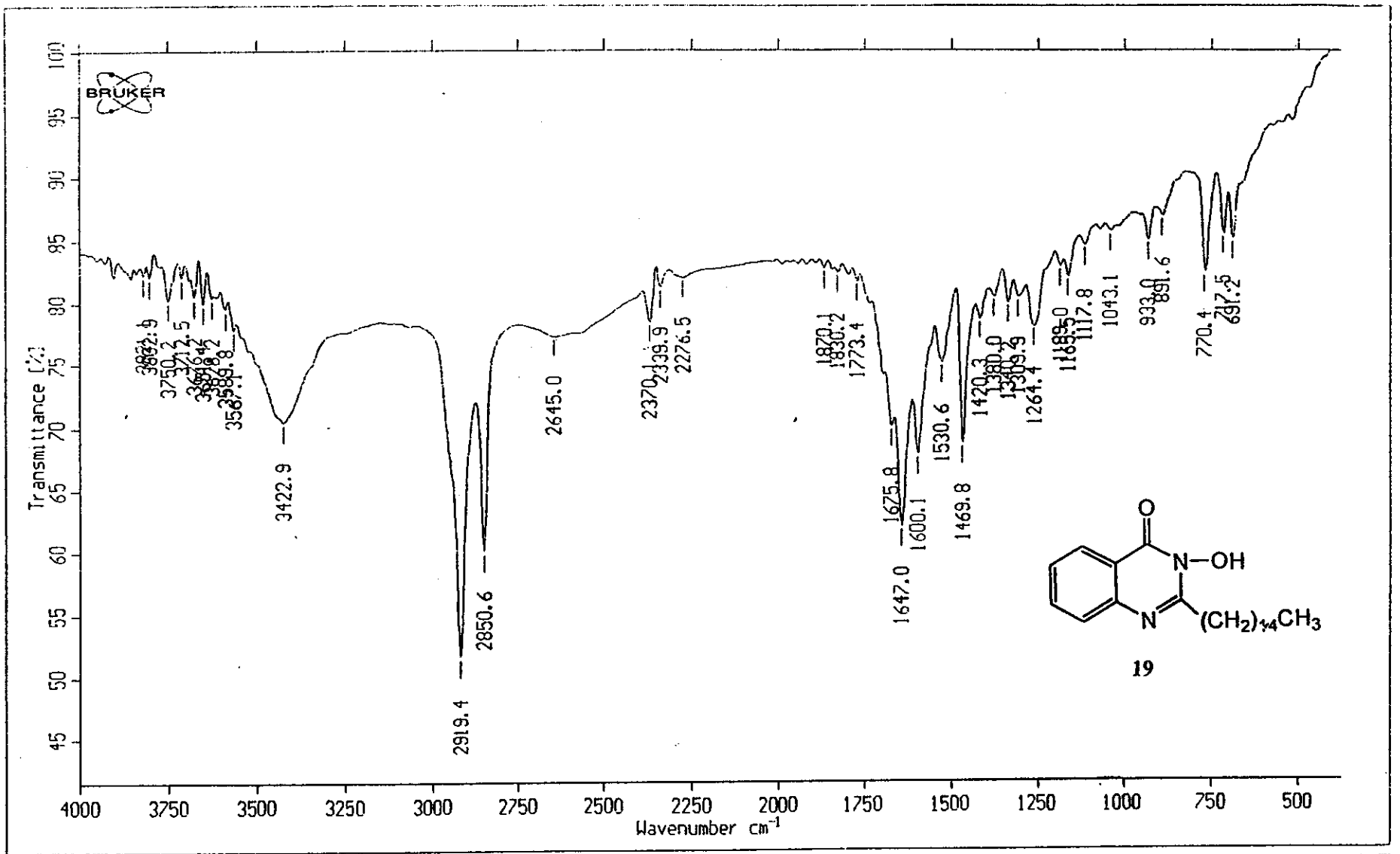
SAMPLE_N.188 10/ 5/2005 9:42:13



18

Fig (16)

SAMPLE_N.186 ·10/ 5/2005 9:37:43



19

Fig (17)

M. AMIN SAMPLE_N.537 24/ 5/2005 16:10: 5

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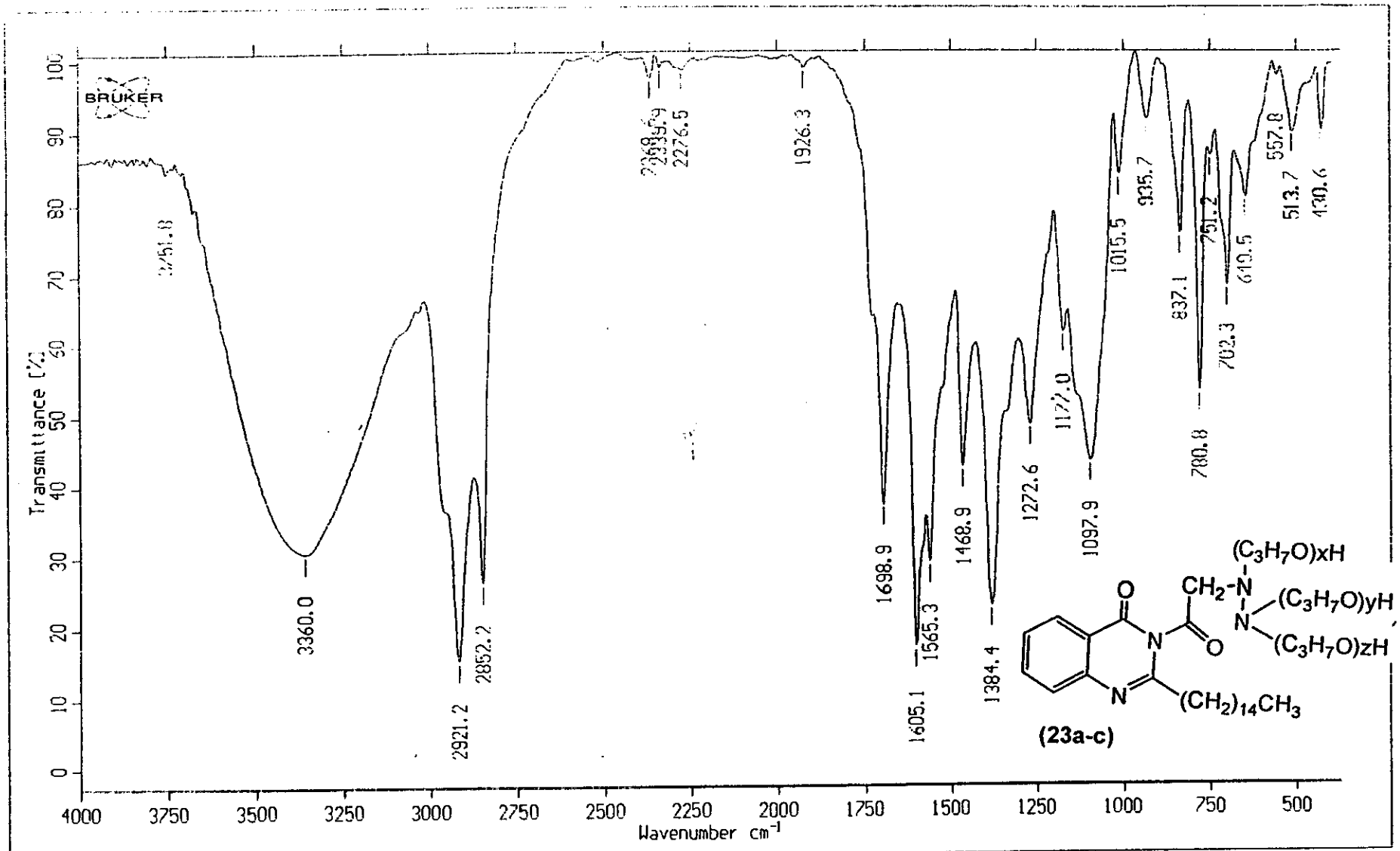

BRUKER
Transmittance [%]
Wavenumber cm-1
3360.0
2921.2
2852.2
1698.9
1605.1
1565.3
1468.9
1384.4
1272.6
1097.9
1015.5
935.7
837.1
780.8
702.3
619.5
557.8
513.7
(C3H7O)xH
(C3H7O)yH
(C3H7O)zH
CH2-N
(CH2)14CH3
(23a-c)
SAMPLE_N. 913 27/ 5/2005 14:31:36

