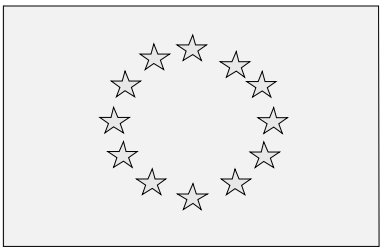
rivm

National Institute
for Public Health and
the Environment



Datasheet
Quinoxaline-2-carboxylic acid-d_4

Reference number : CEC/MAT : 23

Date of preparation : 1994.10.13

date : 2000.10.12

source : CSL

"Bank of Reference Standards"

The Bank of Reference Standards was financially supported by the European Commission

Directorate General "Science, Research and Development DG XII"

Contract MAT 1 - CT92 - 0020

Contract number	:	MAT-CT92-0020[388710]			
Reference number	:	CEC/MAT 23			
Last update	:	1998.01.06	Chemical purity	:	>95 %
Quantity	:	0.106 mg	Isotopic purity	:	d_4 >99 % d_0 <1 %

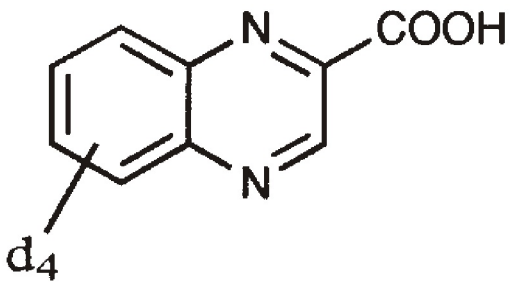


Figure 1. Molecular structure of quinoxaline-2-carboxylic acid-d_4

Name : quinoxaline-[5,6,7,8-d_4]-2-carboxylic acid
Synonym : (benzene ring-d_4)-quinoxaline-2-carboxylic acid
Molecular formula : $C_9H_2D_4N_2O_2$
Molecular weight : 178.183

Long term stability tested on 1997.10.08 : 91.3 ± 5.6 %
(storage 4 °C, analysis HPLC-UV, 6 test on 2 ampoules)

Methods of Characterization:

I UV spectroscopy
II IR spectroscopy
III Mass spectroscopy
IV ^{1}H-NMR spectroscopy

I UV Spectroscopy

Instrument: Hitachi U 3000
Method: Dissolved in ethanol (10mg/l)

