DIETHYL SULPHATE

CONTENTS

SECTION I

PRODUCT CHARACTERISTICS AND SPECIFICATIONS

1.1 General details

Appearance	Colourless, oily liquid, Darkens with age.
CAS. No	64-67-5
Chemical formula	C4H10O4S
Molecular weight	154.18
Odour	Ethereal odour,
Density at 23 deg.C	1.1803
Boiling point at 760 mm pressure	208 deg.C (decomposition)
Flash point	121 deg.,C (open cup)
Refractive index	nd20 = 1.40037
Solubility	Soluble in organic solvents like alcohol and ether Sparingly soluble in water

1.2. Specification

Colour on APHA scale Max.	20
Assay (G.C. % Area) or Hydrolysis by Sodium Hydroxide(%Min)	99.00% min.
Specific gravity at 30 C, (Maximum limits of impurities % by mass)	1.173 + 0.002
Free Acidity as H_2SO_4	0.2

Storage and handling

Diethyl sulphate must be stored in phenolic lined steel drums.

Aluminium, zinc, galvanised iron, lead, nickel or copper and its alloys should not be used. A centrifugal pump is recommended for transfer operations.

PRODUCT APPLICATIONS

Application sector	Dyes and intermediates Pharmaceuticals
Ethylating agent	Used as an ethylating agent in the manufacture of aromatic and aliphatic ethers, amines, amides, esters and imides for dyestuff, pharmaceutical and flavour synthesis.

The following compounds containing reactive methylene groups can be ethylated by Diethyl sulphate.

a) malonic ester
b) acetoacetic ester
c) ethyl acetoacetic ester
d) cyanoacetoacetic ester

Phenyl acetic ester can also be ethylated by Diethyl sulphate using sodium in ether.Benzene cyanide, butyl cyanide, acetophenone isobutylmethyl ketone and camphor can be ethylated by Diethyl sulphate using sodamide in an inert solvent.

The ethyl esters of lauric, cinnamic and stearic acid can be prepared from the acids and Diethyl sulphate. Ethyl benzoate, oxalate, cinnamate stearate and phthalate can be made at 145 deg.C from the sodium salts of respective acids and Diethyl sulphate.

Inorganic esters: It is claimed that esters of weaker inorganic acids such as ethyl nitrite, ethyl sulphite, ethyl mercaptan and ethyl sulphide of high purity can be readily made and in good yield. Other inorganic esters which could be made from Diethyl sulphate are mixtures of mono and Diethyl phosphates and ethyl isocyanate.

Amines: In the preparation of ethylated amines, Diethyl sulphate works very easily, intensely and efficiently.Either one or both ethyl groups may be made use of, forming mono and disubstituted amines, eg. mono and diethyl anilines. N--ethylpiperidine can be similarly synthesised.

Besides, the dye-intermediates that are obtained from the ethylation of amines, Diethyl sulphate offers many others such as diethyl benzene, diethyl acetoacetate as well as variousdyes like Rhodamine-B, diethyl morpholine and chrysophenine.

Other commercial ethylations:

KCN is ethylated to give ethyl isocyanate.Potassium thiocyanate gives ethyl thiocyanate.Morphine and diethyl sulphate in presence of sodium ethoxide and ethanol gives ethyl morphine.

Diethyl sulphate and benzyl magnesium chloride give 70-75% yield of N-propyl benzene.Similarly, N-ethyl benzene can also be obtained in fairly good yield.

In particular,a 60% yield of 1-butyne can be obtained by reacting sodium acetylide and diethyl sulphate in liquid ammonia on the basis of the equation.

$(C_2H_5)_2\ SO_4 + NAC = CHC_2H_2.H_5.C = CH + Na\ C_2H_5.SO_4$

Ethyl carbazole can be prepared by fusing carbazole with KOH and adding diethyl sulphate to the fused mass.

2-Ethoxybenzamide can be obtained by reacting salicylamide with Diethyl sulphate in aqueous alcoholic NaOH solution. O-ethyl caprolactum can be obtained by direct action of Diethyl sulphate on caprolactum in benzene solution.N-ethyl caprolactum can be made simultaneously.

INDIAN MANUFACTURERS

* The Dharamsi Morarji Chemical Company Ltd.,
Mumbai-400 001.

Dharamsi Morarji Chemical Co. Ltd., Ambernath, Maharashtra. has announced expansion of the Diethyl sulphate plant capacity to 6000 tonnes per annum at the Roha plant to meet the increasing demand, particularly in the global market.

IMPORTS/EXPORTS DETAILS

4.1. Present Import level : Around 600 tonnes per annum

Sampe of individual imports of Diethyl Sulphate

Period 2002

Name of the Importers	Quantity in tonnes	Value in Rs.	Country	Date	Port
Desai Specialty Chemicals	16.000	370391	Malaysia	16.04.2002 to 19.04.2002	Mumbai

4.2. Present export level : Around 35 tonnes per annum

SECTION - V

PRICE DETAILS

Present price for Diethyl sulphate	:	Rs.48/- per kg
Taxes and duties	:	Extra as applicable

SECTION VI

INDIAN DEMAND

DES is predominantly used in the Pharmaceutical and Dyestuff sector.

