AR226-3113

Huntingdon Life Sciences

[redacted]

PHYSICO-CHEMICAL PROPERTIES

Company Sanitized. Does not contain TSCA CBI

Report

PHYSICO-CHEMICAL PROPERTIES

Sponsor

DuPont Specialty Chemicals
Jackson Laboratory
Chambers Works
Deepwater
NJ 08023
USA

Research Laboratory

Huntingdon Life Sciences Ltd.
Eye
Suffolk
IP23 7PX
ENGLAND

Draft: 29 October 1998
Final: 2 February 1999

CONTENTS

Huntingdon
Life Sciences

COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

[redacted]

Physico-Chemical Properties

The study described in this report was conducted in compliance with the following Good Laboratory Practice standards and I consider the data generated to be valid.

The UK Good Laboratory Practice Regulations 1997 (Statutory Instrument No 654).

EC Council Directive 87/18/EEC of 18 December 1986 (Official Journal No L 15/29).

OECD Principles of Good Laboratory Practice (as revised in 1997), ENV/MC/CHEM(98)17.

.....Anthony L. Comb.....

A. L. Comb, B.Sc., Ph.D.
Study Director
Huntingdon Life Sciences Ltd.

.....2 February 1999.....

Date

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Huntingdon Life Sciences

QUALITY ASSURANCE STATEMENT

Physico-Chemical Properties

The following have been inspected or audited in relation to this study

Study Phases Inspected	Date of Inspection	Date of Reporting
Protocol	18 August 1998	18 August 1998
Process Based Inspections		
Relative density	25 June 1998	25 June 1998
Freezing temperature	29 June 1998	29 June 1998
Water solubility	23 July 1998	28 July 1998
Surface tension	21 August 1998	21 August 1998
Boiling temperature	22 September 1998	22 September 1998
Vapour pressure	29 September 1998	29 September 1998
Explosive properties	2 October 1998	2 October 1998
Report	4 December 1998	4 December 1998

Protocol: An audit of the protocol for this study was conducted and reported to the Study Director and Company Management as indicated above.

Process based inspections: At or about the time this study was in progress inspections and audits of routine and repetitive procedures employed on this type of study were carried out. These were conducted and reported to appropriate Company Management as indicated above.

Report Audit: This report has been audited by the Quality Assurance Department. This audit was conducted and reported to the Study Director and Company Management as indicated above.

The methods, procedures and observations were found to be accurately described and the reported results to reflect the raw data.

H. Comb, B.Sc.
Principal Auditor,
Department of Quality Assurance,
Huntingdon Life Sciences Ltd.

2 FEBRUARY 1999
Date

Company Sanitized. Does not contain TSCA CBI

RESPONSIBLE PERSONNEL

[redacted]

Physico-Chemical Properties

The following staff member has reviewed this report.

T. C. Cowlyn, EurChem., C.Chem., M.R.S.C.
(Scientific Manager, Product Characterisation, Eye)

The following staff were responsible for the conduct of the work and reporting of the results.

K. Niemtus, L.R.S.C.
(Senior Scientist, Product Characterisation)

P. Woods, H.N.C.
(Scientist, Product Characterisation)

SUMMARY

A study was performed to determine a series of physico-chemical properties of [redacted]. The methods followed were amongst those described in the EEC Methods for the determination of physico-chemical properties (A1-A17), specified in the Annex to Directive 92/69/EEC and the OECD Guidelines for the Testing of Chemicals. The physico-chemical properties which have been determined in this study are detailed below, together with the result obtained for each test.

EEC method no.	OECD method no.	Test	Result
A1	102	Freezing temperature	-1°C
A2	103	Boiling temperature	101.5°C
A3	109	Relative density (D_4^{20})	1.14
A4	104	Vapour pressure	2.34×10^{-4} Pa at 25°C
A5	115	Surface tension (1 g/l solution)	21.5 mN/m at 20°C
A6	105	Water solubility	200 g/l at 10°C 248 g/l at 20°C >284 g/l at 30°C
A14		Explosive properties	Not explosive

INTRODUCTION

This study was designed to determine a series of physico-chemical properties o[redacted]. Information on physico-chemical properties is important in the assessment of the potential effects of a substance both at work and in the environment.

The study was conducted in compliance with EEC Methods for the determination of physico-chemical properties, Directive 92/69/EEC (OJ No. L383A, 29.12.92), Part A and the OECD Guidelines for the Testing of Chemicals. The physico-chemical properties investigated were: freezing temperature, boiling temperature, relative density, vapour pressure, surface tension, water solubility, and explosive properties.

The protocol was approved by Huntingdon Life Sciences Management on 7 July 1998, by the Sponsor on 17 July 1998 and by the Study Director on 13 August 1998.

The experimental phase of the study was undertaken between 24 August 1998 and 23 October 1998.

Location of study : Huntingdon Life Sciences Ltd.
Eye
Suffolk
IP23 7PX
ENGLAND

Primary data from the tests performed at Huntingdon Life Sciences, and a copy of the final report, are stored in the archives of Huntingdon Life Sciences.

