LAMPIRAN A

CONTOH PERHITUNGAN PERSENTASE HASIL SINTESIS

I. Perhitungan berat teoritis

a. Asam Antranilat (BM : 137,14 g/mol)

Penimbangan : 13,7 gram

$$\text{mol asam antranilat} : \frac{13,7}{137,14} = 0,1 \text{ mol}$$

b. *p*-klorobenzoil klorida (BM : 175,02 g/mol, berat jenis : 1,377 g/cm^3)

Volume : 25,42 ml

$$\text{mol } p\text{-klorobenzoil klorida} : \frac{25,42 \times 1,377}{175,02} = 0,2 \text{ mol}$$

c. Hidrazin Hidrat (BM : 50,05 g/mol, berat jenis : 1,03 g/cm^3)

Volume : 2,43 ml

$$\text{mol hidrazin hidrat} : \frac{2,43 \times 1,03}{50,05} = 0,05 \text{ mol}$$

d.Benzaldehida (BM : 106,12 g/mol, berat jenis : 1,05 g/cm^3)

Volume : 2,03 ml

$$\text{mol benzaldehida} : \frac{2,03 \times 1,05}{106,12} = 0,02 \text{ mol}$$

II. Perhitungan persentase hasil sintesis berdasarkan mmol teoritis

Persentase hasil 2-(*p*-klorofenil)-4*H*-3,1-benzoksazin-4-on :

	asam antranilat	+	*p*-kloro-benzoil klorida	⟶	2-(*p*-klorofenil)-4*H*-3,1-benzoksazin-4-on	+	Piridinium klorida
awal	0,1 mol		0,2 mol				
reaksi	0,1 mol		0,1 mol	-	0,1 mol		0,1 mol
sisa	0		0,1 mol		0.1 mol		0.1 mol

BM teoritis = 257,5

Massa teoritis = 0,1 mol x 257,5 = 25,75 gram

Massa praktis = 22,46 gram

$$\% \text{ hasil} = \frac{22{,}46}{25{,}75} \times 100\% = 87{,}24\%$$

LAMPIRAN B

PERHITUNGAN UJI T STATISTIK

Pengaruh Penambahan 4-hidroksi-3-metoksibenzaldehid Dibanding Benzaldehid :

t-Test: Paired Two Sample for Means

	Variable 1	*Variable 2*
Mean	72.66666667	85.66666667
Variance	2.333333333	2.333333333
Observations	3	3
Pearson Correlation	0.142857143	
Hypothesized Mean Difference	0	
df	2	
t Stat	-11.25833025	
P(T<=t) one-tail	0.003898695	
t Critical one-tail	2.91998558	
P(T<=t) two-tail	0.00779739	
t Critical two-tail	4.30265273	

Pengaruh Penambahan 3,4-dimetoksibenzaldehid Dibanding Benzal dehid :

t-Test: Paired Two Sample for Means		
	Variable 1	*Variable 2*
Mean	72.66666667	82
Variance	2.333333333	1
Observations	3	3
Pearson Correlation	-0.981980506	
Hypothesized Mean Difference	0	
df	2	
t Stat	-6.423640548	
P(T<=t) one-tail	0.011693926	
t Critical one-tail	2.91998558	
P(T<=t) two-tail	0.023387851	
t Critical two-tail	4.30265273	

LAMPIRAN C

HARGA Rf OPTIMASI SENYAWA

a. Harga Rf optimasi sintesis senyawa 2-(*p*-klorofenil)-3,1-benzoksazin-4(3*H*)-on

Senyawa	Harga Rf
Asam antranilat	0,48
p-klorobenzoil klorida	0,30
Sampel menit ke-30	0,48 & 0,30
Sampel menit ke-45	0,48 & 0,30
Sampel menit ke-60	0,79 & 0,30
Sampel menit ke-75	0,79 & 0,30

b. Harga Rf optimasi sintesis senyawa 3-amino-2-(*p*-klorofenil)-kuinazolin-4(3*H*)-on

