Hayle Harbour and Estuary (circa. 1960) showing the location of the Associated Octel factory

The Hayle Works of Associated Octel was established in 1939 as the site for the extraction of bromine from sea water. This product was required for the war effort. It was used in the manufacture of octane boosters for aviation gasoline during the Second World War and thereafter in motor gasolines.

The factory was initially owned by the Government (Ministry of Aircraft Production), but designed and operated by British Ethyl Corporation on their behalf.

This article reviews the history of Associated Octel activities at Hayle and covers the entire period of their operation, from 1939 to 1973. The selection of the site, construction, operation and development of the process are considered along with the basic reason for establishing such a faculty in the United Kingdom. The corporate organisation managing the operation is also outlined.

Technical matters are treated in general terms with emphasis placed on those topics felt to be of historical and local interest.

Mining the Sea
The Extraction of Bromine from Sea Water at Hayle

Arthur Fairhurst, B.Sc., C.Chem., F.R.S.C.

Introduction

Bromine was the first chemical to be recovered commercially from sea-water with the exception of salt. It is present at concentrations of up to 65 parts per million (ppm) in U.K. waters. Prior to the discovery of tetraethyl lead (TEL) as a gasoline anti-knock agent the requirement for bromine was limited to the demands of the fine-chemicals industry. World-wide demand for bromine, as ethylene dibromide, was linked to the potential use of TEL in the rapidly expanding petroleum industry. An economic source and plentiful supply of bromine was critical to this expansion.

Ethyl Gasoline Corporation of the USA first examined the possibility of extraction from sea-water in 1924 operating a small pilot plant in partnership with Du Pont at Ocean City, Maryland. The increasing demands of the rapidly growing anti-knock business made it necessary to pursue the extraction of bromine from the sea with some urgency. Dow Chemical Company developed an interest independently, using an alternative process, taking the decision to build a full scale plant at Kure Beach, North Carolina. This was commissioned in 1933 with a design capacity of 2500 tons per year (tpa). Even with bromine in sea water concentrations of 65ppm the project was commercially successful, reducing the production costs significantly. A more detailed account of these developments can be found in the literature. Ref. (1) to (4).

The basic chemistry of the Dow **<u>"alkali" process</u>** involves four separate reactions:

(a) The acidification and oxidation of the sea water with sulphuric acid and chlorine to liberate the bromine.
(sodium bromide) + (chlorine) > (sodium chloride) + (bromine) Reaction 1.

(b) The "blowing out" of the liberated bromine from the sea water by air Reaction 2.

(c) The absorption of the bromine from the air with an alkaline carbonate solution.
(bromine) + (sodium carbonate) > (sodium bromide + sodium bromate) + (carbon dioxide) Reaction 3.

(d) The acidification of the absorption liquors and separation of the liberated bromine by steam distillation.
(sodium bromide + sodium bromate) + (sulphuric acid) > (sodium sulphate) + (water) + (bromine) Reaction 4.

This was the process used at Hayle until 1954.

In 1937 Dow developed a second process, **<u>the "acid" process.</u>**

The two initial stages, the stripping of bromine from sea water, were identical. The third stage involved "fixing" the liberated bromine with sulphur dioxide gas.
(bromine) + (sulphur dioxide) + (water) > (hydrobromic acid + sulphuric acid) + (water) Reaction 5

The acid mixture is scrubbed with fresh water in an absorber, effecting a thousand fold concentration of the original bromine in sea water.

The final stage is the release of bromine from the strong acid liquor by reacting with chlorine and separating the liberated bromine by steam distillation.
(hydrobromic acid + sulphuric acid) + (chlorine) >
(bromine) + (hydrochloric acid + sulphuric acid) Reaction 6
The acid effluent is recycled to the sea water main to acidify the incoming sea water.

The "alkali" process consumes less chlorine but requires far greater quantities of sulphuric acid. The "acid" process requires twice as much chlorine but the recycled acids satisfy more than half the acid demand of the raw sea water. The process plant is also more compact and elegant.

Corporate Background

The corporate history of Associated Octel begins in 1928 when Mr. T.R.A. Bevan became overseas representatives of Ethyl Gasoline Corporation. In 1929 an office was opened in London. Ethyl export business was separated from its domestic, US, business in 1931 when the Ethyl Export Corporation was formed with the responsibility for handling all the TEL business outside North America.

"Ethyl" petrol made slow but steady inroads into the UK market, but by the outbreak of war in 1939 it was popular with motorists. During the same period the aviation industry came to regard TEL as an essential ingredient of high octane aviation gasoline causing the Air Ministry to approach Ethyl Export Corporation in 1935 to see what could be done to provide adequate and secure supplies of TEL for the RAF in the event of war. The American Neutrality Act indicated that it would be difficult to obtain supplies from the US.

At the Ministry request, it was agreed that an established British Chemical Company should be invited to join with Ethyl Export Corporation to set up a Company to manufacture TEL. ICI Ltd were approached and agreed to participate. British Ethyl Corporation (British Ethyl) was thus incorporated in 1936, 50% owned by each party, British Ethyl was to own all "know how" and ICI would provide staff. ICI undertook to construct the plants to designs provided by Ethyl Export and to staff and operate them on behalf of British Ethyl. The agreement between the Air Ministry and British Ethyl provided that the latter should act for the Government throughout the emergency with all product destined for use in aviation gasoline. The project was authorised and funded by Government but with the provision that, at the end of hostilities, the Ministry would offer to sell the plants to British Ethyl on defined terms.

It was never intended that ICI should become a commercial partner in the manufacture of either TEL or bromine. In 1943 British Ethyl became wholly owned by Associated Ethyl with ICI continuing to act as manufacturing agents. In the event, British Ethyl purchased the TEL and bromine plants on 1 Jan 1945 and ICI continued to operate the plants until Dec 31st 1947. ICI therefore operated plants owned by Associated Ethyl from Jan 1945 until the above date.

From 1936 separate negotiations were taking place which led to the formation of Associated Ethyl Co Ltd in 1938. Anticipating more normal times, all the major oil companies were invited to take a financial interest in the development of a growing "TEL" business. Associated Ethyl (Associated) was thus incorporated in September 1938, assuming the rights previously held by Ethyl Export Corporation.

Associated subsequently changed its name to Associated Octel in June 1961 in order to avoid any confusion with its US competitor, Ethyl Corporation, in the world wide market.

This was the background against which all the preliminary activities to the building of Hayle Works took place. Bromine was required to produce Ethylene Dibromide, a scavenger and essential ingredient of the anti-knock compounds used in high octane gasolines. The planning of the project to manufacture TEL was given to Castner Kellner Alkali Co (General Chemicals Division of ICI). Discussions started in 1936 and ICI were charged with obtaining all the basic raw materials. At first they considered that the bromine requirements could be met from the brine sources of ICI Alkali Ltd in mid-Cheshire.

However, the requirements of the Air Ministry continued to increase and it soon became obvious that a sea water source would be required for bromine supplies. In March 1938 the Air Ministry gave instructions to proceed with the preparation of detailed plans and in August of that year British Ethyl were instructed to proceed with the construction of the TEL plant in Cheshire. Following the change of plan and as a matter of urgency Dow Chemical Company of Midland, Michigan, USA, were approached by ICI representatives in order to try to acquire the rights to manufacture bromine by the process used at Kure Beach. In Nov 1938, following a meeting with the Air Ministry, Associated Ethyl were asked formally to approach Dow. In Jan 1939 an agreement was concluded. It was during the course of these negotiations that ICI (Alkali) Ltd initiated a detailed survey of the inshore waters of the UK.

The decision to establish bromine manufacture at Hayle was made early in 1939 but the formal report was only issued in 1940. The actual decision is lost in ICI files but the first references to Hayle, as a potential site, are dated Feb 1939. Given the nature and purpose of the overall project this was rather late in the day and certainly increased the pressure on the staff involved.

