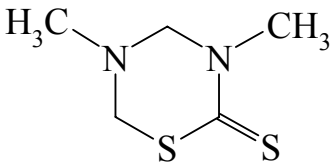
DAZOMET
146



ISO common name	Dazomet
Chemical name	Tetrahydro-3,5-dimethyl-1,3,5-thiadiazine-2-thione (IUPAC); tetrahydro-3,5-dimethyl 2*H*-1,3,5-thiadiazine-2-thione, (CA; *533-74-4*)
Empirical formula	$C_5H_{10}N_2S_2$
RMM	162.3
m.p.	about 104 °C
v.p.	3.7×10^{-4} Pa at 20 °C
Solubility	In water: 3; acetone: 173; ethanol: 15; chloroform: 391; cyclohexane: 400; diethyl ether: 6, (all in g/kg at 20 °C)
Description	The pure material is a white to yellowish sulphurous smelling solid
Stability	Stable to temperatures below 35 °C; unstable above 50 °C; sensitive to moisture, not stable in acidic media
Formulations	Granules

DAZOMET TECHNICAL
*__146__/TC/M/-

1 Sampling. Take at least 100 g.

2 Identity tests

2.1 HPLC. Use the HPLC method below. The retention time of dazomet in the sample solution should not deviate by more than 10 s from that of the calibration solution.

2.2 Infrared. Prepare potassium bromide discs from the sample and from pure dazomet using 1.3 to 1.5 mg material and 300 mg potassium bromide. Scan the discs from 4000 to 400 cm^{-1}. The spectrum obtained from the sample should not differ significantly from that of the standard.

3 Dazomet

OUTLINE OF METHOD Dazomet is dissolved in acetonitrile and determined by high performance liquid chromatography on a reserved phase column using acetonitrile - water - acetic acid as mobile phase, UV-detection at 284 nm and external standardisation (Fig. 15).

REAGENTS

Acetonitrile HPLC grade
Water HPLC grade
Acetic acid glacial, HPLC grade
Dazomet of known content; purity at least 990 g/kg
Mobile phase acetonitrile - water - acetic acid, 150 + 350 + 1 (v/v)
Calibration solution

(i) Direct procedure. Weigh (to the nearest 0.01 mg) in duplicate into separate volumetric flasks (10 ml) about 10 mg (*s* mg) dazomet analytical standard. Add acetonitrile (about 60 ml) and place the flasks in an ultrasonic bath for 10 minutes. Allow to cool to room temperature, and fill to the mark with acetonitrile (Solutions C_1 and C_2).

(ii) Dilution procedure. Weigh (to the nearest 0.1 mg) in duplicate into separate volumetric flasks (100 ml) about 100 mg (*s* mg) dazomet analytical standard. Add acetonitrile (about 60 ml) and place the flasks in an ultrasonic bath for 10 minutes. Allow to cool to room temperature, and fill to the mark with acetonitrile. Transfer by pipette 5.0 ml of these solutions into separate volumetric flasks (50 ml) and fill to the mark with acetonitrile (Solutions C_1 and C_2).

* CIPAC method 2000. Prepared by the German Committee (DAPA). Chairman: W Dobrat. Based on a method supplied by BASF AG, FRG.

APPARATUS

High performance liquid chromatograph equipped with an automatic loop injector (5 µl) and a UV spectrophotometric detector operated at 284 nm
Column stainless steel, 250 × 4.0 or 4.6 mm (i. d.), packed with Nucleosil 100 C_{18}, 5 µm, or equivalent
Electronic integrator or *data system*
Ultrasonic bath

PROCEDURE

(a) Operating conditions (typical):

Stationary phase	250 × 4.0 mm (i. d.), packed with Nucleosil 100 C_{18}, 5 µm
Mobile phase	acetonitrile - water - acetic acid, 150 + 350 + 1 (v/v)
Flow rate	1.0 ml/min
Column temperature	ambient
Injection volume	5 µl
Detector wavelength	284 nm
Retention time	dazomet: about 4 min

(b) Linearity check. Check the linearity of the detector response by injecting solutions with dazomet concentrations 0.5, 1 and 2 times that of the calibration solution. Ensure that the concentrations of the solutions are in the linear range of the detector, otherwise alter the quantities weighed or the dilutions accordingly. Inject each calibration solution at least twice and determine the mean peak area to mass ratios. The single values should differ by less than 0.5 % from the mean value, otherwise repeat the calibration.