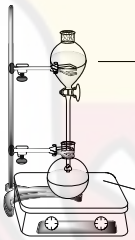
Senyawa	Harga Rf
2-(*p*-klorofenil)-3,1-benzoksazin-4(3*H*)-on	0,71
Sampel jam ke-1	0,70
Sampel jam ke-2	0,71
Sampel jam ke-3	0,71
Sampel jam ke-4	0,70
Sampel jam ke 5	0,38

c. Harga Rf optimasi sintesis senyawa 3-benzilidenamino-2-(*p*-klorofenil)-kuinazolin-4(3*H*)-on

Senyawa	Harga Rf
3-amino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on	0,71
Benzaldehida	0,74
Sampel menit ke-30	0,71 & 0,74
Sampel menit ke-45	0,70 & 0,74
Sampel menit ke-60	0,71 & 0,74
Sampel menit ke-75	0,81 & 0,74

LAMPIRAN D

SKEMA KERJA SINTESIS 2-(*p*-KLOROFENIL)-4*H*-3,1-BENZOKSAZIN-4-ON



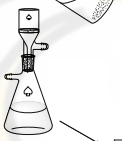
p-klorobenzoil klorida 25,42 ml (0,2 mol)

teteskan sedikit demi sedikit *p*-klorobenzoil klorida ke dalam *beaker glass* yang berisi asam antranilat dalam campuran piridina dan trietilamin sambil diaduk pada suhu kamar selama 1 jam

asam antranilat 0,1 mol (13,7 g) dalam 10 ml piridina dan 50 ml trietanolamina

suspensi 2-(*p*-klorofenil)-4*H*-3,1-benzoksazin-4-on, *p*-klorobenzoil klorida, piridina

saring filtrat, lalu ambil endapannya dan tambahkan larutan $NaHCO_3$ 10% sampai gelembung gas hilang



filtrat :
- Natrium benzoat
- Natrium antranilat
- piridina
- piridium klorida
- air

padatan : 2-(*p*-klorofenil)-4*H*-3,1-benzoksazin-4-on

a. dicuci dengan air
b. direkristalisasi dengan campuran n-hexana dan etil asetat dengan perbandingan 1 : 1
c. dioven

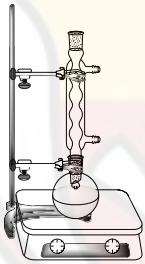
kristal 2-(*p*-klorofenil)-4*H*-3,1-benzoksazin-4-on

uji kemurnian :
- KLT
- titik leleh

identifikasi struktur :
- spektrum UV-Vis
- inframerah
- RMI-^{1}H

LAMPIRAN E

SKEMA KERJA 3-AMINO-2-(*p*-KLOROFENIL)-KUINAZOLIN-4(3*H*)-ON



2-(*p*-klorofenil)-4*H*-3,1-benzoksazin-4-on 12,9 g (0,05 mol) ditambah hidrazin hidrat 2,43 ml (0,02 mol) dalam 100 ml etanol, lalu direfluks dan diaduk selama 5 jam

diamkan sampai dingin

suspensi 3-amino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on, hidrazin, air, dan etanol

padatan : 3-amino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on

filtrat :
- hidrazin
- air
- etanol

a. dicuci dengan air
b. direkristalisasi dengan etanol
c. dioven

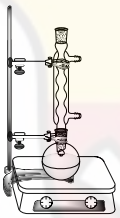
kristal 3-amino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on

uji kemurnian :
- KLT
- titik leleh

identifikasi struktur :
- spektrum UV-Vis
- inframerah
- RMI-^{1}H

LAMPIRAN F

SKEMA KERJA 3-BENZILIDENAMINO-2-(*p*-KLOROFENIL)-KUINAZOLIN-4(3*H*)-ON



3-amino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on 3,07 g (0,01 mol) ditambah benzaldehida 2,03 ml (0,02 mol) dalam 40 ml etanol dan 2 tetes HCl pekat, lalu diaduk selama 1 jam