Fig (18)

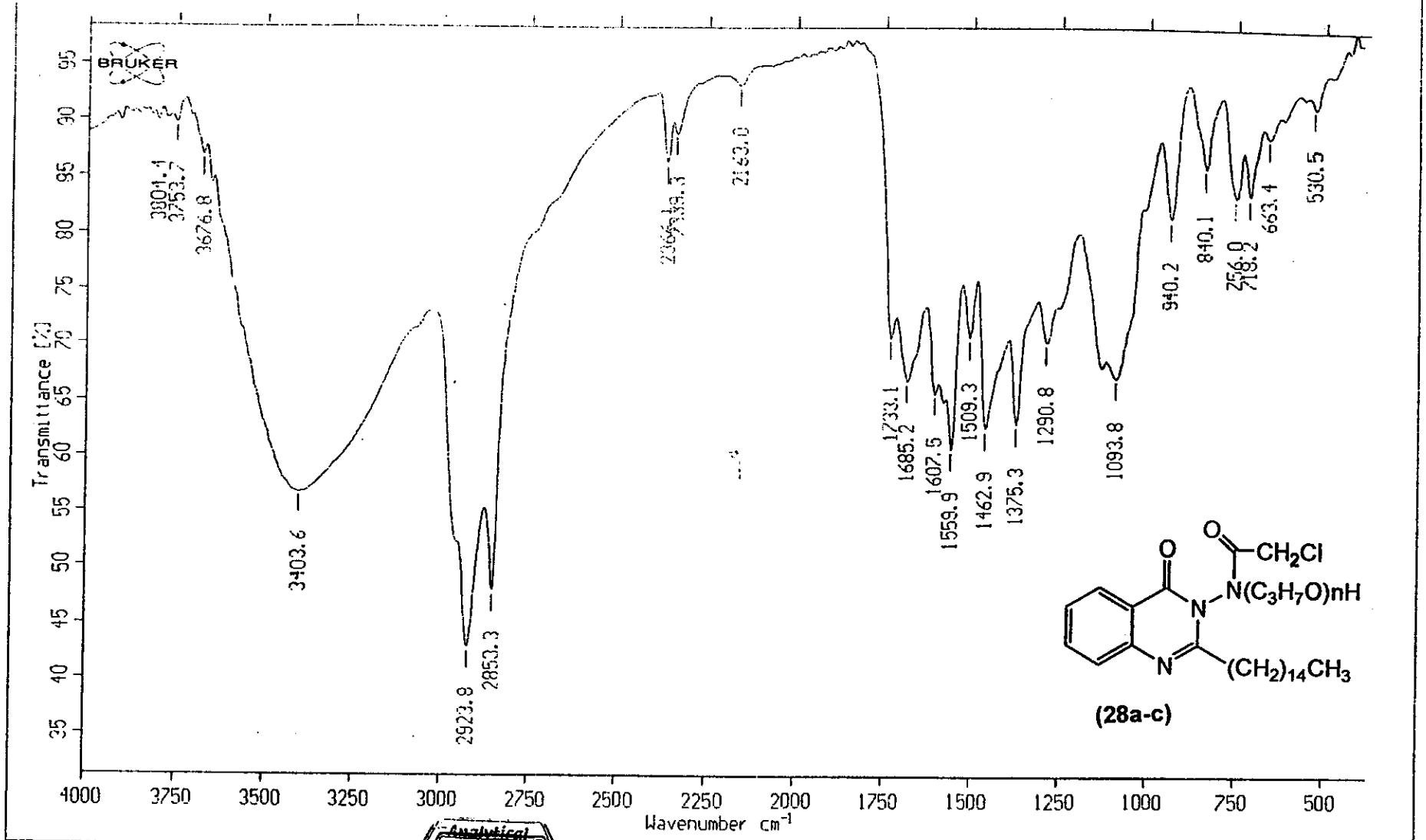


Fig (19)

SAMPLE_N.570 21/ 5/2005 21:35: 6

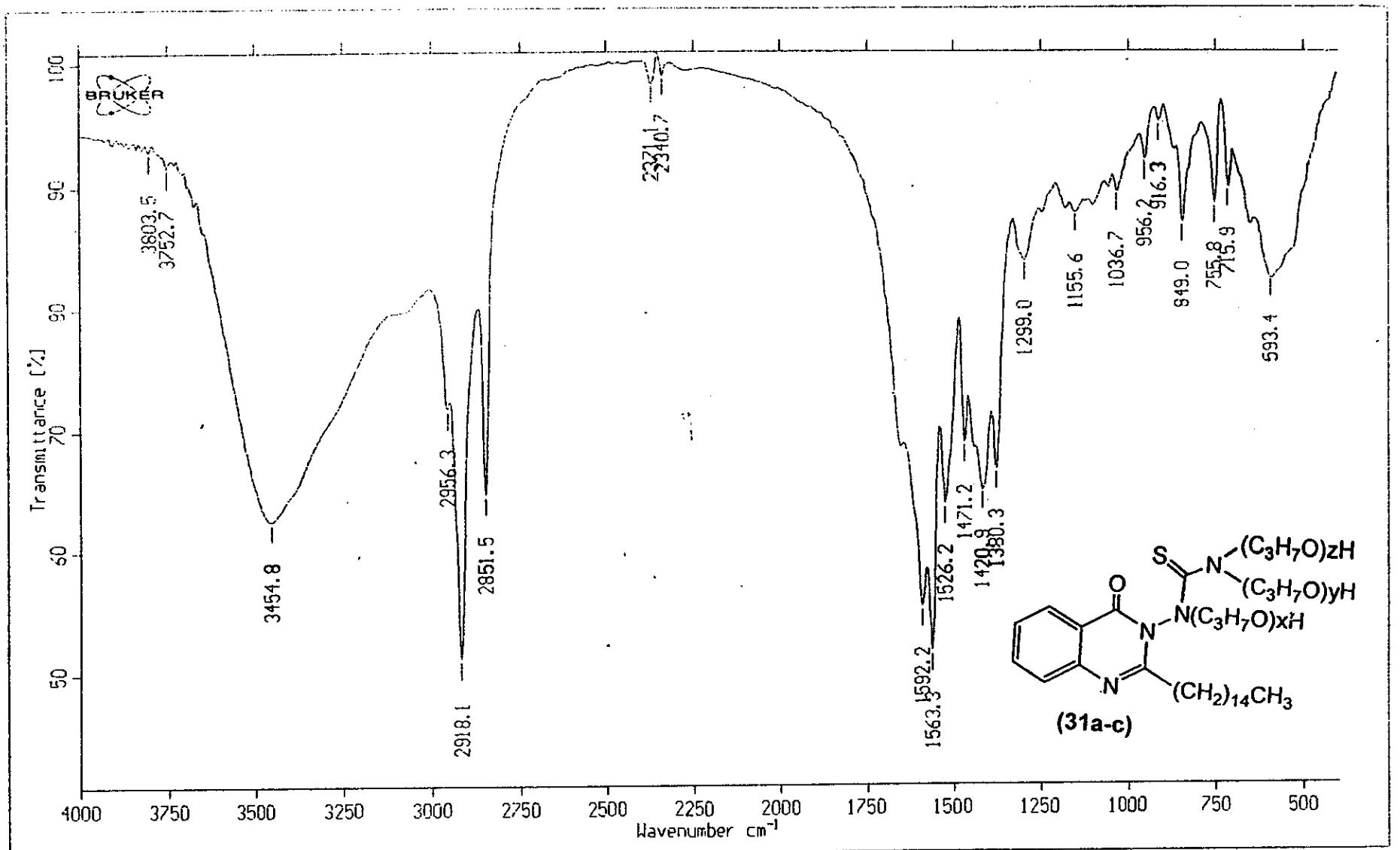

BRUKER
Transmittance [%]
Wavenumber cm-1
3803.5
3752.7
3454.8
2956.3
2918.1
2851.5
2331.7
2340.7
1592.2
1563.5
1526.2
1471.2
1420.9
1380.3
1299.0
1155.6
1036.7
956.2
916.3
849.0
755.8
715.9
593.4
(C3H7O)zH
(C3H7O)yH
N(C3H7O)xH
(CH2)14CH3
(31a-c)
M. AMI
SAMPLE_N. 241
12/ 3/2005 12:34:51

