CHAPTER 9

DEVELOPMENT OF SEPARATION SCHEMES

9.1 Introduction

In chapter 8 interaction parameters for four selected solvents with OCT1 and MBK were determined. These will now be used to synthesize distillation processes to illustrate the ability of the solvents to separate these two azeotropic components.

9.2 Solvent concentration

In extractive and azeotropic distillation the solvent concentration is one of the most important variables. It has a significant influence on process economics but the actual concentration varies considerably from case to case.

A number of different amounts must therefore generally be considered. For extractive distillation equi-molar to four mols of extractive agent per mole of hydrocarbon mixture should be used. The actual amount is usually set by economic trade offs. In azeotropic distillation the composition is usually fixed by the composition of the azeotrope (Berg, 1969:57).

In order to get a feeling for concentration effects, the following table was prepared. As before the ratio of OCT1 to MBK is 0.719 : 0.281. (The relative volatility is that of OCT1 to MBK.)

Table 9.1

Effect of solvent concentration on the relative volatility

Solvent ratio[56]	**Methanol**	**DMF**	**MXEA**	**kerosol 200**
0	1.012			
1	1.849	1.807	1.863	0.722
2	2.628	2.457	2.614	0.652
3	3.311	2.928	3.193	0.621
4	3.897	3.277	3.642	0.603
5	4.398	3.541	3.995	0.592

The figures show that the relative volatility increases favourably with increasing solvent concentration. However, at high concentrations the increase is no longer so marked and the increase in operating and equipment costs as a result of the higher solvent concentration should start to catch up with the improved relative volatility. *The* optimum can only be determined from economic considerations.

9.3 Design parameters

The design variables in this section were fixed for all cases in order to be able to compare the solvents on an equal basis. They do not necessarily represent actual requirements.

The feed stream to the plant is 57200 tons/year (8.8 m^3/hr). On a mol basis it contains 0.719 1-octene with the remainder being 2-hexanone. It is assumed to originate from another distillation unit and will therefore be at its boiling point.

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[56] Moles of solvent per moles of OCT1 / MBK.

The amounts and temperatures of solvent streams depend on the specific solvent and varies from case to case. In extractive distillation different amounts of solvents were tested with a feed temperature of 60 °C. In azeotropic cases the amount of solvent is usually fixed by the composition of the new azeotropes being formed and was assumed to be fed at its boiling point. It became evident that, in the azeotropic cases, increasing the amount of solvent has little effect on the number of stages required.

The general concepts of the different designs are in agreement with the discussion on the topics of extractive and azeotropic distillation given in chapter 3. In extractive distillation the heavy solvent is fed near the top of the column with enough stages remaining above the feed point to ensure that none of it goes overhead. In azeotropic distillation the solvent is either fed along with the feed stream or near the top of the column. The simulations showed that for these cases the latter option gives significantly better results and was used in all cases.

In the azeotropic cases a reflux ratio of 2 was used constantly. Since the slope of the cost versus reflux ratio line is typically relatively flat above the optimum the effect of a higher ratio than the optimum should not be so marked (Douglas, 1988:443). In the extractive cases a reflux ratio of 6 was used because simulations lead the author to suspect a higher minimum reflux for the specific extractive case.

Within these constraints the number of stages and feed point locations were determined by examining the composition and relative volatility profiles within the columns.

The diameters of the columns were determined by specifying the standard Sulzer M250X packing for the columns. The heights were estimated by assuming a constant HETP of 0.3 m.

The specifications are that ca 97.5% of OCT1 be recovered with a purity of 99.99% +. These specifications are applied with respect to OCT1 and MBK in the first main column and were met in all cases.

9.4 Solvent recovery

In the case of methanol, being a volatile solvent, virtually 100% of it is taken overheads with the 1-octene. MXEA forms an azeotrope with both OCT1 and MBK, and the majority of it is found in the bottoms product with MBK. The heavy nature of DMF also necessitated that more than the azeotropic amount be used, with the majority of it found in the bottoms product as well.

For the three azeotropic solvents the solvent recovery process consists of a water wash followed by a water solvent separation distillation. The azeotropic systems all produce a 1-octene / solvent azeotrope as overheads product. The solubility data indicates that the solvents are all infinitely soluble with water. 1-octene losses should be minimal due the fact that it is virtually insoluble in water (0.000410 weight %) (Riddick et al, 1984:191,657,688).

