4-*tert*-ブチルカテコールのラットを用いた
経口投与による2週間毒性試験（混餌試験）報告書

試験番号：0709

APPENDICES

APPENDICES

APPENDIX 1-2

STABILITY OF 4-*tert*-BUTYLCATECHOL IN THE 2-WEEK FEED STUDY

STABILITY OF 4-*tert* -BUTYLCATECHOL IN THE 2-WEEK FEED STUDY

Test Substance : 4-*tert* -Butylcatechol (Wako Pure Chemical Industries, Ltd.)

Lot No. : WKH4921

1. High Performance Liquid Chromatography

Instrument : Shimadzu LC-10 High Performance Liquid Chromatograph

Column : TSK-GEL ODS-80TM (4.6 mmϕ × 15 cm)

Column Temperature: 40 °C

Flow Rate : 1 mL/min

Mobile Phase : Acetonitrile : 5mmol SDS solution (Phospholic acid pH2.2) = 60 : 40

Detector : UV (285 nm)

Injection Volume : 10 μL

Date analyzed	Peak No.	Retention Time (min)	Area (%)
2008.06.24	1	3.080	100
2008.07.18	1	3.080	100

Result: High performance liquid chromatography indicated one major peak (peak No.1) analyzed on 2008.6.24 and one major peak (peak No.1) analyzed on 2008.7.18 No new trace impurity peak in the test substance analyzed on 2008.7.18 was detected.

2. Conclusion: The test substance was stable for the period that the test substance had been used for the study.

APPENDIX 2-1

CONCENTRATION OF 4-*tert*-BUTYLCATECHOL IN FORMULATED DIETS IN THE 2-WEEK FEED STUDY

CONCENTRATION OF 4-*tert* -BUTYLCATECHOL IN FORMULATED DIETS IN THE 2-WEEK FEED STUDY

Analytical Method : The samples were analyzed by high performance liquid chromatography.

Instrument : Shimadzu LC-10 High Performance Liquid Chromatograph

Column : TSK-GEL ODS-80TM (4.6 mmϕ × 15 cm)

Column Temperature: 40 °C

Flow Rate : 1 mL/min

Mobile Phase : Acetonitrile : 5mmol SDS solution (Phospholic acid pH2.2) = 60 : 40

Detector : UV (285 nm)

Injection Volume : 10 μL

	Target Concentration				
Date Analyzed	1250[a]	2500	5000	10000	20000
2008.06.25	1300[b] (104)[c]	2560 (102)	5190 (104)	10700 (107)	21600 (108)

[a] ppm
[b] ppm (Mean measured concentration.)
[c] % (Mean measured concentration/target concentration × 100.)

APPENDIX 2-2

HOMOGENEITY OF 4-*tert*-BUTYLCATECHOL IN FORMULATED DIETS IN THE 2-WEEK FEED STUDY

HOMOGENEITY OF 4-*tert* -BUTYLCATECHOL IN FORMULATED DIETS IN THE 2-WEEK FEED STUDY

Analytical Method : The samples were analyzed by high performance liquid chromatography.

Instrument : Shimadzu LC-10 High Performance Liquid Chromatograph

Column : TSK-GEL ODS-80TM (4.6 mmϕ × 15 cm)

Column Temperature: 40 °C

Flow Rate : 1 mL/min

Mobile Phase : Acetonitrile : 5mmol SDS solution (Phospholic acid pH2.2) = 60 : 40

Detector : UV (285 nm)

Injection Volume : 10 μL

	Target Concentration				
	1250[a]	2500	5000	10000	20000
Coefficient Variation	2.61[b]	2.83	1.38	1.71	0.99

[a] ppm
[b] % (n=7)

APPENDIX 2-3

STABILITY OF 4-*tert*-BUTYLCATECHOL IN FORMULATED DIETS IN THE 2-WEEK FEED STUDY

STABILITY OF 4-*tert* -BUTYLCATECHOL IN FORMULATED DIETS IN THE 2-WEEK FEED STUDY

Analytical Method : The samples were analyzed by high performance liquid chromatography.

Instrument : Shimadzu LC-10 High Performance Liquid Chromatograph

Column : TSK-GEL ODS-80TM (4.6 mmϕ × 15 cm)

Column Temperature: 40 °C

Flow Rate : 1 mL/min

Mobile Phase : Acetonitrile : 5mmol SDS solution (Phospholic acid pH2.2) = 60 : 40

Detector : UV (285 nm)

Injection Volume : 10 μL

	Target Concentration	
Date Analyzed	1250[a]	20000
2008.05.29	1220 (100)[b]	20000 (100)
2008.06.06[c]	1140 (93.4)	18700 (93.5)
2008.06.09[d]	1190 (97.5)	20300 (102)

[a] ppm
[b] % (Percentage was based on the concentration at the date of preparation.)
[c] Animal room samples
[d] Cold storage samples

APPENDIX 1-1

IDENTITY OF 4-*tert*-BUTYLCATECHOL IN THE 2-WEEK FEED STUDY

IDENTITY OF 4-*tert* -BUTYLCATECHOL IN THE 2-WEEK FEED STUDY

Test Substance : 4-*tert* -Butylcatechol (Wako Pure Chemical Industries, Ltd.)