Selection of site

H. Fosset, in his paper "Extraction of bromine from sea water" discusses the topographical and logistical factors determining the selection of a suitable site. (ref 5). In summary they are:

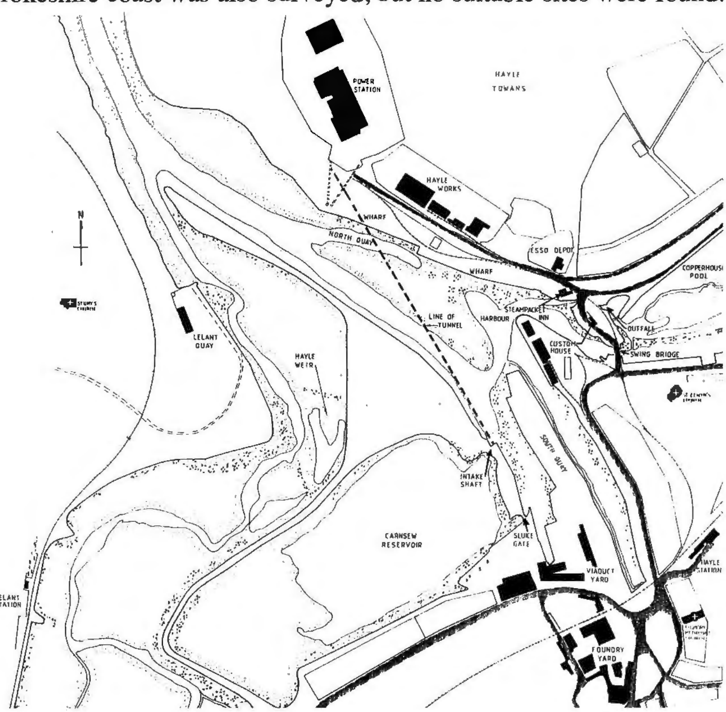
- Sea water as rich in bromine as possible.
- Sea water as clean as possible, free from sea weed, trash and oxidisable matter.
- Sea water as warm as possible.
- Low tidal range.
- Assured continuous supply of sea water.
- Ready availability of a source of cheap power.
- Means to separate sea water intake from discharge.
- Site as close to sea level as possible.
- Road and rail access with adequate distribution services.
- Close as possible to sources of raw materials.
- Availability of labour.

In addition, given the prevailing international situation, It was vital that the site should be as secure as possible from enemy attack by sea or air. A further consideration was that the amenities of the area should not be unduly spoilt by the presence of a chemical factory.

Following the late decision to pursue the extraction from sea water route, the Hydrographic Dept of the Admiralty were consulted to ascertain what information was available on the properties of the sea water around the UK coast. Whilst there was a considerable knowledge about the temperature and salinity of the waters it was unfortunately based on data collected from stations at least 5 miles off-shore. Arrangements were made to collect samples from 12 stations around the coast but closer to land. The deep sea data showed that water with highest salinity and temperature would probably be found around the Cornish coastline. In view of the urgency a detailed study of Cornish coastal waters was carried out during December 1938 and January 1939.

A member of the project team suggested that if a coastal power station could be found that utilised sea water for cooling purposes then advantage might be taken of the elevated temperature of the effluent sea water to improve the efficiency of bromine extraction. This idea focussed attention on Hayle, despite concerns about the salinity of the sea water on an estuarial site. A site visit quickly revealed the great possibilities of the area. It had an established transport infrastructure, with a well established rail system. Alongside the Towans there was also a derelict site which should be more secure than most from enemy action.

An alternative site was found at Deans Quarry, near Manacle Point on the south coast but this had the significant disadvantage that no rail or harbour facilities were available. The Pembrokeshire coast was also surveyed, but no suitable sites were found.



MAP OF HAYLE HARBOUR. (Note the line of the sea water tunnel from Carnsew to the Power Station)

The distance of Cornwall from the source of raw materials and the end-user plant was an economic factor that caused concern. The search for sites nearer to Cheshire continued. The possibility of an ICI site at Fleetwood was explored but the bromine content of the sea water was too low. No sites were found on the North Wales or Cumberland coastline which had advantages to outweigh those of Hayle.

Having focused on Hayle the obvious site was on North Quay, adjacent to and south of the Cornwall Electric Power Company Station, once occupied by the Pentewan Glass Works. The photograph of the area, taken in 1928, highlights the particular site, nestling under the cliffs on the south western edge of the Towans. The stone chimney on the northern boundary is still a prominent landmark today, having been left standing when the plant buildings were demolished in 1973/4. Surveys of the estuarial waters found that water pumped by the Power Company, from a point in the estuary opposite Lelant Quay, had a bromine content sufficient for economic manufacture of bromine for only 4 to 5 hours of each tide. The Carnsew Reservoir was, however, to assume a strategic importance and serve a new purpose, in addition to its traditional function to impound water for sluicing the Harbour channels.

The intake and outfall of the Power Station cooling system were very close to each other and the Company was troubled at low water by pumping their own warm effluent. For this reason they were interested in cooperating in any scheme that would ensure a continuous supply of cold water. When Carnsew was first seen by the ICI Survey Team the possibility of its being adapted to impound the higher tide sea water for use by the Power Station and thus for bromine manufacture was soon realised. The reservoir had two sets of sluices, the seaward sluice, Carnsew Bridge, consisting of vertical sluices fitted into tunnels. The reservoir filled through this source from about 4.5 hours before high water. The other sluice, Blackhouses, consisted of two swing gates fitted with sluicing windows. The inflow started here about 3 hours before high water. When sluicing was to take place the tunnel sluices and sluicing windows were closed on high water and opened 3 hours afterwards. They were then left open for refilling on the next tide. When no sluicing took place, on tides below 16ft., the sluice gates were left open, the reservoir drying out at low water.

The detailed study of the bromine content of the water entering and leaving Carnsew produced encouraging results. It was observed that although the sills of the gates are 30in below OD. there was a ledge of rock inside the reservoir which effectively prevented water from entering its main body until the tide was 6in above OD. at the sluice gates. Water only flowed in through Carnsew Bridge sluice during the intervening period.

In order not to interfere with the sluicing programmes but to provide sufficient water for the Power Station and bromine plant it was proposed that a sill be built to a level 8.5in below OD inside the reservoir across the mouth of the tunnel sluices and to cut back the rocky ledge across the mouth of the sluice gates to the same level. This, in addition to dredging out the centre of the reservoir, would enable a pool of water to be left at low water and to be refilled on each side.

The concern about the bromine content of the sea water and its availability in economic quantities over the whole tidal range was amply demonstrated by the extremely thorough survey carried out in March and April 1939. The dilution effect of the fresh water from the Hayle River was shown to have a greater effect on neap tides than on springs. Bromine content on springs was in excess of 65 ppm but during neaps it was significantly less.

Even so on neap tides, for the greater part of the "filling" period, water entering Carnsew had a content greater than 60ppm. It was calculated that the bromine content of the available water would be suitable for economic bromine manufacture provided the proposed modifications to Carnsew were completed.

The careful assessment and measurement of the factors affecting the bromine content of the sea water was indicative of the attention to detail and high standards that would be required for the efficient operation of the process. The writer can testify to the continuing emphasis placed on the quality and quantity of the available sea water. There were occasions when the dilution effect of the works effluent and a Hayle river, in full spate after heavy rains, combined with the natural effects of neap tides to produce an average content over 24 hours of less than 50ppm.

Site Clearance and Construction

In April 1939 a team from ICI visited the US to develop engineering and process operation plans from the new bromine plant. In May the Air Ministry suddenly doubled the requirements for TEL from that indicated in August 1938. The size of the bromine plant was therefore increased to match. This was indicative of the heightened level of preparedness for war in the post Munich period.

Information about the construction phase is very meagre due to early records being lost. Fortunately, J. Cathie, the Site Engineer and T J W Smith, then Government Auditor on the project, contributed to the record from memory in 1954 by giving their own personal account of events.

ICI were commissioned to build and operate Hayle Works by the Government under Government Contract 3. The operation was subsequently referred to as GC3. Site clearance began on 23rd August 1939. Production commenced on July 24th 1940, some 96 days later than intended judging from the early planning schedules. Taking account of all the circumstances, including the uncertainties of the "phoney war" this was a considerable achievement. Cathie commented on the remarkably free hand he was given and the small amount of interference from his Principals.

Broadly the operations undertaken in connection with the Works were:

Site Clearance.

Construction of roads and other civil engineering works.

Construction of buildings.

Installation of plant and machinery.

Essential to the plant was the supply of sea water from the Power Station and therefore ICI were involved, in varying degrees, in construction of the sea water tunnel from Carnsew to the Power Station uptake, the new Power Station pumphouse at the uptake and the dredging of Carnsew Reservoir.

Christopher Hinton (later Sir Christopher) Chief Engineer of ICI (Alkali) Ltd was a powerful driving force behind the project. ICI negotiated with Harvey & Co, the owners of the land, on behalf of the Air Ministry. In July 1939 suitable Heads of Agreement were confirmed.

Demolition of the old buildings, blasting operations to remove rocks to make room for buildings and the building of the concrete wall surrounding the site (and still in position in 1999) was included in a site clearance contract awarded to A H Dingle of Redruth. Within a week of starting the Chief Engineer was satisfied with neither the arrangements nor progress. He promptly offered incentives to ensure the work was completed to schedule. Work continued until February 1940, by which time approx 80,000 cubic yards of blue elvan rock had been blasted from the cliff area and transported to a dumping site on the Towans.