suspensi disaring lalu ditambah larutan $NaHCO_3$ 10% sampai gelembung gas hilang

suspensi 3-benzilidenamino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on, benzaldehid, air, dan etanol

padatan : 3-benzilidenamino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on

filtrat :
- hidrazin
- air
- etanol

a. dicuci dengan air
b. direkristalisasi dengan etanol
c. dioven

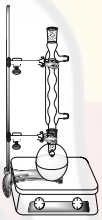
kristal 3-benzilidenamino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on

uji kemurnian :
- KLT
- titik leleh

identifikasi struktur :
- spektrum UV-Vis
- inframerah
- RMI-^{1}H

LAMPIRAN G

SKEMA KERJA 3-(4-HIDROKSI-3-METOKSIBENZILIDENAMINO)-2-(*p*-KLOROFENIL)-KUINAZOLIN-4(3*H*)-ON



3-amino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on 3,07 g (0,01 mol) ditambah 4-hidroksi-3-metoksibenzaldehida 3,04 g (0,02 mol) dalam 40 ml etanol dan 2 tetes HCl pekat, lalu diaduk selama 1 jam

suspensi disaring lalu ditambah larutan $NaHCO_3$ 10% sampai gelembung gas hilang

suspensi 3-(4-hidroksi-3-metoksibenzilidenamino)-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on, 4-hidroksi-3-metoksibenzaldehid, air, dan etanol

padatan : 3-(4-hidroksi-3-metoksibenzilidenamino)-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on

filtrat :
- hidrazin
- air
- etanol

a. dicuci dengan air
b. direkristalisasi dengan etanol
c. dioven

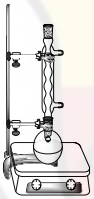
kristal 3-(4-hidroksi-3-metoksibenzilidenamino)-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on

uji kemurnian :
- KLT
- titik leleh

identifikasi struktur :
- spektrum UV-Vis
- inframerah
- ^{1}H-RMI

LAMPIRAN H

SKEMA KERJA
3-(3,4-DIMETOKSIBENZILIDENAMINO)-2-(*p*-KLOROFENIL)-KUINAZOLIN-4(3*H*)-ON



3-amino-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on 3,07 g (0,01 mol) ditambah 3,4-dimetoksibenzaldehida 3,32 g (0,02 mol) dalam 40 ml etanol dan 2 tetes HCl pekat, lalu diaduk selama 1 jam

suspensi disaring lalu ditambah larutan $NaHCO_3$ 10% sampai gelembung gas hilang

suspensi 3-(3,4-dimetoksibenzilidenamino)-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on, 3,4-dimetoksibenzaldehid, air, dan etanol

padatan : 3-(3,4-dimetoksibenzilidenamino)-2-(*p*-klorofenil)kuinazolin-4(3*H*)-on

filtrat :
- hidrazin
- air
- etanol

a. dicuci dengan air
b. direkristalisasi dengan etanol
c. dioven

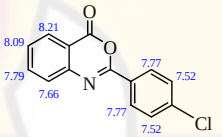
kristal 3-(3,4-dimetoksibenzilidenamino)-2-(*p*-klorofenil)-kuinazolin-4(3*H*)-on

uji kemurnian :
- KLT
- titik leleh

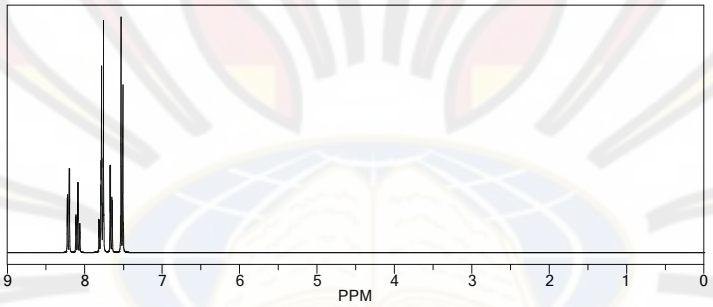
identifikasi struktur :
- spektrum UV-Vis
- inframerah
- ^{1}H-RMI