Lot No. : WKH4921

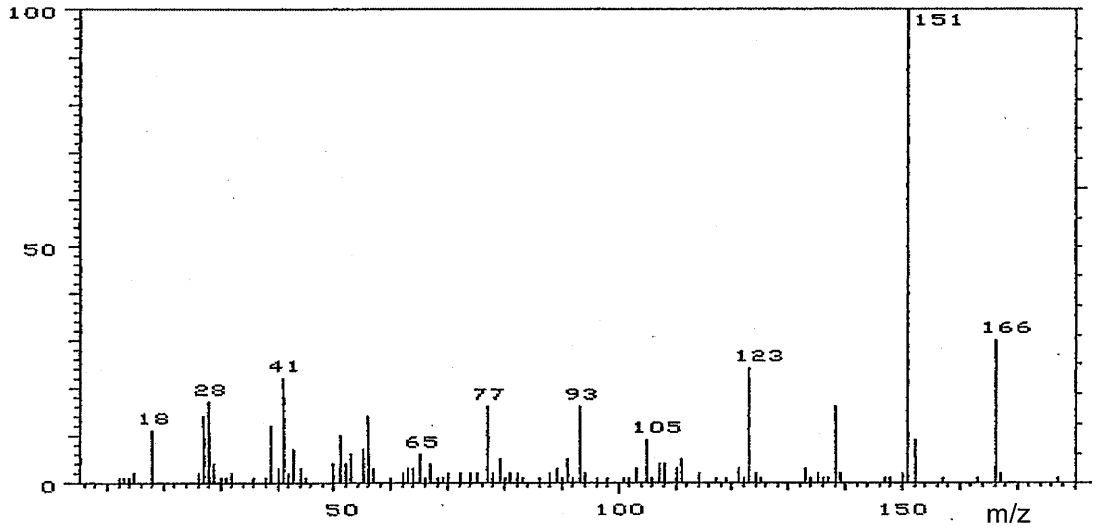
1. Spectral Data

Mass Spectrometry

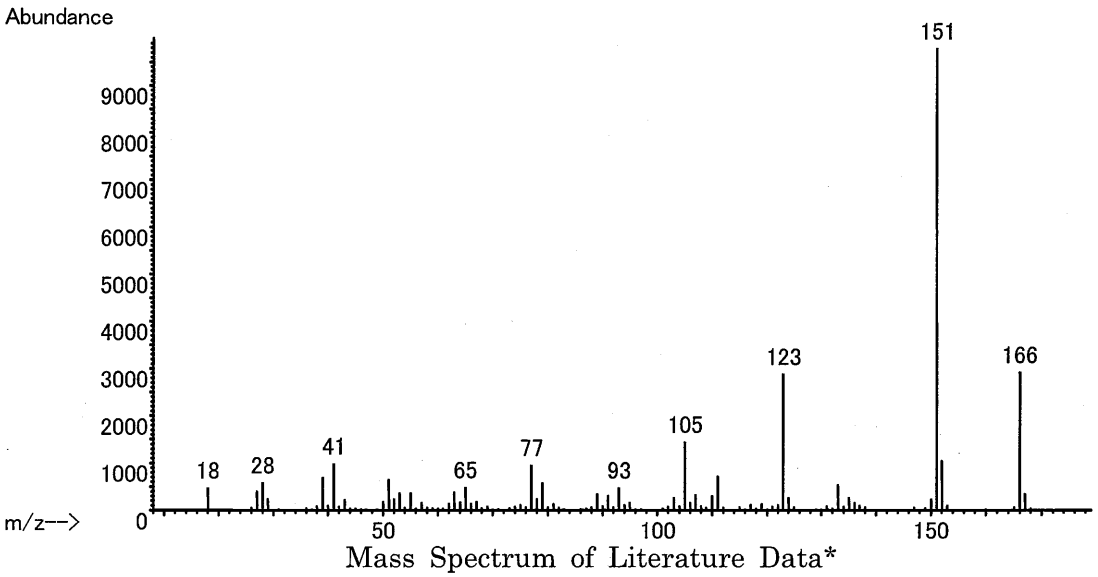
Instrument : Hitachi M-80B Mass Spectrometer

Ionization : EI (Electron Ionization)

Ionization Voltage : 70eV



Mass Spectrum of Test Substance



Mass Spectrum of Literature Data*

Result: The mass spectrum was consistent with literature spectrum.