Site clearance had progressed sufficiently by November 1939 for John Mowlem Construction to lay-out and excavate foundations for all the buildings. The Trussed Concrete Steel Co Ltd were engaged as Design Engineers, with Mowlem the main civil engineering contractors. At the peak of activity Dingles employed 50 men and Mowlem 250 men. 40 local men were directly employed by ICI, early in 1940, to supplement the key staff transferred from Cheshire.

Cornwall Electricity Company Station in 1928. The site of the British Ethyl plant is in the bottom left quadrant, occupied by the buildings of the Pentewan Glass Works.

The sea water system was obviously on the critical path of the entire construction programme and requires detailed comment. At the end of 1939 Dingle secured the contract for the effluent culvert, which runs from the middle of the site in a straight line under the approach road to an outfall on the NW corner of Copperhouse Pool. This work was completed by June 1940 but subsequently, because of poor construction, required regular repair, particularly in the immediate post-war period.

Construction of the sea water tunnel, intake and uptake works and the development of Carnsew justifies a separate paper, only the significant facts are summarised here. The line of the tunnel, from Carnsew to the Power Station is delineated on the Map. Its construction presented some peculiar problems and considerable trouble for the ICI Engineers. The original contract was placed by the Cornwall Electric Power Company with Leightons of London. Their progress lagged well behind that on the works site and was likely to delay the completion of the whole project. After much legal consultation the contract between Leighton and CEP was cancelled (see below). ICI negotiated a new contract with John Mowlem Tunnel Section and work resumed, under ICI control, early in 1940.

Poor initial survey work led to the abandonment of the rock tunnel under the estuary after driving for only 80 yards. The survey indicated rock throughout but at approx 100 yards from the uptake shaft the tunnel broke through the rock and sea water flooded the tunnelled section. A check survey proved that there was a serious and deep fault in the rock and that the rock head around the fault was lower than -120ft. OD. The resultant delay was serious.

The problem was overcome by driving the tunnel on a new and higher line through sand, using cast-iron tubbing and working under compressed air. Work proceeded from each end of the tunnel, using the already constructed uptake shaft and part of the driven rock tunnel, sealing off a 30 yard length at the seaward end, then driving up an incline to the line of the new high level tunnel started from Carnsew. The original borings indicated that the tunnel could be run through rock at a depth of -86ft OD. in which case it was not intended to line it. After 300ft the rock ended. The new tunnel was through sand and shale for 1100ft from Carnsew intake, lined with cast iron tubbing obtained from London Transport. For the remaining 500ft, cut from the uptake end, the tunnel was rock, lined with bricks and backfilled with concrete.

The intake shaft was lined with cast iron tubbing, 10ft diameter. The 10ft dia. section was lined with circular concrete inserts. When the diameter of the shaft reduced to 7ft it was lined with cast iron. All the cast iron tubbing was internally lined with concrete to give a smooth surface.

Dissatisfaction with progress mounted until in Jan 1940 Christopher Hinton took the law into his own hands and personally ordered Leightons off site. Problems still remained because at the end of Feb 1941 the driving of the tunnel was being hampered by the operations of the Power Company's contractor concreting the new pumphouse. These people behaved as if no tunnelling work was in progress and were allowing concrete and water to get into the workings. Work did stop for a period until matters were resolved.

Carnsew Reservoir was the property of Harvey & Co and they entered into an agreement with the Cornwall Electric Power Company on 17th April 1939 in respect of dredging, dam and sill and the amount of water that could be drawn off. The sea water off-take was limited to 2 million gallons per hour. In March 1948 this agreement became a lease, "with the right, licence and liberty to take water for use as circulatory water and to supply water to British Ethyl Corporation". The lease was for a period of 99 years and continued to cover construction of intake works, tunnel, sills and dredging.

Dingle had the contract for the initial dredging. A bund was constructed from the Harbour embankment to the NW side of the reservoir, running in a SW/NE direction. The dredged sand was pumped into the impound. Part of the bund was constructed from rock mined from the tunnel. Work started in the final quarter of 1939 and the additional volume created by dredging was equivalent to the volume of water which could be drawn off between successive high tides.

ICI supplemented Dingles' work by constructing a dredger on-site. A barge was also used. Dingles contract was terminated at the beginning of Feb 1940 when the contract for dredging the pool was transferred from CEP to ICI. The operation continued well into 1941.

A second dredging operation took place between Sept 1950 and Sept 1952. This work was carried out by J & L Eve (Construction) Ltd. The dredged sand was transferred to pits specially dug in the small field between the south bank of the reservoir and the A30 trunk road. The capacity was increased significantly and a further Deed between British Electricity Authority and Harvey & Co increased the permitted off-take to 2.5 million gallons per hour. No further dredging is recorded after this time.

The capacity after dredging, above the -0.7ft OD mark, ranged from 64 million gallons at tides around +8.4ft OD. to 106 million gallons at tides of +12.5ft OD.

The essential equipment at the Power Station uptake, for providing the bulk of water for bromine manufacture, comprised three 24in diameter pumps with a capacity to pump 800,000 gallons per hour, each driven by a 390HP motor. A fourth pump was installed in 1949. The pumphouse was finished and pumps installed just before the tunnel works were completed.

British Ethyl Works, circa 1945, viewed from the North West. The plant buildings, from N. to S. are: Soda Ash mixing, Absorber Fans, Absorber, Blowing out Tower, Steaming Out tower building (concrete), Dibromide reactors and Ethylene Plant and finally Workshops and Offices.

British Ethyl Works, circa 1945, viewed from the South East. Note the compact nature of the site nestling under the Cliff.

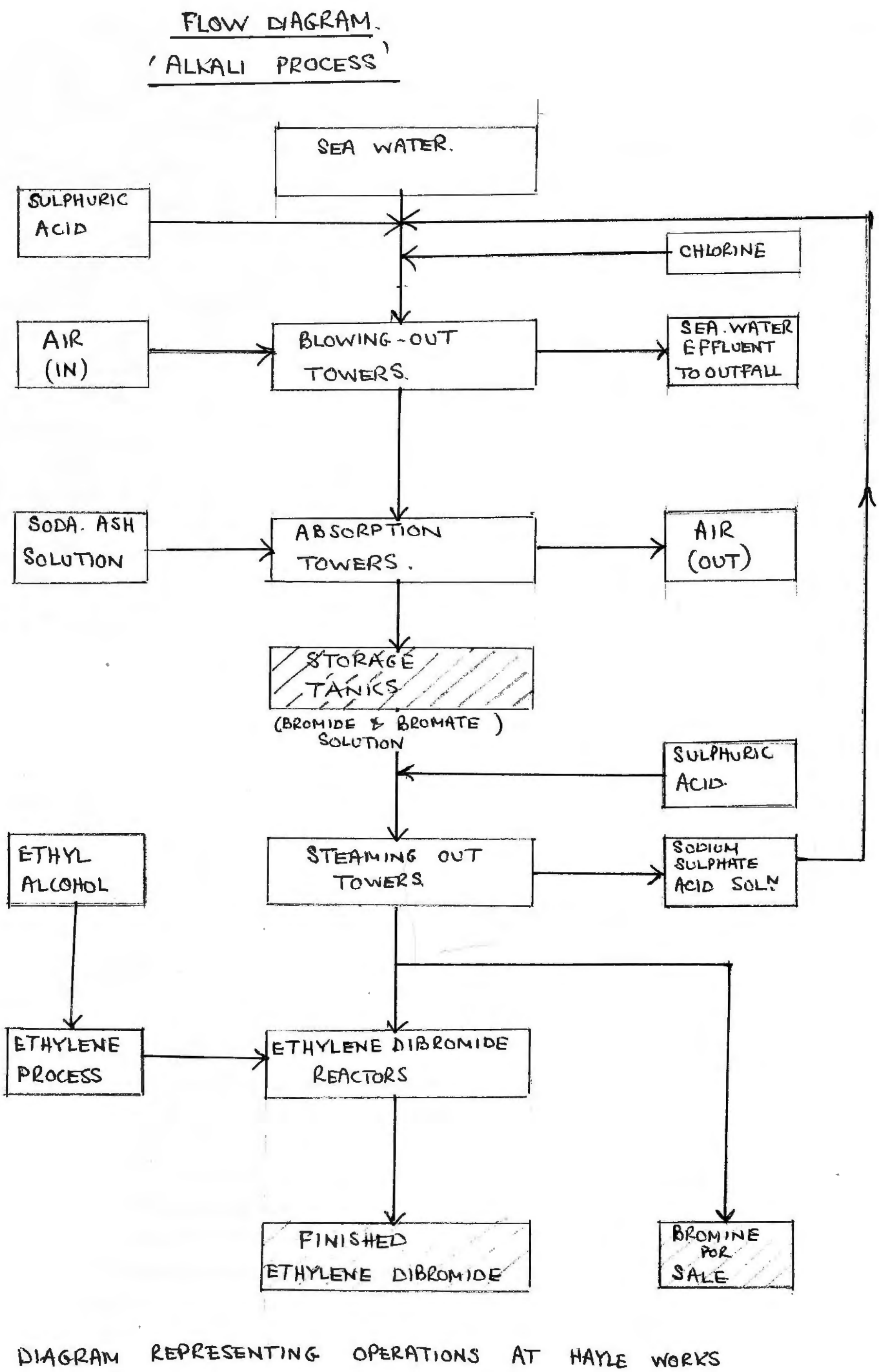
Plant and Process

Photographs show the entire works as constructed and unchanged until 1949. The camouflage is a pertinent reminder of the war-time conditions. To describe the site as compact is an understatement. Diagrams present all the operations in sequence and illustrate the heart of the process.