Results

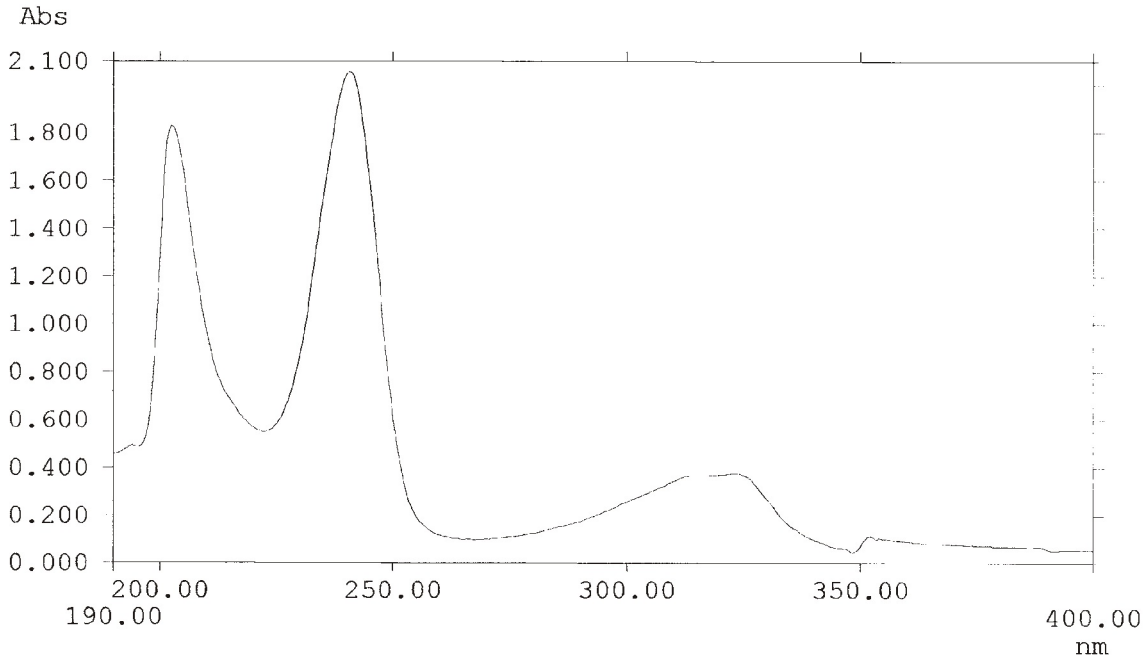


Figure 2. The UV spectrum of quinoxaline-2-carboxylic acid-d_4

Peak no.	wavelength (nm)	absorbance
1	352.00	0.1123
2	323.00	0.3751
3	240.50	2.0549
4	202.50	1.8279

II IR-Spectroscopy

Instrument: Perkin Elmer STIR 1720X
Sampling technique: nujol mull

Results

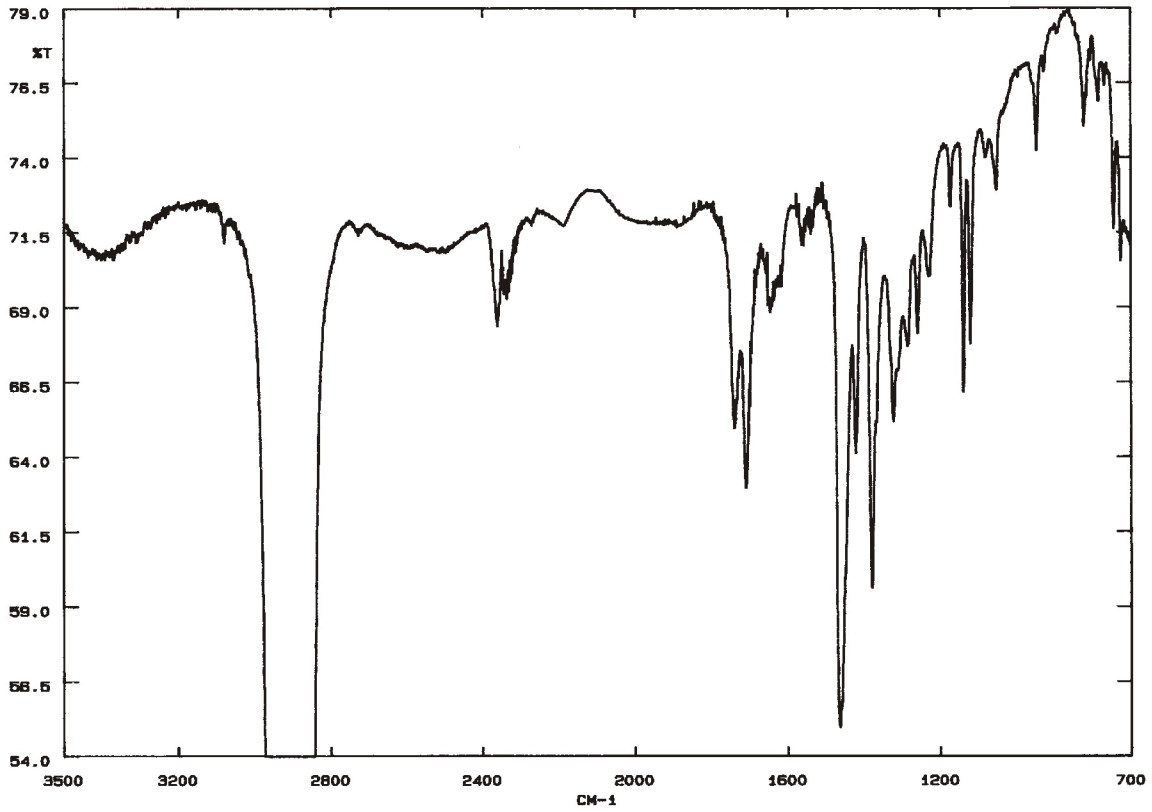


Figure 3. The IR-spectrum of quinoxaline-2-carboxylic acid-d_4

Wavelength (cm-1)	designation
3369	O-H stretch
1739	
1710	C=O stretch
1421	
1322	
1281	
1259	
1230	
1139	
1120	
947	
745	
727	