TEST SUBSTANCE

Identity:	[redacted]
Chemical name:	[redacted]
Intended use:	[redacted]
Appearance:	Pale yellow slurry
Storage conditions:	Room temperature
Lot number:	3
Expiry date:	2 years from date of receipt
Purity:	[redacted]
Date received:	23 June 1998

[redacted] is a suspension at room temperature and separates into its component phases. Hence on sampling the test substance for use in each individual test, the material was warmed to 35 - 40°C in order to yield a homogenous solution.

FREEZING TEMPERATURE
(EEC Method A1, OECD Method 102)

PRELIMINARY TEST

A trial determination was conducted to determine the approximate freezing point and the nature of crystallisation. On cooling the active ingredient was observed to precipitate out of solution, however, at approximately 4°C crystals of ice were observed to form. Consequently the definitive freezing point test was performed.

METHOD

The freezing point was determined by cooling [redacted] at a controlled rate and determination of a sample temperature plateau at the crystallising point.

DEFINITIONS AND UNITS

The freezing temperature of a substance is defined as the temperature (°C) at which the phase transition from liquid to solid state at normal atmospheric pressure takes place.

APPARATUS

Seta Freezing Point Apparatus, Stanhope Seta Ltd.

Calibration: The apparatus is regularly calibrated by means of reference compounds of known freezing point.

PROCEDURE

The Dewar flask was then filled with a freezing mixture of calcium chloride/ice and the sample tube filled to a suitable depth. The sample was stirred continuously and the temperature was measured at 30 second intervals until a plateau of five constant readings were obtained.

The test was performed in duplicate using a fresh test sample on each occasion.

RESULTS

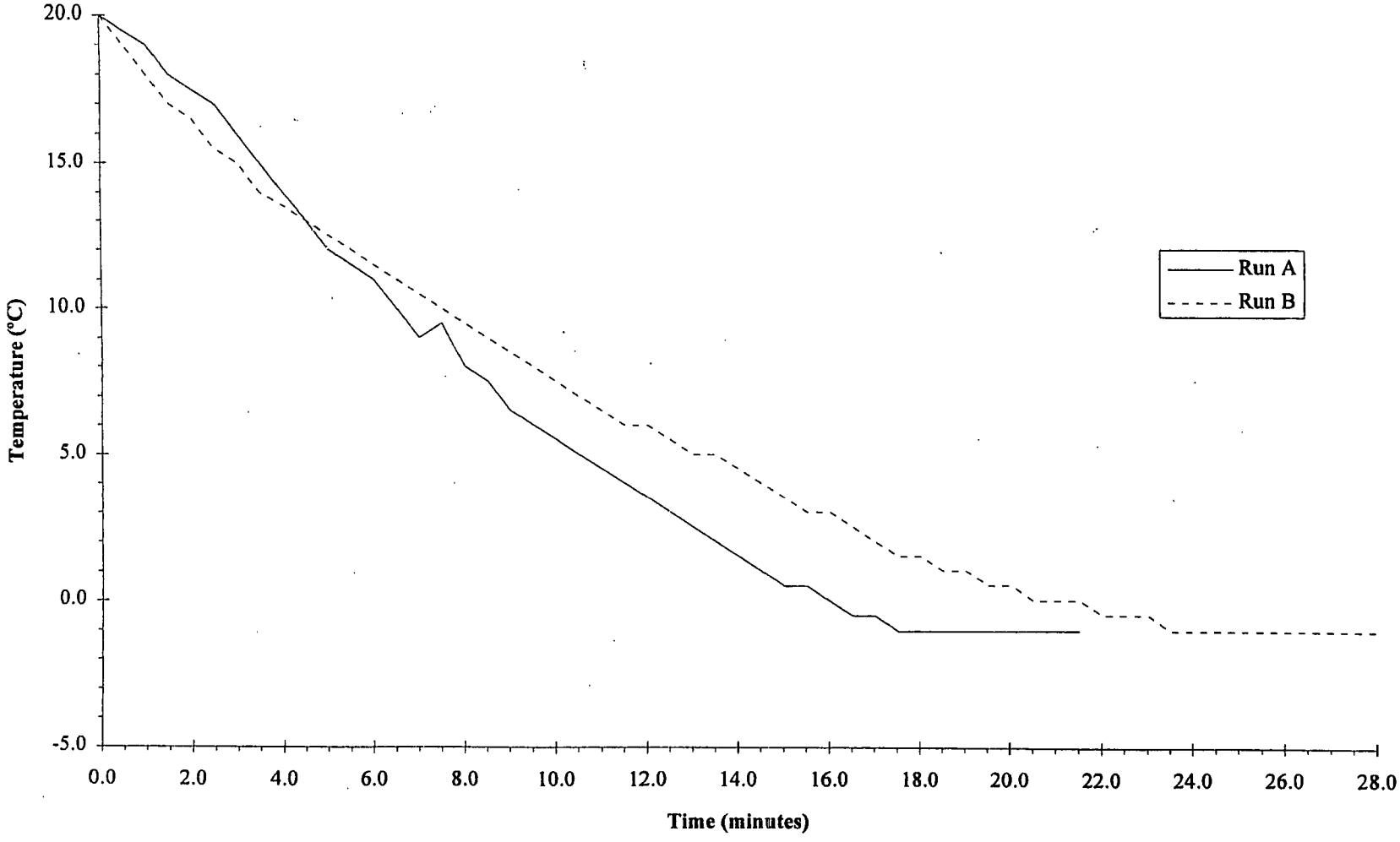
A freezing point for [redacted] of -1°C was determined (mean of -1°C and -1°C) as indicated by a plateau region in the cooling curve at this temperature (Figure1).

