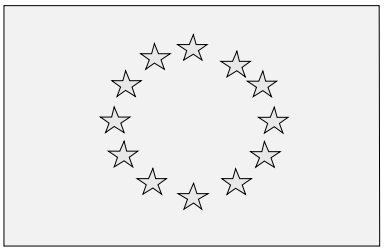
National Institute
for Public Health and
the Environment



Datasheet 1-Desoxycarbadox

Reference number : CEC/MAT : 20

Date of preparation : 1994.06.02

date : 1998.01.06

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 20			
Last update	:	1998.01.06	Chemical Purity	:	>95 %
Quantity	:	1.54 mg			

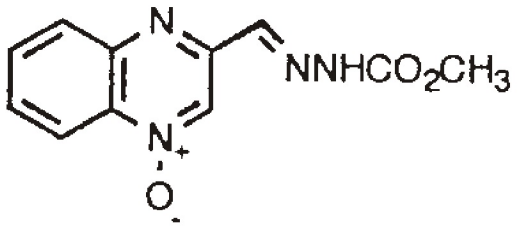


Figure 1. Molecular structure of 1-desoxycarbadox

Name : Methyl 3-(2-quinoxalinylmethylene)-carbazate N^4-oxide
Synonym : 1-Desoxycarbadox
Molecular formula : $C_{11}H_{10}N_4O_3$
Molecular weight : 246.226

Long term stability tested on 1997.11.10 : 97.7 ± 0.8 %
(storage 4 °C, analysis HPLC-UV, 6 tests 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 (20mg/l)

Results

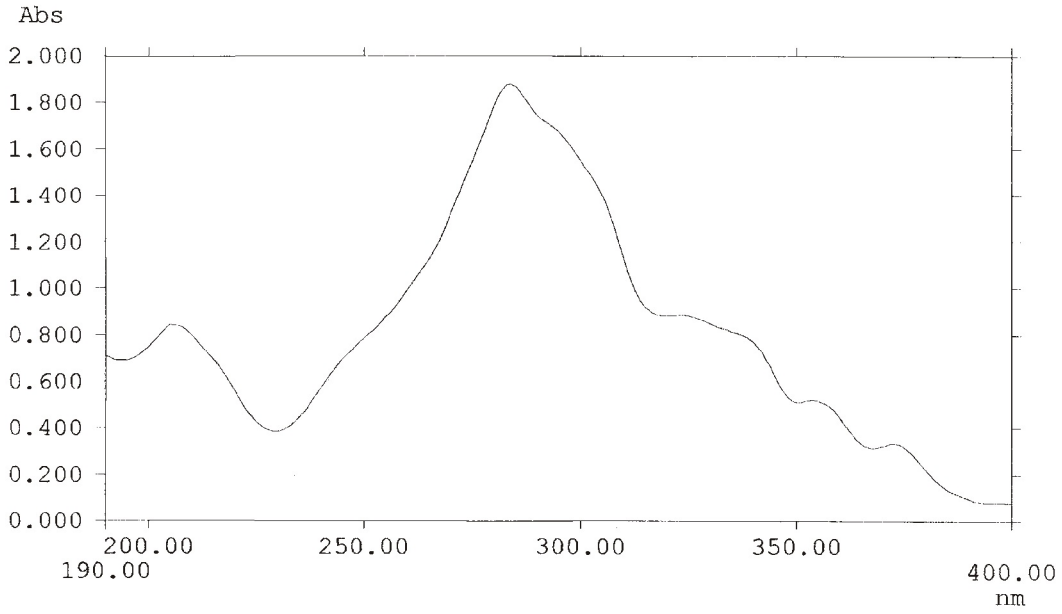


Figure 2. The UV spectrum of 1-desoxycarbadox

Peak no.	wavelength (nm)	absorbance
1	373.00	0.3340
2	284.00	1.8784
3	205.00	0.8443

