National Institute
for Public Health and
the Environment

Datasheet Dimetridazole-d_3

Reference number : CEC/MAT : 31

Date of preparation : 1995.05.11

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 31			
Last update	:	1998.01.06	Chemical purity	:	>95 %
Quantity	:	0.105 mg	Isotopic purity	:	d_3 >99 % d_0 <1 %

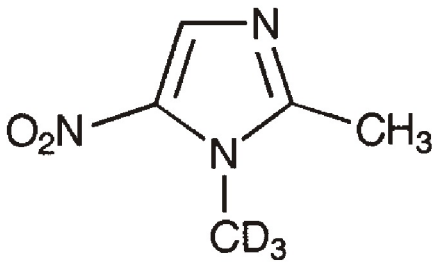


Figure 1. Molecular structure of dimetridazole-d_3

Name : 1,2-dimethyl(1-methyl-d_3)-5-nitroimidazole
Synonym : dimetridazole-d_3
Molecular formula : $C_5H_4D_3N_3O_2$
Molecular weight : 144.148

Long term stability on 1997.10.09 : 93.2 ± 1.1 %
(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

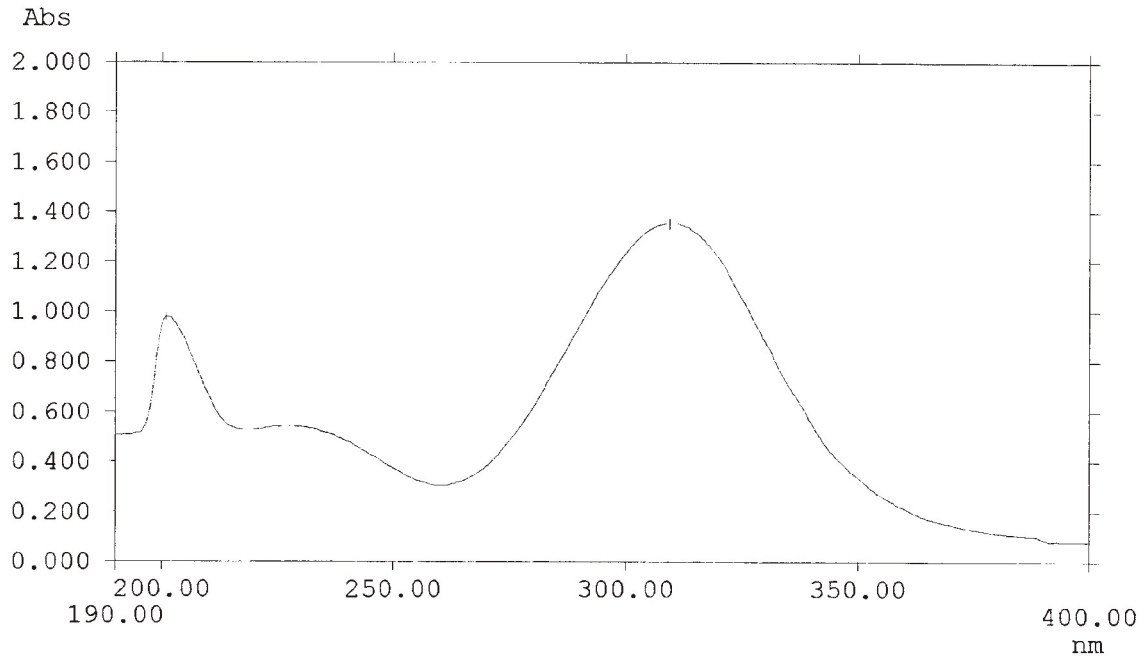


Figure 2. The UV spectrum of dimetridazole-d_3

Peak no.	wavelength (nm)	absorbance
1	309.50	1.3581
2	201.00	0.9806

II IR-Spectroscopy

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

Results

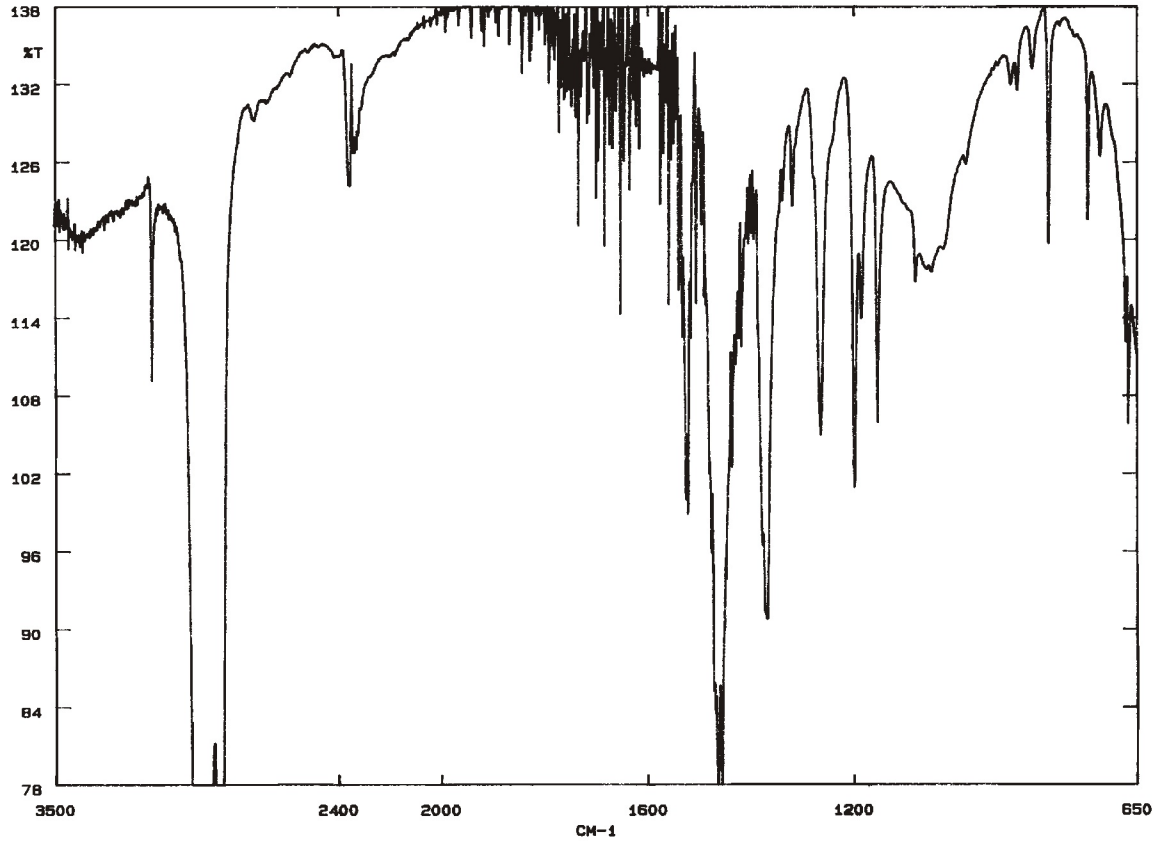


Figure 3. The IR spectrum of dimetridazole-d_3

Wavelength (cm-1)	designation
3122	C-H stretch
1521	NO_2 stretch
1319	
1264	
1199	
1185	
1154	
1080	
822	
746	C-H out of plane deformation