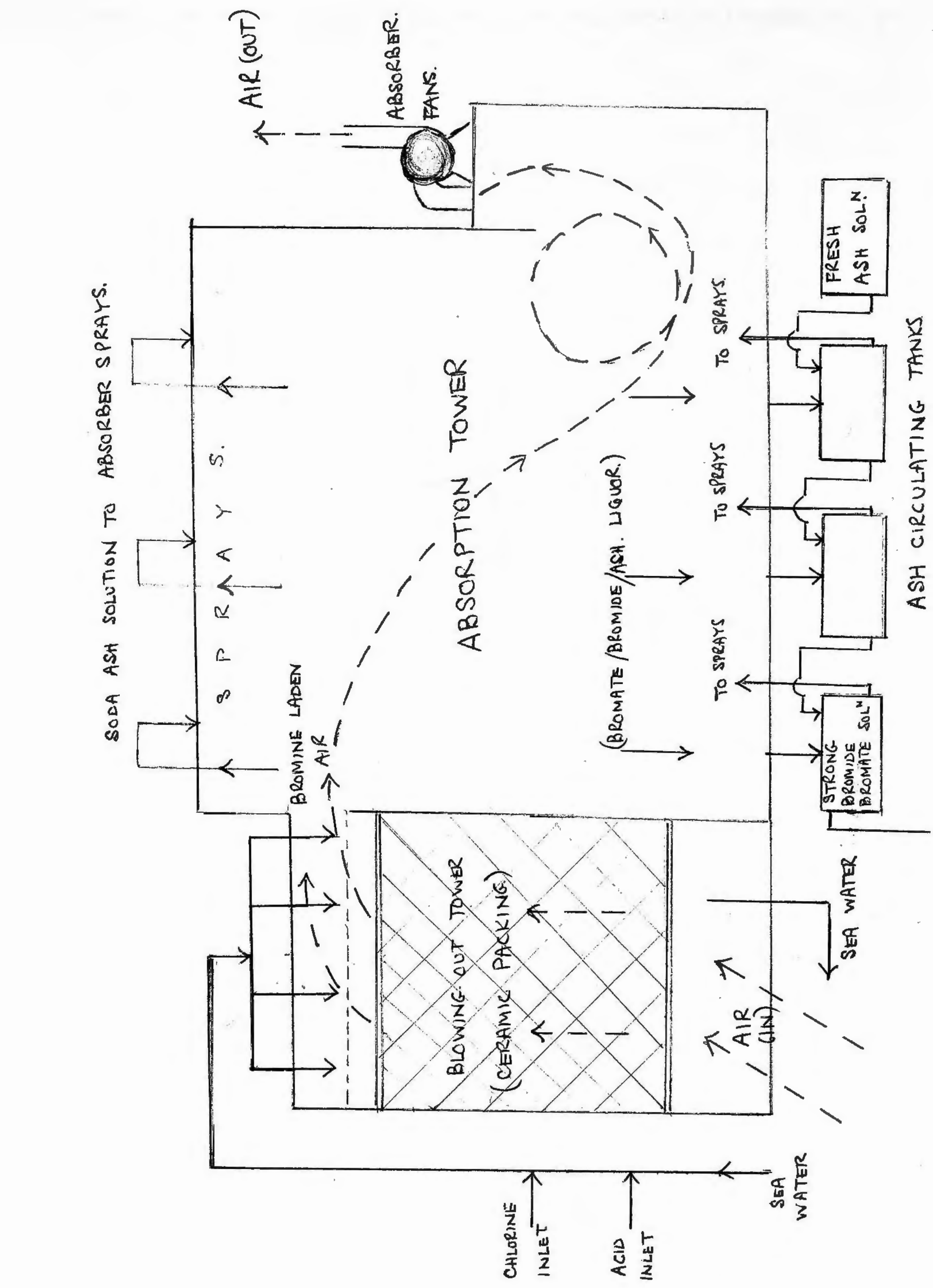
The sea water system is the key element in the whole operation and for practical purposes begins and ends in the Hayle Estuary. Within these outer limits there are Carnsew reservoir, belonging to Harvey & Co, the tunnel to the Power Station, who had the agreement with Harvey & Co, the Power Station circulating system, the Works internal system and the effluent culvert to the estuary, via Copperhouse Pool.

Water pumped from the Power Station is conveyed to the works pumphouse via a 48in diameter cast iron main.

The Blowing Out Tower is the first of the process units and governs the entire rate of production. It is a "stripper" in which the treated sea water meets a counter current of air. The water flows through a series of distribution boxes and over weirs, to ensure even distribution, and then cascades through a brick built tower packed with ceramic rings. Air, in a volume ratio of 70:1, is blown through the tower stripping out the liberated bromine (reaction 2). Before entering the tower the sea water is acidified with dilute sulphuric acid, to ph 3.5, and chlorinated to a pre-determined excess. The injections are made in the vertical section of the sea water main in order to achieve thorough mixing and the optimum liberation of bromine (reaction 1).



Flow Process Chart for the "Alkali" Process



Diagrammatic representation of the principal stages in the alkali-extraction process

The reaction efficiency increases with increasing temperature and the stripping efficiency with higher air: sea water ratios. The type and height of packing in the tower are critical engineering features. All these factors were kept under review. Air is drawn through the towers by the absorber fans located on the northern end of the building.

The function of the Absorption Tower is to remove, as completely as possible, the bromine from the air passing to it from the blowing out tower. This tower is designed for a particular flow rate and is unable to cope with an increased production rate without a loss of efficiency. This inflexibility was an inherent weakness in the process. To ensure complete reaction the absorber is divided into nine individual passes in which the bromine laden air is reacted with a circulating sodium carbonate solution via a series of sprays. (reaction 3). The even distribution of the solution over the whole unit is the key to efficient operation.

Absorption liquor, a solution of soda ash, sodium bromide and sodium bromate, is collected at the base of the tower, from each pass, in an interconnected series of circulating tanks. The bromine content of the liquor is highest at the inlet pass and lowest at the final pass. When the bromine content of the first circulating tank reaches its control strength it is pumped into a storage tank. The contents of the next tank are then transferred into it and so on until the last tank is emptied and ready to be charged with fresh soda ash solution. The whole operation takes about one hour, during which time one section of the unit is out of action.

The Absorption Fans are the second most important item of equipment after the sea water pumps. Throughout the whole life of the alkali process they received a great deal of attention. Four fans were originally installed each having a capacity of 65,000 cubic feet per minute (cfm). In 1942 the originals were replaced by five Howden "Silent Vane" centrifugal fans, with a sixth being installed in 1945. Each fan was cleaned and painted on a routine planned basis.

The absorption liquor in effect stores the bromine extracted from the sea in a thousand fold concentrated form and provides a convenient buffer to smooth out any production variations arising from changes in sea water, either temperature, bromine content or flow rates. The storage tank enables the rest of the process to operate at a steady rate, all other factors being equal.

In the Steaming out Towers elemental bromine is recovered from the alkaline absorption liquor in two sub-stages. The bromine is liberated by the simple expedient of mixing the liquor with sulphuric acid (reaction 4). The unit consists of an upper and lower tower with an acid mixing tank in between. The upper tower acts as an absorber and pre heater, an excess of sulphuric acid is added at the mixing tank, while the liberated bromine is separated by steam distillation, condensed and passed forward either for packing or for conversion to ethylene dibromide.

The towers are of mild steel construction, 42in in diameter, lined with acid resistant bricks and packed with 1in diameter, ceramic, raschig rings, to a height of 14ft. Apart from routine maintenance and the application of newer, acid-resistant cements, the operation was trouble free over its whole life span.

The acid effluent is run off and held in a dilute acid tank, where it is strengthened to the control level by adding conc. sulphuric acid, before being recycled to the incoming sea water main.

