From Laboratory Research to Process Design: A Case Study of Tributyl Phosphate

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Executive Summary

This report presents a case study of the development of tributyl phosphate (TBP) at Prasol Chemicals Limited, following the process from the closing stages of research and development (R&D) through to commercial process design. It is based on an internship undertaken at the company in which I studied the process. Following literature review and experimentation to optimise the synthesis process, the product TBP and recovered *n*-butanol were analysed at the laboratory through Karl Fischer titration, gas chromatography, and acid-base titration. Three validation batches prepared at a gram scale confirmed that the optimised laboratory synthesis produced TBP consistently, at purities of 98.87–99.80% and yields of 72.62–75.51%. However, the recovered *n*-butanol had a mean water content of 13.61%, exceeding the limit acceptable for reuse. This necessitates further research on purifying the recovered solvent. Process design serves as the intermediate stage that scales the product from R&D to manufacturing. At the design stage, a material balance established a throughput of 11.528 MT per MT of product, of which liquid effluent constitutes 6.955 MT, and an energy balance established a design steam rate of 600 kg/hr. The scale of the effluent stream indicates that the balance must be tested against the intended plant's treatment capacity, and a full environmental assessment must be prepared. Based on the material balance, a block flow diagram, process flow diagram, and piping and instrumentation diagram were created. The next stage would be a pilot plant producing TBP at the kilo scale, followed by commercial manufacturing at a ton scale; TBP did not reach these stages during the internship.

1. Introduction

About the Company

Prasol Chemicals Limited is an Indian specialty chemicals manufacturer headquartered in Navi Mumbai, Maharashtra, with dedicated research and development facilities and integrated manufacturing operations. Established in 1992, the company develops and manufactures specialty chemicals, especially acetone and phosphorus-based derivatives. It is involved in sectors such as agrochemicals, pharmaceuticals, home and personal care, paints and coatings, lubricant additives, mining, oil and gas, and performance chemicals.[1]

From Research and Development to Manufacturing

The production of specialty chemicals involves a number of interconnected stages, including research and development (R&D), process design, and manufacturing. R&D focuses on designing and optimising the chemical process, process design or projects constitutes the intermediate stage that scales up the laboratory process, and manufacturing involves carrying out large-scale production at the plant. Successful commercial production depends not only on the underlying chemistry proposed, but also on the ability to design a process that is technically feasible, economically viable, safe, and environmentally responsible.

Moving from R&D to manufacturing presents numerous technical challenges due to differences in scale, equipment, environmental impact, waste management, and operational safety. These challenges necessitate careful process development and engineering design before industrial production can be realised.

Scope of this Report

This report will follow a case study of the development of tributyl phosphate (TBP) conducted during my internship at Prasol Chemicals Limited. It focuses on the transition from late-stage R&D to process design. While the general stages of pilot-scale and commercial manufacturing are introduced to provide context, the development of TBP had not yet progressed to these stages. Owing to the proprietary nature of industrial process development, confidential information has been omitted or generalised where appropriate.

2. The Product

Structure

Tributyl phosphate (TBP) is an organophosphorus compound with molecular formula $C_{12}H_{27}O_4P$. Structurally, TBP consists of a central phosphoryl group (P=O) bonded to three *n*-butoxy groups ($–OC_4H_9$), giving the condensed structural formula $PO(OC_4H_9)_3$. The central phosphorus atom adopts a tetrahedral geometry with one phosphorus–oxygen double bond and three phosphorus–oxygen single bonds. The skeletal structure of TBP is shown in Figure 1.

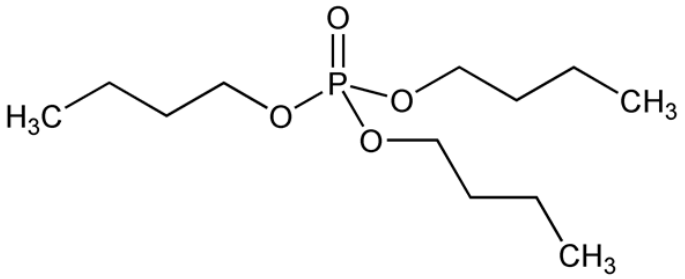


Figure 1. The Skeletal structure of TBP. Vertices represent carbon atoms, while hydrogen atoms attached to carbon are omitted for clarity.[2]

Formation

Tributyl phosphate is synthesised through the esterification of phosphorus oxychloride with *n*-butanol:[3]

$$POCl_3 + 3C_4H_9OH \rightarrow PO(OC_4H_9)_3 + 3HCl$$

Esterification is a chemical reaction in which an alcohol reacts with an acid or an acid derivative to form an ester.[4] During the reaction, each chlorine atom in $POCl_3$ is successively substituted by an *n*-butoxy group through nucleophilic substitution, producing TBP as the desired product and hydrogen chloride (HCl) as a by-product. In this mechanism, the oxygen atom of the *n*-butanol acts as the electron-rich nucleophile, utilizing its lone pairs to attack the central phosphorus atom of $POCl_3$, which serves as the electron-poor electrophile.