CONCLUSION

The freezing point of [redacted] was found to be -1°C.

FIGURE 1

Freezing point curve for [redacted]



BOILING TEMPERATURE
(EEC Method A2, OECD Method 103)

METHOD

The boiling temperature was determined by a modified Siwoloboff method.

DEFINITION AND UNITS

The normal boiling temperature is defined as the temperature (°C) at which the vapour pressure of a liquid is the same as the Standard Pressure.

APPARATUS

Melting/boiling point apparatus: Model B-545, Buchi.

Calibration: The apparatus is regularly calibrated by determination of boiling points of reference materials.

PROCEDURE

A boiling tube (3.2 mm diameter) was filled to a height of 5 - 10 mm with the sample and a boiling capillary was immersed, open end first.

A trial determination was made initially to estimate the boiling point of the substance.

The boiling tube containing the sample and the boiling capillary was inserted into the sample holder and the temperature was raised at approximately 3°C/min to about 10°C below the anticipated boiling point. The temperature was then raised at approximately 1°C/min and the temperature noted at which a rapid continuous stream of bubbles was seen emerging from the inverted open end of the capillary. This temperature represents the boiling point.

The determination was repeated until two measurements were obtained which agreed to within ±1°C of their mean (for boiling temperatures of up to 100°C) and to within ±2°C (for boiling temperatures above 100°C).

RESULTS

The results of duplicate boiling point determinations on [redacted] were as follows:

Barometric pressure: 1014 mbar

	Boiling point (°C)	
	Observed value	Pressure corrected value*
Run I	101.5	101.5
Run II	101.5	101.5

*Pressure correction

$$C_b = 0.00009\ (1013\text{-}P_b)(273 + T_b)$$

where P_b is the ambient barometric pressure (mbar)
and T_b is the observed boiling point (°C)

CONCLUSION

The boiling temperature of [redacted] was found to be 101.5°C.

RELATIVE DENSITY
(EEC Method A3, OECD Method 109)

METHOD

The relative density of [redacted] was determined relative to purified water using a pyknometer at 20°C.

DEFINITION AND UNITS

The relative density (D_4^T) of solids and liquids is defined as the ratio of the mass of a volume of substance to be examined, determined at T°C, and the mass of the same volume of water at 4°C.

APPARATUS

Analytical balance:	Model RC 210P, Sartorius Instruments
Pyknometer:	Glass, nominal 10 cm^3 capacity at 20°C, fitted with capillary stopper (BS 4699)

REAGENTS

Water:	Purified by reverse osmosis and deionising; Elga Prima/Maxima

PROCEDURE

Sample pre-treatment: The test substance was heated to 36°C in order to ensure that the material was homogenous.

Test temperature 20°C

A clean, dry pyknometer was accurately weighed (w_1) then filled to the limits of its capacity with pure water. It was then carefully stoppered without trapping air, dried and re-weighed (w_2). The procedure was then repeated with the same pyknometer using test substance. A Sub-sample of [redacted] was transferred to the test pyknometer. The pyknometer was cooled to the test temperature after which further material was added to ensure that the vessel was full to capacity. It was then carefully stoppered without trapping air, dried and re-weighed (w_3).

Two tests were performed concurrently using separate pyknometers.

Parameters:

mass of pyknometer empty (g) = w_1

mass of pyknometer + water (g) = w_2

mass of pyknometer + test substance (g) = w_3

Calculations:

mass of water to fill pyknometer (g) = $w_2 - w_1 = W_1$

mass of test substance to fill pyknometer (g) = $w_3 - w_1 = W_2$

relative density of test substance = $W_2/W_1 \times \rho_w^T = D_4^T$

where ρ_w^T is the density of water at the temperature of determination (0.998 g/ml)

D_4^T is the relative density of the test material at test temperature compared to water at 4°C

RESULTS

Parameter	Determination 1	Determination 2
w_1	14.67987	14.71513
w_2	24.26925	24.69189
w_3	25.64580	26.13326
W_1	9.58938	9.97676
W_2	10.96593	11.41813
D_4^T	1.14	1.14
$\bar{x}\ D_4^T$	1.14	

CONCLUSION

The relative density (D_4^{20}) of [redacted] was found to be 1.14.

VAPOUR PRESSURE
(EEC Method A4, OECD Method 104)

METHOD

The vapour pressure of [redacted] was determined using a vapour pressure balance.

DEFINITION AND UNITS

The vapour pressure of a substance is defined as the saturation pressure above a solid or liquid substance. At the thermodynamic equilibrium, the vapour pressure of a pure substance is a function of temperature only. The SI unit of pressure is the Pascal (Pa).