Fig (20)

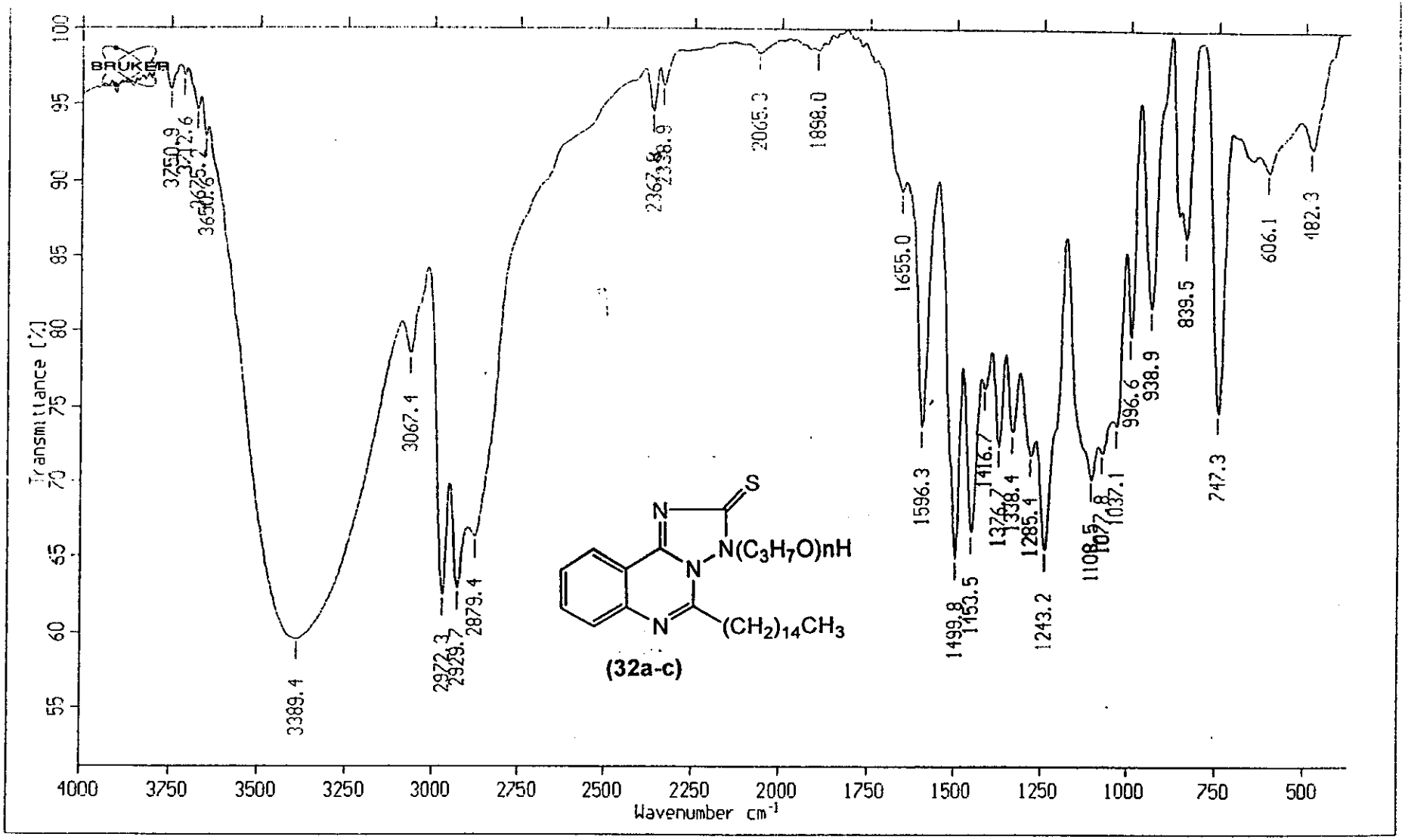


Fig (21)

SAMPLE_N. 618 25/ 5/2005 2:54:42

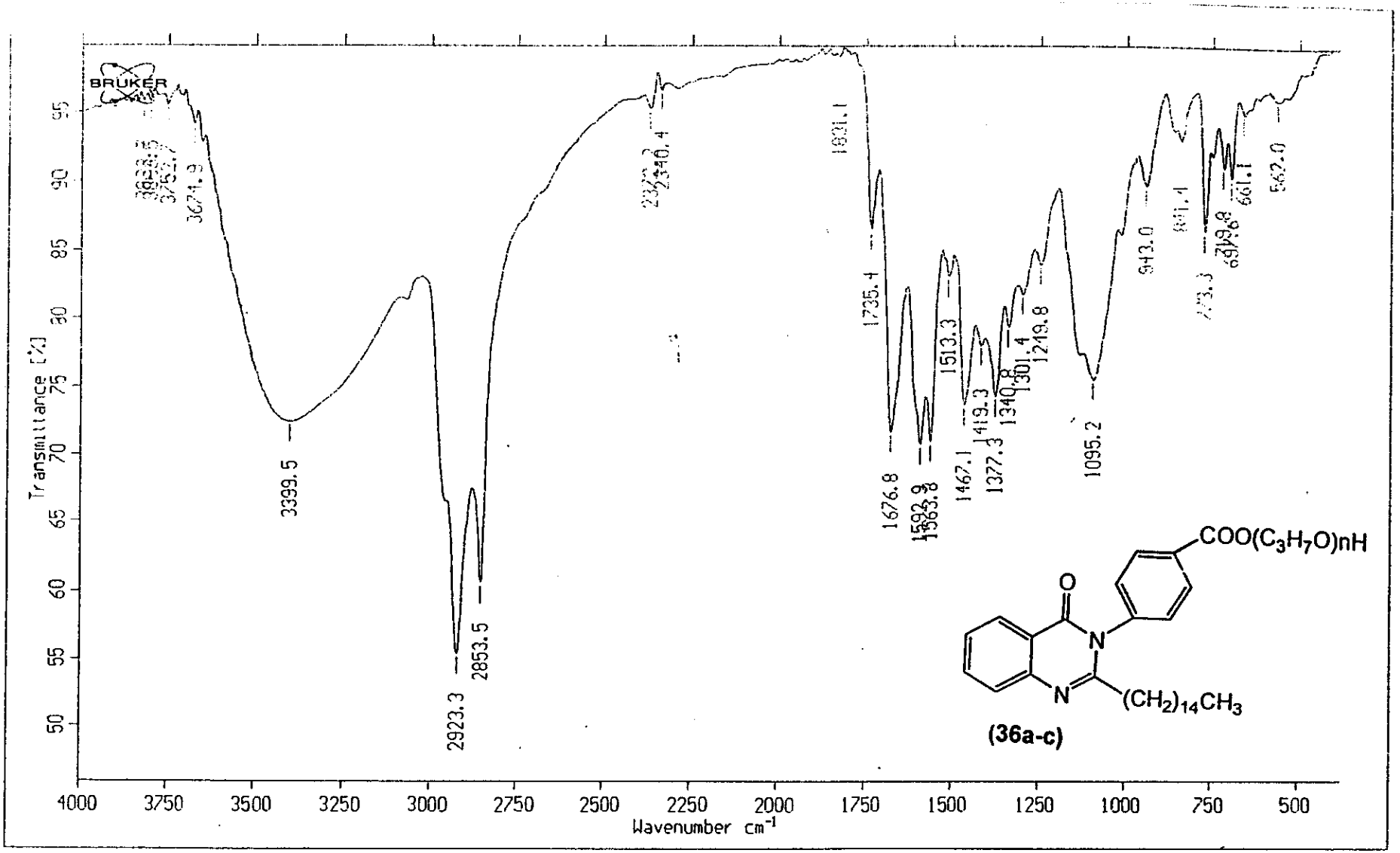
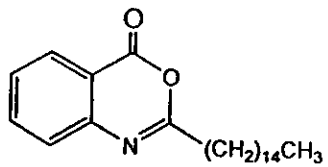


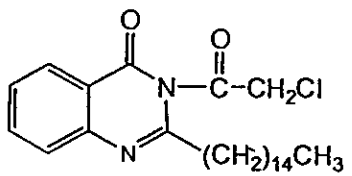
Fig (22) M. AMIN SAMPLE_N.541 24/ 5/2005 16:18:


O
O
N
$(CH_2)_{14}CH_3$

2

2.580
2.495
2.361
2.332
2.311
2.286
1.718
1.721
1.697
1.011
1.228
3.327
2.676
2.650
2.626
1.159
1.162
0.849
0.825
8.104
8.079
7.930
7.900
7.874
7.583
7.562
7.564
18
16
14
12
10
8
6
4
2
ppm
20.21
16.71
50.82
7.30

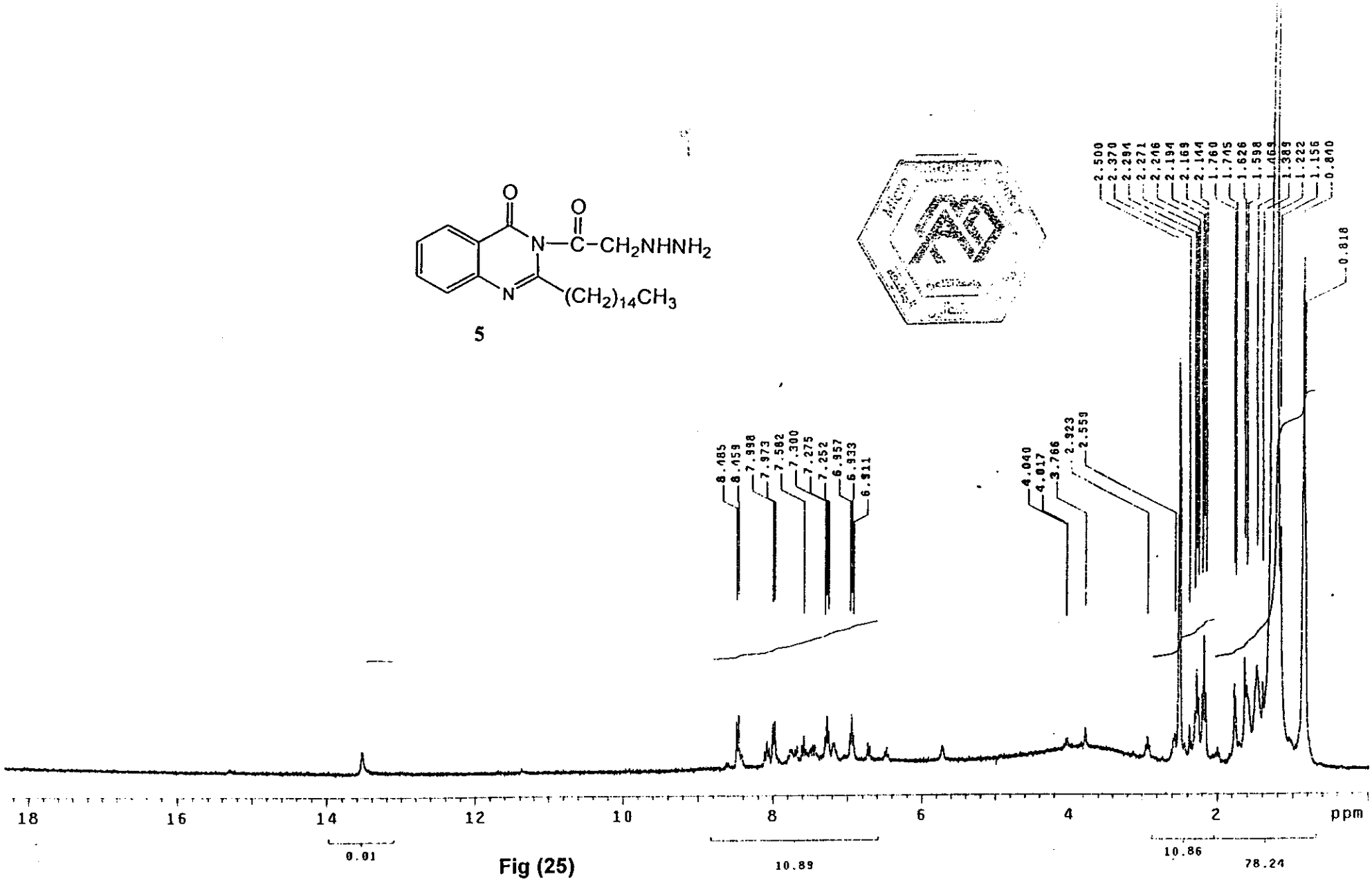
Fig (23)


O
O
N
C
CH2Cl
N
(CH2)14CH3

4

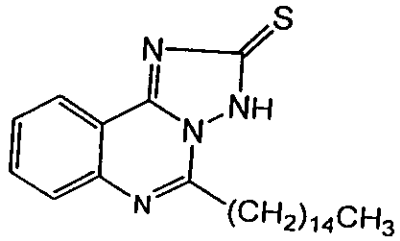
11.170
8.546
8.518
7.976
7.951
7.552
7.526
7.500
7.475
7.112
7.086
7.062
2.497
2.361
2.337
2.313
2.267
2.175
2.150
2.127
2.072
1.460
1.198
1.139
0.819
0.799
18
16
14
12
10
8
6
4
2
ppm
0.66
9.03
8.34
81.97

Fig (24)


O
O
N
C
CH2NHNH2
N
(CH2)14CH3
8.485
8.459
7.998
7.973
7.582
7.300
7.275
7.252
6.957
6.933
6.911
4.040
4.017
3.766
2.923
2.559
2.500
2.370
2.294
2.271
2.246
2.194
2.169
2.144
1.760
1.745
1.626
1.598
1.469
1.389
1.222
1.156
0.840
0.818
18
16
14
12
10
8
6
4
2
ppm
0.01
10.89
10.86
78.24

5

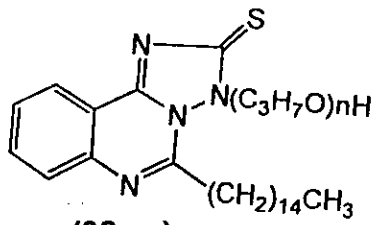
Fig (25)


S
N
NH
N
N
(CH2)14CH3

14

11.108
8.515
8.485
8.088
8.061
7.982
7.955
7.791
7.768
7.740
7.601
7.573
7.477
7.454
7.122
3.354
2.621
2.595
2.569
2.501
2.496
2.481
2.386
2.362
2.339
2.173
1.715
1.692
1.612
1.286
1.220
1.163
0.862
0.844
0.820
0.772
16
14
12
10
8
6
4
2
ppm
1.73
8.68
14.61
10.90
64.08

Fig (27)


N
S
N
N(C3H7O)nH
N
(CH2)14CH3

(32a-c)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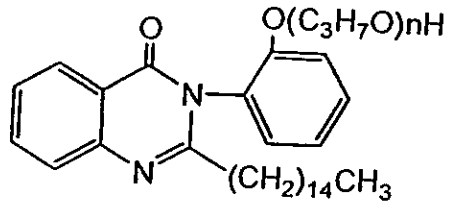
6.918
3.793
3.774
3.602
3.386
3.175
3.030
3.013
2.997
2.500
1.226
1.202
1.190
1.168
1.148
1.127
1.107
1.002
0.989
0.983
0.969
14
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
12.02
7.01
47.57
3.30
30.10

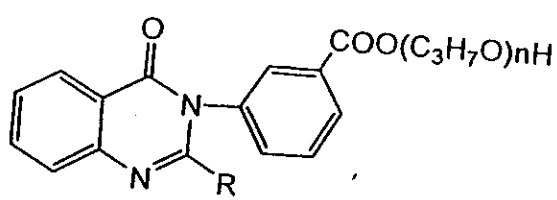
Fig (28)


O
O(C3H7O)nH
N
N
(CH2)14CH3

(35a-c)

8.002
7.977
7.256
3.713
3.566
3.546
3.412
3.381
3.360
3.321
3.310
3.290
3.266
3.250
3.246
3.231
3.226
3.214
3.203
3.188
3.184
2.502
2.496
1.225
1.155
1.133
1.113
1.094
1.055
1.025
1.001
0.983
0.980
0.859
0.841
0.819
10
9
8
7
6
5
4
3
2
1
ppm
9.01
33.10
6.29
51.60

Fig (29)


O
N
N
R
COO(C3H7O)nH

(34a-c)