Although TBP can also be produced through the esterification of phosphoric acid (H_3PO_4) with alcohols, this route is generally not employed for industrial production as H_3PO_4 is significantly less reactive than $POCl_3$, resulting in poor thermodynamic yield and the production of mono- and di-butyl phosphate.[5]

Properties

The principal physical and chemical properties of tributyl phosphate relevant to its manufacture, handling, and industrial applications are summarised in Table 1.

Table 1. Selected physical and chemical properties of tributyl phosphate.[5]

No.	Property	Value
1	Molecular weight	266.31 g/mol
2	Appearance	Colourless liquid
3	Odour	Odourless
4	Density	0.978 g cm^{-3} at 20°C

No.	Property	Value
5	Boiling point	289 °C at 760 mm Hg
6	Flash point	146 °C
7	Water solubility	Slightly soluble
8	Organic solubility	Soluble in a number of organic solvents
9	Toxicity	Toxic if ingested or inhaled, suspected carcinogen

These characteristics are important in industrial processes. Firstly, the three non-polar *n*-butyl groups reduce the overall polarity of the molecule, making it only slightly soluble in water while allowing excellent solubility in many organic solvents. This property is useful in purification. Secondly, the relatively large molecular size and phosphate ester structure, consisting of a highly polar P=O bond, contribute to its high boiling point and low volatility, properties that facilitate safe handling and storage. In addition, as TBP presents a number of safety hazards, appropriate safety measures are required during its manufacture, storage, and transport.

Industrial Applications

TBP is well suited for a variety of industrial applications. Its highly polar P=O bond allows it to act as a Lewis base and bind to uranium and plutonium ions. Hence, it is used to extract nuclear fuel from wastes in nuclear reprocessing.[6] Secondly, because of its central phosphorus atom, TBP serves as an effective flame retardant in hydraulic fuels and textiles. Furthermore, it is used as an antifoaming agent in adhesives and detergents.[7]

3. Research and Development

Objective and Stages

The decision to develop a given chemical process originates from market demand, with buyers' requirements determining which product Prasol undertakes to produce. Once a product is identified, the objective of R&D is to develop a process for its synthesis that meets required quality specifications while maximising yield, ensuring process safety, minimising production costs, and complying with environmental and regulatory requirements.

R&D proceeds through three stages:

1. The first is a literature review, conducted to survey existing patents and published methods for the target compound to avoid infringement and identify possible modifications to established processes.
2. The second stage consists of experimentation, in which the candidate process is run at laboratory scale. Variables such as reaction temperature, reagent stoichiometry,

reaction time, and solvent quantity are systematically adjusted to optimise the process against the objectives above.

3. The third stage is validation, in which multiple batches are manufactured through the optimised process across a series of independent runs to confirm that the process yields consistent results. The batches are as large as possible at a laboratory scale. In the case of TBP, three such validation runs were completed; a report documenting these results was passed to the projects team as the basis for process design and scale-up.

The literature review and experimental stages, along with the precise synthesis process, have been excluded from this report for proprietary reasons. However, the analysis of the validation batches has been covered.

Analytical Techniques

The optimisation and validation of a chemical manufacturing process require accurate analytical techniques to assess product quality. Three techniques were employed across the R&D experiments and subsequent validation of TBP: Karl Fischer titration, gas chromatography, and acid–base titration.

Karl Fischer Titration

Karl Fischer (KF) titration is a volumetric technique used to determine water content. It was applied to assess the water content of recovered *n*-butanol. The titration was conducted twice to minimise random error. The method relies on the Karl Fischer reaction, in which iodine is reduced by sulfur dioxide in the presence of water, a reaction that proceeds only when water is present:[8]

$$I_2 + SO_2 + H_2O \rightarrow 2HI + SO_3$$

In practice, n-butanol was dissolved in a karl-fischer solvent and the karl-fischer solution containing I_2 and SO_2 was added automatically. A pyridine-free karl-fischer solution was used to avoid the odour and toxicity associated with pyridine. The endpoint was detected electrometrically as an excess of free I_2 results in the potential difference between electrodes rapidly dropping to near zero.

The water content is calculated according to the following equation:[8]

$$Water\ content\ (\%) = \frac{V \times F}{m \times 10}$$

where V is the volume of Karl Fischer reagent consumed (mL), F is the reagent factor (mg H_2O mL^{-1}), and m is the mass of the sample (g). The factor refers to the amount of water that reacts with 1 mL of the reagent, determined through calibration.

KF titration was preferred to alternative methods such as loss-on-drying as it is highly selective for water and is not affected by the evaporation of volatile organic compounds. This is important for samples such as recovered *n*-butanol and TBP, where conventional drying

methods may overestimate water content by simultaneously removing the organic solvent. An image of the equipment used for KF titration is shown in Figure 2.