II IR-Spectroscopy

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

Results

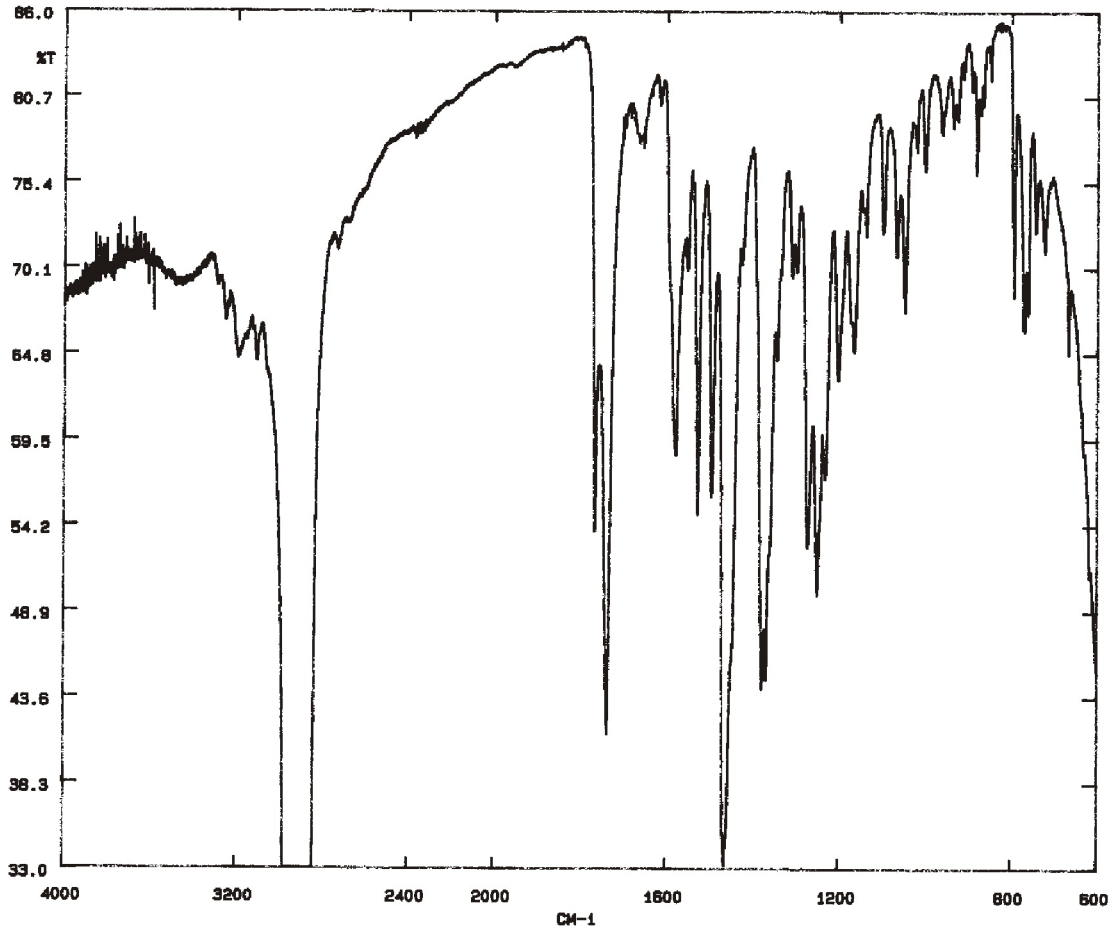


Figure 3. The IR-spectrum of 1-desoxycarbadox

Wavelength (cm-1)	designation
1734	C=O stretch
1576	C=C stretch in aromatic compounds
1526	
1494	C=C stretch in aromatic compounds
1248	
1164	C-O stretch
1046	
794	C-H out of plane deformations
768	C-H out of plane deformations