Methanol and DMF can easily be recovered from a water wash since they do not form azeotropes with water. 2-methoxyethanol, however, does form a 15.3% weight aqueous azeotrope. In this case it may be possible to use this water azeotrope as the solvent stream because the presence of the water may enhance the separation of 1-octene and 2-hexanone.

Previous simulations regarding washes with water, methanol, olefins and azeotropes showed that the liquid-liquid simulations contain significant errors and without the benefit of experimental interaction parameters not even the boundary of the two phased region is established with any accuracy. For these reasons the solvent recovery sections were not modelled for the three azeotropic solvents.

The extractive solvent recovery process was modelled because it does not use water and will therefore be accurate. These simulations therefore include a second column which receives the bottoms from the first main column and separates the octene from the kerosol.

Figures 9.1 and 9.2 illustrate the effective schemes being synthesized.

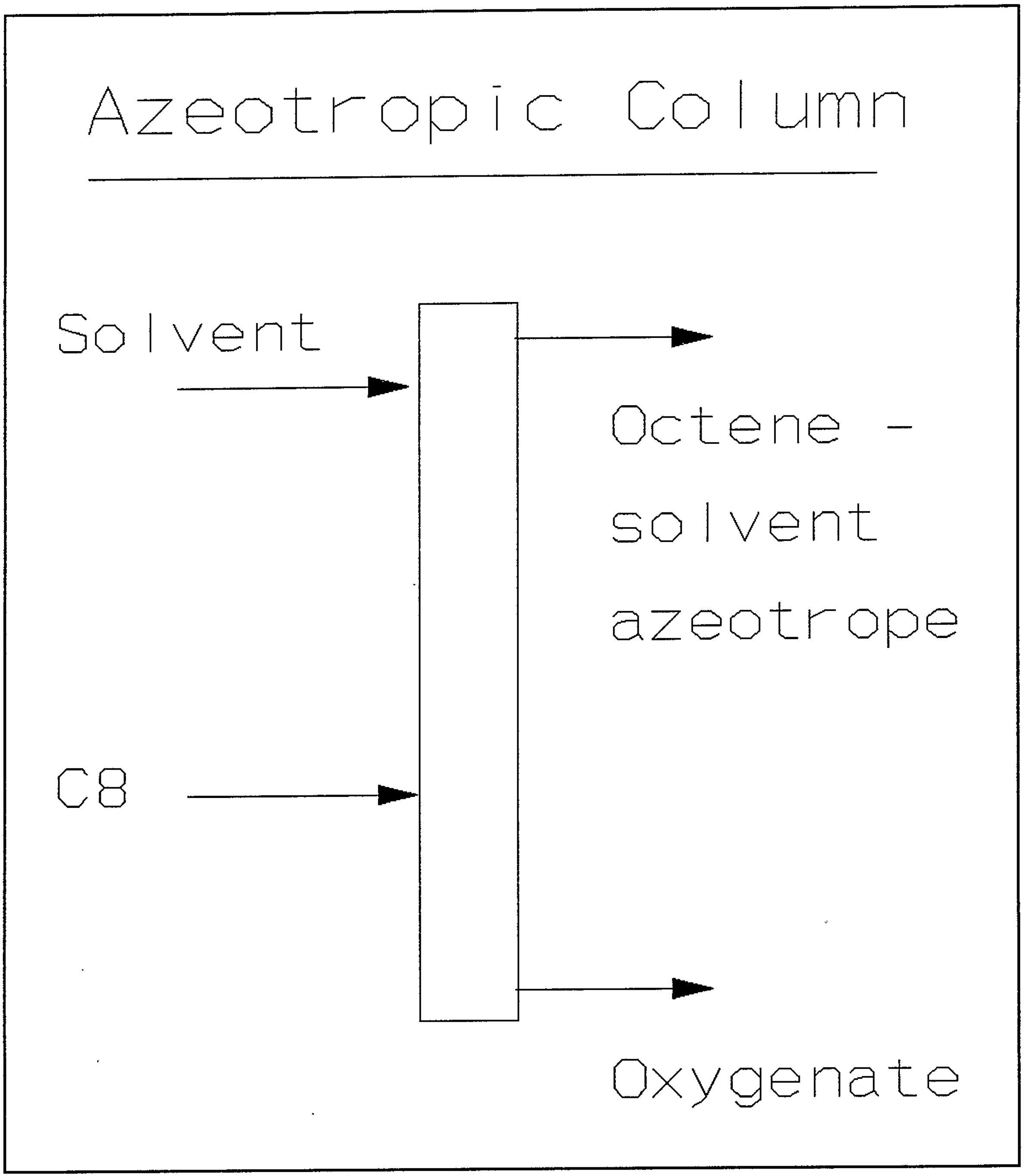

Figure 9.1: Azeotropic column.