Figure 2. The Karl Fischer titrator used for analysis

Gas Chromatography

Gas chromatography (GC) is a separation technique used to determine the composition of volatile mixtures. It was applied to assess the purity of recovered n-butanol and TBP. The sample is vaporised in a heated injector and carried through a capillary column by an inert carrier gas which constitutes the mobile phase. Nitrogen was used as the mobile phase. Separation occurs by differential partitioning between this mobile phase and the stationary phase coating the column: components with a greater affinity for the stationary phase are retained longer, so each component emerges at a characteristic retention time. Identification is made by comparing retention times against known standards.

Detection was carried out using a flame ionisation detector (FID), in which the column effluent is combusted in a flame sustained by hydrogen and air. Organic compounds entering the flame are pyrolysed to form ions, producing a current proportional to the mass of carbon entering the detector. The percent composition of a component is then determined by normalising the area of the peak:[9]

$$Composition\ (\%) = \frac{A_i}{\Sigma A} \times 100$$

where A_i is the peak area of the component of interest and ΣA is the sum of all peak areas in the chromatogram.

Karl Fischer titration was performed before GC analysis as excess water can interfere with chromatographic analysis by affecting sample vaporisation, reducing column performance, and potentially damaging the stationary phase. GC was preferred to alternative techniques such as high-performance liquid chromatography (HPLC) as both n-butanol and TBP are sufficiently volatile and thermally stable to be analysed in the gas phase. An image of the equipment used for GC is shown in Figure 3.



Figure 3. The gas chromatograph used for analysis

Acid-Base Titration

Acid–base titration was used to determine the alkalinity of TBP, a measure of its capacity to neutralise acids. Alkalinity arises from basic species retained from the synthesis, and therefore provides a measure of product purity. The sample was neutralised by hydrochloric acid of 0.1 N concentration, which served as the titrant. Phenolphthalein was used as the indicator, the endpoint being taken as the point at which the pink colouration of the alkaline solution is discharged, corresponding to the transition range of pH 8.2–10.0.[10]

Alkalinity is calculated using the following equation:[11]

$$Alkalinity\ (\%) = \frac{V \times N \times 40}{m \times 10}$$

where V is the volume of titrant consumed (mL), N its normality, and m the mass of the sample (g). Alkalinity is expressed as a mass percentage of equivalent sodium hydroxide, i.e., the titrate is equivalent to a solution of NaOH having that mass percentage in neutralising the acid.

The titration was performed manually, with the titrant delivered from a burette and the endpoint judged visually. This introduces several sources of systematic error absent from instrumented methods: parallax in reading the burette meniscus, variability in drop size near the endpoint, and the subjectivity of visual colour detection. Potentiometric titration would become the appropriate method if a more precise value was necessary.

Results

Karl Fischer Titration

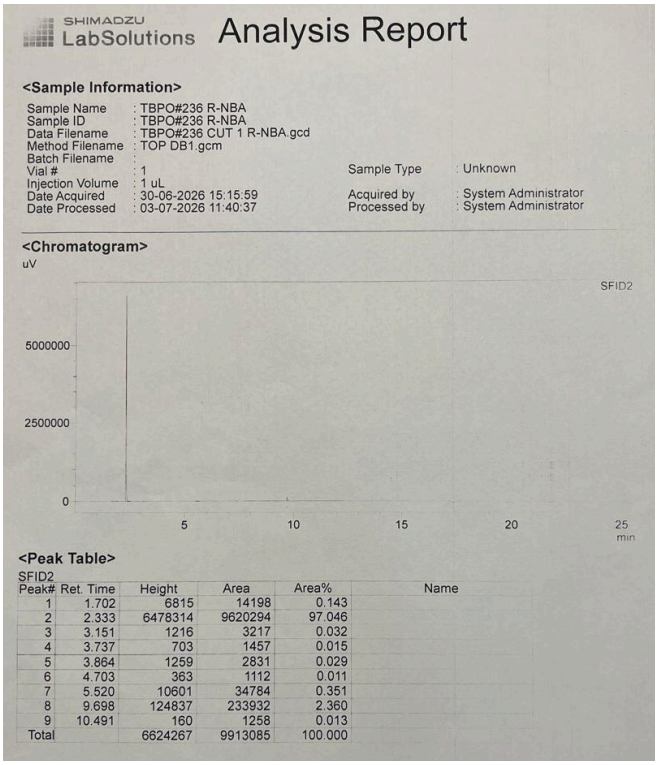
The reagent factor (F) was found to be 4.1182 through calibration. The results of the KF titration for two samples of recovered *n*-butanol are shown in Table 2. The mean water content of the recovered *n*-butanol was found to be 13.61%. This value exceeded the acceptable limit for reuse in TBP production. Thus, further research is required on reducing the water content and increasing the purity of the recovered solvent. At the time of writing, investigations into this process optimisation are ongoing.

Table 2. The results of the KF titration for a sample of recovered *n*-butanol

Trial No.	*Mass of n-butanol (g)*	*Volume of Karl Fischer reagent (mL)*	*Water content (%)*
1	0.1535	5.10	13.48
2	0.2159	7.26	13.74

Gas Chromatography

The results of GC analysis of a sample of recovered *n*-butanol from the first validation batch are shown in Figure 4. The purity of the sample of recovered *n*-butanol, represented by the normalised area of the largest peak, was found to be 97.046%. A secondary peak representing the product TBP had a composition of 2.360%. However, the high water content risks reducing the accuracy of the GC results.