III Mass Spectroscopy

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

Results

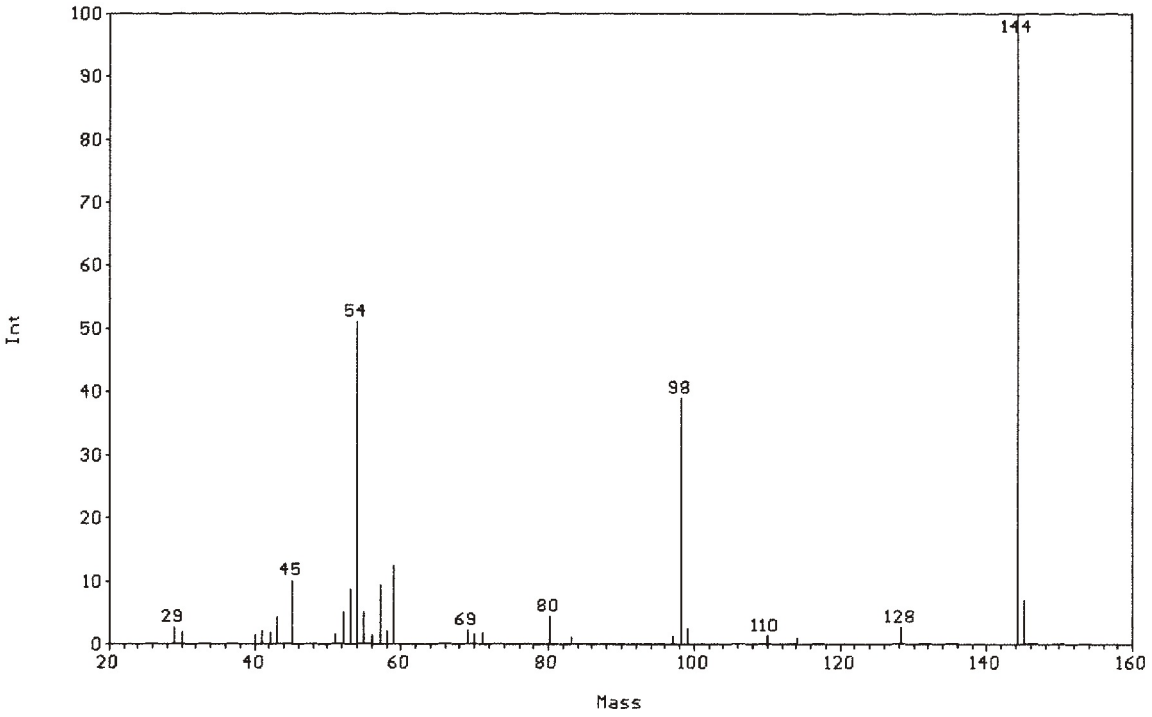


Figure 4. The mass spectrum of dimetridazole-d_3

m/u	percentage	designation
144	100M	
98	39	M - NO_2
59	13	
54	51	
45	10	

III ^{1}H-NMR Spectroscopy

Instrument: Bruker AC250
Solvent: CD_3OD with TMS (δ = 0.0) as internal standard.