Analytical
Micro
Center
التحاليل
الدقيقة

3.326
2.506
2.500
2.494
2.488
2.230
1.185
1.164
1.158
1.057
1.033
1.026
1.011
1.005
0.984
0.862
0.851
0.836
0.832
0.823
0.801
2.513
11
10
9
8
7
6
5
4
3
2
1
ppm
4.57
37.00
5.80
52.63

Fig (30)

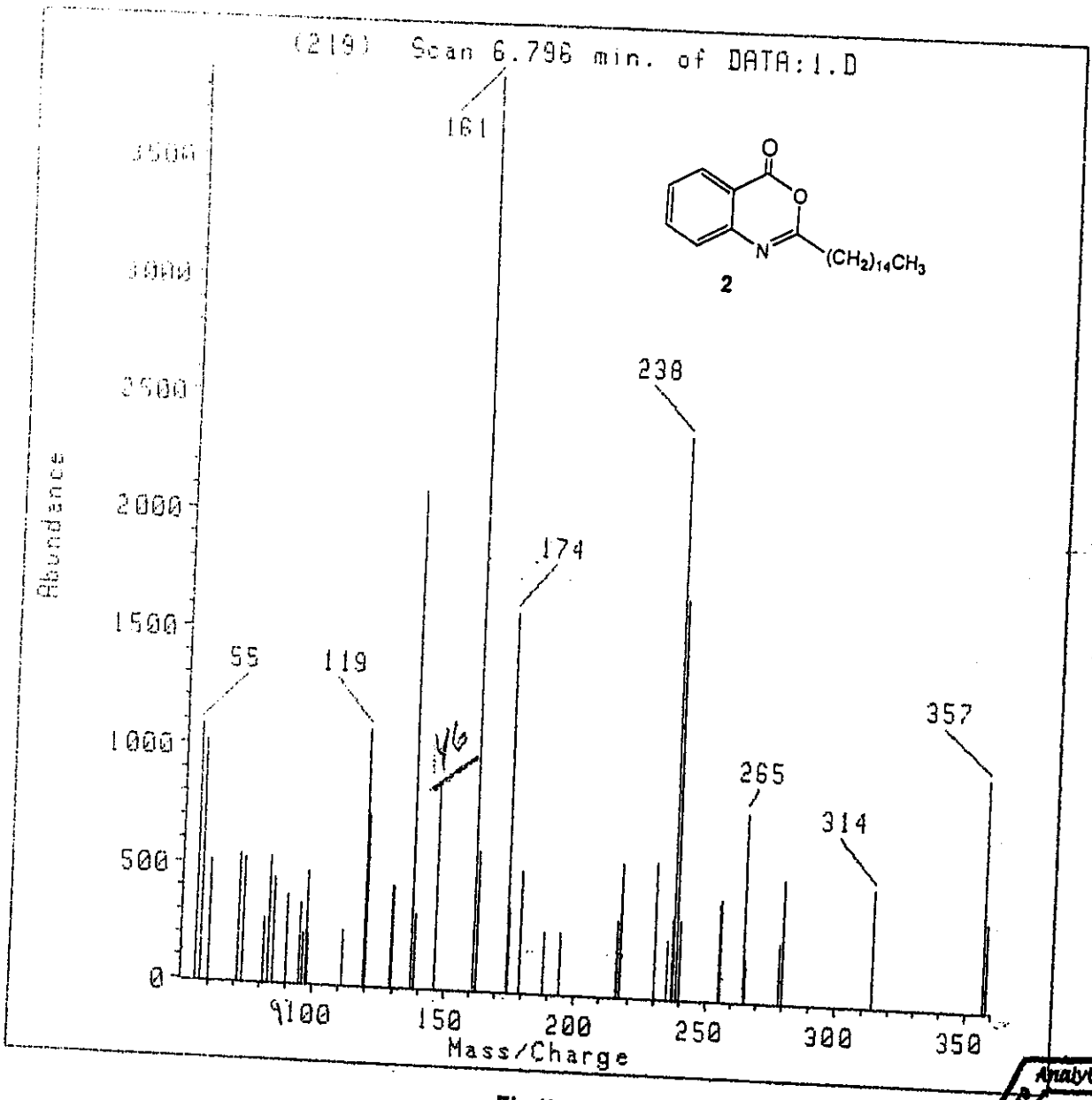

(219) Scan 6.796 min. of DATA:1.D
161
238
174
55
119
46
357
265
314
Abundance
Mass/Charge
2

Fig (31)

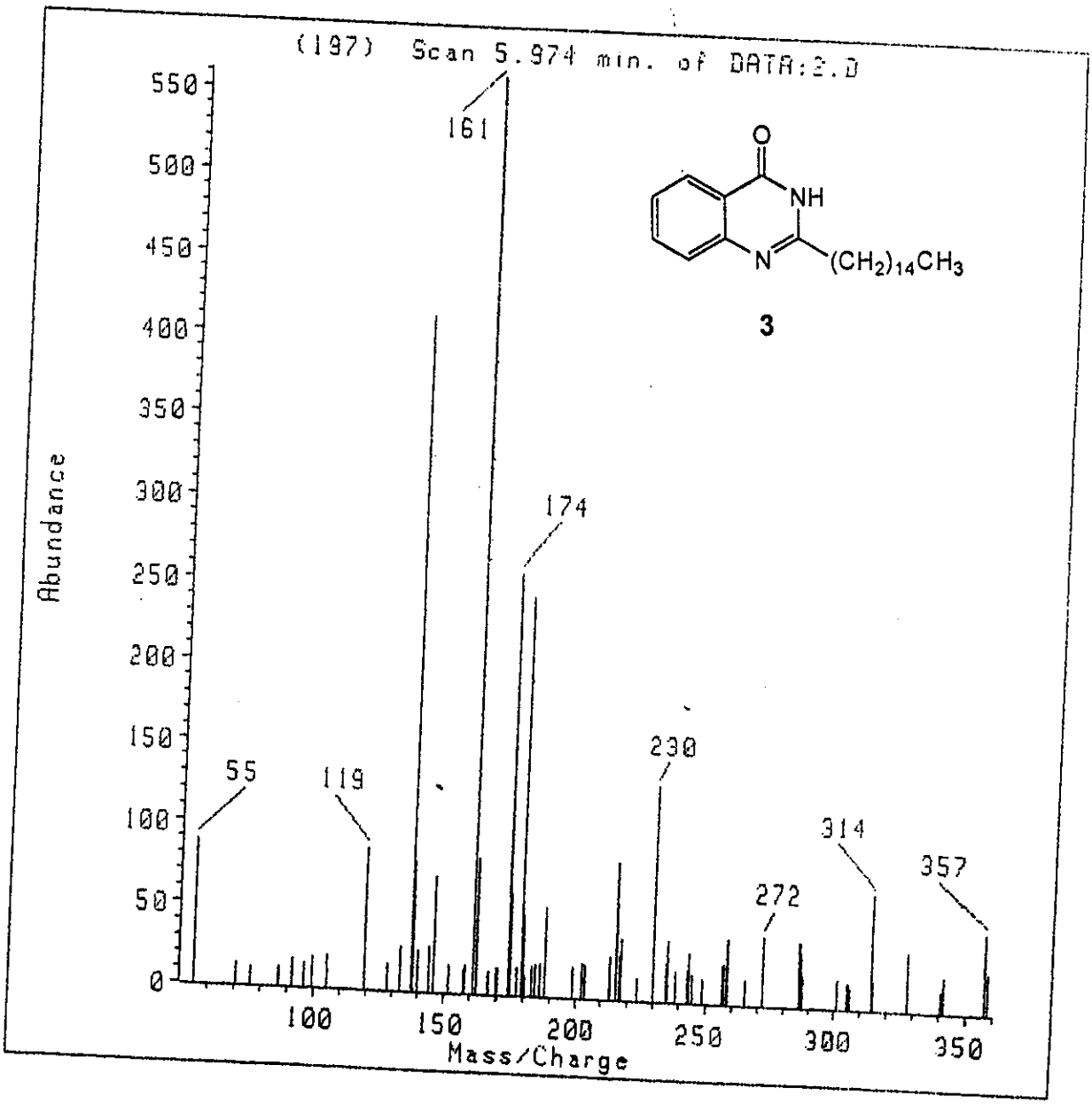

(197) Scan 5.974 min. of DATA:2.D
Abundance
Mass/Charge
161
174
55
119
230
314
357
272
O
NH
N
$(CH_2)_{14}CH_3$
3