III Mass Spectroscopy

Instrument: Kratos MS 25
Sampling technique: Direct probe, 70 ev electron impact

Results

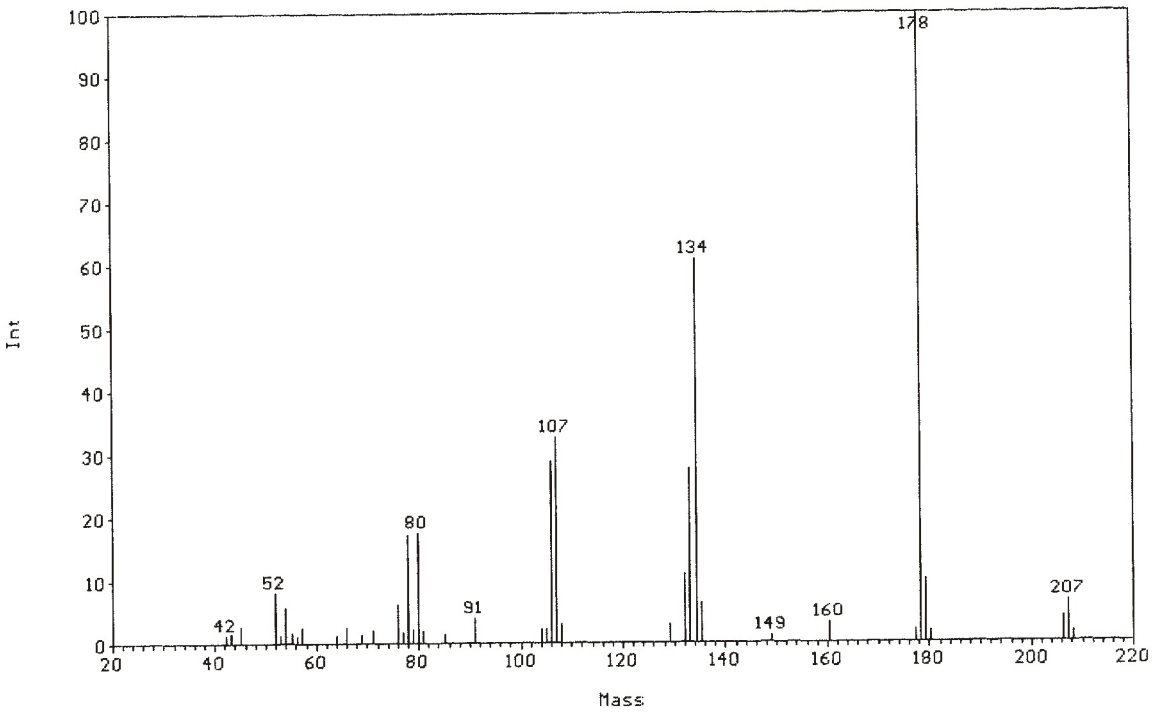


Figure 4. The mass spectrum of quinoxaline-2-carboxylic acid-d_4

m/u	percentage	designation
178	100M	
134	61	M - CO_2
133	28	M - CO_2 H
107	33	M - (CO_2 + HCN ?)
106	29	M - (CO_2H + HCN ?)
80	18	C_6D_4 or $C_4N_2H_4$?
77	17	C_6H_5 ?

III ^{1}H-NMR Spectroscopy

Instrument: GX 400
Solvent: acetone-d_6 + CD_3OD with TMS (δ = 0.0) as internal standard.