LAMPIRAN I

ESTIMASI ^{1}H-RMI 2-(*p*-KLOROFENIL)-4*H*-3,1-BENZOKSAZIN-4-ON



Estimation quality is indicated by color: good, medium, rough

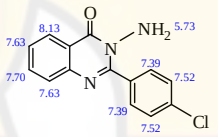


Protocol of the H-1 NMR Prediction:

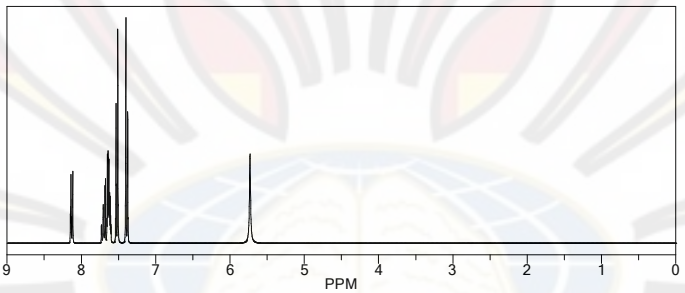
Node	Shift	Base + Inc.	Comment (ppm rel. to TMS)
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl from 1-benzene
		0.22	general corrections
CH	7.66	7.26	1-benzene
		?	1 unknown substituent(s)
		0.21	1 -C(=O)O
		0.19	general corrections
CH	8.21	7.26	1-benzene
		?	1 unknown substituent(s)
		0.87	1 -C(=O)O
		0.08	general corrections
CH	7.77	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		0.21	general corrections
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl from 1-benzene
		0.22	general corrections
CH	7.77	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		0.21	general corrections
CH	7.79	7.26	1-benzene
		?	1 unknown substituent(s)
		0.34	1 -C(=O)O
		0.19	general corrections
CH	8.09	7.26	1-benzene
		?	1 unknown substituent(s)
		0.21	1 -C(=O)O
		0.62	general corrections

LAMPIRAN J

ESTIMASI ^{1}H-RMI 3-AMINO-2-(*p*-KLOROFENIL)-KUINAZOLIN-4(3*H*)-ON



Estimation quality is indicated by color: good, medium, rough

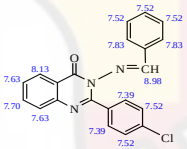


Protocol of the H-1 NMR Prediction:

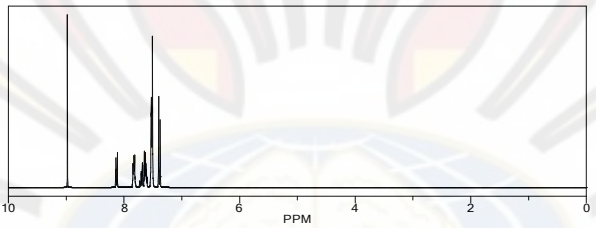
Node	Shift	Base + Inc.	Comment (ppm rel. to TMS)
NH2	5.73	2.00	amine
		3.73	general corrections
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl from 1-benzene
		0.22	general corrections
CH	7.63	7.26	1-benzene
		?	1 unknown substituent(s)
		0.18	1 -C(=O)N
		0.19	general corrections
CH	8.13	7.26	1-benzene
		?	1 unknown substituent(s)
		0.69	1 -C(=O)N
		0.18	general corrections
CH	7.39	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		-0.17	general corrections
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl from 1-benzene
		0.22	general corrections
CH	7.39	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		-0.17	general corrections
CH	7.70	7.26	1-benzene
		?	1 unknown substituent(s)
		0.25	1 -C(=O)N
		0.19	general corrections
CH	7.63	7.26	1-benzene
		?	1 unknown substituent(s)
		0.18	1 -C(=O)N
		0.19	general corrections