Fig (32)

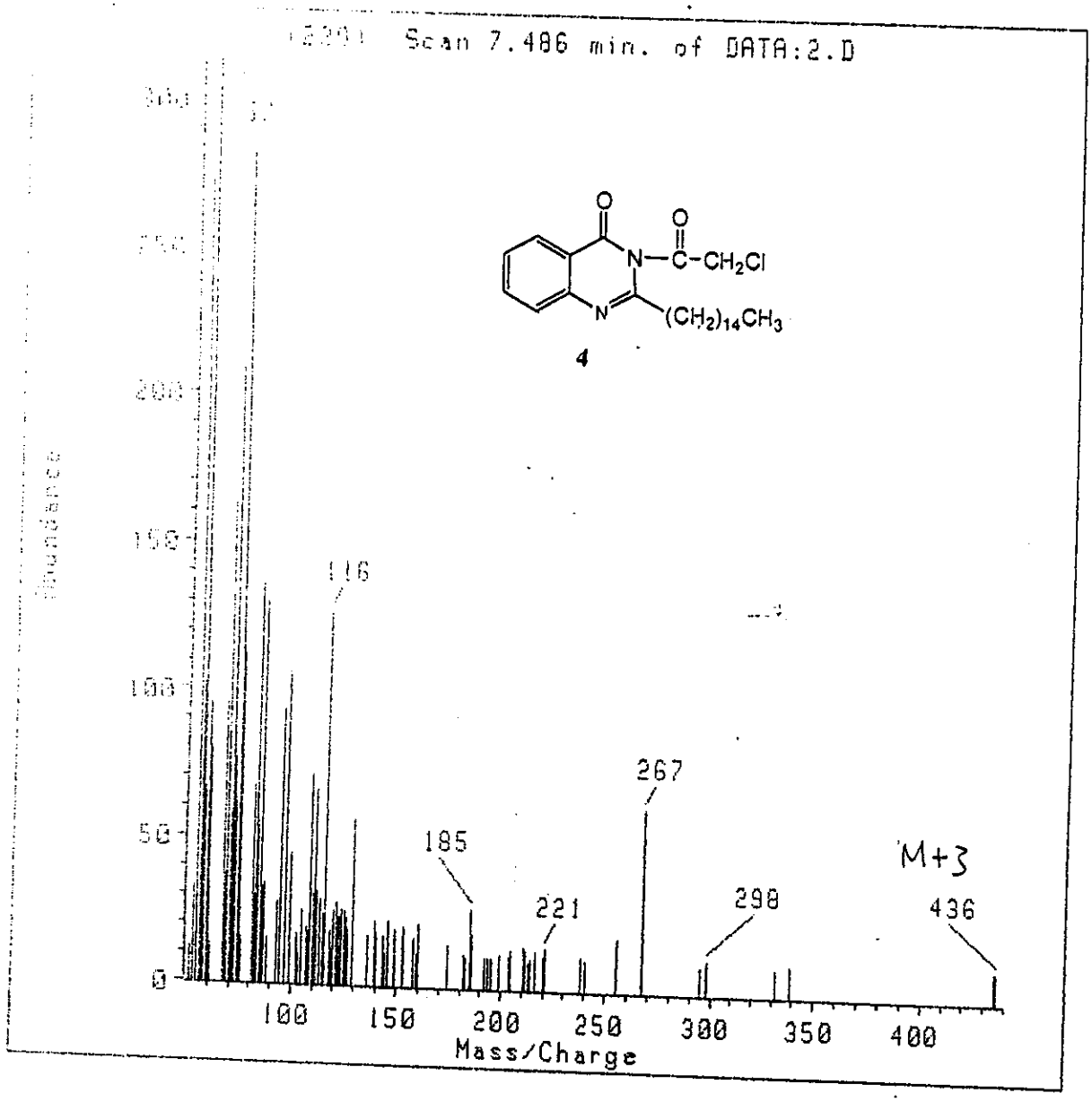
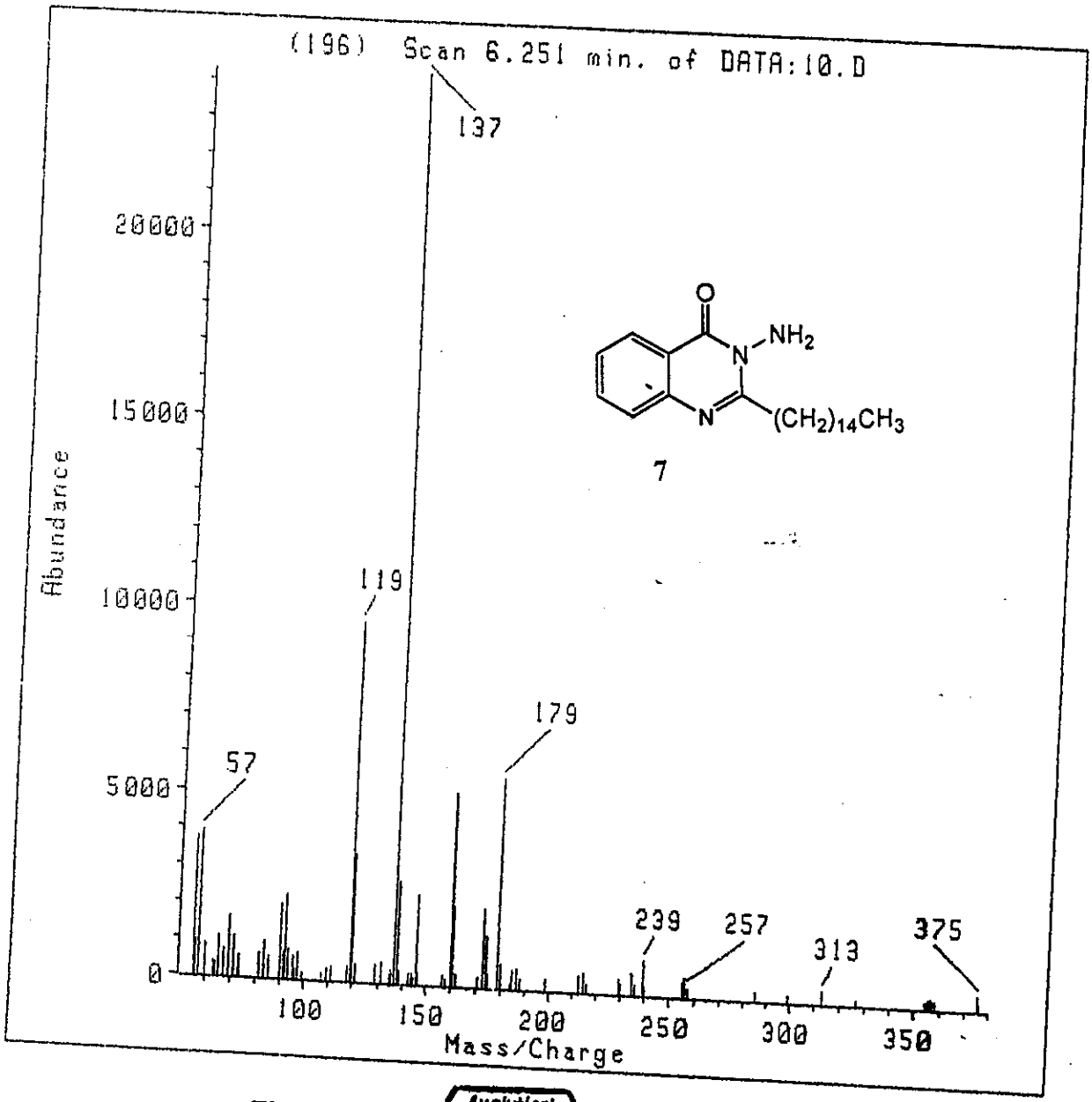
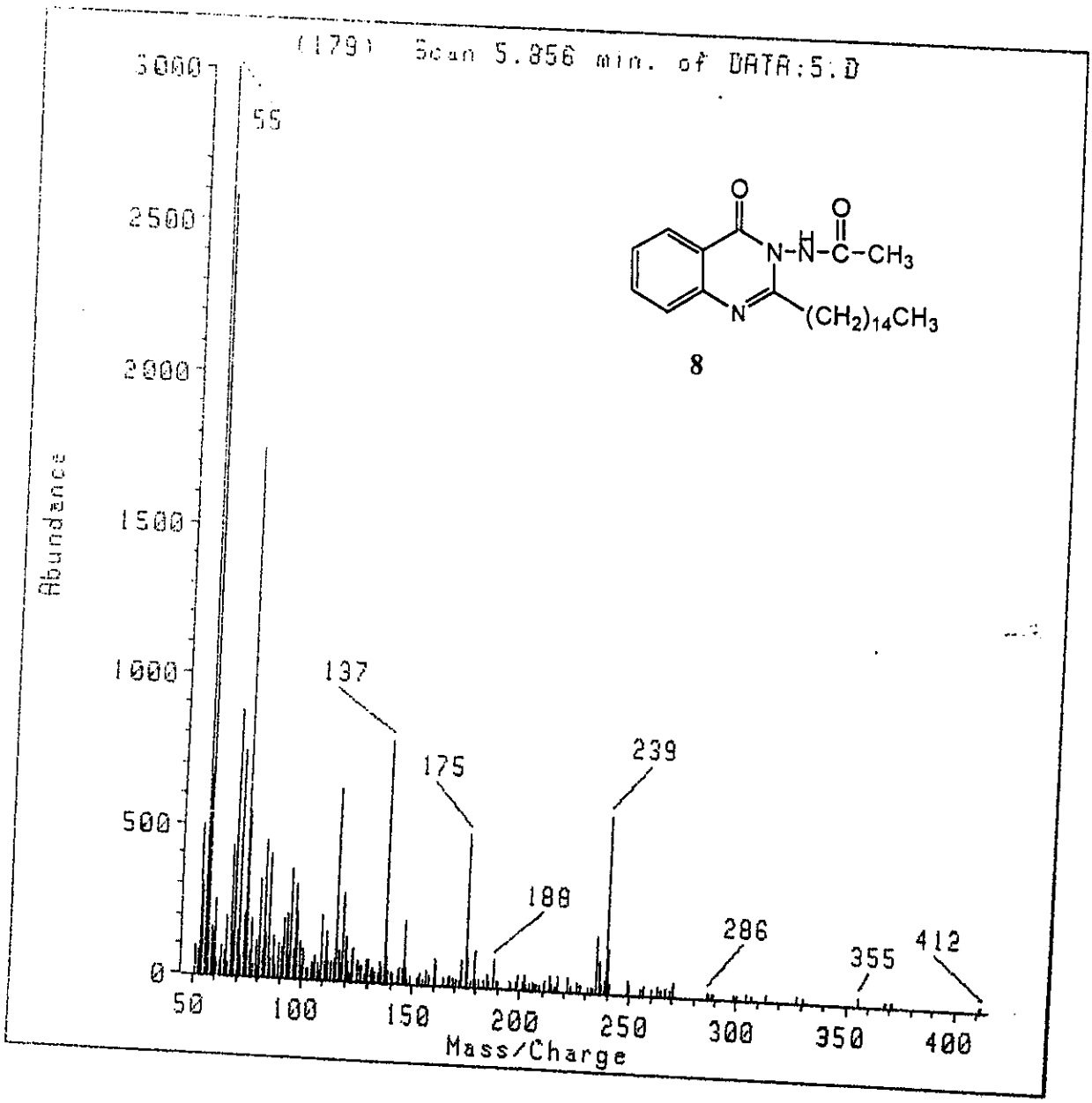