APPARATUS

The vapour pressure balance was constructed by the Department of Facilities Management at Huntingdon Life Sciences. A furnace, containing test substance, is separated from one pan of the microbalance (1 g head, C.I. Electronics) by means of a moveable shutter. This entire assembly is housed in a bell-jar which can be evacuated to a vacuum of $<10^{-5}$ Torr by means of a diffusion pump and a rotary pump connected in series. The pressure within the system is measured by Pirani and ion gauges and the temperature of the furnace by a Type K thermocouple. The signals from the microbalance and thermocouple are sent to a chart recorder.

PROCEDURE

Prior to the determination of the vapour pressure of [redacted] the water in the test substance was removed by evaporation under vacuum at 60°C. The test was subsequently performed on the dried active ingredient.

A check on the stability of the test material at elevated temperatures under nitrogen was performed by differential scanning calorimetry. No evidence of decomposition occurred below 60°C.

The microbalance was calibrated with a NAMAS calibrated 1 mg weight. It was found that 1 µg produced a deflection of 2.78×10^{-3} V.

A quantity of test substance (0.39 g) was added to the furnace. The apparatus was then assembled and evacuated to a pressure of less than 1×10^{-5} Torr (1.3×10^{-3} Pa).

After stabilisation at a given temperature, the shutter was opened to allow a stream of vapour to impact upon one balance pan. The temperature and pan deflection were recorded on a chart recorder. The trace obtained enabled the calculation of mass difference . The furnace temperature was then raised in steps of 1 to 2°C and further measurements taken.

A series of three runs were performed between temperatures of 29 to 52 °C. The same sample was used for each test, with the pressure being kept at less than 1×10^{-5} Torr (1.3×10^{-3} Pa) throughout.

CALCULATIONS

No condensation occurred so the vapour pressure is related to the observed mass difference by the relationship:

$$\text{Vapour pressure} = \frac{\Delta m\, g}{A} \qquad \text{Equation 1}$$

The vapour pressure-temperature relationship is as follows:

$$\log_{10} V_p = \frac{\text{slope}}{T} + \text{intercept} \qquad \text{Equation 2}$$

Consequently, a plot of log V_p versus 1/T(K) should be linear and by extrapolation the vapour pressure at 298.15K (25°C) can be calculated.

Glossary of terms used in equations 1 and 2

A	=	surface area of the aperture (5.952×10^{-6} m²)
g	=	acceleration due to gravity (9.813 m/s²)
Δm	=	mass difference (kg)
T	=	temperature (K)
V_p	=	vapour pressure (Pa)

RESULTS

The full results are detailed in Tables 1 to 3 and a graphical representation in Figure 2.

	Run 1	Run 2	Run 3
Correlation:	-0.99744	-0.99428	-0.99692
Slope:	-4470.6	-4155.4	-4568.8
Intercept:	11.284	10.391	11.671
Log Vp at 25°C:	-3.7100	-3.5461	-3.6524
Vapour pressure at 25°C:	1.95×10^{-4}	2.84×10^{-4}	2.23×10^{-4}

CONCLUSION

The vapour pressure of [redacted] is 2.34×10^{-4} Pa at 25°C.

TABLE 1

Test results from run 1

Temperature (°C)	Mass difference (µg)	Vapour pressure (Pa)	1/Temperature (1/K)	Log vapour pressure
52.0	2.16	0.00356	0.00308	-2.4484
51.0	1.94	0.00320	0.00308	-2.4951
49.5	1.68	0.00277	0.00310	-2.5576
48.5	1.46	0.00241	0.00311	-2.6185
47.0	1.28	0.00211	0.00312	-2.6757
46.5	1.20	0.00198	0.00313	-2.7037
45.0	1.02	0.00168	0.00314	-2.7743
44.0	0.88	0.00145	0.00315	-2.8384
43.0	0.78	0.00129	0.00316	-2.8908
41.5	0.72	0.00119	0.00318	-2.9255
39.5	0.64	0.00106	0.00320	-2.9767
38.0	0.48	0.00079	0.00321	-3.1016
36.0	0.40	0.00066	0.00323	-3.1808
35.0	0.36	0.00059	0.00325	-3.2266
33.5	0.28	0.00046	0.00326	-3.3357
32.5	0.30	0.00049	0.00327	-3.3057
30.0	0.22	0.00036	0.00330	-3.4404

TABLE 2

Test results from run 2

Temperature (°C)	Mass difference (µg)	Vapour pressure (Pa)	1/Temperature (1/K)	Log vapour pressure
29.0	0.26	0.00043	0.00331	-3.3679
29.0	0.28	0.00046	0.00331	-3.3357
30.5	0.34	0.00056	0.00329	-3.2514
32.0	0.38	0.00063	0.00328	-3.2031
33.5	0.44	0.00073	0.00326	-3.1394
35.0	0.48	0.00079	0.00325	-3.1016
36.5	0.54	0.00089	0.00323	-3.0505
38.0	0.60	0.00099	0.00321	-3.0047
39.5	0.70	0.00115	0.00320	-2.9378
41.0	0.78	0.00129	0.00318	-2.8908
42.0	0.96	0.00158	0.00317	-2.8006
44.5	1.16	0.00191	0.00315	-2.7184
45.5	1.34	0.00221	0.00314	-2.6558
47.0	1.56	0.00257	0.00312	-2.5897
49.0	2.06	0.00340	0.00310	-2.4690
51.0	2.60	0.00429	0.00308	-2.3679