Most of the production was converted into ethylene dibromide in a direct copy of the plant built by Dow at Kure Beech. The process is a simple reaction between bromine and ethylene gas, followed by neutralisation and distillation. The plant was constructed exactly in accordance with the Dow drawings but the reactors quickly became a bottle neck. Ways and means of reducing reaction time were sought and it is fortunate that there was a sustained demand for packed bromine. The "batch" process was eventually replaced by a "continuous process" developed and patented by Associated Ethyl. The original plant had a storage capacity of 300 tons, of which 200 tons was for finished product.

Ethylene was produced on site until 1954 by the catalytic conversion of ethyl alcohol. The plant installed in 1940 was precisely that which had been developed at ICI Winnington, Cheshire by Alkali Division for use in the manufacture of polythene, itself a significant historical fact. Operation was trouble free until it was displaced by imported supplies of liquid ethylene from Petrochemicals Ltd. Conversion efficiency was generally in excess of 90%.

A storage tank, blower and heat exchanger were installed on the cliff above the ethylene plant to receive the liquid ethylene from road tankers. The handling plant was commissioned with the first trial shipment on the 12th Sept. 1954. The ease and advantage of receiving liquid ethylene supplies was quickly established, continuing until production of dibromide ceased in 1968.

Raw Materials and Transportation

Essential services, such as fresh water, gas, electricity and labour were readily available in Hayle but most raw materials had to be transported to site. Given the geographical isolation adequate storage for these materials was often an issue.

In spite of war time conditions, particularly transport disruption due to enemy action, there was no very great difficulty in maintaining supplies and stocks at a reasonable level. They did run low at times but there were only two occasions when production was lost due to shortage of materials and these only totalled 48 hours.

Storage capacity was related to annual design capacity of 2500 tons of bromine per year. The gradual increase over the years to 4000 tpa placed a strain on the storage facilities and focussed attention on the supply source, frequency of delivery and the means of transportation. Rail tankers were used to hold stocks of chlorine and sulphuric acid at safe levels while some additional storage for the non-bulk items was rented from Harvey & Co.

The major raw materials were sulphuric acid, chlorine, soda ash and alcohol. There was storage for 350 tons of sulphuric acid. Until the Acid plant was built in 1949 all the sulphuric was supplied by the National Smelting Corporation, Avonmouth or by Gibbs & Finch of Plymouth in company owned rail tankers. Chlorine was supplied in rail tank cars by ICI General Chemicals Division, from their mid-Cheshire factories. With only 40 tons of storage on site and long supply lines (400 miles) stocks were often held in rail cars on Gwinear Road rail sidings until required on site.

Soda Ash was supplied by ICI Alkali, again from mid-Cheshire, in rail wagons. Alcohol was received from Industrial Spirit Supply Co. (later Distillers). The chronic fuel shortages of the post war years continued to threaten the smooth supply of alcohol until 1952. UK sourced material was often supplemented by imports of "Empire Origin".

Naturally, supplies and stocks of alcohol were the subject of intense scrutiny by the Customs and Excise.

Bromine was shipped from site in drums or bottles packed in boxes and transported by road and rail.

Major supplies and finished product were shipped in the rail tanker fleet purchased specifically for the purpose. British Ethyl owned a fleet of 35 RTC's comprising:

For Sulphuric Acid 20 For Chlorine 8 For Dibromide 7

By October 1954 Associated Ethyl had a fleet of 30 RTC's, 10 each for Sulphuric Acid, Chlorine and Dibromide. Sulphuric Acid was being produced on site so fewer acid carrying vehicles were required. Expansion of production facilities at Almwch and Ellesmere Port led to a major expansion in the rail fleet. Associated Ethyl (later Associated Octel) became one of the major fleet owners in the Chemical Industry expanding to 150 vehicles, including 5 for liquid bromine. Bromine RTC's were introduced progressively from 1964.

The Daily Trip Working to St. Erth. Rail Traffic was delivered and collected daily (M to F) from the Works

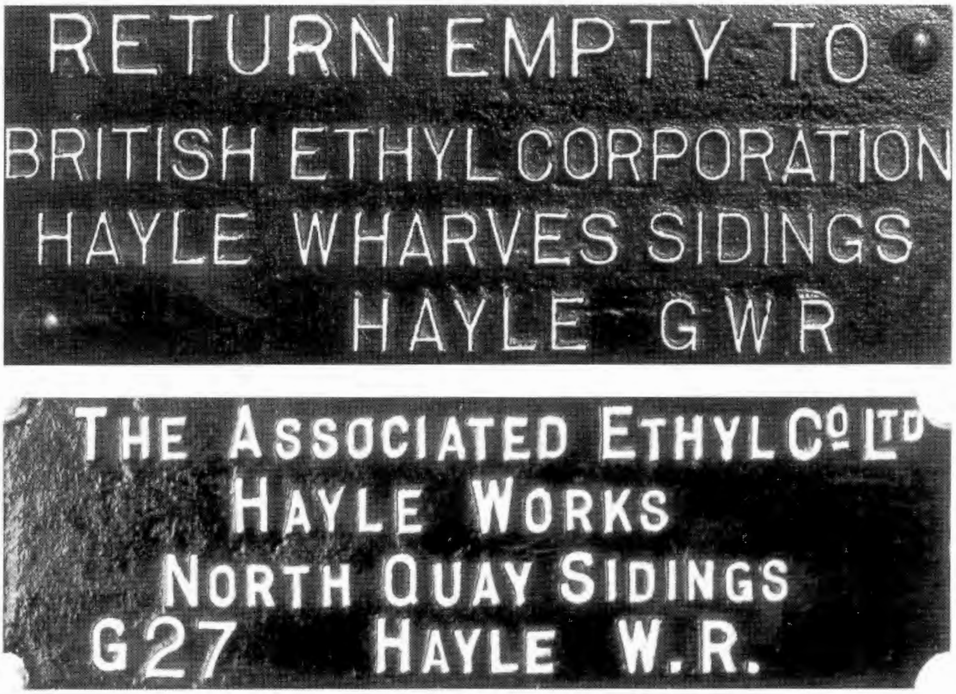
Operating Highlights and Developments

The nature of the process, finished product and intermediates, called for the highest standards of construction and maintenance. The ever increasing demand for its products, from both the Chemical and Oil Industries, as well as the limited production capacity produced a culture of continuing improvement. There was a constant drive to increase production, raise efficiencies and optimise controls which continued until closure in 1973. Towards the end economics became the major driving force.

Hayle operated a new process in a plant designed to produce 2500 tpa, based on a bromine content in sea water of 65ppm. It was appreciated at the design stage that there would be some dilution from the plant effluent and river water but the extent was under estimated. With hindsight the design should have been based on 60ppm bromine. The basic hydrodynamics of the estuary were quickly understood by each successive Works

Manager in the pursuit of even more production.

Early surveys of the mean daily bromine content highlighted the tidal effects and relationship with tide height. On the highest spring tides a mean content of 65.5 ppm was obtained but on neaps this fell to a low of 52ppm. At some stages, particularly immediately after low water, water flowing into Carnsew had a bromine content of only 42ppm. The dilution in the harbour by recycled effluent increased as tide height decreased. Because of the position of Hayle Bar and the prevailing sluicing practices this effect was most pronounced at tides of 16ft and below. Such dilution was the achilles heel of the plant.



Wagon Plates from the first tankers to transport Dibromide from Hayle to Northwich. Note the change of railway status on G27.

Given the variable quality over which there was little control attention was focussed on the quantity of sea water available. Carnsew Reservoir came into full operation in January 1941 permitting a maximum flow of 23,000 gpm. At certain levels in the estuary this quantity could not be sustained for very long and it was necessary to reduce pump speed to avoid problems in the Power Station. When the new Power Station pumps came into operation the maximum sustainable rate increased to 33,000 gpm. This was the limit of the flow permitted under the agreement with Harvey & Co.

In July 1946 discussions between Edmundson Electricity Corporation and British Ethyl resulted in an assurance that following extensions to the Power Station there would be no difficulty in raising the flow to 38,000 gpm. Rates up to 43,000 gpm were achieved.

The integrated sea water systems of Works and Power Station, whilst securing sea water at temperatures higher than those in St. Ives Bay, suffered from the demerit of shortage of supply at times for reasons entirely outside works control. The continuity in rate of supply from the station is governed by contingencies which are part of their normal day to day operations but which nevertheless adversely affect bromine production. Tides were only a minor cause of fluctuations in flow. It was always possible to pump more water on the higher tides because of the higher levels in Carnsew.

The regular pattern of generation at the station, where cooling water demand is proportional to generating load, would have meant a reduction in bromine production between 2200hrs and 0800hrs. Arrangements were made for additional pumping over and above station demands during the periods of low generation. Pumping was via a stand-by condenser or through a 40in diameter by-pass main. The additional water, charged for under an agreed formula, resulted in a drop in the temperature of the water received and this had quite a severe impact on the extraction efficiencies during the colder winter months.