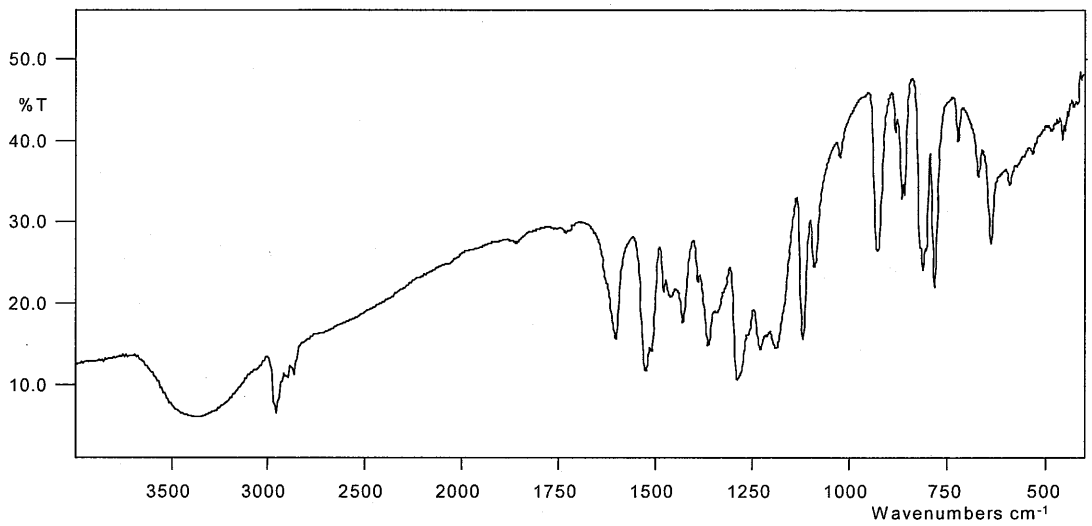
(*McLafferty FW, ed. 1994. Wiley Registry of Mass Spectral Data. 6th ed. New York, NY:John Wiley and Sons.)

Infrared Spectrometry

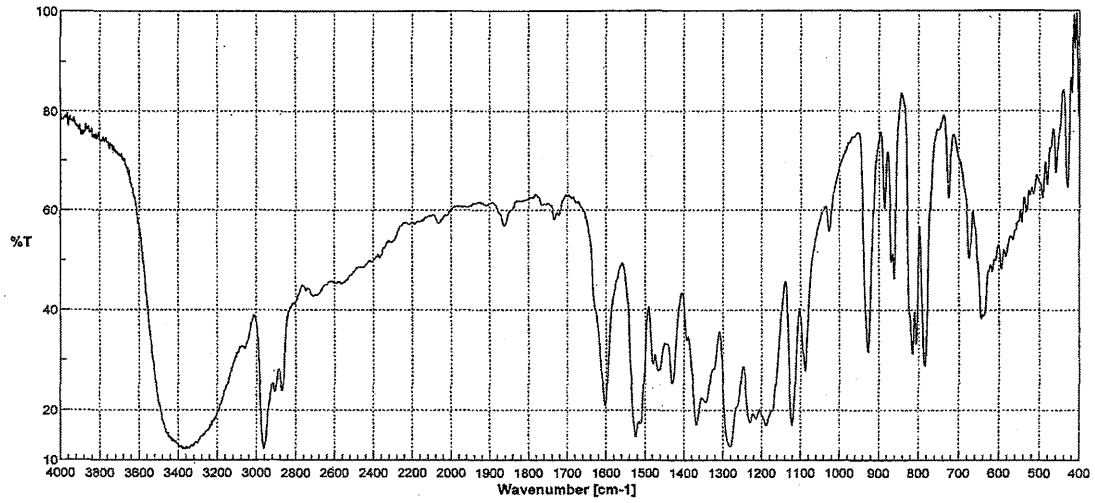
Instrument : Shimadzu FTIR-8200PC Infrared Spectrometer

Cell : KBr

Resolution : 4 cm^{-1}



Infrared Spectrum of Test Substance



Infrared Spectrum of Literature Data*

Result: The infrared spectrum was consistent with literature spectrum.
(*Performed by Wako Pure Chemical Industries, Ltd.)

2. Conclusion: The test substance was identified as 4-*tert* -butylcatechol by mass spectrum and infrared spectrum.