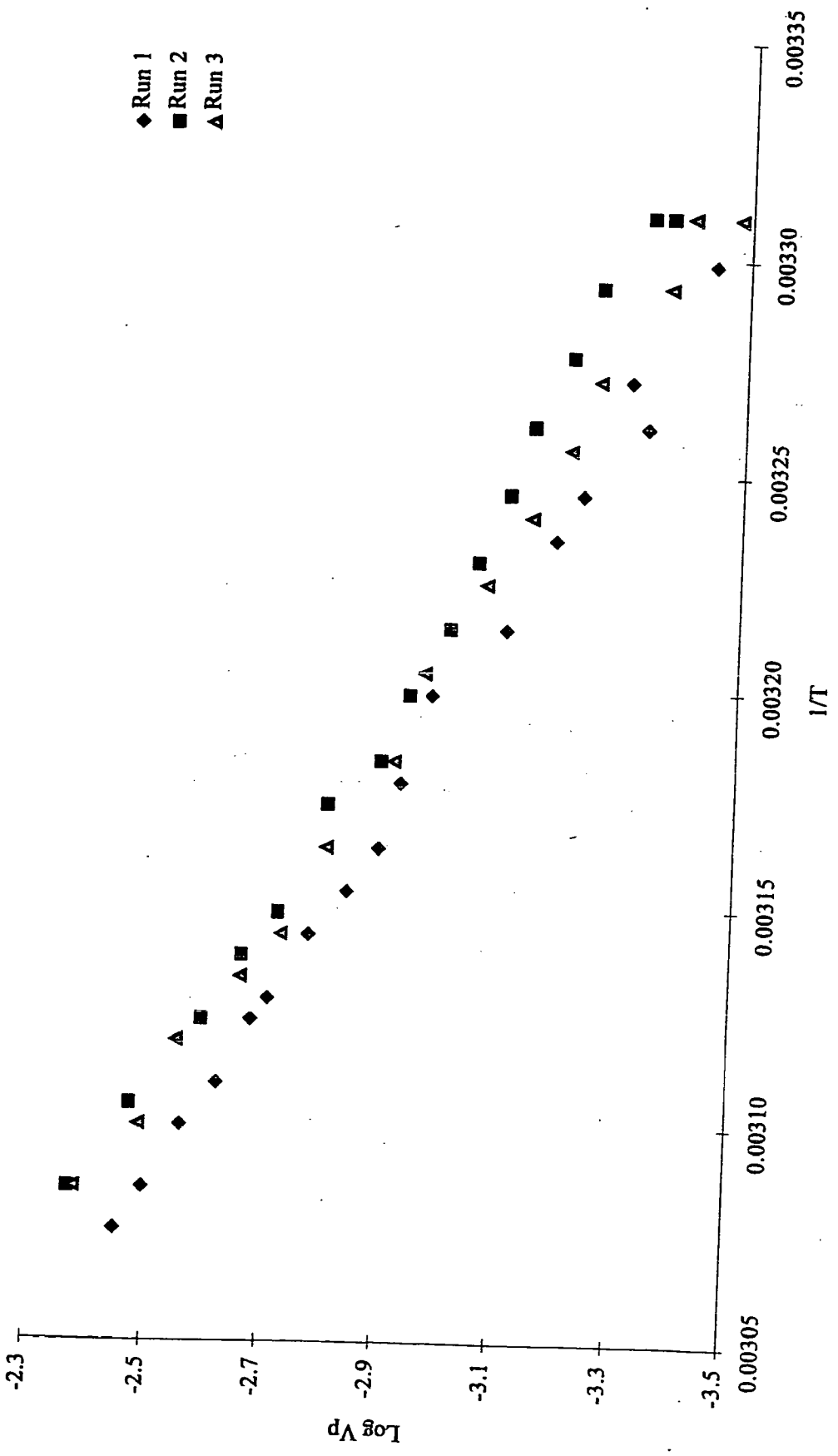
TABLE 3

Test results from run 3

Temperature (°C)	Mass difference (µg)	Vapour pressure (Pa)	1/Temperature (1/K)	Log vapour pressure
29.0	0.20	0.00033	0.00331	-3.4818
29.0	0.24	0.00040	0.00331	-3.4026
30.5	0.26	0.00043	0.00329	-3.3679
32.5	0.34	0.00056	0.00327	-3.2514
34.0	0.38	0.00063	0.00326	-3.2031
35.5	0.44	0.00073	0.00324	-3.1394
37.0	0.52	0.00086	0.00322	-3.0669
39.0	0.66	0.00109	0.00320	-2.9633
41.0	0.74	0.00122	0.00318	-2.9136
43.0	0.96	0.00158	0.00316	-2.8006
45.0	1.14	0.00188	0.00314	-2.7260
46.0	1.34	0.00221	0.00313	-2.6558
47.5	1.72	0.00284	0.00312	-2.5473
49.5	1.98	0.00326	0.00310	-2.4862
51.0	2.54	0.00419	0.00308	-2.3780

FIGURE 2

Graphical representation of runs 1 to 3



SURFACE TENSION
(EEC Method A5, OECD Method 115)

METHOD

The surface tension of an aqueous solution containing 1 g/l of the active ingredient was determined with a surface tension torsion balance using the OECD harmonised ring method.