The outflow of water from the Works presented a different range of problems. These were due more to erosion than to the chemical composition of the effluent sea water. The high flow rates through the culvert and adverse hydraulics over the original outfall were major factors. Pointing damage to the brickwork was however due to the slightly acidic nature of the effluent. Problems in the harbour were too readily attributed to the chemical nature of the effluent but with the massive dilution that took place there must have been other contributory factors.

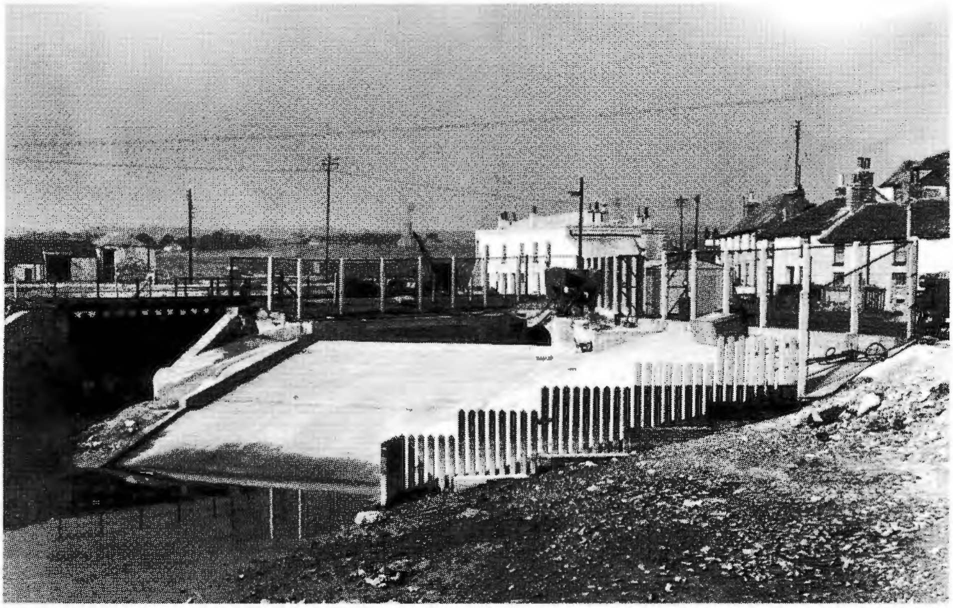
The culvert was initially examined in 1942 after 2 years operation and found to be in good condition. Late in 1945 leakage was believed to be taking place through the brickwork at the Copperhouse end. A thorough survey in Jan 1946 found that mortar had been removed from between the bricks, some bricks had fallen out and inward leakage was taking place. Repair work to culvert and outfall was carried out immediately. The culvert was opened up a year later at that point where the major leakage had been observed for a detailed examination. It was found that there had been a gross departure from the specified mix of concrete that surrounded the brickwork. It was clear that deterioration was largely due to poor workmanship during construction. The greatest attack on the brickwork pointing was at the wind and water line but it was not excessive. There were some patches of bad pointing. The culvert was subsequently routinely inspected and repaired, as required, at every annual shutdown.

From the outset the outfall apron gave trouble. The first repairs were carried out in 1941, when the concrete face blocks were replaced by slag blocks from a nearby derelict cottage. In 1948 the hydraulics of the outfall were studied using a model with a view to making improvements, particularly to the toe. Application of asphalt to the apron was tried on a number of occasions but the adhesion between the surface and the asphalt remained the problem. There was also leakage from the block wall of the sluiceway immediately below the outfall. This undoubtedly arose from the sea water passing through the bed of the outfall. With the progress of time, improved repair techniques and developments in materials of construction the leakage gradually lessened until by 1948 there was little evidence of any. The photographs of the original outfall and the pristine new outfall show very graphically the nature of the problems.

Harvey & Co were always concerned about the effect of the operations on sluiceways and harbour. Discussions were always amicable and a great deal of goodwill existed. ICI and subsequently Associated always investigated their complaints very thoroughly. During 1945 damage to the wall of North Quay was under discussion. Harvey & Co claimed that water of the acidity found at the harbour wall could damage the mortar in the blocks and they dismissed ICI claims that the works effluent was rapidly neutralised by the abundance of normal sea water with the presence of carbonates. It did however transpire that during the construction phase when there was a concern about the impact of blasting, the Consulting Engineer engaged by Harveys reported, "Here the major part of the joints of the masonry near the foot of the wall are open and water issues freely from them when uncovered by the tide. Apparently this is fresh water from a land spring. This condition appears to be old established........ Notwithstanding the open joints, the wall gives no indication of instability."

The Outfall circa 1950 in full spate and undergoing annual repair

The portion of the wall under discussion in 1945 was at the very same point referred to in the 1939 report. A further interesting discovery was that the composition of the mortar used in the building of the harbour wall was "two parts lime and three parts foundry sand, mixed and allowed to stand. Just before using, one part cement was added to four parts of the mixture". This amply demonstrates the local concerns about the proximity of a chemical plant rather than with the limitations of an ageing construction.



The new Apron.
A new apron was constructed in 1955. Note the change in direction of flow, relative to the sluiceway.

On 1st January 1948 Associated took over the operation of the Works assuming full responsibility for all the staff. ICI Alkali ceased to have any involvement from that date. However the Works continued to be referred to as "The ICI" in the environs of Hayle for many years to come.

This important change and the decision to expand production facilities in the UK led to a series of further developments at Hayle. Plans to build a Sulphuric Acid Plant were reported in 1948. A conventional Simon Carves "Contact" plant was constructed and commissioned by July 1949. It was built on the NW corner of the site, to the north of the Absorber (Photograph) to the latest design and had a rated capacity of 46 tpd, a minnow by present day standards. The accompanying sulphur store and handling equipment were located and built on the North Quay, sometime between August 1948 and July 1949. Sulphur was delivered in coastal vessels in consignments of 500 tons and transferred into the melters using a mechanical shovel. Latterly vessels of a size suitable to use Hayle became very difficult to charter.

In April 1948 attention was drawn, yet again, to limited capacity of the works sea water pumps and to their inefficient use of power. It was proposed either to fit the existing Drysdale pump with a new impellor or alternatively to install two new pumps. In the event a new impellor was fitted later the same year. In September 1949 the shaft of the redesigned pump shaft failed and had to be replaced by one of the old design. Two Mather & Platt 26in "Lonovane" pumps were installed in a new pumphouse in 1950, each powered by a 600hp motor and delivering 20,000gpm. The decision to build the pump-house was taken sometime in 1947 but the capital expenditure was only sanctioned in February 1948.

A 1950 aerial view showing the post war developments in the north west corner of the site. The Sulphuric Acid plant and new Pumphouse are clearly visible. The Sulphur Store is located on the Quay (centre).

This increased pumping capacity enabled full advantage to be taken of the increased height of packing in the blowing-out tower. ICI (Alkali) put forward proposals to increase the packing at the end of 1941 and again in 1942 but the Air Ministry would not give their approval. There were obviously greater priorities! When Associated purchased the works in 1945 the proposal was revived. A re-appraisal was made in Oct 1946 and the rings installed in the annual shutdown of Sept 1948. The height of packing was increased by 2.5 ft.

All of these developments were changes to the original design to achieve an increase in production capability. In one way or another each section of the plant was made to operate above the design capacity. Bromine sales relieved the bottleneck at the dibromide plant for instance. Overall, the production capacity was increased by 60% over design. In 1953 and 1954 production exceeded 4000 tpa, a remarkable achievement from a young and enthusiastic team.

Change of Process

1830 hours on 26th October 1954 was an historical moment and just as significant as 24th July 1940. It was the precise time that the "Alkali Process" was shut down for the last time. It had operated for 14 years and 96 days, during which time 43,400 tons of bromine has been produced. Associated had decided to licence the alternative 'Acid Process' from Dow, that which was under development at the time of the first contact with Dow in 1938. The same process was used in the sister facility at Amlwch, Anglesey. The plant closed to allow a major conversion to take place before restarting production in 1956.