Results

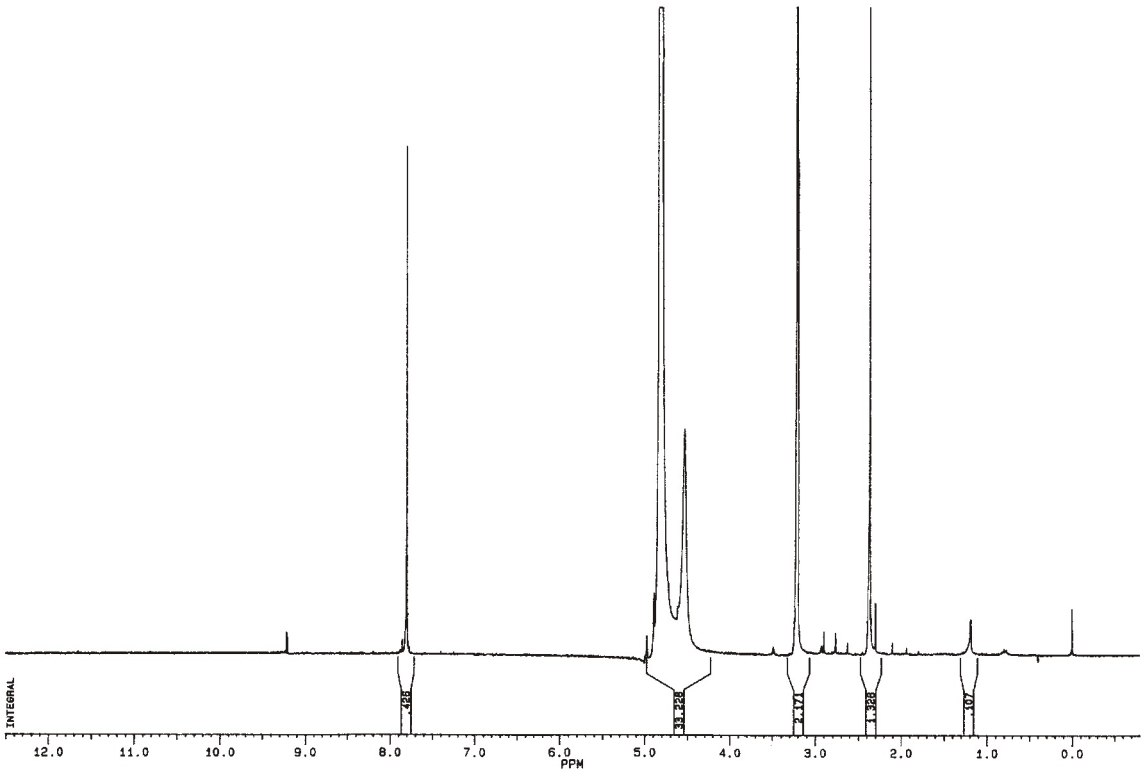


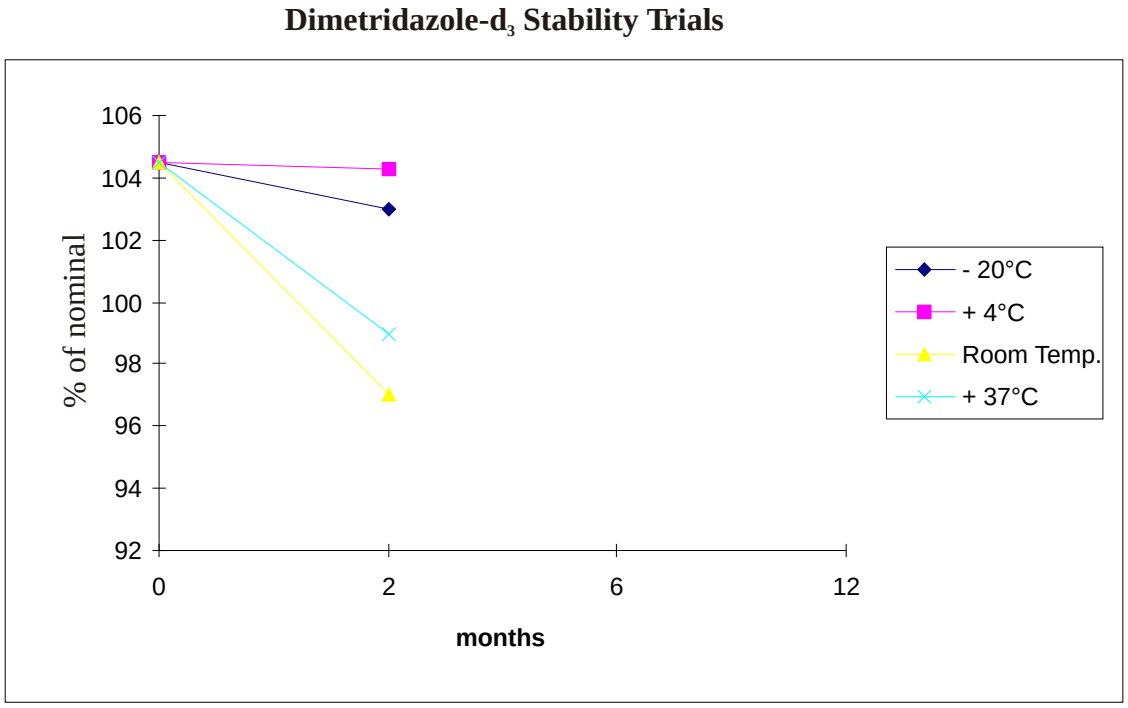
Figure 5. The NMR spectrum of dimetridazole-d_3

Chemical Shift (δ)	number of protons	designation
2.35	3	CH_3
7.8	1	H4

Datasheet BCR-MAT reference standard material Dimetridazole-d_3

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 is under investigation and the results for dimetridazole-d_3 over a period of two months are expressed in the table and chart below.



Results of dimetridazole-d_3 stability trials

The results below are the % recovery (with cv) of dimetridazole-d_3 at 4 different storage temperatures over a period of 2 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)
DMZ-d_3	- 20°C	104.5 +/- 1.0	103.0 +/- 1.5	-	-
	4°C	-	104.3 +/- 0.5	-	-
	Romm Temp.	-	97.0 +/- 3.0	-	-
	37°C	-	99.0 +/- 1.5	-	-

Conclusion

The spectroscopic data is consistant with the proposed structure for all the methods of determination although a little DMF (which was used as solvent during ampouling) was detected by NMR at approximately 2.8 and 2.9 d.
No significant impurities were detected by any of the methods of characterization employed.

The results from the stability trials indicate that dimetridazole-d_3 is acceptably stable over a period of two months at temperatures up to 37 °C although the recoveries at room temperature and 37 °C require further monitoring .