Results

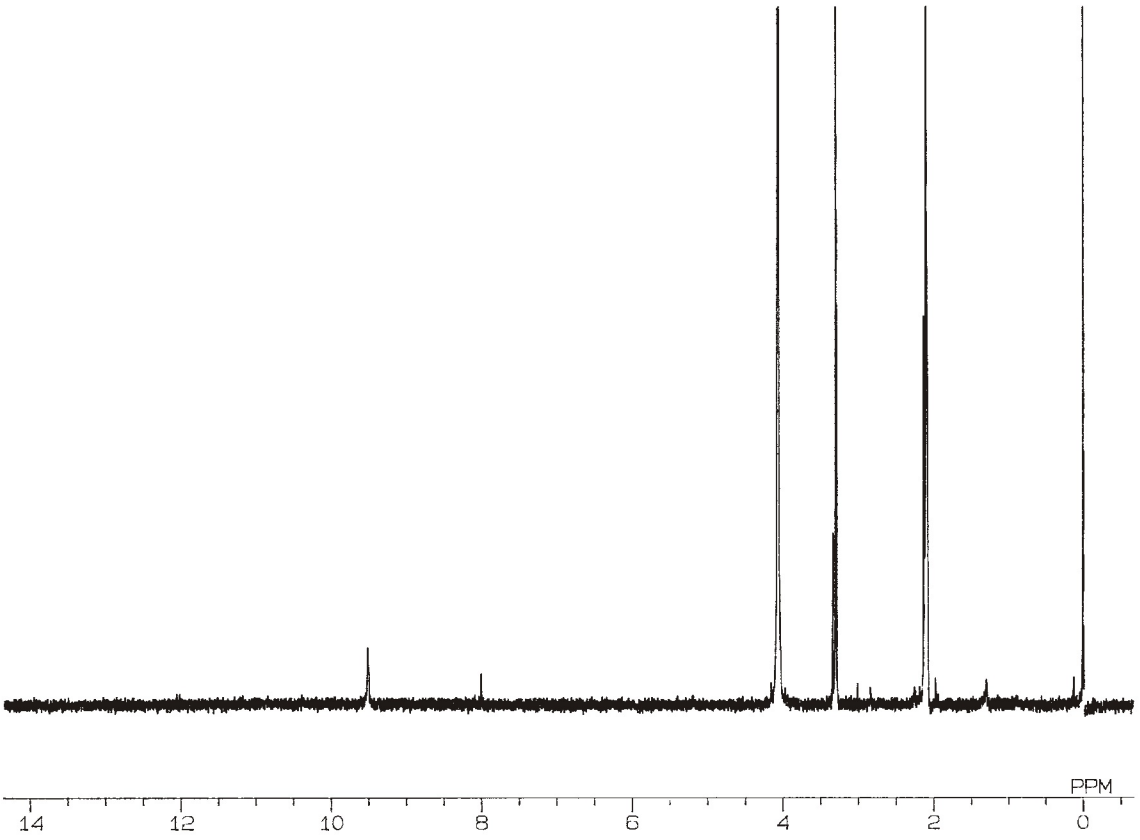


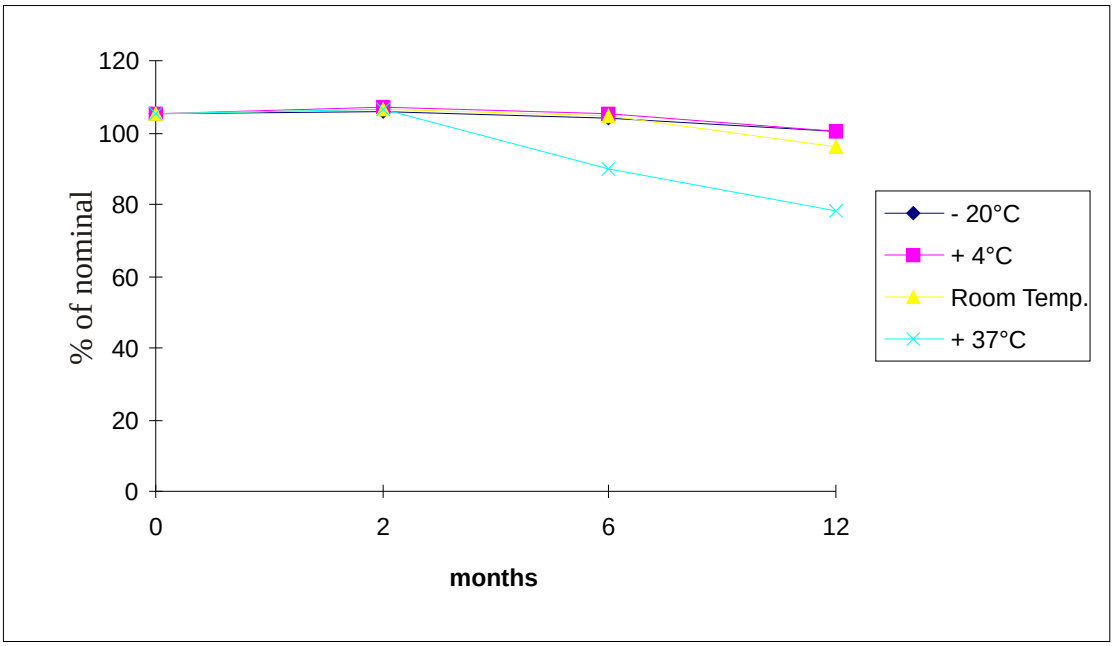
Figure 5. The NMR spectrum of quinoxaline-2-carboxylic acid-d_4

Chemical Shift (δ)	number of protons	designation
9.51	1	H3 (arom.)

Preparation and validation of reference standards

The production of ampoules containing the reference material was described in the final report produced September 1995.
The thermal stability of the compound was under investigation and the results for quinoxaline-2-carboxylic acid-d_4 over a period of 12 months are expressed in the table and chart below.

Quinoxaline-2-carboxylic acid-d_4 Stability Trials



Results of quinoxaline-2-carboxylic acid-d_4 stability trials

The results below are the % recovery (with cv) of quinoxaline-2-carboxylic acid-d_4 at 4 different storage temperatures over a period of 12 months compared with a standard equivalent to 0.1 mg.

	temp. (°C)	t = 0 months (% recovery)	t = 2 months (% recovery)	t = 6 months (% recovery)	t = 12 months (% recovery)
QCA-d_4	- 20°C	105.5 +/- 1.5	106.0 +/- 2.0	104.0 +/- 1.0	100.5 +/- 1.0
	4°C	-	107.0 +/- 1.5	105.5 +/- 1.5	100.5 +/- 1.5
	Room Temp.	-	106.5 +/- 2.0	104.5 +/- 0.5	96.0 +/- 1.0
	37°C	-	106.5 +/- 1.5	90.0 +/- 1.0	78.0 +/- 2.0

Conclusion

The spectroscopic data is consistant with the proposed structure for all the methods of determination although a very small amount of DMF (which was used as solvent during ampouling) was detected by NMR at approximately 2.8 and 2.9 d. There was also a group of 3 unexpected peaks at 206, 207 and 208 m/u (1.5, 6.5 and 4 % resp.) in the MS spectrum which were probably due to some form of bonding or association with the DMF (Note - M + NCH_3 = 207 m/u).
No significant impurities were detected by any of the methods of characterization employed.

The results from the stability trials indicate that quinoxaline-2-carboxylic acid-d_4 is acceptably stable over a period of 12 months except for the ampoules stored at + 37 °C which had lost an average of 15.5 % after 6 months and 28.5 % after 12 months of quinoxaline-2-carboxylic acid-d_4 from each ampoule and those stored at room temperature which had lost an average of 9.5 % after 12 months. It is therefore recommended that these ampoules are stored at - 20 °C or lower before opening and at + 4 °C or lower when in solution. The stability of this compound also requires careful monitoring if it is to be used with confidence as a reference standard.