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Analysis Report

<Sample Information>

Sample Name	: TBPO#236 R-NBA		
Sample ID	: TBPO#236 R-NBA		
Data Filename	: TBPO#236 CUT 1 R-NBA.gcd		
Method Filename	: TOP DB1.gcm		
Batch Filename	:		
Vial #	: 1	Sample Type	: Unknown
Injection Volume	: 1 uL		
Date Acquired	: 30-06-2026 15:15:59	Acquired by	: System Administrator
Date Processed	: 03-07-2026 11:40:37	Processed by	: System Administrator

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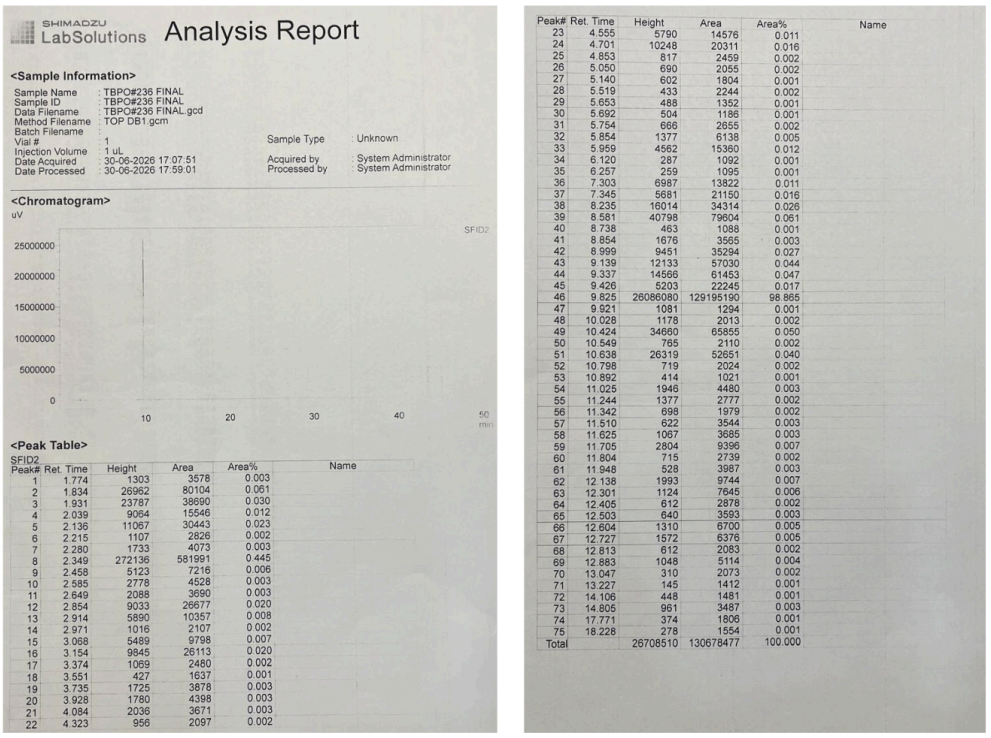
<Peak Table>

SFID2

Peak#	Ret. Time	Height	Area	Area%	Name
1	1.702	6815	14198	0.143	
2	2.333	6478314	9620294	97.046	
3	3.151	1216	3217	0.032	
4	3.737	703	1457	0.015	
5	3.864	1259	2831	0.029	
6	4.703	363	1112	0.011	
7	5.520	10601	34784	0.351	
8	9.698	124837	233932	2.360	
9	10.491	160	1258	0.013	
Total		6624267	9913085	100.000	

Figure 4. Gas chromatogram for a sample of recovered *n*-butanol. The chromatogram graph displays detector response as a function of retention time, while the accompanying peak table lists the retention times, peak areas, and relative area percentages of the detected components. Names have been excluded but significant peaks are identified later.

The results of GC analysis for a sample of the product TBP from the first validation batch are shown in Figure 5. The purity of the sample of TBP was found to be 98.865%. This value, though lower than the purity of the other two validation batches, was sufficient for Prasol's requirements.



SHIMADZU LabSolutions Analysis Report

<Sample Information>

Sample Name	: TBPO#236 FINAL		
Sample ID	: TBPO#236 FINAL		
Data Filename	: TBPO#236 FINAL.gcd		
Method Filename	: TOP DB1.gcm		
Batch Filename	:		
Vial #	: 1	Sample Type	: Unknown
Injection Volume	: 1 uL		
Date Acquired	: 30-06-2026 17:07:51	Acquired by	: System Administrator
Date Processed	: 30-06-2026 17:59:01	Processed by	: System Administrator