DEFINITION AND UNITS

Surface tension (σ) is defined as the free surface enthalpy per unit of surface area and is reported in N/m (SI units) or mN/m (SI subunit).

APPARATUS

Surface tension torsion balance:	White Electrical Instrument Co., Malvern Link, Worcs.
Ring dimensions:	Ring radius 6.37 mm Wire radius 0.150 mm

Calibration: The calibration factor, ϕ_b, by which all instrument readings shall be multiplied, was determined in accordance with:

$$\phi_b = \frac{\sigma_o}{\sigma_g}$$

where

σ_o = value cited in the literature for the surface tension of water (mN/m) at the test temperature
σ_g = measured value of the surface tension of water (mN/m) at that test temperature

Literature value for the surface tension of water at 20°C (σ_o) = 72.7 mN/m.

Measured surface tension of water at 20°C (σ_g) = 72.0 mN/m.

Therefore, the calibration factor, ϕ_b = 1.010

TEST SOLUTION PREPARATION

Test substance (0.4 g) was dissolved in purified water (100 ml) to produce a 1 g/l solution, with respect to the active ingedient.

A second solution was prepared in a similar manner.

PROCEDURE

The test vessel was half filled with liquid, put on the tensiometer test platform, and raised so that the ring was 2 - 3 mm below the liquid surface. The platform was lowered to draw a lamella from the liquid surface. The maximum force which arose just before the lamella was torn off is the surface tension and was recorded. Measurements were made at intervals until a constant value (to within 0.5 mN/m) was recorded. The room temperature was monitored throughout the test.

RESULTS

Test temperature: 20°C

Solution concentration: 1 g/l with respect to the active ingredient

Initial values for the surface tension were obtained as soon as possible after transfer of the aqueous solution to the measurement vessel.

Time (minutes)	Surface tension (mN/m)			
	Measured		Calibration corrected	
	A	B	A	B
0	23.0	24.5	23.0	24.5
10	23.0	24.5	23.0	24.5
20	23.0	25.0	23.0	25.0
30	23.5	25.0	23.5	25.0

From the results there was no apparent time dependence of surface tension after transfer to the measuring vessel.

Overall mean calibration corrected surface tension = 24.0 mN/m

Harkins-Jordan* corrected value = 21.5 mN/m

CONCLUSION

The surface tension of a 1 g/l solution of [redacted] was found to be 21.5 mN/m. As the result is less than 60 mN/m, [redacted] is considered to be surface active.

*HARKINS, W.D. and JORDAN, H.F., J. Amer. Chem. Soc. 52, 1751 (1930).

WATER SOLUBILITY
(EEC Method A6, OECD Method 105)

DEFINITION AND UNITS

The solubility in water is specified by the saturation mass concentration of the substance in water, and is a function of temperature. Solubility is specified in units of mass per volume of solution. The SI unit is kg/m^3; g/l may also be used.

PROCEDURE

[redacted]s a suspension in water at room temperature, which forms a clear solution at elevated temperatures. Due to the apparent significant temperature dependence of the solubility in water, the test was conducted at 10, 20 and 30°C. The following modified procedure was employed at each temperature.

[redacted](20 ml) was added to separate Wheaton vials, which were purged with nitrogen and sealed. Duplicate samples were then stored in water baths at 10, 20 and 30°C. After 4 days, the contents of each vial were centrifuged (2000 rpm for 5 minutes) and the supernatant solutions were separated and transferred to further Wheaton vials. These were then purged with nitrogen, sealed, returned to their respective water baths and stored for a further 3 to 4 days. This equilibration and separation procedure was then repeated until no further solid material separated from solution.

Sub-samples (2 ml) of each of the final supernatant solutions were diluted to volume (100 ml) with purified water, and these solutions were then diluted further (5 or 10 ml to 100 ml, depending on the anticipated level of active ingredient in the sample) with water. Aliquots (5 ml) of the resulting solutions were pipetted into separate 10 ml volumetric flasks, to which purified water (2 ml) and 0.2M aqueous sodium hydroxide (0.5 ml) were added before diluting to volume with acetonitrile. The final solutions were then diluted (5 to 10 ml) with mobile phase for analysis by ion chromatography.

The pH of solutions were measured prior to and following the tests.