(c) Preparation of sample solution
(i) Direct procedure. Weigh (to the nearest 0.01 mg) in duplicate into separate volumetric flasks (10 ml) sufficient sample to contain about 10 mg (*w* mg) dazomet. Add acetonitrile (about 60 ml) and place the flasks in an ultrasonic bath for 10 minutes. Allow to cool to room temperature, and fill to the mark with acetonitrile (Solutions S_{A1} and S_{A2}).
(ii) Dilution procedure. Weigh (to the nearest 0. 1 mg) in duplicate into separate volumetric flasks (100 ml) sufficient sample to contain about 100 mg (*w* mg) dazomet. Add acetonitrile (about 60 ml) and place the flasks in an ultrasonic bath for 10 minutes. Allow to cool to room temperature, and fill to the mark with acetonitrile. Transfer by pipette 5.0 ml of these solutions into separate volumetric flasks (50 ml) and fill to the mark with acetonitrile (Solutions S_{A1} and S_{A2}).

(d) Determination. Inject each sample solution in duplicate and bracket a series of sample solution injections by injections of the calibration solutions as follows: C_1, S_{A1} (double injection), C_2, S_{A2} (double injection), C_1, S_{B1} (double injection), etc. Measure the relevant peak areas. Calculate the mean value of each pair of response factors bracketing the injections of the two samples and use this value for calculating the dazomet contents of the bracketed sample injections.

(e) Calculation

$$f_i = \frac{H_s}{s \times P}$$

$$\text{Dazomet content} = \frac{H_w}{w \times f} \text{ g/kg}$$

where:

f_i = single response factor
f = average response factor
H_s = peak area of dazomet in the calibration solution
H_w = peak area of dazomet in the sample solution
s = mass of the dazomet in the calibration solution (mg)
w = mass of the sample taken (mg)
P = purity of dazomet standard (g/kg)

The dazomet content is the mean value of two sample solutions.

Repeatability r = 16 g/kg at 965 g/kg active ingredient content
Reproducibility R = 41 g/kg at 965 g/kg active ingredient content

Based on a study with 20 participants and 80 values.

DAZOMET GRANULES
*146/GR/M/-

1 Sampling. Take at least 1 kg.

2 Identity tests. As for dazomet technical **146**/TC/M/2.

3 Dazomet. As for dazomet technical **146**/TC/M/3.

Repeatability r = 20 g/kg at 972 g/kg active ingredient content
Reproducibility R = 25 g/kg at 972 g/kg active ingredient content

Based on a study with 22 participants and 88 values.