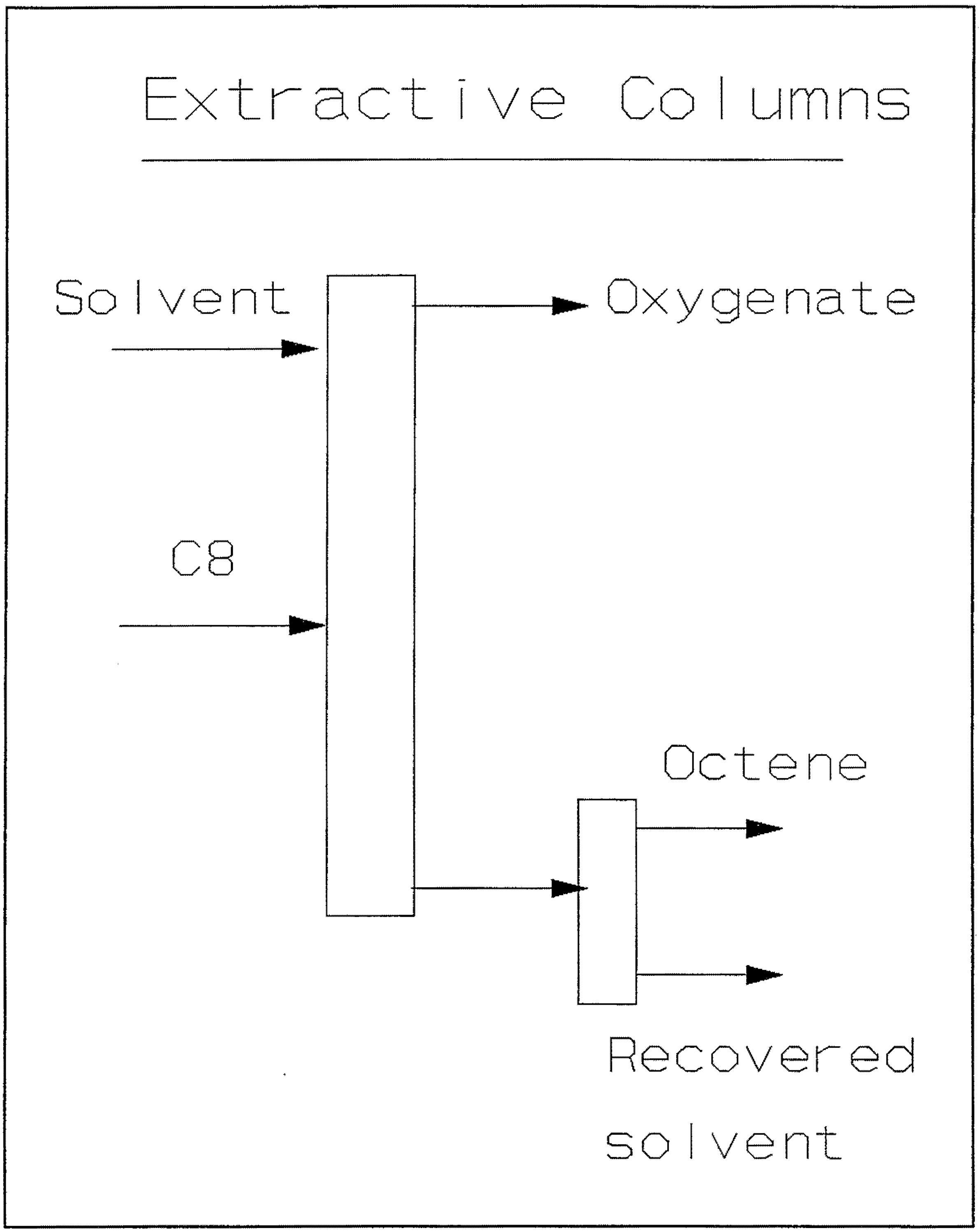

Figure 9.2: Extractive columns.

9.5 Simulation results

The results for the extractive distillation options with kerosol 200 are produced in table 9.2.