III Mass spectroscopy

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

Results

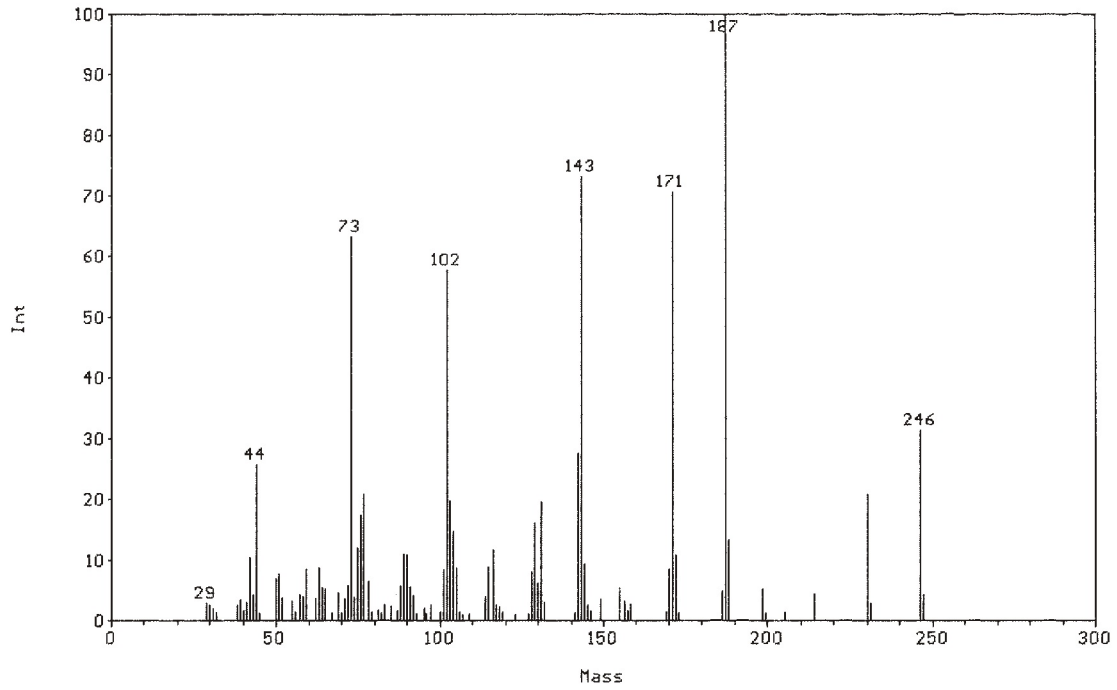


Figure 4. The mass spectrum of 1-desoxycarbadox

m/u	percentage	designation
247	4	M + 1
246	31	M
230	21	M - O
214	4	M - CH_3OH
187	100	M - CH_3OCO
171	71	M - (CH_3OCO + O)
143	73	M - ($NNCH_3OCO$ + O)

