

Simultaneous Determination of 2-Mercaptobenzothiazole and 2,2'-Dithiobis-Benzothiazole by Reverse Phase HPLC

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A New simple, precise, rapid and selective reverse phase high performance liquid chromatographic method is developed for the simultaneous determination of 2-mercaptobenzothiazole (MBT) and 2,2'-dithiobis-benzothiazole (MBTS). The relative standard deviation and recovery for MBT are 0.51% and 98.63%, respectively, and relative standard deviation and recovery for MBTS are 0.51% and 98.66%, respectively.

2-Mercaptobenzothiazole (MBT) and its oxidation product, 2,2'-dithiobis-benzothiazole (MBTS) are used as rubber vulcanization accelerators. Literature survey reveals many methods such as titrimetric (MBT^{1,2} MBTS³), G.C. (MBTS⁴) and mass spectroscopy (MBTS⁵) for their determination individually. However, no attempt has been made yet for the simultaneous determination of MBT and MBTS. In this communication, we propose a simple HPLC method for the rapid and precise simultaneous estimation of MBT and MBTS.

Double distilled water, acetonitrile and THF used were of chromatographic grade. Orthophosphoric acid used was of A.R. grade. Standard samples of naphthalene, MBT and MBTS were procured from E. Merck, Mumbai. The liquid chromatographic system consisted of a WATERS Isocratic 510 pump, with a 7125 Rheodyne valve injector and a 20 microlitre fixed loop equipped with a WATERS 486 UV detector controlled by a Waters 745 Integrator. Microbondapak C18 (10 micron) 30 cm column was used as the stationary phase. The mobile phase used was 40:40:20 (THF:acetonitrile:buffer) v/v and adjusted pH to 4.0 with dilute orthophosphoric acid. Buffer was prepared by dissolving 1.42 g of Na₂HPO₄ in one litre water. Flow rate was kept at 1.0 ml/min with an average operating pressure of 2200 psi and the response was monitored at 240 nm. Standard solution of MBT (2 mg/ml) and MBTS (2 mg/ml) and internal standard solution (naphthalene, 2 mg/ml) were prepared in THF.

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Calibration solutions were prepared by taking different volumes (2.8 ml) of standard stock solution (MBT or MBTS) in four different 50 ml volumetric flasks and 5 ml of internal standard stock solution was added to each flask and was diluted with THF. From the standard stock solutions, mixed standards with internal standard were prepared to contain 80 µg/ml of MBT, 80 µg/ml of MBTS and 200 µg/ml of naphthalene respectively. Then 20 µl of each one was injected five times and the chromatograms were recorded. The retention time of MBT, MBTS and naphthalene were found to be 4.45 min, 8.78 and 6.25 min, respectively. Both MBT and MBTS showed linearity in the range of 80-320 µg/ml respectively. The calibration curves were plotted using the peak area of the standard chromatogram against the concentration. The peak area of the mixed standard chromatogram was compared and the amount of MBT and MBTS were calculated and shown in table-1. The results of recovery analysis (standard addition) are also shown in table-1. From the results it can be revealed that there is a good correlation between the amounts of standards taken and total amounts found at all concentration levels of MBT and MBTS.

The reported HPLC method for the simultaneous determination of MBT and MBTS is simple, rapid and accurate. The reliability and suitability of the method could be seen from the recovery values. therefore, the method can be used in routine quality control analysis of MBT and MBTS.

Table - 1: Recovery Analysis

Chemical	Amount		% error	% Recovery
	Taken	Found	Mean \pm SD	Mean \pm SD
MBT	100	99.21		
	180	179.97		
	260	254.93	1.54 \pm 1.11	98.46 \pm 1.11
	340	330.88		
	420	410.45		
MBTD	100	99.42		
	180	180.59		
	260	255.33	1.45 \pm 0.98	98.67 \pm 1.18
	340	332.97		
	420	409.31		

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