<Chromatogram>

<Peak Table>

SFID2

Peak#	Ret. Time	Height	Area	Area%	Name
1	1.774	1303	3578	0.003	
2	1.834	26962	80104	0.061	
3	1.931	23787	38690	0.030	
4	2.039	9064	15546	0.012	
5	2.136	11067	30443	0.023	
6	2.215	1107	2826	0.002	
7	2.280	1733	4073	0.003	
8	2.349	272136	581991	0.445	
9	2.458	5123	7216	0.006	
10	2.585	2778	4528	0.003	
11	2.649	2088	3690	0.003	
12	2.854	9033	26677	0.020	
13	2.914	5890	10357	0.008	
14	2.971	1016	2107	0.002	
15	3.068	5489	9798	0.007	
16	3.154	9845	26113	0.020	
17	3.374	1069	2480	0.002	
18	3.551	427	1637	0.001	
19	3.735	1725	3878	0.003	
20	3.928	1780	4398	0.003	
21	4.084	2036	3671	0.003	
22	4.323	956	2097	0.002	
23	4.555	5790	14576	0.011	
24	4.701	10248	20311	0.016	
25	4.853	817	2459	0.002	
26	5.050	690	2055	0.002	
27	5.140	602	1804	0.001	
28	5.519	433	2244	0.002	
29	5.653	488	1352	0.001	
30	5.692	504	1186	0.001	
31	5.754	666	2655	0.002	
32	5.854	1377	6138	0.005	
33	5.959	4562	15360	0.012	
34	6.120	287	1092	0.001	
35	6.257	259	1095	0.001	
36	7.303	6987	13822	0.011	
37	7.345	5681	21150	0.016	
38	8.235	16014	34314	0.026	
39	8.581	40798	79604	0.061	
40	8.738	463	1088	0.001	
41	8.854	1676	3565	0.003	
42	8.999	9451	35294	0.027	
43	9.139	12133	57030	0.044	
44	9.337	14566	61453	0.047	
45	9.426	5203	22245	0.017	
46	9.825	26086080	129195190	98.865	
47	9.921	1081	1294	0.001	
48	10.028	1178	2013	0.002	
49	10.424	34660	65855	0.050	
50	10.549	765	2110	0.002	
51	10.638	26319	52651	0.040	
52	10.798	719	2024	0.002	
53	10.892	414	1021	0.001	
54	11.025	1946	4480	0.003	
55	11.244	1377	2777	0.002	
56	11.342	698	1979	0.002	
57	11.510	622	3544	0.003	
58	11.625	1067	3685	0.003	
59	11.705	2804	9396	0.007	
60	11.804	715	2739	0.002	
61	11.948	528	3987	0.003	
62	12.138	1993	9744	0.007	
63	12.301	1124	7645	0.006	
64	12.405	612	2878	0.002	
65	12.503	640	3593	0.003	
66	12.604	1310	6700	0.005	
67	12.727	1572	6376	0.005	
68	12.813	612	2083	0.002	
69	12.883	1048	5114	0.004	
70	13.047	310	2073	0.002	
71	13.227	145	1412	0.001	
72	14.106	448	1481	0.001	
73	14.805	961	3487	0.003	
74	17.771	374	1806	0.001	
75	18.228	278	1554	0.001	
Total		26708510	130678477	100.000	

Figure 5. Gas chromatogram for a sample of TBP. The chromatogram graph displays detector response as a function of retention time, while the accompanying peak table lists the retention times, peak areas, and relative area percentages of the detected components. Names have been excluded but significant peaks.

Acid-Base Titration

Prior to conducting the titration, the HCl solution was standardised and its normality was calculated to be precisely 0.1165 N. The volume of titrant required was 0.6 mL, and the mass of the TBP sample was measured to be 4.6252 g. The alkalinity was calculated to be 0.06%. Due to the methodology of manual titration, the volume is less precise compared to alternatives such as potentiometric titration. However, the estimated alkalinity value was very low, below the threshold at which the product would be considered impure. Hence, the uncertainty introduced by manual endpoint detection does not affect the conclusion drawn, and a manual determination was sufficient.

Validation

The results of the analysis of three validation batches are shown in Table 3:

Table 3. Validation data for TBP

Batch no.	*236/116*	*238/116*	*240/116*
1. Reaction parameters and raw materials			
$POCl_3$ (g)	300	300	300

Batch no.	*236/116*	*238/116*	*240/116*
n-butanol (g)	774	774	774
Temp of addition of $POCl_3$ (°C)	5-10	5-10	5-10
Addition time (h)	4.5	4.5	4.5
Maintaining temp (°C)	20-25	20-25	20-25
Reaction time (h)	5	5	5
2. GC of reaction mass			
Unreacted *n*-butanol (%)	50.12	61.29	63.45
TBP (%)	37.79	38.28	36.31
3. Solvent recover phase			
Recovered *n*-butanol (g)	342	335	311
Purity of recovered *n*-butanol (%)	97.05	98.12	98.97
4. Washing			
Mass of 20% NaOH (g)	723	787	741
Organic layer (g)	770	766	804
Crude TBP (%)	TBP = 44.19 *n*-butanol = 54.27	TBP = 41.26 *n*-butanol = 58.47	TBP = 42.74 *n*-butanol = 56.45
5. Final product			
Pure TBP (g)	262	252	254
Purity TBP (%)	98.87	99.61	99.80
Percentage yield	75.51	72.62	73.41