Scan 7.486 min. of DATA:2.D
Abundance
116
185
221
267
298
436
M+3
100
150
200
250
300
350
400
Mass/Charge
4

Fig (33)


(196) Scan 6.251 min. of DATA:10.D
137
119
179
57
239
257
313
375
20000
15000
10000
5000
0
Abundance
100
150
200
250
300
350
Mass/Charge
O
N
NH2
N
(CH2)14CH3

7

Fig (34)


(179) Scan 5.856 min. of DATA:5.D
Abundance
3000
2500
2000
1500
1000
500
0
55
137
175
188
239
286
355
412
50
100
150
200
250
300
350
400
Mass/Charge
O
O
N-N-C-CH3
H
N
(CH2)14CH3

8

Fig (35)

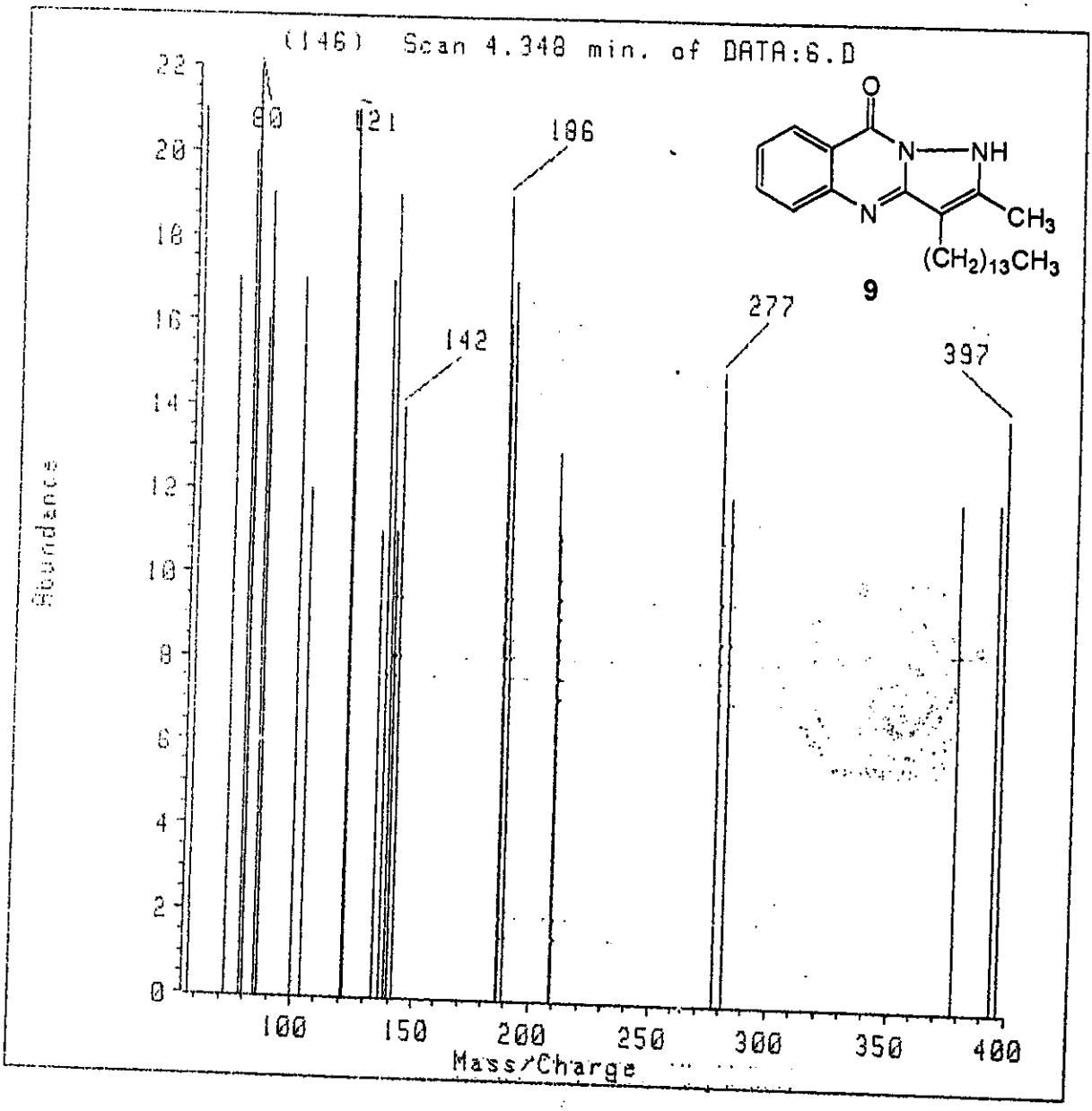
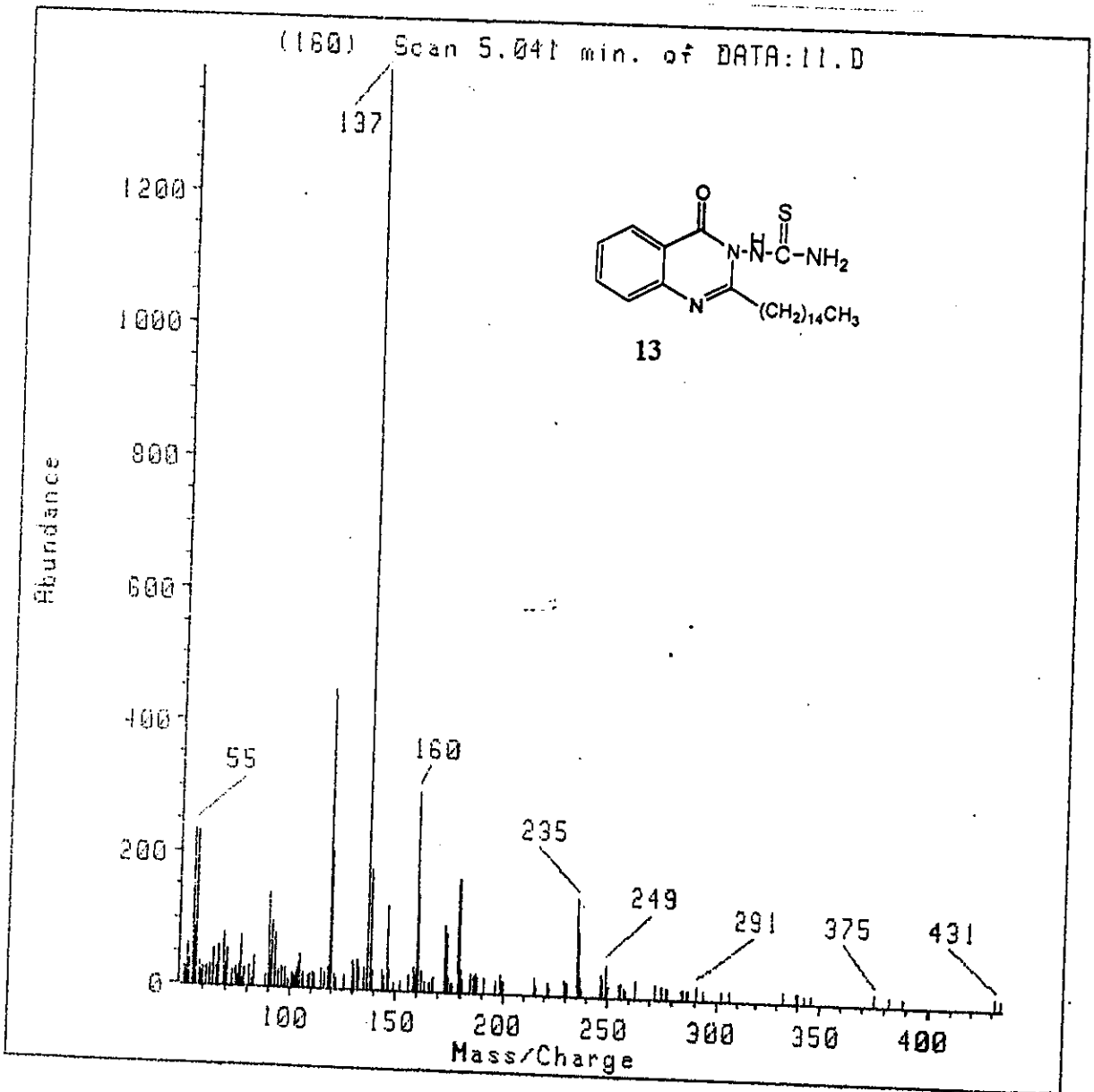