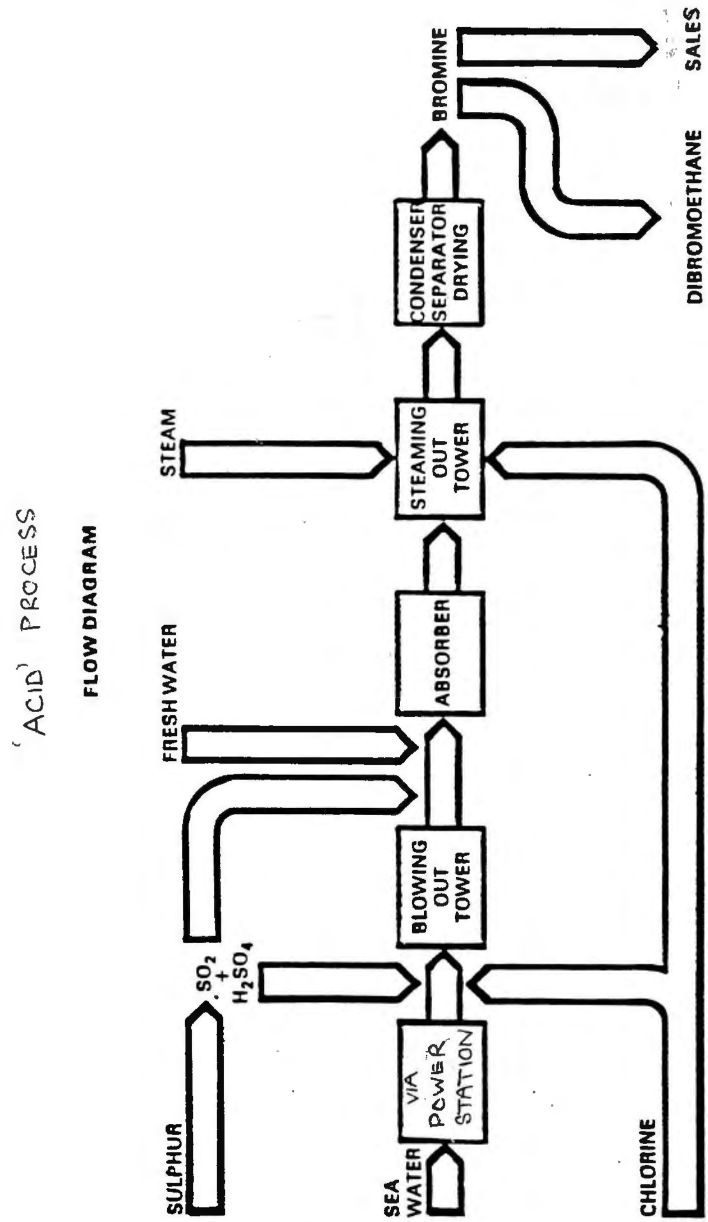
The "Acid process" is more elegant and efficient than the former and a summary is included in this review for the sake of completeness. A more detailed account is contained in the paper "Extraction of bromine from sea water" by H. Fossett (5). Diagrams depict and illustrate the process flows. It will be noted that the treatment of sea water and displacement of bromine is the same in both processes (reactions 1 & 2).

The difference lies principally in the "fixation" of the liberated bromine. The bromine is mixed with gaseous sulphur dioxide and water to produce a mixture of hydrobromic and sulphuric acids (reaction 5) The droplets formed are collected on a fibreglass pad, irrigated with water, to produce an acid liquor containing 10% hydrobromic acid. This is held in storage tanks to await further treatment. An important difference is also the recirculation of the air to the underside of the blowing out towers, a feature which is more environmentally acceptable.

The release of bromine from the acid liquor utilises the original steaming out towers but different chemistry. Chlorine is injected into the liquor in the upper of the two towers, converting the hydrobromic acid to bromine and hydrochloric acid (reaction 6). The

After conversion. View from the East taken in 1956 after conversion to the acid process. Note the new recirculation ducting.

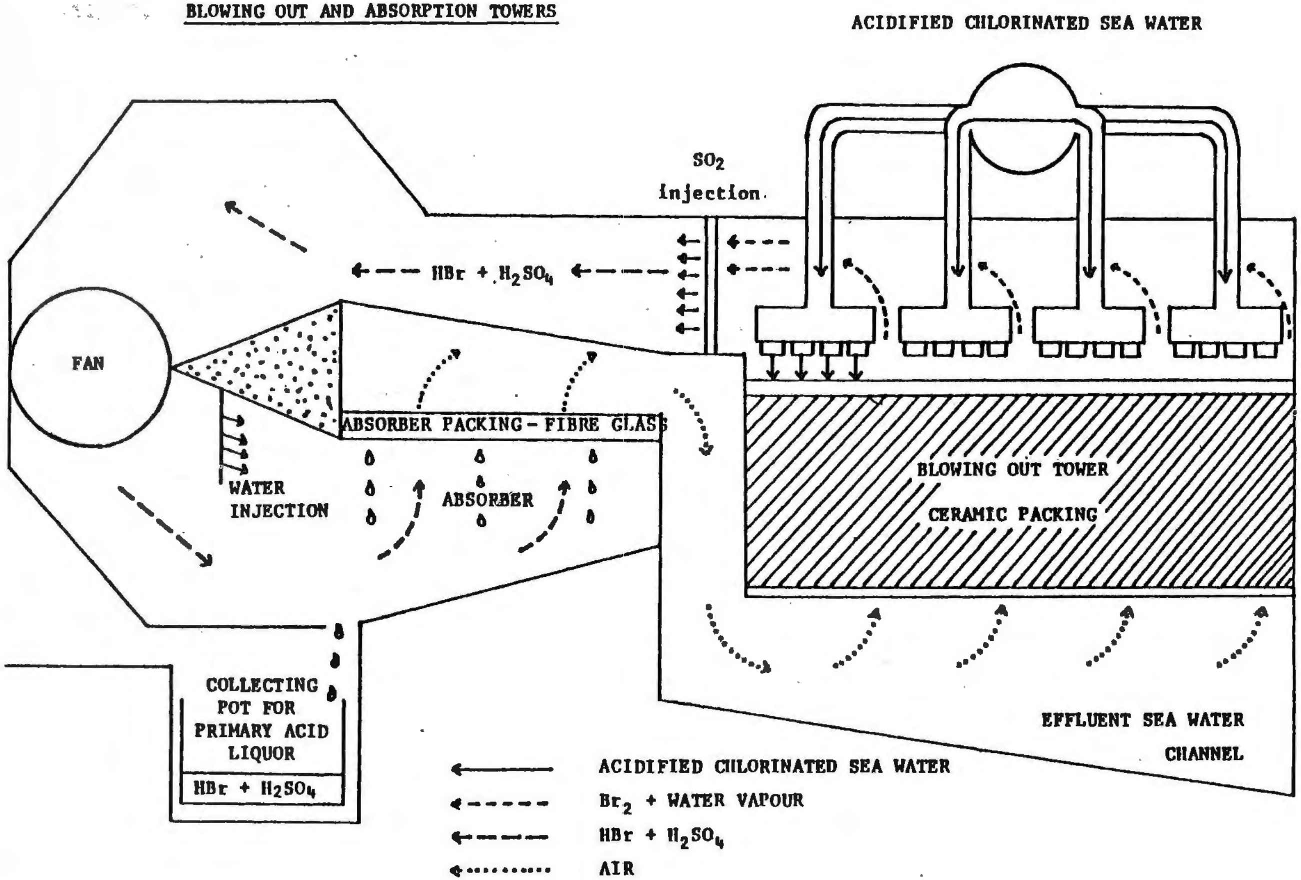
bromine is again separated by steam distillation and condensation while the effluent acids (sulphuric and hydrochloric) are recycled to acidify the incoming sea water. The original plant was modified to accommodate the new process and particularly the absorber. The accompanying photograph shows the compactness of the modified plant and the neat manner in which the recirculation of air is achieved. The capacity of the new process was 5000 tpa.



Flow Process Chart for the "Acid" Process

'ACID PROCESS'

BLOWING OUT AND ABSORPTION TOWERS



Diagrammatic representation of the principal stages in the acid-extraction process

Progressively throughout the 1960's when the Amlwch plant with its more favourable economics expanded to 30,000 tpa there was a swing away from dibromide production to sales of elemental bromine. Production of dibromide ceased in 1968 but the plant was not dismantled until 1973. All the bromine from that time was placed on the UK and European markets except for limited instances when consignments were shipped to China and India in connection with international loan agreements. The introduction of semi-bulk, lead lined, mild steel containers of 7.5 ton capacity and bulk rail tank cars, holding 15 tons net made a great difference to distribution, transportation and costs. Upon closure in 1973 the good-will of the bromine business was transferred to Dow (UK) at Kings Lynn.

The reasons for closure will be obvious to many observers but they are nevertheless interesting. The variable quality of the sea water and its limited availability was always a constraint on production and ruled out any meaningful expansion at Hayle. The first Works Manager wrote in 1945 that any expansion should be at an alternative site. Over the last ten years of operation in particular the economic benefit of receiving warmer water from the Power Station no longer applied. As generation levels fell, due to the high costs of local generation, there was a corresponding increase in pumping costs to Associated. The uncertain future of the Hayle Power Station generated further uncertainty about bromine production next door! In any event unit power costs were significantly higher in the far South West than in North Wales thus adding to the already adverse economics.