The mass of raw materials was scaled as high as possible at a laboratory scale. *n*-butanol was added in excess; hence, GC analysis of the reaction mass showed that the composition of unreacted solvent ranged from 50.12% to 63.45% (along with 36.31–38.28% TBP). This indicated that a substantial proportion of the excess alcohol remained available for recovery. The purity of the recovered solvent ranged from 97.05% to 98.97%, but the water content was too high for Prasol standards, as indicated by KF titration. Further research is required and ongoing to improve the quality of recovered *n*-butanol. Purified product mass ranged from 252 to 262 g, purity from 98.87% to 99.80%, and yield from 72.62% to 75.51%. The

deviation across batches was sufficiently small and yield and purity were sufficiently high for Prasol standards.

4. Process Design

Objective and Stages

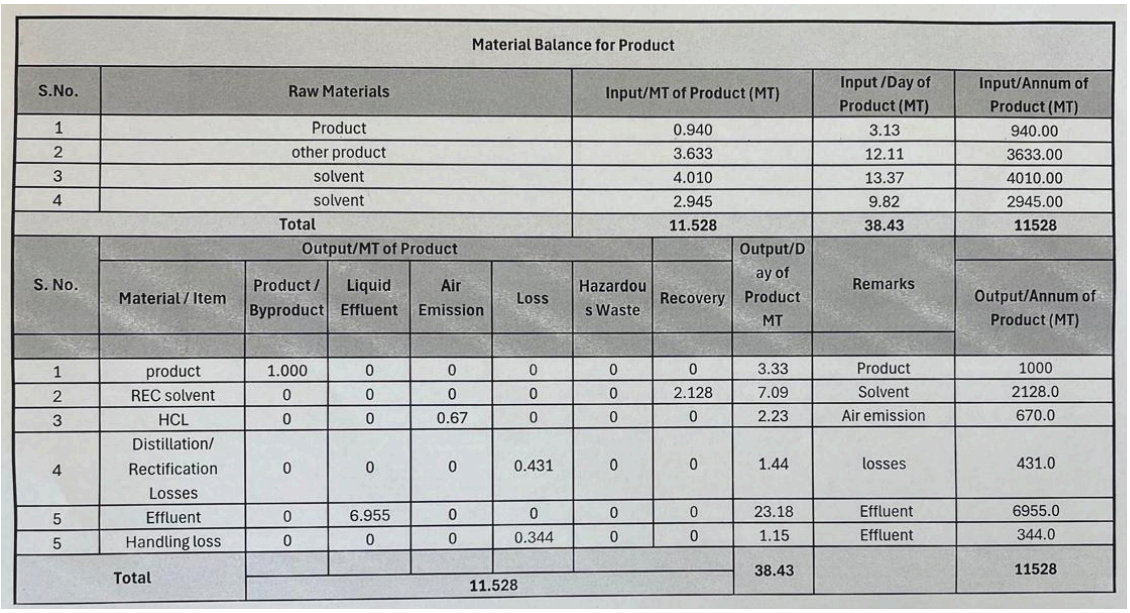
The objective of process design is to transform a laboratory-developed chemical process into a safe, economical, and commercially viable manufacturing process. The validation batches of TBP are at the largest scale achievable in the laboratory but remain at the gram scale. The process design is based on a report presenting the validation data along with the precise synthesis process for a product. The first stage of process design involves the preparation of engineering diagrams that describe the movement of materials and utilities throughout the process. Specifically, beginning with the validated laboratory process, material balances are established and used to develop a series of increasingly detailed design documents, including the Block Flow Diagram (BFD), Process Flow Diagram (PFD), and Piping and Instrumentation Diagram (P&ID).

Once the engineering design has been completed, the process is transferred to a pilot plant, where production is carried out on the kilogram scale. Following successful pilot trials, the process may be scaled up to a manufacturing plant on the tonne scale. TBP did not reach the pilot trial stage during my internship. Hence, this report focuses on the principal elements of the process design prior to this stage.

Material balance

A material balance is a quantitative accounting of all materials entering and leaving a process, based on the principle of conservation of mass: matter is neither created nor destroyed, so the total mass of material fed into a process must be fully accounted for in its outputs. It is created using validation data and forms the basis of process design. The material balance for TBP was created assuming continuous operation rather than batch operation. In batch production, material is charged, processed, and discharged in discrete cycles; in continuous production, material flows through the process without interruption and, at steady state, no mass accumulates within the system.[12] The material balance for TBP is shown in Figure 6. As it is a continuous process, 11.528 MT of raw material enters and 11.528 MT leaves per MT of product. This is likely a consequence of the excess solvent. Only 1.000 MT of the 11.528 MT throughput per MT of product (approximately 8.67% of the output). The largest output stream is the 6.955 MT of liquid effluent produced per MT of product (approximately 60.33% of the output). This quantifies the load that the effluent treatment provisions must be designed to accommodate. Furthermore, the material balance assumes 2.128 MT of recovered solvent per MT of product; this assumes the successful purification of recovered *n*-butanol, as the recovered solvent from the validation batches had an unacceptably high water content. Additionally, 0.67 MT of HCl leaves as air emissions. Thus, a comprehensive report of the

environmental impact of Prasol's TBP production must be prepared, and the material balance must be tested against the estimated effluent treatment capacity of the plant.