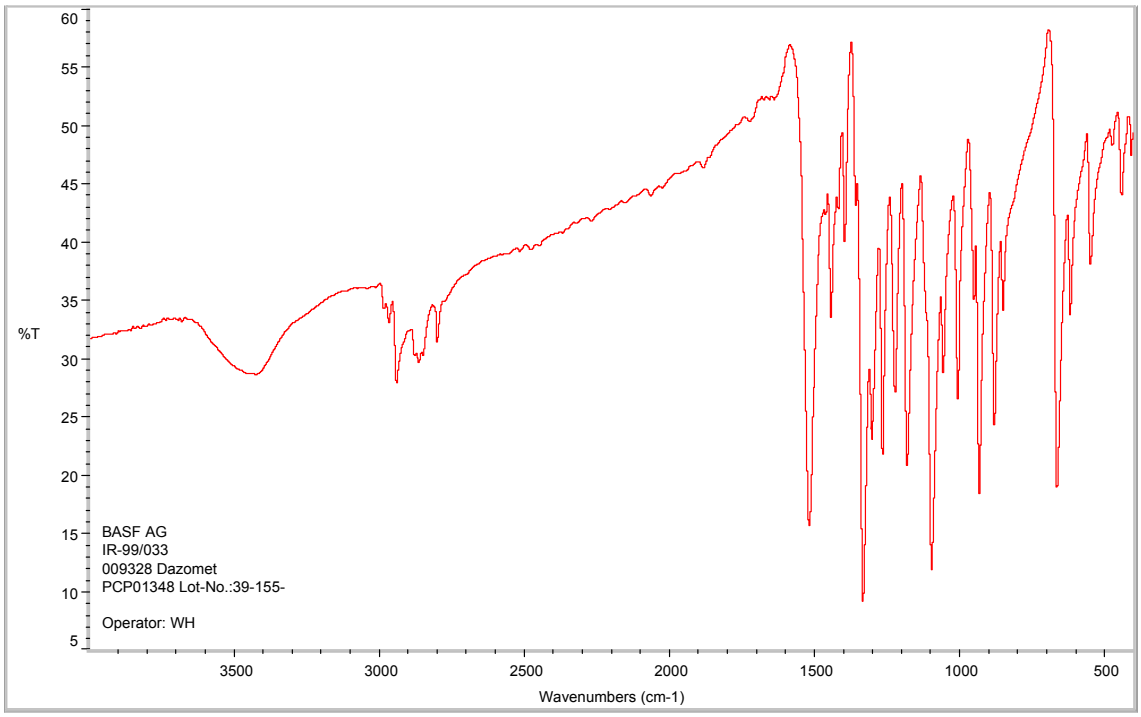


Fig. 14 IR spectrum of dazomet

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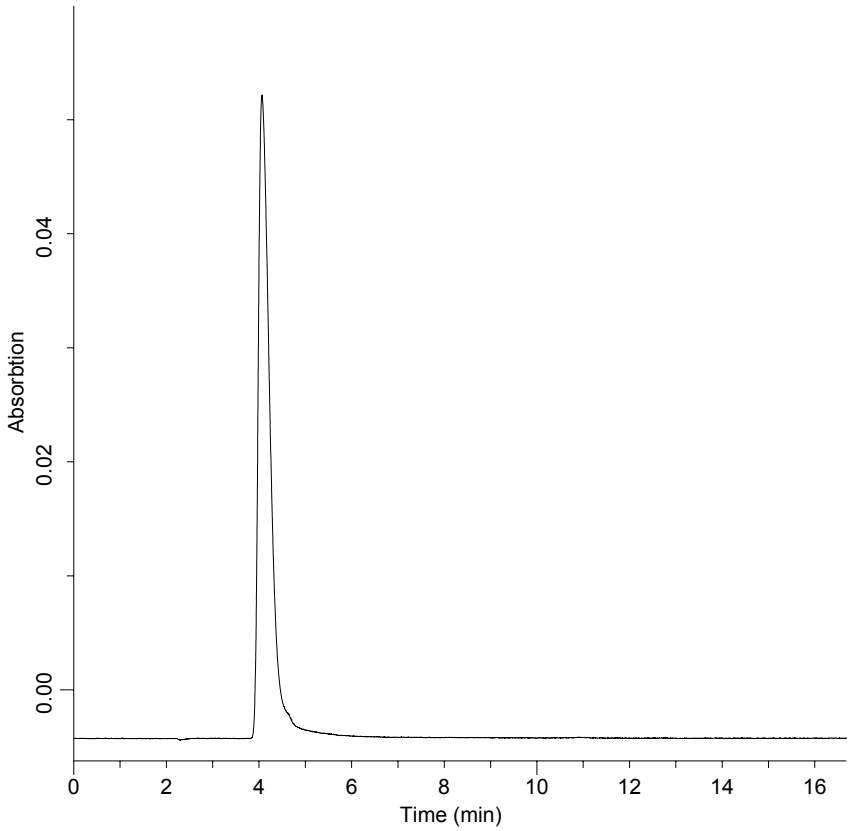

Fig. 15 Typical chromatogram of dazomet analytical standard