Table 9.2: Results for Extractive Options			
Solvent feed (kg-mol/hr)	**180**	**240**	**300**
Solvent feed stage:	4	4	4
Total number of stages:	93	81	72
Feed stage:	31	24	19
Column packed height (m):	27.3	23.7	21.0
Column ID (m):	2.0	2.2	2.4
Reboiler duty (million KJ/HR)	15.3	18.3	21.3

The kerosol recovery columns are as follows:

Table 9.3: Results for Extractive Recoveries			
Solvent feed (kg-mol/hr)	**180**	**240**	**300**
Total number of stages:	20	20	20
Feed stage:	8	8	8
Column packed height (m):	5.4	5.4	5.4
Column ID (m):	2.3	2.4	2.4
Reboiler duty (million KJ/HR)	17.0	17.1	17.2

The recovery columns specifically do not heat the kerosol in the bottoms to its boiling point (as this would cause a flash effect in the main columns if the solvent is recycled and wastes heat). The purpose of the recovery column is just to drive off the 2-hexanone. As a result the duties do not vary much.

The results from the three azeotropic processes are as follows:

Table 9.4: Results for Azeotropic Options

Item	**Methanol**	**DMF**	**MXEA**
Solvent feed (kg-mol/hr)	386	74	156
Solvent feed stage:	4	4	4
Total number of stages:	35	65	48
Feed stage:	10	52	35
Column packed height (m):	9.9	18.9	13.8
Column ID (m):	3.3	1.2	1.4
Reboiler duty (million KJ/HR)	45.5	6.34	9.94

(The relevant PRO/II input files can be found in the appendix.)

9.6 Process costs

In order to be able to compare the different options on the same basis, the column and heat exchanger costs must be estimated. These two costs are often considered to constitute the major equipment costs. The Sasol Process Evaluation Group provided internal cost correlations for the columns and reboilers for this type of plant.

9.6.1 Cost correlations

As a first step in the calculation of the column cost, the area is estimated:

$$\text{Area} = 3.1416 * \text{ID} * (\text{TT} + (0.6901 * \text{ID})) * 1\text{e-}6 \quad (\text{m}^2)$$

where ID is the column inside diameter in mm and TT the tangent to tangent length in mm (roughly the column height).

The price is then estimated from:

Price1 = 10431 + 299.3 * Area

in terms of July 1979 rands. The price must be updated using escalation indices and multiplied with the proper material factor:

October 1993 Price = Price1 * 1919 / 326 * 2.13

where 2.13 is the factor for SS316L.

The accuracy of this correlation is given to be 20%, but for a similar recent plant the average error was in the region of 6%

Based on historical data for a similar plant the column internals are estimated to be about 22% of the vessel cost.

The reboiler areas are estimated from Q=UAdT with Q the reboiler duties given from the simulations, dT about 15K and U about 750 $W/m^2/K$ (Coulson & Richardson, volume 61, 1983:513).

The base price is then obtained from:

Price1 = 5741.55 + 59.5297 * Area

with the area in m^2. Again this must be escalated using:

October 1993 price = Price 1 * 1919/326 * 3.57

where 3.57 is the historical material factor for SS316L.

The heat exchanger estimates are very accurate.

9.6.2 Resulting costs

The costs (in thousands of rands are) are shown in the table below. The column costs include costs for internals as well. The costs for the kerosol options include the cost for the first main column only.

Table 9.5: Actual major equipment costs, Rands			
Option	**Column**	**Heater**	**Total**
kerosol 200, 180	985	593	1736
kerosol 200, 240	958	686	1808
kerosol 200, 300	942	779	1892
methanol	738	1526	2490
DMF	500	316	898
MXEA	457	428	973

9.7 Discussion

The optimum amount of kerosol 200 is shown to lie below the ratio of 3:1. Lower ratios are not included in the table because severe simulation convergence problems start to appear. This indicates that actual operation at these lower ratios will practically probably be difficult. In any case it is clear that extractive distillation can not compare with azeotropic distillation for this system.

While methanol is a convenient solvent with a low price and recovery facilities (water removal) available at Sasol, its low capacity (a high concentration of methanol required) and the resulting high operating costs has a very detrimental effect on its economics.

Economically DMF appears to be the best solvent of the four listed. It is commercially used and therefore not too risky. Two solvent recovery systems will however be required since DMF is found in both column product streams. This will make the process more expensive.

The figures for MXEA also look favourable, but it is also present in both column product streams as it forms an azeotrope with both octene and 2-hexanone. Two separate recovery columns will therefore also be required. More MXEA is also required than DMF.

Figure 9.3 shows that the total costs (divided by 10) and reboiler duties also favours DMF.

It thus appears as that the azeotropic solvent which forms the lowest solvent content azeotrope with octene will be the most economical.