LAMPIRAN K

ESTIMASI ^{1}H-RMI 3-BENZILIDENAMINO-2-(*p*-KLOROFENIL)-KUINAZOLIN-4(3*H*)-ON



Estimation quality is indicated by color: good, medium, rough

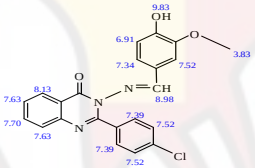


Protocol of the H-1 NMR Prediction:

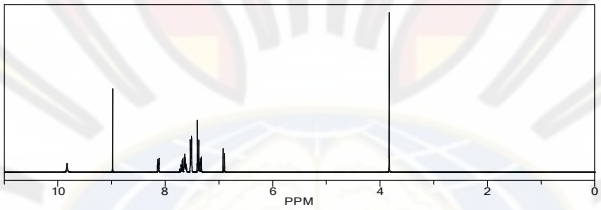
Node	Shift	Base + Inc.	Comment (ppm rel. to TMS)
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl from 1-benzene
		0.22	general corrections
CH	7.63	7.26	1-benzene
		?	1 unknown substituent(s)
		0.18	1 -C(=O)N
		0.19	general corrections
CH	8.13	7.26	1-benzene
		?	1 unknown substituent(s)
		0.69	1 -C(=O)N
		0.18	general corrections
CH	7.39	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		-0.17	general corrections
CH	7.83	7.62	benzylidenimin
		?	1 unknown substituent(s)
		0.21	general corrections
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl from 1-benzene
		0.22	general corrections
CH	7.39	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		-0.17	general corrections
CH	7.83	7.62	benzylidenimin
		?	1 unknown substituent(s)
		0.21	general corrections
CH	7.52	7.29	benzylidenimin
		?	1 unknown substituent(s)
		0.23	general corrections
CH	7.70	7.26	1-benzene
		?	1 unknown substituent(s)
		0.25	1 -C(=O)N
		0.19	general corrections
CH	7.63	7.26	1-benzene
		?	1 unknown substituent(s)
		0.18	1 -C(=O)N
		0.19	general corrections
CH	7.52	7.29	benzylidenimin
		?	1 unknown substituent(s)
		0.23	general corrections
CH	7.52	7.29	benzylidenimin
		?	1 unknown substituent(s)
		0.23	general corrections
CH	8.98	8.11	benzylidenimin
		?	1 unknown substituent(s)
		0.87	general corrections

LAMPIRAN L

ESTIMASI ^{1}H-RMI 3-(4-HIDROKSI-3-METOKSIBENZILIDENAMINO)-2-(*p*-KLOROFENIL)-KUINAZOLIN-4(3*H*)-ON



Estimation quality is indicated by color: good, medium, rough

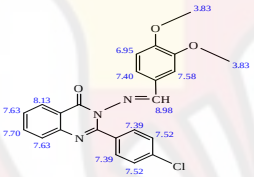


Protocol of the H-1 NMR Prediction:

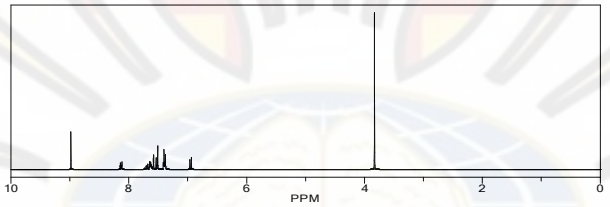
Node	Shift	Base + Inc.	Comment (ppm rel. to TMS)
OH	9.83	5.00	aromatic C-OH
		4.83	general corrections
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl from 1-benzene
		0.22	general corrections
CH	7.63	7.26	1-benzene
		?	1 unknown substituent(s)
		0.18	1 -C(=O)N
		0.19	general corrections
CH	8.13	7.26	1-benzene
		?	1 unknown substituent(s)
		0.69	1 -C(=O)N
		0.18	general corrections
CH	7.39	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		-0.17	general corrections
CH	7.52	7.62	benzylidenimin
		?	1 unknown substituent(s)
		-0.49	1 -O-C from 1-benzene
		-0.17	1 -O from 1-benzene
		0.56	general corrections
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl from 1-benzene
		0.22	general corrections
CH	6.91	7.29	benzylidenimin
		?	1 unknown substituent(s)
		-0.11	1 -O-C from 1-benzene
		-0.53	1 -O from 1-benzene
		0.26	general corrections
CH	7.39	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		-0.17	general corrections
CH	7.34	7.62	benzylidenimin
		?	1 unknown substituent(s)
		-0.44	1 -O-C from 1-benzene
		-0.17	1 -O from 1-benzene
		0.33	general corrections
CH	7.70	7.26	1-benzene
		?	1 unknown substituent(s)
		0.25	1 -C(=O)N
		0.19	general corrections
CH	7.63	7.26	1-benzene
		?	1 unknown substituent(s)
		0.18	1 -C(=O)N
		0.19	general corrections
CH3	3.83	0.86	methyl
		2.87	1 alpha -O-1:C*C*C*C*C*C*1
		0.10	general corrections
CH	8.98	8.11	benzylidenimin
		?	1 unknown substituent(s)
		0.87	general corrections

LAMPIRAN M

ESTIMASI ^{1}H-RMI 3-(3,4-DIMETOKSIBENZILIDENAMINO)-2-(*p*-KLOROFENIL)-KUINAZOLIN-4(3*H*)-ON



Estimation quality is indicated by color: good, medium, rough



Protocol of the H-1 NMR Prediction:

Node	Shift	Base + Inc.	Comment (ppm rel. to TMS)
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl f rom 1-benzene
		0.22	general corrections
CH	7.63	7.26	1-benzene
		?	1 unknown substituent(s)
		0.18	1 -C(=O)N
		0.19	general corrections
CH	8.13	7.26	1-benzene
		?	1 unknown substituent(s)
		0.69	1 -C(=O)N
		0.18	general corrections
CH	7.39	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		-0.17	general corrections
CH	7.58	7.62	benzylidenimin
		?	1 unknown substituent(s)
		-0.49	1 -O-C f rom 1-benzene
		-0.11	1 -O-C f rom 1-benzene
		0.56	general corrections
CH	7.52	7.29	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		0.01	1 -Cl f rom 1-benzene
		0.22	general corrections
CH	6.95	7.29	benzylidenimin
		?	1 unknown substituent(s)
		-0.11	1 -O-C f rom 1-benzene
		-0.49	1 -O-C f rom 1-benzene
		0.26	general corrections
CH	7.39	7.62	benzylidenimin
		0.00	1 -1:C*C*C*C*C*C*1
		-0.06	1 -Cl from 1-benzene
		-0.17	general corrections
CH	7.40	7.62	benzylidenimin
		?	1 unknown substituent(s)
		-0.44	1 -O-C f rom 1-benzene
		-0.11	1 -O-C f rom 1-benzene
		0.33	general corrections
CH	7.70	7.26	1-benzene
		?	1 unknown substituent(s)
		0.25	1 -C(=O)N
		0.19	general corrections
CH	7.63	7.26	1-benzene
		?	1 unknown substituent(s)
		0.18	1 -C(=O)N
		0.19	general corrections
CH3	3.83	0.86	methyl
		2.87	1 alpha -O-1:C*C*C*C*C*C*1
		0.10	general corrections
CH3	3.83	0.86	methyl
		2.87	1 alpha -O-1:C*C*C*C*C*C*1
		0.10	general corrections
CH	8.98	8.11	benzylidenimin
		?	1 unknown substituent(s)
		0.87	general corrections