	Demand (in tonnes per annum)
Dyestuff sector	2300
Pharmaceutical sector	1200
Total demand	3500

Estimated growth rate in demand:-

Pharmaceuticals & Drug Intermediates	9 to 10% per annum
Dyestuff	5 to 6% per annum

SECTION VII

BROAD OUTLINE OF MANUFACTURING PROCESS

Diethyl sulphate is an important by-product of the indirect process for the hydration of ethylene.

Diethyl sulphate, was traditionally prepared from ethyl alcohol and sulphuric acid. The method employed was to distill a mixture of about 2-4 parts by weight of sulphuric acid with one part by weight of ethyl alcohol.The distillate separates into two layers, the upper of which is aqueous alcohol and the lower crude diethyl sulphate as a yellow liquid.

Diethyl sulphate has been made by dropping a mixture of ethyl alcohol and concentrated sulphuric acid on to sodium sulphate at 55 to 65 deg.C, or by heating ethyl alcohol with an alkali metal chlorosulphonate.

A method of making Diethyl Sulphate by passing an inert gas through a hot reaction mixture of ethyl alcohol and sulphuric acid and condensing Diethyl Sulphate and water from the gas was patented.

Other methods of making Diethyl sulphate are as follows.

1. Diethyl sulphate has been prepared by extracting a mixture of fuming sulphuric acid and ethyl alcohol with chloroform and distilling the extract under vacuum. Distillation of the same reaction mixture under high vacuum also gives Diethyl sulphate.

2. Crude Diethyl sulphate may be obtained by reacting gaseous sulphur trioxide with ethyl alcohol.The product so obtained is a mixture of Diethyl sulphate and Ethyl isothionate. There are patent claims for the preparation of pure Diethyl sulphate by this route.

In another variant, the alcohol is treated with sulphur trioxide in a diluent such as carbon tetrachloride and the reaction mixture is then distilled in the presence of a dehydrating agent such as sodium sulphate or phosphorus pentoxide.

Diethyl sulphate is one of the products when ethylene is absorbed in sulphuric acid.

Preparation from Ethyl Alcohol and Chloro sulphonic acid

Absolute alcohol and chlorosulphonic acid with a catalyst are charged to a reactor maintained at sub zero temperature by brine cooling refrigeration in jacket, while stirrer is kept on.The HCl (gas) formed as by-production is absorbed in water in graphite heat exchanger absorbers and the HCl solution is sold in the market.

On completion of addition of reaction, the mass is digested. The mass is transferred to distillation kettle and then subjected to vacuum for distilling off diethyl sulphate.

The process reaction is as follows.

$$2C_2H_5\,OH + HSO_3Cl \longrightarrow (C_2H_5)_2\,SO_4 + HCl + H_2$$

Source of technology

* Indian Institute of Chemical Technology,
  Tarnaka
  Hyderabad

Major plant and machinery suppliers

Name of the equipment	Name of the supplier
Reactors	Chemitherm Plants & Systems P. Ltd., 30, Anandha Street Alwarpet, Chennai-600 018 Texel Fabricators Pvt. Ltd., 335, Sidco Industrial Estate, Ambattur, Chennai-600 098, Tamil Nadu
Distillation column	Chemac Equipments Pvt. Ltd. Regd. Office & Factory: M.J.D'Souza Compound, Safed Pool, Saki Naka Mumbai-400 072 Hydrabad Met Chem. Pvt. Ltd., 34, C.I.E., Phase II, Gandhinagar, Opp. IDPL Colony Hyderabad-500 037

Condensors	Sieco Engineers Pvt. Ltd. #25, Bommasandra Industrial Area, Hosur Road, Bangalore-562 158 Universal Heat Exchangers Ltd., Pollachi Road, Malumachampatti Post, Coimbatore-641 021
Steam boiler	Sri Ranga Industries SF, 739, Ramraj Nagar, Goldwins, Coimbatore-641 014 Firetech Boilers Pvt. Ltd. No.211, 2nd Cross, 38th Main, B.T.M. Layout 2nd Stage, Bangalore-68

SECTION VIII

RAW MATERIALS REQUIREMENTS, UTILITY AND AVAILABILITY

Raw material requirements

Basis one tonne of Diethyl Sulphate

Rectified Spirit	1725 litres
Chlorosulphonic acid	1200 kgs
Catalyst (Thionyl Chloride)	Small quantity

Utility requirements

Basis	One tonne of Diethyl Sulphate
Installed power	220 HP for the project of capacity 600 tonnes per annum.
Fuel	800 litres