ION CHROMATOGRAPHY CONDITIONS

Instrument:	Dionex GP40 Pump Dionex PED-2 Pulsed Electrochemical Detector Perkin-Elmer ISS 200 Autosampler Dionex ASRS-II Membrane Suppressor
Column:	PLRP-S (25 cm x 4.6 mm internal diameter)
Column temperature:	Ambient
Mobile phase composition:	Aqueous solution containing 2mM ammonium hydroxide and 1mM sodium carbonate/acetonitrile (75:25 v/v)
Regenerant solution	50mN aqueous sulphuric acid
Flow rate:	10 ml/min
Injection volume:	100 µl
Detector:	Conductivity mode
Retention time:	Approximately 9 minutes

The peak observed at 9 minutes corresponds to the [redacted] which, from information supplied by the Sponsor, represents [redacted] Consequently the levels of [redacted] in the water solubility test samples will be quantified relative to this component only.

PREPARATION OF CALIBRATION

A stock calibration solution of concentration 135.6 mg/l of active ingredient was prepared by weighing test substance (108.4 mg) into a 100 ml volumetric flask and dissolving in and diluting to volume with purified water. A sub-sample (50 ml) of the stock solution was transferred to a 100 ml volumetric flask, to which 0.2M aqueous sodium hydroxide (5 ml) and purified water (20 ml) were added prior to diluting to volume with acetonitrile. Calibration solutions in the range 13.56 to 135.6 mg/l were prepared by dilutions of the final solution with mobile phase.

CALCULATIONS

The concentrations of [redacted] in the analysed solutions (C_A) were calculated from standards introduced before and after samples (bracketing standards) by the following equation:

$$C_A \text{ (mg/l)} = \frac{\text{sample peak area x standard concentration (mg/l)}}{\text{mean peak area of bracketing standards}}$$

The concentrations of Zonyl® FS-62 in the test solutions (C_B) were calculated from the following equation:

$$C_B \text{ (mg/l)} = C_A \text{ (mg/l)} \times \text{dilution factor}$$

RESULTS

The detector calibration was found to be linear over the range 0 to 135.6 mg/l of standard solutions in mobile phase with a regression coefficient of 0.9986 (Table 4, Figure 3).

Table 5 presents a summary of the results of the tests at 10 and 20°C and shows that the water solubility of [redacted] is 200 g/l at 10°C and 248 g/l at 20°C. Table 6 presents the primary data for these tests.

Samples from the test at 30°C were not analysed by the chromatography system, since on leaving aliquots of the test substance to stand for 4 days clear solutions were obtained. Consequently the solubility of [redacted] at 30°C will be reported as greater than 284 g/l - the concentration of the active ingredient in the neat test substance.

No peaks were observed in the chromatograms of the blank solutions indicating that the analytical method was free from interference.

CONCLUSION

The water solubility of [redacted] was found to be 200 g/l at 10°C, 248 g/l at 20°C and greater than 284 g/l at 30°C.

TABLE 4

Standard calibration for [redacted] by ion chromatography

Standard concentration (mg/l)	Peak area
13.56	8431
27.11	17756
54.22	35827
81.33	52060
108.4	65912
135.6	80589