III ^{1}H-NMR Spectroscopy

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

Results

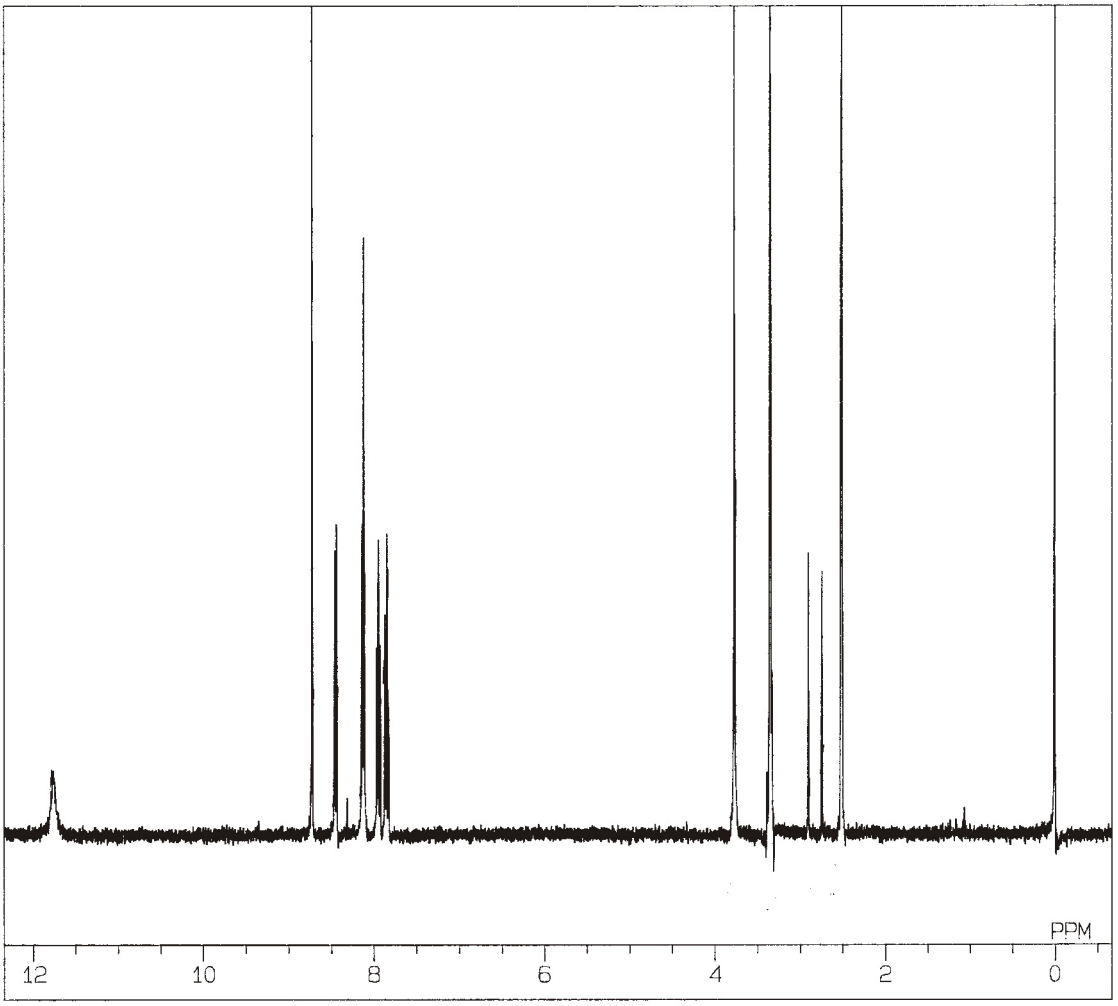


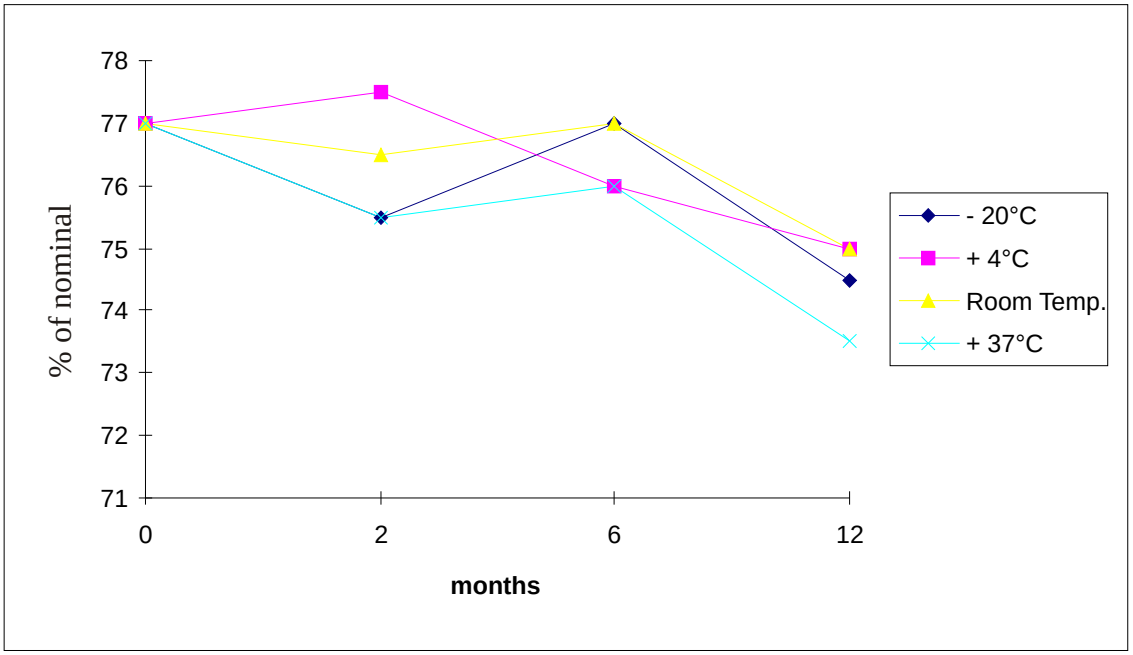
Figure 5. The NMR spectrum of 1-desoxycarbadox

Chemical Shift (δ)	number of protons	designation
3.77	3	CH_3
7.84	1	H6 or H7 (arom.)
7.94	1	H6 or H7 (arom.)
8.11	1	H11
8.13	1	H5 or H8 (arom.)
8.44	1	H5 or H8 (arom.)
8.72	1	H3 (arom.)
11.76	1	NH

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 1-desoxycarbadox over a period of one year are expressed in the table and chart below.

1-Desoxycarbadox Stability Trials



Results of 1-desoxycarbadox stability trials

The results below are the % recovery (with cv) of 1-desoxycarbadox at 4 different storage temperatures over a period of 12 months compared to a standard equivalent to 2 mg.

	temp. (°C)	t = 0 months (% Recovery)	t = 2 months (% Recovery)	t = 6 months (% Recovery)	t = 12 months (% Recovery)
1-DSC	- 20°C	77.0 +/- 1.5	75.5 +/- 4.0	77.0 +/- 2.5	74.5 +/- 1.5
	+ 4°C	-	77.5 +/- 2.5	76.0 +/- 2.0	75.0 +/- 3.5
	Room Temp.	-	76.5 +/- 4.5	77.0 +/- 2.5	75.0 +/- 3.5
	+ 37°C	-	75.5 +/- 1.5	76.0 +/- 1.5	73.5 +/- 3.0

Conclusion

The spectroscopic data is consistant with the proposed structure for all the methods of determination although a small amount of DMF (which was used as solvent during ampouling) was detected by all the methods of characterization* except UV spectroscopy.
No significant impurities were detected by any of the methods of characterization employed.

The results from the stability trials indicate that 1-desoxycarbadox is acceptably stable over a period of one year at temperatures up to 37 °C.

* At approximately 2.8 and 2.9 d in the NMR spectrum, 1680 cm^{-1} in the IR spectrum and 73 m/u in the mass spectrum.