Material Balance for Product

S.No.	Raw Materials	Input/MT of Product (MT)	Input /Day of Product (MT)	Input/Annum of Product (MT)
1	Product	0.940	3.13	940.00
2	other product	3.633	12.11	3633.00
3	solvent	4.010	13.37	4010.00
4	solvent	2.945	9.82	2945.00
Total		**11.528**	**38.43**	**11528**

S. No.	Output/MT of Product							Output/Day of Product MT	Remarks	Output/Annum of Product (MT)
	Material / Item	Product / Byproduct	Liquid Effluent	Air Emission	Loss	Hazardous Waste	Recovery			
1	product	1.000	0	0	0	0	0	3.33	Product	1000
2	REC solvent	0	0	0	0	0	2.128	7.09	Solvent	2128.0
3	HCL	0	0	0.67	0	0	0	2.23	Air emission	670.0
4	Distillation/ Rectification Losses	0	0	0	0.431	0	0	1.44	losses	431.0
5	Effluent	0	6.955	0	0	0	0	23.18	Effluent	6955.0
5	Handling loss	0	0	0	0.344	0	0	1.15	Effluent	344.0
Total		**11.528**						**38.43**		**11528**

Figure 6. The material balance for TBP. The unit MT refers to metric tons. The numbers are estimates and do not necessarily reflect the actual quantities that will be produced in the commercial plant.

Engineering Diagrams

Following completion of the material balance, a series of engineering diagrams was developed to represent the manufacturing process at progressively greater levels of detail:

1. The first diagram prepared was the Block Flow Diagram (BFD), which was prepared in Microsoft Excel. It presents a simplified overview of the process by illustrating the major processing stages in the form of blocks and the flow of materials between them.

2. The second is the Process Flow Diagram (PFD), which was first drawn by hand and subsequently drafted in AutoCAD. Unlike the BFD, the PFD identifies the major items of process equipment, process streams, and principal operating conditions, providing a more detailed description of the manufacturing process. The PFD was initially prepared by hand before being redrawn using AutoCAD.

3. The final stage of process documentation involved the preparation of a Piping and Instrumentation Diagram (P&ID). In addition to the equipment and process streams shown on the PFD, the P&ID specifies the piping network, valves, process instrumentation, and control systems required for plant operation.

As these diagrams were developed directly from the proprietary TBP manufacturing process, they are not included in this report.

Energy and Utility Requirements

Energy balance is used to determine the heating and cooling requirements of each unit operation. For the proposed TBP manufacturing process, steam was selected as the principal heating utility. The quantity of steam that the plant must supply was calculated based on the synthesis process of TBP. The heat required consisted of two components: sensible heat, the energy required to change the temperature of the material, and latent heat, the energy required to effect a phase change at constant temperature. Sensible heat is relevant for steps where temperature changes occur, while latent heat applies to the distillation steps, where material is vaporised. The steam consumption is represented in Figure 7. The greatest steam demand occurred during heating of the reaction mass, which required approximately 1356.6 kg of steam, followed by final product distillation (973.5 kg) and vacuum distillation for recovery of *n*-butanol (890.3 kg). The flow rate of steam (in kg/hr) required at each step was calculated, and the largest value was used: 486.8 kg/hr. To this peak a design margin of 15% is applied, raising the figure to approximately 559.82 kg/hr, which is then rounded up to a design steam rate of 600 kg/hr. This is the estimated amount of steam that will be consumed per hour by the proposed plant. The margin provides a safety allowance against variability in operation, heat losses, and inefficiencies not captured in the idealised calculation.

Steam Consumption.

Line no. 1
Product

Utility - Steam	Steam pressure at BL, Kg/cm2g	3.5	Steam temp, C	150	Latent heat, Kcal/Kg	505