Linear regression (including x = 0, y = 0) $y = 598x + 1320$, $r = 0.9986$

x = concentration
y = peak area

FIGURE 3

Standard calibration for [redacted] by ion chromatography

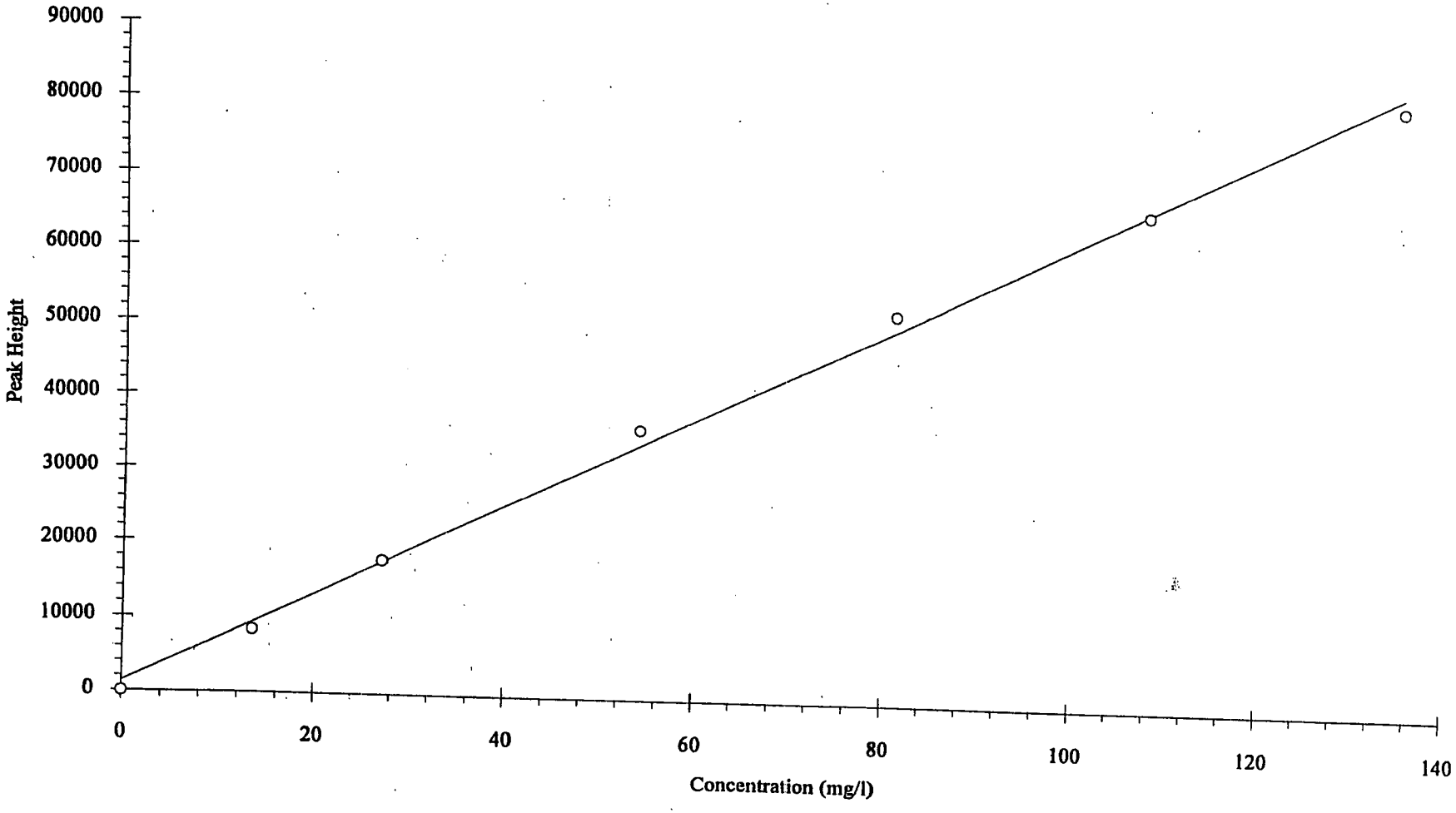


TABLE 5

Measurements of water solubility at 10 and 20°C

Test temperature (°C)	Concentration (g/l)	Mean concentration (g/l)	pH of final saturated solution
10 20	200, 200 248, 249	200 248	0.6, 0.7 0.7, 0.7

Initial pH of test substance = 0.7

TABLE 6

Ion chromatographic analysis of samples from the water solubility test for [redacted]

Sample	Peak area	C_A (mg/l)	Dilution factor	C_B (g/l)
54.22 mg/l std	34623	-	-	-
10°C sample A	61064	99.81	2000	199.6
10°C sample B	61069	99.82	2000	199.6
54.22 mg/l std	31719	-	-	-
54.22 mg/l std	34989	-	-	-
20°C sample A	39772	61,96	4000	247.8
20°C sample B	39960	62.25	4000	249.0
54.22 mg/l std	34623	-	-	-

EXPLOSIVE PROPERTIES
(EEC Method A14)

METHOD

A Koenen test apparatus was used for determination of sensitivity to heat (flame), a fall hammer for determination of sensitivity to shock and a friction test apparatus for determination of sensitivity to friction.

DEFINITION

The material is said to possess explosive properties, if a positive result is recorded on any one or all of the tests, which is defined as follows:

Thermal sensitivity (effect of flame):	An audible explosion, with the steel tube blown into three or more fragments.
Mechanical sensitivity (shock):	An audible explosion or if the material ignites.
Mechanical sensitivity (friction):	An audible explosion, crepitation or bursting into flame.

THERMAL TEST APPARATUS

Koenen apparatus made according to BAM 785-0004 with propane gas supply through calibrated flow meter. The four burners consume 3 - 4 litres/min of propane gas.

PROCEDURE OF THERMAL TEST

[redacted] was poured into new drawn steel tubes 75 mm x 24 mm i.d.. The tubes were closed with an orifice plate (6 mm or 2 mm orifice) and heated at the specified rate on the Koenen apparatus for 5 minutes or until an explosion occurred.

Tests were conducted using 6 mm orifice plates followed by three using 2 mm orifice plates.

THERMAL TEST RESULTS

Duplicate tests were performed with the 6 mm orifice plate and on each occasion the liquid boiled and extinguished the burners. This was considered to occur due to the large content of water in the test substance.

	T1 (seconds)	T2 (seconds)	Observations
2 mm orifice			
Test 1	67	300	Orange flame which turned yellow, tube recovered intact
Test 2	63	300	Orange flame which turned yellow, tube recovered intact
Test 3	53	300	Orange flame which turned yellow, tube recovered intact

Where T1 = time from start of test to flame from nozzle
T2 = time from start of test to explosion, or end of test (300 seconds)

MECHANICAL SENSITIVITY (SHOCK) TEST APPARATUS

Impact hammer apparatus made according to BAM 782-0005 with 10 kg weight and specified sample dies.

PROCEDURE OF SHOCK TEST

40 mm^3 of [redacted] was put in the die assembly and placed on the anvil in the drop hammer apparatus. A 10 kg weight was released from a height of 0.4 m. The test was performed six times using a different sample and die assembly on each occasion.

SHOCK TEST RESULTS

Replicate	1	2	3	4	5	6
Result	N	N	N	N	N	N

Where N = no evidence of explosion or decomposition.

CONCLUSION

[redacted]s not explosive.