9.8 Conclusion

The separation options shown here do indeed verify that all four solvents are able to effect a high degree of separation. The economics of the different options differ significantly.

The main purpose of these processes are to demonstrate the practical separating potential of the solvents. The processes represented here do not necessarily represent optimum configurations and solvent stream flows. In order to accomplish this, accurate VLE data is required for all non ideal component interactions involved (including water recovery systems). The plant feed stream in this chapter also contains only the two components under investigation. Economic values for all the streams should actually be incorporated into a simulation with optimization. Energy integration options within the plant section itself as well as with other sections will also almost surely be available.

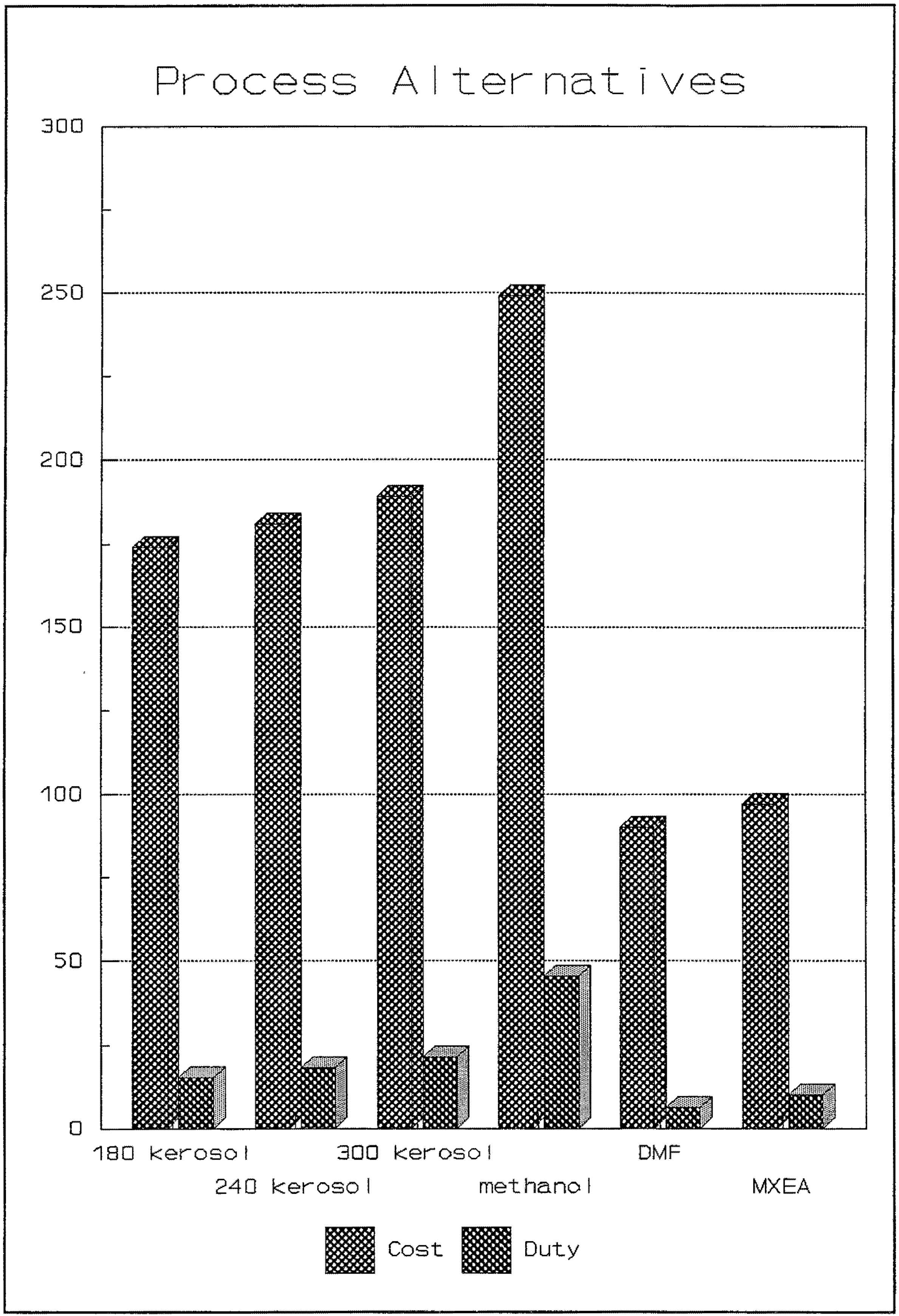

Figure 9.3: Relative costs of the options (Rands/10).