Long supply and distribution lines, high transport tariffs and uncertainty about future railfreight service levels were matters of concern in the 70's as much as in the early 40's. Chlorine was shipped from Ellesmere Port in Cheshire and the nearest customers were in the London area. Amlwch was only 90 miles and an overnight journey from the main manufacturing units in Cheshire.

A leak in the sea water tunnel under the estuary in 1970 and the need for major capital expenditure on an ageing plant triggered a final reappraisal of operations. Inevitably and sadly, an association that started as a strategic response to a war-time emergency was to terminate after 34 years. History had repeated itself. Amlwch with its more efficient modern plant and favourable economics displaced a Cornish enterprise just as it had done when Parys Mountain contributed to the demise of the local copper industry.

People

No record and review of the activities of Associated at Hayle would be complete without a tribute and reference to its workforce. It was a major achievement when in 1940, the plant was commissioned speedily and without undue difficulty, without any assistance from the USA, where a similar plant was already operating. It was a testimony to the thorough training carried out by ICI Alkali and epitomised the "spirit of Hayle".

Detailed records of the number of people employed have been lost over time but it is known that ICI recruited 40 local men in 1940 to supplement the men transferred from Cheshire. Contractors employed 50 men during the site clearance operations whilst John Mowlem employed 250 men at the peak of construction during the first half of 1940. The ICI Site Engineer, J. Cathie, had a team of 5 initially, reinforced at a later date by a further engineer and 2 draughtsmen. The construction team and contractors staff worked up to 80 hours per week as required. No labour problems are recorded. The first contacts with local contractors were made by Brigadier J E T Barbary, Manager of Tucking Mill, an ICI subsidiary at the time.

ICI sent a team of skilled and semi skilled men, in unspecified numbers, from Winnington and Lostock, Cheshire, to assist with the plant installation. The winter of 39/40 was also one of the coldest on record with construction supplies held up on the snow blocked roads over Bodmin Moor for several weeks.

With the exception of the soda ash mixers all the process operators were ICI employees who had trained in and operated the Ammonia Soda process. They were trained in March and April 1940 on Steaming out tower operation using glass models of the actual plant. The ethylene plant operators had been trained on an identical plant at Wallerscote Works. All the process foreman had operated the bromine pilot plant at Lostock and were thoroughly familiar with the Blowing out tower and Absorber operations. By June 1940 all key staff were in post.

The level of commitment to the task in hand is amply illustrated by the Works Manager who, during commissioning, slept on a bed in his office from 24th July until 2nd Aug, averaging only three hours sleep per night. After a brief respite, recovering in a local hospital, he returned on 8th August when he averaged four hours sleep per night until 31st August. This was leadership from the front.

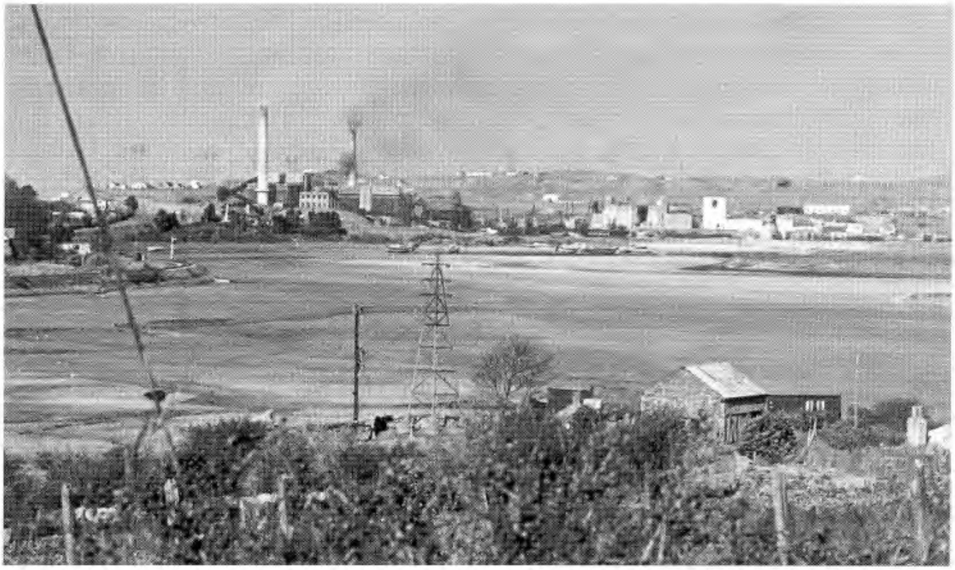
The transfer of employees from the Northwich area of Cheshire was not without its problems, particularly in relation to housing. It is interesting to note that ICI obtained the tenancy of a pool of houses in the Hayle, Lelant and Penzance area, and sublet them to the transferred employees. Many of the key staff stayed with Associated and subsequently transferred to the Amlwch project. They were replaced by local employees. These transfers were important in developing close links between Hayle and the rest of the Company. The effects of geographical isolation were overcome in many and varied ways.

During 1943, when there was a shortage of men, a number of women were recruited for some operations. In the post war period plant work was a male preserve. The pattern of local recruitment during the war years, for routine plant operation, is not recorded. It is estimated that numbers peaked at 125 but by 1965 they had fallen to 85. Labour turnover was virtually zero and the limited vacancies usually involved only secretarial staff. At closure the vast majority had exceeded 20 years continuous service whilst many had achieved 30 years.

Because of its location relative to the rest of the company Hayle had become a self contained unit. Successive young Works Managers were appointed to gain experience before moving into more senior appointments in the organisation. A total of eight Works Managers served at Hayle, six of whom progressed to Associated's top eschelon. Relationships were without exception warm and cordial and because of the underlying company ethos, quite unique.

Once recruited the local employees stayed for the rest of their working lives. This stability and a careful team building produced a camaraderie, social cohesion and loyalty that was referred to throughout the company as "the spirit of Hayle". It was still evident at the many reunions held in the years after closure. The spirit of independence showed through in their singular lack of enthusiasm for trade unions. Such was the team spirit and culture those services were rarely needed. Resourcefulness, reliability and a refreshing flexibility in applying skills were never over exploited. These attributes overcame an innate conservatism which otherwise would have made essential changes difficult to implement. There were many great characters on the Works. Their humour and exploits have been chronicled in numerous in-house publications over the years, making a unique record.

It was cause for satisfaction that following closure all the employees who were not eligible for retirement pensions and wished to continue in employment were able to find alternative work. This was a testimonial to their skills on North Quay.



Then and Now. Views across the Estuary, looking East in 1955 and 1995

The response to the Torrey Canyon emergency in 1967 typifies the approach to a crisis situation. There was a determination to minimise any loss of production in the short term and avoid any contamination of the plant in the longer term. There was no communal wringing of hands in despair but an enthusiastic response for implementing an internally generated idea for a makeshift boom to be placed across the mouth of the Carnsew sluices. Reinforcing rods, 5 gallon drums and hessian sacking were the available materials of construction. The rods were welded end to end to the required span, drums were secured to the rods and a hessian skirt draped over the drums. This Heath-Robinson device was floated into position and with the assistance of the County Fire Brigade, who played their hoses on the water just ahead of the boom during the incoming tides, Carnsew was kept free of oil when the rest of the Harbour became contaminated. Furthermore, this was the only boom in position in Cornwall during the emergency. A manufactured boom was purchased jointly with the Power Station at a later date. The trials with booms in the Camel Estuary came even later.

REFERENCES

Those who wish to explore this subject further may find the following references of interest.

Penwith Museum, Penzance hold some general information and a series of large aerial photographs of the site which show the plant at the various stages of development highlighted in this article.

(1) 'The Recovery of Bromine from Sea water (s.s. Ethyl)'. C.M.A. Stine Industrial Engineering Chemistry, May 1929, Vol. 21, No. 5, pp 434-42.
(2) 'The Commercial Extraction of bromine from Sea Water'. L.C. Stewart ibid. April 1934, Vol 26, No. 4, pp361-9.
(3) 'Minerals from the Sea'. C.M. Shigley J. Metals, Jan 1951.
(4) 'The Seas Around Us.' Rachael Carson.
(5) 'The Extraction of Bromine from Sea water.' H. Fossett. Chemistry and Industry, 9 Oct 1971, pp.1161-71.

ACKNOWLEDGEMENTS

Thanks are due to:

Associated Octel Company for their support in the preparation of this article, particularly for the permission to use photographs and to have access to old documents.

Former colleagues at Hayle, whose skills and endeavours have contributed to the activities recorded herein.

GLOSSARY

p.p.m. parts per million
g.p.m. gallons per minute.
g.p.h. gallons per hour.
c.f.m. cubic feet per minute.
h.p. horse power.