(146) Scan 4.348 min. of DATA:6.D
Abundance
Mass/Charge
80
121
142
186
277
397
100
150
200
250
300
350
400
0
2
4
6
8
10
12
14
16
18
20
22
O
N
NH
N
CH3
(CH2)13CH3
9

Fig (36)


(160) Scan 5.041 min. of DATA:11.D
137
1200
1000
800
600
400
200
0
Abundance
55
160
235
249
291
375
431
100
150
200
250
300
350
400
Mass/Charge
O
S
N-N-C-NH2
N
(CH2)14CH3

13

Fig (37)

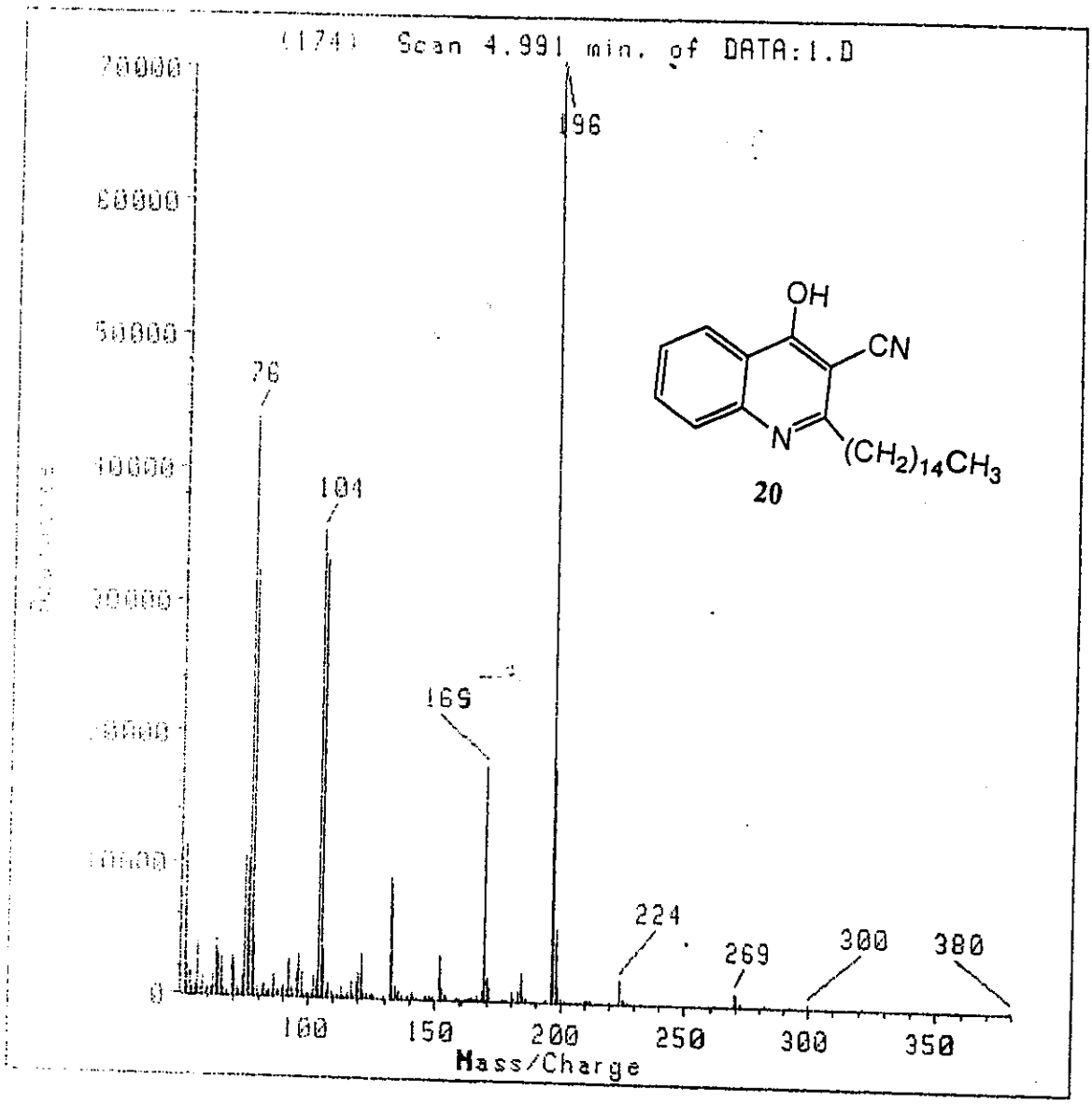

(174) Scan 4.991 min. of DATA:1.D
196
76
104
169
224
269
300
380
100
150
200
250
300
350
Mass/Charge
OH
CN
N
$(CH_2)_{14}CH_3$
20

Fig (38)