Sr. No.	Processing step	Unit Process/Unit Operation	Heat of reaction if Unit Process, Kcal/Kg	Addition Kg	Qty, Kg	Duration of operation, Minutes	Rate of addition	Initial Temp, C	Final Temp, C	Sp heat, Kcal/Kg.C	Sensible heat, Kcal	Latent heat, Kcal/Kg	Latent heat, Kcal	Total heat, Kcal	Steam Latent heat, Kcal/Kg	Steam, Kg	Steam, Kg/Hr
1	Charging IBA / nBA in reactor	Charging		3289	3289	30	6578	30	30	1	0	0	0	0	505	0	0
2	Cooling to 5°C	Cooling			3289	60		30	5	1	0	0	0	0	505	0	0
3	Product Addition +20% NaOH (5–10°C)	Reaction (Unit Process)		940	4229	360	156.7	5	10	1	0	0	0	0	505	0	0
4	Maintaining Reaction	Maintaining			4229	120		10	25	0.8	50,748	0	0	50,748	505	100.5	50.2
5	Heat Reaction mass	Heating			4229	240		25	40	0.8	50,748	150	634,350	685,098	505	1356.6	339.2
6	Vacuum Distillation (Recovery of IBA)	Distillation		-2128	2101	240		40	120	0.8	134,464	150	315,150	449,614	505	890.3	222.6
7	Neutralization	Reaction			2101	60		120	30	1	0	0	0	0	505	0	0
8	Water Washing-I	Washing			2101	30		30	30	1	0	0	0	0	505	0	0
9	Water Washing-II	Washing			2101	30		30	30	1	0	0	0	0	505	0	0
10	Final Product Distillation	Distillation		670	2101	120		30	135	0.8	176,484	150	315,150	491,634	505	973.5	486.8
11	Product Cooling	Cooling			1431	30		135	30	1	0	0	0	0	505	0	0
12	Product Transfer	Transfer			1000	15		30	30	1	0	0	0	0	505	0	0

Maximum	486.8
Margin @ 15%	559.82
Design Steam	600

Figure 7. Steam consumption calculation for the production of TBP. The highlighted rows require energy supplied from steam.

5. Conclusion

This report has followed the development of tributyl phosphate (TBP) at Prasol Chemicals from the closing stage of laboratory research through to the preparation of a commercial process design. The validation batches demonstrated that the developed synthesis process was capable of consistently producing high-purity TBP, with product purities ranging from 98.87% to 99.80% at yields of 72.62–75.51%. Analysis of the product confirmed that

specification was met: gas chromatography returned a purity of 98.865% and manual acid–base titration returned an alkalinity of 0.06% for the first validation batch. Because the measured alkalinity was so low, the imprecision inherent in manual endpoint detection did not affect the conclusion drawn, and a potentiometric determination was not required. By contrast, the Karl Fischer titration of the recovered *n*-butanol returned a mean water content of 13.61%, exceeding the limit acceptable for reuse in TBP production.

At the process design stage, the material balance quantified the consequences of the laboratory process at commercial scale. For 11.528 MT passing through the process, only 1.000 MT is recovered as product (approximately 8.67% of total output), while liquid effluent constitutes the largest single output stream at 6.955 MT (approximately 60.33%). A further 0.67 MT leaves as hydrogen chloride (HCl) in air emissions. The energy balance established the required design rate of steam consumption to be 600 kg/hr.

Three matters were identified as requiring resolution before the process advances. First, the quality of the recovered n-butanol must be improved if the solvent is to be reused; investigation into this optimisation was ongoing at the time of writing. Second, the material balance must be tested against the estimated effluent treatment capacity of the intended plant, given that effluent constitutes the majority of process output. Third, a comprehensive assessment of the environmental impact of production, encompassing the liquid effluent load, the HCl emissions, and the steam requirements, must be prepared.

In conclusion, the study demonstrates how validated laboratory data are transformed into engineering documentation through material and energy balances, process flow diagrams, and utility calculations. These activities form the critical link between chemical research and industrial manufacturing, providing the information required to scale a laboratory process into a safe, economically viable, and commercially implementable production system.

6. Reflections

My role during this internship was to study the development of TBP from the closing stages of laboratory research through to process design. One of the most significant observations was the difference between industrial and academic research. Academic research is primarily directed towards advancing scientific understanding, whereas industrial research is driven by the development of commercially viable products that satisfy technical, economic, and safety requirements. In industry, an analysis is performed to answer a question, and precision need only be sufficient to answer it. This became clear to me in the acid–base titration of the product. Manual endpoint detection is measurably less precise than a potentiometric determination, and my instinct was that the more precise method should be preferred. However, the alkalinity obtained was so far below the threshold for impurity that no plausible error in reading the burette could have altered the conclusion. Furthermore, I was introduced to analytical techniques considerably more advanced than those available in a school laboratory. Karl Fischer titration and gas chromatography allowed for more precise and

accurate analysis than the loss-on-drying or paper chromatography methods I had previously studied.

The internship also highlighted that scaling a chemical process from the laboratory to commercial production is far more complex than simply increasing the quantities of reactants. Material balances, utility requirements, environmental considerations, equipment selection, and process safety must all be incorporated into the engineering design before manufacturing can begin. I linked studying engineering documentation to principles of technical drawing, a subject I had taken during Grades 9–10, as well as my experience using computer-aided design (CAD) for robotics. Finally, the internship demonstrated the interdisciplinary nature of industrial chemical engineering. Successful process development depended upon collaboration between research chemists responsible for process development, chemical and process engineers responsible for scale-up and optimisation, and mechanical engineers involved in the design of manufacturing equipment and plant infrastructure.

Overall, this internship significantly broadened my understanding of how scientific research is translated into industrial practice. It provided practical exposure to analytical chemistry, process development, engineering design, and interdisciplinary collaboration. The experience complemented the theoretical knowledge acquired in school by illustrating its application within an industrial setting, and provided valuable insight into engineering as I prepare to pursue undergraduate studies in this field.

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