Raw material availability

Source of raw material supply

Name of the raw material	Name of the supplier
Chlorosulphonic acid	Sree Rayalaseema Hi Strength Hipo Ltd., Andhra Pradesh The Andhra Sugars Ltd., Andhra Pradesh Dharamsi Morarji Chemicals Ltd.,Maharashtra

SECTION IX

GLOBAL SCENARIO

Global demand Around 60000 tonnes

Global growth rate in demand 2 to 3% per annum

Global manufacturers:

* Union Carbide Corporation,USA.
* Coyne Chemical, USA
* Jarchem Industries Inc., USA

SECTION X

ECONOMIC CAPACITY, PROJECT COST AND PROFITABILITY PROJECTIONS

Economic capacity : 600 tonnes per annum

Project cost Rs.196 lakhs

A**ssessment of project cost**

1. Land

S.No.	Description	Cost Rs.in lakhs
1.1	Cost of land of one acre at Rs.5.5 lakh per acre	5.5
1.2	Cost of levelling, laying internal roads/fencing and compound wall	0.55
	Subtotal	6.05

2. Building

S.No.	Description	Cost Rs.in lakhs
2.1	Factory building of area 320 sq.m. at Rs.3200/sq.m.	10.24
2.2	Non-factory building of area 50 sq.m.at Rs.4500/sq.m.	2.25
	Subtotal	12.49

3. Cost of Plant & Machinery

S.No.	Description	Cost Rs.in lakhs
3.1	Cost of basic plant and machinery	73
3.2	Instrumentation and control	5.48
3.3	Pipelines and valves	7.3
3.4	Structurals for erection	3.65
	Subtotal	89.43
3.5	Octroi, excise duty, sales tax, etc.at 12%	10.73
3.6	Packaging and insurance charges (2%)	1.79
3.7	Transportation charges (2%)	1.79
3.8	Machinery stores and spares (2%)	1.79
3.9	Foundation charges (2%)	1.79
3.10	Installation charges (2%)	1.79
	Total cost of plant and Machinery	109.11

4. Technical know-how fees Rs.2.00 lakhs

5. Miscellaneous fixed assets

S.No.	Description	Cost Rs.in lakhs
5.1.	Electrification	4.1
5.2.	Steam boiler and auxillaries	4.1
5.3.	Water storage tank, borewell etc.	0.76
5.4.	Fuel storage tank	1.14
5.5.	Laboratory equipment	0.76
5.6.	Office machinery & equipment	1.54
5.7.	Material handling equipment, packaging machinery, weigh balance, etc.	1.54
5.8.	Diesel generator	4.43
5.9.	Effluent treatment	1.32
	Total	19.69

6. Preliminary & Pre-operative expenses:

S.No.	Description	Cost Rs.in lakhs
6.1.	Preliminary expenses	1.23
6.2.	***Pre-operative expenses:-***	
6.2.1	Establishment	0.76
6.2.2	Rent rates and taxes	0.76
6.2.3	Travelling expenses	0.88
6.2.4	Interest and commitment charges on borrowings	9.2
6.2.5	Insurance during construction period	1.34
6.2.6	Other preoperative expenses and deposits	-
6.2.7	Interest on deferred payment	-
	Total	14.17

7. Provision for contingency Rs.10.91 lakhs

8. Working capital margin Rs. 21 lakhs

9. Total project cost **Rs.195.39 lakhs**

Say Rs.196 lakhs

10. Means of Finance

Promoter's contribution Rs.78 lakhs
Term loan from financing institutions Rs.118 lakhs
Total project cost Rs.196 lakhs

11. Financial statements

Cost of production

A Variable cost	Rs. in lakhs
Raw material and utilities	143.62
Spares and maintenance	6.55
Selling expenses	14.4
Total variable cost (A)	164.57
B Fixed cost	
Salaries and wages	18
Interest on term loan and working capital loan	32.58
Depreciation	13.77
Administrative expenses	8.64
Total fixed cost (B)	72.99
C. Total cost of production (A+B)	237.56
D. Selling price per kg. (in Rupees)	48
E. Annual sales turnover	288
F. Net profit before tax (E-C)	50.44
G. Breakeven point in %	59%

SWOT ANALYSIS

Strength	Well established technology in India
Weakness	Use restricted to a few regions in the country
Opportunity	Exports
Threat	Involves product like Chlorosulphonic acid which are hazardous

SECTION XII

FACTORS INFLUENCING THE POSTION FOR A NEW INDUSTRY

AND RECOMMENDATIONS

Demand level :

The demand level for the DES is likely to go up steadily in the coming years both in the Indian and the global market. Strong case exists to create additional capacity for DES in the country from the point of view of demand supply scenario.

Recommendations :

From the point of view of the demand supply scenario, new DES project can be favourably considered.

There is only one DES producer and the proximity of the proposed plant to the consuming units would be advantage.